Effect of residual chips on the material removal process of the bulk metallic glass studied by in situ scratch testing inside the scanning electron microscope

Cite as: AIP Advances 2, 042193 (2012); https://doi.org/10.1063/1.4774032 Submitted: 23 October 2012 • Accepted: 14 December 2012 • Published Online: 28 December 2012

Hu Huang, Hongwei Zhao, Chengli Shi, et al.





ARTICLES YOU MAY BE INTERESTED IN

A novel and compact nanoindentation device for in situ nanoindentation tests inside the scanning electron microscope AIP Advances 2, 012104 (2012); https://doi.org/10.1063/1.3676691

Note: A novel rotary actuator driven by only one piezoelectric actuator Review of Scientific Instruments **84**, 096105 (2013); https://doi.org/10.1063/1.4821495

Experimental research on a modular miniaturization nanoindentation device Review of Scientific Instruments **82**, 095101 (2011); https://doi.org/10.1063/1.3632980

AIP Advances



Mathematical Physics Collection

READ NOW

AIP Advances 2, 042193 (2012); https://doi.org/10.1063/1.4774032 Copyright 2012 Author(s)



Effect of residual chips on the material removal process of the bulk metallic glass studied by in situ scratch testing inside the scanning electron microscope

Hu Huang, Hongwei Zhao,^a Chengli Shi, Boda Wu, Zunqiang Fan, Shunguang Wan, and Chunyang Geng College of Mechanical Science & Engineering, Jilin University, Renmin Street 5988, Changchun, Jilin 130025, China

(Received 23 October 2012; accepted 14 December 2012; published online 28 December 2012)

Research on material removal mechanism is meaningful for precision and ultraprecision manufacturing. In this paper, a novel scratch device was proposed by integrating the parasitic motion principle linear actuator. The device has a compact structure and it can be installed on the stage of the scanning electron microscope (SEM) to carry out in situ scratch testing. Effect of residual chips on the material removal process of the bulk metallic glass (BMG) was studied by in situ scratch testing inside the SEM. The whole removal process of the BMG during the scratch was captured in real time. Formation and growth of lamellar chips on the rake face of the Cube-Corner indenter were observed dynamically. Experimental results indicate that when lots of chips are accumulated on the rake face of the indenter and obstruct forward flow of materials, materials will flow laterally and downward to find new location and direction for formation of new chips. Due to similar material removal processes, in situ scratch testing is potential to be a powerful research tool for studying material removal mechanism of single point diamond turning, single grit grinding, mechanical polishing and grating fabrication. Copyright 2012 Author(s). This article is distributed under a Creative Commons Attribution 3.0 Unported License. [http://dx.doi.org/10.1063/1.4774032]

I. INTRODUCTION

Precision and ultra-precision manufacturing has been given great attention in fields of semiconductor, aerospace, automotive, medical, optics, communications, micro-electromechanical systems and so on. Different manufacturing methods, such as single point diamond turning, precision grinding and mechanical polishing, are developing quickly in recent years. However, cost, efficiency and machinability of some difficult-to-machine materials still limit development of precision and ultra-precision manufacturing and its wide applications. One of the main reasons is that material removal mechanism during these manufacturing processes is not clear. Previous researchers have done a lot of work to study material removal mechanism. For example, material removal mechanism of precision and ultra-precision turning,^{1,2} grinding^{3–7} and polishing^{8,9} was investigated by the experiment method as well as the molecular dynamics simulation method,^{10,11} to some extent, improving manufacturing quality and efficiency, and also reducing cost. However, material removal mechanism is still an interesting and significant topic because it is not completely clear.

Scratch testing which is an effective method to characterize surface mechanical properties of materials, especially the thin films and coatings, has been used to study material removal mechanism because it has the similar removal process to single point diamond turning, precision grinding and mechanical polishing.^{12–15} W. B. Gu *et al.* studied characteristics of chipping behaviors and material



^aAuthor to whom correspondence should be addressed; E-Mail: hwzhao@jlu.edu.cn; Tel./Fax: +86-431-8509-4594



FIG. 1. The diagram of the self-made scratch device installed on the stage of the SEM.

removal mechanism during grinding via single and double scratch tests of optical glass BK7.¹² T. Sumitomo *et al.* investigated the feasibility of ultra-precision and nano-grinding machining operations by performing nanoscratching of the cross-section of an amorphous silicon thin film solar panel.¹³ These researches are meaningful to understand the material removal process, such as chip formation, crack formation and propagation, surface generation and damage. However, most of these researches are post-observation, which means that scratch testing first and then morphology observation. The real material removal process is not clear and some meaningful information during the scratch testing is lost.

