

# Strengthening and stress drop of ultrafine grain aluminum after annealing

REN Jiang-wei(任江伟)<sup>1</sup>, SHAN Ai-dang(单爱党)<sup>2</sup>

1. School of Materials Engineering, Shanghai University of Engineering Science, Shanghai 200126, China;

2. School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China

Received 23 October 2009; accepted 27 August 2010

**Abstract:** Ultrafine grain pure aluminum was produced by equal channel angular pressing and cold rolling, the deformed aluminum was annealed at 200 °C for 1 h. The tensile curves of deformed and annealed aluminum show that yield strength of deformed aluminum increases by 100%–300% and its elongation decreases by about 20%. After low temperature annealing, strength of annealed aluminum increases by 20% and elongation decreases by over 50%, the recovery of dislocations may be the main cause of annealing strengthening. In addition, there is an abrupt stress drop in the tensile curves of annealed aluminum and the formation of shear band is responsible for it.

**Key words:** aluminum; ultrafine grain; mechanical properties; heat treatment

## 1 Introduction

Ultrafine grain materials especially nanomaterials have aroused the interests of researchers for their novel physical, chemical and mechanical properties. Severe plastic deformation (SPD) technologies prove effective methods which improve mechanical properties by refining the traditional coarse materials to ultrafine grain or nanocrystalline materials[1–2]. Equal channel angular pressing (ECAP) is one the of the important methods of severe plastic deformation.

Most researches of ultrafine grain materials prepared by ECAP focused on the microstructure evolution and mechanical properties of ultrafine grain materials[3–7]. Heat treatment is a traditional technique to improve the mechanical properties of materials, related researches show that low temperature annealing is beneficial to the microstructure refinement and strengthening of ultrafine grain aluminum. Meanwhile, annealing promotes the stability of subgrain and multiple slip of dislocations[8]. The intermediate annealing is helpful to form equiaxed ultrafine-grains with high angle grain boundaries in the multi-pass ECAP. The content of high angle grain boundaries increases during annealing while the grain size keeps stable. So, the ductility of materials is improved without the cost of strength[9].

However, the influence of low temperature annealing on the mechanical properties of ultrafine grain materials is still to be studied.

In this work, an ultrafine grain material is produced by ECAP on commercially pure aluminum and then subjected to annealing at 200 °C for 1 h. Uniaxial tensile is conducted on deformed and annealed material to explore the influence of low temperature annealing on the mechanical properties of ultrafine grain aluminum.

## 2 Experimental

Commercial pure aluminum (99.8%) was used in this work to avoid the influence of phase transformation during the deformation and annealing. The mechanical properties of raw material were listed in Table 1.

A billet with dimensions of 12 mm×12 mm×80 mm was cut from the raw material and pressed 4 passes in a mould with 90° channel angle. To obtain equiaxed ultrafine grain material with the fewest passes, the billet

**Table 1** Mechanical properties of commercial pure aluminum[10]

| Yield strength/MPa | Ultimate tensile strength/MPa | Elongation/% |
|--------------------|-------------------------------|--------------|
| 30–60              | 80–120                        | 25–50        |

was rotated 90° along its axis after each pass (route Bc). The accumulated equivalent strain in ECAP was 4.62. Then, the billet was cold rolled along the pressing direction and the rolling plane was the Z plane described in Refs.[11–12]. The total reduction was 98%. Finally, deformed aluminum was annealed at 200 °C for 1 h.

Tensile specimens were sliced from the deformed and annealed aluminum by electrode discharge machining. The gauge section of tensile specimen was 3 mm×0.2 mm×10 mm with the long axis parallel to the rolling direction of billet. Tensile test was conducted in a Zwick/Roell BTC-FR020TN.A50 test machine at room temperature with a constant crosshead speed and the initial strain rate was  $3 \times 10^{-6}$  and  $3 \times 10^{-3} \text{ s}^{-1}$ . The stress at 0.2% residual strain was defined as yield strength because of the unapparent yield of pure aluminum during tensile test. Fracture surface was observed by an Hitachi S-520 scanning electron microscope at an accelerating voltage of 20 kV. TEM observation was conducted on a JEOL JEM-200CX transmission electron microscopy at an accelerating voltage of 200 kV. The TEM slices were prepared by standard procedure. The grain size of deformed and annealed aluminum was measured directly from the grains with well-defined grain boundaries.

