

## Cathodoluminescence Studies of Nanoindented CdZnTe Single-Crystal Substrates for Analysis of Residual Stresses and Deformation Behaviour

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### ABSTRACT

Nanoindentation-induced residual stresses were analysed on (111) Te face CdZnTe single-crystal substrates in this study. CdZnTe substrates were subjected to nanoindentation using cube corner indenter geometry with a peak load of 10 mN. Loading rates of 1 mN/s and 5 mN/s were used in the experiments, with a holding time of 10 s at peak load. Residual stresses on the indented region were analysed from load-displacement curves and explained using dislocation generation and elastic recovery mechanisms. Residual stresses were found to be of compressive type, just on the indented surface. The slip lines along the slip directions of this material were clearly visible in the FE-SEM images of the indents. Indents and surrounding surfaces were characterized using the Cathodoluminescence (CL) technique. CL mapping of the indented surface revealed the dislocation generation and their propagation behaviour just beneath the indenter as well as in the surrounding surfaces. The dislocations act as non-radiative recombination centres and quench the CL intensity locally. Dark lines were explained as the presence of dislocations in the material. CL mapping analysis shows that both the rosette glide and tetrahedral glide of dislocations are the primary deformation mechanisms present in CdZnTe. A rosette structure was observed in the CL mapping. CL spectra at 300 K of un-deformed CdZnTe show a peak at 810 nm wavelength, which corresponds to near-band-edge emission. After indentation, the CL spectra show the peak intensity at 814 nm and 823 nm wavelengths at the edge of the indents created with a loading rate of 1 mN/s and 5 mN/s, respectively. These peak shifts from 810 nm were attributed to the tensile residual stresses present in the indented material.

**Keywords:** Nanoindentation; CdZnTe; cube corner indenter; Cathodoluminescence; Infrared

### 1. INTRODUCTION

Single-crystal (111) Te face CdZnTe substrates are used for the growth of HgCdTe epitaxial films due to nearly perfect lattice matching. These HgCdTe films grown on CdZnTe substrates are of strategic importance and used for infrared detectors, IR seekers for missile applications, night vision devices for surveillance and radiation detectors<sup>1</sup>. These detectors require very high efficiency because of its high-end strategic applications. CdZnTe is the base material which requires a smooth surface finish, low dislocation density  $<10^5/\text{cm}^2$ , minimal surface/subsurface damages and impurities free surface to get high-quality epitaxial layers on it<sup>2</sup>. Any damages or defects on the surface will propagate into the epitaxial layers. These defects adversely affect the efficiency of devices. CdZnTe single-crystal substrates require to undergo various mechanical processes like cutting, lapping and polishing before it is ready for growth. These processes may introduce dislocations in the material which will lead to degrading the device quality. To optimize these mechanical processes, a good understanding of dislocation generation and propagation behaviour is required. Also, it requires to have a

good knowledge of residual stresses created in the material due to these mechanical processes, which may also affect the quality of the device at the end<sup>3</sup>. Nanoindentation is one of the technique to find out not only the mechanical properties like elastic modulus, hardness and contact stiffness of the material but also deformation behaviour. Cathodoluminescence (CL) is the popular characterization tool to get the information of dislocation generation and propagation into the material. Dislocations actually act as the recombination centres in the material and quench the luminescence locally<sup>4</sup>. Due to this, the generated dislocations can be seen in the CL imaging as dark lines/spots. Few works have been done on CdZnTe material to see the dislocations and their propagation path by micro/nanoindentation. Leipner<sup>5</sup>, *et al.* have shown the detailed analysis of dislocation rosette patterns on (111), (110) and (001) surface of CdTe by CL. Glide dislocations have been studied by Lu<sup>6</sup>, *et al.* of CdTe. Riviera<sup>7</sup>, *et al.* have studied the dislocation propagation behaviour of CdTe spatially as well as across the depth. Schreiber<sup>8</sup>, *et al.* have explained about the recognition of A(g) and B(g) type of dislocations in CdTe material and rosette pattern was explained by a glide prism model. Though these studies are very useful to find out the mechanical deformation behaviour of the material, none of the studies has explained about the residual stresses developed in this material

