

# **NANOINDENTATION STUDIES OF HgCdTe EPITAXIAL FILMS AND CdZnTe SINGLE CRYSTAL SUBSTRATES**

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**NANOINDENTATION STUDIES OF HgCdTe EPITAXIAL FILMS  
AND CdZnTe SINGLE CRYSTAL SUBSTRATES**

**by**

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Submitted

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

to the



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## **CERTIFICATE**

This is to certify that the thesis entitled “Nanoindentation studies of HgCdTe epitaxial 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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(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, इन्फ्रारेड 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 हैं। 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 सबस्ट्रेट 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 एपिटैक्सियल फिल्मों और CdZnTe सिंगल क्रिस्टल सबस्ट्रेट्स के लिए अलग-अलग लोडिंग / अनलोडिंग दरों के साथ सूचित किया गया है। अनलोडिंग-लोडिंग कर्ब्स के ओपन-जॉ और हिस्टैरिसिस 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 नैनोइंडेंटेशन 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 स्पर्शरिखा 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 हैं। 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

# TABLE OF CONTENTS

<b>CERTIFICATE</b> .....	<b>i</b>
<b>ACKNOWLEDGEMENTS</b> .....	<b>ii</b>
<b>ABSTRACT</b> .....	<b>v</b>
<b>TABLE OF CONTENTS</b> .....	<b>xi</b>
<b>LIST OF FIGURES</b> .....	<b>xv</b>
<b>LIST OF TABLES</b> .....	<b>xxi</b>
<b>SYMBOLS</b> .....	<b>xxii</b>
<b>ABBREVIATIONS</b> .....	<b>xxiii</b>
<b>1 INTRODUCTION</b> .....	<b>1</b>
1.1 Preamble.....	1
1.2 Infrared detection technology.....	3
1.2.1 Infrared Radiation .....	3
1.2.2 Applications of infrared radiations .....	5
1.2.2.1 Military Applications .....	6
1.2.2.2 Research applications .....	7
1.2.2.3 Civilian applications.....	8
1.2.2.4 Industrial applications .....	8
1.2.2.5 Medical application .....	9
1.2.3 Infrared imaging system .....	9
1.2.4 Types of IR detectors .....	11
1.2.4.1 Thermal detectors .....	11
1.2.4.2 Photon detectors .....	12
1.2.5 Materials for IR intrinsic detectors .....	14
1.2.5.1 Hg <sub>1-x</sub> Cd <sub>x</sub> Te epitaxial films.....	15
1.2.5.2 Substrates for HgCdTe epitaxial films .....	17
1.2.5.3 Growth of HgCdTe epitaxial films.....	19
1.2.5.3.1 Liquid phase epitaxy (LPE).....	19
1.2.5.3.2 Molecular beam epitaxy (MBE) .....	20
1.2.5.3.3 Metal-organic chemical vapour deposition (MOCVD).....	21
1.2.5.4 Materials Issues in HgCdTe films for IR sensor applications.....	22
1.3 Motivation .....	24
1.4 Nanoindentation .....	25
1.5 Literature Survey.....	25

1.5.1	Pop-in/Pop-out Phenomenon .....	26
1.5.2	Nano-mechanical properties and deformation behaviour .....	29
1.5.3	Cathodoluminescence studies .....	36
1.6	Objectives and Overview of the Thesis.....	38

