INFLUENCE OF HIGH-PRESSURE TORSION ON MECHANICAL PROPERTIES AND MICROSTRUCTURAL EVOLUTION IN 2197 Al-Li ALLOY

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Abstract

2197 (Al-Li-Cu-Zr-Mn) alloy has been processed by High-pressure torsion (HPT) at applied pressure P of 2GPa and different shear strain γ in the range of 3 turns and 10 turns. Optical microscope (OM) and transmission electron microscope (TEM) provided the detail information of grain size and microstructure of the alloys. Vickers indentation analysis was used to evaluate the micro-hardness of deformed samples. Tension test was employed to obtain the strength σ_b and elongation δ at room temperature. The result show that nice microstructure and properties are achieved when the pressure is 2GPa and the shear strain γ is 10 turns, for the grain sizes in the range of 0.1~0.4 μm, micro-hardness up to 163HV0.2, and σ_b at 591.005 MPa and elongation δ of 8.52%, higher than the peak aging treatment (T8) state of 474.7MPa.

Introduction

Severe Plastic Deformation (SPD) is defined as a metal forming method under an extensive hydrostatic pressure that usually be used to impose a very high strain on bulk solid without the introduction of any significant change in the overall dimensions of the sample and having the ability to produce exceptional grain refinement [1]. Processing through the application of SPD, in procedures, such as high-pressure torsion (HPT), equal-channel angular pressing (ECAP), accumulative roll-bonding (ARB), were developed and successfully applied to obtain an ultra-fine grain (UFG) structure in various materials. The ultra-fine grain structure determines extraordinary properties of such materials [2, 3].For instance the phenomenon of high strain rate superplasticity was demonstrated for the UFG aluminum-based alloys after the ECAP deformation according to the study of R. Z. Valiev [4]. Another example is the nanostructural hierarchy has been observed in aluminium alloy under the research of Peter V. Liddicoat, et al [5]. However, UFG materials are characterized by high internal stresses and non equilibrium structure features. All this factors make them sensitive to the thermal treatment and significant to the commercial application.

The aim of this work is to study the structure and mechanical properties in 2197 Al-Li alloy formed by HPT, its evolution during deformation and also its stability in the course of heat treatment.

Experimental

An ingot of 2197 (Al-Li-Cu-Mn-Zr) alloy was prepared with high-purity Al (99.999%), Li, and intermediate alloys Al-Cu, Al-Zr, Al-Mn using an arc-melting furnace in an argon atmosphere. The chemistry analysis gave a composition in wt% of 1.43% Li, 2.63% Cu, 0.11% Zr, and 0.13% Mn. The ingot was given homogenizing treatment at 530°C for 24h and then water quenching. Disk shaped samples suitable for HPT were cut by spark erosion from the ingot. The diameters of the torsion samples were 10mm, their heights were 2.5mm. HPT was conducted at room temperature with an applied pressure of 2GPa. The lower anvil was machined with a disk shaped depression at the center having a depth of 1.0mm and a diameter of 10mm to impose restrictions on samples, and was rotated with respect to the upper at a rotation speed of 2 rpm for 3, 7 and 10 revolutions. After that, the samples were treated under 450°C for 2h for annealing.

Microstructures were observed using optical microscopy (OM) and transmission electron microscopy (TEM). Mechanical properties were evaluated using Vickers micro-hardness measurement, tensile test. For OM, after polishing the disk samples mechanically to a mirror-like surface, the samples were etched by solution composed by 5% HF and 95% H_2O. For TEM, disks with 3mm in diameter were punched out from the outer part of the HPT samples and ground mechanically to a thickness of 100nm. They were further thinned in a solution of 30%HNO_3-70%CH_3ON under an applied voltage of 5-15 V at a temperature of 253K (-20°C) using a twin-jet electropolishing apparatus. An F-20 transmission electron microscope was operated under an accelerating voltage of 200 kV. The values of Vickers micro-hardness, HV were evaluated systematically using an HXZ-1000 micro-hardness tester with a load of 200g and a dwell time of 10s. Measurements were taken from the center to edge of each disk at position separate by 0.5mm and with four separate hardness measurement recorded at each position. Miniature tensile specimens having 2.5mm gage length, 1mm width and 0.8mm thickness were cut from the 10mm disks at the position of 1.70mm away from the center. Since the machine may introduce surface defect, the tensile samples were carefully polished after the machine. Each specimen was pulled to failure using a tensile testing machine with an initial strain rate of 0.05mm/min.

