CORRESPONDENCE

The Shape of Frank Loops Studied by High-voltage Microscopy

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Abstract

It is shown that extrinsic Frank loops exist in two different forms: one has ⟨110⟩ edges and sharp corners; the other has ⟨112⟩ edges and rounded corners. The former results from growth of the loop, and is stabilized by dissociation of the dislocation core. The latter form results from shrinkage of the loop, and is stabilized by the elastic energy of the dislocation. The observations imply that the absorption of vacancies by a loop occurs by a rate-controlling process different from the absorption of interstitials.

Calculations by Brown (1967) on the basis of anisotropic elasticity suggest that the equilibrium shape of Frank dislocation loops in copper may be hexagonal with somewhat rounded edges along ⟨112⟩ directions in {111} planes. Observations of such loops have shown them to be either irregularly shaped or to adopt a form with straight edges along ⟨110⟩ directions in {111} planes. Previous studies in copper include the observation of proton-irradiated copper (Mazey and Barnes 1968) and quenched copper (Clarebrough, Segall and Loretto 1966). In addition, some observations by Dobson, Goodhew and Smallman (1967) in quenched aluminium show that Frank loops in this material are of the ⟨110⟩ type when formed, but change to a circular shape on annealing. The calculations of Brown predict that in aluminium the loops will have minimum elastic energy if they are almost circular. The elastic equilibrium shapes for copper and nickel resulting from Brown's (1967) calculations are shown in fig. 1; aluminium is elastically so isotropic that the equilibrium shape differs by less than 0·2% from a circle.

The high-voltage microscope provides a very useful means of studying the shapes of Frank loops. It has been shown (Ipohorski, Spring and Goringe 1972) that under suitable irradiation conditions at elevated

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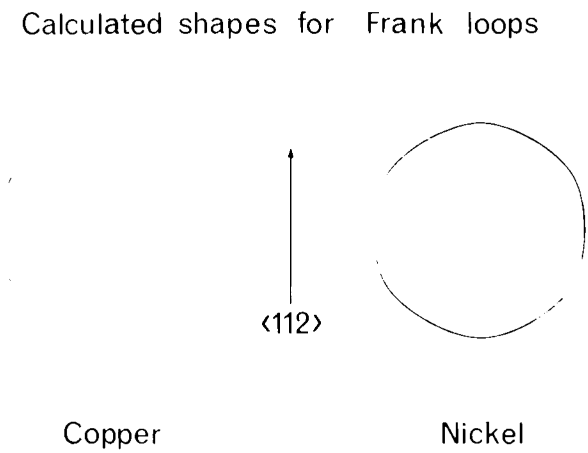
temperatures Frank loops of up to 1 μm in diameter can be grown. These loops result from clustering of interstitials produced by the electron irradiation. With a high-temperature microscope specimen stage, the loops can be made to grow or shrink, and the flux of vacancies and interstitials can be crudely controlled by changing the electron beam intensity and voltage.

