Micro-scale mechanics of the surface-nanocrystalline Al-alloy material

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Abstract Based on the microscopic observations and measurements, the mechanical behavior of the surface-nanocrystallized Al-alloy material at microscale is investigated experimentally and theoretically. In the experimental research, the compressive stress-strain curves and the hardness depth curves are measured. In the theoretical simulation, based on the material microstructure characteristics and the experimental features of the compression and indentation, the microstructure cell models are developed and the strain gradient plasticity theory is adopted. The material compressive stress-strain curves and the hardness depth curves are predicted and simulated. Through comparison of the experimental results with the simulation results, the material and model parameters are determined.

Recent researches show that the high strength nano-structured materials can be fabricated by using some advanced techniques, and the mechanical behavior of the conventional materials can be improved by using a surface-nanocrystalline technique. For example, by using the severe plastic deformation (SPD) method, one can fabricate the nanocrystalline materials[1—5]. The adopted SPD methods mainly include the large torsion method[1], the large pressing method[4] and the ultrasonic shot peening (USP) method[5], etc. The construction features of both the nano-structured block materials and the surface-nanocrystalline materials have been displayed as that they consist of the regular microstructures overall or locally. Usually, the microstructure cell is from tens to hundreds of nanometers, even to microns. Within this length scale, solids used to display a strong size effect. As for the research on the size effect, many and deep investigations are the nanoindentation tests for single crystal metals. Theoretical and experimental researches into the microindentation test show that as the indent depth decreases, the hardness curve displays an obviously going up trend, i.e. the size effect. The size effect was described by using the strain gradient theories[6—9], and the simulation results were

consistent with the experimentally measured results. The researches have shown that the microscale parameter for single crystal metal materials is in the order of microns[10, 11]. However, the nano-crystalline material case investigated here will be very complicated, about which besides the size effect mentioned above, the influences of both the crystal grain size and shape distributions on the material behavior must be considered. In the authors' previous research on the nano-polycrystal Al and the thin film/substrate system[11], the effect of both the crystal grain size and the shape distribution was called the geometrical effect in order to distinguish it from the size effect described only by the microscale parameter of the 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 with the experimental results, the effects of both grain size and the microstructure characteristics on the microscale parameter of the strain gradient theory have been obtained[11].

Up till now, there are few reports on the mechanical behavior of the surface-nanocrystalline material. In ref. [12], by means of the microscopic analysis, the material microstructure features were displayed and the forming mechanism of nanocrystal was interpreted from dislocation motion. Based on the molecular dynamics simulation a relation between the dislocation density and the nanocrystal grain size was obtained[13]. In the present research, the mechanical behavior of the surface- nanocrystalline Al-alloy material (SNCAA) fabricated by using the USP method[5] 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 unidirectional compressive experiment and the nanoindentation experiment will be used to measure the compressive stress-strain curves (overall average mechanical behavior) and the hardness-depth curves (local mechanical behavior). In theoretical research, the corresponding microstructure cell models will be developed and the strain gradient theory will be used to carry out the simulations for both experiments. The material compressive stress-strain curves and the hardness curves will be simulated. Through comparing the experimental results with the theoretical simulation results, both the material microscale parameter and the model parameters will be attained. Finally, through experimental research and the theoretical simulation for the SNCAA material, the mechanical behavior will be displayed for the grain size variations from 100 nanometers to tens of microns.

1   Experiments of mechanical behavior for the SNCAA material

First, through microscopic observation and analysis of the SNCAA material, the specimens of the compressive test and nanoindentation test, by which the material microscale effects must be described, are designed. Second, the compressive experiments and the nanoindentation experiments are performed, and the compressive stress-strain curves and the hardness-depth curves are measured.

