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Low Temperature Superplasticity of High-Strength Ultrafine-Grained Al 7050 alloy

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Abstract. Ultrafine-grained (UFG) Al 7xxx series demonstrates very high strength at room temperature with UTS=845 MPa and hardness Hv=255, which considerably exceeds the value after conventional heat treatment T73, i.e. Hv=165. It is also revealed that with increasing temperature the alloy exhibits rather high ductility with elongation to failure $\delta=170\%$ at $120^\circ C$ and strain rate of $5\times10^{-4}\ s^{-1}$ and typical superplastic behavior with $\delta=480\%$ and $\delta=690\%$ at temperatures $170^\circ C$ and $200^\circ C$ correspondingly. Such values of superplasticity (SP) temperature are considerably lower than that typical of this alloy with micron-sized grains ($T_{SP}=450-500^\circ C$). Such reduction in superplasticity temperature made it possible to preserve ultrafine grains in the alloy structure and provide its high-strength state after SP straining.

1. Introduction
Over the last 20 years, a breakthrough has been achieved in producing Al-based alloys using severe plastic deformation (SPD) techniques [1]. Along with high mechanical strength and even «superstrength», Al alloys with such a microstructure reveal a range of remarkable performance properties such as enhanced fatigue strength, fracture toughness, electric conductivity, etc. [1-3].

Al-based alloys with the UFG structure can also demonstrate superplastic behavior at temperatures much lower and at strain rates higher (more than $10^2\ s^{-1}$) than those appropriate for the coarse-grained materials [2, 4-5]. The general principles of SP, as demonstrated by the UFG materials, have been summarized in a number of reviews and books [1, 2, 4, 5]. The reports note that low-temperature and high-rate SP is very attractive for commercial superplastic forming, as it enables reduced power consumption, increases the service life of a shape-forming tool and reduces the timeframe for production. Besides, lower SP temperature induces a reduction in the microstructural degradation in the UFG materials; as a result, the produced components retain their high level of mechanical and performance characteristics. Recent studies on the model alloys of the Al–Zn and Mg–Li systems showed that the formation of the UFG structure with grain boundaries containing layers formed by segregations of alloying elements may ensure the signs of SP in these alloys, even at room temperature (RT) [6-7]. Good ductility values are observed in the commercial UFG alloy 1570 of the Al–Mg–Sc system (up to 300% at $200^\circ C$), [8] in which the ultimate tensile strength at RT is 950MPa [9].
High-strength aluminum alloys of the system Al-Zn-Mg-Cu (7xxx series) are widely known and at present, there is an avalanche of experimental data on mechanical and corrosion behavior. Despite the fact that a large amount of literature deals with the aging of coarse-grained 7xxx series Al alloys, there is a lack of systematic research on the UFG states and their influence on mechanical and functional properties, including the manifestation of SP at low temperatures and high strain rates.

2. Experimental procedures

In this work, commercial aluminum alloy 7050 of the 7xxx series was used as the material under investigation. The chemical composition of the alloy is Al-6.4Zn-1.7Mg-2.3Cu-0.12Zr-0.10Fe-0.05Si-0.02Ti (wt. %).

After quenching, the disk-shaped samples 20 mm in diameter and 1.4 mm in thickness were subjected to high pressure torsion under a pressure of 6 GPa at RT for maximum refining of the grain structure. As was experimentally stated, the hardness of the UFG alloy discs with the chosen sizes stabilized on the spot located at a half radius when n ~10.

Thus, all the studies on the structure and mechanical properties of the UFG alloy were performed with precision on the half radius of the samples.

A number of initial samples were subjected to conventional treatment Т73 that is recommended for hardening of the 7xxx series alloys in [10-12]. The treatment included quenching and two-stage aging at a temperature of 110°C during 6 h and then 160°C during 16 h.

The thermal stability of the UFG alloy was also investigated, the alloy blanks were annealed at 120, 170 and 200 °C for 10 hours, the hardness was measured after 1, 2, 3, 5, 8 and 10 hours of exposure.

