Effect of severe plastic deformation on the structure and mechanical properties of Al-Cu-Mg alloy

E Khafizova and R Islamgaliev
Ufa State Aviation Technical University, 12 K. Marx Street, 450000 Ufa, Russian Federation
E-mail: ela.90@mail.ru

Abstract. Aluminum Al-Cu-Mg alloy has been subjected to high pressure torsion (HPT) and equal-channel angular pressing (ECAP) at various temperatures. An ultrafine-grained (UFG) structure thermally stable up to a temperature of 175 ºC was produced in all the investigated samples. Simultaneous increase in strength and ductility has been demonstrated in an ECAPed sample in comparison with a coarse-grained sample subjected to standard treatment.

1. Introduction
Al-based alloys as structural materials arouse large interest in the world scientific community due to their light weight and low self-cost. In particular heat-resistant alloys of the Al-Cu-Mg system are very interesting; they are employed to manufacture articles operating at high temperatures. The ultimate tensile strength of these alloys in a coarse-grained state after standard treatment is about 400 MPa. Severe plastic deformation (SPD) can be an effective way to enhance strength and fatigue properties. SPD enables fabricating an ultrafine-grained (UFG) structure in different metal materials [1-3].

It is known that formation of the UFG structure in pure metals can lead to enhancement of strength characteristics. However, the grain refinement in aluminum alloys by SPD techniques has a number of important features because of additional precipitation of second phases and solid-solution hardening, which can result in lower ductility. In particular, in the temperature range 20-150 ºC the quenched samples of aluminum alloys demonstrate decreased deformation ability in comparison with pure metals, and under SPD the samples and billets fail even at the initial stages of deformation [4].

All this creates additional possibilities for enhancing the properties of aluminum alloys, but requires optimization of technological regimes for fabrication of bulk UFG billets to be used as structural materials.

The aim of this work is to increase strength characteristics with retention of sufficient ductility of the alloy of the Al-Cu-Mg system via refinement of grain structure by SPD techniques at different temperatures.

2. The material and research methods
Al-based alloy of the Al-Cu-Mg system cast in USATU was chosen as material for studies. The chemical composition of the alloy was analyzed by an optical and emission technique and is presented in Table 1.
Table 1. Chemical composition of the Al-Cu-Mg, wt. %

|     | Al   | Cu   | Mg   | Mn   | Fe   | Si   | Ni   | Zn   | Ti   | Impurities |
|-----|------|------|------|------|------|------|------|------|------|------------|
|     | 92.8075 | 5.6935 | 0.6935 | 0.3375 | 0.171 | 0.059 | 0.012 | 0.139 | 0.0285 | 0.0584 |

Before SPD, the Al-Cu-Mg billets were subjected to solid solution treatment at 530 ºC for 5 hours followed by water quenching. Disks with a diameter of 20 mm, a thickness of 1 mm were the initial samples to be processed by high pressure torsion (HPT). Cylinders of 20 mm in diameter and 150 mm in length were subjected to equal channel angular pressing (ECAP).

HPT was carried out on a set that is the developed design of the known Bridgman anvil [5]. The sample was placed between the anvils and pressed under an applied pressure of 5 GPa. As a result of surface friction force arising during the bottom anvil rotation, the sample was deformed by shear in the conditions of hydrostatic compression under the action of applied pressure. The disk-shaped samples were subjected to 3 HPT rotations at 20 ºC. Several samples were subjected to combined HPT treatment: 3 HPT rotations at 20 ºC and then additionally 2 HPT rotations at 150 ºC. The combined treatment was chosen on the basis of earlier studies of the HPT effect on the aluminum AK4-1 alloy structure [6]. It was established that more intensive refinement of grain structure took place due to precipitation of strengthening phases during straining at temperatures close to aging ones. During ECAP a workpiece was repeatedly pressed in a special die-set through two channels with identical cross sections intersecting at 90°. ECAP was conducted via the Bc route (after each pass the workpiece was rotated around its longitudinal axis by 90° in a clockwise direction only) in the temperature range 120-200 ºC. The number of passes was selected according to the criterion of maintaining the workpiece integrity. The processing regimes are given in Table 2, the degree of their equivalent strain was calculated by the following formulas:

\[ \varepsilon = \ln\left(\frac{\theta r}{h}\right) \]

for HPT, where \( \theta \) – the angle of rotation, \( r, h \) – the disk radius and thickness, correspondingly;

\[ \varepsilon_N = \frac{2}{\sqrt{3}} \cot\left(\frac{\varphi}{2}\right) \]

for ECAP, where \( N \) – the number of passes, \( \varphi \) – the internal angle, C [3,7].

