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The Microstructure and Fatigue of Ultrafine-Grained Al–Cu–Mg Alloy

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In this work the influence of equal-channel angular pressing on strength and fatigue of an aluminum alloy has been studied. Transmission electron microscopy was applied to determine an average grain size, shape, and size of precipitates. The ultimate tensile strength and fatigue endurance limit of ultrafine-grained and coarse-grained samples were evaluated at 20 $^{\circ}$ C and 175 $^{\circ}$ C.

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1. Introduction

Recently formation of the UFG structure in various metals and alloys to achieve enhanced mechanical properties has been becoming one of the most actively developed research directions of materials science [1-4].

In particular, in order to produce bulk UFG billets for structural application, a number of severe plastic deformation (SPD) techniques was developed. A distinctive feature of the techniques is application of large strains in the conditions of high pressures at relatively low temperatures [1].

At the same time, grain refinement in Al alloys by SPD techniques has a number of important peculiarities. In the temperature range 20–150 °C quenched samples of Al alloys have lower deformability in comparison with pure metals, and in the process of equal-channel angular pressing (ECAP) the samples and billets can fail at the initial stages of deformation. Besides, ageing processes are observed in heat-treatable Al alloys in the temperature range 150–200 °C, which lead to second-phase particles precipitation.

All of this creates additional possibilities for properties enhancement in metal alloys, but it requires optimizing technological regimes of producing bulk UFG billets to be used not only for basic research, but also having innovative potential for structural application.

One of the promising applications of UFG Al alloy is production of gas-turbine engine fan blades. For using Al alloys as materials for fan blades, it is necessary to increase their static and cyclic strength and retain thermal stability to 175 °C, which will open new opportunities for improvement of blade design.

The aim of this work is to study the structure and mechanical properties in ultrafine-grained samples produced by ECAP at different temperatures on the example of aluminum alloy, which is related to a class of thermally stable materials [5].

2. Material and experiment

Heat-treatable alloy 2618 of the Al–Cu–Mg–Si system was chosen as material for studies. The chemical composition in wt% is listed in the Table.

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Cu	Mg	Fe	Ni	Si	Ti	Mn	Zr	Impurities
1.9 - 2.7	1.2 - 1.8	0.8 - 1.4	0.8 - 1.4	0.35	0.02 - 0.1	0.2	0.3	0.1

Chemical composition of 2618, wt%.

Prior to ECAP processing, AK4-1 billets with a diameter of 40 mm and length over 160 mm were subjected to heating at 530 °C for 1 h with subsequent water quenching. ECAP was carried out at different temperatures in the range 120–430 °C on a die-set with an angle of channels intersection 120 °C via the route Bc. The number of passes in each case was determined proceeding from the criterion of billet integrity maintenance.

The structural studies were performed on a transmission electron microscope JEM-2100 equipped with a device for energy dispersive spectroscopy (EDS). Thin foils were prepared on a "Tenupol-5" set by jet electrolytic polishing.

Microhardness measurements via the Vickers technique were performed on a Micromet 5101 set along the diameter of HPT samples under a load of 200 g with dwell time of 15 s.

In order to determine the mechanical properties, cylindrical samples with a diameter of 5 mm and a gage length of 25 mm were used. The tensile tests were carried out on an Instron testing machine at room temperature and a strain rate of 10^{-3} s⁻¹.

High-cycle fatigue (HCF) tests were performed on testing machines — high-frequency pulsators Amsler 100 HFP 5100 and Amsler 300 HFP 5100 (Zwick/Roell) under axial loading of a sample at temperatures T = 20 °C

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and 175 °C according to [6]. The fatigue tests were performed with 30 Hz frequency at a number of cycles $N = 10^7$. The cycle asymmetry coefficient $R_{\sigma} = 0.1$.

3. Results and discussions

The investigations showed that in addition to general tendency to microhardness reduction, there are several distinctive features on the diagram of ECAP temperature dependence of microhardness (Fig. 1)



Fig. 1. Microhardness dependence on ECAP temperature.

First, during ECAP processing at 150 °C maximum values of microhardness are observed. But the samples failed after 3 passes.

Second, a peak near $200 \,^{\circ}$ C is conditioned by the fact that this temperature is recommended for ageing processes in coarse-grained samples [5]. The possible number of ECAP passes increases to 6.

A third peculiarity is slight increase of microhardness after ECAP at 430 °C, where the temperature is slightly lower that the quenching temperature for this alloy. Thus as a result of ECAP carried out at 430 °C, additional coarsening and dissolution of precipitates could take place, which leads to slight solid solution hardening.

Below are the results of structure and properties studies after ECAP at 160 °C, because an equiaxed UFG structure with the smallest grain size was expected in them leading to combination of enhanced ultimate tensile strength and fatigue endurance limit.

After 6 ECAP passes at 160 °C UFG samples were deformed by extrusion at the same temperature to produce elongated billets with a diameter 23 mm and length of 430 mm (Fig. 2).



Fig. 2. View of UFG samples after ECAP at 160 $^{\circ}\mathrm{C}$ and additional extrusion.



Fig. 3. Typical microstructure of the alloy subjected to ECAP at 160 °C and additional extrusion: (a) bright-field image, (b) dark-field image.



Fig. 4. TEM images of the UFG samples with dislocations in grains: (a) bright-field image, (b) dark-field image.

The average grain size in UFG samples was equal to 300 nm (Fig. 3). A large number of dispersed precipitates was observed in the structure, which are effective obstacles for dislocations motion (Fig. 4).

