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Acta Universitatis Carolinae. Mathematica et Physica, Vol. 32 (1991), No. 1, 31--39

Persistent URL: http://dml.cz/dmlcz/142625

TEM Obervation of Dislocation Behavior near Grain Boundaries in Deformed Fe-2wt. %Si Bicrystals

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Received 27 August 1990

In the case of low temperature deformation moving lattice dislocations are trapped by grain boundaries, interact with them and enable thus the slip transmission throughout grain boundaries. In order to ascertain the role of grain boundary geometry in this process, two Fe-2wt.%Si bicrystals containing a special twin and random grain boundary were defomred in tension up to strain $\varepsilon = 0.2$ and 0.4. Dislncation strcutres in bulk and near grain boundaries were studied by TEM. No special deformation zones along grain boundaries were found, which is ascribed to the absence of additional compatibility stresses. The random grain boundary was shown to be more effective sink for moving primary slip dislncation than $\Sigma 3$ twin boundary.

Introduction

The process of slip transfer across grain boundaries [GB] depends on the nature of individual boundaries. The observation and characterization of direct slip transfer through the grain boundaries was performed on various materials using surface slip trace observation [1, 2] and transmission electron microscopy [TEM] [3, 4]. However, dynamic in-situ studies have shown additional discloation-GB interaction like repulsion and re-emission not resulting in simple dislocation transmission [5, 6]. Moreover the compatibility of elastic and plastic deformation on the grain boundary brings about changes in stress distribution especially near grain boundaries [7]. The role played by the grain boundaries is rather complex and cannot be described by simple model. In this paper, the role of grain boundary type in dislocation – grain boundary interactions is studied by means of optical and TEM microscopy. Particular bicrystals were chosen to maintain equal deformation conditions in grains and to avoid redistribution of stresses near grain boundaries.

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Experimental procedure

Fe-2wt. % Si bicrystals were grown from oriented seeds by zone melting technique using induction heating [8]. In order to smooth Si inhomogeneity, the bicrystals were annealed at the temperature 1523K in H_2 atmosphere for 4hours. Two bicrystals containing a special twin boundary $\Sigma 3$ $[011]/70.53°$, GB plane $(2\bar{1}1)_A/(21\bar{1})_B$

