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Czochralski growth of silicon bicrystals

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Résumé. — L'utilisation du silicium polycristallin pour les cellules solaires pose le problème de l'effet des joints de grains sur les propriétés électroniques de ce type de matériau : diffusion des impuretés aux joints de grains, propriétés électriques du joint en relation avec l'existence de liaisons pendantes et d'impuretés. Des bicristaux de silicium où l'interface entre deux grains voisins peut être parfaitement définie ont été préparés pour étudier les propriétés des joints de grains. Les échantillons ont été obtenus par la méthode de Czochralski avec du silicium du type p et du type n.

Abstract. — Utilization of polycrystalline silicon for solar cell applications poses the problem of the effects of grain boundaries on the electronic properties of this type of material : preferential impurity diffusion at the boundary, electrical properties of the boundary in relation to the existence of dangling bonds and impurities. Silicon bicrystals, where the interface between two adjacent grains can be well defined, have been grown to study the properties of the grain boundaries. Such specimens have been realized by the Czochralski growth technique with p-type and n-type silicon.

1. Introduction.

In polycrystalline silicon solar cells, many problems connected with grain boundaries seem, from the technological point of view, to have been almost solved. For instance experimental results concerning dopant diffusion showed that the grain boundaries do not play a significant role in most solar silicon layers with large grain size. In the same way, it is quite well known that the heat treatment with atomic hydrogen minimizes the influence of structural defects upon the electronic properties of silicon polycrystalline layers. However, the structural and electrical mechanisms associated with such phenomena are not really understood.

Some questions about the properties of polycrystalline silicon need to be answered : what is the role of grain boundaries and dislocations ? What is the influence of the impurities ? What is the electrical behaviour of extended defects and impurities ? What are the real phenomena involved in the electrical annihilation of the structure defects ?

The aim of this work was to grow silicon bicrystals [1] as specimens to shed light on the above questions.

This type of specimen satisfies two important conditions :

- the specimens are well defined : a grain boundary can be produced with a good definition of its crystallographic structure and a good control of the impurity quality of the silicon ;

- the material is reproducible : numerous bicrystals of the same type are needed for understanding the fundamental phenomena since they allow the investigation of various treatments on the same well defined grain boundary.

The bicrystals grown in this work are of two types :

- small angle grain boundaries,
- large angle grain boundaries.

2. Small angle grain boundaries.

- Pure flexion boundary.

This kind of misorientation has been chosen to study 60° dislocations.

In these specimens, the [110] axis of each grain are parallel and are used as the growth direction. The

disorientation is realized by setting a three degrees angle between the $[\bar{1}\bar{1}1]$ axis of each grain (Fig. 1). The grain boundary then corresponds to high Miller indexes plan close to $(\bar{1}12)$.

Fig. 1. — Pure flexion grain boundary.

— Pure torsion boundary.

These bicrystals have been grown to study screw dislocations.

In this case, the $[\bar{1}\bar{1}1]$ direction is common to both grains, and an angle of two degrees is set between the $[110]$ direction (Fig. 2). The $[110]$ axis is used as growth direction.

Fig. 2. — Pure torsion grain boundary.

3. Large angle grain boundaries.

Three different misorientations have been grown, corresponding to the so called $\Sigma = 9$, $\Sigma = 13$ and $\Sigma = 25$ twins [2] where Σ is the ratio between the volume of the unit cell of the coincidence lattice and the volume of the unit cell of each grain's lattice.

In our case, if h , k and l are the Miller indexes of the twinning plane, then :

$$\Sigma = h^2 + k^2 + l^2 \quad \text{if} \quad h^2 + k^2 + l^2 = 2n + 1$$

$$\Sigma = \frac{h^2 + k^2 + l^2}{2} \quad \text{if} \quad h^2 + k^2 + l^2 = 2n.$$

The twin $\Sigma = 9$ comes from a rotation of the grains around the common $[011]$ axis. This rotation is $38^\circ 56' 17''$, the boundary plane being (122) (Fig. 3).

For the twins $\Sigma = 13$ and $\Sigma = 25$, the disorienta-

PLANE (122)

Fig. 3. — Twin $\Sigma = 9$.

tion results from a rotation around the common $[001]$ axis.

For $\Sigma = 13$, the rotation is $26^\circ 37' 12''$, the boundary is (510) (Fig. 4) and for $\Sigma = 25$, the rotation is $16^\circ 15' 36''$ and the boundary is (710) (Fig. 5).

PLANE (510)

Fig. 4. — Twin $\Sigma = 13$.

PLANE (710)

Fig. 5. — Twin $\Sigma = 25$.

4. Crystal growth.

The bicrystals have been grown by the Czochralski method [3] using p-type silicon (boron doped) and n-type silicon (phosphorus doped).

2.5 kg of electronic grade silicon are molten at 1 420 °C in a 150 mm diameter silica crucible heated in a graphite resistor furnace. The machine is preheated at 1 000 °C under vacuum, and pulling is accomplished in a 75 l/min flow of highly pure argon.

The silicon bicrystals are grown using two monocrystalline seeds mounted on a special seed holder. This method requires very high precision in the seed orientation, seed cutting and in the design of the seed holder.

For the small angle grain boundaries, the seed are made of two half cylinders cut according to the disorientation. The boundary between the two parts is optically polished and the seed is mounted in the standard seed holder of the growing machine (SILTEC 860C) (Fig. 6).

Fig. 7. — Small angle grain boundary.

Fig. 8. — Micrograph of a torsion grain boundary. Specimen etched for dislocations (Sirtl etch).

timetre. Such a density generally leads to a polycrystalline growth in the Czochralski process, thus the bicrystals cannot be grown over one centimeter length.

For the large angle grain boundaries, a special seed holder has been realized. This seed holder made of graphite can hold two separate seeds cut according to the chosen misorientation (Fig. 9). The growth is then initiated with both seeds by making two separate neckings in order to obtain two dislocations free single crystals. The bicrystal is realized by shouldering both single crystals which will touch each other along the grain boundary. In this case, the grain boundary has no dislocation and the standard Czochralski process can be used to obtain dislocation free crystals. The growth is then performed under automatic diameter control. Large cylindrical specimens of 3 to 5 cm in diameter and several tens of centimetres long have been obtained (up to 90 cm) (Fig. 10). The precision in the orientation of each grain is around 10^{-2} degrees. The grains

Fig. 6. — Seeds for small angle grain boundary.

The growth is then initiated according to the well known Czochralski technic. The specimens are about a centimetre in diameter and a few centimetres long (Figs. 7, 8). This kind of grain boundary is a network of dislocations (edge dislocations for the pure flexion and screw dislocations for the pure torsion). The strong thermal gradients during the Czochralski process cause these dislocations to multiply and the dislocation density is around 10^8 per square cen-

Fig. 9. — Seeds for large angle grain boundary.

Fig. 10. — $\Sigma = 25$ bicrystal.

of the bicrystals are completely dislocation free as shown on figure 11.

Fig. 11. — Lang topograph of a $\Sigma = 25$ bicrystal. The horizontal white line is the grain boundary. The vertical white lines are growth striations ($\times 3$).

5. Conclusion.

Silicon bicrystals have been grown by the Czochralski technique. The results obtained with large angle bicrystals are excellent: the specimens are about 3 cm in diameter and some 10 cm long. Some bicrystals have been produced with no dislocations in the grains.

Bicrystals are good specimens for investigating the different phenomena involving the grain boundaries. Indeed such specimens allow us to isolate one grain boundary and owing to the size of the bicrystal many types of complementary investigation can be performed.

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