EFFECT OF HOT ROLLING AND ANNEALING ON THE STRUCTURE AND PROPERTIES OF Al-Fe-Si-Zr ALLOYS

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Abstract

The aluminum alloys with 1 % Fe, 0.3 % Zr, 0.25 % Si and with 1 % Fe, 0.3 % Zr, 1 % Si (in wt.%), were investigated after hot rolling to a strain of 70% at 300 °C followed by annealing at 400-500 °C during 1 and 3 h. The hot rolling resulted in the elongation of initial grains along the rolling direction. The lamellar microstructure with the transverse size of structural elements of about 0.7-0.8 µm evolved during deformation. The hot rolling was accompanied by strengthening of the both alloys and an increase of the electrical conductivity in the Al -1 % Fe, 0.3 % Zr, 1 % Si alloy. The yield strength after annealing at 400 °C was 160 MPa and 165 MPa for the Al with 1 % Fe, 0.3 % Zr, 0.25 % Si and Al with 1 % Fe, 0.3 % Zr, 1 % Si alloys, respectively. The fine structure after annealing contained dispersed particles with a size of about 4-5 nm, which were identified as Al_3Zr with L12 structure. An increase in the electrical conductivity to 53% IACS confirmed decomposition of solid solution during annealing in the Al - 1 % Fe, 0.3 % Zr, 0.25 % Si and Al - 1 % Fe, 0.3 % Zr, 1 % Si alloys. The strengthening was discussed in terms of the modified Hall-Petch relationship.

Keywords: Al-Fe-Si-Zr alloys, strengthening, electrical conductivity, Al_3Zr particles, Hall-Petch relationship

1. INTRODUCTION

Aluminum alloys of the Al-Fe-Si system are characterised by high electrical conductivity about 60% IACS and are used as power line wires [1]. However, such materials have low mechanical properties and thermal stability of work hardened microstructure. This disadvantages limit application of Al-Fe-Si alloys in thermocables. Significant increase of strength and thermal stability can be achieved by Zr additions due to the formation of the Al_3Zr dispersoids during homogenizing treatment [2]. In additional, an increase of the Fe and Si content is accompanied by the hardening without reduction of the electrical conductivity. The aging of the Al-Fe-Si-Zr alloys with high Fe and Si concentrations leads to hardness and electrical conductivity increase [3]. Note that deformation by conventional methods, such as rolling, leads also to substantial strengthening of the Al-Fe-Si-Zr alloys [2]. Thus, the combination of the alloying and plastic deformation provides good mechanical and functional properties. The microstructure effect on the strength can be described by the modified Hall-Petch relationship. The yield strength reads as follows [4]:

$$\sigma_{YS} = \sigma_0 + \sigma_{part} + k_y\, D^{-0.5} + \alpha\, M\, G\, b\, \rho^{0.5} \quad (1)$$

where:

σ_{YS} - the yield strength (MPa),
σ_0 - lattice friction strength (MPa),
σ_{part} - particle strengthening (MPa),
k_y - the Hall-Petch coefficient (MPa·m$^{0.5}$),
D - the mean grain size (m),
α - constant,
M - the Taylor factor,
G - the shear modulus (Pa),
b - the Burgers vector (m),
ρ - dislocation density (m^{-2}).

The modified Hall-Petch relationship estimates the contribution of the different strengthening mechanisms and microstructure effect on mechanical properties.

The aim of the present research is to investigate the microstructure evolution, electroconductivity change and strengthening mechanisms in Al-Fe-Si-Zr after thermo-mechanical treatment.

2. EXPERIMENT

The aluminum alloys, Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si, were examined. The actual chemical compositions were Al - 0.87 % Fe, 0.41 %Zr, 0.21 % Si and Al - 0.96 % Fe, 0.32 % Zr, 0.75 % Si (all in wt.%). The aluminum alloys were prepared in an electric furnace LAC PT 90/13 (LAC, s.r.o., Rajhrad, Czech Republic) in graphite-chamotte crucibles with cooling at maximum rate. The cast alloys were heated to 300 °C during 15 min and hot rolled at 300 ºC with 70% reduction. After the hot rolling (HR) aluminum alloys were annealed at 400-500 °C during 1 or 3 h with air cooling. The structural observations were carried out on sections perpendicular to the transverse direction (TD) of the rolled samples using a Nova Nanosem 250 (SEM, FEI, Hillsboro, OR, USA) scanning electron microscope equipped with an electron back-scattering diffraction pattern (EBSD) analyzer incorporating an orientation imaging microscopy (OIM) system and a JEOL JEM-2100 (JEOL Ltd., Tokyo, Japan) transmission electron microscope (TEM). The samples for transmission or scanning electron microscopy were electro-polished using an electrolyte of HNO_3:CH_3OH=1:3 at a temperature of -30 °C with a voltage of 20 V. The electrical conductivity was measured by the method of eddy currents using a Constanta K6 device (Constanta, Saint Petersburg, Russia). The specimens with a gauge length of 16 mm and cross section of 1.5 mm x 3 mm were tensioned at an initial strain rate of $2\cdot10^{-3}$ s^{-1} with the tensile axis parallel to the rolling direction.

