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M. W. Bench[1], I. M. Robertson[1], and M. A. Kirk[2]
[1]Dept. of Materials Science and Engineering, University of Illinois at Urbana
Urbana, Illinois 61801
[2]Materials Science Division, Argonne National Laboratory
Argonne, Illinois 60439

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IN SITU TEM OBSERVATIONS OF HEAVY ION DAMAGE IN GALLIUM ARSENIDE

M.W. BENCH,* I.M. ROBERTSON,* AND M.A. KIRK**
*Dept. of Materials Science and Engineering, University of Illinois, Urbana, IL 61801
**Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

ABSTRACT

Transmission electron microscopy experiments have been performed to investigate the lattice damage created by heavy-ion bombardments in GaAs. These experiments have been performed in situ by using the HVEM - Ion Accelerator Facility at Argonne National Laboratory. The ion bombardments (50 keV Ar^+ and Kr^+) and the microscopy have been carried out at temperatures ranging from 30 to 300 K. Ion fluences ranged from 2×10^{11} to 5×10^{13} ions cm^{-2}.

Direct-impact amorphization is observed to occur in both n-type and semi-insulating GaAs irradiated to low ion doses at 30 K and room temperature. The probability of forming a visible defect is higher for low temperature irradiations than for room temperature irradiations. The amorphous zones formed at low temperature are stable to temperatures above 250 K. Post implantation annealing is seen to occur at room temperature for all samples irradiated to low doses until eventually all visible damage disappears.

INTRODUCTION

The damage produced by ion implantation in GaAs has been the subject of extensive investigation for a number of years. Much of this research has focused on the structure of the amorphous layer created at high doses and on the annealing processes required to restore the crystalline lattice structure [1]. Also, a considerable amount of work has focused on the structure of the primary damage and the mechanisms by which this damage builds up to form an amorphous layer [2]. Two basic models for the mechanisms by which amorphization occurs in semiconductors have been proposed. In the first, amorphization occurs by a build up of simple damage during irradiation until the defect density is so great that the region spontaneously collapses to an amorphous state [3]. In the second, small regions are directly amorphized during individual collision cascades and complete amorphization occurs by the accumulation and overlap of these regions [4]. The first model has generally been used to explain the build up of damage for light ions and high temperature, whereas the second has been used to explain the damage created by heavy ions at low temperatures. A more realistic model would be one that allows both mechanisms to occur simultaneously and, as a result, a composite model of amorphous layer production has been developed [5] that includes both and also includes the effects of simultaneous damage annealing. While these models provide a good description of the general mechanisms by which the crystalline-to-amorphous transformation occurs, the exact mechanisms remain controversial.

Most investigations made on the build up of damage toward the production of an amorphous layer have been made by the Rutherford backscattering-channeling technique [6,7,8]. Although much information has been gathered using this technique, it has the limitation that no direct assessment of the actual damage structure can be made. The direct observation of ion implantation induced damage structure is well suited to TEM investigations.

However, as will be shown, the occurrence of room temperature annealing in GaAs complicates the only previously reported low dose investigations [9,10]. Irradiations performed in situ in the HVEM at both 30 K and room temperature have allowed us to perform this investigation without the complications seen by the previous investigators. It is the results of these studies that are reported in this paper.

EXPERIMENTAL

The experiments were performed in the High-Voltage Electron Microscope - Ion Accelerator Facility at Argonne National Laboratory. This facility consists of two accelerators, a 300 keV Texas Nuclear accelerator and a NEC 2 MeV Tandem accelerator, either of which produce ion beams that can be directed onto the HVEM sample position by an ion-beam interface. For these experiments only the first accelerator was used. The details of this facility can be found in reference [11]. The low-temperature experiments have been performed using a double-tilt, liquid-helium cooled, HVEM sample stage which is capable of supporting samples during in situ irradiations and HVEM examination at controlled temperatures between 10 and 300 K. It should be emphasized that it is the powerful capabilities of this facility, cryogenic temperatures and in situ irradiations, that have allowed this investigation to be made.

The samples included both n-type (1 x 10^{18} Si atoms cm^{-3}) and undoped semi-insulating (100) wafers of GaAs. Studies were made in both materials in an attempt to account for the discrepancy between our results and those of previous investigations [9,10]. The TEM samples were prepared using a chemical thinning technique in a static bath solution of 4 parts H_2SO_4, 1 part H_2O_2, and 1 part H_2O at 35° C.

