Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 22(2012) 1035−1040
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Superplastic extensibility deformation of Al−3%Mn alloy with submicrometer grain size

XIAO Xuan[1], GAO Ci[1,2], HOU Jie-shan[2], GUO Jian-ting[2]

1. School of Materials Science and Engineering, Shenyang Ligong University, Shenyang 110159, China;
2. Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

Received 19 April 2011; accepted 22 December 2012

Abstract: Submicrometer-grained (SMG) Al−3%Mn (mass fraction) alloy specimens with initial grain size of ~0.3 μm were produced by ball milling for 3 h. The Al−3%Mn specimens which were cold rolled with a strain rate of 1×10^{-3}− 1×10^{-2} s^{-1} at room temperature show high extensibility to failure more than 2500%. Microstructures of pure Al and Al−3%Mn alloy at as-milled and cold-rolled state were examined using X-ray diffraction and transmission electron microscopy (TEM). Based on the microstructure analysis, it is established that the mechanism of the continued plastic deformation in SMG Al−3%Mn alloy consists of dislocation slip, grain boundary sliding companied by dynamic recovery and recrystallization, and dynamic recrystallization is a main control factor of the large plastic deformation.

Key words: aluminium alloys; mechanical alloying; cold rolling; deformation structure

1 Introduction

Superplastic forming at a higher strain rate or lower temperature is highly desirable in industrial fabrication. There are obvious advantages in conducting superplastic forming at a lower temperature. For example, a lower forming temperature would save energy, improve the surface quality of the formed component, prevent severe grain growth and reduce the level of cavitation and solute loss from the surface layer, maintaining better post-forming properties [1]. It has been shown that Al−Mn alloys are highly suited to plastic deformation process, suggesting that they have potential for the development of wrought Al products. Microcrystalline materials with ultrafine grain size at submicrometer or nanometer scales possess a number of extraordinary physical and mechanical properties [1−9]. However, submicrometer-grained (SMG) materials have some advantages over nanocrystalline structures because bulk specimens are easy to be produced and no problems are associated with residual porosity [2,3]. In recent years, SMG alloys have been fabricated by severe plastic deformations [10], such as high-energy ball milling, electrodeposition, inert gas condensation and equal-channel angular pressing. Compared with other alloying methods, high-energy ball milling, which introduces very large plastic strains leading to grain refinement in coarse grained powder or bulk materials, is feasibility of addition of alloying elements for improvement of their mechanical and physical properties [11].

Plastic deformation in polycrystalline with ultrafine grain size involves dislocation movement, grain boundary motion, and recovery and recrystallization. Grain boundary mobility is commonly considered an important deformation mechanism at elevated temperature because the high activation energy is required for it. However, the occurrence of grain boundary motion at room temperature deformation of nanocrystalline metals has been confirmed by in situ observations recently [12,13].

Deformed metal or alloy contains a large stored energy which will normally revert to a low energy state through structural evolution during recovery and recrystallization [14]. The microstructural changes during recovery, which is primarily associated with dislocation reaction, are relatively homogeneous and usually do not affect the boundaries between the deformed grains. Recrystallization may occur when new defect free grains grow to consume the deformed or recovered microstructure. Moreover, the overlapping of

Corresponding author: XIAO Xuan; Tel: +86-24-83978469; E-mail: hou.js@sohu.com
DOI: 10.1016/S1003-6326(11)61280-6

the two processes may occur at some circumstances [15].

The present investigation are undertaken to explore extensibility behavior and the associated changes of microstructures during cold rolling of submicrometer grained Al−3%Mn alloy specimens prepared by ball milling for different alloying time at room temperature. In addition, observed microstructures leading to large extensibility of the alloy and mechanism of large extensibility are discussed.

