



HAL
open science

TEM AND EBIC INVESTIGATIONS OF POLYCRYSTALLINE SILICON SHEETS GROWN BY THE RAD GROWTH PROCESS

R. Sharko, A. Gervais, C. Texier-Hervo

► **To cite this version:**

R. Sharko, A. Gervais, C. Texier-Hervo. TEM AND EBIC INVESTIGATIONS OF POLYCRYSTALLINE SILICON SHEETS GROWN BY THE RAD GROWTH PROCESS. *Journal de Physique Colloques*, 1982, 43 (C1), pp.C1-129-C1-133. 10.1051/jphyscol:1982118 . jpa-00221774

HAL Id: jpa-00221774

<https://hal.science/jpa-00221774>

Submitted on 4 Feb 2008

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.

TEM AND EBIC INVESTIGATIONS OF POLYCRYSTALLINE SILICON SHEETS
GROWN BY THE RAD GROWTH PROCESS

R. Sharko, A. Gervais and C. Texier-Hervo*

*Laboratoire de Minéralogie-Cristallographie, Université P. et M. Curie,
4, place Jussieu, 75230 Paris Cedex 05, France***Laboratoires de Marcoussis, Centre de Recherches de la C.G.E., Route
de Nozay, 91460 Marcoussis, France*RESUME

Dans cet article, sont présentés des résultats d'une étude par microscopie électronique en transmission de joints de grains électriquement actifs (et inactifs) dans des échantillons de silicium polycristallin élaborés par la méthode RAD. Les joints de grains dans les échantillons extraits après plusieurs mètres de tirage ne sont pas caractérisés par des relations de mâcles parfaites. De nombreux précipités apparaissent dans les joints proches de la surface libre.

ABSTRACT

This article reports on results of observations by transmission electron microscopy (TEM) of electrically active (and inactive) grain boundaries in polycrystalline silicon layers grown by the RAD growth process. In samples taken after a pulling length of several meters, the grain boundaries (GB's) are not characterized by exact twin relationships. Precipitates are present in GB's near the free surface.

I. INTRODUCTION

Polycrystalline layers grown by direct freezing of a silicon film about 100 μm thick on a carbon ribbon by the RAD growth process in view of achieving low-cost solar cells have received continuous attention these last years [1, 2]. Because of the unidirectional solidification occurring during silicon deposition on the carbon substrate, the film contains large columnar grains which are approximately perpendicular to the (011) free surface and extend across the entire thickness of the sample. Usually, these grains are several centimeters long in the $[2\bar{1}\bar{1}]$ growth direction and a few millimeters wide.

The recombinations at such GB's perpendicular to the solar cell surface may act on the photocurrent and the electrical properties of junctions [3]. It has been shown that with large grain sizes, the first effect is not important [4]. An open question is to know if the electrical activity of grain and twin boundaries is related to intrinsic crystallographic structures or to extrinsic defects such as dislocations and precipitates. Combined electron-beam induced-current (EBIC) and TEM studies are particularly well adapted to the problem.

Previous work by C. FONTAINE and A. ROCHER [5] on very early specimens elaborated by the RAD growth process had shown that the main crystal defects electrically active were curved GB's interpreted as step configurations which coincide with mirror planes associated with the exact twin relationship. The specimens studied here were taken after a pulling length of several meters. The curved GB's electrically active under EBIC examinations, although often roughly parallel to the pulling direction, are no more associated to exact coincidence orientations. Two types of samples were studied as reported below : the first variety was taken from an as-grown ribbon, the second one from a solar cell.

II. EXPERIMENTAL

The electrical activity of extended defects was mapped in an electron microscope (SEM) using EBIC contrast. The spatial resolution of the technique largely depends on the volume into which incident electrons are scattered [6]. The resolution of EBIC images is typically of the order of a few micrometers. TEM investigations were carried out on a PHILIPS EM 300 operating at 100 keV. EBIC images were correlated with reflection optical images in order to precisely locate the GB's portion of interest ; the 100 μm thick sample was further ion-thinned either on one side or both sides.

III. RESULTS

a) As-grown samples

A grain boundary electrically active was studied at several locations. The orientation relationship between the two adjacent grains was determined from diffraction patterns by a numerical method ; the deviation from the exact twin relationship $\Sigma 3$ ($R_0 = [011]$; $\theta_0 = 70^\circ 53'$) was found to be a few degrees. For example, in the area represented in figure 1, the couple (axis/orientation angle) was : (0.0721, 0.7204, 0.7058 ; $\theta = 65^\circ 74'$). The orientation of the boundary changes when it is intersected by twin planes or planar defects ; however, the largest portions are close to mirror planes for the exact $\Sigma 3$ twin : $(1\bar{1}\bar{1})\text{I}/(\bar{1}\bar{1}\bar{1})\text{II}$ including the growth direction $[\bar{2}\bar{1}\bar{1}]\text{I}/[2\bar{1}\bar{1}]\text{II}$ (indices I and II refer to those in figure 1). Figure 1 represents the GB seen end-on with defects within 1 μm from it.

