Reprinted from
PHILOSOPHICAL MAGAZINE, Vol. 20, No. 167, p. 937, November 1969

Electron Radiation Damage in a High Voltage Electron Microscope

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[Received 12 June 1969 and after revision 6 August 1969]

ABSTRACT

The factors affecting the nucleation and growth of electron displacement damage in copper are investigated in a high voltage electron microscope. Under certain conditions large faulted loops ($\sim 1\ \mu$) can be obtained. The contrast of the fault fringes enables them to be identified as interstitial in character. The first observation of the unfaulting reaction for extrinsic loops is reported.

THE formation of point defects by electron displacement damage in the Cambridge 750 kv microscope and their subsequent aggregation into loops were first studied by Makin (1968). The present note reports the positive identification of the sign of the damage loops and the effect of the various parameters influencing their formation.

The appearance of the damage in a given material is affected by the precise experimental conditions within the microscope which, because they are not easy to control, makes quantitative deductions about the damage mechanism rather difficult. In particular, the following factors have been found to be important:

(*a*) The *magnitude of the electron flux* striking the specimen affects the growth and resultant size of the loops. Previous measurements of the current were made between the two condenser lenses. To measure the beam current at the specimen level, a Faraday cage was constructed to fit onto the top of the specimen holder. It was found that for a fixed current at the condenser level, the flux at the specimen was critically dependent upon the precise alignment of the microscope, and that continual adjustments of this alignment were essential if a high flux at the specimen was to be maintained. With a 750 μm condenser aperture and a beam current at the condenser level of 4 μA, a flux at the specimen of about 1·5 μA could be obtained. If the alignment was not frequently adjusted, however, this could fall to a value as low as 0·2 μA in a period of time as short as 20 min, with little noticeable effect on the visual intensity on the screen.

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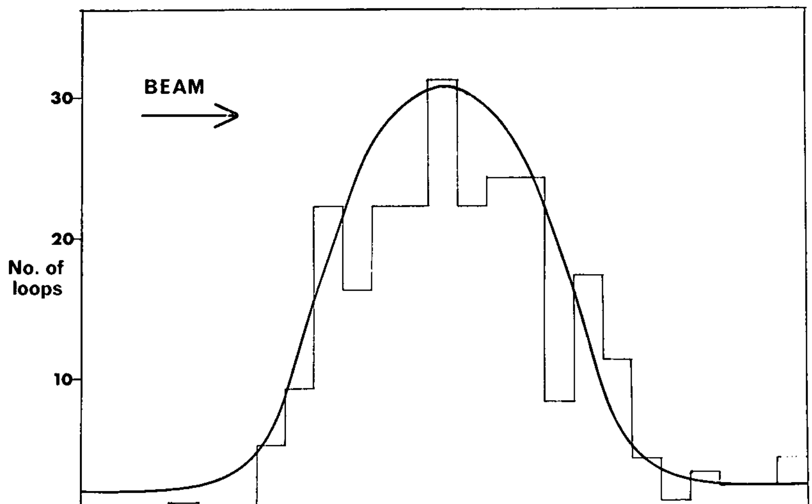
(*b*) *Contamination* films formed on the specimen in the microscope apply stresses to the foil (Pashley and Presland 1962) affecting the stability of the damage loops.

(*c*) The *temperature* of the area under observation is difficult to determine or control; temperatures as high as the melting point of copper have been observed to occur under the normal operating conditions of this microscope (W. M. Stobbs, private communication). It is, however, in general difficult to separate the effects of temperature from those of contamination.

(*d*) In addition, the *thickness* of the area under observation plays an important part. Damage is not observed in thin regions (less than about 1000 Å in copper) suggesting that a fairly wide denuded layer exists near the specimen surfaces. Stereo measurements of a damaged area in pure copper (Johnson Matthey 99·998%) 2500 Å thick show the denuded layer to be about 600 Å wide (fig. 1) although this value appears to be rather variable presumably as a result of temperature variations. In all cases the denuded layer is sufficiently wide to make sign determinations by the strain contrast method very difficult.