3. RESULTS AND DISCUSSION

Typical cast microstructures of the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys are shown in **Figure 1**. The phase composition of the Al-1Fe-0.3Zr-0.25Si alloy includes supersaturated solid solution aluminum matrix and the needle Al_3Fe particles. In contrast, the cast structure of the Al-1Fe-0.3Zr-1Si alloy is characterized by the eutectic formed with the Al_8Fe_2Si phase that has a Chinese script-like morphology. The precipitation of spherical particles, which Belov et al. [2] defined as a Si-rich inclusion, is clearly seen in aluminum alloy with 1 % Si. Note that Zr is in solid solution after casting of Al-1Fe-0.3Zr-Si alloys, so primary Al_3Zr particles don't present in the microstructure.

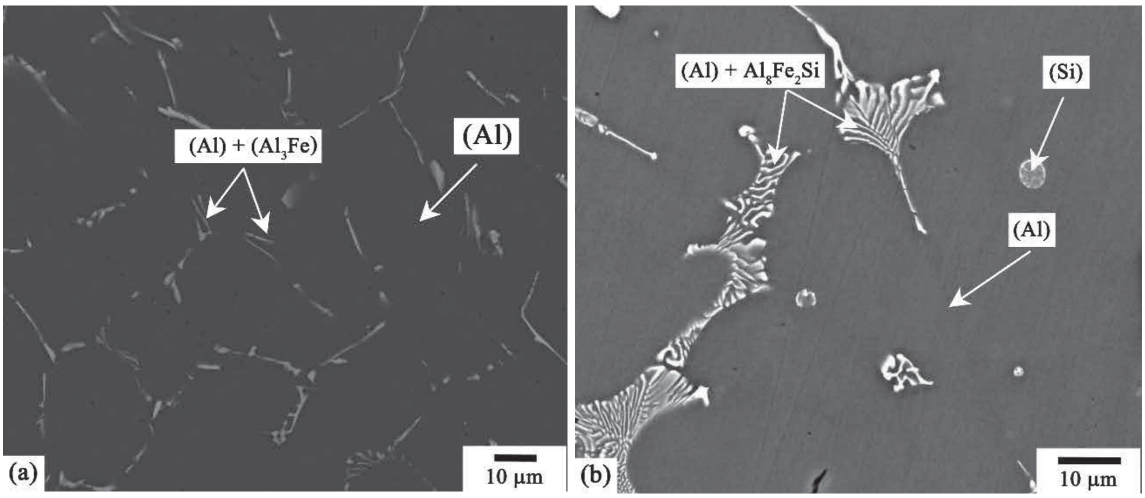

Figure 1 Microstructure of cast aluminum alloys with different Si content: a) Al-1Fe-0.3Zr-0.25Si; b) Al-1Fe-0.3Zr-1Si

Mechanical properties and electrical conductivity after hot rolling and annealing of the Al-1Fe-0.3Zr-Si alloy. The effect of hot rolling and annealing in temperature range 400-500 °C during 1 and 3 h on yield strength (YS) and electrical conductivity of Al-1Fe-0.3Zr-Si alloys is shown in **Figure 2**. The cast alloys are characterized by low yield strength (YS) of 25 MPa and 55 MPa for the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys, respectively. An increase of the Si concentration results in strengthening of aluminum alloy in cast condition. Hot rolling leads to significant strengthening of the alloys and dramatic reduce of plasticity; YS of Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si attain 175 MPa and 200 MPa, respectively.

