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Pileup behavior in sharp nanoindentation of AISI 1045 steel

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Abstract

Experimental measurements have been used to investigate the pileup behavior during nanoindentation with a sharp indenter. The AISI 1045 steels treated by quenching and annealing were examined. The results show that during sharp nanoindentation process, the amount of pileup is related to the residual stress state, the indentation depth and the work hardening. The quenched steel with compressive residual stress will tend to pile up, and the stress-free annealed steel can decrease the pileup height. It is found that the pileup height gradually increases for the two steels as the indentation depth becomes larger. It is also shown that the low work hardening of the two steels can also result in the pileup deformation.

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Keywords: Pileup; Nanoindentation; Atomic force microscopy

1. Introduction

Nanoindentation testing has widely been used as an attractive means to estimate mechanical properties of very different kind of materials. Oliver and Pharr (2004) method is one of the more commonly used methods to analyze nanoindentation load-displacement data. The attractiveness of this approach is that the hardness and elastic modulus can be directly obtained from load-displacement data without the need to image the hardness impression. The hardness and elastic modulus rely heavily on an accurate determination of the contact area. However, it should be noted that the Oliver and Pharr method is based on a purely elastic contact solution derived by Sneddon (1965), and the elastic solution fails to properly describe the pileup behavior of materials around the indenter. When pileup is large, the contact areas deduced from analyses of the load-displacement curves underestimate the true contact areas

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by as much as 60% (Bolshakov et al., 1998), which leads to overestimations of the hardness and elastic modulus. Therefore, it is important to study the pileup behavior of materials.

The pileup behavior in spherical indentation has systemically been researched (Taljat et al., 2004; Rodríguez et al., 2007; Ahn et al., 2001). However, the pileup deformation during sharp indentation is not thoroughly understood. On the other hand, the researches are based on the finite element simulation but not on experiments. The aim of this paper is to provide great detail about the pileup behavior in sharp nanoindentation of AISI 1045 steels treated by different heat processing. In addition, the influence factors on pileup were discussed.

2. Experimental

Specimens used in the present study were cut from a commercial 1045 steel bar, and then machined into samples of dimensions 25 × 15 ×8 mm3. The specimens were quenched at 840 ℃. Some of them were annealed at 650 ℃ to relieve any pre-existing residual stress. All the specimens were polished with 0.5 μm diamond polishing paste, producing a surface roughness of 14.5±3.5nm.

Nanoindentation tests were made by employing TriboIndenter system (Hysitron, Inc) equipped with a Berkovich diamond pyramid indenter which was also used as an atomic force microscopy (AFM) tip and the indented surface was imaged after indentation. All images were collected with a resolution of 256 × 256 pixels, and taken in a 10 μm × 10 μm scan area with a scan rate of 0.5 Hz. The accuracy of load and displacement were 0.1 μN and 0.1 nm, respectively. For quenched and annealed steels, the indenter loads were 3 mN, 5 mN, 7 mN and 9 mN. 3×3 array indents were performed at each peak load. Indentations tests were performed under the constant temperature of 20 ℃.

3. Results and discussion

The load-depth curves for quenched and annealed steels at different peak loads. The resulted loading curves at different peak loads exhibits good reproducibility, which shows the repeatability of the experiments (Zong et al., 2006). For nanoindentation testing, the repeatability of the experiments is very important. If the repeatability of the experiments is poor, the obtained data will be dispersive and difficult to be analyzed. The good repeatability of the experiments in the present study was attributed to the homogeneous of composition and few defects of quenched and annealed steels. Corresponding to the load range between 3 and 9 mN, the indent depths for quenched and annealed steels are 120-245 nm, and 130-260 nm, respectively.