1.1   Observation and analysis of the SNCAA microstructure

Photos of the higher-resolution electron microscopy and TEM about the SNCAA material fabricated by using the USP method are shown in fig. 1. The fabrication was performed in the Institute of Metal Research, Chinese Academy of Sciences[14]. The comparison of the vertical cast figures of the material microstructures before and after the nanocrystallization is given in fig. 1(a). The left half of the figure corresponds to the case before the nanocrystallization, while the right half of the figure corresponds to the
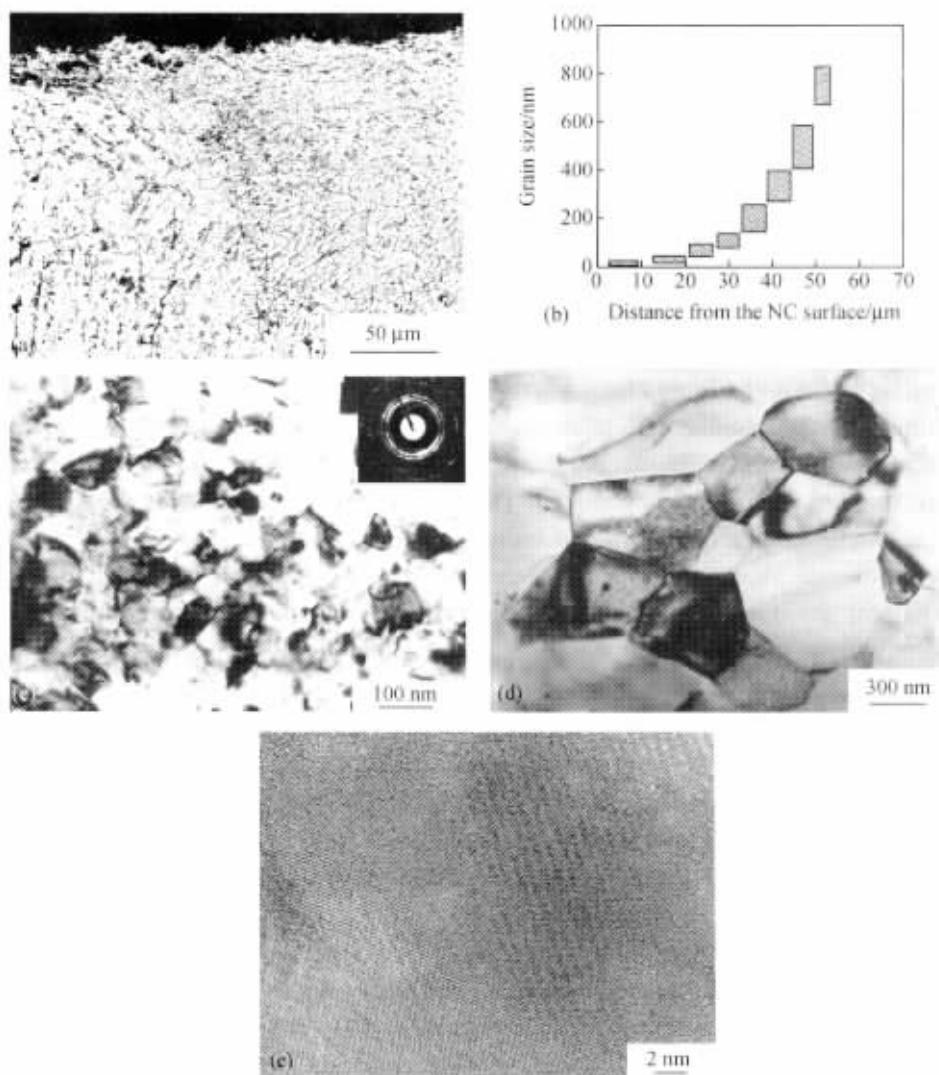

Fig. 1.   Microstructure features of the nanocrystalline Al-alloy material. (a) Comparison of microstructure features; (b) grain size distribution; (c) the formed grains by the nanocrystallization; (d) the formed sub-crystal grains; (e) the profile of the grain boundary zone inside the nanocrystals.

case after the nanocrystallization. Obviously, a regular nanocrystal structure near the surface layer after the nanocrystallization is formed and the sizes of the nanocrystal structures increase as the distance from the nanocrystalline surface increases. Fig. 1(b) shows the statistical results of the grain size distribution. The TEM photo of nanocrystal grains near the surface is shown in fig. 1(c). By using the USP method, the regular nanocrystal grains near the material surface are formed (fig. 1(c)). Fig. 1(d) shows the formed polygon sub-crystal grains at 30 microns from the surface. Differing from the conventional polycrystal material, the nanocrystal grain boundary is a thin layer structure with a certain thickness, as shown in fig. 1(e), a higher-resolution electron microscopic photo.

1.2 The compressive stress-strain curves of the SNCAA material

It is expected that the specimen design and preparation for a compressive test can describe the SNCAA material characteristics. For this reason, two types of specimens were designed. One type covers the influenced region of the nanocrystalline process, while the other one corresponds to the specimens uninfluenced by the nanocrystalline process. Two kinds of specimens, specimen 1 and specimen 2, were cut in turn from nanocrystalline surface to material inside, as shown in fig. 2. Note that fig. 2 is only a sketch, and is only used to interpret the relative location of both specimen 1 and specimen 2. Since each specimen is 1 mm thick and its shape is a cube in 1 mm × 1 mm × 2 mm, actually, specimen 2 is not influenced by the nanocrystalline process at all. However, it is interesting to compare the experimental result of specimen 1 (influenced by the nanocrystalline process) with the experimental result of specimen 2 (uninfluenced by the nanocrystalline process). Each specimen was ground and polished smoothly with sand sheets numbered 600 — 1200 after it was machined. Each type of specimen consisted of 6 specimens. The compressive experiments were performed on a multi-functional microscale materials test machine at the Laboratory of the Mechanics and Engineering Department, Peking University. In the compressive experiments, displacement condition was exerted.
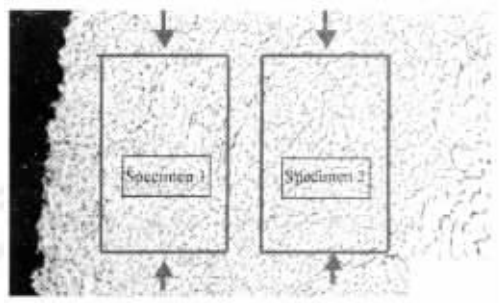

Fig. 2.   The design of compressive specimens.

