NANOINDENTATION STUDIES OF HgCdTe EPITAXIAL FILMS AND CdZnTe SINGLE CRYSTAL SUBSTRATES

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February 2023

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by

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Submitted

In fulfillment of the requirements of the degree of Doctor of Philosophy

to the

INDIAN INSTITUTE OF TECHNOLOGY DELHI

February 2023

CERTIFICATE

This is to certify that the thesis entitled “Nanoindentation studies of HgCdTe epitxial films and CdZnTe single crystal substrates” being submitted by Mr. Hemant Kumar Sharma to the Indian Institute of Technology Delhi for the award of the degree of Doctor of Philosophy in Department of Applied Mechanics is a bonafide research work carried out by him under our supervision and guidance. To the best of our knowledge, the thesis has reached the requisite standard. The research reports and the results presented in this thesis have not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

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ACKNOWLEDGEMENTS

Firstly, I am thankful to my supervisors, **Dr. Rajesh Prasad** and **Dr. Rajesh Kumar Sharma** for their continuous support and guidance for each part of the research being carried out during this period. I am highly grateful to Dr. Rajesh Prasad for enlightening the path of research by guiding and discussing various aspects of this research. He has provided his valuable time even during the weekends and holidays for discussions, as I was a part-time student and was performing official duties during weekdays. I got his constant encouragement during the coursework of my Ph.D. He has always been supportive and motivational during the entire period of my Ph.D. journey at IIT Delhi.

Dr. Rajesh Kumar Sharma was my supervisor, Divisional officer, and Director of my institution, Solid State Physics Laboratory (SSPL). I have learnt the intricacies of liquid phase epitaxy technology under his guidance. Despite his hectic schedule as Director, SSPL, he has agreed to guide me, and I will be highly thankful to him. Under his guidance, I learnt research methodologies, which will be useful for my further research carrier. His immense knowledge of semiconductor materials and devices has motivated me to learn new things in this field. His fatherly behaviour has motivated me to excel not only in the field of science but also in my personal life. I have learnt not only technical but many great personal tracts, which will be useful in my life.

I am thankful to **Dr. Raghvendra Sahai Saxena**, my senior colleague at SSPL. I have learnt the principles of infrared technology from him. He was like my mentor during the whole Ph.D. duration. He has helped me in writing research papers and thesis. Without his help, it was a nightmare for me to complete my thesis in this present form. He has also helped in communicating the research papers and suggested the journals and conferences during the Ph.D. He has also analysed the mathematical data presented in this work. Whenever any help was required, he was always ready. I will be highly indebted for his help.

The epitaxial films have been developed at the MCT-LPE group in SSPL. I wish to record my sincere thanks to all LPE members, **Dr. R. Raman**, **Dr. Rachna Thakur**, **Dr. Chanchal**, **Mr. Varun Sharma**, **Mr. Jayant Ojha**, **Mr. Naryan and Mr. Sanjeev Sharma** for their cooperation during work. I am also thankful to **Mrs.**

Meenakshi Srivastava for providing the CdZnTe substrates used in this study. I wish to thanks the Directors of SSPL, **Dr. Rajesh Kumar Sharma** and **Dr. Seema Vinayak**, for permitting me to pursue Ph.D. and for providing managerial and financial support during the study. I am also thankful to **Dr. Renu Tyagi**, head publication committee and technology director, advanced materials, SSPL, for her support in publishing research papers.

The characterization group of SSPL has provided support for characterizing the samples during this Ph.D. My sincere gratitude to **Mr. Anand**, **Mrs. Garima**, **Mr. Sandeep dalal**, **Mrs. Monika kumari**, **Mrs. Alka Raina**, **Mr. Aman arora** and **Dr. Akhlesh Pandey** for helping to characterize the samples.

As a part-time student, I needed to fulfil my duties at the workplace, apart from doing Ph.D. work. Nanoindenter was not available in my office, and hence used IIT Delhi facilities. Due to this reason, nanoindentation experiments have been usually planned on weekends or holidays. I am thankful to the research scholars of the materials lab, department of material science and engineering, IIT Delhi, for helping me perform my experiments. I sincerely thank **Dr. Aditya Gokhale**, who taught me to operate nanoindenter and helped me perform my experiments. I am also thankful to **Dr. Deepak Chauhan** for helping me to characterize my samples.