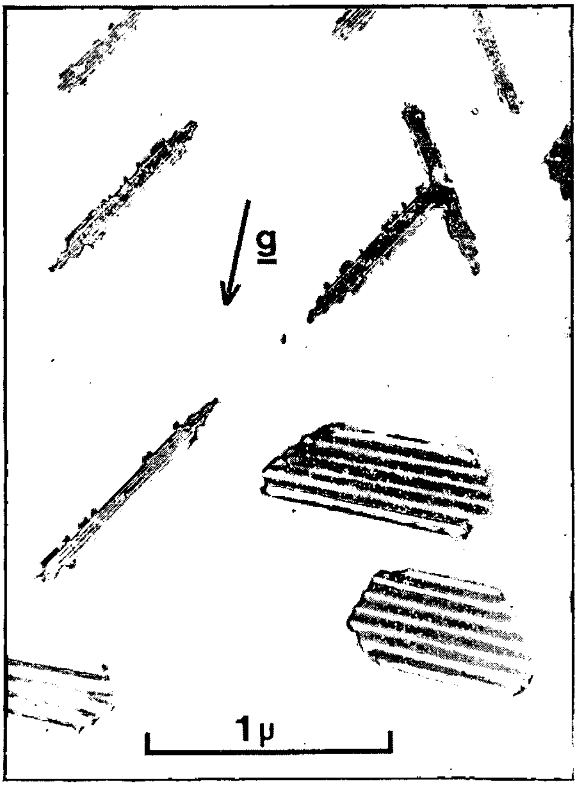
Fig. 1



Distribution of loops in a copper foil 2500 Å thick irradiated with 600 kv electrons.

Under particular combinations of these factors, much larger loops than have previously been observed can be obtained (fig. 2). The loops are initially faulted; occasionally the unfaulting reaction is observed (fig. 3 *a* and 3 *b*). Stereo techniques indicate that the loops lie on the four {111} planes; loops with dimensions as large as 1 μm have been observed. The production of these large loops requires a high beam current ($\sim 1\ \mu$A in a $4\ \mu$m diameter spot) at the specimen, and they can only be grown in clean or lightly contaminated specimens. When a heavily contaminated specimen is irradiated with a high beam current the loops are seen to slip out of the foil before they grow to large size, indicating the importance of stresses due to contamination. These large loops are observed particularly in areas whose thickness does not greatly

Fig. 2

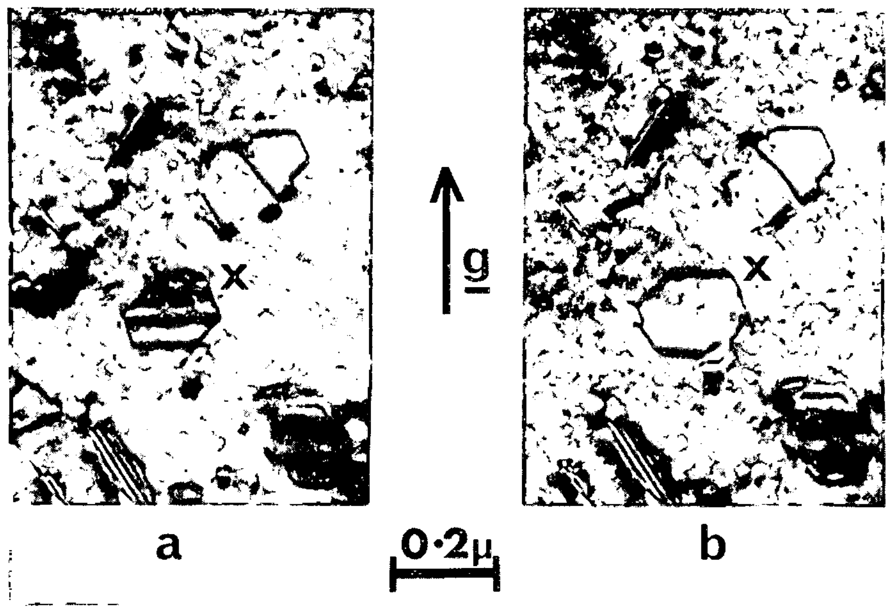


Large faulted loops in pure copper irradiated at 600 kv (110) foil normal; (002) reflection, $s \geqslant 0$.

