SIZE EFFECT CHARACTERIZATION FOR NANOSTRUCTURED MATERIAL IN NANOINDENTATION TEST

Yueguang Wei, Ying Du, Siqi Shu, Chen Zhu
LNM, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, P.R. China

ABSTRACT

Based on the microscopic observations and measurements, the mechanics behaviors of the nanostructured material (the surface-nanocrystallized Al-alloy material) at microscale are investigated experimentally and theoretically. In the experimental research, the hardness-indent depth curves or relations are measured by using both the method of randomly selecting loading points on the specimen surface and the continuous stiffness method. In the theoretical simulation, based on both the material microstructure characteristics and the experimental features of the nanoindentation, the microstructure cell model is developed and the strain gradient plasticity theory is adopted. The material hardness-indent depth curves are predicted and simulated. Through comparison of the experimental results with the simulation results, the material parameters and the model parameters are determined.

INTRODUCTION

Resent researches have displayed that the high-strength nano-structured materials can be fabricated by using some advanced techniques. For example, by using the severe plastic deformation (SPD) method, one can fabricate the nanocrystalline materials [1-3]. The adopted SPD methods include the large torsion method [1], the large pressing method [2] and the ultrasonic shot peening (USP) method [3], etc. Usually, the microstructure cell size is from tens to hundreds of nanometers, even to microns. Within this length scale, solids use to display a strong size effect. On the research of the size effect, many investigators have focused their attention on the nanoindentation problems for single crystal metals. Through theoretical and experimental researches for the nanoindentation size effect, one has found that as the indent depth decreases, the hardness curve displays a obviously going up trend, i.e., the size effect. The size effect was described by using the strain gradient theories [4,5], and the simulation results were consistent with the experimentally measured results. The researches have showed that the microscale length parameter for single crystal metals is in the order of one micron [4, 5]. However, the nanocrystalline material case investigated here will be very complicated, about which besides the size effect mentioned above, additionally the influences of both the crystal grain size and shape distributions on the material behaviors must be considered. In the author's previous research on the nano-polycrystal Al and the thin film/substrate system [6], the effect of both the crystal grain size and the shape distribution was called the geometrical effect for differing from the size effect described only by a single microscale parameter in strain gradient theories. Based on the microstructure cell model and the strain gradient plasticity theory, the size effect and geometrical effect have been studied. Through comparing the predicted results to the experimental results, the effects of both grain size and the microstructure characteristics on the microscale length parameter of the strain gradient theory have been obtained [6].

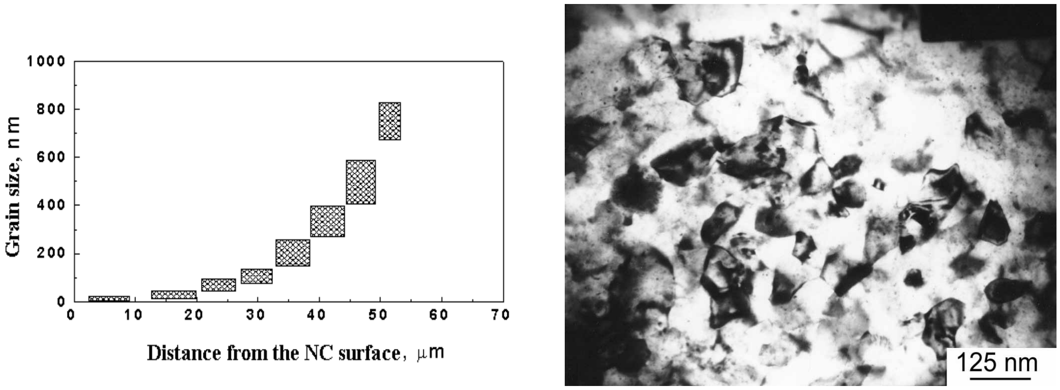
In the present research, the mechanics behavior of the surface-nanocrystalline Al-alloy material (SNCAA) fabricated by using the USP method [3] will be studied experimentally and theoretically based on the microscopic observation, measurement and analysis. In experimental research, the specimens will be designed and prepared according to the microstructure features of the material. The nanoindentation experiment will be carried out to measure the hardness-depth curves. In theoretical research, the corresponding microstructure cell models will be developed and the strain gradient theory will be used to simulate the experiments. The hardness curves will be obtained. Through comparing the experimental results with the theoretical simulation results, both material microscale parameter and the model parameters will be attained.

