JOURNAL OF NANO- AND ELECTRONIC PHYSICS
Vol. 14 No 2, 02001(3pp) (2022)
ЖУРНАЛ НАНО- ТА ЕЛЕКТРОННОЇ ФІЗИКИ
Том 14 № 2, 02001(3cc) (2022)

Quenching Effect on Mechanical Properties of $In_2Se_{2.7}Sb_{0.3}$ Single Crystal

Piyush J. Patel[1,*], Sandip M. Vyas[2], Vimal A. Patel[2], Himanshu Pavagadhi[2], Ravi Varasada[2], Maunik P. Jani[3]

[1] *Department of Physics, Aditya Silver Oak Institute of Technology, Silver Oak University, Ahmedabad, 382481 Gujarat, India*
[2] *Department of Physics, School of Sciences, Gujarat University, Ahmedabad, 380009 Gujarat, India*
[3] *Department of Physics, Faculty of Science, The M.S. University of Baroda, Vadodara, 390002 Gujarat, India*

(Received 11 January 2022; revised manuscript received 18 April 2022; published online 29 April 2022)

III-VI semiconductor compounds are interesting materials for the fabrication of such devices as ionizing radiation detectors, solid-state electrodes, ion batteries, as well as photosensitive heterostructures and solar cells. The structural complexity of In-Se family has motivated us to examine an unexplored composition (In_2Se_3) and its properties with antimony doping (Sb). The purpose of the present work is to study the influence of antimony on the novel configuration In_2Se_3. Ternary semiconductor compounds in the form of single crystals or thin films have attracted considerable interest because of their structural, optical and electrical properties, which allow them to be widely used in many electronic and optoelectronic devices. As the global energy market seeks applications that offer more efficient electronic systems, $In_2Se_{2.7}Sb_{0.3}$ single crystals synthesized by the modified vertical Bridgeman technique pave the way for exciting innovations in solid-state photovoltaic systems and clean energy sectors. We have grown $In_2Se_{2.7}Sb_{0.3}$ single crystal using the Bridgman-Stockbarger technique. The temperature gradient of the system was kept at 60 °C/cm with a growth rate of 0.35 cm/h. Hardness testing is of significant importance in interpreting the mechanical behavior of materials and correlates with other physical properties. Hardness to deformation of as-grown crystals depends on the bond strength and structural perfection. The microhardness measurements of as-cleaved and quenched samples were made by using a Vickers projection microscope. In this paper, the results have been discussed and reported in detail.

Keywords: Bridgman method, Vickers hardness, As-cleavage, Quenching.

DOI: 10.21272/jnep.14(2).02001 PACS numbers: 81.10. – h, 81.40.Cd, 81.40.Ef

1. INTRODUCTION

A selenium-based chalcogenide alloys are preferred because of their unique reversible transformation properties. Such chalcogenide glasses were used as an optical memory device, rectifiers, photocells, switching and xerography etc. To form a glass selenium is an excellent element but it has demerit due to its small lifetime as well as low sensitivity. As a result, some additives are required to improve the property of selenium [1-7]. Antimony is one of the elemental additives to make crystallization of pure non-crystalline selenium [8].

Crystals of metals and alloys were grown by Bridgman-Stockbarger and zone-melt methods widely used to obtain large-size crystals. Various authors [9-13] have been used such methods for In_2Se_3 single crystal. These intermetallic compounds having hexagonal system with $c/a = 2.714$ and high melting point of 890 °C [12, 13]. InSe like InSb is an III-VI intermetallic compound, but their properties are significantly different from InSeSb compound.

In this paper, authors report the microhardness value has been found to be load dependent in low load range (LLR) in case of as-cleaved as well as quenched treated $In_2Se_{2.7}Sb_{0.3}$ samples. The results have been explained in terms of deformation-induced coherent regions.

2. EXPERIMENTAL TECHNIQUE

Indium, selenium and antimony with 5N purity materials were used for the crystal growth. The elemental materials In, Se and Sb were sealed in a quartz ampoule of 1 cm in diameter and 10 cm in length at 10^{-4} Pa. Such sealed ampoule was kept in alloy mixing furnace at a temperature 100 °C greater than the melting point of the material. After the material was in molten form, it was kept in the same state for 48 h. The ampoule was rotated at 10 rpm for 24 h and then gradually cooled to room temperature.