The samples were irradiated in a non-channeling direction with Ar^+ and Kr^+ ions with incident energies of 50 keV. Ion doses ranged from 2 x 10^{11} to 5 x 10^{13} ions cm^{-2}. Dose rates were typically less than 3 x 10^{10} ions cm^{-2} sec^{-1}. The electron microscopy was performed with the HVEM operating at 200 kV. For the most part, micrographs were taken in dark field under dynamical two beam conditions using the fundamental reflection g = (220). It was under these diffracting conditions that the most distinct defect images were observed. The contrast we observe is similar to that seen in silicon by other researchers [12].

To investigate the degree and extent of disordering produced by the cascades pairs of micrographs have been taken using the (200) superlattice and (400) fundamental reflections. The defect contrast using fundamental reflections occurs because of a significant difference in the structure of amorphous and crystalline material. On the other hand, regions high in antisite defects yet still crystalline can be imaged with the (200) superlattice reflection because it is present as a result of the ordering of the Ga and As on distinct sublattices. This effect is comparable to the use of superlattice reflections to directly observe the disordered zones created by cascades in ordered alloys such as Cu_3Au [13].

RESULTS AND DISCUSSION

The results from specific irradiations are summarized in Table 1 and are discussed in detail below. In the table and subsequent text the defect yield is defined as the number of visible amorphous zones produced per incident ion.

Table I
Defect yields

MATERIAL	IRRADIATION TEMPERATURE AND ION		
	300 K 50 keV Kr^+	30 K 50 keV Kr^+	30 K 50 keV Ar^+
N-TYPE	0.18	0.78*	0.17
SEMI-INSULATING	0.19	0.95	-

*limited data

30 K Irradiations with 50 keV Kr^+ and Ar^+ ions

The micrographs presented in figure 1 show that in the semi-insulating material a high density of amorphous zones are produced during ion bombardment at low temperatures; a similar result was obtained for the n-type material. At these ion doses (< 10^{12} ions /cm^2), the degree of spatial overlap of the cascades is negligible and the amorphous zones are formed from isolated individual displacement cascades. The zones are evident immediately following the irradiation and do not develop with time. Comparison of the micrographs reveals that the density of the amorphous zones is higher following the Kr^+ ion than the Ar^+ ion irradiation; the yield difference is between a factor of 3 to 4 times greater for the Kr^+ ion irradiation, see Table 1. The area occupied by the amorphous zones is also