2 Experimental

Elemental Al and Mn powders with a purity of 99.999% or better were used as the starting materials. Al−3%Mn (mass fraction) alloy specimens were prepared. The powders were sealed in a cylindrical stainless steel container under an Ar atmosphere. Mechanical alloying was carried out for 3 h on a high-energy mill, with a ball to powder mass ratio of 15:1. The as-milled powders were pressed under a pressure of about 640 MPa to form a small disk. Plastic deformation of the as-milled Al−3%Mn specimen was done by cold rolling (CR) and a piece of the Al−3%Mn sample (about 6 mm×6 mm×1 mm) cut from the as-milled sheet was rolled at room temperature with a twin-roller apparatus. The strain rate during rolling was controlled to $1\times10^{-3}-1\times10^{-2}$ s^{-1}. Cold rolling resulted in a continuous increase of sample length along the direction of rolling, while the sample width was kept a constant. During repeated rolling (total 6 cycles), the Al−3%Mn-3 h sample became longer and longer, and eventually formed a uniform, long thin ribbon with smooth surface and straight edges with out crack. The degree of deformation of cast pure Al (purity 99.992%) sample after rolling is very small in comparison with that of Al−3%Mn-3 h sample. And many cracks appear at the edges of as-rolled pure Al sample. The total degree of deformation in the sample of as-cast Al is 98%, and more than 2500% in the Al−3%Mn alloy.

The structures of as-milled and as-rolled samples were analyzed by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The X-ray diffraction (XRD) analysis was conducted using Cu K_α radiation with a D/Max-γ A diffractometer equipped with a graphite crystal monochromator. The TEM investigations were carried out on a Philips TEM 420 microscope at 200 kV.

3 Results and discussion

Figure 1 shows a stress—strain curve at room temperature at an initial strain rate of 6.5×10^{-3} s^{-1}. A slight hardness increases in the Al−3%Mn alloy at the initial stage of rolling, as shown in Fig. 1, and no strain hardening effect is seen with further rolling (ε>1000%). To examine the solubility of Mn in Al after ball milling, X-ray diffraction patterns were measured for both as-milled and rolled samples. Only diffraction peaks corresponding to FCC-Al are observed in XRD patterns of all the samples (Fig. 2). This indicates that Mn is very well dispersed in the FCC-Al; moreover, compared with pure Al, the shift of the positions of XRD peaks to low angles reveals the increase of the lattice parameter, further indicating that the Mn atoms are dissolved into the Al lattice. A supersaturated solid solution forms for all the samples.

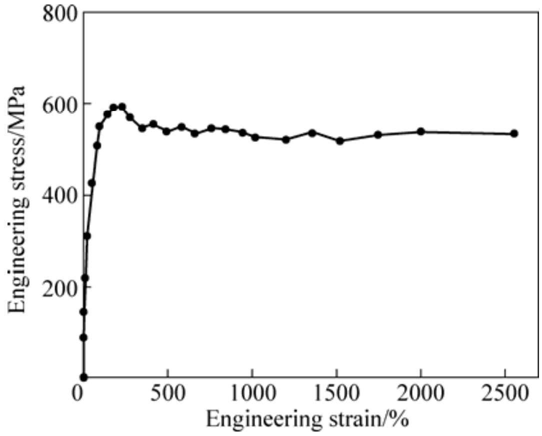

Fig. 1 Stress—strain curve of Al−3%Mn alloy during room temperature rolling at initial strain rate of 6.5×10^{-3} s^{-1}

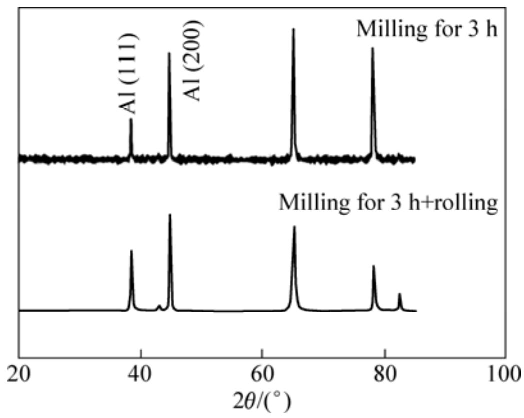

Fig. 2 X-ray diffraction patterns of as-milled Al−3%Mn specimen and milled rolled specimen

