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NEW RESULTS ON DISLOCATION TRANSMISSION BY GRAIN BOUNDARIES IN ELEMENTAL SEMICONDUCTORS

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Résumé : Des expériences de déformation in situ dans le microscope électronique sur des bicristaux de Si et Ge montrent que des dislocations peuvent être transmises par des joints de grand angle même quand leur vecteur de Burgers n'est pas un vecteur translation du second cristal, avec l'aide des concentrations de contrainte en tête d'empilements. Les dislocations sont transmises sous forme de partielles de Shockley, avant de se recombier éventuellement pour former des dislocations complètes. L'élimination des dislocations interfaciales résiduelles conditionne la transmission successive de plusieurs dislocations à partir d'un seul empilement. Ceci peut se faire par glissement sur des systèmes secondaires.

Abstract : HVEM in situ straining experiments on Si and Ge bicrystals show that dislocations can be transmitted across high-angle grain boundaries even when their Burgers vector is not a lattice vector of the second grain, provided that sufficient stress concentrations arise at pile-up tips. Transmitted dislocations appear as Shockley partials, before they eventually recombine to form perfect dislocations. Repeated transmission of several similar dislocations from one pile-up is possible when the interface can be cleared of residual dislocations, which can sometimes be obtained by activating new slip systems.

1 - INTRODUCTION

Much of the experimental evidence for true dislocation transmission across high-angle grain boundaries (GB) has been gained only recently, thanks to in situ observations in well-defined bicrystals /1-8/. As pointed out for example by Hirth /9/ or Smith /10/, the crossing of high-angle GBs by lattice dislocations is likely to be a difficult process, which can take place only when very favourable circumstances are realized, so that in most usual situations, GB crossing is probably by-passed by other dislocation mechanisms. The inherent difficulty of dislocation transmission stems from the ordered structure of GBs and the topological rule which states that the total Burgers vector has to be constant in any reaction. A consequence is that, except for the trivial case when the Burgers vector of incoming dislocations is common to both crystal lattices, a one-to-one dislocation mechanism :

$$b^I \rightarrow b^{II} + b^{GB} \quad (1)$$

requires some extra energy supply proportional to $|b^{GB}|^2$, without forgetting the supplementary energy required to form the step that must be associated with the intergranular residual dislocation, in order to preserve the atomic structure of the GB /11/.

Recent observations in $(1\bar{1}2)\Sigma = 9$ ($[011]$ tilt axis, angle 38.94°) bicrystals of silicon and germanium allowed us to determine the conditions under which dislocation transmission can be observed :

- (i) It is important that the deformation is locally confined in one grain, i.e. no dislocations must be present in the second grain facing the deformed area. In bicrystals which were initially dislocation-free, this could be obtained by careful pre-straining /12/.
- (ii) Slip planes of incident and transmitted dislocations have to match along a common line at the GB plane. Matching planes are always available in symmetric bicrystals.

(iii) The local stress must be very high. In our experiments, an applied shear stress of 50-100 MPa ($\approx 10^{-3} \mu$, μ : shear modulus) was realized in all cases where transmission was observed. The stress is locally reinforced by interactions with other dislocations: transmission always takes place at the tip of pile-ups of ≈ 10 dislocations or more. It is important that the pile-up stress has a large shear component in the slip plane of transmitted dislocations, along the direction of their Burgers vector.

(iv) The Burgers vectors of incident and transmitted dislocations must not be too different in order to minimize the formation energy of the residual dislocation.

(v) Successive transmission of several dislocations from one and the same pile-up critically depends on the evacuation of residual GBDs, whose accumulation would require higher and higher energies. Clearing of the GB can be obtained by the dissociation of residual GBDs into DSC dislocations, (especially if the products are glissile in the GB plane, but evidence for climb was also found /13/) and/or by the activation of new slip systems, usually in the plane of incoming dislocations.

