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NANOMECHANICAL CHARACTERIZATION OF INDIUM PHOSPHIDE EPILAYER USING NANOINDENTATION TECHNIQUE

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Abstract— The Nanomechanical characteristics of InP epilayer grown on GaAs (100) substrate is studied. The mechanical characteristics of the material such as hardness, modulus of elasticity, stiffness, contact depth etc. were studied by Nanoindentation technique with different probe geometries like Berkovich and Vickers. The results show significant variation in the mechanical parameters with respect to the tip geometry and the measured hardness values were observed to increase with the applied load.

Keywords- InP, Nanoindentation, Hardness

I. INTRODUCTION

InP is used in high-power and high-frequency electronics because of its superior electron velocity with respect to the more common semiconductors silicon and gallium arsenide. InP material is especially noted for its high operating speeds, low noise, low voltage and high reliability. The industry is shifting from traditional materials such as Gallium Arsenide (GaAs) to the next generation of high speed circuits using InP. InP is used in both photonic and electronic applications. Typical photonic device types include: lasers, photo-detectors, avalanche photo diodes, optical modulators and amplifiers, waveguide-based devices, quantum photonic devices, and both optoelectronic and photonic integrated circuits as well as new devices for optical communications, switching, networking, signal processing and leading edge material for solar cells because of the direct bandgap of this semiconductor. Hence, an understanding of the physical properties of InP is essential for its wider use. As in other semiconductors, InP exhibits a brittle or ductile behavior depending on the applied stress, temperature, and strain rate. A number of investigators have performed deformation tests in the ductile regime of InP to study its plastic deformation [1-2]. Indium phosphide (InP) is a binary III-V direct bandgap semiconductor compounds. Its natural structure under standard condition is the FCC (zinc blende) structure. With modern growth techniques, InP based devices are now routinely manufactured for technological applications. But to fabricate a highly efficient device, growth of epilayers on suitable substrate needs lots of studies as the lattice mismatch will generate stresses and dislocations between the substrate and epilayers leading to reduction in the device performance. Hence the mechanics of the epilayers at nanoscale have an impact on device performance. So, to improve the characteristics of the epilayers over substrate the study of the mechanical

properties of the epilayer structure is very important and the nanoindentation technique is chosen to study the mechanical properties of the material which will give an in-depth knowledge about the mechanical properties of the epilayers [3-4]. The principal goal of mechanical characterization of thin film systems using depth sensing indentation tests is to extract elastic modulus and hardness of the specimen material from experimental readings of indenter load and depth of penetration. The surface properties studied by nanoindentation can reveal information about elastic recovery, relative hardness and in particular surface flow properties. There are some reports on the surface study and the deformation involved in InP epilayers studied by nanoindentation technique [5-9].

Here, we determine the indentation load-displacement behavior of InP semiconductor alloys using Berkovich indenter and Vickers indenter. A study of the mechanical properties of InP is of interest because of the fundamental importance of this material in the electronics industry. Its larger electron mobility aids in high-speed applications and particularly because of its optical properties, this results from the fact that InP is a direct band-gap material. Understanding the mechanical properties such as hardness of the InP at the nanoscale is crucial for the device applications. The intrinsic deformation behavior of the semiconductor is of the particular interest because they are widely used for the fabrication of electronic device, which operate in large range of stresses and temperatures.

II. EXPERIMENT

The samples investigated were InP epitaxial layer grown on GaAs (100) substrate grown by Metal Organic Vapour Phase Epitaxy (MOVPE) technique of thickness around 300nm. Hardness The Hardness, Elastic modulus, Penetration depth, Stiffness values were calculated from the load-displacement (p-h) data

obtained by the Nanoindentation with T1950 Tribonanoindenter.

The indentation P-h data obtained at each depth were analyzed to determine the hardness, H, and elastic modulus, E, by using the Oliver and Pharr method [3]. In this method, the hardness and modulus are determined as following. Nanoindentation hardness is defined as the indentation load divided by the projected contact area of the indentation. It is the mean pressure that a material will support under load. From the P-h curve, hardness can be obtained at the peak load and given by equation (1) as

$$H = \frac{P_{max}}{A} \quad (1)$$

where A is the projected contact area.

The elastic modulus was calculated using the Oliver-Pharr data analysis procedure [3] and defined as:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E_{sample}} + \frac{1-\nu^2}{E_{indenter}} \quad (2)$$

which is calculated from the contact stiffness S using the following formula:

$$S = \frac{2\beta}{\sqrt{\pi}} E_r \sqrt{A} \quad (3)$$

and E and ν in Equation (2) are the elastic modulus and Poisson's ratio and, β is a constant which depends on the geometry of the indenter ($\beta = 1.034$ for a Berkovich indenter) [9]. The contact area A is determined from the probe area function (Ah_c) where h_c , the contact depth, is given by:

$$h_c = h_{max} - \epsilon \frac{P_{max}}{S} \quad (4)$$

where h_{max} is the maximum indenter penetration depth, and ϵ is a constant depending on the indenter geometry ($\epsilon = 0.75$) [10].

