Deformation mechanisms of $\Sigma = 9$ bicrystals of silicon

M. El Kajbaji, J. Thibault-Desseaux, M. Martinez-Hernandez, Alain Jacques,
A. George

► To cite this version:

M. El Kajbaji, J. Thibault-Desseaux, M. Martinez-Hernandez, Alain Jacques, A. George. Deformation mechanisms of $\Sigma = 9$ bicrystals of silicon. *Revue de Physique Appliquée*, 1987, 22 (7), pp.569-577. 10.1051/rphysap:01987002207056900 . jpa-00245578

HAL Id: jpa-00245578

<https://hal.science/jpa-00245578>

Submitted on 1 Jan 1987

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Classification

Physics Abstracts

61.70JNY — 62.20F — 68.25

Deformation mechanisms of $\Sigma = 9$ bicrystals of silicon

M. El Kajbaji (*), J. Thibault-Desseaux (*), M. Martinez-Hernandez (**), A. Jacques (**) and A. George (**)

(*) Centre d'Etudes Nucléaires de Grenoble, Département de Recherche Fondamentale, Service de Physique, Groupe Structures, 85 X, 38041 Grenoble, France

(**) Laboratoire de Physique du Solide, Unité Associée au CNRS n° 155, E.N.S.M.N., Institut National Polytechnique de Lorraine, Parc de Saurupt, 54042 Nancy, France and LURE, Laboratoire du CNRS conventionné à l'Université de Paris-Sud, 91405 Orsay, France

(Reçu le 7 octobre 1986, accepté le 1^{er} décembre 1986)

Résumé. — Les interactions entre dislocations et joint de grains $\Sigma = 9$ ont été étudiées à l'aide de plusieurs techniques dans des bicristaux de silicium légèrement déformés. La microscopie électronique par transmission révèle des arrangements très complexes au voisinage du joint de grains : empilements, réseaux avec barrières de Lomer-Cottrell, nombreux glissements déviés, inversions locales du signe de la contrainte. Des observations *in situ* par topographie aux rayons X avec faisceau synchrotron suggèrent que les dislocations peuvent dans certains cas traverser le joint $\Sigma = 9$. Sauf pour les dislocations de vecteur de Burgers $a/2$ [011], commun aux deux grains, la réalité d'un mécanisme de transmission directe, à l'échelle atomique, n'est pas confirmée par les observations en MEHR qui montrent que les dislocations se dissocient dans le joint en dislocations du réseau DSC. Ces résultats apparemment contradictoires sont discutés en fonction des conditions de déformation et de la structure de cœur des dislocations.

Abstract. — Interactions between dislocations and $\Sigma = 9$ grain boundaries were studied in slightly deformed silicon bicrystals by several imaging techniques. A very complex dislocation arrangement in the grains close to the grain boundary was revealed by TEM : pile-ups, networks with Lomer-Cottrell barriers, profuse cross-slip, local stress reversals. *In situ* observations by synchrotron X-ray topography suggest the possibility of dislocation transmission by $\Sigma = 9$ grain boundaries. Except for dislocations with the $a/2$ [011] Burgers vector, common to both grains, the reality of a direct transmission mechanism at the atomic scale was not confirmed by HREM investigations which proved that lattice dislocations dissociate in the grain boundary into DSC dislocations. These apparently contradictory results are discussed in terms of deformation conditions and core structures of dislocations.

1. Introduction.

The importance of grain boundaries (GB) in the strength of crystalline materials has long been recognized. There are, however, not so many detailed investigations on interactions and reactions of lattice dislocations with GBs, during plastic deformation. GBs have been proved to be strong obstacles to dislocation motion. They can be important sources of incompatibility stresses from both elastic and plastic origins [1, 2]. They seem to act as sinks for dislocations under some circumstances but they are also claimed to be possible dislocation sources, and it is speculated that GBs might be in favourable cases permeable by dislocations. See for example [3, 4] for a review of these problems.

