Springer

Journal of
Mechanical
Science and
Technology

Journal of Mechanical Science and Technology 23 (2009) 2652~2657

www.springerlink.com/content/1738-494x
DOI 10.1007/s12206-009-0718-4

Nanoindentation of aluminum (100) at various temperatures †

Murugavel Rathinam[1,*], Ramesh Thillaigovindan[2] and Prema Paramasivam[3]

[1]Principal, Paavai College of Engineering, Namakkal, Tamilnadu, India
[2]Sr.Lecturer, Dept. of Mechanical Engineering, National Institute of Technology, Tiruchirappalli, Tamilnadu, India
[3]Research Scholar, Dept. of Computer Applications, National Institute of Technology, Tiruchirappalli, Tamilnadu, India

(Manuscript Received August 25, 2008; Revised May 2, 2009; Accepted June 18, 2009)

Abstract

High temperatures generally affect materials in some form. In this regard, the capability to perform nanoscale measurements at elevated temperatures opens up new possibilities for investigating the temperature dependence of materials' mechanical properties. Particularly, the responses of aluminum's different mechanical properties to indentation at various temperatures have been studied experimentally. In this paper, aluminum response to different room temperatures was examined. The behaviors of a single crystal aluminum during loading and unloading were observed. Nanoindentation experiments on a single crystal aluminum (100) sample at temperatures of 265 K and 388 K were performed with different loading conditions. At the start of the first burst of the dislocation glide, which was indicated by a sudden increase in displacement with no increase in loading, evidence of plastic properties and softening effects on aluminum was identified. The ductile to brittle transition was observed at temperatures below 273 K. Generally, there was a significant increase in the penetration depth and a decrease in hardness, elastic modulus, and elastic recovery as the testing temperature increased.

***Keywords*:** Aluminum; Effect of temperature; Mechanical properties; Nanoindentation

1. Introduction

Temperature changes may have drastic effects on the behavior of materials as demonstrated in the sinking of Titanic, so knowledge in this area is important, and more so, it is a scientific interest. In this regard, a detailed mechanistic understanding of material failure on surface and near surface due to defects initiation and evolution at various temperatures is necessary. High-temperature applications include surface-engineered engine components and bearings, also require the optimization of mechanical properties for the service temperature.

Experiments were performed on Aluminum (A1) (111) surface on a silicon substrate at room temperature [1]. Polycrystalline and single crystal aluminum were used in the experiment. The value of Hardness (H) was found to decrease when the Displacement (h) is high, while the temperature increases [2]. The results of nanoindentation experiment on Fe-3% silicon (Si) at room temperature and at 379 K suggest that the load in which yielding occurs is independent of the temperature between room temperature and 379 K [3]. Simulation of indentation on (111) surface of a gold (Au) sample was done [4]. The simulation temperature was 0 K. The dislocation mechanism was due to the release of compression beneath the indenter and the conservation of volume in the thin film during micro plastic deformation (bursts). It was found that the rotation of the lattice during nanoindentation may account for the pileup of the material. Simulation of indentation and scratching of a single crystal aluminum at room temperature was done [5]. Experiments on Au (111) surface was also carried out [6]. Mean-

† This paper was recommended for publication in revised form by Associate Editor Maenghyo Cho
*Corresponding author. Tel.: +91 4286 200968, Fax.: +91 4286 248658
E-mail address: mrgvel@yahoo.com

while, the current work gives a detailed explanation for the interpretation of a scan line graph.

Experiments on glass, gold, and single crystal silicon at room temperature and 473 K showed that at 473 K, the hardness and elastic modulus of soda lime glass and gold are lower when at room temperature [7]. In contrast, indentation testing of Si (100) at 473 K produced a similar hardness value to that obtained at room temperature, although the modulus was again reduced from 140.3 to 66.0 GPa. Results of the mechanical property characterization of thin films using spherical tipped indenters were presented earlier [8]. The step-by-step transition from elastic to elastoplastic deformation was identified at the initial stage of nanoindentation [9]. Temperature dependency studies for the hardness of a Bulk Single Crystal GaN in comparison with other wide-gap materials were performed earlier [10]. Nanocomposite iron alloy derived from metallic glass at room temperature revealed that although they exhibited high yield strength, they were brittle and did not show tensile elongation [11]. The results revealed that the main mechanism for elevated temperature deformation was grain boundary sliding/rotational processes.