## **2 EXPERIMENTAL TECHNIQUES : GROWTH AND CHARACTERIZATION .....**

<b>2</b>	<b>EXPERIMENTAL TECHNIQUES : GROWTH AND CHARACTERIZATION .....</b>	<b>41</b>
2.1	Introduction .....	41
2.2	Epitaxial Growth of HgCdTe .....	42
2.2.1	VDLPE System.....	42
2.2.2	Epitaxial growth.....	43
2.3	Fourier transform infrared spectroscopy (FTIR).....	47
2.3.1	Instrument design.....	47
2.3.2	Working principle .....	47
2.4	Scanning electron microscopy (SEM).....	49
2.4.1	Instrument design.....	50
2.4.2	Working principle .....	50
2.5	Atomic force microscope (AFM).....	51
2.5.1	Instrument design.....	52
2.5.2	Working principle .....	53
2.6	Cathodoluminescence (CL) microscopy .....	54
2.7	Photoluminescence spectroscopy (PL).....	57
2.7.1	Instrument design.....	58
2.7.2	Working principle .....	58
2.8	High resolution X- Ray Diffraction (HRXRD).....	59
2.9	Indentation.....	61
2.9.1	Historical background .....	61
2.9.2	Nanoindentation .....	62
2.9.3	Nanoindentation Experiments and Characteristics .....	64
2.9.3.1	Load-displacement Characteristics .....	65
2.9.3.2	Cyclic nanoindentation.....	69
2.9.3.3	Continuous Stiffness measurements.....	70
2.9.3.4	Indentation size effect .....	72
2.10	Conclusion.....	74

<b>3</b>	<b>DEFORMATION BEHAVIOUR STUDIES WITH SPHERICAL INDENTER</b> .....	76
3.1	Introduction .....	76
3.2	HgCdTe Epitaxial Films.....	77
3.2.1	Experimental details.....	77
3.2.2	Results and discussions.....	81
3.2.2.1	Single cycle indentation .....	81
3.2.2.2	Cyclic nanoindentation.....	85
3.2.2.2.1	Load-Displacement ( <i>P-h</i> ) Characteristics .....	85
3.2.2.2.2	Depth of indentation with number of cycles .....	94
3.2.2.2.3	Extraction of Mechanical Properties .....	95
3.3	CdZnTe substrates.....	96
3.3.1	Experimental details.....	96
3.3.2	Results and discussions.....	99
3.3.2.1	Single cycle indentation .....	99
3.3.2.1.1	Nano mechanics with varying peak loads .....	99
3.3.2.1.2	Nano mechanics with varying loading rate .....	100
3.3.2.2	Cyclic nanoindentation.....	101
3.3.2.2.1	Load-displacement curves .....	101
3.3.2.2.2	Extraction of mechanical properties .....	105
3.3.2.2.3	Cathodoluminescence (CL) imaging .....	105
3.4	Conclusions .....	110
3.4.1	HgCdTe epitaxial films.....	110
3.4.2	CdZnTe single crystal substrate.....	111
<b>4</b>	<b>DEFORMATION BEHAVIOUR STUDIES WITH SHARP INDENTERS</b> .....	113
4.1	Introduction .....	113
4.2	Cube Corner Indentation .....	114
4.2.1	Experimental .....	114
4.2.2	Results and discussions.....	115
4.2.2.1	Nano mechanics with varying peak load and loading rates .....	115
4.2.2.2	Photoluminescence (PL) studies .....	118

4.2.2.3	Cathodoluminescence (CL) studies.....	119
4.2.2.4	Analysis of residual stresses.....	124
4.3	Berkovich Indenter.....	129
4.3.1	Experimental.....	129
4.3.2	Results and discussions.....	130
4.3.2.1	Nano-mechanics with varying loads and loading rates.....	130
4.3.2.2	Cathodoluminescence (CL) studies.....	132
4.3.2.3	Anisotropic studies.....	133
4.4	HgCdTe epitaxial films.....	134
4.4.1	Experimental.....	134
4.4.2	Results and discussions.....	134
4.4.3	Cyclic nanoindentation.....	137
4.5	Conclusion.....	138
<b>5</b>	<b>CREEP AND STRAIN RATE SENSITIVITY STUDIES.....</b>	<b>140</b>
5.1	Introduction.....	140
5.2	Basics of nanoindentation creep.....	141
5.3	HgCdTe epitaxial films.....	143
5.3.1	Experimental.....	143
5.3.2	Results and discussions.....	145
5.4	CdZnTe single crystal substrates.....	148
5.4.1	Experimental.....	148
5.4.2	Results and discussions.....	150
5.5	Conclusion.....	155
<b>6</b>	<b>CONCLUSIONS AND FUTURE WORK.....</b>	<b>156</b>
6.1	Conclusions.....	156
6.2	Significance and importance of work.....	158
6.3	Suggestions for future work.....	159
	<b>REFERENCES.....</b>	<b>160</b>
	<b>PUBLICATIONS BASED ON PRESENT WORK.....</b>	<b>188</b>
	<b>BIO-DATA.....</b>	<b>192</b>