Hardness was determined at room temperature according to the Vickers method (Hv) using a Buehler Omnimet Micromet-5101; 10 measurements were taken in each area, at a load of 0.5N and a 15 s holding time under load.

The tensile tests were carried out on the test machine Instron. The tests were computer controlled, the temperature, strain and stress were registered. The mechanical tests of the alloy samples with a gauge part of 2.0×1.0×0.8 mm were conducted at RT, 120, 170 and 200 °C in the range of strain rate of $10^{-3}$...$10^{-1}$ s$^{-1}$ to measure the total elongation to failure.

The microstructures were examined with a JEOL JEM 2100 transmission electron microscopy (TEM), operating with an accelerating voltage of 200 kV. The samples intended for foils were first cut using electrical discharge machining, then mechanically thinned to a thickness of 0.15 mm, and finally electrically polished in the solution of 80 % methanol and20% nitric acid on the TenuPol-5 unit at a polishing temperature from –25 °C.

3. Results and discussions

3.1. Grain refinement and thermal stability

Figure 1(a,b) displays TEM images of the homogeneous microstructure after HPT at RT obtained via electron microscopy. The initial coarse-crystalline structure transforms into the UFG structure with an average grain size of 140±15 nm. The circular distribution of spots on the diffraction pattern indicates high-angle misorientations of the grain boundaries formed during HPT (figure 1a). Recent studies into the fine structure showed that the formation of UFG microstructure during HPT at RT in the 7xxx series alloys is accompanied by the formation of nano-sized particles of the μ(Zn$_2$Mg) phase as well as the segregation of Zn and Mg atoms mostly at grain boundaries. As was shown in [11], the presence of grain boundary segregations in the UFG Al alloys may favorably influence the strength and high-strain-rate superplasticity.

After HPT the hardness of the UFG alloy 7050 constituted Hv 255 and this exceeds the alloy hardness after conventional heat treatment Т73 (Hv 165) by ~ 55 %.
No noticeable changes were registered in the UFG structure after annealing for 10 hours at 120°C. The average grain size was 160±10 nm. Long-term annealing at 170 and 200 °C led to a more significant increase of ultrafine grains (figure 1c,d) and their average grain size increased to 230±20 nm and 460±15 nm correspondingly. In addition to grain growth, annealing resulted in coarsening of the particles of μ(Zn2Mg) phase located at GB (figure 1c). Also, annealing led to coarsening of particles in ultrafine grains.

Evolution of the UFG structure well correlates with the nature of the change in the alloy hardness (figure 2). As a result of annealing at 120°C, when microstructural changes were minimal, the alloy hardness decreased to 230 Hv. As the heat treatment progresses at elevated temperatures, the hardness of UFG alloy monotonically decreases and reaches the level of hardness after treatment T73 after 8 hours of annealing at 170 °C and at 200 °C during 2 hours.

Figure 2 demonstrates thermal stability of ultrafine-grained Al alloy after annealing at 120, 170 and 200 °C during 10 hours. It was established that the UFG aluminum alloy retains its thermal stability up to temperatures of 170 °C for 10 hours. At a temperature of 200 °C, noticeable softening begins after 4 hours of annealing; its hardness reduces to 150 Hv.
3.2. Mechanical properties

The formation of UFG state with an average grain size of 150±6 nm and a shape form of 1.2 resulted in the growth of ultimate tensile strength (UTS) to 845±5 MPa, of yield strength (YS) to 818±9 MPa at RT. After conventional treatment T73 the following values were registered in the 7050 alloy: UTS = 580±5 MPa, YS = 516±10 MPa.

To reveal the ability of high-strength material to SP in the lower temperature zone, the tensile tests were performed with strain rates of $10^{-1}$, $10^{-2}$, $5\times10^{-3}$, $10^{-3}$, $5\times10^{-4}$, and $10^{-4}$ s$^{-1}$ at 120, 170 and 200 °C. The temperatures of the thermal stability from 120 to 200 °C were chosen according to the obtained results of microhardness change after annealing (figure 2).