after micro/nano indentation. A few studies on other materials have discussed this residual stress. Lee<sup>9</sup>, *et al.* has reported stress relaxation during plastic deformation by indentation. Yacobi<sup>10</sup>, *et al.* has shown the stress variations due to micro-cracks in thick GaAs epitaxial layers grown on Si. Claudia<sup>11</sup>, *et al.* have measured the residual stresses of strained alumina single-crystal by CL imaging and spectroscopy. Variation of the CL spectrum with stress was reported by Alessandro<sup>12</sup>, *et al.* on N-doped 3C-SiC. Residual stresses were investigated by Wen<sup>13</sup>, *et al.* Thus CL is a powerful tool not only for finding the dislocation generation and propagation but also to analysing the residual stresses present in the materials.

In the present study, nanoindentation-induced residual stresses were investigated using cube corner indenter. CL spectra and CL mapping have been used in this study to examine the residual stresses in the material. This study is done first time on CdZnTe single-crystal substrate to the best of our knowledge.

## 2. EXPERIMENTAL DETAILS

The (111) Te face CdZnTe single-crystal substrate (25 mm X 25 mm, 1200  $\mu\text{m}$  thickness) was used for the nanoindentation studies. Prior to nanoindentation, the substrate was powder polished by 60 nm  $\text{Al}_2\text{O}_3$  powder followed by chemo-mechanical polishing with iodine-based solution<sup>14</sup>. The average surface roughness was found to be 3 nm after chemo-mechanical polishing using Agilent Technologies 6500 AFM as shown in Fig. 1.

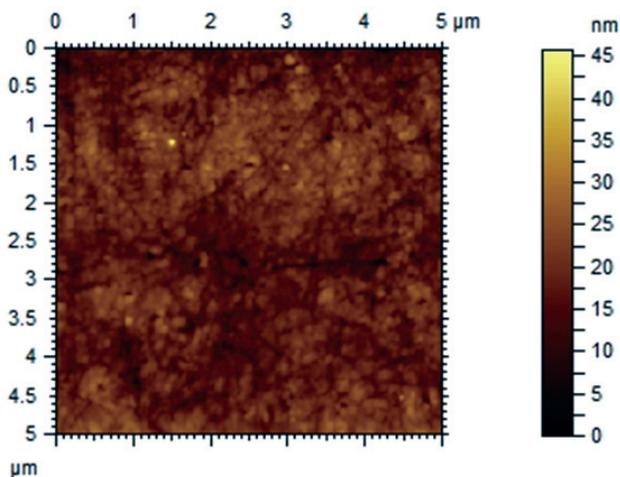


Figure 1. AFM image of polished (111) CdZnTe substrate.

HRXRD measurements was performed on on the surface of the substrate material using a PANalytical make Xpert PRO MRD system with  $\text{CuK}_\alpha$  radiation and 111 diffraction. Figure 2 shows the HRXRD rocking curve of CdZnTe substrate and shows that it is perfectly single-crystalline material having  $\text{FWHM} < 25$  arc-sec.

The nano-indentation experiments were performed using a cube corner indenter with ASMEC, Germany make Universal Nanomechanical Tester. Peak load of 10mN was used for nano-indentation. Loading rates of 1 mN/s and 5 mN/s were used during experimentation with a holding time of 10 s at peak load. Unloading rate of 1 mN/s was used for all experiments.

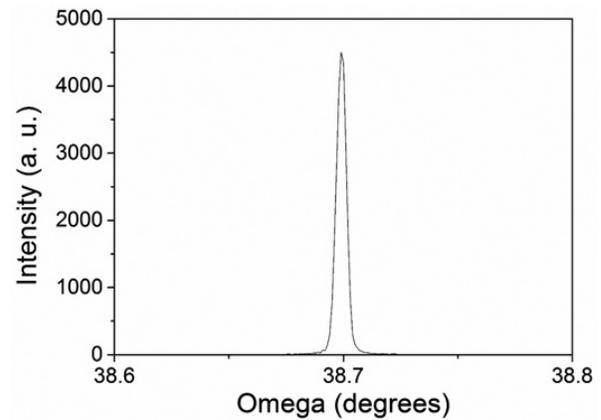


Figure 2. HRXRD rocking curve of (111) CdZnTe single-crystal.