## LIST OF FIGURES

Figure 1.1: Variation of spectral radiance with wavelength demonstrating Plank’s law, Riedl et al. [8] .....	3
Figure 1.2: Infrared spectral band .....	4
Figure 1.3: Images of border areas using (a) visible light (b) IR of same scene showing hidden armymen, IR images using (c) SWIR band (d) using MWIR band [10] ...	6
Figure 1.4: Astronomical images of gas in the Eagle Nebula by Hubble Space Telescope (a) visible radiation (b) infrared radiation [12] .....	7
Figure 1.5: A typical site show in all light conditions with the visible and infrared radiations [13] .....	8
Figure 1.6: Thermal scanning of brain showing brain tumor [15].....	9
Figure 1.7: Schematic of Infrared imaging system .....	10
Figure 1.8: Schematic showing working principle of IR detector .....	12
Figure 1.9: Change of bandgap of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ with $x$ .....	16
Figure 1.10: Variation of lattice parameters of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ and $\text{Cd}_{1-z}\text{Zn}_z\text{Te}$ with varying composition ( $x$ and $z$ ), Capper et al. [29] .....	17
Figure 1.11: Crystal structure of (a) $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ (b) $\text{Cd}_{1-z}\text{Zn}_z\text{Te}$ .....	18
Figure 1.12: Vertical dipping liquid phase epitaxy process for growing HgCdTe epitaxial films, used in this thesis .....	19
Figure 1.13: A typical MBE system for growth of HgCdTe epitaxial films .....	20
Figure 1.14: A typical photograph of MOCVD system [37] .....	21
Figure 1.15: Microscopic images of (a) as-received and polished CdZnTe substrate (b) as-grown and polished HgCdTe epitaxial films .....	22
Figure 1.16: Schematic of dislocation half loops generated during polishing/CMP ...	23
Figure 1.17: Load-displacement curves of (a) MBE grown sample (b) LPE grown sample, Sizov et al. [82].....	26
Figure 1.18: Pop-in phenomenon observed using Berkovich indenter, Martyniuk et al. [85] .....	28
Figure 1.19: Variation of elastic modulus and hardness with the applied load on (111) CdZnTe, Zhang et al. [72] .....	29

Figure 1.20: AFM morphology of indents on (111) Cd <sub>0.96</sub> Zn <sub>0.04</sub> Te with varying peak loads of (a) 0.5, 1 and 2 mN (b) 2, 4, 6 and 8 mN (c) 12 mN (d) 1 mN, Zhang et al. [72].....	29
Figure 1.21: Variation of hardness and elastic modulus of (a) (100) oriented (b) (111) oriented crystal, Li et al. [74].....	30
Figure 1.22: Variation of hardness with different orientations of (110) and (111) plane, Li et al. [87] .....	31
Figure 1.23: Surface topography after grinding (a) (110) (b) CdZnTe (111), Li et al. [87] .....	32
Figure 1.24 : Load-displacement curves with varying peak loads, Martyniuk et al. [85] .....	33
Figure 1.25 : Variation of elastic modulus and hardness as a function of contact depth, Martyniuk et al. [85] .....	33
Figure 1.26: Variation of $h_f/h_{max}$ with contact depth Martyniuk et al. [85] .....	34
Figure 1.27 : CL image of indented surface of CdTe (a) (111) Cd (b) (111) Te face, Leipner et al. [94].....	36
Figure 2.1: Schematic of VDLPE system used for growing HgCdTe films with 4 temperature zones marked as Z1, Z2, Z3 and Z <sub>Hg</sub> .....	42
Figure 2.2: Ternary phase diagram for Te rich growth of HgCdTe films, Brebrick et al.[98] .....	44
Figure 2.3: Process flow chart for the development of HgCdTe epitaxial films .....	44
Figure 2.4: Growth recipe for HgCdTe epitaxial films.....	45
Figure 2.5 : Run to run compositional uniformity achieved in upgraded growth reactor .....	46
Figure 2.6: Schematic of the working principle of FTIR.....	48
Figure 2.7: Typical FTIR spectra of HgCdTe epitaxial film .....	49
Figure 2.8: Schematic of AFM machine .....	52
Figure 2.9: Interaction of high energy electron beam with sample and possible outputs.....	55
Figure 2.10: Schematic of intrinsic and extrinsic CL, Yacobi et al.. [119] .....	56
Figure 2.11: HRXRD rocking curve setup .....	59
Figure 2.12: A typical rocking curve of CdZnTe (111) crystal .....	60