Our purpose in this paper is twofold. First, a brief summary of the results obtained in $\Sigma=9$ bicrystals, that have been already described elsewhere /12/, stresses the point that dislocation transmission in fcc materials proceeds by Shockley partials /14/. Second, two new examples are described. The first one, observed in (155) $\Sigma = 51$ bicrystal of Ge appears to be a rather simple example of a direct one-dislocation mechanism. The second case, observed in a (710) $\Sigma = 25$ Si bicrystal, is much more complex: dislocations start moving in the second grain only after dislocation activity has been observed on several slip systems in the grain of incoming pile-up dislocations. This example could mean that dislocation transmission may be an indirect result of the relaxation of pile-ups formed at the interface.

2 - EXPERIMENTAL METHODS

Symmetric tilt bicrystals were obtained by Czochralski pulling and have been described in /6/. In situ straining experiments were performed in the 1 MeV electron microscope of the CENG operated at 400 kV and using the high temperature deformation stage of Pelissier et al. /15/. Thanks to the two tilt axes available, convenient diffracting conditions could be obtained for each crystal simultaneously. Different stress axes were chosen depending on the transmission reaction which was looked for in any case. Pre-straining was critical. The amount of pre-strain was fixed to introduce few dislocations at the GB plane, confined in one and the same grain. Thinning of the area close to the GB was done by ion milling. More details are given in /6,12/. In situ experiments were carried out at rather low temperatures ($0.5-0.6 T_m$, T_m : melting temperature) in order to be able to measure the velocity of dislocations in real time, which provided a direct estimate of the actual shear stress, using the data of Schaumburg /16/ for Ge and George and Champier /17/ for Si. When possible, in situ experiments were interrupted prior to failure and samples, cooled down with the load applied, could later be observed by conventional TEM to determine the Burgers vectors and details of the slip geometry.

3 - DISLOCATION TRANSMISSION BY THE $\Sigma = 9$ GB

By convention, all reactions are described from crystal I to crystal II, which introduces no physical limitation since bicrystals are symmetric. Superscripts are used to designate the crystal to which a vector or plane belongs, while subscripts designate the cubic basis in which vector components are expressed. For short, dislocations with $b \approx a/2 \langle 110 \rangle$ are called "perfect" although they are actually dissociated in their slip-plane as in fcc crystals, as opposed to isolated Shockley partials with $b \approx a/6 \langle 112 \rangle$, which are observed in some cases.

3.1 - Stress axis $\approx [243]_I / [234]_{II}$

The primary slip system is $(\bar{1}11)_I [110]_I$ with $s:0.42$. At the GB plane dislocations, positively oriented along $[431]_{II}$, glide towards the GB with $\bar{b} = 1/2 [110]_{II} = DC_I$ (in Thompson's notations). Figure 1 shows the simplest configuration that was obtained in Ge after in situ straining at ≈ 803 K, $\tau \approx 85$ MPa. Two Shockley partials (as revealed by weak-beam TEM) have been transmitted in a $(\bar{1}11)_{II}$ plane from the tip of a short pile-up of DC_I dislocations. These two partials are identical with $\bar{b} = 1/6 [211]_{II} = D\beta_{II}$, and each trails an intrinsic stacking fault still linked to the GB.

