

An exploratory study of dislocation relaxation in polycrystalline olivine

by

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"For Wang Linghui, who holds a special place in my heart."

Declaration

The work in this thesis is my own unless otherwise acknowledged. No part of this thesis has been submitted to any other University except ANU.

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Abstract

Motivated by theoretical considerations of damped vibrations of dislocations and the need to better constrain global attenuation models, a laboratory study was undertaken of the attenuation of seismic waves and associated shear modulus dispersion in upper mantle olivine, $(\text{Mg}_{0.9}, \text{Fe}_{0.1})_2\text{SiO}_4$. Polycrystalline olivine specimens were synthesised from laboratory (solution-gelation) and natural (San Carlos) precursors, hot-pressed and pre-deformed in the laboratory under high temperature ($\leq 1300^\circ\text{C}$) and pressure (300 MPa). Compressive deformation experiments were performed for differential stresses up to 290 MPa and 22% strain in the dislocation creep regime of olivine. Torsional deformation experiments of the same materials under similar conditions were performed up to maximum shear stresses of 195 MPa and maximum shear strains of 0.8. Examination of the pre-deformed specimens revealed an average grain size of $\sim 4 - 8 \mu\text{m}$ for sol-gel and $\sim 8 - 17 \mu\text{m}$ for San Carlos olivine. The density of dislocations with [100] and [001] Burgers vectors was increased by as much as an order of magnitude compared to hot-pressed specimens. Crystallographic preferred orientations were weak to random in all specimens. Infrared spectroscopy analysis confirmed that most sol-gel material was essentially dry, but the San Carlos specimens contained between 200 and 550 H/ 10^6 Si.

Dislocation recovery experiments on both types of olivine established that a maximum temperature of 1100°C should allow a relatively stable dislocation density to be maintained during prolonged mechanical testing (> 50 hours). In addition, the recovery experiments demonstrated that there was no discernible difference in the rate of dislocation recovery between polycrystalline sol-gel and San Carlos olivine.

Calculations of resolved shear stress and anelastic shear strain for the easy slip system in olivine as a function of grain orientation, indicated that torsional defor-

mation prior to torsional forced-oscillation testing may generate a population of dislocations more favourably oriented to dissipate strain energy than prior compressive deformation. In addition, models for string-vibration and kink migration of dislocation segments predict a significantly enhanced relaxation strength and dependence of dissipation on oscillation period.

Torsional forced-oscillation tests were performed at high temperature and pressure on hot-pressed and pre-deformed specimens at maximum strain amplitudes approximately less than 10^{-5} within the linear regime, yielding measurements of shear modulus and associated dissipation. A comparison of data from the pre-deformed specimens with a model for grain-size sensitive viscoelastic relaxation (Jackson and Faul 2010) demonstrated enhanced dissipation and modulus dispersion attributable to dislocation relaxation. The strength of this dislocation-related relaxation increased monotonically with frequency and temperature and with increasing dislocation density. The torsionally pre-deformed olivine specimens demonstrated generally greater levels of dissipation and modulus dispersion over a range of temperatures.

The observed relationship between dislocation-related dissipation and the orientation of the stress field during prior deformation suggests the possibility of seismological attenuation anisotropy. If dislocation damping is not grain size sensitive, then its operation in coarser-grained upper mantle olivine may contribute significantly to attenuation ($Q^{-1} \sim 10^{-2}$), especially in regions with relatively high levels of prevailing tectonic stress.

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Nomenclature

Notation

a	Spacing between adjacent Peierls valleys (m)
a_{SiO_2}	Silica activity
α, β, γ	Direction cosines ($\cos a, \cos b, \cos c$)
α	Exponent for the distribution of anelastic relaxation times $D(\tau) \sim \tau^{\alpha-1}$
b	Burgers vector (m)
c	Concentration of hydroxyl ions in Fo_{90} (wt. ppm H_2O or $\text{H}/10^6 \text{ Si}$)
γ	Shear strain
$\dot{\gamma}$	Shear strain rate (s^{-1})
d	Grain size; imaging depth for back-scattered electrons or thickness of a doubly polished FTIR section (m)
D	Diffusion coefficient ($\text{m}^2 \text{s}^{-1}$)
δ	Twist angle (rad)
$\dot{\delta}$	Twist rate (rad s^{-1})
Δ	Anelastic relaxation strength
E_B	Activation energy for relaxation times of background dissipation (J mol^{-1})
E_a	Activation energy for dislocation recovery (J mol^{-1})

E_d	Elastic strain energy (J mol^{-1})
E_{up}	Binding energy of dislocation to its pinning points (J mol^{-1})
ϵ	Strain; molar absorption coefficient ($\text{m}^2 \text{mol}^{-1}$ or $\text{L mol}^{-1} \text{cm}^{-1}$)
$\dot{\epsilon}$	Strain rate (s^{-1})
ϵ_{an}	Anelastic shear strain
f_{O_2}	Oxygen fugacity (Pa)
G	Shear modulus (Pa)
G_U	Unrelaxed shear modulus (Pa)
η	Viscosity (Pa s)
$J(t)$	Creep function
J^*	Dynamic compliance
J_1	Real component of the compliance
J_2	Negative imaginary component of the compliance
J_U	Unrelaxed compliance (ϵ/σ)

k	Rate constant for dislocation recovery ($\text{m}^2 \text{s}^{-1}$); Boltzmann constant ($\text{m}^2 \text{kg s}^{-2} \text{K}^{-1}$)
L	Dislocation line length between pinning points (m)
l	Incident intensity of light transmitted
l_0	Transmitted intensity
M	Total torque (Nm)
n	Stress exponent for creep ($\dot{\epsilon} \sim \sigma^n$)
P	Pressure (Pa)
p	Grain size exponent for diffusion creep ($\dot{\epsilon} \sim d^p$)
Q^{-1}	Strain energy dissipation (internal friction, quality factor or attenuation [= $\tan \delta$])
R	Gas constant ($\text{J K}^{-1} \text{mol}^{-1}$); radius of curvature of dislocation bowed out under applied stress (m)
ρ	3-D dislocation density (m^{-2}) unless otherwise specified.
ρ_k^{eq}	Equilibrium density of kinks of given sign per unit length of dislocation line
S_c, S_t	Schmid factor for compression and torsion
σ	Differential stress (Pa)

$\sigma_{12}, \sigma_{12}^r$	Shear stress, shear stress at radial position r (Pa)
t	Time (s)
T	Temperature (given in °C)
T_0	Oscillation period (s)
τ	Relaxation time
$\log_{10} \tau_L$	Lower limit on the distribution of relaxation times
$\log_{10} \tau_H$	Upper limit on the distribution of relaxation times
$\log_{10} \tau_M$	Maxwell relaxation time
$U(u)$	Peierls potential (J)
U_u	Displacement of a dislocation segment (m)
ω	Angular frequency (Hz)

Terminology

BSE	Back-scattered Electrons
BEI	Back-scattered Electron image
CPO	Crystallographic Preferred Orientation
EBSD	Electron Backscatter Diffraction
EBSP	Electron Backscatter Diffraction Pattern
FE-SEM	Field Emission Scanning Electron Microscope
Fo ₉₀	(Mg _{0.9} ,Fe _{0.1}) ₂ SiO ₄ olivine
FTIR	Fourier Transform Infra-Red (Spectroscopy)
MAD	Mean Angular Deviation
MUD	Multiples of Uniform Distribution
OH	Hydroxyl ion
PSZ	Partially Stabilised Zirconia
Sol-gel	Solution-gelation (often referred to olivine synthesised in the laboratory)
TEOS	Tetra-ethyl Orthosilicate
TEM	Transmission Electron Microscope

*Nature does not hurry,
yet everything is accomplished.*

Lao Tzu (600 – 531 BC)

1

General introduction

1.1 Motivation

In the field of seismology, incorporation of knowledge about the ways that composition and defect structures in upper mantle materials affect seismic shear wave attenuation via solid-state relaxation, has been slowly coming. One major reason for this is that global attenuation models are essentially phenomenological in nature and do not need to invoke physical explanations of the observed increase of attenuation in e.g. the low velocity zone in the upper mantle (80 – 220 km). Another reason is the lack of a significant amount of laboratory data, especially at low frequencies of tele-seismic wave propagation, to constrain and calibrate seismological models of the Earth.

Recently, however, several attempts were made to define 1-D attenuation models from constraints of mineral physics and were compared with observed seismic attenuation measurements of the upper mantle (Faul and Jackson, 2005; Cammarano and Romanowicz, 2008; Dalton et al., 2009; Jackson and Faul, 2010). Nonetheless, the parameters that have a direct and indirect influence on shear wave attenuation are numerous (temperature, pressure, grain size, water, dislo-

cations, melt, composition, etc.) and there is still a long way to go before all contributions to viscoelastic relaxation are adequately understood. The parameter of interest in this study is the contribution to attenuation from dislocation damping. Alternative models of viscoelastic relaxation resulting from vibration of a (pinned) dislocation segment treat the dislocation either as a stretched string (Koehler, 1952; Granato and Lucke, 1956) or consider the role of kink migration and nucleation (Seeger, 1956, 1981).

Relevant laboratory data concerning dislocation-related viscoelastic relaxation come from a single pioneering study (Gueguen et al. 1989) which demonstrated dislocation damping in olivine via torsional forced-oscillation measurements at seismic periods ($0.1 - 10^4$ s) on pre-deformed synthetic forsterite crystals. Unfortunately, dissipation data was limited to only three temperatures (1000, 1200 and 1400°C) and the effect of fast dislocation recovery for temperatures $> 1000^\circ\text{C}$ was not further investigated.

This provided the motivation for further exploration of torsional forced oscillation experiments on pre-deformed Fo_{90} olivine materials at high temperature and moderate gas confining pressure using a specialised attenuation apparatus, built at ANU in the early 1980s.

The apparatus has been continuously modified and improved over time. It was initially capable of measuring the shear mode anelasticity of rocks at low strain amplitude under a gas confining pressure (300 MPa) and room temperature (Jackson et al., 1984; Jackson and Paterson, 1987). A furnace capable of reaching high temperatures (up to 1300°C) was installed by 1992 (Jackson et al., 1992; Jackson and Paterson, 1993). This significant improvement permitted for the first time at ANU shear wave dispersion and attenuation measurements at high temperature on minerals such as olivine. A subsequent modification included the addition of an argon pore pressure control used to determine the effective moduli of fluid-saturated rocks (Lu and Jackson, 1998). Recently (2009) the apparatus was further improved with newly installed and more powerful electromagnetic

drivers and more widely spaced transducer plates. The new drivers provide the generation of larger torque amplitudes enabling the exploration of possible non-linearity for strains above 10^{-5} .

For this project, the possibility of using natural olivine single crystals was considered (Appendix A) but rejected on several grounds including the practical difficulties of accommodating (in an assembly stressed via alumina pistons) the anisotropic thermal expansion of a suitably oriented single crystal, cracking during thermal cycling and partial melting associated with the presence of included secondary phases. Accordingly, high-temperature attenuation experiments were instead carried out on fine-grained hot-pressed and pre-deformed olivine polycrystals rather than single crystals to minimise cracking during thermal cycling and more realistically approach upper mantle conditions.

Based on the successes from earlier research involving synthesis of olivine-rich aggregates, polycrystalline olivine from two different sources were used; solution-gelation (sol-gel) olivine, fully prepared and synthesised in the laboratory and San Carlos olivine, derived from a natural precursor. Both materials of 'realistic' Fo₉₀ composition were relatively fine-grained ($\sim 10\ \mu\text{m}$) and essentially melt-free. They were exposed to a chemical environment similar to which this material is inferred to be in the upper mantle of the Earth, by controlling the oxygen fugacity (f_{O_2}), silica activity (a_{SiO_4}) and very low water (OH) content. In anticipation of attenuation experiments, selected specimens were pre-deformed either in compression or torsion to generate desired populations of dislocations. Each such specimen was then subjected to micro-strain forced-oscillation to measure its dissipation and shear modulus dispersion – twin manifestations of viscoelastic relaxation.

Most notably, this research documents a full characterisation of each specimen, after every step, for reliable grain size, microstructures including defects, crystallographic preferred orientation, Infrared spectroscopy for OH determination and plastic flow behaviour after pre-deformation. With this information,

interpretations of the viscoelastic relaxation of dislocations from torsional forced-oscillations can be more convincingly made.

1.2 Thesis structure

The bulk of the research in Chapters 3, 4, 5 is presented in scientific manuscript format with abstracts given at the beginning and interim conclusions at the end. The references are combined at the end of the thesis after the Appendix chapter. Some repetition was unavoidable, especially between the general procedures chapter 2 and each subsequent chapter. Please note that during the writing of the thesis, two parts have already been submitted for publication. These are Chapter 3 and Appendix E.

The chapters comprise the following:

- Chapter 2 outlines the general procedures used to synthesise the materials, how they were pre-deformed in compression and torsion and how they were mechanically tested in the attenuation apparatus. Procedures for microstructural analysis (dislocation decoration and imaging, electron backscatter diffraction, OH content analysis, density measurements, etc.) are also given.
- Chapter 3 explores the dislocation recovery kinetics in compressively pre-deformed sol-gel and San Carlos olivine for a range of temperatures (1100 – 1500°C) and annealing durations (10 min to 50 hours) to constrain the maximum temperature feasible for attenuation experiments on pre-deformed materials with stable dislocation microstructures.
- Chapter 4 presents the results from hot-pressing and deformation experiments of sol-gel and San Carlos olivine. Electron backscatter diffraction data on e.g. crystallographic preferred orientations and microstructural data are given of the pre-deformed specimens before and after attenuation experiments. In addition, theoretical considerations concerning the resolved

shear stress and resulting anelastic shear strain for different geometries of pre-deformed single crystal and for polycrystalline olivine are presented. This provided the motivation for carrying out forced oscillation experiments on both compressively and torsionally pre-deformed specimens.

- Chapter 5 presents the results from all the attenuation experiments. Two hot-pressed specimens with measured low dislocation density were intended to provide a baseline against which data for the pre-deformed specimens of higher dislocation densities should be compared. Linearity of dislocation-controlled mechanical behaviour was explored to higher oscillating strain amplitudes and complementary torsional microcreep test results are given to assess the extent to which the viscoelastic relaxation is recoverable (i.e. anelastic). The chapter concludes with implications for dislocation damping in the upper mantle.
- Chapter 6 provides a summary of key results and future studies.
- Appendices 1 – 11 are a collection of related experimental projects, theoretical considerations and additional data presented for reference.

*When it is obvious
that the goals cannot be reached,
don't adjust the goals,
adjust the steps.*

Confucius (551 – 497 BC)

2

General procedures

2.1 Olivine sample preparation

2.1.1 Olivine synthesis in the laboratory

Synthetic olivine samples were systematically prepared in the laboratory from natural or sol-gel derived powders. Olivine aggregates were synthesised with consistent grain size, porosity and orthopyroxene content. Without contamination, the sol-gel olivine starting material would only have contained none to very small trace amounts of impurity atoms (e.g. TiO, CaO, MnO, NiO, etc) and water content, close to or below the detection limits of the ion microprobe and infra-red spectrometer respectively (Jackson et al., 2002; Faul et al., 2004).

2.1.1.1 Sol-gel olivine

The procedure for creating synthetic iron-bearing olivine aggregates was first developed and applied by Cooper and Kohlstedt (1984) and subsequently refined independently at Utrecht University (McDonnell et al., 2002; Kellermann Slotemaker and De Bresser, 2006) and at the Australian National University (ANU) (Jackson

et al., 2002; Faul and Jackson, 2007). The procedure, outlined here, is the one used at ANU.

Prior calibration of the Mg and Fe nitrates to oxides was necessary to determine the correct conversion factor. This was done as follows. The Mg and Fe nitrates were each heated in three steps to remove all water and other volatiles. First, a small amount of each (~ 2 g) was slowly heated separately on a hotplate at 150°C in a Pt crucible of known mass to avoid an instantaneous reaction. Then each batch of nitrate was decomposed by heating over a Bunsen burner until the smoke cleared. The last step involved the heating in a box furnace at 1000°C for several hours to make sure all water and nitrates were lost. To obtain the conversion factor for MgO, the remaining material was weighed and the mass of the Mg nitrate in the crucible was divided by the mass of the remaining product in the crucible. Likewise, the same was done for Fe_2O_3 . For TEOS (Tetra-ethyl Orthosilicate) the calibration was done by forming a gel during a set of firing stages and the final result was weighed. However, since the procedure is more complicated, the actual difference in molecular weight was calculated and used instead.

Sol-gel olivine was then synthesised by adding the right amounts of Mg, Fe nitrates (see Table 2.1) into a glass beaker containing 240 ml of ethanol. The use of an ultrasonic bath and magnetic stirrer ensured complete dissolution of the components. Upon dissolution, TEOS was added and the mixture stirred continuously for 16 hours. To initiate gelation, the pH was lowered by adding 2 ml of nitric acid. The hotplate was set to low heat while the gel was stirred into a solid, dry mass for the next 16 to 20 hours. The magnetic stirring lug was removed to allow the gel to dry at a moderate temperature of 200°C for 24 hours on the hotplate. The material was subsequently removed from the glass beaker in batches and transferred into a platinum crucible. In moderate amounts, the gel was heated over a Bunsen burner to release nitric acid and water. The release of these chemicals was demonstrated by the release of brown smoke. The platinum

crucible containing the sol-gel was left in a box furnace for 1 hour at 550°C. Care was taken during these drying steps that no Mg and Fe enriched residues were left attached to the crucible, keeping the final gel of homogeneous and expected consistency.

Silica under-saturated sol-gel An attempt to synthesise silica-undersaturated olivine polycrystals was also made. The followed sol-gel procedures were the same as described earlier, however with a modified recipe given in Table 2.2. After the last firing step at 1400°C and 50% CO / 50% CO₂ gas mix the pellets were visually inspected to contain tiny brown spots inferred to be magnesiowüstite (MgO). Repeated pellitising, firing under these conditions and re-grinding improved the homogeneity and mixing of final product substantially. Unfortunately, after many failed attempts of hot-pressing the material (including one failed attempt to deform it) ended the investigation of the influence of silica activity.

Table 2.1: Sol-gel recipe for obtaining a silica over-saturated Fo₉₀ rock.

	Mass (g)	Conversion factor	Total mass (g) [†]		
MgO	24.411	6.360	155.25	Mg(NO ₃) ₂ · 6H ₂ O	99%
Fe ₂ O ₃	5.373	5.080	27.30	Fe(NO ₃) ₃ · 9H ₂ O	98+%
SiO ₂	20.216	3.467	70.2 [‡]	Si(OC ₂ H ₅) ₄	98%

[†] Molecular weight as given on the bottle[‡] 0.12 g excess to ensure Si saturationTable 2.2: Sol-gel recipe for obtaining a silica under-saturated Fo₉₀ rock.

	Mass (g)	Conversion factor	Total mass (g) [†]		
MgO	12.3711	6.360	78.680	Mg(NO ₃) ₂ · 6H ₂ O	99+%
Fe ₂ O ₃	2.7229	5.080	13.832	Fe(NO ₃) ₃ · 9H ₂ O	99+%
SiO ₂	9.9060	3.467	34.344	Si(OC ₂ H ₅) ₄	98+%

[†] Molecular weight as given on the bottle

2.1.1.2 San Carlos olivine

San Carlos olivine powders of a fine grain size fraction (10 – 38 μm) have been previously prepared by Tan et al. (2001). In brief, selected olivine megacrysts of near gem quality from the San Carlos locality have been carefully hand picked, avoiding any inclusions. Via a process of crushing and sieving for smaller grain size factions, the resulting powder was purified via optical inspection, heavy liquid and magnetic separation. The final powder was further sorted for grain size by sedimentation from suspension in ethanol. An olivine fraction of $\sim 10 - 38 \mu\text{m}$ grain size was recovered from the sediment after one day with $< 10 \mu\text{m}$ olivine material still in suspension.

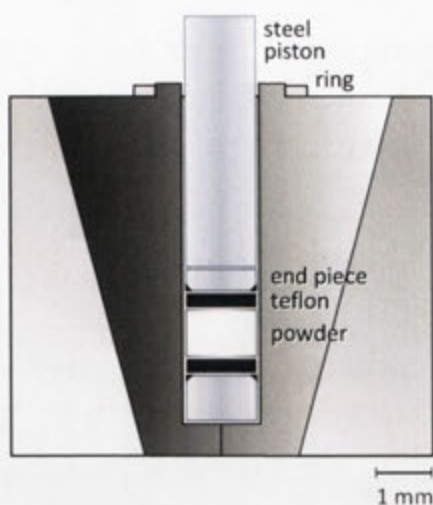


Fig. 2.1: Schematic of the jig for cold pressing olivine powder into solid pellets

2.1.2 Cold pressing and preliminary heat treatment

Using an agate mortar and pestle, the relatively dry sol-gel and San Carlos powders were ground to a fine powder and pelletised using a steel cold press jig. The pellets were exposed in a controlled atmosphere furnace at 50%/50% mix of CO/CO_2 at 875°C for 10 to 12 hours to initiate the reaction to form olivine. Typically, the temperature was ramped up at $5^\circ\text{C}/\text{min}$ and down at $4^\circ\text{C}/\text{min}$ before taking out the pellets at 600°C . The pellets were subsequently reground using the mortar and pestle and re-pelletised. Using the same gas mix, the material was fired at 1400°C for a duration of 16 hours to achieve crystallisation of fully anhydrous Fo_{90} . The resulting sol-gel and San Carlos pellets were white and hard when reground. A normal batch of olivine powder allowed roughly 15 ~ 3.1 g pellets to be cold pressed (see Fig. 2.1). A differential pressure of about 10 MPa (~ 1500 lb/in²) was applied on ~ 3.1 g of olivine powder in the jig for 2 minutes. The pellets were fired at 1400°C overnight (16 hour) for a subsequent hot pressing experiment under 300 MPa confining pressure. Generally, 15 pellets allowed preparation of three ~ 30 mm samples for deformation and attenuation experiments. The pellets were stored in an oven at 110°C pending hot-pressing.

2.1.3 Hot-isostatic pressing experiments

A stack of five cold-pressed and pre-fired pellets were wrapped in foil with two end-discs, all of $\text{Ni}_{70}\text{-Fe}_{30}$ composition and 0.07 mm thickness and pushed inside a mild steel jacket. The sample was sandwiched between hollow LuxaloxTM alumina pistons as shown in Fig. 2.2. The whole specimen assembly was then inserted into a thick-walled pressure vessel equipped with a furnace capable of reaching temperatures over 1300°C. The top steel piston used for each experiment possessed a similar bore hole. A Type-R Pt/Pt,13%Rh thermocouple was inserted into the alumina upper piston to measure the temperature immediately adjacent to the top of the specimen and to provide servo-control of the furnace by a Eurotherm controller.

Every new furnace was calibrated at the desired temperatures (and pressures) before or after a deformation experiment. A calibration was performed using a hollow LucaloxTM alumina assembly so that a long thermocouple could be traversed through the hot-zone of the furnace. The furnace has three separate windings of molybdenum wire and the power output was controlled in each winding to obtain a flat temperature profile in the hot-zone. After a successful calibration, the new furnace settings, voltage and current readings and temperature profiles were recorded for the subsequent experiment.

The hot-pressing experiment was commenced by allowing Ar gas to enter the pressure vessel at bottle pressure. It was standard procedure to purge the air from the system twice before ramping up in pressure by using the first and second stage gas booster pumps (operating in series via compressed air) and an oil intensifier. The target gas confining pressure was 300 MPa for all runs. The last 100 MPa increase in pressure was reached via the thermal pressure of the gas (from suppressed thermal expansion in a restricted volume) during furnace heating to 1300°C. A typical hot-pressing experiment lasted for 24 hours, during which the porosity of the olivine aggregate was reduced to < 1%. The furnace was heated and cooled at constant rates of 600°C/hour and 300°C/hour, respectively.

The pressure was dropped incrementally down to 50 MPa by the time the furnace had cooled down to 700°C.

2.1.4 Specimen recovery

2.1.4.1 Acid dissolution and cutting

On most occasions, after each hot-press and deformation run, the specimen was recovered from its mild steel jacket so that the loading pistons could be reused. Generally, to preserve the maximum length of the olivine samples, the specimen assemblies were dissolved in a diluted solution of one part nitric acid (70%) and three parts water. After the metal jacket has dissolved away, the ceramic alumina pistons were cleaned ultrasonically first in water, then ethanol and finally acetone. They were oven dried in preparation for the next experiment. It was possible the specimen may have been affected by acid dissolution, sometimes indicated by mild discolouration at its cracked ends. In some cases, the most cracked and altered ends were ground off. The specimens were generally precision ground to the standard 11.5 mm diameter so any adhering flakes of metal after acid dissolution were cleanly removed. All samples were stored inside a ~150°C oven and re-fired under standard conditions (1400°C and 50% CO/ 50% CO₂) before the next experiment.

The two samples (T0434 and T0436) torsionally pre-deformed at the University of Minnesota were not subjected to acid dissolution but instead cut from the jacketed specimen assembly for easy and safe transportation back to ANU. The samples appeared to contain significantly cracked ends. To avoid the risk of losing more material and to preserve their lengths for subsequent attenuation experiments, their jackets were instead precision-ground to an average thickness of 0.25 mm inclusive of nickel-iron foil of 0.07 mm thickness. One hot-pressed San Carlos specimen (H6695) received the same treatment twice – on recovery from attenuation experiments during which the furnace had failed. Two furnaces had failed in succession and melted a portion of the jacket close to the specimen. On

both occasions the microstructure was checked for any anomalous grain growth or alterations. A substantial increase in porosity was found but no longer after subsequent successful forced-oscillation testing.

For an overview of the number and rheology of all the specimens, view Table H.1 in Appendix H.1 and Tables 4.1 and 4.2 in Chapter 4.

2.1.4.2 Sectioning

Each specimen was transversely sectioned at the upper end. Routinely, a 1-2 mm slice was sawed off using a diamond wafering blade and cut into quadrants for a full microstructural analysis. These included, but were not limited to the following. The first quadrant was placed in a epoxy block and polished for oxidative-decoration of dislocations (see section 2.4). The second quadrant was prepared for Infrared Spectroscopy (IR) (see section 2.7). A third quadrant was prepared as a thin section ($\sim 30\text{ }\mu\text{m}$ thick) for a first order qualitative observation of the microstructures, although in some cases an initial estimate of the grain size was obtained from it too. Finally, for some deformed specimens, the fourth quadrant was used for Electron Backscatter Diffraction (EBSD) (see section 2.6.2). This slab was sawn, ground and polished to allow examination of sections normal to the compressive axis, or in the tangential plane of maximum shear (see Fig. 2.6a), for the compressively and torsionally deformed specimens, respectively.

2.2 Rock deformation experiments

2.2.1 General overview

Compressive deformation of precision-ground cylindrical rock specimens was performed in the same triaxial testing machine in which the hot-pressing experiments were carried out. The deformation apparatus is a custom designed prototype built according to specifications as described in Paterson (1990). Its design allows gas pressures up to 700 MPa to be safely reached. However beyond 300 MPa the

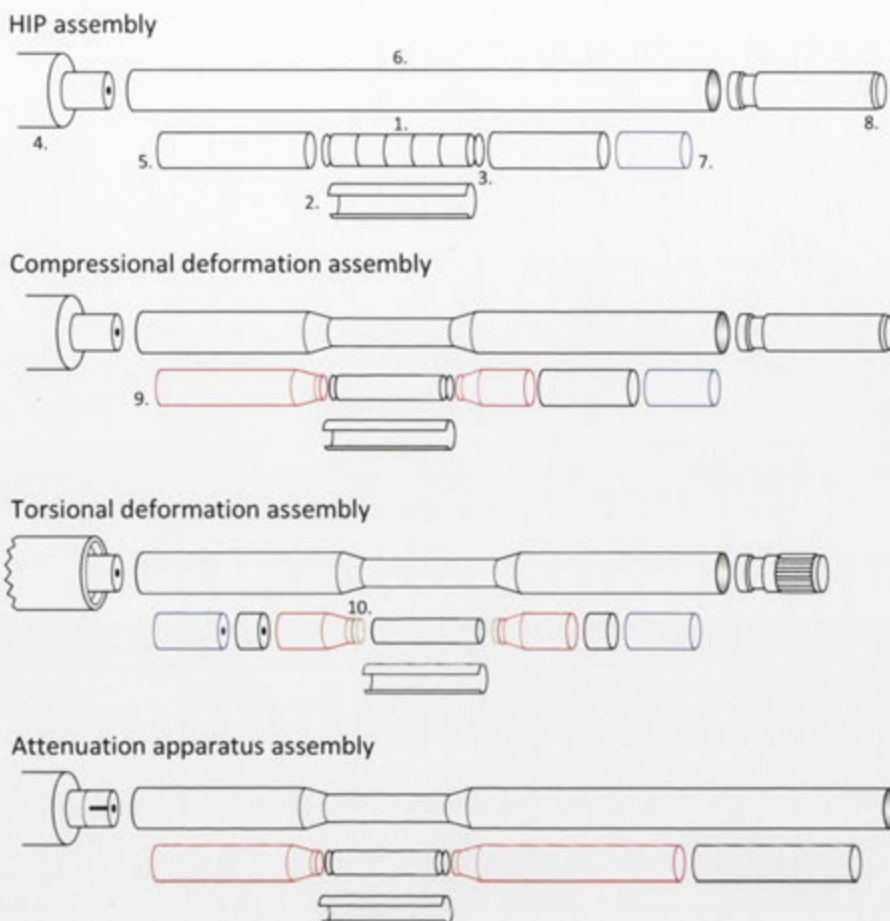


Fig. 2.2: The number of specimen assemblies used in this study. Most assemblies are comprised of the following: 1. specimen, 2. Ni₇₀Fe₃₀ foil, 3. Ni₇₀Fe₃₀ discs, 4. top steel piston, 5. alumina loading pistons, 6. mild steel jacket, 7. PSZ, 8. steel end piston. The deformation and attenuation assemblies have tapered alumina loading pistons instead (9) and the torsional deformation assembly has additional porous alumina discs (10)

polyurethane o-rings in the system will normally give way to a significant leak rate. The apparatus allowed for deformation in compression but not in torsion and was operated by a computer interface loaded with LabView Software. The software permitted manual and automatic control of the advancing actuator supplying a desired force against the specimen assembly.

2.2.2 Compressional deformation

2.2.2.1 Overview

Typically, compression experiments on olivine were carried out between 1200°C and 1300°C up to a differential stress of ~ 300 MPa. High temperature and large differential stresses provide access to the rheological regime in which defects such as vacancies and dislocations are mobile in the crystal grains, thus facilitating plastic deformation. The differential stress was generally not allowed to exceed the confining pressure – in order to minimize the risk of brittle behaviour as described by the Mohr-Coulomb criterion. However, the exact point at which brittle behaviour overtakes ductile behaviour is not well constrained. As a rule of thumb, the length of a precision ground specimen was more than twice its diameter to maintain a relatively uniform state of stress in the specimen – minimizing the stress ‘shadow’ that tends to result from the lateral constraint imposed by frictional stresses at the ends of the specimen (Paterson and Wong, 2005). In this study sol-gel and San Carlos olivine specimens of around 30 mm in length were deformed between 2% and 22% strain. More strain was not attempted to preserve the length of the specimens which were needed for subsequent attenuation.

The specimen assembly is similar to a hot-isostatic pressing assembly but with loading pistons that taper towards a diameter of 11.5 mm or in some cases 10 mm depending on the diameter of the precision ground specimen (see Fig. 2.2). The specimen was wrapped and sandwiched between Ni₇₀-Fe₃₀ foils for oxygen fugacity control. A partially stabilised zirconia (PSZ) piston was loaded at the bottom end of the assembly. Its unique properties (high thermal shock resistivity and resistance to cracking) ensured the initial impact of the advancing piston did not cause any damage.

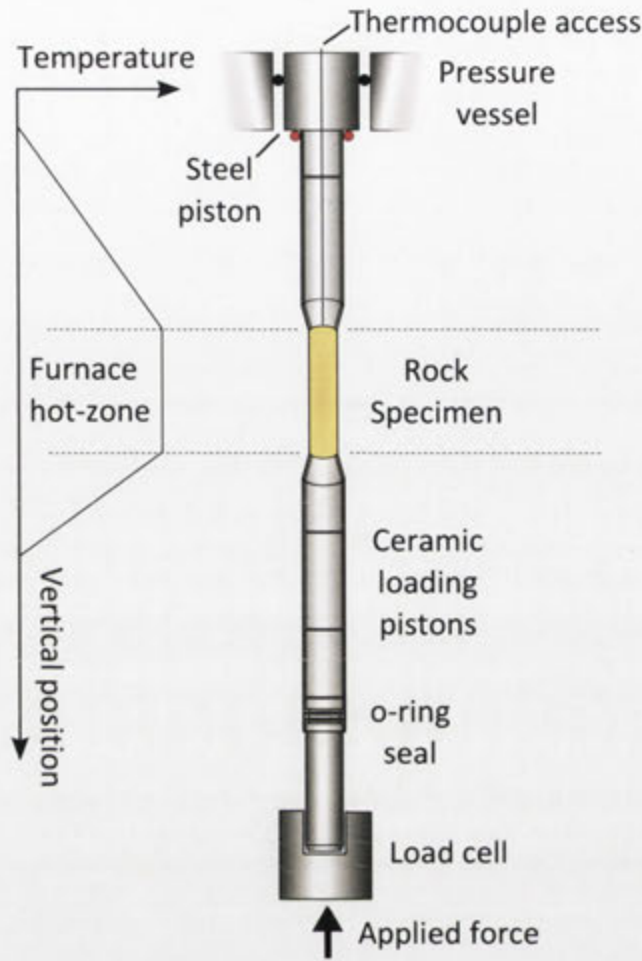


Fig. 2.3: Overview of compressional deformation for a specimen assembly loaded into a pressure vessel, subjected to an applied force from below.

2.2.2.2 Procedures

The specimen assembly was loaded into the pressure vessel to engage with the o-ring seal between the upper steel piston and the furnace plug that forms part of the ‘wall’ of the pressure vessel (see Fig. 2.3). Purging, pressurization and heating of the furnace was carried out exactly as described in section 2.1.3. Once the desired temperature and pressure conditions were reached for the olivine specimen, the actuator was slowly advanced manually in order to gently establish contact between the load cell and the lower steel loading piston. Such contact was indicated by the first appearance of a non-zero signal from the load cell. The

touch point was obtained once the internal load cell registered a non-zero signal. Before compressive deformation began, safety limit switches above and below the moving arm of the loading system were checked (and repositioned) to allow only a few millimeters of upward displacement. During the course of the experiment the upper safety limit switch was re-checked that it was not touching, as a touch would halt the experiment.

The compressive deformation experiments were performed under computer control in order to approximate creep (constant stress) experiments. This was done by servo-control of the axial load involving comparison of the load cell output with the instantaneous value of the prescribed load and adjustment of the actuator drive to minimise the difference (i. e. error signal). The LabView software on the adjacent computer registered the output voltages in the load cell and actuator demand and converted the value into a force via a load cell calibration factor of 72.6 kN/V, previously established from a deformation experiment on a spring of known stiffness (a constant independently measured). A load of 8 kN, for example, corresponded to an actuator demand of ~ 0.1 V. Additionally, the operating software recorded the position of the advancing piston, temperature and pressure during the course of the experiment.

A deformation experiment was set up by pre-programming a series of ramps and dwells of progressively increasing force using the same software. The desired compressive stress was calculated based on the applied force on the initial cross-sectional area of the specimen. The first set of ramps for relatively small stresses paired up with long dwells up to 6000 seconds to obtain rheology data at relatively low strain rates in olivine. Subsequent increments towards higher stress were accompanied by dwells of shorter durations. Depending on the strain rate in the dislocation creep regime, the shortest dwell at the highest stress step was only 50 seconds during which time strains between 1 and 2% were obtained. Generally, for each step close to 2% strain was desired to ensure steady-state creep and to avoid complications of strain hardening.

The experiments were terminated once the pre-programmed phase of the test was completed. The system kept a constant load on the specimen until the actuator was manually withdrawn. Simultaneously, the furnace was switched off to quench the sample quickly and preserve the dislocation microstructures. On one occasion (for sample D6646), the furnace was allowed to cool at a rate of 600°C/hour with the actuator still touching the specimen assembly until the temperature reached 1000°C and the actuator was lowered. This was an attempt to allow the sample to continue to deform during cooling to investigate whether a higher dislocation density could be obtained.

2.2.2.3 Data analysis

The displacement versus time data from the experiments, recorded by the Lab-view software, were processed according to the following procedures. A preliminary overview of the stress, strain and strain rate data can be estimated from best fits to the dwell curves of displacement versus time. The strain in the sample for each dwell was calculated as the change in displacement over the original length of the sample. The observed strain rate can be estimated as

$$d\epsilon/dt = (1/\lambda)dz/dt \quad (2.2.1)$$

where λ is the initial length of the specimen and dz/dt is the instantaneous value of the slope of the $z(t)$ trajectory. However, this simplified analysis did not take into account several contributing factors that affect the deformation of the olivine specimens. These corrections include apparatus distortion during deformation due to shortening of the loading column by ~ 0.015 mm/kN; a correction for the calculated stress to subtract the fraction of the load supported by the mild steel jacket surrounding the specimen; and a correction of the stress taking into account an increasing cross-sectional area of the specimen during shortening (assuming constant volume).

The data was processed as follows. Firstly the raw displacement (mm) ver-

sus time (s) and load (kN) versus time (s) data were plotted. The trends were carefully inspected for linear displacement-time segments between successive load-versus-time ramps of increasing stress. Any curvature in the slopes, indicating strain hardening, was avoided. The carefully picked time segments for each dwell were processed with the applied corrections (e.g thermal expansion, apparatus distortion, specimen diameter, etc) to produce the strain, strain rate and stress data. The result was plotted as log (strain-rate) versus log (stress). An additional post-processing correction would normally be applied to take into account the evolution of the grain size during deformation. However, it was unlikely that any further grain growth occurred in the dislocation creep regime of olivine due to the relatively short duration of the load dwells (200 – 50 seconds) for the relatively high strain rates ($\sim 10^{-5} - 10^{-4} \text{ s}^{-1}$).

2.2.3 Torsional deformation

2.2.3.1 Overview

Torsional deformation experiments were carried out at the University of Minnesota, USA on a Paterson-type deformation apparatus capable of both compressive and torsional deformation at high temperature and Ar confining gas pressure (Paterson and Olgaard, 2000). The procedures are largely similar to a compression experiment as described in the previous section. A geometrically compatible Paterson furnace from ANU was installed and used for testing at high temperatures (up to 1350°C). Before the experiments began and the specimen assemblies were prepared, the furnace was re-calibrated for flat temperature profiles at 1200, 1250, 1300 and 1350°C within measurement uncertainty of less than $\pm 1^\circ\text{C}$.

The schematic of the specimen assembly for the torsion apparatus is shown in Fig. 2.2. The specimen assembly was composed of LucaloxTM alumina loading pistons tapered at the inner end to match the specimen diameter and two porous alumina discs (20% porosity) to make up the waisted section sandwiching the

sample in between. A PSZ piston was used at each end of the assembly instead of one for the compression assembly, as an extra precaution. The specimen was wrapped in a $\text{Ni}_{70}\text{-Fe}_{30}$ foil for oxygen fugacity control but without any $\text{Ni}_{70}\text{-Fe}_{30}$ foil discs. Preliminary tests using $\text{Ni}_{70}\text{-Fe}_{30}$ and solid alumina discs have shown that significant amount of slip occurred between the olivine polycrystal and foil – alumina piston interfaces thereby leaving the sample undeformed. To overcome this problem porous alumina discs (20% porosity) were employed to provide better grip and minimise the chance of slip occurring. In addition, one sol-gel and one San Carlos olivine sample were ground to a smaller (10 mm) diameter for torsion tests. For a specimen of smaller diameter, a given level of applied torque results in higher (radially variable) shear stresses.

Before the experiment could proceed, it was necessary to consider the desired type – a constant twist rate experiment versus a constant torque experiment. Due to problems experienced with slip on the sample and alumina disc interfaces from preliminary experiments, a constant twist rate experiment was chosen to allow for shear stress (or internal torque) drops at a given constant twist rate. A constant torque experiment keeps the shear stress constant, but cannot be maintained when slip occurs on one of the sample or piston interfaces.

In this study, an attempt was made to deform cylindrical olivine specimens in the dislocation creep regime. While it was desirable to reach a high shear stress to allow multiplication of as many dislocations as possible, the objective was to generate a population of more favourably oriented dislocations in the torsionally pre-deformed specimens for subsequent torsional forced-oscillation experiments. Obtaining a high shear strain was a secondary objective so that a microstructural interpretation of the development of a crystallographic preferred orientation (CPO) in olivine could be made. Acquiring useful rheology data for sol-gel and San Carlos olivine was not considered a major objective. Hence, in this study such data will need to be interpreted with caution. Because the experiments were conducted at constant twist rate and involved some slip at the alumina/specimen

interfaces, it was unlikely that steady-state creep was reached during the course of the two more successful experiments.

2.2.3.2 Procedures

Torsion experiments in this study were performed on long cylindrical hot-pressed and precision-ground sol-gel and San Carlos olivine specimens. The furnace was installed into the pressure vessel and the specimen assembly was inserted into place. The bottom steel piston was fitted with grooves that mated with the torsion actuator. The vessel was closed up and pressurised to 50 MPa. On the front panel, the Eurotherm temperature controller at Minnesota lacked the ability to pre-programme heating and cooling ramps and dwells. Hence the temperature was manually increased incrementally to higher output power values until the desired temperature was reached, while at the same time pressurising to 300 MPa using the Ar intensifier.

The deformation experiment was controlled by Labview software loaded onto the adjacent computer. An initial small and constant twist rate of 0.03 mrad s^{-1} was set in order to avoid the loss of cohesion or friction between the porous alumina disc and sample interfaces. The software recorded the external and internal torque, maximum shear stress, maximum shear strain (at the cylindrical surface – as clarified in section 2.2.3.3), temperature and pressure as a function of time. The shear strain rate was not recorded but was routinely calculated from an automatically updating plot of shear strain versus time. The twist rate was increased by 0.02 mrad s^{-1} increments in order to achieve large enough shear stresses and to produce faster strain rates. While a specimen could have been left to deform at low twist rates to avoid slip, it would not have been deforming in the dislocation creep regime, required to obtain a reasonable dislocation density. As long as the shear stress was increasing, the specimen was inferred to be adequately gripped by the loading pistons.

Each run involved substantial deformation within the dislocation creep regime

(as shown by microstructural observations), however slip continued to be problematic especially towards higher shear stresses. Sometimes patience was needed when a drop in shear stress was registered. Keeping the twist rate constant appeared to be crucial in order to observe a return to increasing shear stress indicating the specimen was being gripped again. Any attempt to increase the twist rate caused further slip. Likewise attempting to reduce the twist rate only reduced the shear stress further. Generally, it was unpredictable at what shear stress or constant twist rate, unrecoverable slip between the gripping sample-alumina discs interfaces occurred, which is why both experiments did not terminate at the same shear stress. Where possible, some time was allowed to reach steady-state deformation before increasing the twist rate again.

Upon termination of the experiments, the furnace was switched off to quench the dislocation microstructures. The specimen assemblies were recovered and examined. The jacket crimp around the specimen, which formed as soon as the assembly was exposed to high confining pressure, was used as a strain marker from which the shear strain was independently measured.

2.2.3.3 Data analysis

For a torsion deformation test, there are a number of geometrical complications that do not exist in a compression test. The analysis given here is a summary from Paterson and Olgaard (2000).

For a cylindrical specimen of uniform diameter d , being deformed in torsion, the shear strain (γ) and strain rate ($\dot{\gamma}$) each varies linearly with radius within the specimen. The maximum shear strain at the surface of the cylinder is given by $\gamma = d\theta/2l$ where θ is the twist angle. For power-law creep with stress exponent n , the shear stress σ_{12}^r varies non-linearly with radial position r according to the expression

$$\sigma_{12}^r = (2r/d)^{1/n} \sigma_{12} \quad (2.2.2)$$

where σ_{12} is the shear stress at the rim. For high exponent $n \approx 10$ the shear stress

remains nearly constant for half the distance between centre and rim. The total torque for a solid cylinder undergoing power-law creep can then be expressed as

$$M = \frac{\pi d^3 \sigma_{12}}{4(3 + 1/n)} \quad (2.2.3)$$

The stress exponent n was experimentally determined from shear strain rate (s^{-1}) or angular displacement rate (mrad s^{-1}) versus the internal torque (Nm) stepping tests. The rate of twist $d\theta/dt$ versus the total torque (M) is given by

$$n = \frac{\delta \ln \dot{\gamma}}{\delta \ln M} = \frac{\delta \ln \dot{\theta}}{\delta \ln M} \quad (2.2.4)$$

The measured torque was not corrected for by the contribution of the mild steel jacket but such a correction would in practice be ~ 0.5 Nm to the measured torque values (Bystricky et al., 2006). The processed data was plotted as equivalent strain rate ($\dot{\gamma}/\sqrt{3}$) versus equivalent stress ($\sigma_{12} \cdot \sqrt{3}$) appropriate for comparison with a compressive test (Shrivastava et al., 1982). Plots of the unprocessed data can be found in Appendix B.

2.3 Attenuation experiments

2.3.1 Overview

Seismic-frequency torsional forced-oscillation experiments, otherwise referred to as attenuation experiments, were carried out at ANU using a custom-built apparatus. The experimental procedure involves study of the torsional oscillation of an experimental assembly comprising the rock specimen and associated torsion rods, and an elastic standard of known compliance, at periods of 1 – 1000 s, under conditions of high temperature ($\leq 1300^\circ\text{C}$) and moderate confining pressure (200 MPa). The default driver amplitudes for twist corresponded to average maximum shear strains of 10^{-5} keeping the rock response within the linear re-

gion. This region exists under certain conditions when the relationship between the applied oscillating stress time derivative is linearly proportional to the time derivative of the response of the oscillating strain and are in phase for genuinely elastic behaviour. Later modifications to the apparatus allowed exploration outside the linear regime for larger drive amplitudes for torsion and hence larger maximum shear strains (see Chapter 5).

In its default configuration, the apparatus was capable of measuring the shear modulus, G and the phase lag δ between oscillating stress and strain (or the dissipation $Q^{-1} = \tan \delta$) of olivine. In addition, microcreep tests were carried out to provide additional information on the elastic and anelastic recovery behaviour of each sol-gel and San Carlos olivine specimen at a given temperature.

Before each experiment, specimens were generally re-fired at 1000°C for 16 hours under controlled atmosphere conditions to remove adsorbed water but keep the dislocation density. They were subsequently heat treated at 180°C in air prior to mechanical testing.

2.3.2 Experimental set up

Many aspects of the attenuation apparatus and its operation have been described in Jackson and Paterson (1987) and Jackson (1993). A summary is given here regarding the current standardised mode of operation and any specifics pertaining to current experiments.

The specimen assembly for attenuation experiments is shown in Fig. 2.2. The assembly was notably longer than those for hot-pressing and deformation but also varied in length from run to run, depending on the sample length and the addition of thicker solid alumina discs. In addition, the assembly was later modified with a thinner waisted section to test 10 mm diameter specimens (as opposed to the standard 11.5 mm, see Table H.2 for specimen dimensions). Two hollow LucaloxTM pistons tapered to this diameter and sandwiched each specimen in between the alumina discs. The specimens were wrapped in Ni₇₀-Fe₃₀ foil and

an $\text{Ni}_{70}\text{-Fe}_{30}$ disc of 0.07 mm thickness was located at each end of the specimen. These foils also served to reduce interfacial stress associated with differential thermal expansion and to prevent chemical reaction between the specimen and torsion rods. Unfortunately, on one occasion, the top $\text{Ni}_{70}\text{-Fe}_{30}$ disc was not properly inserted during assembly and was dislodged inside the mild steel jacket, leaving the sample in direct contact with the solid alumina disc (specimen D6701, attenuation run A1232).

The jacketed specimen assembly (Fig. 2.2) was lowered into the pressure vessel (Fig. 2.4) to mate with and seal against the lower half of the experimental assembly (Fig. 2.4). The bottom half of the assembly is composed primarily of a steel elastic standard of known elastic modulus and negligible internal friction. Upon pressurisation, the two halves were coupled to respond as a rigid unit to torsion from two electromagnetic drivers housed at the bottom of the assembly. The entire assembly was connected through two horizontal lever arms to four sensitive capacitance displacement transducers. The displacement ($d_1(t)$) measured by the upper pair of transducers represents the twist of the specimen assembly (including torsion rods) whereas the difference in displacement between the lower and upper pairs of transducers, $d_{12}(t) = d_2(t) - d_1(t)$ reflects the twist of the elastic standard. These displacements were measured on the computer in the time domain. A Fourier transform function converted these displacements to the frequency domain for determination of the relative amplitudes and phase of $d_1(t)$ and $d_{12}(t)$.

A three-zone furnace was installed around the specimen assembly inside the attenuation apparatus with the hot zone in the region of the olivine specimen. Prior to every attenuation experiment the furnace was re-calibrated with a hollow alumina assembly allowing the use of a traversing thermocouple. It was thus possible to ensure flat temperature profiles across the entire hot zone for temperature intervals of 50°C from 1300°C to 600°C and then for 200°C intervals to room temperature.

A similar reference assembly was previously tested at various corresponding temperatures to provide for the subtraction of the contributions of the (steel / alumina) torsion rods to the overall compliance of the specimen assembly. The reference assembly was nearly identical to the specimen assembly except for a high-grade LucaloxTM polycrystalline alumina control specimen in place of the olivine specimen in the waisted section. The two Ni₇₀-Fe₃₀ discs were co-located at one end of the control specimen which was wrapped in the same Ni₇₀-Fe₃₀ foil (Jackson et al., 2009). Corrections for any dimensional differences between the olivine and alumina control specimens were made during the processing of the torsional forced-oscillation data of each sol-gel and San Carlos specimen (see section 2.3.4).

2.3.3 Experimental conditions

2.3.3.1 Preliminaries

Once the specimen assembly, with a precision-ground sol-gel or San Carlos specimen, was loaded inside the attenuation apparatus, a series of preliminary procedures were carried out in preparation for the experimental run at 200 MPa and maximum desired temperature. For each run, the system was purged twice before pressurisation. After purging the pressure was increased to 150 MPa for the collapse of the jacket into longitudinal and circumferential steel grooves in the steel members at either end of the specimen assembly. After jacket collapse, the whole experimental assembly was raised at room pressure by 1.5 mm to account for thermal expansion of the column and allowing the assembly to freely oscillate at high temperature. In addition, the outer transducer plates were aligned parallel with and equally spaced from the central plate at each transducer station with the aid of an oscilloscope and were subsequently fixed in place. Such ideal alignment of the transducer plates ensures optimal sensitivity for torsion and discrimination against flexure.

In practice, it was necessary to offset the transducer plate spacings for repro-

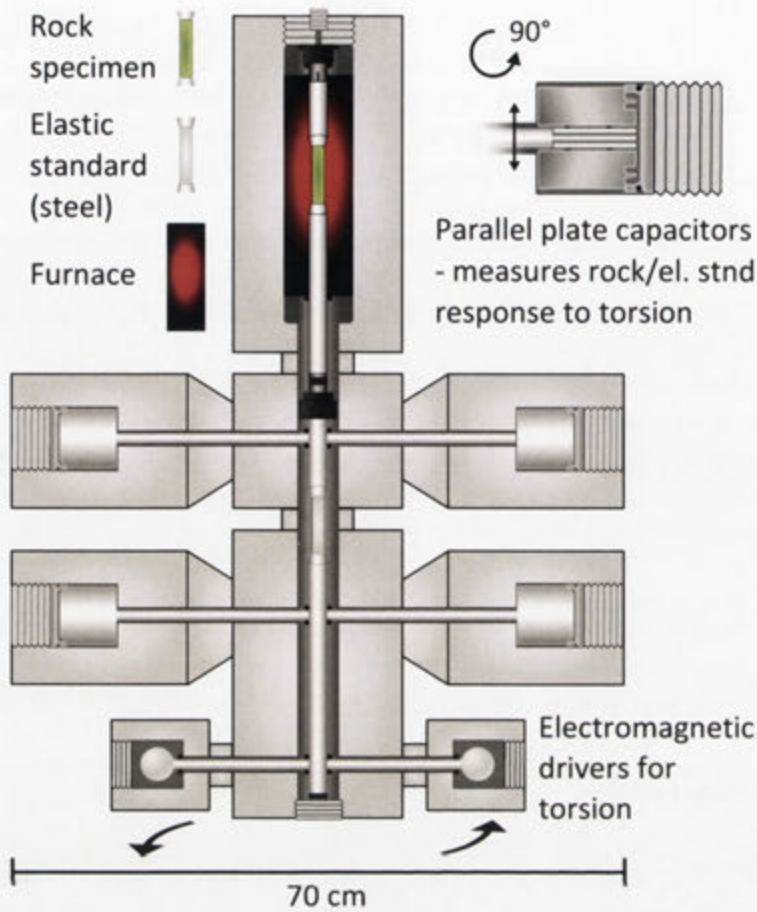


Fig. 2.4: Schematic of the attenuation apparatus. The electromagnetic drivers at the bottom provide oscillating torque amplitudes at seismic periods to the entire assembly, including elastic standard and rock specimen. The displacements are measured as a function of time at the four transducer stations (parallel plate capacitors).

ducible distortions associated with operation at peak pressure and temperature, as follows. The apparatus was once again gradually pressurised to 130 MPa allowing the o-rings to condition. The furnace was switched on and programmed to ramp up in temperature at 300°C/hour to the desired temperature (1100°C for most deformed specimens). During heating, pressure gradually increased with temperature to ~160 MPa assuming no gas leaks occurred. The system was subsequently pressurised in stages using the Ar intensifier to reach 200 MPa. Once the plate spacings for the individual transducers were measured, the fur-

nance was allowed to cool at the same rate of 300°C/hour. At room temperature and pressure conditions, the mean of the plate spacings was calculated to obtain the corrections needed to offset the outer transducer plates relative to the central ones for high temperature and pressure experimentation.

2.3.3.2 Experimental runs

Each data acquisition run for a given temperature, consisted of four sets of prior and subsequent calibration measurements to determine the transducer sensitivities to torsional and flexural mode displacements.

Once the initial calibrations were completed, torsional forced-oscillation measurements were taken over either 1 hour, 5.3 hour or 9 hour runs at each temperature, starting at the highest temperature. The short 1 hour runs were conducted over a range of standard oscillation periods namely 1.28, 3.84, 6.40, 11.52, 21.76, 47.36 and 101.12 seconds. For the longer experimental runs, compliance and loss for three additional periods – 213.76, 467.20 and 999.68 seconds – were measured. Before each cycle at a specific period, a period of pregeneration was allowed to ensure steady state during the actual oscillation measurements. The difference between the 5.3 hour and 9 hour runs is that for the 5.3 hour run, records of only 8 successive cycles were collected, rather than 16. The specific periods shown above were chosen to avoid the worst of the aliasing caused by under-sampled low-level 50 Hz electrical noise.

In addition to torsional forced-oscillation measurements commonly at 50°C intervals, regularly every 100°C a torsional microcreep was run for either a short period of 5,000 seconds or 10,000 seconds. During this test, the torque successively assumed values of 0, +L, 0, -L, 0 at five equal time intervals for the duration of the test (L ~0.1 Nm is a representative value of the steady torque employed in the microcreep tests). The results from the microcreep test provided information on the nature of the recovery of anelastic strain. After the removal of the steady positive and negative torque, a close approach to the original (un)deformed state

of the specimens was expected.

Each specimen assembly containing an olivine polycrystal was first subjected to prolonged annealing (tens of hours) at the maximum desired temperature, typically 1300°C for hot-pressed samples and 1100°C for pre-deformed samples and 200 MPa confining pressure. During this time, short 1 hour measurements comprised of seven oscillation periods between 1 and 100 seconds were taken to monitor the evolution of the mechanical behaviour of the rock. Once the behaviour of the olivine polycrystal stabilised with no further increase or decrease in the compliance or loss, data acquisition began.

Due to a maximum imposed temperature of 1100°C for most of the pre-deformed specimens, additional oscillation data between 1100 – 1000°C was obtained at 25°C intervals (and between 1000 – 900°C for T0434, run A1271). Once a temperature of 600°C was reached, only 1 hour experimental runs were carried out to room temperature. Once at room temperature, the pressure was dropped from 200 MPa to 100 MPa for another 1 hour run. The purpose of this was to check for the opening of cracks and pores at lower confining pressure which would affect the measured compliance and loss of the specimen. If no change was registered, it was inferred that most cracks had been annealed at high temperature and that little thermal cracking had occurred during the subsequent staged cooling under pressure to room temperature. At the end of an experiment, the furnace was switched on and the apparatus repressurised to 200 MPa to return to the previous maximum temperature (at 300°C/hour) for several other short or long experimental runs to check for reproducibility of the data.

2.3.4 Data processing

Routine processing of the raw forced-oscillation data yielded estimates of the torsional compliance of the entire specimen assembly (inclusive of steel/alumina torsion rods) normalised by that of the elastic standard and phase lag between $d_{12}(t)$, representing the distortion of the elastic standard and $d_1(t)$, a measure

of the viscoelastic response of the specimen assembly. The output data was subsequently processed by another script which, broadly speaking, subtracted the complex compliance of the reference assembly from that of the assembly containing the olivine specimen. The LucaloxTM alumina control specimen of the reference assembly was modelled as mildly viscoelastic (Lakki et al., 1998)*.

Because these two assemblies inevitably varied geometrically and were still uncorrected for jacket and foil contributions, certain corrections had to be applied. These corrections were entered in the input files and were the following. The first correction removed the difference in assembly length (ΔL), the second took into account the difference in vertical position of the two assemblies relative to the pressure vessel ($\Delta Z'$), the third was a correction for the difference in the length of the waisted section (ΔLW), the fourth correction was the extra length above 6 mm for the solid alumina discs ($\Delta LDSK$) and finally the fifth correction took into account a difference in thickness for the Ni₇₀-Fe₃₀ foil end discs (ΔLF). In addition to these corrections, the olivine specimen length, its bare diameter and jacketed diameter were specified. The run conditions were entered last, for pressure, temperature and number of oscillation measurements (7 or 10). Finally, the compliance and phase angle data were processed with known material and thermal properties of steel and alumina – adjusted as appropriate for the effects of pressure and temperature. The steel jacket and foil liner were modelled with previously acquired data concerning the temperature-dependent viscoelastic behaviour of mild steel. After processing, the shear modulus G and strain energy dissipation Q^{-1} were obtained for the sol-gel or San Carlos olivine specimens.

2.3.5 Least-squares fits to extended Burgers model

In a paper by Jackson and Faul (2010) a revised approach was described to fit strain-energy dissipation Q^{-1} and the associated shear modulus G as functions of oscillation period T_0 , temperature T , grain size d for essentially dry polycrystalline

*Testing is underway to find a better elastic standard of known shear modulus

olivine specimens, the same types employed in this research. Any material, such as olivine, will experience elastic and non-elastic deformation in response to an applied stress. Non-elastic, time dependent deformation has two components, recoverable (anelastic) and non-recoverable (viscous) deformation. Both elastic and viscous components can be described by spring (elastic) and dashpot (viscous) components in the Maxwell and Kelvin-Voigt models. The Kelvin-Voigt model is responsible for recoverable anelastic behaviour whereas viscous behaviour entirely comes from the Maxwell element. Both can be combined to form the Burgers model for an anelastic solid built-in with both a short- and long-term viscous response. The Burgers model with creep function is given by Faul and Jackson (2005) as

$$J(t) = J_U \left\{ 1 + \Delta \int_0^\infty D(\tau) [1 - \exp(-t/\tau)] d\tau + t/\tau_M \right\} \quad (2.3.1)$$

where J_U is the unrelaxed compliance (inverse of the unrelaxed shear modulus G_U), $\tau_M = \eta J_U$ is the Maxwell relaxation time proportional to the viscosity η , Δ is the relaxation strength associated with the normalised distribution of anelastic relaxation times $D(\tau) = \alpha \tau^{\alpha-1} / (\tau_H^\alpha - \tau_L^\alpha)$ with $\tau_L < \tau < \tau_H$ and zero elsewhere, and $0 < \alpha < 1$ (Minster and Anderson, 1981), where α is the frequency dependence.

For linear viscoelastic behaviour described in Chapter 5, the Laplace transform of the creep function Eqn. (2.3.1) gives the dynamic compliance $J^*(\omega)$ with respectively real and imaginary parts

$$J_1(\omega) = J_U \left\{ 1 + \Delta \int_0^\infty D(\tau) d\tau / (1 + \omega^2 \tau^2) \right\} \quad (2.3.2)$$

and

$$J_2(\omega) = J_U \left\{ \omega \Delta \int_0^\infty \tau D(\tau) d\tau / (1 + \omega^2 \tau^2) + 1/\omega \tau_M \right\} \quad (2.3.3)$$

The shear modulus G and strain energy dissipation (attenuation) Q^{-1} are then

given as functions of angular frequency $\omega = 2\pi/T_0$ or oscillation period T_0 by

$$G(\omega) = [J_1^2(\omega) + J_2^2(\omega)]^{-1/2} \quad (2.3.4)$$

and

$$Q^{-1} = J_2(\omega)/J_1(\omega) \quad (2.3.5)$$

A modification to parameterisation of the unrelaxed compliance J_U was employed by Jackson and Faul (2010) to include its variation with temperature and pressure as

$$J_U(T, P) = [G_U(T_R, P_R) + (T - T_R)\partial G/\partial T + (P - P_R)\partial G/\partial P]^{-1} \quad (2.3.6)$$

where the elements with subscript R are the reference conditions explained in the following.

