Cyclic nanoindentation studies of HgCdTe epitaxial films

Hemant Kumar Sharma, Rajesh Kumar Sharma, Raghvendra Sahai Saxena, Aditya Gokhale and Rajesh Prasad

1 Solid State Physics Laboratory, Lucknow Road, Timarpur, Delhi, India
2 Indian Institute of Technology, Hauz Khas, New Delhi, India
E-mail: rksraje@mse.iitd.ac.in

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Abstract

Hg_{1−x}Cd_{x}Te (x≈0.29) epitaxial films have been subjected to cyclic nanoindentation using spherical indenter from tribological aspects related to the development of polishing process for preparing defect free surface. Different loading/unloading rates of 0.5, 1 and 4 mNs−1 with a peak load of 10 mN were used for 10 nanoindentation cycles. An open jaw shape was observed in the load-displacement curve for loading/unloading rate of 0.5 mNs−1, while hysteresis loops were observed for 1 and 4 mNs−1 loading/unloading rates. This phenomenon is explained in light of the regimes of elastic/anelastic deformation and smooth plastic flow. Pop-in during first loading cycle was observed at loading/unloading rates of 1 and 4 mNs−1, which was attributed to elasto-plastic transition. Multiple pop-in events of low extent were also found in the form of serrations in load-displacement curves for loading/unloading rates of 0.5 and 1 mNs−1. Based on these observations, the maximum load and minimum loading rate during polishing process for this material has been suggested. The mechanical properties of these films, such as contact stiffness and hardness have been extracted for different loading/unloading rates. The effect of indenter geometry on deformation behaviour using Berkovich indenter is also reported.

1. Introduction

Hg_{1−x}Cd_{x}Te is a strategic material for the development of high performance infrared detectors due to its excellent optical and electrical properties [1–4]. The bandgap of Hg_{1−x}Cd_{x}Te can be tailored from −0.6 eV to 1.6 eV by varying its composition (x) and hence it can be tuned for use in all the important infrared bands [2]. Liquid phase epitaxy (LPE) is one of the most favoured techniques for growing epitaxial HgCdTe films for fabricating high performance IR detectors due to its capability of producing high quality material. However, the films produced using LPE generally require post-growth surface preparation with chemo-mechanical polishing (CMP) due to inherently obtained micro-terraces on their surface [5]. HgCdTe, being a soft/fragile material, is prone to mechanical damage during the CMP process.

During the fine polishing, the crystalline surfaces undergo periodic deformation due to mechanical interaction with the nano-sized abrasive particles as well as with the polishing pad material. The cyclic loading and unloading of the near-surface material is expected to alter the mechanical properties therein. A knowledge of the near-surface deformation characteristics can therefore help optimize the polishing properties.

Nanoindentation has emerged as a suitable techniques to study the deformation behavior of the material in the near-surface region with varying loads and other related parameters [6–12]. It has been previously used for primarily studying the mechanical properties of HgCdTe alloys by several researchers [13–17]. The elastic modulus and hardness of the Hg_{0.7}Cd_{0.3}Te single crystal were studied using nanoindentation experiments by Irwan et al [13]. Fang et al [14] performed nanoindentation measurements on thick epilayers (5–100 μm) of HgCdTe grown on CdTe substrate by ISOVPE process and also on CdZnTe substrate by horizontal LPE technique for estimating hardness. Sizov et al [15] performed a study on nanoindentation-induced phase transformation on Hg_{0.8}Cd_{0.2}Te deposited on CdZnTe single crystal substrate by LPE technique. Martyniuk et al [16] studied the mechanical properties of MBE grown Hg_{0.7}Cd_{0.3}Te thin film using nanoindentation.
experiments. Zhenyu et al. [17] studied ultra-high hardness and improved ductility for nanotwinned Hg_{0.22}Cd_{0.78}Te (111) wafers lapped by abrasive paper with a mesh size of 2000 using nanoindentation experiments. These studies although explored several useful properties of the HgCdTe films, they were focussed mainly on single cycle nanoindentation and lacked in providing the desired parameters and deformation behaviour during actual lapping and polishing induced cyclic loading/unloading, which could better be studied using cyclic nanoindentation. The cyclic nanoindentation can provide crucial information on the evolution of mechanical properties with repeated loading-unloading of the near surface material [12, 18, 19].

