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Annealing twinning and the nucleation of recrystallization at grain boundaries

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Experimental evidence is presented which shows that, in three different low stacking fault energy materials, annealing twins form at grain boundaries during the very early stages of recovery following deformation. These observations provide the basis for the suggestion that twinning at grain boundaries during recovery might stimulate nucleation of recrystallization in low stacking fault energy materials. The experimental observations also lead to the implication that the density of recrystallization nuclei formed in such materials may be directly related to the strength of the deformation texture.

1. Introduction

The formation of annealing twins during recrystallization and grain growth is an important feature in the development of the final microstructure of many face centred cubic (f c c) materials of low stacking fault energy.

The work of Peters [1] on a Cu–10 wt % Sn alloy and of Peters and Reid [2] on an AISI 310 steel, for instance, has demonstrated the importance of higher order twins to the development of annealing textures during grain growth following recrystallization. Other results, concerning the importance of chains of higher (up to fourth) order twins to the development of recrystallization textures, have been reported for deformed and annealed Ag single crystals by Hoekstra *et al.* [3]. Recent data on Cu, in this case obtained through high voltage electron microscopy (HVEM) studies [4], have again emphasized the importance of twinning in the recrystallization process and in the development of texture.

These and other investigations have tended to provide evidence of the importance of annealing twinning at or beyond the stage of recrystallization where nuclei have become well established. However, very little information is available which indicates whether or not annealing twinning may actually be instrumental in the successful development of individual recrystallization nuclei.

Recently, however, Huber and Hatherly [5, 6] have produced experimental evidence of what they have termed "recovery twinning" in brasses. These authors have suggested that intragranular bundles of very fine annealing twins which form during the early stages of recovery may, by subsequent growth, lead to the development of recrystallization nuclei.

In a previous paper [7] it was suggested that twinning decomposition at grain boundaries in deformed low stacking fault energy materials might facilitate nucleation of recrystallization. Experimental observations which support this earlier suggestion are presented and discussed in the following sections. These observations are from three different low stacking fault energy materials.

2. Experimental details

Three different alloys were investigated during the current work. They were an AISI 310 stainless steel, a 20 wt % Cr, 25 wt % Ni, 0.97 wt % Nb steel containing 0.097 wt % (C + N), and a texture free α-brass containing 15 wt % Zn. Specimens of both types of stainless steel were initially given a solution treatment anneal.

A variety of different cold rolling and subsequent annealing treatments was used to produce microstructures characteristic of the very early stages of recrystallization. Specimens of AISI 310 were cold rolled by either 20% or 50% reduction in thickness and the Nb stabilized steel was cold rolled by 20%, prior to annealing. Specimens of the texture free brass were given a cold rolling of 50% reduction in thickness. Following defor-

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mation, stainless steel specimens were annealed for suitable times at 1173 K, while brass specimens were annealed at 573 K.

Electron microscopy of thin foils produced by standard techniques was performed on a JEOL 100C instrument.

3. Experimental results

3.1. General

All three of the materials investigated contained deformation structures characteristic of lower stacking fault energy materials, including deformation twins and the general absence of any well defined cell or subgrain structures.

For both types of stainless steel the recrystallization anneals led to precipitation at grain boundaries prior to any detectable nucleation of recrystallization. In the AISI 310 steel large $M_{23}C_6$ precipitates were formed, while in the Nb stabilized steel high densities of much smaller grain boundary Nb(CN) were precipitated.

One further feature of the Nb stabilized steel was the presence of a distribution of large relatively equiaxed Nb(CN) particles which had not been dissolved during the solution treatment anneal. Some of these particles were several μm in diameter and, together with some of the quite small (0.2 μm diameter) undissolved Nb(CN) particles, were found to be preferential sites for the nucleation of recrystallization. Frequently, those small (~ 0.2 μm diameter), Nb(CN) particles which did not act as nucleation sites were centres around which there was accelerated recovery, leading to local formation of small, relatively sharply defined subgrains.

