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RADIATION INDUCED PRECIPITATION IN SILICON DURING
HIGH VOLTAGE ELECTRON MICROSCOPE OBSERVATION

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ABSTRACT

Two modes of electron beam radiation induced precipitation have been observed in high purity, Lopex (low oxygen, dislocation free), silicon. (1) At room temperature a whisker-like precipitate structure developed along the dislocation lines. (2) During hot stage observations long needle-shaped precipitates oriented in $\langle 110 \rangle$ directions were formed.

I. INTRODUCTION

In high voltage electron microscopy, radiation damage occurs when the electron beam voltage exceeds the threshold value required for an electron to eject a matrix atom.¹⁻² This effect should be especially pronounced in foils made from the lighter elements, and as pointed out by Makin and Sharp¹ the effect may complicate studies of annealing and diffusion processes. However, little work dealing with radiation induced effects in the high voltage microscope has been reported in the literature. In the case of silicon, formation of small defect clusters attributed to electron damage has been reported by Thomas.³ This paper describes radiation induced precipitation in high purity silicon crystals. This precipitation effect was observed in specimens already containing precipitate colonies tentatively identified as $\alpha\text{Fe}_3\text{Si}$.⁴ The precipitate colonies and the conditions under which they nucleate and grow are described in detail in ref. 4 and will not be repeated here.

2. EXPERIMENTAL

The silicon crystals were obtained from Texas Instruments Company. The material was graded as Lopex (low oxygen, dislocation free silicon). The crystals were Czochralski grown in the form of cylindrical rods of 1 in. dia. x 6 in., with [111] growth axis. The resistance of the crystals was 50 Ω -cm and the conductivity was n type. For details of the specimen preparation see reference 4. The experiments were done on a Hitachi high voltage microscope, equipped with a hot stage, and operating at 650 kV.

3. RESULTS AND DISCUSSION

3.1 Precipitation on Dislocations

Figure 1 shows three micrographs of a $\langle 100 \rangle$ type precipitate colony⁴ in a (110) foil. The dendritic shaped colony is located on the [010] plane and the enveloping dislocation has a [010] Burgers vector. The effect of electron radiation damage while under observation in the microscope was to cause further precipitation around the edges of the colony in the form of fine whiskers. The micrographs were all recorded within a period of about 10 minutes, during this time the new whisker-like precipitate structure had developed along the dislocation line. As this precipitation was limited to areas of the foil illuminated by the electron beam, it was concluded that this effect is caused by the high energy electrons. The 650 kV beam voltage is far above the threshold voltage required for an electron to eject a silicon atom from a lattice position by direct collision. For silicon this threshold energy can be estimated to be about 170 keV.⁵

The time intervals between the three exposures in Fig. 1 are only a few minutes. Considering that the foil temperature is at most a few 100°C, the precipitation has occurred very rapidly. The precipitates were formed on the outside of the enveloping dislocation line only, i.e. on the tension side of the interstitial edge loops. The stress field of the dislocation may cause this preferential precipitation outside the loops, in addition the area inside of the dislocation is probably already depleted of the precipitating species.⁴ Perhaps because of the large Burgers vector, this effect is much more pronounced for a $\langle 100 \rangle$ than for a $\langle 110 \rangle$ Burgers vector dislocations.

Individual precipitates cannot be resolved, but they appear to be closely spaced fine needles growing in a direction normal to the dislocation line.

As the colony in Fig. 1 extends almost from one surface to the other it can be concluded that this radiation induced precipitation occurs with equal strength throughout the thickness of the foil. The whisker-like precipitates have not been identified, but as the individual precipitates in the colony have been tentatively identified as $\alpha\text{-Fe}_3\text{Si}$,⁴ it is suggested that the electron beam causes additional iron precipitation.

3.2 Radiation Damage at Higher Temperatures

A different mode of radiation induced precipitation occurred during annealing of a foil in the high voltage electron microscope hot stage. Typical results are presented in Figs. 2a to d. Figure 2 shows that many long needles have been formed after annealing for about one hour and fifteen minutes at about 700°C. This foil contained, in addition to the colony shown in Fig. 2, several other precipitate colonies. However, needles were formed only in the colonies illuminated by the electron beam. Thus, this precipitation effect, like the one described above, was caused by electron radiation damage.

The needle growth rate was strongly temperature dependent. The diagram in Fig. 2e shows the heat treatment given to the foil. Figure 2a and d are taken before and after the heat treatment respectively, the two other exposures are indicated in the diagram. The micrograph in Fig. 2b was recorded after about 60 minutes at 670°C, only a few needles had developed. By raising the temperature to about 750°C the nucleation and growth rate of the needles was greatly increased, Fig. 2c.

The contrast oscillations along the needles marked by arrows in Fig. 2c show that these needles were strongly inclined to the foil surfaces. All needles were oriented in $\langle 110 \rangle$ directions. The very long ones were parallel to the foil surfaces. Some of the needles were up to 10 μm long, the diameter varied from about 40 $\text{\AA}$ to about 200 $\text{\AA}$.

Figure 2b also suggests that some of the needles may have been nucleated at the dislocation line. However, Fig. 2c and d show that needles have been nucleated in regions outside the colony, thus this radiation induced needle precipitation is expected to take place also in the as received material. No identification of these needles has yet been made. However, further iron precipitation again seems like the most promising possibility.

ACKNOWLEDGEMENTS

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REFERENCES

1. M. J. Makin and J. V. Sharp, J. Matls. Sci. 3, 360 (1968).
2. V. E. Cosslett, Modern Diffraction and Imaging Techniques in Materials Science, S. Amelinckx, R. Gevers, G. Remaut and J. Van Landuyt, editors, (North-Holland Publishing Company 1970) p. 341.
3. G. Thomas, Phil. Mag. 17, 1097 (1968).
4. E. Nes and J. Washburn
5. M. J. Makin, Phil. Mag. 18, 637 (1968).

FIGURE CAPTIONS

Fig. 1. Precipitation along the dislocations due to electron radiation damage. The colony arm is of $[010]$ type and the three micrographs were exposed at the time intervals of a few minutes.

Fig. 2. Hot stage observations showing the formation of needle-like precipitates due to radiation damage. (a) $\langle 110 \rangle$ type precipitate colonies in a (111) foil, the micrograph is taken before the heating started. (b) After annealing the foil for about 60 minutes at 670°C . (c) This micrograph was taken about 10 minutes later than exposure b at 750°C , note the contrast oscillations along the needles marked by arrows. (d) Taken after cooling down to room temperature. (e) Diagram showing the heat treatment of the foil.

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Figure 1

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Figure 2a and b

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Figure 2c and d

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Figure 2e

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