

Microstructure characteristics of as-surface nanocrystallized 1420 aluminum alloy by high-energy shot peening^①

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Abstract: A nanostructured surface layer was fabricated on 1420 aluminum alloy by high-energy shot peening. Microstructures were characterized by X-ray diffractometer (XRD), transmission electron microscope (TEM) and high-resolution electron microscope (HRTEM), and microhardness measurement was conducted along the depth from top surface layer to matrix of the sample peened for 30 min. The results show that a nanocrystalline layer about 20 μm in thickness is formed on the surface of the sample after high-energy shot peening, in which the grain size is changed from about 20 nm to 100 nm. In the surface layer of 20 ~ 50 μm in depth, the microstructure consists of sub-micron grains. The surface nanocrystallization is accomplished by dislocation slip. The microhardness of the top surface nanostructured layer is enhanced obviously after high-energy shot peening (HESP) compared with that of the coarse-grained matrix.

Key words: 1420 alloy; aluminum alloy; high-energy shot peening; surface nanocrystallization; microstructure; microhardness

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1 INTRODUCTION

Nanocrystalline materials have attracted great concern in scientific field^[1-3]. These materials are structurally characterized by fine grain size and large amount of grain boundary area (and volume). Nanocrystalline materials have unusual and extraordinary mechanical and physical properties that are fundamentally different from, and often far superior to those of their conventional coarse-grained polycrystalline counterparts. Severe plastic deformation (SPD) is an effective processing method for the fabrication of nanocrystallization structures by imposing intense plastic strains into metals and alloys^[4-7]. Compared with other processing techniques, such as inert gas condensation and high-energy ball milling, SPD can produce large bulk samples and the produced nanocrystalline samples are free from any residual porosity and contamination. Recently, several severe plastic deformation processes are available for producing high plastic strain, including equal channel angular pressing (ECAP)^[5-7], high pressure torsion (HPT)^[8], multipass-coining^[9], and repetitive corrugation and strengthening (RCS)^[10]. However, most of these techniques are still difficult for practical application in conventional engineering materials.

A SPD based mechanical treatment for surface nanocrystallization (SNC), high-energy shot peening (HESP), is recently proposed^[11]. It has been demonstrated that a nanocrystalline surface layer can be formed through intensive strain and high strain rate during the process of HESP^[12-14]. The strains present gradient distribution in the surface layer, changing from the maximum at the top surface to zero far into the matrix^[15]. Microstructure observation at different depths from the surface was conducted to investigate the structural evolution at different stages of strain. The microstructures of the surface layers of 1420 aluminum alloy before and after HESP treatment were characterized.

2 EXPERIMENTAL

The experimental material is 1420 aluminum alloy plate of 3 mm in thickness, and its chemical compositions (mass fraction, %) are 2.3Li, 4.6Mg, 0.25Mn and balance Al. The alloy plate was cut into pieces with dimension of 100 mm \times 100 mm. The samples were polished with SiC papers and then solution-treated at 520 $^{\circ}\text{C}$ for 1 h in a vacuum furnace. Microscopic examination results show that the initial grain size is about 30 μm . Sur-

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face nanocrystallization was conducted on the sample by high-energy shot peening in vacuum for 5 and 30 min, respectively. The material of shot is stainless steel and the shot diameter is 8 mm.

The X-ray diffraction analysis was carried out on a Rigaku D/max 2400 X-ray diffractometer, with Cu K α radiation. The microstructures in given depth from the surface to the matrix of the sample were observed by H-800 transmission electron microscope(TEM) and JEOM-2010 high resolution transmission electron microscope(HRTEM). The sample for TEM observation was finally thinned by ion-milling. Cross-section microhardness measurement was conducted along the depth from the surface of the sample by MVK-H3 Vickers hardometer. The load is 0.1 N, and the loading-time is 10 s.

3 RESULTS AND DISCUSSION

3.1 Microstructures

The XRD patterns of 1420 aluminum alloy samples before and after HESP treatments are shown in Fig. 1. It is observed that after HESP treatment, there is evident broadening of the Bragg reflection profiles, which might be attributed to grain refinement and the microstrain development.