Total 10 indents were made for each loading rate separated by a distance of 40  $\mu\text{m}$ . The average data for each loading rate is reported here. Hardness and elastic modulus have been calculated from the load-displacement curve using the Oliver and Pharr method<sup>15</sup>.

Cathodoluminescence (CL) measurements were carried out at 300 K using an optical detection system (MonoCL3, Gatan) installed with Field emission scanning electron microscopy (Supra55, Zeiss).

## 3. RESULTS AND DISCUSSIONS

Load-displacement curves for the loading rate of 1 mN/s and 5 mN/s has been shown in Fig. 3. The peak load of 10 mN was used during both loading rate experiments. It is evident from the figure that it contains mainly three parts. One is loading at a specified loading rate till a peak load of 10 mN is achieved, second is the holding for 10 s at a peak load of 10 mN, and third is the unloading part.

It is observed from Fig. 3 that penetration depth (deformation) of indenter into the material for both loading rates are same up to a peak load of 0.61 mN, and after crossing this load, the penetration depth is found more for the loading rate of 1 mN/s, till peak load is reached. Hence, a lower plastic work is observed during the loading rate of 1 mN/s. Penetration depths, as the load reached to its peak value (10 mN), are 1699 nm and 1672 nm for the loading rate of 1 mN/s and 5 mN/s, respectively.

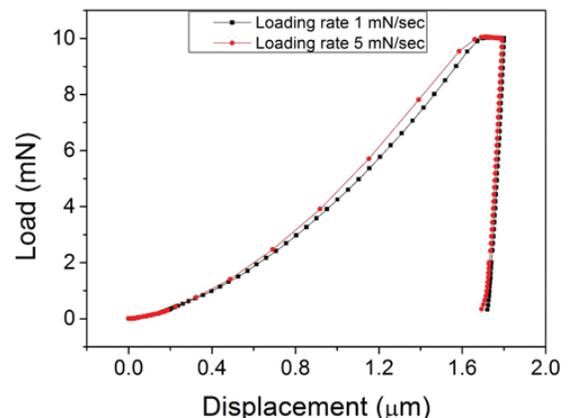


Figure 3. Load-displacement curves for different loading rates.

Orawan's relationship states that the dislocation density of mobile dislocations increases with increasing strain rates as per Eqn. (1)

$$\varepsilon'_p = ab\rho_m v \quad (1)$$

where  $\rho_m$  is the mobile dislocation density,  $b$  is Berger's vector,  $v$  is an average velocity of dislocations and  $\varepsilon'_p$  is plastic strain rate. With increased dislocation density, dislocations start piling up on each other. This piling effect leads to low deformation and the generation of residual stress in the material. Bull<sup>16</sup>, *et al.* and Khan<sup>17</sup>, *et al.* has also explained a similar mechanism for the generation of residual stresses in the material. The elastic recovery during unloading has been studied in both the cases and found that recovery is 5.58 % of total indentation depth (1.79  $\mu\text{m}$ ) for the loading rate of 5 mN/s and 4.4 % of total indentation depth (1.80  $\mu\text{m}$ ) for the loading rate of 1 mN/s. The more elastic recovery is observed due to higher residual stresses present in the material with the increase of loading rates. Xu<sup>18</sup>, *et al.* has performed finite element simulations for determination of residual stresses, generated in the material during nanoindentation. Authors have reported that the ratio of elastic recovery to maximum penetration depth ( $h_e/h_{max}$ ) varies linearly with the ratio of residual stress to yield stress ( $\sigma_r/\sigma_y$ ). The ratio of elastic recovery to maximum penetration depth ( $h_e/h_{max}$ ), increases with the increase of residual compressive stresses.  $h_e/h_{max}$ , calculated from load-displacement curves and found 0.00558 and 0.004425 for loading rate of 5 mN/s and 1 mN/s, respectively. Yield stress ( $\sigma_y$ ) was kept constant during experimentation. Compressive residual stresses found to be increased with increase of loading rate on the indent area, as the ratio of  $h_e/h_{max}$  increases. Hence, this validate our findings of presence of residual stresses from load-displacement curves