Table 2. Processing regimes.

|                | Number of ECAP passes / number of HPT rotations | Total true degree of strain, \( \varepsilon_\Sigma \) |
|----------------|-----------------------------------------------|--------------------------------------------------|
| HPT 20 ºC      | 3                                             | 5.24                                             |
| HPT 20 ºC + 150 ºC | 5                                           | 5.74                                             |
| ECAP 125 ºC    | 4                                             | 4.6                                              |
| ECAP 150 ºC    | 6                                             | 6.9                                              |
| ECAP 175 ºC    | 10                                            | 11.5                                             |
| ECAP 200 ºC    | 10                                            | 11.5                                             |

Qualitative and quantitative analysis of the microstructure of the initial coarse-grained alloy was performed using an optical microscope OLYMPUS. To create an optical contrast, the surface of mechanically polished objects was etched in Keller’s reagent (1 HF+1.5 HCl+2.5 HNO\(_3\)+95 H\(_2\)O ml).

The UFG structure was investigated using a transmission electron microscope (TEM) JEM-2100. The samples for TEM studies were prepared by twin jet electro-polishing on a Tenupol-5 in an electrolyte of the following composition: 400 ml of acetic acid, 300 ml of orthophosphoric acid, 200 ml of nitric acid and 100 ml of water. The average grain size, as well as the size of particles, was
determined by the random linear intercept method on dark-field and bright-field images of the microstructure.

The Vickers microhardness was measured on a Buehler Omnimet tester across a sample diameter with a step of 1 mm, load of 0.1 kg, holding time of 10 s. Tensile tests were conducted on the machine for small samples testing [8] with a gage length of 4.0 mm, thickness of 0.5 mm and width of 1.5 mm.

X-ray diffraction (XRD) analysis was performed on a “Rigaku” diffractometer with CuKα radiation. To determine the lattice parameter of the Al-Cu-Mg alloy, X-ray reflections with centroids in the 2θ angle range 34 ÷ 47º were taken, and the Nelson - Reilly extrapolation procedure was used.

Thermal stability was determined by measuring the microhardness after annealing for 30 minutes at temperatures of 100, 150, 175, 200, 250, 300 °C.

3. Results and discussion

The structure of the Al-Cu-Mg alloy after standard treatment, which involved heating at 530 °C for 1 hour, followed by quenching in water and aging at 180 °C for 14 hours, was characterized by equiaxed grains with an average size of 72 µm.

Transmission electron microscopy showed that the coarse-grained (CG) structure of the initial alloy completely transformed into an ultrafine-grained one (UFG) after SPD treatment. Particularly, after HPT at 20 °C grains with an average size of 140 nm were observed in the structure. After further annealing at 250 °C for 1 hour, the average grain size in the UFG samples increased up to 250 nm (Figure 1a).

After the combined HPT the grain structure was strongly refined to 66 nm, while after additional annealing at 250 °C the average grain size increased to 170 nm (Fig. 1b). In the structure of both samples after annealing at 250 °C numerous particles with an average size of 50 and 30 nm, respectively, were observed, which obviously retarded the grain growth at elevated temperatures.

In the samples subjected to 4 ECAP passes at 125 °C a band structure with an average band width of 250 nm (Figure 2a) as well as separate areas were observed, in which an ultrafine-grained structure was formed with an average size of 200 nm (Figure 2b).

After 6 ECAP passes at 150 °C, an ultrafine-grained structure with an average grain size of 400 nm was observed in the aluminum alloy (Figure 3). Thickness extinction contours were observed in grain boundaries, which testified to reduction of internal stresses and relaxation of crystalline lattice microdistortions (Figure 3a). Numerous precipitates with an average grain size of 75 nm were found too.

The UFG structures after ECAP treatment at 175 °C (Figure 4a) and 200 °C (Figure 4b) were similar, the grains with an average size of 500 nm and particles of 50-75 nm were observed.
Figure 2. The alloy microstructure after ECAP at 125 °C, 4 passes: (a) band structure, (b) ultrafine-grained structure

XRD analysis of samples subjected to the standard treatment showed that peaks corresponding to Al$_2$Cu, Al$_2$CuMg precipitates in the Al matrix alongside with the peaks belonging to it. After HPT the peaks of Al$_2$Cu, Al$_2$CuMg phases were not observed, which implied their complete dissolution during quenching and HPT.

Figure 3. (a, b). Typical microstructure after ECAP at 150 ºC, 6 passes

Figure 4. The Al-Cu-Mg microstructure after ECAP:
   a) 175 ºC, 10 passes; b) 200 ºC, 10 passes.

However, the peaks of these phases were observed in the samples after ECAP, indicating that the applied strain was insufficient for their dissolution.