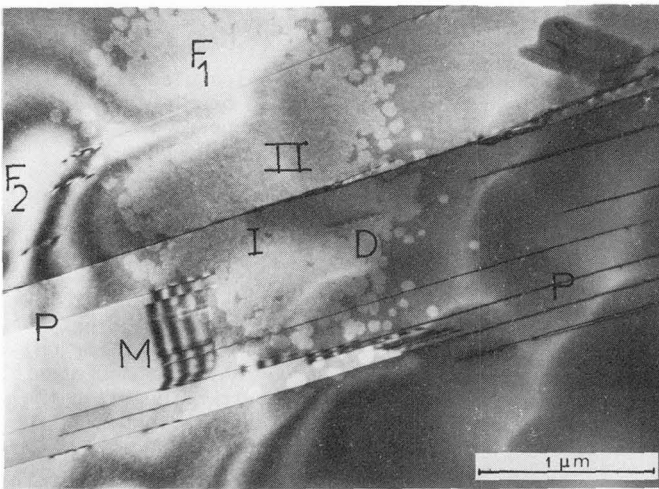


Fig.1 : grain boundary and defects in its vicinity
 $G = 02\bar{2}(I)$ and $G = 02\bar{2}(II)$
 (I) and (II) refer respectively to grains I and II.

Among these defects a few isolated and perfect 60° dislocations D (direction $u = [\bar{1}\bar{1}0]\text{I}$, Burgers vector $b = 1/2 [0\bar{1}\bar{1}]\text{I}$) have been observed. Stacking faults F_1 $(\bar{1}\bar{1}\bar{1})\text{II}$, F_2 $(111)\text{II}$ are present in the upper grain II. Their translation vector \vec{T} determined from three extinctions in two-beam conditions are perpendicular to these planes. A Schockley partial dislocation oriented along $[101]\text{II}$ with a Burgers vector $b = 1/6 [\bar{1}\bar{2}\bar{1}]\text{II}$ is present at the intercept of the F_2 stacking fault with the GB. The large width of the contrasts at the faults, especially in F_1 ($\sim 2 \mu\text{m}$) must result of large stresses to overcome the backstress of the partial dislocations [7]. Many $\Sigma 3$ twins with a $[011]$ common axis are visible in the lower grain I. These boundaries do contain many partial dislocations, some of them being stepped with facets oriented along $(1\bar{1}\bar{1})\text{I}$ (P) and $(112)\text{I}$ (M). Images taken with a common reflection should extinct the thickness fringes (fig.2a). This is observed for $(\bar{2}20)\text{I}/202(\text{II})$ but a slight residual contrast on $(112)\text{I}$ facet is present in the following common reflection (fig.2b): $(202)\text{I}/220(\text{II})$ and $(11\bar{1})(\text{I})/\bar{1}\bar{1}\bar{1}(\text{II})$. These fringes with an α character [8] may be interpreted as a rigid body translation or associated to partial dislocations in the interface [9, 10]. The partials at the intercept between facets P and M have a Burgers vector $1/6 (\bar{1}\bar{2}\bar{1})\text{I}$ determined from extinctions with reflections $111(\text{I})$ and $202(\text{I})$; these dislocations

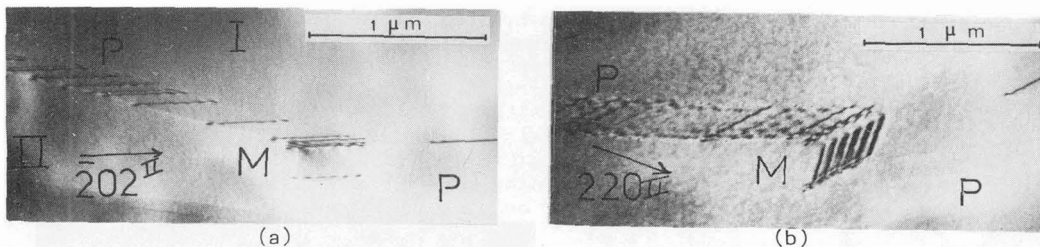


Fig.2 : M(112)I facets showing fringes with common reflection : $(202)I/(220)II$.

are of the glide type in boundary P. Partials with $b = 1/6 (2\bar{1}1)I$ and the glide type $1/6 (1\bar{2}1)I$ are present in the finely stepped boundary P. Figure 3 presents a commonly encountered distribution of microtwins consisting of large $(11\bar{1})I$ interfaces and limited at their tip by $(112)I$ facets. The dotted EBIC signal usually observed in the interior of a grain could be associated with that crystallographic defect structure related to the $(112)I$ facet.