The successful pulling of large size, dislocation-free, precisely oriented silicon bicrystals provided a very good opportunity to undertake a new study of interactions between dislocations and a high-angle GB of well defined and fully ordered atomic structure. The $\Sigma = 9$ GB was selected for that purpose. Its atomic structure has been studied in detail [5-8] and can be described as a periodic arrangement of A and B structural units made up of five and seven atom rings. A and B units correspond to each other in a glide $(1/4 [41\bar{1}]_I)$ mirror $((1\bar{2}2)_I)$ symmetry (Fig. 1). The structure has no dangling bond and each A or B unit can be viewed as the core of a perfect Lomer dislocation. GB dislocations are con-

Fig. 1. — [011] projection of the $(\bar{1}\bar{2}2)$ $\Sigma = 9$ symmetric tilt boundary. — CSL unit cell, - - - DSCL unit cell (nodes at the center of the cell at $a/4$ [011] above the plane of the figure are not represented, ■ atoms in the plane of the figure, • atoms $1/4$ [011] above.

veniently described in the DSC lattice whose basis vectors expressed in the lattice I cubic basis are :

$$\begin{aligned} \text{dsc}_1 &= 1/18 [41\bar{1}]_I \\ \text{dsc}_2 &= 1/9 [\bar{1}\bar{2}\bar{2}]_I \\ \text{dsc}_3 &= 1/6 [121]_I. \end{aligned}$$

Experiments reported on here combined several imaging techniques with very different resolutions and fields of view : optical microscopy, sometimes after chemical etching, X-Ray Topography (XRT), conventional electron microscopy (TEM) and high resolution electron microscopy (HREM). Each technique was used after or during (*in situ* observations) a suitable mechanical treatment (paragraph 2). It is very clear indeed that dislocation mechanisms close to GBs are various and complex and that special simple configurations must be tailored in order to yield information on local mechanisms.

Special attention was paid to the accommodation or absorption of dislocations in a perfect $\Sigma = 9$ boundary and to the possibility of transmission of dislocations from one grain to the other. The former process is achieved *via* the dissociation of the incoming dislocation of Burgers vector $\mathbf{b}$ into GB dislocations with Burgers vectors of the DSC lattice, with the total Burgers vector preserved :

$$\mathbf{b} = \Sigma \mathbf{b}^{\text{GB}}. \quad (1)$$

A simple form of the transmission process can be written as :

dislocation I $\rightarrow$ dislocation II + GB dislocation
with

$$\mathbf{b}^{\text{I}} = \mathbf{b}^{\text{II}} + \mathbf{b}^{\text{GB}}. \quad (2)$$

Usually GB dislocations introduce associated steps in the GB plane. In the following these are defined and determined according to King and Smith [9].

It must be stressed that may be none of these two processes is really important if the mechanical behaviour of polycrystals is considered. For this problem predominant may be the very special dislocation arrangements formed inside the grains, in the vicinity of GBs. A few words about such arrangements observed in the yield region of Si bicrystals will be said first.

2. Experimental.

$\Sigma = 9$ bicrystals were obtained by Czochralski pulling using a seed cut in an accidentally occurred second-order twin. Both grains and the GB plane are dislocation-free. Crystals contain about 3×10^{17} oxygen atoms per cm^3 .

During deformation experiments, stress was applied along $[\bar{2}6, 7, 20]_I$, i.e. a « single slip » orientation, parallel to the GB plane and equivalent for both crystals. Two kinds of mechanical tests were performed.

i) Compression tests at constant strain rate ($\dot{\epsilon} \approx 6 \times 10^{-6} \text{ s}^{-1}$) in the temperature range $720^\circ\text{C} - 850^\circ\text{C}$. Most of tests were stopped in the yield region and samples cooled down under load in order to freeze-in the dislocation arrangements for TEM and HREM observations. Dimensions of compression samples were $14 \times 4.25 \times 4.25 \text{ mm}^3$.

ii) Creep tests in tension with thinner ($\sim 0.7 \text{ mm}$) samples convenient for transmission XRT. Typical conditions were $\sigma \approx 45 \text{ MPa}$ (σ , nominal stress), $690^\circ\text{C} \leq T \leq 800^\circ\text{C}$. *In situ* observations were possible thanks to the synchrotron radiation of LURE-DCI and using a hot deformation stage described in [10]. The X-Ray beam was monochromatized by 220 reflection on a Ge crystal (+ n , + n setting) selecting a $\lambda \approx 0.08 \text{ nm}$ wavelength. The instant of topograph exposure could be chosen by the permanent control of the diffracted image with a TV system. Images were recorded on Ilford L4-100 μm nuclear plates. The two grains of the bicrystal were imaged either alternately with 220 reflections (changing the Bragg setting from one grain to the other took about 2 min) or simultaneously with common $1\bar{1}3_I/\bar{1}\bar{1}3_{II}$ reflections. A typical exposure time was 20 s with DCI operating at 1.85 GeV, 200 mA.

