JOURNAL OF MATERIALS SCIENCE **39** (2004) 2851 – 2854

Nanostructural formation of fine grained aluminum alloy by severe plastic deformation at cryogenic temperature

JINGOU YIN
Northwest Institute for Nonferrous Metals Research, P.O. Box 51, Xi'an, Shaanxi 710016, People's Republic of China
E-mail: jingouyin@hotmail.com

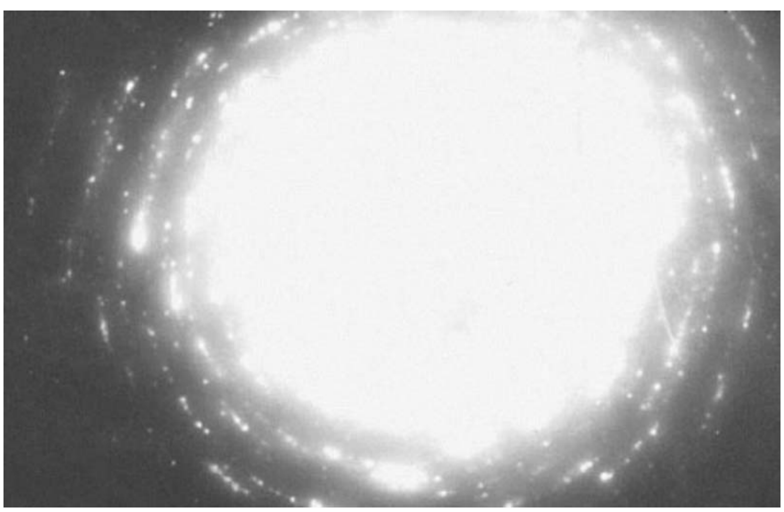
JIAN LU
Baoji Nonferrous Metals Works, Baoji, Shannxi 721014, People's Republic of China

HONGTAO MA
200 Broun Hall, Auburn University, AL 36849, USA
E-mail: mahongt@auburn.edu

PENGSHENG ZHANG
Northwest Institute for Nonferrous Metals Research, P.O. Box 51, Xi'an, Shaanxi 710016, People's Republic of China

Severe plastic deformation (SPD) is currently thought to be a promising method in grain refinement, especially in fabricating ultrafine-grained (UFG) metal bulk with less porous defect and no contamination [1–4]. There are various approaches based on SPD, for example, equal channel angular pressing (ECAP) [2, 3], high pressure torsion (HPT), accumulative rolling bonding (ABR) [5], repetitive corrugation and

(a)

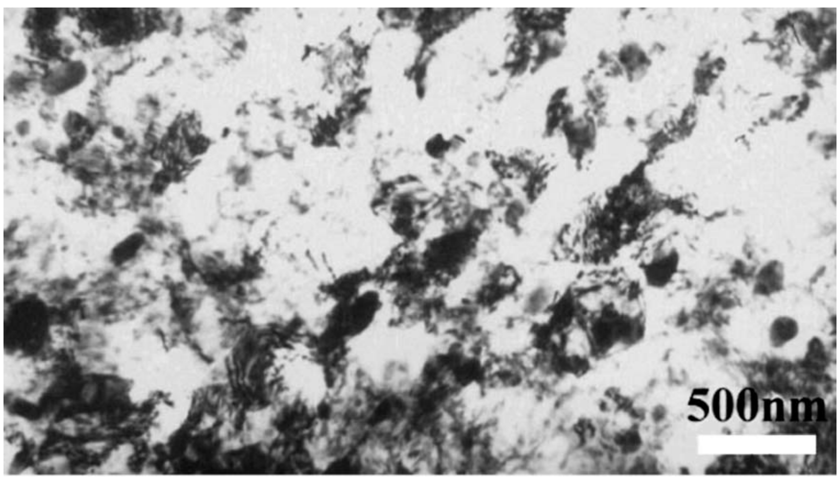


(b)