Fig. 3 shows the compressive stress-strain curves, measured from the experiments based on the above-mentioned two types of specimens. There is a small difference among the measured results for the same type of specimen (fig. 3). The compressive stress-strain curves obtained from specimen 1 are obviously higher than those of specimen 2. Moreover, the yield points of the materials before and after nanocrystallization are not very obvious. It seems at $s_Y \approx 200$ MPa that the curves

turn low, and then become soft slowly. The compressive stress-strain curve of the original Al-alloy material (before the nanocrystallization) becomes soft quickly as strain increases. When compressive strain reaches about 1.5%, the material loses the hardening feature. However, the compressive stress-strain curve of the nanocrystalline material still displays a strong hardening characteristic.
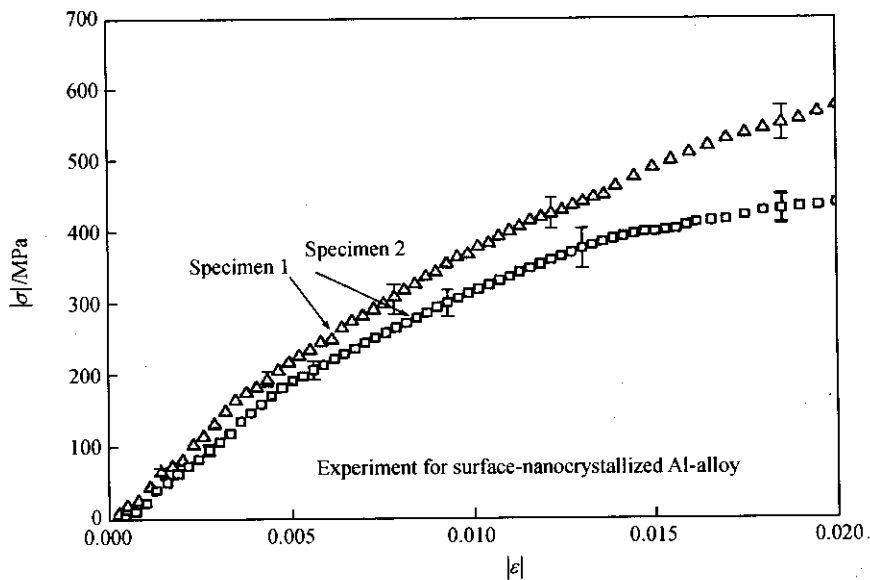

Fig. 3.   Compressive stress-strain curves before and after the nanocrystallization.

1.3   Nanoindentation test of the SNCAA material

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). The present experimental project is different from that in ref. [11]. In ref. [11], for the nanocrystalline Al, the indented cross-section was parallel to the nanocrystalline surface. In the present experiment, initially, the indenter tip will be located at either the grain region or the grain boundary region. However, there is a great possibility of the indenter tip located at the grain region, because the grain region is much larger than the grain boundary region on the indented surface. Consider that when the indenter tip is located 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. A simplified model for the nanoindentation test of the polycrystals is presented and developed, as shown in fig. 4. When the indent depth is small, the plastic slip deformation occurs within the indented grain, while the other grains outside

the indented grain only undergo the elastic deformation. As the indent depth further increases or as the indenter tip approaches the grain boundaries, the effective stress or effective strain within the grain boundary region attains a critical value at which the slip systems of neighboring grains will be switched on.
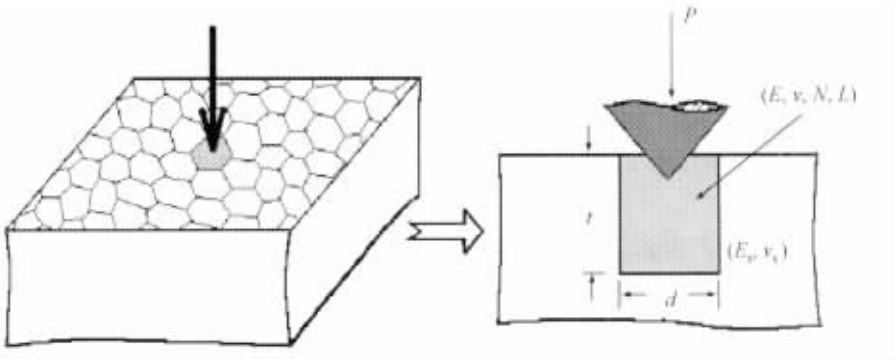

Fig. 4.   Microstructure cell model for the nanoindentation test.