The TEM observations of the microstructures of polycrystalline pure Al and Al−3%Mn-3 h sample are shown in Fig. 3. The most common feature, a random distribution of equiaxed grains is observed in these specimens. The diameter of equiaxed grains in pure Al is in the range of 320−500 nm while diameter of equiaxed grains in Al−3%Mn-3 h is in the range of 200−400 nm. The grain boundaries of pure Al present smooth extinction contours, indicating a much low density of extrinsic grain boundary dislocations. And, very rare dislocation is observed within grains (Fig. 3(a)). In Al−3%Mn specimen, the grains and their grain boundaries are well defined with some parts of the boundaries containing extinction contours (Fig. 3(b)). It appears that the grain structure is not completely stable since many grain boundaries meet at acute angles (as arrows shown). In the Al−3%Mn sample, grains with high density dislocations are frequently found, as shown in Fig. 3(c). In this case, individual dislocation could hardly be distinguished, whereas dislocation tangles are distributed almost homogeneously and a few cell walls are observed. The grain boundaries can be well distinguished with extinction contours found at some parts of them. Figure 3(d) shows an example of the grains with sharply defined grain boundaries with no dislocation observed within the grains.

Transmission electron microscopy reveals that the microstructures after cold rolling are indicative of significant dynamic evolution.

For the cold rolled Al−3%Mn specimen, the typical structures are presented in Fig. 4. A reasonably equiaxed grain structure is observed in some areas shown in Fig. 4(a) and the grains markedly elongated are observed in other areas as shown in Fig. 4(b). Microdiffraction patterns of the elongated grains labeled *A* and *B* in Fig. 4(b) are given in Fig. 4(d) and (e), respectively. These patterns demonstrate that there are large misorientations between the individual elongated grains in Fig. 4(b), indicating the presence of large internal stress. In both of these structures, some grain boundaries with irregular extinction contours are visible along all or part of their length, indicating the nonequilibrium character of the boundaries, as reported also for other SMG alloys [2,16]. The dark contrast near grain

