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TEM INVESTIGATION OF GRAIN BOUNDARIES IN POLYCRYSTALLINE SILICON

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Résumé
Une étude par microscopie électronique en transmission de la structure de joints de grains dans du silicium polycristallin est présentée, pour deux types de joints voisins des désorientations de coïncidence $\Sigma = 7$ et $\Sigma = 9$. Dans le joint $\Sigma = 7$, un réseau de dislocations secondaires existe ainsi qu'une série de marches. Dans le joint $\Sigma = 9$, nous avons trouvé un système de franges $\alpha$ (causé par une translation des deux grains le long du plan du joint) et un réseau de dislocations secondaires dont la structure dépend des conditions de diffraction.

Abstract
The structure of two near-coincidence boundaries (close to a $\Sigma = 7$ and a $\Sigma = 9$ misorientation) occurring in commercial poly-silicon has been examined in TEM. Both the structure of secondary dislocation networks as well as the occurrence of $\alpha$-fringes (indicating a relative translation of the crystals along the boundary) were investigated. The near $\Sigma = 7$ boundary is found to contain a simple network in addition to a regular array of steps. The near $\Sigma = 9$ boundary exhibits $\alpha$-fringes and a dislocation network which appears as a different structure for different diffraction conditions.

1. Introduction
In recent years increasing interest in the structure of grain boundaries in semiconductor materials has developed because of the important role of boundaries in determining the electrical properties of polycrystalline material. In particular low-angle boundaries as well as some special high-angle boundaries, close to a coincidence-site-lattice (CSL) orientation, have been investigated by TEM [1 to 5]. Much of this work was carried out using specially prepared bicrystals with the aim of determining the dislocation structure in the boundaries as well as the state of relative translation of the crystals at the boundary plane. In this paper we present some preliminary results on the structure of two inclined near-coincidence boundaries occurring in commercial polycrystalline Si.

2. Experimental details and results
Thin foils of commercial polycrystalline Si (Silso, Heliotronic) were prepared by ion beam thinning of thin sections cut from a large ingot having an average grain size of 1 mm. Specimens prepared in this way were found to contain irregularities in thickness on a scale of $\sim$ 20 $\mu$m. No preferential attack at grain boundaries could be established. The specimens were examined in a Philips EM-400 electron microscope operating at 120 kV; the results for two special boundaries (close to a $\Sigma = 7$ and a $\Sigma = 9$ misorientation) are presented below. Some important geometrical characteristics of these two boundaries are summarised in table I.

The near $\Sigma = 7$ boundary
From an analysis of diffraction patterns it is found that this boundary has a deviation of $6.2^\circ$ from the exact $\Sigma = 7$ misorientation (see table I) around a common $<511>$ axis. (The $\Sigma = 7$ coincidence orientation can also be obtained from a $158.21^\circ$ rotation around a common $<511>$ axis). In addition to this tilt deviation there is approximately a $1^\circ$ rotation around a common $<132>$ axis, orientated nearly
Table I. Geometrical characteristics of the Σ = 7 and the Σ = 9 boundaries

<table>
<thead>
<tr>
<th>Boundary</th>
<th>Deviation Δθ</th>
<th>Rotation angle/axis</th>
<th>DSC Burgers vectors</th>
<th>common g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Σ = 7</td>
<td>6.2°</td>
<td>38.21°/[111]</td>
<td>$\frac{a}{14}[132], \frac{a}{14}[563]$</td>
<td>111,331</td>
</tr>
<tr>
<td>Σ = 9</td>
<td>0.5°</td>
<td>38.94°/[110]</td>
<td>$\frac{a}{18}[174], \frac{a}{9}[221], \frac{a}{6}[121]$</td>
<td>220,311</td>
</tr>
</tbody>
</table>

Fig. 1 (a) DF image of an inclined near Σ = 7 boundary using a common 111 diffraction vector. (b) The same boundary with the dislocations seen end-on. Arrows indicate steps in the boundary. (c) BF image with a common (111) diffraction vector emphasizing the α-type fringes. Scale marker 50 nm.