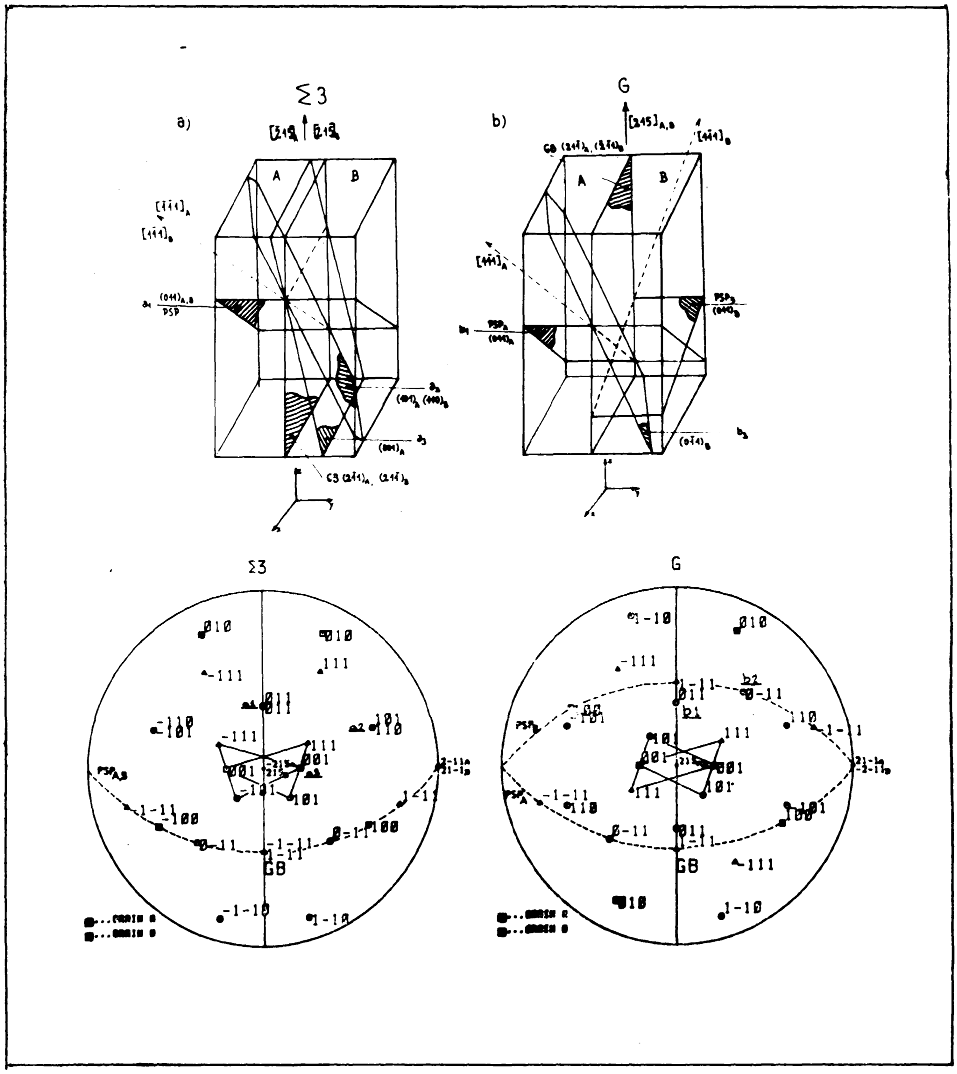

Fig. 1. Bicrystal geometry and stereographic plots along deformation axes. a1, a2, a3, b1, b2 denote foil planes cut in particular bicrystal and PSP primary slip planes.

and general grain boundary G [215]/180°, GB plane $(21\bar{1})_A/(\bar{2}\bar{2}1)_B$ were prepared (Fig. 1). The samples for tensile deformations were spark cut from annealed bicrystals to the shape of right angle prisms 3 × 5 × 40 mm. It is of particular importance that both bicrystalline grains are identically oriented for easy glide. Slip direction of primary slip systems lies in the boundary plane. The samples $\Sigma 3$ and G differs only by GB structure and by the orientation relationship of primary slip planes [PSP] in adjoining grains. They are parallel in $\Sigma 3$ bicrystal but nearly perpendicular in G bicrystal (the slip plane-GB intersection lines make an angle 80°).

Tensile tests were carried out on an INSTRON 1326 testing machine at room temperature in air with constant strain rate $5 \cdot 10^{-5}\ s^{-1}$. Totally 2 samples from each bicrystal were deformed to the strains $\varepsilon = 0.02$ and 0.04 respectively. Surface slip-patterns were observed by an optical microscope equipped with Nomarski interference contrast during and after tests. 3 differently oriented foils for TEM were cut from each sample and thinned electrolytically in double - jet apparatus METALTHIN.

Standard 3 mm disk foils were examined using double tilt goniometer stage in a PHILIPS EM 420 microscope operating at 120 kV. Bright and dark field diffraction imaging techniques were used to analyze discloation arrangements. Burgers vectors of lattice dislocatinos were determined by $g \cdot b = 0$ criterion together with trace analysis.

Results

TENSILE TESTS

A simple model based calculation [7] of additional stresses due to incompatibility of elastic and plastic deformation at the grain boundary has shown that there is no stress redistribution in symmetrical $\Sigma 3$ bicrystal and there are fine stress changes only in G bicrystal consequently to the slight elastic and plastic shear strain incompatibility. Equal homogeneous plastic shear strain on primary slip systems was supposed in both grains. The parameters of deformation curves (flow stress, yield point, hardening rate) were very similar for $\Sigma 3$ and G bicrystals. Excellent slip line continuity across GB was found for $\Sigma 3$ bicrystal (Fig. 2), where slip systems $[\bar{1}\bar{1}1]_A/(001)_A$ and $[1\bar{1}1]_B/(011)_B$ were identified as primary ones. On the contrary, almost no slip traces continuity of primary systems $[1\bar{1}1]/(011)_{A,B}$ was observed across general grain boundary. Furthermore, there is a region of secondary slip $[111]_B/(011)_B$ 50 – 100 μm wide along GB at strains higher than $\varepsilon = 0.02$.