NANOINDENTATION EXPERIMENTS FOR THE SNCAA MATERIAL

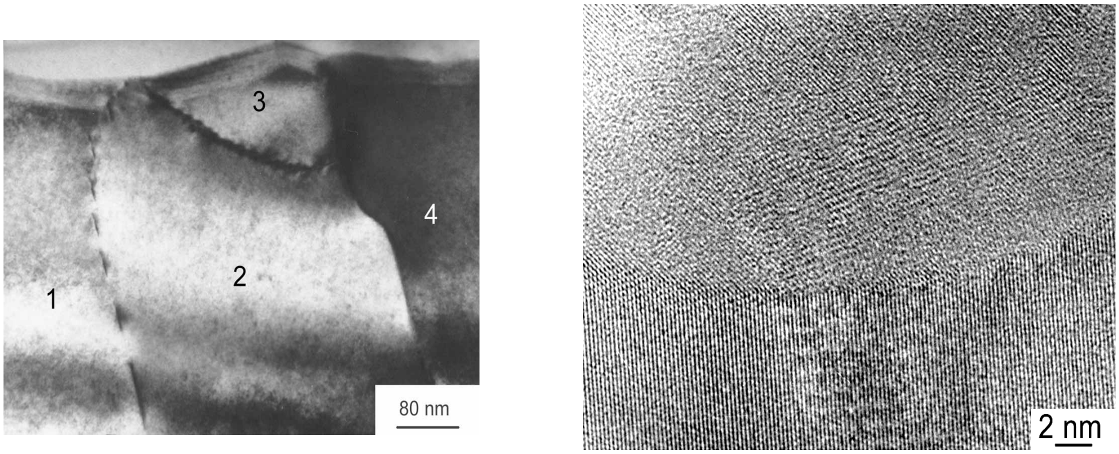
Through microscopic observation and analysis for the SNCAA material, the specimens of nanoindentation test, through which the material microscale effects should be reflected, are designed. Photos of the higher-order resolution electron microscopy and TEM about the SNCAA material fabricated by using the USP method are shown in figure 1. The fabrication was performed in the Metal Institute, Chinese Academy of Sciences. From figure 1, a regular nanocrystal structure near the surface layer after the nanocrystallization is formed. Figure 1(a) shows the statistical results of the grain size distribution. The sizes of the nanocrystal structures increase as the distance from the nanocrystalline surface increases. The TEM photo of nanocrystal grains near surface is shown in figure 1(b). From figure 1(b), by using the USP method, the regular nanocrystal grains near the material surface are formed. Figure 1(c) shows the formed sub-crystal grains at 30 microns from surface. Differing from the conventional polycrystal material, the nanocrystal grain boundary is a thin layer structure with the certain thickness, as shown in figure 1(d), by a higher-resolution electron microscopy photo.

In the present research, the specimens were cut vertically to the nanocrystalline surface. On the cross-section, the nanoindentation experiments were performed within a region from 30 to 60 microns to the nanocrystalline surface, where the grain sizes are from 100 nanometers to 1 micron (submicron region). Consider that when the indenter tip locates at a grain region, and when the horizontal size of the contact zone is smaller than the horizontal size of the indented grain, the plastic slip zone will be constrained within the grain region due to the grain boundary constraint.

Nanoindentation experiments are carried out at the State Key Laboratory of Friction and Lubrication, Tsinghua University, and at the Metal Institute, Chinese Academy of Sciences, respectively. The instrument of the former is CSM-Nanoindenter (made in Switz). The nanoindentation test method is the discrete point selection method [7]. The instrument of the latter is MTS-Nanoindenter-XP (made in USA). The nanoindentation test method is the continuous stiffness method, i.e., the hardness-depth curve is measured through continuously indenting at a fixed point.