It should be noted that the X-ray diffraction patterns of the samples after HPT are substantially different from those after standard treatment. Particularly, changes in the integral background radiation intensity, width and intensity of X-ray peaks were observed on the diffraction patterns.
The lattice parameter changes significantly depending on the used processing regimes (Table 3), which is obviously due to the precipitation and dissolution of strengthening particles. The minimum size of the coherent scattering domain was observed after HPT at 20 °C and after ECAP at the lowest temperature of 125 °C. The greatest root-mean-square values of the lattice micro-distortions were detected after HPT at 20 °C.

### Table 3. Results of X-ray analysis

| State | Treatment   | Lattice parameter, Å | Size of coherent scattering domains, nm | Root-mean-square micro-distortions of the crystal lattice, 10^-4 | Lattice volume |
|-------|-------------|----------------------|----------------------------------------|---------------------------------------------------------------|---------------|
| CG    | Quenching   | 4.04761              | 98.4                                   | 4.8                                                           | 66.313        |
| UFG   | HPT at 20 °C| 4.04313              | 39.9                                   | 32.7                                                          | 66.092        |
|       | Combined HPT| 4.04676              | 42.4                                   | 33.2                                                          | 66.271        |
|       | ECAP at 125 °C | 4.04443          | 45                                     | 21.5                                                          | 66.156        |
|       | ECAP at 150 °C | 4.04723          | 62.3                                   | 27.6                                                          | 66.294        |
|       | ECAP at 175 °C | 4.04909          | 47.4                                   | 15.3                                                          | 66.386        |
|       | ECAP at 200 °C | 4.05074          | 55.1                                   | 16.7                                                          | 66.466        |

The initial sample of the Al-Cu-Mg alloy had a microhardness of 980 MPa. The samples after the standard treatment displayed an increase in microhardness up to 1480 MPa. After SPD the microhardness enhanced considerably. The highest values were obtained in the samples after HPT at 23 °C (2320 MPa) and combined HPT (2790 MPa). As for ECAP, the maximum microhardness equal to 1912 MPa was observed at 125 °C.

In order to investigate thermal stability of the structure, the UFG samples were subjected to annealing for 30 minutes in the temperature range of 100-300 °C. The most thermally stable HPT samples were those fabricated by the combined regime. As for ECAP processed samples, the samples ECAPed at 125 °C and 150 °C exhibited increased microhardness values in the temperature range up to 175 °C.

Table 4 presents the results of tensile tests on the Al-Cu-Mg samples before and after treatment by SPD techniques. After the tests the highest tensile strength was observed in UFG samples produced by ECAP at 125 °C; they also retained a sufficiently good ductility – about 11 %, while the HPT samples were very brittle despite their high tensile strength up to 800 MPa.

It should be noted that the increased strength values observed in ECAP and HPT samples are due to the strong refinement of a grain structure and presence of fine particles of strengthening Al$_2$Cu, Al$_3$CuMg phases.

### Table 4. Mechanical properties of the Al-Cu-Mg alloy at room temperature.

| Treatment          | $\sigma_{0.2}$, MPa | $\sigma_{V}$, MPa | $\delta$, % |
|--------------------|---------------------|------------------|-------------|
| ECAP 125 4 passes  | 610±38              | 635±35           | 11±1        |
| ECAP 150 6 passes  | 588±27              | 624±25           | 9±0.4       |
| ECAP 175 10 passes | 436±31              | 460±21           | 14±1        |
| ECAP 200 10 passes | 352±18              | 420±30           | 13.5±0.5   |
| Standard treatment | 300±45              | 400±15           | 6±1         |
| Combined HPT       | 785±45              | -                | -           |
| HPT                | 720±38              | -                | -           |
4. Conclusions
In this paper the effect of severe plastic deformation on the structure and mechanical properties of ultrafine-grained Al-Cu-Mg samples was studied, which allowed drawing the following conclusions:

1. During combined treatment by HPT at two different temperatures, grain refinement to 66 nm in size and microhardness of 2790 MPa have been achieved in the Al-Cu-Mg alloy.

2. 4 ECAP passes at 125 ºC are insufficient to produce a homogeneous ultrafine-grained structure, while 6 ECAP passes at 150 ºC result in fabrication of ductile UFG samples with high ultimate tensile strength and microhardness.

3. The analysis of the temperature dependence of microhardness proves that the microstructure of UFG samples is thermally stable up to a temperature of 175 ºC, above which a considerable microhardness decrease was observed due to beginning of grain growth and coarsening of precipitates.

Thus, enhanced strength characteristics of the alloy of the Al-Cu-Mg system were achieved via SPD technique application. Primary ductility was retained due to application of an ECAP technique. Further optimization of SPD regimes can improve the mechanical properties.

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