Subsequent annealing treatment leads to the strength decrease regardless of the annealing temperature and time. Maximum YS after hot rolling with annealing comprises 160 MPa in the Al-1Fe-0.3Zr-0.25Si alloy and 165 MPa in the Al-1Fe-0.3Zr-1Si alloy after the annealing treatment at 400 °C during 3 h. Moreover, annealing was accompanied by electroconductivity increase. The best electrical conductivity was observed in the Al-1Fe-0.3Zr-0.25Si alloy after hot-rolling with annealing at 400 °C for 3 h and attains 53% IACS (cf. with 45% IACS in cast condition). Note that plastic deformation leads to change in the peak aging conditions from 450 °C to 400 °C [3].

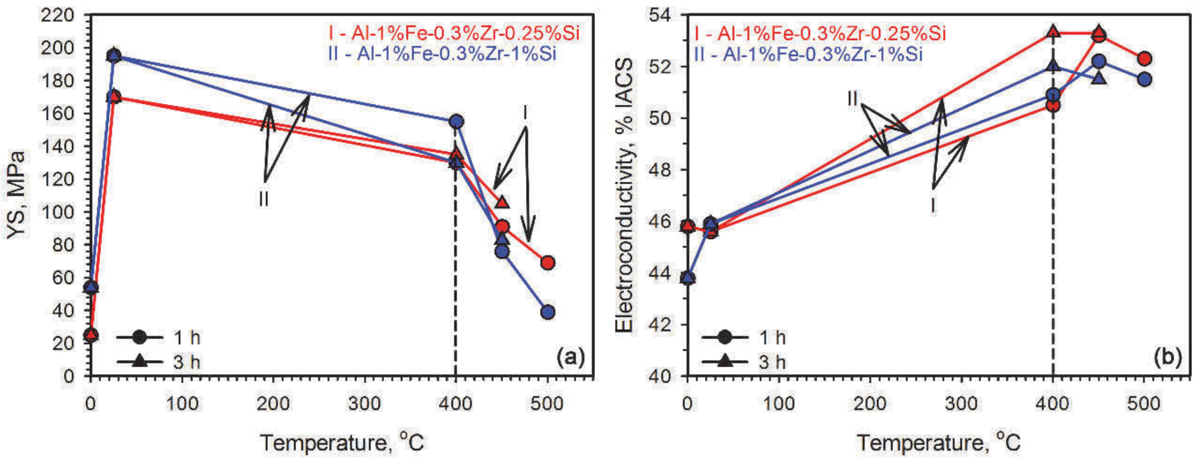

Figure 2 Temperature effect on the yield strength (YS) (a) and electrical conductivity (b) of the Al-1Fe-0.3Zr-Si alloys with different Si content.

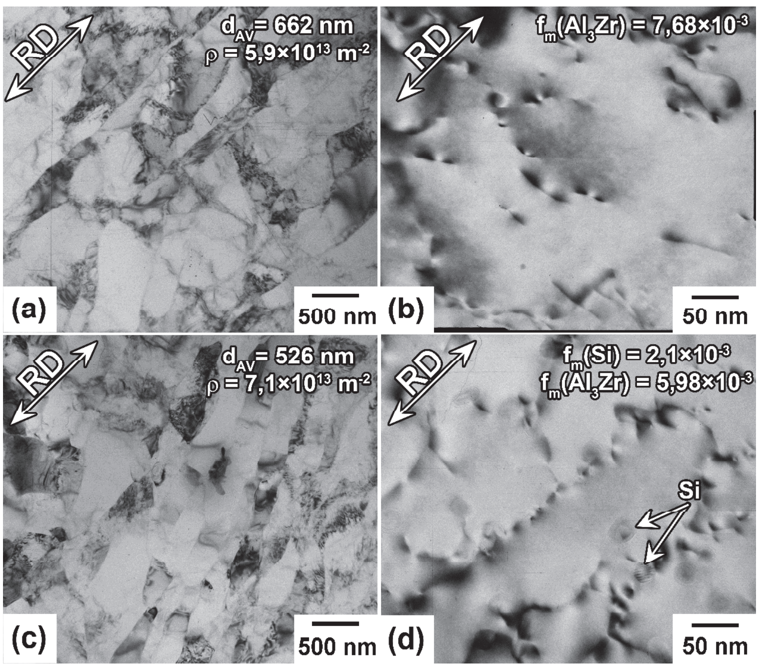

Figure 3 TEM micrographs of the Al-1Fe-0.3Zr-0.25Si (a, b) and the Al-1Fe-0.3Zr-1Si (c, d) alloys after hot rolling; d_{av} is the average subgrain size, ρ is the dislocation density, f_m is the mass fraction of particles calculated using Thermocalc Software

Fine microstructure of the Al-1Fe-0.3Zr-Si alloys after hot rolling and annealing. Significant change of the mechanical and functional properties of aluminum alloys is associated with the microstructure evolution during hot rolling and annealing treatment. **Figure 3** illustrates typical deformation microstructure of the Al-1Fe-0.3Zr-Si alloys after hot rolling at 300 °C.