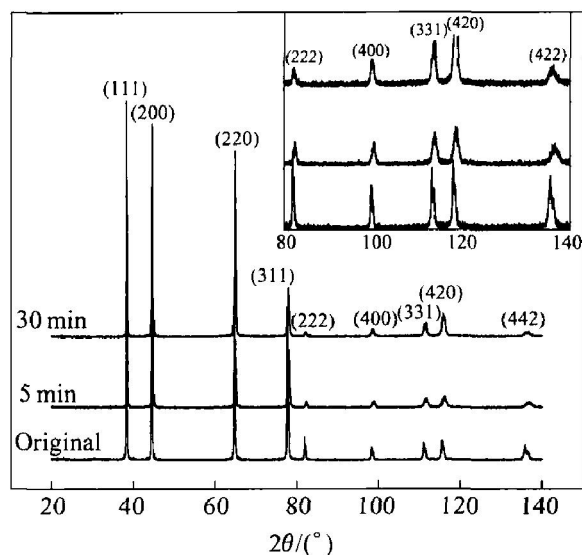


Fig. 1 XRD patterns for Al alloy samples after HESP treatment for various periods of time

The increase of HESP treatment duration does not change significantly the grain size of the surface layer, but increases the thickness of the nanocrystalline layer.

The microstructure of the sample after HESP treatment for 30 min was also observed. Fig. 2 shows a TEM micrograph of the layer in a depth of about 100 μm from the top surface layer of sample. A great number of dislocation tanglings are formed inside the grains. Because of the high stack fault energy of aluminum alloy, the dislocation is diffi-

cult to dissociate and the cross-slip is easy to take place, and hence the dislocations interact and tangle with each other frequently. Therefore, the inhomogeneously distributed dislocations result in the formation of high dislocation density area and low dislocation density area, which is the initial stage of dislocation cell. With an increase in plastic deformation, the tangled dislocations transform into dislocation cells and further develop into subgrains.

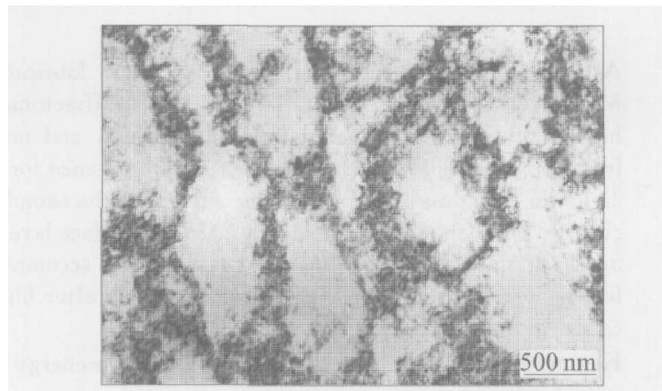


Fig. 2 TEM micrograph showing dislocation cells and tanglings

TEM micrograph(Fig. 3) clearly shows that subgrains are separated by dislocation walls (about 50 μm in depth from the top surface). The subgrains are formed by dislocation tangling. With an increase in strain, the dislocations on the cell boundaries interact with each other, resulting in the formation of the dislocation wall. With an increase in dislocation density and a decrease in dislocation spacing, the dislocation wall can evolve into subgrain boundary or the low angle boundary.

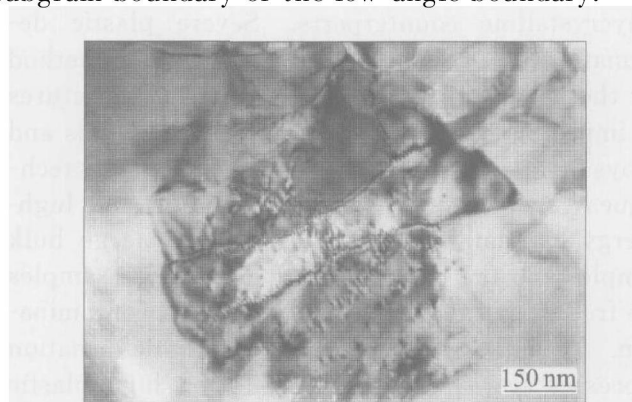


Fig. 3 TEM micrograph showing dislocation walls in subgrains

TEM micrograph in Fig. 4 shows the grain sizes are about 80 - 120 nm at an increased strain (about 20 μm in depth from the top surface). The contrast is obviously different in grains, which indicates the existence of high internal stresses.