The extended Burgers model consists of several parameters that provide a physical background for the fitting. In brief, these parameters are; 1. The value $G_{UR}(= 66.5 \text{ GPa})$ of unrelaxed shear modulus G_U of olivine at the reference conditions of pressure and temperature, here taken as $P_R = 0.2 \text{ GPa}$ and $T_R = 1173 \text{ K}$ (900°C); 2. The temperature derivative of the unrelaxed shear modulus $\delta G_U/\delta T$; 3. The anelastic relaxation strength Δ_B of the high-temperature background; 4. The exponent α for the distribution $D(\tau) \sim \tau^{\alpha-1}$ of anelastic relaxation times associated with the monotonic background; 5-6. the lower and upper limits, at the reference conditions, for the distribution of anelastic relaxation times ($\log_{10} \tau_{LR}$ and $\log_{10} \tau_{UR}$) fixed at [2] and [6.5]; 7. the Maxwell time for viscous relaxation ($\log_{10} \tau_{MR}$); 8. grain size (d) and finally 9. the activation energy for background dissipation (E_B). The fit was constrained by setting a few other parameters for the reference, fully dense, and pure Fo_{90} sample of given grain size d_R . An activation volume of $10^{-6} \text{ m}^3 \text{ mol}^{-1}$ (Durham et al., 2009) was used and $\partial G/\partial T$ and $\partial G/\partial P$ – the established anharmonic values were given as -13.6 MPa K^{-1} and 1.8 respectively.

The adjustable parameters (α , Δ_B , $\log_{10} \tau_{MR}$, E_B and sometimes G_{UR}) were refined through the Levenberg-Marquardt least-squares algorithm involving minimisation of the objective function

$$\chi^2 = \chi_G^2 + \chi_{1/Q}^2 \quad (2.3.7)$$

with $\sigma(G)/G = 0.03$ and $\sigma(\log_{10} Q^{-1}) = 0.05$. In addition, the average misfit was determined as

$$(\chi^2/2N)^{-1/2} \quad (2.3.8)$$

where N is the number of (G, Q^{-1}) data pairs being fitted per specimen. Acceptable fits with misfit values of ≤ 1.5 were sought with the parameter constraints given above. For several specimens the fit was improved by excluding the shortest period oscillation data at 1.28 and 3.84 seconds for relatively lower temperatures $< 1025^\circ\text{C}$. Any data below $\log_{10} Q^{-1}$ values of -2.0 were generally excluded systematically.

The modelled results of the hot-pressed, compressively or torsionally pre-deformed sol-gel and San Carlos samples are intended for extrapolation to the conditions of seismic-wave propagation in the Earth's upper mantle. In order to provide a comprehensive overview of the viscoelastic behaviour, the experimental models were evaluated for ranges of temperature and oscillation period broader than those accessed experimentally.

2.4 Dislocation recovery

2.4.1 Overview

High temperature static annealing experiments were carried out on compressively pre-deformed sol-gel and San Carlos olivine samples to determine the rate of dislocation recovery. The method used to image dislocations for the light and electron microscope is described by Kohlstedt et al. (1976) and Karato (1987),

respectively. The rate of dislocation recovery with the rate constant k was determined in this study similar to previous studies (Kohlstedt et al., 1980; Karato and Ogawa, 1982; Karato et al., 1993). The following sections describe in detail how the samples were prepared and analysed.

2.4.2 Sample preparation and experimentation

The prepared quadrants, which were mounted in epoxy (as described in section 2.1.4.2), were ground and polished. The first stage of polishing involved the removal of deep grooves left behind from the diamond wafering blade. Polishing was done by hand firstly using a 600 # grade Silicon Carbide (SiC) of mean particle size of 14 μm followed by SiC of 1200 # grade of mean particle size of 5 μm . The epoxy blocks were subsequently cored from below and mounted on a Kent Mark 2 (Engis Ltd.) polisher. A hard disc-shaped polishing cloth, branded as Kermet Microtex 600-K, was used on the rotating polishing wheel. Firstly a 6 μm diamond paste was added and the samples were mechanically polished for 1 hour. On a new cloth, 3 μm diamond paste was added for another 1 hour polish. The samples were then transferred to a Kent mark 3 polisher fitted with a soft Buehler Mastertex polishing cloth and were polished for another 30 minutes using an alumina slurry of 0.05 μm particle size and a 1 hour polish on a new soft cloth using 0.05 μm colloidal silica.

After the first round of polishing, olivine cubes of ~ 2 mm edge were cut using a diamond wafering blade from the polished quadrant and removed from the epoxy. Two cubes, one from the centre and one from the rim, were retained unannealed in order to obtain the averaged initial dislocation density information.

The first set of annealing experiments was done on the pre-deformed sol-gel olivine cubes followed by the next set of annealing experiments on San Carlos olivine. For every experimental run, one cube was placed into a small Pt bucket hanging from a long alumina tube. Several thin Pt wires were threaded through the bottom of the bucket to minimize contact between olivine and platinum and

minimise iron loss from the olivine. The assembly was lowered into a controlled atmosphere furnace left at a constant base temperature of 600°C. Immediately, CO and CO₂ gas flow controllers were switched on to ensure appropriate partial pressures of these dominant gas species. A mixture containing 70% CO and 30% CO₂ was used to provide redox conditions similar to those prevailing during hot-pressing and compressive deformation within Ni₇₀-Fe₃₀ foil wrappers (see Appendix F). This buffer lies roughly in between the petrological quartz-fayalite-magnetite and quartz-fayalite-iron buffers. Heating and cooling ramp rates varied between 12°C/min and 50°C/min for faster ramp rates above 1250°C target temperatures.

While for long duration and moderate temperature experiments the extra recovery during cooling and heating would not have significantly affected the final dislocation density results, this mattered for the highest temperature annealing experiments due to faster dislocation recovery kinetics. Hence, minor corrections to the calculated rate constants (k) for dislocation recovery were applied to take this consideration into account. The correction values applied to ρ_f , propagated to k , are listed in Chapter 3, section 3.3. Without these corrections, ρ_f would be artificially low and hence k artificially higher.

In total, 10 annealing experiments were performed on sol-gel olivine (D6532) and 7 on San Carlos olivine (D6701) between 1100°C and 1550°C for different durations (10 min up to 50 hours). For sol-gel olivine, an additional run was carried out at 1200°C/50 hours at an oxygen fugacity of 1.7×10^{-6} Pa, away from $f_{O_2} = 8.6 \times 10^{-4}$ with a 70% CO and 30% CO₂ gas mix, to estimate an effect on the final dislocation density. For the sol-gel specimen, multiple annealing experiments of various durations (3-50 h) were conducted at 1300°C. All the olivine samples were given oxidative-decoration treatment as described in the next section.

2.4.3 Decoration of dislocations by oxidation

The annealed and as-deformed olivine cubes of both sol-gel and San Carlos origin were placed in a box furnace under normal atmospheric conditions and quickly heated to 900°C. The samples were left to oxidise for 45 minutes in a Pt crucible and subsequently retrieved (Kohlstedt et al., 1976; Karato, 1987). During this process, grain boundaries and dislocations at the surface are preferentially oxidised to produce iron oxide precipitates, most likely magnetite or hematite.

The oxidised olivine cubes were mounted in epoxy. Each sample was placed with its previously polished surface faced down onto adhesive tape. A cup was placed around it into which liquid epoxy was poured and left to harden around the olivine sample overnight. Every olivine sample was prepared in its own epoxy block and re-polished with an alumina slurry (0.05 μm) on the Kent Mark 3 polisher using a soft cloth. Previous experimentation with pre-polished oxidised olivine cubes demonstrated optimal visibility of dislocations for a 10 minute polish at a given rotation speed of 50 turns per minute on the polishing wheel (polishing away about 2 - 6 μm of material). During the polishing, a fresh scoop of alumina slurry was added every two minutes. Progress of removing the iron-oxide coating on the surface of each sample was occasionally checked under the light microscope. In the rare event that the iron-oxide layer was still covering the grains after 10 minutes of polishing, up to two more minutes of polishing was allowed to obtain a surface showing only the decorated grain boundaries and dislocations (this is because the roughness of the surface could leave a fraction of depressed grains coated with the iron-oxide layer). At 1000 $\times$ magnification, dislocations and grain boundaries were clearly visible under the light microscope.

The polished samples remained in their respective epoxy blocks and were carbon coated under vacuum for 4 - 5 pulses. The thickness of the coating was not critical as opposed to electron backscatter diffraction (EBSD) preparation, but needed to be thick enough to avoid charging of the surface once inside the field emission scanning electron microscope (FE-SEM). A solid-state type back-

scattered electron (BSE) detector was inserted under the pole piece. The FE-SEM dislocation imaging steps are described in the next section.

2.5 Dislocation imaging using back-scattered electrons

2.5.1 Imaging conditions

This study used the Hitachi S-4300SE/N Schottky (field) emission FE-SEM for dislocation imaging performed under the following conditions. A low accelerating voltage of 5 kV was used to reduce imaging depth by minimising the electron interaction volume (see Appendix G). A short working distance of 10 mm was chosen to allow the BSE detector to be closer to the sample surface thereby capturing as many low angle backscattered electrons as possible. A large aperture with setting '1' was used, equivalent to 100 μm which allowed more electrons from the electron beam to pass through to interact with the specimen. Image quality was further improved by selecting an average condenser lens setting of '6' (equivalent to $\sim 10^{-10}$ A specimen irradiation current) for a reasonable spot size. Allowing a too high current to pass through the condenser lens led to increased noise making it harder to focus on the specimen. A gun brightness setting of '3' was used. The emission current was routinely measured at 43 μA and the specimen current was around 0.160 – 0.180 nA as measured by an external pico ammeter (~ 0.3 – 0.4 nA in the Faraday cup). In short, it measured the portion of the probe current conducted through the specimen to the ground. The reading depends on the average atomic number of the sample.

Once the olivine sample was brought into focus, the electron beam was aligned and astigmatism was corrected in a secondary electron image, the BSE detector was inserted for dislocation imaging. The image signal was modified for maximum contrast and moderate brightness to optimally highlight the dislocations

and grain boundaries. The dislocations and grain boundaries appear brighter under BSE conditions due to an average higher atomic number of iron-oxide precipitates in relation to the surrounding olivine. The INCA Automate+ software was capable of taking control of the microscope beam and stage and was used to image areas within a pre-defined rectangle or along a line. Preliminary tests showed that for areas taken at magnifications between $3000\times$ and $10,000\times$, dislocation information was not sufficiently imaged below $6000\times$ and no additional dislocations were imaged at magnifications higher than $8000\times$. Hence, up to 4 rectangular areas, randomly located, with each being an array of 16 to 36 back-scattered electron images were recorded at a default magnification of $8000\times$ for dislocation density determination. The individual images in the grid had an original resolution of 2048×1536 pixels which was preserved by extracting the images from the master file using the INCA Batch software. The microstructural images were stitched together using the Adobe Photoshop photomerge feature into one large high-resolution image for each area. These large images were subsequently ready to be processed for extraction of dislocation density and grain size information.

2.5.2 Data processing

2.5.2.1 Dislocation density determination

In total, 19 sol-gel and San Carlos samples were imaged using the FE-SEM equipped with a BSE detector. Areas, composed of high resolution BSE images, were processed as follows to obtain the dislocation density information (see Fig. 2.5). Most of the image processing was done using open source software called ImageJ[†]. High contrast grey-scale images (Fig. 2.5a) were inverted (Fig. 2.5b) and processed with a threshold filter. The original 16-bit image was converted to a 2-bit grey-scale image after a threshold operation (Fig. 2.5c). Given small variations between the beam conditions of different imaging sessions, the

[†]<http://rsbweb.nih.gov/ij/>

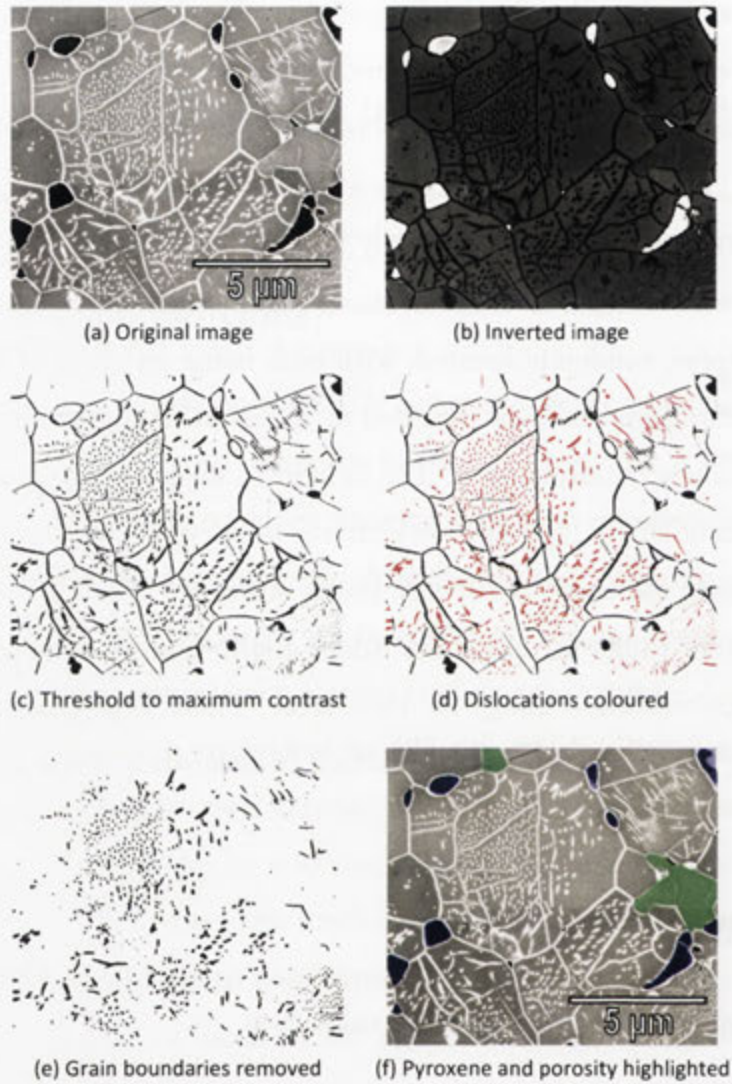


Fig. 2.5: Procedure for obtaining the 2D dislocation density of the as-deformed olivine specimen. Panels (a) to (e) show how the original SEM image is inverted, processed with a contrast filter of an appropriate threshold, manually coloured with a colour replacement tool so that a colour threshold filter can be applied to remove the grain boundaries. Panel (f) shows the manual highlighting of the porosity (dark blue) and pyroxene grains (green) that are excluded from the calculated area. To get the 3D dislocation density result, the line lengths are measured in panel (e) instead of the number dislocations

threshold filter setting for each image was manually adjusted at high magnification on a region of poorly visible dislocations to ensure no dislocation information was lost during this operation. Two noise filters were used. The first removed all single pixel outliers and the second removed all remaining clusters of 5 or fewer pixels which were systematically checked for potential dislocations in the original overlaid image.

The dislocations were manually highlighted using a colour replacement tool from black to red as shown in Fig. 2.5d. The following features were avoided during this manual operation. Dislocations in array configurations were not considered free dislocations but rather recovery microstructures. These dislocations, that were not counted, had to be parallel to each other and not wider apart than the full length of a dislocation in such an array. Other sub-structures that were avoided were long straight decorated segments often crossing through grains (these were probably decorated micro-cracks or scratches), connected grain boundaries, pores and shadows from surface contamination.

The number of dislocations and the approximate imaged dislocation lengths were measured using an add-on to ImageJ called 'Bezier curve ROI[†]'. This add-on attempted to locate the largest separation between two points on each object (i.e. dislocation). It then fitted a parametric curve to the end points of the object and any undulations in between. This function provides the cumulative length of all dislocations.

Two approaches were used to calculate the dislocation density. Initially the '2-D' dislocation density was determined from the number of dislocations over an area (m^{-2}). An improved estimation was obtained from the dislocation length information in the processed back-scattered electron images. The true '3-D' dislocation density was determined from the dislocation length over the 'imaging volume' (also in m^{-2}).

Previously, a correction rather than direct measurement was applied to ap-

[†]<http://www.optinav.com/Measure-Roi.htm>

proximate the 3-D dislocation density as $\rho_{3-D} = 2N/A = 2 \cdot \rho_{2-D}$ (Karato, 2008). However, this correction assumed that a dislocation can be randomly oriented, while from observation clusters of dislocations appear to exist in preferential configurations (e. g. in parallel arrays).

Additionally, from geometrical considerations, imaged or projected dislocation lengths l could not be the true dislocation lengths. In order to obtain the true dislocation length l_0 , a value for the imaging depth or depth of information d at 5 kV accelerating voltage was needed (see Appendix G). In brief, the 3-D dislocation density ρ can be expressed as

$$\rho = (1/A)\Sigma \left[\sqrt{1 + (l/d)^2} \right] \quad (2.5.1)$$

where Σ is the sum of the lengths of all dislocations and A is the total area imaged.

Details for evaluation of uncertainty for dislocation density measurement and of imaging depth d are given in Appendix G or refer to Chapter 3.

2.6 Electron backscatter diffraction (EBSD)

2.6.1 Overview

EBSD is a microstructural or crystallographic technique used to examine the crystallographic orientations of grains in ceramics or metals. Preferred orientations and grain textures provide information on the history of deformation of a material and EBSD can also be used to study defects, mineral phases and grain boundary morphologies. The main advantage is that sample preparation is relatively easy, unlike for the transmission electron microscope, and similar / complementary information on the operating crystal slip systems in a deformed material can be obtained.

EBSD is extensively used in mineral studies for various dynamic settings in

the Earth (e. g. Mainprice et al. 2008; Tasaka et al. 2008; Skemer et al. 2009), for seismological models (e.g. Vonlanthen et al. 2006; Becker et al. 2008; Gibert and Mainprice 2009) or for deformation mechanisms of the upper mantle mineral olivine in particular (e.g. Bruhn et al. 2005; Jung et al. 2006; Warren et al. 2008).

In this study, the ZEISS UltraPlus FE-SEM with EBSD capability was used to analyse grain orientations in compressionally and torsionally pre-deformed sol-gel and San Carlos olivine. The software used was provided by Oxford Instruments called HKL CHANNEL (version 5.0.9.0). The software package includes the following programmes; Tango for examination of grain properties and orientations, Mambo for plotting (inverse) pole figures of grain orientations and Salsa for determination of the orientation distribution function and associated fabric strength indicator (J-Index). Additional functions used include the capability to stitch multi-maps and obtain histograms of grain misorientations (for an M-Index calculation see Skemer et al. 2005).

Here, the primary objective was to determine the fabric strength and CPO of the deformed olivine aggregates and to make a comparison between the mode of deformation (compression versus torsion). In addition, a question arose during attenuation experiments on the torsionally deformed samples, whether annealing was having an effect on the highly strained microstructures at moderate temperatures. No EBSD mapping was attempted on a compressively deformed sol-gel sample, see Faul et al. 2011 instead.

2.6.2 Specimen preparation

As indicated in section 2.1.4.2, EBSD samples were prepared from several pre-deformed and equivalent attenuation specimens. From each pre-deformed specimen and those obtained after mechanical testing in the attenuation apparatus, a transversely sectioned quadrant of 2 mm thick was taken and emplaced in an epoxy block. The cylindrical specimens that were pre-deformed in torsion (T0436, A1231 and T0434, A1271) were polished on the tangential side close to the rim.

This permitted microstructural observation of the shear plane in the zone of highest strain. The embedded olivine sections were manually polished using SiC grit of increasing grade (600#, 14 μm and 1200#, 5 μm). Subsequent polishing was done on polishing machines fitted with rotating wheels that were covered with a hard cloth and 6 μm diamond paste followed by 3 μm diamond paste. Both mechanical polishing steps lasted 1 hour each. The final step included a 30 minute polish with an alumina 0.05 μm slurry followed by a 1 hour mechanical polish using colloidal silica (0.05 μm particle size) both carried out on a soft cloth. The final polishing result was inspected under the light microscope before taken to the electron microscope.

Related to a study presented in Appendix E, two EBSD sections were given the same polishing treatment described above, but were subsequently oxidised in air as described in section 2.4.3. After oxidative decoration they were repolished for 10 minutes using either an alumina or colloidal silica slurry. In the first instance for sol-gel olivine, dislocations were made visible using the same polishing procedure which has been used for the dislocation recovery experiments (see section 2.4.3) using an alumina slurry. EBSD appeared to be possible on an oxidized and decorated sample while concurrently discriminating between grains of high and low dislocation density. Point-indexing of grain orientations was successful using this method, however the sample was severely scratched from the alumina polish.

A trial polish of 10 min using colloidal silica on an oxidised San Carlos sample yielded a better indexing rate of grain orientations while grains of high and low dislocation density were still clearly discernible in grain-orientation contrast images (using the forward-scattered electron detector). This technique may be used in a future project by directly mapping an area of grains of known dislocation density (an improvement which could be built upon the preliminary study presented in Appendix E). However for accurate, high magnification, dislocation density measurements, separate electron backscattered imaging was necessary

(see section 2.5.1).

A prepared EBSD section ideally required a consistent thin carbon coat yet avoiding charging of the sample surface to minimize obstruction to diffraction and to obtain a high indexing rate (where 100% is perfect). In practice, however, since no film thickness monitor was available for the carbon coat machine, it was only possible to judge the thickness of the coating from a piece of filter paper placed in the coater with the sample under vacuum. For all subsequent EBSD carbon coating, the coated filter paper was used as a reference.

2.6.3 Conditions used to carry out EBSD

EBSD was carried out using techniques to provide the best diffraction patterns (Kikuchi bands) with an acceptable indexing rate for olivine (see e.g. Deal et al. 2005 for more information). After mechanical polishing and carbon coating, the axially pre-deformed specimen section was oriented inside the chamber of the electron microscope with the compression axis perpendicular to the mapped area (z direction) and the torsionally deformed specimen sections were oriented with the shear direction and shear plane in the x and y directions respectively (Fig. 2.6a,b,c). The stage was lowered to 6.5 mm and tilted by 70°. A Nordlys hkl CCD camera, fitted with a phosphor screen and a forward-scattered electron detector, was inserted up to about 10 mm away from the sample (Fig. 2.6d). The operating ZEISS SmartSEM software was configured to use a default aperture of 30 μm . A high current setting was enabled by default for improved imaging conditions. Astigmatism, focus and alignment were checked under the geometric conditions described above to provide an average working distance of 24.5 mm. At 20 kV accelerating voltage, probe current was nominally 397 pA (~ 200 pA measured) and a measured beam current between 124 μA for a new tungsten filament to 340 μA from an ageing filament. Tilt correction was applied via scan control from the HKL CHANNEL software.

The HKL software was used for grain orientation contrast imaging, manual

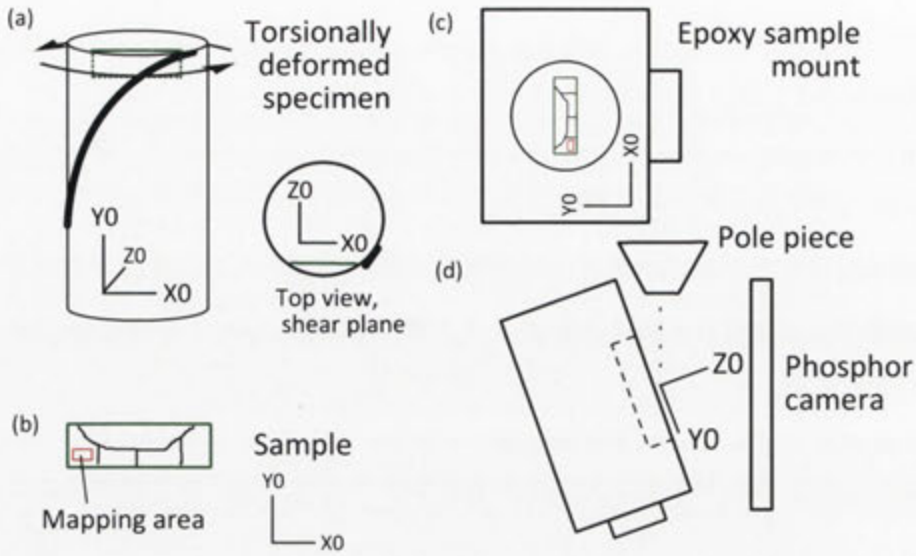


Fig. 2.6: EBSD set up for a polished section. (a) A tangential section is cut from the torsionally pre-deformed specimen and a laboratory reference frame is chosen. Here the X_0 direction is the shear direction and Y_0 is the normal to the shear plane. (b) After polishing, a mapping area is identified in relation to visible cracks (shown in black). (c) The section is mounted keeping in mind its orientation with respect to the reference frame. (d) the mounted section is inserted in the electron microscope chamber and the stage is tilted by 70° . The phosphor camera is inserted which is used to image the diffraction patterns when the electrons hit the section from the pole piece.

point indexing and for mapping projects. At low magnification but with only the sample in view, an electron-backscattered pattern (EBSP) was taken to define the background across the camera when measuring individual grains. The quality of the EBSP depends on several settings in the HKL software. A moderate pixel binning resolution of 4×4 was used with low gain. In addition, a setting of 3 frames noise reduction with a timing per frame of 60 to 80 ms gave the best compromise between successful EBSP indexing whilst keeping the time to capture patterns as short as possible. Further settings, optimised for olivine indexing using the HKL software, include the number of reflectors at 75, a hough space at 120 and 7-8 bands to be detected for accurate diffraction pattern recognition.

2.6.4 Orientations of low and high dislocation density grains

The orientation of low and high dislocation density grains was determined from point-indexing pre-oxidised and decorated sol-gel olivine (D6532) and San Carlos (D6701) specimens. Grain orientation contrast images were captured for a number of random areas at a magnification of $4000\times$ for fine-grained sol-gel olivine and at $3000\times$ for the slightly coarser-grained San Carlos olivine sample. Spot mode was used to position the beam onto a desired grain previously identified with a high or low to no dislocation density (using the forward-scattered electron detector). Up to 30 grains per area of each subset were indexed this way, saved into two separate bins for high and low dislocation density. Grains of moderate dislocation density were avoided in this study. For sol-gel olivine, a total number of 585 grains were manually indexed whereas for San Carlos 443 grains were indexed for roughly equal proportions of low and high dislocation density grains. HKL Tango was used to construct pole figures (see Appendix E for more information on this research project).

2.6.5 EBSD data acquisition via mapping

Several deformed sol-gel (T0436, A1231) and San Carlos (D6701, T0434, A1271) samples were prepared for overnight EBSD mapping sessions. The mapping process describes the automatic indexing of orientations in an array of points in a rectangular region at constant grid spacing (defined as the step size). All but the smallest grains (that were missed due to a too coarse step size) were indexed to determine the CPO. As a rule of thumb, at least 8 indexing points were required to be inside a grain in order to detect a single grain accurately. A step size was determined accordingly. Depending on the grain size, step size values varied between $0.5 - 1\ \mu\text{m}$. Larger step size sped up mapping and facilitated indexing of larger areas (and more grains) within a restricted amount of time, but grain boundaries were less well resolved. A 3×3 grid of connected areas was generally

chosen to cover an reasonable area within the allowed time available.

Table 2.3: EBSD mapping results for all specimens

	D6701	T0434	T0436	A1231	A1271
Map layout	3×3	3×3	4×1	3×3	3×3
Step size (μm)	1.0	0.6	0.5	0.4	0.6
Timing per frame (ms)	60	80	80	80	80
Mean time (ms)	250	311	1070	313	322
Total area (mm ²)	0.2106	0.0545	0.0140	0.0253	0.0578
Final index rate (%)	85	74	78	77	76

Up to four test areas of around 25×25 pixels on a representative area were run to determine which step size was ideal and whether each point was indexing properly. The geometry of the system was refined in the HKL software if the maps did not finish with an acceptable (< 70%) indexing rate. The timing per frame for capturing diffraction patterns was increased if indexing did not improve, although generally it was not increased beyond 80 ms to avoid indexing only a partial map in the time available. Average times were between 0.250 and 0.350 s allowing 9 areas of roughly 170 × 120 pixels to be captured during 15 hours overnight. Table 2.3 summarises the run conditions for each sample.

2.6.6 Data processing using HKL software

CPO data, amongst other microstructural information, were extracted from EBSD maps. Firstly, the unchanged EBSD map data were loaded in HKL Mambo. The first modification applied was the removal of solitary non-indexed pixels to match the orientation of their neighbours. The next step was to remove any systematic misindexing due to pseudosymmetry in the individual grains. For olivine, the most common misindexing occurs at the [100] and [-100] orientations, for which the diffraction patterns are similar and 60° apart due to a pseudo 3-fold symmetry. The maps were subsequently noise reduced by taking small iterative steps

converting non-indexed pixels into orientations while continuously consulting a band contrast map (see Brewer and Michael 2010). Afterwards, all small clusters of pixels were individually checked and removed if they did not appear to be true grains.

A one point-per-grain sub-set of data was extracted from the EBSD map data to plot the pole figures in HKL Tango. Inverted pole figures were plotted for either compression directions or shear directions in the crystallographic reference frame. In a deformed grain, dislocation alignment gives rise to small variations in crystallographic orientations. Hence, misorientation axes of sub-grains (misorientation angles between 2 and 10°) were also plotted in the crystallographic reference frame to provide some information on the internal grain deformation mechanisms.

2.6.6.1 Grain size determination

Grain area information was extracted from the built-in grain analysis function in HKL Mambo. For olivine, grains with grain boundary misorientation angles > 10° were analysed and all grains truncated by the map edge were excluded from the final grain size determination. In addition, the grain size with a corrected diameter was determined similarly to the method described in Chapter 4 rather than relying on the built-in function in the HKL software. Histograms were constructed to display distributions in grain size, for comparison between hot-pressed or pre-deformed specimens with specimens sectioned and analysed after attenuation experiments.

2.7 FTIR analysis

2.7.1 Overview

The water (OH) content in the olivine aggregates was analysed using an infrared spectrometer. Normally for olivine, the region of interest is in the infrared domain

between wavenumbers 4000 and 3000 cm^{-1} in which specific vibration frequencies of OH-related species in olivine contribute to the absorbance of infrared light at specific wavenumbers.

Much research has been carried out on determining the OH content in olivine since Paterson (1982) and there are several methods currently employed. Bulk measurements are typically taken using unpolarised light while single grain measurements are carried out using polarised light (Lemaire et al., 2004). Additional corrections have to be made using polarised light as the absorbance in olivine is anisotropic, i. e. varies with crystallographic orientation (Sambridge et al., 2008; Kovács et al., 2008). For bulk measurements, an averaged absorbance is measured across many grains and grain boundaries. Either an integration under the absorbance spectrum curve is performed (e. g. Demouchy 2010) or the peak height of the approximately symmetric curve is measured in order to calculate the water concentration. Both methods should give approximately similar results. The calculation requires the knowledge of an appropriate value for the molar absorption coefficient which has been calibrated by Paterson (1982) but has been refined over the recent years to yield 2 to 4 times higher OH measurements in olivine (Bell et al., 2003; Aubaud et al., 2009). Hence, care should be taken when comparing OH measurements from this study to older studies.

Unlike single crystal olivine measurements, an aggregate often displays a contribution from OH present in the grain boundaries, pores, fluid inclusions and in silicate glass. These microstructural features characterise a broad band of absorbance over a range of wave numbers red-shifted towards a lower frequency, roughly centered on 3400 cm^{-1} . A broad band of absorption may hide minor absorbance of structurally bound OH (Berry et al., 2005) in the olivine grains although this depends on the quality of the spectrum (signal to noise) and the baseline correction.

2.7.2 Procedures

In this study, only bulk measurements with limited spacial resolution could be performed using a Vertex 80V FT Infrared spectrometer fitted with a 632.8 nm IR laser and pyroelectric DigiTect™ DLaTGS detector covering a spectral range of 12,000 – 250 cm⁻¹. The spectral resolution was better than 0.2 cm⁻¹. The sample chamber could be vented to a vacuum of 1.6 hPa and contained a sample holder positioned directly in the path of the IR beam, nominally 1.5 mm wide. The measurements were performed on 19 double-sided polished sections of average 400 μm thickness for all pre-deformed or hot-pressed and attenuation specimens (A1127 which was 167 μm thick). The IR sections were sandwiched between a stub with a 2 mm hole and a doughnut-shaped metal foil.

Before a sample measurement could be taken, a background measurement using the Opus software package (v 6.5) was performed on an uncovered disc with a 2 mm diameter hole to set an absorption baseline. The absorbance was measured as

$$A(\lambda) = \log_{10}(I/I_0) \quad (2.7.1)$$

where I_0 is the intensity in the no-sample spectrum at each wavelength λ and I is the corresponding intensity for an inserted sample.

A single channel measurement confirmed that the IR beam was passing through the hole unobstructed. The vacuum chamber was subsequently vented and the first olivine IR section was inserted in place. Under vacuum, full spectrum measurements were taken from 8000 to 0 cm⁻¹ for 64 scans. The raw spectrum was saved and a subsequent baseline correction was applied as follows. A region between 4000 and 2400 cm⁻¹ was magnified and the baseline was pinned on the curve for the following wave numbers (cm⁻¹): 4000, 3900, 3800, 3700, 3100, 2800, 2700, 2550, 2400. Correction of the curve was avoided in specific regions of interest where a broad band or structural absorbance might occur. The averaged value for peak absorbance in the broad band was measured for a range of wave

numbers ($\pm 50 \text{ cm}^{-1}$) around 3400 cm^{-1} to avoid bias from the noise in the curve. It was possible to measure both the rim and the central region for each cylindrical sample.

Once an IR absorbance value was measured per sample, the associated water concentration was calculated using the Bouguer-Beer-Lambert law approach.

$$A = \epsilon \cdot c \cdot d \quad (2.7.2)$$

where A is the absorbance, ϵ is the molar absorption coefficient ($\text{l mol}^{-1} \text{ cm}^{-1}$), c is the concentration of water (wt. ppm) and d is the length (or thickness) of the material (cm). The absorbance is normalised to 1 cm thickness and the expression is rearranged to solve for the unknown concentration of water.

For olivine of given measured density ($\rho = 3.330 \text{ g cm}^{-3}$, see Chapter 4 section 4.4.3) the hydrogen content can then be expressed as weight proportion of H_2O

$$c = (M_{\text{H}_2\text{O}} \cdot A) / (\epsilon \cdot d \cdot \rho) \quad (2.7.3)$$

where $M_{\text{H}_2\text{O}}$ is the molecular weight of H_2O of 18.02 g mol^{-1} , A is the absorbance at peak height (around 3400 cm^{-1} for a broad band) and d is the thickness in cm. The molar absorption coefficient ϵ is $80 \text{ l mol}^{-1} \text{ cm}^{-1}$ (Leschik et al., 2004), calibrated for water in silicate glass. To convert from h wt. ppm H_2O to n H atoms / 10^6 Si atoms in Fo_{90} , a conversion factor of $n/h = 16.32$ was applied.

The sources for error in the determination of OH content in the olivine specimens are section thickness, olivine density, absorbance and the method / baseline correction.

2.7.3 Alternative procedure

Ideally, the water content in an olivine aggregate is measured per grain using a small aperture under unpolarised infrared light. If the aperture can be made small enough, this allows absorbance to be measured within single grains away

from ‘imperfections’ such as grain boundaries or inclusions. Unfortunately, this technique requires a specialised IR spectrometer which was not available at the time of this research.

2.8 Olivine density measurements

2.8.1 Overview

Olivine density measurements were performed on a Mettler H20 balance with a precision of 0.00001 g. The process involves the submersion of a sample in ethanol. An MgO standard of known density ($\rho = 3.584 \text{ g cm}^{-3}$) was used to calculate the density of ethanol followed by the determination of the sample density via

$$\rho_{\text{ethanol}} = \frac{\rho_{\text{MgO}}(M_{\text{MgO}}^{\text{air}} - M_{\text{MgO}}^{\text{ethanol}})}{M_{\text{MgO}}^{\text{air}}} \quad (2.8.1)$$

$$\rho_{\text{sample}} = \frac{\rho_{\text{ethanol}} \cdot M_{\text{sample}}^{\text{air}}}{M_{\text{sample}}^{\text{air}} - M_{\text{sample}}^{\text{ethanol}}} \quad (2.8.2)$$

where ρ is the density of the object in g cm^{-3} and M the mass in g.

The density measurements were only performed on a few selected specimens. The objective was to identify whether the hot-pressed specimens were compromised by porosity after high-temperature forced-oscillation testing. The specimens selected for these measurements were two attenuation specimens previously hot-pressed (sol-gel A1127 and San Carlos A1279). No density measurements were performed on pre-deformed specimens, which were assumed to be close to fully dense from deformation.

2.8.2 Procedures

Firstly, the standard and the olivine sample were weighed in air. The MgO standard was then fitted in a Pt cage suspended in a glass beaker containing

ethanol. The submersed MgO standard and cage were weighed together followed by weighing the cage alone. Likewise, the olivine sample was fitted inside the cage and suspended in ethanol for both to be weighed together and the empty cage alone. Each step described was repeated at least five times to obtain an estimation of the error in mass. Errors were propagated into a calculated density. Typically the uncertainty in the olivine density was of the order of 1 part in 10^{-4} .

*Truth uttered before its time,
is always dangerous.*

Mencius (371 – 289 BC)

3

Dislocation Recovery in Fine-grained San Carlos and Sol-gel Olivine polycrystals

3.1 Abstract

The rate of static dislocation recovery in Fo_{90} olivine has been studied under conditions of high temperature and controlled atmosphere in compressively deformed polycrystals hot-pressed from synthetic (sol-gel) and natural (San Carlos) precursor powders. The sol-gel olivine, containing a small fraction of orthopyroxene, was deformed to a final strain of 19% with a maximum differential stress of 266 MPa whereas the San Carlos specimen was deformed to 15% strain and 260 MPa differential stress. Small samples cut from these deformed materials were annealed under high-temperature, controlled atmosphere conditions, for different durations to allow partial recovery of the dislocation sub-structures. Oxidative-decoration of the microstructural features, followed by backscattered electron imaging at 5kV and image analysis, was used to determine dislocation density. The variation of dislocation density ρ with time t at absolute temperature T was fitted to a second-order rate equation, in integral form, $1/\rho(t) - 1/\rho(0) = kt$ with

$k = k_0 \exp(-E_a/RT)$. The activation energy E_a of the recovery process is 240 ± 43 kJ mol⁻¹ and 355 ± 81 kJ mol⁻¹ for sol-gel and San Carlos olivine polycrystals respectively. The measured rates are one to two orders of magnitude lower than those reported in previous studies on natural single crystal olivine. The difference may be explained by several factors such as high dislocation densities measurable from large areas at high magnification for the SEM and the technique used to estimate dislocation densities. Comparison between fine-grained sol-gel olivine and the coarser-grained San Carlos olivine aggregate did not indicate that grain boundaries play an important role in dislocation recovery, but the absence of grain boundaries might also have contributed to the high recovery rates previously measured for single crystals.*

3.2 Introduction

The rheological, diffusional, electrical and seismological properties of olivine, the most abundant mineral in the Earth's upper mantle, continue to be of primary interest for experimental research (Mei and Kohlstedt, 2000; Karato and Jung, 2003; Jackson et al., 2004; Du Frane et al., 2005; Dohmen et al., 2007; Spandler and O'Neill, 2010). Much recent work has focussed on the use of pure polycrystalline iron-bearing olivine, (Mg,Fe)₂SiO₄, synthesized from laboratory reagents (Jackson et al., 2002; McDonnell et al., 2002; Faul and Jackson, 2007). The benefit of using this synthetic equivalent to upper mantle rocks is the capability to work with polycrystals of controlled grain size and purity. However, using synthetic instead of natural material for experimental research requires re-examination of some of its material properties mentioned above. One important aspect was the evolution of dislocation configurations and dislocation recovery kinetics in olivine during static recovery at high temperature.

Climb-controlled annihilation of dislocations operates as pairs of dislocations

*Revised version published in *Physics and Chemistry of Minerals*, accepted 19 Nov. 2010 (<http://dx.doi.org/10.1007/s00269-010-0410-3>)

with opposite Burgers vectors meet via the diffusion of vacancies. The conditions favourable for dislocation climb occur at medium to high temperatures ($T > 0.4 T_m$). At these temperatures the lattice resistance to vacancy diffusion is sufficiently overcome. Dislocations can also anneal via cross-slip which occurs as a stress assisted process at high temperatures (Poirier and Vergobbi, 1978). Not all dislocations annihilate during recovery. Some dislocations glide and climb into stable positions in the crystal lattice leading, for example, to the formation of dislocation tilt walls. Overall, these recovery processes lead to a reduction in the free dislocation density, particularly during static recovery.

Dislocation recovery is dependent on many factors including impurity / vacancy concentrations, temperature, pressure, time, oxygen fugacity and possibly silica activity. Temperature, time and the activities of the chemical components of the system (e.g. f_{O_2} , a_{SiO_2} for olivine) are the most important controllable variables whereas pressure gives smaller contributions. The extent to which silica activity plays a role is not yet understood, but if dislocation recovery is diffusion controlled, there will be a dependency of the vacancy concentration (and point defects) on these activities (Stocker, 1978; Nakamura and Schmalzried, 1983).

Several studies on dislocation recovery have been carried out on natural olivine single crystals (Kohlstedt et al., 1980; Karato and Sato, 1982; Bai and Kohlstedt, 1992; Karato et al., 1993; Karato and Jung, 2003), but none so far have examined polycrystalline material derived from sol-gel and San Carlos olivine precursors. These two types of materials were deformed, annealed and analysed using the latest high resolution scanning electron microscope imaging techniques covering large areas. This study offers a systematic method for determining the true dislocation line lengths and hence the true dislocation density measured in a volume of material. The results offer a solid prediction for reduction of dislocation density (free dislocations) in sol-gel and San Carlos olivine for different temperatures and annealing times. The recovery rate of dislocations was calculated to determine the activation energy and the process controlling the rate of dislocation recovery.

Some constraints on the dislocation recovery mechanisms were evaluated.

3.3 Experimental procedure

Fully synthetic dry iron-bearing (Fo_{90}) olivine has been prepared via a solution-gelation (sol-gel) method detailed in studies by (Jackson et al., 2002; Faul and Jackson, 2007). Fe and Mg nitrates and TEOS (tetraethyl orthosilicate) were dissolved in an ethanol solution. Gel formation resulted from a change in pH and slow heating above room temperature. A series of steps followed including the dehydration of the gel, cold pressing, firing and grinding of olivine pellets at different temperatures and f_{O_2} . The last firing was done at 1400°C and f_{O_2} of 7×10^{-10} bar. To reduce the porosity, a stack of several fired pellets was wrapped in a $\text{Ni}_{70}\text{Fe}_{30}$ foil, loaded in a steel jacket and hot pressed at 1300°C and 300 MPa confining pressure for 24 hours. The resulting cylindrical specimen was a dense olivine aggregate of $2.7\text{ }\mu\text{m}$ mean grain size with 12% orthopyroxene (opx) as silica buffer and less than 1% porosity (specimen #6532 of Faul and Jackson 2007). The hot-pressed specimen was ground to cylindrical shape, fired at 1200°C and deformed stepwise in axial compression with progressively increasing stress including a final step involving 2% strain at 266 MPa differential stress. Total strain was about 19% (Faul et al., 2011). The deformation experiment was carried out at 1250°C and 300 MPa confining Ar pressure. The deformed specimen was recovered and sectioned from the upper end. Small 2 mm by 2 mm cubes were cut and put in epoxy for polishing in anticipation of the annealing experiments.

The San Carlos olivine samples were similarly prepared for annealing experiments. Inclusion-free grains were hand-picked from a suite of San Carlos olivine phenocrysts under the light microscope (see Tan et al. 2001). The grains were crushed, sieved and further size sorted by sedimentation from suspension to separate out the 10 to $38\text{ }\mu\text{m}$ grain size fraction. Pellets were cold pressed from the resulting San Carlos powders and given the same treatment of firing and hot pressing until a long cylindrical sample was prepared for compressive deforma-

tion. It was deformed at 1250°C and 300 MPa Ar confining pressure (run #6701) to 15% strain at 260 MPa differential stress. Likewise, a 2 mm slice was cut from one end of the cylindrical sample from which the annealing samples were prepared.

The dislocation recovery experiments were performed at temperatures of 1100–1500°C and annealing times of 3 – 50h under controlled oxygen fugacity conditions at atmospheric pressure utilising CO and CO₂ gas flow controllers. The oxygen fugacity and temperature were monitored in real time with an oxygen sensor and a thermocouple respectively and logged for the duration of the experiment. The f_{O_2} conditions were chosen to simulate the conditions during planned seismic wave attenuation experiments for which the specimen will be wrapped in an Ni₇₀Fe₃₀ foil (Jackson et al., 2002). Hence, a fixed mixture of 70% CO and 30% CO₂ approximating the Ni₇₀/Fe₃₀ – Fo₉₀ olivine redox buffer was used in all experiments, unless otherwise indicated (see Appendix F). This consideration was judged to be more important than isolating the effect of the oxygen fugacity on the rate of dislocation recovery as investigated by Karato and Sato (1982) where a slight negative trend was resolved but with large uncertainty. The oxygen sensor reported consistent f_{O_2} readings for the duration of the annealing experiments which match the calculated f_{O_2} values within 0.2 log units. For each annealing run, an olivine sample was placed in a platinum cup fitted with a grid of thin platinum wires on the bottom to ensure minimal contact with the cup and therefore minimal iron loss.

The temperature was ramped up at rates varying from 12°C/min up to 50°C/min for higher target temperatures ($\geq 1250^\circ\text{C}$). After a specified dwell time at the target temperature, the furnace was quenched at an estimated rate of 50°C/min to prevent any further dislocation recovery during cooling. Although this cooling rate was relatively slow, it was decided not to quench samples more rapidly in water in order to avoid major cracking and/or the generation of new dislocations. As a result, significant levels of dislocation recovery would have

occurred during the comparatively long ramp times and these must be taken into account particularly for the shortest experiments (0.5 – 5 hours). The emerging constraints on the temperature dependence of the rate constant allowed a correction for this effect.

The continuous temperature ramps were approximated as 50°C steps. Time spent at steps 0 – 1100°C was ignored, based on the observation that dislocation recovery rate was unmeasurable at such temperatures. Taking 50°C/min for heating and cooling ramp rates, positive corrections to the final dislocation density were made for $T \geq 1250^\circ\text{C}$ for sol-gel olivine, exceeding 0.4%, as follows: 1350°C (0.45%), 1400°C (0.71%), 1450°C (1.10%). For San Carlos olivine these corrections are: 1350°C (0.94%), 1400°C (1.91%), 1450°C (4.20%), 1500°C (8.27%). Note that the number of dislocations that anneal during heating will be larger than the number of disappearing during cooling. This was taken into account by approximating the final dislocation density of the samples as the initial dislocation density in order to calculate the correction during cooling. The San Carlos corrections were greater due to a higher dislocation recovery rate at high temperature.

Table 3.1: Summary of recovery experiments on sol-gel olivine.

Specimen ^a	T	f_{O_2}	t^b	2-D ρ_f^c	3-D ρ_f^d	Recovery	$\log(k, \text{m}^2\text{s}^{-1})^e$
		10^{-6}		10^{12}	10^{12}		
	$^{\circ}\text{C}$	Pa	h	m^{-2}	m^{-2}	%	
Fo-01	1450	75	3	0.6	4.1 ± 0.6	51 ± 8	-16.95
Fo-02	1400	23	3	1.2	5.1 ± 0.6	40 ± 9	-17.14
Fo-03	1350	6.4	3	1.7	6.4 ± 0.7	24 ± 11	-17.46
Fo-04	1300	1.7	3	2.2	6.5 ± 0.8	23 ± 12	-17.49
Fo-05	1300	1.7	8	1.7	5.6 ± 0.7	34 ± 10	-17.71
Fo-06	1300	1.7	16	1.5	5.0 ± 0.5	41 ± 8	-17.86
Fo-07	1300	1.7	50	1.7	3.5 ± 0.6	59 ± 8	-18.02
Fo-08	1200	0.086	50	1.4	3.9 ± 0.4	54 ± 6	-18.13
Fo-08*	1200	1.7	50	1.3	4.0 ± 0.4	53 ± 7	-18.11
Fo-09	1100	0.0029	50	2.7	7.1 ± 0.5	16 ± 10	-18.90

^a Average grain size after deformation is $(3.8 \pm 0.2) \times 10^{-6}$ m

^b Time at maximum temperature. Corrections to k were made for the duration of heating and cooling ramps (see text)

^c Final 2-D dislocation density (with $\rho_i = \sim 2.8 \times 10^{12} \text{ m}^{-2}$)

^d Final 3-D dislocation density (with $\rho_i = (8.5 \pm 0.9) \times 10^{12} \text{ m}^{-2}$)

^e Rate constant, k based on 3-D dislocation density calculation.

* Annealing experiment carried out away from the $\text{Ni}_{70}/\text{Fe}_{30}$ – Fo_{90} buffer

Table 3.2: Summary of recovery experiments on San Carlos olivine.

Specimen ^a	T	f_{O_2}	t^b	2-D ρ_f^c	3-D ρ_f^d	$\log(k, \text{m}^2\text{s}^{-1})^e$
		10^{-6}		10^{12}	10^{12}	
	$^{\circ}\text{C}$	Pa	h	m^{-2}	m^{-2}	
SC-01	1500	230	0.5	1.3	3.7 ± 0.6	-16.05
SC-02	1450	75	1	1.4	4.0 ± 0.9	-16.38
SC-03	1400	23	2	1.4	3.4 ± 1.6	-16.56
SC-04	1350	6.4	5	1.9	4.8 ± 1.2	-17.19
SC-05	1300	1.7	16	1.5	3.6 ± 1.1	-17.49
SC-06	1200	0.086	20	3.4	7.9 ± 1.8	-18.27
SC-07	1100	0.0029	50	4.1	9.1 ± 1.2	-18.90

^a Average grain size after deformation is $(13.0 \pm 0.47) \times 10^{-6}$ m

^b Time at maximum temperature. Corrections to k were made for the duration of heating and cooling ramps (see text)

^c Final 2-D dislocation density (with $\rho_i = \sim 5.2 \times 10^{12} \text{ m}^{-2}$)

^d Final 3-D dislocation density (with $\rho_i = (11.4 \pm 2.9) \times 10^{12} \text{ m}^{-2}$)

^e Rate constant, k based on 3-D dislocation density calculation

3.3.1 Imaging and determination of dislocation density

The decoration of dislocations was performed using a variation on the oxidative-decoration technique initially developed by Kohlstedt et al. (1976) and later used with the Scanning Electron Microscope (SEM) by Karato (1987). Earlier dislocation recovery studies, (Durham et al., 1977; Kohlstedt et al., 1980; Karato and Ogawa, 1982) used the oxidative-decoration technique for Light Microscope (LM) imaging. Our samples were prepared for Field Emission Scanning Electron Microscope (FE-SEM) imaging by grinding about 80 μm from the surface of the annealed specimen using silicon carbide, thereby removing any surface contamination. The grinding was followed by a fine polish with a 0.05 μm alumina slurry. The polished surfaces were then oxidised at 900 $^{\circ}\text{C}$ for 45 minutes. After

oxidation, another polish was performed for 10 minutes using the same alumina slurry, previously determined to guarantee the best result. The iron-oxide surface coating was removed ($< 1\ \mu\text{m}$) and the most decorated region of dislocations was about 2 - 6 μm deep. The samples were cleaned and observed under the LM at the highest magnification ($1,000\times$) to check that the grain boundaries and dislocations are clearly visible.

The Hitachi 4300 SE/N FE-SEM was used to image the decorated dislocations at an acceleration voltage of 5 kV. Working distance used was 10 mm, the condenser lens was set at 6 (equivalent to $\sim 10^{-10}$ A specimen irradiation current), aperture was set to 100 μm , the emission current was 43 μA , the imaging current was about 0.4 nA and gun brightness was set to 3. Images of 2048×1536 pixels were captured in a rectangular grid pattern. In order to establish optimal imaging conditions for dislocation density measurements, selected regions were imaged at different magnifications. From $3,000\times$ to $8,000\times$ magnification, the apparent dislocation density increased by 75% as a higher magnification allowed previously overlapping dislocations to be resolved into distinct lines. Beyond $8,000\times$ there was little change. So this magnification was used for all subsequent dislocation density measurements.

The FE-SEM images were prepared for dislocation density measurements using public domain software called ImageJ[†]. Several stages in the procedure are shown in Fig. 3.1. High contrast images (Fig. 3.1a) were inverted (Fig. 3.1b) and processed with a threshold filter. The threshold operation converts a grey-scale image to a binary image (Fig. 3.1c) and the threshold value was manually adjusted to produce sharp dislocation images which remained continuous. This adjustment was required because, despite care taken to impose standard imaging conditions, fluctuations in the electron beam produced slightly different image brightness and contrast levels between weekly sessions. Two noise filters were used to remove all groups of five or fewer pixels. Such small clusters of pix-

[†]<http://rsbweb.nih.gov/ij/>

els were not considered to be representative of dislocations but rather specks of contamination on the sample surface for instance.

In the process of distinguishing individual dislocations from other features, in this study dislocation walls were excluded. Walls were identified either as relatively long, often straight, solid lines crossing a grain or as arrays of solitary dislocations. The dislocations were highlighted using a colouring tool (Fig. 3.1d) and all other structures (e.g. grain boundaries, dislocation walls, pores, etc.) were removed using a colour filter (Fig. 3.1e). Lastly, the porosity and opx grains were manually highlighted and their areas subtracted from the total area to be counted (Fig. 3.1f).

From such processed images containing only free dislocations, dislocation density was estimated by invoking an add-on called 'Bézier curve ROI' for imageJ[†]. It fits a parametric curve to the end points of an object and any undulations in between. This way, the 'projected' length of every individual dislocation was calculated – the sum of which gives the cumulative length of all dislocations.

The 'projected' dislocation length was important to know, because the dislocation density can be quantified in two ways. The first method involves counting the number of dislocations N imaged. When the number of dislocations was divided by the area, the uncorrected 2-D dislocation density was obtained (see Fig. 3.2a). However, dislocations are line defects and hence the 'true' dislocation density should be estimated by measuring the cumulative length of dislocation lines and dividing by the volume. This was referred to as the 3-D dislocation density, considered to be more accurate than a simple dislocation count over an area. However, it should be noted that previously the 3-D dislocation density might have been approximated as $\rho_{3-d} = 2N/A$ (see section 3.5.2) (Karato, 2008).

To obtain the 3-D dislocation density from the FE-SEM images, the true dislocation line length l_0 must be calculated from the projected length l and the depth of information d . This depth is not the electron penetration depth (see

[†]<http://www.optinav.com/Measure-Roi.htm>

Appendix G), but rather the depth from which backscattered electrons were returned to the backscatter detector situated above the sample. Fig. 3.2 highlights the difference between using different accelerating voltages and how l_0 was calculated. In this study, $d = 0.095 \mu\text{m}$ was used at 5 kV (again, see Appendix G). The 3-D dislocation density was calculated as the sum of l_0 over the volume of material (area times d) expressed as

$$\rho_{3-D} = (1/A) \sum \left[\sqrt{1 + (l/d)^2} \right] \quad (3.3.1)$$

where A is the total area imaged.

Total areas imaged for each dislocation density measurement were around $4000 \mu\text{m}^2$. Up to four such areas were measured per sample. The uncertainty in the data was obtained by dividing the total area imaged per sample into small areas each of about $300 - 500 \mu\text{m}^2$. The dislocation density was then calculated as the mean of those for all areas. The standard error was given by twice the standard deviation over the square root for the total number of measurements. Error propagation was used to carry the uncertainty from the dislocation density into the rate constant, k . Fig. 3.4 shows an indication of error in the measurements of k for both sol-gel and San Carlos olivine. The large error bars towards lower temperature reflect the difficulty of determining the difference between the initial and final dislocation density for only a small reduction.

Alongside characterizing FE-SEM images for dislocations, a lower magnification ($\times 1,000$) light microscope was also used in this study. Thin sections of two oxidised samples from the San Carlos olivine samples were prepared, one from the as-deformed material and the other from a sample annealed at 1200°C . Four areas, each $6640 \mu\text{m}^2$, were analysed per sample. Dislocations were then hand traced and counted to produce uncorrected 2-D dislocation densities for the deformed and annealed sample (SC-06). The 3-D dislocation density, from the light microscope images, was calculated using the thickness of the thin section, determined by focusing successively on the top and bottom surface of the section.

The difference in focal position multiplied by the value of the refractive index of Mg-rich olivine (~ 1.65) yields the thickness of the thin section – confirmed by the birefringent colours of olivine throughout the section.

The grain size of the original deformed sol-gel sample #6532 was determined from the FE-SEM images (as shown in Fig. 3.1a). Unfortunately, the grain boundaries can be discontinuously decorated by oxidation creating potential for two or more grains to be counted together, in some instances, leading to an over-estimation of the grain size. Therefore, the grain boundaries were hand traced to ensure accurate grain size determination. The grain size of the original deformed San Carlos sample #6701 was determined via Electron Backscatter Diffraction (EBSD). The images containing grain size information were processed using ImageJ which can treat single grains as particles. The area of every grain was measured and the equivalent diameter was determined. A correction (multiplication by a factor $4/\pi$, see Jackson et al. 2002) was applied to account for sectioning bias.

3.3.2 Recovery kinetics

There are a number of equations that describe particular recovery mechanisms based on distinct rate-limiting steps. These recovery mechanisms include diffusion controlled dislocation glide, dislocation cross-slip, dislocation climb controlled by vacancy bulk diffusion or core diffusion or solute drag. The rate of dislocation recovery by dislocation glide, climb or cross slip can be expressed as

$$\sqrt{(\rho_f/\rho_i)} = [1 + t/\tau]^{-m} \quad (3.3.2)$$

where ρ_i and ρ_f are the initial and final dislocation densities respectively. τ is a relaxation time parameter containing an Arrhenius term involving the activation energy for diffusion or cross slip. In Eqn. 3.3.2, $m = 1$ for dislocation glide recovery with lateral jog drift, $m = 1/2$ for climb controlled by vacancy bulk

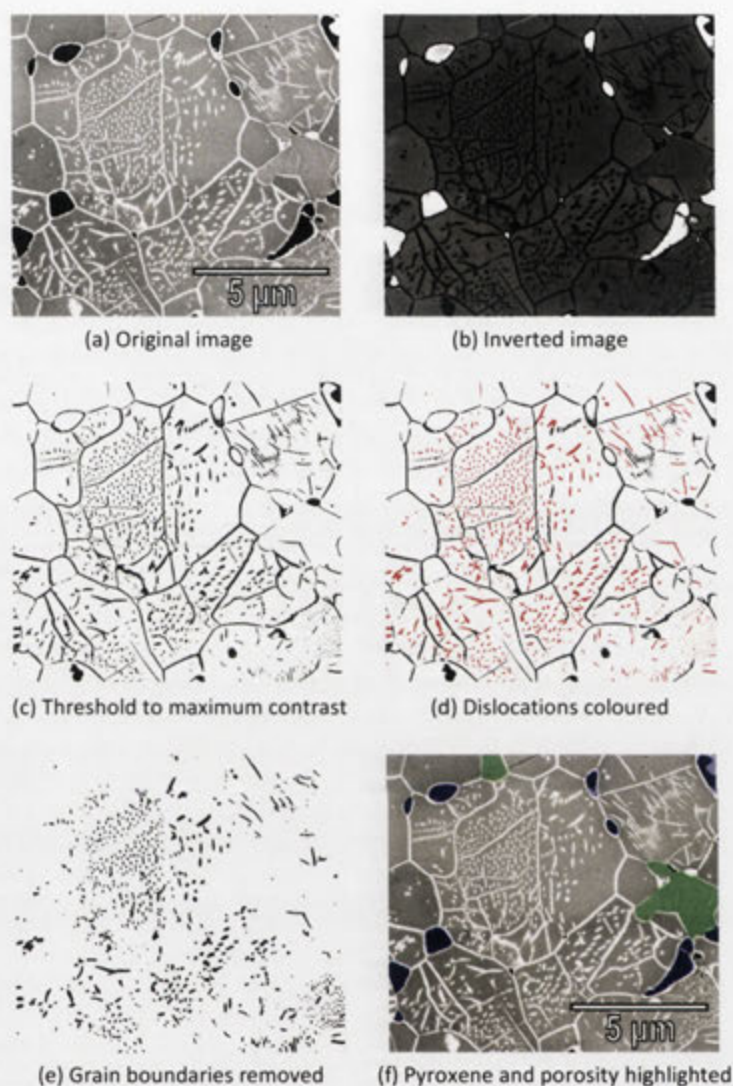


Fig. 3.1: Procedure for obtaining the uncorrected 2-D dislocation density of the as-deformed olivine specimen. Panels (a) to (e) show how the original SEM image was inverted, processed with a contrast filter of an appropriate threshold, manually coloured with a colour replacement tool so that a colour threshold filter can be applied to remove the grain boundaries. Panel (f) shows the manual highlighting of the porosity (dark blue) and pyroxene grains (green) that were excluded from the calculated area. To get the 3-D dislocation density result, the line lengths were measured in panel (e) instead of the number of dislocations (see Eqn. 3.3.1)

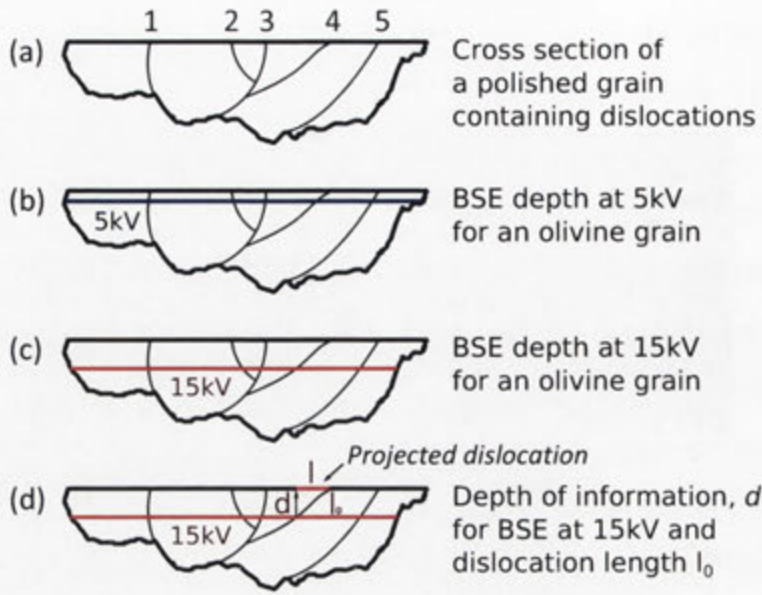


Fig. 3.2: Method used to determine the depth of information, d and hence the true dislocation line length, l_0 . (a) The uncorrected 2-D dislocation density was determined by counting the number of dislocations over an area. (b – c) d is more shallow with a lower accelerating voltage. (d) l_0 was determined from d and l respectively

diffusion, $m = 1/4$ for climb controlled by vacancy core diffusion and $m = -1$ for cross slip (Nes, 1995). The equation was based on quantifying the growth of the network of increasingly widely spaced dislocations and jogs.