TEM OBSERVATION

Some suitable foils, which we did not succeed to perforate in the GB vicinity, were used to study bulk dislocation structures. The latter consist of tangles of primary dislocations mostly in the form of edge dislocation dipoles and prolonged screw

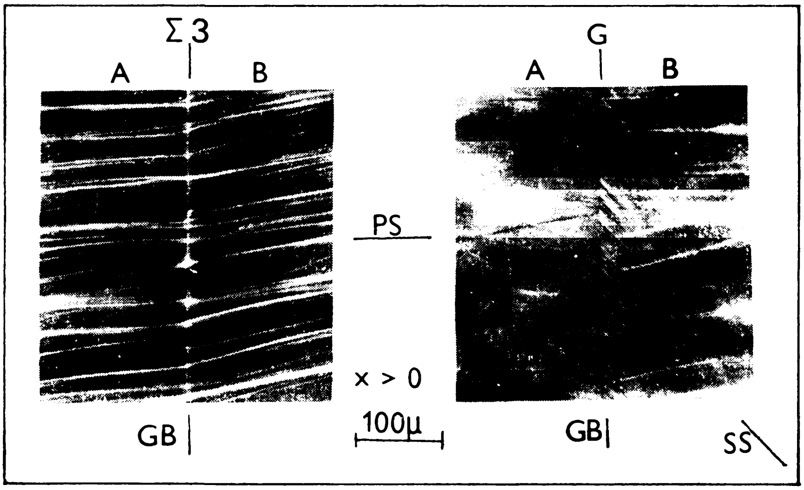

Fig. 2. Surface slip pattern of bicrystals $\Sigma 3$ and G strained to $\varepsilon = 0.01$ and $\varepsilon = 0.02$ respectively.

dislocation segments. Only rarely, the bands of secondary dislocations were observed (Fig. 3). There is not essential difference between samples strained to $\varepsilon = 0.02$ and 0.04 except of higher density of lattice dislocations in the latter case.

$\Sigma 3$ bicrystal

Dislocation arrangements near GB can be best observed on foils a1 cut along primary slip planes $(011)_{A,B}$ (Fig. 4). Grain boundary lies perpendicularly to the foil surface in this case. In addition to primary dislocations $b = 1/2[\bar{1}\bar{1}1]_A$, $1/2[1\bar{1}1]_B$ some extended screw dislocation segments $b = 1/2[\bar{1}1\bar{1}]_A$, $1/2[11\bar{1}]_B$ moving in the same slip plane were found. There is a narrow zone $1-2$ μm wide along the grain boundary, which is free of dislocation tangles but filled with very long primary screw dislocations. Structural defects in deformed $\Sigma 3$ grain boundary were best studied on a2, a3 foils with PSP and GB inclined to the foil surface (Fig. 5). Two sets of grain boundary dislocations α, β and one set of trapped primary slip dislocations [TLD] γ exhibit strong contrast in the grain boundary.

G bicrystal

Dislocation tangles in the vicinity of the grain boundary were observed on foils b1 (foil surface parallel to the PSP_A) and b2 ($PSP_{A,B}$ and GB inclined to the foil surface). They consist of dislocation segments from two independent slip systems. There is no dislocation free zone at the boundary and, on the contrary, a lot of dislocation segments hanging on GB can be seen (Fig. 6). The boundary itself is full of trapped lattice dislocations and no structural defects were recognized.