I will not forget to express my sincere thanks to the management of IIT Delhi, who gave me admission to pursue my Ph.D. from IIT. I feel proud to be a research scholar of this institute of eminence, one of the India's top institutes. During my research, I got support from all relevant sections of the institute, including P.G. Section, Security, Dean (academics) section, central library, etc.

My gratitude also goes to a few M.Tech. Students who have helped me during coursework. I am thankful to **Mr. Monu Nath**, **Mr. Puneet Sharma**, **Mr. Nanhe Gupta** and **Mr. Divya Prakash Dubey** for helping me and providing relevant literature for the completion of the coursework.

I wish to thank my friends **Dr. Sandeep Mahajan**, Scientist, C-MET, Hyderabad and **Dr. Ashok Yogi**, Executive Director, Rajasthan Tourism Development Corporation (RTDC), for providing me moral and technical support during the tenure of my PhD.

I am highly grateful to my father, **Shri Manohar Lal Sharma**, my mother, **Smt. Chandra Kala Sharma** and my big brother **Mr. Hari Shankar Sharma** who has encouraged me to pursue my Ph.D. and excel in the field of research. They have shown their confidence in me and motivated me during the entire period of my Ph.D.

I wish to acknowledge my hearty thanks to my wife **Divya,** and my son **Arnav.** My wife has supported my studies by taking more family responsibilities. At one time, it was difficult for her to manage my studies, her job and child care; she left her job and put my studies on top priority list. I am highly obliged to her. My Son has missed his father many weekends during the research work. Sometimes, he used to come to IIT along with me so that he could get my time during driving. He has sacrificed his childhood time; still, as a child, he motivated me to complete my Ph.D. so that he can hear my name as Dr. Hemant Kumar Sharma.

Lastly, I am thankful for the almighty for providing the courage to pursue my higher studies.

(Hemant Kumar Sharma)

ABSTRACT

Semiconductor materials are the backbone of today's technology era. The revolutionary electronic devices, such as microprocessors, field programmable gate arrays (FPGA), dynamic random access memories (DRAM), micro-electro mechanical system (MEMS) based sensors, optoelectronic devices, infrared detectors, night vision devices, monolithic microwave integrated circuits (MMIC) etc. are made of the semiconductor materials. These materials undergo a variety of mechanical processes for the fabrication of crystalline wafers and epitaxial heterostructures, such as slicing, lapping, and mechanical and chemo-mechanical polishing. While performing these processes, the materials are subjected to various mechanical loading conditions, which may lead to deformation or defect-generation. The electrical properties of these materials are critically influenced by the extended surface or subsurface defects generated during these processes. Also the performance of most of the semiconductor devices degrades with the presence of bulk or surface defects in the material. Apart from the mechanical damage, a local phase transformation may also take place during the application of applied stress, which may lead to change in the electrical and mechanical properties as well. Therefore, understanding the changes in mechanical properties and deformation behaviour due to mechanical processes is important for the development of high-performance semiconductor devices. Nanoindentation is a widely used technique, particularly for crystalline materials, for extracting their mechanical properties, such as hardness and elastic modulus, and studying the surface or subsurface deformation behaviour. In the present work, nanoindentation studies are performed on HgCdTe epitaxial films and CdZnTe single crystal substrates, which are one of the most demanding semiconductor materials for the development of infrared detectors.

The first chapter of the thesis contains the basics of infrared, various types of infrared detectors, materials, and growth techniques of HgCdTe epitaxial films. Materials issues in HgCdTe epitaxial films are discussed for the application of infrared detectors, followed by motivation for nanoindentation studies. A literature survey regarding nanoindentation studies of HgCdTe epitaxial films and CdZnTe single crystal substrate is also included.

The second chapter discusses the liquid phase epitaxy (LPE) process in detail for the growth of HgCdTe epitaxial films used in this study. This chapter also contains the working principle and instrumentation of various characterization tools, which were used in this study. Issues related to the measurement and analysis of load-displacement characteristics have been discussed in detail, including those for cyclic nanoindentation and continuous stiffness measurements.