Alternatively, one can express dislocation recovery kinetics empirically using the general n th-order dislocation recovery rate equation

$$\frac{d\rho}{dt} = -\rho^n k \quad (3.3.3)$$

where k is the rate constant. This integrates for $n \neq 1$ to

$$1/\rho_f^{n-1} - 1/\rho_i^{n-1} = kt(n-1) \quad (3.3.4)$$

and for $n = 1$ to

$$\rho_f/\rho_i = e^{-kt} \quad (3.3.5)$$

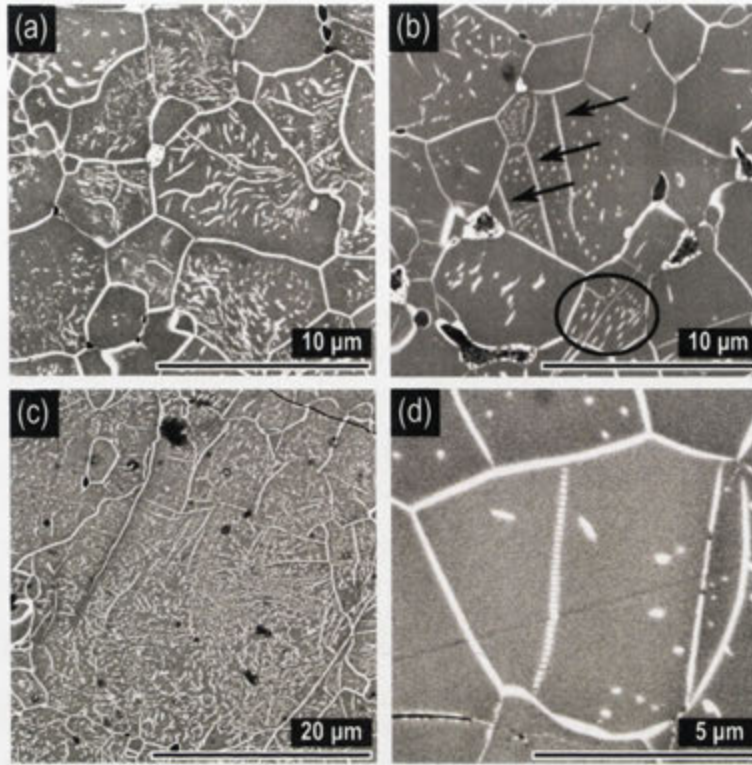


Fig. 3.3: (a) Backscattered image of a section of the deformed sol-gel olivine. (b) Backscattered image of the same material after annealing at 1300°C for 50 hours. Arrows indicate dislocations organised into walls. The circle indicates a region with many parallel dislocation lines. (c) A typical grain in the San Carlos olivine packed with closely spaced dislocations prior to annealing. (d) Example of a dislocation tilt wall sub-structure after annealing at high temperature with dislocations separated by just 130 nm

The rate constant k is given by

$$k = k_0 \exp\left(\frac{-E_a}{RT}\right) \quad (3.3.6)$$

where E_a is the activation energy for dislocation recovery, R is the gas constant, T is the temperature and k_0 is a constant. The results of the annealing experiments are summarized in Table 3.1 and 3.2.

3.4 Results

3.4.1 Dislocation microstructures

Overall, there was no significant increase in grain size through prolonged high temperature annealing, indicating a stable grain-scale microstructure for both the sol-gel and San Carlos olivine samples. The average grain size for the deformed sol-gel olivine specimen is $3.8 \pm 0.2 \text{ } \mu\text{m}$ (795 grains) and $13.0 \pm 0.47 \text{ } \mu\text{m}$ (1275 grains) for deformed San Carlos olivine. The porosity ranges from 2 – 3% for the deformed and annealed sol-gel samples except for the highest temperature annealing ($> 1400^\circ\text{C}$) which caused an increase in porosity to as much as 14% throughout the sample. The same occurred at very high temperature in the coarser grained annealed San Carlos samples but was less pronounced, except at the edges of the samples. The porosity was generally between 2 – 3% but for samples SC-02 and SC-01 (at 1450°C and 1500°C) porosity increased to 5% in the central region.

Fig. 3.3 shows the typical arrangements of dislocations before (a) and after annealing (b,d) in sol-gel olivine. Dislocation density was significantly lower in the annealed samples and the grain boundaries appear to have straightened as well. Fig. 3.3a shows that most grains contain a large concentration of dislocations with some preferential azimuthal orientation within each grain. Dislocation alignment was more pronounced after annealing at high temperature (Fig. 3.3b). Fig. 3.3d shows that most dislocations have been annihilated with the remainder having moved into stable sub-structures. A few olivine grains contain almost no dislocations. The opx grains were generally dislocation-free, possibly the result of high temperature phase transformation. Fig. 3.3c shows a large olivine grain with a high dislocation density typical of the deformed San Carlos specimen. The initial dislocation density for San Carlos is $11.4 \pm 2.9 \text{ } \mu\text{m}^{-2}$, a little higher than that for sol-gel olivine of $8.5 \pm 0.9 \text{ } \mu\text{m}^{-2}$.

When observing isolated dislocation segments inside grains one must keep in mind that the imaged area is a representation of a 3-D microstructure. Individual

dislocation lines must terminate at an impurity, pore space, grain boundary or another dislocation. However, the depth of information at 5 kV was sufficiently low (see Appendix G) that only the near-surface parts of plunging dislocations can be seen. Dislocation walls as seen in Fig. 3.3 are less abundant in the sol-gel material (both deformed and annealed) than in the San Carlos samples with larger grain size (e.g. Fig. 3.3c for a large grain in the as-deformed San Carlos specimen).

Information concerning the slip plane and direction of slip for dislocations cannot be determined from the FE-SEM images.

3.4.2 Dislocation recovery rate

The initial dislocation density ρ_i , measured on each of the deformed specimens, was found to be uniform within experimental error (Tables 3.1 and 3.2). These values of ρ_i , in combination with those (ρ_f) measured on the annealed specimens, were used to calculate the second-order rate constant k (Eqn. 3.3.4) with $n = 2$. The rate constants thus estimated for the sol-gel and San Carlos olivines are presented in Tables 3.1 and 3.2, respectively, and the recovery rates based on the 3-D dislocation densities are plotted against reciprocal absolute temperature in Fig. 3.4. Note that the 3-D dislocation densities reported here are comparable to values measured in similar polycrystalline olivine (Karato and Jung, 2003) but are higher by one order of magnitude compared to single crystal olivine measurements (Durham et al., 1977; Karato and Ogawa, 1982; Karato et al., 1993).

This study used the second-order rate law as previously employed by Kohlstedt et al. (1980) and Karato and Ogawa (1982). The physical reason for choosing a second order rather than first order rate law is that second order kinetics imply dislocation-dislocation annihilation rather than dislocation adsorption at grain boundaries, a process which would be described by first order kinetics. The former, rather than the latter was believed to have occurred judging from microstructural observations. Conveniently, the second-order rate constant has the

same unit as the diffusivity (m^2s^{-1}), facilitating a comparison between the two quantities.

Fig. 3.4 shows broadly similar recovery rates for sol-gel (with $E_a = 240 \pm 43$ kJ mol^{-1} , $k_0 = 1.73 \times 10^{-10} \text{ m}^2\text{s}^{-1}$) and San Carlos olivine with some divergence at high temperature, indicating a larger activation energy (355 ± 81 kJ mol^{-1} , $k_0 = 1.85 \times 10^{-6} \text{ m}^2\text{s}^{-1}$) for San Carlos olivine. The uncertainty was calculated for both the error in the regression as well as for each data point. The four data points for the sol-gel at 1300°C are measurements for different annealing durations (3,8,16,50 hours). The two overlapping data points at 1200°C and 50 hours annealing are for different CO/CO_2 gas mixes and therefore different oxygen fugacities. While there might be an effect of the oxygen fugacity on the dislocation recovery rate, for these experiments the effect will most likely be within experimental uncertainty. The lone data point (green) was obtained from a light microscope study of the decorated San Carlos samples (deformed and annealed, SC-06) and indicates a significantly higher ‘apparent’ rate constant.

Fig. 3.5 provides a complementary view of the same data and the second-order recovery model for the sol-gel olivine specimen. The percentage of recovery was calculated for this model based on parameter values for k_0 and E_a determined via Fig. 3.4. Each dashed curve represents a particular duration of annealing. This figure highlights the implications of the dislocation recovery model for the stability of dislocation microstructures during prolonged high-temperature testing.

The two data points at 1200°C ($10^4/T = 6.8$) and 50 h in Fig. 3.5 are those measured at different oxygen fugacity levels (Fo-08 and Fo-08* in Table 3.1). For the data point closer to the 50 hour curve the gas mixture was varied to achieve the same f_{O_2} as for the data point at 1300°C . All other data were obtained under f_{O_2} conditions given by a constant gas mix of 70% $\text{CO}/30\%$ CO_2 . The 1200°C results have been taken to demonstrate that the effect of f_{O_2} was small enough to be within the uncertainty of the measurements (of about $0.1 \times 10^{-14} \text{ m}^2\text{s}^{-1}$ for 1.3 orders of magnitude difference in oxygen fugacity).

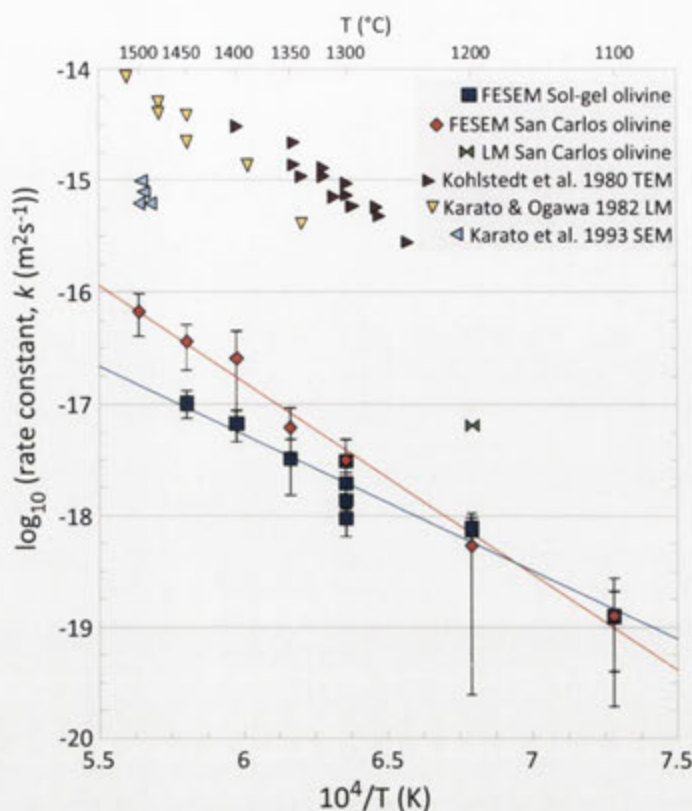


Fig. 3.4: Rate constant versus the reciprocal of temperature (3-D calculation, Table 3.1 and 3.2). Light Microscope data; Karato and Ogawa (1982), Karato et al. (1993) and this study. TEM data; Kohlstedt et al. (1980) and Karato et al. (1993). SEM data; Karato et al. (1993). All previous studies used San Carlos single crystals. FE-SEM data; this study (polycrystalline sol-gel and San Carlos olivine). All the data was obtained under atmospheric pressure. A linear regression line was fitted to each of the data sets from this study

3.4.3 Uncertainty in measured dislocation density

High dislocation density leads to overlapping dislocations that make counting difficult (although this was improved for imaging at 5kV relative to higher accelerating voltages) and increases uncertainty in dislocation density measurements. In addition, scatter in the data results from the heterogeneity in the dislocation distribution between grains. Fig. 3.3a, for example, shows a representative area where a few grains contain no dislocations. The proportion of dislocation-free grains increases with increasing dislocation recovery at higher temperature, thereby enhancing the uncertainty in the data (see Fig. 3.4).

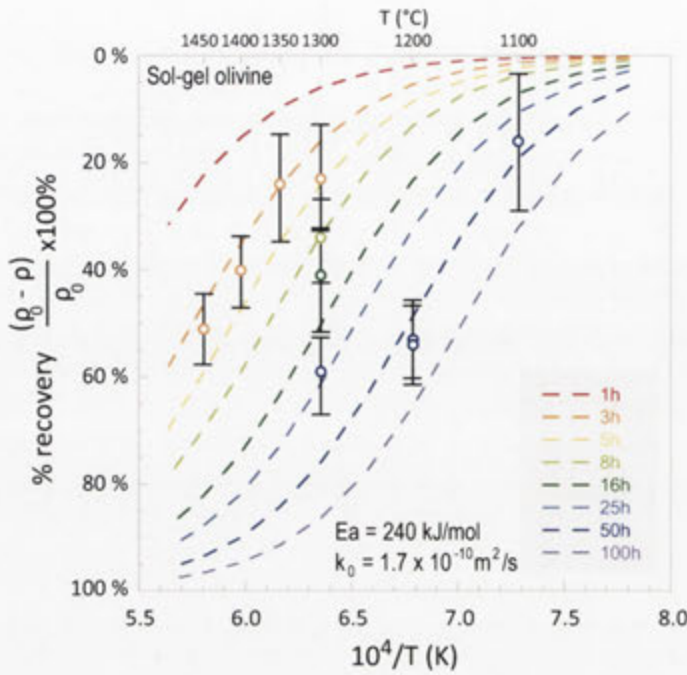


Fig. 3.5: Percent recovery versus the reciprocal of temperature. The annealing time is indicated with a different colour for each curve (calculated via Eqn. 3.3.6). Each data point is correspondingly colour coded accordingly to its annealing time. All the data was obtained under atmospheric pressure only

This study found that larger areas for dislocation measurements provide more robust results but come at the cost of more time and resources. During an initial calibration, where the dislocation density was measured over different areas at a constant magnification of 8,000 $\times$, it was found that the dislocation density for total areas of 10,000 to 20,000 μm^2 did not significantly change. However, dislocation density measurements for areas down to 500 μm^2 show a variability in dislocation density to more than $\pm 2 \mu\text{m}^{-2}$.

As a final check on image processing using the various contrast filters, dislocations in one grain were counted manually at 11,000 $\times$ magnification on a raw image. The dislocation density using the method described earlier at 8,000 $\times$ was about 15% less than the manual recount, comparable with the statistical uncertainty of 7 – 18% in Table 3.1, therefore the tabulated results may be a slight under-estimate, but acceptable given the practical limitation that twice as many images need to be processed for 11,000 $\times$ magnification. This slight increase in

dislocation density will not yield a statistically significant decrease in the rate constant.

3.5 Discussion

3.5.1 Microstructural interpretation of dislocations

The deformed fine grained sol-gel olivine (and to a lesser extent San Carlos olivine) shows some grains devoid of dislocations even before annealing. These grains may have been in an orientation unfavourable for internal deformation by dislocation creep, reducing the number of generated dislocations. Hence, it cannot be said with certainty whether annealing can allow the elimination of all dislocations from a grain.

The presence of isolated dislocation walls after high temperature annealing suggests that these were stable sub-structures unlikely to undergo further recovery (see Fig. 3.3d). Dislocation walls may act as attractors for annihilation of free dislocations of opposite sign, especially if these sub-structures develop early on during recovery or have already formed via dislocation creep during deformation. Fig. 3.3b, however, shows several isolated dislocations persisting in the region between dislocation walls after 50 hours at 1300°C. Occasionally, a network of dislocation walls was observed in the annealed samples. Individual dislocations in walls can be resolved at the highest magnification of the FE-SEM in some rare cases. Typically, the BSE image quality does not improve beyond 20,000 $\times$ magnification. Dislocations making up these walls were typically less than 50 nm apart. More widely spaced dislocations occur in walls that must be of lower misorientation angle. Dislocation wall structures indicate recovery and reorganisation of dislocations during annealing. When the spacing between dislocations in a dislocation structure becomes too small, it becomes indistinguishable from a grain boundary in the backscattered images. As mentioned before, such structures were not included in the dislocation density estimation.

Fig. 3.3b demonstrates some other dislocation sub-structures which were not seen in the original deformed material. The observable decrease in the average number of curved dislocations between figures 3.3a (e.g. centre grain), 3.3b and 3.3d in statically annealed samples gives an indication of significant dislocation climb (Lui and Evans, 1997).

3.5.2 Comparison with other work

In Fig. 3.4, the rate constant $k = (1/\rho_f - 1/\rho_i)/t$ calculated from the change in dislocation density for both sol-gel and natural San Carlos polycrystalline olivine is lower by 1 – 2 orders of magnitude than for single crystal San Carlos olivine (Fo₉₂) from a LM study by Karato and Ogawa (1982) and a single crystal TEM study by Kohlstedt et al. (1980). k is lower by over one order of magnitude compared to a small set of data obtained by Karato et al. (1993) for single crystal San Carlos olivine. The activation energy calculated for dislocation recovery in single crystal San Carlos olivine is 398 ± 59 kJ mol⁻¹ (Karato and Ogawa, 1982) which compares quite well with that for San Carlos olivine determined over a wider temperature range in this study. For the study of single crystal San Carlos olivine by Karato et al. (1993) no activation energy was obtained but Kohlstedt et al. (1980) obtained a value of 328 ± 31 kJ mol⁻¹. In addition, Yan (1992) obtained an activation energy of 291 kJ mol⁻¹ in natural olivine, whereas a much higher value of 586 ± 126 kJ mol⁻¹ for a natural deformed peridotite was obtained by Goetze and Kohlstedt (1973), not plotted in Fig. 3.4.

The dislocation density for other studies was obtained using different techniques. The LM was used for imaging in the study by Karato and Ogawa (1982). The LM has the limitation that it can image only up to a magnification of 1,000 $\times$ and therefore with a high dislocation density, many dislocations were potentially missed. TEM imaging carried out by Kohlstedt et al. (1980) was done at high magnification (10,000 $\times$) but only on small areas (24 times 54 μm^2), complicating the determination of a robust average for a markedly heterogeneous dislocation

distribution. In only two of the previous studies was the 3-D dislocation density estimated via measurement of dislocation line lengths. This has no relevance to special instances where dislocations were imaged end-on (as in suitably oriented single crystals (Karato et al., 1993)).

The San Carlos polycrystal of the present study has a larger grain size than the sol-gel olivine and may likely contain higher concentrations of trace elements due to its natural origin. However, we measured a rate constant broadly consistent with that of sol-gel olivine. Only at high temperatures is k systematically different indicating a somewhat more strongly temperature dependent rate constant for San Carlos olivine.

Since a similar rate constant was observed for olivine of larger grain size, prepared from the natural precursor, a single measurement of the rate constant was performed based on images recorded in a petrographic light microscope. Uncorrected 2-D dislocation analysis of the thin section photos using the hand trace method described in section 3.3.1 produced dislocation densities of 8.0×10^{10} and $7.7 \times 10^{10} \text{ m}^{-2}$ respectively for the deformed and annealed sample, SC-06 with k equal to $5.6 \times 10^{-18} \text{ m}^2 \text{ s}^{-1}$ (see plotted value in Fig. 3.4). Calculating the 3-D dislocation density, using a measured thickness of the thin sections as $\sim 16.5 \text{ }\mu\text{m}$ for the deformed sample and $\sim 9.9 \text{ }\mu\text{m}$ for SC-06, yields an apparent recovery rate for the light microscope study of $5.0 \times 10^{-18} \text{ m}^2 \text{ s}^{-1}$, comparable with the uncorrected 2-D value, indicating that a much smaller depth of information determined for FE-SEM dislocation imaging has a greater effect on calculating k (see Appendix G).

The rate constant of dislocation density kinetics for the light microscope study is one order of magnitude higher than the recovery rate from the analysis using high magnification FE-SEM images. This suggests that the difference lies in the capacity of the latter technique to resolve a higher number of individual dislocation structures. The higher FE-SEM dislocation densities, which apply equally to ρ_f and ρ_i , translate directly into lower recovery rates through Eqn.

3.3.4.

It is interesting to note that the rate constant from the SEM study by Karato et al. (1993) is lower by one order of magnitude than the Karato and Ogawa (1982) LM study, also consistent with the notion that more dislocations were counted at higher resolution. Karato et al. (1993) propose that their focus on a particular slip system was the cause for a lower recovery rate and that the thermodynamic environment may also be important, including silica activity which was controlled by an opx buffer in their study. The former might be possible, but should be investigated further. The latter is unlikely, however, because opx buffering was also used in the present study for sol-gel olivine. The silica activity was not deliberately fixed for the San Carlos olivine of the present study. However, microstructural analysis does not indicate the presence of MgO and previous analysis showed some 1 – 2% opx in the same material (Jackson et al., 2002). Therefore, opx-buffering may have contributed to lower recovery rates in Karato et al. (1993) and in our study but was unlikely to be the only contributing factor.

The difference between an uncorrected 2-D and a corrected 3-D dislocation density calculation from Table 3.1 and 3.2 would produce up to half an order of magnitude increase in recovery rate if k had been calculated from 2-D densities. This may contribute towards an explanation for a lower rate constant in this study using the 3-D results. Unfortunately, previous results cannot be simply recalculated to a 3-D result because the raw images were required to measure the dislocation lengths (see Appendix I).[§] The depth of information will also be different depending on the accelerating voltage used in SEM or section thickness for LM and TEM studies. It is not known whether previous dislocation densities were given a geometric correction of the type $\rho_{3D} = 2(N/A)$ where N is the number of dislocations and A the area (Karato, 2008). Nonetheless, this correction is only an assumption because dislocations in olivine are not randomly oriented but

[§]Raw images from this study are available upon request

confined to the slip systems in olivine on which they multiply. The large errors produced by this calculation can be reduced if the total length of dislocation lines for a given volume is measured (Karato and Jung, 2003), as done in this study.

Finally, Karato and Sato (1982) have shown there may be an effect of oxygen fugacity on dislocation recovery. However, results from two experiments labelled Fo-08 and Fo-08* (Table 3.1 and 50 hour curve for 1200°C in Fig. 3.5), separated by over one order of magnitude in f_{O_2} are indistinguishable within uncertainty.

3.5.3 Recovery mechanisms

Static dislocation recovery is driven by the reduction in the strain energy within and possibly between grains. It is commonly believed that dislocations in olivine can migrate at high temperature via two mechanisms – glide, climb or a combination of both – when no external stress is applied. Obstacles to dislocation glide may be interstitial atoms, trace elements or other dislocations, all of which could need to be overcome by dislocation climb requiring the diffusion of Si, O, Mg, and Fe but controlled by the slowest species, Si (Dohmen et al., 2002). When the temperature is high enough, dislocations of opposite sign will be attracted to each other and climb over obstacles to annihilate. Either climb or glide is the rate limiting step controlling dislocation recovery.

The dislocation structures observed in the deformed polycrystalline olivine samples, #6532 (sol-gel) and #6701 (San Carlos), are quite similar to structures seen in natural single crystal olivine that underwent dislocation creep (e.g. Durham et al. 1977; Bai and Kohlstedt 1992). Some of the recovered dislocation sub-structures such as dislocation walls are also comparable to those in annealed specimens from previous studies (Bai and Kohlstedt (1992, Fig. 11) and Karato and Ogawa (1982)). The similarities indicate that the dislocation creep and recovery mechanics are comparable for all olivine materials.

The activation energies for dislocation recovery determined in all the investigations of San Carlos olivine are very similar around 350 kJ mol⁻¹ whereas sol-gel

olivine has an activation energy of $\sim 240 \text{ kJ mol}^{-1}$ and therefore a less pronounced temperature dependence on the rate constant. However, this lower activation energy is not associated with a higher rate constant (Fig. 3.4). With an absence of impurity defects in our sol-gel olivine (c.f. Al, Mn and Cr as detected in San Carlos olivine (Spandler and O'Neill, 2010)), one might have expected greater mobility of dislocations and a larger value for the rate constant during annealing compared with San Carlos, especially towards lower temperatures where glide is more important. Since this was not observed, impurity defects were apparently not limiting the rate of dislocation recovery.

Recovery could also depend on whether dislocations are dominated by an edge or screw character, affecting dislocation recovery kinetics via differences in the nature and behaviour of jogs and kinks. A straight dislocation segment also undoubtedly behaves differently from a curved segment. In most cases, dislocations were of mixed nature and at least gently curved. Only in one single crystal study was a particular slip system in olivine controlled and investigated (Karato et al., 1993). The other single crystal data potentially involve multiple slip systems. It is not clear, however, why the dominant easy slip system in olivine, (010)[100], would actually contribute to a lower rate constant as measured by Karato et al. (1993).

One other factor which could relate to the low measured recovery rates is the small grain size of our polycrystals. The way dislocations interact with each other in the presence of grain boundaries remains largely unknown. However, small grain size might affect the overall dislocation mobility via back-stress as embedded in the Hall-Petch relation (see e.g. Hull and Bacon 2001). In addition, plastic deformation is characterised by the heterogeneous distribution of dislocations in grains and their participation in strain avalanches (Csikor et al., 2007). Consequently, understanding dislocation interactions with grain boundaries is critical to understanding crystal plasticity and recovery. The dislocation recovery mechanism in the relatively fine-grained materials in this study might involve

a higher internal resistance from grain boundaries that complicates glide, climb and annihilation of dislocations not seen in single crystal olivine experiments. For instance it could be that residual internal stresses act as long range obstacles to easy dislocation mobility within grains during annealing at high temperature. In addition, dislocation arrangements (e.g. tilt walls) within grains also tend to screen long range stress fields (Moretti et al., 2008). Lastly, the high initial dislocation densities in this study could conceivably reduce the recovery rate by initial entanglement of dislocations.

3.5.4 Dislocation recovery related to diffusion experiments and creep

If dislocation climb is the dominant recovery process, then dislocation recovery kinetics would be related to diffusion rates of the appropriate species in olivine. Studies of electrical conductivity in olivine suggest that magnesium vacancies and small polarons ($\text{Fe}_{\text{Mg}}^\bullet$) are the major charge carriers and that silicon interstitials are the slowest diffusing species (Smyth and Stocker, 1975; Roberts and Tyburczy, 1993; Du Frane et al., 2005). Single crystal olivine diffusion experiments have shown that Fe-Mg diffusion (Jaoul et al., 1995; Chakraborty, 1997; Dohmen et al., 2007) is the fastest followed by oxygen (Jaoul et al., 1980; Reddy et al., 1980; Gerard and Jaoul, 1989), and finally silicon (Houlier et al., 1988, 1990; Dohmen et al., 2002). For temperatures between 1300 – 1500°C. Dohmen and Chakraborty (2007) reports Fe-Mg diffusion coefficients ($\log_{10}D$, m^2s^{-1}) between -16 and -15 and Dohmen et al. (2002) reports oxygen diffusion coefficients ranging between -19.5 and -18, whereas silicon diffusion coefficients range between $\log_{10}D = -21.5$ and -19.5. Karato and Ogawa (1982) suggested that the rate constant, k can be related to the diffusivity, D as

$$k = \alpha' D \quad (3.5.1)$$

where α' is approximately 300 (Karato and Ogawa, 1982, see, appendix). The assumptions for this derived equation are that all dislocations will form dislocation dipoles and annihilate (second order kinetics), that the mean distance of dislocations is proportional to the mean distance of the dislocation pair, that a Weertman climb-controlled creep mechanism operates and that jogs are saturated with vacancies. With this equation, our measurements of k indicate diffusion coefficients close to that for oxygen. At 1400°C a value of -21.25 ($\log_{10}D$, m^2s^{-1}) for the silicon diffusion coefficient would yield an equivalent value of -18.77 for the rate constant of dislocation recovery, roughly two orders of magnitude slower than observed in this study.

In addition, other studies have established that E_{creep} is similar to E_{diff} for Si diffusion in dry single crystal olivine (e.g. $E_{\text{creep}} = 523 \text{ kJ mol}^{-1}$ Goetze (1978) and $E_{\text{diff}} = 529 \text{ kJ mol}^{-1}$, Dohmen et al. 2002). This activation energy is much higher than the one obtained from static dislocation recovery experiments in the present and previous studies. A lower value of $358 \pm 28 \text{ kJ mol}^{-1}$ was reported for silicon diffusion in water-bearing olivine with $\sim 45 \text{ ppm H}_2\text{O}$ (Costa and Chakraborty, 2008). However, Fourier Transform Infrared Spectroscopy (FTIR) showed no water was present in the sol-gel olivine, whereas $423 \text{ H}/10^6 \text{ Si}$ ($\sim 26 \text{ wt. ppm H}_2\text{O}$) was measured in the San Carlos specimen. Overall, this complicates any comparison between static recovery and creep in olivine and the mechanism for dislocation recovery remains unclear.

3.6 Implications for high temperature experimentation

This study was motivated by the need to have a stable dislocation density during prolonged high temperature forced oscillation measurements of seismic wave attenuation on olivine polycrystals hot-pressed from either sol-gel or San Carlos derived precursor powders. We conclude that the temperature should not exceed

1100°C if the dislocation density is to be maintained within 20% throughout prolonged (~ 50 h) attenuation measurements (Fig. 3.5). Previous high temperature experiments of dislocation damping in pre-deformed single crystal forsterite by Gueguen et al. (1989) may have been complicated by substantial dislocation recovery. For example, a 20% reduction in dislocation density would be expected after only 1 hour at 1400°C (Fig. 3.5), the maximum temperature for the attenuation experiments of (Gueguen et al., 1989). Larger rate constants for the single crystal olivine (comparable to Karato et al. (1993) would imply even more rapid changes in dislocation density.

3.7 Conclusion

In this study, samples of polycrystalline olivine, hot-pressed from either sol-gel or natural San Carlos precursor powders have been deformed and annealed under a controlled atmosphere at various temperatures up to 1500°C. The observed decrease in dislocation density as a function of temperature and time is adequately described by an empirical second order law for the rate constant. The associated activation energy for dislocation recovery under static conditions is 240 ± 43 kJ mol⁻¹ for sol-gel olivine and 355 ± 81 kJ mol⁻¹ for San Carlos olivine.

In this study a new and consistent method for estimating the three-dimensional dislocation density in iron-bearing olivine has been presented. This approach contributes half an order of magnitude increase in dislocation density and a corresponding decrease in recovery rate. The uncertainty in the imaging depth, d should be noted however and further work is required to refine its value.

The recovery rates for both data sets are markedly lower compared to previous studies on single crystals by 1 – 2 orders of magnitude. This discrepancy could be partly related to the imaging techniques used by the various authors: LM, TEM and SEM as opposed to a high resolution FE-SEM in this study. It has been demonstrated that imaging dislocations using the LM at 1,000 $\times$ will count only $\sim 1\%$ of the dislocations imaged by FE-SEM at 8,000 $\times$. This causes an apparent

increase of about two orders of magnitude of the rate constant for dislocation recovery. In addition, there may be an effect of grain size on the rate constant, where fine-grained aggregates have higher internal back-stresses exerted on the grain boundaries, slowing down dislocation (glide) kinetics. The small difference in recovery rates between sol-gel and San Carlos olivine at higher temperatures are within experimental uncertainty.

Microstructural analysis reveals dislocation re-organisations including straightening of dislocations and formation of tilt walls that accompany the reduction in free dislocation density. While it is not clear if there is a particular species of vacancy responsible for controlling dislocation climb, it appears to be controlled by fast diffusing oxygen. The commonly assumed slowest diffusing species in olivine, Si appears not to control the static dislocation recovery rate.

*There is nothing true anywhere,
the true is nowhere to be seen;
If you say you see the true,
this seeing is not the true one.*

Hui-Neng (638 – 713)

4

Dislocation damping in polycrystalline olivine I: Specimen fabrication, prior deformation and microstructural characterisation

4.1 Abstract

Several fine-grained polycrystalline olivine specimens were prepared from solution-gelation (sol-gel) and San Carlos precursors and pre-deformed in triaxial compression or torsion to investigate the effect of the resulting dislocation populations on anelastic relaxation at high temperature and moderate pressure. Hot isostatic pressed olivine aggregates of each type served as a baseline against which the pre-deformed specimens of higher dislocation density were compared. Two sol-gel specimens were pre-deformed in compression to a maximum differential stress and total strain of 280 MPa / 2.3% and 290 MPa / 22% respectively. The San Carlos specimen was pre-deformed in compression to 227 MPa differential stress

and 15% total strain. The dislocation density in the cylindrical sol-gel specimens increased from $1.0 \mu\text{m}^{-2}$ (hot-pressed) to a maximum of $11.4 \mu\text{m}^{-2}$. One specimen each of the sol-gel and San Carlos materials was pre-deformed in torsion to maximum shear stress/strain conditions of $165 \text{ MPa} / \leq 0.8$ (inhomogeneous deformation) and $195 \text{ MPa} / 0.5$, respectively. Complications due to interfacial slip resulted in only a modest increase in dislocation density up to $3.3 \mu\text{m}^{-2}$ in the region of maximum shear strain possibly due to recovery processes taking over during deformation. During successive deformation and attenuation experiments, the specimens maintained relatively constant average grain sizes between $\sim 3 - 17 \mu\text{m}$ and water contents for San Carlos olivine between $\sim 200 - 550 \text{ H}/10^6 \text{ Si}$, whereas sol-gel olivine was relatively dry. Dislocation densities decreased during annealing and mechanical testing at 1100°C more than expected from complementary static recovery experiments (Chapter 3). No reduction in dislocation density was measured for a torsionally pre-deformed San Carlos specimen after the attenuation experiment to a more conservative maximum temperature of 1000°C . Electron backscatter diffraction (EBSD) on each pre-deformed specimen before and after attenuation experiments showed only weak crystallographic preferred orientations but provided evidence for $(010)[100]$ slip. The torsionally pre-deformed specimens showed noticeable anisotropic thermal expansion upon heating/cooling and a tendency to untwist in response to stresses residual from the prior deformation at high temperature. Lastly, geometrical considerations of compressively and torsionally pre-deformed olivine concluded that for the easy slip system $(010)[100]$ higher resolved shear stress and thus larger anelastic shear strain is expected in a torsionally pre-deformed aggregate subsequently tested in torsional oscillation. The contribution from dislocation damping may hence be greater in these specimens, however also anisotropic.

4.2 Introduction

Seismologically observable attenuation in the upper mantle, where temperatures approach or exceed the solidus, is associated with wave speed variability. The correlation between low wave speeds and high attenuation (e.g. Anderson and Hart 1978a) is suggestive of solid-state viscoelastic relaxation. However, the topic has mostly been avoided in global attenuation models (e.g. Birger (2006); Cammarano and Romanowicz (2008)) where few efforts have been made to jointly interpret seismologically observed attenuation and velocity with temperature and composition of upper mantle rocks (Dalton et al., 2009). Some recent models, however, do consider the effect of the geotherm and partial melt distributions to explain local and regional attenuation (Artemieva et al., 2004; Faul and Jackson, 2005; Dalton and Ekström, 2006; Aizawa et al., 2008). Interim models for seismological application come from laboratory experiments on fine-grained olivine. These experiments have revealed grain-size sensitive viscoelastic relaxation attributable to grain-boundary sliding, augmented by the effects of partial melting (e.g. Cooper 2002; Faul et al. 2004). Dissipation from hydrogen in point defect sites has not yet been investigated in the laboratory but attempts have been made to model the effect indirectly (e.g. Karato 2003; Cammarano and Romanowicz 2008).

Dislocations may also play an important role in solid-state viscoelastic relaxation (e.g. Minster and Anderson 1981) and thus are capable of contributing to seismic shear wave attenuation Q_s^{-1} in the previously deformed / deforming upper mantle (Anderson et al., 1965; Anderson and Hart, 1978b). Extrapolation of lab-based flow laws (e.g. Mei and Kohlstedt 2000; Hirth and Kohlstedt 2003; Faul et al. 2011) and observed seismic anisotropy suggest that the upper mantle deforms by dislocation creep. Seismic anisotropy in highly deformed olivine polycrystals is also known to be related to the development of crystallographic preferred orientations (CPO) (Karato, 2008) which are thought to persist in the

upper mantle (Zhang and Karato, 1995; Tommasi et al., 2000; Mizukami et al., 2004). Furthermore, plastic anisotropy in forsterite, for instance, can be exceeded by a factor of ~ 100 (Darot and Gueguen, 1981) and strong wave attenuation is believed to result from simple shear strain giving rise to dislocation glide, whereas weak attenuation, in turn, results from pure shear strain favouring dislocation climb (Birger, 2006).

It follows that the dislocation microstructure of the upper mantle should reflect the prevailing tectonic stress associated with on-going deformation, in particular, the dislocation density ρ should be proportional to the square of the prevailing shear stress σ^2 (e.g. Minster and Anderson 1981). Cooler parts of the lithosphere may have a frozen-in fabric and dislocation density – subject to annealing kinetics (Chapter 3 and Karato and Ogawa 1982; Karato et al. 1993). However, it should be noted that dislocation density is the least stable microstructure in a deformed olivine polycrystal at temperatures $> 1000^\circ\text{C}$.

There are two main models in the literature that describe mechanical damping of dislocations that contribute to seismic attenuation – the stretched string vibration model (Koehler, 1952; Granato and Lucke, 1956; Minster and Anderson, 1981) and the kink migration model (Seeger, 1956; Karato, 1998; Jackson, 2007).

The string vibration model is based on a dislocation line pinned at each end by impurities or other dislocations, and occupying a minimum energy configuration in the absence of stress. For a favourably oriented applied stress, the dislocation line bows out under a counteracting tension force in the dislocation line. The displacement is accompanied by a small dissipation of strain energy. Deviations from linear stress-strain behaviour occur when the dislocation line bows out beyond its equilibrium curvature and dislocation multiplication by the Frank-Read mechanism results (Frank, 1980).

The kink migration model is more physically realistic as it takes into account the variation of dislocation line energy with position in the crystal lattice (the

Peierl's Potential). A dislocation segment is considered to lie in a low-energy valley but bows out under an applied oscillating stress and migrates over the energy barrier into the next valley, by migration of pre-existing (geometrical) kinks.

Variations of the models exist for nucleation and / or migrating pinning points (Yamafuji and Bauer, 1965; Granato and Lucke, 1981; Seeger, 1981). Such models can be simplified to similar expressions for the anelastic relaxation strength and associated estimates of the relaxation time.

However, few relevant laboratory data to test such models exist for specimens in which the dislocation microstructure has been systematically manipulated. Only in the study by Gueguen et al. (1989) have comparative forced-oscillation measurements been performed on un-deformed and pre-deformed specimens of single-crystal forsterite. Attenuation from vibrating dislocations was found to increase with decreasing frequency towards unrealistic upper mantle Q^{-1} values of 0.1 from torsional forced oscillation measurements at 1400°C (Gueguen et al., 1989). However, no reference was made to the possibility that the dislocation density varied markedly during the high-temperature experiments or whether comparison with upper-mantle conditions allowed for the expected effect of pressure through the activation volume. This study presents in two parts a more systematic approach to obtain new seismic-frequency data of Q^{-1} and shear modulus G dispersion in un-deformed and pre-deformed olivine polycrystals.

In this Chapter (Part I) a full microstructural examination is given of two suites of fine-grained and somewhat coarser-grained polycrystalline olivine prepared from sol-gel and San Carlos precursors. They were selectively hot-pressed and pre-deformed by dislocation creep in compression or torsion to produce contrasting populations of dislocations. Microstructural changes and water (i.e. OH) content after deformation and after annealing prior to / during high temperature forced-oscillation and microcreep experiments are documented. Using electron backscatter diffraction (EBSD) on the compressively and torsionally pre-deformed

samples, the slip systems upon which dislocations have multiplied are identified. A theoretical analysis of the variation of the resolved shear stress for easy dislocation glide in a single crystal pre-deformed in compression and torsion. In addition, the anelastic shear strain for these two deformation scenarios is calculated for an idealised aggregate composed of crystallites, assuming uniform stress (Sachs, 1927).

The seismic-frequency experimental data are reported in part II in Chapter 5 on these hot-pressed and pre-deformed olivine aggregates. For the pre-deformed sol-gel specimens, a marked increase in anelastic strain energy dissipation above $> 1000^{\circ}\text{C}$ is observed. However, torsionally pre-deformed San Carlos olivine with moderate dislocation density, tested at a conservative maximum temperature of 1000°C , reveals the highest levels of seismic attenuation in this study.

4.3 Experimental procedures

4.3.1 Sample preparation and hot-pressing

A detailed overview of sample preparation is given in Chapter 2 and only a brief summary is given here. Solution-gelation (sol-gel) olivine material was prepared by dissolution of Fe and Mg nitrates in ethanol to which tetraethyl orthosilicate was added (Jackson et al., 2002; Faul and Jackson, 2007). A second type of olivine material was prepared from hand-picked San Carlos xenocrysts, crushed and grain-size sorted (to $10\text{--}38\text{ }\mu\text{m}$) by sieving and sedimentation from suspension in ethanol (Tan et al., 2001). Cold-pressed pellets were hot-pressed at 1300°C , 300 MPa Ar confining pressure and $f_{\text{O}_2} \sim 10^{-8} - 10^{-9}$, buffered by a $\text{Ni}_{70}\text{--Fe}_{30}$ foil. The porosity was reduced to less than 1%. Microstructural analysis was systematically performed from a thin $\sim 1\text{ mm}$ slice from the upper end of the specimens. The cylindrical specimens were precision ground in preparation for subsequent deformation and attenuation experiments, and then heat-treated at 1400°C in a controlled atmosphere furnace to eliminate any newly adsorbed water.

Tables 4.1 and 4.2 contain a summary of the run conditions of the specimens. Specimens that were hot-pressed are denoted by Hxxx. Likewise compressively and torsionally pre-deformed specimens are denoted by Dxxx and Txxx respectively. These three types of specimens were subsequently mechanically tested in the attenuation apparatus for low-amplitude forced oscillations and are given by new run numbers (Axxx).

4.3.2 Triaxial compressive deformation

Compressive deformation experiments were carried out at a constant temperature of 1200°C or 1250°C and 300 MPa confining pressure at the Australian National University. A description of the apparatus is given in Chapter 2 and by Faul and Jackson (2007) including calibration procedures for apparatus and furnace.

The olivine specimens were deformed under servo-controlled load using Lab-view software on the adjacent computer for a series of ramps and dwells of increasing loads. The first deformation experiment (D6618) was a test experiment with a final 600 s ramp to 29 kN (~ 280 MPa) and a dwell of 1200 s for 2.3% strain to produce dislocations in the dislocation creep regime. Sol-gel specimen D6646 was deformed with two steps of increasing stress in the diffusion regime and one final step in the dislocation creep regime at 290 MPa differential stress producing a total amount of 22% strain and a high dislocation density ρ_i (see Table 4.3). San Carlos specimen D6701 was deformed in 7 steps of increasing stress (three of which in the diffusion creep regime, see Faul and Jackson 2007). The maximum differential stress was 227 MPa with a total strain of 15%. The experiments were terminated with a furnace quench to preserve the dislocations and microstructure. Generally, care was taken to ensure steady-state creep was achieved during each dwell. The data was inspected for any strain hardening in the dwell segments. This meant that the first data point for D6701, with the lowest differential stress, had to be discarded.

4.3.3 Torsional deformation

Torsional deformation experiments were carried out at the University of Minnesota on several cylindrical sol-gel and San Carlos hot-pressed specimens of different dimensions. In preparation for the torsional deformation tests, the hot-pressed sol-gel olivine specimens were re-fired as described in section 4.3.1. Similarly, the hot-pressed San Carlos olivine specimens were re-fired but at a lower temperature of 1200°C to prevent further grain growth in these ‘wetter’ specimens.

A Paterson gas-medium torsion apparatus (Paterson and Olgaard, 2000) was used for these experiments, servo-controlled via Labview software. Experiments were carried out at a constant rate of angular displacement (twist rate), see e.g. King et al. 2010. Preliminary torsional experiments on hot-pressed sol-gel and San Carlos olivine (H6602 and H6688) of 11.5 mm diameter were unsuccessful due to persistent slip at the sample-piston interfaces at temperatures up to 1250°C. The San Carlos sample cracked from a sudden jacket puncture during a second attempt at 1350°C and 300 MPa confining pressure and could not be reused.

Two precision-ground specimens of 10 mm diameter were tested next; a San Carlos olivine (T0434) and sol-gel olivine (T0436). Torsional deformation of a cylindrical specimen of smaller diameter would yield larger maximum shear stresses for a given torque value. The samples were jacketed and sandwiched between two porous alumina discs (of ~20% porosity) to obtain better grip during twisting. The specimens were wrapped in Ni₇₀-Fe₃₀ foil but no Ni₇₀-Fe₃₀ foil discs were placed at the ends of the specimens.

An initial constant twist rate of 0.03 mrad s⁻¹ was used, progressively increased by 0.02 mrad s⁻¹ steps. Maximum values of the twist rate were 0.12 mrad s⁻¹ (San Carlos) and 0.15 mrad s⁻¹ (sol-gel) corresponding to maximum values of the radially variable shear strain rates and shear stresses of $2.3 \times 10^{-5} \text{ s}^{-1}$ / 195 MPa at 1250°C and $2.8 \times 10^{-5} \text{ s}^{-1}$ / 165 MPa at 1300°C respectively. Upon recovery of the specimen assembly, the strain marker (jacket crimp) indicated

that most shear strain in the sol-gel specimen was achieved in the upper end, to a measured value of $\gamma = 0.8$ whereas the lower end remained almost un-deformed. The San Carlos specimen was more uniformly deformed to a maximum shear strain of $\gamma \sim 0.5$ (see Appendix B for photographs of the recovered assemblies).

Slip at the sample-alumina disc interfaces posed a problem throughout the experiments and was characterised by sudden drops in shear stress for a given constant twist rate. Under watchful control, increasing the constant twist rate was avoided as soon as the shear stress started to drop, to avoid unrecoverable loss of cohesion at the specimen ends. Both the sol-gel and San Carlos samples were deformed to a maximum possible twist rate for a period of about 6 hours. On both occasions, however, a sudden drop in shear stress ended the experiments and the furnace was quenched. Evidence for shearing between the porous alumina discs and olivine surfaces was found on the outside of the jacket.

4.3.4 Preparation for micro-strain mechanical testing

The compressively pre-deformed specimens were recovered by acid dissolution of the jacket and foil liner, reground and re-fired at high temperature, 1400°C for sol-gel and 1200°C for San Carlos olivine, in a controlled oxygen environment to remove any adsorbed moisture. The torsionally pre-deformed samples, however, were treated differently: the enclosing metal (foil and jacket) was precision ground down to a thickness of 0.25 mm (enough to keep the original Ni₇₀-Fe₃₀ wrapper in place) and the jacketed specimens were placed inside a vacuum oven at 100°C pending each attenuation experiment. Each specimen was loaded into a new mild steel jacket with long tapered alumina loading pistons. Tens of hours of annealing established stable mechanical behaviour at a constant confining gas pressure of 200 MPa and peak maximum temperature of either 1200, 1100°C or 1000°C before forced-oscillation / microcreep measurements at 25°C and 50°C decrements down to room temperature. Table 4.3 lists the total annealing times at temperatures $\geq 1000^\circ\text{C}$ for each specimen with new run number, Axxxx. In

addition, the calculated final dislocation densities ρ_{fcalc} are listed, based upon the dislocation recovery laws of sol-gel and San Carlos olivine given in Chapter 3.

4.3.5 Sectioning and EBSD preparation

A transverse slice of 1 mm thickness was cut from the upper end of each specimen, hot-pressed, deformed or recovered from the attenuation apparatus, for microstructural examination and any cracked ends were ground optically flat and parallel. The equivalent post-attenuation specimens (A1156, A1161, A1232 respectively) were prepared for EBSD in the same way as described above.

The torsionally pre-deformed specimens (T0434 and T0436) were sectioned tangentially at the outer specimen surface which contained the shear plane of maximum shear stress. The equivalent post-attenuation specimens (A1271 and A1231 respectively) were sectioned the same way.

All samples were embedded in epoxy, then polished for a series of steps of decreasing polishing grit size with a final colloidal silica (0.05 μm) polish for 1 hour on a soft cloth using a Kent Mark 3 (Engis Ltd.) polisher.

4.3.6 EBSD run conditions

A Zeiss Ultraplus FE-SEM, equipped with electron backscatter diffraction (EBSD) capability, was used in this study. Run conditions, EBSD mapping and noise reduction treatment of the data are described in Chapter 2, section 2.6.

4.3.7 Determination of grain size and water content

The grain size for each sol-gel and San Carlos specimen was determined using two methods: grain distribution analysis from EBSD maps and oxidation-decoration of the grain boundaries in backscattered electron images (Kohlstedt et al., 1976; Karato, 1987). The process of decorating and imaging grain boundaries (and

dislocations) is described in Chapters 2 and 3. For EBSD, grain boundaries were associated with misorientations $>10^\circ$. The average grain size was calculated from the grain size distributions as the diameter of a circle of equivalent area with a sectioning bias correction of $4/\pi$ (Jackson et al., 2002).

In addition, OH content of the olivine specimens during the various stages of experimentation was determined using a fourier transform infrared (FTIR) spectrometer. Again, the procedures are outlined in Chapter 2, section 2.7.

The grain size and water content results are summarised in Table 4.1 for the compressively pre-deformed specimens, Table 4.2 for the torsionally pre-deformed specimens and in Table 4.3 for the post-attenuation specimens.

Table 4.1: Compressive deformation experiments and hot-pressed specimen data on sol-gel and San Carlos olivine

Run number	Run conditions	Deformation conditions	Grain size	[H ₂ O] [¶]
	T (°C), P (MPa)	σ (MPa), ϵ^* (%), $\dot{\epsilon}$ (s ⁻¹)	(μm)	(H/10 ⁶ Si)
Sol-gel				
H6585	1300, 300	n/a	3.12±0.09	nd [§]
San Carlos				
H6695	1300, 300	n/a	11.10±0.15	555(34)
Sol-gel				
D6618 (H6607)	1200, 300	280, 2.3% total strain	5.00±0.30	950 [†] (58)
Sol-gel				
D6646 (H6611)	1250, 300	56.2, 0.87, 1.42×10 ⁻⁶	4.78±0.25	18(1)
		115.5, 0.72, 3.22×10 ⁻⁶		
		280.6, 2.63, 5.67×10 ⁻⁵		
		290 [‡] 12.8, –		
		22% total strain		
San Carlos				
D6701 (H6694)	1250, 300	53.9, 0.27, 1.27×10 ⁻⁶	12.95±0.47	423(26)
		76.8, 0.41, 1.28×10 ⁻⁶		
		103.0, 0.49, 2.14×10 ⁻⁶		
		134.6, 1.11, 5.87×10 ⁻⁶		
		172.1, 2.56, 1.83×10 ⁻⁵		
		208.7, 2.94, 5.65×10 ⁻⁵		
		227.8, 3.00, 1.02×10 ⁻⁴		
		15% total strain		

* Only strain values during constant loading (steady state creep)
† Water content measured from the pale yellow top-end of the specimen
‡ Deformation continued during furnace cooling at 600°C/hour until 1000°C
§ Water content below the detection limit
¶ Water content in wt. ppm H₂O between round brackets

Table 4.2: Torsional deformation data for sol-gel T0436 and San Carlos T0434. Both experiments were done in a series of fixed-torque/twist-rate steps.

T0436 1300°C [†]		Measured water content: 95 H/10 ⁶ Si (6 wt. ppm H ₂ O)				
Programmed	dθ/dt	M*	$\dot{\gamma}$	$\dot{\epsilon}_{eqv}$ [§]	τ	σ _{eqv} [¶]
twist rate (mrad s ⁻¹)	(rad s ⁻¹)	(Nm)	(s ⁻¹)	(s ⁻¹)	(MPa)	(MPa)
0.03	3.06×10 ⁻⁵	8.9	4.60×10 ⁻⁶	2.66×10 ⁻⁶	45.2	78.3
0.05	5.51×10 ⁻⁵	13.8	8.27×10 ⁻⁶	4.77×10 ⁻⁶	70.3	121.8
0.07	8.00×10 ⁻⁵	16.7	1.20×10 ⁻⁵	6.93×10 ⁻⁶	85.2	147.6
0.09	1.29×10 ⁻⁴	19.6	1.93×10 ⁻⁵	1.12×10 ⁻⁵	100.0	173.2
0.10	1.31×10 ⁻⁴	20.5	1.98×10 ⁻⁵	1.14×10 ⁻⁵	105.0	181.9
0.12	1.47×10 ⁻⁴	21.6	2.20×10 ⁻⁵	1.27×10 ⁻⁵	110.0	190.5
0.13	1.58×10 ⁻⁴	23.5	2.37×10 ⁻⁵	1.37×10 ⁻⁵	120.0	207.8
0.14	1.75×10 ⁻⁴	30.3	2.62×10 ⁻⁵	1.51×10 ⁻⁵	155.0	268.5

T0434 1250°C [†]		Measured water content: 209 H/10 ⁶ Si (13 wt. ppm H ₂ O)				
Programmed	dθ/dt	M*	$\dot{\gamma}$	$\dot{\epsilon}_{eqv}$ [§]	τ	σ _{eqv} [¶]
twist rate (mrad s ⁻¹)	(rad s ⁻¹)	(Nm)	(s ⁻¹)	(s ⁻¹)	(MPa)	(MPa)
0.03	3.05×10 ⁻⁵	15.7	4.62×10 ⁻⁶	2.67×10 ⁻⁶	80.3	139.1
0.05	5.39×10 ⁻⁵	23.5	8.17×10 ⁻⁶	4.71×10 ⁻⁶	120.0	207.8
0.07	7.77×10 ⁻⁵	26.4	1.17×10 ⁻⁵	6.78×10 ⁻⁶	135.0	233.8
0.09	1.03×10 ⁻⁴	28.8	1.56×10 ⁻⁵	8.98×10 ⁻⁶	147.0	254.6
0.10	1.09×10 ⁻⁴	31.3	1.66×10 ⁻⁵	9.57×10 ⁻⁶	160.0	277.1
0.11	1.19×10 ⁻⁴	35.2	1.80×10 ⁻⁵	1.04×10 ⁻⁵	180.0	311.8
0.12	1.33×10 ⁻⁴	38.8	2.01×10 ⁻⁵	1.16×10 ⁻⁵	195.0	342.9

[†] Grain size after deformation: 6.68±0.65 μm

[‡] Grain size after deformation: 6.10±0.24 μm

* Peak torque at the end of each twist step

[§] Equivalent or effective strain rate ($\dot{\epsilon}_{eqv} = 1/\sqrt{3} \cdot \dot{\gamma}$)

[¶] Equivalent or effective stress ($\sigma_{eqv} = \sqrt{3} \cdot \tau$)

Table 4.3: Microstructural information from attenuation experiments on sol-gel and San Carlos olivine

Runs	T_{max}	$t_{anneal}^{\dagger}$	ρ_i	ρ_f	$\rho_{fcalc}^{\dagger\dagger}$	$\rho_{fcalc}^{\dagger\dagger}$	d	$[H_2O]^{§§}$
	°C	hour	μm^{-2}	μm^{-2}	μm^{-2}	μm^{-2}	μm	H/10 ⁶ Si
Sol-gel								
A1127 (H6585)	1200	33	1.0	1.0	—	—	3.1	31(2)
A1156 (D6618)	1100	52	4.8	1.9	4.0	0.9	4.9	468(29) [§]
A1161 (D6646)	1100	34	7.1	5.1	6.1	1.4	5.1	9(1)
A1231 (T0436)	1100	55	2.4	1.3	2.3	0.8	5.7	27(2)
San Carlos								
A1279 (H6695)	1200 [¶]	60	0.4	0.5	—	—	17.3	284(17)
A1232 (D6701)	1100	68	11.4	7.2	9.6	1.0	8.6	190(12)
A1271 (T0434)	1000	61	3.3	3.3	3.3	2.8	8.8	241(15)

[†] Total annealing time at T_{max}

[§] Water content measured from the discoloured upper end of the specimen

^{§§} Water content in wt. ppm H₂O between round brackets

[¶] Specimen has been briefly at > 1300°C on two occasions during successive furnace problems

^{††} Calculated using $E_{sol-gel} = 240 \text{ kJ mol}^{-1}$, $k_0 = 1.73 \times 10^{-10} \text{ m}^2 \text{s}^{-1}$ and $E_{SanCarlos} = 355 \text{ kJ mol}^{-1}$, $k_0 = 1.85 \times 10^{-6} \text{ m}^2 \text{s}^{-1}$ (see Chapter 3)

^{‡‡} Calculated using $E_a = 389 \text{ kJ mol}^{-1}$, $k_0 = 2.13 \times 10^{-3} \text{ m}^2 \text{s}^{-1}$ (Karato and Ogawa, 1982)

4.4 Microstructural characterisation

4.4.1 BSE imaging and grain size distributions

Oxidative-decoration and polishing procedures of dislocations are described in Chapter 2, section 2.4.3 and by Karato et al. (1986).

Some typical dislocation substructures are presented in panel (a) in Fig. 4.1 for the torsionally deformed San Carlos (see Chapter 3 for microstructures in compressively deformed olivine). The large grain in the image centre contains a moderate density of dislocations whereas some neighbouring grains appear de-

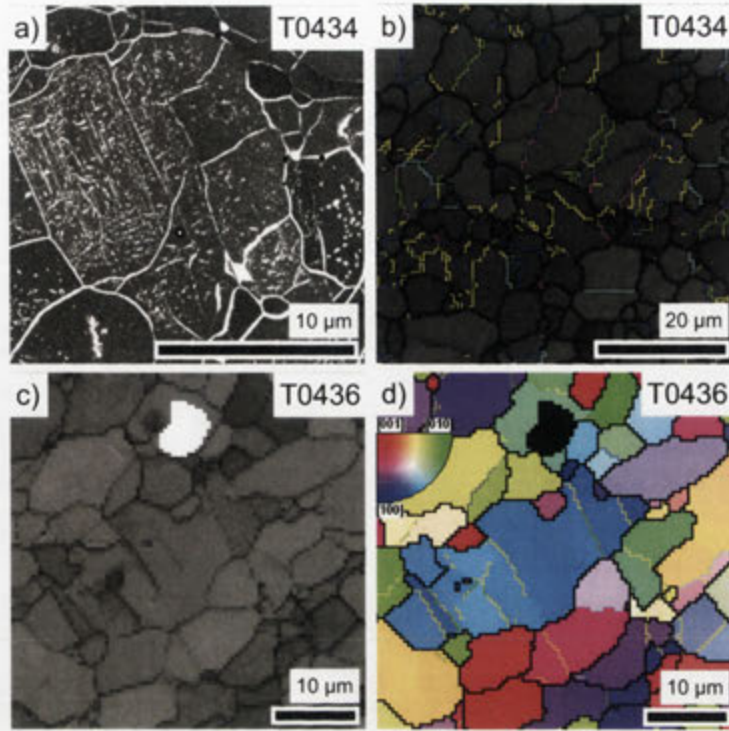


Fig. 4.1: Dislocation sub-structures and grain microstructures as shown in the torsionally pre-deformed samples T0434 and T0436. Note the differences in scale. (a) A backscattered electron image of decorated grain boundaries and dislocations in T0434 obtained at an accelerating voltage of 5 kV. (b) A portion of an EBSD band contrast map for T0434 showing misorientation angles in sub-grain boundaries for $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries for $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black. (c) A band contrast image of T0436 (d) An inverse pole figure map for the shear direction of the same region for all grain orientations additionally showing sub-grain boundaries using the same colour coding given in (b).

void of dislocations. There are straight dislocations present, identified by the long parallel white decorated lines and circular dislocations present, which are imaged perpendicular to the polished surface. Some dislocations appear to form dislocation walls. Higher misorientations lead to sub-grains, and an EBSD map in Fig 4.1b reveals many of these. As standard practice, misorientations between grains of $> 10^\circ$ (blue) were considered grain boundaries. However, the background band contrast image in panel (b) does not always reveal the amount of misorientation as shown by similar shades of grey on either side of a misorientation boundary

up to $> 20^\circ$ (aqua blue). Several grains do not appear to contain any sub-grain boundaries. Although some features in the band contrast image appear to be boundaries no misorientation is picked up by EBSD and are thus likely to be microcracks.