In situ observations were supplemented with detailed investigations of frozen-in dislocation configurations using the conventional Lang technique. When the dislocation density was too high, topographic work was complemented by etch pits.

In these creep experiments, dislocation loops were created in one grain from a scratch or micro-indentations. (The possibility of confining starting disloca-

tions in one grain is a great advantage of XRT experiments and is particularly useful to study slip propagation from one grain to the other when dislocations reached the GB plane. Similar situation could not be achieved in samples prepared for TEM or HREM because of the higher dislocation density required).

All deformation experiments were conducted under reducing atmosphere (10 % H_2 , 90 % N_2).

TEM and HREM works were done with JEOL 200 CX microscopes. Two foil orientations were used to resolve the complex 3-dimensional dislocation arrangement close to the GB in deformed bicrystals [11]. In HREM the common [011] axis had to be parallel to the electron beam in order to resolve the GB structure and only those dislocations which run along [011] could be studied. With the chosen stress axis, dislocations of the primary slip systems — i.e. of highest Schmid factor, s — $(11\bar{1})_I [1\bar{1}0]_I$ and $(1\bar{1}1)_{II} [10\bar{1}]_{II}$ $s: 0.47$ fulfill this condition, and also those of the second slip system in primary planes $(11\bar{1})_I [011]_I$, $(1\bar{1}1)_{II} [011]_{II}$ ($s: 0.38$) of particular interest since the Burgers vector is common to both crystals. Dislocations lying in $(1\bar{1}1)_I$ and $(11\bar{1})_{II}$ could also be observed by HREM (Fig. 2).

3. Results.

3.1 PRELIMINARY REMARKS. — i) With the chosen stress axis, compatibility at the GB is achieved for both elastic and plastic strains. As plastic strain

Fig. 2. — Deformation geometry (tensile sample). Sketch of $\{111\}$ slip planes. s : corresponding Schmid factors. $(11\bar{1})_I$ and $(11\bar{1})_{II}$ feel negligible shear stresses and are not represented.

increases, in agreement with similar observations in Ge [12], the GB plane remains a plane of symmetry of the sample, [011] stands as the tilt axis of the bicrystal, but the misorientation angle continuously evolves, decreasing in the case of tension, increasing in compression. Therefore, in order to create small and localized perturbations of the equilibrium structure of the $\Sigma = 9$ GB, the deformation must be restricted to its very first stage i.e. in the upper yield region.

ii) Little information is gained from stress-strain curves: bicrystals behave like single crystals of same orientation, with equal upper yield stress. The lower yield stress and hardening rate in the stage I of easy glide are slightly higher in bicrystals but these hardly significant differences may be attributed to different end-effects (because of the different strain induced rotation of primary slip planes in bicrystals and single crystals) rather than to a specific influence of the GB. This similar behaviour was expected since compatibility conditions are fulfilled.

iii) Slip trace analysis by optical microscopy gives a rough information about slip distribution at the scale of the sample gauge length. At strains beyond the lower yield point, only primary slip traces are visible. In the yield region deformation proceeds by sharp isolated slip bands and is highly inhomogeneous starting from sample ends, as usually observed in dislocation-free Czochralski silicon single crystals. This is interesting for our concern since slip lines are seen to cross the GB i.e. apparent one-to-one correspondances at the boundary between slip planes in the two grains are observed [13]. This is a proof that plastic deformation can propagate across the $\Sigma = 9$ GB but Burgers vectors cannot be determined and the resolution is much too low to give any information on the transfer mechanism: a single slip line may consists of several tens of activated slip planes. Noteworthy, however, is the appearance, at this stage, of $(11\bar{1})_{II}/(1\bar{1}1)_I$ traces in connection at the GB plane with primary slip lines $(11\bar{1})_I/(1\bar{1}1)_{II}$, respectively.