Increasing the deposition temperature from 523 K to 973 K on the deposited Ti-Si-N coatings. led to a significant increase in hardness [12]. Micro materials measuring technology explained the technique for higher-temperature nanoindentation [13]. For copper (111) and Silicon (100) substrates, the first elastic response appeared only before the penetration depth of 4.5nm in the case of thin films [14]. Nanoindentation experiments on single crystal ionic materials with special attention devoted to elastic response revealed that the in-plane interactions played a key role [15].

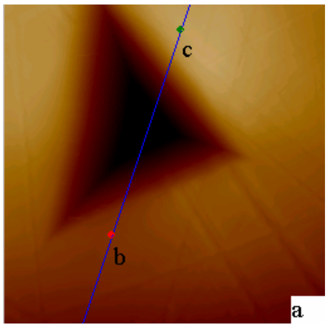
2. Methodology

Experiments were performed using a nanoindentation setup with a Berkovitch tip of 100 nm radius at the temperatures of 265 K and 388 K. In the experiments at elevated temperatures, an insulating material was used to protect the piezoelectric setup of the indenter. A small heater was added with the sample holder to maintain a constant temperature. The heating stage consisted of an insulating ceramic block attached to the nanotest sample holder. With the heater at a higher temperature, the temperature increase behind the ceramic block was normally less than 1 degree. The sample surface was brought to a constant temperature for each experiment. The temperature of the sample surface was measured before the indentation. A new attachment was made to circulate dry nitrogen gas to eliminate the formation of ice during the low-temperature experiments. The loading rate was held constant for each experiment. The sample was 10 mm diameter and 2 mm thick. It took about 15 minutes to reach the required temperature. Imaging was done with a 2 μN force. The topography and gradient images were captured to show the surface morphology after the indentation. In situ, built in Atomic Force Microscopy (AFM) imaging provided the capability to observe and quantify the material damage while minimizing the time for material recovery. During the course of the instrumented indentation process, a record of the depth of penetration was made over a complete loading cycle using depth sensing indentation, and then the area of the indent was determined using the known geometry of the indentation tip. While indenting, various parameters such as load and depth of penetration were measured. A record of these values was plotted on a graph to create a load-displacement curve. These curves were used to extract the mechanical properties of the material.

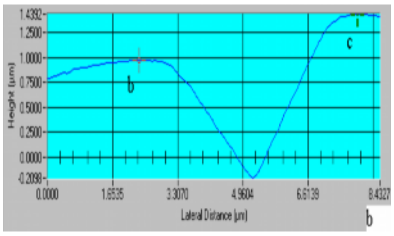
3. Results and discussion

Pileup around an indentation is clearly observed. Figs. 1(a) and 1(b) are the AFM images. Figs. 1(a) and 1(b) show the pileup effect in the case of indentation at a load of 10000 μN and a loading rate of 2000 μN/s. The pileup at a low temperature was due to the resistance for dislocation. The position 'c' on Fig. 1(a) shows the topmost regions of the pileup.

The multiple force-displacement curves in Fig. 2 are the results of experiments at various locations of the sample for the same experimental parameters. Figs. 2(a), 2(b), and 2(c) are the result of experiments with various parameters. The various colors are used only to identify the different curves. Figs. 2(a)-(c) show that the elastic recovery is at a minimum for aluminum at these load conditions, as compared to Cu. The initial burst, however, is clearly observed. From Fig. 2(c), the little visible initial burst is noted for 265 K only at this load and rate. The clear initial burst curve for the direct comparison at 50 μN and 10 μN/s cannot be obtained. Moreover, the curves at 265K show brittle behavior because they have withstood a higher load before the first yield compared with the



(a) topography image at 10µ



(b) Pileup asymmetry

Fig. 1. AFM images of the sample after indentation at 388 K.

curves obtained at 388K. From the initial burst curves, it is observed that the initial burst occurred very early as the material was accommodated inside instead of being piled up.