In the 50% cold rolled and annealed α-brass specimen, a number of examples were found of the clusters of "recovery twins" reported by Huber and Hatherly [5, 6]. At the correct $\langle 1\,1\,0 \rangle$ matrix orientation, diffraction patterns of such areas showed streaking along $\langle 1\,\bar{1}\,1 \rangle$ directions with intensity maxima at twin positions.

These clusters of "recovery twins" were not observed as frequently as appears to have been the case in the alloys investigated by Huber and Hatherly [5, 6]. However the applied level of deformation in the current work was also somewhat smaller than those which were employed by Huber and Hatherly [5, 6].

Finally, in all three materials, in addition to the types of grain boundary nucleation processes documented in the next section, normal microbulging processes [8, 9] were also observed.

3.2. Twinning at boundaries

Fig. 1a shows a small region of grain boundary in a specimen of AISI 310 steel which had been deformed 50% and annealed for 60 sec at 1173 K. The small bulge which can be seen in this grain boundary, impinged at both ends on large boundary

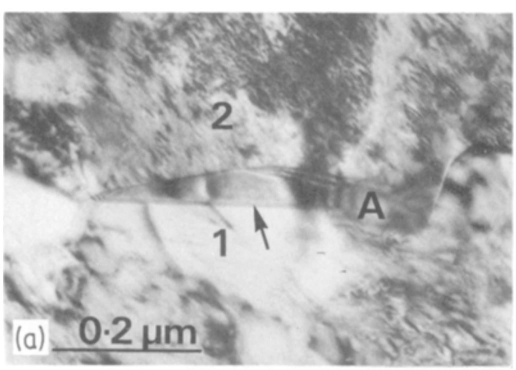
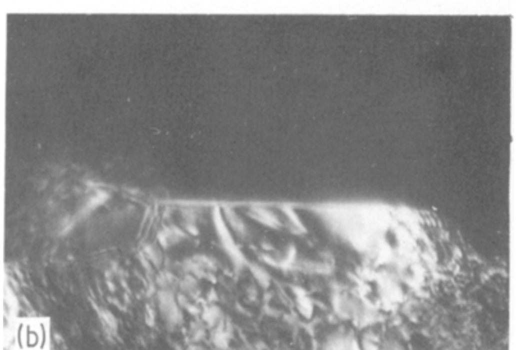
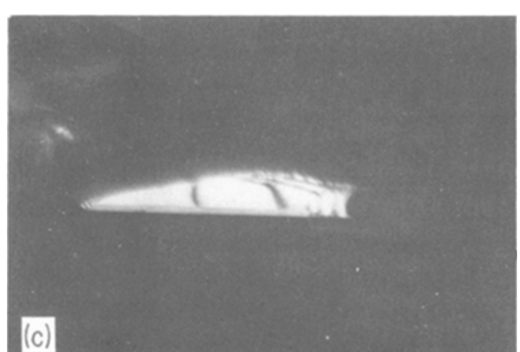


Figure 1 A grain boundary region in AISI 310, 50% deformed and annealed for 60 sec at 1173 K: (a) small twinned volume at the boundary, with the coherent twin interface arrowed and an $M_{23}C_6$ precipitate visible at A, (b) 2 0 0 matrix dark-field image of the region shown in (a) and (c) $1\,\bar{1}\,\bar{1}$ twin dark-field image of the region shown in (a).





$M_{23}C_6$ precipitates, one of which is clearly visible at A. Using dark-field microscopy, the orientation of the small, low dislocation density volume within the bulge was confirmed to be the twin of that in grain 1. The interface (arrowed) which constituted the base of the bulge was the coherent twin boundary. The 2 0 0 matrix and $1\,\bar{1}\,\bar{1}$ twin centered dark-field images shown in Fig. 1b and c, respectively, illustrate more clearly the separate orientations of the matrix and twin related bulge regions. This example indicates that twinning at grain boundaries can occur at a very early stage in the local recovery that characterizes the early stages of a recrystallization anneal and, further, that such twinning can be associated with the formation of small bulges on grain boundaries.