The deformation substructures in the present alloys look like typical hot-worked ones consisting of dislocation subboundaries with mainly low-angle misorientations. Band-like configurations almost parallel to the rolling direction (RD) prevail in the microstructure observed in the longitudinal section. The average size of transverse and longitudinal subboundary spacing (d_{av}) for the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys was 0.7 µm and 0.8 µm, respectively. The high dislocation density ρ about $6\cdot10^{13}$ m^{-2} and $7\cdot10^{13}$ m^{-2} observes into subgrain in alloys with 0.25 wt.% Si and 1 wt.% Si, respectively.

Annealing at 400 °C during 3 h is accompanied by recovery and dislocation density reduction. Annealing treatment led to the supersaturated solid solution decomposition and the dispersed Al_3Zr particles precipitated (see the selected area diffraction pattern in **Figure 4b**). This spheroidal precipitates have cubic L12 structure with a size about 4-5 nm and coherence with matrix according to cube-cube relationship. In addition, the Si-rich particles form during annealing in the Al-1Fe-0.3Zr-1Si alloy (**Figure 4d**). The Si-rich particles are characterized by globular shape with a diameter about 30-40 nm. Note that dispersed precipitates provide stabilization deformation structure and inhibit primary recrystallization development in the Al-1Fe-0.3Zr-Si alloys.

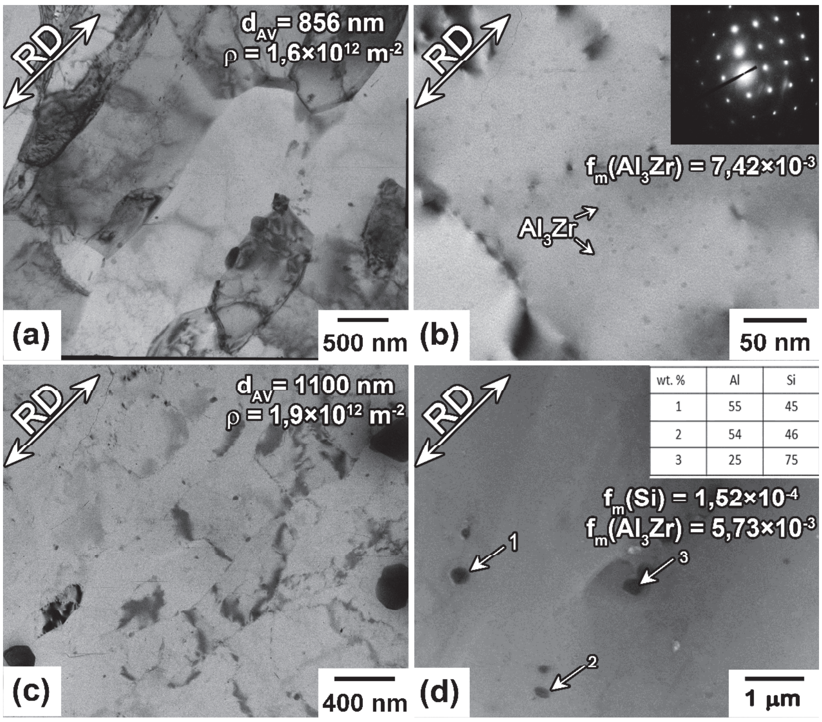

Figure 4 TEM micrographs of the Al-1Fe-0.3Zr-0.25Si (a, b) and the Al-1Fe-0.3Zr-1Si (c, d) alloys after hot rolling and annealing at 400 °C for 3 h; d_{av} is the average subgrain size, ρ is the dislocation density, f_m is the mass fraction of particles calculated using Thermocalc Software

Strengthening of the Al-1Fe-0.3Zr-Si alloys. The dislocation (substructural) strengthening of the Al-1Fe-0.3Zr-Si alloys can be calculated by the Taylor relationship (the third term in **Eq. 1**). The constants of σ_0 = 10 MPa, k_y = 0.2, α = 0.24, G = 25.4 GPa, b = 0.286 nm and M = 3.06 [4] were used for the calculation of the strengthening contributions to the overall YS. The grain size D was estimated using the EBSD-map and reaches 15.2 µm and 14.1 µm after hot rolling, and 19.8 µm and 24.2 µm after annealing for the Al-1Fe-0.3Zr-

0.25Si and Al-1Fe-0.3Zr-1Si alloys, respectively. The contributions of different strengthening mechanisms to the overall strength as calculated using **Eq. 1** for the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys after hot rolling (HR) and subsequent annealing (HR+A) at 400 °C for 3 h are presented in **Table 1**. The dispersion strengthening was estimated as a difference between experimental YS and calculated strengthening including the lattice friction strength, the substructural strengthening, and the grain boundary strengthening.