Nanoindentation experiments are carried out at the State Key Laboratory of Friction and Lubrication, Tsinghua University, and at the Institute of Metal Research, Chinese Academy of Sciences, respectively. The instrument of the former is CSM-Nanoindenter (made in Switzerland). The nanoindentation test method is the discrete point selection[10] method. 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.

(i) Experimental results of the discrete point selection method.   By randomly selecting the loading points on the specimen surface and carrying out the nanoindentation test, the obtained hardness-depth relation is shown in fig. 5, where D is the distance from the selected indent point to the nanocrystalline surface. Two hardness relations corresponding to different distance D are given in fig. 5(a) and (b). Fig. 5 shows that the difference between the measured hardness-depth relations at two distances is small. This implies that the differences among the nanocrystalline grain sizes are small. The hardness value sharply increases as the indent depth decreases when the indent depth is smaller than 200 nanometers (fig. 5), i.e. the 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[15].

(ii) Experimental results of the continuous stiffness method.   Fig. 6 shows the measured hardness-depth curves at the several loading points and at D=60 microns. Fig. 6(a) and 6(b) correspond to the experimental curves for two different coordinate scales, respectively. Although the curves correspond to the different loading points on the
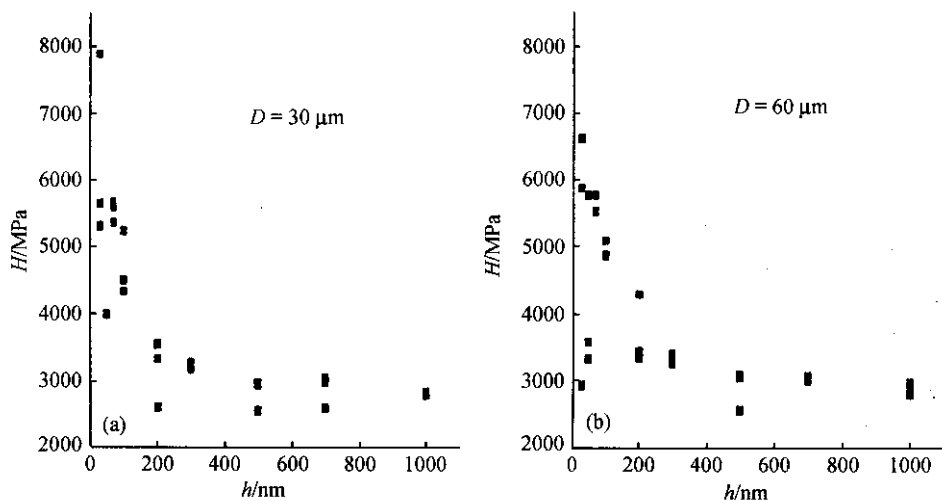
Fig. 5.   Hardness-indent depth relations measured by using the discrete selection points method. *D* is distance from indent location to the nanocrystalline surface.
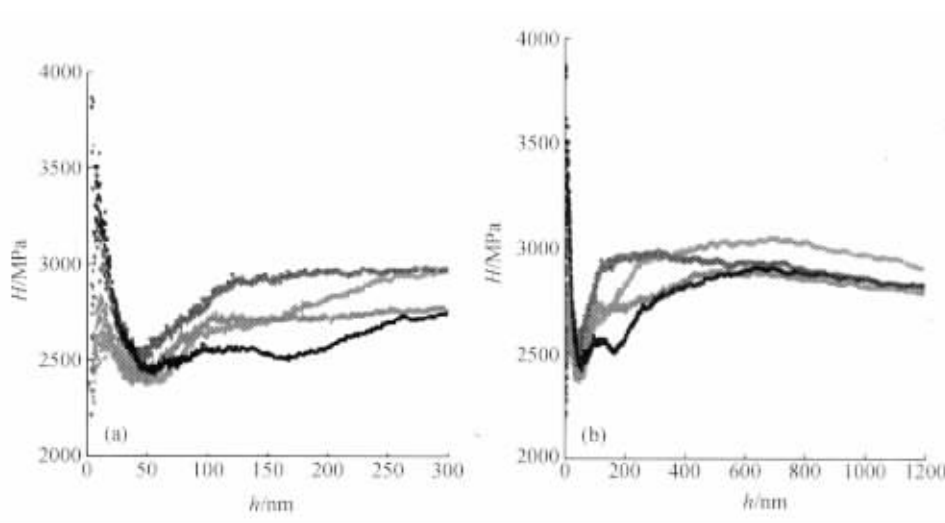

Fig. 6.   Hardness-indent depth curves measured by using the continuous stiffness method (*D*=60 nanometers). (a) and (b) correspond to the same data file and the different horizontal coordinate scales.

specimen surface, the measured results have a similar feature, which implies that the tests are repeatable (fig. 6). Comparing fig. 6 with fig. 5, clearly both results by adopting two kinds of indentation test methods have a big difference. Fig. 6 shows that as the indent depth decreases, especially, when the indent depth is smaller than 100 nanometers, the variation law of the hardness curves is very complicated. When the indent depth is smaller than 50 nanometers, the results display a strongly going up trend. When the 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 be consistent with each other.