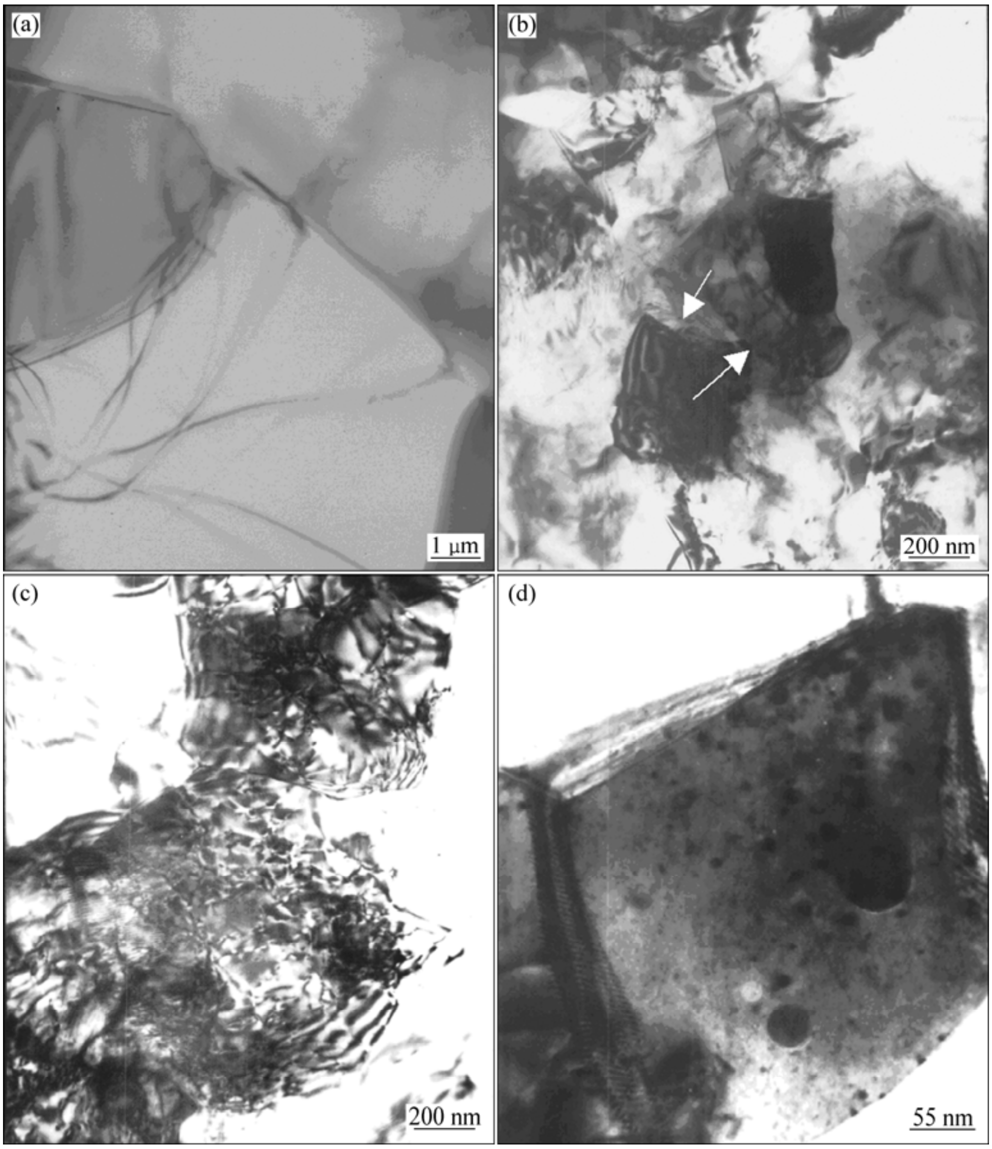

Fig. 3 TEM microstructures of pure Al (a), Al−3%Mn with equiaxed grains (b), dislocation cells and tangles (c) and sharply defined grain boundaries (d)

