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ON THE ELECTRICAL ACTIVITY OF THE $(\bar{1}11)$ - $(\bar{1}\bar{1}2)$ STEPS IN THE SILICON $\Sigma = 3$ TWIN BOUNDARY

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Résumé - Des mesures de courant induit par faisceau d'électrons (EBIC) dans un microscope électronique à balayage, ont été réalisées sur des marches $(\bar{1}11)$ - $(\bar{1}\bar{1}2)$ de la macle $\Sigma = 3$ du silicium. Les deux plans d'interface ainsi que les deux types de dislocations qui les séparent ne présentent fréquemment aucune activité électrique détectable. Une telle activité apparaît cependant quand la densité de marches est élevée. La microscopie électronique en transmission (TEM) montre alors que des nano-précipités décorent une grande proportion des dislocations formant les arêtes des marches.

Abstract - Electron beam induced current (EBIC) measurements in the scanning electron microscope have been performed on a $\Sigma = 3$ twin boundary with $(\bar{1}11)$ - $(\bar{1}\bar{1}2)$ steps in silicon. The two interface planes, together with the two different dislocations at their intersections, frequently show no detectable electrical activity. Such an activity however appears when the step density is high. In this case, transmission electron microscopy (TEM) shows that a large proportion of the step dislocations is decorated by nano-precipitates.

1 - INTRODUCTION

Grain boundaries (GBs) in silicon usually exhibit an electrical activity, the origin of which may be intrinsic (due to the structural mismatch) or extrinsic (due to the impurities) /1/. Among the possible electrically active structures, the silicon dangling bond has been shown to play a noticeable role /2/. However, many other bond configurations (with angle and length distortions) are also present in the GBs and the question arises whether they also induce an electrical activity (i.e. deep levels). A large amount of work has been performed in this respect on the stepped $\Sigma = 3$ twin boundary, because this GB has the peculiarity of carrying highly distorted bonds within the frame of the most simple coincidence relationship /3-9/. The $(\bar{1}11)$ - $(\bar{1}\bar{1}2)$ step is characterized by the presence of the "incoherent" $(\bar{1}\bar{1}2)$ twin plane and by the fact that there is a rigid body translation (RBT) associated to this facet while there is none for the $(\bar{1}11)$, which induces the presence of dislocations at the step edges. The atomic structure of the step has been described by Bourret and Bacmann /5/ and can be summarized as follows: (i) dangling bonds are absent, (ii) the $(\bar{1}\bar{1}2)$ facet and the dislocations include reconstructions along the $\langle 110 \rangle$ tilt axis, and (iii) the dislocations have two different core configurations. The electronic levels associated to the $(\bar{1}\bar{1}2)$ boundary /6-8/ and to the whole step (including the dislocations) /9/, have been the objects of computer simulations which indicated that they should not fall into the band gap. The aim of this paper is to present the electrical activity observed on this boundary in a real material, by means of the electron beam induced current (EBIC) mode of the scanning electron microscope (SEM) /10/. We have mentioned, in a previous paper /11/, EBIC results obtained on a stepped $\Sigma = 3$, where indeed some of the steps showed no electrical activity. The example presented in /11/ is further developed here, and its implications are detailed. Moreover, the effects of an electrically activating anneal on this GB are also discussed.

2 - EXPERIMENTAL DETAILS

2.1. Experimental procedure

We observed stepped $\Sigma = 3$ boundaries in two directionally solidified large grained polycrystals, with either boron or aluminium doping. However, we present hereafter only one representative case, where EBIC measurements and transmission electron microscopy (TEM)

could be carried out on the same regions. This twin boundary comes from the Al-doped material ($N_{Al} = 2 \times 10^{17} \text{cm}^{-3}$) /12,13/. The activating anneal is made at 900°C for 2 h in vacuum in a sealed ampoule, it is followed by a fast cooling ($\approx 5^\circ\text{C s}^{-1}$); this procedure has been shown to have the effect of increasing the GB electrical activity by creating metallic precipitates in the GB-plane, the metals (Cu and Ni) coming mainly from contamination during sample preparation /11,14/. This treatment is then applied to the stepped $\Sigma = 3$ to check the behaviour of the steps regarding the precipitation phenomenon. In this respect, two neighbouring samples with exactly the same GB patterns - thanks to the columnar structure of the ingot - have been studied together, one in the as-grown state and the other in the annealed state. A Schottky contact has been deposited by aluminium evaporation on both of them in the same run and the EBIC experiments have been performed with exactly the same beam and imaging conditions. The ohmic contacts are obtained with deposition on the back surface of an Al-Ga alloy after scrapping; all preparations are carried out at room temperature.