Shape preferred orientation of grains is only apparent in the torsionally pre-deformed sol-gel T0436 in panels (c) and (d) in Fig. 4.1. Panel (c) shows a band contrast image of a typical region obtained from the upper end of the specimen close to the outer radius characterised by maximum shear strain estimated at $\gamma \sim 0.8$. Some low angle sub-grain boundaries ($> 2^\circ$) are visible as yellow lines in panel (d) and only few $> 5^\circ$ boundaries (lime green). The low density of sub-grain boundaries in (c) and (d) compared with (b) cannot be explained by control on grain size as the average grain size between the two specimens is very similar (given in Table 4.2). However the dislocation density in the torsionally pre-deformed sol-gel specimen is somewhat lower compared to the pre-deformed San Carlos olivine specimen.

Finally, the coloured grains in panel (d) represent shear directions of grains in the crystallographic reference frame for olivine shown by the legend. The large grain in the middle shows slightly different shades of blue between the sub-grain boundaries as indicated. Other blue to purple to light blue grains share similar orientations in terms of the shear direction. However, other grains (orange, red, yellow, green) appear randomly oriented.

The porosity and opx distribution in these samples is very low, $< 1\%$ (not shown in Fig. 4.1).

4.4.2 Grain size distributions

As described in section 4.3.7, grain sizes were determined using EBSD or decorated grain boundary imaging for those specimens not analysed using EBSD. Normally, the EBSD method is preferred due to better resolution of all grain orientations while using the oxidative-decoration technique may cause some grain

boundaries not to decorate well (Durham et al., 1977) or alternatively can be mistaken as sub-grain boundaries. To test this, the average grain size was determined for T0436 using both techniques on the same quadrant. The results were 5.70 ± 0.45 (ox-dec) and 6.68 ± 0.65 (EBSD), which implies both techniques can be used to adequately resolve the grain size within measurement error.

The grain size distributions for all pre-deformed and post-attenuation specimens are shown in Fig. 4.2. The specimens for which the EBSD method was used are indicated with a † attached to the specimen label for the corresponding histogram of each histogram. The figure is divided up into three columns for the (1) hot-pressed, (2) compressively pre-deformed and (3) torsionally pre-deformed specimens (shown in blue) with their equivalent attenuation run numbers (shown in red). Each grain size distribution is normalised to the number of grains measured, indicated by N_1 and N_2 . Average grain size per specimen is given as well. Identical vertical scales are used to facilitate comparison.

Sol-gel olivine was synthesised in the laboratory with near-identical initial grain size distributions, opx and porosity contents, facilitating a comparison with the resulting distributions of the pre-deformed specimens to the original hot-pressed result. The grain size distribution for hot-pressed sol-gel olivine (H6585, A1127) in Fig. 4.2a is narrow and little microstructural change has occurred after annealing at 1200°C and mechanical testing for attenuation measurements. The compressively pre-deformed sol-gel olivine specimens in panels (2a) and (2b) do not show any sign of major microstructural change after annealing and mechanical testing at 1100°C either, but do indicate some broadening of the grain size distribution indicating some grain growth has occurred during deformation. The torsionally deformed sol-gel specimen that was deformed to the highest equivalent strain shows a continued trend of broadening towards larger grain size (T0436) and a somewhat bimodal distribution. However, after attenuation (A1231) the specimen appears to have lost some of the larger grains possibly indicating that some recrystallization occurred during annealing at 1100°C.

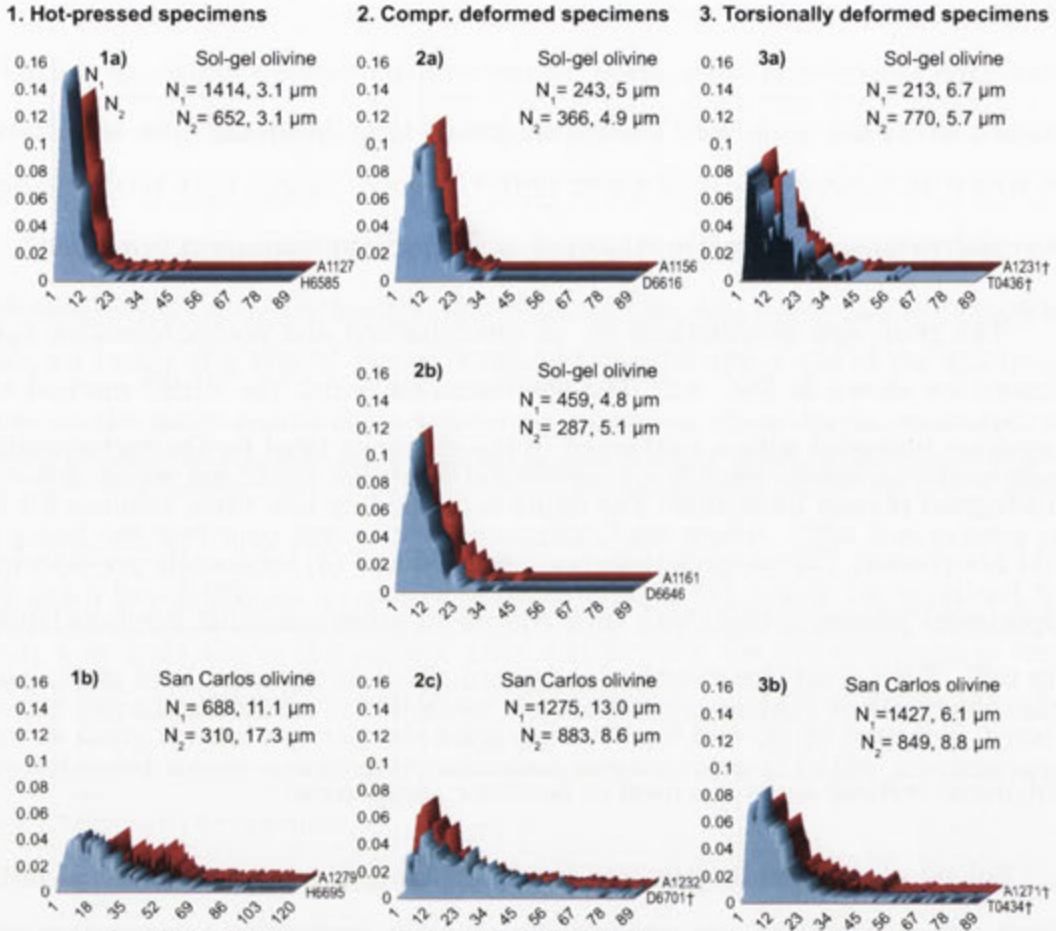


Fig. 4.2: Normalised grain size distributions of all specimens before (blue) and after attenuation (red). N_1 and N_2 show the number of grains and average grain size respectively for blue and red. Numbers across the histogram bases are grain sizes in micrometres. The † indicates that the grain size distribution for that specimen was obtained from an EBSD map rather than via oxidative-decoration of grain boundaries.

The grain size distributions for San Carlos olivine have to be interpreted with more caution as the initial grain size distribution of the hot-pressed San Carlos, shown in panel (1b) in Fig. 4.2, is not necessarily representative of the original grain size distributions of the pre-deformed specimens shown in panels (2c) and (3b). In general, the distributions are broad especially for the hot-pressed and compressively pre-deformed specimens, but slightly less so for the torsionally pre-deformed specimen (T0434). Starting with panel (1b), compared with the

original hot-pressed specimen (H6695), significant grain growth has occurred after prolonged annealing at 1200°C for ~ 60 hours and mechanical testing (A1279). The odd grain size distribution may also reflect the two prior uncontrolled excursions to high temperature ($> 1300^{\circ}\text{C}$) for this specimen caused by furnace and thermocouple malfunctions. The grain size distribution of the compressively pre-deformed San Carlos specimen (D6701) in panel (2c) is comparable to the hot-pressed specimen (H6695) in (1b). However, after annealing at a maximum temperature at 1100°C and mechanical testing, average grain size has been reduced. Finally, in panel (3b) only minor grain growth can be observed after annealing at a maximum temperature of 1000°C (A1271) in comparison with the grain size distribution of the pre-deformed specimen (T0434).

4.4.3 FT Infrared Spectroscopy and density measurements

The FTIR spectra for all doubly polished thin sections can be found in the Appendix J. The measured concentrations of molecular water are listed in Tables 4.1 and 4.2 for the pre-deformed specimens and Table 4.3 for the same specimens after attenuation experiments. One sol-gel sample reported anomalously high water content values of 950 H/10⁶ Si (58 wt. ppm H₂O) (D6618) after deformation and 468 H/10⁶ Si (29 wt. ppm H₂O) (A1156) after attenuation. These values were measured from a section cut from the relatively discoloured and porous (~ 4 %) upper end of the specimen so these values were most probably not representative of the entire specimen. The same specimen that came before from hot isostatic pressing was found to have an OH content below the detection limit of the spectrometer (see Appendix J).

Sample density was measured on two hot-pressed specimens (sol-gel A1127 and San Carlos A1279) after an attenuation experiment at 1200°C and 200 MPa confining pressure. The results were

- $3.33005 \pm 0.00026 \text{ g cm}^{-3}$ (A1127)
- $3.33261 \pm 0.00021 \text{ g cm}^{-3}$ (A1279)

By comparison, fully dense Fo₉₀ would have a density of 3.34 g cm⁻³ (Gribble and Hall, 1992). Hence, the final porosity in the specimens was 0.30% and 0.24% respectively.

4.5 Deformation of sol-gel and San Carlos olivine

4.5.1 Overview

The three mechanisms operating during high temperature creep in olivine are diffusion, dislocation and Peierls creep for increasing differential stresses. In all cases, the total strain rate is the sum of the strain rates from each contributing deformation mechanism (Poirier, 1985). The respective flow laws for diffusion, dislocation and Peierls creep of ‘low-OH’ olivine are as follows

$$\dot{\epsilon}_{diff} = A_d \sigma^n d^{-p} \exp(-E_d/RT) \quad (4.5.1)$$

$$\dot{\epsilon}_{dis} = A_c \sigma^n \exp(-E_c/RT) \quad (4.5.2)$$

$$\dot{\epsilon}_P = A_P \sigma^n \exp\left(-\frac{E_P}{RT} \left(1 - \left(\frac{\sigma}{\sigma_P}\right)^r\right)^q\right) \quad (4.5.3)$$

(Frost and Ashby 1982 and Faul et al. 2011, and references therein), where $\dot{\epsilon}$ is the axial (or for torsion, equivalent) strain rate (s⁻¹). A is a material-dependent constant which additionally includes the dependency on water content and melt fraction (MPaⁿ s⁻¹ μm^{-p}). d is the grain size (μm). E_d , E_c and E_P are the activation energies for diffusion, dislocation and Peierls creep (J mol⁻¹). σ is the differential stress, whereas σ_P is the resistance to glide at zero K (MPa) (Frost and Ashby, 1982). n and p are the stress and grain size exponents whereas q and r are the exponents in Peierls law. Finally, R is the gas constant (J K⁻¹ mol⁻¹) and T is the temperature in K.

Fitting a flow law to an olivine aggregate is well established for triaxial compressive deformation experiments but is less commonly employed for olivine specimens deformed in torsion (see e.g. Bystricky et al. 2006). Firstly, the torsional

stress and strain rate vary with distance from the torsion axis. Secondly, if intermittent slip occurs on the specimen-alumina disc interfaces during deformation, it may have an undesired softening effect (unstable localisation) and cannot be easily corrected for. Thirdly, it is assumed that the rheological properties of the cylindrical specimen are homogeneous throughout. However, any radial variation of the effective deformation mechanism might be more important (e.g. for grain size). This implies that a transition exists with increasing radial distance from the torsional axis from diffusional creep to dislocation creep.

The stress exponent n from a torsion test can be estimated in a way that is analogous to axial deformation tests via a log-log plot of $\dot{\theta}$ versus M

$$n = \frac{\delta \ln \dot{\gamma}}{\delta \ln M} = \frac{\delta \ln \dot{\theta}}{\delta \ln M} \quad (4.5.4)$$

where M is the total torque, $\dot{\gamma}$ is the shear strain rate and $\dot{\theta}$ is the angular displacement rate (Paterson and Olgaard, 2000).

Note that in this study the specimens subjected to triaxial compression, were deformed only to low strains to preserve their length for the subsequent attenuation experiments presented in Chapter 5.

4.5.2 Triaxial compression experiments

The following hot-pressed specimens were compressively deformed for this study; Sol-gel olivine H6607 (D6618), H6611 (D6646) and San Carlos olivine H6694 (D6701). However, useful stress-strain rate data could only be processed for the latter two. These two deformation experiments were carried out at 1250°C and 300 MPa confining pressure.

The rheology data of sol-gel (D6646) and San Carlos (D6701) olivine, shown in Table 4.1, are presented in a strain rate (s^{-1}) versus stress (MPa) figure (Fig. 4.3). Note that only a limited amount of data was acquired from just two deformation experiments. At lower differential stress, the data lie in the same trend despite

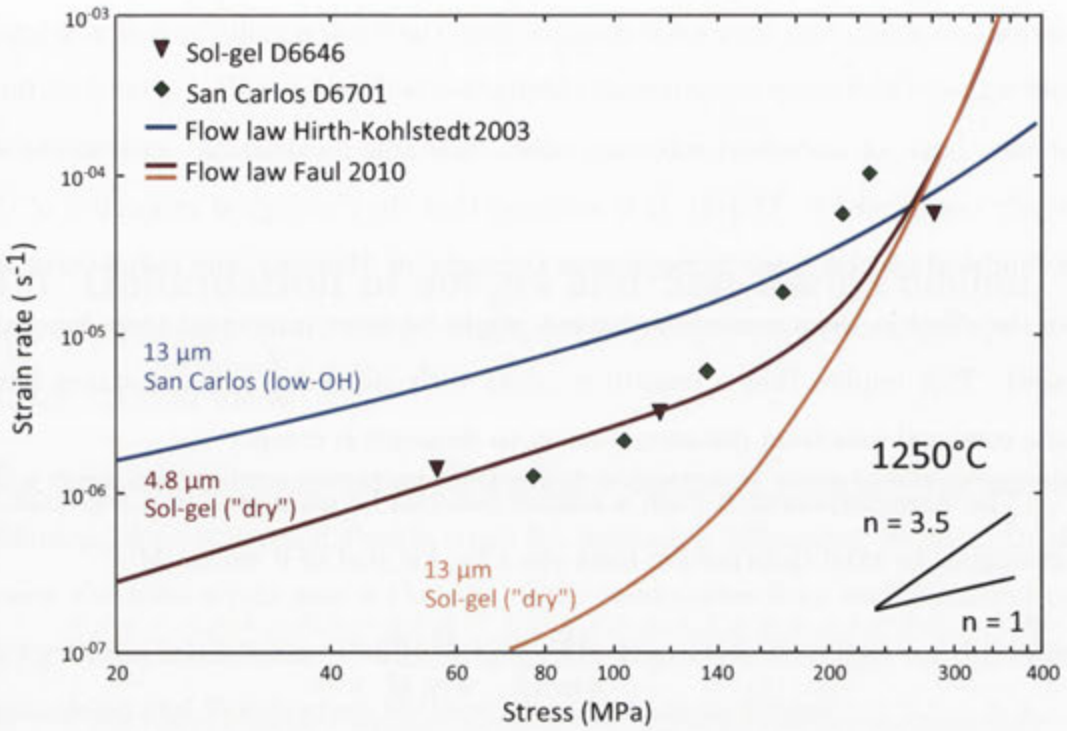


Fig. 4.3: Strain rate versus stress data on a log-log scale for the compressively pre-deformed materials D6646 and D6701 at 1250°C. A comparison of the creep data is made with two combined melt-free exponential creep flow laws for grain sizes of 4.8 and 13 μm and essentially dry rheology (Faul and Jackson, 2007; Faul et al., 2011) and a combined dislocation creep flow law from Hirth and Kohlstedt (2003) for a grain size of 13 μm and 'low-OH' rheology.

different grain sizes of either specimen (Fig. 4.1). At low stresses, the deformation regime is dominated by grain size sensitive diffusion creep for a stress exponent on σ of $n \approx 1$. The San Carlos specimen (D6701) demonstrated a transition to more strongly stress-sensitive deformation at ~ 140 MPa differential stress. At the highest differential stress (227 MPa) it deformed up to half an order of magnitude faster than the sol-gel specimen (D6646).

Two relatively new flow laws, determined in previous studies, are shown. These are combined diffusion creep and Peierls exponential or powerlaw creep flow laws (Fig. 4.3). They were recalculated for different grain sizes and stress exponents of respectively 13 μm and $n = 3.5$ for powerlaw creep (bright red curve) (Hirth and Kohlstedt, 2003) and 4.8 μm and $n_p = 2$ (dark red curve) for Peierls

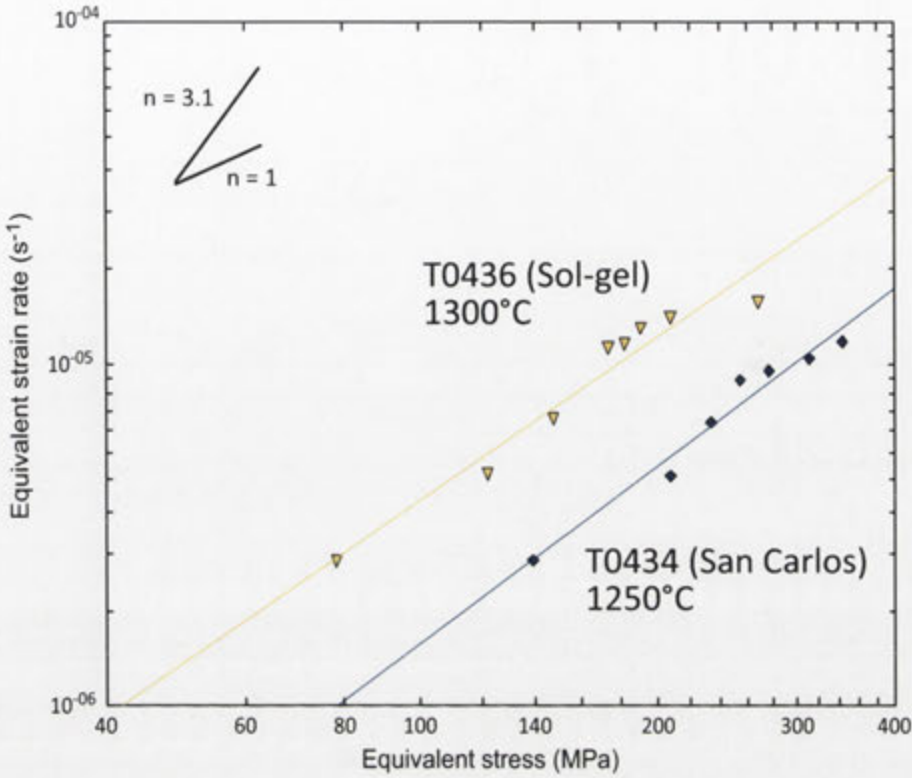


Fig. 4.4: Creep data from torsion experiments on sol-gel and San Carlos olivine at 1300°C and 1250°C respectively. The axes show the equivalent (effective) strain rate and stress for easier comparison with the compressively pre-deformed samples. The two trend lines are best fits through the data. No transition to creep with higher stress exponent at higher applied stress is observed. The slopes of the data sets are close to $n = 1.7$.

creep ($n \sim 8$ for equivalent powerlaw creep) (Faul et al., 2011). The rheology data from the sol-gel specimen D6646 is relatively well fitted by the combined flow law from Faul et al. (2011). However, the San Carlos D6701 rheology data is fitted by neither flow law very well.

4.5.3 Torsion experiments

The mechanical results of two torsionally deformed olivine specimens, both measuring 10 mm in diameter, are shown in Fig. 4.4. To compare with compressional creep experiments, the equivalent stress presented, is $\sqrt{3}$ times the shear stress and the equivalent strain rate is $1/\sqrt{3}$ times the shear strain rate (Shrivastava

et al., 1982). The sol-gel olivine specimen (T0436) was tested at 1300°C whereas the slightly ‘wetter’ San Carlos specimen (T0434) was tested at 1250°C. The stress exponents were fitted via Eqn. 4.5.4 and are $n \approx 1.6$ and $n \approx 1.7$ respectively. These low values are reflected by the shallow slopes in Fig. 4.4. Additional plots of the shear strain versus time and internal and external torque versus shear strain are shown in Fig. B.1 in Appendix B.

4.6 EBSD results

4.6.1 Pole figures and inverse pole figures

A one-point-per-grain analysis of crystal orientation was carried out from orientation data presented in maps of the pre-deformed and equivalent post-attenuation sol-gel and San Carlos specimens. The pole figures in Fig. 4.5 show the orientations of each of the crystal axes for olivine [100], [010] and [001] in the laboratory reference frame. Multiples of uniform misorientation (MUD) values up to 2 are considered mostly weak for olivine (e.g. Jung et al. 2009).

In addition, to the right of the pole figures are shown misorientation distributions of the uncorrelated (not neighbouring) grains. Such a distribution is a measure of fabric strength from which the M-index value can be calculated (Skeemer et al., 2005). For completeness, J-index values are also reported (Bunge, 1982). They were calculated using HKL Salsa (Oxford Instruments) from an orientation distribution function (ODF) with $L_{max} = 20$ as the highest harmonic for the series expansion. A Gaussian half-scatter width of 5° was used. For each specimen, the number of grains (N) measured is reported together with, for pre-deformed specimens, actual or equivalent strain. Note that more than 200 grains are required to make a robust estimate of fabric pattern and strength (Ben Ismail and Mainprice, 1998).

The following observations can be made. The sol-gel specimen T0436 (Fig. 4.5a), that was pre-deformed in torsion to a maximum equivalent strain of 46%

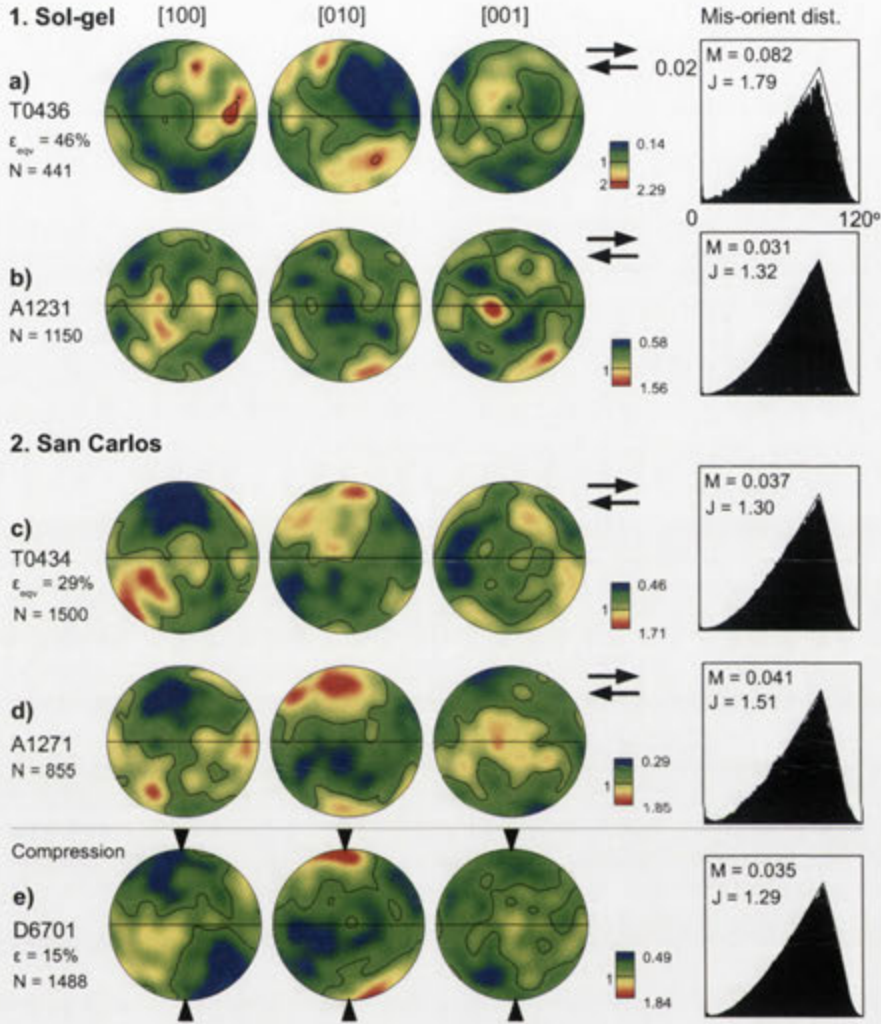


Fig. 4.5: Pole figures showing the principal directions in olivine ([100], [010] and [001]) for the pre-deformed and post-attenuation specimens with associated misorientation distribution histograms. The pole figures are presented as lower hemisphere equal area projections with a half-scatter width of 20° and cluster size of 5° . The sense of shear is indicated by the arrows. For torsion, in (a) to (d) the north (south) poles correspond to the shear plane normal. For compression, the north (south) pole corresponds to the compression direction. The colour coding refers to the density of data points and the contour lines to multiples of uniform distribution. Since a one-point-per-grain analysis is done, N refers to the number of grains. In addition, the (equivalent) strain is given per deformed specimen. The histograms show uncorrelated misorientations for olivine between $0 - 120^\circ$. The insets show the fabric strength indicators: M- and J-indices.

($\gamma = 0.8$), shows a moderately strong CPO for a maximum MUD of 2.29, M-index of 0.082 and J-index of 1.79. By comparison, a hot-pressed olivine aggregate can have an M-index of 0.01, while an aggregate with strong texture could show an M-index of 0.44 (Skemer et al., 2005). The [010] axes tend to lie perpendicular to the shear plane and [100] maxima appear inclined to the shear direction and shear plane. Tens of hours of annealing T0436 at 1100°C associated with attenuation experiment A1231 (Fig. 4.5b) considerably weakened the preferred orientation.

The San Carlos specimen T0434 (Fig. 4.5c) was pre-deformed in torsion to a somewhat lower maximum equivalent strain of 29% ($\gamma = 0.5$). The respective MUD, M- and J-index values are hence also lower than for T0436. There is a suggestion of [100] maxima inclined to the shear direction but less obvious are the associated [010] axes perpendicular to the shear plane. The weak fabric patterns are generally similar in Fig. 4.5d after tens of hours annealing at 1000°C and an attenuation experiment, suggesting little microstructural change. However, the texture indices (J-, M-index) became marginally higher.

The San Carlos olivine specimen D6701 (Fig. 4.5e) was pre-deformed in tri-axial compression. When orientations are plotted with the reference frames for the two different stress states as shown, some of the maxima and minima fall in the same parts of the equal area projections as for the torsionally pre-deformed specimens. However, a comparison would be meaningless because the expected CPO patterns for olivine deformed in compression is different to that from simple shear or torsion experiments (Nicolas et al., 1973; Wenk et al., 1991). D6701 was pre-deformed to 15% accompanied with a low J- and M-index values. A sol-gel sample of smaller grain size, deformed to approximately the same amount of compressive strain does not show any CPO (Faul et al., 2011).

Inverse pole figures of these specimens are shown in Fig. 4.6. These pole figures are presented in a similar way to previous EBSD work on olivine aggregates deformed in shear (Jung et al., 2006). Both the sol-gel and San Carlos torsionally pre-deformed samples and their post-attenuation counterparts are plotted to show

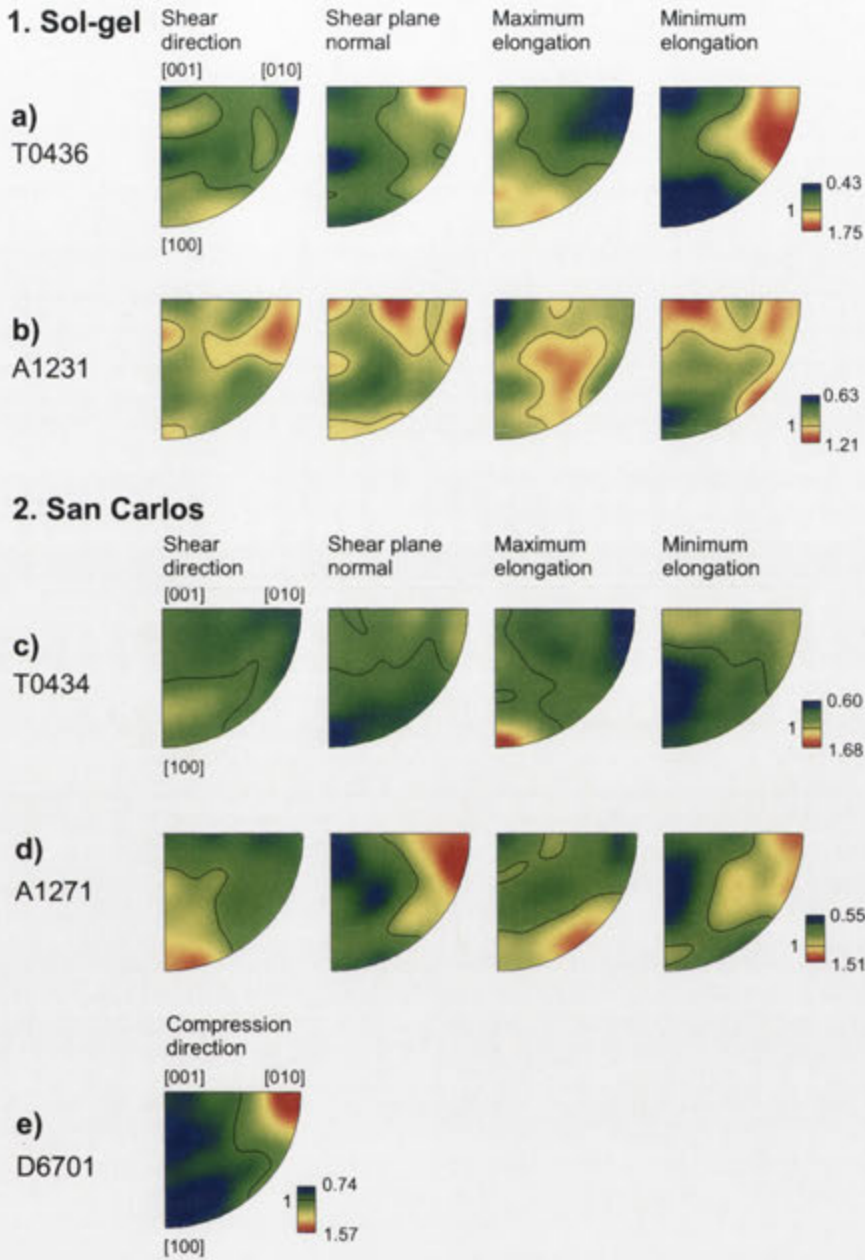


Fig. 4.6: Inverse pole figures of the pre-deformed specimens. For (a) to (d), the figures are shown for the selected shear direction, shear plane normal, maximum elongation of the strain ellipsoid and minimum elongation of the strain ellipsoid, 90° away from the maximum. In (e) the compression direction is chosen for a compressively pre-deformed specimen. A half-scatter width of 20° with 5° cluster size is used. The colour coding refers to the density of data points and the contour lines refer to the multiples of uniform distribution.

four directions characterising stress and strain relative to the [100], [010] and [001] crystallographic co-ordinate system. The two applied stress axes (shear direction and normal to the shear plane) and of major and minor axes of the calculated strain ellipsoid are shown. The orientation of the finite strain ellipsoid was measured from the angle of rotation the originally straight strain marker (crimp in the jacket) assumed to be the same for the post-attenuation EBSD sections. For all inverse pole figures (Fig. 4.6a-d) the maximum MUD indicates that the texture is weak to random, especially for Fig. 4.6b, A1231. Some weak patterns can be observed but should be interpreted with caution. Shear directions are given for completeness, but demonstrate essentially the same information in Fig. 4.5.

In Fig. 4.6e, an inverse pole figure showing compression direction relative to crystal axes is shown for the compressively pre-deformed specimen D6701. There is a weak clustering of compression direction towards [001] for a narrow range of MUD.

4.6.2 Low-angle grain interior misorientations

Rotation axes for grain interior low angle ($2\text{-}10^\circ$) misorientation boundaries are plotted in Fig. 4.7. The analysis was carried out using all available data pairs for $2\text{-}10^\circ$ misorientations rather than a one-point-per-grain analysis. The number of measurements are indicated by n and varies depending on the number of sub-grain boundaries present. Generally, fairly strong maximum multiples of uniform distributions are obtained with clustering of low angle rotations either sub-parallel to the [001] or [010] directions. Few to no rotations are measured with axes close to [100] (Fig. 4.7a-d). Sol-gel olivine shows little change after annealing at 1100°C . However, San Carlos olivine shows an initial preference for low-misorientation axes lying near to [010] after deformation but a switch to both orientations, [001] and [010] is observed after annealing (1000°C) and torsional forced oscillation measurements. The compressively pre-deformed San

Carlos specimen shows strong clustering at $[001]$ (Fig. 4.7d). Additional information was obtained from observation of sub-grain boundaries in relatively large grains of the San Carlos material using the light microscope. Essentially, a grain containing sub-grain boundaries was placed in extinction. It was then rotated 45° from extinction and the interference colour of the grain is noted. Knowing the length-slow direction in the sensitive tint plate, if the colour in the grain is one order higher, then the slow crystallographic direction is identified (i.e. $[100]$ in olivine). For 50 grains analysed, $> 90\%$ demonstrated dislocation boundaries sub-parallel to (100) plane (or perpendicular to $[100]$ direction, Fig. 4.7c).

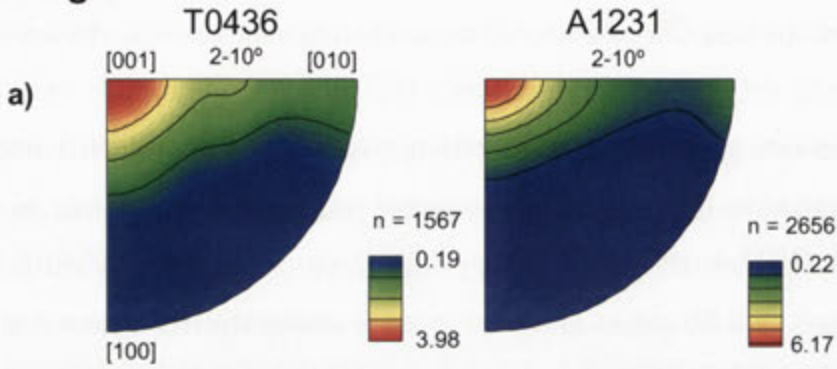
4.7 Discussion

4.7.1 Mechanisms of deformation and microstructural recovery

Deformation of olivine in the dislocation creep regime is mainly controlled by the activation of a number of slip systems. Depending on the physical and chemical conditions of deformation, slip systems requiring the lowest critical resolved shear stress to activate will generally make the largest contributions to the total strain (Castelnau et al., 2008). These slip systems, in turn, control the CPO development of all the grains in an aggregate.

Low strain, plastic deformation of grains is unlikely to involve a lot of grain back-rotation (Karato, 2008) to accommodate the developing shear on a slip plane for a given slip system. Therefore, the fabric strength will be weak and should allow a comparison of high and low dislocation density grains with Schmid factor for a given slip system (Appendix E). This was done using EBSD via determination of grain orientations with visibly high and low dislocation density (whereas moderate density grains were avoided to produce a sharp contrast). The two subsets of EBSD data were plotted in (inverse) pole figures and compared to a predicted distribution of Schmid factors for one of olivine's slip systems with

1. Sol-gel



2. San Carlos

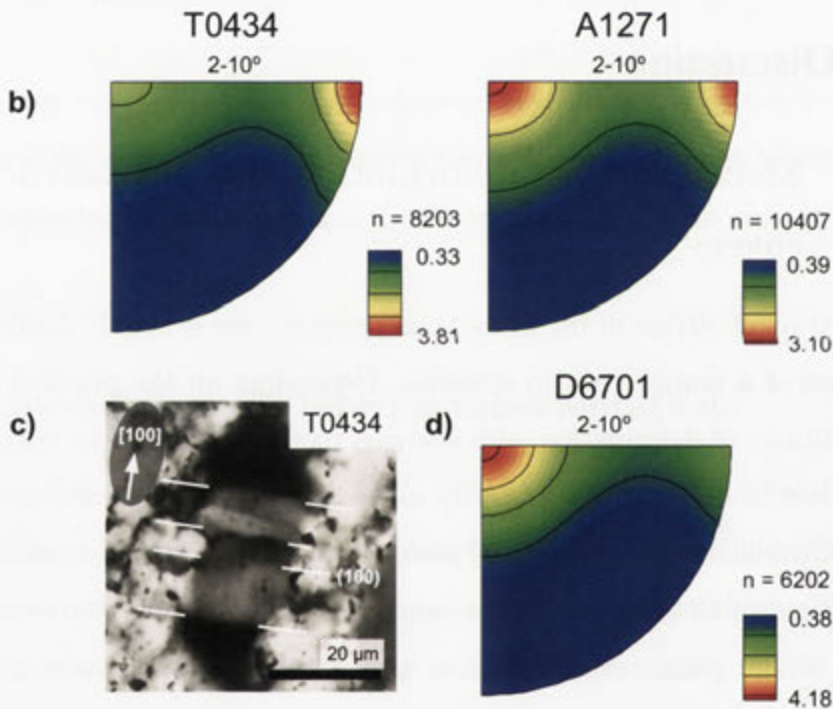


Fig. 4.7: Plots of $2-10^\circ$ misorientation angle data for rotations in the crystallographic reference frame for each pre-deformed specimen (a), (b) and (d). A half-scatter width of 20° with 5° cluster size is used. The colour coding refers to the density of data points and the contour lines refer to the multiples of uniform distribution. A transmitted light micrograph in (c) shows a large San Carlos olivine grain (T0434) for which the orientation of the sub-grain boundaries (composed of dislocation segments) has been identified as indicated by the arrow.

smallest burgers vector – (010)[100]. The results, however, were somewhat ambiguous and indicated, as expected, that stress and strain distributions between grains during (compressive) deformation are not uniform.

Conversely, any grain undergoing high strain plastic deformation that was originally favourably oriented with a high Schmid factor for a given slip system ends up being unfavourably oriented with a low Schmid factor to an imposed stress (Karato, 2008). This gives rise to dynamic recrystallization and grain growth depending on the heterogeneous distribution of local stress and strain on grain boundaries in a deforming aggregate (Zhang et al., 2000).

4.7.1.1 Microstructural interpretations

In this study, the microstructures of the torsionally pre-deformed sol-gel and San Carlos specimens (Fig. 4.1) indicate dislocation multiplication and sub-boundary formation has occurred but the dislocation densities after deformation were lower (2.4 and $3.3 \mu\text{m}^{-2}$ respectively, Table 4.3) than those in some of the lower strain compressively deformed specimens ($\sim 7 - 11 \mu\text{m}^{-2}$ for D6646 and D6701). The dislocation density during steady state plastic deformation is known to initially increase markedly but not much further after 5% strain but the ‘steady-state’ density does depend on applied differential stress (Durham et al., 1977). The sol-gel olivine specimen T0436 was deformed to lower equivalent stress than the San Carlos specimen T0434 and has fewer sub-grain boundaries (Fig. 4.1d). However, both have still deformed to higher (equivalent) stress than some of the compressively deformed samples although softening due to slip likely affected the outcome. Nonetheless, T0436 that was deformed to higher shear strain developed a visible shape preferred orientation of grains (Fig. 4.1c) while T0434 (Fig. 4.1b) did not.

FT infrared spectroscopy did not reveal any structural water peaks which suggests the broadband infrared absorbance due to OH vibrations was mainly measured from non-structural sites in the olivine aggregates (see Appendix J for

all FTIR spectra). Light microscope and scanning electron microscope examination (with x-ray microprobe analysis) on D6701 did not reveal any melt present in the pores. Generally, a reduction in water concentration in the olivine aggregates after annealing and mechanical testing is measured (see Tables 4.1, 4.2 and 4.3).

4.7.1.2 Rheological observations

The rheology data of both compressively pre-deformed and torsionally pre-deformed olivine specimens are presented in Fig. 4.3 and Fig. 4.4.

Compressively pre-deformed specimens The few sol-gel olivine (D6646) data points in Fig. 4.3, from this study, plot somewhat lower but close to either combined flow laws by Hirth and Kohlstedt (2003) or Faul et al. (2011). Note that Hirth and Kohlstedt (2003) also discuss the possibility of dislocation accommodated grain boundary sliding as a deformation mechanism in polycrystalline olivine, with stress and grain size exponents of 2-3 and 1-2. However, for simplicity its implications are not discussed here.

Relatively fine grained ($\sim 13 \mu\text{m}$) San Carlos olivine (D6701) was significantly weaker than sol-gel olivine (D6646) at higher differential stresses. The rheology data appears to follow the same curve demonstrated by a flow law of sol-gel olivine from a previous study, showing evidence for possible Peierls exponential creep (Faul et al., 2011). However, the data is offset towards higher strain rates for given differential stresses. Given the larger grain size of the San Carlos olivine, it is surprising it plots near finer-grained sol-gel olivine for grain size dependent diffusion creep. The San Carlos data does not compare very well with a flow law (for $13 \mu\text{m}$ grain size) determined from previous ‘dry’ San Carlos olivine compressive deformation experiments (Hirth and Kohlstedt, 2003). However, it should be noted that the Hirth and Kohlstedt (2003) flow laws were based on grain sizes from light microscope measurements. A study has shown that EBSD measurements or orientation contrast images generally give smaller grain sizes than light microscopy measurements (Fliervoet et al., 1999). The deformed San

Carlos olivine (D6701) of this study contained an OH content of $\sim 400 \text{ H}/10^6 \text{ Si}$ ($\sim 26 \text{ wt. ppm H}_2\text{O}$) which may have made it weaker in the higher differential stress exponential creep regime.

Torsionally pre-deformed specimens Results from specimens T0434 and T0436, pre-deformed in torsion, show an exceptionally different rheology at 1250°C and 1300°C . Note that comparison with the compressively pre-deformed specimens is only possible for specimen T0434 deformed at 1250°C . This specimen, of somewhat smaller grain size and lower water content than D6701, may have shown reasonable strain rates for diffusional creep deformation for about 140 MPa equivalent stress. However, at higher stresses no transition to powerlaw or exponential creep is observed. The same is true for the sol-gel T0436. Nonetheless, microstructural evidence (Fig. 4.1) and dislocation density results demonstrate that both T0434 and T0436 must have been deformed at least partially in the dislocation creep regime. However, there was the possibility that for a given stress, the density of free dislocations first increased with increasing strain but reached a maximum and decreased as dislocation recovery processes took over at high temperature. Dislocation recovery sub-structures, as those observed in Fig. 4.1, suggest that dislocations reorganised into walls, which led to the formation of sub-grains, recrystallising the material.

It must be noted that torsional deformation only approximates simple shear deformation as there is always a finite contribution from axial compression affecting the microstructural results (Karato, 2008).

4.7.1.3 CPOs and slip systems in olivine

The slip systems that have been identified in olivine include $(010)[100]$, $(001)[100]$, $(0kl)[100]$ and $(hk0)[001]$ (Carter and Ave Lallemant, 1970; Nicolas et al., 1973; Durham et al., 1977) and their CPO development for axial compressive and (simple) shear deformation have been modelled for three or more operating slip systems (Ribe and Yu, 1991; Wenk et al., 1991; Castelnau et al., 2008). Transmission

electron microscopy studies have identified dislocations of both [100] and [001] Burgers vectors in natural olivine (Green and Radcliffe, 1972; Phakey et al., 1972). Both of these vectors also have been observed in sol-gel olivine deformed in the exponential (or powerlaw) creep regime (Faul et al., 2011).

Fig. 4.5 shows weak Type-A CPO (Jung et al., 2006; Karato, 2008) for most specimens but strongest for the torsionally pre-deformed sol-gel T0436. After annealing and torsional forced oscillation measurements, this material shows far weaker texture. This may be a result of pervasive recrystallisation observed in sections of A1231.

The pre-deformed San Carlos specimen T0434 was not subjected to the same peak temperature as T0436 for the attenuation experiment. During the tens of hours of annealing at 1000°C the kinetics of dislocation recovery and grain boundary migration were slow enough to avoid any significant microstructural changes (Fig. 4.5c and d). Comparing A1271 with T0434, all texture strength indicators (maximum MUD, M- and J-index) are higher after attenuation. This is difficult to understand – there is a slight possibility that the differences may be an artefact from tangential sectioning at a slightly different radius of the cylindrical specimen (see Chapter 2, section 2.2.3.3). It is unlikely that at 1000°C the low-amplitude torsional forced oscillations would have further deformed the specimen.

Unlike triaxial compressive deformation experiments on sol-gel olivine (e.g. 18% total strain) (Faul et al., 2011), San Carlos specimen D6701 (15% total strain) shows some indication of a CPO that can be associated with a particular set of slip systems, although this could have questionable significance because the patterns are weak as compared to most other deformed specimens for a maximum MUD value of 1.84.

The inverse pole figures (Fig. 4.6) for the torsionally pre-deformed olivine aggregates broadly agree with the type-A CPOs from Jung et al. (2006) deformed to somewhat higher shear stress of $\gamma = 1.2$, although showing very weak CPOs. Particular agreement is observed for a weak clustering of the [010] axes sub-

parallel to the shear plane normal, also observed for the minimum elongation of the strain ellipsoid. A1231 in Fig. 4.6b with an MUD between 0.63 and 1.21 is clearly demonstrating the most random patterns for each direction of interest. D6701 shows a hint of $[010]$ axes sub-parallel to the compression direction, also indicative of Type-A slip possibly involving some grain back-rotation during deformation.

The clear misorientation-axis pattern shown for pre-deformed specimens in Fig. 4.6 and preserved in annealed materials, indicates activation of the $(010)[100]$ slip system. A small rotation around $[010]$ is more indicative of slip on the (001) plane in the $[100]$ direction corresponding to activation of $(001)[100]$ slip system. These are the two easier slip systems in olivine characterising a Type-A CPO in the pole figures (Fig. 4.5 and 4.6). The third harder slip system $(010)[001]$ would require low angle axis pair rotations around $[100]$, not obvious in Fig. 4.7. In terms of dislocation sub-structures, a twist boundary of dislocations with $[100]$ and $[001]$ Burgers vectors would satisfy a twist $[010]$ twist axis. For a tilt boundary, rotation around $[001]$ would satisfy dislocations with $[100]$ and $[010]$ Burgers vectors. However, the latter corresponding to the greatest unit cell distance in olivine is rarely seen. The additional information obtained from light microscope analysis on T0434 (Fig. 4.7c) suggested sub-grain boundaries are roughly perpendicular to the $[100]$ direction in most grains. This is compatible with a rotation axis along $[001]$ on these boundaries.

4.8 Prospects for damping by dislocation glide in pre-deformed olivine

A brief Schmid factor analysis is given next to estimate the anelastic compressive and torsional strains for an olivine single crystal and randomly oriented crystallites in an aggregate (for detailed calculations, see Appendix C). The analysis is based on the results from Fig. 4.5, 4.6 and 4.7, from previous observations

of active slip systems in olivine (e.g. Carter and Ave Lallemant 1970; Durham et al. 1977; Karato 1988) and Appendix E that the dominant (high temperature) slip system in 'low-OH' olivine is (010)[100]. From this idealised scenario for a single slip system and assuming a homogeneous stress distribution between crystallites, a determination is made whether prior torsional deformation, as opposed to prior compressive deformation, might be expected to enhance the dissipation of shear strain energy (by bowing of favourably oriented dislocations) in torsional oscillation. This analysis provided the motivation to test both torsionally and compressively pre-deformed specimens for dislocation damping, the results of which are presented in Chapter 5.

4.8.1 Single crystals

Fig. 4.8 shows a slice from a single crystal olivine with an arbitrary crystallographic direction $[\alpha, \beta, \gamma]_c$ serving as the axis for uniaxial compression with compressive stress σ_c in (a) and for torsion with shear stress σ_t at radius r in (b). Each of these applied stresses are related through the direction cosines α , β and γ and azimuth θ to the resolved shear stress σ_{12} for (010)[100] dislocation glide. The components of the stress and strain tensors in a rotated reference frame $x'\{x'_1, x'_2, x'_3\}$ are related to those in the crystal co-ordinate frame xx_1, x_2, x_3 by expressions of the form

$$\kappa'_{ij} = a_{ik}a_{jl}\kappa_{kl} \qquad \kappa_{ij} = a_{ki}a_{lj}\kappa'_{kl}$$

where $a_{ij} = \cos \angle(x'_i, x_j)$ and summation is to be done over all values (1, 2 and 3) of repeated suffices (Nye, 1985).

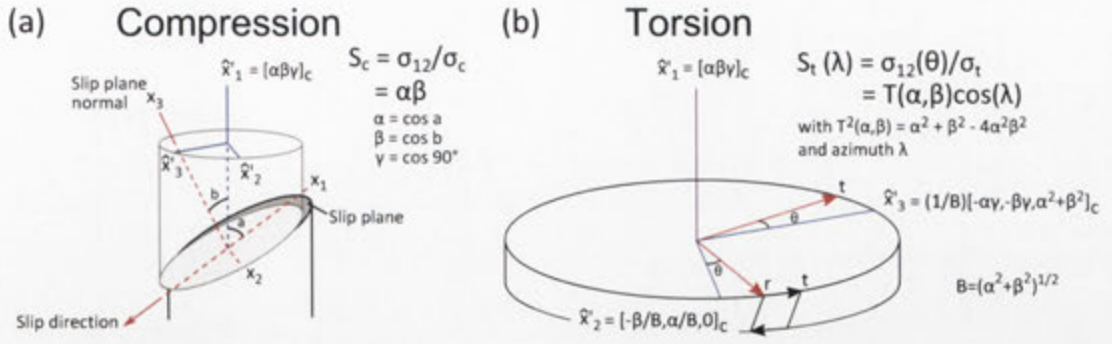


Fig. 4.8: Co-ordinate systems for consideration of compression parallel to and torsion around $[\alpha, \beta, \gamma]_c$. (a) A single crystal undergoing axial compression parallel to an arbitrary compression axis $[\alpha, \beta, \gamma]_c$. (b) A schematic of a single crystal in torsion oriented for twist around the same arbitrary axis. Note how the resolved shear stress changes relative to the slip direction with changing azimuthal position. See the text for explanation of the symbols.

4.8.1.1 Uni-axial compression

The resolved shear stress σ_{12} for (010)[100] slip for a single crystal deformed in axial compression parallel to $[\alpha, \beta, \gamma]_c$ is simply

$$\sigma_{12} = a_{11}a_{12}\sigma'_{11} = \alpha\beta\sigma_c \quad (4.8.1)$$

where σ_c is the compressive stress. The geometric function $S_c = \alpha\beta$ is known as the Schmid factor (e.g. Hull and Bacon 2001).

The contours of constant S_c are plotted in Fig. 4.9a for the (010)[100] slip system. The ideal orientation for a maximum S_c for this slip system is when $\alpha = \beta = 1/\sqrt{2}$ ($\alpha^2 = \beta^2 = 0.5$, compression along $[110]_c$).

Within the linear regime, (010)[100] dislocation glide in response to the resolved shear stress σ_{12} yields a proportional simple shear strain $\epsilon_{12} = \epsilon_{21}$ equivalent to an axial anelastic strain, per unit of dislocation density, given by

$$\epsilon_{an} = \epsilon'_{11} = 2\alpha\beta\epsilon_{12} \sim S_c^2\sigma_c \quad (4.8.2)$$

Note that only two of the direction cosines are independent as $\alpha^2 + \beta^2 + \gamma^2 = 1$.

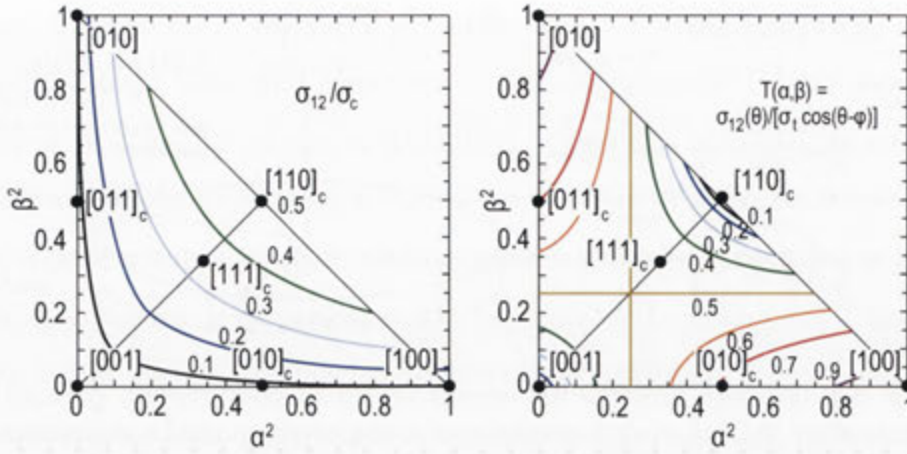


Fig. 4.9: Contours of resolved shear stress for $[100](010)$ glide for compression parallel to $[\alpha, \beta, \gamma]_c$. (a) The amplitude of the azimuth-dependent resolved shear stress for torsion (b) around the same axis.

4.8.1.2 Torsional deformation

Fig. 4.8b shows the co-ordinate systems relevant for torsion around an arbitrary axis $[\alpha, \beta, \gamma]_c$. The orthogonal system $\{\hat{x}'_1, \hat{x}'_2, \hat{x}'_3\}$ is related to the torsional co-ordinate frame $\{\hat{x}'_1, \hat{r}(\theta), \hat{t}(\theta)\}$ by expressions containing the relevant direction cosines a_{ij} given in Appendix C. The resolved shear stress for $(010)[100]$ dislocation glide is accordingly

$$\sigma_{12}(\theta) = -T(\alpha, \beta) \cos(\theta - \phi) \sigma_t \quad (4.8.3)$$

where

$$T^2(\alpha, \beta) = \alpha^2 + \beta^2 - (2\alpha\beta)^2 \quad \tan \phi(\alpha, \beta, \gamma) = (\alpha^2 - \beta^2)/2\alpha\beta\gamma$$

The contours of the normalised amplitude of the azimuth-dependent resolved shear stress $S_t = |\sigma_{12}(\theta)/[\sigma_t \cos(\theta - \phi)]|$ are plotted in Fig. 4.9b for the given slip system. It is apparent that torsional deformation around either the $[100]$ or $[010]$ axes will provide the maximum amplitude of the azimuth-dependent resolved shear stress.

The resolved shear strain for the linear anelastic regime is deduced (see Appendix C) to be

$$\begin{aligned}\epsilon_{an} &\cong T^2(\alpha, \beta) \cos^2(\theta - \phi) \\ &\cong (1/2)T^2(\alpha, \beta)[1 + \cos 2(\theta - \phi)]\end{aligned}\quad (4.8.4)$$

Anelastic relaxation by (010)[100] glide in pre-deformed olivine crystals has been determined for both compression (Eqn. 4.8.2) and torsion (Eqn. 4.8.4). For subsequent torsional deformation on a previous compressively pre-deformed olivine crystal, the anelastic relaxation is averaged over azimuth λ and normalised by elastic strain (neglecting azimuthal variation of the unrelaxed shear modulus) to give $\rho S_t^2(\alpha, \beta)$ where ρ is the dislocation density. For (010)[100] slip, the dislocation density ρ is proportional to σ_{12}^2 . Hence $\rho \propto S_c^2 \sigma_t^2$ and $\epsilon_{an} = S_c^2 S_t^2(\alpha, \beta)$. Equivalently, for prior torsional deformation $\rho \propto S_t^2 \sigma_t^2$ and $\epsilon_{an} = S_t^4(\alpha, \beta)$.

From this analysis, the resulting anelastic shear strains (for (010)[100] slip) can be estimated for either prior compressive deformation (Fig. 4.10a) or torsional deformation (Fig. 4.10b). In Fig. 4.10a, the amplitude of the contours for the azimuthally averaged torsional anelastic strain are significantly lower with a broad saddle extending from $(\alpha^2, \beta^2) \sim (0.15, 0.85)$ through a shallow local minimum near $(0.35, 0.35)$ to $(0.85, 0.15)$. Meanwhile, Fig. 4.10b demonstrates an azimuthally averaged torsional anelastic strain distribution of broadly similar shape to that of the resolved shear stress distribution in Fig. 4.9b as the history of previous torsional deformation is repeated.

Significantly, a crystal that is ideally oriented for (010)[100] slip in compression parallel to $[110]_c$, experiences zero resolved shear stress for glide on the same slip system in torsion. A compromise can be obtained for dislocation damping in a single crystal olivine pre-deformed in axial compression by pre-deforming it along $[111]_c$ to generate a population of (010)[100] dislocations that are also favourably oriented over subsequent torsional forced-oscillation measurements around this axis (see Gueguen et al. 1989).

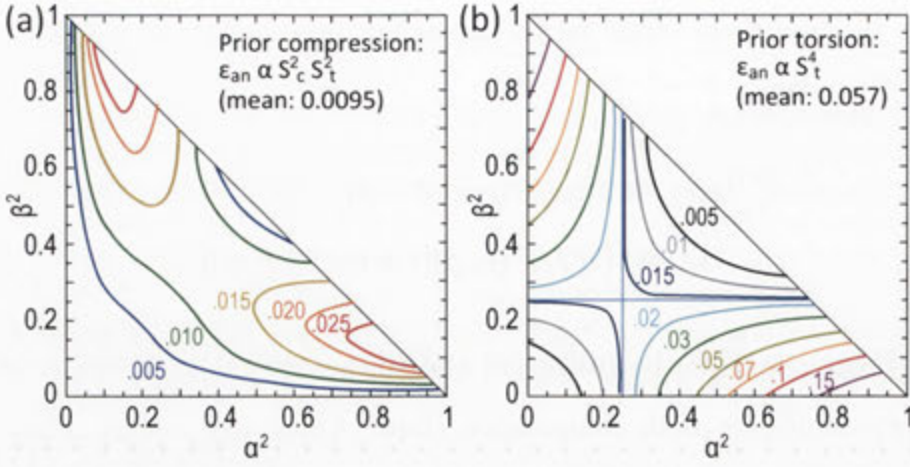


Fig. 4.10: Azimuthally averaged torsional anelastic strain after prior compressive deformation of single crystal olivine (a) and after prior torsional deformation of the same single crystal (b) around $[\alpha, \beta, \gamma]_c$ with (010)[100] slip.

4.8.2 Averaged anelastic shear strain in an aggregate

Thus far, the potential anelastic compressive and torsional shear strains have been analysed for a single crystal subject to compression along or torsion around a specific axis $[\alpha\beta\gamma]_c$. For an aggregate composed of randomly oriented crystallites, each exposed to the same uniform stress, the shear strains may be averaged for all possible orientations $[\alpha\beta\gamma]_c$. An expression for the averaged anelastic strain for both the compression and torsion cases is derived. In spherical co-ordinates the result is

$$\bar{\epsilon}_{an} = \int_0^\pi \int_0^{2\pi} p(\theta, \phi) \cdot \epsilon_{an} \cdot d\theta d\phi \quad (4.8.5)$$

where $p(\theta, \phi)d\theta d\phi = (\sin \phi/4\pi)d\theta d\phi$ is the probability that the compression or torsion axis unit vector lies within the solid angle subtended by an element of area on a unit sphere bounded by curves with θ and $\theta + d\theta$ and ϕ and $\phi + d\phi$, where $\phi = \cos^{-1}(\gamma)$ and $\theta = \tan^{-1}(\beta/\alpha)$ (note that the angles θ and ϕ , thus defined, are not those of Eqns. 4.8.3 and 4.8.4 – see Appendix C).

Re-expressed in terms of the angles θ and ϕ , the anelastic shear strain esti-

mated for compressive deformation becomes

$$\begin{aligned}\epsilon_{an} &\cong (\alpha\beta)^2 \\ &= 1/4(\sin^2 \phi \sin 2\theta)^2\end{aligned}$$

Following the trivial integration over θ and a subsequent integration by parts (Appendix C), the average strain is found to be $\bar{\epsilon}_{an} = 1/15$. The result compares to a maximum $[110]_c$ single crystal value of $(0.5)^2 = 1/4$.