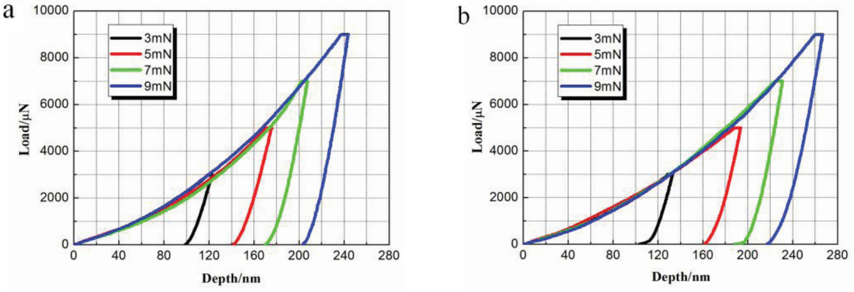

Fig. 1. Load-depth curves for quenched and annealed 1045 steels at different peak loads (a) quenched 1045 steel; (b) annealed 1045 steel

Fig. 2 shows AFM morphologies of pileup in quenched and annealed 1045 steels at a peak load of 9mN. The two steels exhibit similar morphologies at the other peak loads. It is obvious that significant pileup occurs in both two steels. In addition, it can be observed that the materials pile up mainly along the flat faces of the indents, and not the corners.

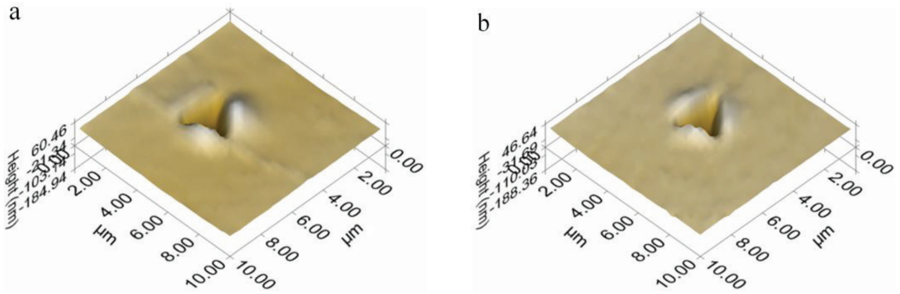

Fig. 2. Materials pileup in quenched and annealed steels at a peak load of 9mN (a) quenched 1045 steel; (b) annealed 1045 steel

Fig. 3 and Fig. 4 are AFM profile images of indents at different peak loads for the quenched and annealed steels. The left hand side of each indent profile represents the indent corner, while the right hand side shows the midpoint of the pileup. The amount of pileup can be characterized by the height of the pileup relative to the undeformed surface. Fig. 3 and Fig. 4 also demonstrate that almost no pileup occurs at the corners of the indents. As the load on the indenter is increased, namely, the indentation depth increases, the pileup height for the two steels both becomes larger. The pileup height of the quenched steel ranges between 19 and 36 nm, and that of the annealed steel ranges between 15 and 30 nm. The pileup height of the former is larger than that of the latter at each peak load. It is clear that annealing treatment can decrease pileup height. Note that the pileup height of the two steels accounts for a large proportion in the indentation depth and should be taken into account to obtain accurate measurement.

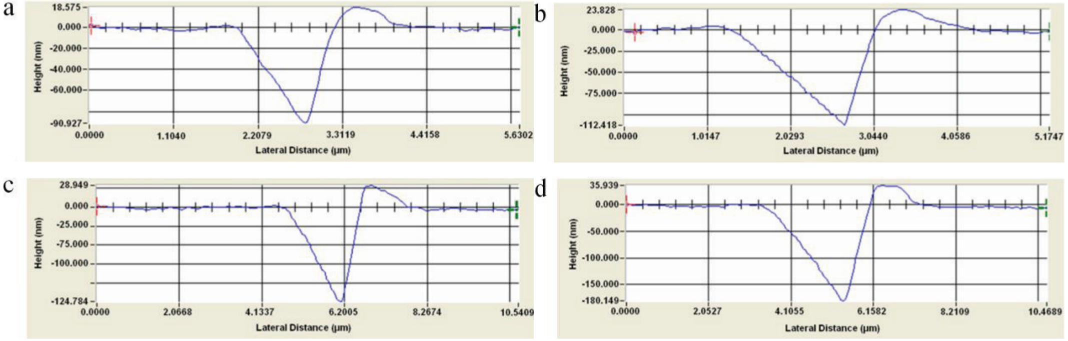

Fig. 3. AFM profiles of indents at different peak loads for quenched 1045 steel (a) 3mN; (b) 5mN; (c) 7mN; (d); 9mN

