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Effect of Loading Rate on Creep Properties of HgCdTe Epitaxial Films

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ABSTRACT

Nanoindentation creep studies were performed on $Hg_{1-x}Cd_xTe$ (x~0.29) epitaxial films using different loading rates of 0.5 $mN.s^{-1}$, 1 $mN.s^{-1}$, 2 $mN.s^{-1}$ and 4 $mN.s^{-1}$, keeping a constant peak load of 10 mN. A constant hold time of 20 sec at peak load was maintained for all experiments. The effect of loading rate on creep behaviour of material has been investigated. Creep displacement had shown increasing trend with increase of loading rates. Stress exponents were extracted using creep curve fitting with an empirical equation. A strong dependence of loading rate on stress exponent was observed. The value of stress exponent was found varying in the range 0.60-1.76, 0.96-2.23, 0.98-2,87 and 0.90-2.81 for loading rates 0.5 $mN.s^{-1}$, 1 $mN.s^{-1}$, 2 $mN.s^{-1}$ and 4 $mN.s^{-1}$, respectively. The change of stress exponent was attributed to change of creep mechanism. Hardness and elastic modulus were extracted from load-displacement curves and it was found that with the increase of the loading rate hardness increases, while elastic modulus remains constant. A correlation between variation of hardness and creep displacement has also been presented.

Keywords: Berkovich indenter; Creep; HgCdTe epitaxial films; Nanoindentation

1. INTRODUCTION

$Hg_{1-x}Cd_xTe$ (x~0.29) epitaxial films are used for the development of strategic devices like infrared detectors, night vision cameras and Imaging Infrared seekers for missiles[1,2]. The as-grown epitaxial films grown by liquid phase epitaxy (LPE) has inherent terraces features on the surface[3]. The epitaxial layer surface should be free from terraces, scratches and subsurface defects for device fabrication. Various mechanical processes like cutting, lapping and polishing are required to get the desired shape and surface of epitaxial layer[4]. During these processes, the $Hg_{0.71}Cd_{0.29}Te$ epitaxial films are subjected to various forces for long time that may lead to creep deformation. Different loading rates are used during these mechanical processes and hence systematic study of the variation of creep deformation on loading rates is essential to optimise the process parameters.

Nanoindentation is a technique to find out the mechanical properties like stiffness, hardness, elastic modulus and creep properties apart from deformation behaviour of the material especially for thin films[5–8]. Feng[9], *et al.* have reported the creep studies of Al and In metals. Creep studies of single crystal W and GaAs was reported by Syed[10], *et al.* Ding[11], *et al.* has reported the indentation size effect on creep deformation for bulk metallic glasses. Wang[12], *et al*. has studied the creep behaviour of polycrystalline Cu thin films. All these studies are very useful to understand the creep behaviour of the respective materials, and also provides understanding of creep deformation in general, but lack in providing information for $Hg_{0.71}Cd_{0.29}Te$ epitaxial films. No studies of the creep deformation for $Hg_{0.71}Cd_{0.29}Te$ epitaxial films have been done so far to the best of our knowledge. In this paper, creep deformation studies are performed using different loading rates to understand the effect of loading rate sensitivity on creep properties.

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2. EXPERIMENTAL WORK

$Hg_{0.71}Cd_{0.29}Te$ epitaxial films grown by liquid phase epitaxy (LPE) on lattice matched CdZnTe substrates were used in nanoindentation studies[3]. As grown films were first polished by 60 nm Al_2O_3 power to remove terraces from the surface followed by chemo-mechanical polishing (CMP) using an Iodine based solution[4]. After CMP, the average surface roughness of the samples was ~3 nm, measured using Agilent Technologies 6500 AFM. In all the samples, HgCdTe epitaxial layers were having a thickness of 17-20 μm as determined by the fringe pattern in Fourier Transform Infrared (FTIR) Transmittance Spectrum by a Varian 610 microscope with a 15X objective and using liquid nitrogen-cooled HgCdTe PC detector.