No sudden discontinuity during loading (pop-in) and during unloading (pop-out) has been observed in these load-displacement curves. The calculated values of elastic modulus and hardness of loading rate of 1 mN/s (5 mN/s) and were found  $39.7 \pm 2$  GPa ( $39.2 \pm 1.9$  GPa) and  $0.99 \pm 0.03$  GPa ( $1 \pm 0.02$  GPa), respectively. The values of elastic modulus and hardness are matching for both loading rate of 1 mN/s and 5 mN/s and not showing the dependency of loading rates.

Figure 4(a) and 4(b) shows SEM images of indents using the loading rate of 1 and 5 mN/s, respectively. No cracks

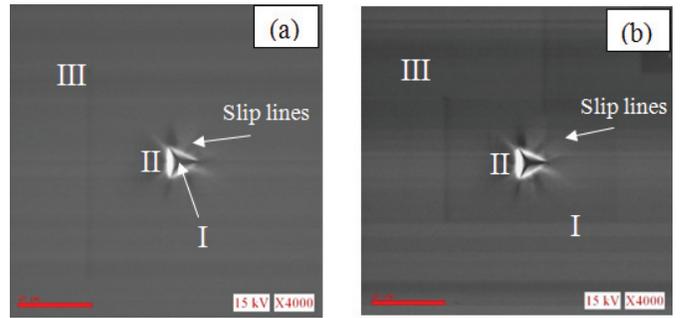


Figure 4. SEM images of indent formed with a loading rate of (a) 1 mN/s (b) 5 mN/s.

around indents were observed in SEM images. Slip lines can be clearly seen in these images. These slip lines are along to slip directions of this material, which are corresponding to the directions of  $[\bar{1}01]$ ,  $[\bar{1}10]$ ,  $[01\bar{1}]$ ,  $[10\bar{1}]$ ,  $[1\bar{1}0]$  and  $[0\bar{1}1]$ . More dense slip lines are observed in the SEM image of indent used by loading rate of 5 mN/s than loading rate of 1mN/s. This observation confirms more dislocation density present in the indented material with higher loading rate.

Figures 5(a) and 5(b) shows the CL spectra of CdZnTe taken at 3 different positions as shown in Fig. 4 for the loading rate of 1 mN/s and 5 mN/s, respectively. Position III is far from indent which is an unreformed region. Position II corresponds to the near edge of indent and position I is taken at the centre of indent.

CL spectra show a peak at 810 nm at 300 K for undeformed CdZnTe, which is due to band edge emission. No peak is seen at the centre of indent, using both loading rates of 1 mN/s and 5 mN/s. Dislocation density at the centre of the indent is high enough to quench nearly all CL emission, which can also be seen as a dark area at the indent region in CL images. CL spectra at the edge of indent show the band edge peak with lower intensity compared with the un-deformed region. This is because of dislocations have propagated beyond the indent and acted as a source of non-radiative recombination centre. It is noted that CL peak intensity is lower in case of higher loading rate of 5 mN/s compared to 1 mN/s. Jian<sup>19</sup>, *et al.* has reported that the CL intensity of the deformed region

