INFLUENCE OF SEVERE PLASTIC DEFORMATION ON MICROSTRUCTURE, STRENGTH AND ELECTRICAL CONDUCTIVITY OF AGED Al–0.4Zr(wt.%) ALLOY

T.S. Orlova¹,², A.M. Mavlyutov², T.A. Latynina², E.V. Ubyivovk³, M.Yu. Murashkin³,⁴, R. Schneider⁵, D. Gerthsen⁵ and R.Z. Valiev³,⁴

¹Ioffe Institute, Russian Academy of Sciences, ul. Politekhnicheskaya 26, St. Petersburg, 194021 Russia
²Saint Petersburg National Research University of Information Technologies, Mechanics and Optics, Kronverksky Pr. 49, St. Petersburg 197101, Russia
³Saint Petersburg State University, Universitetskiy Pr. 28, St. Petersburg 198504, Russia
⁴Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, K. Marx str. 12, Ufa 450000, Russia
⁵Laboratory for Electron Microscopy, Karlsruhe Institute for Technology, Karlsruhe D-76128, Germany

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Abstract. Microstructure evolution of an Al–0.4Zr(wt.%) alloy after isothermal aging (AG) and subsequent high pressure torsion (HPT) and its impact on strength and electrical conductivity has been investigated. Microstructure was characterized by X-ray diffraction, electron backscatter diffraction, transmission electron microscopy (TEM) and electron energy-dispersive X-ray spectroscopy in TEM. The initial Al–0.4Zr(wt.%) alloy obtained by combined casting and rolling presents solid solution of Zr in Al matrix. Aging at 375 °C for 60 h leads to formation of uniformly distributed metastable Al₃Zr precipitates with the average diameter of 13 nm, resulting thereby in a decrease of strength σUTS from 128 to 95 MPa and in increase of conductivity from 50.7 to 58.8% IACS at ambient temperature. The subsequent HPT processing leads to grain refinement and partial dissolution of the Al₃Zr precipitates that is accompanied by enrichment of solid solution by Zr atoms and by coarsening of the remaining Al₃Zr precipitates. The combination of AG and HPT provides the strength and the conductivity at ambient temperature which do not decrease under annealing up to 230 °C. Moreover, additional strengthening accompanied by an increase in conductivity was found for AG–HPT samples after annealing at Tₐ=230 °C for 1 h, that provides the best combination of the strength σUTS=142 MPa and the conductivity of 58.3% IACS. Contribution of different possible mechanisms into strength and charge scattering are analyzed on the basis of specific microstructural features. The analysis indicates a suppression of strengthening by the Orowan mechanism in AG and AG–HPT samples. In all the studied states, i.e. initial, after AG, and subsequent HPT, grain boundary strengthening is found to be the main strengthening mechanism.

1. INTRODUCTION

Aluminum alloys are widely used for production of wires for overhead power transmission lines due to good combination of light weight, reasonable electrical conductivity, and high corrosion resistance. Requirements to materials used for electrotechnical applications, and especially for power lines, are steadily increasing: these materials should combine high electrical conductivity and sufficient strength at the service temperature up to 150–230 °C [1]. Recently a good combination of strength (ultimate tensile strength ~360 MPa) and electrical conductivity (~56% IACS) was achieved for Al–Mg–Si alloys through a complex thermomechanical treatment involving severe plastic deformation (SPD) sequentially at room temperature and then at el-
evated temperatures. Such treatment leads to grain refinement and the purification of the Al matrix due to the formation of secondary phase precipitates, that, on the one hand, provides an increase in grain-boundary strengthening and involves precipitate strengthening by the Orowan mechanism, and, on the other hand, increases the electrical conductivity [2–4]. However, the Al–Mg–Si alloys have serious disadvantage: their long-time operating temperature does not exceed 90 °C [5].

