FORMATION OF STRUCTURE OF THE ALUMINUM ALLOY BASED ON Al-Zn-Mg-Ni-Fe SYSTEM DURING THERMOMECHANICAL TREATMENT

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Abstract

This work investigates the formation of the structure of high-strength aluminum alloy based on an Al-Zn-Mg-Ni-Fe system with an iron content of more than 0.5 wt.% during thermomechanical treatment including hot and cold longitudinal rolling with a reduction ratio of 40 and radial-shear rolling with a reduction ratio of 8.16. Microstructural investigations showed that iron and nickel are joined into the eutectic Al_9FeNi phase at all stages, and the non-equilibrium T phase has no influence on technological plasticity. A hardness measure showed significant strengthening after rolling and precipitation ageing in mode T6 and T4.

Keywords: High-strength aluminum alloys, thermomechanical treatment, microstructure, hardness

1. INTRODUCTION

High-strength Al-Zn-Mg alloys with additions of iron and nickel (nickalins) have been attracting great research interest due to their high castability and competitive mechanical properties. These alloys have a hardening matrix with zinc, magnesium, and eutectic Al_9FeNi phase contributing to their unique characteristics. In particular compared to conventional 7xxx alloys, nickalins can be successfully subjected to argon-arc welding [1, 2]. It is important to note that the eutectic (Al) +Al_9FeNi is similar to (Al)+Al_3Ni but contains less nickel and iron as an alloying element. The recycled aluminum alloys and aluminum produced by electrolysis with an inert anode can be used as a feedstock due to high content of an iron. In favour of this approach, there is a 2618 aluminum alloy based on an Al-Cu-Mg system with Al_9FeNi phase; it is used as a heat resistant alloy and has high deformability [3, 4].

Any wrought hardenable aluminum alloy should be properly processed to obtain the required properties. The most frequently used wrought semi-finished products are sheets and rods that are conventionally obtained by longitudinal rolling (LR) and extrusion. One of the prospective ways to obtain small-diameter rods is by radial-shear rolling (RSR). This method is acknowledged as an effective method of forming a sub-microcrystalline structure with high mechanical properties. The RSR method relates to screw rolling with high supply angles (18-20°), allowing it to be applied to metals and alloys that are hard to deform, including high carbon iron-based alloys and hypereutectic silumins [5, 6]. There is a little information about the processing of aluminum alloys. One of the most informative and recent publication [7] discusses the processing of Al-4.4Cu-1.6Mg and demonstrates the achievement of subgrain structure. In addition, the grain growth and destruction of billets during processing at temperatures above 380 °C were noted in that work.

Nickalins should be carefully studied. It is considered as promising alloys which can replace 7xxx alloys and they should have competitive strength, which primarily depends on the structure. Therefore, it is relevant to study the structural behaviour of nickalins during RSR and to compare them with thin sheet structures.

This work aims to determine the formation of the structure of novel high-strength aluminum alloy based on Al-Zn-Mg-Ni-Fe system during thermomechanical treatment including the conventional LR method and the newer RSR method.

2. EXPERIMENT

The experimental alloy with a calculated composition of Al-6.8%Zn-2.8%Mg-0.5%Fe-0.6%Ni-0.15%Zr was melted in an induction furnace in graphite-chamotte crucibles using 99.85% pure aluminum, 99.98% pure zinc, 99.9% pure magnesium, and Al-20 wt.% Ni, Al-10 wt.% Fe, and Al-15 wt.% Zr master alloys. The molten metal was poured into the graphite flat cross-section mould (40 × 140 × 180 mm) and metallic round cross-section mould (40 × 250 mm). The chemical composition of alloy obtained by spectral analysis is given in **Table 1**.

Table 1 Chemical composition of experimental alloy (wt.%) [2]

Zn	Mg	Ni	Fe	Zr	Al
6.54	2.33	0.67	0.52	0.16	Balance

Thermodynamic calculations including the composition of the polythermal section, the determination of the phase transformation temperatures, and the analysis of non-equilibrium crystallization were performed using Thermo-Calc software (TTAL5 database) [8]. Thermomechanical treatment was conducted according to scheme shown in **Figure 1**. Heat treatment was carried out in an electric muffle furnace; the temperature was maintained with accuracy about 3 K. Hot rolling and cold rolling were conducted on laboratory rolling mills. The rods were rolled on a 14-40 RSR mill.

