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The Relation Between Dislocation Density and Crystal Crosscut in β-Sn Grown by Modified Bridgman Method

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Abstract

Crystals β-Sn are grown from ingotsof 99.99% prity by modified Bridgman method under 10^{-3} torr pressure and oriented by Laue back reflection method. The relation between crystal crosscut and dislocation density calculated using etch hillock technique is investigated. It is found that the dislocation density increases with increasing crystal crosscut.

1. Introduction

The first suggestion of dislocations has been provided by observations [1, 2] in the nineteenth century that the plastic deformation of metals proceeded the formation of slip bands or slip packets, wherein one portion of a specimen sheared with respect to another. Taylor, Orowan, and Polanyi [3] introduced dislocations into physics in the 1930's. By the late, the investigation methods of individual dislocations could be divided into four main groups [4]. The first method, known as the surface method, is based on the formation of etch pits or hillocks at the site where a dislocation meets the surface. One-to-one correspondence between etch hillocks or pits and dislocations has been proposed by Chockley and Read [5] in 1949 and first observed by Batterman [6] in 1957. The second method is x-ray difraction topography. This method introduces local differences at dislocations in the scattering of x-rays. The other method used by Hedges and Mitchell [4] is the decoration method. Apart from these methods, the dislocations could be observed by means of electron microscopy in which the dislocations are studied in specimens of 0.1-1 micron thick.

In recent years, extensive studies have been conducted to understand the individual dislocation motions and the relation between the dislocation density and the deformation characteristics. A number of investigations have been made by Hirokowa et.al. [7] to confirm one-to-one correspondence between dislocations and etch hillocks which was introduced by Chockley and Read [5] and Batterman [6], using the modified double etch method. Corresponding to this, Düzgün [8] investigated dislocation density using the same method in β-Sn single crystals. It is generally observed that dislocation density depends on crystal shape. Ojima and Hirokowa [9] and Düzgün et.al. [10] showed that the dislocation density varies with the crystal shape. The same results have been obtained by Kojima et.al. [11], Tisivinky [12], Fukutomi and Takatori [13]. Experimental observations about this matter are extremely numerous and in many cases, confusing and contradictory.

The purpose of the present study is to investigate the relation between dislocation density and the crystal crosscut in β - Sn single crystals which grown by modified Bridgman method.

2. Experimental Procedure

2.1. Preparation of Specimen

The white tin used in this study was obtained from Merck Detining Co. From which crystals were grown by modified Bridgman method under 10^{-3} torr pressure using the crystal growth apparatus set up by Düzgün [8] as seen in Figure 2.1. The crystals are 11.34, 22.05, 30.17 mm in crosscut and 30 to 50 mm long. The crystal orientations are found to be close to the [110] direction.

2.2. The Formation of Etch Hillocks

As determined from Düzgün [8], the specimens were chemically polished for one hour at room temperature by a solution which consisted of 1 part HNO_3, 1 part CH_3COOH and 4 parts glycerine. After this procedure, the crystals were etched for 70 s with a solution which consisted of 100 cm^3 % 37 HCl, 100 gr. NH_3NO_3, 500 cm^3 distilled water and 5×10^{-5} mol $CuSO_4.5H_2O$. As a result of etching we calculated the dislocation densities by means of etch hillocks occuring in the crystal surfaces of different crystals with different crystal crosscuts. Results are given in Table 3.1.

Table 3.1. Relation between dislocation density and crystal crosscut.