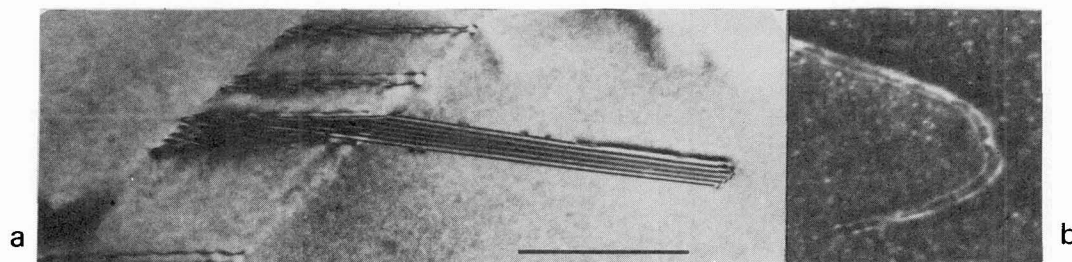


Fig.1 - Transmission of a pair of Shockley partials across a $\Sigma = 9$ GB in Ge. (a) general view. $\bar{g}:022$. Marker : $1 \mu\text{m}$ (b) weak beam micrograph of the two partials, $g, 3g, \bar{g}:202$

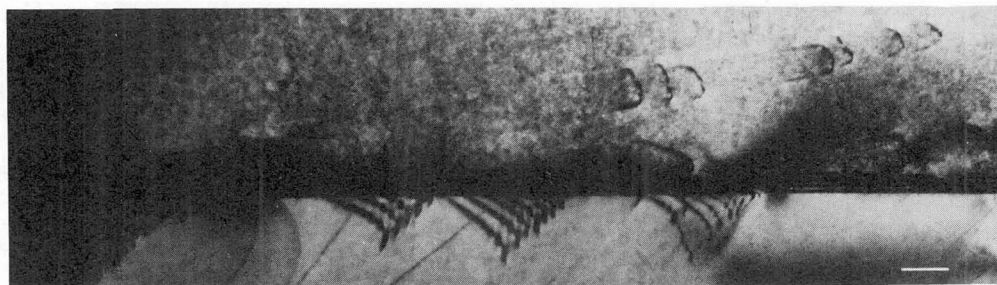


Fig.2 - Transmission of perfect and partial dislocations across a $\Sigma=9$ GB in Si. $\bar{g} : 220_1/022_{11}$. Marker : $1 \mu\text{m}$

Another sequence, observed in the HVEM at 690 K, 150 MPa, showed that up to nine separate partials can be emitted in ≈ 2 mins while, in the same time, about twenty dislocations have joined the pile-up. It appears that the majority of transmitted partials are $D\beta^{II}$ dislocations, so that they cannot recombine to form perfect dislocations. The simplest cyclic reaction consists in converting two $1/2[110]_I$ dislocations into three identical partials and one GB dislocation with $b^g = 1/18[4\bar{1}\bar{1}]_I$, a unit DSC dislocation glissile in the $\Sigma = 9$ GB. Such a cycle leaves the GB free of any residual GBD at the pile-up tip, since the $\Sigma = 9$ coincidence relationship exactly gives :

$$2 DC^I = b^g + 3 D\beta^{II}$$

It has been checked that b^g dislocations were actually created during in situ straining and that the $D\beta^{II}$ partial was the most stressed at the pile-up tip. It must be noticed that this scheme does not lead to the transmission of perfect dislocations, but rather to micro-twinning, with the reservation that the stacking of slip planes of transmitted dislocations has not been determined.

3.2 - Stress axis $[\bar{1}\bar{2}2]_I, [\bar{1}\bar{2}2]_{II}$

The primary slip system is $(11\bar{1})_I, [101]_I$ with $s:0.41$. When piling up against the GB, dislocations align parallel to the tilt axis and this case allowed us to study the asymmetric transmission :

$$DA^I \text{ in the } \gamma^I \text{ plane} \rightarrow DA^{II} \text{ in the } \gamma^{II} \text{ plane}$$