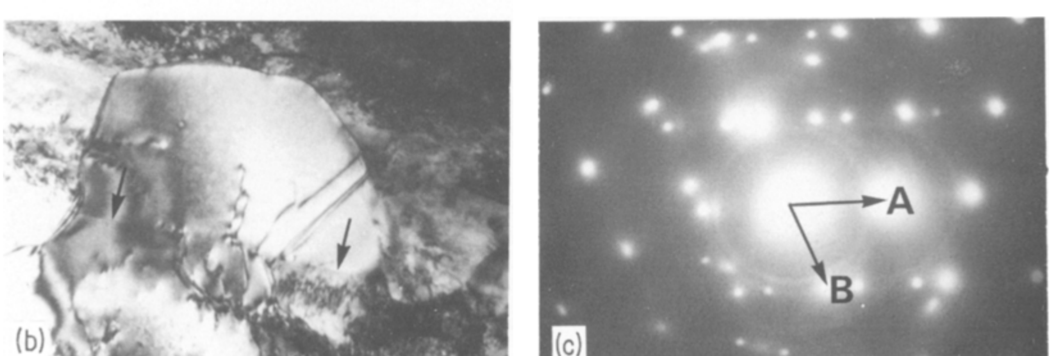
Both of the parent grains shown in Fig. 1 (grains 1 and 2) had $\langle 0\,1\,\bar{1}\rangle$ virtually parallel to the foil normal and, therefore, parallel to the rolling plane normal. In addition, both grains exhibited cumulative changes in orientation (mainly around $\langle 0\,1\,\bar{1}\rangle$) between the grain boundary and grain centre regions, being $\sim 16^\circ$ over a distance of $5\,\mu$m in grain 2 and $\sim 25^\circ$ over $10\,\mu$m in grain 1. As might have been expected, the misorientation across the boundary between grains 1 and 2, differed from point to point along its length.

A further example showing the formation of a twin oriented bulge on a grain boundary during the early stages of recrystallization is given in Fig. 2. In this case, the material was 50% rolled brass annealed for 1800 sec at 573 K. It would appear from the micrographs that the bulge, which was almost completely comprised of twin oriented material, may have been a viable nucleus for recrystallization, being $\sim 0.75\,\mu$m in diameter (along the coherent twin plane; arrowed in Fig. 2a). An important point illustrated by this example and which will be discussed further in the next section, is shown more clearly in Fig. 2b. The coherent interface (arrowed) between the twin oriented bulge and the deformed parent grain has effectively sealed off the twin region from most of the dislocation structures in the parent matrix.

The example shown in Fig. 2 again concerns deformed grains of near $\langle 1\,1\,0\rangle$ rolling plane normal. In this case the boundary between the original deformed grains had a misorientation close to that characteristic of a $\Sigma 9$ coincidence boundary (39° about $\langle 1\,1\,0\rangle$). Therefore, as can be seen from the diffraction pattern of Fig. 2c, the volume within the bulge was $\Sigma 3$ related to the parent grain and very close to $\Sigma 3$ related to the grain into which it had extended.

Both of the examples given in Figs 1 and 2 have illustrated rather early stages in the recovery of

Figure 2 A $\Sigma 3$ twin oriented bulge on a grain boundary in 50% rolled α-brass annealed for 1800 sec at 573 K: (a) the coherent twin interface is arrowed, (b) the same area as in (a), but tilted to show more clearly the discontinuity in dislocation density across the coherent twin interface (arrowed) and (c) $\langle 1\,1\,0\rangle$ axis selected area diffraction patterns from the boundary region shown in (a) and (b). In this pattern the common $\langle 1\,\bar{1}\,1\rangle$ for the twin and parent grains is indicated along A while the nearly common $\langle \bar{1}\,1\,1\rangle$ of the twin and the grain into which the bulge is growing is indicated along B.