In this paper, deformation behaviour of near surface regions of epitaxial HgCdTe (x ~ 0.29) films on CdZnTe substrates as a function of different cyclic nanoindentation conditions is reported for the first time to the best of our knowledge. This work is primarily aimed for providing mechanical parameters for tribological interests related to the CMP processes.

2. Experimental details

Hg_{1−x}Cd_xTe (x=0.29) epitaxial films grown by VDLPE (vertical dipping liquid phase epitaxy) process [20] on lattice matched (111) B (Te face) surface of CdZnTe substrates were used in this work. Prior to indentation, the films were polished by 60 nm alumina powder initially to remove about a 2 μm surface layer followed by CMP on soft pad using iodine based solution developed previously at SSPL [21]. This process yields a smooth surface with minimal surface damage. Prior to nano-indentation experiments the surface condition of the samples was evaluated using Agilent Technologies 6500 AFM.

The thickness of the epilayers was determined from FTIR Transmittance spectrum using a Varian 610 FTIR microscope with a 15X objective and using liquid nitrogen cooled HgCdTe PC detector. The crystalline perfection was evaluated using a PANalytical make XpertPRO MRD system with CuKα radiation and 333 diffraction.

The cyclic nano-indentation experiments were performed using a spherical indenter having 10 μm tip diameter with ASMEC, Germany make Universal Nanomechanical Tester. A maximum of the 10 mN load has been used for cyclic nanoindentation with loading and unloading rates (hereafter referred to as L/UR) of 0.5, 1, and 4 mN s\(^{-1}\). Unloading has been done till 1 mN load before reloading the material so that point of contact of indenter and sample does not change. Initially the cyclic nanoindentation was performed for 100 cycles. It was observed that the penetration depth of the indenter starts saturating to about 0.7 μm after 10 cycles. Hence, all cyclic indentation experiments were performed for 10 cycles. Initial experiments with variable holding times at peak load of 10 mN ascertained that a holding time of 20 s at peak load was sufficient to minimize the contribution of creep during unloading. In all such cases the ratio of strain rates (signified by the slope of displacement vs. time curves) just after and before the start of unloading was observed to be > 60, with strain rate being negative from the start of unloading, as would be clear from the results presented later on. A holding time of 100 s was given after every unloading for correcting any thermal drift [22].

Some cyclic nanoindentation experiments were also performed with varying load range (ΔP = P_{max} − P_{min}), keeping P_{max} at 10 mN, L/UR 0.5 mN s\(^{-1}\) and ΔP values of 3, 6 and 9 mN. Cyclic nanoindentation with 1 mN s\(^{-1}\) L/UR was also repeated with a Berkovich indenter to examine the effect of indenter geometry.

Stiffness has been extracted from cyclic load-displacement curves using Oliver and Pharr method [22], in which the initial slope of unloading curve is described as Stiffness. It has been a general practice in the literature (for example [23]) to use penetration depths at the end of each cycle to estimate the hardness in cyclic nanoindentation experiments. The hardness is the overall resistance of the material to indentation which could only be estimated after the indenter has stopped penetrating further at a specified load. In case of cyclic indentation, the repeated loading can lead to ratcheting [24], resulting in the indenter penetrating higher than the maximum penetration depth during a single indentation. The application of Oliver and Pharr method, therefore, cannot be applied to estimate the hardness during cyclic nanoindentation experiments. In our case even very long holding times of up to 400 s did not result in stopping the penetration and the minor cracks started appearing around the indentation mark. Under such circumstances it was considered appropriate to estimate the hardness to a first approximation from a constant elastic modulus value estimated in [16] using the following expressions [22]:

\[
E_r = \frac{1}{2} \left( \frac{\pi}{A_p} \right)^{1/2} S
\]

(1)

\[
H_n = \frac{F_p}{A_p}
\]

(2)

where \(A_p\) is the effective contact area (=2πR_h, with \(R\) & \(h\) being the indenter radius and contact penetration depths) at peak load, \(S\) is stiffness, \(E_r\) is reduced modulus [22], \(H_n\) is indentation hardness and \(F_p\) is peak load.
during indentation. Here effective $P_c$ estimated from equation (1) can be substituted in equation (2) to estimate the indentation hardness using the known value of $E_p$.