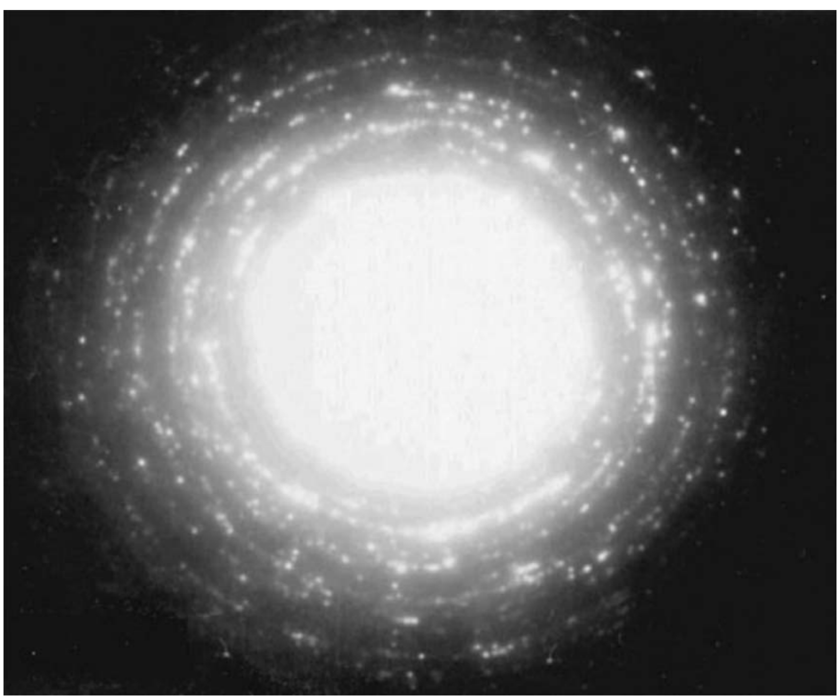
Figure 1 TEM feature of microstructure by continuous MAF at liquid nitrogen temperature: (a) microstructure and (b) the corresponding EDP.

straightening (RCS) [6], and multi-axis forging (MAF) [1, 6]. HPT can easily decrease the grain size of many metals to nanometers regime [1]. However, the mechanical property is inhomogeneous caused by the gradient deformation from center to peripheral part by HPT process [7], in addition it can only produce very small samples. MAF has some advantages over other SPD techniques [6, 8] in producing homogeneous large bulk with fine structure, but it has the limitation for grain refinement since the process is always carried out at elevated temperature. Lower temperature can hinder the dynamic recovery and consequently improve the grain refinement effects [6]. However, little work has been done so far to fabricate UFG metals by extreme low temperature MAF due to the poor workability for most metals at low temperature.

In this work, under the condition of constrained deformation, nanostructural grains have been formed by large MAF process at cryogenic temperature based on commercial 7075 aluminum alloy which is known for its poor ductility at low temperature. The attempt of this research is to give rise to the feasibility in fabricating nanostructural bulk metals with homogeneous structure by a novel method through studying the grain refinement. The composition of commercial 7075 aluminum alloy is Zn: 5.6 wt%, Mg: 2.3 wt%, Cu: 1.6 wt%, aluminum in balance. In current experiment there are two different deformations, continuous MAF and single axis compression forging, leading from high temperature to extreme low temperature. However, there is no annealing process during the entire procedure. Before cryogenic forging, hot extruded bulk of 7075 alloy was subjected to continuous MAF first at 430 °C, followed by MAF at room temperature to gain fine-grained structure. Then a sample in 40 mm × 40 mm × 40 mm was cut off from the bulk for continuous MAF after being immersed in liquid nitrogen for 15 min with copper clad. After continuous MAF smaller samples were cut

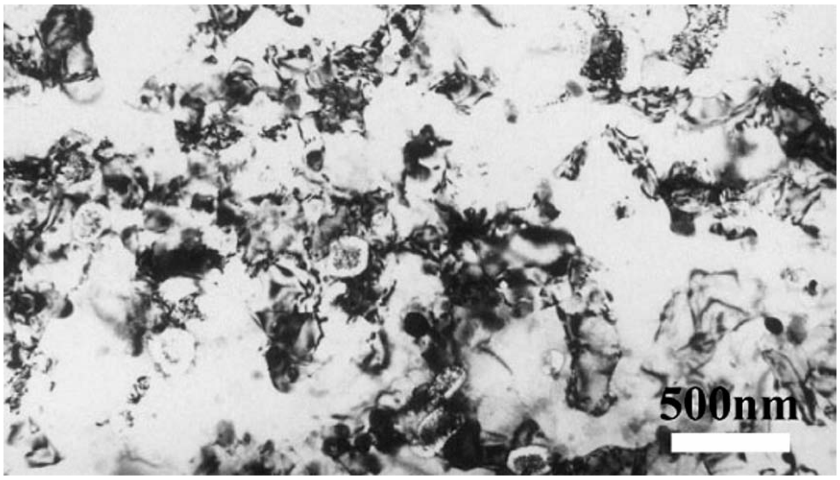


(a)