The nano-indentation experiments were performed using a Berkovich indenter with ASMEC, Germany make Universal Nano mechanical Tester. A maximum of the 10 mN load has been used for all nanoindentation experiments. Loading rates of 0.5 $mN.s^{-1}$, 1 $mN.s^{-1}$, 2 $mN.s^{-1}$ and 5 mN s^{1} were used during experimentation. Hold time during peak load for creep studies was maintained for 20 s in all experiments. During unloading, 60s hold time was provided at 1 mN for thermal drift correction[5].

Each experiment was repeated to 10 times to get an average value of the creep deformation as well as the extracted values of hardness and elastic modulus. The maximum displacement, observed after 20 s holding time, was ~0.9 µm, which is less than 10 % of the total thickness of the epitaxial layer (17-20 µm) and hence, substrate effect can be ignored[13].

3. RESULTS AND DISCUSSIONS

Load-displacement curves were obtained from nanoindentation experiments. Figure 1 shows the load-displacement curves using different loading rates. No pop-in or pop-out phenomenon was observed for any of the loading rates. It may be noted, that there is a significant increase in creep displacement with an increase of loading rates, as shown in Fig. 1.

Creep displacement has been obtained from load-displacement curves by providing holding time of 20 s at peak load. Fig. 2 shows the creep displacement curve for different loading rates. Initial points of creep displacement and time are aligned at a single point for comparison.

The creep displacement was found increasing sharply during the initial timings. The displacement rate during this period was decreasing. This is similar to the transient state of creep during uniaxial loading. After a few seconds, a slow increase in creep displacement was observed. The displacement rate was nearly constant during this period. This is analogues to steady-state creep. It may also be noted that creep displacement increases with increase of loading rates. During the low loading rate, the material has more time available to reach the peak load. As creep displacement takes place simultaneously during loading also, more time leads more creep displacement. This results in the lesser creep displacement during holding time.

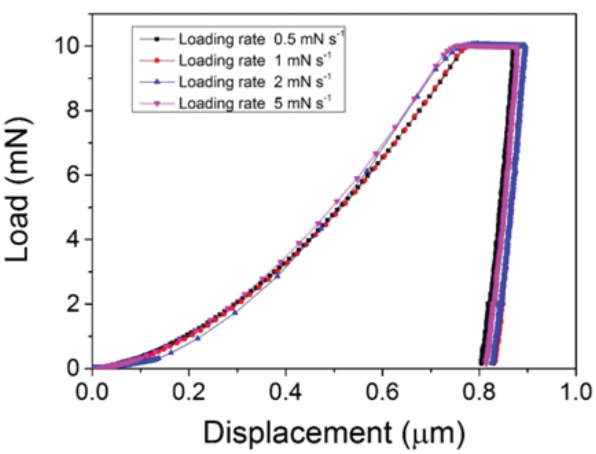

Figure 1. Load displacement curves using different loading rates of 0.5, 1, 2 and 5 mN s1.

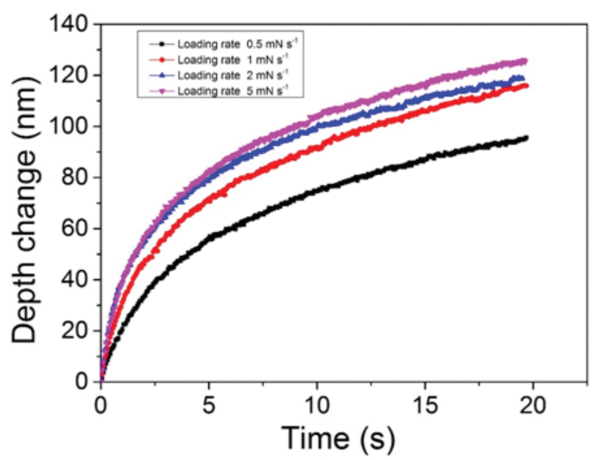

Figure 2. Creep displacement with time for different loading rates.