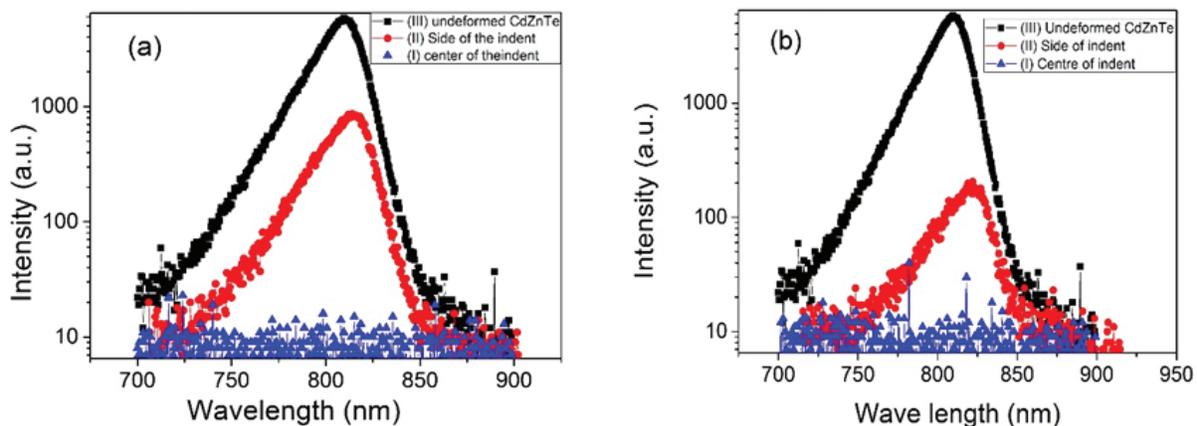


Figure 5. CL spectra on and around indent formed with a loading rate of (a) 1 mN/s, and (b) 5 mN/s.

decreases with the increase of dislocations, generated during deformation. Our results of CL spectra are consistent with the findings of Jian<sup>19</sup>, *et al.* Higher loading rate increases the dislocation density as discussed previously and hence quench more emission intensity. This CL result is also validating the explanation of increased residual stresses generated in the material with increased loading rates.

It is also observed that peak positions have shifted from 810 nm (un-deformed region) to 814 nm for deformation created by loading rate of 1 mN/s and 823 nm for the loading rate of 5 mN/s, at position II. This shifting to higher wavelength confirms the tensile stress in the material. Yacobi<sup>10</sup>, *et al.* have also similar findings on GaAs grown on Si, where the increase of residual stresses in the material were deduced from the peak shift towards higher wavelengths. Claudia<sup>11</sup>, *et al.* also studied the relationship of the shifting of CL intensity peaks with stress in alumina single-crystal and explained it due to the presence of residual stresses in the material. Sharma<sup>3</sup>, *et al.* has reported that band gap of semiconductor material increases with the presence of compressive stresses and decreases with the presence of tensile stresses. In CdZnTe, band gap is decreasing around indent edge and hence, confirms the presence of tensile residual stresses. Pagliaro<sup>20</sup>, *et al.* found that tensile residual stresses are created in radial the direction of indent created in 316L stainless steel during nanoindentation. Boyce<sup>21</sup>, *et al.* has studied about residual stresses and remnant damaged caused by hitting a spherical hard body on flat surface. Their study suggest that hitting point is having compressive residual stresses and nearby radially surface is having tensile residual stresses. Hence, these studies confirm the presence of tensile residual stresses on the vicinity of indent.

It may also be noted that the CL peak shift is more towards higher wavelengths with an increase of loading rate. This behaviour also confirms that residual tensile stresses in the material increases with the increase of loading rates. Tiwari<sup>22</sup>, *et al.* also found similar results for deformed copper bars.

CL images as shown in Figs. 6 (a) and 6 (b), which shows the dark lines, are actually the dislocations present in the material. These dislocations act as non-radiative recombination centre as discussed earlier and hence appear dark lines in the CL images. Previous studies have shown that the primary slip system of zinc blende structured CdZnTe is  $\{111\}, \langle 1\bar{1}0 \rangle$ .

