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Radiation Induced Removal of Stacking Faults in Quenched Aluminium

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Abstract

The effect of neutron irradiation on specimens of quenched aluminium containing Frank sessile dislocation loops has been studied by means of electron microscopy. The Frank loops were found to transform into perfect loops at doses less than 10^{17} nvt. A possible reason for the removal of the stacking faults is the displacement of a number of atoms at the faults, leading to the passage of a Shockley partial. Unfaulting induced by stress fields from dislocations, released during the irradiation, can also be important.

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1. Introduction

Transmission electron microscopy has during recent years been extensively applied to the study of point defect clusters in many substances. In quenched aluminium perfect prismatic dislocation loops, Frank sessile loops (enclosing a stacking fault), double-faulted loops and voids can be identified [1]. Under suitable experimental conditions almost all loops in quenched aluminium are of the Frank type and exist in a metastable state in spite of the high value of the stacking fault energy [1, 2]. If quenched material containing vacancy clusters is irradiated some interaction between the vacancy clusters and the defects produced by the irradiation is expected, which perhaps can provide information about the damage process. The effect of neutron irradiation on aluminium after various treatments, including quenching, has been studied by Piercy and Whitton [3], but their results on quenched material seem to be inconclusive, however. A series of neutron irradiations and electron microscopic studies of quenched aluminium was therefore undertaken. Only room temperature irradiation facilities were available.

2. Experimental technique

Specimens were prepared from zone-refined aluminium in the form of pellets with a thickness of 0.5 mm and a diameter of 2.3 mm and were subsequently annealed for an extended length of time in a high vacuum. The specimens were drop-quenched a number at a time from approximately 560 ^{o}C into distilled water. The temperature of the quenching bath was adjusted to a few different temperatures in the range 10 ^{o}C to 30 ^{o}C in order to obtain specimens with dislocation loops of different mean sizes. Ageing after quenching proved to substantially increase the number of Frank loops present in the specimens. An ageing treatment of one hour at 125 ^{o}C prior to irradiation was adopted as a standard treatment . Electron microscopic examination of specimens in the quenched and aged state showed a fairly uniform distribution of Frank sessile dislocation loops, see Fig. 1. The proportion of perfect loops was generally less than 5 %, provided the specimens were very carefully annealed prior to quenching.

Quench-aged specimens were neutron irradiated in sets of 6 to different fast neutron doses from 10^{16} to 10^{19} nvt. Some annealed specimens were also irradiated to high doses. During the irradiation the specimens were sealed in helium-filled flat aluminium cans, surrounded by cooling water at 20 °C. Some of the neutron doses were checked by means of In and Ni monitors. Irradiated and corresponding unirradiated control specimens were examined by electron microscopy on the day following the day of irradiation.

3. Results

The smallest neutron doses left the microstructure apparently unchanged. After slightly increased doses, however, a change in loop character could be observed; i.e. perfect prismatic loops with $b = \frac{a}{2}\langle 110\rangle$ appeared instead of the Frank loops with $b = \frac{a}{3}\langle 111\rangle$. Micrographs from specimens irradiated to approximately $3 \cdot 10^{16}$ nvt (>0.2 MeV) are shown in Figs. 2 and 3. Some of the perfect loops formed apparently coalesce by combined glide and conservative climb processes as already noted by Hirsch et al. [4] in quenched aluminium. Clustering of loops having different $\frac{1}{2}\langle 110\rangle$ -type Burgers vectors was also observed (e.g. at A in Fig. 2). As seen from Fig. 3, most of the faulted loops remaining after irradiation to moderate doses are of the double-faulted type. The proportion of such loops prior to irradiation was of the order of 10 %. The behaviour of voids could not be followed. Small voids were observed after quench-ageing and after low-dose irradiations.