For torsional deformation, the potential anelastic shear strain of each crystallite varies with azimuth about the torsional axis $[\alpha, \beta, \gamma]_c$ resulting in an azimuthally averaged value of

$$\begin{aligned}T^2(\alpha, \beta)/2 &= (1/2)[\alpha^2 + \beta^2 - (2\alpha\beta)^2] \\ \epsilon_{an} &= (1/2)[\sin^2 \phi - \sin^4 \phi \sin^2 2\theta]\end{aligned}$$

The average of this quantity over randomly oriented grains is

$$\bar{\epsilon}_{an} = \frac{1}{8\pi} \int_0^\pi \int_0^{2\pi} \sin^3 \phi - \sin^5 \phi \cdot \sin^2 2\theta \cdot d\theta d\phi \quad (4.8.6)$$

Integrating this expression twice yields a value for the averaged anelastic shear strain as $\bar{\epsilon}_{an} = 3/15$, compared to a maximum single crystal value for $[010]$ and $[100]$ of $1/2$.

As deduced from Fig. 4.10 and the analysis above, the averaged anelastic relaxation for prior compressive deformation in terms of Schmid factor is $[S_c^2 S_t^2]_{av} = 1/105$. However, the averaged anelastic relaxation for prior torsional deformation is $[S_t^4]_{av} = 6/105$, six times more effective.

4.9 Implications for dislocation damping

Assuming that dislocations multiply during deformation on preferred slip planes with restricted Burgers vectors and if the same dislocations are favourably oriented to vibrate and dissipate strain energy during low-amplitude forced oscillations (Granato and Lucke, 1956, 1981), then dislocation damping should be measurable in the laboratory (e.g. Gueguen et al. 1989). Moreover, the given geometrical analysis for Schmid factor and resolved shear stress in single and polycrystalline olivine is suggestive of enhanced anelastic shear strain in torsionally pre-deformed specimens. Because strain increases linearly outwards from the torsional axis (Paterson and Olgaard, 2000) a torsionally pre-deformed material would also be expected to have its dislocation density biased to be high in regions that will exert most leverage on subsequent measurements of forced torsional oscillation.

4.9.1 Torsionally pre-deformed specimens tested in high-temperature torsional forced oscillation

While favourably oriented dislocations in a torsionally pre-deformed specimen may contribute to higher dislocation damping, neither sol-gel T0436 nor San Carlos T0434 had developed very high dislocation densities. In addition, from inspection of the strain markers on the jackets, sol-gel T0436 appeared to have deformed inhomogeneously to $\gamma \sim 0.8$ at the upper end down to $\gamma \sim 0.1$ at the lower end. Because the dislocation densities were only moderate, it may be possible that the transition from diffusion creep near the torsional axis to dislocation creep at the rim of the cylindrical specimen was not fully achieved. Dislocation multiplication, which weakly occurred, was also likely subject to fast recovery kinetics at high temperature.

The torsionally pre-deformed specimens were quenched with their respective deformed microstructures frozen in. Evidence for a stored potential energy in the

deformed microstructures was encountered when T0436 and T0434 were reheated to 1100°C and 1000°C respectively. Two effects were measurable – anisotropic thermal expansion during heating, attributed to the fabric of the pre-deformed material and the gradual ‘untwisting’ of the specimen during high-temperature annealing – possibly associated with recrystallization.

A simple calculation of the axial stretches resulting from anisotropic thermal expansion of a single crystal olivine between $T_0 = 25$ and $T = 1100^\circ\text{C}$ can provide an indication of the maximum possible resulting twist. The elongation of an initial length l is given by $l(T) = l(T_0) \exp[\int_{T_0}^T \alpha(T) \cdot dT]$ where $\alpha(T) = a_0 + a_1T + a_2/T^2$ and a_0 , a_1 and a_2 are the thermal expansion coefficients (Fei, 1995). Hence, the axial stretches are calculated from thermal expansion on each axis as $\epsilon_{11} = 0.0104$, $\epsilon_{22} = 0.0149$ and $\epsilon_{33} = 0.0127$. The largest shear strain γ in the plane (110) is thus given by $(\epsilon_{22} - \epsilon_{11})/2 = 0.0023$. Such a shear strain in the tangential plane for a specimen of 5 mm radius r and 30 mm length L would yield a twist of $\phi = \gamma L/r \sim 0.0138$ rad. The corresponding maximum achievable displacements measured by the capacitance transducers for a distance of 231.1 mm off-axis would be 3.2 mm for optimally oriented single-crystal olivine. However, substantially smaller strains and displacements would be expected for a polycrystal with only a modest CPO. This effect was evident for torsionally pre-deformed San Carlos T0434 upon heating to 1000°C. Between 1000 – 700°C the twist was measured as 9.7×10^{-4} rad, which would imply a shear strain of $\sim 1.6 \times 10^{-4}$. The anisotropic thermal expansion required an additional offset of the transducer alignment.

The other noticeable effect was a tendency of T0436 to continuously cause drift in one direction between the transducer plates at 1100°C, not observed by the compressively deformed specimens. This happened to be in the opposite direction into which it was pre-deformed. A steady opposing DC torque of incrementally decreasing amplitude was applied to counteract this effect down to 1000°C where it was much reduced. However, during the initial ~ 50 hours of annealing at 1100°C much of the microstructure may have been reset (Fig. 4.5b).

4.9.2 All pre-deformed specimens

Processes such as a reduction in dislocation density, grain growth and recrystallisation evident during prolonged annealing at 1100°C intended to establish stable mechanical behaviour before forced oscillation measurements began. The initial and final dislocation density of the attenuation samples are given in Table 4.3 along with annealing times and the calculated values of expected final dislocation densities (see Chapter 3). The torsionally pre-deformed sol-gel T0436 experienced a reduction in dislocation density of more than 50% compared to the initial measured value. A larger reduction in dislocation density was measured only in the weakly compressively deformed sol-gel D6618, which has a high uncertainty in its dislocation density measurements. The other two compressively pre-deformed specimens D6646 and D6701 all experienced a higher dislocation recovery rate than predicted although not by the same amount as T0436 and D6618. The torsionally pre-deformed San Carlos T0434 did not undergo any apparent reduction in dislocation density at 1000°C. The difference in pressure (200 MPa) was unlikely to affect the dislocation recovery kinetics much. For a given activation volume, the rate constant k for dislocation recovery in single crystal olivine would be expected to decrease by < 0.5 log units for an increase in pressure to 200 MPa (Karato et al., 1993).

On the other hand, application of an alternative dislocation recovery model for single-crystal olivine (Karato and Ogawa, 1982) systematically overestimates the observed rate of dislocation recovery (Table 4.3). The oxygen fugacity conditions would have been approximately the same for the Ni₇₀-Fe₃₀ foil-wrapped specimen as in the complementary controlled-atmosphere ($P = 0.1$ MPa) static dislocation recovery experiments. Thus, the only remaining possible cause could be partly due migrating grain boundaries sweeping dislocations during preliminary low-amplitude forced oscillations at high temperature – a dynamic component to otherwise static recovery kinetics.

Both grain growth and recrystallization operated during annealing at peak

temperature prior to attenuation measurements. This is reflected in Fig. 4.2 for all San Carlos specimens but only the torsionally pre-deformed sol-gel olivine. In some cases grain growth was active (Fig. 4.2, panels 1b and 3b) and in other cases recrystallization led to a reduction in mean grain size (Fig. 4.2, panels 3a and 2c). There does not appear to be any systematic trend. T0434 experienced some grain growth at 1000°C while the dislocation density remained constant within measurement error (see Chapter 3). It may be worth noting that the biggest changes occurred in the San Carlos olivine specimens, with a relatively wetter (and weaker) rheology. For instance, the largest change in grain size is found to be for H6695 / A1279 likely assisted by a reduction in water concentration from 555 H/10⁶ Si to 284 H/10⁶ Si. This specimen, as mentioned earlier, had an unusual history with uncontrolled excursions to temperatures probably locally higher than 1300°C. It developed a significant micro-crack density as observed under the light microscope before it was reground and rerun for an attenuation experiment (see Appendix I). The new grain sizes for all the post-attenuation samples were assumed to have been reached during the initial tens of hours of annealing while establishing stable mechanical behaviour. Subsequent forced-oscillation data acquisition for periods of 1 – 1000 s were believed to have been carried out for a relatively stable grain size in the specimens.

The dynamic microstructural evolution of the pre-deformed olivine polycrystals must be taken into account when analysing shear modulus G and attenuation data Q^{-1} presented in Chapter 5. Any indication for larger Q^{-1} above the background dissipation from anelastic grain boundary sliding or mobility of water defects in the pre-deformed specimens, compared to hot-pressed specimens of lower dislocation density, provides evidence for dislocation damping.

4.10 Summary

Seismic attenuation in olivine is dependent on many physical mechanisms that contribute to strain energy dissipation. In order to study the effects of one com-

ponent, the effect of dislocation damping, controlled laboratory conditions were required. Two types of synthetic olivine materials were chosen for this study – ‘low-OH’ fine-grained sol-gel olivine and coarser-grained San Carlos olivine. Both types were hot-pressed from powders, pre-deformed in compression and torsion and subjected to tens of hours of annealing at high temperature prior to attenuation measurements.

In this Chapter, a detailed microstructural and rheological examination was given to document the evolution of dislocation density, grain size and water content of all the specimens to provide a background against which seismic forced oscillation data will be interpreted (see Chapter 5). In addition, a theoretical analysis was developed to compare compressive and torsional pre-deformation in an attempt to identify conditions conducive to dislocation damping in olivine. In summary, the following results were obtained.

- Sol-gel and San Carlos olivine aggregates were successfully pre-deformed in compression. San Carlos olivine with larger grain size ($\sim 13 \mu\text{m}$) was weaker than sol-gel olivine for high differential stresses consistent with exponential creep rather than powerlaw creep. However, it was stronger for low differential stresses.
- Similar olivine aggregates were pre-deformed in torsion to relatively high equivalent stress and strain but developed only moderate dislocation densities. A weak shape preferred orientation was observed in the sol-gel sample. Intermittent slip at the sample-alumina disc interfaces and mostly diffusion creep throughout the bulk of the specimens may have contributed to low stress exponents and no apparent transition into the dislocation creep regime.
- Dislocation recovery kinetics inferred from the post-attenuation specimens appeared faster than predicted from the complementary static recovery experiments. While two compressively pre-deformed specimens kept a rel-

atively high dislocation density, texture in the torsionally pre-deformed sol-gel specimen was completely reset removing pre-existing CPO and a low dislocation density was reached close to that of a hot-pressed specimen. The remaining torsionally pre-deformed San Carlos specimen was mechanically tested at a conservative 1000°C to avoid similar microstructural re-adjustment.

- Grain size evolution during annealing prior to attenuation experiments occurred in most specimens, although more in the ‘wetter’ San Carlos specimens. There does not appear to be any systematic trend but all changes during annealing prior to forced oscillation measurements must be driven by a reduction in stored potential energy in the quenched pre-deformed specimens.
- CPOs in the pre-deformed specimens were generally weak to random but strongest in the torsionally pre-deformed sol-gel sample with the highest fabric strength, quantified by the M- and J-indices. Activation of multiple slip systems was found, most notably (010)[100] and (001)[100].
- For simplicity, (010)[100] was taken to be the active slip system for theoretical considerations of damping by dislocation glide. It was found that dislocations generated during prior compressive deformation experience a substantial resolved shear stress in subsequent torsional oscillation experiments only for orientations in a swath through $[111]_c$.
- Assuming a uniform stress distribution on crystallites in an aggregate, prior torsional deformation was determined to be 6 times more effective than prior compressive deformation for anelastic relaxation in torsion.

Despite the experimental challenge to successfully prepare and pre-deform large ~ 30 mm long sol-gel and San Carlos cylindrical aggregates for attenuation measurements at high temperature and moderate confining pressure, these were

the right materials to use in order to obtain greater insight into anelasticity and seismology of the upper mantle in tectonically active regions of the Earth where anomalously high Q^{-1} values are measured.

*The great virtue of man lies
in his ability to correct his mistakes
and to continually make a new man of himself.*

Wang Yang-Ming (1472 – 1529)

5

Dislocation damping in polycrystalline olivine II: Laboratory measurements of shear modulus dispersion and attenuation with seismological implications

5.1 Abstract

Torsional forced oscillation tests of olivine polycrystals described in this chapter were carried out on specimens for maximum temperatures of 1200°C (hot-pressed) and 1000 – 1100°C (pre-deformed) providing dislocation densities of 0.5 – 11.4 μm^{-2} . Dislocation damping mechanisms via string vibration and kink migration were presented as background to the experimental search for dislocation damping. The comparison of behaviour of un-deformed and pre-deformed materials was intended to reveal the additional viscoelastic relaxation attributable to

stress-induced migration of dislocations. However, complications in the interpretation of data for some specimens forced a change of plan. An alternative baseline for comparison of ‘un-deformed’ and compressively and torsionally pre-deformed sol-gel and torsionally pre-deformed San Carlos olivine was chosen – a Burgers model based on an extensive suite of experiments performed on essentially dry melt-free olivine, including the hot-pressed sol-gel of this study. Significant enhancement of the dissipation and modulus dispersion for the pre-deformed specimens is observed, greatest for the torsionally pre-deformed San Carlos specimen, tested only up to 1000°C at which the dislocation sub-structures were preserved. Enhancement of background rather than development of peaks suggests that the stress-induced migration of dislocations might couple closely with the dominant grain-boundary sliding relaxation mechanism. Excursions to larger maximum strains $\sim 10^{-4}$ demonstrated a clear deviation from linear stress-strain behaviour and a strain dependence on Q^{-1} – probably an artefact associated with flow of gas pressure medium within the displacement transducers. Complementary torsional microcreep tests showed that the torsionally pre-deformed specimens had the highest creep rates. Any non-linearity of dislocation relaxation potentially caused by the unpinning of dislocation segments, at strain amplitudes significantly $> 10^{-5}$ was beyond the resolution of these experiments. The observed relationship between dislocation-related dissipation and the orientation of the stress field during prior deformation suggests the possibility of seismological attenuation anisotropy. While dislocation damping has been demonstrated in the laboratory for pre-deformed fine-grained olivine polycrystals, it remains to be seen whether upper mantle materials, deforming in steady-state creep, have sufficiently high dislocation densities for a significant contribution to dislocation damping.

5.2 Introduction

Two suites of Fo₉₀ olivine polycrystals were successfully hot-pressed from sol-gel and San Carlos precursors in the laboratory in anticipation of deformation in

the dislocation creep regime and subsequent micro-strain torsional oscillation / microcreep testing. After hot isostatic pressing, the sol-gel specimens had an average grain size between 3 – 4 μm whereas the San Carlos material was more coarse-grained ($\sim 13 \mu\text{m}$). Microstructural characterisation before and after each deformation and attenuation experiments demonstrated tendencies towards both grain growth and recrystallisation, more pronounced in wetter specimens.

Sol-gel and San Carlos specimens were deformed in either compression or torsion at high stresses to provide access the regime of dislocation creep. The resulting dislocation densities were up to an order-of-magnitude higher than for undeformed, hot-pressed specimens (Chapter 4). Sub-grain development was more pronounced in the somewhat coarser grained San Carlos materials than in the finer grained sol-gel specimens. Nonetheless, sub-boundaries dominantly parallel to the (100) plane, demonstrated misorientation axes involving rotation around [001], suggestive of (010)[100] slip. A weak shape preferred orientation was apparent in the torsionally most-highly pre-deformed sol-gel. Electron backscatter diffraction mapping of selected pre-deformed and post-attenuation olivine specimens demonstrated only weak to random crystallographic preferred orientations (CPO) most closely resembling Type-A patterns (Jung et al., 2006).

Hence, an analysis was presented of the resolved shear stress for dislocation glide on the favoured (010)[100] slip system and of the resulting resolved shear strain for compressive and torsional deformation geometries. It suggested that the population of dislocations resulting from prior deformation in torsion might be more favourably oriented for glide and hence viscoelastic relaxation during the subsequent micro-strain torsional oscillation / microcreep testing than those generated by prior compressive deformation.

In addition, static dislocation recovery experiments, conducted under controlled atmosphere (0.1 MPa) on the same pre-deformed sol-gel and San Carlos materials of high dislocation density, demonstrated that new rate constants k for dislocation recovery in polycrystalline olivine are one or two orders of magni-

tude lower than in single crystal olivine (Karato and Ogawa, 1982; Karato et al., 1993). Nonetheless, for the duration of > 50 hour annealing at peak temperature to stabilise mechanical testing for forced oscillation / microcreep measurements, temperatures greater than 1100°C were not feasible for maintenance of 'stable' dislocation microstructures. At 1100°C a reduction of $\sim 20\%$ in dislocation density was expected for a duration of 50 hours of annealing for both sol-gel and San Carlos olivine (Chapter 3), acceptable for testing anelastic and viscoelastic relaxation of dislocations at the highest accessible temperature.

Following recovery of the pre-deformed specimens after forced-oscillation / microcreep testing, it was found that the dislocation densities were generally lower than predicted by the static recovery kinetics for maximum temperatures of 1100°C . The torsionally pre-deformed sol-gel, tested at 1100°C , was most seriously affected, having undergone persuasive microstructural change, including loss of its crystallographic preferred orientation (CPO). However, the dislocation microstructure remained essentially unchanged for a specimen tested to the lower maximum temperature of 1000°C . Thus the measurement of micro-strain viscoelastic behaviour at high temperatures whilst maintaining a stable dislocation density is very challenging. The necessary compromise between temperatures high enough for appreciable viscoelastic relaxation, yet low enough to avoid extensive static dislocation recovery results in a narrow window ($900 - 1100^{\circ}\text{C}$) for such measurements.

In Chapter 4 (Part I) suites of sol-gel were prepared and characterised for an exploratory study of dislocation damping. Hot isostatic pressed specimens of each were tested to establish the baseline behaviour of dominantly grain-boundary relaxation at low dislocation densities. Compressively and torsionally pre-deformed specimens were used with populations of dislocations potentially less- and more favourably oriented for activation in the forced-oscillation / microcreep tests. This systematic approach seeks to identify and provide insight into the mechanical response of reasonably stable dislocation microstructures during the high-

temperature forced oscillation / microcreep testing.

In this Chapter (Part II), shear modulus dispersion and attenuation data on pre-deformed sol-gel and San Carlos olivine are presented for the first time. Previous attenuation data on melt-free hot-pressed synthetic olivine of various grain size have demonstrated a sub-solidus viscoelastic relaxation associated with a broad absorption band likely to be associated with strain energy dissipation from grain boundary sliding (Tan et al., 2001; Jackson et al., 2002; Cooper, 2002; Faul and Jackson, 2005). Here a search was performed for additional viscoelastic relaxation predicted by the string vibration and kink migration models.

Micro-strain forced oscillation experiments conducted on un-deformed and pre-deformed specimens revealed an enhanced absorption background suggesting a close involvement of dislocations in the dominant grain boundary relaxation processes. Linearity and recoverability was accessed through the variation of forced-oscillation strain amplitudes and torsional microcreep tests. Finally, possible seismological implications are briefly explored.

5.3 Viscoelastic relaxation related to stress-induced dislocation motion

The Burgers model for linear viscoelastic rheology, introduced in Chapter 2, section 2.3.5 provides a phenomenological description of several parameters, such as the distribution of relaxation times $D(\tau) \sim \tau^{\alpha-1}$ and anelastic relaxation strength Δ . A characteristic relaxation time and strength can be predicted for the anelastic (recoverable) and viscoelastic (irrecoverable) vibration and motion of dislocations. There are two scenarios in which dislocation damping can be envisaged: the vibrating-string and kink migration models. Linearity is assumed by the models presented here. A detailed review of these models and implications are given by (Jackson, 2007), a brief summary is given here.

5.3.1 Dislocation relaxation models for linear behaviour

5.3.1.1 Vibrating string model

The vibrating string model has been developed for mechanical dislocation damping since Koehler (1952); Granato and Lucke (1956). A dislocation line with length L and Burgers vector b is pinned at two ends and bows outwards from its glide plane when an applied shear stress is exerting a force σb per unit length (Fig. 5.1a). This displacement is counteracted by the tension in the dislocation line, equivalent to the elastic strain energy per unit length $E_d \sim (1/2)Gb^2$ where G is the (unrelaxed) shear modulus. The dislocation continues to bow out under the influence of a continuous steady shear stress σ until the equilibrium curvature with radius R is reached and $R = Gb/2\sigma$ (Fig. 5.1a). Linear behaviour is only guaranteed without dislocation multiplication. Thus it is required that $(\sigma/G) < (b/d)$ where d is the grain size which imposes an absolute upper limit on the dislocation length.

For a pinned oscillating dislocation, an equation of motion expressing the force balance on unit length of dislocation is given by Granato and Lucke (1956). When the relaxation strength is $\ll 1$, the expressions of the relaxation time τ and strength Δ are approximated as

$$\tau \sim (1/6)BL^2/Gb^2 \quad (5.3.1)$$

$$\Delta = (1/6)\rho L^2 \quad (5.3.2)$$

(Granato and Lucke, 1956), where B is the drag coefficient and ρ is the dislocation density.

5.3.1.2 Kink migration models

Unlike the vibrating string model, the kink migration models do not assume constant elastic strain energy E_d . Instead E_d varies with an average value with

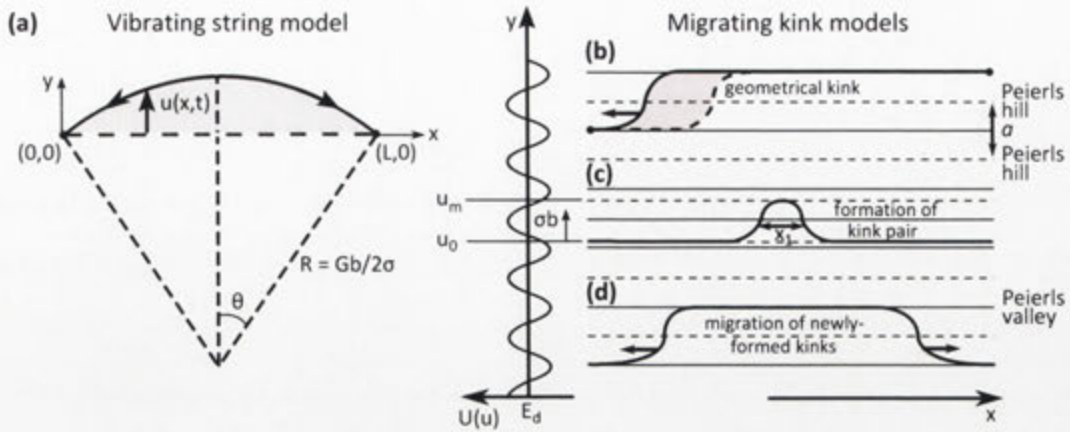


Fig. 5.1: String vibration and kink migration models for a stress-induced glide of dislocation segments in response to an externally imposed shear stress. (a) A dislocation line of length L bows out by an applied oscillating stress for a distance $u(x,t)$. Longer period forced-oscillations allow the dislocation line to bow out further than for shorter periods. An equilibrium is reached when the inward force from line tension $f = 2Gb^2/R$ equals the applied force $F = \sigma b$. The equilibrium curvature is thus $R = Gb/2\sigma$. (b-d) Modulation of the energy E_d of a straight dislocation by the Peierls potential $U(u)$ affecting nucleation and migration of kinks (redrawn after Seeger (1981); Jackson (2007)).

position u inside the crystal lattice. This is described by the Peierls potential $U(u)$ (Fig. 5.1b,d). For silicate minerals, strong covalent bonding and/or a large unit cell ensures large values of $U(u)$ thereby keeping dislocation lines preferentially inside low-energy Peierls valleys (Karato, 1998). However, a periodic applied stress may push part of a dislocation line over a high-energy valley by the lateral motion of a pre-existing (geometrical) kink (Fig. 5.1d and see e.g. Seeger 1956). From a similar equation of motion given by Granato and Lucke (1956), new expressions for the relaxation time and strength were derived for geometrical kink migration by Brailsford (1961) as

$$\tau = (L/\pi)^2/D_k \quad (5.3.3)$$

$$\Delta \approx (4/5\pi^4)\rho L^2(ab^2G/kT) \quad (5.3.4)$$

where D_k is the kink diffusivity expressed with a Boltzmann exponential term with the free energy of kink migration, a is the spacing between adjacent hills

of maximum Peierls potential energy and k is the Boltzmann constant. In this evaluation Brailsford (1961) allowed for the thermal generation, recombination and stress-induced migration and diffusion of kinks.

The analyses given above of the migration of geometrical kinks and vibration of a pinned dislocation line, happen to share the same scaling characteristics whereby $\tau \propto L^2$ and $\Delta \propto \rho L^2$.

In addition, as noted by Karato (1998); Jackson (2007), the maximum anelastic strain resulting from the migration of geometrical kinks can be expressed as

$$\epsilon_{an} = b\rho L \sin \phi \cos(\phi/2) \quad (5.3.5)$$

where ϕ is the angle between an initial dislocation line pinned across several Peierls valleys (and peaks) and along a valley (or peak). The expression can be compared to strains obtained in this study for given dislocation densities in the pre-deformed materials.

A more extensive analysis of the formation and migration of kink pairs (Fig. 5.1c) of finite width is given by Seeger (1981). The resulting expressions for the relaxation time and strength are

$$\tau = [kT/(\rho_k^{eq})^2 D_k](L/2a^2 E_d)(1 + \rho_k^{eq} L) \quad (5.3.6)$$

$$\begin{aligned} \Delta &= \rho L^2 b^2 G_u / 12 E_d \\ &\approx (1/6) \rho L^2 \end{aligned} \quad (5.3.7)$$

where ρ_k^{eq} is the equilibrium density of kinks of given sign per unit length of dislocation line (including a Boltzmann exponential term with the free energy of kink migration) and G_u is the unrelaxed shear modulus. As observed, the relaxation strength is identical to that of the vibrating string model. This is reflected in the geometrical considerations of both models. In addition, the relaxation times can also be compared. The drag coefficient in the vibrating string model becomes equivalent to $B = 6kT/\rho_k^{eq} D_k a^2$ for high kink densities in the high temperature

regime for the kink migration model.

Computed relaxation times for Seeger's (1981) kink model of dislocation mobility as a function of the reciprocal of temperature are presented by Jackson (2007, Fig. 12) for a given set of parameters. The calculations suggest migration of geometrical kinks could result in anelastic relaxation within the seismic frequency band for typical upper mantle temperatures. However, kink-pair formation and migration exhibit much longer relaxation times ($\sim 10^4 - 10^{6.5}$) at the highest plausible upper mantle temperatures (~ 1400 °C) unless the effective activation energy is reduced by 100 kJ mol^{-1} for given dislocation lengths between $1 - 100 \text{ }\mu\text{m}$ (Jackson, 2007).

In summary, the analysis given above suggests that dislocation damping would provide a substantial relaxation strength $\Delta \sim 0.1 - 1$ for favourably oriented dislocations undergoing stress-induced migration and vibration. Large values are expected when the dislocation density approaches that of a three-dimensional (Frank) network, especially for closely-spaced dislocations in walls of sub-grains (Nowick and Berry, 1972). In addition, the internal friction Q^{-1} can be generalised to accommodate a wide distribution of dislocation segment lengths for relaxation times $\tau(L) \sim L^2$. If $Q^{-1} \sim T_0^{-\alpha}$ from laboratory experiments, then it is required that the distribution of dislocation segment lengths $p_L(L)$ adopt the form $p_L(L) \sim L^{2\alpha-4}$, strongly favouring short dislocation lengths (Minster and Anderson, 1981; Karato, 1998; Jackson, 2007).

5.3.2 Transition from anelastic to viscoelastic behaviour

Dislocation relaxation likely approaches non-linearity towards higher temperatures and/or higher stress amplitudes. At the beginning of the previous section 5.3.1.1, an expression was derived that imposes an upper limit on linear stress-strain behaviour: $(\sigma/G) < (b/d)$. Given a maximum dislocation length for polycrystalline olivine in this study ($L = d = 5 \times 10^{-6} \text{ m}$) and a Burgers vector of $5 \times 10^{-10} \text{ m}$, the critical elastic strain amplitude is approximately 1×10^{-4} . How-

ever, dislocation segments are generally much smaller than the average grain size due to pinning on other dislocations or impurities – expanding the field of linear behaviour. Thus dislocation multiplication appears unlikely for current experiments, given the small grain size and standard strain amplitudes between 10^{-6} and 10^{-5} .

However, processes such as the unpinning of dislocation lines resulting in irrecoverable viscous behaviour, may have well played a role in modifying the density of free dislocations during static annealing at relatively high temperature ($\leq 1000^\circ\text{C}$). The unpinning of dislocations during forced-oscillation testing would enable larger irrecoverable strains to be reached. A critical stress for unpinning (at 0 K) was estimated as

$$\sigma_{up} = E_{up}/abL \quad (5.3.8)$$

where E_{up} is the binding energy of the dislocation to the pinning point(s). For a temperature above absolute zero, σ_{up} will be somewhat lower for thermally-assisted unpinning, proportional to $\exp[-(E_{up} - \sigma abL)/kT]$ (Nowick and Berry, 1972, pp. 364-365). In this study, for a range of maximum dislocation lengths $L = (1 \text{ to } 10) \times 10^{-6} \text{ m}$, an activation energy of $\sim 400 \text{ kJ mol}^{-1}$ and $a \sim b \sim 5 \times 10^{-10} \text{ m}$, would yield a range of unpinning stresses to be of order 0.1 – 1 MPa corresponding to shear strain amplitudes of order 10^{-6} to 10^{-5} . Hence, a more strongly frequency-dependent attenuation ($Q^{-1} \sim \omega^{-1}$ in the viscous regime) is expected for laboratory experimentation (Karato and Spetzler, 1990; Jackson, 2007).

5.4 Experimental methods and data analysis

5.4.1 Brief overview of olivine specimens

As described in Chapter 4, two types of fine-grained synthetic specimens were used for seismic-frequency measurements: sol-gel derived and natural San Carlos

derived olivine. They were prepared from powders and hot-pressed into dense aggregates. Several selected hot-pressed specimens were subsequently deformed in compression or in torsion (at the University of Minnesota) as described in Chapter 4. Note that hot-pressed are denoted by Hxxxx. Likewise compressively and torsionally pre-deformed specimens are denoted by Dxxxx and Txxxx respectively. Attenuation runs on those specimens are identified by Axxxx.

Each cylindrical specimen, whether simply hot-pressed or also pre-deformed, was precision-ground to 11.5 or 10.0 ± 0.001 mm diameter and maximum length (in the range 24 – 33 mm). Some specimens were cut from their previous assembly and retained their original mild steel jacket and Ni₇₀-Fe₃₀ foil wrapper (T0434, T0436, H6695) in order to minimize the possibility of fragmentation of the specimen. Each such foil-wrapped and jacketed specimen was precision ground to a foil and jacket thickness of ~ 0.25 mm. These specimens were kept in a vacuum oven at $\sim 100^\circ\text{C}$ and transferred to an ordinary oven at $\sim 180^\circ\text{C}$ in air prior to an experiment. Other specimens that were treated by acid dissolution (in diluted nitric acid), were re-fired at 1000°C , lower than the usual 1200 – 1400 $^\circ\text{C}$, to avoid (significant) dislocation recovery and similarly heat treated at $\sim 180^\circ\text{C}$ in air awaiting attenuation experiments. These procedures ensured the elimination of adsorbed water and any nitrates residual from acid dissolution of the foil and jacket. For more details, see Chapter 2.

5.4.2 Mechanical testing procedures

All specimens except T0434 and H6695 were newly wrapped with Ni₇₀-Fe₃₀ foil and sandwiched between tapered LucaloxTM alumina torsion rods (of either 11.5 or 10.0 mm minimum diameter) with 70 μm thick Ni₇₀-Fe₃₀ discs at either end. The lengths (l_{ass}) of the specimen assemblies are given in Table H.2 in Appendix H.

In one of the specimens recovered following forced oscillation/microcreep measurements (A1232), it was found that one of the foil disks had inadvertently been

dislodged during assembly, leaving the ground alumina torsion rod in direct contact with the specimen (A1232). Compliance and phase angle information from ‘double-foil’ and ‘foil-free’ reference assemblies (see Jackson et al. 2009) were averaged to process the mechanical data of this specimen (see section 5.4.3). In addition, the hot-pressed San Carlos specimen (A1279) experienced a complicated history whereby it was exposed to temperatures possibly $> 1300^{\circ}\text{C}$ during two furnace failures and had developed a substantial increase in micro-crack density (see Appendix I).

A switch to 10.0 mm diameter specimens was made part-way through this project because preliminary torsion deformation experiments failed to deform olivine specimens of 11.5 mm diameter. These preliminary experiments, conducted with and without foil disks, were plagued by persistent slip at the alumina-specimen interfaces. More successful were torsional deformation experiments on 10.0 mm diameter cylindrical specimens.

Each specimen assembly was inserted into the attenuation apparatus as described in Chapter 2 and a Pt/Pt10%Rh type-R thermocouple was lowered into the hollow alumina piston to a position 5 mm above the specimen to monitor the temperature of the hot-zone. Initial pressurisation to 200 MPa Ar confining pressure at room temperature, caused the jacket to collapse into longitudinal and tangential grooves in the steel torsion members at each end of the specimen assembly, ensuring that the entire assembly thereafter behaves as a monolithic mechanical unit. The capacitance displacement transducer plates were systematically offset to allow for reproducible distortions caused by pressurisation and heating of the specimen assembly. A period of annealing (30 – 70 hours) at a given peak temperature and 200 MPa confining pressure and short period (1 – 100 s) forced oscillation measurements followed, to monitor any temporal evolution of mechanical response to oscillating torque – presumably reflecting stabilisation of the microstructure. After stable conditions were established, forced oscillation measurements for 10 selected periods between 1 and 1000 s were taken for

temperatures typically spaced by 25 or 50°C during slow staged cooling to room temperature. The furnace was consistently cooled at a rate of 300° h⁻¹ to avoid cracking in the alumina torsion rods. During routine forced-oscillation measurements, the maximum shear stress was ~ 0.4 MPa corresponding to a maximum shear strain of 10^{-5} (see Table 5.1) at 1100°C. Modifications to the apparatus during the course of this project enabled excursions to higher torque amplitudes and thus higher strains to test for any amplitude dependence diagnostic of non-linear behaviour.

Complementary torsional microcreep tests were conducted to provide information on the extent of recovery of the non-elastic strain after removal of the steady torque. Typically the response becomes elastic below 900°C. Detailed information on how a microcreep test is performed is given in Chapter 2 and as described by Jackson (2000).

5.4.3 Data processing

The raw displacement data from each pair of displacement transducers were first processed by scaling the displacement amplitudes $d_2(t)$ and $d_1(t)$ for the upper and lower transducer stations to yield the same difference ($d_2(t) - d_1(t)$) associated with distortion of the elastic standard. $d_1(t)$, thus scaled, measures the response of the specimen assembly to the prescribed amplitude of oscillating torque. The complex torsional compliance and phase angle were hence determined and compared to experimental results of a reference assembly fitted with an alumina control specimen in place of the rock specimen. In processing the compliance and phase angle data from the experimental assemblies to obtain the shear modulus and dissipation for the specimen, generally small corrections are made for geometrical differences between the specimen and reference assemblies as described in Chapter 2 and listed in Table H.2 in Appendix H.

The resulting shear modulus and attenuation data were subsequently data for each specimen were subsequently fitted to a background-only extended Burgers model. In brief, the following strategy was used (see Chapter 2 for a full description). Generally, only oscillation data for temperatures $\geq 900^\circ\text{C}$ were modelled, except for A1271 (T0434) for which the maximum temperature was only 1000°C . The relaxation times defining the anelastic absorption band (τ_L, τ_H) are referred to values at the reference conditions of pressure ($P_R = 0.2 \text{ GPa}$), temperature ($T_R = 900^\circ\text{C}$ and grain size d_R equal to the measured average grain size. The value of the unrelaxed shear modulus at the reference conditions ($G_{UR} = G_U(T_R, P_R)$) was taken as 66.5 GPa consistent with accepted high-frequency data concerning the elastic properties of Fo_{90} olivine and their temperature and pressure derivatives (-13.6 MPa K^{-1} and 1.8 respectively). The activation volume is only of minor importance as $P = P_R$ throughout the data analysis presented here. A very small contribution of PV to the activation enthalpy ($E + PV$) exists.

Several parameters were fixed to reduce ambiguity and constrain the physical model. This is highlighted in Table 5.1 by the square brackets. The unrelaxed

shear modulus was not permitted to exceed the established reference value of 66.5 GPa. The interval ($\log_{10} \tau_{LR}$, $\log_{10} \tau_{HR}$) was specified as fixed values of [-2], [6.5] for a general best fit, allowing the anelastic absorption band to just span the observational 1 – 1000 s window for all temperatures between 800 and 1200°C.

The other parameters (Δ_B , α , $\log_{10} \tau_{MR}$, E_B and sometimes G_{UR}) shown in Table 5.1, were refined using the Levenberg-Marquardt algorithm, which minimises the misfit (χ^2) of the modulus and dissipation data through iterative adjustment of the values of the model parameters. The given parameters describe respectively the anelastic relaxation strength for the background dissipation, the shape of the distribution $D(\tau) \sim \tau^{\alpha-1}$ of the anelastic relaxation times, the Maxwell relaxation time, the activation energy for the temperature-dependent relaxation times and the unrelaxed shear modulus at reference conditions. The average misfit is given by the value of $(\chi^2/2N)^{1/2}$, where N is the number of (G, Q^{-1}) data pairs being fitted. The a priori errors were specified as $\sigma(G)/G = 0.03$ and $\sigma(\log_{10} Q^{-1}) = 0.05$ and perseverance in optimising the fit generally yielded a satisfactory model with $(\chi^2/2N)^{1/2}$ not much greater than 1. The quality of the fits were systematically improved per specimen by excluding the most serious outliers, occasionally short period data and generally the most scattered data below $\log_{10} Q^{-1} = -2$. For the torsionally pre-deformed specimens A1231 (T0436) and A1271 (T0434), the data were appreciably more scattered with higher values of the misfit parameter.

Table 5.1: Extended Burgers model parameters. The data comprise N (G, Q^{-1}) pairs for uniformly spaced oscillation periods between 1 and 1000 s and temperatures of 900-1200°C

	γ_{max}^*	G_U GPa	$\partial G_U / \partial T$ MPa K ⁻¹	Δ_B	α	$\log_{10} \tau_{LR}$	$\log_{10} \tau_{HR}$	$\log_{10} \tau_{MR}$	E_B kJ mol ⁻¹	χ_G^2	$\chi_{1/Q}^2$	N	$(\chi^2/2N)^{1/2}$
Sol-gel													
A1127 (H6585)	$\sim 10^{-5}$	60.39 (.27)	[-13.6]	1.895(.146)	.3241(.0090)	[-2]	[6.5]	5.408 (.079)	321.6 (8.2)	67.0	89.5	66	1.18
A1156 (D6618)	4.6×10^{-6}	64.58 (.25)	[-13.6]	.7153 (.051)	.3045 (.0088)	[-2]	[6.5]	5.819 (.083)	481.1 (12.8)	52.5	113.3	66	1.26
A1161 (D6646)	1.0×10^{-5}	65.08 (.28)	[-13.6]	2.031 (.160)	.3531 (.0097)	[-2]	[6.5]	5.141 (.071)	423.5 (9.8)	25.0	61.2	65	0.66
A1231 (T0436)	4.6×10^{-6}	[66.5]	[-13.6]	2.482 (.212)	.4039 (.0102)	[-2]	[6.5]	5.646 (.093)	465.9 (10.1)	149.8	158.0	61	2.52
San Carlos													
A1279 (H6695)	1.0×10^{-5}	[66.5]	[-13.6]	2.105 (.132)	.3816 (.0076)	[-2]	[6.5]	6.351 (.082)	436.1 (7.9)	51.6	114.3	65	1.28
A1232 (D6701)	7.4×10^{-6}	65.21 (.25)	[-13.6]	.3417 (.034)	.3929 (.0104)	[-2]	[6.5]	7.031 (.134)	538.8 (15.1)	23.4	155.5	61	1.47
A1271 (T0434)	7.8×10^{-6}	64.67 (.30)	[-13.6]	1.931 (.210)	.2517 (.0122)	[-2]	[6.5]	4.081 (.071)	339.1 (15.0)	45.8	161.9	53	1.96

* Maximum shear strains for 1000 s period oscillations at 1100°C, except for A1271 at 1000°C

† Square brackets indicate fixed parameters and round brackets indicate the calculated uncertainty

‡ Grain size d_R , equivalent to grain size of each specimen (see Chapter 4), $G_{UR} = 66.5$ GPa, $T_R = 900^\circ\text{C}$, $P_R = 0.2$ GPa, $V^* = 6 \times 10^{-6}$ m³ mol⁻¹, $\sigma G/G = 0.03$, $\sigma(\log_{10} Q^{-1}) = 0.05$

5.5 Results

5.5.1 Strain-amplitude dependence of G and $1/Q$

More powerful electromagnetic drivers were installed during the later stages of experimentation, which provided access to larger values of torque and hence also strain, by almost an order of magnitude. Forced torsional oscillation tests (1 – 100 s periods) were carried out on two olivine specimens at two different temperatures involving the progressive doubling of the torque amplitude for a total range of $8\times$ (Fig. 5.2).

A systematic steepening of the $G(\log_{10} T_0)$ trends with increasing strain amplitude is observed with an intersection in the trends near 30 s (Fig. 5.2a,c). Data below maximum shear strain values of 2×10^{-5} show smaller deviations indicating an approximation to linearity.

There are also consistent trends for attenuation (Fig 5.2b,d). Between maximum shear strains of 10^{-6} and $\sim 1.4 \times 10^{-5}$ there is little dependence of Q^{-1} on the shear strain as demonstrated earlier (Berckhemer et al., 1979; Gueguen et al., 1989; Jackson et al., 1992). However, for maximum shear strain values $> 10^{-5}$ the relation of $Q^{-1}(\epsilon)$ becomes much more significant (where ϵ is the shear strain). For maximum shear strain amplitudes substantially $> 10^{-5}$, Q^{-1} has increased by over half an order of magnitude towards 100 s period forced-oscillations significantly.

5.5.2 Torsional microcreep tests

Torsional microcreep tests were performed on several specimens at different temperatures to qualitatively document the viscoelastic response of olivine to the application and removal of a constant torque (shear stress) (Jackson, 1993, 2000; Jackson et al., 2004). The raw data are presented in Fig. 5.3. A steady applied torque assumed values of 0, +L, 0, -L and 0, respectively, for consecutive time

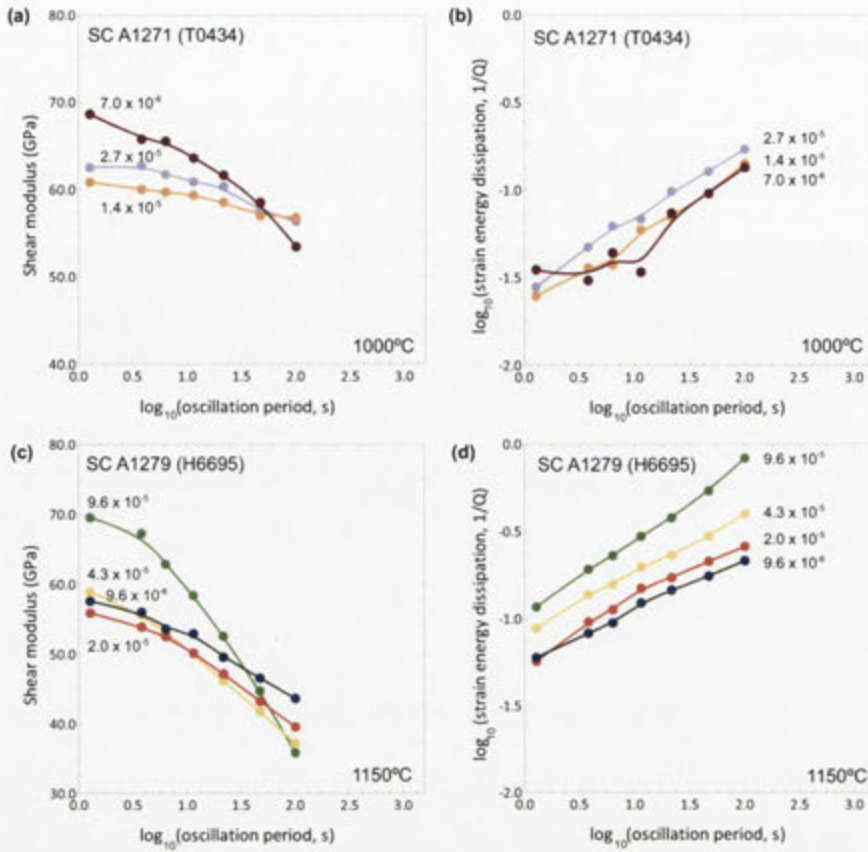


Fig. 5.2: Shear strain amplitude tests showing processed shear modulus and attenuation data at maximum shear strains across a range of forced-oscillation periods (1 – 100 s). (a,b) Three runs of ~ 1 hour each performed on a torsionally pre-deformed San Carlos A1271 at 1000°C. (c,d) Four runs, including one at the highest possible drive amplitude, performed on a hot-pressed San Carlos specimen A1279 at 1150°C. Maximum shear strains measured at 101 s oscillations are shown near each series of data, colour-coded per run.

intervals of either 1000 or 2000 s. For each switching of the torque, the response consists of the creep function for that event superimposed on suitably time-shifted responses from the prior episodes of switching. Obviously, the steepness of the curves in Fig. 5.3 reflect the creep rate (steeper means faster creep).

Most specimens (whether hot-pressed or pre-deformed) demonstrated increasingly stronger viscoelastic response with increasing temperature and diminishing partial recovery after removal of the steady torque (Fig. 5.3). This is most noticeable for the 1150°C curve of specimen A1279 (H6695) (Fig. 5.3d) but also

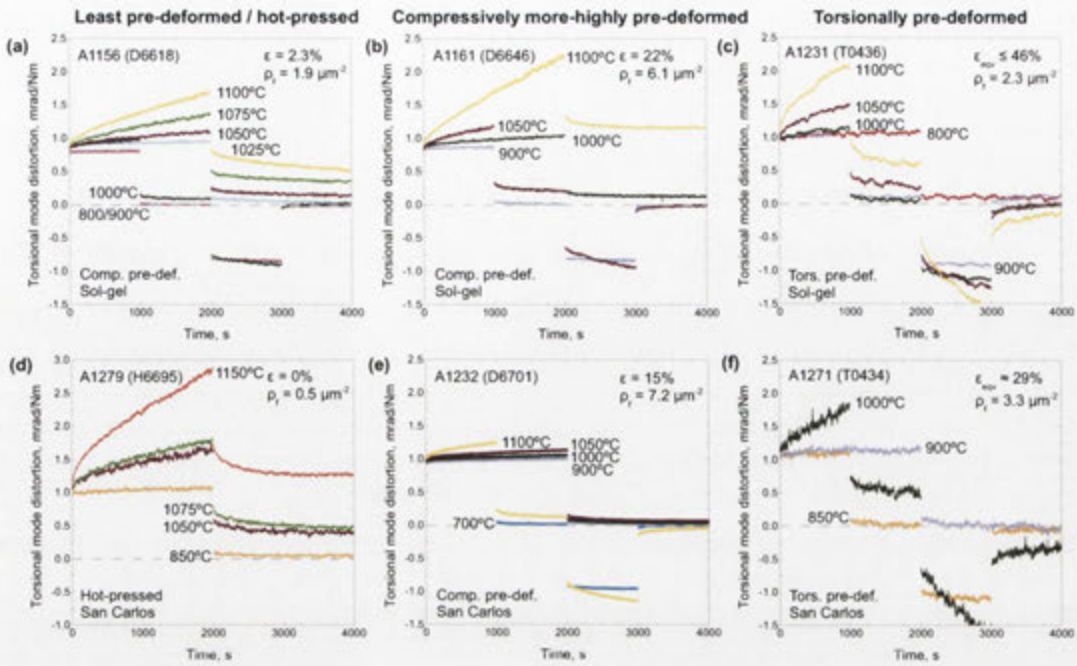


Fig. 5.3: Torsional microcreep records plotted as angular distortion versus time for a selected number of specimens (a) to (f). Scales are the same except for A1279 (d). Partial recovery of the creep strain can be observed following the termination of an applied steady torque after the first 1000 or 2000 s interval. For short interval records, an equal and opposite torque can be seen applied at 2000 s followed by another period of relaxation at 3000 s. The curves are colour-coded to temperature as shown. For each specimen, the pre-deformed strain is given by ϵ or equivalent strain ϵ_{eqv} for torsionally pre-deformed specimens. $\epsilon = 0$ for the hot-pressed specimen. Final dislocation density is indicated by ρ_f .

quite noticeable down to 1050°C for most specimens. The onset of appreciable anelastic relaxation at 900°C is most apparent for A1161 and A1271 (Fig. 5.3b, 5.3f) where only minor anelastic relaxation is seen after the removal of the steady torque at 1000 s and 2000 s respectively, followed by a full recovery.

The compressively more-highly deformed sol-gel specimen A1161 (Fig. 5.3b) with higher dislocation density experienced a high creep rate at 1100°C and 1050°C followed by a greater amount of inelastic deformation at 1100°C compared to A1156 (Fig. 5.3a). The torsionally pre-deformed sol-gel specimen (A1231) in Fig. 5.3c demonstrated even higher creep rates at 1100°C and 1050°C despite a lower dislocation density. A distortion of about 2 mrad Nm⁻¹ was measured

for A1231 after 1000 s at 1100°C compared to only ~ 1.4 mrad Nm⁻¹ for the compressively less deformed sol-gel A1156. However, A1232 demonstrates comparatively similar recovery levels. The last recovery segment demonstrates that the specimen approximately returned to its previously deformed state.

By comparison, the San Carlos torsionally pre-deformed specimen A1271 showed significant creep close to a distortion value of 1.75 mrad Nm⁻¹ at only 1000°C for a moderate dislocation density (Fig. 5.3f). These results show that the viscoelastic shear strain of these specimens, pre-deformed in torsion, can apparently be larger than in the compressively pre-deformed olivine specimens or a hot-pressed specimen shown in Fig. 5.3d. However, at 1075°C, hot-pressed San Carlos olivine (A1279) deformed with a creep rate somewhat higher than the more fine-grained weakly pre-deformed sol-gel A1156, indicating the weaker rheology of San Carlos olivine (likely due to a higher water concentration). The compressively pre-deformed San Carlos specimen A1232 in Fig. 5.3e is an unusual case in that demonstrated very low creep rates, even at 1100°C and almost immediate recovery after removal of the steady torque. As noted earlier, a complication during assembly was discovered for this specimen. The creep segments for some specimens appear noisier than in others, probably because of deterioration in transducer alignment in some experiments.

5.5.3 Forced-oscillation tests

5.5.3.1 General observations

Shear modulus G and attenuation (strain energy dissipation) Q^{-1} results from all mechanically tested specimens are presented in Fig. 5.4 and Fig. 5.5. The scales are equivalent in all plots for easy comparison. A systematic monotonic variation of G and Q^{-1} with oscillation period and temperature is observed for each specimen with a dependence of Q^{-1} upon oscillation period given by $Q^{-1} \sim T_0^\alpha$. Essentially elastic behaviour is evident below 900°C for most specimens but significant dissipation and modulus dispersion are observed at lower temperatures

for torsionally pre-deformed San Carlos, A1271 (Fig. 5.5e).

For each panel of Fig. 5.4 and Fig. 5.5, the continuous curves represent the Burgers background-only fits (Table 5.1) to $N(G, Q^{-1})$ data pairs. The broken curves represent the same model, extrapolated as appropriate, to higher and lower temperature outside the range of constraining measurements. The open symbols represent data points excluded from the least-squares analysis but have been plotted for completeness.

The same monotonic variation of Q^{-1} with oscillation period and temperature is demonstrated in Arrhenius plots (Q^{-1} against $1/T$) for a series of fixed oscillation periods (Fig. 5.6). The data are fitted by the same extended Burgers model but the model curves are not extrapolated beyond the measured data for clarity. Fig. 5.6 confirms the absence of a dissipation peak localised in the $1/T$ space for each mechanically tested specimen.

While the activation energies for the compressively and torsionally pre-deformed specimens (Fig. 5.6c,d,e) are generally high, the activation energy for the hot-pressed San Carlos olivine specimen is not as low as for the hot-pressed sol-gel specimen (Table 5.1). The unusual low Q^{-1} data for San Carlos A1232 (Fig. 5.6d) presented the highest activation energy for all experiments, which explains why the extrapolated 1150°C and 1200°C curves are still higher than the hot-pressed equivalent (Fig. 5.5b,d). Lastly, there is no obvious dissipation peak visible superimposed on a monotonic background typically seen in melt-bearing olivine (e.g. Jackson et al. 2004). The sol-gel and San Carlos olivine data are described in more detail in the following sections.

5.5.3.2 Sol-gel olivine data

The shear modulus data for the hot-pressed sol-gel specimen (A1127, Fig. 5.4a) show a generally tight fit to the extended Burgers model. The shear modulus of 42 GPa measured at 1100°C and 1000 s period provides a benchmark against which data for the pre-deformed specimens can be compared. For the weakly

compressively pre-deformed sol-gel A1256, the shear modulus $G = 41.2$ GPa. This value continues to decrease for the compressively more-highly pre-deformed specimen A1161 ($G = 33.9$ GPa) and for the torsionally pre-deformed specimen A1231 ($G = 33.4$ GPa). It is noteworthy that the optimal background-only Burgers fit includes a value of G_{UR} 7% lower than expected at 900°C for a fully dense olivine aggregate (66.5 GPa). Also, the excluded low temperature shear modulus data ($< 900^\circ\text{C}$) are systematically underestimated by the background-only model.

The attenuation data of A1127 (Fig. 5.4b) are also generally well-fitted by the background-only Burgers model at higher temperatures. Below 900°C, however, there appears to be a ‘plateau’ with $\log_{10} Q^{-1} \sim -2$. It has been suggested by Jackson and Faul (2010) that both the Q^{-1} plateau and the 7% modulus deficit might be explained by incorporation of a broad dissipation peak attributable to elastically accommodated grain-boundary sliding and associated modulus dispersion.

The weakly pre-deformed sol-gel A1156 shows a generally good model fit to both both shear modulus and attenuation, although the shear modulus may be slightly over-estimated at 1100°C (Fig. 5.4c). Thus for this specimen of relatively low dislocation density, the data are broadly comparable with those for the undeformed specimen (A1127). Nonetheless, at 1100°C for 1000 s oscillations slightly higher Q^{-1} values were obtained and marginally lower values of the modulus data than for the undeformed specimen. The transition towards viscous behaviour is seen by the steepening of the $\log_{10} Q^{-1}(\log T_0)$ curves (for lower Maxwell relaxation times) for $T \geq 1050^\circ\text{C}$. Interestingly, since the $\log_{10} Q^{-1}(\log_{10} T_0)$ data for different temperatures are more widely spaced than observed in Fig. 5.4b, attenuation became less than the hot-pressed specimen below 1050°C.

The shear modulus data for the compressively more highly pre-deformed specimen (A1161, Fig. 5.4e) are generally fitted very well to the extended Burgers model (a (G, Q^{-1}) outlier at 1075°C and 1000 s was excluded from the fit). Again,

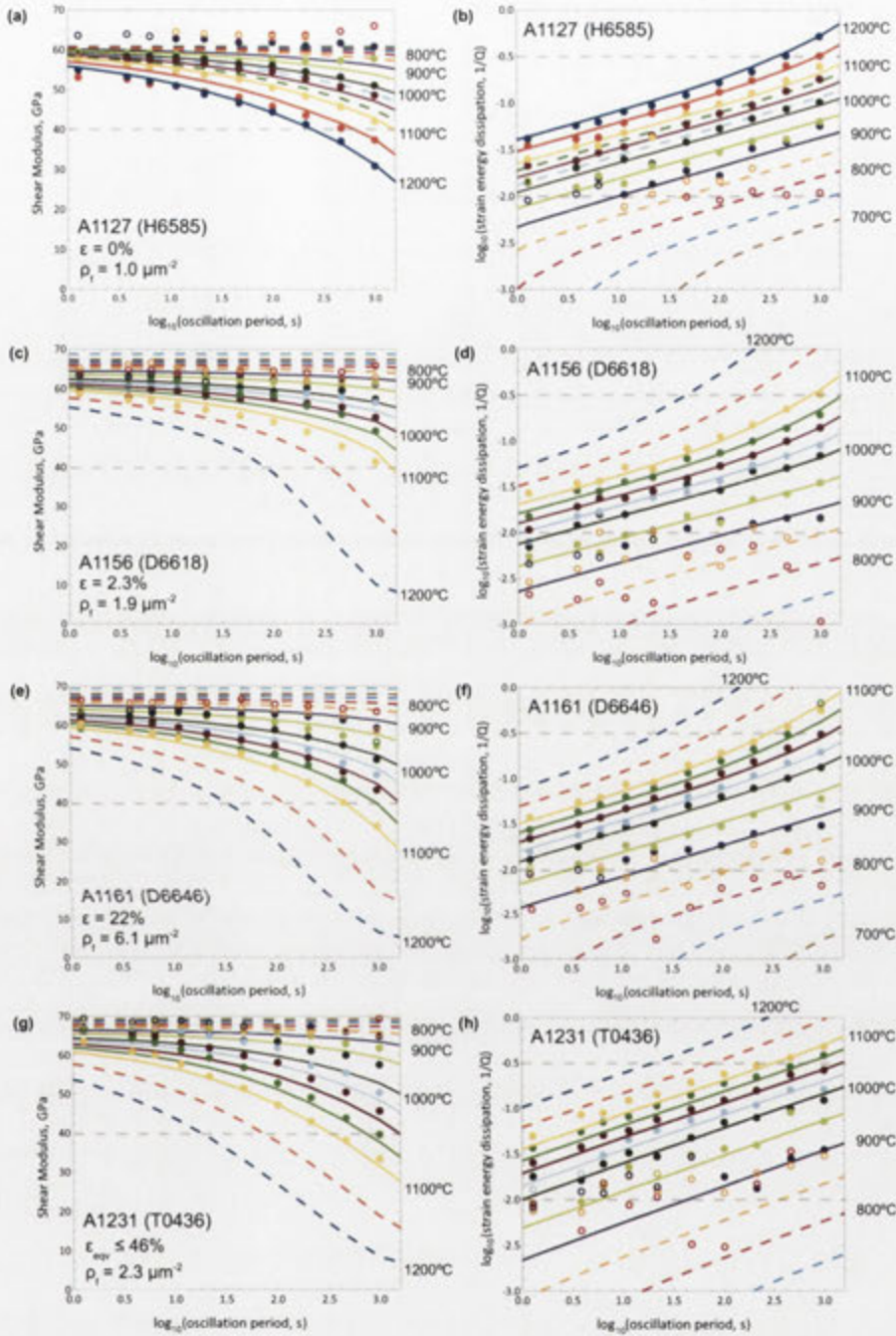


Fig. 5.4: Results of mechanical testing of all sol-gel specimens (a) – (h). Shear modulus versus log oscillation period data at each of a series of temperatures are shown in column on the left. Log dissipation versus log oscillation period data for the same series of temperatures are shown in the column on the right. The solid data symbols are modelled by the extended Burgers model (continuous curves) whereas the open data symbols were excluded. The dashed curves at higher and lower temperatures were extrapolated from the model based on the best fit parameters given in Table 5.1 per specimen. Temperature labels for each curve are shown next to each plot.

the transition towards viscous behaviour is observed for long-period-oscillations at the higher temperatures. Q^{-1} values approach 1 for 1100°C, the highest observed for any of the sol-gel specimens.

The torsionally pre-deformed specimen A1231 (Fig. 5.4g and h) appears similar to the previous pre-deformed specimens in several ways despite the fact that final dislocation density was moderately low. The shear modulus data in (g) are very similar to those of Fig. 5.4e although the quality of the fit deteriorates significantly below 1025°C for long period oscillations and all short period oscillation measurements. The unrelaxed shear modulus at 900°C had to be fixed for the Burgers fit, to avoid a physically unreasonable value of $G_{UR} > 66.5$ GPa for the reference conditions. The Q^{-1} data shows a general better fit down to 1000°C but with a lot of scatter below 950°C. The maximum Q^{-1} lies in between the compressively pre-deformed specimens.

5.5.3.3 San Carlos olivine data

For the San Carlos specimens, the maximum temperature to which mechanical testing was performed varied per specimen; 1200°C (A1279), 1100°C (A1232) and 1000°C (A1271) for reasons described elsewhere.

The G and Q^{-1} data for hot-pressed specimen A1279 are generally well constrained by the optimal Burgers model (Fig. 5.5a,b) except for some 1 s oscillation period data towards lower temperatures (similarly problematic for the other San Carlos specimens). In order to avoid unrealistic high values, the unrelaxed shear modulus was fixed at the standard value for the reference conditions (Table 5.1). The attenuation data in Fig. 5.5b however, appeared very scattered for temperatures below 1000°C and not many low-temperature data were used to constrain the extended Burgers model. The kink in the $\log_{10} Q^{-1}(\log_{10} T_0)$ curve near the longest oscillation period (1000 s) at 1200°C is an artefact from inadequate modelling of the transition from anelastic to viscous behaviour to be addressed in future work.