Figure 2.13: Schematic of typical nanoindenter instruments [(a) UMIS and (b) Triboindenter Manuals] .....	63
Figure 2.14: Schematic load-displacement curves obtained using a nanoindentation cycle .....	65
Figure 2.15: Illustration of pop-in and pop-out events in load-displacement curve ....	66
Figure 2.16: Schematic of indentation on a surface.....	67
Figure 2.17: Experimental $P-h$ curve of cyclic nanoindentation (a) Open jaw type shape (b) Hysteresis loop type shape, Saraswati et al. [124] .....	69
Figure 2.18: Schematic of CSM curve.....	70
Figure 2.19: Applied forces pattern during QCSM, ASMEC manual.....	71
Figure 2.20: Indentation Size Effect on hardness measurements schematic .....	72
Figure 3.1: Schematic of load-time graph showing $\Delta P$ .....	78
Figure 3.2: (a) AFM image of chemo-mechanical polished HgCdTe epitaxial layer. (b) FTIR spectrum of a typical HgCdTe epitaxial film and (c) HRXRD rocking curve (333 diffraction) scan on a typical CMP polished HgCdTe epitaxial layer over an area of $15 \times 15 \text{ mm}^2$ .....	80
Figure 3.3: (a) Load-displacement curves of HgCdTe epitaxial film with varying loading rate (b) Hertzian fit during loading .....	82
Figure 3.4: Typical SEM images of spherical indents after nanoindentation with L/UR of $4 \text{ mNs}^{-1}$ .....	83
Figure 3.5: Load-displacement curve of cyclic indentation of HgCdTe films for 100 cycles .....	85
Figure 3.6: Load-time-displacement curves with loading and unloading rates of (a) 0.5, (b) 1 and (c) $4 \text{ mNs}^{-1}$ and load-displacement curves with loading and unloading rates of (d) 0.5, (e) 1 and (f) $4 \text{ mNs}^{-1}$ .....	86
Figure 3.7: Schematic of anelastic strain/recovery due to dislocations .....	87
Figure 3.8: Schematic of (a) open jaw type (b) hysteresis type loop in a load vs displacement ( $P-h$ ) characteristic for cyclic nanoindentation.....	88
Figure 3.9: Magnified view of cyclic load displacement curves for L/UR of (a) 1 (b) $4 \text{ mNs}^{-1}$ .....	90
Figure 3.10: Exponential behaviour of anelastic recovery region of the first unloading curve for L/UR of $4 \text{ mNs}^{-1}$ .....	91