The obtained material hardness values shown in fig. 5 and fig. 6 when the indent depth is larger than 600 nanometers are obviously higher than that of the conventional polycrystal Al-alloy material (~1.5 GPa)[15].

It is worth pointing out that the difference of both indentation experimental results is mainly due to the difference of both experimental methods. Each experimental method has its own advantages and disadvantages, which have been discussed in detail in ref. [10].

2 Simulations of size effect and geometrical effect for the SNCAA material

Since the size effect cannot 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 compressive stress-strain curves and the hardness-depth curves for the SNCAA material. Fleck-Hutchinson's strain gradient plasticity theory[6,10,16] will be adopted. 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.

2.1 Compressive stress-strain curve

From the measured compressive stress-strain curves of specimen 1 (influenced by the nanocrystalline process) and specimen 2 (uninfluenced by the nanocrystalline process) shown in fig. 3, a large difference of both experimental curves can be observed, and can be described and simulated based on the following considerations:

(1) For a simple and overall consideration (uniformization), the materials of both specimen 1 and specimen 2 have different mechanical parameters, such as yield strength, etc. Based on the consideration, the difference of both experimental curves can be described by adopting the conventional theory and adopting different material parameters. However, the overall yielding strength of material (related with the grain size) after the nanocrystallization cannot be obtained.

(2) For a fine consideration, suppose that the grains of both specimen 1 and specimen 2 have the same mechanical parameters, but the grain sizes are different, and the constraint effects by grain boundary on the plastic slip zones are different. The phenomenon can be described by presenting a microstructure cell model and adopting the strain gradient theory. The effect of the grain size on the material behavior can be obtained. In the present paper, the analysis model is set up based on the consideration (2).

Through observation of the microstructure features of the SNCAA (fig. 1 (c), (d) and (e)) and the design of the compressive specimens (fig. 2), a microstructure cell model for compressive test problem as shown in fig. 7 is adopted. The grain shape is simplified as a cylinder and the grain arrangement is simply described according to a periodic manner. The connection of grain with grain is by a thin grain boundary layer. The effective radius and height of a representative grain are described by B_1 (note that when top and bottom surfaces of the cylinder are exerted by the uniform displacements, the solution is independent of the cell height). The thickness of the grain boundary layer is denoted by $2B_2$. Through observation of the micro-construction features of the nanocrystalline material, one can find that in the process of the nanocrystallization, an original large grain is broken up into many small grains, and the great number of the dislocations are produced inside the grain and moved to the new grain boundaries. Therefore, in the present simplified model, the grain is simplified as an elastic-plastic material conforming to a power-law hardening relation, and is also described by using the strain gradient theory. The material power-law hardening relation of the stress-strain curve is written as follows:
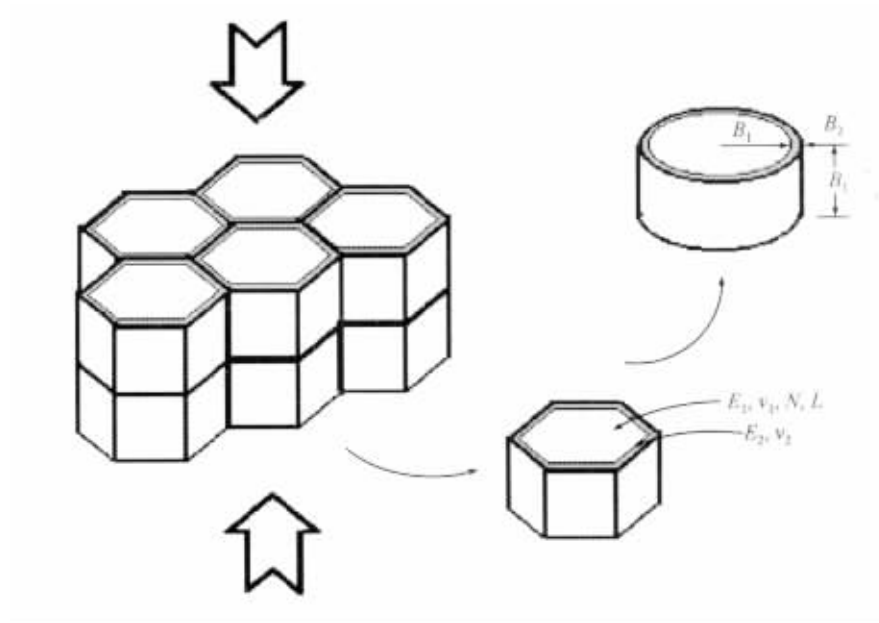

Fig. 7.   Microstructure cell model for compressive test of nanocrystal material.

$$s = E_1 e, \ s \leqslant s_Y; \ s = s_Y \left(\frac{E_1 e}{s_Y} \right)^N, \ s > s_Y. \tag{1}$$

The related parameters are: E_1 (Young's modulus), n_1 (Poisson's ratio), N (power-law hardening exponent), s_Y (yield strength) and L (microscale parameter in strain gradient theory[6,10]). The grain boundary layer is elastic, and can be described by using the

conventional elastic theory. The related parameters are: E_2 (Young's modulus), n_2 (Poisson's ratio). The size ratio of the elastic-plastic material region with the elastic material region is B_1/B_2, which can be expressed as the volume fraction of the grain zone or the grain boundary zone.