3.2 TEM OBSERVATIONS OF THE DISLOCATION ARRANGEMENT NEAR THE GRAIN BOUNDARY. — Even restricted at the upper yield point, deformation induces a very high dislocation density in the GB plane ($\approx 5 \times 10^5 \text{ cm} \cdot \text{cm}^{-2}$), where practically all dislocations are aligned parallel to the traces of available slip planes, and in a stripe of material extending to about $50 \mu\text{m}$ on each side of the GB. This makes difficult any detailed analysis of interactions between dislocations and the $\Sigma = 9$ boundaries. Interesting features rather concern the arrangement of dislocations close to the GB [11], which can be summarized as follows.

i) Most dislocations belong to the primary slip

systems but all $1/2\langle 110 \rangle$ Burgers vectors could be found.

ii) Dislocations form pile-ups against the GB plane. Such pile-ups are usually short (less than 10 dislocations).

iii) Long pile-ups are relaxed by cross-slip which is very common and provides a way to homogenize the distribution of dislocations.

iv) As in semiconductor single crystals, secondary slip systems are activated in the yield region, because of the high stress level reached at the beginning of deformation (upper yield point) before the fast multiplication of primary dislocations has allowed to achieve the imposed strain rate at a reduced stress level (lower yield point). When secondary dislocations are stopped at the boundary and crossed by primary slip bands, networks can form as shown in figure 3 with Lomer-Cottrell dislocations resulting from the reaction of the former two slip systems. Such arrangements frequent in the GB area of bicrystals are never observed in single crystals at this stage of deformation.

v) A close inspection of dislocation contrast and curvature reveals very complex internal stress fields, which may have not been expected in this fully compatible situation. This point is described in [10]. It appears that at a given point the sign of the stress exerted on a given dislocation is sometimes reversed during the deformation, probably because a new pile-up has formed in the vicinity, in the same grain or in the other one. Consequently the observation of an arc of dislocation bowing out of the GB plane is by no means a proof that this dislocation has come from the opposite grain i.e. has crossed the GB.

Fig. 3. — Dislocation networks with formation of Lomer-Cottrell barriers according to the reaction :

(Marker 1 μm).

Fig. 4. — Dislocation configuration suggesting the transmission of dislocations by $\Sigma = 9$ GB according to :

The configuration has been stabilized by reactions with other dislocations ; see [11] for a detailed description. (Marker 1 μm).

vi) With this reservation in mind, we have however observed configurations which seem to point that dislocation transmission through $\Sigma = 9$ GB is possible, although difficult. A good example is given in figure 4. The transmission reaction observed is one of those suggested by the XRT experiments described below.

Such configurations are very seldom and most configurations extending in the two grains with an apparent coincidence at the GB are very likely to have been formed by dislocations running towards the GB from both grains. It has not been determined if such a meeting is driven by internal stresses or if it is fortuitous. In the latter case it may also be argued that a close meeting at the GB may stabilize in the two grains extended configurations that would otherwise have been relaxed, by cross-slip, for example.

3.3 SYNCHROTRON X-RAY TOPOGRAPHY EXPERIMENTS AND THE TRANSMISSION OF DISLOCATIONS ACROSS $\Sigma = 9$ BOUNDARIES. — Figure 5 illustrates the development of dislocation loops produced at micro-indentations in grain I and followed *in situ* by Synchrotron XRT. The observed slip systems are the three (sometimes four) systems of highest Schmid factors. It appears clearly that when leading dislocations reached the GB plane, they were stopped and their accumulation developed long range stresses in grain II which induced strong and diffuse white and black contrast. In the present case no dislocation could be detected in grain II developing in these highly stressed regions.

Figure 6 shows a more positive result. Here dislocations were created from a scratch and they

Fig. 5. — a-c) Sequence of synchrotron X-Ray topographs. $1\bar{1}3_I/1\bar{1}3_{II}$ common Bragg reflection. Dislocation groups are created from Vickers micro-indentations. $T = 700^\circ\text{C}$, $\sigma = 45\text{ MPa}$ (Marker : 1 mm), d) Lang topograph, detail.

Fig. 6. — Transmission of dislocations with $a/2 [011]$ Burgers vector. $\Sigma = 9$ bicrystal deformed 25 mm at $T = 715^\circ\text{C}$, $\sigma = 45\text{ MPa}$. Lang topograph (Marker 1 mm).

accumulate over a large fraction of the GB area. Whilst most dislocations were stopped, a few dislocation groups were able to develop in the second grain, running from the GB towards the interior of the grain on $(1\bar{1}1)_{II}$ planes. These groups do not consist of primary dislocations but of dislocations having the common $1/2 [011]$ Burgers vector.