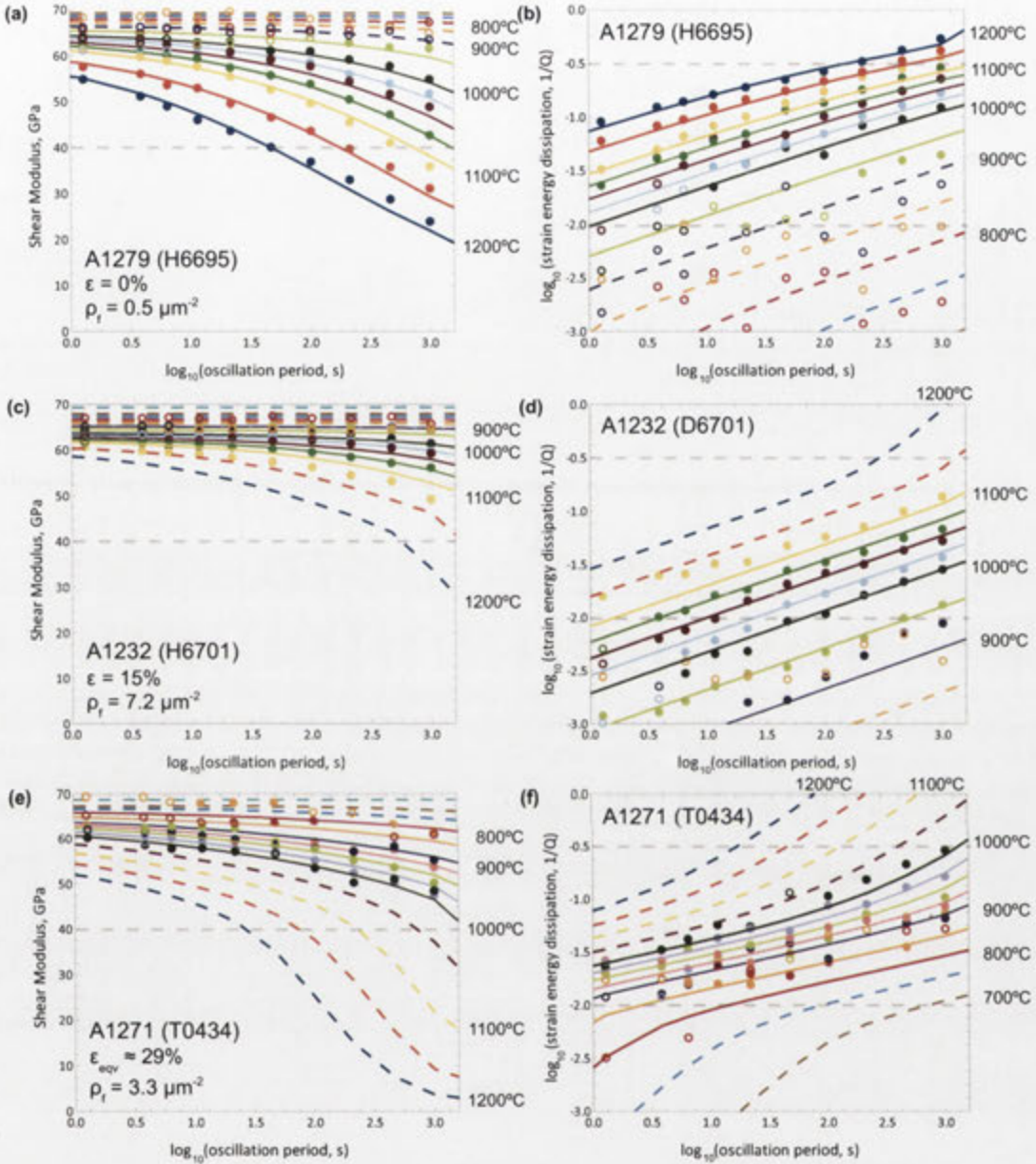


Fig. 5.5: Results of mechanical testing of all San Carlos specimens (a) – (f). Shear modulus versus log oscillation period data at each of a series of temperatures are shown in column on the left. Log dissipation (shear wave attenuation) versus log oscillation period data for the same series of temperatures are shown in the column on the right. The solid data symbols are modelled by the extended Burgers model (continuous curves) whereas the open data symbols were excluded. The dashed curves at higher and lower temperatures were extrapolated from the model based on the best fit parameters given in Table 5.1 per specimen. Temperature labels for each curve are shown next to each plot.

The unusually high G and low Q^{-1} data for the compressively pre-deformed specimen A1232 of high dislocation density were modelled as shown in Fig. 5.5c,d. The most interesting result is that unlike other specimens the data appears to be well resolved down to $\log Q^{-1}$ values of -3. Microstructurally, this specimen did not appear unusual (Chapters 4, 3 and Appendix E). However, analysis of these data is complicated by the previously mentioned displacement of one of the foil discs and remains to be addressed in future work.

Torsionally pre-deformed specimen A1271 of moderate dislocation density, tested to 1000°C, shows the lowest shear modulus and highest attenuation data obtained in this study (Fig. 5.5e,f). Unfortunately, the data appears significantly scattered and lower temperature data (down to 800°C) had to be included as much as possible to constrain the Burgers model fit. Onset of significant viscous behaviour (with a very low Maxwell relaxation time) at temperatures as low as 950°C is unlikely (and not clearly required by the data), but fixing $\log_{10} \tau_{MR}$ at a higher value resulted in straighter $\log_{10} Q^{-1}(\log_{10} T_0)$ curves that underestimate the dissipation measured at long oscillation periods. Future work might be able to improve upon this model. Nonetheless, in comparison with hot-pressed San Carlos specimen A1279 (Fig. 5.5b), at all temperatures the attenuation data in Fig. 5.5f plot significantly higher. The sol-gel olivine $\log_{10} Q^{-1}$ versus $\log_{10} T_0$ trends generally steepen significantly at long periods – unlike those for the San Carlos material (Fig. 5.5e).

5.5.3.4 Comparison of data for sol-gel and San Carlos olivine

Differences and similarities between the two types of olivine prepared from natural and laboratory precursors are described next. These comparisons are complicated to some degree by uncertainties concerning porosity and / or cracking in the hot-pressed San Carlos specimen A1279 and the processing of data for the compressively pre-deformed San Carlos specimen A1232 with the displaced foil disk. Accordingly data for the other specimens will be emphasised in seeking to

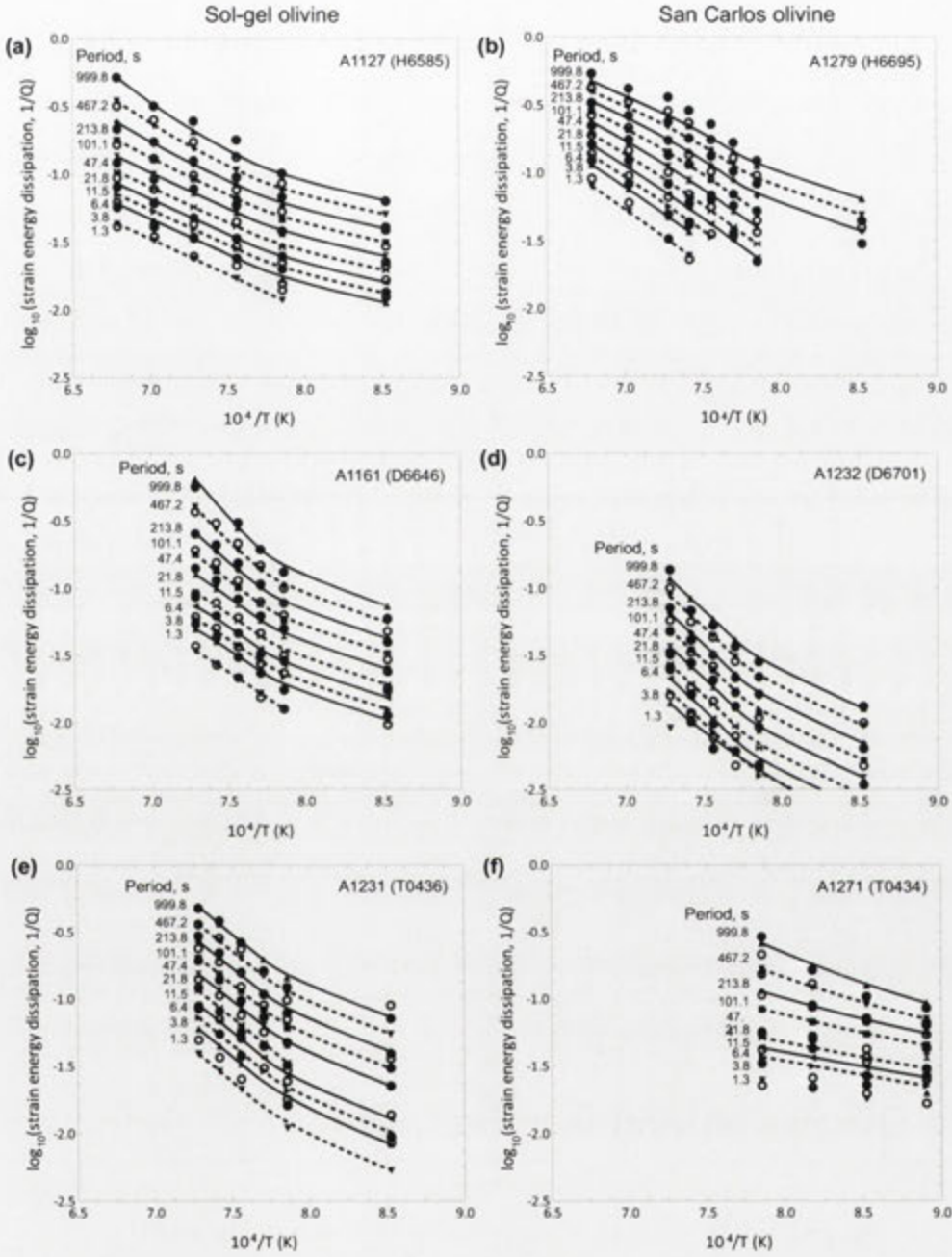


Fig. 5.6: Arrhenius plots of log dissipation versus reciprocal absolute temperature (K) at each of a series of oscillation periods (1.3, 3.8, 6.4, 11.5, 21.8, 47.4, 101.1, 213.8, 467.2 and 999.8 s). The first column shows the sol-gel olivine specimens whereas the second column shows the San Carlos specimens. Furthermore, (a,b) are results from the hot-pressed olivines, (c,d) are results from the most compressively pre-deformed olivines and (e,f) are results from the torsionally pre-deformed olivines. The scales are the same for easy comparison.

identify significant trends.

The pre-deformed specimens generally display lower shear modulus and higher attenuation than the hot-pressed specimens, especially towards longer oscillation periods. Both hot-pressed specimens were tested to 1200°C. For 1000 s forced oscillations, no significant differences can be found in maximum levels of attenuation (Fig. 5.4b, 5.5b), however hot-pressed San Carlos olivine shows substantially lower values of the shear modulus at the higher temperatures (Fig. 5.5a). Another difference can be found in the slope of the attenuation curves for the range of oscillation periods for A1279 (Fig. 5.5b) compared to A1127 (Fig. 5.4b). The hot-pressed sol-gel (A1127) Q^{-1} data appear to steepen upwards at longer oscillation periods than the hot-pressed San Carlos Q^{-1} data for each temperature. This is reflected by the difference in the $\log_{10} \tau_M$ values in Table 5.1 (5.4 versus 6.4). Note, however, that the Burgers fit is not ideal between 1025 and 1150°C for A1279 (Fig. 5.5b).

Attenuation measurements between the compressively pre-deformed and torsionally pre-deformed specimens are not dramatically different for sol-gel olivine. However, the more homogeneously torsionally pre-deformed San Carlos olivine specimen, which was more microstructurally stable during $\leq 1000^\circ\text{C}$ testing, shows significantly higher attenuation than any other pre-deformed specimen.

5.6 Discussion and interpretations

5.6.1 Strain amplitude dependence and linearity

As pointed out by Berckhemer et al. (1979); Jackson (1993), linearity between the application of a sinusoidally time-varying stress and the sinusoidal strain response forms the basis for linking viscoelastic rheology with seismology. Therefore, it was important for these experiments to test whether the conditions of mechanical testing remained within or close to the linear regime. Outside the linear regime, towards higher shear strains, a strain-amplitude dependence of the

dynamic compliance is expected, which severely complicates any interpretation of the shear modulus and attenuation (Gueguen et al., 1989). Thus far, linearity has been demonstrated for shear strains up to 5×10^{-5} (Berckhemer et al., 1979) and was disproved for larger shear strains above 10^{-4} (Goetze and Brace, 1972). In addition, extensive work at ANU was carried out at strains generally $\leq 2 \times 10^{-5}$ (e.g. Jackson et al. 1992 – Jackson 2007) but the transition to linear behaviour has not yet been clearly demonstrated.

In this study experiments were routinely performed with strains $\leq 10^{-5}$ for 1000 s period oscillations at $\leq 1100^\circ\text{C}$ (Table 5.1) – with one exception, a maximum strain amplitude of 1.6×10^{-5} was reached for the hot-pressed San Carlos specimen A1279 at 1200°C . However, the previously mentioned access to larger torque amplitudes has been exploited in selected tests at higher strain amplitudes (Fig. 5.2). These tests reveals a progressive steepening of the $G(\log_{10} T_0)$ trends with increasing strain amplitude. The fact that the differences are greatest at short periods where the inferred values of the shear modulus are implausibly high, suggests that the effects of argon gas flow between the parallel capacitance plates may have been underestimated for large displacement amplitudes (Jackson and Paterson, 1993).

Fig. 5.2b,d demonstrates the relationship of Q^{-1} on the oscillation period for increasing maximum shear strains. Up to maximum shear strains of 1.4×10^{-5} , Q^{-1} values have not yet significantly increased within statistical uncertainty. However, greater dependence of the strain on attenuation began to occur near 2×10^{-5} . Data from current experiments appear to be just within the linear regime for maximum shear strains.

5.6.2 Dislocation density changes during mechanical testing

Prior to the micro-strain mechanical testing of the pre-deformed specimens, a study was carried out to determine the dislocation recovery kinetics in similar

compressively deformed sol-gel and San Carlos aggregates (see Chapter 3). The study pointed out that dislocation recovery kinetics in a fine grained olivine aggregate are at least an order of magnitude slower than in a deformed single crystal (Karato and Ogawa, 1982; Karato et al., 1993). This consideration suggested a maximum temperature of 1100°C to which mechanical testing could proceed without losing a significant population of dislocations. Specifically, it was inferred that about 50 hours annealing at 1100°C for acceptable stable mechanical response prior to mechanical testing, would lead to a reduction in dislocation density of no more than 20%. This was considered an acceptable trade-off that avoided narrowing too severely the window for experimental observation of viscoelastic behaviour.

5.6.3 Interpretation of obtained Burgers model parameters

Table 5.1 presents the extended Burgers model parameter values for all specimens which provided a best fit to the mechanical data. Caution should be exercised when comparing individual model parameters as there is substantial covariance between them. As evident, many values for the unrelaxed shear modulus G_U approach or are fixed at the value for the reference (66.5 GPa). The relaxation strength for background dissipation (Δ_B) is similar (around a value of 2) for most specimens except for sol-gel A1156 and a very low relaxation strength for San Carlos A1232. The dependence of Q^{-1} upon oscillation period given by $Q^{-1} \sim T_0^\alpha$, where α is mostly between 0.3 – 0.4, slightly higher values than those obtained in previous studies (Berckhemer et al., 1982; Jackson et al., 2004; Faul and Jackson, 2005) except Tan et al. (2001). The Maxwell relaxation times (specified as $\log_{10} \tau_{MR}$) for viscous response are tightly bunched for the sol-gel specimens (5.14 – 5.82). Generally larger values are seen for San Carlos olivine. From observation of the data of torsionally pre-deformed San Carlos A1271, the low $\log_{10} \tau_{MR}$ is arguably not required by the data (as seen in Fig. 5.5).

The activation energies in Table 5.1, constrained by fixed $\log_{10} \tau_L$ and $\log_{10} \tau_H$ appear to vary between 320 kJ mol⁻¹ (hot-pressed sol-gel A1127) and ~ 480 kJ mol⁻¹ (compressively pre-deformed sol-gel A1156), ignoring the value from the problematic (G, Q^{-1}) data of the compressively pre-deformed San Carlos specimen A1232. An activation energy of 440 ± 50 kJ mol⁻¹ was obtained from dislocation damping in single crystal forsterite (Gueguen et al., 1989). Values obtained for melt-free hot-pressed sol-gel and San Carlos olivine were around 400 kJ mol⁻¹ and in melt-bearing, 520 kJ mol⁻¹ (Jackson et al., 2004). Similar activation energies were obtained for grain boundary diffusion creep experiments by, e.g. Hirth and Kohlstedt 1995, who obtained a value of 315 kJ mol⁻¹ for natural low melt-bearing olivine (0.5 – 1 vol. %). Diffusion creep experiments on dry, melt-free sol-gel olivine provided a much higher activation energy of 484 kJ mol⁻¹ (Faul and Jackson, 2007), which is more consistent with most olivine specimens in this study but not with the hot-pressed sol-gel specimen. An activation energy of 360(8) kJ mol⁻¹ was reported by Jackson and Faul (2010) from a more recent global Burgers fit to a suite of both sol-gel and San Carlos olivine specimens. Whether viscous dislocation creep can occur during the forced-oscillation testing, depends upon whether stresses are high enough for dislocation to break away from their pinning points (see Jackson 2007). Activation energies for dislocation creep are generally higher at around 530 kJ mol⁻¹ (see e.g. Hirth and Kohlstedt 2003 for a summary on relatively recent work).

It should be noted that the Maxwell relaxation time $\log_{10} \tau_{MR}$ at 900°C, exhibits a strong trade-off with the activation energy E as evident in the following equation

$$\begin{aligned} \tau_M(T, P, d) = & \tau_{MR}(d/d_R)^m \cdot \exp[(E/R)(1/T - 1/T_R)] \\ & \cdot \exp[(V/R)(P/T - P_R/T_R)] \end{aligned} \quad (5.6.1)$$

5.6.4 A dissipation peak?

Comparable to previous observations of melt-free sol-gel and San Carlos olivine (Tan et al., 2001; Jackson et al., 2002, 2009), a systematic monotonic variation of G and Q^{-1} with oscillation period and temperature was observed. A shear modulus deficit ($G_{UR} < 66.5$ GPa) and a dissipation plateau near $\log_{10} Q^{-1} = -2$ can both be explained by modifying the distribution $D(\tau)$ of anelastic relaxation times to allow some degree of localisation of dissipation and associated modulus dispersion, potentially attributable to elastically accommodated grain-boundary sliding (Jackson and Faul, 2010). More apparent dissipation peaks were seen in melt-bearing olivine with rounded grain edges in triple junctions (Faul et al., 2004). Possible evidence for a broad dissipation peak or ‘plateau’ is most noticeable in the low temperature data $< 900^\circ\text{C}$ for sol-gel specimens A1127, A1161, A1231 (Fig. 5.4b,f,h). The scatter in the San Carlos olivine data, however, is too great to make out any existence of a dissipation peak superimposed on the broad background dissipation.

5.6.5 Setting a baseline for data comparison

Distinguishing the additional attenuation attributable to dislocation relaxation from the relatively high Q^{-1} background associated with grain-boundary relaxation processes in the fine-grained polycrystals of the present study is more challenging than in the previous study of single crystal forsterite (Gueguen et al., 1989).

The hot-pressed specimens of sol-gel and San Carlos olivine were intended to provide a baseline against which the torsional forced oscillation data of the pre-deformed specimens could be compared. However, because of the previously-mentioned difficulties with micro-cracks and porosity caused by accidental uncontrolled and unconfined heating of the San Carlos specimen (A1279), the use of the Burgers model of Jackson and Faul (2010) is preferred, evaluated at the

appropriate grain sizes, as a more robust baseline for undeformed material (Fig. 5.7).

5.6.6 Dislocation damping

In this study, sol-gel olivine from laboratory precursors was the most ideal material to use due to its extremely low water content and stable grain microstructure, whereas the ‘wetter’ San Carlos olivine results may be affected by slight differences in average grain size, water content and a possible small melt fraction (~ 0.0001 , not observed using the scanning electron microscope with secondary electrons).

The Burgers model by Jackson and Faul (2010) provides an alternative baseline against which the pre-deformed specimens can be compared, thereby avoiding the suspicious data of hot-pressed San Carlos specimen (A1279). The baseline is given by a background plus peak fit to (G, Q^{-1}) data for a suite of essentially dry and melt-free sol-gel and San Carlos olivine (including the hot-pressed sol-gel specimen A1127 of this study). Fig. 5.7 shows the model in comparison with the three most highly pre-deformed specimens in this study. These specimens are the compressively more-highly pre-deformed sol-gel (A1161, 5.7a,b), the torsionally pre-deformed sol-gel (A1231, 5.7c,d) and the torsionally pre-deformed San Carlos (A1271, 5.7e,f). The model (G, Q^{-1}) curves are identical for specimens A1161 and A1231, being evaluated at their geometric mean grain size of $5.4 \mu\text{m}$. The model (G, Q^{-1}) curves for specimen A1271 are evaluated for a grain size of $8.8 \mu\text{m}$ equivalent to that of the specimen.

The shear modulus measured for each the pre-deformed specimens is more strongly period dependent than the baseline model for the undeformed materials (Fig. 5.7). Furthermore, there is a systematically higher G at low temperature for the torsionally deformed specimens which may reflect anisotropy associated with its fabric (see Chapter 4).

The dissipation measured for each of the pre-deformed specimens is substantially higher than the baseline model, especially at relatively high temperatures.

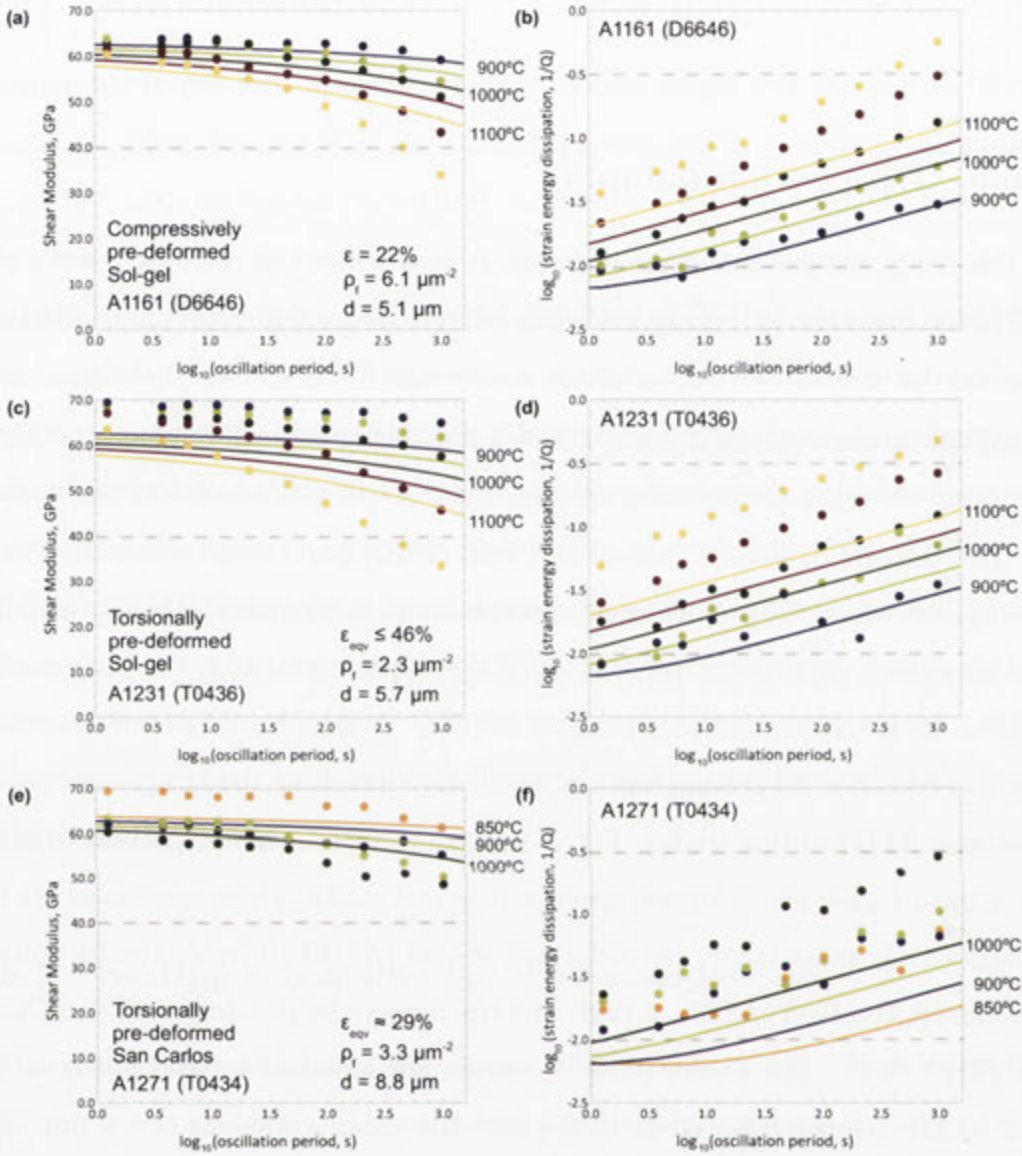


Fig. 5.7: Results of mechanical testing for three selected pre-deformed specimens (a) – (f). Shear modulus versus log oscillation period data at each of a series of temperatures are shown in column on the left. Log dissipation (shear wave attenuation) versus log oscillation period data for the same series of temperatures are shown in the column on the right. The continuous curves represent the Burgers model with background and peak dissipation by Jackson and Faul (2010) based on 204 ($G, 1/Q$) data pairs for essentially melt-free olivine (including H6585, i.e. A1127). For the compressively more-highly pre-deformed and torsionally pre-deformed sol-gel specimens (panels a – d) a global fit for an averaged grain size was performed ($5.4 \mu\text{m}$) whereas for the torsionally pre-deformed San Carlos specimen (panels e – f) a global fit for $8.8 \mu\text{m}$ grain size is shown.

Pre-deformed San Carlos A1271, with a better preserved microstructure from deformation than A1231, demonstrates higher strain energy dissipation across all oscillation periods and all temperatures (850 – 1000°C) compared to the baseline model.

A comparison between the baseline Burgers model and the pre-deformed specimens in this study can be better demonstrated quantitatively as follows. A quantity R can be calculated for the strain energy dissipation of each specimen at given temperatures as

$$\begin{aligned} R &= \log_{10} Q_{def}^{-1} - \log_{10} Q_{JF}^{-1} \\ &= \log_{10}[Q_{def}^{-1}/Q_{JF}^{-1}] \end{aligned} \quad (5.6.2)$$

where Q_{def}^{-1} is the dissipation of the pre-deformed specimen and Q_{JF}^{-1} is the dissipation of the model by Jackson and Faul (2010). The values of R have been calculated at each oscillation period at every temperature. The values were subsequently averaged over the entire range of oscillation periods to give a single value $\bar{R}$ at every temperature. These are summarised in Table 5.2.

The overall dislocation-related enhancement of dissipation is apparent in positive $\bar{R}$ values except for the more scattered low-temperature data for the sol-gel specimens. At temperatures $> 1000^\circ\text{C}$ the pre-deformed sol-gel specimens A1161 and A1231 demonstrate increasingly higher dissipation averaged over all oscillation periods. Surprisingly, the torsionally pre-deformed sol-gel specimen A1231 with a final dislocation density of $1.3 \mu\text{m}^{-2}$, close to that of a hot-pressed specimen, still consistently dissipated more strain energy at every temperature $> 1000^\circ\text{C}$ compared to the compressively more-highly pre-deformed sol-gel A1161 with a final dislocation density of $5.1 \mu\text{m}^{-2}$. This observation suggests a population of more favourably oriented dislocations from prior deformation in torsion contributed to enhanced anelastic relaxation and higher attenuation.

This is even more convincingly demonstrated for the torsionally pre-deformed San Carlos (A1271) (with $3.3 \mu\text{m}^{-2}$ dislocation density) in Table 5.2. For all

Table 5.2: $\bar{R}$ values from a comparison between the pre-deformed specimens and the Burgers model fit of Jackson and Faul (2010) for different temperatures.

Temperature (°C)	Specimens		
	A1161	T0436	T0434
1100	0.415	0.518	–
1075	0.400	0.454	–
1050	0.315	0.372	–
1025	0.239	0.257	–
1000	0.031	0.046	0.534
975	–	–	0.428
950	-0.154	-0.107	0.313
925	–	–	0.455
900	0.075	0.111	0.408
850	0.033	0.180	0.496
800	-0.206	0.080	0.389

temperatures significantly higher $\bar{R}$ values are apparent compared to the model – over half-an-order of magnitude higher at 1000°C.

The simplified model of Chapter 4 (Part I) for dislocation relaxation suggested that prior torsional deformation is six times more effective than prior compressive deformation for anelastic relaxation in torsion (see Chapter 4, section 4.8). This prediction is evidently broadly consistent with the experimental observations.

5.6.6.1 Preliminary interpretations

The following preliminary interpretations can be made from the comparison of the Burgers model by Jackson and Faul (2010) and the three most-highly pre-deformed olivine polycrystals in Fig. 5.7 and Table 5.2.

Firstly, enhancement of background dissipation rather than development of peaks suggests that the stress-induced migration of dislocations might couple closely with the dominant grain-boundary sliding relaxation mechanism by pro-

viding an additional means of achieving the required accommodation at grain boundaries. The enhanced dissipation is associated with stronger dispersion of the shear modulus. Secondly, any indication of non-linearity associated with dislocation damping is swamped by effects interpreted as probable artefacts arising from inadequate correction of data for torques exerted by the displacement transducer plates, with displacement amplitudes of unprecedented scale.

5.6.6.2 Implications of dislocation damping in the upper mantle

Although no extrapolation is needed towards a different range of oscillation periods for a comparison with seismic data, higher temperatures in regions of the upper mantle ($> 1100^{\circ}\text{C}$ in this study), larger grain sizes, higher pressures than laboratory pressure ($P_R = 0.2 \text{ GPa}$) and different generally lower, dislocation densities under mantle conditions, all need to be taken into account to allow any realistic comparison to conditions of the upper mantle.

The recent improved Burgers model by Jackson and Faul (2010), used here as a baseline, contains a grain-size sensitivity allowing extrapolation to mantle grain sizes. Dissipation Q^{-1} and shear modulus dispersion G associated with grain size sensitive relaxation mildly but systematically decrease with increasing grain size for any given fixed temperature. In Fig. 5.7 this mild effect can be seen between the Burgers model for $5.4 \mu\text{m}$ grain size and $8.8 \mu\text{m}$ grain size for a given temperature. This implies that the contribution from dislocation damping (assumed to be grain size independent) should become relatively more important in coarse-grained mantle material.

An extrapolation to upper mantle pressure (e. g. 3 GPa , for 100 km depth) is also taken into account by the Jackson and Faul (2010) Burgers model. As reported in their study, using an indicative value of $10^{-6} \text{ m}^3 \text{ mol}^{-1}$ of the activation volume, the estimated relaxation times increase by $8.1\times$ with increasing pressure ($P_R = 0.2 \text{ GPa}$ to $P = 3 \text{ GPa}$) at 1340°C . Additionally, the shear modulus G is expected to systematically increase by more than 5 GPa across the same pres-

sure range, through a combination of anharmonic and viscoelastic effects. New techniques for measuring strain energy dissipation and shear modulus dispersion at high pressure (~ 5 GPa, upper mantle pressures) at high temperature (up to 1723°C) are currently being explored using a multi-anvil (Deformation-DIA) configuration coupled with a synchrotron x-ray radiation source (Li and Weidner, 2007).

In this study, the observed relationship between dislocation-related dissipation and the orientation of the stress field during prior deformation suggests the possibility of seismological attenuation anisotropy. Evidence for CPO and dislocation creep in the upper mantle (e.g. Zhang and Karato (1995); Karato (2008)) suggest that populations in equilibrium with the prevailing tectonic stress are generated by glide in preferred directions on specific slip systems. Hence, strongest attenuation for shear waves could be expected for shear waves with a polarisation that maximises the resolved shear stress for glide of these populations of dislocations (e.g. SH waves in the oceanic upper mantle with polarisation parallel with plate motion).

It is thus possible that dislocation damping could contribute significantly ($Q^{-1} \sim 10^{-2}$) under upper mantle conditions, especially in regions with relatively high levels of prevailing tectonic stress (e.g. in deeper levels of the lithosphere), as opposed to regions of localised deformation where recrystallisation is expected to occur. However, it must be noted that as dislocation density scales with σ^2 , generally much lower dislocation densities will be expected at the lower deviatoric stresses of ongoing tectonic deformation. This results in a decreased contribution from dislocation damping to seismic shear wave attenuation.

5.7 Conclusions

A study was carried out to characterise the effect of dislocation density on dissipation and shear modulus dispersion in pre-deformed polycrystalline olivine. Fine-grained olivine powders were prepared from laboratory (sol-gel) and natu-

ral (San Carlos) precursors and were compressively and torsionally pre-deformed. The resulting specimens of each type (sol-gel and San Carlos) were mechanically tested for torsional forced-oscillations in the tele-seismic range (1 – 1000 s periods) at 200 MPa confining pressure. To avoid the risk of a significant reduction of dislocation density, a complementary static recovery study (Chapter 3) demonstrated that mechanical testing had to be restricted to temperatures of $\leq 1100^{\circ}\text{C}$. However, generally greater reductions in dislocation density were measured after attenuation experiments than anticipated. Nonetheless, final dislocation densities in the specimens were still up to a half-an-order of magnitude higher than the hot-pressed specimens.

Maximum strain amplitudes between 5×10^{-6} and 1×10^{-5} were reached during 1000 s forced-oscillations at 1100°C and maximum stress amplitudes up to 1 MPa. Previous work and results from this study indicate that routine testing was carried out under essentially linear conditions avoiding significant dislocation multiplication. An experiment at $\leq 1000^{\circ}\text{C}$ on a torsionally pre-deformed San Carlos specimen, however, demonstrated the highest levels of dissipation but no reduction in dislocation density.

Evidence from torsional microcreep tests confirmed larger anelastic shear strains in the pre-deformed specimens compared to the hot-pressed equivalents. The torsionally pre-deformed San Carlos specimen demonstrated the highest creep rate for at a maximum temperature of 1000°C . The torsionally pre-deformed specimens however, were also the most mechanically unstable specimens, especially above 1000°C where a tendency of the specimen to untwist occurred.

Generally, a monotonically frequency and temperature dependent background dissipation and shear modulus dispersion is observed similar to previous hot-pressed relatively dry and melt-free olivine (Tan et al., 2001; Jackson et al., 2002, 2004; Jackson and Faul, 2010). A baseline comparison was made with a Burgers model by Jackson and Faul (2010) for previous tested specimens described above, evaluated for appropriate grain sizes for the pre-deformed specimens in

this study. Enhancement of background dissipation up to half-an-order of magnitude is observed at 1100°C for the more-highly pre-deformed sol-gel specimens and at 1000°C for the torsionally pre-deformed San Carlos specimen. The lack of development of peaks suggests that the stress-induced migration of dislocations might couple closely with the dominant grain-boundary sliding relaxation mechanism. The obtained high activation energies of the pre-deformed specimens compare to dislocation creep values ($\sim 450 \text{ kJ mol}^{-1}$), possibly implicating relaxation mechanisms with stress-induced migration of dislocation kinks.

The observed enhancement of dissipation in the torsionally pre-deformed specimens compared to a compressively more-highly pre-deformed specimen (of higher dislocation density), suggests that the orientation of the stress state influences damping of dislocations that are associated with preferred slip system(s), leading to seismological attenuation anisotropy in the upper mantle.

*Tell me and I'll forget;
show me and I may remember;
involve me and I'll understand.*

Chinese proverb, unknown origin



Conclusions and future studies

The research presented here comprises systematic exploratory study of possible dislocation damping in pre-deformed polycrystalline olivine to better constrain global attenuation models and understand viscoelastic relaxation in the upper mantle of the Earth.

Models of stress-induced migration of dislocation segments suggest significant relaxation in the seismic frequency band with a high background relaxation strength. This provided motivation for a pioneering study on dislocation damping in olivine in the laboratory on synthetic pre-deformed forsterite single crystals (Gueguen et al., 1989). Data were collected over $0.1 - 10^4$ s period forced oscillations at three temperatures, 1000, 1200 and 1400°C in a vacuum. A pronounced increase in dissipation Q^{-1} for a broad absorption band was observed in the pre-deformed crystals towards high temperature. However, the authors questioned whether the high levels of measured dissipation might have resulted from non-linearity in the stress-strain behaviour. In that study, neither this, nor the question of dislocation recovery during high-temperature mechanical testing were further investigated.

In this study at the ANU, new upper mantle analogue materials were prepared

for attenuation measurements. Both laboratory sol-gel and natural San Carlos derived Fo_{90} specimens were synthesised and hot-isostatically pressed into dense cylindrical aggregates in a controlled chemical environment (f_{O_2} , a_{SiO_4} , essentially dry). Several specimens were compressively pre-deformed in the dislocation creep regime at high temperature (1250 – 1300°C) and moderate gas confining pressures (300 MPa) to create suitably dense populations of dislocations (up to 10× higher dislocation density for a hot-pressed specimen). The rheological data of the deformed fine-grained specimens indicated a classic transition from diffusion to dislocation creep. The San Carlos specimen appeared to be significantly weaker at high differential stresses (> 180 MPa) than essentially dry and melt-free sol-gel olivine.

Small cubes were cut from compressively deformed sol-gel and San Carlos specimens. These samples were annealed under controlled-atmosphere conditions between 1100 and 1500°C for different durations in order to measure the rate of dislocation recovery. Following annealing, the dislocations were decorated by oxidation at moderate temperature in air then imaged across large areas using back-scattered electron field emission SEM.

A new method for determining the ‘3-D’ dislocation density was presented based on cumulative line length measurements and an estimation of the imaging depth using Monte Carlo electron interaction simulations for back-scattered electrons. The dislocation recovery measurements produced rates that are 1 – 2 orders of magnitude lower than those from previous studies on single crystals. This discrepancy could be partly related to the imaging techniques used by the various authors or the influence of internal back-stresses exerted by the grain boundaries in the polycrystals, slowing down dislocation (glide) kinetics.

Most importantly, the results from the dislocation recovery study allowed prediction of the reduction in dislocation density during high temperature stabilisation of mechanical behaviour prior to forced-oscillation measurements. At 1100°C for a duration of 50 hours of annealing, a reduction in dislocation density

would be 20% for both sol-gel or San Carlos olivine – an acceptable compromise for attempting to measure a contribution from dislocation damping above a background of strong grain-size-sensitive viscoelastic relaxation.

Additionally, a sol-gel and a San Carlos specimen were torsionally pre-deformed at high temperature and gas confining pressure. The strain marker in the jacket of the specimens demonstrated that, despite slip at the sample-piston interfaces, the cylindrical olivine aggregates were deformed to shear strains of 0.5 but only with moderate dislocation density (up to $3\times$ higher than a hot-pressed specimen). Despite the lower dislocation density compared to compressively pre-deformed specimens, geometrical considerations of damping by dislocation glide suggested that prior torsional deformation may be significantly more effective than prior compressive deformation for anelastic relaxation in torsion.

Both hot-pressed and pre-deformed specimens were tested in torsional forced oscillation at 200 MPa pressure and temperatures to 1200°C, at periods of 1 - 1000 s and strain amplitudes $\lesssim 10^{-5}$ within the linear regime. The (G, Q^{-1}) data for the more-highly pre-deformed specimens were compared to a Burgers model by Jackson and Faul (2010) fitted to experimental data for grain-size sensitive viscoelastic relaxation of hot-pressed, essentially dry and melt-free olivine. The data for both compressively and torsionally pre-deformed olivine demonstrated significantly enhanced dissipation Q^{-1} and shear modulus G dispersion. The absence of dissipation peaks suggests that the stress-induced migration of dislocations might couple closely with the dominant grain-boundary sliding relaxation mechanism.

Higher creep rates in complementary microcreep tests, dissipation and dispersion were measured for the torsionally pre-deformed specimens, in particular for a San Carlos olivine specimen tested to only 1000°C to keep its dislocation density close to that developed from pre-deformation.

A contribution of dislocation relaxation to seismic attenuation was observed in the laboratory, most significantly for torsionally pre-deformed fine-grained olivine

aggregates. This would suggest that solid-state anelastic relaxation from stress-induced dislocation migration in the deforming / deformed upper mantle is possible and potentially anisotropic. Under upper mantle conditions of larger grain size, but lower dislocation density in equilibrium with the prevailing tectonic stress, dislocation relaxation may significantly enhance the dissipation and shear modulus dispersion from grain-boundary relaxation processes.

Plans for future work include reassessment of the data for two problematic San Carlos specimens of this study (A1232, A1279). This will probably lead to further experimental work on these specimens or similarly prepared replacements.

Additional investigations of dislocation damping could benefit from prior deformation of synthetic olivine aggregates of a wider range of grain sizes in order to more clearly separate intra- and intergranular contributions to the viscoelastic relaxation. Such materials would ideally be essentially dry and melt-free to avoid additional complications. If it were possible to obtain large inclusion-free olivine crystals, these could also be pre-deformed and tested. Finally, it may be worthwhile to synthesise / pre-deform and test multi-phase materials with more realistic compositions of olivine and pyroxene found in the upper mantle, increasing the fraction inter-phase grain boundary contact, which may lead to more or less dissipation.

A

Single crystal olivine as candidate for dislocation damping

After the sol-gel olivine specimens were pre-deformed in compression and mechanically tested in the attenuation apparatus, single crystal olivine was briefly considered as the next best alternative to attempt to more conclusively demonstrate dislocation damping. The attractive advantage of using pre-deformed single crystals is that there is no contribution from grain boundary dissipation during forced-oscillation measurements at forced-oscillation periods (1 - 1000 s). Therefore, three large (~ 1 cm length and diameter) Pakistani olivine crystals were bought. Unfortunately, they appeared to be far from gem quality. One of the crystals, Perm169, was examined using EBSD and the universal stage to resolve the crystal faces shown in Fig. A.1a.

One crystal face was sectioned and doubly polished for FT infrared spectroscopy (Fig. A.1b). No effort was made to determine the OH content, but the absorbance is significant. Several structural peaks at specific wave numbers are present. These peaks correspond to the following: 3704 cm^{-1} is a serpentine peak (Berry et al., 2005), 3600 cm^{-1} indicates H at an Si-vacancy site (Lemaire

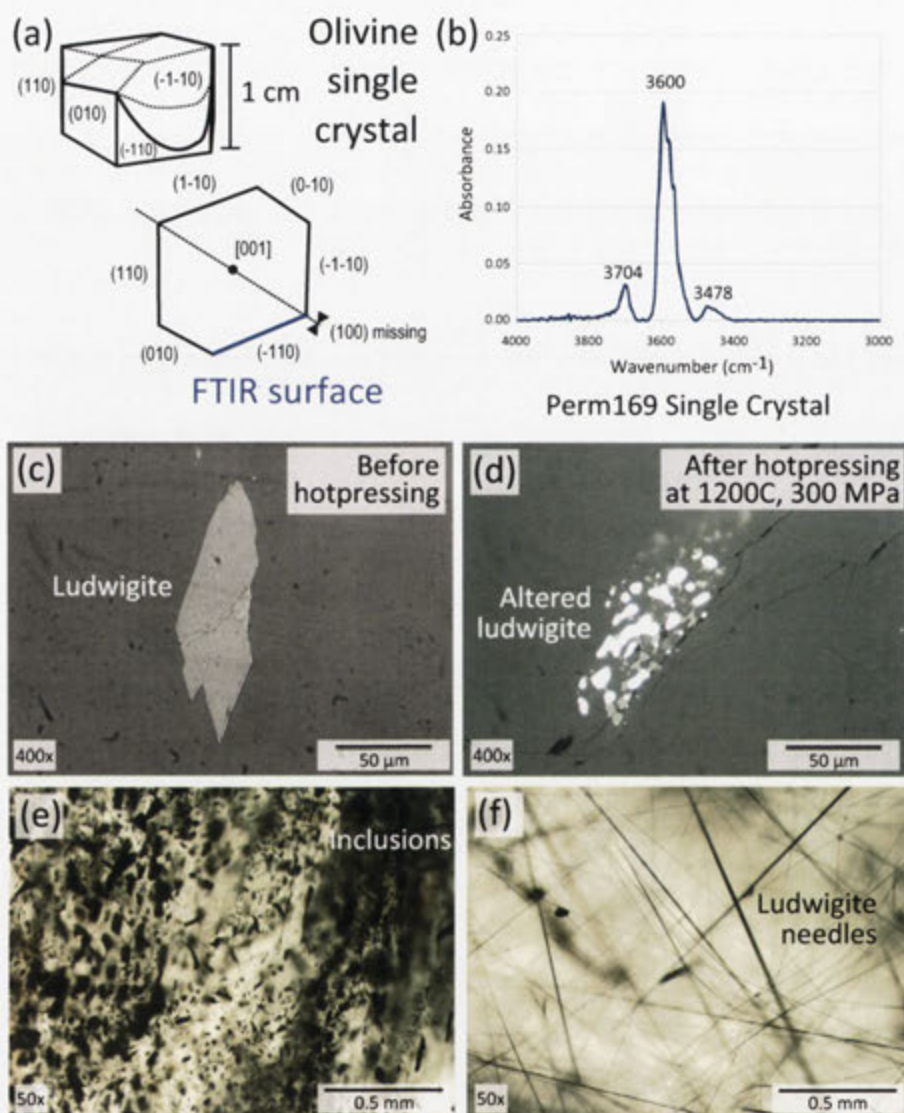


Fig. A.1: Results from a single crystal olivine as a candidate for deformation and attenuation experiments. (a) Identification of the crystal faces of Perm169 and indication of the surface for FTIR measurements shown in (b). The subsequent panels are light microscope images of a section prepared from the single crystal. (c) and (d) show the before and after results of a hot-pressing experiment at high temperature. (e) and (f) show respectively inclusions and borate needles present in the olivine single crystal.

et al., 2004; Berry et al., 2005) for a silica activity < 0.5 or H at a Mg site if the silica activity is buffered at 1 (Lemaire et al., 2004) and lastly 3478 cm^{-1} may correspond to a Ti planar defect (Berry et al., 2005).

Another thin section was prepared for the light microscope (Fig. A.1c,e,f). Thin black needles were abundantly observed and reported* to be a borate mineral called ludwigite (nominally Mg_2FeBO_5), which is consistent with the geological setting in which these olivine single crystals were found – namely in a hydrothermal environment. Numerous inclusions were also observed (Fig. A.1e).

To test the degree to which these impurities will react inside the olivine single crystal, a high temperature hot-pressing experiment was performed. A single crystal cube was sandwiched between two slices of sintered sol-gel olivine. The whole was loaded into a specimen assembly with alumina pistons on either side and lowered into the pressure vessel. The specimen was exposed to 300 MPa of Ar confining pressure and subsequently heated to 1200°C for 24 hours. The single crystal was recovered and sectioned again for the light microscope. The result is shown in Fig. A.1d. The original borate mineral appeared to have reacted with the olivine showing distinct melt pockets.

Given the impure crystals and their relatively short lengths, it was decided to abandon the deformation of single crystal olivine for attenuation experiments. The contributions from melt, cracks and impurities would have significantly complicated interpretations of the seismic data.

*<http://www.mineralminers.com>

B

Torsional deformation of sol-gel and San

Carlos olivine

B.1 Evidence for slip

The unprocessed data from the torsion experiments are plotted for maximum shear strain against time and torque against maximum shear strain for both San Carlos (Fig. B.1a,b) and sol-gel (Fig. B.1c,d) olivine respectively. The coloured slopes in Fig. B.1a,c represent constant shear strain rates, proportional to the programmed constant twist rate steps (the original record is continuous, see figure caption). The strain rates were obtained from linear regression of the slopes. A similar plot of twist angle against time would allow determination of the twist rates. The programmed and measured twist rates were comparable for low values, but not for larger values (see Chapter 4, Table 4.2). The cause for this may be variable friction during the experiments due to either an effect of the apparatus or the alignment of the assembly. This translates as a compliance effect for an internal twist equal to the external twist minus the compliance. However, because the compliance is a function of time or strain, the internal twist rate is also equal

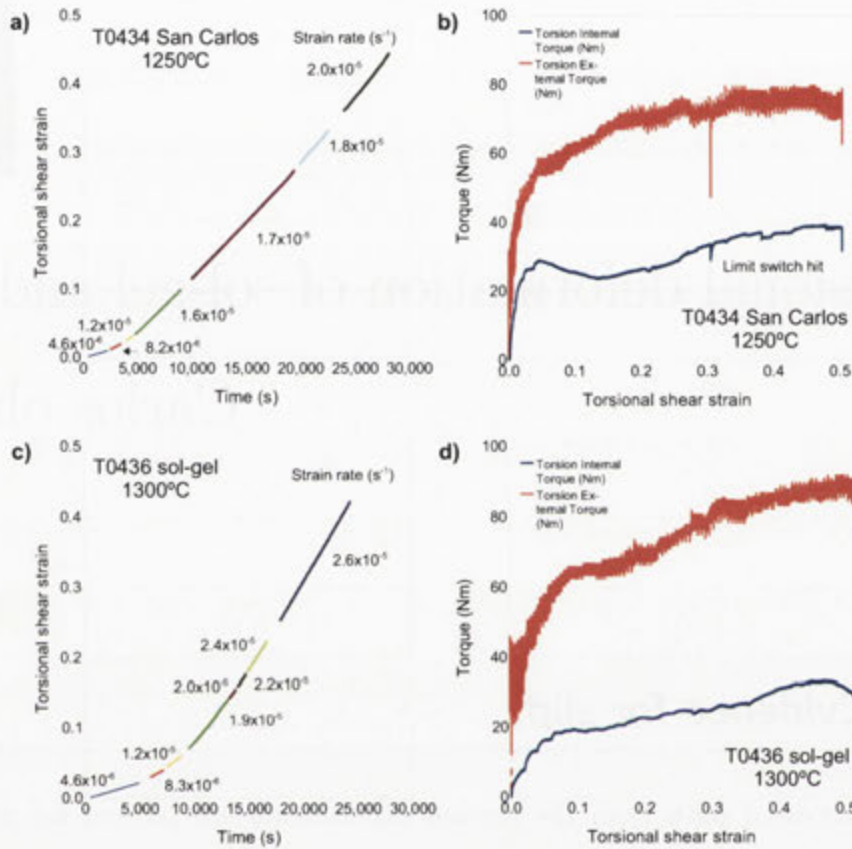


Fig. B.1: Shear strain versus time and torque versus shear strain figures for two torsionally pre-deformed olivine specimens T0434 (San Carlos) and T0436 (sol-gel). In (a) and (c) the coloured slope segments reflect the maximum strain rate, as labelled to each curve segment and proportional to each programmed constant twist rate step. The original record is continuous. The gaps between the slope segments, from one twist rate to another, facilitated more accurate determinations of the shear strain (and twist rates). In (b) and (d) the internal and external torque are plotted as a function of shear strain. Due to changes in the internal friction during the experiments these two curves are not the same. In (b) a safety twist angle limit was briefly hit at a shear strain of $\gamma \sim 0.3$ as indicated.

to the external twist rate minus the compliance twist rate. The issue is currently investigated by attaching a thermocouple to an internal piston and linking it to a rotary variable differential transformer to measure the effect of the compliance (Private communication with Mark Zimmerman, 17-07-2010).

Fig. B.1b,d shows the difference between the internal and external torque during the experiments. The increase observed in the external torque versus decrease in the internal torque is a reflection of changes in friction during the experiments (as discussed above).

Comparing both plots in Fig. B.1, a large drop in the internal torque is seen for San Carlos olivine for almost $\gamma = 0.1$ shear strain. A critical point was reached after stepping up the twist rate to 0.1 mrad s^{-1} . Upon recovery of the internal torque (or shear stress), the twist rate was further incrementally increased for the duration of the experiment until the coupling between the porous alumina discs and the olivine specimen was lost. Similar behaviour was observed for sol-gel olivine.

Photographs of the recovered specimen assemblies (Fig. B.2) show that slip did indeed occur at the interfaces. However, the jacket crimp strain marker (originally straight) demonstrates that both specimens were deformed in torsion. The direction of twist is indicated by the arrows. The sol-gel olivine in Fig. B.2a deformed non-uniformly along the length of the specimen with more shear strain at the upper end than at the lower end.

B.2 Early microstructural observations

Subsequent preliminary observations of the deformed microstructures are shown in Fig. B.3 for prepared thin sections ($\sim 10 \text{ }\mu\text{m}$ thick) under the light microscope. Crystallographic preferred orientations (CPO) of grains are identified by the change in birefringence colours when rotating the stage through 90° . This is strongest in sol-gel olivine (Fig. B.3a,b,c,d), sectioned on the tangential side close to the rim of the cylindrical specimen. This permitted microstructural ob-

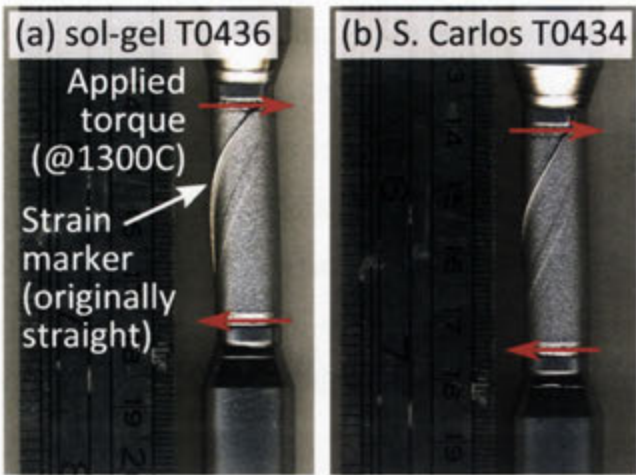


Fig. B.2: Photographs of the recovered specimen assemblies after torsional deformation. The arrows indicate the direction of twist. The crimp in the jackets (originally a straight line parallel to the cylinder axis) is the strain marker from which the shear strain in the specimen was measured. At the upper specimen interface with the porous alumina disc, localised shearing occurred (i.e. slip).

servation of the shear plane in the zone of highest strain. The shear direction and sense is indicated by the arrows. San Carlos olivine was similarly sectioned but does not show a strong CPO (Fig. B.3e,f). The grain size is slightly coarser in San Carlos olivine and lacks the shape preferred orientation clearly present in the sol-gel specimen. Using an additional sensitive tint plate (i.e. the full-wave plate inserted for all micrographs in Fig. B.3), the slow crystallographic direction in olivine [100] was found to be in the direction indicated by the arrow (Fig. B.3d).

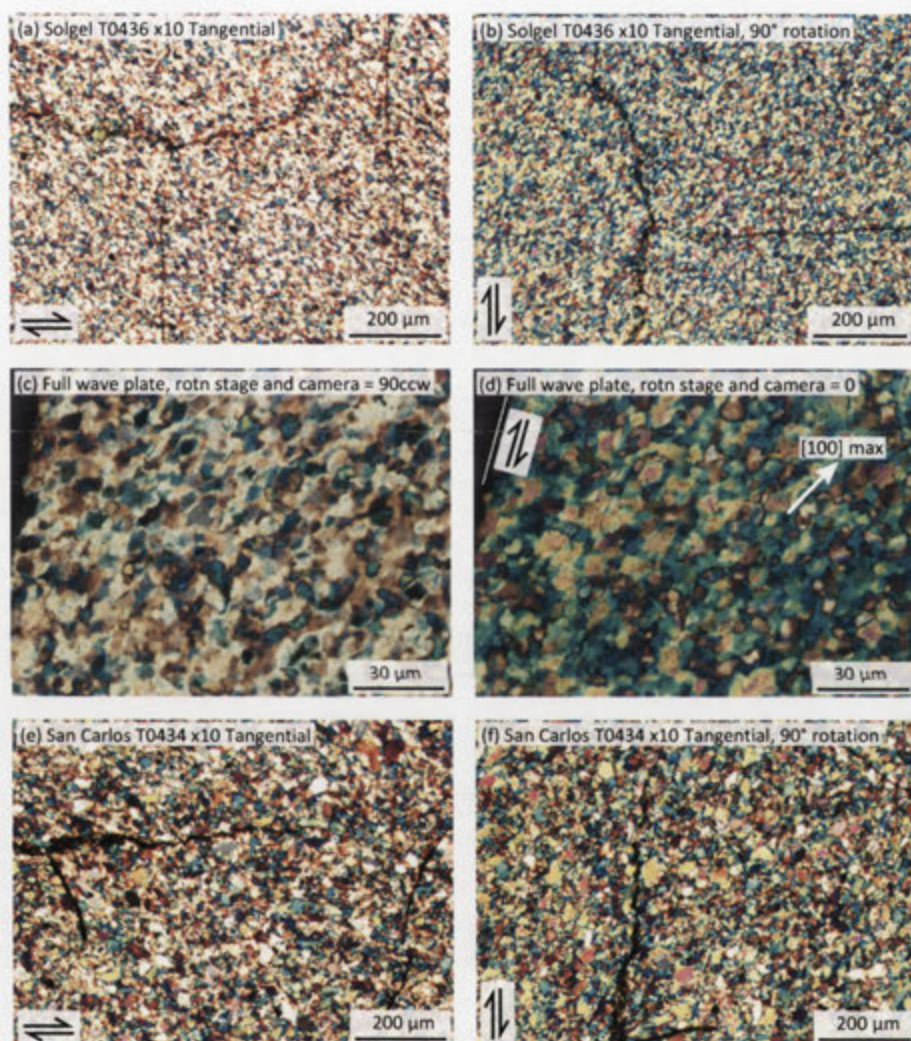


Fig. B.3: Light microscope birefringence images of torsionally pre-deformed sol-gel olivine (a) – (d) and San Carlos olivine (e) – (f). The thin sections were prepared from the regions of maximum shear strain. The shear directions are shown by the arrows. In panels (c,d) the camera is rotated 90° with the stage.



Prospects for damping by dislocation glide in pre-deformed olivine

The Schmid factor analysis presented in Chapter 4 is shown here in more detail for torsional deformation of a single crystal and polycrystal. In addition, more steps are shown for the determination of the averaged anelastic shear strain for randomly oriented crystallites pre-deformed in compression and torsion. Most of these calculations were provided by Professor Ian Jackson.

C.1 Torsional deformation

Recalling from Chapter 4, section 4.8, the orthogonal system $\{\hat{x}'_1, \hat{x}'_2, \hat{x}'_3\}$ is related to the torsional co-ordinate frame $\{\hat{x}'_1, \hat{r}(\theta), \hat{t}(\theta)\}$ (Fig. 4.8) via the following

expressions containing the relevant direction cosines a_{ij}

$$\begin{aligned}
 \hat{r}(\theta) &= \cos \theta \cdot \hat{x}'_2 + \sin \theta \cdot \hat{x}'_3 \\
 &= (1/B) \{ \cos \theta [-\beta, \alpha, 0] + \sin \theta [-\alpha\gamma, -\beta\gamma, (\alpha^2 + \beta^2)] \} \\
 &= (1/B) [-\beta \cos \theta - \alpha\gamma \sin \theta, \alpha \cos \theta - \beta\gamma \sin \theta, (\alpha^2 + \beta^2) \sin \theta] \\
 \hat{t}(\theta) &= -\sin \theta \cdot \hat{x}'_2 + \cos \theta \cdot \hat{x}'_3 \\
 &= (1/B) [\beta \sin \theta - \alpha\gamma \cos \theta, -\alpha \sin \theta - \beta\gamma \cos \theta, (\alpha^2 + \beta^2) \cos \theta]
 \end{aligned}$$

where $B = (\alpha^2 + \beta^2)^{1/2}$.

