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Vacancy Tetrahedra in Copper due to Electron Irradiation in the High-voltage Microscope

By M. IPOHORSKI† and M. S. SPRING‡
Metal Physics Group, Cavendish Laboratory, Cambridge

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ABSTRACT

Under suitable conditions of irradiation in the high-voltage microscope, stacking-fault tetrahedra have been found to form in copper specimens. These defects are thought to be of vacancy type, in contrast to the more usually observed displacement damage, which is of interstitial type.

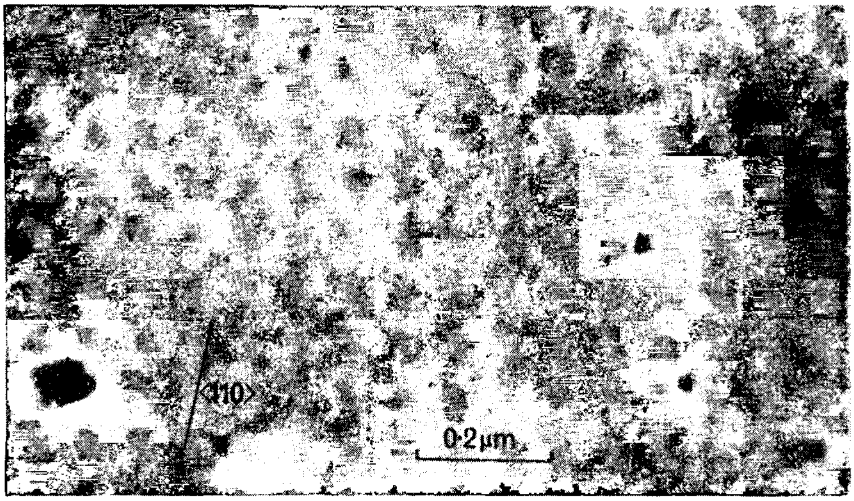
RECENTLY, there has been considerable interest in radiation damage in metals produced by the displacement reaction during observation in the high-voltage electron microscope. The damage takes the form of point-defect clusters whose size varies with the conditions of irradiation. Careful study of the clusters shows a homogeneity of size and form, suggesting the presence of a single population of one type of defect. Generally, the damage clusters have been assumed to be of interstitial type (Makin 1968) because the mobility of the interstitial point-defect is much higher than that of the vacancy; the larger clusters have been determined to be of interstitial type (Ipohorski and Spring 1969). Most of the vacancies generated by the displacements are annihilated by collision with interstitials; the remainder have usually been assumed to remain in the foil as single vacancies or small voids, since no evidence of visible vacancy clusters has previously been reported. It is also interesting to note that irradiation of aluminium containing small vacancy clusters produced by quenching and annealing prior to observation causes these clusters to shrink and disappear (Shoaib and Segall 1970). This note describes observations made on the Cambridge high-voltage microscope which suggest that visible vacancy type clusters may be produced in copper under suitable conditions of irradiation.

The specimens used were standard 2·3 mm diameter discs of Johnson Matthey 99·999% pure copper single crystal, which were irradiated with a beam of 1 μamp in a 5 μm diameter spot at 600 kev. Figures 1 (*a*) and 1 (*b*) show the effect of irradiating thin (*ca.* 50 nm) areas at a temperature of 200°C for 20 min. (The Ward (1967) double tilting and heating specimen