### 3 Results and discussion

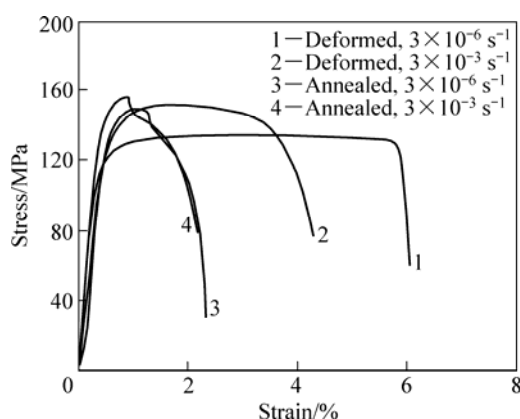
The tensile curves of deformed and annealed aluminum are shown in Fig.1. It is obviously that the mechanical properties of aluminum change greatly after severe deformation by analyzing the data in Fig.1 and Table 1. Comparing with raw state, the yield strength of deformed aluminum increases by 100%–300% and ultimate strength increases by 50%, but the elongation of deformed aluminum decreases by over 80%, which are the typical characteristics of ultrafine grain materials[1]. Compared with deformed aluminum, the strength of annealed aluminum increases and the elongation decreases distinctly. In addition, the strength increases

and the elongation decreases with increasing strain rate in deformed and annealed aluminum. The mechanical properties of deformed aluminum are more obviously influenced by the strain rate than those of annealed aluminum. The strength of annealed aluminum is about 20% higher than that of deformed aluminum at the same strain rate. The elongation of annealed aluminum is over 50% lower than that of deformed aluminum at the same strain rate. Besides the difference of mechanical properties, there are significantly different tensile behaviors between deformed and annealed aluminum, deformed aluminum reveals obvious homogenous deformation and annealed aluminum reveals an interesting phenomenon—stress drop—during tensile test. Necking phenomenon and dimples can be seen on the fracture surfaces of deformed and annealed aluminum, as shown in Fig.2. The dimples in annealed aluminum are less than those in deformed aluminum.

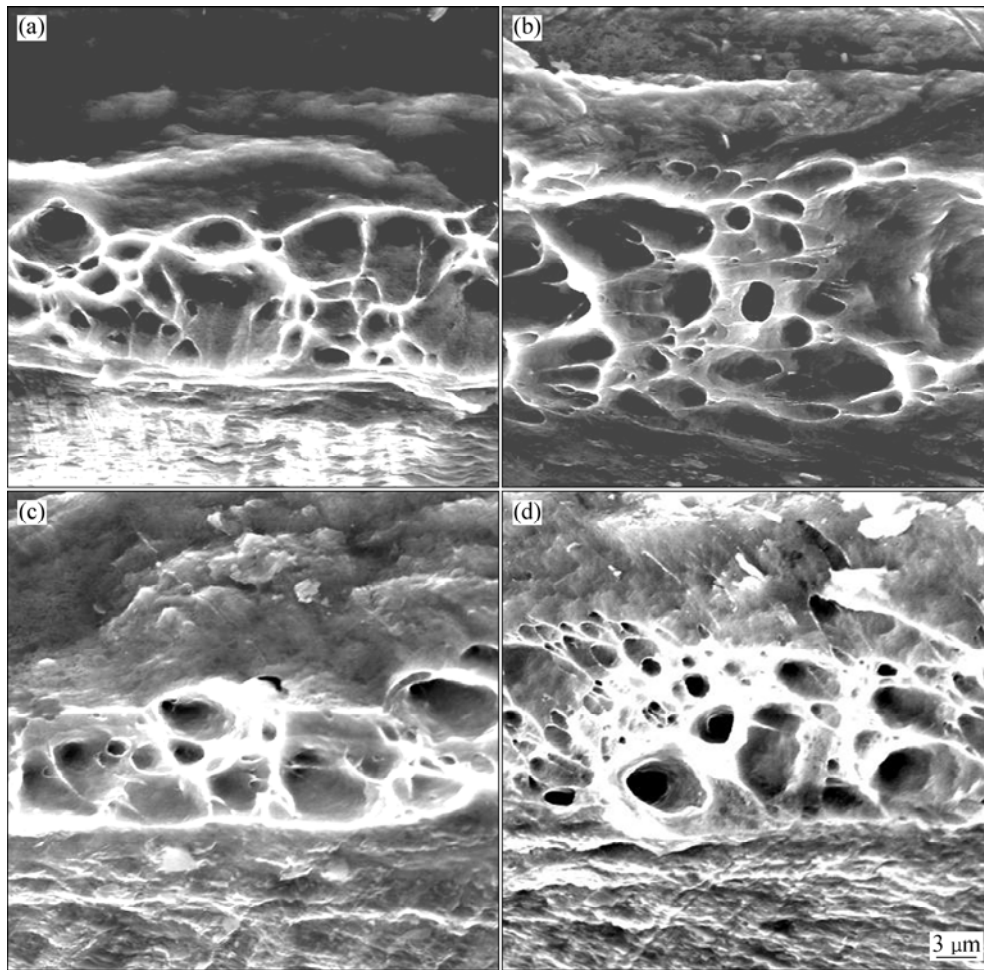
To demonstrate the cause of tensile behavior difference, the fine structures of deformed and annealed aluminum before tensile test are observed by TEM. The typical TEM images are shown in Fig.3. The grains are elongated in deformed and annealed aluminum, grain size of deformed and annealed aluminum is measured from TEM pictures. The average sizes of deformed and annealed aluminum are about 700 nm×280 nm and 830 nm×310 nm, respectively. The grains grow slightly during low temperature annealing, but the distinct contrast difference of TEM image implies the marked annihilation of dislocations.