3. Results and discussions

Figure 1(a) depicts an AFM image of a typical HgCdTe epitaxial layer sample after CMP process Average surface roughness of the samples was found to be around 3 nm. Figure 1(b) depicts the FTIR Transmittance spectrum of a typical samples used in this work. The fringe pattern is utilized to estimate the thickness of epitaxial layers [25]. The epitaxial layer thickness of the samples used were between 17–20 μm. Figure 1(c) shows HRXRD rocking curves over a typical sample. The FWHM of ~40 s of arc indicates a good crystalline perfection for these LPE grown HgCdTe layers. The dislocation etch pit density in such samples is of ~4 × 10^6 cm⁻² indicating an inter-dislocation separation of ~50 μm. Since the nano-indenter diameter is 10 μm, it is generally expected that the initial loading would be in a near perfect crystal region. The penetration depth of ~0.7 μm for >17 μm thick films having a surface roughness of ~3nm was considered adequate for determination of mechanical parameters from the cyclic nano-indentation data. We would now present/discuss the results of nanoindentation studies.

3.1. Load-displacement (P-h) characteristics

Figures 2(a)–(c) depicts the variation of load (P) and displacement (h) with time at L/UR of 0.5, 1 and 4 mNs⁻¹ respectively using a spherical indenter. The corresponding P-h characteristics are shown in figures 2(d)–(f).

The major observations from figure 2 are: (a) Unloading-reloading curves show open jaw shape with L/UR of 0.5 mNs⁻¹ while hysteresis loops are seen therein with 1 and 5 mNs⁻¹, (b) A pop-in (sudden discontinuity during loading) was observed during first loading for L/UR 1 and 5 mNs⁻¹ but was absent in case of L/UR of 0.5 mNs⁻¹ and (c) Serrations were observed with L/UR of 0.5 and 1 mNs⁻¹ and were rarely seen for 4 mNs⁻¹.

The open jaw shape observed in figure 2(d) can be can be understood in terms of a combination of elastic and plastic deformation during loading and primarily elastic/anelastic recovery during unloading [18, 23]. The elastic/anelastic deformation is primarily recovering in the unloading part of the cycle and additional plastic deformation is occurring over and above the expected elastic deformation during the subsequent loading for the next indentation cycle. The hysteresis type loops observed in figures 2(e) and (f) are similar to the observations made in earlier studies including [18, 23, 26], where different explanations have been given for this behaviour. However, the most relevant one is by Saraswati et al [18] who have reported open jaw shaped P-h characteristics for pure gold samples, while the hysteresis type loops were observed for the Ca doped gold. They proposed that Ca atoms segregate around deformation induced dislocations forming the Cottrell atmosphere and thereby making their slip harder in subsequent indentation cycles. Once the applied load overcomes the resistance of Cottrell atmosphere, the displacement becomes faster. This phenomenon can result in hysteresis type loops in displacement versus load graphs. We, however, would like to point out here that the work hardening, or more precisely the dislocation entanglement, in general is expected to result in the same behaviour. Impurities would restrict the dislocation slips even if they do not segregate around dislocations to form Cottrell atmosphere, which would be expected more in high temperature treated samples. We would like to further emphasize that the explanation of hysteresis like loop would require consideration of unloading behaviour of the previous cycle as well, as depicted in figure 3 schematically. It can be appreciated that during unloading initially there would be an instantaneous elastic recovery followed by a slow anelastic recovery [27]. Subsequently during loading for the next cycle initially the dislocation slip may be restricted due to dislocation pinning on other dislocation segments, impurities, stacking faults, grain boundaries, microcracks etc. However, after a specific load marked as $P_{c, anelast}$ a smooth plastic flow is expected. It can now be appreciated that both anelastic recovery and restricted dislocation slip have to be significant in order to observe the hysteresis type loop. Observation of this looping behaviour of course is subject to the magnitude of L/UR. In case of very slow loading/unloading, the loops in P-h characteristics would not be observed as both plastic and anelastic deformations/recovery may keep completing within the loading/unloading time spans. We can now understand the open jaw structure observed instead of a hysteresis type loop for the same maximum load of 10 mN in case of 0.5 mNs⁻¹ L/UR. The indentation induced deformation in all the three cases is nearly same after holding the peak load for about 20 s on first indentation. The maximum penetration depths of about 0.515 μm, 0.505 μm and 0.495 μm can be extracted from figures 2(d)–(f) for the three cases. The variation observed is due to different overall loading times of 40 s, 30 s and 22.5 s respectively given in the three cases. It can be presumed that the anelastic recovery process is faster than the unloading at the rate of 0.5 mNs⁻¹, as no anelastic recovery region is observed therein. To test this hypothesis, we further analysed the anelastic recovery region of the first unloading curve for L/UR of 4mNs⁻¹. The data was fitted with an exponential decay of the characteristic time (τ) of ~2.46 s with $R^2 \sim 0.998$, as depicted in figure 4. Similar regions were found to be roughly linear in case of (a) subsequent cycles of the
same data and (b) for all the cycles for L/UR of 0.5 and 1 mN s$^{-1}$. It can be appreciated that since the dislocation density in the deformed region would increase with successive cycles of indentation [18], the dislocation bending responsible for anelastic recovery [27] would successively decrease thereby reducing the extent of anelastic deformation. In case of slower unloading, the exponential recovery anyway is not expected. We would also like
Figure 2. Load-time-displacement curves with loading and unloading rates of (a) 0.5 mN s\(^{-1}\), (b) 1 mN s\(^{-1}\) and (c) 4 mN s\(^{-1}\) and load-displacement curves with loading and unloading rates of (d) 0.5 mN s\(^{-1}\), (e) 1 mN s\(^{-1}\) and (f) 4 mN s\(^{-1}\).