Figure 2 is a general view of a configuration formed in Si at 840 K, ≈ 100 MPa. A few DA^{II} dislocations have been emitted in $(11\bar{1})_{II}$ planes of grain II from the tips of two pile-ups of DA^I dislocations. However, near the pile-ups, extended stacking faults trailed by γA^{II} Shockley partials could also be identified. An additional interesting feature was the observation of dislocations with $b = 1/1[0\bar{1}\bar{1}]_I = CA^I$, gliding in $(11\bar{1})_I$ planes of crystal I. These loops expanded from the pile-up tip immediately after the dislocation transmission events. One possibility is to convert one pile-up dislocation plus the next leading partial γA^I into one perfect dislocation, $DA^{II} = \gamma A^{II} + D\gamma^{II}$, one b^g dislocation, which has glided away in the GB, and one perfect CA^I dislocation which can be re-emitted in crystal I (in the $(100)_I$ cube plane and then in $(11\bar{1})_I$:

$$DA^I + \gamma A^I = DA^{II} + b^S + CA^I$$

This, however, does not achieve a cycle and the next possible steps /12/ appear to be energetically more difficult. Anyway in these experiments, the transmission always stopped after a few dislocations.

4 - DISLOCATION TRANSMISSION BY THE $\Sigma = 51$ GB

The $\Sigma = 51$ GB has the same [011] tilt axis as $\Sigma = 9$, but a lower angle, 16.10° . The GB plane is $(\bar{1}\bar{5}5)_I/(\bar{1}\bar{5}5)_{II}$ and the stress axis was chosen to be $[532]_I/[523]_{II}$. In situ straining was done at ≈ 743 K, ≈ 85 MPa. As shown in Fig.3, activated dislocations are mainly $b = 1/2[\bar{1}\bar{1}0]_I = CD^I$ gliding in $(\bar{1}\bar{1}1)_I$, (s:0.34). The Burgers vector can be determined from in situ observations, thanks to cross-slip events from the pile-up plane towards $(\bar{1}\bar{1}1)_I$ planes. Transmission of perfect dislocations takes place in $(\bar{1}\bar{1}1)_{II}$. The first dislocation is emitted in crystal II from the tip of a six-dislocation pile-up and in the next 90 sec, three other dislocations are transmitted, while only four new dislocations have joined the pile-up, out of which four dislocations have cross-slipped. Transmitted dislocations move fast out of the field of view but are clearly revealed by the traces they leave at the surfaces of the thin foil. Those slip traces vanish after a few seconds.

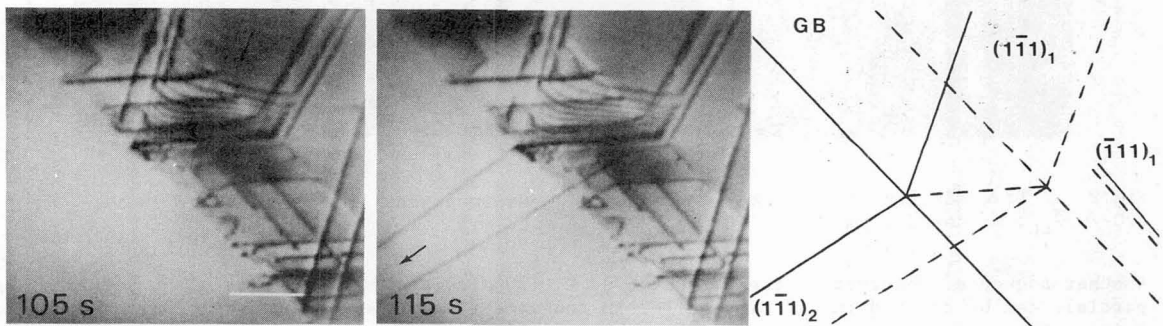


Fig.3 - Transmission of perfect dislocations across a $\Sigma = 51$ GB in Ge. HVEM experiment. Times in seconds from the beginning of recording are given for each frame. See text for details. $\bar{g} : 022$. Marker : $1 \mu\text{m}$

Although the Burgers vector of transmitted dislocations could not be determined, the observed reaction is most probably :

$$CD^I \Rightarrow 1/34[\bar{4}\bar{3}\bar{3}]_I + CD^{II}$$

This reaction has to be decomposed in four steps, following the scheme successfully used in the $\Sigma = 9$ cases /7,12/ :

- (i) Incorporation of the leading partial, αD^I , in the GB plane, which needs a reaction with the existing residual GBD, except for the first dislocation of the pile-up.
- (ii) Transmission of the leading partial, αD^{II} , in grain II, which modifies the residue.
- (iii) Incorporation of the trailing partial $\alpha \alpha^I$, which again modifies the residue.
- (iv) Transmission of the trailing partial, $\alpha \alpha^{II}$, in grain II and so on...