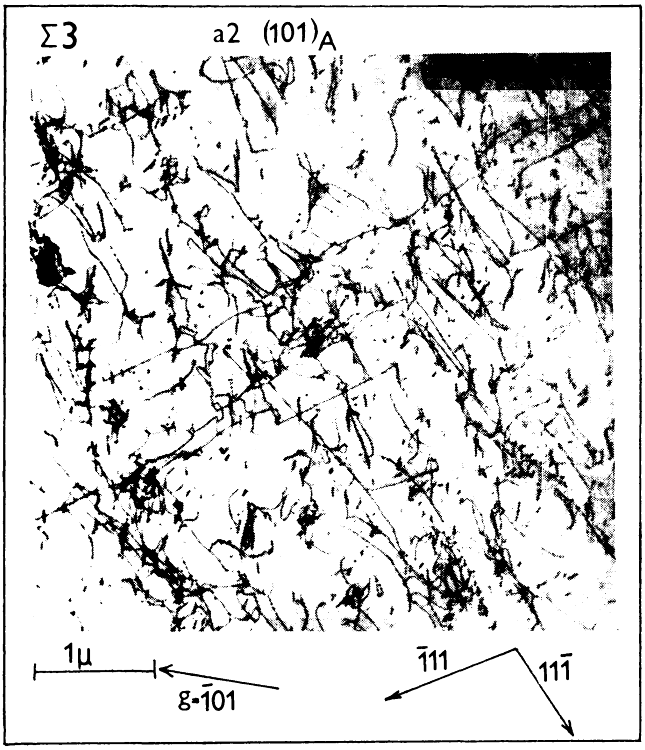

Fig. 3. Dislocation tangles far from grain boundary, foil a2, $\varepsilon = 0.02$.

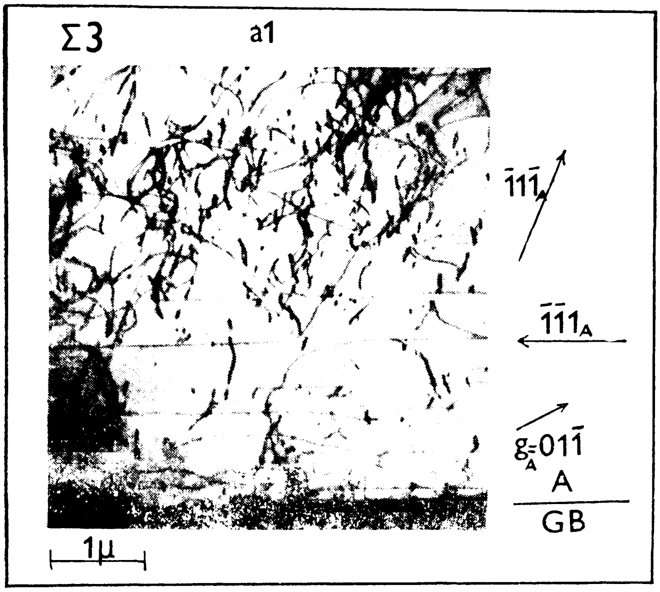

Fig. 4. Dislocation arrangements near $\Sigma 3$ grain boundary, foil a1, $\varepsilon = 0.02$.