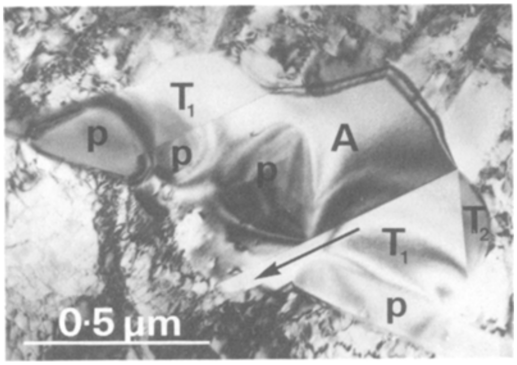


Figure 3 A recrystallization nucleus forming at a boundary in AISI 310, 20% deformed and annealed for 600 sec at 1173 K. Large $M_{23}C_6$ particles (marked "p") delineate the original boundary position. The nucleus is a continuation of the matrix orientation in region A and elsewhere is comprised of primary twin variant T_1 and second order twin T_2. One of the primary twins has grown back into the parent grain (arrowed).

regions where the twin related bulges have been separated from their parent grains by essentially immobile coherent twin interfaces. The example given in Fig. 3, while illustrating both a rather later stage in local rearrangement and a more complicated nucleation event, indicates that incoherent twin segments appear to be capable of growing back into their deformed parent grains. The bowing segment of grain boundary shown in this figure lay between two grains in a sample of AISI 310 stainless steel deformed by 20% and annealed for 600 sec at 1173 K. Large $M_{23}C_6$ precipitates have formed during this anneal and delineate the original position of the grain boundary. In region A of the bulge, the orientation was simply a continuation of that in the matrix. There was no interface between the bulged region and the local, recovered dislocation substructures. The remainder of the bulge comprised two separated areas of one primary twin variant (T_1) and a single small region of second order twin (T_2). The relevant feature in this micrograph is the obvious intrusion of one of the segments of primary twin (arrowed), into the parent grain. The indication is that there had been growth of this twin through migration of the incoherent interface segments, in addition to any growth associated with the migration of the high angle bulge interface.

Fig. 4a shows an apparently established recrystallization nucleus of $\sim 2\,\mu$m in diameter in a specimen of the Nb stabilized austenitic stainless steel which had been rolled by 20% and subsequently annealed for 90 sec at 1173 K. The nucleus straddled a boundary between two deformed grains. The original position of this boundary has been delineated by the line of small Nb(CN) particles running across the nucleus. The nucleus (A), which contains an annealing twin (B), is itself very close to being $\Sigma3$ related to the lower, unrecrystallized grain (C). The small facets (arrowed) in the lower part of the recrystallization interface are indicative of the proximity of a special orientation relationship.

The recrystallization nucleus, the twin within it, and the lower, unrecrystallized grain all possessed orientations with ⟨1 1 0⟩ close to the foil normal