At any step, the energy barrier can be roughly estimated by

$$\Sigma b^2 = \Sigma b^2 \text{ products} - \Sigma b^2 \text{ reactants},$$

expressed in units of b_p^2 , the square of the Shockley partial Burgers vector. Up to the 5th transmitted dislocation, the proposed reactions are the easiest if only transmission towards $(\bar{1}\bar{1}1)_{II}$ is permitted, as suggested by observations. When the number of transmitted dislocations increases, the energy barriers increase because residual dislocations accumulate at the GB plane. However in this particular case, the energy barriers are very low ($\leq 0.12 b_p^2$) for the first transmitted perfect dislocation and increase only slowly so that the most difficult step needs only $0.5 b_p^2$ to complete the transmission of the 4th CD dislocation. Such a barrier is commonly involved, and overcome, in the $\Sigma = 9$ case.

The emission of glissile GB dislocations with $b = n \times 1/102[\bar{1}0, \bar{1}, 1]_I$ ($n = 1, 2, \dots, 5$) could sometimes lower the energy of residual GBDs, but would make the transmissions much more difficult. Note that Skrotzki /8/ did not observe those GBDs in deformed $\Sigma = 51$ bicrystals of Ge.

The Σ^2 criterion neglects the stacking fault energies and this simple scheme does not explain why transmitted dislocations look perfect and why isolated partials are not observed, contrarily to the $\Sigma = 9$ case. A slightly more elaborate treatment was done, taking into account the string action of the stacking faults on partial dislocations, and also the fact that dislocation interactions strongly depend on the character of dislocation lines. This calculation shows that, at least for the first dislocation, the force which is required to transmit the leading partial in grain II (≈ 400 MPa) is much larger than both the force needed to incorporate the trailing Ca^I partial in the GB (≤ 100 MPa) and the force needed to transmit the Ca^{II} partial in grain II (≈ 200 MPa). It is then believed that these two steps immediately follow the first one, therefore preventing an extended stacking fault to form. The same calculation in the $\Sigma = 9$ case predicts, on the contrary, that the first step is not the most difficult.

5 - DISLOCATION TRANSMISSION BY THE $\Sigma = 25$ GB

The $\Sigma = 25$ GB has about the same angle as $\Sigma = 51$, 16.26° , but a different tilt axis $[001]$. The GB plane is $(710)_I / (7\bar{1}0)_{II}$ and the stress axis was chosen to be $[\bar{4}30]_I / [\bar{3}40]_{II}$. Our purpose with this geometry was to check the possibility of transmission of $(\bar{1}11)_I$, $[101]_{II}$ dislocations from $(\bar{1}11)_I$, $[101]_I$ pile-ups. The difference between the two Burgers vectors is very small, being a unit vector of the DSC lattice which is, in addition, glissile in the GB plane, but the most stressed slip planes (Schmid factors : 0.34 and 0.46, respectively) do not match at the GB plane, their lines of intersection with the GB are at an angle of $\approx 8.21^\circ$.