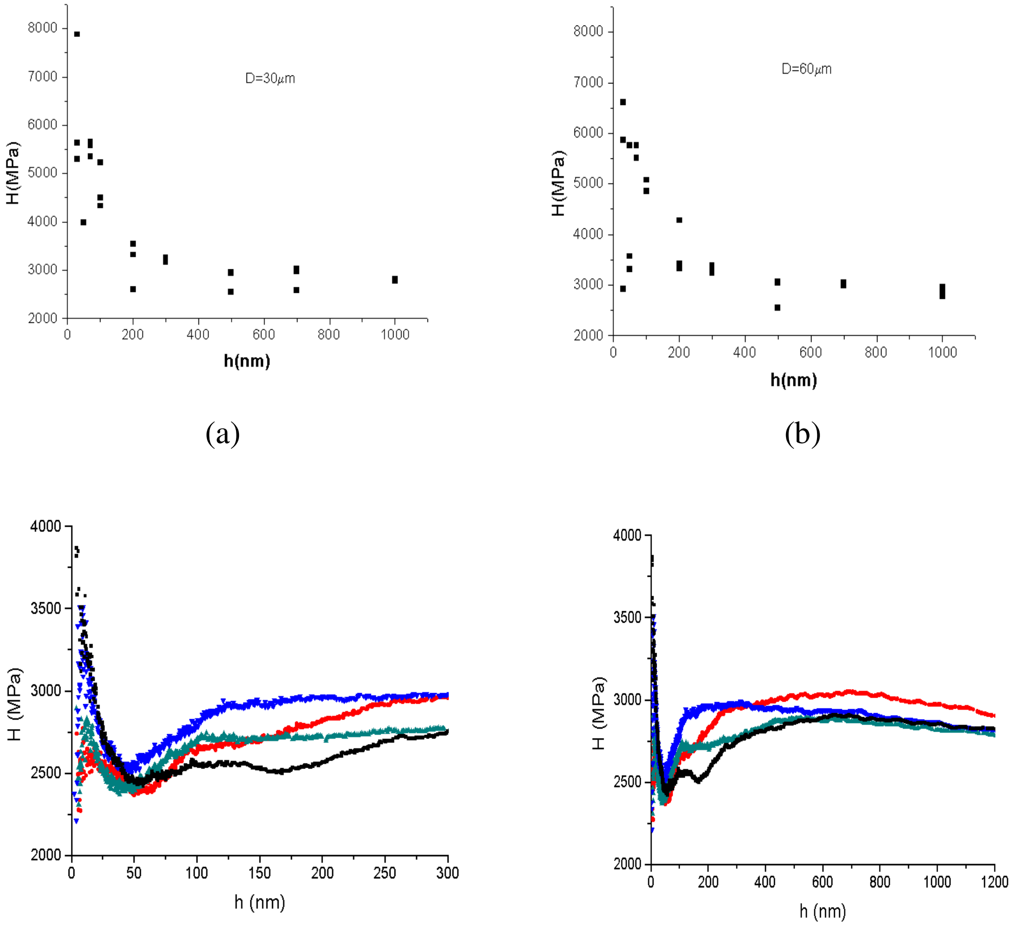
(a) Grain size distribution (b) The formed grains by the nanocrystallization



(c) The formed sub-crystal grains (d) The profile of the grain boundary zone

Figure 1. Microstructure features of the nanocrystalline Al-alloy material.

By randomly selecting the loading points on the specimen surface and carrying out the nanoindentation test, the obtained hardness-depth relations are shown in figure 2(a) and (b), where D is the distance from selected indent point on the specimen surface to the nanocrystalline surface. Two hardness relations corresponding different distance D are given in figure 2(a) and (b). From figure, the difference between the measured hardness-depth relations at two distances is small. This implies that the differences among the nanocrystalline grain sizes within that region are small. From figure 2(a) and (b), the hardness value sharply increases as indent depth decreases when the indent depth is smaller than 200 nanometers, i.e., size effect phenomenon is obvious. When the indent depth is larger than about 600 nanometers, the hardness value decreases slowly as indent depth increases. However, the hardness value at 600 nanometers of indent depth is still far higher than that of the conventional Al-alloy material. Figure 2(c) and (d) show the measured hardness-depth curves by using the continuous stiffness method at the several loading points for D=60 microns. Figure 2(c) and (d) correspond to the experimental curves for two different coordinate scales, respectively. From figure 2(c) and (d), although the curves correspond to the different loading points on the specimen surface, the measured results have the similar feature, which implies that the tests are repeatable. Comparing figure 2(c) and (d) with figure 2(a) and (b), clearly both results by adopting two kinds of indentation test methods have a big difference. From figure 2(c) and (d), as indent depth decreases, especially, when indent depth is smaller than 100 nanometers, the variation law of the hardness curves is much complicated. When the indent depth is smaller than 50 nanometers, the results display a strongly going up trend. When indent depth is larger than 400 nanometers and as the indent depth increases, both hardness results by adopting two kinds of the indentation test methods tend to consistent with each other.