Fig. 5 shows a TEM micrograph illustrating the equiaxed microstructure in the nanometer re-

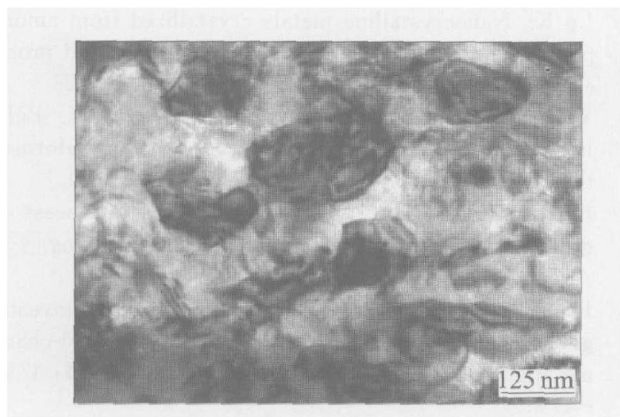


Fig. 4 TEM micrograph of layer in depth of about 20 μm from top surface layer

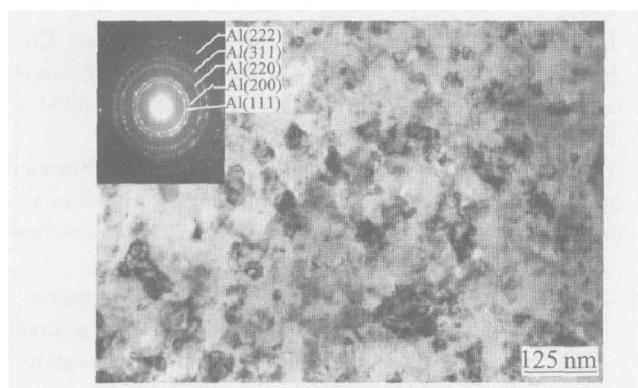


Fig. 5 TEM micrograph showing nanocrystal grains and corresponding selected area electron diffraction pattern

gime. The image is taken at the outer surface of the layer (about 5 μm deep from the top surface). The average grain size is determined to be 10 – 50 nm. Some grain boundaries are visible, but most of them are poorly defined. The corresponding selected area electron diffraction (SAED) pattern is also presented. The ring-like electron diffraction pattern shows that grain orientation is random, which indicates highly misoriented boundaries.

The above mentioned results show that the microstructures change from subgrains far from the top surface layer to grains about 100 nm with high angle grain boundaries to grains about 20 nm near the top surface layer. This indicates that with an increase in strain, deformation structure undergoes the changes from dislocation tanglings to dislocation cells to dislocation walls and finally subgrains. As strain increases, a large number of dislocations slip to subgrain boundary, resulting in an increase in disordered orientation among subgrains, and evolve into high angle grain boundary.

Fig. 6 shows a HRTEM micrograph of the layer in a depth of about 5 μm from the top surface layer of sample after HESP treatment for 30 min. It shows that the grains orient randomly and are characterized by high angle grain boundary, which further proves that the grain disordered orientation

increases and the subgrains with low angle boundary evolve into nanocrystallines with high angle boundary with an increase in strain.