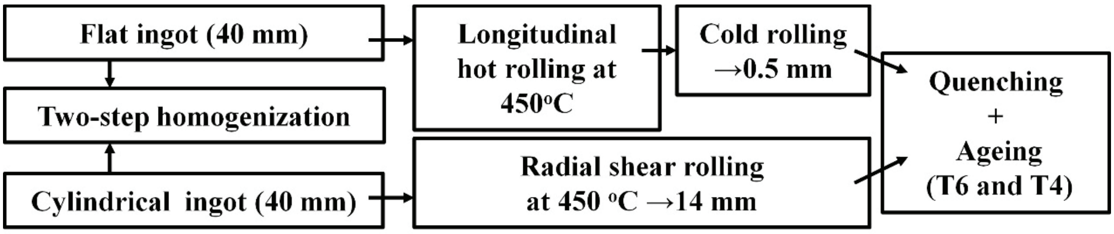


Figure 1 Scheme of thermomechanical treatment

Ingots and wrought semi-finished products (**Figure 2**) were subjected to microstructure analysis by scanning electron microscopy (SEM) TESCAN VEGA 3 and electron microprobe analysis (Oxford AZtec). The specimens were polished using a diamond suspension with 9, 6, and 3 μm fractions. Quantity analysis of microstructures including calculation of the volume fraction of particles and their distribution depending on size was carried out with ImageJ software.