Results and Discussion

Investigated material comprised of the coarse grain of low dislocation density and the grains were reasonably equiaxed with an average size of about 213μm, as shown in Fig.1 (a). Using HPT method for refining structure to nanostructure size needs the application of large strain, usually with an equivalent strain ε_{eq} more than 10 (according to the relation $\varepsilon_{eq} = 2\pi N r / t\sqrt{3}$ (1), where N is the torsion revolutions, r is the distance from the center of the disk and h is the thickness of the disk [6]). Electron microscopy analysis of samples prepared from deformed disks exposed to different strains revealed formation of various structural characteristics in relation to strain applied and selected location on the disk.

Fig. 1 shows microstructures near the edges of the disks after straining through (b) 3 turns, (c) 7 turns and (d) 10 turns, respectively. After rotation through 3 turns, the original grain can be seen indistinctly, but the grains were too small to be identified.

With the increasing of revolutions number, the homogeneities in the microstructure of material tend to be in-homogeneity. After homogenizing treatment, the majority of globular phase δ' (Al_3Li) and δ (AlLi) dissolved into the matrix. A small quantity of phase δ' can be seen mainly in the boundary. Deformed by HPT through 7 turns under 2 GPa, tiny phase δ' and δ appeared again. Because of the small quantity of element Zr, the phase β' (Al_3Zr) deformed as dispersoid particles [7]. Globular dispersoid particles have the pinning effect on the grain boundary, and are benefit for refining grain and inhibition of recrystallization. The grain was too small to be observed by OM, and TEM method was employed.

The TEM photos were shown in Fig. 2. After deformed by HPT, the grains were refined to under 200nm. After rotation through 3 turns, there was a high dislocation density in the center of the disk but the grains were equiaxed. But when the rotations number was increasing, the grains turned to be anisometric. The microstructure consists of small grains with sub-micrometer sizes and many of the grains are surrounded by curved or ill-defined grain boundaries. All of the SAED analysis exhibits well-defined ring patterns, indicating that the grain boundaries are in high angles of misorientation and the grain size was nanometer lever. Energy spectrum analysis shows that linear T_1 (Al_2CuLi) phase precipitated in the grain internal and at the grain boundaries when the pressure is 2GPa. With increasing the revolutions number, the grain size was decreasing, and the dislocation density was increasing when N = 7, and then decreasing when N = 10.

All the materials processed by SPD method were sensitive to the thermal treatment. To examine the thermostability, the samples produced by HPT were annealing treated at 450°C for 2h. Fig. 3 shows the microstructure taken by OM of annealed. From Fig. 3 (a), globular phase δ' of uneven size, abundant of the dark oblique needle phase, along with perpendicular needle phase has dissolved out inside the grain and at the grain boundary. It is certain that two needle phases have precipitated: plenty of T1 phase (Al_2CuLi) and a small quantity of θ' phase (Al_2Cu). When the revolution number was increasing to 7, the needle phase T1 and θ' tends to be homogenization, and the quantity of global phase δ' precipitated on the boundary was increasing, as shown in Fig. 2 (b). Looking at Fig.2 (c), the needle phase was shorter and thicker. Due to exist of Zr in 2197 Al-Li alloy, that the δ' phase cladding on the T1 phase formed δ'/ T1 compound phase. The grain size was about 50μm. Compared with the samples without HPT process, the disks has more homogenizing and smaller needle phase.

Mechanical properties

According to the equation (1), the deformation degree at HPT depends on the radius and the thickness of the disk. If the disks have the same thickness as in our case, the deformation degree increases with increasing distance to the center of disk that leads to changes of the structure and properties. In this connection, heterogeneity of microhardness values of disks under hydrostatic pressure of 2GPa, after 3, 7, 10 revolutions was studied.