The alloy of mixed material (sample) was then placed in a Bridgeman (vertical) furnace for the crystal growth. For the best crystal growth, a growth velocity of 0.35 cm/h and a temperature gradient of 60 °C/cm were used. To study the Vickers microhardness, $In_2Se_{2.7}Sb_{0.3}$ crystals were cleaved (1-2 mm thick) into slices at ice temperature with the (111) cleavage plane to minimize stress-free dislocation.

For the quenched sample, the material was sealed under 10^{-2} Pa pressure and then kept in a preheated furnace of 200 °C for 24 h. Then, after the sample was quenched, it was lowered into an ice bath (ice temperature). Vickers microhardness measurements were carried out using a pyramidal diamond indenter on the cleavage plane. The indentation marks were made with different loads ranging from 10 to 1000 mN for fixed azimuthal orientations of the indenter to avoid anisotropic variation. The 35 s constant indentation time was kept for the quenched as well as as-cleaved samples. The results are discussed below.

* physicsathgce@gmail.com

 © 2022 Sumy State University

3. RESULTS AND DISCUSSION

Fig. 1 shows the square pyramidal shape of the indentation mark. The diagonals of the indentation mark were measured by a micrometer eyepiece having least count of 0.19 micron.

For the decisive constant indentation time, the Vickers hardness (VHN) was measured between 5 to 60 s under the 1000 mN applied load at room temperature. Here, it was observed that the 1000 mN load is high enough to be insensitive to the hardness number.

Fig. 2 shows measurements of *Hv* versus *t* (time) at room temperature for $In_2Se_{2.7}Sb_{0.3}$ crystals. From the figure, it is seen that the VHN increases with time and becomes independent of time (constant) starting from 35 s. Hence indentation time of 35 s was kept constant to study the hardness versus load for as-cleaved as well as quenched $In_2Se_{2.7}Sb_{0.3}$ crystals, as shown in Fig. 3.

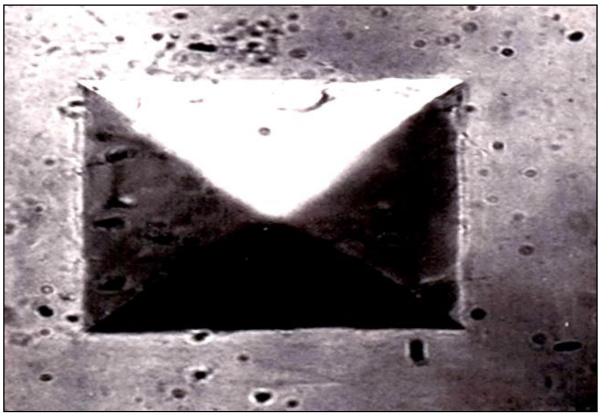

Fig. 1 – Vickers hardness indentation mark

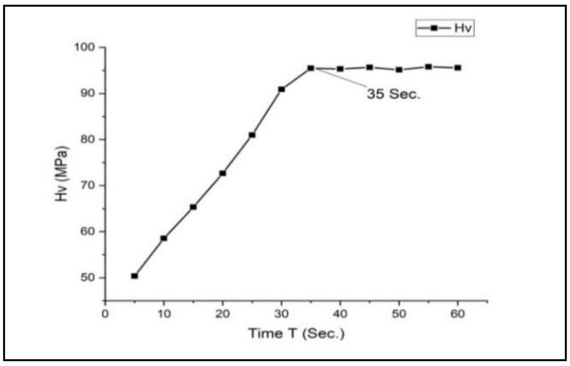

Fig. 2 – Plot of hardness *Hv* versus loading time *t*

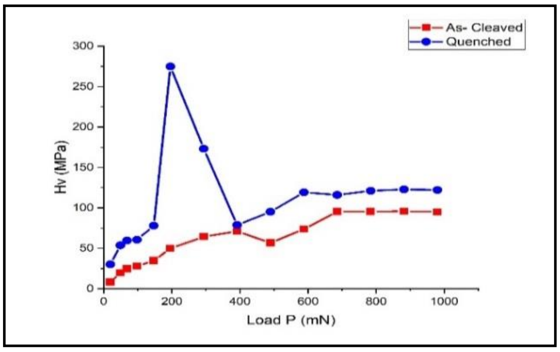

Fig.3 – Variation of microhardness with applied load

Table 1 – Microhardness value of $In_2Se_{2.7}Sb_{0.3}$ single crystal

Sample name	Treatment	Hardness HV (MPa)	(%) change
$In_2Se_{2.7}Sb_{0.3}$	As-cleaved	95	–
	Quenched	125.82	13.24 % (increase)

Table 1 shows the independent hardness (VHN) of $In_2Se_{2.7}Sb_{0.3}$ crystals for as-cleaved and quenched samples. The LLR dependence of the hardness is well known. Here, hardness is seen to be independent of load for loads in a range only above 700 mN, such region of load is known as high load region (HLR), which represents bulk hardness for the material. From Fig. 3, the Vickers hardness for as-cleaved and quenched $In_2Se_{2.7}Sb_{0.3}$ crystals is found to be 95 MPa and 125.82 MPa, respectively.