From dimensional analysis, the independently parametrical relation of the compressive stress-strain curve can be expressed as

$$\frac{|\boldsymbol{s}|}{\boldsymbol{s}_Y} = f\left(|\boldsymbol{e}|; \frac{E_1}{\boldsymbol{s}_Y}, \frac{E_2}{E_1}, \boldsymbol{n}_1, \boldsymbol{n}_2, N, \frac{B_2}{B_1}, \frac{L}{B_1}\right). \tag{2}$$

Relation (2) shows that the compressive stress-strain relation depends on the many material parameters and the size ratio of grain region with grain boundary region. The microstructure cell model (fig. 7) and the strain gradient theory were adopted to carry out the finite element simulation for the compressive experiment, and the compressive stress-strain curves for certain values of the parameters were obtained. For the simplified axi-symmetrical model shown in fig. 7, the nine-node displacement isoparametrical element and the 2×2 Gauss point distribution scheme[16] were adopted in the calculation.

Fig. 8 shows the variations of the simulated compressive stress-strain curves for several microstructure cell size values and the material parameter values. For metal material, in the calculation, the adopted material parameters are given in fig. 8. Regarding the model parameter ratio B_2/B_1, from microscopic observations of the SNCAA, its value is within the region 0.03—0.06. In the calculation, take its value as 0.03 and 0.06, respectively. Fig. 8 shows the compressive stress-strain curves for several
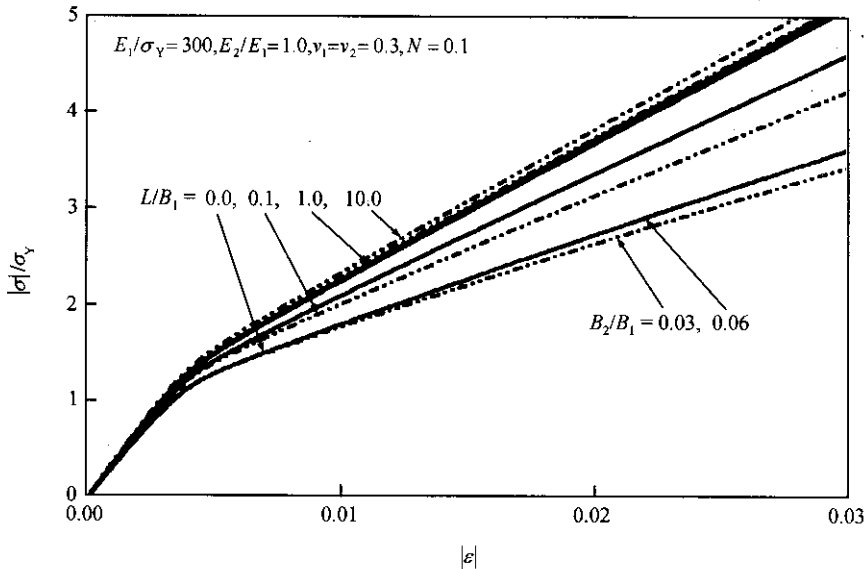

Fig. 8.   Theoretical simulation results of compressive stress-strain curves.

L/B_1 values. Clearly, when the grain is very large, corresponding to small L/B_1 value, the compressive stress-strain curves are low. As the grain dwindles, the compressive stress-strain curves rise up. As the grain continuously dwindles and becomes so small as to be comparable with the micro-scale parameter, L, the effect of the continuous decrease in grain size on the material strength is very weak.

Fig. 9 shows the simulation results and the comparisons with the average experimental results for $L=0.5$ micron[9] when B_1 is equal to 0.1 and 100 microns, respectively. It is worth pointing out that when the grain size is 100 microns (original grain size of the polycrystal Al-alloy), the simulation results and the experimental results (specimen 2) correspond to each other; however when the grain size is 0.1 micron, it is difficult to obtain the corresponding experimental results. The experimental results of specimen 1 given in fig. 9 only interpret the increase in the material strength due to the nanocrystallization. Through comparison with the simulation results, the experimental results of specimen 1 seem equivalent to the strength enhanced for the 0.1 micron, an average grain size case.

