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Hardness anisotropy of InP

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Hardness anisotropy measurements in InP single crystals were made using a Knoop hardness tester. The data indicate that hardness is essentially independent of the direction in the (1 1 1) plane, but shows variation with direction in the (1 0 0) and (1 1 0) planes. Contrary to previous observations in crystals of cubic structure, in which the Knoop hardness depends only on the direction of indentation, the hardness of InP is dependent on both plane and direction. The (0 0 1) ⟨1 1 0⟩ combination shows the maximum Knoop hardness number of 430 ± 10.

1. Introduction

Since 1949, when Daniels and Dunn [1] showed that it was possible to measure the hardness anisotropy of metallic single crystals, most work has been done on fcc, bcc, and hcp crystals. During this time, efforts were made to explain hardness anisotropy based on the Schmid Law for slip [1–4]. Experimentally, Garfinkle and Garlick [2] observed for several bcc metallic crystals and for LiF that the Knoop hardness Number (KHN) is dependent essentially on the long direction of indentation and not on the plane of indentation, while the KHN is additionally dependent upon the plane of indentation for hcp crystals. The later works [3, 4] corroborated these results for a wide group of metallic and nonmetallic materials. Brooks *et al.* [3] tried to relate this orientation dependence to the primary slip mode. However, Chin *et al.* [4] showed that for cubic crystals the hardness anisotropy cannot be used to determine the primary slip mode.

In this study, we have attempted to extend the work on hardness anisotropy of cubic crystals to the zinc-blende structure. Hardness measurements of this structure are of interest because of the observed asymmetric dislocations [5] and asymmetric cracking [6]. Abrahams *et al.* [5] observed that the arrangement of misfit dislocations along the two ⟨1 1 0⟩ directions lying in the (0 0 1) junction plane is anisotropic in nature. The misfit dislocations tend to be uniformly distributed along one of the ⟨1 1 0⟩ directions, while in the other ⟨1 1 0⟩ direction there is a tendency for periodic banding of the dislocations. Subsequently Olsen *et al.* [6] showed that the cracking directions follow the asymmetry of the zinc-blende structure. Hence, it was felt that an attempt should be made to see if this asymmetry, might extend to the hardness anisotropy, i.e. to see if the ⟨1 1 0⟩ directions lying in the {0 0 1} plane differ in hardness, and also to compare the hardness anisotropy with that observed in the other cubic systems. We chose InP for the study because it is one of the most promising materials for semiconductor applications.

2. Experimental

The single crystals of InP were prepared by the "liquid encapsulated Czochralski method" as described by Bachmann *et al.* [7] After orientation using a Laue camera, samples approximately 0.1 in. thick were cut on a diamond saw parallel to the (0 0 1), (1 1 0), (1 1 1) planes. The cut samples were ground using 600 grit polishing paper and finally polished with Syton to remove the worked surface. Subsequent re-evaluation by the Laue back-reflection technique showed the spots to be sharp.

All indentations were made on a Kentron microhardness tester equipped with a Knoop diamond indentor and a calibrated eyepiece. All tests were standardized by using a 50 g load and completing each identation within 20 sec. Indentation lengths were measured at a magnification of × 500.

Indentations were made on the (0 0 1), (1 1 0), and (1 1 1) planes. Virtually all of the indentations were crack free. The average value of seven readings for each direction was calculated and a standard deviation was computed. The directions were picked on the basis of the major crystallographic directions for each orientation.

3. Results

Fig. 1 shows the variations of KHN with direction of the long axis of the indentor for the three crystallographic planes. As one can see, the hardness on the (0 0 1) and (1 1 0), is very much dependent on both the direction and the plane of indentation which is at variance with the observation on other cubic systems [1,3,4]. It can also be seen that in the (1 1 1) plane the hardness is fairly uniform and almost independent of direction. On the other hand, the (0 0 1) and (1 1 0) planes show a very hard ⟨1 0 0⟩ direction with the other directions being much softer. Results for the (0 0 1) plane also show that the Knoop hardness numbers of the orthogonal ⟨1 1 0⟩ directions are virtually the same.

From the above data, the values of a number of indentation directions common to two or more indentation planes were extracted and are listed in Table I. Again, it is apparent that the hardness is dependent on both the plane and the direction of indentation. Further, it can be seen that the (0 0 1) ⟨1 0 0⟩ is the hardest plane and direction combination. Also, for a given direction, common to both planes, the (0 0 1) plane is harder than the (1 1 0) plane.