2.2 Electron beam induced current

Electron-hole pairs are created by the incident electron beam of the SEM and they are separated in the electric field of the Schottky junction to form the electron beam induced current (EBIC), which is used, in turn, to modulate the image intensity of the CRT display of the SEM. Recombining defects then appear on the image with varying grey levels, on a white background, depending on their recombination strength. The EBIC contrast can be interpreted provided it is obtained in low injection conditions (excess carrier density small compared to the equilibrium carrier density) /10/. The minimal detectable density of intrinsic states can be evaluated supposing that the minority carrier capture cross section of these states is similar to the one currently assigned to the dangling bonds (10^{-16}cm^2 /16/). In the case of a grain boundary, using the computed numerical relationships between the density of states and the recombination velocity given by Dugas /15/, it can be estimated to be about 10^{11}cm^{-2} ; while in the case of a dislocation, Kittler and Seiffert have found it to be $\approx 2 \times 10^8 \text{cm}^{-1}$ /16/. The EBIC technique is then particularly efficient to detect electrical activity in the GB case, since the average atomic density in a silicon plane (10^{15}cm^{-2}) and the density of states associated to dangling bonds in /2/ (10^{12}cm^{-2}) are higher than the detection limit; however it is less interesting in the dislocation case, because the mean atomic density along a line in silicon ($\approx 3 \times 10^7 \text{cm}^{-1}$) is lower than the detection limit. In this last situation, only deep levels generated by segregated or precipitated impurities can be detected, since they exhibit higher density and capture cross sections /16/. This fact will be of prime importance in the discussion.

2.3 Transmission electron microscopy

The samples are mechanically thinned by the back surface to $\approx 30 \mu\text{m}$, and then ion milled on both sides, until the EBIC-studied zones are transparent to the electrons. The microscope used (JEOL 2000 FX) has a point to point resolution of 0.28 nm, which allows to obtain lattice images along the $\langle 110 \rangle$ GB tilt axis.

3 - RESULTS

We use the same notations as Bourret and Bacmann /5/ to refer to the two kinds of step dislocations. Following this, the dislocation situated at point P in fig.8 of ref.5, is called "P" here and is situated at the left edges of the $(\bar{1}\bar{1}2)$ boundary planes in all the figures presented in this text (arrow " \downarrow " in fig.1), and the one situated at point Q in ref.5 is called "Q" here and lies on the right hand side of the $(\bar{1}\bar{1}2)$ facets in the figures of this paper (arrow " \uparrow " in fig.1).

3.1 Electron beam induced current

Figure 1 shows different images of the stepped $\Sigma = 3$, recorded at a primary beam voltage of 20 kV. The strong contrast on the secondary electron image (Fig.1.a) is due to the chemical etching prior to the Schottky contact deposition, it can be verified that it induces no EBIC dark contrast. The two EBIC images (as-grown, fig.1.b; annealed, fig.1.c) show that the anneal systematically darkens the contrast in zones that were already active, while it very seldom gives contrast to zones previously inactive; when a dark spot is present in only one of the two images (1.b or 1.c), it indicates more likely a difference between the two sample microstructures than one between the two states of treatment.

Zone (A, B) in fig.1 shows EBIC features typical of most of the steps: no EBIC contrast on the $(\bar{1}\bar{1}1)$ interface plane, a varying one on $(1\bar{1}2)$, and none also on the "P" side of the $(1\bar{1}2)$ interface (\downarrow) and a strong one on the "Q" side of this facet. Zone C shows an original step with no EBIC contrast at all before annealing (fig.1.b); actually, the two kinds of planes and the two kinds of dislocations appear to be electrically inactive in this case.