Figure 3.11: Magnified view of cyclic load-displacement curves for (a) L/UR of 0.5 mNs <sup>-1</sup> showing serrations (b) L/UR of 4 mNs <sup>-1</sup> showing absence of serrations.....	92
Figure 3.12: Incremental cyclic indentation from 3 mN to 10 mN peak load .....	93
Figure 3.13: (a) Variation of indentation depth with number of cycles (b) Change of indentation depth per cycle (dh/dN) with number of cycles.....	94
Figure 3.14: (a) Incremental penetration depth per cycle with number of cycle (2) variation of $\beta$ with load variance .....	95
Figure 3.15: (a) AFM image of polished (111) B CdZnTe single crystal substrate (b) HRXRD graph of HgCdTe epitaxial layers grown on CdZnTe substrate .....	98
Figure 3.16: Load-displacement curve of varying peak loads.....	99
Figure 3.17: AFM image of spherical indent using loading rate of 1 mNs <sup>-1</sup> and peak load (a) 10 mN (b) 50 mN .....	100
Figure 3.18: Load-displacement curves with varying loading rates .....	101
Figure 3.19: Load-time-displacement (a-c) and load-displacement curves (d-f) for L/UR of 0.5, 5 and 25 mNs <sup>-1</sup> respectively .....	102
Figure 3.20: SEM image of indent on CdZnTe (111) substrate using peak load of 50 mN and loading rate 0.5 mNs <sup>-1</sup> .....	103
Figure 3.21: Hertzian fit curve during first loading of CdZnTe (111).....	104
Figure 3.22: CL image of the single cycle indentation at a peak load of 50 mN .....	106
Figure 3.23: (a) Internal apex and (b) External apex tetrahedron of the {111} planes of zinc blende structure .....	106
Figure 3.24: Dislocation loops emission at the beginning of deformation .....	107
Figure 3.25: CL images with peak load of (a) 10 mN (b) 50 mN.....	108
Figure 4.1: Illustration of indenter geometries (a) Cube corner (b) Berkovich, Agilent technologies .....	114
Figure 4.2: Load-displacement curves with varying peak loads, using cube corner indenter .....	115
Figure 4.3: SEM images of indents with cube corner indenter using peak loads of (a) 10 (b) 20 (c) 50 and (d) 100 mN.....	116
Figure 4.4: Variation of Hardness with peak load .....	117
Figure 4.5: (a) Load-displacement curve with varying loading rates (b) Variation of indentation hardness with contact depth.....	117

Figure 4.6: P L mapping image around indented regions for 50 mN peak load using cube corner indenter .....	118
Figure 4.7: CL images of cube corner indents formed by peak loads of (a) 10 (b) 20 (c) 50 (d) 100 mN.....	120
Figure 4.8: Slip band formation after nanoindentation (a) SEM image using cube corner indenter at 100 mN peak load, (b) AFM image of spherical indenter at 50 mN load (c) CL of indent of cube corner at 100 mN load.....	121
Figure 4.9: Schematic of material flow around the indent after nanoindentation ....	122
Figure 4.10: CL images of indents using loading rate of (a) 1 (b) 2 (c) 5 and (d) 10 mNs <sup>-1</sup> .....	123
Figure 4.11: Load-displacement curves for different loading rates using cube corner indenter .....	125
Figure 4.12: SEM images of indent formed using cube corner indenter with loading rates of (a) 1 and (b) 5 mNs <sup>-1</sup> .....	126
Figure 4.13: CL spectra on and around indent formed with a loading rates of (a) 1 and (b) 5 mNs <sup>-1</sup> .....	127
Figure 4.14: Load-displacement curves of (a) varying loads (b) varying loading rates, using Berkovich indenter .....	130
Figure 4.15: Variation of hardness with varying loading rates.....	131
Figure 4.16: CL images of indents using Berkovich indenter at a peak load of (a) 10 (b) and 50 mN .....	132
Figure 4.17: Variation of indentation hardness and elastic modulus along different angles on the surface of CdZnTe .....	133
Figure 4.18: Load-displacement curve of HgCdTe epitaxial films .....	134
Figure 4.19: Magnified view of load-displacement curve shown in Fig. 4.18 .....	134
Figure 4.20: SEM images of indents using loading rates of (a) 0.5 and (b) 2 mNs <sup>-1</sup> , showing higher deformation laterally for 0.5 mNs <sup>-1</sup> .....	135
Figure 4.21: Variation of hardness with loading rate of HgCdTe films .....	136
Figure 4.22: Load-displacement curves for cyclic nanoindentation of HgCdTe epitaxial films with Berkovich indenter .....	137
Figure 5.1: Illustration of strain rate sensitivity of the material (a) varying loading rate (b) descent rate of indenter for strain rate insensitive material (b) descent rate of indenter for strain rate sensitive materials, mayo et al. [190].....	143