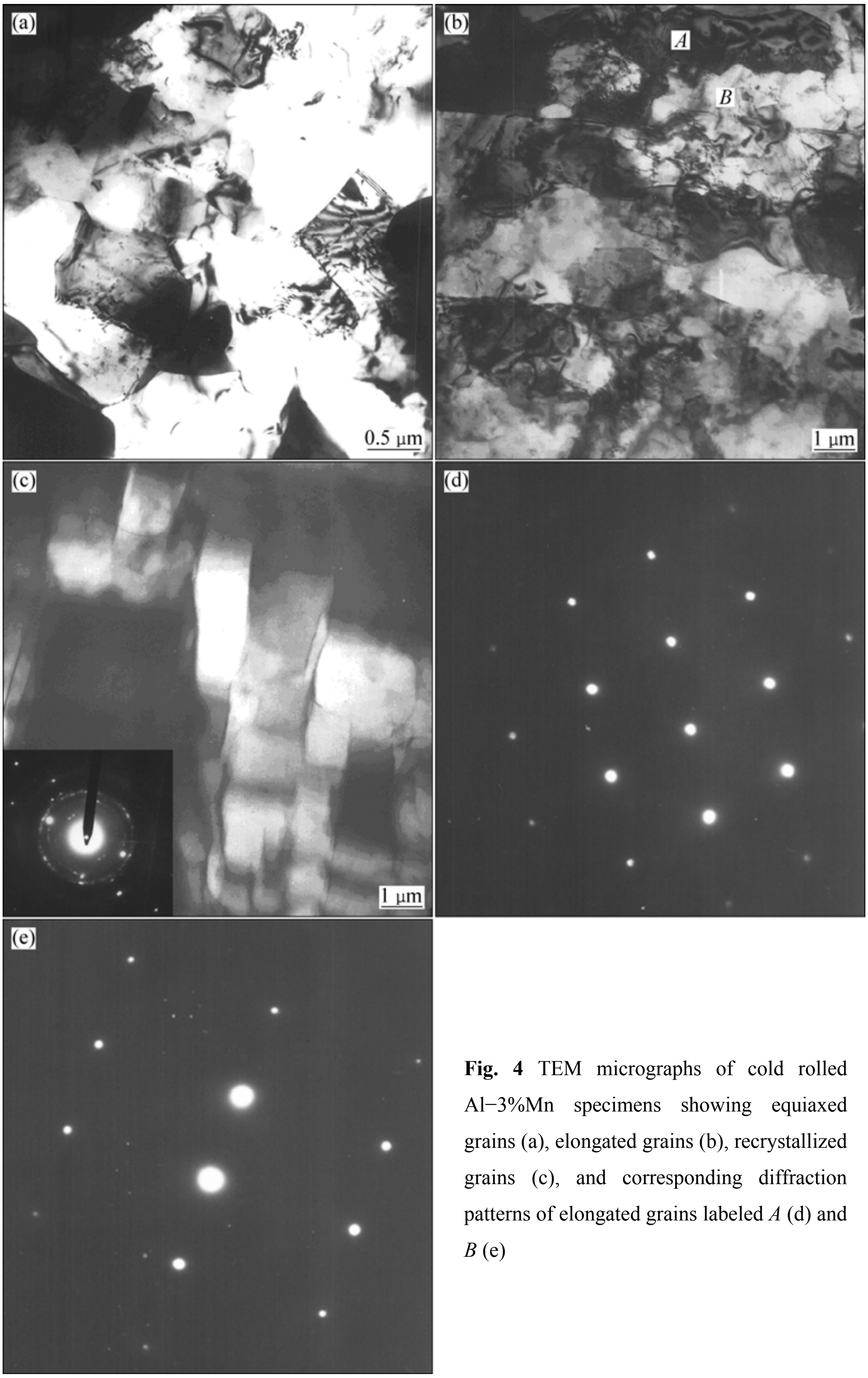

Fig. 4 TEM micrographs of cold rolled Al−3%Mn specimens showing equiaxed grains (a), elongated grains (b), recrystallized grains (c), and corresponding diffraction patterns of elongated grains labeled *A* (d) and *B* (e)

boundaries in parts of the micrographs suggests a high dislocation density. And, some fine subgrains, which should be recrystallized grains, are observed in some elongated grain structure. Figure 4(c) shows a well-developed recrystallized microstructure with the grain boundaries exhibiting low misorientations.

Under tensile test condition, two fundamental requirements must be fulfilled in order to achieve superplasticity in conventional metals: 1) a small grain size, typically less than 10 μm, and 2) a temperature of test above $0.5T_m$ (where T_m is the absolute melting temperature) [16]. However, a further reduction in grain size, to the submicron level or lower, offers the potential advantages of increasing the overall ductility prior to failure while at the same time decreasing the temperature associated with superplastic flow. Similar tendency occurs under cold-rolling test condition. During rolling at room temperature, conventional coarse-grained polycrystalline Cu exhibits an extension of 800%, while nanocrystalline Cu prepared by electrodeposition technique exhibits a superplastic extensibility of 5100% (measured with the change of thickness) [17]. In our investigation, the elongations exceed 2500% during cold rolling at room temperature. Such a high extensibility maybe attribute to the microstructural characteristics and low strain rate (about 10^{-3} s^{-1}).

The TEM observations show that the large extensibility of Al−Mn alloy is related to several deformation mechanisms. The grain boundary diffusion in SMG aluminum alloys is accelerated, compared with that of aluminum alloys with fine grains (1−10 μm) [3]. Commonly, the mechanisms of the superplasticity of aluminum-based alloys with SMG microstructure are related to grain boundary sliding and intragranular deformation during enhanced diffusion. When grain boundary sliding or slip plays a decision role, the microstructure during superplastic deformation will consist of dislocation-free grains, as confirmed in many superplastic alloys. TEM images (Fig. 4) show a rare distribution of dislocations within the grains but high density close to the grain boundaries, suggesting that the dislocations originate from the grain boundaries. Detailed observation indicates that some of the well-developed boundaries have low misorientations, which is beneficial to the grain boundary sliding or slip. Thus, it is reasonable to conclude that grain boundary sliding plays an important role in producing large extensibility in our alloys. The presence of large internal stress between grains also gives evidence for the grain boundary sliding (Fig. 4(b)). Furthermore, dislocation slip is also a control factor during plastic extensibility deformation in the Al−3%Mn specimen. Figure 4(b) shows an area of elongated grains, indicating that the active dislocation slip of the alloy definitely contributes to superplastic deformation. At as-milled state, the Al−3%Mn specimen shows high density of dislocations within some grains (Fig. 3(c)). During cold rolling, the dislocation slip in grains leads to the directional elongation of the grain. Under this condition, the grain boundaries act as a source for dislocation nucleation and growth under the deformation conditions.