Figure 3. Schematic hysteresis type loop in a load vs displacement (P-h) characteristics for cyclic nanoindentation.
to mention here that the data presented in figure 4 appears an incidental right condition for observation of exponential anelastic recovery, as the unloading time of 2.25 s is less than the time constant observed. It would be interesting to plan future experiments to study the anelastic deformation/recovery in similar materials using cyclic nano-indentation experiments with faster unloading rates. At present a further discussion on this issue is considered beyond the scope of the current study.

The pop-in observed in nanoindentation $P$-$h$ characteristics during loading has been attributed primarily to three phenomenon [28–30]: (a) Onset of elasto-plastic transition, (b) Phase Transformation and (c) Crack formation. No cracks were observed around the spherical indentation marks during a careful scanning electron microscopic (SEM) observation of the samples. An SEM picture of a typical indent is shown in figure 5. The pressure exerted in the indented region during loading/unloading at a load $P$ can be estimated as $p = P/A_z$; with the area of contact for a spherical indenter, $A_z \approx 2\pi R_i h_c$, where $R_i$ is the indenter radius and $h_c$ is the contact depth [31]. Accordingly, the onset of pop-in has been found to be $\approx 1.8$ GPa as presented in table 1 for both the cases. This is in complete agreement with an earlier study by Martyniuk et al [16] carried out using Berkovich indenter.

Phase transformation from B3 (zinc blende) to B9 (cinnabar) structure at a pressure of 1.4 GPa and 2.2 GPa respectively has been reported for HgTe [32] and Hg$_{0.8}$Cd$_{0.2}$Te [33] bulk single crystals. CdTe on the other hand has been reported to have a phase transformation from B3 to B1 (sodium chloride) structure at 3.6 GPa. Although no similar studies have been made for Hg$_{0.7}$Cd$_{0.3}$Te, the above facts indicate that in case any pressure induced phase transformation would occur in Hg$_{0.7}$Cd$_{0.3}$Te it would be at pressure values further higher than

![Figure 4. Exponential behaviour of anelastic recovery region of the first unloading curve for L/UR of 4 mNs$^{-1}$.](image)

![Figure 5. Typical SEM images of spherical indents after cyclic nanoindentation with L/UR of 1 mNs$^{-1}$.](image)
2.2 GPa. In view of the facts that (a) the onset of pop-in has been observed for pressure values around 1.8 GPa for Hg₀.₇Cd₀.₃Te which is well below the expected phase transformation pressure for this material and (b) there is no pop-out event characteristic of phase transformation during unloading \[16, 31\], it can be concluded that the pop-in is related with the onset of plasticity herein.