In Fig. 3, it is observed that the brittle behavior of the material at low temperature is not visible since the load is high. Therefore, the brittleness cannot be seen from the curves obtained at a higher load. Even the initial burst is not visible at higher load. Figs. 4(a) and (b) show the curves obtained at 10000µN and 2000µN/s for the purpose of comparison during the loading and unloading process to observe the maximum penetration depth and elastic recovery. It is found that during the unloading process or the recovery process, the material emerges out very steeply at 388K, and the recovery process stops abruptly at this temperature. In the case of 265K, however, the recovery rate is less and rebounds sharply at the end of the recovery process.

Tables (1) and (2) show that the maximum penetration depth increases due to the increase in temperature, as the material softens at higher temperatures.

Each data set is the result of experiments at various locations as a sample for a given temperature. At a high temperature, the hardness and modulus are reduced due to softening, which is also evident from the increase in the depth of indentation in the same loading conditions. Elastic recovery increases with the

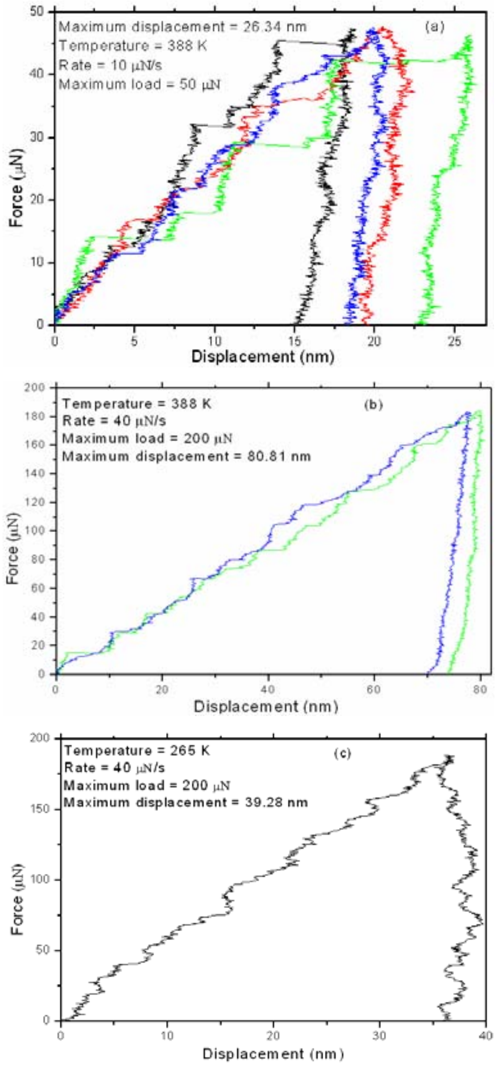


Fig. 2. Comparison of initial burst, (a) 50 µN, 10 µN/s and 388 K, (b) 200µN, 40 µN/s, 388K, (c) Brittle behavior at 265 K, 200 µN, 40 µN/s.

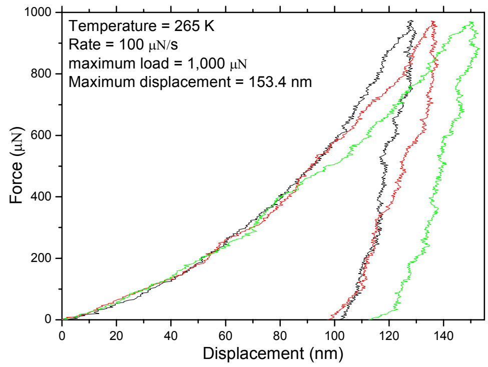


Fig. 3. Shows burst at 1000 µN, 100 µN/s, and 265 K.

Table 1. Comparison of data for different temperatures at 200 μN, 40 μN/s.

Temp\constants	h_{max} nm	H Gpa	E Gpa
265 K	68.34	1.3	631.48
	90.03	0.82	41.67
	79.94	0.96	25.65
388 K	128.2	0.26	273.9
	112.3	0.36	1194
	95	0.47	840.16

Table 2. Comparison of data for different temperatures at 10,000 μN, 2000 μN/s.