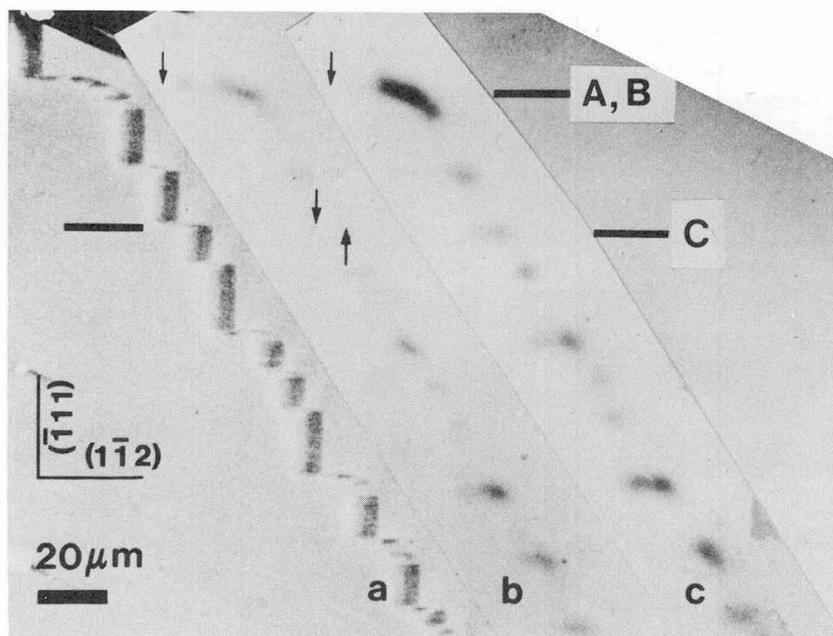


Fig. 1 - SEM images of the $\Sigma = 3$ stepped boundary, (a) secondary electrons, (b) EBIC on the same area in the as-grown sample, and (c) EBIC in the annealed sample. Arrows indicate examples of step dislocations - "P" (\downarrow) and "Q" (\uparrow) - which show no electrical activity. See TEM of zone (A,B) in figs. 2 and 3. (The plane indices refer to the left hand side crystal).

3.2 Transmission electron microscopy

Zone (A,B) (fig.1) from the as-grown sample has been observed by TEM. Figure 2 shows its well recognizable shape (compare to fig.1.a). A light EBIC spot in the $(1\bar{1}2)$ interface (fig.1.b) thus appears to be associated to point-like TEM strain contrasts (fig.2.A). Microsteps are actually existing on the left hand side of this $(1\bar{1}2)$ boundary, as witnessed by a slight deviation to the exact plane orientation (fig.2.A), which do not induce a recombination detectable by EBIC (fig.1.b). The recombining part of the boundary (fig.2.B) is a curved interface neatly farther from the $(1\bar{1}2)$ exact orientation. A lattice image of this zone (fig. 3) shows that it is made of no other structures than $(\bar{1}\bar{1}1)$ - $(1\bar{1}2)$ steps, and further, that the strain contrasts visible in fig.2 are associated with precipitates. The particle shown is likely amorphous; it seems to have nucleated in a "Q" dislocation core and to have grown at the expenses of the nearby $(1\bar{1}2)$ interface.