Nanoindentation studies using spherical indenter geometries are included in the third chapter of the thesis. Both single and multi-cycle studies are reported with varying loading and unloading rates for HgCdTe epitaxial films and CdZnTe single crystal substrates. Open-jaw and hysteresis loop type shapes of unloading-loading curves are explained in light of elastic and anelastic deformation regimes and smooth plastic flow. Sudden discontinuity in load-displacement curves, called pop-in, is observed during first loading and analysed due to the elastoplastic transition of materials. A significant ratcheting effect is also observed during cyclic nanoindentation. Variation of hardness and stiffness with the number of cycles as well as loading and unloading rate is reported here. Cathodoluminescence (CL) studies were performed on CdZnTe and investigated the deformation behavior. Observed tangential and tetrahedral glides are explained in detail. Based on these studies, maximum loads and minimum loading rate during the polishing process of these materials has been suggested.

Nanoindentation studies are also executed for sharp indenters like Berkovich and cube-corner with varying peak loads and loading rates and reported in chapter four. The effect of peak loads and loading rates on mechanical properties is included in this thesis. Detailed deformation studies are performed on indented CdZnTe using photoluminescence (PL) and CL imaging. Residual stress analysis was executed with the help of CL spectra and concluded the presence of tensile residual stresses around the indent. An increase of hardness with loading rate is explained using CL spectra.

The fifth chapter of this thesis includes nanoindentation creep studies with varying loading parameters. Based on indentation data, stress exponent and loading rate sensitivities were extracted for both HgCdTe epitaxial films and CdZnTe single crystal substrates. It was found that strain rate sensitivities of both materials are by and large constant with varying loading parameters.

Keywords: Nanoindentation, HgCdTe, CdZnTe, Berkovich indenter, Spherical indenter, Cathodoluminescence, Pressure assisted phase transformation, Cyclic nanoindentation

सार

सेमीकंडक्टर सामग्री आज के प्रौद्योगिकी युग की रीढ़ हैं। क्रांतिकारी इलेक्ट्रॉनिक उपकरण, जैसे कि माइक्रोप्रोसेसर, फील्ड प्रोग्रामेबल गेट एरेज़ (FPGA), डायनेमिक रैंडम एक्सेस मेमोरी (DRAM), माइक्रो-इलेक्ट्रो मैकेनिकल सिस्टम (MEMS) आधारित सेंसर, ऑप्टोइलेक्ट्रॉनिक डिवाइस, इंफ्रारेड डिटेक्टर, नाइट विजन डिवाइस, मोनोलिथिक माइक्रोवेव इंटीग्रेटेड सर्किट (MMIC) आदि सेमीकंडक्टर सामग्री से बने होते हैं। ये सामग्री क्रिस्टलीय वेफर्स और एपिटैक्सियल हेटरोस्ट्रक्चर के निर्माण के लिए विभिन्न प्रकार की यांत्रिक प्रक्रियाओं से गुजरती हैं, जैसे कि स्लाइसिंग, लैपिंग और मैकेनिकल / केमो-मैकेनिकल पॉलिशिंग। इन प्रक्रियाओं को निष्पादित करते समय, सामग्रियों को विभिन्न यांत्रिक लोडिंग स्थितियों के अधीन किया जाता है, जिससे विरूपण/defects उत्पन्न हो सकते है। इन सामग्रियों के विद्युत गुण इन प्रक्रियाओं के दौरान उत्पन्न विस्तारित सतह/उपसतह दोषों से गंभीर रूप से प्रभावित होते हैं। इसके अलावा अधिकांश अर्धचालक उपकरणों का प्रदर्शन सामग्री में थोक या सतह दोषों की उपस्थिति के साथ खराब हो जाता है। यांत्रिक क्षति के अलावा, लागू तनाव के आवेदन के दौरान एक स्थानीय चरण परिवर्तन भी हो सकता है, जिससे विद्युत और यांत्रिक गुणों में भी परिवर्तन हो सकता है। इसलिए, उच्च-प्रदर्शन अर्धचालक उपकरणों के विकास के लिए यांत्रिक प्रक्रियाओं के कारण यांत्रिक गुणों और विरूपण व्यवहार में परिवर्तन को समझना महत्वपूर्ण है। नैनोइंडेंटेशन एक व्यापक रूप से उपयोग की जाने वाली तकनीक है, विशेष रूप से क्रिस्टलीय सामग्री के लिए, उनके यांत्रिक गुणों को निकालने के लिए, जैसे कठोरता और लोचदार मापांक और सतह/उपसतह विरूपण व्यवहार के अध्ययन के लिए। वर्तमान कार्य में, HgCdTe एपिटैक्सियल फिल्मों और CdZnTe सिंगल क्रिस्टल सबस्ट्रेट्स पर नैनोइंडेंटेशन अध्ययन किया गया है, जो इन्फ्रारेड डिटेक्टरों के विकास के लिए सबसे अधिक मांग वाले अर्धचालक पदार्थों में से एक है।