Recently low-alloyed Al alloys with Zr additives are considered as promising materials to meet the requirement of high heat resistance and good conductivity, which can be achieved by formation of dispersed precipitates of the metastable Al₃Zr phase, which is accompanied by a purification of the Al matrix [6–9]. Usually, Al–Zr(0.1–0.4 wt.%) alloys with the total Zr content in solid solution are obtained by different technologies and then aged at 300–450 °C for formation of dispersed nanoscale Al₃Zr precipitates. The uniformly distributed Al₃Zr nanoparticles are stable and hinder the grain growth at elevated temperatures providing good thermal stability of the properties up to 150–230 °C. At the same time, the Al–Zr alloys demonstrate much lower strength compared to the Al–Mg–Si alloys [2–4,6–9]. Therefore, it is very important to enhance strength of the Al–Zr alloys while keeping high level of thermal stability and good electrical conductivity. One of the approaches to enhance their strength could be grain refinement by severe plastic deformation which is effective for the Al–Mg–Si alloys.

This work presents for the first time the results on the influence of high pressure torsion (HPT) on microstructure and resulting functional properties (strength, electrical conductivity, and heat resistance) for preliminary aged Al–0.4Zr(wt.%) alloy. Contributions of different possible strengthening mechanisms to strength and charge scattering mechanisms to resistivity are analyzed on the basis of specific microstructural features for the initial state, the states after aging, and after aging with subsequent HPT processing.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

An Al–0.4Zr(wt.%) alloy with the chemical composition 99.25Al, 0.393Zr, 0.023Si, 0.242Fe, 0.018Zn, 0.026V, 0.05–balance (wt.%) was obtained in the form of rod by the method of combined casting (C–casting) and rolling (R–rolling) [10,11]. After the C–R processing Zr atoms are mainly dissolved in the Al matrix [12]. Blanks in the form of cylinders 9.5 mm in diameter and 8 mm in height were cut from the initial alloy, pressed under a pressure of 6 GPa to a height of 1.5 mm, and subjected to subsequent isothermal annealing at 375 °C for 60 h. Part of the blanks after the long-term annealing were subjected to severe plastic deformation by high pressure torsion (HPT) under a hydrostatic pressure of 6 GPa to 10 revolutions at room temperature (RT) [13,14]. As a result of such treatment, samples in a shape of discs with a diameter of 20 mm and a thickness of 1.5 mm were obtained. The true strain at the distance of 5 mm from a disc center was ε=6.6 [14].

A comparative study of the relationship between the microstructure and strength and electrical conductivity of the Al–0.4Zr (wt.%) alloy was carried out for three states: initial (C–R samples), after aging by long-term isothermal annealing (AG samples), and after long-term isothermal annealing followed by HPT processing (AG–HPT samples).

To determine the thermal stability of the properties of the AG–HPT samples, an additional short-term annealing was carried out for 1 h at various temperatures in the range from 90 to 400 °C.

Microstructure of the samples was studied by X-ray diffraction (XRD), electron backscatter diffraction (EBSD), transmission electron microscopy (TEM) and electron energy-dispersive X-ray spectroscopy (EDX). The XRD measurements were taken on the Bruker D8 DISCOVER diffractometer in a standard regime of symmetric ϕ-2θ scanning. Lattice parameter a, average size of coherent-scattering regions (Dav), elastic microdistortion level (Δε<2>) were determined from the diffraction patterns. The dislocation density Ldis was estimated as [15]:

\[ L_{\text{dis}} = 2\sqrt{3} \left(\frac{\varepsilon^2}{2}\right) / \langle D_{\text{av}} \rangle b, \]

where b is the Burgers vector.

The EBSD studies were performed using the scanning electron microscope Zeiss Merlin on the area of ~1200 μm² with a scan step of 0.2 μm to determine the grain size distribution, the average grain size (dav), the distribution of grain boundaries between the adjacent grains on their misorientation angle (θ), and the fraction (f15) of high-angle grain boundaries (HAGBs) with a misorientation angle θ≥15°. The details of EBSD analysis are presented in [16].