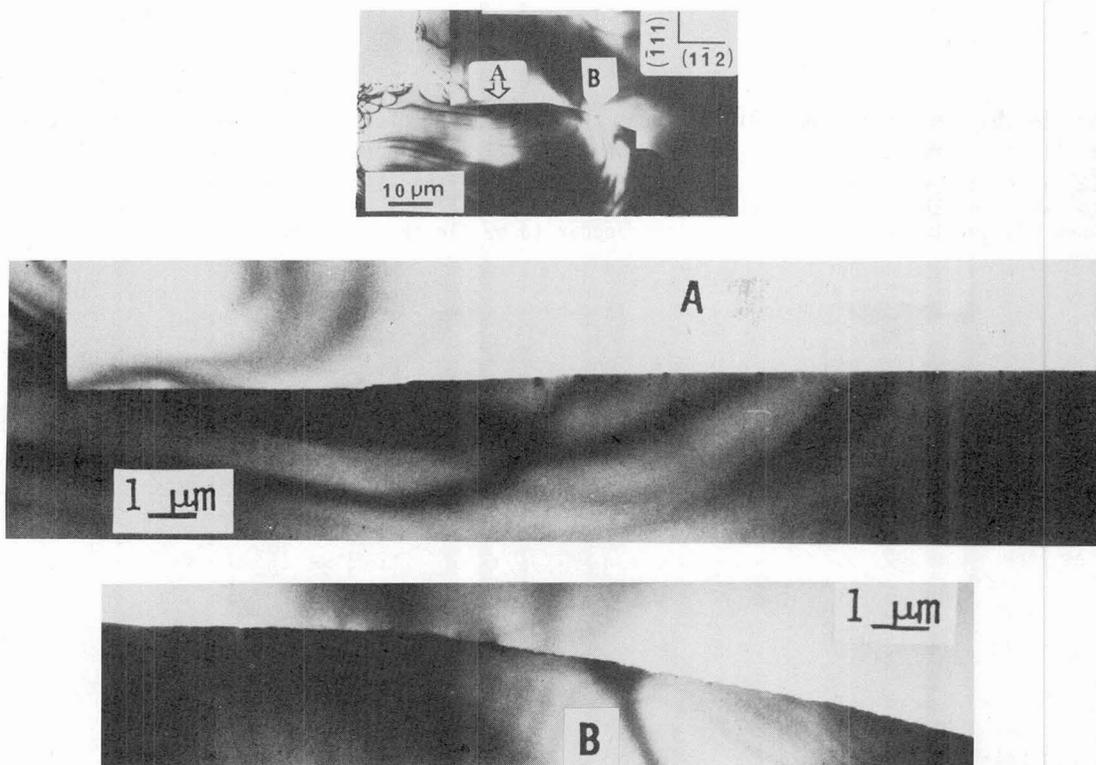


Fig. 2 - Low magnification TEM images of zones A and B of fig.1 (the plane indices refer to the lower grain).

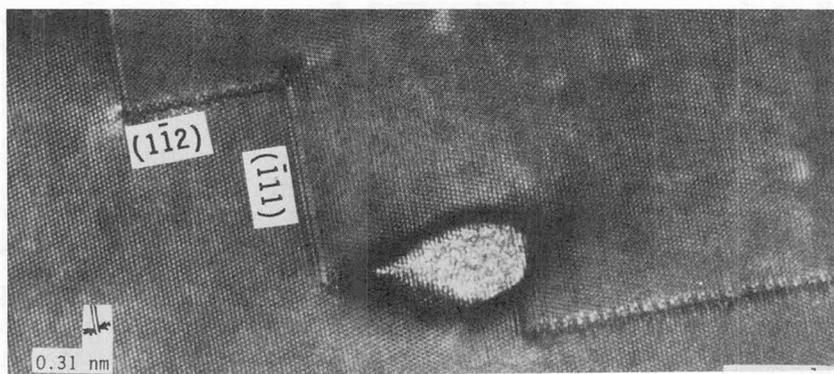


Fig. 3 - High magnification image of zone B of figs. 1 and 2.

4 - DISCUSSION AND CONCLUSION

4.1 The precipitate nucleation at the $\Sigma = 3$ steps

In the as-grown sample, the high magnification observations show that precipitates nucleate exclusively on the step dislocations, and more frequently on the "Q" type of these defects. The EBIC contrast after annealing indicates that precipitation (*metallic* /14/) occurs also in these regions, and not at the $(\bar{1}\bar{1}2)$ "incoherent" boundary planes.

4.2 The origin of the observed electrical activity at the $\Sigma = 3$ steps

The correlation between the EBIC and TEM images shows that precipitates are always present in the $\Sigma = 3$ steps when an EBIC contrast is observed. Moreover the presence of these precipitates appears to be associated to that of the dislocations at the step edges. On the other hand, all the different components of the step may be observed with no EBIC contrast. One is then tempted to conclude that the $\Sigma = 3$ step is electrically active only when decorated by precipitates, i.e. that it exhibits no intrinsic recombination activity.

4.3 On a possible passivation by the dopant (Al) and the metallic impurities

Aluminium is known to passivate the GB electrical activity after diffusion below the eutectic-formation temperature (577°C) /17/. However no such mechanism is reported when aluminium is added to the melt where, on the contrary, a strong increase of the GB electrical activity is even observed after annealing /18/; moreover, we have also observed electrically inactive $\Sigma = 3$ steps in the B-doped material. Therefore Al-passivation is not likely in the studied samples.

Cu is also known to passivate GBs /19/ and dislocations /20/, after low-temperature diffusion, and is found to be one of the contamination metals in the preparation procedure /14/. However, it is known to precipitate at the defects during the heat treatment and to induce there a strong recombination activity /14/. As a number of non-recombining areas subsist after annealing, this hypothesis seems unlikely, i.e., the non-activity observed seems to be indeed of intrinsic origin.

4.4 About the possibility of recombination below the EBIC sensibility

As an isolated dislocation would not show intrinsic recombination activity in EBIC, even if there were one dangling bond per atom in its core /16/, the step dislocations "P" and "Q" would also show none when isolated between two inactive facets. In order to check their intrinsic activity, one then needs a noticeable concentration of these defects with no precipitates, a situation which is rare in current polycrystalline silicon. A definite conclusion thus cannot be drawn on this particular point.

In the case of the "incoherent" $(\bar{1}\bar{1}2)$ interface however, the EBIC sensibility is high and the experiments show unambiguously that this interface has no recombining activity, i.e., that its deep level density, supposing a capture cross section of 10^{-16}cm^2 , must be lower than approximately 10^{11}cm^{-2} (10^{-4} Si monolayers).

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