The direction cosines relating the local torsional reference frame to the crystal frame are as follows

$$\begin{aligned}
 a_{11} &= \cos \angle(x'_1, x_1) = \alpha \\
 a_{12} &= \cos \angle(x'_1, x_2) = \beta \\
 a_{13} &= \cos \angle(x'_1, x_3) = \gamma \\
 a_{21} &= \cos \angle(r, x_1) = (-1/B)(\beta \cos \theta + \alpha\gamma \sin \theta) \\
 a_{22} &= \cos \angle(r, x_2) = (1/B)(\alpha \cos \theta - \beta\gamma \sin \theta) \\
 a_{23} &= \cos \angle(r, x_3) = (1/B)(\alpha^2 + \beta^2) \sin^2 \theta \\
 a_{31} &= \cos \angle(t, x_1) = (1/B)(\beta \sin \theta - \alpha\gamma \cos \theta) \\
 a_{32} &= \cos \angle(t, x_2) = (-1/B)(\alpha \sin \theta + \beta\gamma \cos \theta) \\
 a_{33} &= \cos \angle(t, x_3) = (1/B)(\alpha^2 + \beta^2) \cos \theta
 \end{aligned}$$

The resolved shear stress for (010)[100] is thus

$$\begin{aligned}
 \sigma_{12}(\theta) &= a_{31}a_{12}\sigma'_{31} + a_{11}a_{32}\sigma'_{13} \\
 &= (a_{31}a_{12} + a_{11}a_{32})\sigma_t
 \end{aligned}$$

where σ_t is the torsional shear stress at specified radial position r . Hence

$$\begin{aligned}
 \sigma_{12}(\theta) &= (1/B) [\beta(\beta \sin \theta - \alpha \gamma \cos \theta) - \alpha(\alpha \sin \theta + \beta \gamma \cos \theta)] \sigma_t \\
 &= (-1/B) [2\alpha\beta\gamma \cos \theta + (\alpha^2 - \beta^2) \sin \theta] \sigma_t \\
 &= (-1/B) [(2\alpha\beta\gamma)^2 + (\alpha^2 - \beta^2)^2]^{1/2} [\cos \phi \cos \theta + \sin \phi \sin \theta] \sigma_t \\
 &= (-1/B) [(2\alpha\beta\gamma)^2 + (\alpha^2 - \beta^2)^2]^{1/2} \cos(\theta - \phi) \sigma_t \\
 &= -T(\alpha, \beta) \cos(\theta - \phi) \sigma_t
 \end{aligned}$$

where

$$T^2(\alpha, \beta) = \alpha^2 + \beta^2 - (2\alpha\beta)^2 \quad \tan \phi(\alpha, \beta, \gamma) = (\alpha^2 - \beta^2)/2\alpha\beta\gamma$$

For axial compression, the simple shear strain resulting from (010)[100] slip can be determined as $2\epsilon_{12} = 2\epsilon_{21} = \partial u_1 / \partial x_2$ proportional to $\sigma_{12}(\theta)$. Hence the contribution of slip to torsional shear strain is

$$\epsilon'_{31}(\theta) = (a_{31}a_{12} + a_{32}a_{11})\epsilon_{12}(\theta) = A(a_{31}a_{12} + a_{32}a_{11}^2)\sigma_t \quad (\text{A-1})$$

So the potential elastic torsional shear strain varies with azimuth around $[\alpha, \beta, \gamma]$ as

$$a_{31}a_{12} + a_{32}a_{11}^2 = T^2(\alpha, \beta) \cos^2(\theta - \phi) = (1/2)T^2(\alpha, \beta)[1 + \cos 2(\theta - \phi)]$$

C.2 Averaged anelastic shear strain in an aggregate

As we have seen, for olivine crystallites, the shear strains may be averaged for all possible orientations $[\alpha\beta\gamma]_c$.

The problem can be approached in detail by determining a relationship between an arbitrary reference frame and the unit vector $[\alpha\beta\gamma]_c$ for all possible rotations in a unit sphere. The relationship between (α, β, γ) and the angles (θ, ϕ)

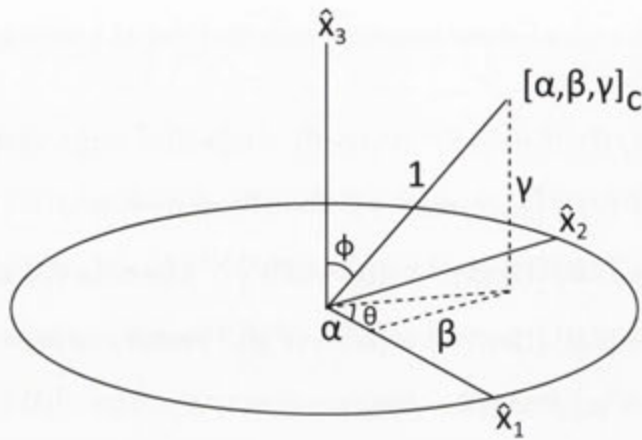


Fig. C.1: Orientation of the unit vector $[\alpha\beta\gamma]_c$ in spherical co-ordinates.

may be expressed in spherical co-ordinates as

$$\alpha = \sin \phi \cos \theta \quad \beta = \sin \phi \sin \theta \quad \gamma = \cos \phi$$

Equivalently

$$\phi = \cos^{-1}(\gamma) \quad \theta = \tan^{-1}(\beta/\alpha)$$

Note that the angles θ and ϕ , thus defined (Fig. C.1) are different from those describing latitude around the torsional axis in Fig. 4.8. There are two types of rotations considered here. One is the rotation about $\hat{x}_3$ through an angle θ to $\theta + d\theta$ and the other one is through an angle ϕ to $\phi + d\phi$. An area element on the unit sphere swept out by these two rotations is given by

$$dA = \sin \phi \cdot d\theta d\phi \quad (\text{A-1})$$

The total angular area of a sphere is 4π steradians or equivalent to $1/(4\pi)$ of the fractional area. Hence the probability that the unit vector lies within the solid angle subtended by the element of area given in Eqn. (A-1) is

$$p(\theta, \phi) = \sin \phi / (4\pi)$$

and the average value of any quantity $X(\theta, \phi)$ is

$$\bar{X} = \int_0^\pi \int_0^{2\pi} p(\theta, \phi) \cdot X(\theta, \phi) \cdot d\theta d\phi \quad (\text{A-2})$$

Using Eqn. (A-2), it is now possible to calculate average values of anelastic strain for uni-axial compression and torsion.

C.2.1 Deformation in compression

For compression we have

$$\begin{aligned} X(\theta, \phi) &= \epsilon_{an} \cong (\alpha\beta)^2 \\ &= (\sin^2 \phi \sin \theta \cos \theta)^2 \\ &= 1/4(\sin^2 \phi \sin 2\theta)^2 \end{aligned}$$

so that for the average anelastic strain

$$\begin{aligned} \bar{\epsilon}_{an} &\cong (1/16\pi) \int_0^\pi \int_0^{2\pi} \sin \phi \cdot \sin^4 \phi \sin^2 2\theta \cdot d\theta d\phi \\ &= (1/16\pi) \int_0^\pi \sin^5 \phi \cdot d\phi \int_0^{2\pi} \sin^2 2\theta \cdot d\theta \\ &= (1/16) \int_0^\pi \sin^5 \phi \cdot d\phi \end{aligned}$$

Integrating by parts

$$\begin{aligned} \int_0^\pi \sin^5 \phi \cdot d\phi &= - \left[\frac{\sin^4 \phi \cos \phi}{5} \right]_0^\pi + \frac{4}{5} \int_0^\pi \sin^3 \phi \cdot d\phi \\ &= \frac{4}{5} \left\{ - \left[\frac{\sin^2 \phi \cos \phi}{3} \right]_0^\pi + \frac{2}{3} \int_0^\pi \sin \phi \cdot d\phi \right\} \\ &= \frac{4}{5} \cdot \frac{2}{3} [-\cos \phi]_0^\pi \\ &= \frac{4}{5} \cdot \frac{4}{3} \\ &= \frac{16}{15} \end{aligned}$$

Therefore the maximum averaged anelastic strain for a polycrystal deformed in compression is

$$\bar{\epsilon}_{an} \cong (1/16)(16/15) = 1/15 \quad (\text{A-3})$$

compared to a maximum $[110]_c$ single crystal value of $(0.5)^2 = 1/4$.

C.2.2 Deformation in torsion

The potential anelastic strain for torsion of each crystallite varies as $T^2(\alpha, \beta) \cos^2 \lambda$ with azimuth $\lambda = (\theta - \phi)$ about the torsional axis $[\alpha, \beta, \gamma]_c$ resulting in an azimuthally averaged value of

$$\begin{aligned} T^2(\alpha, \beta)/2 &= (1/2)[\alpha^2 + \beta^2 - (2\alpha\beta)^2] \\ &= (1/2)[\sin^2 \phi - \sin^4 \phi \sin^2 2\theta] \end{aligned}$$

The average of this quantity over randomly oriented grains is

$$\begin{aligned} \bar{\epsilon}_{an} &\cong \int_0^\pi \int_0^{2\pi} \left(\frac{\sin \phi}{4\pi} \right) \frac{1}{2} (\sin^2 \phi - \sin^4 \phi \cdot \sin^2 2\theta) \cdot d\theta d\phi \\ &= \frac{1}{8\pi} \int_0^\pi \int_0^{2\pi} \sin^3 \phi - \sin^5 \phi \cdot \sin^2 2\theta \cdot d\theta d\phi \end{aligned}$$

Evaluating the inner integral

$$\begin{aligned} \int_0^{2\pi} \sin^3 \phi - \sin^5 \phi \cdot \sin^2 2\theta \cdot d\theta &= \int_0^{2\pi} \sin^3 \phi \cdot d\theta - \int_0^{2\pi} \sin^5 \phi \sin^2 2\theta \cdot d\theta \\ &= [\theta \sin^3 \phi]_0^{2\pi} - \sin^5 \phi \int_0^{2\pi} \sin^2 2\theta \cdot d\theta \\ &= 2\pi \sin^3 \phi - \pi \sin^5 \phi \\ &= \pi(2 \sin^3 \phi - \sin^5 \phi) \end{aligned}$$

and evaluating the outer integral where $\int_0^\pi \sin^3 \phi \cdot d\phi = 4/3$ and $\int_0^\pi \sin^5 \phi \cdot d\phi = 16/15$ as before (see Eqn. (A-3))

$$\begin{aligned}\bar{\epsilon}_{an} &\cong \frac{1}{8\pi} \int_0^\pi \pi(2 \sin^3 \phi - \sin^5 \phi) \cdot d\phi \\ &= 3/15\end{aligned}\tag{A-4}$$

which is the averaged anelastic strain for a polycrystal deformed in torsion compared to a maximum single-crystal value for [010] and [100] of 1/2.

Similar procedures for orientational averaging are involved in calculating the quantities $[S_c^2 S_t^2]_{av}$ and $[S_t^4]_{av}$ for use in Chapter 4.

D

Some notes on dislocation elasticity theory

Both the stress and strain imposed on a material as well as on a dislocation are described by second order tensors in elasticity theory. This means that both contain nine independent components that entirely describe the state of deformation depending on the degree of anisotropy of the material. In both cases the tensor can be broken up into two parts. One describes the principal, mean, components of the stress or strain (i.e. normal to the surface) and the other part describes the deviatoric components. For example, deviatoric stresses cause shear for the case of the stress tensor. In other words, the normal components cause volume changes and the deviatoric components cause shape changes. The stress and strain are related to each other by a fourth order tensor with 81 independent components. This is the compliance or the stiffness tensor, where the compliance is multiplied by the stress tensor to give the strain tensor and the stiffness tensor is multiplied by the stress tensor to give the strain tensor. These two expressions are equivalent to Hooke's law for anisotropic elastic materials.

In the context of mechanical dislocation damping whereby a pre-deformed

material experiences a viscoelastic response to torsional forced-oscillations, the expressions given may be rewritten for oscillating stress. These simplified results indicate that dislocations will respond to shear stresses (S-waves) but much less to compressional stresses (P-waves). Many of these theoretical considerations are reproduced from Hull and Bacon (2001).

For an anisotropic solid with 81 independent components, the involved tensor calculations can be first approximated by considering an isotropic solid to obtain a generalised version of the stress and strain field of a dislocation. For a uni-axial stress case we have

$$\epsilon_{11} = \frac{\sigma_{11}}{E} \quad \epsilon_{22} = \frac{-\nu\sigma_{11}}{E} \quad \epsilon_{33} = \frac{-\nu\sigma_{11}}{E}$$

where $\epsilon_{22} = \epsilon_{33}$ and $\nu = -\epsilon_{22}/\epsilon_{11}$, or Poisson's ratio. Likewise, there are three ϵ_{ii} contributions for σ_{22} and σ_{33} respectively. Combining them gives the following three relations.

$$\begin{aligned} \epsilon_{11} &= \frac{\nu}{E} \left(\frac{\sigma_{11}}{\nu} - \sigma_{22} - \sigma_{33} \right) \\ \epsilon_{22} &= \frac{\nu}{E} \left(\frac{\sigma_{22}}{\nu} - \sigma_{11} - \sigma_{33} \right) \\ \epsilon_{33} &= \frac{\nu}{E} \left(\frac{\sigma_{33}}{\nu} - \sigma_{11} - \sigma_{22} \right) \end{aligned}$$

Therefore, for an isotropic cube-shaped material the general stress-strain equations are

$$2G\epsilon_{ij} = \sigma_{ij} - \left(1 - \frac{2G}{3K}\right) \bar{\sigma} \delta_{ij}, \quad \sigma_{ij} = \lambda \epsilon_{kk} \delta_{ij} + 2G\epsilon_{ij} \quad (\text{A-1})$$

where G is the shear modulus, K is the bulk modulus, $\bar{\sigma}$ is the mean stress $\sigma_{kk}/3$, δ_{ij} is the Kronecker delta and λ is the Lamé constant given as $\lambda = (3K - 2G)/3$.

D.1 Screw dislocation

The elastic distortion around a straight screw dislocation of infinite length can be approximated using the general equations in A-1. The screw displacement is shown in Fig. D.1 by a vertical jump in an otherwise perfect cylinder. This distance equals the dislocation Burgers vector, b . Since the only displacement is in the vertical (z) direction, $u_x = u_y = 0$. In the z direction the displacement varies from 0 to b with the angle θ . This is expressed as

$$u_z = \frac{b\theta}{2\pi} = \frac{b}{2\pi} \cdot \tan(y/x)$$

The equivalent stress field for a screw dislocation can be found using Eqn. A-1. The result is the following:

$$\begin{aligned}\sigma_{xx} &= \sigma_{yy} = \sigma_{zz} = \sigma_{xy} = \sigma_{yx} = 0 \\ \sigma_{xz} &= \sigma_{zx} = -\frac{G \cdot b}{2\pi} \cdot \frac{y}{x^2 + y^2} \cdot \frac{\sin \theta}{r} \\ \sigma_{yz} &= \sigma_{zy} = -\frac{G \cdot b}{2\pi} \cdot \frac{x}{y^2 + x^2} \cdot \frac{\cos \theta}{r}\end{aligned}$$

Or in polar co-ordinates we get the simple expression:

$$\sigma_{\theta z} = \sigma_{z\theta} = \frac{G \cdot b}{2\pi r} \quad (\text{A-1})$$

From these equations it is apparent that for a screw dislocation there are no tensile or compressive components and the stress field is basically pure shear. However, there is a limitation for these expressions. When approaching the dislocation core (or centre of the cylinder), the stresses (and strains) will go to infinity since they are proportional to $1/r$. This can be empirically approximated by limiting the rise of stress / strain towards the dislocation core to some maximum instead of infinity.

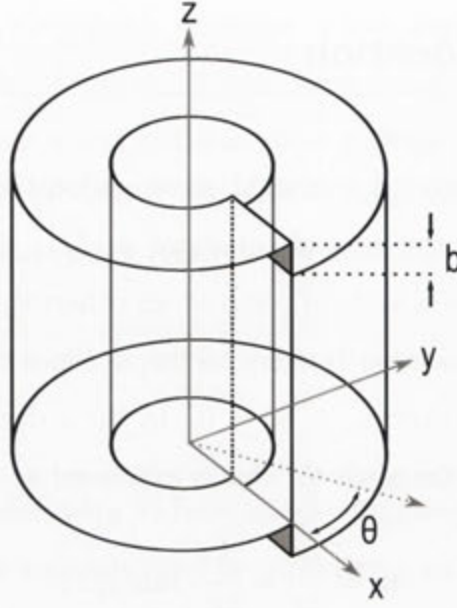


Fig. D.1: A schematic of a straight infinite screw dislocation. b represents the Burgers vector

D.2 Edge dislocation

Similarly, for an edge dislocation, the same stress field expressions can be derived based on a distorted cylinder as shown as in Fig. D.2. Here the deformation is simply plane strain deformation. Note that in this case there is no displacement in the z direction. The expressions for a stress field of an edge dislocation are as follows

$$\sigma_{zz} = \sigma_{zx} = \sigma_{yz} = \sigma_{zy} = 0$$

$$\sigma_{xx} = -B \cdot y \frac{3x^2 + y^2}{(x^2 + y^2)^2}$$

$$\sigma_{yy} = -B \cdot y \frac{x^2 - y^2}{(x^2 + y^2)^2}$$

$$\sigma_{xy} = \sigma_{yx} = B \cdot x \frac{x^2 - y^2}{(x^2 + y^2)^2}$$

$$\sigma_{zz} = \nu \cdot (\sigma_{xx} + \sigma_{yy})$$

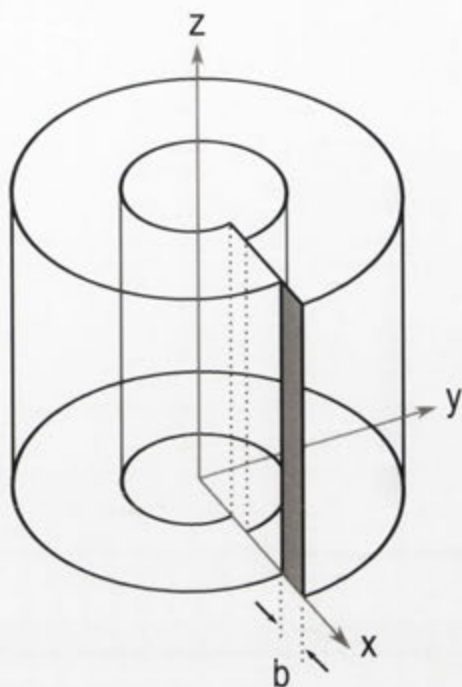
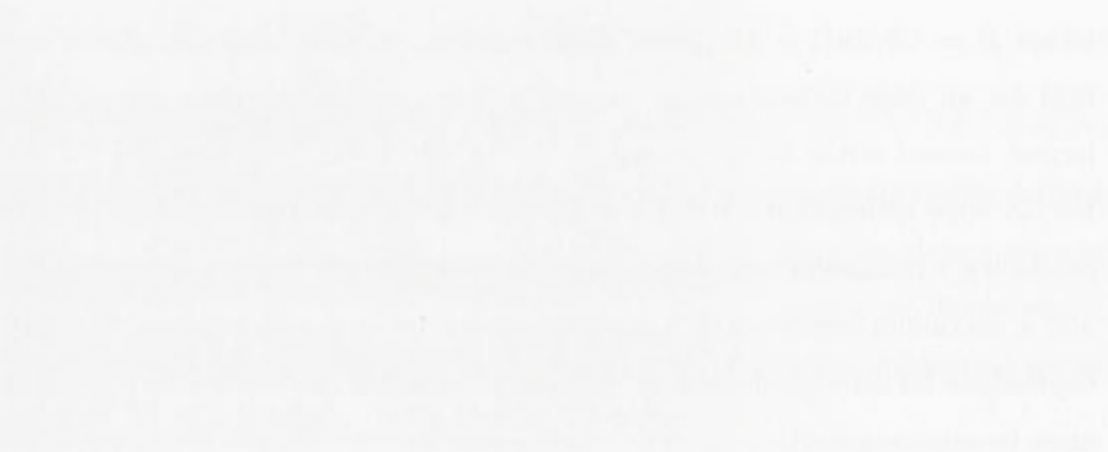


Fig. D.2: A schematic of a straight infinite edge dislocation. b represents the Burgers vector

where $B = Gb/2\pi(1 - \nu)$. From these equations it is evident that the stress field for an edge dislocation has both dilational and shear components. The largest normal stress is σ_{xx} acting parallel to the Burgers vector (Fig. D.2). For an edge dislocation with the y direction normal to the slip plane, σ_{xx} is producing a maximum compressional stress above the slip plane with ($y = +ve$) and a maximum tensile stress below the slip plane ($y = -ve$). As before, these expressions for an edge dislocation become invalid near the dislocation core and must be approximated.



Slip system and EBSD analysis on compressively deformed synthetic sol-gel and San Carlos olivine aggregates

E.1 Introduction

* Olivine, the main constituent of the Earth's upper mantle, has been thoroughly studied in the past several decades to understand how crystallographic preferred orientations (CPO) develop under upper mantle conditions due to prevailing plate tectonic forces (Ben Ismaïl and Mainprice, 1998; Tommasi et al., 2000; Jung et al., 2006; Karato, 2008). Lattice or crystallographic preferred orientation development commonly occurs in olivine during plastic deformation (i.e. high temperature dislocation creep) where individual grains rotate and / or recrystallise, promoting anisotropy in seismic measurements (Karato, 1988; Lee et al., 2002; Drury and Pennock, 2007).

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Several slip systems potentially operate in olivine during plastic deformation. According to the von Mises criterion, a minimum of 5 slip systems is needed to deform a material without changing its volume. A high Schmid factor (with a maximum value of 0.5) is attributed to a slip system when the geometrical orientation of the compression direction on an olivine crystal is favourable to cause slip on one of the atomic slip planes. Studies on the fabric and seismic anisotropy of olivine have shown that dominant slip systems firstly depend on the critical resolved shear stress (CRSS) and angle of stress relative to a slip system. Other factors can be the presence of water and possibly trace elements.

The type-A CPO typically develops in low-OH natural olivine (single crystal and polycrystalline) for high temperature plastic deformation ($> 1000^{\circ}\text{C}$) (Green and Radcliffe, 1972; Goetze, 1978; Ben Ismaïl and Mainprice, 1998; Jung and Karato, 2001). Slip system models employing up to three active slip systems (Ribe and Yu, 1991; Wenk et al., 1991) confirm that this CPO type is associated with dominant slip in the $[100]$ direction on the (010) slip plane. Additional slip systems may be operating in parallel with, for example, several planes intersecting the $[100]$ direction and is often summarised as pencil glide on $(0kl)[100]$ (Passchier and Trouw, 1998). Other CPO types are summarised in Jung et al. (2006) for a variety of water concentrations.

Two end-member models describe the interactions between grains during deformation, the uniform strain (Taylor) and uniform stress (Sachs) models (e.g. Winther et al. 1997; Dawson and Wenk 2000). In the Taylor model, some grains require higher grain boundary stresses than others to produce uniform strain. In the Sachs model, some grains in favourable orientations deform more than neighbouring grains, some unfavourably oriented grains do not deform at all. In general, a positive correlation between dislocation density (ρ) and Schmid factor is more likely to be expected if grain interactions obey the Sachs model rather than the Taylor model (Karato and Lee, 1999). However, it is likely that the deformation involves heterogeneous stress and strain as implemented in the

Self-Consistent model for a limited number of slip systems (Wenk et al., 1991; Tommasi et al., 2000). More recent modelling requires activation of four essential slip systems in olivine plus another degree of freedom to describe the microscopic stress heterogeneities (Castelnau et al., 2008).

This study investigates to what extent the Taylor and Sachs models are applicable to compressively deformed fine-grained olivine aggregates, prepared from laboratory and natural precursors, by comparing dislocation density with Schmid factor for several slip systems in olivine.

E.2 Method

Solution-gelation (sol-gel) olivine material was prepared from dissolution of Fe and Mg nitrates in an ethanol solution (Jackson et al., 2002; Faul and Jackson, 2007). A second type of olivine material was prepared from crushed San Carlos phenocrysts (Tan et al., 2001). Both were fired at 1400°C at a 50/50 CO/CO₂ gas mix and hot-pressed in 70%Ni-30%Fe foil at 1300°C and 300 MPa Ar confining pressure in a Paterson gas-medium apparatus (Paterson, 1990). The porosity is reduced to 1 – 2%. After successful hot-pressing, one cylindrical sol-gel (H6529) and one cylindrical San Carlos specimen (H6694) were obtained.

Compressive deformation experiments were carried out on both specimens using the same Paterson apparatus at 300 MPa confining pressure and 1250°C. The sol-gel sample was deformed following a series of ramps and dwells of increasing loads (Faul and Jackson, 2007, run D6532, Fig. 8). Care was taken to make sure steady-state creep was achieved for every dwell and the confining pressure was not exceeded. The maximum load step was equivalent to a differential stress of 266 MPa and the total strain was 18.9%. The San Carlos specimen was deformed to a maximum differential stress of 227 MPa and a total strain of 15% (run D6701). The experiments were terminated with a furnace quench to preserve the microstructure and dislocations. Table E.1 contains a summary of the rheological data of the deformed samples described above.

Table E.1: Rheology data of sol-gel and San Carlos samples deformed at 1250°C

Sample	ϵ_{total}	σ_{max}	$\dot{\epsilon}_{max}$	d	ρ_{low} / ρ_{high}	ρ_{mean}
		(MPa)	(s ⁻¹)	(μm)	(μm^{-2})	(μm^{-2})
Sol-gel	18.9	266	1.1×10^{-4}	4.2 ± 0.2	$1.1 \pm 0.8 / 15.1 \pm 1.7$	8.5 ± 0.2
San Carlos	15	227	1.0×10^{-4}	13.0 ± 0.5	$1.9 \pm 1.1 / 15.7 \pm 2.7$	11.4 ± 2.9

ρ is the dislocation density

Both samples were sectioned perpendicular to the compressive axis and polished using colloidal silica (0.05 μm) and subsequently oxidised at 900°C for 45 min to decorate dislocations (Kohlstedt et al., 1976; Karato, 1987). Subsequent polishing for 10 min using either 0.05 μm alumina slurry (sol-gel olivine) or colloidal silica (San Carlos olivine) removed the surface oxide layer (< 1 μm thick) and about 2 - 6 μm of material to find the region of the best decorated grains and dislocations. The 3-D dislocation density was determined following the procedures outlined in Chapter 2, section 2.4.

A Zeiss Ultraplus Field Emission Gun Scanning Electron Microscope (FE-SEM), equipped with EBSD capabilities, was used in this study. An accelerating voltage of 20 kV was used with a nominal probe current of 400 pA. The stage was tilted to 70° creating a working distance of around 25 mm. An objective aperture of 30 μm with High Current mode was used. Images were collected on a CCD camera, equipped with a forward-scattered electron detector (FSD). A step size of 1 μm was used for automated EBSD mapping on the coarser grained San Carlos material. At these settings, acceptable Mean Angular Deviation (MAD) values were obtained (<0.500) and 85% of data points were indexed.

The raw, stitched, multi-map for San Carlos olivine was given the following noise reduction treatment to obtain CPO data. Initially, wild spikes were removed, followed by a series of individual iteration steps starting with single pixel

outliers. Subsequent manual noise reduction was constantly checked by relying on an overlay band contrast image of the microstructure so no artifacts were introduced. An additional correction was made for systematic misindexing of the [100] and [-100] orientation (due to pseudosymmetry) in olivine. Finally, a one-point-per-grain analysis was performed to produce the (inverse) pole figures

Manual indexing of high and low dislocation density grains for both the sol-gel and San Carlos samples was also performed, guided by OC images. Only one-point-per-grain was indexed for every grain of high and low dislocation density. Backscattered electron images (BEI) of the same grains confirmed the FSD on the Zeiss FESEM identified the same high and low dislocation density grains. Grains of moderate dislocation density were avoided and a portion ($\sim 20\%$) of the indexed grains were analysed for dislocation densities.

The grain size for the San Carlos sample was determined from an EBSD map showing only grain boundaries of $>10^\circ$ misorientations. The average grain size was calculated as the diameter of a circle of equivalent area with a sectioning bias correction of $4/\pi$ (Jackson et al., 2002).

E.3 Results

Fourier transform infrared (FTIR) measurements confirmed the deformed sol-gel sample had a low OH content (< 10 wt. ppm H_2O , below the detection limit) (Costa and Chakraborty, 2008) but an averaged water concentration of 26 wt. ppm H_2O was measured for San Carlos olivine aggregate (D6701).

E.3.1 Microstructural observations

Fig. E.1 shows typical fine-grained olivine microstructures imaged using forward- and back-scattered electron detectors of a heterogeneous distribution of dislocations. For Fig. E.1b a simple binary contrast filter was applied to highlight the dislocations, which were manually recoloured in red. 3-D dislocation density

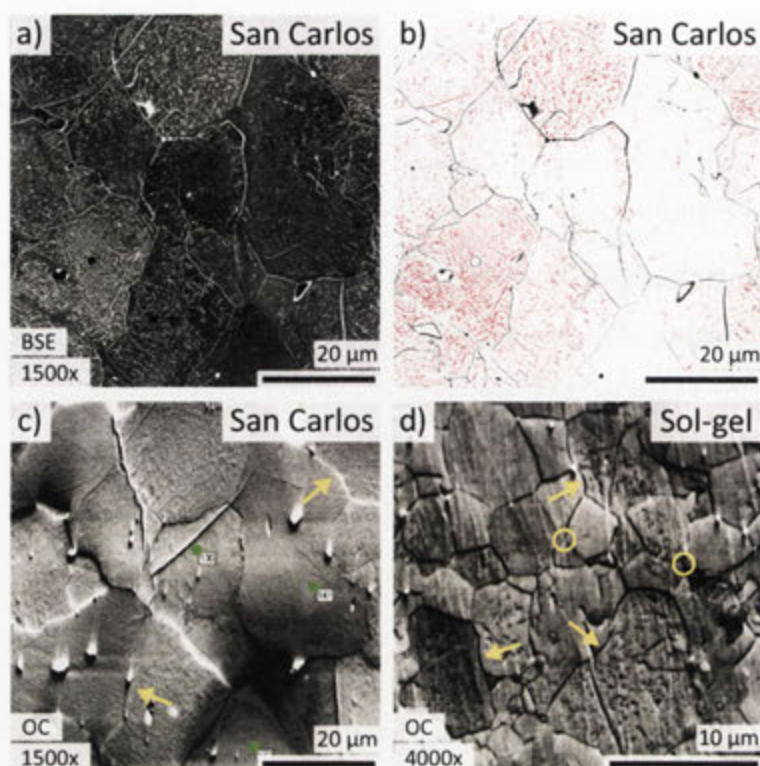


Fig. E.1: (a) Backscatter image (5 kV) of San Carlos olivine showing grain boundaries and internal dislocation density. (b) Same area as a. but dislocations highlighted in red. Used to determine the 3-D dislocation density. (c) Inverted orientation contrast image (20 kV) of San Carlos of the same area to directly compare grains of low and high dislocation density. Three grains show each a manual indexed point indicated by the green square symbols. These grains are indeed low dislocation density grains as seen in b. The yellow arrows indicate grain boundary bulging into high dislocation density grains. (d) Inverted orientation contrast image for sol-gel olivine illustrating a similar heterogeneous density distribution of dislocations amongst grains. The yellow arrows show grain boundary bulging as in c. and the circles point out quadruple junctions. The sol-gel olivine specimen has a smaller grain size than San Carlos olivine.

determination was performed from such processed images. The orientation contrast images were captured to facilitate identification of high and low dislocation density grains during manual indexing. Several low dislocation density grains in Fig. E.1c still show their original markers as shown by the green square symbols. These grains compare accurately with the same grains of low dislocation density in the BSE image (Fig. E.1a).

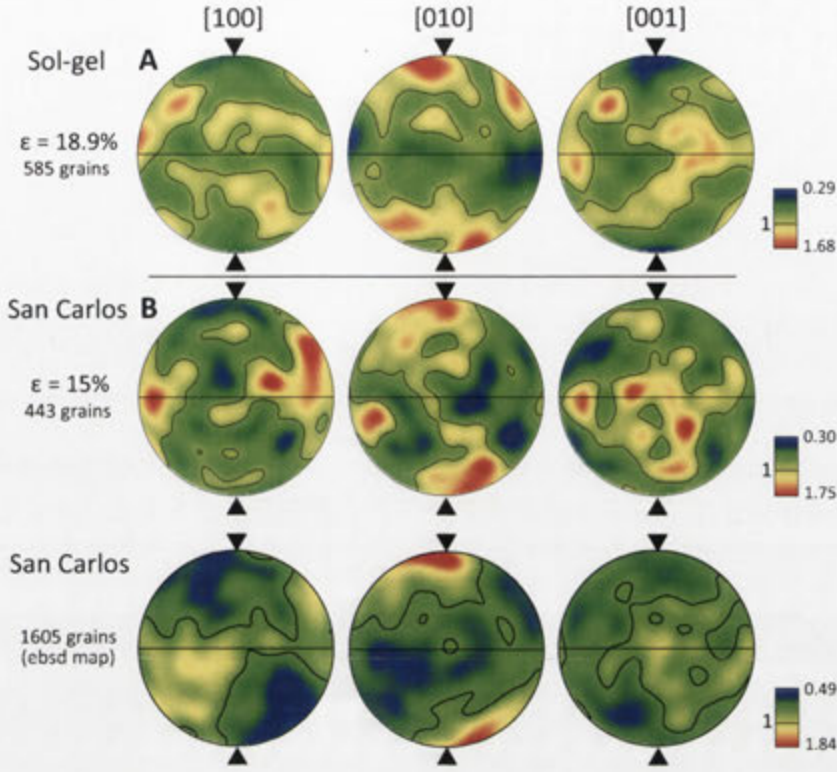


Fig. E.2: Pole figures for all data. The north pole is the compression direction. The half-scatter width is 20° with 5° clustering. An equal area, lower hemisphere projection was used. One-point-per-grain data from the EBSD map for San Carlos olivine is similar to the pole figure pattern obtained by point-indexing high and low dislocation density grains. In all cases, the patterns are weak with low maximum MUD values.

E.3.2 CPOs

Pole figures were obtained from manual indexing of grains of high and low dislocation density from several areas captured on orientation contrast images (see Fig. E.1c and d). Fig. E.2 shows the CPO for the full datasets of manual per-grain measurements on sol-gel and San Carlos olivine plus a dataset from a mapped area of 1605 grains for San Carlos olivine. Note that the overall patterns for preferred orientation are weak for current olivine samples deformed in compression up to $\sim 19\%$ strain. Multiples of uniform distribution (MUD) shown in the CPO patterns only have maximum values up to 1.75 and should be considered when comparing the CPO patterns from this study to others. Generally, Fig. E.2

shows some indication of sub-parallel girdles or point maxima for [100] perpendicular to the compression direction, [010] point maxima are sub-parallel to the compression direction and there is a general scatter of orientations for [001].

In addition, the misorientation index (M-index) was calculated for both samples from the uncorrelated misorientation angles between grains (Skemer et al., 2005). This is a measure of fabric strength complementary to the J index, which in turn is calculated from the distribution of discrete crystallographic orientation data in Euler angle space but is sensitive to the number of grains measured. Both the deformed San Carlos specimen and the sol-gel specimen have M values of 0.03, calculated using a bin of 1.4° . For comparison Skemer et al. (2005) show that an undeformed hot-pressed sample has an M index of 0.01 whereas an olivine aggregate, deformed to a shear strain of $\gamma = 0.6$ (equivalent to 35% compressive strain), was shown to have an M value of 0.17.

In contrast, the CPOs strengthen considerably when data is divided into subsets for high and low dislocation density grains (Fig. E.3). Both samples show for high dislocation density grains a [001] girdle normal to the compression direction. The pattern is complementary for low dislocation density grains with point maxima of [001] orientations parallel to the compression direction. San Carlos olivine shows a concentration of [010] orientations sub-parallel to the compression direction for high dislocation density grains, but this is not evident in sol-gel olivine. The CPOs for [010] between sol-gel and San Carlos olivine do not agree for low dislocation density grains either: sol-gel olivine shows point maxima close to parallel to the compression direction whereas San Carlos olivine shows no distinct CPO at all. Finally, [100] grain orientations for high dislocation density grains in sol-gel olivine are possibly clustered in two separate girdles at 45° to the compression direction but San Carlos olivine does not clearly show the same pattern. The pole figures for [100] of low dislocation density grains in both materials, however, show a girdle perpendicular to the compression direction. For comparison, a predicted CPO is shown for high and low Schmid factor subsets of

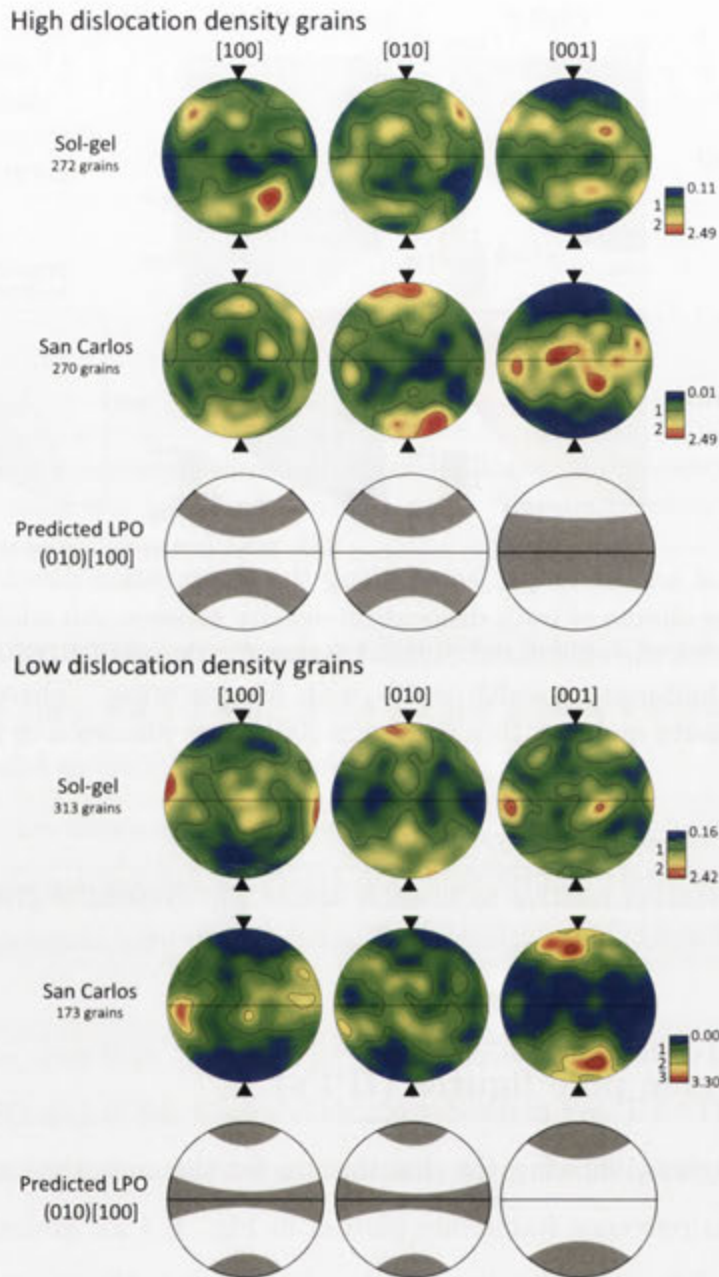


Fig. E.3: Pole figures for the high and low dislocation density grain fractions. The north pole is the compression direction. The half-scatter width is 20° with 5° clustering. An equal area, lower hemisphere projection was used. The CPO patterns in the pole figures between high and low dislocation density appear complementary to each other for both sol-gel and San Carlos olivine. There is some similarity in the CPO patterns among both materials, especially for [100] and [001], although a difference in patterns exist for [010]. In addition, predicted complementary pole figures for high (shaded regions) and low (empty regions) Schmid factors for (010)[100] slip is shown for a random orientation distribution of grains.

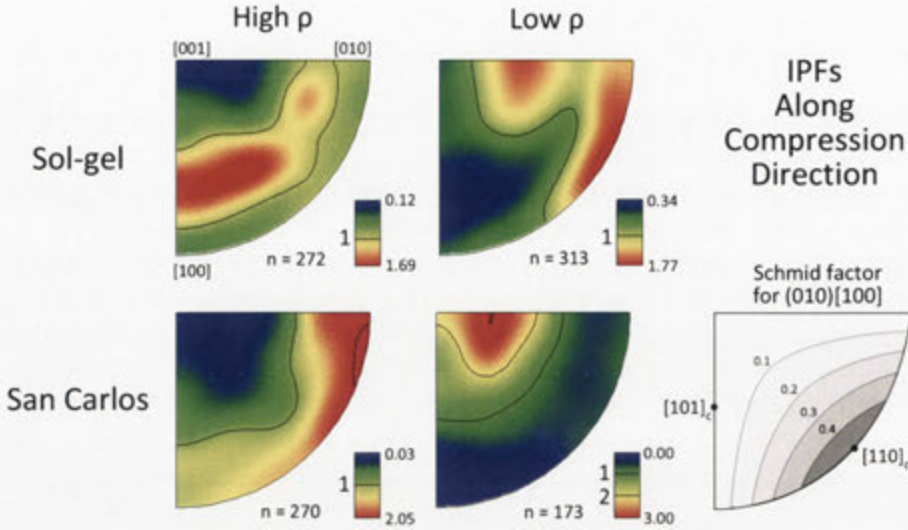


Fig. E.4: Equal area IPFs projected along the compression direction for sol-gel and San Carlos olivine of both dislocation density subsets. An additional inverse pole figure shows the calculated Schmid factor for the (010)[100] slip system in olivine. The half-scatter width is 20° with 5° clustering. There are no high dislocation density grains with a dominant [001] axis, also seen in Fig. E.3

the (010)[100] slip system only.

An interpretation relative to possible active slip systems is given in the discussion.

E.3.3 Inverse pole figures (IPFs)

Inverse pole figures, showing the distribution for the compression direction in the crystal-axis reference frame, are plotted in Fig. E.4 for grains divided into high and low dislocation density subsets. In addition, the insert figure shows contouring of Schmid factor calculated for the (010)[100] slip system in olivine.

San Carlos olivine shows stronger CPO than sol-gel olivine with maximum MUD between 2 and 3 and a more distinct complementarity of low and high dislocation density grains. Few grains of high dislocation density parallel have [001] axes to the compression direction. Such orientations have Schmid factors for (010)[100] slip from 0 to 0.1. The strongest concentration of a compression direction for high dislocation density grains in San Carlos olivine lies between

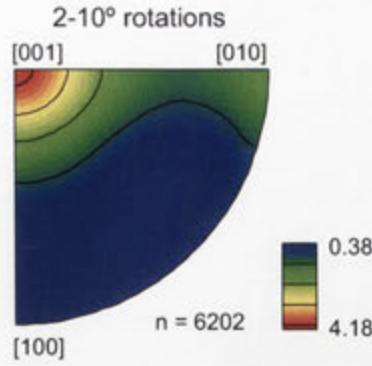


Fig. E.5: An equal area IPF of the rotation axes for misorientations at low-angle ($2 - 10^\circ$) internal boundaries shown in the crystallographic reference frame. Data was obtained from the San Carlos EBSD map. The half-scan width is 20° . Most rotations have occurred near $[001]$.

$[010]$ and $[110]_c$, which corresponds to $(010)[100]$ Schmid factor values of 0.3. However, high dislocation density grains are also present with the compression direction parallel to $[010]$, so low Schmid factors.

Sol-gel olivine shows a different pattern, albeit weaker, with few compression axes in high dislocation density grains near high Schmid factor values at $[110]_c$; actually compression axes in low-dislocation density grains broadly cluster near $[110]_c$.

In addition, axes from grain interiors for low misorientation ($2-10^\circ$) boundaries from the EBSD map of San Carlos olivine are shown in Fig. E.5. The figure shows misorientation-axis geometries are dominantly near the $[001]$ axis and some near $[010]$.

E.4 Discussion

E.4.1 CPO development during tri-axial compression

Compression experiments, in this study, show that overall CPO development is very weak for low to moderate strain (Fig. E.2) for both sol-gel and San Carlos olivine. The patterns should be compared to theoretical low to high strain

deformation models of olivine for up to 2 or 3 slip systems (Wenk et al., 1991; Ribe and Yu, 1991; Tommasi et al., 2000) and previous uni-axial, high strain (58%), deformation experiments on a synthetic dunite (Nicolas et al., 1973).

In this study, for low strains, we show that overall preferred orientation is near random as demonstrated by the low maximum MUD vales in Fig. E.2.

E.4.2 CPO in dislocation density subsets

By separating high from low dislocation density grains we solely attempt to compare Schmid factor with dislocation density for the grain population in both olivine samples. The grain orientations of both groups of high and low dislocation density grains show distinct CPO patterns and quite noticeably some complementarity of the CPO between the two groups. This is most pronounced in San Carlos for the [001] direction (Fig. E.3). Such an observation can be used to infer that grains in favourable orientations deform via one or two 'easy' slip systems and develop high dislocation densities. On the other hand, low dislocation density grains in unfavourable orientations for slip will presumably deform little.

If this inference is true, then a signature of (010)[100] slip (the easiest system in olivine) should be observable in the CPO for grains of high dislocation density. As the schematic pole figures in Fig. E.3 demonstrate, for high (010)[100] Schmid factor, the CPO should be characterised by two girdles for [100] and [010] at 45° to the compression direction; such girdles possibly exist for sol-gel olivine but not for San Carlos olivine. A girdle normal to the compression direction should occur for [001]. A pronounced feature of this type is seen for San Carlos olivine, a weaker equivalent for sol-gel olivine. Complementary patterns should be observed for low dislocation density given that the overall CPO is essentially random (see Fig. E.2). San Carlos samples with low dislocation density show a [100] concentration parallel to the compression direction but this is not clear for sol-gel, low dislocation density grains. Sol-gel olivine shows low dislocation density maxima

of $[010]$ parallel to the compression direction. Overall the patterns are suggestive of $(010)[100]$ slip but there are clear discrepancies.

A comparison of the data can be made with slip systems other than $(010)[100]$. Concerning pole figures of high dislocation density grains only, the slip system $(001)[100]$ should show 45° girdles for $[100]$ and $[001]$ and $[010]$ perpendicular to the compression direction. In an IPF for the compression direction, concentrations for high dislocation density grains are expected to lie near $[101]_c$ for $(001)[100]$ maximum Schmid factor. Alternatively, for the slip system $(010)[001]$ pole figures of high dislocation density grains should show 45° girdles for $[010]$ and $[001]$ with $[100]$ perpendicular to the compression direction; IPF for the compression direction should show a concentration for high dislocation density grains at $[011]_c$. Neither of these two patterns are obvious in Fig. E.3 for high dislocation density grains. However, IPFs for compression direction in high dislocation density grains (Fig. E.4) do show a clustering towards $[101]_c$, especially in the sol-gel olivine case, suggestive that $(001)[100]$ was active.

Misorientation axis data ($2 - 10^\circ$ rotations) are plotted in Fig. E.5 for all San Carlos grains from the EBSD map. Most low-angle boundaries have misorientation axes dominantly near $[001]$ but also some near $[010]$. Such rotations indicate grain interior deformation occurred via the activation of multiple slip systems. While $[001]$ rotations predominantly suggest $(010)[100]$ was activated, evidence for $[010]$ rotations suggest $(001)[100]$ was also active in several grains analysed via EBSD mapping. However, TEM observations have found both $[100]$ and $[001]$ Burgers vectors in sol-gel olivine (Faul et al., 2011). In terms of dislocation substructures, a twist boundary of dislocations with $[100]$ and $[001]$ Burgers vectors would satisfy a twist $[010]$ twist axis. For a tilt boundary, rotation around $[001]$ would satisfy dislocations with $[100]$ and $[010]$ Burgers vectors. However, the latter corresponding to the greatest unit cell distance in olivine is rarely seen.

In addition, when data in Fig. E.5 are broken up into three subsets of grains of only high Schmid factor (≥ 0.4) for the three most quoted slip systems in

olivine; (010)[100], (001)[100] and (010)[001] (Wenk et al., 1991; Tommasi et al., 2000; Castelnau et al., 2008), misorientation-axis geometries (not shown) remain dominant near the [001] axis for all three subsets. This suggests that a model for uniform stress (Sachs) cannot be valid as olivine grains appear to deform on a slip system which would otherwise be inactive given the external compressive stress. This could also explain the discrepancies between Schmid factor and dislocation density in Fig. E.3 and Fig. E.4.

Hence, we conclude that the dominant slip system has been (010)[100] in deformation of our olivine specimens even though the patterns in the pole figures are not perfect. Similar Schmid factor – dislocation density observations have previously been made for olivine deformed in shear (Karato and Lee, 1999; Lee et al., 2002). In addition to multiple operating slip systems, other factors may also have played a role such as grain boundary sliding in our fine grained samples and it may be possible that slip in high Schmid factor grains may have been suppressed by surrounding grains which adds to a case for heterogeneous stress. Lastly, the cases of weak complementarity between the two dislocation density subsets may reflect the choice to exclude grains of moderate dislocation density from Fig. E.3 and E.4.

E.4.3 Comparison with previous studies

The dislocation density distribution and sub-structures that evolve during deformation of our fine-grained olivine appear to be different than in olivine aggregates undergoing dynamic recrystallisation under high-strain simple shear (Karato and Lee, 1999; Lee et al., 2002). Commonly, a heterogeneous distribution of dislocation density is observed in a low-OH deformed olivine polycrystals. This may result from varying intra-granular stresses between grains, where grains which are favourably oriented are more likely to deform. However, from high-strain deformation experiments, such grains are less likely to dominate the final preferred orientations caused by grain boundary migration (Zhang et al., 2000).

In this study, some evidence for grain boundary bulging from low to high dislocation density grains is observed (see Fig. E.1) but not at the expense of a significant increase in grain boundary area or an increase in low dislocation density grains. Grain shapes are not typical of high temperature stress-induced grain boundary migration (SIGBM) and vary between equigranular polygonal or mild interlobate structures (Passchier and Trouw, 1998).

It is not well known to what degree the microstructural state of stress and strain plays a role between grains (Taylor, 1938; Karato and Lee, 1999). However, recently a Second-Order estimate model by Castelnau et al. (2008) has managed to approximate the stress heterogeneities on the macroscopic flow stress in polycrystalline olivine. This model essentially falls halfway between the Taylor (uniform strain) and Sachs (uniform stress) end member models. The Second-Order model highlights an important fact, which is the localisation of stress and strain rate in an olivine polycrystal and that they often have varying higher and lower values than the imposed macroscopic values. All the grains in the model are allowed 5 degrees of freedom for the three known slip systems in olivine and two estimated ones. While the details of the chosen parameters are debatable, the effects of strain localisation, fluctuations and stress gradients in and between grains are important to consider when trying understand microstructural change in an olivine aggregate.

The results from this study have several implications for seismic attenuation measurements in the upper mantle. Models have shown that forced vibrations on pinned dislocation lines can dissipate seismic strain energy (Granato and Lucke, 1956; Lucke and Granato, 1981). The results from this study suggest that any CPO inferred from seismic anisotropy in olivine is directly linked to the presence of high dislocation density grains. It is likely that dislocations, with a dominant [100] Burgers vector, will cause anisotropy in seismic attenuation measurements.

E.5 Conclusion

This study for weakly strained ($\sim 15\text{-}19\%$) synthetic olivine aggregates shows a near-random CPO, supported by low M-index values. Despite this, a heterogeneous distribution of grains with high and low dislocation density was observed. An overall near-random CPO facilitated identification of operating slip systems in olivine as a complementarity is expected between grain populations of high and low dislocation density.

Patterns of preferred orientation from point-indexed grains of high and low dislocation density and misorientation axis data from an EBSD map demonstrated some correlation between Schmid factor and dislocation density. The data suggest that grains of high dislocation density are more favourably oriented to deform via the dominant slip system $(010)[100]$. The correlation is not perfect, however, as seen in Fig. E.4 by a higher concentration of high dislocation density grains for the compressive axis near $[010]$ rather than $[110]_c$ in San Carlos olivine and a distribution towards $[101]_c$ for sol-gel olivine. This is more suggestive for a correlation between high dislocation density and high Schmid factors for $(001)[100]$ slip. A complementarity between the subsets in Fig. E.3 would not appear at all if multiple slip systems operated evenly to the same degree.

Furthermore, the CPO data described above re-affirm that neither the Sachs model of uniform stress nor the Taylor model of uniform strain are adequate to describe plastic deformation and textural evolution of olivine. Most likely plastic deformation is more comparable to a Second-Order micromechanical model used by Castelnau et al. (2008). Heterogeneous stress provides an explanation for grains in unfavourable orientations to the external compressive stress (i.e. with low Schmid factor) to deform. Variation in dislocation density between grains indicates that strain is heterogeneous.

Lastly, if the $[100]$ Burgers vector dominates the dislocations in high-dislocation-density olivine grains, deforming via $(010)[100]$ slip, this could lead to anisotropy

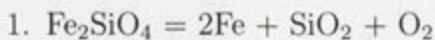
in seismic attenuation measurements.

F

Calculating the Ni₇₀Fe₃₀-opx-olivine buffer

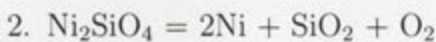
The Ni₇₀-Fe₃₀ foil wrappers for hot-pressing, deformation and attenuation experiments provided an oxygen fugacity buffer for olivine at high temperature.

In order to recreate the same conditions in a 1 atmosphere controlled environment furnace for dislocation recovery experiments, a corresponding CO/CO₂ gas mix buffer had to be determined from olivine-orthopyroxene-metal equilibria, presented first. The relevant reactions are



$$\Delta G_1^0(T,1) = 564800 - 144.4T$$

$$\Delta V_{s1}^0 = -0.942$$



$$\Delta G_2^0(T,1) = 485700 - 179.2T$$

$$\Delta V_{s2}^0 = -0.671$$



$$\Delta G_3^0(T, 1) = -6710 - 0.31T$$

$$\Delta V_{s3}^0 = -0.362$$

with units: temperature T (K), pressure P (bar), volume change for solid phases ΔV_s^0 ($\text{J bar}^{-1} \text{ mol}^{-1}$) and ΔG^0 (J mol^{-1}). The respective free energies for the forward reactions are from Rubie et al. (1993) with updated molar volume differences from Smyth and McCormick (1995).

F.1 Fe/Ni partitioning between olivine and metal phases

The exchange reaction between Fe and Ni can be obtained by subtracting (2) from (1) to give



$$\Delta G_4^0(T, 1) = \Delta G_1^0(T, 1) - \Delta G_2^0(T, 1)$$

$$\Delta V_{s4}^0 = \Delta V_{s1}^0 - \Delta V_{s2}^0$$

Equilibrium at T and P requires that

$$\Delta G_4^0(T, P) = 0 = \Delta G_4^0(T, 1) + P\Delta V_{s4}^0 + RT \ln [a_{\text{Fe}, \text{me}}/a_{\text{Ni}, \text{me}}]^2 \cdot [a_{\text{Ns}, \text{ol}}/a_{\text{Fa}, \text{ol}}]\}$$

so that

$$a_{\text{Ns}, \text{ol}}/a_{\text{Fa}, \text{ol}} = [a_{\text{Ni}, \text{me}}/a_{\text{Fe}, \text{me}}]^2 \exp \left[-\frac{\Delta G_4^0(T, 1) + P\Delta V_{s4}^0}{RT} \right] \quad (\text{A-1})$$

where 'Ns' represents the Ni_2SiO_4 component of olivine.

This expression demonstrates a unique olivine composition in equilibrium with the Ni-Fe alloy of given composition. The activities a are related to the mole

fraction X and activity coefficient γ by expressions in the form

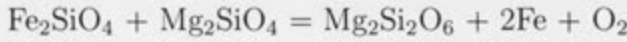
$$a_{Fe,me} = \gamma_{Fe,me} X_{Fe,me}$$

$$a_{Fa,ol} = \gamma_{Fa,ol} X_{Fa,ol}$$

The activity coefficients for olivine and metal solid solutions are given by Rubie et al. (1993) and Rammensee and Fraser (1981), respectively. For the simple case of an ideal solid solution, all activity coefficients are equal to 1.

F.2 Oxygen fugacity for olivine-opx-metal equilibrium

Oxygen fugacity is also fixed for an equilibrium between coexisting olivine, orthopyroxene and metal. Again, the relevant reaction is obtained by adding reactions (1) [or (2)] and (3)



$$\Delta G_6^0(T, 1) = \Delta G_1^0(T, 1) + \Delta G_3^0(T, 1)$$

$$\Delta V_{s6}^0 = \Delta V_{s1}^0 - \Delta V_{s3}^0$$

and equilibrium requires

$$\Delta G_6^0(T, P) = 0 = \Delta G_6^0(T, 1) + P\Delta V_{s6}^0 + RT \ln \left[f_{\text{O}_2} [a_{Fe,me}]^2 \cdot \frac{a_{En,me}}{a_{Fo,ol} \cdot a_{Fa,ol}} \right]$$

So that

$$\ln f_{\text{O}_2} = - \left[\frac{\Delta G_6^0(T, 1) + P\Delta V_{s6}^0}{RT} \right] - 2 \ln \left[\frac{(a_{Fe,me})^2 \cdot a_{En,opx}}{a_{Fo,ol} \cdot a_{Fa,ol}} \right] \quad (\text{A-1})$$

The relevant activity coefficients γ are as follows

$$\begin{aligned}\ln \gamma_{Fa,ol} &= (1 - X_{Fa,ol})^2(600 + 0.0013P)/T \\ \ln \gamma_{Fe,me} &= (1 - X_{Fe,me})^2[-3.33 + 0.22(4X_{Fe,me}) - 1]\end{aligned}$$

from various sources shown in Rubie et al. (1993, Table 2). They are multiplied to the mole fractions of each species to give the respective activities. The activity $a_{En,opx} \approx X_{En,opx}$ and $a_{Fo,ol} \approx X_{Fo,ol}$. The activities of nickel in olivine and metal are controlled by the partitioning and the activities of iron in olivine and metal and thus require no extra consideration. The representative results are shown in Fig. F.1.

F.3 Implications for annealing experiments

The oxygen fugacity for the olivine-opx-metal equilibrium can now be reproduced in a controlled atmosphere furnace at room pressure using CO and CO₂ gas mixes as an oxygen buffer. The relevant reaction for this gas mixture is



$$\Delta G_8^0(T, 1) = -282420 + 86.82T$$

(Kubachewski and Evans, 1965). For equilibrium we have

$$\Delta G(T, P) = 0 = \Delta G^0(T, 1) + RT \ln \left[\frac{f_{\text{CO}_2}}{f_{\text{CO}} \cdot f_{\text{O}_2}^{1/2}} \right]$$

Thus the gas mixture required to yield a specified f_{O_2} is given by

$$\frac{f_{\text{CO}_2}}{f_{\text{CO}}} = f_{\text{O}_2} \exp \left[-\frac{\Delta G_8^0(T, 1)}{RT} \right]$$

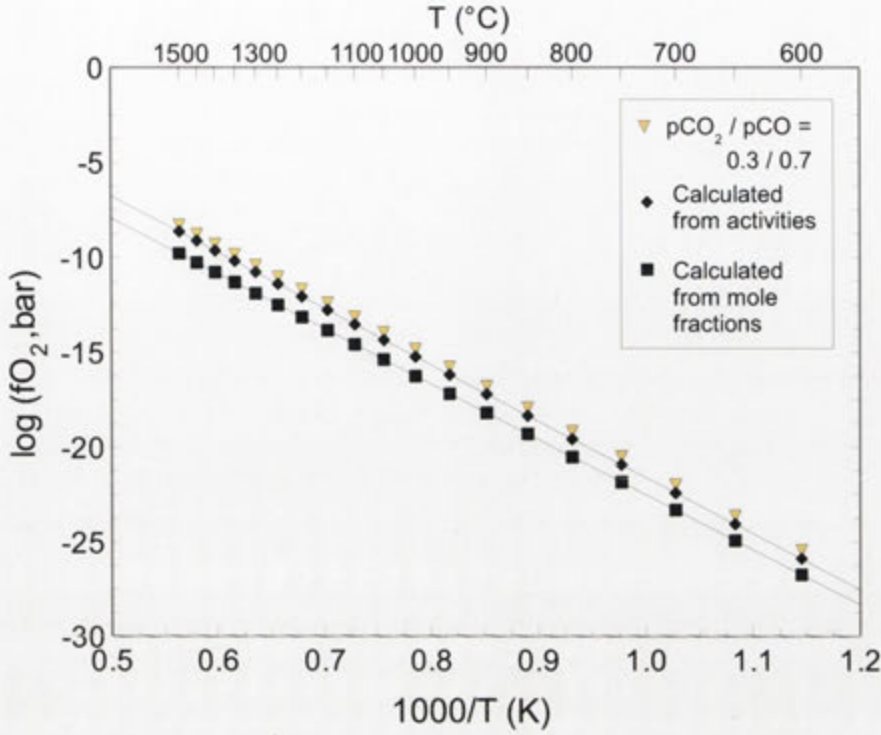


Fig. F.1: Oxygen fugacity buffers calculated from the mole fractions and activities for an equilibrium of coexisting phases of olivine, orthopyroxene and Ni-Fe metal. In addition, the range of oxygen fugacity with reciprocal temperature is plotted for a gas mix of 30% CO₂ and 70% CO, used for the dislocation recovery experiments (see Chapter 3).

For an ideal gas mixture (likely to be a good approximation), the activity coefficients are set to 1 to give

$$\frac{p_{\text{CO}_2}}{p_{\text{CO}}} = f_{\text{O}_2}^{1/2} \exp \left[-\frac{\Delta G_s^0(T, 1)}{RT} \right] \quad (\text{A-1})$$

Hence it was determined that a gas mix of 30% CO₂ and 70% CO approximately reproduces the olivine-opx-metal buffer for a Ni₇₀-Fe₃₀ foil in contact with olivine over a range of temperatures (room temperature to 1500°C) (see Fig. F.1).



The data points in Figure 1 show a linear relationship between $\log t$ and $1/T$. The slope of the line is approximately -1.5×10^4 K. This indicates that the annealing process follows an Arrhenius law, where the rate of annealing is proportional to $\exp(-U/kT)$, with U being the activation energy. The intercept of the line on the $\log t$ axis is approximately 0.5, which corresponds to a time of about 3.2 seconds at the reference temperature of 1000 K.

Figure 1 shows the relationship between the logarithm of the annealing time ($\log t$) and the reciprocal of the annealing temperature ($1/T$). The data points are plotted for various temperatures and times, showing a linear trend.

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Estimating the depth of information for BEI used in calculating the 3-D dislocation density

The electron penetration depth and depth of information, d was approximated using Monte Carlo (MC) electron interaction simulations. The programme that was used is called CASINO or monte CARlo SIMulation of electroN trajectory in sOlids*. Its operation is summarised by Drouin et al. (2007).

