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STRUCTURE AND PROPERTIES OF CREEP SUBBOUNDARIES

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Résumé - On décrit les propriétés des sous-joints de fluage à partir d'observations et expériences diverses réalisées dans un microscope électronique dans de l'aluminium et un alliage aluminium-zinc. Leur géométrie, champs de contraintes associés, leurs propriétés dynamiques et leur rôle dans la résistance au fluage sont examinés.

Abstract - The properties of creep subboundaries are described as derived from observations and experiments in the electron microscope for aluminium and an aluminium-zinc alloy. Their geometry, associated stress fields, their dynamic properties and their contribution to the creep strength are reviewed.

Among the different types of interfaces, subboundaries are simple ones. At low misorientations at least, the constituent dislocations are visible by means of conventional electron microscopy and several of their properties can be known. The present paper reports the characteristics of subboundaries which develop during stage II of creep in Aluminium and an Al-Zn alloy. The static properties (detailed geometrical description), dynamic ones (mobility ...), and their connection with the constitutive equations of creep are studied here below.

EXPERIMENTAL DETAILS

The results presented here were obtained, using 99.99 % Al single crystals in the 112 orientation, crept at 150 °C and 400 °C /1/, 99.3 % pure Al in polycrystalline sheets of .5 mm thickness tested between 80 and 200 °C /2/, and Al-11 wt % Zn cylindrical polycrystals crept at 210 and 215 °C /3/. All were deformed in tension up to stage II of creep. Conventional electron microscope studies were carried out (TEM and SEM), as well as in situ creep experiments in a high voltage electron microscope (HVEM) /4, 5/ and dislocation pinning experiments in the load applied state /3/ in Al-Zn. The latter techniques provide useful informations about the substructure properties under load, and are very complementary /6/.

SUBBOUNDARY STRUCTURE

The possible geometries of subboundaries (i.e. periodic networks with no long range stress fields) are analyzed in detail, in /7/. The first experimental observations of subboundaries were performed by decoration techniques in NaCl and KCl /8/.

Creep subboundaries appear in stage II, when the crystal is divided into subgrains of several microns in diameter.

In the single crystals /1/, the symmetrical orientation with respect to glide, develops at 150 °C a characteristic substructure illustrated on fig. 1. A cross-grating of two types of cylinders approximately parallel to the directions of the primary Burgers vectors (i.e. [101] and [011]) appears on a (111) longitudinal section. The (112) normal cross section shows that the corresponding crystal blocks are elongated along the [111] direction. They are misoriented by 1 or

2° with respect to each other, around $[21\bar{1}]$ and $[12\bar{1}]$ respectively. They are rotated alternatively in the positive and negative directions around these axes, so as to maintain the symmetrical sample orientation as it is strained. They are divided into smaller subgrains as seen in TEM, with weak relative misorientations (smaller than $.5^\circ$). Some of the subboundaries between the cylinders have been analysed. They appear as periodic dislocation networks. Some networks appear to be of pure twist type, lying in the $(11\bar{1})$ plane with 3 Burgers vectors at 120° . The mesh sizes are of the order of some 100 Å. A more complete identification of the other types of subboundaries is underway.

At 400°C , on the contrary, the temperature is high enough to allow glide on the unusual $[110]$ (001) slip system as evidenced by the slip lines at the sample surface. Since it has a higher Schmid factor than the 111 primary systems of intermediate temperatures, it is privileged at 400°C . A rotation of the lattice occurs around $[1\bar{1}0]$, as expected in single glide, and the tensile axis evolves from $[112]$ towards $[111]$. The subgrains appear as flat blocks parallel to (110) planes as evidenced by SEM on $(1\bar{1}0)$ sections (see the stereographic projection of Fig. 1). Subboundaries exhibit higher tilt angles than previously (5 to 6°). The dislocations are therefore much closer to each other (around 20 Å) and more difficult to identify but seem to belong to tilt boundaries with $[110]$ as rotation axis (M. Carrard, to be published).