In order to reveal material removal mechanism in depth, dynamic observation of the whole material removal process is required. So, in situ scratch devices which can be installed on the stage of the scanning electron microscope (SEM) were developed, and adhesion, wear and removal processes of materials were studied by researchers.^{16–20} Previous research results enhance understanding of wear and removal mechanism of materials.

In this paper, a compact and novel in situ scratch device was introduced and it has the ability to realize dynamic observation of the material removal process inside the SEM in real time. The long range scratch was realized by integrating the parasitic motion principle (PMP) linear actuator. More information about the PMP linear actuator can be found in reference 21. In order to keep the similarity between the scratch process and manufacturing processes of single point diamond turning, precision grinding and mechanical polishing, indenter surface ahead was selected instead of edge ahead to carry out scratch testing of the sample.

Formation, growth and flow of chips on the rake face of the Cube-Corner indenter during the scratch testing were observed and studied inside the SEM firstly. Then, emphasis was put on studying effect of residual chips on the material removal process of the bulk metallic glass during the scratch testing by in situ observation. Residual chips often exist during the manufacturing processes, which affect stress states of the tool and the sample surface and further affect formation and flow of chips as well as the surface quality of the sample. Study of effect of residual chips on the material removal process by in situ scratch testing will contribute to precision and ultra-precision manufacturing of materials.

II. EXPERIMENTAL SET-UP

Fig. 1 is the diagram of the self-made scratch device installed on the stage of the Zeiss SEM (EVO 18). The device has the dimensions of 167 mm × 130 mm × 53 mm, and the main frame is made of Aluminum 7075. The device mainly consists of five components—the z axis coarse positioner for coarse adjustment of the indenter, the z axis precision piezo actuator for fine penetration and withdraw of the indenter, the PMP linear actuator for y axis long range scratch up to several millimeters, the x axis precision piezo actuator for x axis scratch with the range of 12μ m, and the load and displacement sensors for measuring of the penetration load and depth. The PMP linear actuator can easily reach the large velocity over 40 μ m/s with the driving voltage of 100 V under a low driving frequency of



FIG. 2. The schematic diagram of the scratch testing.

5 Hz. Different velocities can be obtained by changing the driving voltages and driving frequencies. Also positive and negative movements along the *y* axis are available. The indenter axis has a tilt angle of 17 degrees to the surface of the SEM stage, proving a good observation angle for the sample surface. The Zr-based bulk metallic glass was adhered to the stage of the scratch device by the copper foil tape. More information about the sample can be found in reference 22 and 23.

Fig. 2 is the schematic diagram of the scratch testing, indicating the relative movement between the indenter and the sample surface. During the scratch testing, the indenter is motionless and the sample will move with the movement of the stage along the negative *y* axis. Instead of the commonly used Berkovich indenter, the Cube-Corner indenter with a curvature radius of about 150 nm was used because of its sharp tip which can improve the visualization of the scratch zone.

III. EXPERIMENTS

A. Dynamic observation of the material removal process

In situ scratch testing of the bulk metallic glass was carried out by the developed scratch device. The main processes were as follows. The pulse signal was sent to the stepper motor via the motor driver, and the stepper motor drove the indenter to close the sample surface. During this process, location of the indenter and the sample surface was observed via the SEM. When the indenter was near the sample surface, stop the stepper motor. The normal force was set by the software and the *z* axis precision piezo actuator drove the indenter to penetrate into the sample surface. The closed-loop control method was developed to keep constant of the normal load during the scratch testing. The driving frequency of 3 Hz and the driving voltage of 60 V were selected to drive the PMP linear actuator. During the whole process of the scratch testing, the contact zone was observed by the SEM in real time.