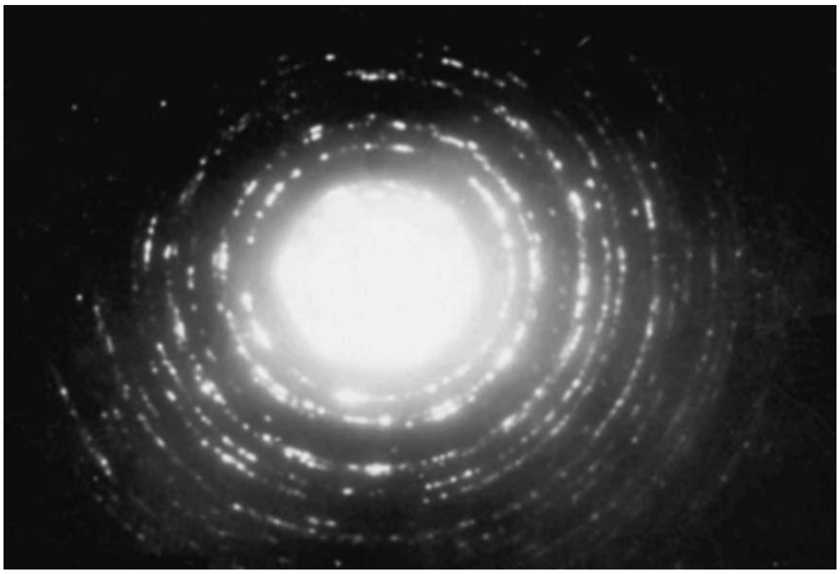


(b)

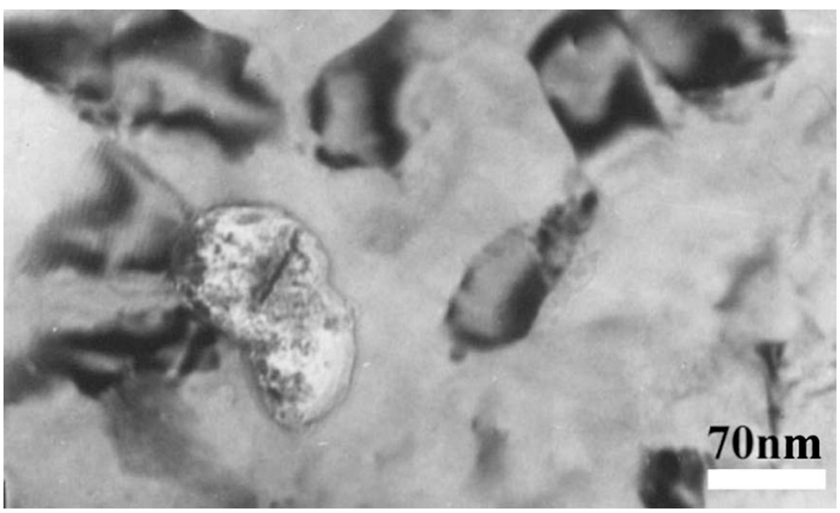
Figure 2 TEM microstructural morphology of first single compression forging at liquid nitrogen temperature: (a) microstructure and (b) the corresponding EDP.



(a)



(b)



(c)

Figure 3 The TEM microstructures of second single compression forging at liquid nitrogen temperature: (a) microstructure, (b) the corresponding EDP, and (c) feature of nanostructured grains.

off in dimension 10 mm × 10 mm × 10 mm with copper clad for a two-step compression forging. The samples were subjected to a single forging along height direction with reduction of over 60%, followed by a second single forging in a different deformation axis with reduction of over 60%. Each time before forging, samples were kept in liquid nitrogen for 15 min. The strain rate for all forging was 10^2–10^3 s^{-1}. Transmission electron microscopy (TEM) studies were conducted using Jeol 200CX microscope at 100 KV. Thin foils for TEM observations were prepared by jet electropolishing at room temperature.