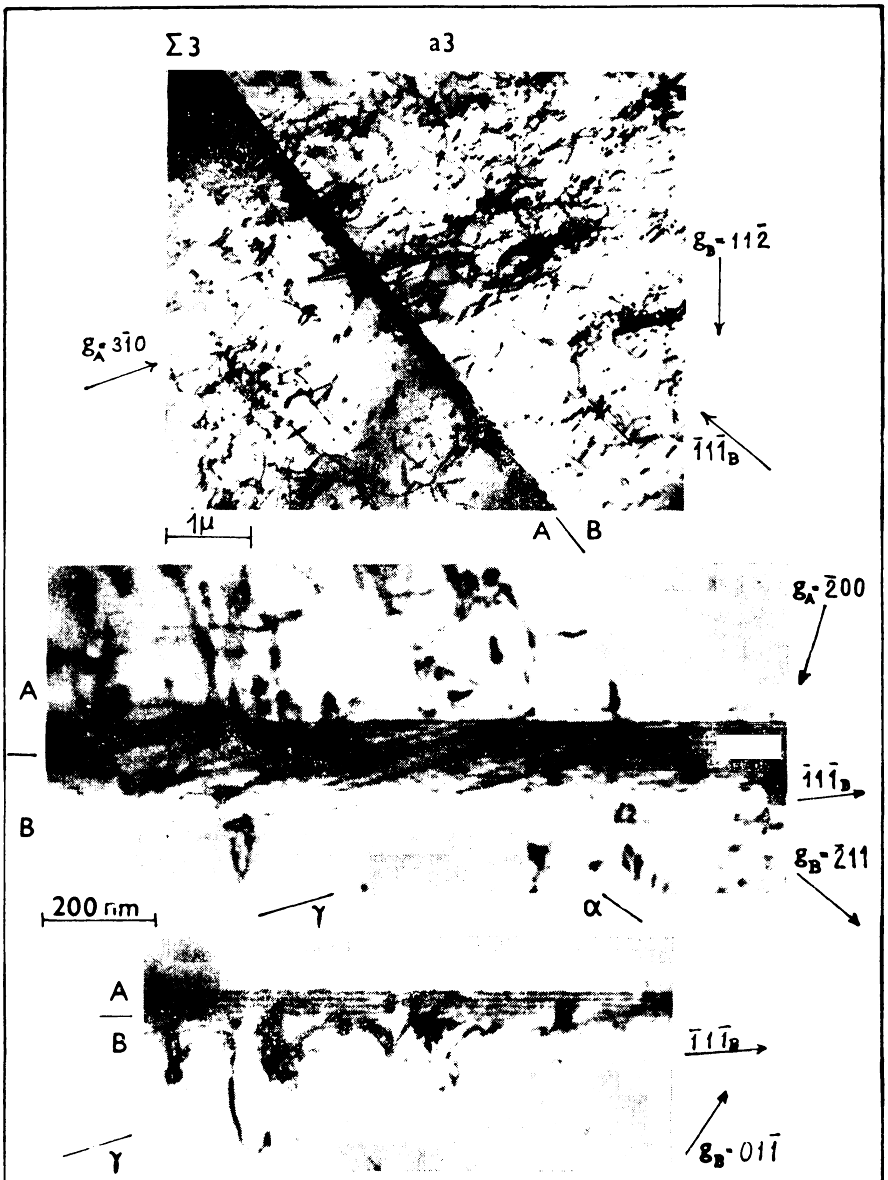

Fig. 5. Grain boundary dislocations in $\Sigma 3$ bicrystal, foil a3; $\varepsilon = 0.04$. Markings at α, γ, δ show directions of dislocation lines.

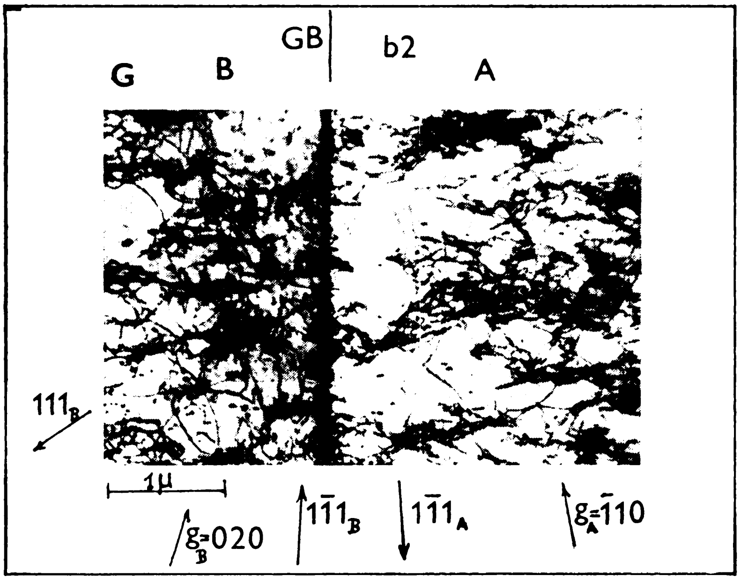

Fig. 6. Dislocation arrangements near random G boundary, foil b2, $\varepsilon = 0.04$.

Discussion

Being able to act as a barrier to moving crystal dislocations, grain boundaries impede the slip propagation throughout the crystal. However, the grain boundary may be „hard" or „soft" or in between depending on the actual crystallography of the system. The chosen $\Sigma 3$ twin boundary is considered to be of the soft type due to its high degree of symmetry. On the contrary, the random G boundary should be more effective barrier to the slip propagation. The results of surface slip observation support this concept. Similar deformation behavior of $\Sigma 3$ and G bicrystals is caused by the lack of additional compatibility stresses. The occurrence of the secondary slip region along the G boundary we ascribe to the discontinuity of PSP in adjacent grains. The primary slip can be, on the other hand, easily transmitted to the secondary slip in the neighbouring grain due to theirs nearly common slip plane-GB intersection lines.

