

Nanoindentation-induced Deformation Mechanisms in Germanium

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CERTIFICATE

This thesis, to the best of my knowledge and belief, does not contain any results previously published by another person or submitted for a degree or diploma at any university except where due reference is made in the text.

David J. Oliver

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Abstract

Germanium (Ge), a Group IV elemental semiconductor, is an important electronic material used in many technological applications. Although it is frequently considered to be a classic brittle material, deforming elastically under mechanical stress up to the point of fracture, in practise this is not the case. Instead, under indentation with a sharp tip, plastic deformation plays a dominant role and other deformation mechanisms may be activated. In the literature there is some controversy as to what is the dominant indentation response of Ge at room temperature, shear-induced plasticity or high-pressure phase transformation. This thesis addresses that controversy by investigating the indentation response of germanium over a range of loading regimes and sample preparation conditions. A diverse range of responses is observed, shedding light on the behaviour of Ge at nano- and microscale contact events.

A wide range of techniques has been employed in this work to investigate the sharp contact response of Ge. Instrumented nanoindentation with a sharp diamond tip has been used to introduce mechanical damage at small scales. Features of the indentation force-displacement ($P-h$) curve can be linked to changes induced in the material. A number of techniques have been applied to characterise the damage produced, including cross-sectional transmission electron microscopy (XTEM), micro-Raman spectroscopy, atomic force microscopy (AFM), scanning electron microscopy (SEM), and focussed ion beam (FIB) analysis. In addition, high-energy ion implantation has been used to introduce structural defects and disorder or to completely amorphise the material.

Loading conditions are found to profoundly effect the deformation response of Ge. Rapid loading rates promote the formation of high-pressure phases during indentation, due to the rate-limited nature of shear plasticity mechanisms. These high-pressure phases transform to amorphous Ge (a-Ge) or metastable crystalline phases on load release. At high maximum load values, cracking becomes an important response. Lateral cracking in the vicinity of the indent is found to cause spallation and debris expulsion, resulting in a dramatic ‘giant pop-in’ event observed in the $P-h$ curve.

Implantation-induced disorder is found to have a pronounced effect on the mechanical properties of Ge. Implantation-induced defects in crystalline Ge lower the hardness and elastic modulus, suppressing cracking and causing enhanced plasticity and quasi-ductile extrusion. In ion-implanted a-Ge, high-pressure phase transformation is the dominant indentation response. Intriguingly, this phase transformation results in the formation of crystalline Ge on unloading.

Finally, it is found that the deformation response can be altered by confining Ge in the form of a thin film. Thin films of crystalline Ge on Si deform by high pressure phase

transformation, resulting in the formation of a-Ge on unloading. The threshold film thickness at which this occurs is associated with the geometry of the stress fields under the indenter.

These results show that a diverse range of indentation responses are possible in Ge and that the dominant response can be controlled via loading conditions and sample preparation. End phases of a-Ge and Ge-III are obtained under appropriate conditions with novel electronic, optical, and chemical properties. Furthermore, many of the findings here should be generalisable to other technologically important covalent semiconductors, opening new avenues of research.

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Table of acronyms

| | |
|---------------|--|
| a-Ge | amorphous Ge |
| a-Si | amorphous Si |
| AFM | atomic force microscopy |
| BSE | back-scattered electron |
| BF | Bright field |
| c-Ge | crystalline Ge |
| c-Si | crystalline Si |
| DAC | diamond anvil cell |
| DF | Dark field |
| DP | diffraction pattern |
| DLTS | deep-level transient spectroscopy |
| FESEM | field-emission SEM |
| FIB | focussed ion beam |
| hda-Ge | high-density amorphous Ge |
| LVDT | linear variable differential transformer |
| MEMS | microelectromechanical systems |
| NEMS | nanoelectromechanical systems |
| PAS | positron annihilation spectroscopy |
| SADP | selected area diffraction pattern |

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| SEM | scanning electron microscopy |
| TEM | transmission electron microscopy |
| UMIS | ultra-micro indentation system |
| XTEM | cross-sectional transmission electron microscopy |

Publications

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