Sample No:	Crystal crosscut S(mm^2)	Dislocation Density N(cm^{-2})
1	11.34	2.6×10^5
2	22.05	4.6×10^5
3	30.17	8.89×10^5

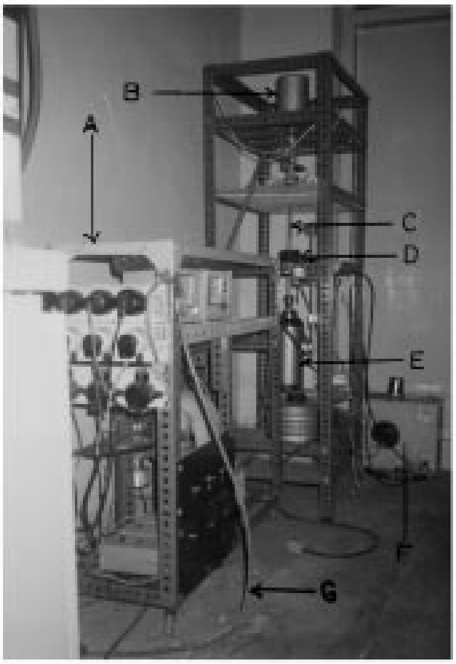


Figure 1. Crystal growth apparatus A-Vacuum pump, B-Protecting header, C-Quartz pipe, D-Furnace, E-Liquid nitrogen, F-Power amplifier, G-Connecting cable.

2.3. Photographic Magnification

The investigations of etch hillocks produced on crystal surfaces were examined with photographic magnification. The etch hillocks were photographed with a metal microscope having an optical arrangement [8, 14]. The observed etch hillocks patterns are given in Figure 2.2.

3. Experimental Results

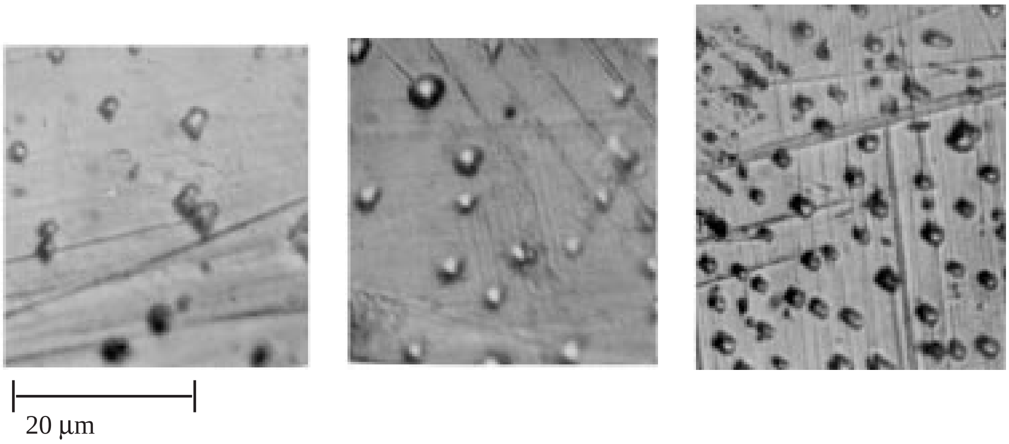
3.1. Geometry of Etch Hillocks

As seen in Figure 2.a, b and c, etch hillocks produced on the etched crystal surfaces $\{001\}$ have a pyramidal shape. The edges of basal squares of hillocks are in the $\langle 001 \rangle$ direction and their four oblique faces are parallel to the $\{101\}$ planes [7, 15].

3.2. The Relation Between Dislocation Density and Crystal Crosscut

Dislocation density is defined as the total length of dislocations per unit volume [8, 9, 10, 11, 16]. The dislocation density in well annealed crystals is usually between $10^6 - 10^8$

cm^{-2}. Hull [4], Young and Savage [17] showed that the dislocation density in Cu single crystals is reduced to about $10^2, 10^3$ cm^{-2} with very careful treatment in crystals grown by modified Bridgman method. The dislocation density in white tin single crystals has been obtained as low as 10^3 cm^2 by Ojima and Hirokawa [9]. In the present study, the calculated dislocation densities as a function of the crystal crosscut are shown in Table 3.1 and in Fig. 3.1. It can be seen that the dislocation density increases with increasing crystal crosscut.