Temp\constants	h_{max} nm	H Gpa	E Gpa
265 K	707.87	0.76	54.64
	668.26	0.87	93.92
	697.82	0.77	53.66
	683.13	0.83	43.1
388 K	992.3	0.42	16.5
	1054.9	0.4	12.1
	1004.2	0.44	12.8

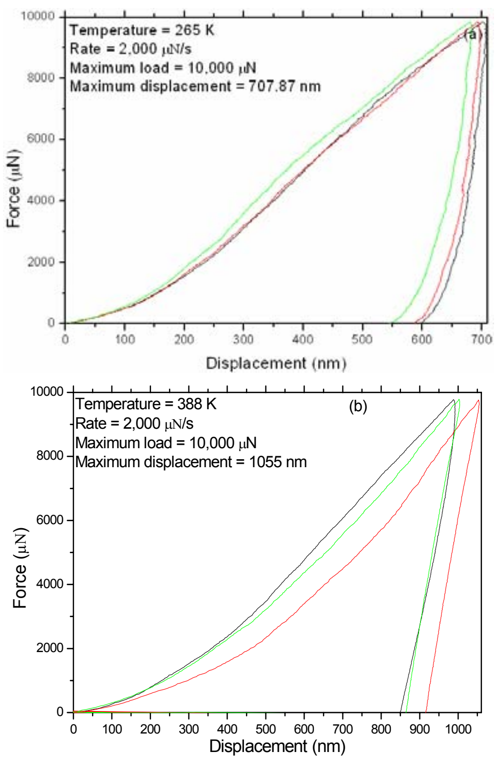


Fig. 4. The softening effects at 10000 μN and 2000 μN/s, (a) 265 K, (b) 388 K.

penetration depth as the temperature increases. The plastic range also increases with the increasing temperature, resulting in the increased failure of the material at higher temperatures. As such, the elastic recovery is reduced at higher temperatures. The hardness decreases, and displacement increases as the temperature increases.

It is also noted that the difference between plastic displacement ($h_{plastic}$) and maximum displacement (h_{max}) is less at a smaller loading rate. It shows that the loading rate also affects the range of plasticity. The hardness and displacement increase as the temperature increases. Furthermore, the value of the elastic modulus (E) is high when the difference between $h_{plastic}$ and h_{max} is large. It is observed that the H value does not depend on the difference between $h_{plastic}$ and h_{max}. The value of H is found to decrease when h_{max} is high, as noted from Tables (1) and (2) in line with the studies of Smith et al. [2].

The h_{max} in Tables (1) and (2) show the softening effects at different temperatures. The measured amount of softening enables the selection of proper materials for different thermal loading conditions. At higher temperatures, the material is subjected to higher dislocation mobility and greater plastic deformation. However, the deformation is global, unlike with the multi-bursts (brittleness) due to local dislocation motion at low temperatures.

There are clear strain bursts showing the breakout of dislocations. Based on h_{max} in the tables, significant softening may occur at a sufficiently high stress at higher temperatures. The elastic recovery is also reduced at higher temperatures.

4. Conclusions

The experiments performed show that a significant softening of aluminum occurred at higher temperatures, as it is observed from the penetration depths read from the curves. It is also noted that the elastic recovery decreased at higher temperatures. In addition, the reduction in modulus occurred as the temperature and penetration depth increased. The capability to perform nanoscale measurements at elevated temperatures opens up the possibility for similar studies. The complete elastic range was found. The onset of the first burst of the dislocation glide, which is indicated by a sudden increase in displacement without an increase in loading, was clearly observed. The start of plastic deformation was viewed from the periodic bursts. From the difference in penetration depth for different temperatures, it was noted that there was a significant increase in depth at a higher temperature. The hardness and elastic modulus also dropped at higher temperatures. The brittle behavior was seen at negative temperatures similar to copper. It was observed that the temperature variation influences the hardness and modulus values significantly. At a high temperature, the hardness and modulus decreased due to softening, as it was evident from the increase in the depth of indentation for the same loading conditions. Pileup around the indentation was clearly observed. The recovery process shows that the energy was stored in the material after the plastic deformation. It seems that the load at which yielding occurs may be independent of the temperature between room temperature and 388 K. As evident from the experiments, the rate of indentation also affects the modulus and the hardness value of the material. The modulus is high, and the penetration depth is less at lower temperatures.