TEM investigations were carried out using a JEOL JEM 2100 microscope and FEI OSIRIS microscope equipped with an EDX SuperStem detector for local chemical measurements. Thin foils for TEM observation were prepared by mechanical polishing fol-
followed by double-jet electropolishing in a regime similar that used in [16].

Uniaxial tensile tests were performed on a Shimadzu AG-XD Plus machine with a constant strain rate of 5·10⁻⁴ s⁻¹. For mechanical tests the samples were cut in the shape of blades with a gauge width of 2 mm and a gauge length of 6 mm. The cutting scheme and sample configuration are presented in [17]. Sample straining was recorded using a TRView 55S video extensometer. At least 3 samples were tested for each state. Vickers hardness was measured using Shimadzu HMV-G microindentation tester with application of a load of 1 N for 15 s. Each sample was measured not less than 15 times along its length.

Electrical resistivity was measured by a standard four probe technique at 77K (ρₜₐₚ), as well as at a number of intermediate temperatures in a range of 100–300K, a measuring error being <2%. The temperature of the sample was controlled by a silicon diode with an accuracy of ±0.03K. A linear approximation of the obtained data was used to determine electrical resistivity at 293K (ρ₂₉₃). More details of electrical resistivity measurements by the four probe technique are provided in [18].

3. RESULTS AND DISCUSSION

3.1. Microstructure characterization

The results of the XRD analysis are presented in Table 1. As seen, the lattice parameter a slightly decreases after aging and subsequent HPT processing. Dislocation density Lₐₕ substantially decreases after aging, then restores up to 4.3·10⁻¹² m² after subsequent HPT processing. The XRD patterns (not shown in the paper) demonstrate the presence of the Al₃Zr (D₀₂₃) phase in about the same small amount in all the studied states: C–R, AG and AG–HPT, any traces of the metastable Al₃Zr (L₁₂) phase being absent.

The TEM microstructure images of the Al–0.4Zr alloy in AG and AG–HPT states are shown in Fig. 1 and Fig. 2, respectively. It can be seen that uniformly distributed secondary phase precipitates are formed upon aging of the initial (C–R) alloy. Selected area electron diffraction (the insert in Fig. 1) and local EDX analysis in TEM (Fig. 1c) confirm that they belong to metastable Al₃Zr phase (L₁₂). The average size of these nanoparticles was determined on the basis of more than 200 particles (Fig. 1b) to be equal to dₚ = 13±2 nm. Their concentration was determined with allowance for the thickness of the TEM foils (300–400 nm) to be equal to nₚ = (3.6±0.6)·10¹⁶ m⁻³. The latter corresponds the average distance 65±5 nm between adjacent particles. Along with these small nanoparticles there are a number of larger particles (Fig. 1c) which are most likely formed in the process of casting and belong to the D₀₂₃ phase according to XRD data.

Estimation of the total volume fraction of nanoparticles of the metastable Al₃Zr phase, which was made from TEM images, gives Vₚ = 0.42±0.06 vol.% that corresponds to a concentration Cₐₕ = 0.05±0.04 wt.% of remaining Zr atoms in solid solution in these samples (Table 2). After HPT processing, the average size of metastable Al₃Zr particles substantially increased up to dₚ = 56±23 nm and their concentration dramatically decreased to (6.50±0.80)·10¹⁸ m⁻³ (Fig. 2). Estimation from TEM images gives Vₚ = 0.06±0.01 vol.% and Cₐₕ = 0.34±0.01 wt.% Zr for AG–HPT samples (Table 2). It means that HPT processing leads to a dissolution of most Zr, Al nanoparticles (dₚ~13 nm), that is accompanied by coarsening of the remaining ones. It should be noted that since the thickness of the TEM films is not exactly defined and can vary in the limit 300 – 400 nm, the obtained estimates of Vₚ and, hence, of Cₐₕ from TEM images are very approximate. In addition, initially present large D₀₂₃ particles are fragmented into smaller ones (Fig. 2). Further, we will consider only numerous precipitates of the metastable L₁₂ phase, omitting a much smaller number of D₀₂₃ phase particles.