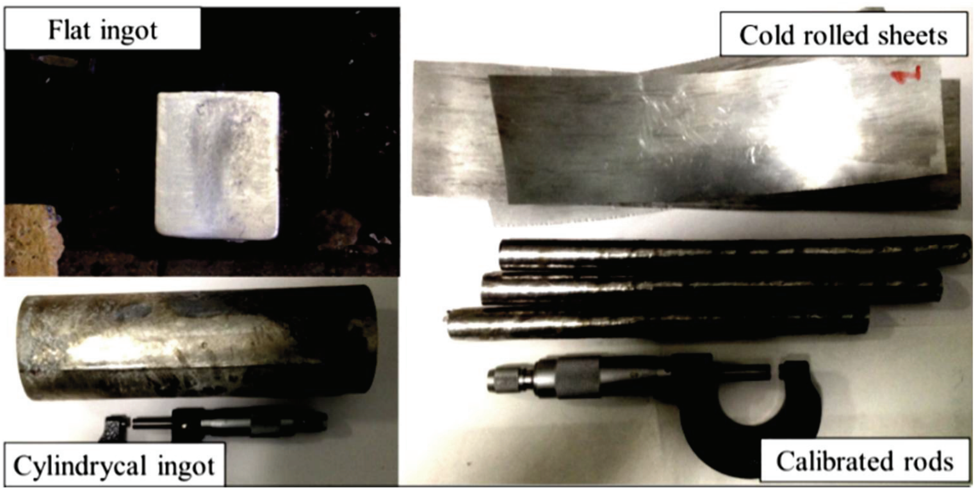


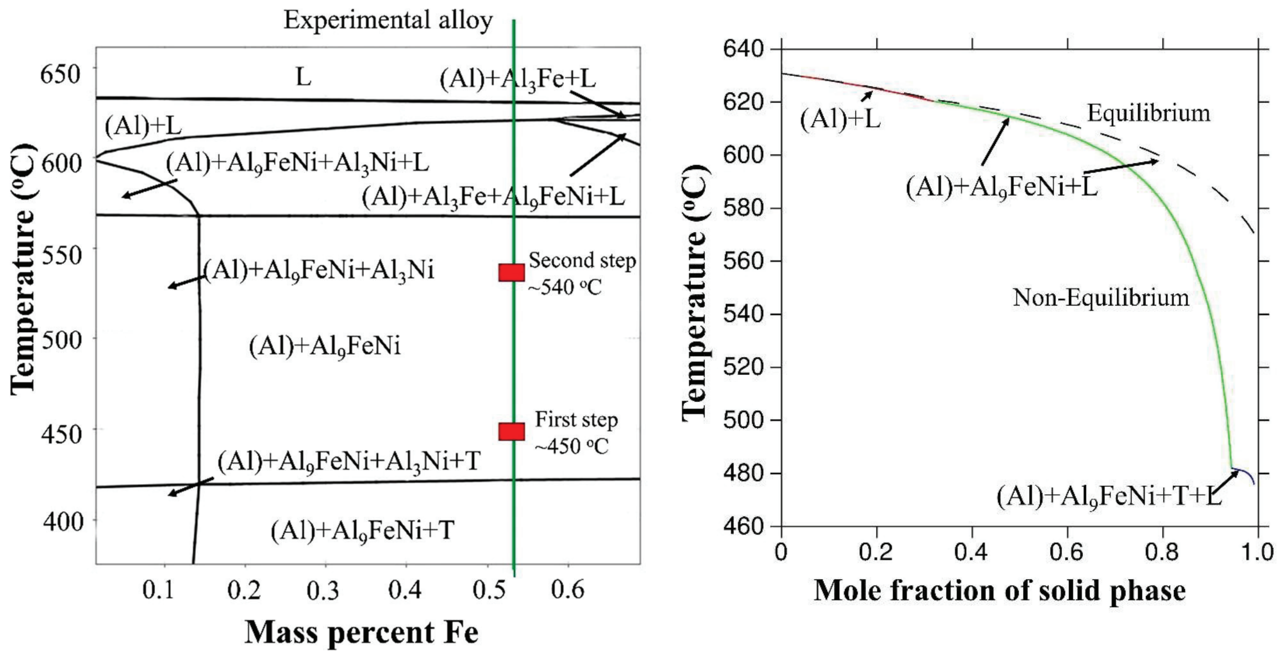
Figure 2 Study subjects

The Vickers hardness (HV) was evaluated using a Wilson Wolpert 930 N hardness tester at the following parameters: the load was 50 N and the holding time was 15 s.

3. RESULTS AND DISCUSSION

3.1. Calculations

According to the polythermal section (**Figure 3a**), the contents of iron and nickel satisfy the requirements for the formation of the equilibrium L→(Al)+Al_9FeNi eutectic reaction. A high equilibrium solidus (**Table 2**) makes it possible to conduct high-temperature annealing (at about 540 °C) to spheroidize the Al_9FeNi phase. Compared to high-strength Al-Zn-Mg-Cu alloys, which have a wide equilibrium crystallization range (up to 180 °C), due to the low eutectic temperatures of (Al)+$Al_2Mg_3Zn_3$ (T) (489 °C) and Al-$MgZn_2$ (M) (470 °C) [9], nickalins have an equilibrium crystallization range that is about 2.5 times lower, which makes them weldable. Moreover, the high content of copper in alloy such as 7150 contributes to the formation of S (Al_2CuMg) phase, which is hard to dissolve in (Al) matrix due to its low diffusivity coefficient in aluminum [10, 11]. Non-equilibrium crystallization was considered using the Scheil-Gulliver model. This simulation shows that non-equilibrium crystallization ends with L → (Al) + Al_9FeNi + T at 485 °C (**Figure 3b**). The formation of T phase increases the crystallization range at 87 °C and constrains the primary first step of annealing (about 450 °C under non-equilibrium solidus) to dissolve it in the (Al) matrix.



a) b)

Figure 3 Fragment of polythermal section of Al-Zn-Mg-Fe-Ni system (6.54 wt.% Zn, 2.33 wt.% Mg, and 0.67 wt.% Ni) (a) and mole fraction of solid - temperature diagram during non-equilibrium crystallization (b) [3, 4]

Table 2 Phase transformation temperatures calculated at Thermo-Calc

T_L (°C)	T_S (°C)	ΔT (°C)	T_{NS} (°C)	ΔT_{NS} (°C)	T_{SS} (°C)
632	565	67	485	147	417

T_L - liquidus; T_S - solidus; ΔT - crystallization range; T_{NS} - non-equilibrium solidus; ΔT_N - non-equilibrium crystallization range; T_{SS} - solvus