Using the Nanoindentation technique, tests were performed in order to measure the Hardness, Elastic modulus of the samples and to build a depth profile. On the InP samples, load-controlled depth profiles were performed using the three side pyramidal Berkovich indenter and four side pyramidal Vickers indenter probes with radius of curvature of 50nm and 100nm respectively.

The indents were made on the sample with specified load function, keeping a specified hold time. The unloading curve segment obtained in the experiment was considered purely elastic (Oliver Pharr 1992) and the mechanical properties such as Elastic modulus, Hardness and contact depth values were calculated at loads 3000 μ N, 5000 μ N, and 7000 μ N. The Hardness and the reduced modulus were studied from the Load-displacement plot.

III. RESULTS AND DISCUSSION

A. Load- Displacement curve

Fig.1 and 2 shows the comparative typical load versus displacement (p-h) curves of InP epilayer at different loads of 3000 μ N, 5000 μ N, and 7000 μ N

using both the Vickers indenter and Berkovich indenter. The maximum indentation depth is obtained at about 116, 250 and 187 nm by Vickers indenter and 122, 175 and 220 nm by Berkovich indenter at loads 3000, 5000 and 7000 μ N, respectively.

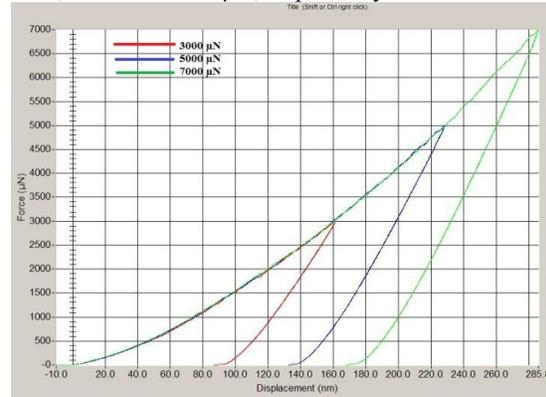


Figure 1. Load-Displacement curves for three different loads performed on InP epilayer using Vickers indenter

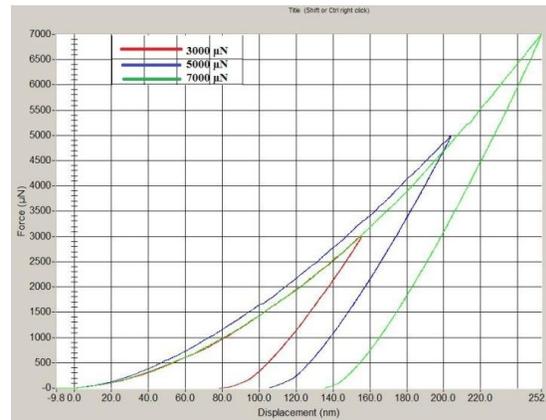


Figure 2. Load-Displacement curves for three different loads performed on InP using Berkovich indenter

B. LOAD Vs HARDNESS

Fig. 3 shows the variation of hardness values with the applied load for InP epilayers. For lower load, the hardness value is low, as the load increases the hardness also increases. The hardness values varying drastically with load between 3000 to 7000 μ N. With increased applied load, the depth of indentation increases and the effect of the surface layer becomes less and for larger loads, when the impression reaches a depth at which an undisturbed layer of material exists, the value of the hardness becomes independent of the load. It is interesting to note that in all the cases, the hardness increases with increase of load. This nonlinear relation can be represented by Meyer's equation $P = kdn$, where n is the indentation size effect (ISE) coefficient, k the material constant, d the diagonal length and P is the applied load. The increase of hardness with load is the characteristics often referred to an indentation size effect.

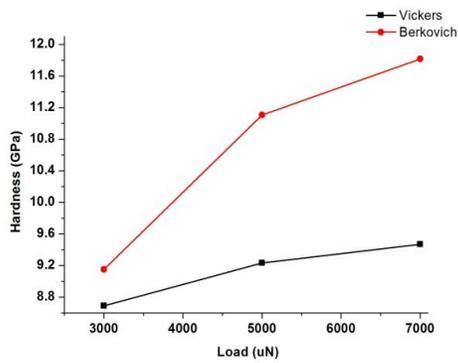


Figure 3. Variation of hardness with applied load

C. CONTACT DEPTH VS HARDNESS

The fig. 4 shows the variation of hardness as a function of contact depth for the InP epilayers. The hardness value drastically changes with contact depth and as maximum depth of penetration obtained with increasing applied load, the hardness value also increases. Goryunova [11] has calculated microhardness for the bulk sample of InP as 5.35(±0.40) GPa using the Knoop indenter. The hardness of the InP epilayer is considerably higher than the bulk value of InP, which could be due to the strain relaxation at the interface of the epilayer. Raman spectroscopic analysis has also confirmed that the strain decreases with increasing thickness, which is mainly due to the strain relaxation at the interface [6].