Fig. 3 is the main results obtained from the video recorded during the scratch testing. More information about the whole process can be found from the video 1 (BMG-in situ scratch inside the SEM-1.mpg). In order to describe the results conveniently, two regions, the region A and the region B, are defined. In Fig. 3(a), the indenter has penetrated into the sample surface and there are some chips around the indenter. In Fig. 3(b), the sample has moved a distance along the negative y axis and residual scratch appears. The chips in the region A of Fig. 3(a) remain where they are, but the chips in the region B of Fig. 3(a) residue on the surface of the indenter. Compared with Fig. 3(a) and Fig. 3(b), new chips present in Fig. 3(c) and Fig. 3(d) in the region B, forming the lamellar chips and accumulating on the surface of the indenter with the increase of scratch distance.

The lamellar chips grow bigger in the region B of Fig. 3(e). Comparing Fig. 3(e) and Fig. 3(d), another obvious difference is observed that chips present again in the region A of Fig. 3(e). The

AIP Advances 2, 042193 (2012)



FIG. 3. SEM images obtained from the video recorded during the scratch testing (enhanced online) [URL: http://dx.doi.org/10.1063/1.4774032.1].

formed chips in the region A of Fig. 3(e) residue on the surface of the residual scratch in Fig. 3(f). Just like Fig. 3(e), chips appear and grow again in the region A of Fig. 3(g) and Fig. 3(h). Also, the lamellar chips on the rake face of the indenter grow bigger. In Fig. 3(i), the long scratch forms behind the indenter.

In Fig. 3, lamellar chips are observed obviously during the scratch process. Possible reasons for formation of the lamellar chips here are as follows: 1) the step motion and the backward motion of the PMP linear actuator, which promote break of chips; 2) the Cube-Corner indenter with the negative rake; 3) the low thermal conductivity of the BMG; 4) formation of shear bands around the indenter; 5) the relatively low scratch speed; 6) dry scratching without lubrication; 7) surface defect and nonuniform of the BMG. Though the chips are lamellar, they can keep a whole and remain on the rake face of the indenter, indicating that adhesiveness of the BMG sample exists because of the local high temperature and press. The exact reason for the lamellar chips and adhesiveness will be studied in the future via more contrast tests. The lamellar chips were also post-observed after the conventional cutting of the BMGs by previous researchers.^{24,25} Dynamic observation of the material removal process inside the SEM will be a powerful tool for further studying the chip formation mechanism of the BMG during the cutting.

Fig. 4 is the SEM images before and after the indenter is away from the sample surface. More information about the whole process can be found from the video 2 (BMG-in situ scratch inside the SEM-2.mpg). Comparing Fig. 4(a) and Fig. 4(b), the conclusion can be obtained that most of lamellar chips remain on the surface of the indenter after the indenter has been away from the sample surface, which also indicates that adhesiveness of the BMG sample exists.



FIG. 4. SEM images before and after the indenter is away from the sample surface (enhanced online) [URL: http://dx.doi.org/10.1063/1.4774032.2].



FIG. 5. SEM images obtained from the video recorded, illustrating the formation and flow of new chips when lots of lamellar chips were accumulated on the rake face of the Cube-Corner indenter (enhanced online) [URL: http://dx.doi.org/10.1063/1.4774032.3].

B. Effect of residual chips on the material removal process

In Fig. 3(e), 3(g), and 3(h) and Fig. 4, chips appear in the region A when lots of lamellar chips were accumulated on the rake face of the indenter. So, the question is that what will happen when the indenter with lamellar chips as shown in Fig. 4(b) is used for the subsequent scratch testing. Formation and flow of new chips under this condition are studied in this paper. Following the previous scratch testing mentioned in section III A, a new scratch testing was carried out with the used indenter as shown in Fig. 4(b) and the scratch process was dynamically observed and captured by the SEM.

Fig. 5 gives some images obtained from the video recorded. More information about the whole process can be found from the video 3 (BMG-in situ scratch inside the SEM-3.mpg). From Fig. 5(a) to 5(f), the lamellar chips on the rake face of the indenter don't vary obviously. But two new chips are observed in the region A and the region B, and they grow and become bigger and bigger from Fig. 5(a) to 5(f) with the increase of scratch distance. In Fig. 5(f), the big and continuous chip with the length more than 10 μ m forms. Comparing the residual scratch behind the indenter in Fig. 3 and Fig. 5, formation of the continuous chip may be resulted from the shallow scratch depth, which agrees well with the result in reference 20. During the scratch process, the chip in the region A only becomes bigger and bigger but nearly don't change the direction, while the chip in the region B has variational directions with the increase of chip length. From the Fig. 5(a) to 5(c), the chip in the



FIG. 6. A diagram illustrating the effect of residual chips on the material removal process of the BMG.

region B has the trend to close the lamellar chips while the chip in the region B moves far away from the lamellar chips gradually from the Fig. 5(d) to 5(f).