Fig. 1 shows the TEM feature of microstructure of the sample subjected to continuous MAF at liquid nitrogen temperature. Equiaxed UFG grains can be found. The average size of grains was less than 500 nm. Extensive extinction contour in the vicinity of the boundary indicated the existence of internal stress. The near ring-like electron diffraction pattern (EDP) indicates the existence of the high angle boundaries as well as fine grains. Like typical microstructural UFG metals, few dislocations can be found within grains, even around particles, although dislocation cells can be observed on grain boundaries.

Fig. 2 gives the TEM microstructural morphology of the first single compression forging at liquid nitrogen temperature. The microstructure here is quite different from that as shown in Fig. 1 in that the sharp boundary of original equiaxed grains became obscure. However, elongated grains could be hardly observed (Fig. 2a). High density of dislocations and dislocation cells was found both in matrix and around particles. The ring-like EDP indicates the existence of much finer microstructure as well as an increase of misorientation between adjacent grains as showed in Fig. 2b.

Fig. 3 shows the TEM microstructures subjected to second single compression forging based on the first one. Homogeneous UFG structure was found (Fig. 3a). The previous high density of dislocations and dislocation cells became disappeared. The previous obscure microstructure became sharp boundary grains. Grains were still equiaxed. No elongated microstructure could be even observed. Grains in size were from less than 100 nm to a few hundred nanometers. The average grain size is less than 200 nm as shown in Fig. 3a which is much smaller than that in Fig. 1a. The apparent ring-like EDP compared with that in Fig. 2b indicates the more random misorientation between adjacent grains, as showed in Fig. 3b. Fig. 3c gives the morphology of grains in nanometers. The original large inclusion could be even broken into nanometer regime, as the light area in the center shown in Fig. 3c.

In this study, commercial 7075 aluminum alloy showed good compressive ductility during the constrained deformation at extreme low temperature by continuous MAF and two separate single forging processing without interval annealing. However, failure would occur during deformation at such low temperature without proper constrain. This experiment also demonstrates that large repetitive deformation at low temperature is possible for 7075 alloy, which is critical to further refinement. Tsuji *et al*. studied the microstructural evolution of UFG pure aluminum subjected to large deformation by compression at room temperature at different deformation speeds. They found that the heat generated from deformation coarsened the grains [9]. Our experiment showed that the extreme low temperature greatly hindered grain coarsening. This was also confirmed by other researchers [3, 6]. However, their results were all concluded from experiments of pure metals with good ductility, like pure aluminum and pure copper.

In summary, we obtained nanostructured bulk based on commercial 7075 aluminum alloy by MAF at liquid nitrogen temperature, which gives the possibility of gaining nanostructural grains with other alloys. The grain refinement could be furthered by repeating of multiple forging at cryogenic temperature without interval annealing.

References

1. R. Z. VALIEV, R. K. ISLAMGALIEV and I. V. ALEXANDROV, *Prog. Mater. Sci.* **45** (2000) 103.
2. V. M. SEGAL, *Meter. Sci. Eng. A* A **197** (1995) 157.
3. Y. IWAHASHI, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Acta Mater.* **46** (1998) 3317.
4. S. FERRASE, V. M. SEGAL, K. T. HARTWIG and R. E. GOFORTH, *Metall. Mater. Trans.* **28A** (1997) 1047.
5. Y. SAITO, H. UTSUNOMIYA, N. TSUJI and T. SAKAI, *Acta Mater*. **47**(2) (1999) 579.
6. J. Y. HUANG, Y. T. ZHU. H. JIANG and T. C. LOWE, *ibid*. **49** (2001) 1497.
7. A. P. ZHIYAEV, S. LEE, G. V. NURISHLAMOVA, R. Z. VALIEV and T. G. LANGDON, *Scripta Mater.* **44** (2001) 2753.
8. W. CHEN, D. FERGUSON and H. FERGUSON, in "Ultrafine Grained Materials," edited by R. S. Mishira, S. L. Semiatin, C. Suryanrayana, N. N. Thadhani and T. C. Lowe (TMA, Warrendale, PA, 2000) p. 235.
9. N. TSUJI, T. TOYODA, Y. MINAMINO, Y. KOIZUMI, T. YAMANE, M. KOMATSU and M. KIRITANI, *Meter. Sci. Eng.* A, in press.

Received 21 October
and accepted 13 November 2003