Fig. 3 demonstrates the EBSD maps of the studied C–R, AG, and AG–HPT samples. The distribu-

<table>
<thead>
<tr>
<th>State</th>
<th>a, Å</th>
<th>&lt;ɛ²&gt;₁/₂, %</th>
<th>DₓRD, nm</th>
<th>Lₐₕ, m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–R</td>
<td>4.05104±0.00014</td>
<td>0.0190±0.0020</td>
<td>245±50</td>
<td>9.4·10⁻¹²</td>
</tr>
<tr>
<td>AG</td>
<td>4.05028±0.00005</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>AG–HPT</td>
<td>4.05024±0.00003</td>
<td>0.0100±0.0024</td>
<td>283±13</td>
<td>4.3·10⁻¹²</td>
</tr>
</tbody>
</table>
Influence of severe plastic deformation on microstructure, strength and electrical...
Table 2. Structural parameters of the Al–0.4Zr(wt.%) alloy. \( d_{av} \) – average grain size, \( f_{\geq 15} \) – percentage of high-angle grain boundaries, \( d_{pt} \) – average size of second phase particles, \( n_{pt} \) – concentration of second phase particles, \( V_{pt} \) – volume fraction of second phase particles, \( C_{sol}^{el} \) – concentration of solute Zr atoms.

<table>
<thead>
<tr>
<th>State</th>
<th>EBSD data</th>
<th>TEM data</th>
<th>estimated from ( p_{293} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( d_{av}, \mathrm{nm} )</td>
<td>( f_{\geq 15}, % )</td>
<td>( d_{pt}, \mathrm{nm} )</td>
</tr>
<tr>
<td>C–R</td>
<td>1200±550</td>
<td>25</td>
<td>–</td>
</tr>
<tr>
<td>AG</td>
<td>2100±1150</td>
<td>47</td>
<td>13±2</td>
</tr>
<tr>
<td>AG–HPT</td>
<td>820±220</td>
<td>78</td>
<td>56±23</td>
</tr>
</tbody>
</table>

Aging leads to formation of microstructure with equiaxed grains, the average grain size increases to \( d_{av} \approx 2.1 \mu \mathrm{m} \). It should be noted that the grain size distribution in this state is not uniform: there are quite large grains with sizes of 3–6 \( \mu \mathrm{m} \) and a large number of small grains with a size below 1 \( \mu \mathrm{m} \). The distribution of GBs on misorientation angle becomes bimodal (Fig. 4e) with fraction of HAGB \( f_{\geq 15} = 47\% \) (Table 2). The subsequent HPT processing results in grain refinement to \( d_{av} = 820 \pm 220 \) nm, the fraction \( f_{\geq 15} \) increases up to 78\%. It should be noted that the average grain size remains within 0.8 – 2.1 \( \mu \mathrm{m} \) in all the three studied states of the Al–0.4Zr alloy.

3.2. Strength, its thermal stability and electrical conductivity

The results of mechanical tensile tests and microhardness measurements for the studied C–R, AG, and AG–HPT states are presented in Fig. 5 as well as in Table 3. As is seen, the characters of change of conventional yield point \( \sigma_{y}^{exp} \), ultimate tensile strength \( \sigma_{UTS} \), and microhardness \( H_{v} \) are similar: these mechanical characteristics somewhat decrease after aging, then after HPT processing they are restored nearly to the initial level (Fig. 5a). Relative elongation to failure is kept on the same level \( \delta = 26–28\% \) for all these states.