From above observations, it can be seen that the cold-rolled Al−3%Mn specimen shows typical dislocation- free bands with an average width of ~0.12 μm, typical recrystallized microstructure. It is the most interesting that the recrystallized temperature in SMG aluminide alloy is close to room temperature, which is lower than that in conventional aluminide alloy. It should be attributed to the raw material, high purity Al powder, which can markedly decrease the recrystallized temperature [5,18]. In the TEM tests (Fig. 4(b)), clear subboundaries are observed, which means that dynamic recovery has occurred during superplastic deformation of this material. After rolling several cycles, the equiaxed grain size of Al−3%Mn-3 h specimen grew to 0.6−0.9 μm, with the elongated grain in a width of 0.3−0.5 μm. Thus, during cold rolling, dynamic recovery and recrystallization led to a network of highly nonequilibrium grain boundaries through dislocation rearrangement and subsequently these boundaries transformed gradually into a more equilibrated configuration accompanied by some limited grain growth.

Strain hardening was not pronounced in the SMG Al−3%Mn alloys during cold rolling, owing to the absorption of dislocation by grain boundaries because of accelerated diffusion. On the other hand, the cavity behavior is important because suppression of the formation and growth of cavities is directly related to the large extensibility. The general model of cavitation, based on the idea that stress concentrates at a grain boundary which is accompanied by grain boundary sliding and results in decohesion of the boundary interface, is commonly accepted [19]. During deformation, dynamic recovery is an effective accompany process to relive such concentration stresses [20]. Considering the competition between the opening of a cavity by grain boundary sliding and concentration of stress, the cavity will hardly nucleate when the latter is predominated. So, the mechanism for continued plastic deformation in SMG Al−3%Mn alloy should be the grain boundary sliding and dislocation slip accompanied by dynamic recovery and recrystallization. Moreover, it needs to be noted that dynamic recrystallization is a main control factor in the Al−3%Mn alloy.

4 Conclusions

1) The SMG Al−3%Mn alloys produced by ball milling exhibit high plastic extensibility to failure during cold rolling at low strain rate and room temperature. Appropriate alloying time, e.g. 3 h, leads to the extensibility towards the value of more than 2500%.

2) Microstructure analysis of milled and rolled alloys indicates that mechanism of continued plastic deformation in SMG Al−3%Mn involves dislocation slip, grain boundary sliding companied by dynamic recovery and recrystallization, and dynamic recrystallization is a main control factor of the large plastic deformation.