It clearly seen that the main contribution to YS is made by the particle strengthening regardless of thermo-mechanical treatment and chemical composition. The particle strengthening is provided by Al_3Zr, Si particles and eutectic. The volume fraction of Al_3Zr particles estimated using Thermocalc Software for the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys at 300 °C (the temperature of HR) is 0.77% and 0.63% mass (**Figure 3**). Thus, the deformation at 300 °C provides the solid solution decomposition and Al_3Zr particles precipitate. However, a short treatment time does not provide complete decomposition of solid solution [2, 3]. Thus, Al_3Zr particles precipitate during subsequent annealing as well. In addition, the solid solution in the Al-1Fe-0.3Zr-1Si alloy is supersaturated by Si due to accelerated cooling [2, 3] and Si particles with a size about 30 nm precipitate in aluminum matrix during hot deformation (**Figure 3d**). This is good agreement with electroconductivity change and TEM observation. Plastic deformation should lead to electrical conductivity degradation due to dislocation density increase, but stability or rise of conductivity is observed in the aluminum alloys after hot rolling in experiment (**Figure 2**). The electroconductivity increase can result from dispersed Al_3Zr and Si particle precipitate. A small increase in the particle strengthening in the Al-1Fe-0.3Zr-0.25Si alloy results from an increase in the volume fraction of the dispersed Al_3Zr particles (**Figure 4**). Dramatic decrease by 25 MPa in the particle strengthening in the Al-1Fe-0.3Zr-1Si alloy can be associated with a coarsening of dispersed Si particles as observed in TEM (**Figure 4d**). Note that annealing after hot rolling leads to significant degradation of the dislocation strengthening to 7 MPa in both alloys, while the grain size strengthening slightly decreases by about 10 MPa.

Table 1 The strengthening contribution to the yield strength of the Al-1Fe-0.3Zr-Si alloys after hot rolling (HR) and subsequent annealing at 400 °C for 3 h (HR+A) as calculated by **Eq. (1)**

Strengthening contributor	**Al-1Fe-0.3Zr-0.25Si**		**Al-1Fe-0.3Zr-1Si**	
	HR (MPa/%)	**HR+A** (MPa/%)	**HR** (MPa/%)	**HR+A** (MPa/%)
Lattice friction strength	10/6	10/7	10/5	10/8
Grain size strengthening	52/30	45/33	54/27	41/31
Dislocation strengthening	41/25	7/5	45/23	7/6
Particle strengthening	67/39	74/55	87/45	62/55
Experimental YS	170/100	135/100	195/100	130/100

4. CONCLUSION

Hot rolling of the Al-1Fe-0.3Zr-Si alloys resulted in the band-like subgrains elongated along the rolling direction with an average of transverse and longitudinal subboundary spacings about 0.7 µm and 0.8 µm in the alloys with 0.25 wt.% Si and 1 wt.% Si, respectively. Hot rolling was accompanied by strengthening of the both alloys due to substructure strengthening. The annealing at 400 °C during 3 h resulted in the yield strength decrease to 135 MPa and 130 MPa for the Al-1Fe-0.3Zr-0.25Si and Al-1Fe-0.3Zr-1Si alloys, respectively. Annealing at 400 °C led to the decomposition of the solid solution and to increase of the electrical conductivity to 53% IACS in the Al-1Fe-0.3Zr-0.25Si alloy and 52% IACS in the Al-1Fe-0.3Zr-1Si alloy. The microstructure after annealing contained dispersed Al_3Zr particles with a size of about 4-5 nm. The YS decreased after annealing in the Al-1Fe-0.3Zr-0.25Si alloy resulted from the dislocation strengthening decrease. Dramatic decrease in

YS in the Al-1Fe-0.3Zr-1Si alloy can be attributed to a decrease in both the dislocation strengthening and the particle strengthening.

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