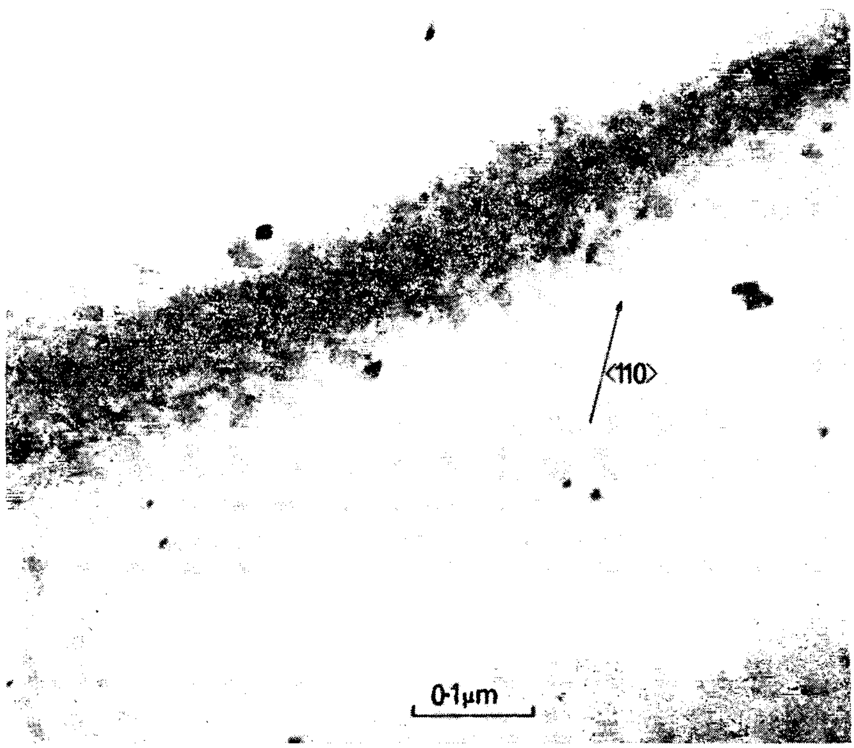
† Permanent address: Departamento de Metalurgia, Comisión Nacional de Energía Atómica, Buenos Aires, Argentina.

‡ Present address: Architecture Research Unit, University of Edinburgh.

Fig. 1



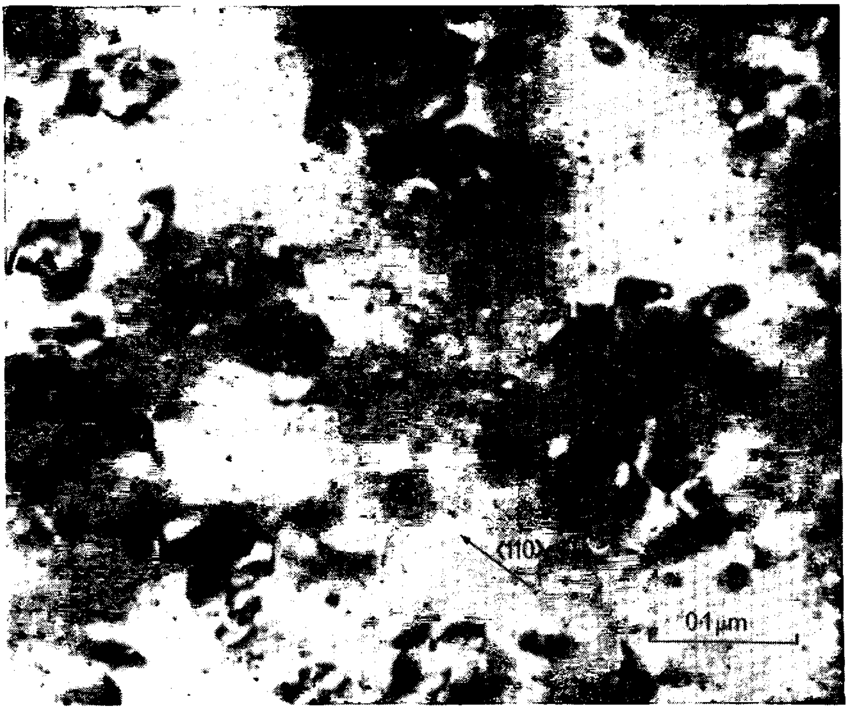
(*a*)