Irradiation to 10^{19} nvt caused some further changes in the specimen microstructure, see Fig. 4. The perfect loops assumed more irregular shapes and, in addition, small loops were formed close to the large loops. The small loops were probably formed from displacement cascades, the larger loops acting as sinks for interstitials, enhancing the damage. Well annealed control specimens did not show any resolvable defects after irradiation to doses greater than 10^{19}. Results of neutron irradiation of annealed aluminium reported in the literature are conflicting [5, 6]. The perfect loops tend to be oriented between the pure edge orientation and the {120} planes. Orientation on {120} planes was observed by Makin and Hudson in the case of parallelogram shaped loops [7]. Our results are summarized in the table below, where N_F and N_P are the numbers per

cm^3 of faulted and perfect loops respectively, D_F and D_P the corresponding mean diameters and C_V the total vacancy concentration determined from loop data. Specimens type A and B were quenched to 22 °C and 9 °C respectively.

Details of loops in quench-aged and irradiated specimens

Specimen type	Dose nvt	N_F cm^{-3}	N_P cm^{-3}	D_F Å	D_P Å	C_V
A	unirr.	$4 \cdot 10^{13}$	$1 \cdot 10^{12}$	1200	-	$0.9 \cdot 10^{-4}$
A	$3 \cdot 10^{16}$	$1 \cdot 10^{13}$	$2 \cdot 10^{13}$	1200	1200	$0.9 \cdot 10^{-4}$
A	10^{19}	0	$3 \cdot 10^{13}$	-	~1400	$1.3 \cdot 10^{-4}$
B	unirr.	$1 \cdot 10^{14}$	$5 \cdot 10^{12}$	600	-	$0.6 \cdot 10^{-4}$
B	$3 \cdot 10^{16}$	$1 \cdot 10^{14}$	$1 \cdot 10^{13}$	600	600	$0.6 \cdot 10^{-4}$

Bulk annealing experiments on the quench-aged specimens showed the Frank loops to anneal out by climb whilst maintaining their stacking faults in agreement with the annealing behaviour described in the literature [8]. The greater stability of double-faulted loops is also confirmed. Fig. 5 shows an area of a specimen annealed at 160 °C, where double-faulted loops have grown owing to the relatively high vacancy concentration, whereas regular Frank loops have shrunk.

The effect of deformation on the Frank loops was studied to some extent, since unfaulting of Frank loops due to moving dislocations has been observed in thin foils [9, 10]. Plastic deformation by compression to different amounts down to 2 % was found to cause sweeping out of all the Frank loops and the formation of very few perfect loops.

4. Discussion

The total number of interstitials formed at a neutron dose of the order of 10^{16} is quite insufficient to annihilate all the Frank loops. The following mechanisms are therefore conceivable to explain the change in loop character:

(a) Nucleation of perfect loops close to the Frank loops and growth of these perfect loops at the expense of the Frank loops.

(b) Transformation of the Frank loops into perfect loops by a glide mechanism:

$$\frac{1}{3}[111] + \frac{1}{6}[11\bar{2}] = \frac{1}{2}[110]$$

If new perfect loops were nucleated, the growth of some of them should be observed upon heating specimens in which Frank loops remain after irradiation. Transformation of Frank loops into perfect loops was actually observed in a few specimens when they were heated in the electron beam. The transformation appeared to be similar to the unfaulting occasionally observed in quenched unirradiated specimens, but in the irradiated specimens all the loops transformed. The change of loop character is therefore assumed to take place according to mechanism (b).

The propagation of a Shockley partial, which removes the stacking fault, has been discussed by Cotterill [1] and by Saada [2]. As the partial expands the total energy first increases and then passes through a maximum at a certain critical size, above which the partial continues to grow spontaneously. Thermal activation alone is not sufficient to cause any appreciable unfaulting of Frank loops up to temperatures at which the loops disappear by climb.

Since the irradiations were performed at room temperature a part of the point defects formed will certainly migrate to the Frank loops. The diffusion of point defects to the loops is not expected to have any drastic effect on the character of the loops, however. If the unfaulting is caused by the displacement cascades, it must be assumed to occur by the displacement of a number of atoms at the stacking faults, leading to the formation of a Shockley partial of sufficient size. On the further assumption that one primary collision event causes unfaulting of a loop if it occurs within a certain range from a loop, this range can be estimated from the dose at which unfaulting of loops of a certain size is first observed. As a rough estimate a range of 80 Å is obtained, which is of the expected order of magnitude of the radius of a displacement cascade if long range effects, for example channeling, are not taken into account. The above point of view is supported by the effect of a change in average loop size. The same neutron dose which caused unfaulting of the majority of the loops in type A specimens was found to have a very limited effect on the smaller loops in the type B specimens.