When the billet is pressed through the channel corner in ECAP, pure shear strain is introduced and shear bands form. The amount of high angle grain boundary is high in shear bands. During the multi-pass ECAP with route Bc, shear plans and shear directions interact, so the formed shear bands interact with high angle grain boundaries. Meanwhile, the severe plastic strain introduced by ECAP makes dislocations multiply. The high density dislocations interact and tangle in their migration, so cell structures form and a large number of dislocations are absorbed by subgrain boundaries. Finally, equiaxed ultrafine-grain microstructure develops under the control of grain subdivision mechanism[13], and the amount of high angle grain boundary is high in ultrafine-grain microstructure. The microstructure is continually refined and the amount of high angle grain boundary increases under the rolling strain. The 50%–90% rolling strain leads to the formation of grain boundaries with high angle ranging from 10° to 30°[14]. Grain boundaries are in nonequilibrium and high energy state.

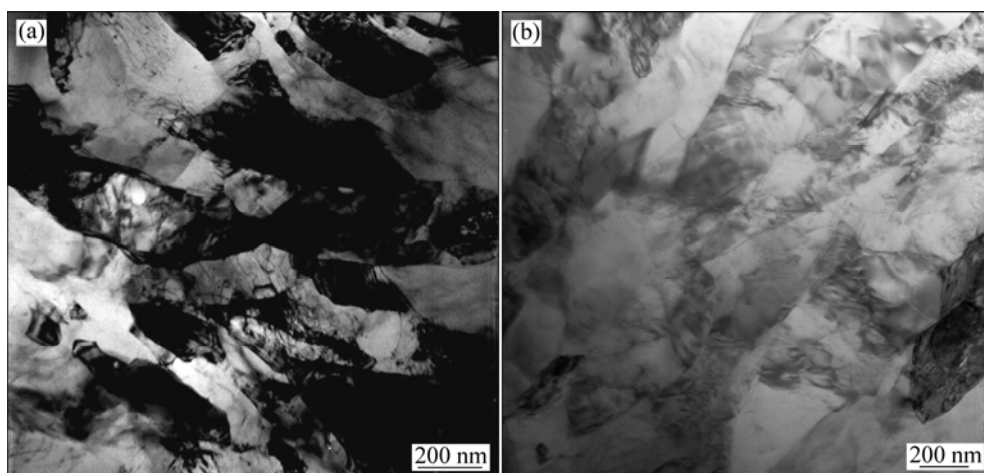
The plastic deformation occurs when the stress in slip system reaches a critical value. The pileup of dislocations in boundaries results in stress concentration



**Fig.1** Stress — strain curves of deformed and annealed aluminum at different strain rates



**Fig.2** Fracture morphology of deformed aluminum at strain rate of  $3 \times 10^{-6} \text{ s}^{-1}$  (a)  $3 \times 10^{-3} \text{ s}^{-1}$  (b) and annealed aluminum at strain rate of  $3 \times 10^{-6} \text{ s}^{-1}$  (c) and  $3 \times 10^{-3} \text{ s}^{-1}$  (d)



**Fig.3** TEM images of deformed (a) and annealed (b) aluminum before tensile

and is helpful to slip. The grain size of ultrafine microstructure is relatively small, so the distance between high angle grain boundaries is short. In addition, high angle grain boundaries are effective sinks of dislocations. Therefore, mobile dislocations in ultrafine materials are

not enough, which leads to the phenomenon that strength of deformed state is higher than that of raw state. Moreover, the lack of mobile dislocations makes the dislocations tangle difficultly, so necking occurs in early stage in tensile test and ductility decreases[15].

Recovery or recrystallization occurs in the annealing of ultrafine-grain materials. Dislocations in grain boundaries rearrange to form more stable low angle grain boundaries when ultrafine grains recover. The low temperature annealing of severe plastic deformed aluminum shows that the width of low angle grain boundaries narrows when annealed at 150 °C for 30 min, which means the dislocations at boundaries rearrange and the density of dislocations inside grains decreases obviously[16]. High angle grain boundaries in ultrafine grains decrease the number of dislocation source. In tensile test, the yield strength has to increase to activate new dislocations and the dislocations bear strain. So, the decreasing density of intragranular dislocations in annealing results in the decrease of elongation[16]. Meanwhile, the decrease of dislocation density after annealing makes mobile dislocations fail to satisfy applied strain rate. The dislocation absorption of grain boundary increases and stress concentration in grain boundary decreases after annealing, which makes the deformation difficult to extend to adjacent grains, therefore, heterogeneous deformation occurs. The formation of shear bands may contribute to the stress drop in tensile test of annealed aluminum[15].