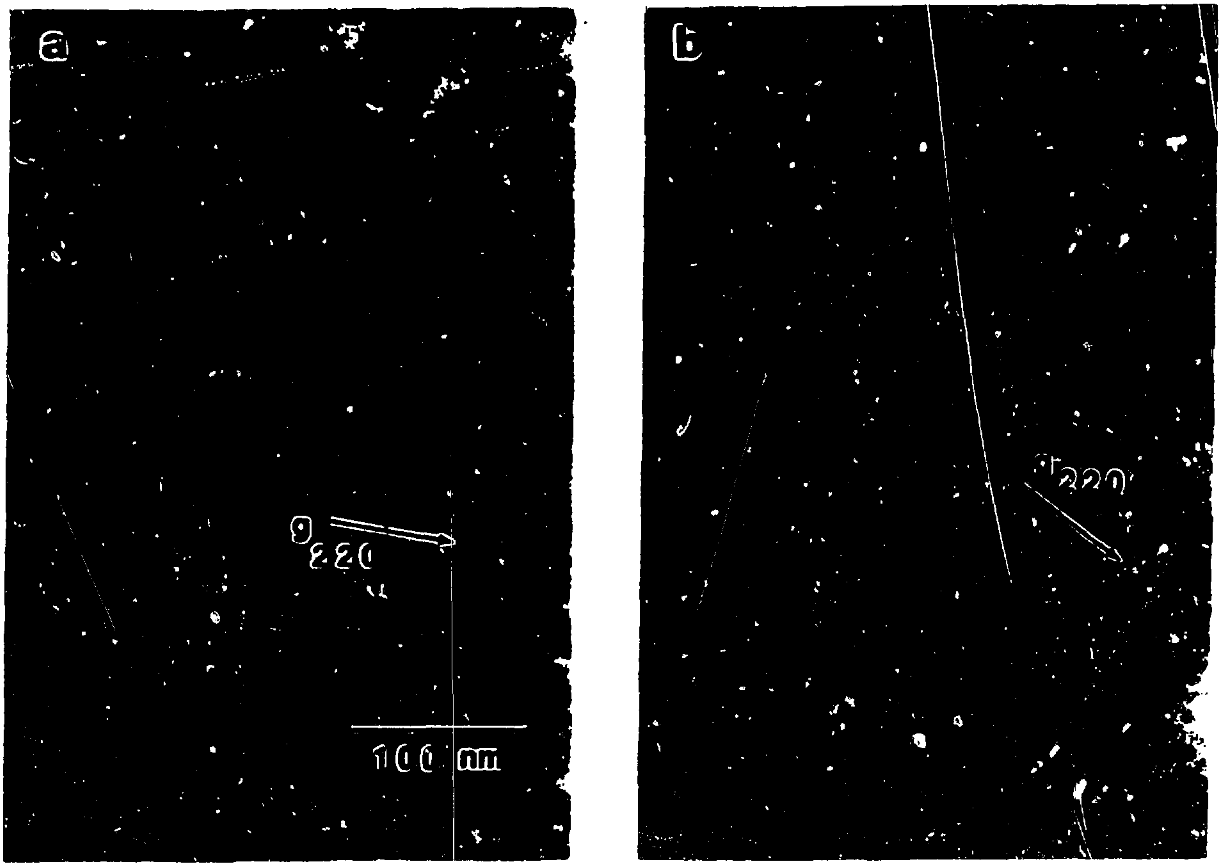

Fig 1. Comparison of the amorphous zones produced at 30 K by (a) Kr^+ and

dependent on the mass of the incoming ion, being on average larger for the Kr$^+$ ion irradiation. Zones produced by the Kr$^+$ ions have an average area of 36.8 nm^2 but can be as large as 100 nm^2, whereas the average area for zones produced by Kr$^+$ ion irradiations is 30.4 nm^2 and the largest 75 nm^2. The yield and size differences are readily understood in terms of the cascade parameters for the different ions.

Crystallization of the amorphous zones during warm-up from 30 K to 300 K

Annealing of the materials from 30 to 300 K caused the individual zones to crystallize. The yield after warm-up drops by a factor of about 4 and is now equal to the yield at room temperature immediately after irradiation. The micrographs in Figure 2 show the results of a step anneal from 30 to 300 K, the zones remain as the temperature is increased to 250 K. Above 250 K the zones start to recrystallize, with the yield decreasing by a factor of 0.44 between 250 and 285 K. Area size distributions show no change with increasing temperature, indicating that the size of the zone does not dictate the order in which the zones crystallize. The loss of amorphous zones at temperatures > 250 K corresponds with one of the recovery peaks in the electrical properties as determined by resistivity measurements [14]. The defect responsible for the recovery has not been unambiguously identified.