Qualitatively different is the case depicted in figure 7. A higher dislocation density was developed in the scratched grain and large resulting elastic strains put out of contrast parts of the dislocated zone. At some places at the GB, groups of dislocations belonging to several slip planes were seen to develop in the second grain. Etch pits (Fig. 7b) give a better view of these areas. Burgers vectors could be determined and suggested the following transmission reactions :

$$\mathbf{b}^I = \frac{a}{2} [011]_I (11\bar{1})_I \rightarrow \mathbf{b}^{II} = \frac{a}{2} [011]_{II} (1\bar{1}1)_{II} \quad (1)$$

$$\mathbf{b}^I = \frac{a}{2} [\bar{1}10]_I (11\bar{1})_I \rightarrow \mathbf{b}^{II} = \frac{a}{2} [\bar{1}01]_{II} (1\bar{1}1)_{II} + \mathbf{b}^{GB} = \frac{a}{3} [\bar{1}2\bar{2}]_I = +3 \mathbf{dsc}_2 \quad (2)$$

$$\mathbf{b}^I = \frac{a}{2} [\bar{1}10]_I (11\bar{1})_I \rightarrow \mathbf{b}^{II} = \frac{a}{2} [\bar{1}10]_{II} (11\bar{1})_{II} + \mathbf{b}^{GB} = \frac{a}{18} [25\bar{5}]_I = \mathbf{dsc}_1 + \mathbf{dsc}_2 \quad (3)$$

Fig. 7. — Transmission of several types of dislocations. $\Sigma = 9$ bicrystal deformed 35 min at $T = 700^\circ\text{C}$, $\sigma = 45\text{ MPa}$. a) Lang topograph (Marker 1 mm). b) Typical etch pit configurations.

$$\begin{aligned} \mathbf{b}^I &= \frac{a}{2} [110]_I (\bar{1}11)_I \rightarrow \\ \mathbf{b}^{II} &= \frac{a}{2} [101]_{II} (\bar{1}11)_{II} + \\ &+ \mathbf{b}^{GB} = -\mathbf{dsc}_2. \quad (4) \end{aligned}$$

(The signs are for dislocations, oriented positively along $+\frac{1}{\sqrt{2}} [011]$, gliding towards the GB in crystal I and away from it in crystal II in a deformation in tension).

The main result of SXRT experiments is that transmission of slip through $\Sigma = 9$ GBs is difficult and requires large stress concentrations. A. Jacques [14, 15] has calculated shear stresses exerted on all available slip systems of grain II by dislocations piled-up against the GB in grain I, with the result that pile-up stresses are actually effective to help all reactions listed here.

However, all these reactions are not likely to occur in such simple a form. Because of its limited resolution, XRT cannot prove that transmission is a one-to-one dislocation process. It may be argued as well that pile-up stresses are able to activate pre-existing dislocation sources in the vicinity of the GB. Such indirect mechanisms would also account for the apparent continuity of slip traces. Sources could be grown-in defects, such as swirl defects, which are too small to be detected by XRT and whose density is so low that there is very few chance to see them by TEM.

Especially the direct transmission of primary dislocations (reaction 2) appears to be very unlikely. Reaction 2 would create a GB dislocation with a very large Burgers vector ($|\mathbf{b}^{GB}| = 5.43 \text{ \AA}$), the motion of which would in addition require pure climb in the GB plane. Furthermore in this very case, the pile-up stress is much more efficient at remote distance than at the spot of incoming head dislocation.

The direct transmission of dislocations with the common $1/2 [011]$ Burgers vector, on the contrary, has been proved in Ge $\Sigma = 9$ bicrystals by *in situ* HVEM experiments [13] and reaction 4 has received some support from TEM observations (Fig. 4).

3.4 HREM OBSERVATIONS OF DISLOCATION DISSOCIATION IN $\Sigma = 9$ BOUNDARIES. — So far HREM observations have been done only in bicrystals deformed in compression at 850 °C. They gave the following results :

i) primary dislocations and dislocations with $b = a/2 [011]$ in primary slip planes have been found in both grains, inhomogeneously distributed along the GB. As they are aligned parallel to the GB plane, those dislocations are — and will be referred to as — 60° and screw dislocations, respectively. 60° dislocations are often arranged in pile-ups ;

Fig. 8. — a) 60° dislocation entering the GB. b) The 90° partial has been dissociated in the GB plane, emitting a $\mathbf{b}_g$ dislocation. (It is noticeable that this dissociation took place in the electron microscope, which proves the high mobility of $\mathbf{b}_g$ dislocations).