The comparison of dislocation structures found in bulk and near grain boundaries does not reveal any special deformation zone along grain boundaries neither in $\Sigma 3$ nor in G bicrystals. The fact that such zones are often reported by other authors [9] suggests a great role of additional and pile-up stresses, which were not observed in our study. The existence of narrow dislocation tangles free zone in $\Sigma 3$ bicrystal we explain by image forces, which act on dislocations near GB in an anisotropic medium. According to Khafallah et al. [10], who calculated these forces for $\Sigma 3$ twin in iron, all dislocation segments are repelled from GB with exceptions of screws b = $1/2[\bar{1}\bar{1}1]_A$, $1/2[1\bar{1}1]_B$, which are attracted. Data for G configuration are not available, nevertheless, an attractive image forces are proposed for most of random grain boundaries and b = $1/2<111>$ dislocations. This is in a good agreement with our results. The adjusted distance d = 0.3 – 1 μm, on which these image forces may be felt by moving dislocations is approximately equal to the width of dislocation tangles free zones.

Main difference between $\Sigma 3$ and G boundary deformation lies in the density of TLD observed in grain boundaries. This is relatively low in $\Sigma 3$ boundary, where in addition to TLD, secondary grain boundary dislocations and rotational Moire fringes were resolved. Especially low amount of trapped primary slip dislocations and observed events of dislocation transmission supports the idea that $\Sigma 3$ boundary in this configuration is not an effective sink for these dislocations. They seem to be easily transmitted to the neighbouring grain in spite of the supposed attractive image force between grain boundary and screw dislocation.

$$1/2[\bar{1}\bar{1}1]_A \rightarrow 1/2[1\bar{1}1]_B \tag{1}$$

This process is documented on Fig. 5b.

In G bicrystal, on the contrary, the amount of TLD is so high, that the identification of individual line defects in GB was hardly possible. The presence of secondary slip dislocation segments in tangles near G boundary (up to 200 μm away from GB) corresponds to the surface slip line pattern showing double slip zone as well. Short dislocation segments hanging on the grain boundary come from both primary and secondary slip systems which have nearly common intersection line with GB plane. We consider them to be relics of these reactions:

$$\begin{aligned} 1/2[1\bar{1}1]_A &\rightarrow 1/2[111]_B + \vec{b}_{res1} \qquad (2) \\ 1/2[1\bar{1}1]_B &\rightarrow 1/2[111]_A + \vec{b}_{res2} \end{aligned}$$

According to the latest results [11,6] every dislocation transmission must be accomplished by its decomposition into GBD and back recombination of lattice dislocation even in the case of special high symmetry $\Sigma 3$ system. Such process is rather difficult due to the limited diffusion at room temperature. This is the reason, why the reaction 1 in $\Sigma 3$ bicrystal takes place regularly ($\vec{b}_{res} = 0$), while reactions 2 do not seem to be important in G bicrystal deformation. Nevertheless, we assume the increased amount of secondary dislocations near GB to have its origin in the reaction 2. There were no secondary dislocations near $\Sigma 3$ boundary in spite of the same resolved shear stress and latent hardening. Low additional compatibility stresses in the G bicrystal further support this idea. The effectivity of reaction 2 depends sensitively on the value of pile-up stresses (Baillin et al. [6] estimated the pile up stress due to grain boundary in Si to be 10 times higher than critical resolved shear stress in particular slip system). The lack of strong pile-ups in Fe-Si bicrystals suppresses the effectivity of this mechanism in present experiment.

Conclusions

Two Fe-2wt.%Si bicrystals $\Sigma 3$ (special twin and random G boundary) with all grains oriented for single slip were deformed in tension. Dislocation structures in bulk and near grain boundaries were observed by TEM and compared to the surface slip pattern.

No special deformation zone along grain boundaries was found, which is linked to the lack of additional stresses there. The origin of narrow dislocation tangles free zone along $\Sigma 3$ grain boundary is ascribed to image forces between grain boundary and dislocations.

The type of the grain boundary seems to be essential in bicrystal deformation not only in a consequence of strain compabibility at grain boundaries, but also due to reactions with lattice dislocations. The random grain boundary G was shown to be more effective sink for moving $1/2\langle 111\rangle$ primary slip dislocations than $\Sigma 3$ twin boundary.

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