The results of electrical conductivity measurements at 293K \((1/p_{293})\) are shown in Fig. 6a. As expected the purification of the Al matrix from the Zr solute atoms during the aging provides a marked increase in electrical conductivity up to 58.8\% IACS. After the subsequent HPT processing the conductivity decreased to 55.8\% IACS. The latter is caused mainly by partial dissolution of the \( \mathrm{Al}_{3} \mathrm{Zr} \) phase.

The tests for thermal stability of strength were also carried out by short annealing for 1 h at different annealing temperatures \( T_{an} \) in the range 90–400 °C. The results are shown in Fig. 6b. The \( H_{v} \) does not decrease with increasing \( T_{an} \) up to \( T_{an} = 230 \) °C, demonstrating good thermal stability. Moreover, some additional strengthening (up to ~16\%) as a result of the annealing in the range 90–230 °C takes place. Similar strengthening by annealing was found for commercially pure Al, preliminary nanostructured by severe plastic deformation [17,18,19]. This phenomenon was explained by relaxation of non-equilibrium HAGBs in UFG microstructure during annealing that impedes the onset of plastic flow under subsequent loading [17]. The AG–HPT samples have also UFG structure with the predominant amount of HAGBs, so the observed strengthening by annealing in them may be of the same origin as in pure Al. In addition, a possible contribution to strengthening from grain boundary segregation, which could occur.

Fig. 3. EBSD maps of Al–0.4Zr(wt.%) alloy in C–R state (a), after AG treatment (b), and after AG–HPT treatment (c).
Fig. 4. Grain size distribution (a, b, c) and grain boundary misorientation angle distribution (d, e, f) of Al–0.4Zr(wt.%) alloy in C–R state (a, d) after AG treatment (b, e) after AG–HPT treatment (c, f).

Fig. 5. a – experimentally obtained values of microhardness ($H_V$), ultimate tensile strength ($\sigma_{UTS}$) and proof stress ($\sigma_{0.2}$) of Al–0.4Zr(wt.%) alloy in initial C–R state, after aging (AG–state) and subsequent HPT processing (AG–HPT state); b – experimentally obtained values of the proof stress ($\sigma_{0.2}$) in comparison with theoretically estimated contributions from grain size strengthening ($\sigma_{GB}$), solid solution hardening ($\sigma_{SS}$), precipitation hardening ($\sigma_{Or}$), and dislocation strengthening ($\sigma_{dis}$). $\sigma_0$ – the Peierls-Nabarro stress.

cur during annealing of the HPT processed samples, cannot be also excluded from the consideration. Further investigation of the fine structure of GBs is needed. It is important that annealing can provide not only additional strengthening, but also increase in electrical conductivity. As a result of the annealing of the AG–HPT samples at $T_{an}$=230 °C for 1 h, the best combination of strength $\sigma_{UTS}$=142 MPa and conductivity (~58.3% IACS) was achieved (Table 3).

### 3.3. Analysis of strengthening and charge scattering

In the temperature range 77 – 300K, the electrical resistivity of an alloy can be described by the
Matthiessen’s rule [20], according to which it is equal to the sum of contributions from different scattering mechanisms that do not affect each other:

\[
\rho_{\text{alsy}} = \rho_{\text{pure}} + N_v \Delta \rho_{\text{vac}} + L_{\text{dis}} \Delta \rho_{\text{dis}} + S_{\text{GB}} \Delta \rho_{\text{GB}} + \sum C_i \Delta \rho_{\text{sol}} + \Delta \rho_{\text{pt}}
\]

