Implant isolation of ZnO

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We study ion-irradiation-induced electrical isolation in *n*-type single-crystal ZnO epilayers. Emphasis is given to improving the thermal stability of isolation and obtaining a better understanding of the isolation mechanism. Results show that an increase in the dose of 2 MeV ¹⁶O ions (up to ~ 2 orders of magnitude above the threshold isolation dose) and irradiation temperature (up to 350 °C) has a relatively minor effect on the thermal stability of electrical isolation, which is limited to temperatures of ~ 300 – 400 °C. An analysis of the temperature dependence of sheet resistance suggests that effective levels associated with irradiation-produced defects are rather shallow (< 50 meV). For the case of implantation with keV Cr, Fe, or Ni ions, the evolution of sheet resistance with annealing temperature is consistent with defect-induced isolation, with a relatively minor effect of Cr, Fe, or Ni impurities on the thermal stability of isolation. Results also reveal a negligible ion-beam flux effect in the case of irradiation with 2 MeV ¹⁶O ions, supporting high diffusivity of ion-beam-generated defects during ion irradiation and a very fast stabilization of collision cascade processes in ZnO. Based on these results, the mechanism for electrical isolation in ZnO by ion bombardment is discussed. © 2003 American Institute of Physics.

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I. INTRODUCTION

Single-crystal ZnO has recently attracted significant research interest as a potential material for (opto)electronic device applications.¹ In future ZnO-based technology, bombardment with energetic ions can be used for electrical isolation of closely spaced devices. Indeed, it has recently been shown that ion irradiation under appropriate conditions can render ZnO highly resistive, with sheet resistances R_s of $\sim 10^{10}$ Ω/sq ,² as required for electrical isolation. Such an increase in the value of R_s is typically attributed to irradiation-induced degradation of carrier mobility as well as the trapping of free carriers at deep centers associated with ion-beam-produced defects (so-called *defect isolation*).³ Previous studies,² however, have revealed a relatively poor thermal stability of electrical isolation, where R_s of ion-irradiated ZnO effectively recovers during postimplantation thermal annealing at temperatures above ~ 300 °C. Better thermal stability is obviously desirable if ZnO is to be used for high-temperature/high-power electronics in cases when device operating/processing temperatures can be as high as several hundred degrees Celsius.

Previous studies of electrical isolation in other compound semiconductors have shown that the thermal stability

of isolation can be improved by several approaches.^{3,4} These include (i) increasing ion dose, (ii) carrying out ion irradiation at elevated temperatures to promote the formation of more stable defect complexes, and (iii) implantation of impurities which form thermally stable deep levels in the band gap and trap free carriers (so-called *chemical isolation*).

In this article, following our previous brief report,² we present a more detailed study of electrical isolation in single-crystal ZnO. Emphasis is given to improving the thermal stability of isolation and obtaining a better understanding of the isolation mechanism. Section III A examines the effects of ion dose and irradiation temperature on the thermal stability of ion-beam-produced damage. The influence of Cr, Fe, or Ni impurities, introduced by ion implantation, on the electrical properties of ZnO is studied in Sec. III B. Finally, in Sec. III C, we discuss additional results on the ion-beam flux effect and the temperature dependence of R_s , which help one to understand more fully the mechanism of electrical isolation in ZnO by light-ion bombardment.

II. EXPERIMENT

The *n*-type single-crystal wurtzite ZnO epilayers used in this study were ~ 0.6 - μm -thick, epitaxially grown on *a*-plane sapphire substrates by molecular-beam epitaxy at Osaka Institute of Technology. A further description of

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TABLE I. Implant conditions used in this study to introduce Cr, Fe, or Ni atoms into ZnO. All implants were carried out at RT. Calculated values of projected ion ranges (R_p) are also given.

