Formation and evolution of dislocation structures in Fe-3%Si single crystals under monotonous and sequential loading.

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ABSTRACT

Tensile tests are performed on Fe-3%Si single crystals in a multiple slip orientation, namely the $[1\bar{1}0]$ axis, to promote the development of a cell structure. At low strains ($\epsilon < 15\%$), the resulting dislocation arrangement is composed of tangles distributed at random. This pattern evolves toward two families of dislocation sheets which constitute a mosaic like structure at higher levels of deformation. The sheets are composed of elementary tangles aligned in directions which are approximatively parallel to the trace of the active slip planes. When the deformed samples are further sheared at 90° from the tensile axis, the observed pattern is homogeneous only at low prestrains. For tensile deformations higher than 25%, microbands are formed and appear superimposed to the prestrain cell structure. The flow localization related to microband formation results from the destabilisation of the dislocation arrangement pertaining to the tensile deformation.

INTRODUCTION

Metals are known to develop under plastic deformation, at microscale, organized dislocation structures. At a temperature below half the melting point, more or less well defined cell structures are frequently observed in face-centred cubic as well as in body-centred cubic materials. While the reason for the tangling process is still a subject of controversy, the morphology and the contents of the dislocation structures are rather well documented. For instance, single slip promoted during cyclic or monotonous deformations gives rise to dipole walls which orientation is perpendicular or at least very inclined with respect to the active slip plane (1-3). By contrast,

during monotonous deformation, dislocation sheets, which lie roughly parallel to the active slip planes, become predominant when more than one slip system are active (3-5). These sheets are composed of dislocations with at least two different Burgers vectors and partly of debris, resulting from the interaction between them. The first part of the present note is focused on the formation of dislocation sheets in iron-silicon single crystals. The development of a cell structure is described in details for tensile deformation in a multiple slip orientation.

The second objective of this work is to investigate the stability of the dislocation pattern during a subsequent, and different, sollicitation, i.e.: under a change of strain path. The procedure is similar to the well-known latent hardening tests (for a review see MICHEL and CHAMPIER (6)), except that usually prestrains are restricted to low values and in single slip orientations. While latent hardening tests permit the interactions between dislocations of two particular slip systems to be inferred, the present aim is to investigate the interaction between newly active slip systems and the dislocation distribution resulting from large prestrains in multiple slip conditions.

MATERIALS AND METHODS

The iron 2.9% silicon single crystals obtained by the strain-anneal method have a plane orientation almost perfectly parallel to the (110) plane. The tensile samples spark-cut from the thin resulting sheets ($e=0.28$ mm) are aligned with the $[\bar{1}10]$ axis. The crystal orientation is chosen in order to promote the activity of dislocations with two different Burgers vectors, namely $\mathbf{b}_1=[\bar{1}11]$ and $\mathbf{b}_2=[\bar{1}\bar{1}1]$ (Fig.1). The mostly sollicitated slip planes are of $\{112\}$ types for both Burgers vectors, but the activity on these planes depends on their critical resolved shear stress and seems doubtful for the present material (3). It is rather believed that the $(0\bar{1}1)$ and (101) slip planes for $\mathbf{b}_1$ (respectively $(0\bar{1}1)$ and (101) for $\mathbf{b}_2$) are sufficiently

Fig.1 : Orientation of the specimens during pre and subsequent loading

loaded to accommodate the deformation. In any case, a completely equivalent activity is expected for the two Burgers vectors because they lie in symmetrical directions with respect to the tensile axis.

Small shear samples (10 mm high) are cut from the central part of the $40 \times 10 \times 0.28 \text{ mm}^3$ tension specimens. The deformation in tension is homogeneous up to about 10%; above this prestrain a neck develops. In the latter case the shear specimens are taken out from the localized zone and prestrains are estimated from the decrease in the sample section. It has been verified that the crystallographic orientation of the thin foils remains unchanged even in the necked region. The shear test, described elsewhere (3,7,8), is performed in the [001] direction, i.e.: at 90° from the tensile direction. The higher Schmid factor is obtained on two coplanar and previously latent slip systems, namely $[11\bar{1}](1\bar{1}0)$ and $[111](1\bar{1}0)$. The active part of the shear samples is limited by the thickness of the rolled material, the space between the grips (1 mm) and its length which corresponds to the final width of the prestrained sample ($h < 10 \text{ mm}$). Unfortunately these dimensions are too small to enable the load and the deformation to be measured accurately. As a consequence, the flow laws during pre and subsequent deformations are not obtained in this experimental campaign.

Dislocation structures were observed using a 200kV transmission electron microscope (T.E.M.). The samples were prepared with the usual electropolishing techniques (8). All the thin foils are parallel to the sheet plane. Dislocation densities are estimated by dividing the number of intercepts with two sets of parallel lines by the foil thickness ($\approx 2000 \text{ \AA}$) and the total length of the grid lines.