The calculated chemistry of the (Al) matrix at 450 °C is 6.6 wt.% Zn, 2.63 wt.% Mg, 0.16 wt.% Zr. This composition was used to define the phase composition of experimental alloy at ageing temperature (140 °C) to predict the phase composition and volume fraction of hardening phases (**Table 3**). According to the calculation, the microstructure of wrought semi-finished products after ageing in mode T6 will contain dispersion-hardened (Al) matrix and Al_9FeNi phase with a volume fraction of 3.7%.

Table 3 Phase composition (wt.%) of (Al) matrix at ageing temperature 140 °C calculated at Thermo-Calc

Phase	Q_M (%)	Q_V (%)	Al	Mg	Zn	Zr
(Al)	89.32	89.3	99.1	0.60	0.16	0.16
T	10.68	10.7	15.21	22.22	61.14	0.00

Q_M - mass fraction; Q_v - volume fraction

3.2. Microstructure

As predicted, the as-cast structure consists of (Al) matrix and eutectic phases: equilibrium Al_9FeNi phase and non-equilibrium T phase. These phases have an intermittent vein morphology, located around dendritic cells (**Figure 4a**). According to the multilayered distribution map **(Figure 4b)**, zinc and magnesium have partially dissolved in the (Al) matrix, which will reduce the time during isothermal holding in the first step of homogenization. There are no primary Al_3Zr (DO23) crystals because all of the zirconium has apparently dissolved in the aluminum during non-equilibrium crystallization. Al_3Zr (L12) modification is expected to be an effective means of preventing recrystallization during rolling and pre-quenching heating [12]. After two-step homogenization which took 6 hours, the Al_9FeNi phase develops a fine morphology (**Figure 4c**) with a volume fraction of 3.7% and predominant size of 1-3 μm^2 (**Figure 6a**). According to the results of microprobe analysis (**Table 4**), secondary T phase precipitated as fine particles less than 1 μm in size in the centre of dendritic cells.

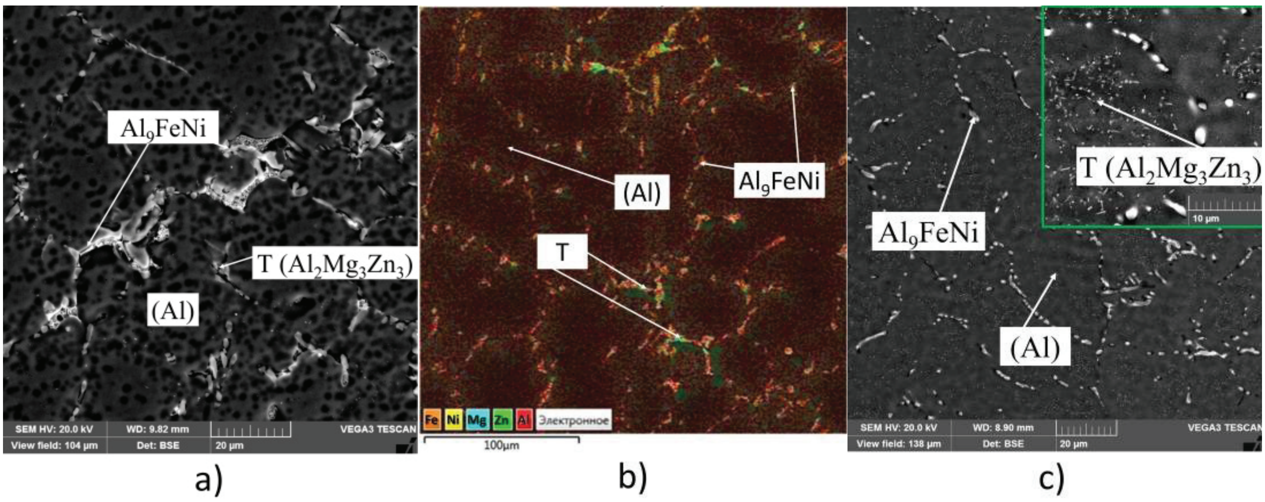


Figure 4 Microstructure of ingot from experimental alloy: (a) as-cast SEM; (b) as-cast multilayered distribution map; (c) after two-step homogenization