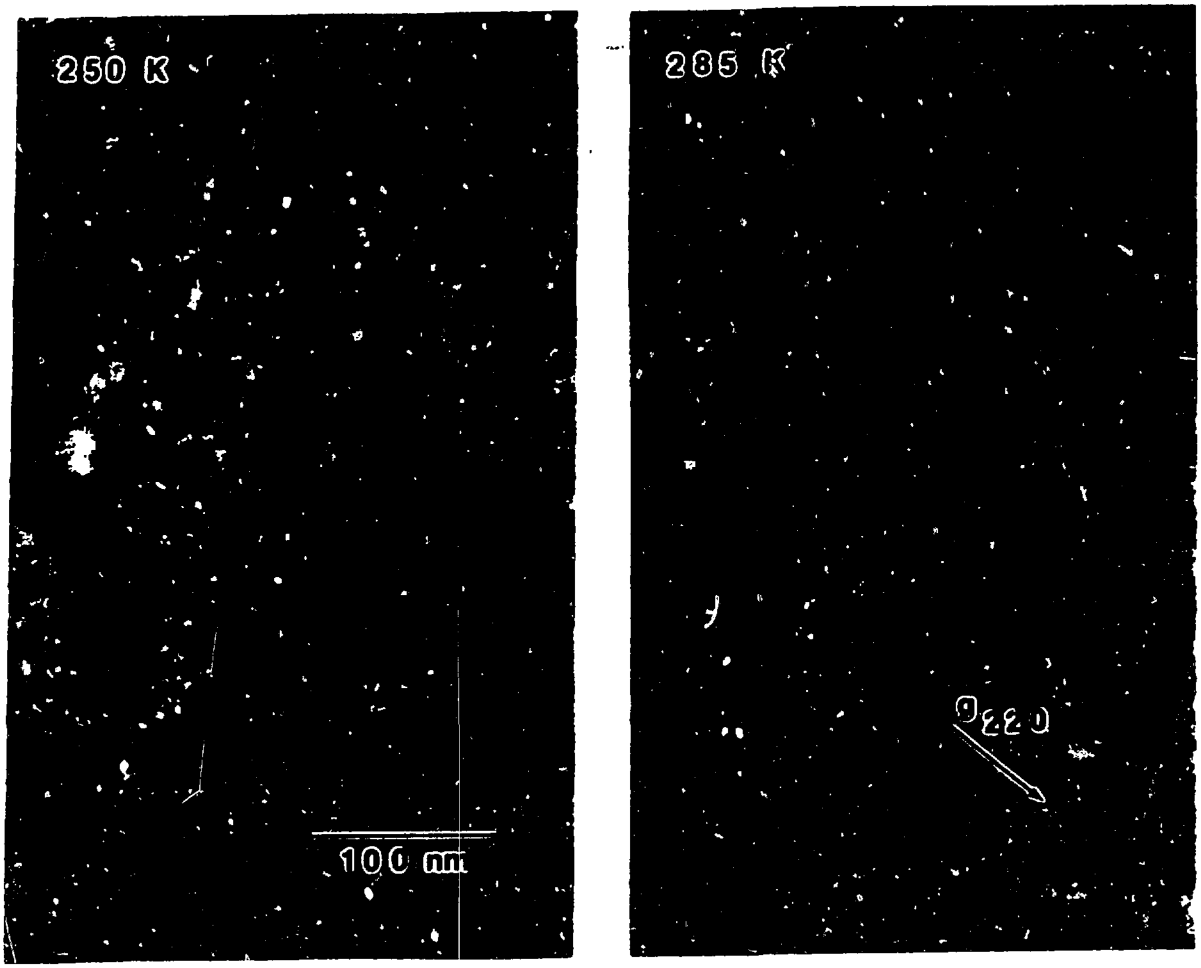

Figure 2. Density of defects as a function of annealing temperature from 30 to 300 K. The sample was irradiated at 30K with 50 keV Ar$^+$ ions to a dose of

Crystallization of amorphous zones created during room temperature irradiations

For both n-type and semi-insulating GaAs, the yield obtained from room temperature irradiations is lower than the yield obtained from low temperature irradiations, see Table 1. These zones are again formed from isolated displacement cascades. The room temperature yield decreases with time at room temperature. It is this room temperature crystallization effect which explains why previous work found no zones at these ion doses. The crystallization rate also increased under the electron beam; this is probably a consequence of beam heating rather than collisional assistance. An example of the room temperature annealing in semi-insulating GaAs is shown in Fig 3. The micrographs in Fig 3(a and b) compare the same region of sample 30 and 60 minutes after irradiation. The zones indicated by the arrows are not present in the second micrograph. After 20 hours, the yield has decreased from 0.19 to 0.09. Further time at room temperature removes all isolated amorphous zones. Analysis of the zone area distributions showed that there was no preferential recovery of either large or small isolated zones. The low yield at room temperature and the results of earlier work can now be understood in terms of the competition between creation and crystallization of the amorphous zones. At high ion doses (>10^{13} ions cm^{-2}) and at higher dose rates where the displacement cascades overlap both spatially and temporally (i.e. within the lifetime of the amorphous zone) stable amorphous zones can form. The greater stability of amorphous zones formed under these irradiation conditions is not understood but may reflect