This material has mainly dissociated  $\alpha$ ,  $\beta$  and screw dislocation segments<sup>23</sup>. The dislocation motilities of  $\alpha$  and  $\beta$

dislocations are nearly the same in this material reported by Sieber<sup>7</sup>, *et al.* Dislocations move in this material by either rosette glide or tetrahedral glide mechanism. When Berger's vector of dislocations are parallel to the surface, dislocations movement is through rosette glide and appears as straight lines in CL image. The tetrahedral glide of dislocations takes place in the material when Burger's vector is inclined with the surface. The possible slip directions on the surface of (111) CdZnTe, are  $[\bar{1}01]$ ,  $[\bar{1}10]$ ,  $[01\bar{1}]$ ,  $[10\bar{1}]$ ,  $[1\bar{1}0]$  and  $[0\bar{1}1]$ . There are three more slip directions in this material, which are inclined with the surface and are  $[101]$ ,  $[110]$ , and  $[011]$ . Dislocations moving in these directions make six glide prisms. Rosette and tetrahedral glide are responsible for the development of a rosette structure. This rosette structure is consistent with SEM images where slip lines are also forming a rosette structure. As these are massive dislocation generation beneath and around the indenter, even 1 mN/s loading rate is sufficient to generate dislocations, which are able to quench nearly all CL intensity. Due to this reason, increased dislocation density due to the higher loading rate is indistinguishable in CL image. No significant change in dislocation propagation behaviour has been observed in the CL images of loading rates of 1 mN/s and 5 mN/s, respectively.

#### 4. CONCLUSION

single-crystal (111) Te face CdZnTe substrate was subjected to nanoindentation using cube corner indenter with different loading rates of 1 mN/s and 5 mN/s. The load-displacement curves analysis shows the presence of compressive residual stresses on the indented region, when indented. Elastic modulus and hardness were found to be independent of loading rates. No cracks were found around indents for both loading rates of 1 mN/s and 5 mN/s, observed from FE-SEM images. CL spectra were obtained at three places of indents (on the indent, edge of the indent and far from indent) for both loading rates. CL peak observed at 810 nm at far from indent (un-deformed region) was found due to band edge emission. CL spectra on the indent show no peak, which was found due to a massive dislocations generation during indentation. The peak corresponding to band edge emission at the edge of indent was found to be shifted to higher wavelengths in CL spectra, which were attributed to the presence of residual tensile stresses present in the material. Higher loading rates (5 mN/s) shifts CL peak to higher wavelengths (823 nm) compared to low wavelength shift (814 nm) for low loading rate (1 mN/s). This was found due to higher residual tensile stresses generated in the material. These studies will be useful for optimization of mechanical processes like lapping and polishing of CdZnTe substrates for infrared detector applications.

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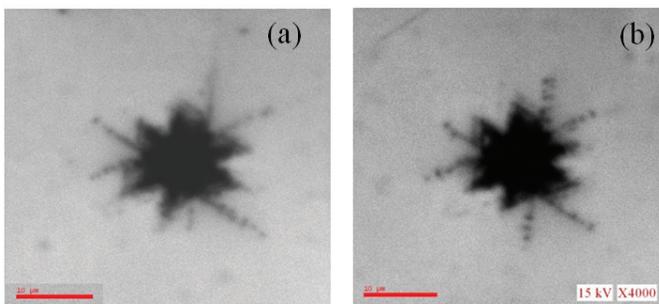


Figure 6. CL image of indent formed using a loading rate of (a) 1 mN/s, and (b) 5 mN/s.

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In the present study, he participated in the analysis of the obtained results.

**Mr Aditya Gokhale**, received his BTech from Nagpur University in 2011, MTech from NIT Nagpur in 2014 and currently pursuing PhD in IIT Delhi. His areas of interest includes Deformation behaviour of materials (hexagonal closed pack metals), texture of materials, materials characterization and nanoindentation based small volume study.

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In the current study, he has provided his valuable inputs for understanding cathodoluminescence (CL).

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In the current study, he has provided valuable suggestion for analysis of the results apart from reviewed the results.

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In the current study, he has reviewed the experiments, results and provided valuable inputs for analysis of the results. He has also contributed in understanding and explaining residual stresses and its effect on band gap of semiconductor materials.