Table 4 Chemical composition (wt.%) of aluminum matrix obtained by microprobe analysis

Processing state	Zn	Mg	Fe	Ni	Al
As-cast	5.89	1.68	0.07	0.06	Balance
As homogenized	7.99	2.91	0.08	0.03	Balance

After rolling, we obtained high-quality thin sheets (0.5 mm, average reduction ratio = 1.33 for one pass, total reduction ratio = 40) and calibrated rods (14 mm, average reduction ratio = 1.7 for one pass, total reduction ratio = 8.16).The microstructures of hot-rolled sheets (**Figure 5a**) and cold-rolled sheets after ageing in mode T6 are similar. They consist of fine, relatively globular Al_9FeNi particles uniformly distributed in (Al) matrix. The size of the particles is about 1-2 μm^2 (**Figure 6b**). Eutectic particles in calibrated rods are larger (**Figure 5b**). The predominant size is 2 μm. However, some particles are 3-15 μm^2 in size (**Figure 6c**). The microstructure was obtained at the centre of the specimen and the picture will probably be similar in terms of structure after LR in the peripheral zone, because the RSR method is well-known for its hard fragmentation of the peripheral zone [5, 13].

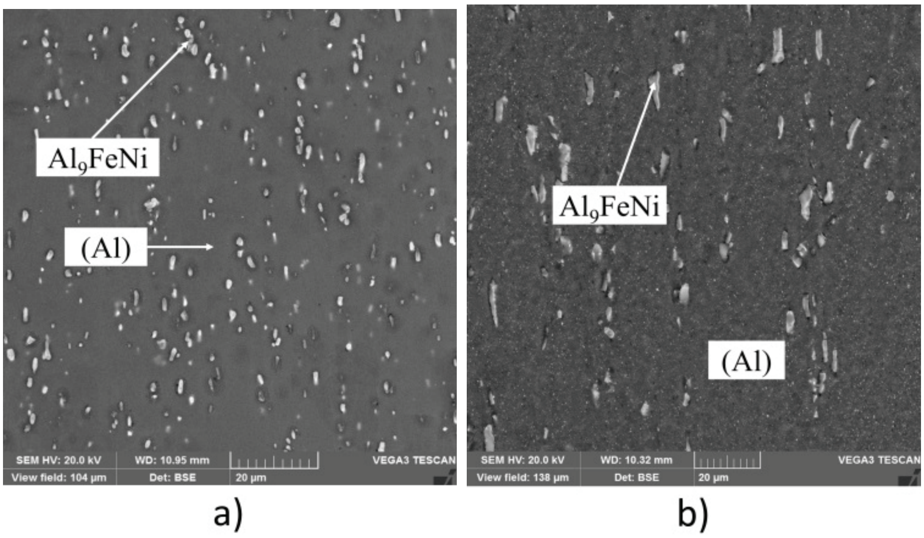
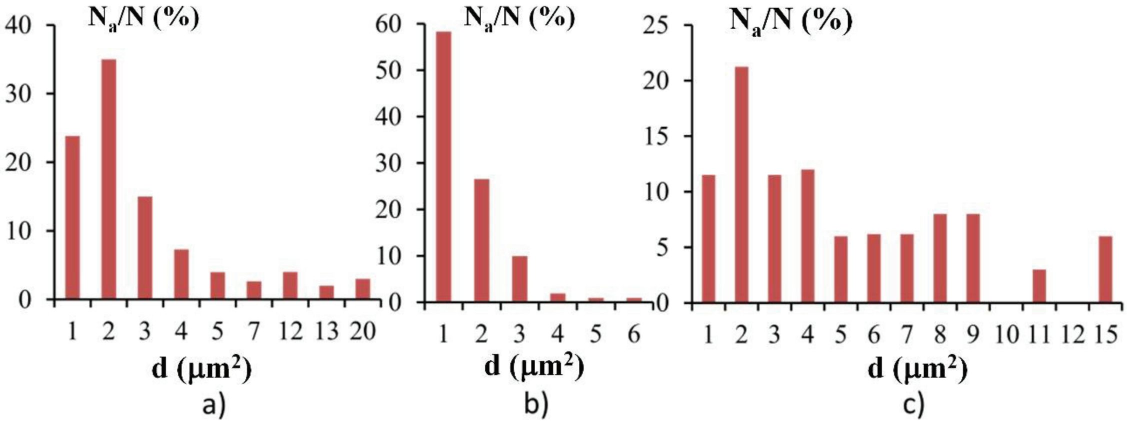