थीसिस के पहले भाग में इन्फ्रारेड की मूल बातें, विभिन्न प्रकार के इन्फ्रारेड डिटेक्टर, इन्फ्रारेड डिटेक्टर सामग्री और HgCdTe एपिटैक्सियल फिल्मों की विकास तकनीक शामिल हैं। HgCdTe एपिटैक्सियल फिल्मों में सामग्री के मुद्दे पर और इसके बाद इन्फ्रारेड डिटेक्टरों के अनुप्रयोग के लिए चर्चा की गई है। इसके बाद नैनोइंडेंटेशन अध्ययन के लिए प्रेरणा दी गई है। इस भाग में HgCdTe एपिटैक्सियल फिल्मों और CdZnTe सिंगल क्रिस्टल सब्सट्रेट के नैनोइंडेंटेशन अध्ययन

के संबंध में साहित्य सर्वेक्षण शामिल किया गया है। इस अध्ययन में प्रयुक्त HgCdTe एपिटैक्सियल फिल्मों के विकास के लिए लिक्विड फेज एपिटैक्स (एलपीई) प्रक्रिया पर विस्तार से चर्चा की गई है। इस अध्ययन में उपयोग किए गए विभिन्न लक्षण वर्णन उपकरणों के कार्य सिद्धांत को शामिल किया गया है। भार-विस्थापन विशेषताओं के मापन और विश्लेषण से संबंधित मुद्दों पर विस्तार से चर्चा की गई है,

थीसिस के अगले भाग में गोलाकार इंडेंटर ज्यामिति का उपयोग करते हुए नैनोइंडेंटेशन अध्ययन शामिल हैं। एकल/बहु-चक्र दोनों अध्ययनों को HgCdTe एपिएटैक्सियल फिल्मों और CdZnTe सिंगल क्रिस्टल सबस्ट्रेट्स के लिए अलग-अलग लोडिंग / अनलोडिंग दरों के साथ सूचित किया गया है। अनलोडिंग-लोडिंग कर्व्स के ओपन-जॉ और हिस्टैरिसीस लूप प्रकार के आकार को लोचदार / एनालास्टिक विरूपण और चिकनी प्लास्टिक प्रवाह के प्रकाश में समझाया गया है। लोड-विस्थापन वक्रों में अचानक असंतुलन, जिसे पॉप-इन कहा जाता है, पहली लोडिंग के दौरान देखा गया और सामग्री के इलास्टो-प्लास्टिक संक्रमण के कारण विश्लेषण किया गया। चक्रीय नैनोइडनेटेशन के दौरान एक महत्वपूर्ण रैचिंग प्रभाव देखा गया। इस भाग में चक्रों की संख्या के साथ-साथ लोडिंग/अनलोडिंग दर के साथ कठोरता और कठोरता की भिन्नता की सूचना दी गई है। CdZnTe पर कैथोलोलुमिनसेंस (CL) अध्ययन किए गए और विरूपण व्यवहार की जांच की गई। देखे गए स्पशरिखा और चतुष्फलकीय ग्लाइडों के बारे में विस्तार से बताया गया। इन अध्ययनों के आधार पर इन सामग्रियों की पॉलिशिंग प्रक्रिया के दौरान अधिकतम भार और न्यूनतम रहने की दर का सुझाव दिया गया है।