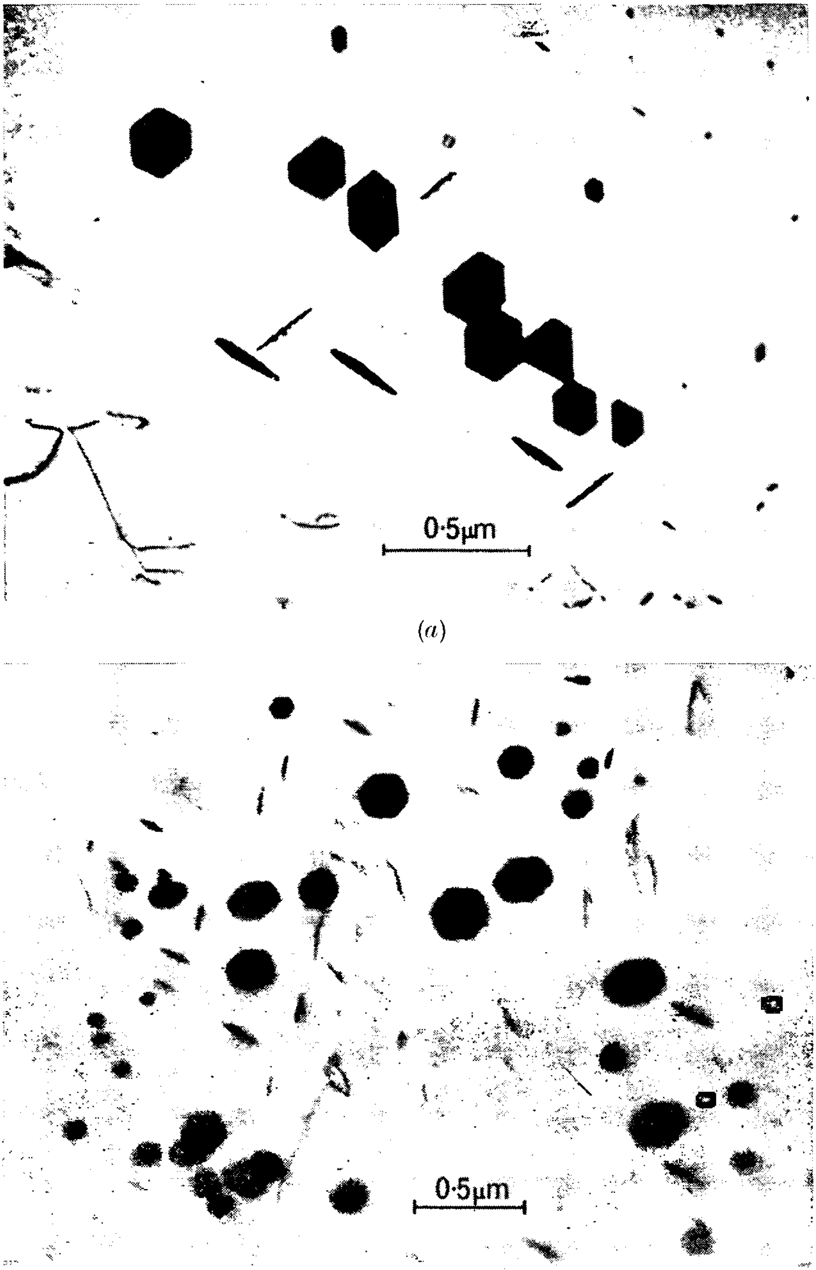
Fig. 1



The equilibrium shape of Frank loops in copper and nickel, computed by the Wulff construction on the basis of the elastic energy as described by Brown (1967). The shape for aluminium cannot be distinguished from a circle.

We wish to report the results of experiments on electron-irradiated copper. Spark-cut, annealed, disc specimens of Johnson-Matthey 99·999% pure copper were irradiated in the Cambridge high-voltage microscope with 600 kev electrons. Thick areas (approximately 300 nm thick or more) were chosen for irradiation, and the irradiation temperature was approximately 200°C. Large Frank loops were produced. These were often irregular in shape, probably due to stresses in the foil resulting from contamination (see Ipohorski and Spring 1969, also Mazey and Barnes 1968). In some specimens, however, good regular loops could be formed: they were always of the $\langle 110\rangle$-edged, sharp-cornered form (fig. 2 *a*). When such loops were allowed to anneal at the same temperature for about 10 min, away from the electron beam, they were transformed to the rounded $\langle 112\rangle$-edged form (fig. 2 *b*). Further electron irradiation very rapidly returned them to the original form and the process could be repeated several times, the loops eventually becoming unstable, unfaulting and slipping from the foil. Figure 3 shows a comparison of the equilibrium elastically-controlled shape from fig. 1 with the equilibrium shape of an annealed loop, correcting for viewing normal to the $\{111\}$ plane of the loop. The predicted and observed

Fig. 2



(*a*)

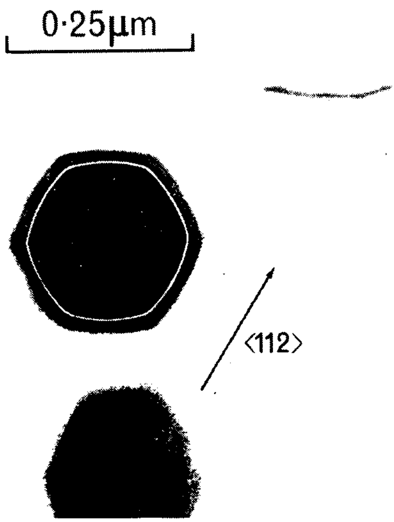
(*b*)

(*a*) Frank loops in copper as grown by electron irradiation at 200°c. The slip traces are parallel to 110 directions. (*b*) Frank loops in copper after annealing at 200°c for 10 min without irradiation. The slip traces are parallel to 110 directions.

shapes are in close agreement, although the observed shape is slightly more rounded than the theoretical shape.

From these experiments it is difficult to say with certainty whether the ⟨110⟩-edged loop is *unstable*, but unable to change shape because core diffusion is too slow; or *metastable*, requiring energy to be converted to the ⟨112⟩-edged form. It is clear that the ⟨112⟩-edged form is stable or metastable, as the calculations based on elasticity show.

Fig. 3



Comparison between observed and calculated loop shapes.

It seems probable that the ⟨110⟩-edged form is metastable, since earlier calculations (Brown 1967) suggest that the probable energy of a jog in the dissociated Frank dislocation is great enough to stabilize that form. When the loop is growing, jogs in the extended Frank dislocation run to the corners of the loop and it is forced into the ⟨110⟩-edged form. This clearly implies that the growth of Frank loops is controlled by the *nucleation* of jogs on the extended dislocation. However, when the loop is shrinking, jogs run in from the corners and the loop is forced towards the ⟨112⟩-edged form. When it is in that form, jogs are in plentiful supply. The Frank dislocation cannot dissociate when it is parallel to a ⟨112⟩ direction, so it would appear that the shrinkage of the loops is controlled by the *motion* of jogs in the undissociated Frank dislocation. The experiments clearly show that the absorption of vacancies occurs by a different process from the absorption of interstitials; or more generally, that growth and shrinkage of loops are not symmetrical processes.

When the jogs are more closely spaced than the length over which the extended Frank dislocation is constricted, the energy per jog will become very small, as it will not include the energy of constriction. Thus it is possible for both the ⟨110⟩ and ⟨112⟩ forms to be stable or metastable, and for the Frank loop as a whole to have two equilibrium (or, more correctly, meta-equilibrium) shapes. Clearly further studies of different metals, combined with theoretical analysis, could lead to a determination of the energy associated with jogs in Frank dislocations.

This note confirms and extends the preliminary account of this work presented at the VIIth International Congress on Electron Microscopy, Grenoble, 1970 (Spring and Brown 1970).

Note added in proof.—Observations of Frank loops in the ⟨112⟩ form were briefly reported by D. J. Mazey and R. S. Barnes, VIth Int. Cong. for Elect. Microscopy, Kyoto, 1966, p. 377. A fuller account is in preparation by Mr. D. J. Mazey.

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