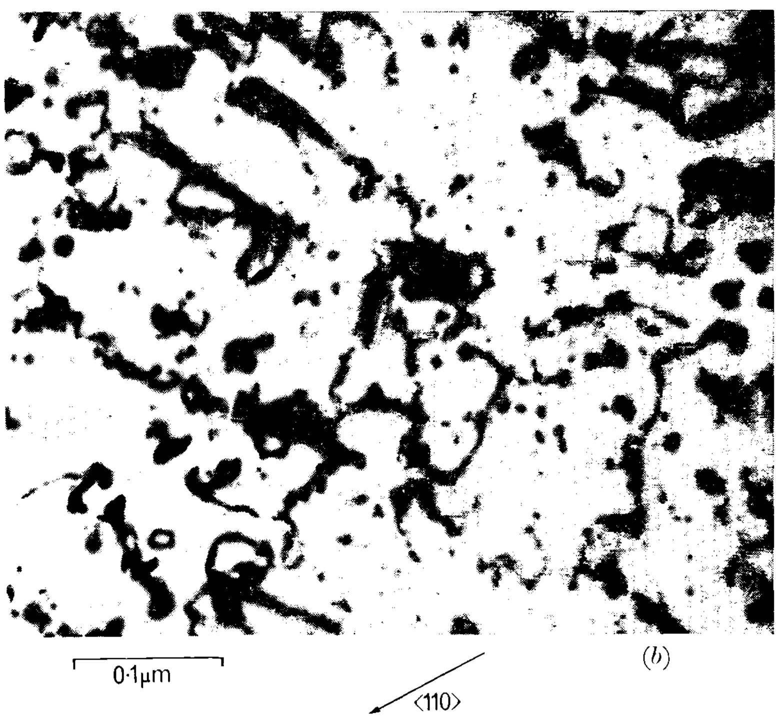
(*b*)

Areas of pure copper irradiated at 200°C: ⟨110⟩ foil normal: thickness about 50 nm.

Fig. 2



(*a*)



(*b*)

Areas of Cu 0·1 at. % Si irradiated at *ca.* 200°c; thickness 250 nm. (*a*) ⟨110⟩ foil normal, (*b*) ⟨100⟩ foil normal.

cartridge was used for these experiments.) A small number of stacking-fault tetrahedra can be seen, some of which are of the double type previously observed by Eades and Rojo (1968) in quenched gold specimens. These defects are thought to be of vacancy type because:

(i) The surface denuded layer for interstitial defects is about 60 nm at each surface at 20°C (Ipohorski and Spring 1969) and is expected to increase as the interstitial velocity increases with temperature (Brown 1969), making it unlikely that the defects are of interstitial nature.

(ii) In thicker foils under these irradiation conditions, the interstitial defects take the form of very large (up to 1 μm) faulted loops, quite different in appearance from the stacking-fault tetrahedra. A low density of interstitial defects is to be expected on theoretical grounds under high temperature conditions (Brown, Kelly and Mayer 1969).

(iii) All previous observations of stacking-fault tetrahedra have been of vacancy type.

Small quantities of impurity atoms also appear to encourage the formation of vacancy clusters. Figure 2 (*a*) shows a $\langle 110 \rangle$ normal foil of copper containing 0·1 at. % silicon, irradiated at *ca.* 200°C. There are quite clearly two distinct populations of defects: large irregular dislocation loops and small defects which are triangular in this section. $\langle 100 \rangle$ foils show a square section for these small defects (fig. 2 *b*), consistent with $\{111\}$ tetrahedra. The existence of two populations of large and small defects strongly suggests that the large loops are of interstitial origin, and the small tetrahedra of vacancy type. Similar behaviour has been observed in copper containing 0·1 at. % beryllium.

The results above are consistent with the assumption that for irradiation of thick ($\sim$150 nm) specimens of pure copper at room temperature, vacancies remain in the foil as single point-defects or very small clusters which are unable to grow because the vacancy velocity is much less than the interstitial velocity. Thus the number of interstitials arriving at an incipient vacancy cluster is, for moderate times of irradiation, greater than the number of vacancies arriving. If the interstitial concentration is reduced by using a thin specimen, and the vacancy velocity is increased by raising the temperature of irradiation, visible vacancy clusters are able to form. The role of impurity atoms in vacancy cluster formation is not clear, but the impurities may well act as nucleating agents for vacancy clusters as they do for interstitial clusters, besides possibly favouring the formation of tetrahedra from voids; tetrahedra give much stronger microscope contrast and are expected to be more resistant to attack by interstitials.

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