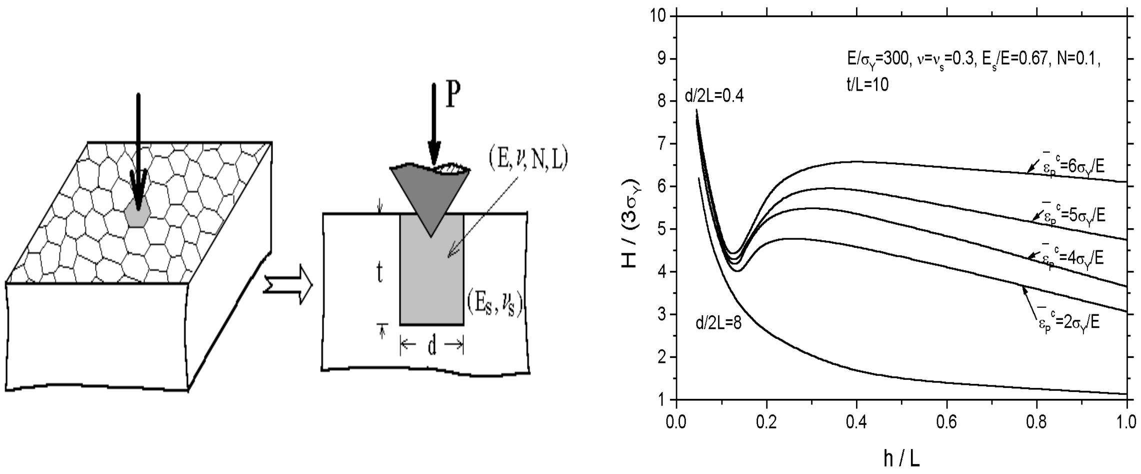
(a) (b)

(c) (d)

Figure 2. Hardness-indent depth relations measured by using the discrete selection points method ((a), (b)) and the continuous stiffness method ((c), (d)).

SIMULATIONS OF SIZE EFFECT AND GEOMETRICAL EFFECT FOR THE SNCAA MATERIAL

Since the size effect can not be simulated by using the conventional elastic-plastic theory, here the strain gradient plasticity theory combined with the microstructure cell models will be adopted to model and simulate the hardness-depth curves for the SNCAA material. Fleck-Hutchinson's strain gradient plasticity theory [4] will be adopted here. In the fundamental relations of the strain gradient plasticity theory, an additional length-scale parameter, L (SG theory), is included compared with the conventional elastic-plastic theory. At the macroscale, the strain gradient effect is very weak and can be neglected, however at the microscale (micron or sub-micron), this effect is important. Usually L is called the microscale parameter.



(a) (b)

Figure 3. Microstructure cell model for the nanoindentation test (a) and hardness-depth simulation results of the SNCAA material (b).

A simplified model for the nanoindentation test of the polycrystals is presented, as shown in figure 3(a). The indented grain region is treated as elastic-plastic solid governed by strain gradient theory. The region the other grains occupy can be treated as an equivalent uniform and isotropic elastic body. According to self-consistent theory [8], one can find the Young's modulus and Poisson's ratio of the equivalent elastic body. The obtained Young's modulus of the equivalent elastic body is 0.671 times the average Young's modulus of single crystal Al-alloy material. From previous research, the size effect result was not sensitive to the ratio value [6]. For the constrained plastic slip deformation of the grain of the conventional metals, the grain can be approximately treated as the uniform and isotropic elastic-plastic material, simultaneously the strain gradient effect is considered. Differing from the previous case [6], the grain size along the indenting direction is large, and the extension of plastic slip zone is mainly constrained by grain boundaries on two side faces of grain. In the simplified model, besides the grain size and the micro-scale parameter L, another parameter needs to be introduced in order to describe the condition of the plastic slip zone across the grain boundary. Here we consider that when effective plastic strain at grain boundary is over a critical value, $\bar{\varepsilon}_P^c$, the plastic zone will penetrate the grain boundary. The condition of plastic slip zone across the grain boundary can be expressed as

$$\bar{\varepsilon}_P \,|_{\Gamma} = \bar{\varepsilon}_P^c \qquad (1)$$

where Γ stands for the grain boundary.