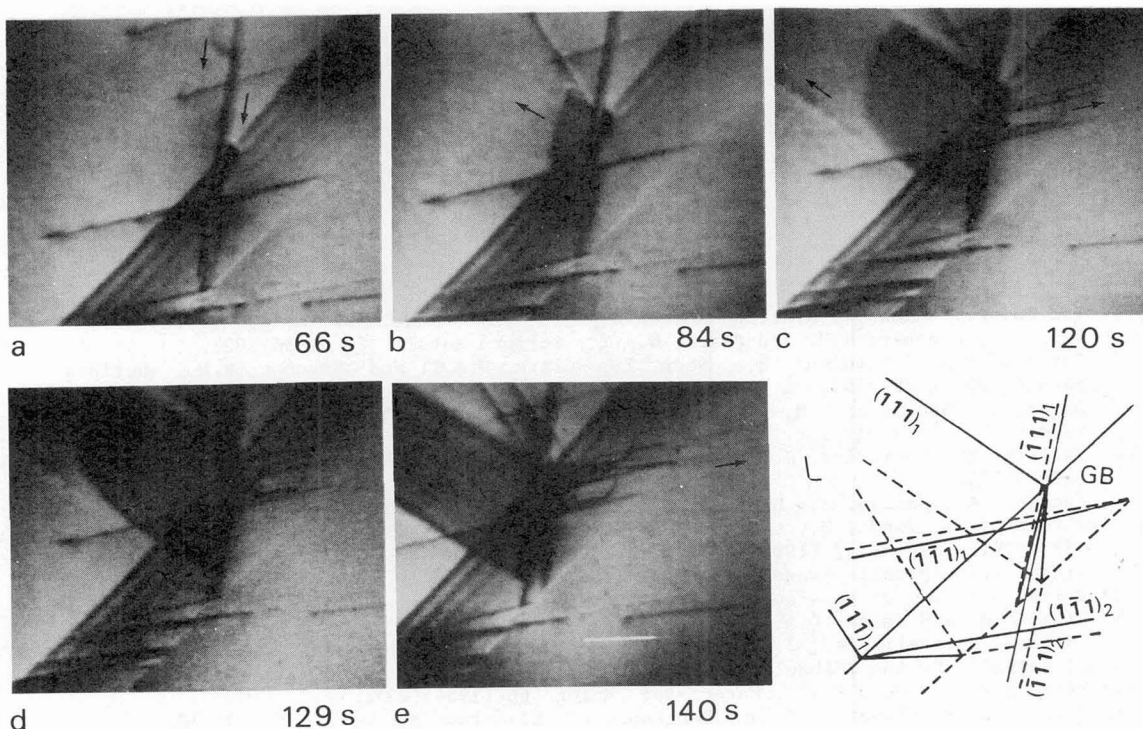


Fig.4 - Activation of secondary slip systems and transmission of dislocations at a $\Sigma = 25$ GB in Si. HVEM experiment. Times in seconds from the beginning of recording are given for each frame. See text for details. $g : 004$. Marker : $1 \mu m$

What was observed at 940 K, ≈ 116 MPa, was totally different! $1/2[101]_I$ dislocations, gliding from top of Figure 4a in a $(\bar{1}11)_I$ plane -viewed nearly end-on and vertical- formed a pile-up against the GB plane. The Burgers vector is known from the observation of cross-slipping out of the pile-up in $(11\bar{1})_I$ (arrow). At the pile up tip, dislocations are more compressed near the upper surface of the thin foil (dark contrast). From that point, which is also very near the arriving point of the cross-slipping dislocation, a partial dislocation with $b = 1/6[\bar{1}2\bar{1}]_I$ developed in the $(111)_I$ plane, trailing an intrinsic stacking fault (Figure 4b). Some seconds later, this $(111)_I$ plane was swept by several perfect dislocations, with $\bar{b} = 1/2[1\bar{1}0]_I$, moving from the pile-up tip. These dislocations have created the slip traces which are visible on figures 4c and d. Next, two dislocations developed in $(\bar{1}11)_I$ planes, -viewed end-on on the right side of the pile-up- and were probably trapped in the GB plane. They probably have $\bar{b} = 1/2[011]_I$, but do not seem to take any part in the following. In the mean time, the faulted area was increased by the motion of the Shockley partial, towards the left. Figure 4d shows a new dislocation cross-slipping on the right side of the pile-up and a modification of the dark area at the impact point of the pile-up.