The possibility that the Frank loops are converted into perfect loops by dislocations moving close to the loops or intersecting the loops cannot be excluded, however. Dislocations can perhaps be locked by small vacancy clusters when the specimen is subjected to quenching stresses and subsequently be released during the irradiation. An attempt to decrease the effect of stresses was made in a separate experiment by electropolishing a number of quenched specimens almost to the final thickness prior to irradiation. The result of the irradiation was, however, very similar to that in 0.5 mm thick specimens.

The effect on material purity and quenching conditions on the character of the dislocation loops formed in aluminium has been discussed in several papers [11, 12, 13]. If dislocation movements cause removal of stacking faults in the present case, the extent of dislocation movements may also influence the results of quenching experiments on aluminium.

Acknowledgement

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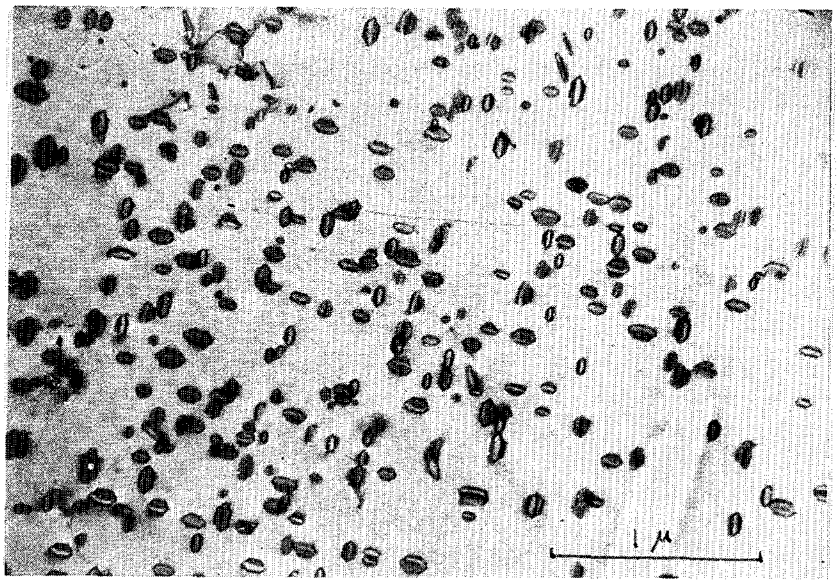

Fig. 1. Area of quench-aged specimen. Specimen orientation [100].

Fig. 2. Quench-aged specimen irradiated to $3 \cdot 10^{16}$ nvt, showing faulted and perfect loops. Clusters of loops at A. Specimen orientation [110].

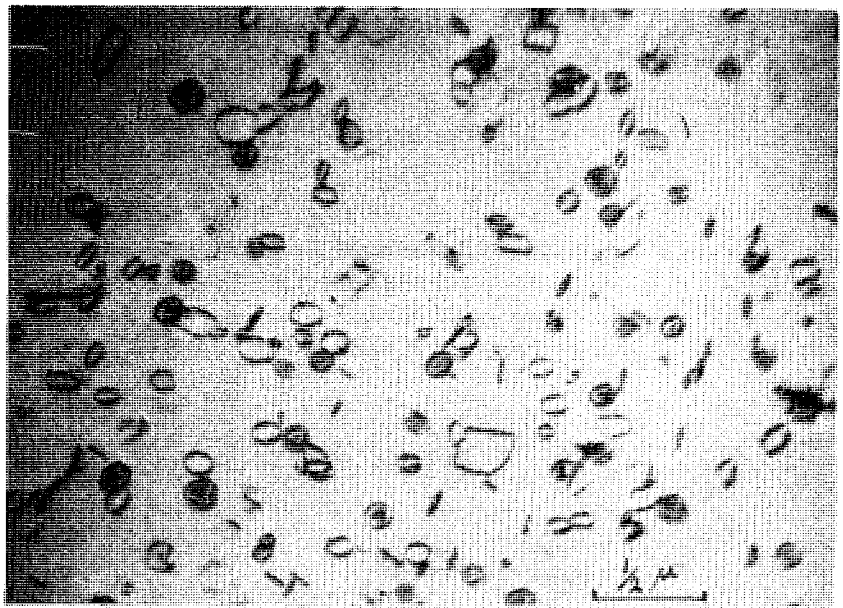

Fig. 3. Quench-aged specimen irradiated to $3 \cdot 10^{16}$ nvt, showing double-faulted and perfect loops.