RESULTS

The dislocation arrangement after tensile deformation

At low prestrains ($\epsilon < 15\%$), the dislocation structure appears essentially composed of long screw dislocations with the expected Burgers vectors (Fig.2), the two families being equally and homogeneously distributed. The dislocation density estimated at 14% tensile strain is 10^{14} m^{-2} . Tangles are also visible and frequently associated with the small loops resulting from the movement of the screw dislocations (9,10). The distribution of the tangles is of interest : when the long screw segments are mentally eliminated, the debris appear randomly placed in the material. In other words, at small strains an heterogeneous organization of the dislocations builds up but with no favoured orientation.

At higher deformations, a well formed cell structure is found (Fig.3). This mosaic like pattern is composed of two families of dislocation sheets obviously associated with the two active slip systems¹. They are constituted of elementary tangles, which length

¹ It should be emphasized that whatever the effective slip planes are (of $\{112\}$ or $\{110\}$ types) for a given Burgers vector, the same trace of the slip system will be observed on the picture, its direction being the Burgers vector itself.

Fig.2 : Typical dislocation structure after low deformation in tension. The tensile axis (T.D.) as well as the trace of the active slip planes are indicated on the picture. The beam (B) is normal to the thin foil.

equals the cell size, and placed end to end. As an average, a sheet seems parallel to the trace of the active slip plane because the individual segments lie in it, but deviations from this main orientation are frequent. The elementary tangles contain dislocations of the two active slip systems. Some of them are not completely formed so that the sheets do not cross the entire sample. A less well finished aspect of the dislocation arrangement is observed at some places (Fig. 4) where a high density ($\rho=3 \times 10^{14} \text{ m}^{-2}$) of entangled but still

Fig.3 : Mosaic structure resulting from a high level of tensile strain ($\epsilon=26\%$) in a double slip orientation. The dislocation sheets lie approximatively parallel to the active slip plane.

Fig.4 : Transition between the low strain dislocation arrangement and the cell structure.

distinguishable dislocations are locally covering up to 50% of the surface (typical width of $0.5 \mu\text{m}$). This network is similar to the low deformation dislocation arrangement but more dense. The elementary tangles described above appear within this structure by some process of dislocation concentration because firstly the width of these elements ($d < 0.2 \mu\text{m}$) is smaller than the one of the networks and secondly the dislocation density is higher in the tangles ($\rho > 10^{15} \text{ m}^{-2}$, Fig. 4). Besides, the density in the cell interiors ($\rho \approx 5 \times 10^{13} \text{ m}^{-2}$) is of the same order or even lower than the value measured at $\epsilon = 14\%$.

The dislocation arrangement after the change of strain path.

The structure formed by 13% prestrain in tension followed by 7% shear deformation at 90° from the tensile axis is exhibited on figure 5. The resulting structure looks like a cell structure. The surrounding tangles contain dislocations related to prestrain and shear strain, i.e.: the four Burgers vectors are present. Such a kind of cellular arrangement resembles the cell configuration created by tensile deformation in the $[100]$ axis when four slip systems are activated together (11). Of importance is the fact that the structure is homogeneous all over the thin foils observed for these conditions. It can be concluded that the strain was not localized.

A completely different situation arises when prestrain is high enough to develop the mosaic like structure (Fig.6). A slight deformation in shear promotes strain localization in form of microbands superimposed to the previously constructed dislocation arrangement, while the remaining structure appears practically not affected by the subsequent deformation. These microbands lie in a plane roughly parallel to the newly activated slip plane $(1\bar{1}0)$ but a deviation as high as 15° may be observed (Fig.6). It is hard to estimate the strain in the microbands because, as noted above, the sheets which may serve as markers are not straight. Moreover the microbands are not equally

spaced within the samples, usually they are isolated but some clusters of up to five microbands are sometimes observed.

DISCUSSION

Monotonous deformation

From the T.E.M. observations at different prestrains it can be concluded that the sheet formation is a multiple steps process. At the beginning, dislocation segments entangle at random on their slip planes

Fig.5.: Dislocation arrangement after change of deformation mode for a low prestrain. Dislocations with the four Burgers vectors are observed. The trace of the slip plane activated during tension or shear deformation are marked.

Fig.6 : Microbands superimposed to the mosaic like structure resulting from shear deformation after a high tensile prestrain.

around debris like loops and reaction products related to the two active slip systems. At higher levels of strain, i.e.: at higher dislocation densities, the segments cluster in the elementary tangles which compose the final sheets. The progressive opening of cell interiors within the highly concentrated dislocations structure (Fig. 4) requires the disappearance of some parts of the network. This may occur either when locally pinned segments cross the obstacles while others accumulate elsewhere or by some drift of the interlaced segments to what will compose the cell boundaries. Such a drift process was reported for *in situ* deformation of iron foils (12), but it cannot be concluded that this process is operative here. Rather, the formation of the dislocation pattern is believed to occur as follows. It should first be noted that the long screw dislocations seen on the pictures are mobile dislocations even if their movement is impeded by the Peierls stress. The alignment of cells with the trace of an active slip plane (e.g.: from lower left to upper right on figure 4) suggests that the mobility of the screw segments is higher in these channels. The elementary tangles are created when the moving dislocations surround the segments of the other family which speed is thereby reduced. In other words, the sheet embryos are created by a statistical mechanism close to the 'hard spots' formation proposed by KOCKS (13). The same holds for the other slip system and the final pattern results from the crossing of the channels related to the two families. This does not mean that the strain is entirely concentrated in the cells. In fact the deformation may be similar in cell interiors and cell boundaries except that a low speed is sufficient when a high density of dislocations accommodates the strain.