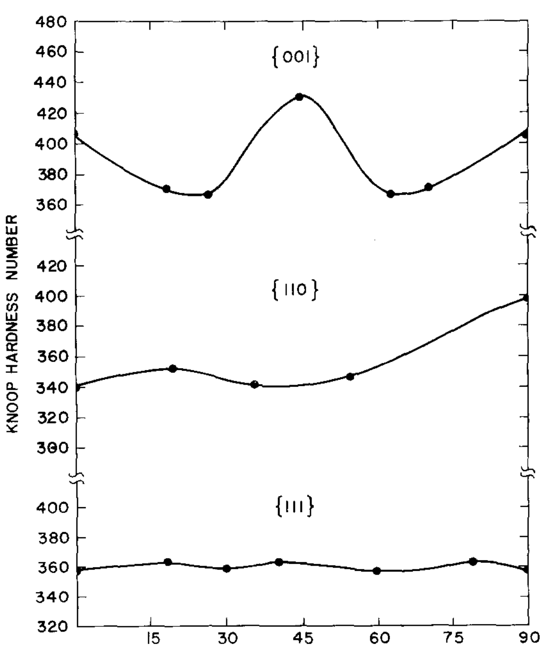

Figure 1 Variation of KHN with direction of identation on three planes.

TABLE I Comparison of KHN data of InP with direction

Direction	Plane	Knoop hardness number (KHN)
⟨1 0 0⟩	{0 0 1}	430 ± 10
	{1 1 0}	299 ± 13
⟨1 1 0⟩	{0 0 1}	406 ± 11
	{1 1 0}	341 ± 15
	{1 1 1}	358 ± 11
⟨1 1 2⟩	{1 1 0}	347 ± 14
	{1 1 1}	359 ± 12

Table II is a list of the average hardness values of the planes and directions which were measured The hardness of each direction in a given plane is the average of all the indentations of all equivalent directions. For example, all the indentations of the ⟨1 0 0⟩ directions were averaged together and a standard deviation was calculated. This resulted in the averaging of from 28 to 56 indentations for each major crystallographic direction.

TABLE II Knoop hardness data for InP

Plane	Direction	Ave. KHN
(0 0 1)	⟨1 1 0⟩	406 ± 11
	⟨2 1 0⟩	370 ± 8
	⟨3 1 0⟩	367 ± 9
	⟨1 0 0⟩	430 ± 10
(1 1 0)	⟨1 $\bar{1}$ 0⟩	341 ± 15
	⟨2 $\bar{2}$ 1⟩	353 ± 16
	⟨1 $\bar{1}$ 1⟩	341 ± 14
	⟨1 $\bar{1}$ 2⟩	347 ± 14
	⟨1 0 0⟩	399 ± 13
(1 1 1)	⟨1 0 $\bar{1}$⟩	358 ± 11
	⟨3 $\bar{1}$ $\bar{2}$⟩	364 ± 11
	⟨2 $\bar{1}$ $\bar{1}$⟩	359 ± 12

4. Discussion

The observation that the zinc-blende structure exhibits asymmetric dislocations [5] and asymmetric cracking [6] along ⟨1 1 0⟩, does not appear to result in an asymmetry of the KHN in this direction, i.e. in the (0 0 1) plane the KHN of the [1 1 0] and [1 $\bar{1}$ 0] directions is essentially identical. This is perhaps not surprising since the stress pattern beneath a hardness indentation is rather complex, with several slip systems expected to be activated simultaneously. Thus any subtle difference between the two directions could be swamped by the complex stress pattern.

The hardness of the samples differ somewhat from that reported by Borshchevskii *et al.* [8] They used a PMT-3 instrument with a 50g load. Their crystals were obtained from polycrystalline ingots and polished along the "vertical cleavage planes," and got a hardness value of 410 ± 2. Since in InP the cleavage plane is (1 1 0) [9], their hardness compares favourably only with that for the ⟨0 0 1⟩ direction in the (1 1 0) plane. Unfortunately, their set-up was not sensitive enough to detect any hardness anisotropy in InP.

The most interesting observation from the present study is that, unlike the other cubic systems, the KHN of the zinc-blende structure is dependent on both the plane and direction. Whether the asymmetric behaviour of the dislocations may in some way be responsible, requires further investigation.

5. Conclusions

Knoop hardness was determined for InP on several crystallographic planes and with the long axis of the indentor aligned in various crystallographic directions. Results are summarized below:

(1) Unlike other cubic systems, the KHN is dependent on both the direction and plane of indentation.

(2) The (0 0 1) ⟨1 0 0⟩ is the hardest plane-direction combination.

(3) The hardness is essentially uniform in the (1 1 1) plane.

(4) No anisotropy could be detected in the ⟨1 1 0⟩ directions.

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