From Fig. 5, the conclusion can be obtained that when lots of chips were accumulated on the rake face of the Cube-Corner indenter, formation location and flow direction of new chips will be changed. Fig. 6 is a diagram to illustrate the effect of residual chips on the material removal process of the BMG. On the one hand, accumulated chips obstruct formation and flow of new chips with the previous way, and fewer chips are continuously accumulated on the rake face of the indenter. On the other hand, new chip formation location and flow direction appear. As shown in Fig. 6(b), when large obstruction exists on the scratch direction, materials will flow laterally and downward to find new location and direction for formation of new chips.

From Fig. 4(b), Fig. 5 and Fig. 6, two suggestions can be given. Firstly, the indenter should be cleaned after one scratch testing especially when long scratch testing is carried out on the soft materials because of the residual chips, which will result in area and geometry errors during the subsequent indentation testing and scratch testing according to definition of indentation hardness and scratch hardness.^{26,27} Secondly, for precision and ultra-precision manufacturing, the tool should also be measured and cleaned after one manufacturing process. Otherwise, the tool radius will be changed because of the residual chips, and also residual chips will affect the manufacturing status, formation and flow of new chips, the tool life as well as surface quality of the sample. Currently, the above two points are not given much attention, but from the observed results, they should be paid more attention because residual materials often exist on surfaces of the indenter and the machining tool.

In addition, fluctuations of the lateral force were observed in conventional scratch testing,^{20,28} but up to now the reason is not very clear. From Fig. 4(b), Fig. 5 and Fig. 6, another possible reason can be given that residual chips affect the formation and flow of new chips. During the scratch process, chip accumulation, change of chip types, change of chip location and flow as well as chip exfoliation happen occasionally, which will change the material removal process of the subsequent scratch testing and further they will affect the lateral force. Effect of factors mentioned above on the lateral force will be studied when the new in situ scratch device with the function of quantitative measurement of the lateral force is developed in the future.

IV. CONCLUSIONS

In this paper, a novel in situ scratch device was presented to study the material removal process of the Zr-based BMG inside the SEM. Emphasis was put on studying effect of residual chips on the material removal process of the BMG. Via preliminary experiments and analysis mentioned above, conclusions can be obtained as follows:

(1) The parasitic motion principle linear actuator proposed in reference 21 is feasible for the application of in situ scratch testing inside the SEM.

042193-7 Huang et al.

- (2) Lamellar chips were formed and accumulated on the rake face of the Cube-Corner indenter and they can keep a whole during the scratch testing. When the indenter is away from the sample surface, the lamellar chips still remain on the surface of the indenter. These two points express adhesiveness of the BMG during the scratch testing.
- (3) When lots of chips are accumulated on the rake face of the Cube-Corner indenter and obstruct forward flow of materials, materials will flow laterally and downward to find new location and direction for formation of new chips.
- (4) Residual chips will change the status of the indenter or the machining tool, and further affect the material removal process. So, after one scratch testing, the indenter should be cleaned before a new indentation or scratch testing, especially when long scratch testing is carried out on the soft materials. And also, after one manufacturing process, the tool should also be cleaned. Otherwise, the testing results and the manufacturing quality will be affected because of residual chips on the surface of the indenter or tools.

This paper studied effect of residual chips on the material removal process of the Zr-based BMG by in situ scratch testing inside the SEM. More detailed and quantitative researches, such as effects of manufacturing parameters, tool parameters and manufacturing directions on manufacturing quality, effects of manufacturing parameters, tool parameters on the minimum manufacturing thickness, and so on, will be carried out by the developed in situ scratch device in the future.

ACKNOWLEDGMENTS

This research is funded by the National Natural Science Foundation of China (Grant No. 50905073, 51275198, 51105163), Special Projects for Development of National Major Scientific Instruments and Equipments (Grant No. 2012YQ030075), National Hi-tech Research and Development Program of China (863 Program) (Grant No. 2012AA041206), Key Projects of Science and Technology Development Plan of Jilin Province (Grant No. 20110307), and Graduate Innovation Fund of Jilin University (Grant No. 20121080).