From the microstructure cell model (see figure 3(a)), the fundamental relations of the strain gradient theory [4,7], as well as the condition of the plastic slip zone across the grain boundary (see formula (1)), the independently parametrical relation of hardness depth can be dictated as

$$\frac{H}{3\sigma_Y} = g(\frac{h}{L};\ \frac{d}{2L}, \frac{t}{L}, \frac{E}{\sigma_Y}, \frac{E_s}{E}, \nu, \nu_s, N, \bar{\varepsilon}_P^c) \qquad (2)$$

The parametrical relation (2) can be obtained in detail by using the finite element numerical simulation. The detailed discussion of the finite element method is given in [9, 10]. Figure 3(b) shows the simulated hardness-depth curves by adopting the microstructure cell model and the strain gradient theory, corresponding to several strength values of the plastic zone across the grain boundary, $\bar{\varepsilon}_P^c$, and $E_s/E=0.67$ from the self-consistent analysis. When the critical value of the effective plastic strain, which describes the grain boundary strength, varies from twice to six times the yield strain, the predicted hardness curves have the similar characteristics with the experimentally measured curves (compare figure 3(b) to figure 2(c) and (d)). Obviously, the theoretical model captures the fundamental features of the problem. If take as L=0.5 micron in figure 3(b) and consider the yield strength $\sigma_Y = 200$ Mpa, the theoretical simulation results in figure 3(b) are consistent with the experimental results in figure 2(c) and (d). Figure 3(b) also shows the result when grain size is very large, i.e., for the case of d/2L=8, i.e., the extension of plastic slip zone is not constrained as the indent depth increases. This result describes the case of the pure size effect.

CONCLUSIONS

The mechanics behaviors of the SNCAA material fabricated by using the USP method have been investigated from both experimental and theoretical respects. The experimental results have shown that with the nanocrystallization of the grain size, the mechanics properties of the Al-alloy polycrystal material have been improved considerably. The ascents of hardness curves are the prominent manifestation of the improvement in mechanics property. In the theoretical research, the hardness curves have been modeled, and the results are basically consistent with experimental measurements. Through comparing the experimental results with the simulation results, the microscale length parameter included in the strain gradient theory and model parameters have been predicted.

ACKNOWLEDGEMENTS

This work was supported by the National Natural Science Foundation of China (Grant No. 19925211), and jointly supported by "Bai Ren Plan" of Chinese Academy of Sciences.

REFERENCES

1. Valiev RZ, Korznikov AV, Mulyukov RR. *Mater. Sci. Eng.*, **A168**, 141(1993).
2. Valiev RZ, Islamgaliev RK, Alexandrov IV. *Prog. in Mater. Sci.*, **45**, 103(2000).
3. Lu K, Lu J. *J. Mater. Sci. & Tech.*, **15**, 193(1999).
4. Fleck NA, Hutchinson JW. *Adv. in Appl. Mech.*, **33**, 295(1997).
5. Gao H, Huang Y, Nix WD, Hutchinson JW. *J. Mech. Phys. Solids*, **47**, 1293(1999).
6. Wei Y, Wang X, Zhao M, Cheng CM, Bai Y. *Acta Mechanica Sinica*, **19**, 59(2003).
7. Wei Y, Wang X, Wu X, Bai Y. *Science in China (Series A)*, **44**, 74(2001).
8. Hill R. *J. Mech. Phys. Solids*, **13**, 89(1965).
9. Wei Y, Hutchinson JW. *J. Mech. Phys. Solids*, **45**, 1253(1997).
10. Chen JY, Wei Y, Huang Y, Hutchinson JW, Hwang KC. *Eng. Fracture Mech.*, **64**, 625(1999).