Strain rate and stress can be calculated from load-displacement curves from Eqn. (1) and Eqn. (2), respectively[14]:

$$\varepsilon' = \frac{\dot{h}}{h} \quad (1)$$

$$\sigma = \frac{P}{h^2} \quad (2)$$

where P is the peak load, h is the instantaneous penetration depth. $\dot{h}$ (dh/dt) is calculated from h, which is extracted out from fitting empirical relations as shown in Eqn. (3) to creep displacement–time graph[14].

$$h_t = h_i + \rho(t - t_m{}^m) + \sigma t \quad (3)$$

where h_i, ρ, t_m, m and σ are fitting parameters. Stress exponent can be calculated from the slope of ln ($\dot{\in}$) and ln (σ)[14]. As shown in Fig. (3), All curves were fitted into the empirical relation of Eqn. (3) and found that very good fits were obtained for all loading rates, having goodness of fit $R^2 > 0.999$.

Stress exponents were calculated for each loading rate by using the slope of ln ($\dot{\in}$) and ln (σ) as shown in Fig. 4. It is observed that the slope of these curves was not constant and have different values for the initial portion of curves and for later portion of curves. Stress exponent was found different indicating the possibility of change of creep mechanisms during this transition of its value. It is reported that the value of stress exponent ~1 represents diffusional creep and 3-5 represent dislocation creep[15].

In $Hg_{0.71}Cd_{0.29}Te$ epitaxial films, the value of stress exponent was found varying in the range 0.60-1.76, 0.96-2.23, 0.98-2,87 and 0.90-2.81 for loading rates 0.5, 1, 2 and 4 mN.s^{-1}, respectively. The variation of stress exponent values may be due to change of creep mechanism from diffusional to dislocation type[15]. Also, it is observed that stress exponent has a strong dependency on loading rate. Stress exponent has shown increasing trend with increasing loading rate to 2 mNs^{-1} and after that it shows the decreasing trend. Very high loading rates may initiate number of dislocation interactions and reactions that may lead low-stress exponent value.

Hardness and elastic modulus have also been extracted from load-displacement curves to understand the creep displacement effect on these properties. Increasing loading rates lead to increased hardness, measured from Oliver-Pharr method[5].

Increasing loading rates lead to less available time for material to creep during loading and hence, more creep displacement has been observed during holding time. This leads to more contact depth, hence more hardness has