Moreover, the P-h characteristics are also expected to follow the Hertzian law for mechanical contacts prior to the pop-in events, that is \( P \propto h^{3/2} \)[28]. Figure 6 depicts the regions of P-h characteristics following Hertzian law. It is evident that in both the cases, where the pop-in was observed, the P-h characteristics by-and-large followed the \( P \propto h^{3/2} \) relationship till the observation of pop-in event. However, \( P-h \) curve corresponding to \( L/UR \) of 0.5 mNs\(^{-1}\) starts deviating from the Hertzian law at very low load values (i.e. \( \sim 0.6 \) mN). At 0.6 mN load the maximum Hertzian contact pressure of \( \sim 2.4 \) GPa and a maximum shear stress of \( \sim 0.7 \) GPa can be estimated [34]. Even the shear stress at the surface of the sample is of \( \sim 200 \) MPa. In view of the fact that the Critical Resolved Shear Stress (CRSS) for HgCdTe at yield point has been reported to be 8–12 MPa [35], one can expect an instantaneous dislocation nucleation at the sample surface during indentation process. However, it appears that during the slow indentation process only the dislocation nucleation, propagation and multiplication can keep pace with the loading rate. At higher loading rates, on the other hand, the stress is primarily elastic/anelastic and spreads over a larger depth. A sudden burst of dislocation generation/propagation can occur after a critical stress. We understand that this is a critical information for the tribological interests related to the development of polishing processes for preparing defect free surfaces. For example, from the current study it can be inferred that the loading pressures of less than 1.8 GPa and loading rates of \( > 1 \) mNs\(^{-1}\) (The maximum value can be experimentally determined) can preferably be used for the polishing processes of HgCdTe.

Serrations in P-h characteristics can be regarded as multiple pop-in events of smaller extent which are helpful in a gradual release of strain in the material. Serrations events are obviously expected to be slower than any major pop-in events where a sudden burst and movement of dislocations can be expected [16]. The slower indentation process has therefore shown both serrations as well as early deviation from Hertzian elastic behaviour. In case of \( L/UR \) of 5 mNs\(^{-1}\) the stress rate is high enough to produce smooth flow of dislocation surpassing all barriers encountered due to other dislocations, alloying, impurities etc. It is, therefore, further stressed that the optimization of tribological conditions during surface preparation requires that a maximum load and a minimum loading rate are maintained during any surface preparation process, these parameters would be the functions of the size of polishing particles though.

### 3.2. Depth of indentation with number of cycles

Ratcheting effect [24] was also observed in all the cyclic nanoindentation experiments as depicted in figure 7, where a continuous increase in the penetration depth is observed for every indentation cycle with all the curves.
showing two distinct stages of deformation. In the secondary stage the incremental deformation tends to attain a steady progress. Figure 7(a) depicts the variation of indentation depth with number of indentation cycles. It is observed that after initial three indentation cycles the incremental indentation depth starts saturating as seen in figure 7(b). It was again observed that both the maximum penetration depth and the incremental penetration are much higher in case of 0.5 L/UR case, suggesting again that under slower loading a higher ratcheting effect is expected. On the other hand, the saturation values for the other two cases are quite lower and similar.

In another experiment, the effect of overall unloading was also studied with constant peak load of 10 mN and L/UR of 0.5 mNs$^{-1}$, for the three cases where unloading was carried out to 7, 4 and 1 mN, correspondingly for a load variation ($\Delta P$) of 3, 6 and 9 mN (figure 8(a)). Here the steady state incremental penetration per cycle in the second stage ($\beta$) was found to vary almost linearly with $\Delta P$ as depicted in figure 8(b) with extrapolated value of $\beta$ approaching a negligible low value. It can be further concluded that the loading/unloading also plays a crucial role in the incremental deformation in this material and the same may be critically considered for optimizing any CMP process for this material.

3.3. Extraction of mechanical properties

No definite trend in the variation of stiffness was observed with number of cycles with a large scatter in data in case of L/UR of 0.5 and 1 mNs$^{-1}$. However, for higher L/UR of 4 mNs$^{-1}$, the value of stiffness has been nearly constant. Table 2 summarises the Stiffness and corresponding indentation hardness values (extracted using equation (1) and (2)) for the three cases along with the standard deviation therein. The Indentation Hardness values obtained are in broad agreement with the values of 1.2–0.6 GPa reported in [16] under varying load condition.

A large scatter in stiffness data extracted from $P$-$h$ curves in case of L/UR of 0.5 and 1 mNs$^{-1}$ can be attributed to the errors in slope estimations due to instabilities during deformation as well as recovery resulting in serrations observed therein. The smooth $P$-$h$ curves for L/UR of 4 mNs$^{-1}$ have on the other hand resulted in a

Figure 7. (a) Variation of indentation depth with number of cycles (b) Change of indentation depth per cycle ($dd/dN$) with number of cycles.