References

[1] CHOKSHI A H, MUKHERJEE A K, LANGDON T G. Superplasticity in advanced materials [J]. Mater Sci Eng R, 1993, 10: 237−274.
[2] WANG J, IWAHASHI Y, HORITA Z, FURUKAWA M, NEMOTO M, VALIEV R Z, LANGDON T G. Enhanced grain growth in an Al−Mg alloy with ultrafine grain size [J]. Mater Sci Eng A, 1996, 216: 41−46.
[3] VALIEV R Z, KRASILNIKOV N A, TSENEV N K. Plastic deformation of alloys with submicron-grained structure [J]. Mater Sci Eng A, 1991, 137: 35−40.
[4] LIU F C, MA Z Y. Contribution of grain boundary sliding in low-temperature superplasticity of ultrafine-grained aluminum alloys [J]. Scripta Mater, 2010, 62: 125−128.
[5] MA Z Y, LIU F C, MISHRA R S. Superplastic deformation mechanism of an ultrafine-grained aluminum alloy produced by friction stir processing [J]. Acta Mater, 2010, 58: 4693−470.
[6] SHENG L Y, ZHANG W, GUO J T, WANG Z S, OVCHARENKO V E, ZHOU L Z, YE H Q. Microstructure and mechanical properties of Ni_3Al fabricated by thermal explosion and hot extrusion [J]. Intermetallics, 2009, 17: 572−577.
[7] LEE S, UTSUNOMIYA A, AKAMATSU H, NEISHI K, FURUKAWA M, HORITA Z, LANGDON G T. Influence of scandium and zirconium on grain stability and superplastic ductilities in ultrafine-grained Al−Mg alloys[J]. Acta Mater, 2002, 50: 553−564.
[8] XU C, FURUKAWA M, HORITA Z, LANGDON T G. Severe plastic deformation as a processing tool for developing superplastic metals [J]. Journal of Alloy and Compound, 2004, 378: 27−34.
[9] MA Y, FURUKAWA M, HORITA Z, NEMOTO M, VALIEV R Z, LANGDON T G. Significance of microstructure control for superplastic deformation and forming [J]. Mater Trans JIM, 1996, 37: 336−339.
[10] LANGDON T G. The characteristics of grain refinement in materials processed by severe plastic deformation[J]. Rev Adv Mater Sci, 2006, 13: 6−14.
[11] SHENG L Y, XI T F, LAI C, GUO J T, ZHENG Y F. Effect of extrusion process on microstructure and mechanical properties of Ni_3Al-B-Cr alloy during self-propagation high-temperature synthesis [J]. Transactions of Nonferrous Metals Society of China, 2012, 22: 489−495.
[12] SOER W A, de HOSSON J T M, MINOR A M, MORRIS J M Jr, STACH E A. The effect of solute Mg on grain boundary and dislocation dynamics during nanoindentation of Al−Mg thin films [J] Acta Mater, 2004, 52: 5783−5790.
[13] SHAN Z W, STACH E A, WIEZOREK J M K, KNAPP J A, FOLLSTAEDT D M, MAO S X. Grain boundary mediated plasticity in nanocrystalline nickel [J]. Science, 2005, 308: 356−357.
[14] HUMPHREYS F J, HATHERLY M. Recrystallization and related annealing phenomena [M]. Oxford, UK: Pergamon, 1996.
[15] VANDERMEER R A, GORDON P. The influence of recovery on recrystallization of aluminum [C]//HIMMEL L. Recovery and Recrystallization of Metals. New York: Interscience Publishers, 1963: 211.
[16] ZHOU F, LIAO X Z, ZHU Y T, DALLEK S, LAVERNIA E J. Microstructural evolution during recovery and recrystallization of a nanocrystalline Al−Mg alloy prepared by cryogenic ball milling [J]. Acta Mater, 2003, 51: 2777−2791.
[17] LU L, SUI M L, LU K. Superplastic extensibility of nanocrystalline copper at room temperature [J]. Science, 2000, 287: 1463−1466.
[18] LANGDON T G. The mechanical properties of superplastic materials [J]. Metall Trans A, 1982, 13: 689−701.
[19] NIEH T G, WADSWORTH J, SHERBY O D. Superplasticity in metals and ceramics [J]. Trans AIME, 1968, 242: 654−660.
[20] SHERBY O D, WADSWORTH J. Superplastic-recent advances and future direction [J]. Prog Mater Sci, 1989, 33: 169−221.

亚微米晶 Al−3%Mn 合金的超塑延展性变形行为

肖 旋[1]，高 慈[1,2]，侯介山[2]，郭建亭[2]

1. 沈阳理工大学 材料科学与工程学院，沈阳 110159；
2. 中国科学院 金属研究所，沈阳 110016

摘 要：采用球磨法制备晶粒尺寸为 0.3 μm 的亚微米晶 Al−3%Mn(质量分数)合金。Al−3%Mn 合金在室温下轧制时，表现为极高的延展性(超过 2500%)。采用透射电镜(TEM)观察球磨态和冷轧态的纯铝和 Al−3%Mn 合金组织；采用 X 射线衍射对比分析组成，发现连续塑性变形机制包括位错滑移和晶界滑动，同时还有动态回复和再结晶，而动态再结晶是大塑性变形的主要控制机制。

关键词：铝合金；机械合金化；冷轧；变形组织

(Edited by YANG Hua)