In Fig. 3, the LLR variation of hardness is due to strain hardening of the surface layers in metals and alloys. It plays an important role for the progressive loading of crystals, and the hardness of quenched samples increases due to quenched phenomena.

The load-dependent hardness variation in LLR has been explained in terms of penetration depth of the indenter and strain hardening of surface layers. The hardness of this material shows empirically two parts: one at a load less than the peak load, and the other in the intermediate load range [14, 15].

As load increases, the depth of penetration increases and hence plot in Fig. 3 reflects the characteristic response of the zone depth of penetration made by the indenter. The hardness variation in LLR and intermediate load range are explained in terms of deformation-induced coherent regions [16, 17].

Coherent region in $In_2Se_{2.7}Sb_{0.3}$ crystals extend to depth penetration, at LLR, dislocation creates interacting jogs with progress of penetration. So, an increase in hardness in LLR is due to work hardening, and in the intermediate region, hardness decreases after the peak due to work softening.

Thus, it can be seen that the strain hardening due to quenching is more in the $In_2Se_{2.7}Sb_{0.3}$ crystal with respect to as-cleaved. The Vickers hardness number increases by 13.24 % from 125.82 MPa, which is 13.24 % more than the hardness observed in the as-cleaved sample. The softer the crystal, the more mobile the dislocations will be, resulting in a crystal showing a higher tendency to strain. So that quenching leads to considerable hardening of the crystals.

The hardness value is maximum at its peak and has complex variation with increasing load, becoming constant at sufficiently high loads. Shah et al. [18] reported that the bulk characteristic microhardness is usually represented by the value in the saturation region.

Vyas et al. [19] reported for SnSe and SnS single crystals that quenching is known to have the most pronounced effect on the crystal boundaries of surfaces. And they also noted that, in the general nature of the load dependence of hardness, the effect of quenching on the magnitude of hardness and on the extent of the coherent region also follows similar trends in crystals.

Shah et al. [18] reported for $InBi_{1-x}Sb_x$ single crystals that the quenching effect is known to increase dislocation density and to create a complex network of immobile dislocations acting as a strong barrier to the motion of new dislocations.

4. CONCLUSIONS

The surface microhardness is a load-dependent quantity, its variation is quite prominent in LLR, and only at sufficiently high applied loads it becomes virtually independent of load. The hardness peaks observed in the *Hv* versus load plots can be explained in terms of deformation-induced coherent regions. The value of the independent microhardness of 125.82 MPa of the quenched sample increases by 13.24 % with respect to the value of the independent microhardness of 95 MPa of the as-cleaved sample.

ACKNOWLEDGEMENTS

We are grateful to DRS-SAP and DST-FIST programs sponsored by the Department of Physics, School of Science, Gujarat University, Ahmedabad, Gujarat, India. Authors are also thankful to Dr. P.R. Vyas and Prof. P.N. Gajjar, Head of physics department, school of sciences, Gujarat University, Ahmedabad for their constant encouragement. We are highly thankful to Assistant Professor Dr. Om P. Joshi (Department of English, Bhakta Kavi Narsinh Mehta University, Junagadh) for his valuable suggestions to improve English language of the manuscript.