Figure 8. (a) Schematic of load vs. cycle showing load variation (b) variation of secondary stage indentation depth per cycle with load variation.
very little scatter in the data. It can also be observed from Table 2 that the stiffness values are decreasing with increasing L/UR, while indentation hardness is increasing correspondingly with L/UR. With higher loading rate one can expect a higher density of dislocations around the indent as compared to that with the slower loading \[36\]. Under such situation one can expect a correspondingly higher hardness. The stiffness is a purely elastic property which is related with the shortrange atomic order and is not expected to depend largely on deformation. Nevertheless, the shortrange elastic deformation associated with dislocations can result in lowering the stiffness.

### 3.4. Effect of indenter geometry

Load displacement characteristics of the cyclic-nanoindentation using Berkovich indenter with L/ULR of 1 mN s\(^{-1}\) is shown in figure 9. The sharp Berkovich indenter has been reported to induce elastoplastic transition in HgCdTe at a low load of \(\sim 50\) μN \[16\] and hence no pop-in has been observed in our case. However, the hysteresis like loops and serrations similar to the case of spherical indenter were observed, the extent of recovery and hysteresis was comparatively a bit lower though. This is consistent with the understanding of a higher deformation with a sharp indenter compared to a spherical one. Observation of serrations in case of similar L/UR with both the indenter geometries is also consistent with our earlier hypothesis of smooth flow of dislocations being dependent on a critical stress rate.

### 4. Conclusions

LPE grown HgCdTe epitaxial films were subjected to cyclic nanoindentation with varying loading unloading rates (L/UR) of 0.5, 1 and 4 mNs\(^{-1}\) for 10 cycles and also with changing load variation (\(\Delta P\)). A summary of the main results is as follows:

(a) The load displacement (P-h) curves during the first loading followed the Hertzian law for mechanical contacts before pop-in events observed at 5.7 and 5.3 mN respectively for loading rates of 1 and 4 mNs\(^{-1}\), indicating an elastic/anelastic deformation regime therein. However, for the loading rate of 0.5 mNs\(^{-1}\) virtually no pop-in event was observed and the P-h curves deviated from the Hertzian law right from a load of 0.6 mN indicating the onset of plastic flow at a very low load.

| L/UR (mN s\(^{-1}\)) | Stiffness (mN μm\(^{-1}\)) | Indentation Hardness (GPa) |
|-----------------------|-----------------------------|---------------------------|
| 0.5                   | 198.8 ± 17.8                | 0.82 ± 0.15               |
| 1                     | 190.2 ± 13.8                | 0.89 ± 0.12               |
| 4                     | 174.3 ± 2.8                 | 1.05 ± 0.03               |

Figure 9. Load-displacement curves for cyclic nano-indentation with Berkovich indenter for L/UR of 1 mNs\(^{-1}\).
(b) The anelastic recovery rate of 2.46 s estimated from the exponential behaviour of the $P-h$ characteristics during first unloading at 4 mN s$^{-1}$ indicated/confirmed the slowness of dislocation movement/stretching processes compared to the instantaneous elastic deformation.

(c) More incremental deformation of the material was observed for slower L/UR of 0.5 mN s$^{-1}$ due to higher ratcheting effect in this case. It is also emphasized that the estimation of hardness from the varying penetration depths can be erroneous as it results in subsequent determination of large variation of elastic modulus.

(d) No definite trend in variation of stiffness with number of cycles was observed, but it was found decreasing with increasing L/UR. Indentation hardness showed increasing trend with increasing L/UR, indicating deformation being more confined to the indented region compared to an extended deformation with lower L/UR cases.

The following general conclusions can be made from this study:

(a) The open-jaw and hysteresis-like-loops observed in load displacement curves under different L/URs can be explained in terms of elastic/anelastic deformation/recovery and smooth plastic flow regimes.

(b) An analysis of load displacement characteristics obtained under suitable experimental conditions can reveal crucial tribological information, such as the maximum particle load and a minimum loading rate to be used for optimization of CMP processes for semiconductor materials where minimization of surface damage is crucial.

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ORCID iDs

Rajesh Kumar Sharma https://orcid.org/0000-0003-0624-1182

Raghvendra Sahai Saxena https://orcid.org/0000-0002-6129-7771

Rajesh Prasad https://orcid.org/0000-0002-5006-8236

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