- ¹ J. W. Yan, Z. Y. Zhang, and T. Kuriyagawa, Int. J. Mach. Tool Manu. 49, 366 (2009).
- ² F. Klocke, A. Demmer, and M. Heselhaus, Int. J. Mater. Prod. Tec. 20, 231 (2004).
- ³ T. T. Öpöz and X. Chen, Int. J. Mach. Tool Manu. 63, 31 (2012).
- ⁴H. Huang and Y. C. Liu, Int. J. Mach. Tool Manu. 43, 811 (2003).
- ⁵S. Agarwal and P. Venkateswara Rao, Int. J. Mach. Tool Manu. 48, 698 (2008).
- ⁶S. Agarwal and P. Venkateswara Rao, Int. J. Mach. Tool Manu. 50, 1077 (2010).
- ⁷Z. Y. Zhang, Y. W. Meng, D. M. Guo, L. L. Wu, Y. J. Tian, and R. P. Liu, Int. J. Adv. Manuf. Tech. 46, 563 (2010).
- ⁸ Y. Chen, L. C. Zhang, and J. A. Arsecularatne, Int. J. Mach. Tool Manu. 47, 1615 (2007).
- ⁹A. Vijayakumar, T. Du, K. B. Sundaram, and V Desai, Microelectron. Eng. 70, 93 (2003).
- ¹⁰ B. Lin, S. Y. Yu, and S. X. Wang, J. Mater. Process. Tech. **138**, 484 (2003).
- ¹¹X. S. Han, Y. Z. Hu, and S. Y. Yu, Appl. Phys. A **95**, 899 (2009).
- ¹² W. B. Gu, Z. Q. Yao, and X. G. Liang, Wear **270**, 241 (2011).
- ¹³T. Sumitomo, H. Huang, and L. B. Zhou, Int. J. Mach. Tool Manu. **51**, 182 (2011).
- ¹⁴ B. Denkena, J. Koehler, and A. Moral, J. Mater. Process. Tech. **210**, 1827 (2010).
- ¹⁵ R. L. Kobrick, D. M. Klaus, and K. W. Street, Jr., Wear **270**, 815 (2011).
- ¹⁶ S. V. Prasad and T. H. Kosel, J. Mater. Sci. Lett. **3**, 133 (1984).
- ¹⁷ P. Hedenqvist, M. Olsson, and S. Jacobson, Surf. Coat. Technol. 41, 31 (1990).
- ¹⁸ P. Nledengvist and S. Hogmark, Tribol. Int. **30**, 507 (1997).
- ¹⁹ M. Yoshino, T. Aoki, T. Shirakashi, and R. Komanduri, Int. J. Mech. Sci. 43, 335 (2001).
- ²⁰ J. Michler, R. Rabe, J.-L. Bucaille, B. Moser, P. Schwaller, and J.-M. Breguet, Wear **259**, 18 (2005).
- ²¹ H. Huang, H. W. Zhao, Z. J. Yang, J. Mi, Z. Q. Fan, S. G. Wan, C. L. Shi, and Z. C. Ma, Rev. Sci. Instrum. 83, 055002 (2012).
- ²² L. Y. Chen, Z. Xue, Z. J. Xu, J. Q. Chen, R. X. He, X. P. Nie, Q. P. Cao, X. D. Wang, S. Q. Ding, and J. Z. Jiang, Adv. Eng. Mater. 14, 195 (2011).
- ²³ H. Huang, H. W. Zhao, Z. Y. Zhang, Z. J. Yang, and Z. C. Ma, Materials 5, 1033 (2012).
- ²⁴ M. Bakkal, A. J. Shih, and R. O. Scattergood, Int. J. Mach. Tool. Manu. 44, 915 (2004).
- ²⁵ M. Q. Jiang and L. H. Dai, Acta Mater. **57**, 2730 (2009).
- ²⁶ W. C. Oliver and G. M. Pharr, J. Mater. Res. 7, 1564 (1992).
- ²⁷ S. Graça, R. Colaço, and R. Vilar, Tribol. Lett. 31, 177 (2008).
- ²⁸ J. G. Wang, B. W. Choi, T. G. Nieh, and C. T. Liu, J. Mater. Res. 15, 913 (2000).