where \(\rho_{\text{pure}} = 2.7 \ \Omega \cdot \text{m} [21]\) is the electric resistivity of a single-crystalline defect-free aluminum, \(\Delta \rho_{\text{vac}} (\Omega/\text{at.\%})\), \(\Delta \rho_{\text{dis}} = 2.7 \times 10^{-8} \ \Omega \cdot \text{m}^2 [22]\), \(\Delta \rho_{\text{GB}} = 2.6 \times 10^{-16} \ \Omega \cdot \text{m}^2 [22]\) are the contributions from unit vacancy concentration, unit densities of dislocations, and grain boundaries in Al, respectively, \(\Delta \rho_{\text{sol}} (\Omega/\text{wt.\%})\) is the contribution from unit concentration of \(i\)-th impurity in the solid solution, \(N_v\) (at.\%) is the vacancy concentration, \(L_{\text{dis}}\) (m\(^{-2}\)) is the dislocation density, \(S_{\text{GB}}\) (m\(^{-1}\)) is the bulk density of GBs, \(C_i\) (at.\%) is the concentration of \(i\)-th solute atom; \(\Delta \rho_{\text{pt}}\) – the contribution originating from the secondary phase precipitates.

On the basis of the obtained changes in microstructure (Table 2) and literature data for \(\Delta \rho_{\text{GB}}\) Zr \(\rho_{\text{GB}} = 15.8 \ \Omega \cdot \text{m/ wt.\%} [23]\), we estimated the electrical resistivity of Al–0.4Zr for the three studied state: C–R, AG, and AG–HPT and compared the estimates with the experimentally obtained values of resistivity at 77K (Fig. 6a). For the comparison we chose low temperature measurements, because at 77K the influence of thermal fluctuations is much lower compared to the ambient temperature and effect of microstructure changes on the resistivity is more evident. The contribution originating from the secondary phase particles can be calculated as an effective reduction of the conducting volume [24]. Since even maximum possible \(V_p\) in Al–0.4Zr (wt.\%) is very small (0.49 vol.\%) [12], this contribution is negligible for both AG and AG–HPT states. Contribution of vacancies to the electrical resistivity is also negligible because they are very quickly annealed in Al alloys even at room temperature [25].

Results of estimation of \(L_{\text{dis}} \Delta \rho_{\text{dis}}, S_{\text{GB}} \Delta \rho_{\text{GB}}, \) and \(C_i \Delta \rho_{\text{sol}}\) are presented in Fig. 6a. A detailed procedure of similar estimations is presented for the Al–Mg–Si system in [4]. There is good agreement between the experimental data and theoretical estimates for C–R and AG samples and substantial discrepancy for the AG–HPT state. As noted above, the thickness of the TEM samples is not exactly defined, the obtained estimates of \(V_p\) and \(C_{\text{sol}}\) are very approximate. In addition, in the AG–HPT sam-

Table 3. Mechanical and electrical properties of Al–0.4Zr (wt.%) alloy.

<table>
<thead>
<tr>
<th>State</th>
<th>(H_v), MPa</th>
<th>(\sigma_{\text{xx}}), MPa</th>
<th>(\sigma_{\text{UTS}}), MPa</th>
<th>(\delta), %</th>
<th>(\omega), % IACS</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–R</td>
<td>469±10</td>
<td>117±2</td>
<td>128±2</td>
<td>26±1</td>
<td>50.7</td>
</tr>
<tr>
<td>AG</td>
<td>399±22</td>
<td>72±6</td>
<td>95±4</td>
<td>27±2</td>
<td>58.8</td>
</tr>
<tr>
<td>AG–HPT</td>
<td>439±11</td>
<td>96±2</td>
<td>118±2</td>
<td>28±1</td>
<td>55.8</td>
</tr>
<tr>
<td>AG–HPT–AN(230°C)</td>
<td>463±7</td>
<td>137±2</td>
<td>142±1</td>
<td>18±1</td>
<td>58.3</td>
</tr>
</tbody>
</table>