ii) all dislocations are dissociated into Shockley partials, with an intrinsic stacking fault. In compression a 60° dislocation glides towards the GB with the 90° partial as the leading one ;

iii) 60° dislocations dissociate in the GB plane into GB dislocations with the smallest DSCL vectors as Burgers vectors. The dissociation process was established to be the following :

When the 90° partial enters the GB it dissociates first according to (Fig. 8) :

$$\mathbf{b}_{90^\circ} \rightarrow \mathbf{b}_c + \mathbf{b}_g$$

with

$$\mathbf{b}_c = \frac{a}{9} [1\bar{2}2]_I = -\mathbf{dsc}_2$$

$$\mathbf{b}_g = \frac{a}{18} [41\bar{1}]_I = \mathbf{dsc}_1$$

which reaction occurs through the glide of the dislocation $\mathbf{b}_g$ in the GB plane.

It appears that the trailing 30° partial is then able

to enter the GB, forming at the impact point a residual dislocation of Burgers vector :

$$\mathbf{b}_r = \mathbf{b}_c + \mathbf{b}_{30}$$

which in turn dissociates into these two components, which implies that at least one of them is mobile, a process which involves climb in the GB.

iv) When two 60° dislocations enter the GB, the one from grain I and the other from grain II, their dissociation creates two $\mathbf{b}_g$ dislocations of opposite signs, which may annihilate since they are highly mobile in the GB plane ($\mathbf{b}_g^I + \mathbf{b}_g^{II} = 0$), two identical $\mathbf{b}_c$ dislocations and two 30° dislocations $\mathbf{b}_{30}^I$ and $\mathbf{b}_{30}^{II}$, whose components of Burgers vector parallel to the GB cancel, whilst normal components add, with the possible reaction :

$$\mathbf{b}_{30}^I + \mathbf{b}_{30}^{II} = \mathbf{b}_c \quad (\text{Figs. 9, 10})$$

This reaction would explain the observations of groups of three $\mathbf{b}_c$ dislocations, which can be formed by the motion of $\mathbf{b}_{30}$ only and are expected if the impact points of $\mathbf{b}_{60}^I$ and $\mathbf{b}_{60}^{II}$ at the GB are not too far from each other. There is no evidence that $\mathbf{b}_c$ dislocations are mobile in the deformation conditions investigated so far.

v) Other reactions can be found. For example, the intermediate configuration $\mathbf{b}_r$ can be modified by a moving dislocation $\mathbf{b}_g$ of opposite sign :

$$\mathbf{b}_r' = \mathbf{b}_r + \mathbf{b}_g \quad (\text{Fig. 9})$$

vi) Screw dislocations consist of two partial 30° dislocations. As these do not dissociate in the GB

Fig. 9. — Sketch of dislocation reactions in the GB. Burgers vectors are presented on a projected view of the DSC lattice.

Fig. 10. — Typical view of the GB in a $\Sigma = 9$ Si bicrystal deformed beyond the upper yield point at $T = 850^\circ\text{C}$, $\dot{\epsilon} = 8 \times 10^{-6} \text{ s}^{-1}$.

one could expect the formation of pairs of two $\mathbf{b}_{30l}$, $\mathbf{b}_{30t}$ dislocations in the GB plane. Such pairs have never been observed, which is taken as an indirect evidence of the easy transmission of dislocations with $\mathbf{b} = a/2 [011]$ by $\Sigma = 9$ GB. However, non-dissociated screw dislocations cannot be detected by HREM and it is not known whether transmission occurs after recombination in the GB plane, which would require extra energy for the collapse of the stacking fault or if the leading partial is transmitted first, which also requires energy since an intermediate residual GB dislocation has then to be created before being annihilated by the trailing partial when this is in turn transmitted.

vii) The net result of dislocation interactions with $\Sigma = 9$ GB is the accumulation of $\mathbf{b}_c$ dislocations in the GB plane (Fig. 10). If these dislocations were homogeneously distributed, a subgrain boundary of the same tilt axis would superimpose to the initial $\Sigma = 9$ GB. In agreement with observations, the angle of the resulting new GB would increase with strain, in compression.

viii) Associated steps could be directly measured. A $\mathbf{b}_c$ dislocation is associated to equal and opposite step vectors in the two grains i.e. the average GB plane is not sheared and, if this is taken as the reference, $\mathbf{h}_c = 0$.