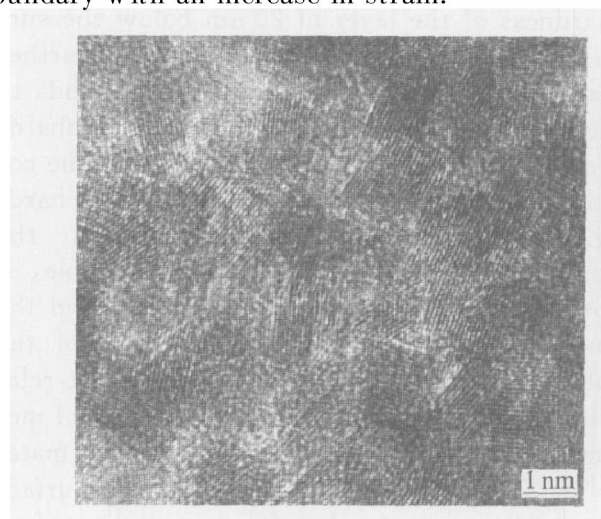


Fig. 6 HRTEM micrograph of layer in depth of about 5 μm from top surface

The above experimental results show that nanostructure can be formed by high-energy shot peening. The thickness of the nanocrystalline surface layer varies from a few micrometer to about 20 μm depending upon the treatment duration. Owing to the different amount of the plasticity, grain size increases from about 20 nm at the top surface layer, gradually to more than 100 nm at a depth of about 20 μm . Submicron crystalline layer is formed in the depth of 20 – 50 μm from the top surface layer.

3.2 Hardness variation

Fig. 7 shows the microhardness variation along the depth from the treated surface of the sample by high-energy shot peening for 30 min. In the top surface nanostructured layer, microhardness is enhanced obviously, and the hardness decreases gradually with an increase in depth. Compared

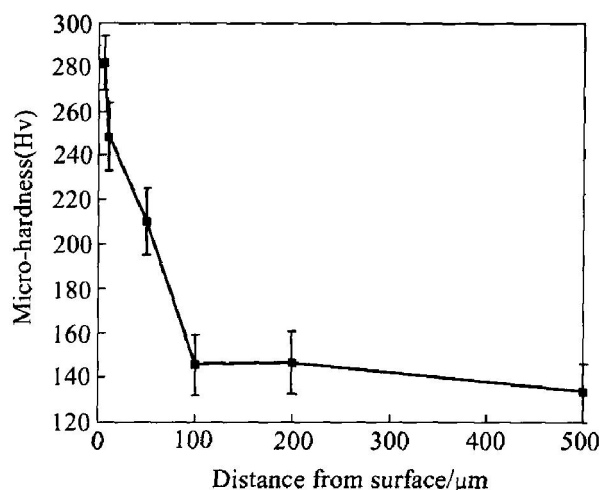


Fig. 7 Hardness variation along depth after HESP for 30 min

with that of the matrix of sample, the microhardness of the top surface nanostructured layer is about twice that for the coarse-grained matrix, and the hardness of the layer of 20 μm below the surface is enhanced obviously too. With the further increase in depth, the hardness variation tends to be steady. The increase of the surface microhardness after HESP treatment is attributed to the coaction of the grain refinement and the work-hardening. From the relationship between the microstructure and the property of the sample, it can be found that the grain size increases and the hardness decreases along with the depth of the sample, which consists with the Hall-Petch relationship and corresponds to the study result of mechanical properties of other ultrafine-grained materials^[16]. So it can be concluded that the surface nanocrystallization is helpful to the surface strengthening of materials.

4 CONCLUSIONS

1) The nanocrystalline layer is successfully prepared on the surface of 1420 aluminum alloy specimen by high-energy shot peening. The surface nanocrystallization is related to the amount of the plastic deformation. Grain size increases from about 20 nm at the top surface layer, gradually to more than 100 nm at a depth of about 20 μm . Sub-microstructure is formed in the layer of 20 ~ 50 μm below the surface.

2) The surface nanocrystallization is realized by dislocation slip during HESP treatment.

3) Mechanical property measurement indicates a significant increment of hardness in the surface layer with nanostructures after the high-energy shot peening treatment. Compared with that of the matrix of sample, the microhardness of the top surface nanostructured layer is about twice that for the coarse-grained matrix.

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