It is worth stressing that if the conventional elastic-plastic theory is adopted, from dimensional analysis, the form of the stress-strain parametrical relation can be obtained by letting $L/B_1=0$ in formula (2), while B_2/B_1 can be expressed as the volume fraction of the grain boundary zone, and its value is very small (when B_1 is at submicron, B_2/B_1 is about 0.03 from microscopic measurement). Therefore, the size effect displayed in the stress-strain relation cannot be simulated by adopting the conventional elastic-plastic theory, and the simulated results are not consistent with the experimental results (variation of grain size from submicon to tens of microns). This is the reason why the
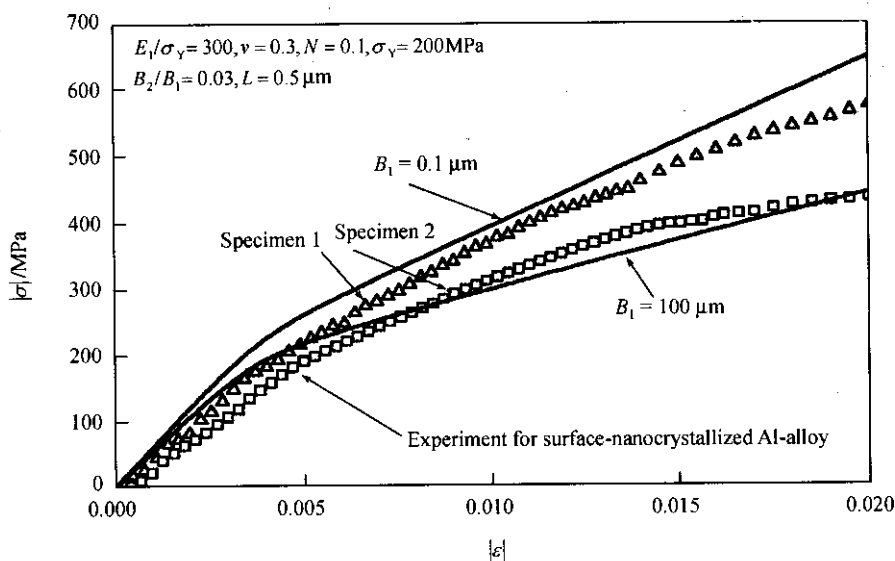

Fig. 9.   Comparison of the simulation results with the experimental results.

strain gradient theory is adopted.

2.2   Hardness-depth curves in nanoindentation test

The nanoindentation experimental results, size and geometrical effects given in the first section can be simulated by developing a microstructure cell model (fig. 4) and by using the strain gradient theory. In the authors' simulations for the nanocrystalline Al (in the vertical direction of the nanocrystalline surface)[11], a combination model of both the material microstructure cell and the strain gradient theory was adopted. Here we adopt the same model to simulate and analyze the SNCAA material when the indent direction is parallel to the nanocrystalline surface. The model is shown in fig. 4. The simplified consideration of the model can be described as follows: When the grain size is within the submicron region (100 nanometers to 1 micron), the length scale is at the same quantity level as the size-sensitive depth of the nanoindentation. The nanoindentation test phenomena can be described as that when the indenter acts on a grain surface, as the indent depth gradually increases, near the indenter tip the plastic slip zone size increases and expands to the grain boundary within the indented grain region. However, due to the resistance of the grain boundaries, the plastic slip zone is constrained to the indented grain region for a certain indent depth scope, while the other grains outside the indented grain only undergo the elastic deformation. The region the other grains occupy can be treated as an equivalent uniform and isotropic elastic body. According to self-consistent theory[17], one can find the Young's modulus and Poisson's ratio of the equivalent elastic body. The related problem of the self-consistent method can be summarized as that for the equivalent modulus problem of a polycrystal material when each grain is a cylinder in shape, consider that a cylinder is located in an infinite-large equivalent elastic body, solve the problem to find the equivalent modulus. 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. Considering the grain shape as the ellipsoid, the Young's modulus of the equivalent elastic body is 0.614 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[11]. 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. The simplified model is shown in fig. 4. Differing from the previous case[11], 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 the grain. In the simplified model, there are totally three governing parameters. Besides the grain size and the micro-scale parameter L included in the strain gradient theory discussed previously, 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 the effective plastic strain at grain boundary is over a critical value, $\overline{e}_P^c$, the plastic zone will pass through the grain boundary and extend to the neighbour grain zone. The condition of the plastic slip zone across the grain boundary can be expressed as

$$\bar{\boldsymbol{e}}_P \mid_G = \bar{\boldsymbol{e}}_P^c, \tag{3}$$

where $\boldsymbol{G}$ stands for the grain boundary.

From the microstructure cell model (see fig. 4), the fundamental relations of the strain gradient theory[6, 10], as well as the condition of the plastic slip zone across the grain boundary (see formula (3)), the independently parametrical relation of hardness depth can be dictated as

$$\frac{H}{3\boldsymbol{s}_Y} = g\left(\frac{h}{L}; \frac{d}{2L}, \frac{t}{L}, \frac{E}{\boldsymbol{s}_Y}, \frac{E_s}{E}, \boldsymbol{n}, \boldsymbol{n}_s, N, \bar{\boldsymbol{e}}_P^c\right). \tag{4}$$

Note that since the total plastic region only concerns a few grains, and the plastic region is infinitely small relative to the whole material region, the effect of the plastic zone across the grain boundary on the Young's modulus of infinitely large equivalent elastic body E_s can be neglected.

