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Study of evolution of microstructure and mechanical properties in aluminum alloy 1570 with the addition of 0.5 % hafnium

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ABSTRACT

Introduction. Aluminum alloys are in high demand with the aerospace industry. From the viewpoint of various performance characteristic combinations, high-magnesium aluminum alloys with the addition of transition metals, such as *Zr* and *Sc*, are among the most future-oriented alloys. Alloy *1570* is one of the most popular in this group. Recent studies demonstrated the positive effect of 0.5 % hafnium addition on as-cast structure. **Study objective is** to study the effect of the addition of 0.5% hafnium on the structure and properties of aluminum alloy 1570 during thermomechanical treatment. **The study addresses** the effect of cold rolling, homogenization, and recrystallization annealing on mechanical properties and microstructure of the specimens from alloy 1570 and similar alloy with 0.5 wt. % hafnium addition. **Study methodology:** for the study, ingots were cast from alloy 1570 with and without additions of 0.5 wt. % of hafnium. The resulting ingots were homogenized for 4 h at 440 °C, followed first by hot rolling and then cold rolling. Cold-rolled specimens were annealed at temperatures 340 °C to 530 °C with a holding time of 3 hours. The homogenized, cold-rolled, and annealed specimens were examined using transmission and light microscopy. In addition, homogenized and cold-rolled specimens were subjected to uniaxial tensile tests to determine the mechanical properties of the studied alloy. **Results and discussion.** It is revealed that in an alloy containing hafnium, after homogenization annealing, there is a slight decrease in the average particle size and an increase in its total proportion in comparison with alloy *1570*. In general, 0.5 % hafnium addition does not significantly affect the mechanical properties. The number of nanoparticles in both alloys increases, as does the yield strength compared to the as-cast state. When heated, both alloys demonstrate an increase in plasticity and a decrease in strength characteristics. Studies of the annealing effect on the grain structure of the studied alloys showed that hafnium increases the tendency of alloy 1570 to recrystallize. However, additional research is required to determine the reasons for this phenomenon.

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Introduction

Aluminum alloys are widely used in various industries due to its high corrosion resistance, weldability, and low density [1–5]. In particular, in the aerospace industry, *Al-Mg* alloys, known in foreign literature as *5XXX* series of alloys, are one of the most common groups of aluminum alloys [6, 7]. These alloys are in high demand because the addition of magnesium enhances its mechanical properties through solid solution strengthening [8, 9]. The addition of scandium further improves its mechanical properties [10–12].

Scandium has low solubility in supersaturated aluminum solid solution, with a solubility of 0.35 % at equilibrium conditions and 655 °C [13]. However, if the cooling rate is high enough after casting, excess scandium can be dissolved in the aluminum matrix. Heating the alloy between 250 °C and 350°C causes the supersaturated solid solution to decompose and leads to the precipitation of Al_3Sc, which has a spherical morphology with a radius ranging from 2 to 20 nm [14–16]. These particles have an $L1_2$-type lattice and minimal mismatches with the aluminum matrix, which ensures its coherence. Such nanoparticles provide strengthening, which occurs due to particles intercepting by dislocations. The strengthening effect is based on the *Orowan* mechanism when the nanoparticle sizes range from 1.5 to 4 nm [17–19]. Moreover, scandium is a potent structure modifier, and its refinement capability is due to the L_{12} structure of primary intermetallic compound Al_3Sc formed in the liquid phase and the minimal mismatch between crystalline lattice and aluminum solid solution [13, 14]. It is worth noting that the modifying effect appears only when the scandium concentration reaches 0.6%, when primary Al_3Sc particles begin to form in the liquid [14].

However, as the temperature rises to 400 °C, scandium nanoparticles formed during solid solution decomposition start coagulating and increasing in size. When particle reaches a critical diameter of 30–40 nm, it loses its coherence, and the strengthening effect disappears [16]. This is a significant limit for the use of scandium alloy. For example, it reduces the temperature of the homogenization and hot deformation processes, which inevitably affects its efficiency and leads to higher energy costs [20].

Minor zirconium additions are used to improve the thermal stability of Al_3Sc-type nanoparticles [21]. Zirconium can form a shell around Al_3Sc particles as it is partially soluble in it. This shell inhibits the growth of Al_3Sc nanoparticles at elevated temperatures as zirconium has a lower diffusion coefficient than scandium [22]. Additionally, zirconium reduces scandium concentration, which is needed to form primary Al_3Sc intermetallic compounds in the liquid phase, contributing to the as-cast structure modification [23, 24].

One of the classic aluminum alloys with a high *Mg* content and *Sc* and *Zr* additives, successfully used in industry, is *1570* alloy [25, 26]. However, even with the presence of zirconium, Al_3Sc particles still do not have sufficient thermal stability to retain its size during high-temperature homogenization and further hot deformation [20]. One way to solve this problem is to add hafnium to the *1570* alloy. Hafnium has an even lower diffusion coefficient than zirconium [22] and partially dissolves in Al_3Sc particles [27], creating thermal stabilizing shells around it [22]. The joint addition of hafnium and zirconium is highly effective for thermal stabilization of Al_3Sc particles [28, 29].

The effect of combined hafnium and zirconium additions on the thermal stabilization of Al_3Sc particles has been mainly studied for lean aluminum alloys, but aluminum alloys with a high *Mg* content have several specific features. Firstly, magnesium slightly accelerates the decomposition kinetics of aluminum solid solution supersaturated with scandium [30]. Secondly, it stimulates an increase in the critical size of nanoparticles, after which its coherence is lost [13, 31]. Therefore, studying the hafnium effect on Al_3Sc particles in commercial aluminum alloys with a high *Mg* content is of utmost interest.