On the contrary, other GB dislocations have non-zero associated steps :

$$\begin{aligned} \mathbf{h}_g &= d \quad \text{with} \quad d = a/9 [\bar{1}22]_{\text{I}} \\ \mathbf{h}_{30} &= -1.75 d \\ \mathbf{h}_t &= -1.75 d. \end{aligned} \quad (\text{Fig. 10})$$

Reactions can lead to more complex dislocations with non unit DSC Burgers vectors. As explained by El Kajbaji [16] the formation of such complex configurations can be understood by considering not only Burgers vectors but also the associated steps.

Remarks :

1) In present HREM observations most dislocations that could be detected lie in primary slip planes, $(11\bar{1})_{\text{I}}$ or $(1\bar{1}1)_{\text{II}}$. These dislocations are particular in the sense that Shockley partial dislocations lying in these planes have Burgers vectors which belong to the DSC lattice. This not the case for dislocations lying in other $\{111\}$ planes [17].

2) Models of the core structure of DSC dislocations parallel to $[011]$ have been deduced from HREM observations [16]. They will be detailed elsewhere. The result to be mentioned here is that these dislocations do not seem to have dangling bonds. Particularly $\mathbf{b}_c$ and $\mathbf{b}_{30}$ dislocation cores could be reconstructed in the GB plane in a way very similar to that of 90° and 30° partials respectively in the bulk.

3) Dislocation-GB interactions do not seem to be affected by oxygen atoms dissolved in Czochralski grown Si crystals. Few precipitates were observed by HREM. Large ones containing Cu are related to accidental contamination during experiments and can be avoided with sufficient care, smaller ones could not be identified. Yet, most dislocations and GB areas appear to be « clean ».

4. Discussion.

4.1 GRAIN BOUNDARY HARDENING : GB INDUCED DISLOCATION INTERACTIONS. — TEM observations have revealed a very special situation in the grains, close to the GB : at the onset of plastic flow and whereas the deformations of the two grains are fully compatible, formation of dislocation networks with Lomer-Cottrell barriers have been found, a situation typical of the stage II of the hardening curve in single crystals, and also profuse cross-slip, a stress-release mechanism typical of stage III. It appears therefore that a thin stripe of material is in a much

more advanced state of deformation than the rest of the bicrystal.

Clearly this cannot induce a large hardening of the total sample since the affected volume is rather small compared with the total volume. The heavily deformed stripe is not even continuous along the GB and one question may be whether it expands over the entire GB length and thickens towards grain interior when strain increases or whether it remains confined in some parts of the GB area. The presents authors idea is that this « advanced state of deformation » could be a transient phenomenon specific to semiconductors or materials with a very low initial dislocation density i.e. exhibiting marked yield point phenomena. As discussed in [11], it appears that secondary slip bands necessary to built networks have not developed at the GB but moved towards it and stopped at it. As strain increases beyond the upper yield point, the stress drop should decrease secondary slip activity whilst primary slip becomes more and more homogeneous. It is however possible that the rather particular stress field built up near the GB initiates or develops secondary slips in the area. This point has to be elucidated by further investigations in more heavily deformed bicrystals.

Noteworthy, the observed situation is not typical of any special GB. It has been also observed in $\Sigma = 25$ (001) tilt bicrystals [11] and only requires that the GB is not transparent for dislocations.

4.2 REACTIONS BETWEEN DISLOCATIONS AND $\Sigma = 9$ GB : THE ALTERNATIVE BETWEEN DISSOCIATION INTO DSC DISLOCATIONS AND TRANSMISSION. — Results obtained by SXRT and HREM seem to be contradictory. No evidence was obtained by HREM for any of the transmission reactions suggested by SXRT except for the rather trivial case of dislocations with the common $a/2$ $[011]$ Burgers vector.

A possible conclusion might be that for other reactions, the transmission process is indirect. The possible activation of pre-existing sources (to be determined !) by pile-up stresses has already been mentioned. HREM suggests another possibility : as GB dislocations accumulate, reactions between elemental dislocations like $\mathbf{b}_c$, $\mathbf{b}_g$, $\mathbf{b}_{30}$... might occasionally produce a convenient nucleus for a $a/2 \langle 110 \rangle$ dislocation that could be emitted in one of the two grains. Such a mechanism, however, has not yet been identified.