The parametrical relation (4) can be obtained in detail by using the finite element numerical simulation. The detailed discussion of the finite element method is given in refs. [10, 11]. Fig. 10 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, $\bar{\boldsymbol{e}}_P^c$, and $E_s/E = 0.67$ from the self-consistent analysis[11,17]. When the critical value of the effective plastic strain, which describes the grain boundary strength, varies from twice to six times the yield
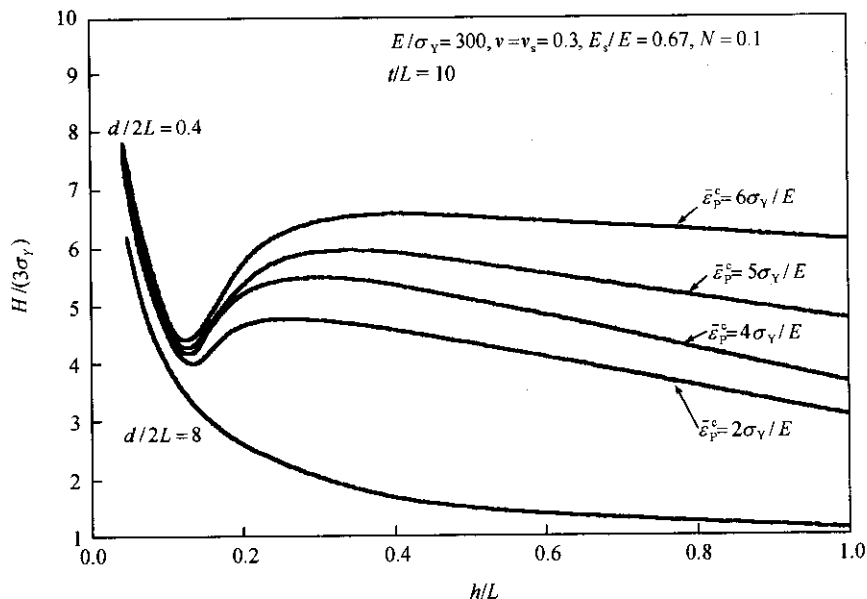

Fig. 10.　Hardness-depth simulation results of the SNCAA material by adopting the microstructure cell model.

strain, the predicted hardness curves have similar characteristics to the experimentally measured curves (comparing fig. 6 with fig. 10). Obviously, the theoretical model captures the fundamental features of the problem. If we take L=0.5 micron in fig. 10 and consider the yield strength s_Y = 200 MPa, combining the compressive stress-strain curve of the Al-alloy material (fig. 3), the theoretical simulation results in fig. 10 are consistent with the experimental results in fig. 6. Fig. 10 also shows the result when the grain size is very large, i.e. for the case of $d/2L$ = 8. The extension of plastic slip zone is not constrained as the indent depth increases. The result describes the case of the pure size effect.

3 Summary and discussion

The mechanical behavior of the SNCAA material fabricated by using the USP method has been investigated both experimentally and theoretically. Through observation of the microstructure of the material and through combining the microstructure features, the specimens have been designed, by which the size effect can be described, and both microscale compressive experiments and the nanoindentation experiments have been performed, respectively. The compressive stress-strain curves and the hardness-depth curves have been measured. 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 obviously. The ascents of both stress-strain curve and hardness curve are the prominent manifestation of the improvement in mechanics property. In the theoretical research, taking the observed microstructure features of the nanocrystalline material and the characteristics of the compressive and indentation experiments into account, the microstructure cell models have been developed and the strain gradient theory has been adopted to simulate the experimental processes. The compressive stress-strain curve and the hardness curve have been obtained, which are basically consistent with experimental measurements. Through comparing the experimental results with the simulation results, the microscale parameter and model parameters have been predicted. Through experimental and theoretical researches for the SNCAA material, a basic purpose for basically understanding the material mechanical behavior has been attained.

Through observations of the SNCAA material microstructure, one has found that near the nanocrystalline surface the grain size is very small (several nanometers). However, the effectiveness of both the microstructure cell models and the strain gradient theory adopted in the present research is difficult to be confirmed when the grain size is smaller than submicron. Although the strain gradient theory can be applied to the small scale to compare with the conventional elastic-plastic theory, the simulated results can only be taken as a rough estimation for the mechanical behavior of the nanocrystalline material when the grain size is smaller than submicron. Therefore, in order to investigate the mechanical behavior of the surface-nanocrystalline material near the nanocrystalline surface, one still needs to further explore the more reasonable and effective microscopic

theories and methods.

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