G.1 Introduction

The electron penetration depth value depends on three variables; the sample density, accelerating voltage and sample composition. These are related to each other through the equation derived by Kanaya and Okayama (1972), as restated by Goldstein et al. (1984).

$$R_{KO} = 0.0276AE_0^{1.67}/(Z^{0.889}\rho) \quad (\text{A-1})$$

*<http://www.gel.usherbrooke.ca/casino/>

where R_{KO} is the electron penetration depth in μm , ρ is the target density in g/cm^3 , E_0 is the electron accelerating voltage in keV, Z is the average atomic number of the target and A , the average atomic mass is in g/mol . The calculated depth of information, d (see Eqn. 3.3.1) is approximately 0.3 of the R_{KO} range (Goldstein et al., 1984, pp. 72-89).

The depth of information relates to the interaction volume for backscattered electrons (BSEs) which return to the sample surface through elastic collisions, then escape to land on the BSE detector. However, there much uncertainty in estimating the maximum depth from which the majority of BSEs returned. Not all electrons return back to the detector, especially electrons escaping at high angles. Also BSE electrons which interact with the iron oxide precipitates at dislocations might need to be described by a different d .

The problem was addressed experimentally by Karato and Lee (1999) by taking a single crystal olivine in the $[101]_c$ orientation and deforming it so that the $(001)[100]$ and the $(100)[001]$ slip systems were both activated. When the crystal was cut along the (010) plane, the dislocation lines made an angle of 45° to this surface and the 'effective thickness,' or depth of information was calculated from the apparent length of lines in the SEM images on the assumption that all dislocations were straight and parallel to one of the shear directions. The authors obtained a result of $(0.9 \pm 0.1) \times 10^{-6}$ m for 15 keV backscattered electrons which was later refined to a lower value of $\sim 0.6 \times 10^{-6}$ m (personal communication, Prof. Shun Karato, 2009). Eqn. A-1 predicts a maximum electron penetration depth of 1.99×10^{-6} m for Fo_{90} at 15 kV (with $3.3 \text{ g}/\text{cm}^3$ specimen density). The Monte Carlo approach predicts a maximum depth of information for BSEs of about $d \approx 0.62 \times 10^{-6}$ m, which is not too far off the measured value after applying the 0.3 factor predicted by Goldstein et al. (1984).

In this study, a lower accelerating voltage of 5 kV was used to limit the imaging of dislocation lines to a thinner surface layer (Fig. G.1). The geometry of dislocations is less discernible but measuring the length and number of disloca-

tions was more reliable because dislocation overlap is lessened. Unfortunately, in this study we do not have a 'calibration' specimen of the type employed by Karato and Lee (1999) to calibrate a depth value for the backscattered-electron imaging.

G.2 Approach

Using the CASINO software, olivine ($\text{Fe}_{0.1}\text{Mg}_{0.9}\text{SiO}_4$) was represented by a layer of 'custom' density of 3.33 gcm^{-3} . The layer was set at $2 \times 10^6 \text{ nm}$ thick which was the average thickness of each olivine block. The simulations were run at 5 kV and 15 kV simulating trajectories for up to 100,000 electrons. The tilt was 0° and the beam radius was estimated at 1.5 nm for a field emission SEM. The following default physical models were employed. The total and partial cross sections were estimated via Mott by interpolation. The effective section ionisation was calculated according to 'Casnati'. The Ionisation potential was calculated according to Joy and Luo (1989). A random number generator was run using the method described by Press et al. (1986). The directing cosin and dE/dS calculations were performed using equations developed by Soum et al. (1979) and Joy and Luo (1989). The distribution settings for electrons were left at their respective default values. The simulation that was run at 5 kV, presented a maximum depth for back-scattered electrons (in red) in addition to a maximum electron penetration depth of lost electrons (in blue, as shown in Fig. G.1). The depth of information d was determined from a histogram of the Z_{max} distance of back-scattered electrons in the olivine layer for a 95% confidence interval, corresponding roughly to energy levels down to 40% in the interaction volume.

G.3 Results

Using Eqn. A-1 at 5 kV, $R_{KO} = 0.32 \text{ }\mu\text{m}$. This is supported by running a Monte Carlo simulation (Fig. G.1a) which also predicts a depth of information for BSEs

of $d \approx 0.095 \mu\text{m}$, with the factor from Goldstein et al. (1984) of $d \sim 0.3R_{KO}$.

Fig. G.1 illustrates clearly that the depth sampled by BSEs is very dependent on the accelerating voltage. The depth of information from the Monte Carlo simulations, for both incident energies, can be combined with the measured projected lengths, l to calculate 3-D dislocation densities. These can then be compared for consistency at different accelerating voltages. The dislocation density of the FESEM images in the inset of Fig. G.1 was obtained, not via the usual process of filtering, but via hand-tracing each dislocation line to avoid any artefacts that result from applying a series of contrast filters. The first large grain measured at both 5 and 15 kV indicates a ratio for l_0 to be 1:2 for a consistent dislocation density. The next grain measured a ratio of 1:1.7 for l_0 and a third gave a ratio of 1:1.9 for l_0 . A higher ratio of l_0 at 5 to 15 kV implies an equivalent higher ratio of d between 5 and 15 kV. The measurements differ significantly from both the ratio from the Monte Carlo simulations ($95/600 = 1/6$) and the prediction from Eqn. A-1 $(5/15)^{1.67} = \sim 0.16$.

G.4 Discussion and conclusion

There are several complications that may explain the above observations. Firstly, the atomic number of the decorated dislocation lines (iron oxides) is far higher than the enclosing olivine which reduces the electron penetration depth. To simulate this accurately, one would need complex electron interaction models like those of Hennig and Denk (2007). Secondly, there are also considerations related to the acceptance angle of the BSE detector and the escape-angle distribution of BSEs (Murata, 1976; Goldstein et al., 1984). BSEs notably emerge from the surface of the specimen away from the primary electron source area due to changes in travel directions from elastic collisions in the material. The further out, the less energy the BSE generally has. Therefore, it is preferable for highest image resolution to select only the high energy BSEs. In addition, the escape probability of a BSE decreases gradually with penetration depth. There is no cut off value.

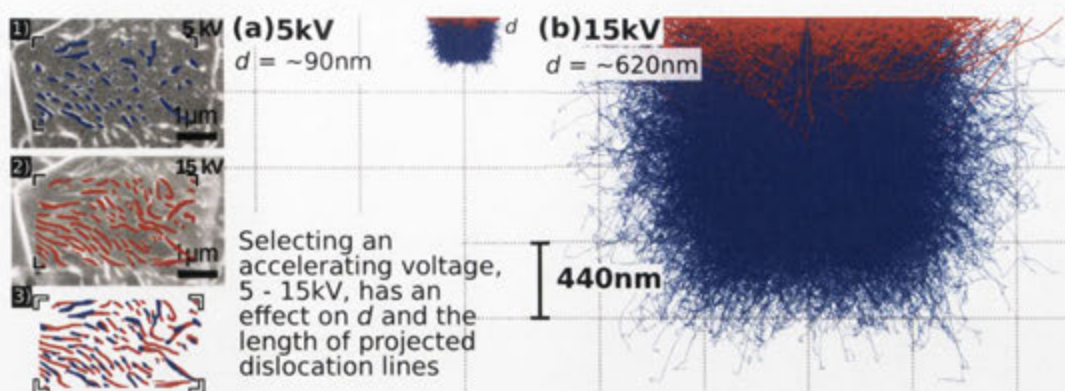


Fig. G.1: Monte Carlo electron interaction simulations at (a) 5 kV and (b) 15 kV accelerating voltage for a solid with composition and density of Fe_{90} . The red lines represent the backscattered electron paths. The value for depth of information at 95% confidence interval is shown in bold on the vertical scale in each case. The blue lines indicate the travel paths for the primary electrons that change direction due to elastic scattering events. Both simulations are shown at the same scale for comparison. The inset shows a comparison between 5 kV and 15 kV accelerating voltage with maximum contrast for the same region. Note that the dislocations in (2) are longer and that some sets of dislocations can be found which are not present in (1). (3) shows the comparison of hand traced dislocations in the area highlighted by the dislocation lines in (1) and (2)

This means that a single value cannot accurately define the depth of information (Goldstein et al., 1984). One must also take into account that the ratios of l_0 , calculated from 5 and 15 kV BSE images, are a lower estimate because at higher kV the dislocations overlap significantly which makes it very difficult to draw the entire length of each dislocation.

Lastly, the measured value of l appears to have a small effect on the calculated recovery rate and activation energy. For example, at 1450°C with $d = 0.095 \mu\text{m}$, $k = 9.9 \times 10^{-18} \text{ m}^2\text{s}^{-1}$. With $d = 0.3 \mu\text{m}$, $k = 3.4 \times 10^{-17} \text{ m}^2\text{s}^{-1}$. This effect is related to the fraction of short dislocations for which l approaches d . When there is a large fraction of long dislocations, the dependence on d is greater (i.e. shorter d produces relatively higher ρ_f and therefore lower k). For dislocations approaching $1 \mu\text{m}$ in length, d can be effectively ignored. The way k is correlated with d and the length distribution of dislocations imaged in each experiment points to the high uncertainty in E_a . Despite all this, however, using a depth

of information, $d = 0.095 \mu\text{m}$, is still a good approximation for obtaining an accurate estimate of the 3-D dislocation density.

H

Tables

H.1 Hot isostatic pressing experiments

Before hot isostatic pressing, the sol-gel specimens were typically reacted at 875°C in a controlled atmosphere furnace (95%CO₂ / 5%CO gas mix) and both sol-gel and San Carlos specimens were fired twice (with 50%CO₂ / 50% CO gas mix) at 1400°C for 16 hours to remove all adsorbed moisture. Table H.1 lists the run conditions and dimensions of all the hot-pressed specimens (sol-gel and San Carlos). Successful runs permitted the specimens to be reused for subsequent experiments (deformation/attenuation). The samples were typically re-fired after deformation in preparation for attenuation experiments, as described in detail in Chapter 2. Run H6668 was the hot-pressing of the Pakistani single crystal (see Appendix A).

H.2 Specimen assemblies for attenuation experiments

Table H.2 lists the various corrections that result from subtracting the specimen assembly from the ‘double foil’ reference assembly (A1117). These were used in processing the compliance and phase angle data to shear modulus and attenuation

Table H.1: Hot-pressed olivine runs

Run number	Run conditions T (°C), P (MPa), t (hour)	Stack length (mm)	Ground dimensions l × d, (mm)
sol-gel			
H6529	1300, 300, 24	47	24.9
H6585	1200, 300, 23	?	?
H6602 (failed)	1300, 300, 24	44.55	32.8×11.73
H6605 (failed)	1300, 300, 24	41	n/a
H6607	1300, 300, 24	42.44	30.92×11.63
H6611	1300, 300, 24	44	33.3×11.59
H6612 (unused)	1300, 300, 24	?	32.89×11.56
H6667	1300, 300, 24	37	33.3×10.39
H6668 (s. xl)	1200, 300, 24	n/a	n/a
San Carlos			
H6688	1300, 300, 24	45.38	35.80×11.5
H6691	1300, 300, 24	43.82	33.0×10.0
H6694	1300, 300, 24	39.7	32.70×11.5
H6695	1300, 300, 24	41.29	31.44×10.12

as a function of oscillation period. These corrections include differences between:

1. length of the respective assemblies (l_{ass}),
2. the vertical positions of the two assemblies relative to the pressure vessel (z'),
3. the length of the waisted sections (l_w),
4. the excess length of the solid alumina discs above a nominal value of 6.0 mm for the specimen assembly (l_{dsk}),
5. the thickness of the Ni₇₀-Fe₃₀ foil discs (l_f).

Additionally, the following information was given: 1. length of the olivine specimen (l_r), 2. diameter of the olivine specimen (d_r) and finally 3. the final jacketed diameter of the specimen (d_{rj}). Other required information including the properties of the elastic element, and the viscoelastic behaviour of the steel jacket and LucaloxTM alumina in addition to a complete description of the assembly geometry remained the same for each experiment.

Table H.2: Specifications of sol-gel (SG) and San Carlos (SC) olivine specimens for attenuation experiments

Run			l_{ass}	z'	l_w	l_{dsk}	l_f	l_r	d_r	d_{rj}
H	D	A	mm	mm	mm	mm	mm	mm	mm	mm
Reference		A1117	338.21	24.30	35.85	5.87	0.14		11.50	12.28
H6585(SG)		A1127	338.23	25.00	35.87	5.87	0.14	30.00	11.50	12.28
H6607(SG)	D6618	A1156	335.83	22.70	33.55	5.31	0.14	28.10	11.50	12.28
H6611(SG)	D6646	A1161	336.02	23.80	33.76	8.13	0.14	25.49	11.50	12.28
H6667(SG)	T0436	A1231 [†]	342.05	28.60	39.00	9.30	0.14	29.56	10.00	11.39
H6694(SC)	D6701	A1232 ^{*‡}	336.46	23.00	33.41	9.30	0.07	23.97	9.98	10.90
H6691(SC)	T0434	A1271 [†]	342.96	29.20	39.99	9.30	0.14	30.55	10.00	11.31
H6695(SC)		A1279 [†]	339.44	26.00	36.44	9.30	0.14	27.00	10.00	11.05

See text for explanation of the column headers
* Single Ni₇₀-Fe₃₀ foil disc
[†] Ni₇₀-Fe₃₀ foil wrapper only matches specimen length, not solid alumina discs
[‡] Extra Ni₇₀-Fe₃₀ foil wrapper only matching specimen length



Microstructural images of sol-gel and San Carlos olivine

Selected backscattered electron images showing areas at 5 kV accelerating voltage for the hot-pressed sol-gel and San Carlos specimens before (a) and after (b) forced-oscillation mechanical testing are presented in Fig. I.1 and Fig. I.2 respectively. Three to four other uncropped areas such as these have been imaged and processed to obtain the mean dislocation density information. Note the low density of decorated dislocations in the hot-pressed specimens.

Pre-deformed and post-attenuation sol-gel and San Carlos specimens are presented in Fig. I.3 and Fig. I.4 showing initially high and sometimes subsequently lower densities of decorated dislocations after annealing at high temperature for long durations and attenuation experiments.

Please note that these images have been significantly downsized from their original resolutions and hence cannot be used to estimate the dislocation density. The original files are available upon request. However, each image is at least 60 MB in size.

In addition, transmitted light microscope images are given in Fig. I.5 of pre-

pared thin sections from the upper end of the hot-pressed San Carlos specimen H6695, from the lower end of this specimen after a failed attenuation experiment A1217 due to furnace malfunction (and melting of the jacket near the specimen) and from the upper end of that specimen after an attenuation experiment A1279. For comparison a photomicrograph of the torsionally pre-deformed San Carlos specimen A1271 after attenuation is also given. It is clear that the two uncontrolled (and unconfined) excursions to temperatures $> 1300^{\circ}\text{C}$ caused significant cracking in the originally hot-pressed San Carlos specimen H6695. The porosity, shown by the distribution of small black spots, after a failed attenuation experiment (A1217) and a successful attenuation experiment (A1279) also appears higher than for San Carlos specimen A1271 (Fig. 1.5d). While the porosity for the entire specimen A1279 is low ($\sim 0.24\%$), as calculated from an olivine density test via immersion in ethanol, the presence of micro-cracks may have contributed to an unusual low shear modulus dispersion during forced-oscillation testing.

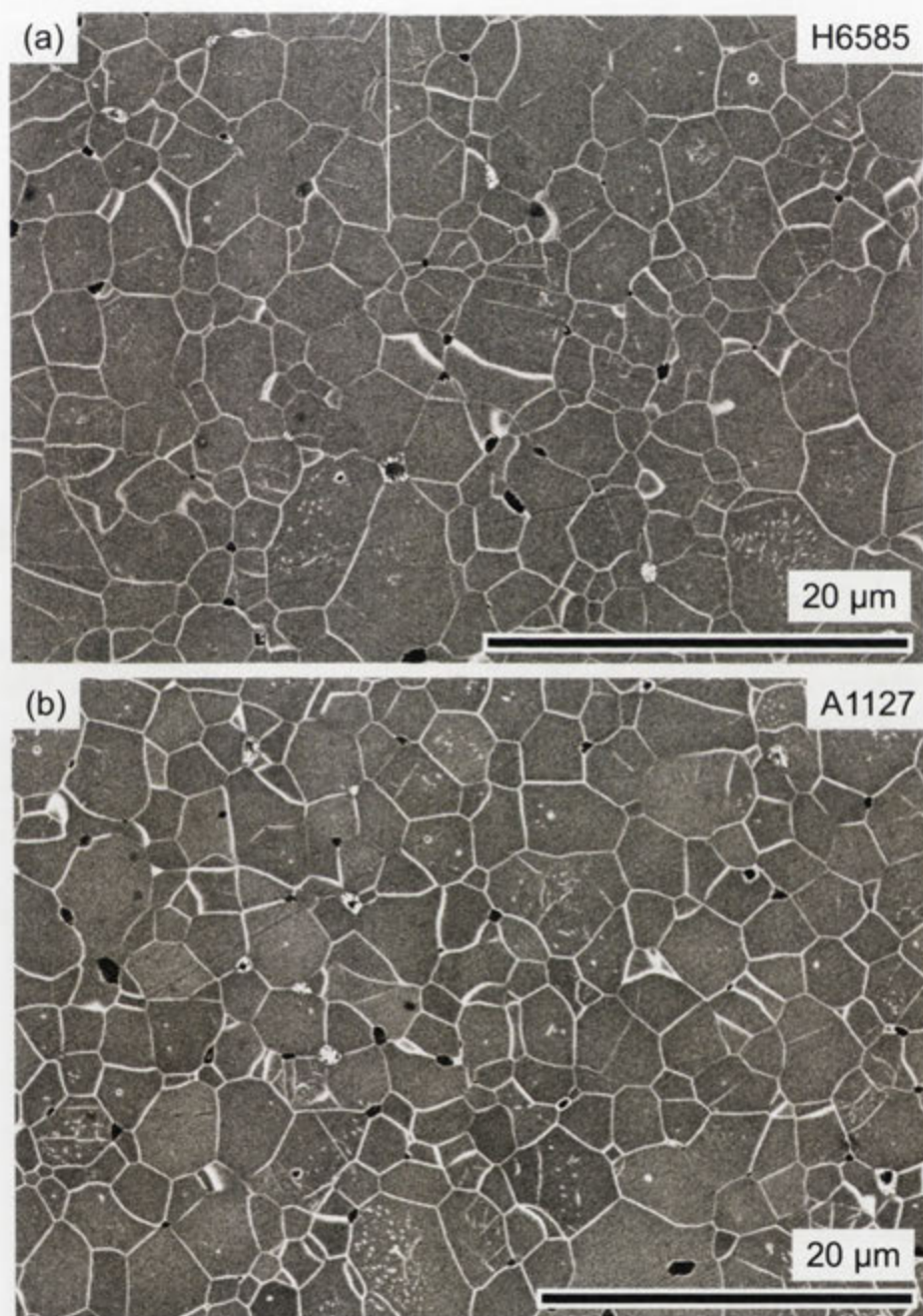


Fig. I.1: Selected and cropped areas imaged at 5 kV using the Hitachi FE-SEM for a hot-pressed sol-gel specimen before (a) and after (b) forced-oscillation mechanical testing.

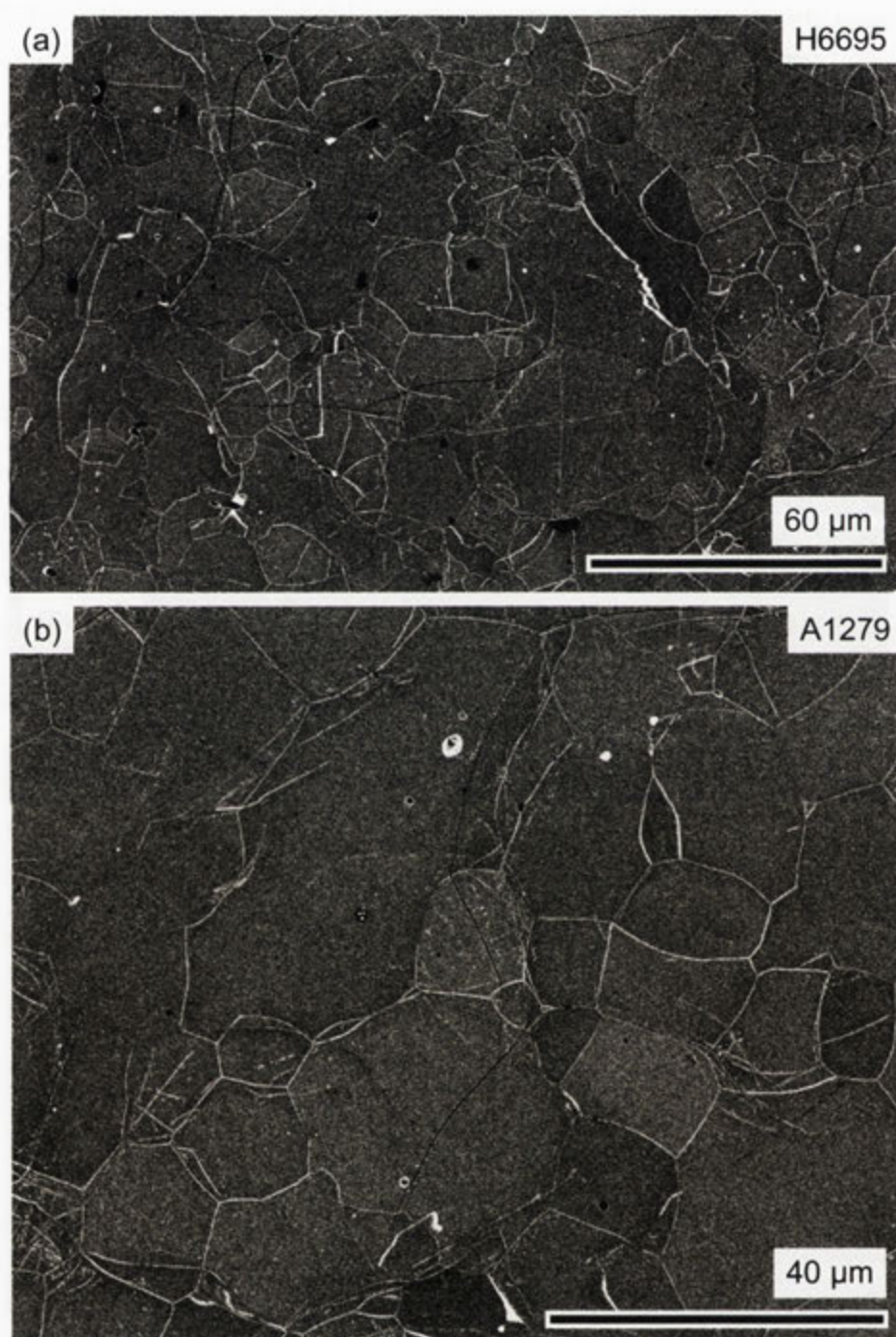


Fig. I.2: Selected and cropped areas imaged at 5 kV using the Hitachi FE-SEM for a hot-pressed San Carlos specimen before (a) and after (b) forced-oscillation mechanical testing.

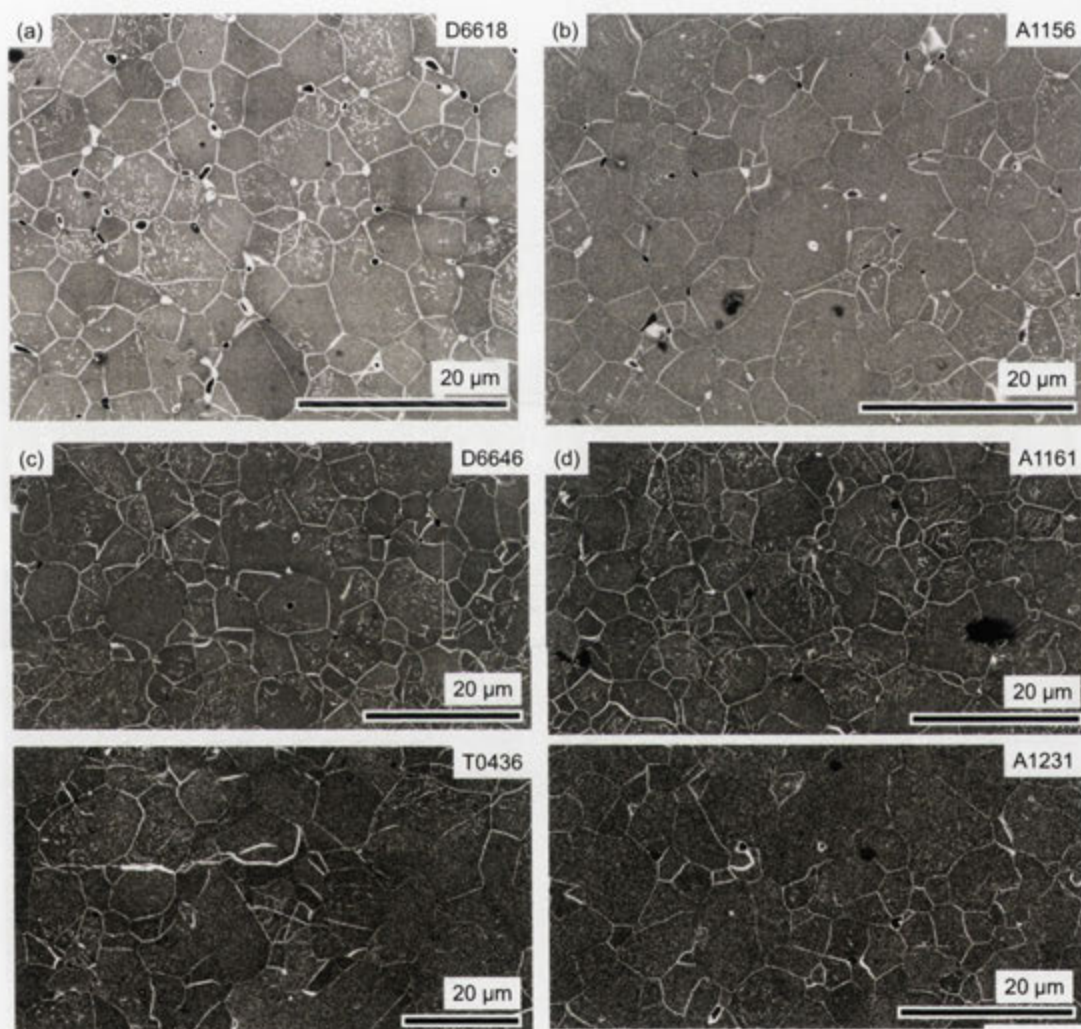


Fig. I.3: Selected and cropped areas imaged at 5 kV using the Hitachi FE-SEM for pre-deformed sol-gel specimens before (a) and after (b) forced-oscillation mechanical testing.

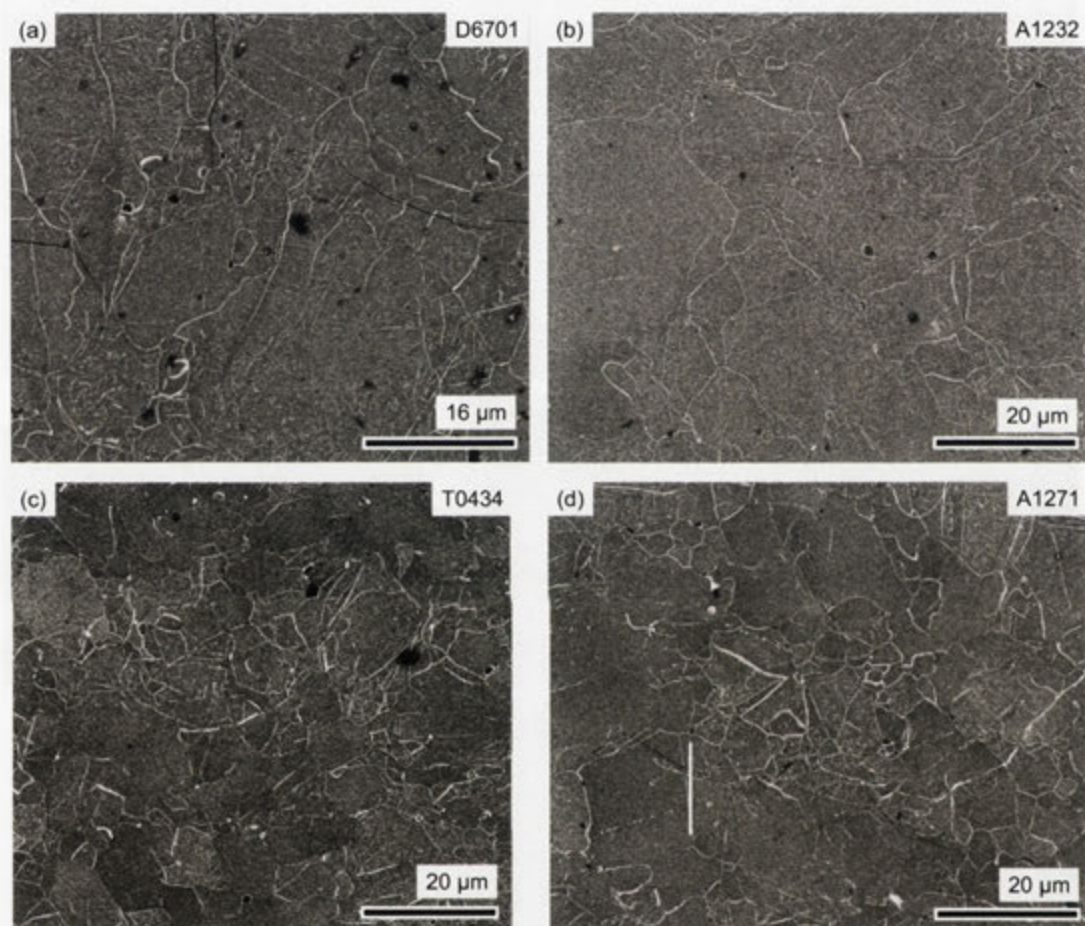


Fig. I.4: Selected and cropped areas imaged at 5 kV using the Hitachi FE-SEM for pre-deformed San Carlos specimens before (a) and after (b) forced-oscillation mechanical testing.

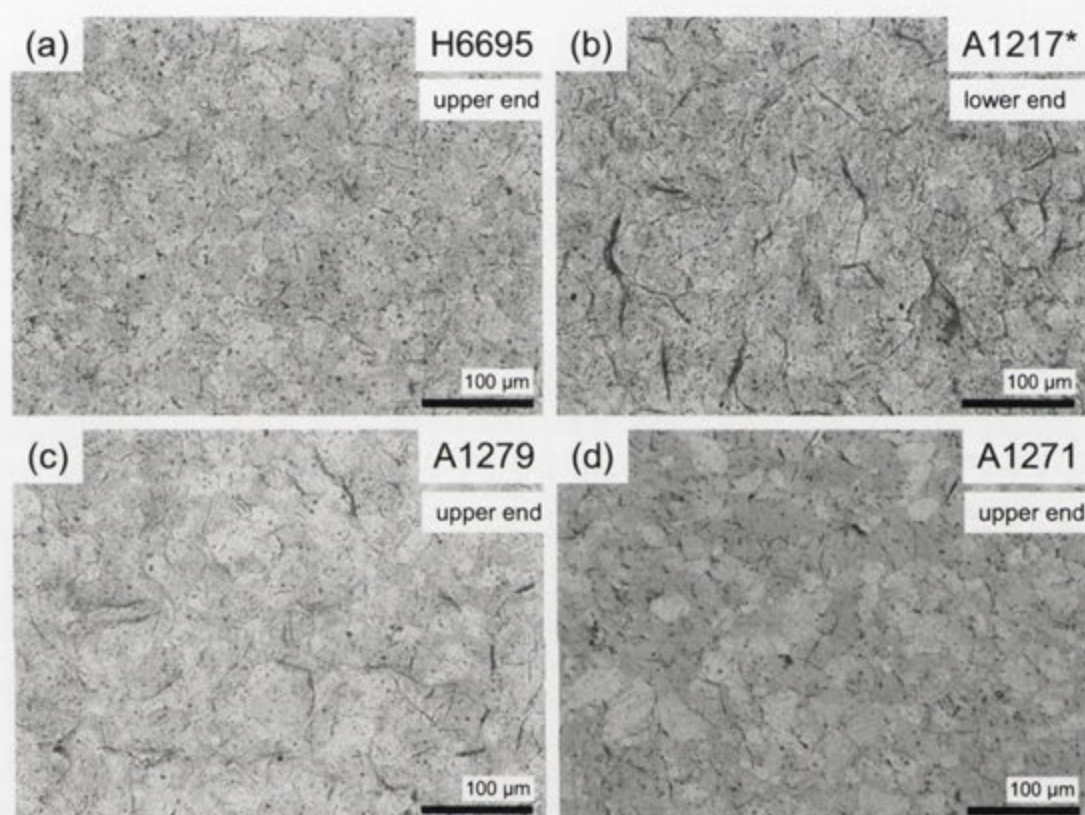


Fig. I.5: Transmitted light photomicrographs showing the development of significant density of micro-cracks after the hot-pressed San Carlos specimen (a) was exposed to temperatures $> 1300^{\circ}\text{C}$ after the first furnace malfunction (b) and after a second furnace malfunction and subsequent attenuation experiment (c). On both occasions, the lower end of the specimen was exposed to extremely high temperatures that melted the mild steel jacket around it. For comparison, in panel (d), a photomicrograph of the torsionally pre-deformed San Carlos specimen after attenuation is given.



FT Infrared absorbance spectra

Infrared absorbance spectra of all specimens (after hot isostatic pressing, pre-deformation and attenuation experiments) are shown in Fig. J.1, J.2 for sol-gel olivine and Fig. J.3 for San Carlos olivine. The spectra are all normalised to 1 cm for sample thickness. The average OH content was determined for a peak broadband absorbance around 3400 cm^{-1} where visible in each spectrum (see Chapter 4, section 2.7). The degree of noise in the spectra resulted from infrared light scattering off grain boundaries and pores. This appears to be significant in the mildly compressively pre-deformed sol-gel specimen D6618, A1256. Other specimens showing noisy spectra are the hot-pressed San Carlos (H6695 – A1279), and the compressively pre-deformed San Carlos specimen (H6694 – D6701).

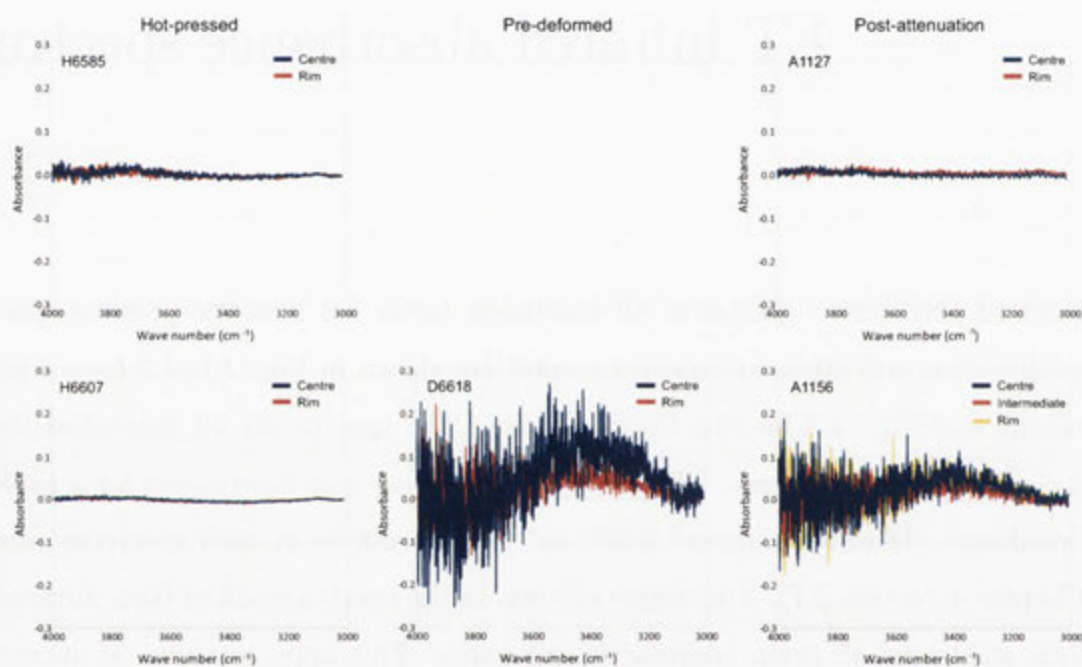


Fig. J.1: First set of absorbance spectra of sol-gel olivine specimens during the various stages of experimentation. Two bulk measurements were taken, one at the centre and one at the rim of the cylindrical specimen. The IR beam had a nominal width of 1.5 mm. The spectra are normalised to 1 cm sample thickness.

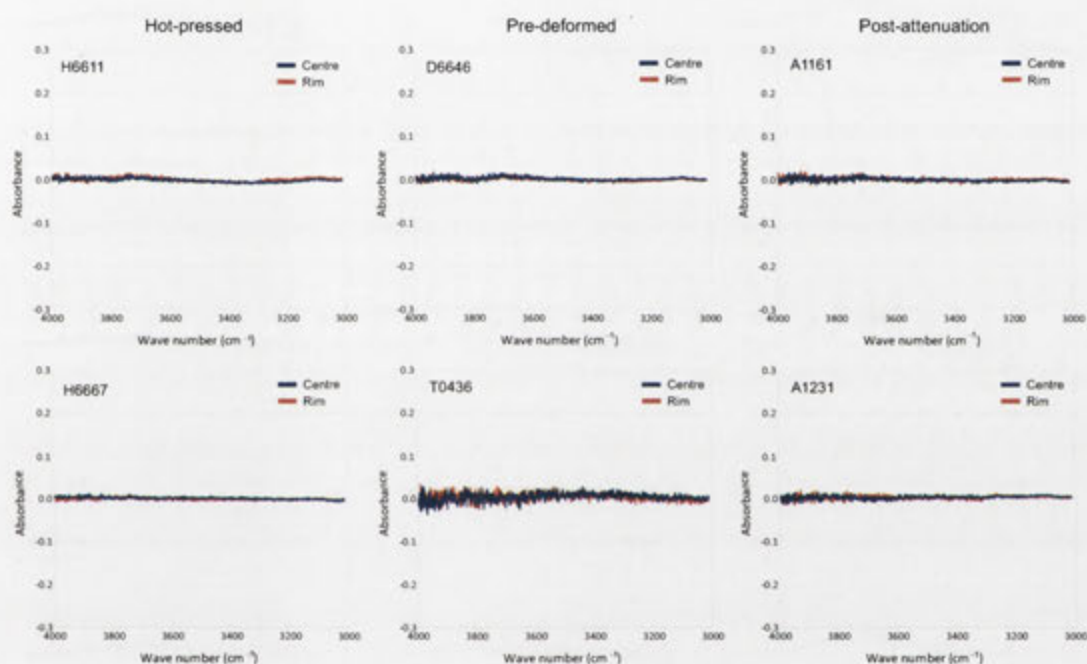


Fig. J.2: Second set of absorbance spectra of sol-gel olivine specimens during the various stages of experimentation. Two bulk measurements were taken, one at the centre and one at the rim of the cylindrical specimen. The IR beam had a nominal width of 1.5 mm. The spectra are normalised to 1 cm sample thickness.

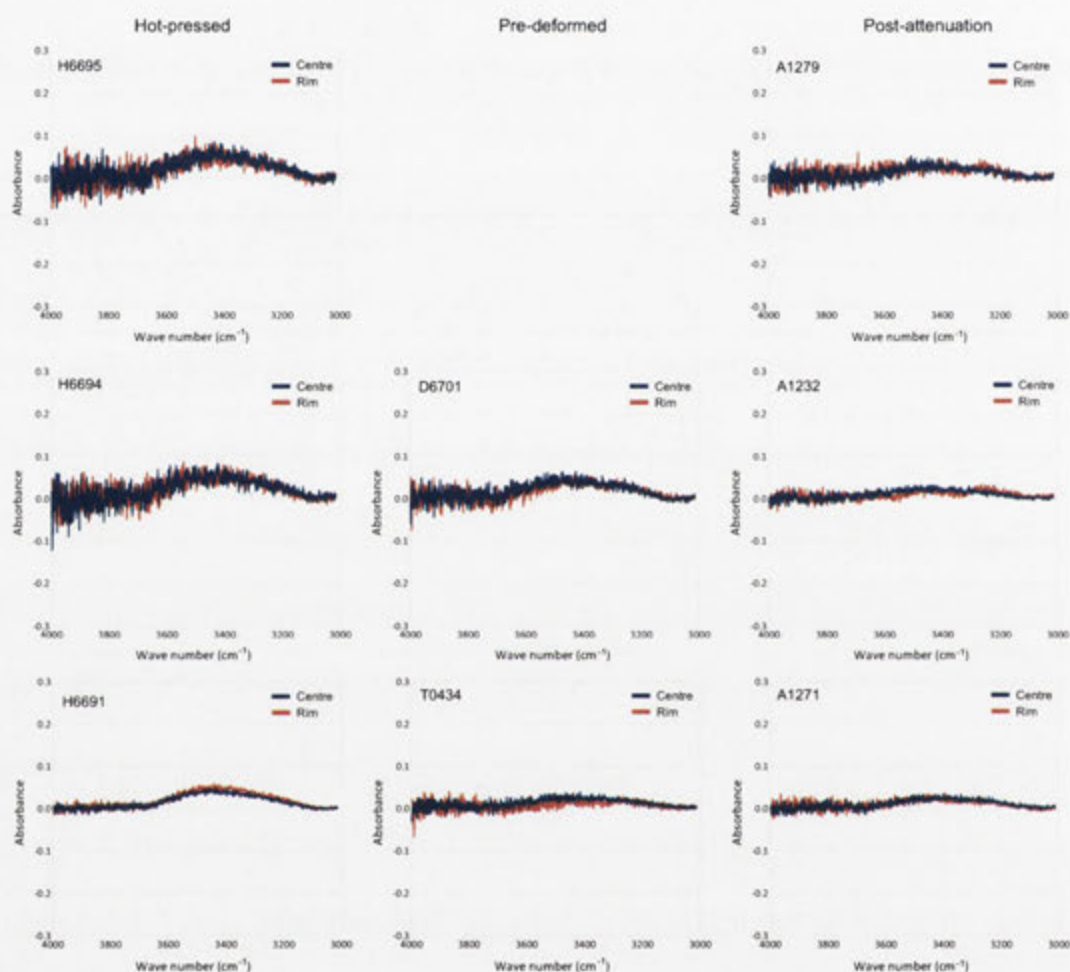


Fig. J.3: Absorbance spectra of all San Carlos olivine specimens during the various stages of experimentation. Two bulk measurements were taken, one at the centre and one at the rim of the cylindrical specimen. The IR beam had a nominal width of 1.5 mm. The spectra are normalised to 1 cm sample thickness.

K

EBSD map data

Figures K.1, K.2, K.3, K.4 and K.5 show the raw EBSD orientation data in inverse pole figure maps for the torsionally pre-deformed specimens and for the specimens after annealing and mechanical testing. In addition, EBSD data of a compressively pre-deformed San Carlos specimen are shown. The second map in each figure is noise-reduced showing the band contrast with highlighted low and high misorientation angles (see figure captions for legend).

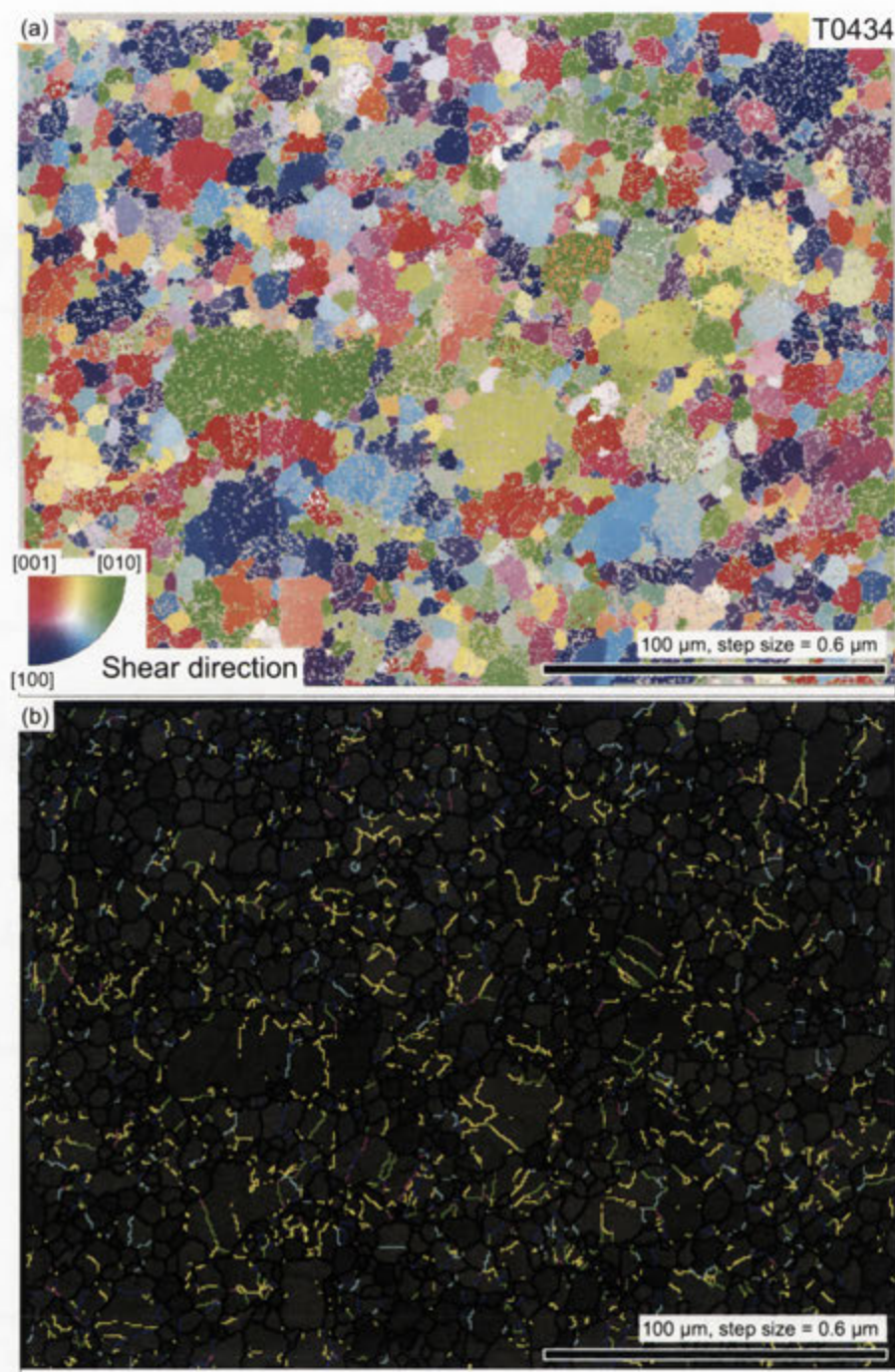


Fig. K.1: EBSD map data for torsionally pre-deformed T0434. (a) Raw orientation data displayed for all Euler angles. The light grey pixels represent non-indexed regions. (b) A noise-reduced band contrast map showing sub-grain boundaries with low misorientation angles $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries with high misorientation angles $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black.

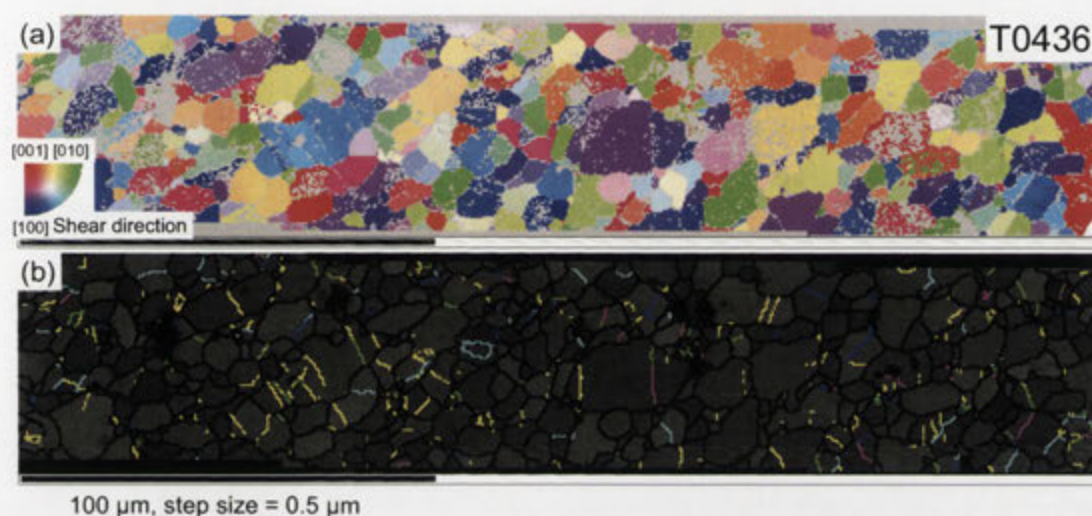


Fig. K.2: EBSD map data for torsionally pre-deformed T0434. (a) Raw orientation data displayed for all Euler angles. The light grey pixels represent non-indexed regions. (b) A noise-reduced band contrast map showing sub-grain boundaries with low misorientation angles $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries with high misorientation angles $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black. Unusually low indexing rate possibly caused by an inferior quality polish allowed only a small map to be completed in an overnight acquisition.

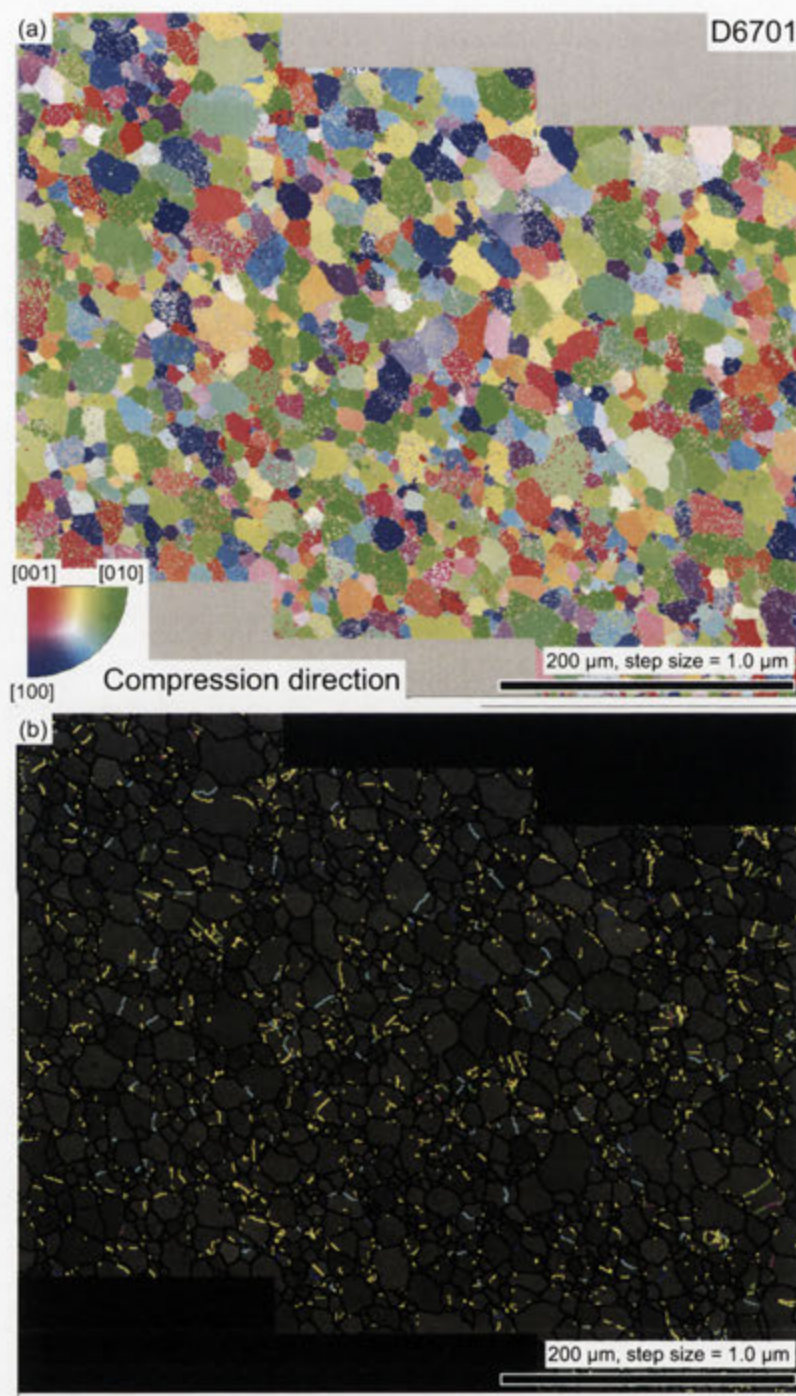


Fig. K.3: EBSD map data for torsionally pre-deformed T0434. (a) Raw orientation data displayed for all Euler angles. The light grey pixels represent non-indexed regions. (b) A noise-reduced band contrast map showing sub-grain boundaries with low misorientation angles $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries with high misorientation angles $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black.

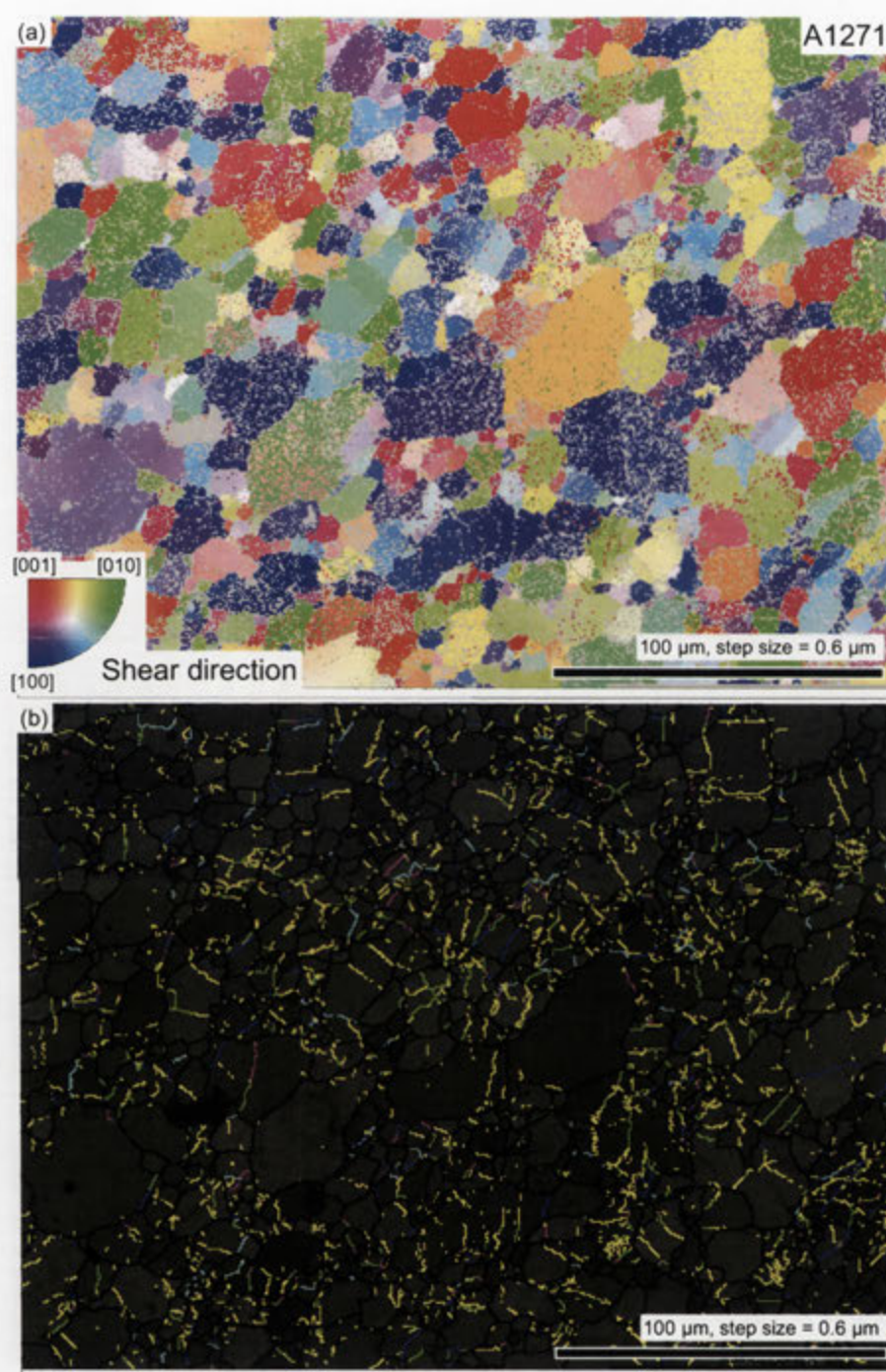


Fig. K.4: EBSD map data for torsionally pre-deformed T0434. (a) Raw orientation data displayed for all Euler angles. The light grey pixels represent non-indexed regions. (b) A noise-reduced band contrast map showing sub-grain boundaries with low misorientation angles $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries with high misorientation angles $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black.

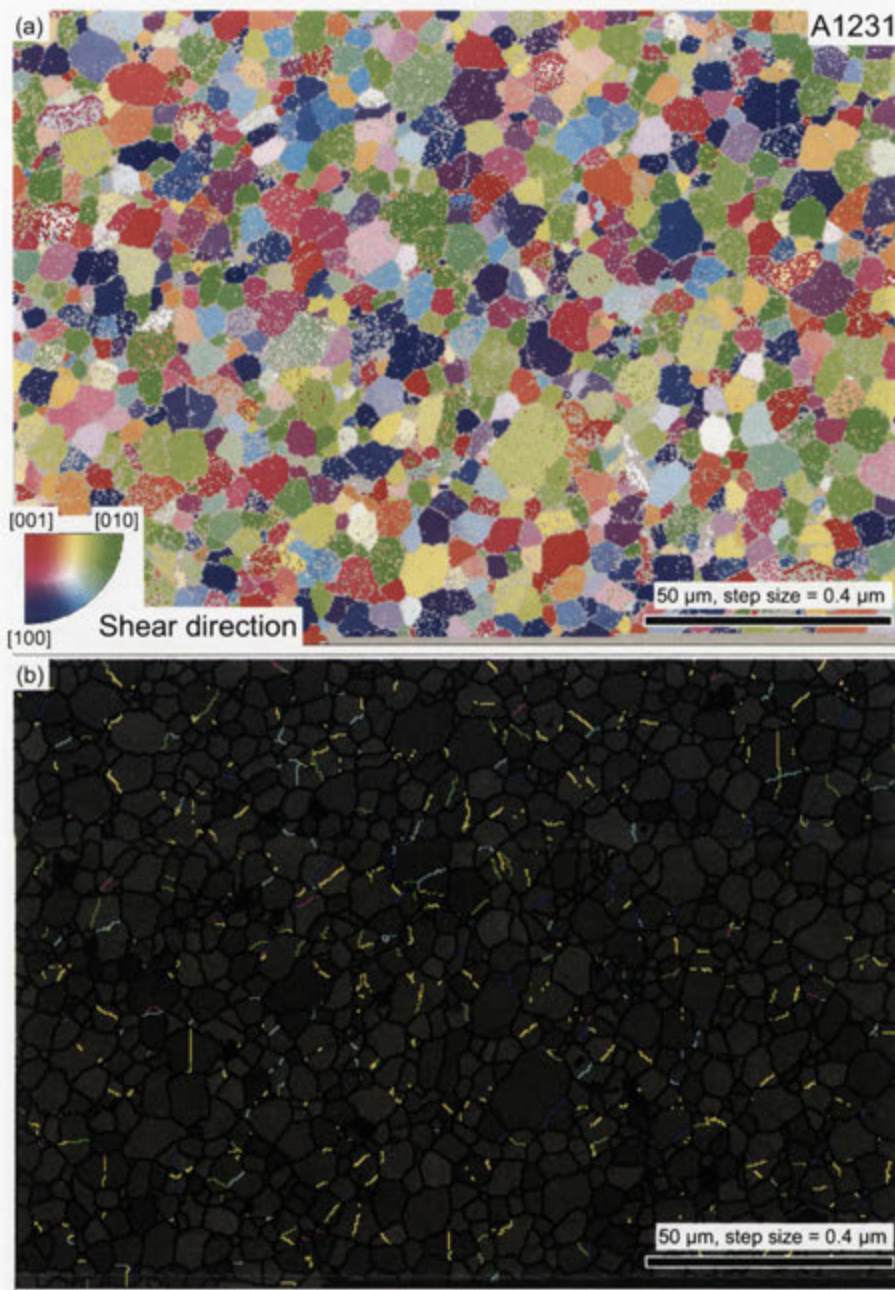


Fig. K.5: EBSD map data for torsionally pre-deformed T0434. (a) Raw orientation data displayed for all Euler angles. The light grey pixels represent non-indexed regions. (b) A noise-reduced band contrast map showing sub-grain boundaries with low misorientation angles $> 2^\circ$ yellow, $> 5^\circ$ lime green, $> 8^\circ$ purple, and grain boundaries with high misorientation angles $> 10^\circ$ blue, $> 20^\circ$ aqua and $> 30^\circ$ black.

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