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EDGE-ON ION IRRADIATION OF ELECTRON MICROSCOPE SPECIMENS*

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ABSTRACT

A special technique is described for in situ transmission electron microscope (TEM) experiments involving simultaneous ion irradiation, in which the resultant phenomena are observed as in a cross-section TEM specimen. That is, instead of ion-irradiating the film or foil specimen normal to the major surfaces and observing in plan view (i.e., in the same direction), the specimen is irradiated edge-on (i.e., parallel to the major surfaces) and is observed normal to the depth direction with respect to the irradiation. The results of amorphization of Si, irradiated in this orientation by 1 or 1.5 MeV Kr, are presented and briefly compared with the usual plan view observations. The limitations of the technique are discussed and several experiments which might profitably employ this technique are suggested.

INTRODUCTION

Generally when in situ ion irradiation experiments in a TEM are performed, the specimen and hence the resulting phenomena are seen essentially in plan view because the irradiation and viewing directions are both more or less normal to the surface of the specimen. The typical disposition of the specimen with respect to the electron and ion beams for such an in situ ion irradiation experiment in the high voltage electron microscope (HVEM) of the HVEM-Tandem Facility at Argonne is depicted in Fig. 1, for which the angle between the incident ion and electron beams $\phi = 33$ deg. In addition, because of the requirement that the TEM specimen be relatively thin, a few tenths of μm or even much less for heavy metals, which is a distinct advantage when high energy implantation effects are to be avoided, such deep implantation-type experiments have not been possible in situ. Geometrically at least, these problems can be circumvented by ion irradiating the specimen at 90 degrees with respect to the viewing direction; i.e., by irradiating an edge of the specimen, parallel to, rather than normal to, the major surfaces of the specimen. This turns the specimen into a kind of dynamic cross-section specimen as far as the observer is concerned. The disposition of the specimen with respect to the electron and ion beams for this situation is depicted in Fig. 2. While there are a number of problems with actually doing this and also with interpreting the results quantitatively, results of each of two experiments will be presented in order to demonstrate the principle. The experiments involve the amorphization of Si at room temperature during edge-on bombardment by Kr ions of incident energies of 1.0 and 1.5 MeV. Several other experiments are suggested, involving the configuration described here, from which meaningful quantitative information may be derived. As with any well designed in situ experiment, the advantage of performing a study in this way is the high degree of experimental efficiency and physical sense which may result.

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Fig. 1. Usual plan view configuration in the HVEM at Argonne for simultaneous ion and electron irradiation experiments. $\phi = 33$ deg.

EXPERIMENTAL DETAILS

In order to perform an edge-on ion irradiation experiment in a TEM, several conditions must be met. The experiment involves a somewhat unconventional specimen configuration and a specimen holder must allow access of the ion beam to the specimen edge. Each of these is discussed briefly below. In the experiment itself, ideally the ion and electron beams should be more or less at a right angle, as illustrated in Fig. 2, in order to allow simultaneous ion irradiation and TEM analysis under substantially optimal conditions. There are twelve installations in the world now in which a TEM is interfaced to one or more ion accelerators. Of these, only two are configured in a way which is essentially ideal, in the sense of Fig. 2, for this type of experiment: the facilities at CSNSM-CNRS—Orsay, France (120 kV Philips EM400 with 5–200 kV ion accelerator) and at the Central Research Institute of Electric Power—Komae, Japan (200 kV JEOL 200CX with a 20 kV ion accelerator). For the remaining ten installations, nine of which are in Japan, the angle between the ion and electron beams ranges from 18–45 degrees. In the case of the HVEM-Tandem Facility, this angle is 33 degrees (i.e., the specimen must be tilted 57 degrees from horizontal during the ion irradiation, which makes simultaneous observation impossible with most existing specimen holders). The procedure for conducting the edge-on irradiation experiment in such a case is described in the third part of this section.

Specimen preparation

For the demonstration experiments described in this paper, the specimen material was Si which was thinned by chemical polishing in gently agitated solution of HNO_3 (65 pct), HF (25 pct) and H_3PO_4 (10 pct) at room temperature. The starting material was a Si wafer, 4 deg off (111), from which 3 mm diameter discs were core-drilled and chemically polished to a thickness of about 100 μm . Each specimen was then lacquered with a commercial stopoff, completely on one side and in the shape of a crescent around about 2/3 of the edge on the other. Further polishing resulted finally in a uniformly thin area which usually polished away from the open portion of the crescent. Stopping this process at the right moment is critical, of course, in which case the cross-section of the Si at an edge within the crescent is very satisfactory. Frequently it was possible to estimate the specimen thickness from observation of thickness fringes very near the specimen edge, especially if a piece of the Si had broken off, leaving a flat edge. For such an estimate, the specimen must be tilted in the TEM into a known, strong two beam condition so that the corresponding

Fig. 2. Ideal configuration in a TEM for simultaneous electron and edge-on ion irradiation experiments.

extinction distance ξ_g is well established.

Specimen holder

An ambient temperature, single tilt holder for the HVEM specimen was modified so that an ion beam could be admitted to the edge of the specimen with the specimen in its normal position in the holder. This modification consisted of machining a 2 mm wide notch in the holder at the specimen position and removing approximately one quarter of the specimen retaining ring. The specimen was mounted as in a normal holder, but with the edge to be ion irradiated facing these cutouts in the holder.