Sample	Ion	Energy (keV)	Dose (10^{14} cm^{-2})	Beam flux ($10^{10} \text{ cm}^{-2} \text{ s}^{-1}$)	R_p (Å)
1	^{52}Cr	100	1	~ 8	478
1	^{52}Cr	225	2	~ 5	1069
1	^{52}Cr	500	4	~ 7	2442
1	^{52}Cr	1000	8	~ 10	5039
2	^{56}Fe	100	1	~ 3	444
2	^{56}Fe	225	2	~ 2	953
2	^{56}Fe	500	4	~ 3	2210
2	^{56}Fe	1000	8	~ 13	4564
3	^{58}Ni	100	1	~ 258	407
3	^{58}Ni	225	2	~ 258	890
3	^{58}Ni	500	4	~ 103	2071
3	^{58}Ni	1000	8	~ 52	4190

growth conditions can be found elsewhere.⁵ As-grown epilayers had a room-temperature (RT) free electron concentration of $\sim 10^{17} \text{ cm}^{-3}$, an effective Hall mobility of $\sim 80 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, and a R_s of $\sim 1 \text{ k}\Omega/\text{sq}$. Resistors of $\sim 3.5 \times 3.5 \text{ mm}^2$ in size were prepared with InGa eutectic ohmic contacts on two opposite sides of each sample.

These ZnO resistors were bombarded in an ANU 1.7-MV-tandem accelerator (NEC, 5SDH-4). During ion bombardment, samples were tilted by $\sim 7^\circ$ off the surface normal direction to minimize channeling. To study defect-induced isolation, irradiation with 2 MeV $^{16}\text{O}^+$ ions at 20 or 350 $^\circ\text{C}$ was performed. This energy of O ions was chosen to place the damage peak (as well as implanted O atoms) deep into the sapphire substrate, beyond the ZnO layer, given that the projected ion range of 2 MeV ^{16}O ions is $\sim 1.97 \mu\text{m}$ (as calculated with the TRIM code).⁶

To study possible chemical isolation, multiple-energy implantation with ^{52}Cr , ^{56}Fe , or ^{58}Ni ions was carried out at RT under conditions given in Table I. Such a multiple-energy implantation scheme resulted in a relatively uniform profile

FIG. 1. Depth profiles of ^{56}Fe ions implanted into ZnO with different ion energies, as indicated in the legend, to doses of 1×10^{14} (for 100 keV), 2×10^{14} (for 225 keV), 4×10^{14} (for 500 keV), and $8 \times 10^{14} \text{ cm}^{-2}$ (for 1000 keV). A solid line represents the total profile of Fe atoms after the multiple-energy Fe implant sequence. Results of TRIM simulations (see Ref. 6).

FIG. 2. Evolution of sheet resistance as a function of temperature of isochronal annealing (for 60 s in an Ar ambient) of ZnO epilayers bombarded at RT with 2 MeV O ions with a beam flux of $\sim 2 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ to different doses. The evolution of sheet resistance of an as-grown (unirradiated) sample during annealing is also shown for comparison.

of Cr, Fe, or Ni atoms throughout the entire $\sim 0.6\text{-}\mu\text{m}$ -thick surface layers of ZnO. This can be seen from Fig. 1, illustrating the depth profiles in the case of multiple-energy implantation with ^{56}Fe ions.

During ion irradiation, R_s was measured *in situ* after each dose step using a Keithley 619 electrometer. Postirradiation isochronal annealing was carried out in a rapid thermal annealing (RTA) system for 60 s at temperatures from 100 up to 1000 $^\circ\text{C}$ in an Ar ambient at atmospheric pressure. Before each annealing step, InGa ohmic contacts were removed with methanol to avoid possible degradation of the quality of ZnO films stimulated by the presence of InGa eutectic on the ZnO surface during thermal annealing.

III. RESULTS AND DISCUSSION

A. Thermal stability of damage

As alluded to earlier, previous studies² have revealed that defect-induced electrical isolation of ZnO is unstable to RTA at temperatures above $\sim 300^\circ\text{C}$. In this section, we investigate how critical irradiation parameters such as ion dose and sample temperature influence the thermal stability of isolation.