In the Aluminium sheets crept at intermediate temperatures, the following features of subboundaries were obtained /2/:

- Pure tilt boundaries, or pure twist ones consisting of a square grid of two screw dislocation families are observed but seldom.
- Boundaries of mixed character, with 3 Burgers vectors at 120° are the most frequent ones, lying in a variety of planes. The dislocation segments appear to lie in their respective slip planes and the general configuration of the networks is presented on fig. 2 a.
- The Frank formula which expresses that a dislocation array does not exhibit any long range stress field, seems to be satisfied for the periodic sections of the networks. This condition together with the previous ones can lead to various extreme subboundary configurations as the subboundary plane is rotated away from the Burgers vectors plane (see fig. 2 b, c, d). The equilibrium of line tensions at nodes in the network is thus not fulfilled. This indicates that in aluminium at least, the weak dislocation dissociation decreases the core energy by a larger amount than the subsequent increase of line energy /9/. Some of these configurations had been predicted by Ball and Hirsch /10/.

In the Al-Zn polycrystal, subboundaries were also observed. They have been analysed and most of them consist of 5 dislocation families /3/. An example is shown on fig. 3. It was also observed that the misorientation across subgrains increases with strain /3/ in agreement with other measurements /12/.

Thus, according to the type of sample and creep conditions, subboundaries consisting of one to five dislocation families (out of 6 possible ones in the FCC structure) are present. This can be explained by the particular geometry of the single crystal, the various deformation compatibility requirements in the polycrystals; they are two dimensional in the sheet case and more severe for the cylindrical samples of Al-Zn.

SUBBOUNDARY STRESS FIELDS

As mentioned above, they were already checked through the Frank criterion. A different experimental approach has been developed as well /11/. When it is possible to pin the dislocation arrangements under load, defects exhibit bowed shapes under the effects of the local or effective stresses present in the crys-

Fig. 2 - Sketch of subboundaries with 3 bs at 120° . a) general case; b), c), d) extreme possible cases [18]. X_1 , X_2 , X_3 dislocation segments.

tal. The following technique was used in the case of Al-Zn. At creep temperatures of 210 or 250 °C, the zinc is in solid solution and the alloy creeps in the same way as aluminium (i.e. forming subgrains). The pinning technique used, consists in quenching and ageing the specimen under load such that zinc precipitates onto the dislocations to freeze them as they are during the creep test [1]. The sample is then unloaded and foils are prepared for TEM. From the dislocation curvatures, orders of magnitude of local effective stresses can be derived.

They were measured at the subboundary and as one moves away from it towards the subgrain interior. Results are presented on fig. 4, which were obtained from several subgrains after creep at 250 °C under an applied stress of 2.4 MPa and at a strain of 7 %. The sign of the local stress has not been measured and all values are plotted as positive. It appears that very high local stresses exist at given points of the subboundaries, several times larger than the applied stress and these decrease as a function of the distance from it. In addition, a given subboundary exhibits a limited number of points with a highly concentrated stress, if

Fig. 3 - Sketch of a subboundary with 5 dislocation families as observed in Al-Zn.

Fig.4 - Normalized local stresses around subboundaries. $\epsilon = 7\%$ /3/.

any /11/. It has also been observed that the maximum values of local stresses in subboundaries increase as strain increases, for given creep conditions, as stress increases for a given temperature and strain and as temperature increases for a given stress and strain /13/. It therefore appears that as dislocations are heterogeneously stored in the crystal, a non homogeneous stress distribution is built up. It is thought that these areas of high effective stresses correspond to points at which the periodicity of the subboundary network is interrupted as a result of deformation: extrinsic dislocations enter the network while others are leaving it thus altering its regularity.

DYNAMIC PROPERTIES OF SUBBOUNDARIES

These were observed during in situ creep experiments performed in a HVEM /4/: the aluminium polycrystalline sheets were submitted to macroscopic creep tests. Then, microsamples were cut out of them, with the same tensile axis, and further deformed in the microscope at the same temperature and strain rate as in stage II of the preceding test.