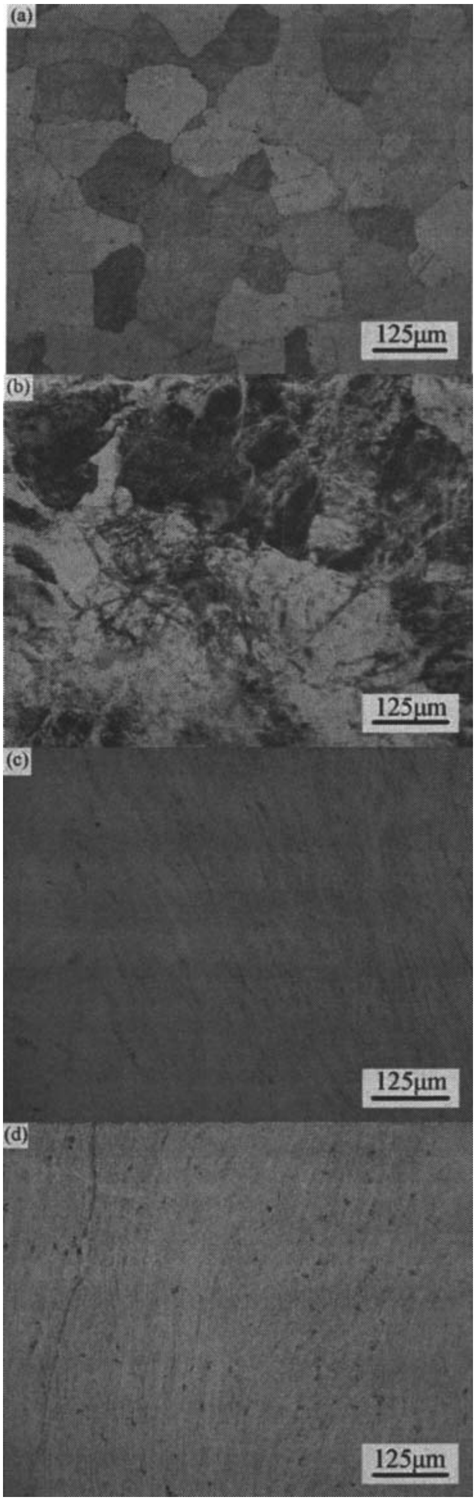

Figure 1. Microstructures in the peripheral regions of disks (a) unprocessed and processed by HPT through (b) 3 turns; (c) 7 turns and (d) 10 turns under pressure of 2GPa taken by OM.

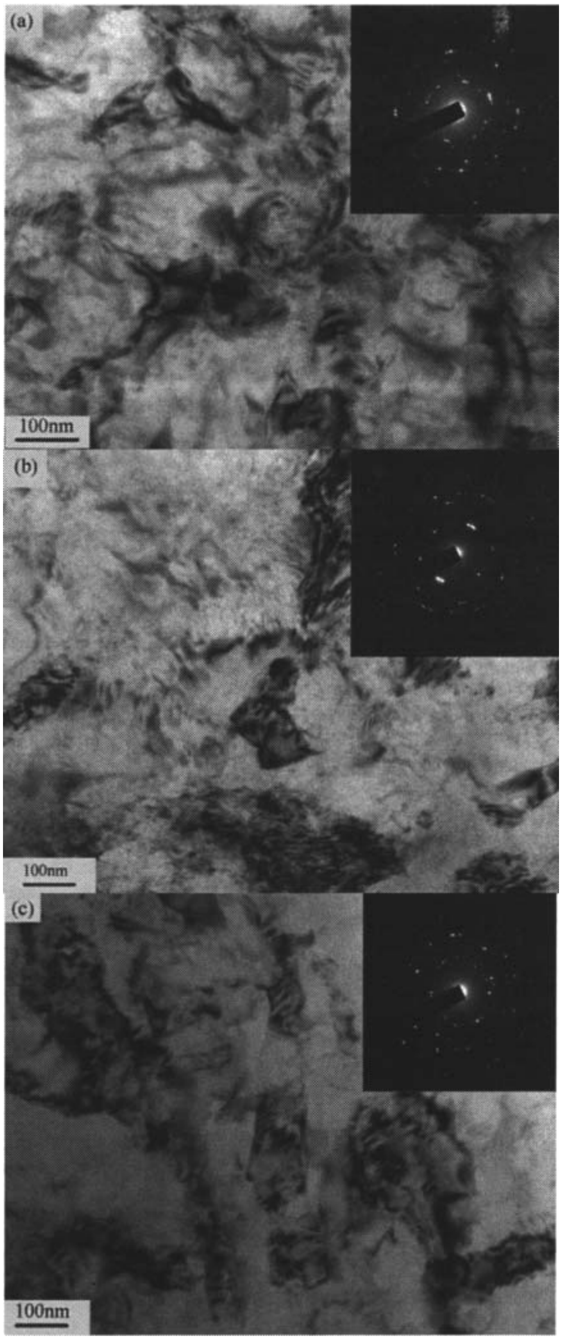

Figure 2. Microstructures of disks in the peripheral regions processed by HPT after (a) 3 turns, (b) 7 turns and (c) 10 turns under pressure of 2GPa.