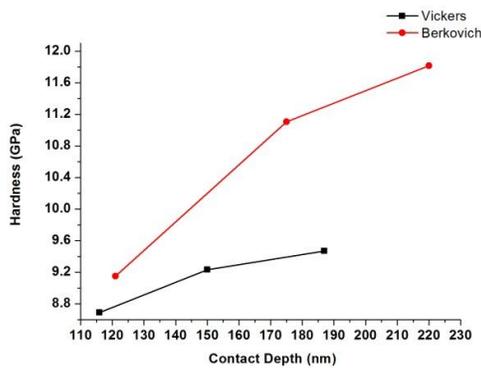


Figure 4. Hardness as a function of contact depth

D. LOAD VS REDUCED MODULUS

The fig. 5 shows the variation of modulus of elasticity as a function of applied load. The observed data shows that the modulus of elasticity decreases with the maximum depth of penetration obtained with increasing applied load.

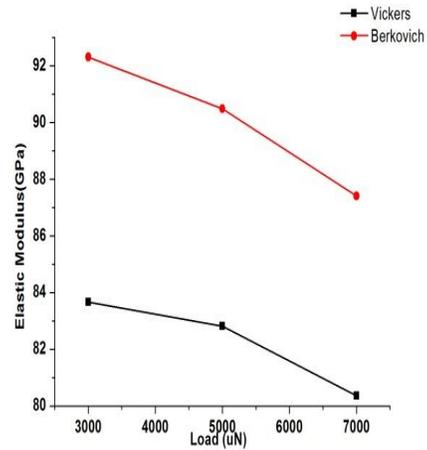


Figure 5. Modulus of elasticity as a function of load

E. TOPOGRAPHICAL IMAGE OF NANOINDENTATION

Fig. 6(a) and (b) shows the SPM images of the InP epilayer at a peak load of 5000 μN with both Berkovich and Vickers indenter probe.

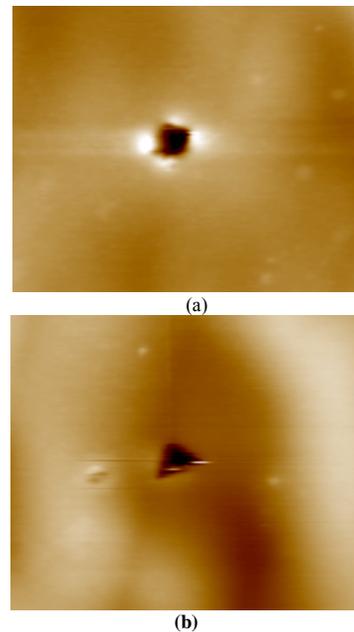


Figure 6. SPM images showing the indentation impression on the InP epilayers (a) using Vickers indenter (b) using Berkovich indenter

Fig. (a) and (b) shows the typical 3D topographical in situ image of the InP epilayer. By indenting with Vickers and Berkovich probe, from fig. 7(a and b) it is clearly observed the piling up in the materials and is due to the reason that the most of the plastic deformation occurred near the indenter and a distinct stress propagation in the sample. The large piling up near the facet of the indentation mark is a further evidence of plastic behaviour of InP film.

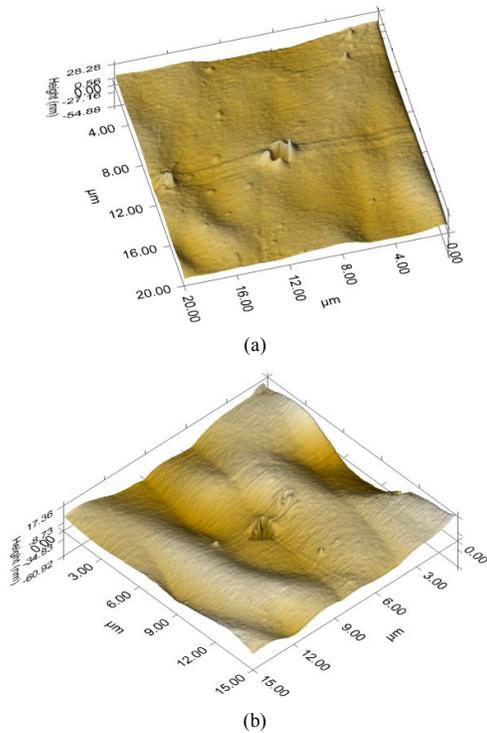


Figure 7. 3D topographical image of the indented surface using (a) Vickers indenter and (b) Berkovich indenter

Also Almeida [8] explains that residual impressions with pile up at the surface have been observed for indentations in the elastic regime for InP at the instrument resolution. He explained the fact of pilling up by attributing to its plastic deformation of the native oxide layer. The observed pile up is formed by the flow of oxide out from the pit during the indentation. Due to broken bonds in the oxide layer, some oxide adheres to the diamond tip, leading to the formation of the roughness observed on the pile-up rim and the deformation diameter. This unexpected plastic deformation on the native oxide layer causes an increase in the real contact area between the tip and the surface under pressure.