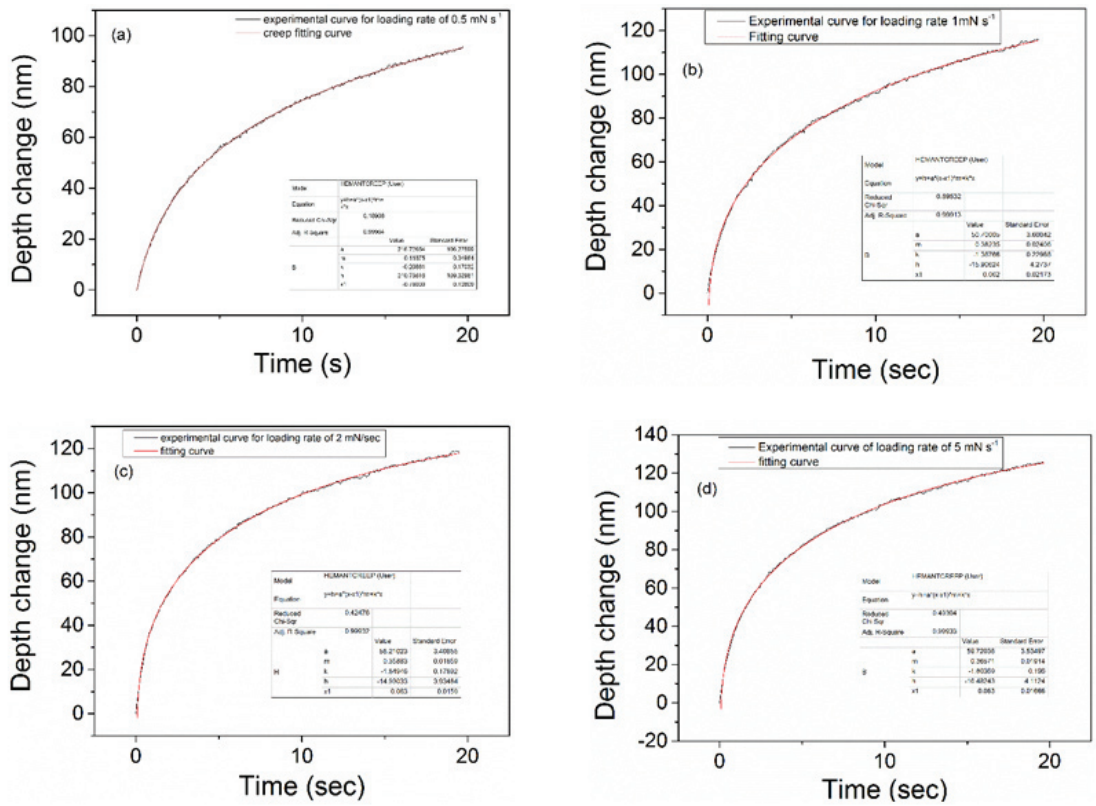

Figure 3. creep fitting curves with experimental data for loading rates (a) 0.5 mN.s^{-1}, (b) 1 mN.s^{-1}, (c) 2 mN.s^{-1}, and (d) 5 mN.s^{-1}.