1. Effect of ion dose

It has previously been shown that the thermal stability of electrical isolation in other compound semiconductors can be improved by increasing ion dose.^{3,4} Hence, we have investigated the effect of ion dose on the thermal stability of isolation in ZnO. Figure 2 shows the evolution of R_s as a function of temperature of isochronal annealing of ZnO epilayers bombarded at RT with 2 MeV O ions to different doses. It should be noted that all ion doses from Fig. 2 are above (or equal to) the so-called threshold isolation dose Φ_{th} ,⁷ which is the ion dose necessary to create a concentration of deep levels (trapping free carriers) comparable with the initial concentration of free carriers. Figure 2 reveals a relatively small increase in thermal stability with increasing ion dose up to ~ 2 orders of magnitude above Φ_{th} . Indeed, it can be

FIG. 3. (a) Dose dependence of sheet resistance (measured at 350 °C) of ZnO irradiated with 2 MeV O ions at 350 °C with a beam flux of $\sim 2 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$. (b) Evolution of sheet resistance (measured at RT) as a function of temperature of isochronal annealing for 60 s of a ZnO sample bombarded at 350 °C with a beam flux of $\sim 2 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ to a dose of $1 \times 10^{16} \text{ cm}^{-2}$.

seen from Fig. 2 that, in all the cases studied, R_s experiences a fast decrease for annealing temperatures above ~ 350 °C. This suggests that an increase in ion dose (and a corresponding increase in the concentration of ion-beam-produced defects) has a relatively minor effect on the thermal stability of defect-induced isolation in ZnO.

2. Effect of irradiation temperature

Previous ion implantation studies of other semiconductors, such as Si and GaAs, have shown that the type and thermal stability of ion-beam-produced defects can strongly depend on irradiation temperature.^{3,4,8} Typically, the higher the substrate temperature during ion irradiation, the more likely it is that thermally stable defect structures form as a result of ion bombardment. Hence, it is instructive to investigate the influence of irradiation temperature on the thermal stability of electrical isolation in ZnO.

Shown in Fig. 3(a) is the ion dose dependence of R_s of a ZnO resistor irradiated with 2 MeV O ions at 350 °C. It is seen from Fig. 3(a) that, in this case of ion irradiation and R_s measurements at an elevated temperature, R_s increases ~ 2 orders of magnitude up to $\sim 10^5 \text{ } \Omega/\text{sq}$ with increasing ion dose up to $\sim 2 \times 10^{14} \text{ cm}^{-2}$. For ion doses from $\sim 2 \times 10^{14}$ up to $\sim 2 \times 10^{15} \text{ cm}^{-2}$, R_s is essentially independent of ion dose. Finally, for doses above $\sim 2 \times 10^{15} \text{ cm}^{-2}$, R_s decreases, which can be attributed to the onset of pronounced hopping conduction with increasing the concentration of ion-beam-produced defects. It should be noted that such a relatively low maximum R_s value of $\sim 10^5 \text{ } \Omega/\text{sq}$ shown in Fig.

FIG. 4. Evolution of sheet resistance as a function of temperature of isochronal annealing (for 60 s in an Ar ambient) of ZnO epilayers bombarded with Cr, Fe, or Ni ions, as indicated in the legend. See Table I for the details of implant conditions. The evolution of sheet resistance of an as-grown (unirradiated) sample during annealing is also shown for comparison.

3(a) is related to the fact that the measurement of R_s was performed (*in situ*) at 350 °C. Indeed, R_s measurements at RT of samples irradiated at 350 °C reveal larger R_s values. For example, in the case of a sample irradiated with 2 MeV O ions at 350 °C to a dose of $4 \times 10^{14} \text{ cm}^{-2}$, the value of R_s measured at RT is $\sim 2 \times 10^6 \text{ } \Omega/\text{sq}$, which is ~ 4 orders of magnitude lower than that for the case of RT irradiation to the same ion dose.

Figure 3(b) shows the evolution of R_s (measured at RT) as a function of temperature of isochronal annealing of a ZnO resistor bombarded at 350 °C with 2 MeV O ions to a dose of 10^{16} cm^{-2} . It is seen from Fig. 3(b) that such high-dose light-ion implantation at an elevated temperature results in a R_s of $\sim 4 \times 10^6 \text{ } \Omega/\text{sq}$ directly after ion irradiation. Figure 3(b) also shows that an increase in irradiation temperature up to 350 °C has only a minor effect on the thermal stability of electrical isolation. Indeed, in Fig. 3(b), R_s experiences a dramatic decrease as a result of annealing at temperatures above ~ 400 °C.

These results show that an increase in implantation temperature (up to 350 °C) and ion dose (up to ~ 2 orders of magnitude above Φ_{th}) has only a minor effect on the thermal stability of ion-beam-produced defects responsible for electrical isolation of ZnO. Hence, *defect-induced* isolation of ZnO by ion irradiation currently faces difficulties if desirable device processing and/or operating temperatures are above ~ 400 °C.