बेरकोविच और क्यूब-कॉर्नर जैसे तेज इंडेंटर्स के लिए अलग-अलग पीक लोड/लोडिंग दरों के साथ नैनोइंडेंटेशन अध्ययन भी रिपोर्ट किए गए है। इस थीसिस में यांत्रिक गुणों पर पीक लोड और लोडिंग दरों का प्रभाव शामिल है। इंडेंटेड CdZnTe पर photoluminescence (PL) और CL इमेजिंग का उपयोग करके विस्तृत विरूपण अध्ययन किया गया। अवशिष्ट तनाव विश्लेषण को सीएल स्पेक्ट्रा की मदद से निष्पादित किया गया और इंडेंट के आसपास तन्य अवशिष्ट तनाव की उपस्थिति का निष्कर्ष निकाला गया। सीएल स्पेक्ट्रा का उपयोग करके लोडिंग दर के साथ कठोरता में वृद्धि को भी समझाया गया है।

अलग-अलग लोडिंग मापदंडों के साथ नैनोइंडेंटेशन creep घटना की जांच की गई। इंडेंटेशन डेटा के आधार पर, HgCdTe एपिटैक्सियल फिल्मों और CdZnTe सिंगल क्रिस्टल सबस्ट्रेट्स

दोनों के लिए स्ट्रेस एक्सपोनेंट / लोडिंग रेट सेंसिटिविटी निकाली गई। यह पाया गया कि दोनों सामग्रियों की तनाव दर संवेदनशीलता अलग-अलग लोडिंग मापदंडों के साथ स्थिर है।कीवर्ड: नैनोइंडेंटेशन, HgCdTe, CdZnTe, बर्कोविच इंडेंटर, गोलाकार इंडेंटर, कैथोडोल्यूमिनेसिसेंस, प्रेशर असिस्टेड फेज ट्रांसफॉर्मेशन, साइक्लिक नैनोइंडेंटेशन

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SYMBOLS

h	Plank's constant
c	Speed of light
k	Boltzmann's constant
$w(\lambda,T)$	Spectral radiant emittance
T	Object's temperature
E_g	Bandgap
GPa	Giga pascal
H	Hardness
P	Load
P_{max}	Peak load
h_c	Contact depth
h_f	Final depth
A_C	Contact area
ϵ	Geometrical factor
E_r	Reduced modulus
ω	Angular frequency of the oscillation
δ	Phase angle
ΔF	Force amplitude
K	Damping coefficient
ΔP	Load variance
R_i	Indenter radius
p	Pressure
$\dot{\varepsilon}_p$	Plastic strain
ρ_m	mobile dislocation density
B	Burgers vector
v	Average velocity of dislocations
σ_y	Yield stress
σ	Applied stress
$\dot{\varepsilon}$	Strain rate
m	Strain rate sensitivity
n	Stress exponent
S	Stiffness
h_{max}	Maximum penetration depth

ABBREVIATIONS

IR	Infrared
SWIR	Short wave infrared
MWIR	Medium wave infrared
LWIR	Long wave infrared
ROIC	Readout integrated circuits
HgCdTe	Mercury cadmium telluride
CdZnTe	Cadmium zinc telluride
LPE	Liquid phase epitaxy
VDLPE	Vertical dipping liquid phase epitaxy
CMP	Chemo-mechanical polishing
P-h curves	Load-displacement curves
CL	Cathodoluminescence
PL	Photoluminescence
L/UR	Loading/unloading rate
FTIR	Fourier transform infrared spectroscope
HRXRD	High resolution X-ray diffraction
CSM	Continuous stiffness measurement
QCSM	Quasi Continuous stiffness measurement
ISE	Indentation size effect
FWHM	Full width at half maxima
SRS	Strain rate sensitivity
SEM	Scanning electron microscope
AFM	Atomic force microscope
MBE	Molecular beam epitaxy
MOCVD	Metal-organic chemical vapor deposition