On the other hand, it must be pointed out that the deformation geometry was not strictly equivalent in the two kinds of experiments reported above. HREM experiments prove the dissociation into DSC dislocations of 60° dislocations gliding towards the GB with the 90° partial in leading position. In tension, the leading partial would be the 30° partial which does not dissociate in $\Sigma = 9$ GB and could be

therefore more easily transmitted in the opposite grain. Configurations resulting from the transmission of the leading partial, without prior recombination in the GB plane, have been analysed in some details in [15, 18]. Such configurations, if they exist, could be detected by HREM only if they are sufficiently stable, which should not be the case for screw dislocations, the only studied case identical in tension and compression. Direct transmission of primary dislocations appears unlikely in any case but indications supporting reaction 3 (paragraph 3.3) should be carefully looked for in samples deformed in tension.

Another point may be emphasized: the probability to freeze-in a dislocation that has been transmitted sufficiently close to the GB in order that it can be detected by HREM is very small if, as assumed in [15], it glides away from the GB under the externally applied stress. Further investigations by *in situ* experiments, with the much better resolution of HVEM could be helpful especially to study

dislocations not parallel to the tilt axis. This work is under progress, with the cooperation of Baillin, Bacmann and Pelissier at the CENG.

This work will be continued by investigations of samples deformed in different conditions, mainly in order to look for the temperature dependence of DSC dislocations mobility: a lower deformation temperature could impede the motion of 30° partials in the GB plane, a higher temperature could, on the contrary, promote the motion of b_c dislocations, a condition for a complete accommodation of plastic strain at the GB plane.

Acknowledgments.

We are indebted to J. J. Aubert (LETI-CENG), who provided the bicrystals, and to J. J. Bacmann (DMG-CENG), H. Kirchner and A. Korner (University of Vienna) for fruitful discussions. Two of us (A. J. and A. G.) thank also the staff of LURE and the group « Anneaux » of the Laboratoire de l'Accélérateur Linéaire d'Orsay.

References

- [1] ZAOUI, A. in *Dislocations et déformation plastique* (Les Editions de Physique) 1980 p. 307.
- [2] REY, C., ZAOUI, A., *Acta Metall.* **30** (1982) 523.
- [3] SMITH, D. A., *J. Physique* **43** (1982) C6-225.
- [4] PRIESTER, L. in *Joints de Grains dans les Matériaux* Carry le Rouet (Les Editions de Physique) 1984, p. 231.
- [5] KRIVANEK, O., ISODA, S., KOBAYASHI, K., *Philos. Mag.* **36** (1977) 931.
- [6] PAPON, A. M., PETIT, M., BACMANN, J. J., *Philos. Mag. A* **49** (1984) 573.
- [7] D'ANTERROCHES, C., BOURRET, A., *Philos. Mag. A* **49** (1984) 783.
- [8] MÖLLER, H. J., *Philos. Mag. A* **43** (1981) 1045.
- [9] KING, A. H., SMITH, D. A., *Acta Crystal. A* **36** (1980) 335.
- [10] GEORGE, A., MICHOT, G., *J. Appl. Cryst.* **15** (1982) 412.
- [11] MARTINEZ-HERNANDEZ, M., Thèse de 3^e Cycle, INPL, Nancy (1986);
- MARTINEZ-HERNANDEZ, M., KORNER, A., KIRCHNER, H. O. K., GEORGE, A., to be published.
- [12] BACMANN, J. J., GAY, M. O., DE TOURNEMINE, R., *Scripta Metall.* **16** (1982) 353.
- [13] BAILLIN, X., PELISSIER, J., BACMANN, J. J., JACQUES, A., GEORGE, A., *Philos. Mag. A* **55** (1987) 143.
- [14] JACQUES, A., Thèse de Docteur Ingénieur, INPL, Nancy (1984).
- [15] JACQUES, A., GEORGE, A., BAILLIN, X., BACMANN, J. J., *Philos. Mag. A* **55** (1987) 165.
- [16] EL KAJBAJI, M., Thèse de Doctorat de l'Université de Grenoble (1986).
- [17] KING, A. H., FU-RONG CHEN, *Mater. Sci. Eng.* **66** (1984) 227.
- [18] GEORGE, A., JACQUES, A., 5th Intern. Symp. on *Structure and Properties of Dislocations in Semiconductors*, Moscow (1986), to be published.