(a) **(b)** **(c)**

Figure 2. Etch patterns on the top face of crystals (a) sample 1, (b) sample 2, (c) sample 3.

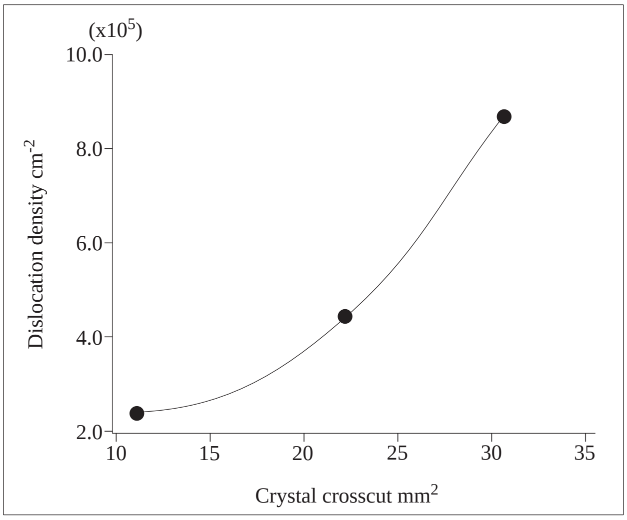


Figure 3. Dislocation density against crystal crosscut.

4. Results and Discussion

Two principal mechanisms for introducing dislocations during the crystal growth have been suggested [4]. These are formation and subsequent movement of dislocation loops by collapse of vacancy platelets and heterogeneous nucleation of dislocations by local internal stresses. It can be seen from Figure 3.1, dislocation density increases with increasing crystal crosscut. This increase is explained by assuming that vacancies escape to the surface in thin crystals, so the vacancies can not collapse to form dislocations. Meanwhile, thin crystals have few subgrain boundaries. Thus a sufficient supersaturation of vacancies to form dislocations in the subgroun boundaries is reduced and dislocation density decreases. These results have been confirmed by Kojima et.al. [11] and Düzgün et.al. [10] with the results obtained in Antrasin and in Cd single crystals, respectively. In Table 4.1, the results from various investigation are given. The results in this study are the same as those of researchers [18, 19]. In contrast, the results are different from those of Ojima and Hirokowa [9]. They showed that the dislocation density in triangular thin plates of $\beta - Sn$ single crystals decreases with increasing crystal width. This case has been interpreted by means of the crystal growth conditions and crystal shape [20]. The dislocation density increases by means of the adhesion force which is produced with the interaction of crystal surfaces and crucible [9, 21]. Meanwhile, the crystals which have corners produce internal stresses. It is expected that the internal stresses are connected with dislocation density [7, 11, 20, 21]. In this study the grown crystals are cylindrical shape and have no corners. The internal stresses are less than those needed for plastic deformation. So, there was no effect on the dislocation density.

Table 4.1. The relationships between dislocation density and crystal width or crosscut in the present study and previous investigations.

Specimen	Crystal width or Crystal crosscut (mm^2 or mm)	Dislocation Density (cm^{-2})	Year	References
β-Sn	2.1 10.3 29.6	8.2×10^5 8.1×10^4 4.0×10^3	1979	Ojima and Hirokawa [9]
Antrasin	1.6 ∅ 4.0 ∅ 7.5 ∅	1.2×10^6 2.7×10^6 4.3×10^6	1984	Kojima et.al. [11]
Cd	2.0 6.3 8.3 9.2	1.4×10^6 5.8×10^6 7.0×10^6 9.2×10^6	1990	Düzgün et.al. [10]
β-Sn	11.34 22.05 30.17	2.6×10^5 4.6×10^5 8.8×10^5	1997	present study

In conclusion, the relation between the dislocation density and crystal crosscut in $\beta - Sn$ single crystals grown by modified Bridgaman method was investigated. We found that the dislocation density increases with crystal crosscut.

5. Acknowledgement

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