In particular, in the structure of UFG samples two types of particles were revealed. The first type was of a globular shape, and the average grain size was about 20 nm (Fig. 5a). According to the EDS analysis these particles contained mainly aluminum and copper (Fig. 6), i.e. they referred to the particles of the Al_2Cu phase, which are strengthening ones for this system of alloys.



Fig. 5. Dark-field images of particles: (a) Al_2Cu , (b) Al_2MgCu .

The second type of particles with the average size of about 70 nm (Fig. 5b) had a slightly elongated shape and contained mainly aluminum, magnesium and copper. They can be referred to Al_2MgCu phase particles according to the EDS analysis and morphological characteristics, which are also found in the investigated alloy (Fig. 7) [5].

As a result of testing at room temperature, the UFG 2618 alloy produced by ECAP at 160 °C and subsequent extrusion demonstrated the enhanced ultimate tensile strength of 460 MPa with elongation to failure up



Fig. 6. EDS analysis for particles displayed in Fig. 4a.



Fig. 7. EDS analysis for particles presented in Fig. 4b.

to 7% (Fig. 8). The 2618 alloy in the UFG state exhibited the ultimate tensile strength over 20% higher than that in a coarse-grained sample subjected to standard treatment (370 MPa) (Fig. 8), but the ductility reduced more than twice.

The enhanced strength of UFG samples at room temperature is a result of strong grain refinement and dispersion hardening. Decrease of elongation to failure can be explained by internal stresses around particles and near grain boundaries, which prevent nucleation, propagation and motion of dislocations. A short strain hardening stage is observed in the UFG samples. As it is known, at the strain hardening stage the dislocation density increases. In the UFG material the distance between grain boundaries is far smaller, therefore dislocations achieve the opposite grain boundary faster and annihilate. This can explain a short-term stage of strain hardening in the UFG samples and, thus, a small uniform deformation.

The tensile tests of UFG 2618 samples carried out at elevated temperatures showed that the ultimate tensile strength reduces slightly to 430 MPa. This confirms the thermal stability of mechanical properties of AK4-1 alloy in the UFG state to a temperature of 175 °C (Fig. 9).



Fig. 8. The variation of engineering stress with engineering strain for UFG samples at room temperature.



Fig. 9. The variation of engineering stress with engineering strain for UFG samples at 175 $^{\circ}\mathrm{C}.$

The experimental data obtained during HCF tests were used for determining numerical values in equation which describes the dependence of the number of cycles to failure on stress:

$$N = C\sigma^{-n}.$$
 (1)

The experimental data on HCF tests at $20 \,^{\circ}\text{C}$ and $175 \,^{\circ}\text{C}$ and results of their modeling are presented in the diagrams (Figs. 10, 11).

As it follows from the above-written data (Fig. 10), the alloy with the UFG structure at T = 20 °C has a higher fatigue endurance limit (by $\approx 15\%$) on the basis of $N = 10^7$ cycles as compared to the coarse-grained one after standard treatment.

Alongside with that when the testing temperature increases to 175 °C on the basis of $N = 10^7$ cycles in the UFG alloy, the fatigue endurance limit reduces from $\sigma_{\rm max} = 257$ MPa to $\sigma_{\rm max} = 204$ MPa (Fig. 11).



Fig. 10. Dependence of the the maximum stress on number of cycles at = 20 °C. Curve 1 – coarse-grained structure; curve 2 – UFG-structure.



Fig. 11. Dependence of the the maximum stress on number of cycles at = 175 °C.

It is known that grain refinement can increase the fatigue properties in some metals and alloys with a UFG structure [7–9].

At the same time, in some publications it was mentioned that the fatigue limit in Al alloys subjected to ECAP is close to the values observed in coarse-grained materials [10, 11]. In other works [12] much higher fatigue properties were achieved experimentally in UFG samples.

It is evident that the fatigue properties of Al alloys are sensitive not only to grain refinement, but also to other structure peculiarities, in particular to chemical composition that determines contributions of solid solution and precipitation hardenings. Alloying element atoms and dispersed particles can prevent grain boundary migration, which is one of structure relaxation mechanisms in the process of fatigue tests manifesting itself as grain growth [8] and twinning [9] leading to material softening.

Thus, as compared to coarse-grained samples subjected to standard treatment, enhanced fatigue properties at 20 °C in ECAPed samples are conditioned by the combination of the UFG structure and precipitation hardening due to particles with a grain size of 20 nm (Fig. 5a). In the process of fatigue testing at an elevated temperature of 175 °C these particles coarsen, as a result the fatigue endurance limit decreases.

4. Conclusions

- 1. The ECAP processing at $160 \,^{\circ}\text{C}$ allowed forming the UFG structure with an average grain size of 300 nm with particles of the Al₂Cu phase, which contributed to enhanced ultimate tensile strength of 460 MPa with a ductility of 7%. Ultimate tensile strength decreased slightly to 430 MPa after tensile tests at an elevated temperature of $175 \,^{\circ}\text{C}$.
- 2. Formation of a UFG structure in the Al–Cu–Mg alloy resulted in the increase of fatigue endurance limit at 20 °C by 15% to $\sigma_{\rm max} = 257$ MPa, as compared to the fatigue endurance limit $\sigma_{\rm max} = 222$ MPa for coarse-grained samples subjected to standard treatment. With the testing temperature increase to 175 °C, the fatigue endurance limit in the UFG state reduced to $\sigma_{\rm max} = 204$ MPa.

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