The destruction of subboundaries (as well as their formation) is sometimes observed. Some examples have been reported previously /14/ in which, a square grid of screw dislocations (pure twist boundary in a 100 plane) was destroyed: the applied stress pulled both dislocation families apart on their glide planes of $\{110\}$ type. But numerous experiments performed under similar conditions have shown that this phenomenon occurs rather seldom. In addition the newly emitted dislocations account for less than 1 % of the total strain. However, the migration of subboundaries is more commonly observed. This process has been evidenced a long time ago in the experiments of Washburn and Parker /15/. An extensive study was undertaken by Exell and Warrington, during creep of Aluminium between 400 and 600 $^\circ C$, using X ray topography and a Nomarski interferometer /16/. It also occurs at lower temperatures as seen during in situ straining. The dislocation traces visible at the foil surface behind the moving boundaries, indicate that its constituent segments move by glide. Migration takes place over large distances (a significant fraction of the subboundary diameter) with a low speed (10^{-4} to $10^{-1} \mu m/s$). The dislocation segments having 3 Burgers vectors at 120° (see above), two families glide in different $\{111\}$ planes while the third one glides in a $\{100\}$ type plane. Indeed there are not three intersecting 111 planes in the FCC structure. The geometry of this glide process is sketched on Fig. 5.

The locus of the boundary nodes are $\langle 011 \rangle$ directions which as a rule, are not perpendicular to the network plane. Therefore the nodes move along this plane during migration so that the constituent dislocations move towards triple junctions, are crossing through them and are continuously transferred from one subboundary to the adjacent one. This complex three dimensional glide mechanism, which involves a cooperative slip on three different planes, is possible in this case because 3 dislocation families only are involved.

In the Al-Zn polycrystal, migration has been observed too /11/. The surface tilts connected to it were observed by straining a specimen to 15 %, polishing it and restraining it another 2 %. The surface was examined using Nomarski interference contrast. An example of such observations can be found in /17/. In this case, since subboundaries consist of five dislocation families as reported above, and that they are not always situated in glide planes, migration cannot occur by glide only.

Under these various experimental conditions, the amount of strain due to migration could be estimated. It is always a small fraction of the total strain i.e. 5 to 10 % in agreement with other measurements /16/.

Fig. 5 - Scheme of subboundary migration. SP and FP: subboundary and foil planes. x_i : dislocation segments. P_i , y_i : corresponding slip planes and slip traces /4/.

Fig. 6 - Local creep curve in a given subgrain. In situ experiment in the HVEM. Al 200 °C /18/.

SUBBOUNDARIES AND CREEP RATE

Since a small amount of strain only is due to boundary migration or destruction, the motion of individual dislocations is responsible for most of it. It could be directly studied in aluminium polycrystalline sheets with the help of in situ experiments, and this provided useful informations about the possible influence of subboundaries on the rate controlling process during stage II of creep /5/.

The direct observation of moving dislocations through subgrains provides useful informations on their densities, on the effective obstacles to their motion, as well as the corresponding waiting times involved. Dislocations are observed to glide through the subgrains at temperatures between 80 and 200 °C. This could be deduced from the traces left at the foil surfaces by moving defects. The flight time through subgrains could be measured from dynamic recordings and was found to be shorter than 1/50 s. The mean free path of the dislocations is larger than the subgrain size since moving dislocations are observed to cut through subboundaries. This is a much slower process and waiting times larger or much larger than 1 s can be measured at the boundaries. A local creep curve could be built from such observations in the following way: each dislocation which shears the subgrain provides an amount of strain $\epsilon = b/L$ if b is its Burgers vector. By counting the number of passing dislocations as a function of time, the curve of Fig. 6 could be established. It shows that successive groups of 5, 4, 1 and 5 dislocations respectively, have been gliding through the observed subgrain (2 μm in diameter) during the observation period of 2000 s. An average deformation rate of $7.5 \cdot 10^{-7} s^{-1}$ could be derived. This experiment illustrates the jerky nature of glide under such creep conditions /18/.

Since the rate at which the crystal creeps, seems to be directly connected to the cutting through mechanism of mobile dislocations through subboundaries, this process was studied in detail both experimentally and theoretically. In situ experiments /5/ and pinned dislocation arrangements /3/ show that a crossslip mechanism is involved for dislocation insertion or extraction. The critical event in extraction is the breaking of the first node by the escaping dislocation. Orders of magnitude of the critical stress have been computed /5/ and though they are smaller than the Orowan stress, they are much larger than the applied one.

The barriers to dislocation motion for high temperature creep in aluminium are the subboundaries. They are constructed as straining proceeds. In the same time, the crystal builds up large local internal stresses which add to the applied stress and help dislocation emission at privileged points in the boundaries.

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