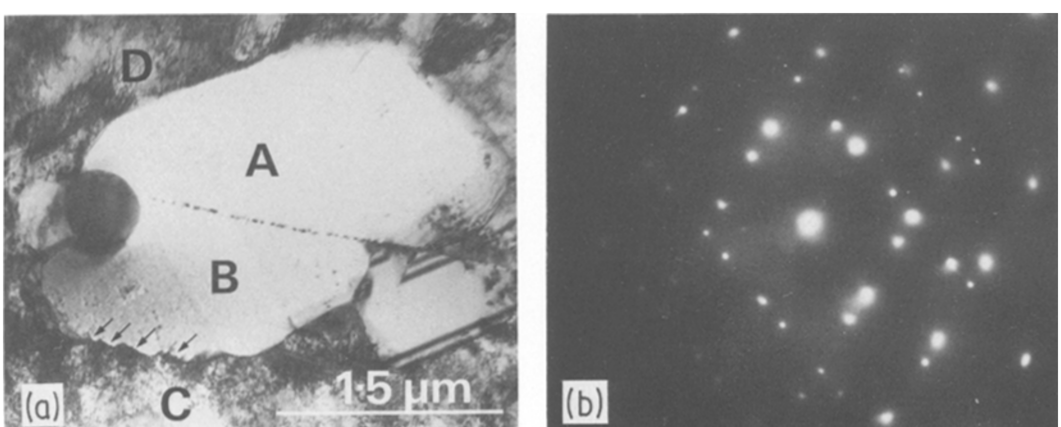


Figure 4 A recrystallization nucleus at a grain boundary in a 20% deformed Nb-stabilized steel specimen annealed for 90 sec at 1173 K: (a) the nucleus (A), which contains an annealing twin (B), is near to a $\Sigma3$ twin orientation with respect to grain C and (b) a diffraction pattern taken from areas A, B and C. The proximity of $\Sigma3$ and $\Sigma9$ misorientations is apparent, cf Fig. 2c.

and, therefore, the rolling plane normal. Hence, the diffraction pattern given in Fig. 4b, which contains information on the orientation of all three areas (A, B and C) is more or less identical to that shown in Fig. 2c for the brass specimen. The interface between the annealing twin contained in the nucleus and the lower, unrecrystallized grain is near in misorientation to that characteristic of a $\Sigma 9$ boundary. No similar, simple relationship existed between the orientation of the nucleus and that of the upper unrecrystallized grain (D). For this latter grain the low index zone nearest to being parallel to the rolling plane and foil normals was, again, $\langle 1\ 1\ 0 \rangle$.

The example shown in Fig. 4 is further evidence that incoherent interfaces around near $\Sigma 3$ twin oriented nuclei are mobile and that, irrespective of any involvement in stimulation of nucleation, their movement can contribute to the early stages of growth of a recrystallization nucleus.

4. Discussion

The observations presented in the previous section have shown that annealing twins can form at very early stages during annealing following deformation. Further, the results show that the formation of annealing twins, during recovery, at the boundaries between deformed grains is not a process which is confined to a single alloy system. Further emphasis of the generality of these results comes from the fact that the grain boundaries in question varied from those containing high densities of large precipitates (AISI 310) or high densities of small precipitates (Nb steel), to those which were precipitate free (α-brass).

The growth of nuclei through migration of segments of interface close to $\Sigma 3$ in misorientation has been illustrated by the examples shown in Figs 2 and 4. The close proximity of this particular special orientation does not appear to have been a barrier to nucleus growth. Moreover, as mentioned in connection with the example shown in Fig. 1, grain boundaries may border orientation gradients in the deformed material. In such environments, nuclei and parent grains which are initially $\Sigma 3$ twin related will quickly lose this special orientation relationship with continued nucleus growth.

From examples such as were shown in Figs 1 and 2, it also appears that grain boundary annealing twins can form at a sufficiently early stage during a recrystallization anneal to affect nucleation behaviour. In considering this possibility, brief mention should first be made of one aspect of the theory of twin formation. Fullman and Fisher [10], in considering grain growth in copper, suggested that twinning in the corners of growing grains would be facilitated if the event led to replacement of segments of high energy boundary by segments of lower energy grain boundary. These arguments appeared to be supported by the thermal etching observations presented by these authors. Dash and Brown [11], in a study of annealing twin formation at advancing recrystallization interfaces in a deformed and annealed Ni–Fe alloy, presented evidence which led to their drawing similar conclusions on the driving forces associated with the twinning process. There is no reason to suspect that similar dictates did not govern the type of twinning reported in the present work.

Experimental observations of the type shown in Fig. 2 have indicated that the twinning operation can effectively seal off a small area of the (transformed) grain boundary from most links with the dislocation structures present in the adjacent parent grain. Thus, the numbers of dislocations linked to a transformed segment of boundary plane will be less than those which were linked to the segment of untransformed boundary plane. In such circumstances the twinning operation will effect a local increase in the difference in stored energy density across the high angle boundary, within the region defined by the twin segment.