Procedure for edge-on ion irradiation

Prior to the specimen holder modification just described, several preliminary experiments were performed utilizing a small adapter which allowed the specimen to be mounted in the holder at an inclination of 40 deg with respect to the horizontal holder position. Thus for the ion irradiation, the holder was tilted +17 deg away from the ion beam so that the specimen was edge-on with respect to the ion beam and could still be observed during the irradiation. For intermittent image recording, the specimen holder was tilted to -37 deg so that the specimen was nearly horizontal. With a specimen in the modified holder described above, for which mounting the specimen is considerably simplified, the holder must be tilted +57 deg away from the ion beam for edge-on irradiation. In this particular holder the specimen may still be observed during the irradiation, however, if the edge to be observed is near the center of the usual specimen position. After various dose intervals, the ion irradiation is stopped momentarily and the specimen tilted back to horizontal for image recording. For the demonstration experiments reported here, the ion irradiation was 1.0 or 1.5 MeV Kr at a flux of $3.4 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$. The threshold for displacement of Si by electrons corresponds to an operating potential for the microscope of approximately 170 kV. The HVEM was operated at 150 kV (below threshold) in the first experiment and at 1 MV (well above threshold) in the second experiment.

RESULTS AND DISCUSSION

In the first experiment a Si crystal was irradiated edge-on in the modified specimen holder, nominally at room temperature, with 1.5 MeV Kr at a flux of $3.4 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$, results of which are shown in Fig. 3 after three ion doses: (a) $1.0 \times 10^{14} \text{ cm}^{-2}$, $1.5 \times 10^{14} \text{ cm}^{-2}$ and (c) $2.0 \times 10^{14} \text{ cm}^{-2}$. The thickness of the specimen was approximately 300 nm as determined by counting thickness fringes in a strong two beam (220) bright field image. The HVEM was operated at 150 kV, below threshold for Si displacement by electrons. Because a single tilt holder is employed, it was not possible to reproduce exactly the same diffraction condition for each of the three micrographs of Fig. 3. However, the development of damage as a function of depth in the specimen and of ion dose is clear from this sequence. TRIM calculations for 1.5 MeV Kr on Si [1] yield a broad band of large energy deposition roughly from 400–800 nm depth; the calculation ignores, however, the finite dimension of the Si (the vertical dimension in Fig. 2). Selected area diffraction patterns from this peak damage zone indicate that a large fraction of the material in this zone is amorphized by the ion dose of $2.0 \times 10^{14} \text{ cm}^{-2}$ (Fig. 3c). In a subsequent experiment performed in the usual plan view configuration (as depicted in Fig. 1) involving a Si crystal of approximately the same thickness and 1.5 MeV Kr, the Si was completely amorphized by an ion dose of approximately $8 \times 10^{14} \text{ cm}^{-2}$. At this point it is difficult to assess with any accuracy the role of the free surfaces in the edge-on configuration from this comparison, but of course this is a very important question.

In the second demonstration experiment another Si specimen was irradiated, nominally at room temperature, with 1 MeV Kr at a flux of $3.4 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$. In this case the specimen adapter described above was employed in a single tilt holder with the HVEM operated at 1 MeV, well above threshold for Si displacement by electrons. The micrograph of Fig. 4 shows the result of an ion dose of about $2 \times 10^{15} \text{ cm}^{-2}$. In this micrograph the ion irradiated edge is shown at the top of the micrograph. A well developed amorphous zone is evident, now seen as a band of good electron transparency compared to "deeper" material which has been heavily damaged by the 1 MeV electron beam. Any misorientation of the specimen with respect to the incident ion beam may also produce a thin damage layer over all of one surface of such a crystalline specimen. Such misorientation would be evident only when not superimposed on a damage structure associated with the simultaneous high energy electron irradiation.

OTHER POTENTIAL EXPERIMENTS

It should be possible to observe individual ion tracks (defect clusters and/or amorphous zones) in a Si specimen for very low dose ion irradiations in the edge-on configuration. Such capability would allow, for example, the effects of ion mass and energy and of specimen temperature on the nature of such tracks to be established empirically by in situ experiments.

Fig. 3. Development of amorphization in Si irradiated edge-on with 1.5 MeV Kr to doses of (a) $1.0 \times 10^{14} \text{ cm}^{-2}$, $1.5 \times 10^{14} \text{ cm}^{-2}$ and (c) $2.0 \times 10^{14} \text{ cm}^{-2}$. Ions impinge onto the specimen edge at left parallel to the plane of foil. Note development of peak damage zone. HVEM operated at 150 kV.

Fig. 4. Si specimen ion irradiated edge-on with 1 MeV Kr to dose of about $2.0 \times 10^{15} \text{ cm}^{-2}$. Ions impinge onto edge at top of micrograph, parallel to plane of foil. HVEM operated at 1 MeV. Note amorphous band and zone of dense damage. Magnification uncertain.

It may also be possible to study development of microstructure during and subsequent to deep implantation, such as the development of a silicide with implantation of a transition element into Si at a moderate temperature. In such an experiment the role of the free surfaces, for example as defect sinks (the top and bottom surfaces in Fig. 2) again may be crucial. In order to assess this role in part, we are modifying a TRIM code to take account of the finite dimension of the edge-on specimen (vertical dimension in Fig. 2) in order to estimate the damage distribution as a function both of depth (distance in from the edge) and vertical position in the specimen, neglecting defect mobility.

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