exceed twice the denuded layer width. If these conditions are not all satisfied, damage similar to that observed by Makin or some intermediate stage is observed. In particular, the occurrence in some cases of large loops near the edge of a specimen changing continuously to the small loops in thicker regions, indicates that the large loops are of the same nature as those observed by Makin, development to large dimensions occurring only when all the conditions are favourable.

After prolonged irradiation, most of the large loops on the two {111} planes making an angle of ~35° to the {110} foil (near to the plane of maximum resolved shear stress at 45°) will eventually slip, leaving predominantly loops whose normal is parallel to the foil. These loops, nucleated in the centre region of the foil, can be grown until they intersect the specimen surfaces and the sign then determined by examination of the contrast of the first fringe at the top surface (Hashimoto, Howie and Whelan 1960). Figure 4 shows one of the loops examined; the strain

Fig. 3

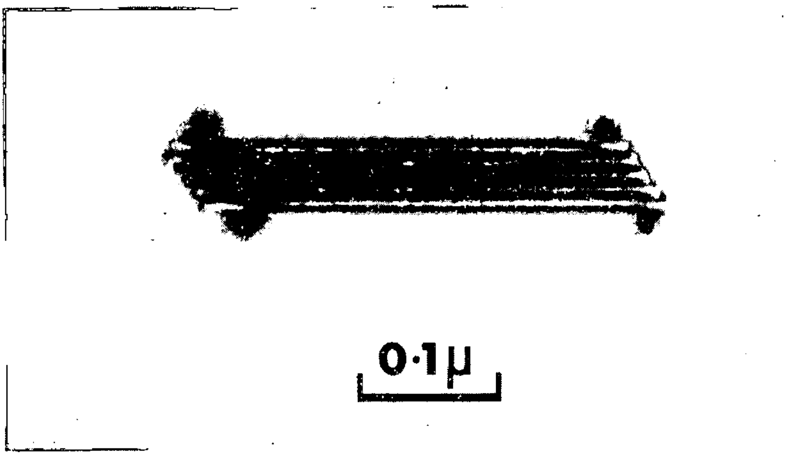


The unfaulting reaction; loop X is faulted in (*a*), and unfaulted in (*b*) shortly after. (110) foil normal; (002) reflection, $s \geqslant 0$.

contrast where the two partial dislocations bounding the loop reach the foil surfaces is clearly visible. The top edge of such loops was determined both from bright field-dark field pairs and by stereo methods. About twelve loops in different specimens were examined; all were found to be *interstitial* in character, in accordance with the picture proposed by Makin. Since the loops are interstitial, the unfaulting reaction is especially interesting as this is, to the author's knowledge, the first experimental evidence that the Shockley partial bounding an extrinsic fault is mobile.

As the vacancies are less mobile than the interstitials, a large number of them must remain in the foil. No evidence of their presence in pure copper has been seen at the present resolution—however in Cu doped with certain impurities, small tetrahedra have been observed (~30 Å) in addition to the large loops. Higher resolution work is envisaged in an attempt to discover whether such tetrahedra or other vacancy clusters can be found in the pure material. The effect of impurities on the nucleation of the interstitials is also being investigated.

Fig. 4



Faulted loop intersecting the foil surfaces.

ACKNOWLEDGMENTS

The authors are indebted to Dr. V. E. Cosslett and his team for the use of the high voltage microscope, and to Dr. M. J. Goringe and Dr. L. M. Brown for helpful discussions and suggestions.

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