If the conditions for nucleus formation by microbulging (local strain induced boundary migration) are taken to be those suggested by Bailey and Hirsch [8], then the critical bulge radius necessary for successful nucleation (R_c) will be given by

$$R_c = \frac{2E}{\Delta G_v}$$

where E represents the energy per unit area of bowing high angle interface and ΔG_v the local free energy difference across this interface. Following the above discussion it will be clear that a decrease in E and an increase in ΔG_v, effected by twinning, will facilitate nucleus formation from smaller bulges than would be the case if no twinning took place. Nucleation will, therefore, be favoured by the operation of such twinning processes.

At this point it is pertinent to suggest an explanation for the fact that the annealing induced twinning decomposition at grain boundaries, which is reported in the present paper, only took place following deformation. To this end, the work of Goodhew and co-workers [12, 13] is relevant. These authors have shown that annealing of [1 1 0] axis gold bi-crystal specimens containing twist boundaries of various misorientations leads to the formation of island grains surrounded by tilt boundaries. In certain cases a twinning decomposition was found to have occurred as the result of rotation of these twist boundaries into tilt orientations during annealing. However, these decompositions only occurred for bi-crystal specimens with certain initial misorientations, being those close to $\Sigma 9$, $\Sigma 11$ and $\Sigma 99$ coincidence site lattice misorientations.

It is therefore tempting to suggest that the decompositions observed in the current work only became feasible as a result of favourable local changes in grain boundary misorientation consequent to lattice rotations generated during deformation. The difference in orientation between grain boundary and grain centre regions, reported for the example shown in Fig. 1, is a result of such orientation changes. Further, the twinned grain boundary region in brass shown in Fig. 2 corresponded to decomposition of a boundary of near $\Sigma 9$ character and is therefore consistent with the above noted data concerning twinning decomposition at boundaries in gold [12, 13].

An additional element which may be involved in the stimulation of such decomposition processes concerns the stored energy removed from the twin volume by the twinning process itself. Meyers and Murr [14], in an analysis of the twinning process, suggested that the removal of such stored energy would favour the twinning decomposition. Therefore, this may be a further factor involved in stimulating the operation of twinning in the deformed as opposed to in the originally well annealed materials.

It is impossible to decide from the current work whether or not some element of boundary migration was necessary before the operation of the twinning decompositions which were observed. However, as has been shown elsewhere [9] there is almost always at least a small amount of boundary plane readjustment during the very early stages of annealing of deformed materials, even in areas where there may be no subsequent nucleation of recrystallization. These small amounts of boundary movement may be sufficient to stimulate twin formation.

Finally, the observations suggest that twin formation during the early stages of annealing is favoured at boundaries which lie between grains with $\langle 1\,1\,0 \rangle$ close to the rolling plane normal. Hence, it is possible that the total number of such twinning events will increase with increases in the strength of the "brass"-type $(1\,1\,0)[\bar{1}\,1\,2]$ deformation textures characteristic of these low stacking fault energy materials. Given the likelihood of nucleation of recrystallization following early twinning of the above type, this suggests that there may be a direct connection between the density of nuclei formed and the strength of a deformation texture. This connection would be quite separate from the more obvious association between nucleation density and strength of texture, which arises from the general dependence of critical nucleus size on the level of stored energy of cold work.

5. Conclusions

Experimental evidence has been presented which shows that annealing twins are formed at grain boundaries during the early stages of annealing in three different cold rolled, low stacking fault energy materials. The alloys investigated were an AISI 310 stainless steel, a Nb stabilized stainless steel and an α-brass.

The observations which have been reported suggest that the formation of small twin segments at grain boundaries may occur sufficiently early in an anneal to affect behaviour during nucleation of recrystallization. It has been argued that such twinning events may stimulate nucleation of recrystallization.

From the results of the present investigation it appears that there may be a direct link between the density of nuclei formed during recrystallization and the texture of a deformed low stacking fault energy material.

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