A complete description of hardness data can be achieved by potting the hardness values of the disks against the distance from the disk center. Fig. 4 shows hardness values of different testing conditions used in this investigation.

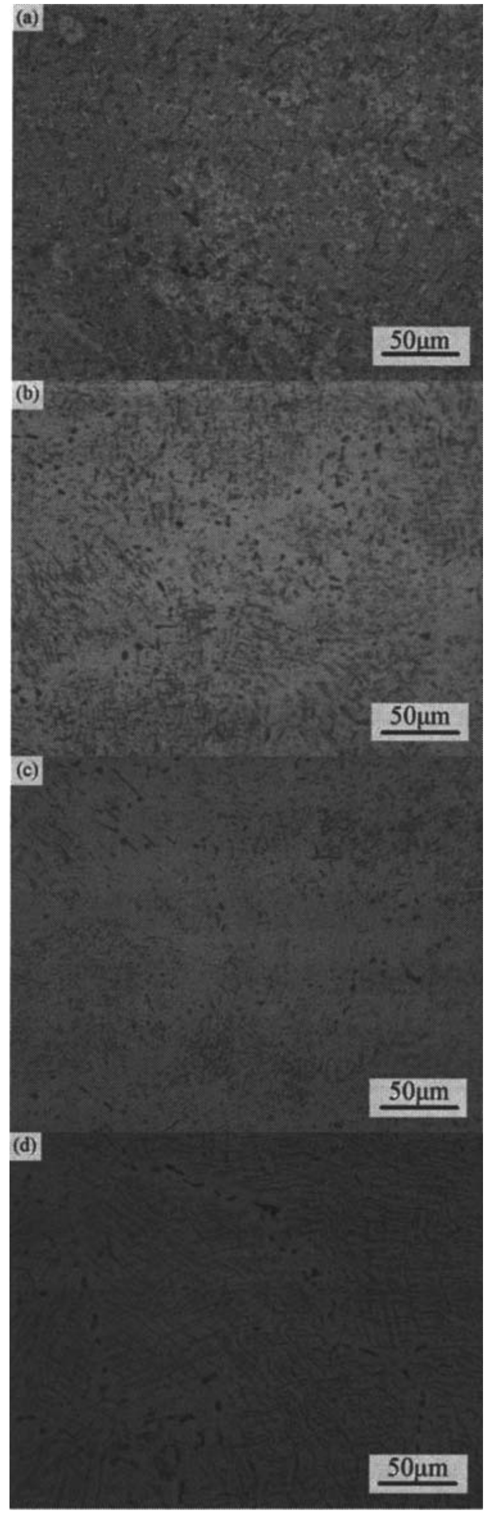

Figure 3. Microstructures disks annealed at 450°C for 2h through (a) 3 turns; (b) 7 turns; (c) 10 turns under pressure of 2GPa after processed by HPT; (d) without HPT processing taken by OM.