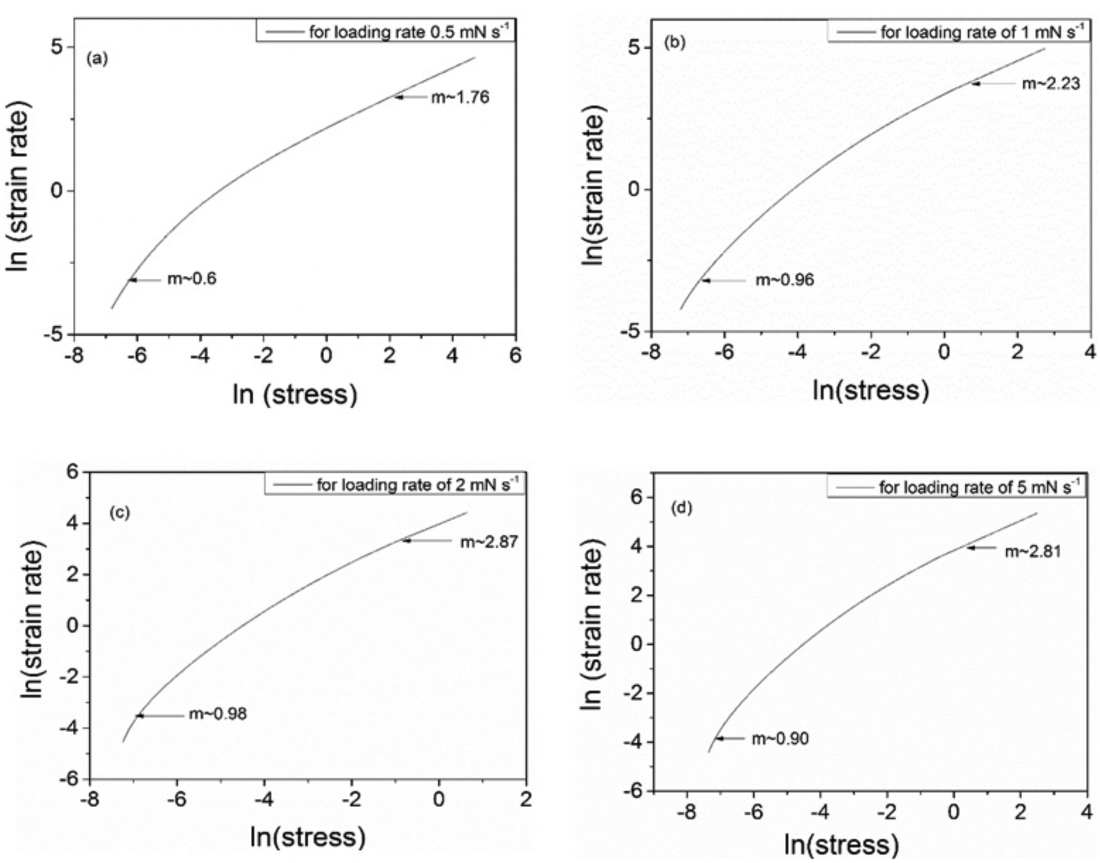

Figure 4. Curve between ln(strain rate) and ln(stress) for extraction of stress exponent for loading rates (a) 0.5 mN.s^{-1}, (b)1 mN.s^{-1}, (c) 2 mN.s^{-1}, and (d) of 5 mN.s^{-1}.

observed. Chudoba[16], *et al.* reported that holding time at peak load during nanoindentation of metals like Al, Au etc. is necessary to calculate the correct value of hardness. In the present study, the creep displacement is higher than the required value (increase in depth should be less than 1% of total indentation depth in 1 minute[16]), even with 20 sec of hold time. Hence, the hardness calculated from above method may not be the correct hardness of the material, as the material is continuously creeping even after 20 sec of holding time. These hardness values may be used for comparison as the condition of calculating hardness for all experiments are same. Elastic modulus values were found nearly constant for all loading rates. A higher value of elastic modulus towards higher loading rate may be an artifact of creep. As the material is not getting sufficient time for creep during loading, creep effect may be present during unloading that may lead to overestimation of stiffness[5,17], results in higher elastic modulus.

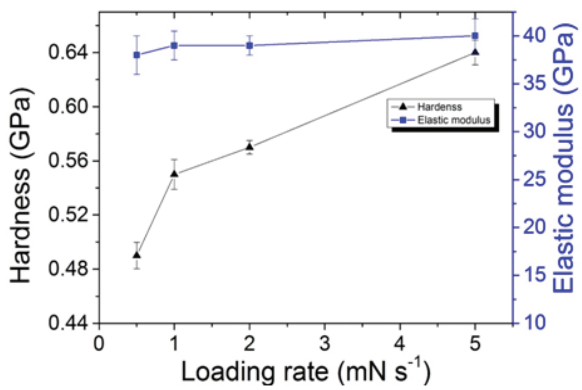

Figure 5. Variation of hardness and elastic modulus with loading rate.

4. CONCLUSION

Effect of loading rates on creep deformation behaviour has been studied on $Hg_{1-x}Cd_xTe$ (x~0.29) epitaxial films using nanoindentation technique. Loading rates of 0.5, 1, 2 and 4 mN.s^{-1}were used during experimentation. No pop-in or pop-out has been observed from load-displacement curves which shows absence of strain burst. Increased Creep displacement was observed with increase of loading rates. Different stress exponents were observed for all loading rates, which may be due to change in creep mechanism. Stress exponent was found to increase with increase of loading rate till 2 mN.s^{-1} followed by decreasing trend. The hardness of the material has shown increasing trend with increased loading rates. Elastic modulus values were found unaffected with varying loading rates. These studies will be useful for optimisation of mechanical process parameters for preparation of $Hg_{1-x}Cd_xTe$ (x~0.29) epitaxial films for infrared detectors.

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CONTRIBUTORS

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In the current study, he has performed exhaustive literature survey for planning of experiments and performed all nanoindentation experiments.

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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 helped in analyzing the 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 characterisation and nanoindentation based small volume study.
In the current study, he has supported in making experimental setup and helped in performing experiments.

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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 explaining creep phenomenon.