Figure 3 demonstrates the typical stress-strain curves taken after mechanical testing (at 120, 170 and 200 °C) of the UFG alloy processed by HPT at RT. As the strain rate reduces, the ductility of the alloy increases. The maximum values of ductility 170 % are achieved at a strain rate of $5\times10^{-4}$ s$^{-1}$. At a testing temperature of 120 °C the ultimate tensile strength retains high in the whole range of rates. The hardness after testing at 120 °C is 230 Hv for strain rate $10^{-2}$ s$^{-1}$ and 200 Hv for rate $5\times10^{-4}$ s$^{-1}$.

The growth of the testing temperature to 170 °C results in the achievement of unusually high strain rate ductility - 480 %, the strength retains at a rather high level, as shown in figure 3. When the strain rate reduces, the ductility decreases, which is evidently connected with the recovery processes taking place during the long-term static testing. However, at 170 °C in the whole rate range the high ductility values are achieved. The hardness after testing at 170 °C is 195 Hv.

The further increase of the temperature of tensile test to 200 °C leads to the growth of ductility to 690 %, the strength values reduce considerably, as shown in figure 3. The hardness after testing at 200 °C decreases to the hardness values after heat treatment T73 and is 165 Hv.
Figure 3. Engineering tensile curves of UFG Al 7050 alloy. Tensile testing at: 120, 170 and 200 °C.

Figures 4 demonstrates the curves of flow stress and elongation vs strain rate that are plotted against the results of static testing executed at 120, 170 and 200 °C. When the deformation temperature increases, the stress reduces, which is typical of superplastic materials [2,4,13]. The strain rate decrease during testing at 120 °C in the whole range of rates results in the reduction of the flow stress and the increase of the ductility, as shown on figure 3.

The testing temperatures of 170-200 °C are marked with the reduction in the flow stress value in the rate range from $10^{-2}$ to $5\times10^{-4}$ s$^{-1}$.

Figure 4. Dependence of elongation and flow stress on strain rate based on the results of static tests at 120, 170, and 200 °C of UFG Al 7050 alloy.

The maximum elongations are achieved at 170 °C and a strain rate of $5\times10^{-4}$ s$^{-1}$ and at 200 °C and strain rate of $10^{-3}$ s$^{-1}$. It should be noted that at 170 °C there are no clear maximums of ductility. However, currently, in a number of production-oriented industries the task has been set to refocus manifestations of superplasticity towards the region of low temperatures and high strain rates [14,15,16]. Therefore, according to the results of these studies, it is possible to determine the strain rate range where the material exhibits high values of plasticity (elongation near 300 %) and retains high values of strength (Hv near 190-200): strain rate from $10^{-3}$ to $10^{-4}$ s$^{-1}$ at a temperature of 170 °C.
4. Summary and conclusions

1. The formation of UFG state with an average grain size of 140±15 nm resulted in the growth of microhardness in the 7050 alloy to Hv255±12, in UTS to 845±5 MPa, in YS to 818±9 MPa at RT. After conventional treatment T73 the values are Hv=160, UTS=580±5 MPa, YS=516±10 MPa.

2. The ultrafine-grained aluminum alloy has been established to retain its thermal stability up to temperatures of 170 °C for 10 hours. At a temperature of 200 °C, noticeable softening begins after 4 hours of annealing, its hardness reduces till 150HV.

3. Tensile tests are performed at 120, 170 and 200 °C in the strain rate range from 10^-2 to 10^-4 s^-1 to determine the high values of ductility and strain rate sensitivity. The maximum elongations of 170 % are achieved at 120 °C and a strain rate of 5×10^-2 s^-1, 480 % - at 170 °C and a strain rate of 5×10^-4 s^-1, at 200 °C and a strain rate of 10^-3 s^-1 the elongations achieved 690 %. The ultrafine-grained alloy retains an increased level of hardness after mechanical tests in the temperature range 120-200 °C.

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