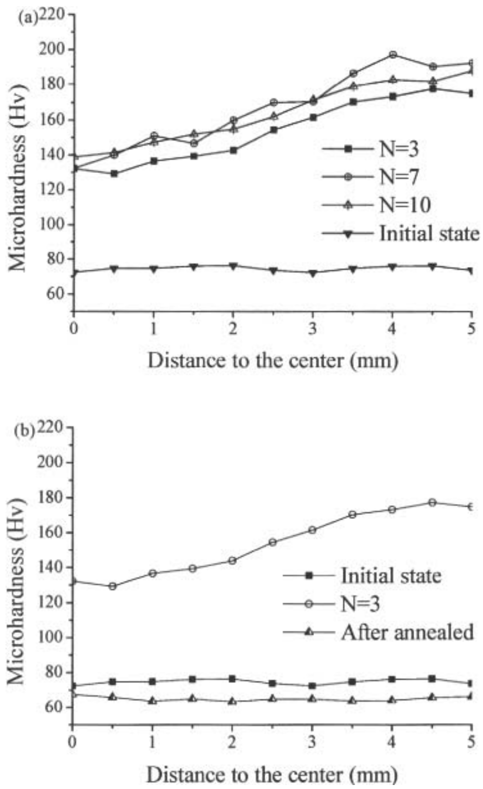

Figure 4. The average Vickers micro-hardness, Hv, versus distance from the center of the 2197(Al-Li-Cu-Zr-Mn) alloy disks: (a) disk unprocessed by HPT, and disks after 3, 7, 10 revolutions under hydrostatic pressure of 2GPa; (b) disks unprocessed by HPT, disks produced by HPT under 2GPa after 3 revolutions, and disks been annealed.

Table I. Values of Ultimate Strength and Elongation of Different States

Samples		Ultimate strength, MPa	Elongation, %
Initial state	0T	308.649	26.30
P=2GPa	3T	586.381	6.43
	7T	588.715	8.43
	10T	591.005	8.52
Annealed	10T	360.430	26.29

The bottom line of noted initial state denotes the unprocessed condition. It is apparent from Fig.4 (a) that (1) hardness increases sharply after processed by HPT, increasing more than 50%; (2) hardness increases with the distance from the disk center for all numbers of revolutions; (3) hardness increases with the numbers of revolutions under 2GPa; (4) hardness of the disks center were more or less the same. That is because according to the equation (1), in the center of the disk, the strain was zero. That was equivalent to applying pressure to the disks without any torsional. That was consistent with the microstructure observed in Fig. 2.

After processed by HPT, the disks were annealing treated at 450°C for 2h. The grain size increased obviously, as shown in Fig. 4 (b). The hardness tends to be homogenizing and decreased sharply compared with the state produced by HPT and the homogenizing treated ingot. The values of other conditions were more or less the same.

Values of tensile strength and elongation to failure acquired from tensile are shown in table 1. Compared with the initial state of 308.649MPa, the ultimate strength increased sharply to 591.005MPa when processed by HPT. The value is higher than the aging treatment peaking state and some high strength aluminum alloy [7]. The maximum elongation was 8.52%. Although it was decreased, it was good enough. With the increasing of rotation number, the strength was increasing. If increasing the rotation number or the pressure, it is hoped that the ultimate strength would break though 650MPa. After annealed, the ultimate strength of samples decreased to 360.430MPa, but the elongation increased at the same time.

Discussion

High-pressure torsion method at increasing rotation number was applied to refine microstructure. After processed by HPT the globular phase δ' (Al_3Li) and β' (Al_3Zr) precipitated out. Globular dispersoid particles have the pinning effect on the grain boundary, are the benefit to grain refining and inhibition of recrystallization. And inhibiting recrystallization can make alloys presenting strong texture. The increasing of strength was due to the grains refining as well as the globular dispersoid particles' pinning effect. Hardness values would reach to saturation point when the pressure and rotation number increased. After the annealed, according to Fig.3 and Fig.4, the grains grew obviously, and needle phase T1 and θ' are responsible to the decrease of hardness value. Also they maybe the cause of elongation's mounting up. The globular phases may be responsible to the increase of hardness value when processed by HPT along with the work hardening.

Conclusions

2197 Al-Li alloy material was successfully processed at room temperature using HPT and the following conclusions were obtained.

1. High-pressure torsion method is an effective method to refine microstructure. True nanometer grain sizes of <200 nm were achieved in the alloy after produced by HPT when pressure was 2GPa, revolution number >7.
2. The analysis of microstructure shows that lobular dispersoid particles' pinning effect was equally important to the grain refinement to the HPT strengthening function. In addition, the needle phase T1 and θ' in annealed samples tends to be homogenization and smaller after HPT process. They are also the cause of the elongation increase.
3. Microhardness measurements were taken after HPT on disks subjected to a range of rotation strains from 3 turns to 10 turns. There was a significant strengthening after 3 turns. Radially inhomogeneous hardness values were shown and the hardness values increased to a maximum of 190HV0.2 at the edge of the disk sample. According to the tensile test

results, after processed by HPT, the Ultimate strength was increasing sharply to 591.005.

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