REFERENCES

1. S. Sharma, S. Sharma, P. Kumar, R. Thangaraj, M. Mian, *Mater. Today: Proc.* **4**, 10446 (2017).
2. D. Eddike, A. Ramdani, G. Brun, J. Tedenac, B. Liautard, *Mater. Res. Bull.* **33**, 519 (1998).
3. M. Spiesser, R.P. Gruska, S. Subbarao, C.A. Castro, A. Wold, *J. Solid-State Chem.* **26**, 111 (1978).
4. J. González-Leal, P. Krecmer, J. Prokop, S. Elliott, *J. Non-Crystall. Solids* **326**, 416 (2003).
5. A. Seddon, W. Pan, D. Furniss, C. Miller, H. Rowe, D. Zhang, T. Benson, *J. Non-Crystall. Solids* **352**, 2515 (2006).
6. E.R. Shaaban, M.A. Kaid, E.S. Moustafa, A. Adel, *J. Phys. D* **41**, 125301 (2008).
7. P. Sharma, S. Katyal, *J. Non-Crystall. Solids* **354**, 3836 (2008).
8. K. Bindra, N. Suri, M. Kamboj, R. Thangaraj, *Thin Solid Films* **516**, 179 (2007).
9. J. Sanghera, I. Aggarwal, *J. Non-Crystall. Solids* **6**, 256 (1999).
10. T.M. Jani, G.R. Pandya, C.F. Desai, *Cryst. Res. Technol.* **28**, K40 (1993).
11. G.R. Pandya, S.M. Vyas, *Cryst. Res. Technol.* **28**, 163 (1993).
12. J. Ye, S. Soeda, Y. Nakamura, O. Nittono, *Jpn. J. Appl. Phys.* **37**, 4264 (1998).
13. P. Patel, S.M. Vyas, V. Patel, H. Pavagadhi, M. Solanki, M.P. Jani, *AIP Conf. Proc.* **1675**, 030030 (2015).
14. J.R. Pandya, L.J. Bhagia, A.J. Shah, *Bull. Mater. Sci.* **5**, 79 (1983).
15. C.F. Desai, P.H. Soni, S.R. Bhavsar, *Bull. Mater. Sci.* **22**, 21 (1999).
16. H. Buckle, *Rev. Met. Paris* **48**, 957 (1951).
17. Braunovic, *The Science of Hardness Testing and Its Research Applications* (West Brook and Conrad: ASM Ohio) (1973).
18. D. Shah, G.R. Pandya, S.M. Vyas, M.P. Jani, *Turk. J. Phys.* **31**, 231 (2007).
19. S.M. Vyas, G.R. Pandya, C.F. Desai, *Indian J. Pure Appl. Phys.* **33**, 191 (1995).

Вплив загартування на механічні властивості монокристала $In_2Se_{2.7}Sb_{0.3}$

Piyush J. Patel[1], Sandip M. Vyas[2], Vimal A. Patel[2], Himanshu Pavagadhi[2], Ravi Varasada[2], Maunik P. Jani[3]

[1] *Department of Physics, Adity Silver Oak Institute of Technology, Silver Oak University, Khyati Foundation, Gujarat, India*

[2] *Department of Physics, School of Sciences, Gujarat University, Ahmedabad 380009, Gujarat, India*

[3] *Department of Physics, Faculty of Science, The M.S. University of Baroda, Vadodara 390002, Gujarat, India*

Напівпровідникові сполуки III-VI є цікавими матеріалами для виготовлення таких приладів як детектори іонізуючого випромінювання, твердотільні електроди, іонні акумулятори, а також фоточутливі гетероструктури та сонячні елементи. Структурна складність сімейства In-Se спонукала нас до дослідження невивченого складу In_2Se_3 та його властивостей із легуванням сурмою (Sb). Метою даної роботи є вивчення впливу сурми на нову конфігурацію In_2Se_3. Потрійні напівпровідникові сполуки у вигляді монокристалів або тонких плівок викликають значний інтерес через свої структурні, оптичні та електричні властивості, які дозволяють їх широко використовувати в багатьох електронних і оптоелектронних пристроях. Оскільки світовий енергетичний ринок шукає застосування, які пропонують більш ефективні електронні системи, монокристали $In_2Se_{2.7}Sb_{0.3}$, синтезовані за модифікованою вертикальною технікою Бріджмена, відкривають шлях для захоплюючих інновацій у твердотільних фотоелектричних системах та секторах чистої енергії. Ми виростили монокристал $In_2Se_{2.7}Sb_{0.3}$ за методикою Бріджмена-Стокбаргера. Температурний градієнт системи підтримували на рівні 60 °C/см зі швидкістю росту 0,35 см/год. Випробування твердості має важливе значення для інтерпретації механічної поведінки матеріалів і корелює з іншими фізичними властивостями. Твердість до деформації вирощених кристалів залежить від міцності зчеплення та досконалості структури. Вимірювання мікротвердості розщеплених і загартованих зразків проводили за допомогою проекційного мікроскопа Віккерса. У статті результати досліджень обговорені та докладно викладені.

Ключові слова: Метод Бріджмена, Твердість за Віккерсом, Розщеплення, Загартування.