B. Implantation of Cr, Fe, or Ni

Previous isolation studies of compound semiconductors³ have shown that thermally stable electrical isolation can be achieved by implantation of impurities which form deep levels in the band gap and trap free carriers (so-called chemical isolation). Some previous studies have also suggested that transition metals in ZnO can form relatively deep levels in the band gap.⁹ Hence, in this section, we study the effect of Cr, Fe, or Ni impurities, introduced by ion implantation, on electrical properties of single-crystal ZnO.

Relatively uniform depth profiles of Cr, Fe, or Ni impurities (with a bulk concentration of $\sim 2\text{--}5 \times 10^{19} \text{ cm}^{-3}$) were achieved by a multiple-energy implantation scheme,

FIG. 5. Dose dependence of sheet resistance of ZnO samples irradiated with 2 MeV O ions at RT with different values of beam flux.

described in more detail in Sec. II and summarized in Table I. Shown in Fig. 4 is the evolution of R_s as a function of temperature of isochronal annealing of ZnO resistors bombarded with Cr, Fe, or Ni ions. It is seen from Fig. 4 that, directly after ion irradiation (i.e., before postirradiation annealing), the R_s of all three samples reaches the values of $\sim 10^9$ – 10^{10} Ω/sq .¹⁰ Such a dramatic increase in the value of R_s is due to ion-beam-produced lattice defects, which is similar to the case of bombardment with 2 MeV O ions described above. Figure 4 also shows that, similar to the case of irradiation with the other ion species discussed above and in Ref. 2, annealing at temperatures above ~ 300 °C effectively recovers implantation-induced defects, resulting in a dramatic decrease in R_s . Interestingly, Fig. 4 also shows that, for annealing temperatures from ~ 600 to 900 °C, R_s is lower than that of as-grown ZnO, indicating the formation of shallow (rather than deep) levels as a result of implantation of Cr, Fe, and Ni impurities (as well as ion-beam-produced lattice defects) and subsequent thermal annealing at temperatures up to 1000 °C. Hence, the introduction of Cr, Fe, or Ni into single-crystal ZnO by ion implantation and subsequent thermal annealing at temperatures up to ~ 1000 °C does not result in an improvement in the thermal stability of chemical isolation. At present, more work is needed to study whether thermal budgets larger than those used in the present study can electrically activate Cr, Fe, or Ni impurities implanted into ZnO, resulting in the formation of deep levels in the band gap. Additional work is also highly desirable to study the effect of other impurities which can potentially form thermally stable deep acceptor levels necessary for successful electrical isolation of *n*-type ZnO.

C. Mechanism of isolation

In this section, we present additional results which provide an insight into the mechanism for electrical isolation in ZnO. We also discuss possible reasons for a relatively poor thermal stability of isolation in this material.

A better understanding of defect processes responsible for electrical isolation can often be provided by studying the influence of ion-beam flux. Shown in Fig. 5 is the evolution of R_s of ZnO resistors bombarded at RT with different fluxes of 2 MeV O ions. Figure 5 reveals a negligible beam flux effect with the variation in the flux value over an order of

FIG. 6. Arrhenius plots of sheet resistance of ZnO epilayers bombarded with 2 MeV O ions at room temperature with a beam flux of $\sim 2 \times 10^{12}$ $\text{cm}^{-2} \text{s}^{-1}$ to different doses.

magnitude. This is consistent with results of previous studies^{11–16} where it has been shown that ion-beam-generated point defects are highly mobile in ZnO at least at RT and above. Indeed, a high defect diffusivity in ZnO results in a very fast stabilization of collision cascades produced by energetic light ions. In this case of very efficient dynamic annealing (i.e., defect migration and interaction during ion irradiation), a negligible flux effect is typically observed. This has recently been discussed in Ref. 17, where the influence of beam flux on the process of electrical isolation in another wide band gap semiconductor—GaN—has been considered in detail. Hence, the negligible flux effect in ZnO, coupled with efficient dynamic annealing processes, strongly suggests that centers responsible for isolation are not simple Frenkel pairs but the product of defect migration and interaction processes.