Moreover, the migration of high angle grain boundary slows down in most cases because of the conflict of growing grains. In addition, deformed grains have similar crystallographic orientation. The conflict makes the high angle grain boundaries with high mobility become low angle grain boundaries with low mobility[14]. The stability enhancement of grain boundary and subgrain boundary after annealing increases the tensile strength. So, the ductility decreases and mechanical properties are less sensitive to strain rate in annealed aluminum.

## 4 Conclusions

1) Yield strength of pure aluminum increases by 100%–300% and the elongation decreases by about 20% after four-pass ECAP and 98% cold rolling.

2) Compared with deformed aluminum, strength of aluminum increases by 20% and elongation decreases by about 50% after annealing at 200 °C for 1 h. The recovery of dislocations is the main cause of anneal strengthening.

3) There is an obvious stress drop in the tensile strength of annealed aluminum and the formation of

shear band is responsible for it.

## References

- [1] SURYANARAYANA C. Nanocrystalline materials [J]. *International Materials Reviews*, 1995, 40(2): 41–64.
- [2] HYOUNG S K, MIN H S, SUN I H. Plastic deformation analysis of metals during equal channel angular pressing [J]. *Journal of Materials Processing Technology*, 2001, 113(1/3): 622–626.
- [3] WU Shi-ding, AN Xiang-hai, HAN Wei-zhong, QU Shen, ZHANG Zhe-feng. Microstructure evolution and mechanical properties of fcc metallic materials subjected to equal channel angular pressing [J]. *Acta Metallurgica Sinica*, 2010, 46(3): 257–276. (in Chinese)
- [4] WANG Jin-feng, BAI Pu-cun, ZHANG Xiu-yun, HOU Xiao-hu, REN Yong-gang. Investigation of effect of tensile property and fracture behavior of pure aluminum during ECAP [J]. *Journal of Aeronautical Materials*, 2009, 29(3): 33–38. (in Chinese)
- [5] ZHANG Guo-ping. Influence of strain rate on microstructure of pure aluminum in the process of equal channel angular pressing [J]. *Transactions of Materials and Heat Treatment*, 2008, 29(1): 111–115 (in Chinese)
- [6] WANG Li-zhong, WANG Jing-tao, GUO Cheng, CHEN Jin-de. Microstructure and properties of ultrafine-grained aluminum-based alloy processed by equal channel angular pressing [J]. *Journal of Xi'an Jiaotong University*, 2004, 38(5): 457–460, 473. (in Chinese)
- [7] XU Zun-ping, CHENG Nan-pu, CHEN Zhi-qian. Mechanical properties and finite element simulation of equal channel angular pressing of 7050 Al alloy [J]. *Journal of Materials Engineering*, 2008, (8): 1–4. (in Chinese)
- [8] OH-ISHI K, ZHILYAEV A P, MCNELLEY T R. Effect of strain path on evolution of deformation bands during ECAP of pure aluminum [J]. *Materials Science and Engineering A*, 2005, 410/411: 183–187.
- [9] CHANG J Y, SHAN A D. Intermediate annealing of pure aluminum during cyclic equal channel angular pressings [J]. *Journal of Materials Science*, 2003, 38(12): 2613–2617.
- [10] MONDOLFO L F. Aluminum alloys: Structure and properties [M]. London: Butterworths, 1976: 497.
- [11] SEGAL V M. Materials processing by simple shear [J]. *Materials Science and Engineering A*, 1995, 197(2): 157–164.
- [12] FURUKAWA M, HORITA Z, LANGDON T G. Factors influencing the shear patterns in equal-channel angular pressing [J]. *Materials Science and Engineering A*, 2002, 332(1/2): 97–109.
- [13] SEGAL V M. Equal channel angular extrusion: from macromechanics to structure formation [J]. *Materials Science and Engineering A*, 1999, 271(1/2): 322–333.
- [14] HUGHES D A, HANSEN N. High angle boundaries formed by grain subdivision mechanisms [J]. *Acta Materialia*, 1997, 45(9): 3871–3886.
- [15] HUNG P C, SUN P L, YU C Y, KAO P W, CHANG C P. Inhomogeneous tensile deformation in ultrafine-grained aluminum [J]. *Scripta Materialia*, 2005, 53(6): 647–652.
- [16] HUANG X X, HANSEN N, TSUJI N. Hardening by annealing and softening by deformation in nanostructured metals [J]. *Science*, 2006, 312(5771): 249–251.

(Edited by FANG Jing-hua)