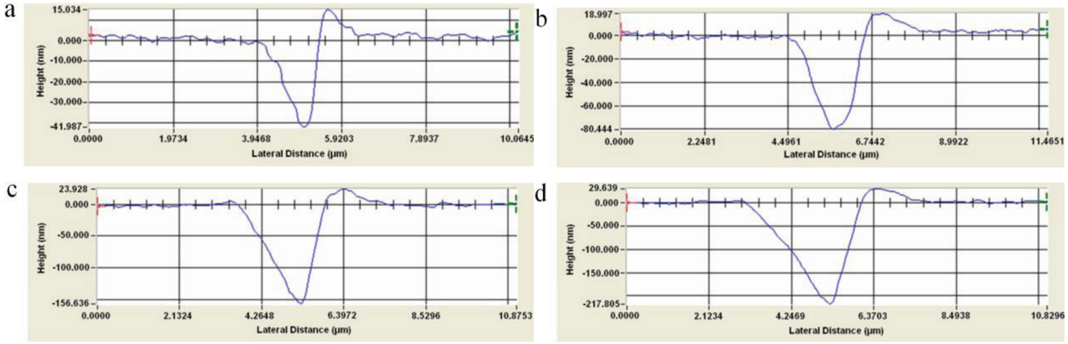

Fig. 4. AFM profiles of indents at different peak loads for annealed 1045 steel(a) 3mN; (b) 5mN; (c) 7mN; (d); 9mN

Pileup deformation is affected by residual stress in materials (Xu et al., 2005; Chen et al., 2006; Khan et al., 2011). For compressive residual stress, the plastic zone is more defined near the impression crater, where the materials are pushed out to the surface of indenter, which increases the pileup of materials around the indenter; whilst tensile residual stress will tend to pull the materials away from the surface of indenter and thus decrease the amount of pileup. In the present study, compressive residual stress was generated on the surface of quenched 1045 steel (Zhu et al., 2010). Therefore, a significant pileup was observed. For annealed 1045 steel, any pre-existing residual stress was released completely, which results in a decrease in the height of pileup compared with the quenched steel. It is noted that even the annealed steel with zero stress there was pileup around the indents, which indicated that pileup is generated due to combined effects not only the residual stress. Moreover, pileup deformation also depends upon work hardening. After polishing, the quenched and annealed steels slightly work hardened. During indentation, the materials around the indenter were not hard enough to inhibit materials flowing upwards, which lead to pileup deformation.

For spherical indentation, the pileup behavior depends on the normalized depth of penetration, i.e. the ratio of indented depth to indenter radius, the ratio of the modulus to the yield strength, the work hardening, and the contact friction between the indenter and materials (Taljat et al., 2004). For the sharp indentation in the present study, the penetration depth and work hardening also affect the pileup behavior. Concretely, large penetration depth and low work hardening will increase the pileup amount. However, further study should be made to understand whether the ratio of the modulus to the yield strength and the contact friction between the indenter and materials produce an effect on the pileup behavior.

4. Conclusions

Experimental tests have been carried out to investigate the effect of residual stress, indentation depth and work hardening on the pileup behavior of AISI 1045 steel during sharp nanoindentation. It has been found that pileup occurs both in the quenched steel with compressive residual stress and stress-free annealed steel. After annealing, the pileup height can be reduced. The pileup also depends on the indentation depth. As the indentation becomes larger, the pileup height increases. Furthermore, pileup deformation is also attributed to the low work hardening of the quenched and annealed 1045 steels. Collectively, these observations show that pileup is complex phenomenon which is still to be further studied.

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