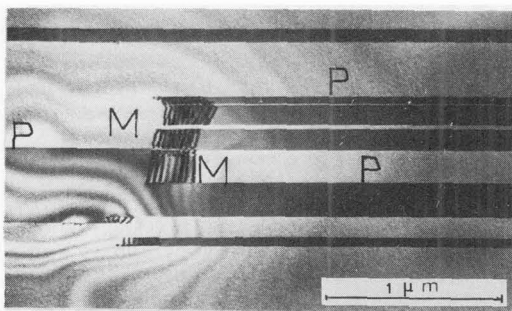


Fig.3 : distribution of microtwins.

b) Solar cell samples

100 μm thick samples were ion-thinned on both sides selecting either electrically active or inactive GB's. Blank EBIC contrasts were connected through SEM observations to flat boundaries without defects (dislocations, microtwins) in the interface or within a few micrometers from it. This result has already been reported by many authors concerning $\Sigma 3$ twins ; however, in the case of concern the GB is a general one as defined by the following (axis/rotation angle) pair : $(0.21, 0.34, 0.57 ; \theta = 38^\circ 4)$.

In opposition to that result, strong EBIC contrast arises whenever a general GB is intercepted by many planar defects. Figure 4 illustrates such a situation where a general GB noted J3 ($[11\bar{1}]$ common axis, $\Sigma 73$) cuts first and second order twins.

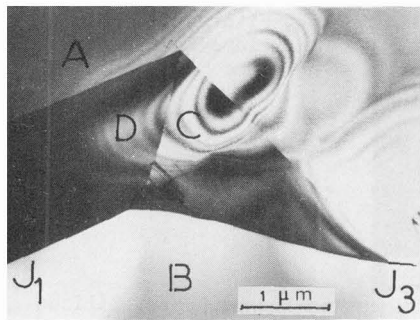


Fig.4 : 011 common axis ; the relationships between regions denoted A,B,C,D are the following : A/C, $\Sigma 3$, $(11\bar{1})A/(11\bar{1})C$; A/D, $\Sigma 3$, $(11\bar{1})A/(11\bar{1})D$; C/D, $\Sigma 9$, $(12\bar{2})C/(12\bar{2})D$; J3 = A/B, $(223)A/(455)B$.

In the region named C, many microtwins with (112) interfaces previously described are present. No dislocations are visible in the GB's where orientation changes strongly within 1 μm in length.

In order to look at GB's close to the p-n junction, samples were ion-thinned mostly on one side. Furthermore, by thinning step by step, it was possible to explore the inhomogeneous contrast presented by EBIC images along an interface (figure 5).

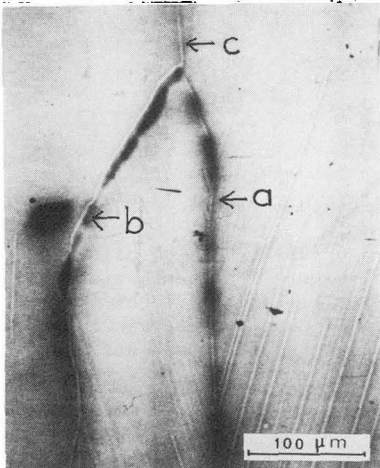


Fig.5 : inhomogeneous EBIC contrast along boundaries.

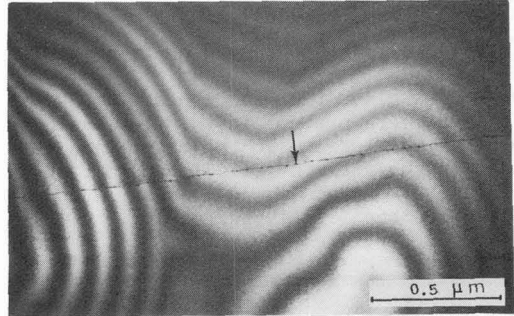


Fig.6 : precipitates in a boundary (arrow).