perpendicular to the boundary plane. The 6.6 nm spacing of the set of dislocation lines shown in fig. 1, assuming a Burgers vector of the type $a/14<132>$, has to be associated with the latter rotation. However, since this rotation causes a twist deviation, a second set of dislocations is expected which has not been detected. It is of interest to note the more or less regular discontinuities in spacing of the dislocations in fig. 1a. These are interpreted as a series of steps occurring in the boundary plane. Evidence for steps is presented in fig. 1b in which the boundary is imaged edge-on. Since the deviation from the Σ = 7 misorientation is rather large for this boundary, it may be difficult to distinguish between dislocation network images and moiré fringes originating from double diffraction [6]. However, we can exclude the possibility that the network image in fig. 1a is generated by double diffraction because similar micrographs were obtained with different diffracting vectors. Moreover, the strain contrast of the individual dislocations can be observed in fig. 1b, where the dislocations are imaged in the end-on position.

Because of the relatively large deviation from the Σ = 7 misorientation, the weak α-fringes (parallel to the intersection of the boundary with the foil surface in fig. 1a) cannot be definitely interpreted in terms of a rigid translation of the crystals away from the Σ = 7 coincidence position. Using the same g strongly developed α-type fringes have also been obtained near the Bragg condition, as is illustrated in fig. 1c.

The near Σ = 9 boundary

Fig. 2 shows the curved overall shape of this boundary imaged under two-beam conditions using a common 311 diffraction vector. Since the deviation from Σ = 9 is small in this case (see table I), the strong α-fringes indicate an appreciable translation component along the direction of g, the magnitude of which has not yet been established. Similar results were found for the common g = 220, and it appears that the rigid translation for both common diffraction vectors is about the same.
Fig. 2 (a) DF image of a curved near \( \Sigma = 9 \) boundary between crystals 1 and 2, using a common 311 diffraction vector (arrowed). Scale marker 2 \( \mu \text{m} \).

Fig. 2 (b) Enlargements of the boundary areas indicated p, q and r in (a). Scale marker 0.5 \( \mu \text{m} \).
Fig. 3 DF image of boundary area q in fig. 2 using the 220 common diffraction vector. Scale marker 0.5 μm.

Fig. 4 DF weak-beam images of the boundary area close to q in fig. 2. The images in (a) and (b) are taken with a 111 diffraction vector from crystal 2 (diffraction condition (g, 4g)) and from crystal 1 (diffraction condition (g, 2g)), respectively. Scale marker 0.2 μm.
along the length of the boundary (differences in fig. 2 are due to slight changes in local orientation of the bicrystal).

In fig. 2 also a simple dislocation network consisting of a single row of dislocations can be observed. The projected direction of the dislocation lines is nearly constant along the boundary plane, but the spacing of the dislocation lines varies with location. However, the set of dislocations visible in fig. 2 forms part of a more complex network which can be made visible under different diffraction conditions. This phenomenon is illustrated in figures 3 and 4. In fig. 3 the network is imaged using the common \( g = 220 \), showing that the set of dislocations in fig. 2 is part of a set of narrowly spaced dislocations in the same direction. Employing a non-common \( 111 \) diffraction vector, it can be seen in fig. 4 that this set of closely spaced dislocations belongs to a network. In fig. 4a, in which the boundary plane is nearly perpendicular to the incident beam, the network is observed to be of rectangular shape. On the other hand, fig. 4b emphasizes the different contrast of the closely and widely spaced dislocations in one direction. One possible reason for the different contrast is that the widely spaced set of dislocations is probably associated with steps in the boundary plane. It should be noted that the spacing of the network visible in fig. 4 is of the correct order of magnitude to explain the deviation from the \( \Sigma = 9 \) misorientation (table 1). However, a satisfactory explanation, including a Burgers vector determination of the various dislocations involved, cannot yet be presented. Although moiré effects due to double diffraction cannot be entirely excluded, it is believed that such effects do not play any significant role in determining the contrast shown in fig. 4.

3. Conclusion

It has been shown that \( \alpha \)-fringes, characteristics of a rigid translation at the boundary, and dislocation networks occur in two near-coincidence boundaries (close to \( \Sigma = 7 \) and \( \Sigma = 9 \)) in commercial polycrystalline Si in the as-grown condition. Depending on the diffraction conditions employed, the near \( \Sigma = 9 \) boundary exhibits a dislocation network imaged as a single row of dislocations or a more complex network.

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References