Transmission itself last appears on figure 4e. While another dislocation has cross-slipped on the right side of the pile-up, a burst of several dislocations suddenly moved in grain II on -at least- two parallel $(\bar{1}11)_{II}$ planes. Transmission soon stopped while intense dislocation activity continued in crystal I, mainly on $(\bar{1}11)_I$ and $(111)_I$ slip planes. Unfortunately, transmitted dislocations have moved out of the field of view and their Burgers vector could not be determined.

This complex chain of reactions has only been observed once and is far from being understood. Space is insufficient here to give a detailed discussion. The emission of the Shockley partial in $(111)_I$ is readily explained. Then, a possibility is that a node formed by the reaction of this partial with a cross-slipping $1/2[101]_I$ dislocation can decompose, creating new dislocation segments with $b = 1/2[1\bar{1}0]_I$ in the faulted plane and with $\bar{b} = 1/2[011]_I$ in the $(11\bar{1})_I$ cross-slip plane. When the latter dislocation reaches the GB plane, it could easily be converted into one $1/2[011]_{II}$ dislocation gliding in $(\bar{1}11)_{II}$, which matches $(\bar{1}1\bar{1})_I$ at the GB plane.

If this explanation, which has still to be more firmly validated, is correct, that would mean that slip plane matching at the GB is needed for dislocation transmission, at least in the range of moderate temperatures which has been investigated so far.

The above analysis assumes that glide is the only mechanism for dislocation motion. Higher temperatures would increase the importance of climb, which might be relevant for the dispersion of residual GBs and to overcome imperfect plane matching /10,13,14/.

REFERENCES

- /1/ Van An, F., Bulenkov, N.A. and Andreeva, A.V., Phys. Stat. Sol.(a) 88, (1985) 429.
- /2/ Shen, Z., Wagoner, R.H. and Clark, W.A.T., Scripta Metall. 20 (1986) 921.
- /3/ Bamford, T.A., Hardiman, B., Shen, Z., Clark, W.A.T. and Wagoner, R.H., Scripta Metall. 20 (1988) 253.
- /4/ Baker, I., Schulson, E.M. and Horton, I.A., Acta Metall. 35 (1987) 1533.
- /5/ Paidar, V., Pal-Val, P.P. and Kadeckova, S., Acta Metall. 34 (1986) 2277.
- /6/ Baillin, X., Pelissier, J., Bacmann, J.J., Jacques, A. and George, A., Phil Mag. A 55 (1987) 143.
- /7/ Jacques, A., George, A., Baillin, X. and Bacmann, J.J., Phil. Mag. A 55 (1987) 165.
- /8/ Skrotzky, W., Wendt, H., Carter, C.B. and Kohlstedt, D.L., Acta Metall., 36 (1988) 983 ; Phil. Mag. A 57 (1988) 383.
- /9/ Hirth, J.P., Metall. Trans. A 3 (1972) 3047.
- /10/ Smith, D.A., J. de Physique 43 (1982) C6-225.
- /11/ King, A.H. and Smith, D.A., Acta Cryst. A 36 (1980) 335.
- /12/ Baillin, X., Pelissier, J., Jacques, A. and George, A., Phil. Mag. A (to be published)
- /13/ El Kajbaji, M. and Thibault-Desseaux, J., Phil. Mag. A 58 (1988) 325.
- /14/ King, A.H. and Chen F.-R., Mater. Sci. Engng. 66 (1984) 227.
- /15/ Pelissier, J., Lopez, J.J. and Debrenne, P., Electron. Microsc. 4 (1980) 30.
- /16/ Schaumburg, H., Phil. Mag., 25, (1972) 1429.
- /17/ George, A. and Champier, G., Phys. Stat. Sol.(a) 53 (1979) 529.