Now it must be emphasized that the process is self-catalytic because when a high number of dislocations of low mobility are present, like in the sheets, the opportunity for tangling increases. As a consequence, the stress required to promote the deformation inside the cluster also increases and the segments in the cell interiors move faster - their density decreases - leaving more 'concave loops' (13) by surrounding the clusters. The final alignment of the tangles, and therefore the formation of the sheets, results from the alignment of the cell interiors which compose the free channels. Such a process is compatible with the pattern observed in other deformation conditions : while the mosaic like structure is due to the equi-activity on the two slip systems in a double slip orientation, elongated cells are expected when one slip system dominates like in stage II for single crystal (2) or in grains of polycrystals (8). Indeed, in the last cases, the creation of free channels is more efficient in the direction of the main slip system.

Change of strain path.

The change of strain path promotes flow localization. This was already reported for exactly the same deformation sequence in polycrystals (8) ; from the present results it can be concluded that the localization in polycrystals is related to the destabilization of the intragranular structure and not to grain boundary effects. Concerning single crystal the same trend was observed in fcc and invoked for hcp metals (e.g.: 14 and 15) so that the destabilization is

not due to some specific behaviour of screw segments in bcc materials.

It was observed on polycrystals (8,16) that after a high deformation along the second part of the strain path the dislocation pattern connected to prestrain vanishes while a new structure is established. This evolution seems quite general. Already BASINSKI and JACKSON (17) noted for copper that dislocation distributions are unstable to slip on a single system unless they have been formed on that system and they attributed the inhomogeneous deformation to this structural instability. Present results show that the evolution from the prestrain dislocation arrangement to the new pattern occurs either homogeneously, at low prestrains, or with a sharp strain localization. As a consequence, the microband formation appears to be one of the different mechanisms used by the material to modify adequately the dislocation organisation. This means that the formation of microbands is a transient process.

The key for plastic instability lies in the decrease of the local flow stress related to the microstructural evolutions. It is clear that the disappearance of the prestrain dislocation structure implies a decrease of the dislocation densities pertaining to the previously active slip systems, but it should be noted that dislocation densities increase on the new slip systems. As a consequence, the flow localization appears only when the recovery term exceeds the hardening one. This is particularly true when the hardening component is low and that the prestrain structure recovers easily. The onset of plastic instabilities inherent to cross-loading may be predicted if these hardening and recovery terms are correctly estimated. An attempt to determine the critical prestrain on the basis of a single parameter model (the dislocation density) is given elsewhere (18). In view of these considerations, it appears that latent hardening tests are particularly subjected to flow localization. Indeed, the dislocation arrangement after single slip is composed of dipoles and multipoles which are unstable by nature, and the subsequent deformation is also performed in single slip, i.e.: with a low hardening. The last condition is respected in the present deformation sequence : in the worst case, the coplanar slip promoted at reloading leads to a hardening of the same order of magnitude than self-hardening, but it is rather believed that microbands are manifestations of single slip (in which case two types of microbands, associated with the two Burgers vectors, are required to accommodate the deformation, this will explain the clusters of microbands). The surprising point here is the fact that tangles, composed of interlaced dislocations belonging to at least two slip systems, recover so easily at reloading. The paradox is less difficult to solve if the tangles contain essentially concave loops (13,19), as suggested by the formation process described above, indeed, the natural tendency of the loops is to collapse. In such a case the driving force for annihilation increases when the loops are smaller or, in other words when the width of the tangles decreases. The observed behaviour at low prestrains compared to high prestrains is in agreement with this view. This implies that the dislocation arrangement, and not only their density, has an influence on the recovery term. However it is difficult to separate the effects of these two parameters from *post mortem* TEM observations and some further works are required in this field.

CONCLUSIONS

The dislocation organization in Fe-3%Si single crystals sollicitated in a double slip orientation is a two step process. Initially, dislocations accumulate at random around obstacles created during the deformation. Then, the free dislocations are progressively swept out of the cell interiors while the less mobile dislocations are tightened in the sheets. The cells interiors correspond to free channels for the movement of the mobile segments and the final sheets result from the alignment of elementary tangles surrounding these channels.

Specimens containing the above structure are sheared at 90° from the tensile axis in order to activate two coplanar slip systems which were latent during prestrain. After low prestrains, the four Burgers vectors are present and distributed uniformly in the materials. By contrast when the tensile deformation exceeds 25%, strain localization is observed through microband formations superimposed to the previous cell structure. These microbands result from the recovery related to the disappearance of the prestrain dislocation structure.

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