A GB with a "flat" interface but general orientation, ($[117]$ common axis, $(34\bar{2})$ plane boundary, close to $\Sigma 19$) without planar defects or dislocations around it exhibited a small recombination effect probably due to small precipitates (about 20 nm in size). These precipitates seem to be aligned along a common direction in the boundary where no dislocation contrast was observed. A similar situation arises in location (a) in figure 5. A "flat" $\Sigma 27$ twin boundary shows (figure 6) small dotted contrasts (~ 10 nm) when seen end-on, which may result from precipitates aligned in the boundary. The associated EBIC contrast is small in opposition to what happens in location (b) in figure 5. In this case, the interface changes its orientation due to a complex micro-twin configuration which intercepts the GB. Furthermore, a pronounced silicon carbide precipitation is present in the interface (figure 7).

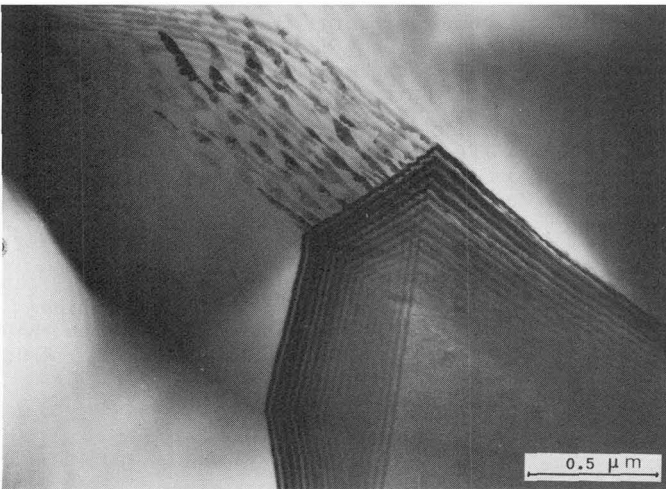


Fig.7 : silicon carbide precipitates in a boundary close to the free surface.

Notice the finely stepped nature of the thickness fringes in the boundary. Silicon carbide is thought to be present in that sample because of the proximity of the free surface (unpublished results). Silicon carbide interfaces were not observed in specimens ion-thinned equally on both sides.

IV. CONCLUSION

Coherent twin boundaries as it has been previously shown by several authors do not present electrical activity. The same result presently occurs for "uniform" general grain boundaries.

When mixed GB's are encountered, due to their intercept by planar defects, the electrical activity distinctly arises. These defects result from stress inhomogeneity probably during the growth process since large stacking fault widths are observed in as-grown samples. Most of the defects which may be ascribed an electrical activity are partial dislocations and incoherent boundaries. Due to the EBIC resolution their respective influence is generally difficult to evidence. However, it seems that a concentration of (112) incoherent $\Sigma 3$ interfaces might result in dotted EBIC contrasts within grains.

Preliminary results have indicated the presence of silicon carbide films in the grain boundaries and impurity precipitates inside the grains near to the surface (i.e. in the vicinity of p-n junction).

Further experiments in progress will be aimed at evaluating the contribution of defects and impurities to the electrical activity of the boundaries.

REFERENCES

- [1] C. BELOUET, J. HERVO, R. MARTRES, Ngo Tich PHUOC, M. PERTUS, 13th IEEE Photovoltaic Specialist Conf. (1978) 131.
- [2] C. TEXIER-HERVO, M. MAUTREF, S. MAKRAM-EBEID, C. BELOUET, Proc. 3th Photovoltaic Solar Energy Conf., Cannes (1980) 563.
- [3] Y.S. TSUO, J.B. MILSTEIN, T. SUREK, Grain Boundaries in Semi-Conductors/Pike/Seager/Leamy, editors (1982) 155.
- [4] C. BELOUET, E. FABRE, S. MAKRAM-EBEID, Ngo Tich PHUOC, C. TEXIER, Proc. 2nd Photovoltaic Solar Energy Conf., Berlin (1979) 114.
- [5] C. FONTAINE, A. ROCHER, Journal of Microscopy 118 (1980) 105.
- [6] H.J. LEAMY, L.C. KIMERLING, S.D. FERRIS, Scanning Electron Microscopy, 1 (1978) 717.
- [7] H. ALEXANDER; K. WESSEL, Journal de Physique C, 2 (1978) 114.
- [8] R.C. POND, J. Microsc. 116 (1979) 105.
- [9] A.M. PAPON, M. PETIT, G. SILVESTRE, J.J. BACMAN, in Ref. 3, p. 27.
- [10] C. FONTAINE, D.A. SMITH, in Ref. 3, p. 39.