It is interesting to consider the position of the energy levels associated with lattice defects responsible for isolation. Figure 6 shows Arrhenius plots of R_s of ZnO resistors irradiated with 2 MeV O ions to different doses. The activation energies E_a derived from these $R_s(1/T)$ dependencies, also shown in Fig. 6, can often be used to roughly estimate the position of defect-induced levels in the band gap. Figure 6 reveals rather small effective activation energies $E_a < 50$ meV, which may suggest that effective levels associated with irradiation-produced defects are relatively shallow. However, more work, such as temperature-dependent Hall effect measurements, is currently needed to better understand the electronic properties of complex ion-beam-produced defects in single-crystal ZnO.

It is instructive to compare the isolation behavior of ZnO with that of other semiconductors. For example, previous studies^{3,18,19} have shown that ion irradiation of *n*-type GaAs or GaN creates a highly resistive material. The isolation process in these materials (i.e., GaAs and GaN) has been attributed to the formation of simple point defects, such as lattice vacancies, interstitials, as well as antisite defects, which are carrier traps with electronic levels close to the midgap. The thermal stability of ion-irradiation-induced electrical isolation in GaAs and GaN matches the expected annealing behavior where annealing temperatures scale with the melting point T_{melt} of the material.^{3,18} For example, electrical isola-

tion in GaAs ($T_{\text{GaAs}}^{\text{melt}} = 1240^\circ\text{C}$) and GaN ($T_{\text{GaN}}^{\text{melt}} \approx 2500^\circ\text{C}$) can be stable to thermal annealing at ~ 600 and $> 900^\circ\text{C}$, respectively.^{3,18} In addition, activation energies extracted from $R_s(1/T)$ dependencies for GaAs and GaN suggest the formation of deep carrier traps with levels close to the midgap.^{3,20}

The results of the present study reveal a quite different behavior of electrical isolation in ZnO in comparison to that in other semiconductors such as GaAs and GaN, discussed above. In particular, electrical isolation produced in ZnO by ion bombardment under a rather wide range of implant conditions is unstable to RTA at temperatures above ~ 300 – 400°C , whereas one can expect that significantly larger thermal budgets would be necessary to anneal irradiation-produced defects in ZnO, given its melting point of $\sim 1975^\circ\text{C}$. Indeed, previous Rutherford backscattering/channeling studies¹⁴ have shown that temperatures above 1000°C are needed to fully recover extended defects produced in ZnO by bombardment with keV Cr, Mn, or Bi ions at RT, which is consistent with the expected annealing behavior when temperatures of $\sim 2/3$ of the melting point (in K) are typically needed to remove implantation-induced extended defects in semiconductors (suggesting annealing temperatures of $\sim 1225^\circ\text{C}$ for ZnO).¹⁸

The fact that defect-induced isolation is thermally unstable indicates that (i) RTA recovers defect complexes associated with deep acceptor levels responsible for irradiation-induced dramatic changes in R_s and/or (ii) RTA results in the transformation of some ion-beam-produced defects, with deep levels in the band gap before annealing, into other defect structures which have shallow donor levels and, hence, significantly reduce the value of R_s . Results of the present study cannot distinguish between these two scenarios, and, at present, more work is obviously needed to better understand the electrical properties of ion-beam-produced defects in ZnO.

IV. CONCLUSIONS

In conclusion, we have studied the evolution of sheet resistance of single-crystal ZnO during irradiation with 2 MeV O ions at 20 or 350°C as well as during postimplantation annealing. Results have shown that an increase in the dose of 2 MeV ^{16}O ions (up to ~ 2 orders of magnitude above the threshold isolation dose) and irradiation temperature (up to 350°C) has a relatively minor effect on the thermal stability of electrical isolation, which is limited to temperatures of ~ 300 – 400°C . In addition, the introduction of Cr, Fe, or Ni into single-crystal ZnO by ion implantation with subsequent annealing at temperatures $\leq 1000^\circ\text{C}$ does not result in an improvement in the thermal stability of elec-

trical isolation. More work is highly desirable to study the effect of other impurities which can potentially form thermally stable deep acceptor levels in ZnO if desirable device processing and/or operating temperatures are above $\sim 400^\circ\text{C}$.

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