Table 1 given below compares the various mechanical parameters obtained such as Hardness, Elastic modulus, stiffness for the InP sample. The discrepancies among the mechanical parameters by various indentation methods are mainly due to specific tip-surface contact configuration and stress distribution inherent to each type of indenter tip. From the data shown in the tables 1 it is cleared that the hardness for the epilayer varies with load. The hardness value increases with the increasing load whereas the elastic modulus values decreases with increased load and the fact mainly attributed to the indentation size effect.

Table 1: The The Hardness, Elastic modulus, Penetration depth, Stiffness values of InP epilayer determined using Vickers and Berkovich

Load (μN)	Hardness (GPa)		Elastic Modulus (GPa)		Depth of penetration (nm)		Stiffness ($\mu\text{N}/\text{nm}$)	
	Vickers	Berkovich	Vickers	Berkovich	Vickers	Berkovich	Vickers	Berkovich
3000	8.687	9.150	83.665	92.316	116.02	121.44	55.488	56.486
5000	9.232	11.105	82.819	90.488	150.58	174.24	68.782	69.908
7000	9.468	11.816	80.354	87.415	186.60	219.26	77.973	78.600

IV. CONCLUSION

Mechanical behaviour of InP epilayers were investigated using Nanoindentation with both Berkovich and Vickers diamond indenters. The hardness values of the InP epilayers were found to lie between 8.6 GPa and 11 GPa and the stiffness of the epilayer found to lie between 55 $\mu\text{N}/\text{nm}$ and 78 $\mu\text{N}/\text{nm}$. The measured hardness and stiffness values were observed to increase with the applied load owing to indentation size effect. InP epilayers shows pilling up phenomena due to the indentation effect and it is attributed to the further evidence of plastic behaviour of InP film.

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REFERENCES

[1] B. Leonardus, A. Bayu and P. Pirouz, “Brittle-to-ductile transition temperature in InP,” *Phys. Status Solidi A*, Vol 207, No. 5, pp.1190–1195 December 2010.

[2] G. Patriarche and E. Le Bourhis, “In-depth deformation of InP under a Vickers indenter,” *Journal of Material Science*, Vol. 36, pp. 1343–1347, 2001.

- [3] W.C.Oliver, G.M. Pharr., "An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments," *Journal of materials research.*, Vol 7, No.6, June 1992.
- [4] E. Le Bourhis, and G. Patriarche, "Nanoindentation response of compound semiconductors," *phys. stat. sol. (c)* 4, No. 8, pp. 3002–3009 May 2007
- [5] R. Navamathavan, D. Arivuoli, G. Attolini, C. Pelosi, Chi Kyu Choi., "Mechanical properties of InAs/InP semiconductor alloys," *Applied Surface Science*, Vol 253, Issue 5, pp. 2657–2661, December 2006.
- [6] R.Navamathavan, , D. Arivuoli , G. Attolini, C. Pelosi., "Nanoindentation studies of (111) GaAs/InP epilayers," *Applied Surface Science* Vol 180, Issues 1–2, pp. 119–125 August 2001.
- [7] C. Coupeau, J. Grilh'E , "Role of dislocation structure on nanomechanical properties," *Journal Of Materials Science Letters* Vol. 19 pp. 259– 262, 2000.
- [8] C. M. Almeida, R. Prioli and F. A. Ponce, "Effect of native oxide mechanical deformation on InP nanoindentation," *Journal Of Applied Physics* Vol. 104, pp.113509-6 Dcember 2008.
- [9] J. Yan, J. Tamaki, H. Zhao and T. Kuriyagawa,"Surface and subsurface damages in nanoindentation tests of compound semiconductor InP," *J. Micromech. Microeng.* Vol. 18, pp.105018-10, September 2008
- [10] Sheng-Rui Jian , Te-Hua Fang , Der-San Chuu, "Nanomechanical characterizations of InGaN thin films", *Applied Surface Science*, 252, 3033–3042, (2006)
- [11] N.A. Goryunova, J.C. Anderson, "The chemistry of diamond like semiconductors," Chapman & Hall, London, 1965.