Fig. 6. a – experimentally obtained values of electrical conductivity (\(\omega\)) at RT and electrical resistivity at 77K (\(\rho_{\text{alsy}}\)) for Al–0.4Zr (wt.%) alloy in initial C–R state, after aging (AG–state), and subsequent HPT processing (AG–HPT state) in comparison with theoretically estimated contribution to resistivity from grain boundaries (\(\rho_{\text{GB}}\)), dislocations (\(\rho_{\text{dis}}\)), solute Zr atoms (\(\rho_{\text{sol}}\)) electrical resistivity of coarse-grained pure Al (\(\rho_{\text{pure}}\)); b – microhardness (\(H_v\)) of AG–HPT samples versus annealing temperature.
for the small \( V_{pt} \) values like in our study. For estimation of contribution from the Orowan mechanism, we proceed with the values of \( \Delta V_{pt} \) (Table 2).

Strain hardening can be calculated as:

\[
\sigma_{ss} = M \alpha \psi \beta \theta_{cr}^{1/2},
\]

(6) where \( M = 3.06 \) is the Taylor factor [33], \( \alpha = 0.33 \) is the dislocation interaction parameter [34], \( G = 26 \) GPa is the shear modulus, \( b = 2.86 \) Å is the Burgers vector, \( L_{ds} \) is the dislocation density.

Grain boundary strengthening can be calculated from Hall-Petch relation [26]:

\[
\sigma_{gb} = K d_{av}^{-1/2},
\]

(7) where \( K = 0.07 \) MPa m \(^{1/2} \) is the Hall-Petch coefficient [27] and \( d_{av} \) is the average grain size. For some specific microstructures the grain boundary strengthening is better described by the modified equation [35]:

\[
\sigma_{gb} = K \sqrt{1 - f_{gb}} / d_{av},
\]

(8) where \( f_{gb} \) is the fraction of grain boundaries with the misorientations lower than a certain critical angle \( \theta_{cr} \) that do not contribute to grain boundary strengthening. Eq. (8) takes into account the fact that grain boundaries with low misorientations are of dislocation character and do not participate in grain boundary strengthening [35].

The contribution to hardening from Zr dissolved in solid solution was estimated as [36]:

\[
\sigma_{ss} = k Z_{\text{sol}}^{2/3},
\]

(9) where \( k Z_{\text{sol}} = 9 \) MPa wt. \( 2/3 \) is calculated from the data of [37].

Using Eqs. (4–9) we estimated the contributions from all the possible mechanisms to the total strengthening, which are presented in Fig. 5b by diagrams in comparison with the experimentally obtained \( \sigma_{gb}^{\exp} \). For the initial C–R state there is a good agreement between theoretically estimated \( \sigma_{ss}^{\exp} \) and experimentally obtained \( \sigma_{gb}^{\exp} \) values. The best agreement was obtained when \( \sigma_{gb}^{\exp} \) was estimated with equation (8) for \( \theta_{cr} = 5 \). It is reasonable because most of GBs in the initial C–R state are low angle GBs and GBs with low misorientation angles could not participate in GB strengthening.

For the AG and AG–HPT states, the difference in estimates of \( \sigma_{gb} \) with Eq. (7) and Eq. (8) was rather small. As seen in Fig. 5b, a good agreement of experimental and theoretical values of \( \sigma_{gb} \) for the AG and AG–HPT states are achieved without contributions to strengthening from secondary phase precipitates, precipitate bypass by dislocation looping (Orowan mechanism), or a combination of these two mechanisms in coarse-grained, precipitation-strengthened alloys at ambient temperature [29]. It was shown in [30], in Al–Zr alloys with precipitations larger than 4.0 nm in diameter the Orowan mechanism is predominant, hence in our case \( \sigma_{ss} = \sigma_{cr} \), where \( \sigma_{cr} \) is Orowan stress [29]:

\[
\Delta \sigma_{cr} = \frac{0.4 Gb \ln(2\pi / b)}{\pi \lambda} \frac{1}{\sqrt{1 - \nu}},
\]