Figure 5 Microstructure of experimental alloy after ageing in mode T6: (a) thin sheet (0.5 mm); (b) rods (14 mm)



N_a/N -ratio of the number of particles of size *a* to the total number of particles; d - area of particles (μm^2)

Figure 6 Histograms of the particle size distributions of Al_9FeNi particles in the experimental alloy after two-step homogenization (a), LR (b) and RSR (c)

3.3. Hardness

Each sample was subjected to five measurements and the result was taken as an average value. The standard deviation was no more than 1% for each sample. The load was 50 N (5 kg). According to measured hardness (**Figure 7**), the experimental alloy was subjected to cyclical structural changes during the rolling-ageing treatment. There is a high deformation strengthening after hot and cold longitudinal rolling: the HV5 value is

180 that is similar with value in T6 mode. Ageing according to T4 mode allowed obtaining the HV5 value 145. The hardness of as-rolled rods is much lower, because of processing at high temperature. But after ageing in T4 and T6 mode it is about 5 HV higher than the value in thin sheets. Apparently the high strengthening rate appeared due to combination of deformation hardening, precipitation hardening and reinforcing by Al_9FeNi particles.

Owing to the fact that modern studies of Al-Zn-Mg-Cu alloys assume a limitation of the iron content, the addition of Sc, and an increase in the duration of thermomechanical treatment [1, 10, 12, 15], nickalins are a potential candidate to replace them due to their high processability and economical alloying.

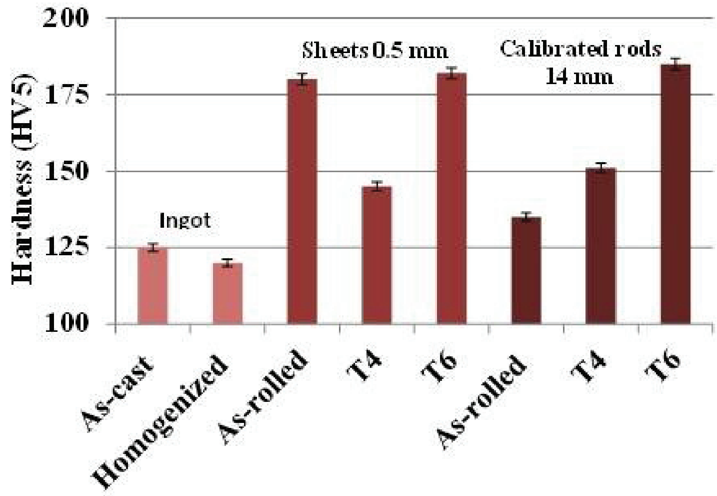


Figure 7 Hardness of the experimental alloy depending on a processing way

4. CONCLUSION

High-strength aluminum alloy based on Al-Zn-Mg-Ni-Fe system with a content of iron above 0.5% was passed through longitudinal hot and cold rolling with an reduction ratio of 40 and radial-shear rolling with an reduction ratio of 8.16. Iron is joined to Al_9FeNi particles, which do not inhibit the deformation.

The temperatures of thermomechanical treatment were proved by a calculation method with Thermo-Calc software. Two-step homogenization leads to precipitation of zinc and magnesium into T phase with a size of less than 1 μm in the centre of dendritic cells of (Al).

The size and morphology of eutectic particles in experimental alloy obtained as radial-shear rolled rods is a little larger and coarser than in thin sheet. However, a hardness measure showed significant strengthening after rolling and precipitation ageing in mode T6 and T4.

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