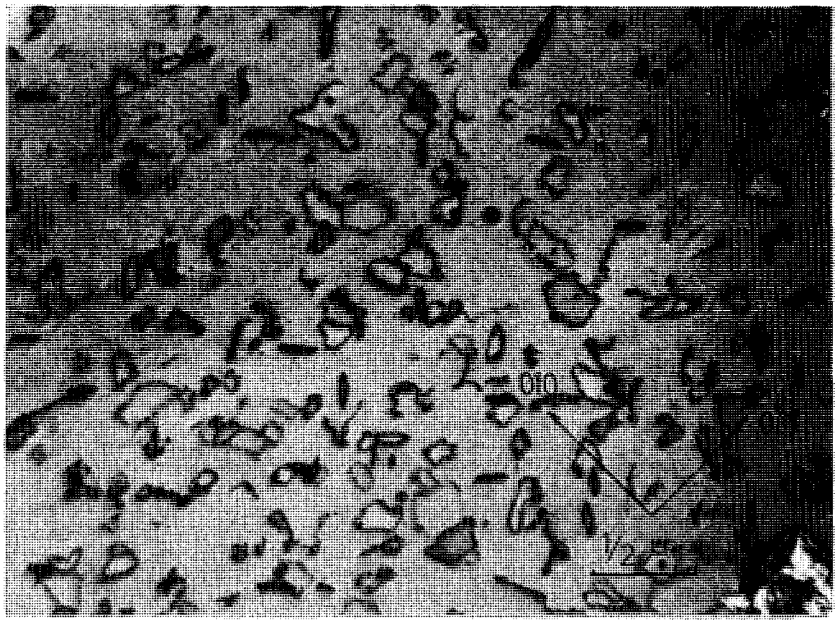

Fig. 4. Quench-aged specimen irradiated to 10^{19} nvt, showing perfect loops and small clusters. Specimen orientation [100].

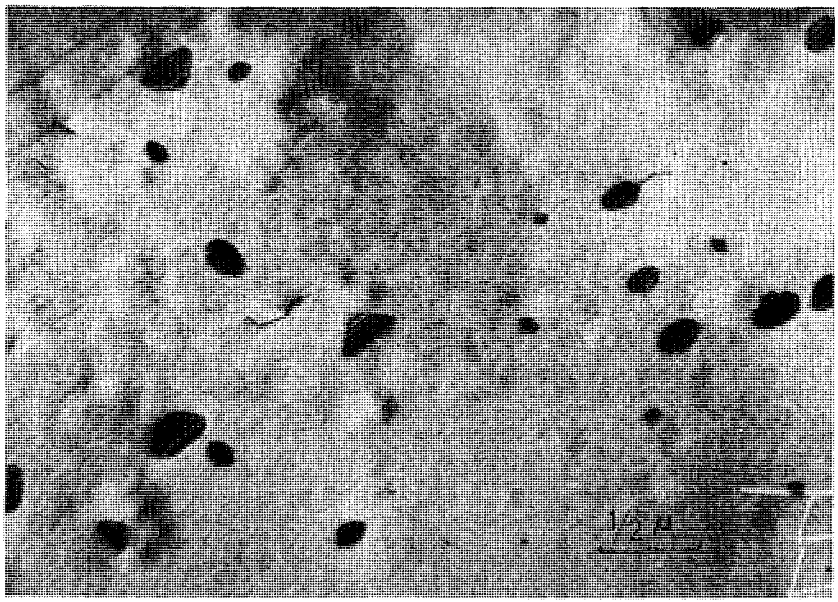

Fig. 5. Quench-aged specimen annealed at 160 $^{\circ}$C for 10 minutes. Specimen orientation [100].

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