(4) where \( \nu = 0.345 \) is Poisson’s ratio of Al [31], \( f = \sqrt{2/3} \langle r \rangle \) is the mean radius of a circular cross section in a random plane for a spherical precipitate [32], and \( \lambda \) is the inter-precipitate spacing in the Orowan mechanism, which can be calculated from the following equation [28]:

\[
\lambda = 2 \pi \left( \frac{\pi}{2V_{pt}} - 1 \right),
\]

(5)
nanoparticles. This result points out that the most probably Orowan mechanism does not operate in the AG and AG–HPT samples. Our results are in a good agreement with the results of [12], where microhardness of Al−Zr alloys with 0.2−0.5Zr wt. % (obtained by combined casting and rolling) did not increase after their long-term annealing at temperatures of 300−650 °C despite the formation of nanoscale AlZr precipitates. No analysis of strengthening was done in [12]. On the other hand, it was shown that the strengthening by the Orowan mechanism perfectly works for the coarse grained Al−0.1Zr alloy with the concentration 0.1Zr at.% corresponding to 0.33Zr wt.% (obtained by non-consumable electrode arc-melting) [37,38] and in the case of Al−Zr alloys with 0.1 and 0.2 wt.% Zr fabricated by casting [6]. In Al−0.1Zr [37,38] the size of AlZr precipitates and their volume fraction were similar to that in the AG samples (this work). Despite the operation of the Orowan mechanism in the aged Al−0.1Zr (at.%) samples [37,38], their maximum strength achieved was not higher than that of the AG and AG–HPT samples. Due to the coarse grained structure, the initial Al−0.1Zr(at.%) [37,38] and Al−Zr with 0.1 and 0.2 wt.% Zr samples [6] had very low strength. Our results testify that strengthening by the Orowan mechanism is suppressed in the AG and AG–HPT samples. A probable reason for this is the small average grain size (0.8−2.1 μm) in these samples. The AG–HPT and AG samples have ultrafine grained (UFG) and close to UFG microstructure, respectively, resulting in higher volume fraction of grain boundaries and triple junctions which have enhanced energy. Therefore, significant amount of precipitates are formed therein and do not contribute to the Orowan strengthening due to overlap with the grain boundary strengthening mechanism [39]. To understand the nature of this phenomenon (suppression of strengthening by the Orowan mechanism), it is necessary to carry out additional studies including a deep theoretical analysis.

4. CONCLUSIONS

The microstructure evolution and resulting change in strength and electrical conductivity after long-term aging at 375 °C and subsequent treatment by high pressure torsion have been studied for the Al−0.4Zr (wt.%) alloy obtained by combined casting and rolling. The following conclusion can be drawn.

Aging by long-term annealing leads to formation of nanoscale secondary phase precipitates with the average size 13 nm that is accompanied by purification of the Al matrix. Such a microstructure provides good electrical conductivity (58.8% IACS), but the strength σUTS decreasing from 128 to 95 MPa. Subsequent HPT processing results in grain refinement to ~0.8 μm and dissolution of most AlZr nanoparticles accompanied by coarsening the remaining nanoparticles to ~56 nm. The HPT processing provides increase in strength (σUTS=118 MPa) and decrease in conductivity (ω=55.8% IACS). Strengthening by annealing for 1 h in the temperature range 90−230 °C was observed for the HPT processed samples. The best combination of strength (σUTS=142 MPa) and conductivity (ω=58.3% IACS) was found for the AG–HTP samples after additional annealing at 230 °C for 1 h.

Analysis of contributions of different possible strengthening and charge scattering mechanisms was made on the basis of specific microstructural features of the Al−0.4Zr (wt.%) alloy for all the studied states. It is shown that in all the studied states: C−R, AG, and AG−HPT, the electrical resistivity is mainly controlled by the concentration of Zr in solid solution and the strength is mainly controlled by grain boundary strengthening. Strengthening by the Orowan mechanism is suppressed in AG and AG−HPT samples despite the presence of dispersed nanoscale AlZr precipitates.

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