This work is an initial and important step towards the study of materials' properties with respect to temperature at the nanoscale in order to predict better their behavior. Continuous research in this area will enable researchers to solve a large number of problems caused by materials due to unpredicted environmental conditions. Aluminum has a special importance due to its extensive increasing applications at present and in the future.

References

[1] A. Gouldstone, H. J. Koh, K. Y. Zeng, A. E. Giannakopoulos and S. Suresh, Discrete and continuous deformation during nanoindentation of thin films, *Acta Mater*. 48 (2000) 2277-2295.

[2] Roger Smith, D. Christopher and S. D. Kenny, Defect generation and pileup of atoms during nanoindentation of Fe single crystals, *Phys. Rev. B*, 67 (2003) 245405.

[3] D. F. Bahr, D. E. Wilson and D. A. Crowson, Energy considerations regarding yield Points during indentation, *J. Mater .Res*. 14 (6) (1999) 2269.

[4] J. A. Zimmerman, C. L. Kelchner, P. A. Klein, J. C. Hamilton and S. M. Foiles, Surface Step effects on nanoindentation, *Phys. Rev. Lett*. 87 (16) (2001) 165507-1.

[5] R. Komanduri, N. Chandrasekar and L. M. Raff, MD Simulation of indentation and scratching of single crystal aluminum, *Wear*, 240 (2000) 113-143.

[6] J. D. Kiely, J. F. Jarausch, J. E. Houston and P. E. Russell, Initial stages of yield in nanoindentation, *J. Mater. Res*. 14 (6) (1999) 2219.

[7] J. F. Smith and S. Zhang, High temperature nanoscale mechanical property measurements, *Surface Engineering*, 16 (2) (2000) 143-146.

[8] M. V. Swain and J. Mencik, Mechanical property characterization of thin films using spherical tipped indenters, *Thin Solid Films*, 253 (1-2) (1994) 204-211.

[9] Yu. I. Golovin and S. N. Dub, Stepwise transition from elastic to elastoplastic deformation at the initial stage of nanoindentation, *Doklady Physics*, 48 (2003) 612-614.

[10] I. Yonenaga, T. Hoshi and A. Usui, High Temperature hardness of Bulk Single Crystal GaN in comparison with other wide-gap materials *J. Phys.: Condens.Matter*, 12 (2000) 10319-10323.

[11] D. J. Branagan, Y. L. Tang, A. V. Sergueeva and A. K. Mukherjee, Low-temperature superplasticity in a nanocomposite iron alloy derived from a metallic glass, *Nanotechnology*, 14 (2003) 1216-1222.

[12] Xiao Dong Zhang, Temperature dependence of the structure and mechanical properties of TI-SI-N coatings, *ME Graduate student Conference*, 2003.

[13] High temperature Nanotesting, Micro materials measuring nanotechnology, www.http://freespace.virgin.net/micro.materials/HITEMP.HTMUT.HTM.

[14] S. Suresh, T. G. Nieh and B. W. Choi, *Scripta Materiala*, 41 (9) (1999) 1999.95-957.

[15] J. Fraxedas, S. Garcia Manyes, P. Gorostiza and F. Sanz, Nanoindentation: Toward the Sensing of atomic interactions, *PNAS*, 99 (2002) 5228.

Murugavel Rathinam received his Bachelor of of Engineering degree (B.E) in Mechanical Engineering from University of Madras, India in 1993. He then received his Master's of Engineering degree (M.E) in Engineering Design from the Government College of Technology in 1997 and his Ph.D. from The Hong Kong Polytechnic University, Hong Kong, in 2004. He served as a Senior Engineer (R&D) in TATA Engineering and Locomotive Company. He is a recipient of the International Fellowship

Award from the Hong Kong government. He was also awarded as excellent teacher. Dr. Rathinam is currently the Principal and a professor of Paavai Institutions, India. He has over 16 years of experience in the fields of teaching, research, and the industry. He has enormous international exposure in foreign universities in many countries, and he likewise serves as an editor and reviewer of international journals and books. Dr. Rathinam's interests include nanotechnology, design, mechanics, materials, solid state electronics, innovative projects, and methodologies.