Figure 5.2: Thermal drift correction during creep experiment .....	144
Figure 5.3.: Load-displacement curves using different loading rates of 0.5, 1, and 5 mN s <sup>-1</sup> .....	145
Figure 5.4 : Creep displacement with time for HgCdTe epitaxial films using different loading rates .....	146
Figure 5.5: Strain rates variations with displacement with varying loading rates for HgCdTe epitaxial films.....	145
Figure 5.6: Creep fitting curves with experimental data for HgCdTe using loading rates of (a) 0.5 (b) 1 and (c) 5 mNs <sup>-1</sup> .....	147
Figure 5.7: Curve between ln (strain rate) and ln (stress) for extraction of stress exponent of HgCdTe for loading rates of (a) 0.5 (b) 1 and (c) of 5 mNs <sup>-1</sup> .....	148
Figure 5.8: Creep displacement with time for varying loading rates for CdZnTe.....	149
Figure 5.9: Creep data and fitting curve for CdZnTe using loading/unloading rate of for (a) 0.5 (b) 1 (c) 5 mNs <sup>-1</sup> .....	150
Figure 5.10: Curve between ln (strain rate) and ln (stress) for extraction of stress exponent of CdZnTe for loading rates (a) 0.5 (b) 1 and 5 mNs <sup>-1</sup> .....	151
Figure 5.11: Creep with time for CdZnTe using varying peak loads of 10, 50 and 100 mN .....	152
Figure 5.12: Creep data and fitting curve for CdZnTe using peak loads of (a) 10 (b) 50 and (c) 100 mN .....	152
Figure 5.13: Curve between ln (strain rate) and ln (stress) for extraction of stress exponent for CdZnTe using peak load of (a) 10 (b) 50 and (c) 100 mN .....	153
Figure 5.14: Creep displacement with varying holding time at a peak load of 10 mN .....	154

## LIST OF TABLES

Table 2-1 Area function ( $F(h_c)$ ) and geometrical factor ( $\epsilon$ ) for different Indenter geometries [42].	68
Table 3-1: The indentation pressure estimated at the onset of pop-in observed in Fig. 3.5 (b).	83
Table 3-2: Stiffness extracted from load-displacement curves (Fig. 3.3) and corresponding values of indentation hardness	96
Table 3-3. The pressure calculated at the onset of pop-in observed in Figs. 3.19 (d- f).	104
Table 3-4: Stiffness values and their corresponding hardness values extracted from load-displacement curves (Fig. 3.19)	105
Table 4-1: Comparison of hardness, elastic modulus and pop-in load for varying indenter geometries	131

## 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
$\epsilon$	Geometrical factor
$E_r$	Reduced modulus
$\omega$	Angular frequency of the oscillation
$\delta$	Phase angle
$\Delta F$	Force amplitude
$K$	Damping coefficient
$\Delta P$	Load variance
$R_i$	Indenter radius
$p$	Pressure
$\dot{\epsilon}_p$	Plastic strain
$\rho_m$	mobile dislocation density
B	Burgers vector
$v$	Average velocity of dislocations
$\sigma_y$	Yield stress
$\sigma$	Applied stress
$\dot{\epsilon}$	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
<i>P-h</i> curves	Load-displacement curves
CL	Cathodoluminescence
PL	Photoluminescence
L/UR	Loading/unloading rate
FTIR	Fourier transform infrared spectroscopy
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