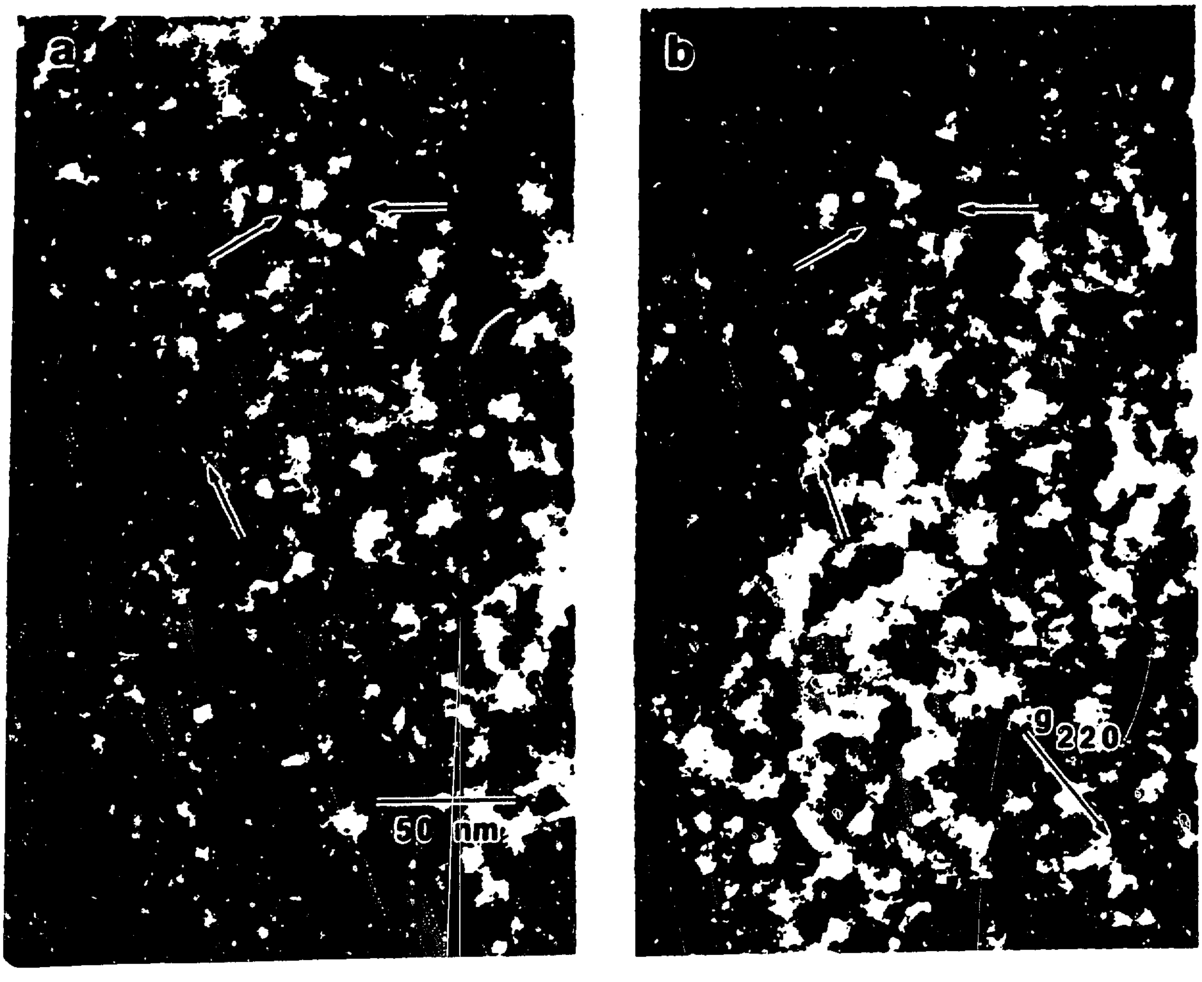

Figure 3. Room temperature annealing at (a) 30 and (b) 60 minutes after the irradiation. The arrows in (a) and (b) mark defects that have disappeared. The sample was irradiated to a dose of 9×10^{11} ions cm^{-2} with 50keV Kr^+ ions.

the degree of antisite disorder surrounding the amorphous zones. Diffusion of the defect responsible for the room temperature recovery is expected to be dependent on the degree of antisite disorder, being slower the greater this disorder. Measurements of the extent of the antisite disorder zone associated with the amorphous zone are complicated, however, a preliminary comparisons of images taken using the (200) superlattice and (400) fundamental reflections indicate that the antisite disordered zones, at least in the low temperature irradiations, are larger than the amorphous zones. The degree of antisite disorder associated with amorphous zones and its dependence on ion dose and dose rate is the subject of further investigation.

SUMMARY

Amorphous zones have been observed to form in both semi-insulating and n-type GaAs from single isolated displacement cascades. The yield from room temperature irradiations is less than that from low temperature irradiations due to the continual room temperature crystallization of the amorphous zones. The zone crystallization rate is not related to size of the amorphous region but may be related to the size and degree of the antisite disorder zone that is associated with the amorphous zone.

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