The effect of adding 0.5 % hafnium to the *1570* alloy was studied in the as-cast state. It was found that 0.5 % hafnium addition stimulates as-cast structure modification and leads to the complete termination of discontinuous decomposition of aluminum solution supersaturated with scandium [32, 33]. Discontinuous decomposition during ingot cooling down is a negative process when Al_3Sc needle-shaped precipitates are formed [34–36]. Such particles are usually semi-coherent to the aluminum matrix and do not contribute significantly to strengthening compared to equiaxed dispersed phases formed during heat treatment. After discontinuous decomposition, the aluminum supersaturated solid solution contains no scandium, which is needed for Al_3Sc nanoparticle formation during subsequent thermomechanical treatment [12, 34].

Considering the ability of 0.5 % hafnium to inhibit discontinuous decomposition, it is worth investigating its effect on the microstructure and mechanical properties of the *1570* alloy in the as-cast state and during subsequent thermomechanical treatment. Most *1570* alloy products are thin-walled and are produced from sheets that are supplied in annealed or cold-rolled states, depending on the required properties. Therefore, it makes sense to study the effect of 0.5 % hafnium on the microstructure and mechanical properties of the *1570* alloy after these types of treatment. To achieve the study objective, the following tasks need to be addressed: studying nanoparticle formation during *1570* alloy homogenization annealing, as its size and number will dictate alloy structure and properties during subsequent thermomechanical treatment stages. Additionally, the effect of 0.5 % hafnium on mechanical properties and grain structure needs to be studied for cold-rolled and annealed states.

Study methodology

The study focused on the *1570* aluminum alloy and its version with the addition of 0.5 % hafnium. These alloys were chosen based on its chemical composition, which is listed in Table 1. The production of these alloys was carried out in the lab induction furnace *UI-25P*, and the resulting ingots had dimensions of 20×40×400 mm. Molten metal was cast into a steel chill mold with water cooling at a melt temperature of 720–740 °C.

Table 1

Chemical composition of the studied alloys, %

Alloy	*Al*	*Si*	*Fe*	*Mn*	*Mg*	*Ti*	*Zr*	*Sc*	*Hf*
1570	base	0.17	0.26	0.4	6.1	0.03	0.07	0.25	–
1570 0.5*Hf*	base	0.15	0.32	0.42	6.36	0.01	0.04	0.2	0.52

Specimens preparation technology

Ingots casting

Ingots weighing 5 kg each were produced; with 3 ingots cast per each chemical composition. The charge stock used included *A85* grade aluminum, *MG90* grade magnesium, and alloying compounds such as *Al-Sc2*, *Al-Zr5*, *Al-Hf2*, and *Mn90Al10* tablets. The content of elements was determined according to various standards such as *GOST 25086*, *GOST 7727*, *GOST 3221*, *ASTM E 716*, and *ASTM E 1251* using an atomic emission spectrometer *ARL 3460*. The required concentration of stock materials, including hafnium, was calculated theoretically since there is a currently no *GOST* covering hafnium additive. After solidification, the ingots were removed from the mold and water chilled.

Homogenization annealing

Homogenization annealing at 440 °C for 4 hours dissolves non-equilibrium eutectic and improves chemical homogeneity in aluminum solid solution. Uniaxial tensile tests were conducted on homogenized specimens.

Rolling

The studied specimens underwent a rolling process. It is important to note that the commercial production of *1570* alloy sheets involves hot rolling above the recrystallization temperature followed by cold rolling. The laboratory rolling process used the same practice in order to produce sheet material. Initially, the specimens were hot rolled in a *Duo* reversing mill. The process reduced the thickness from 40 mm to 5 mm at a temperature of 440 °C and a roll rotation speed of 3 m/min. After every three passes, the ingots were heated back up to the initial rolling temperature. The overall reduction percentage was 88 %. It is worth mentioning that the hot rolling temperature was the same as the homogenization temperature. This is because a higher temperature may cause particle coagulation, at least in the alloy without hafnium content. Lower temperatures may result in a loss of plasticity [20]. Rolling a small ingot that has already been heated after homogenization, including its heating in the furnace, takes no more than 30 minutes. The microstructure and properties of the alloy were not examined after this operation since it was not a finishing operation, and short-term heating did not significantly affect Al_3Sc particles. After reaching a thickness of 5 mm, the tapes underwent cold rolling to 2 mm thick. The percentage of reduction during cold rolling was 95 %.

Cold rolled tape annealing

The cold-rolled tape from the tested alloys was annealed after rolling to examine how the hafnium content affects the recrystallization process. Furthermore, an additional series of annealing was conducted on the cold-rolled tape to investigate the mechanical properties of the alloy (Table 2).

Table 2

Annealing modes of cold-rolled tape

Annealing for recrystallization verification	Annealing for mechanical properties analysis
470 °C – 3 hours	340 °C – 3 hours
500 °C – 3 hours	440 °C – 3 hours
530 °C – 3 hours	470 °C – 3 hours
550 °C – 3 hours	530 °C – 3 hours

It is worth noting that the annealing temperature for high-magnesium alloy can be chosen from a wide range of temperatures (340 to 530 °C), depending on the required mechanical properties level and the desired combination of strength and plastic properties, as well as the contents of scandium, zirconium, and hafnium. This is precisely why these temperature values were chosen for the present study.

Methods for studying the microstructure and mechanical properties of specimens

Transmission electron microscopy

The fine structure of the specimens was analyzed using a *JEOL* analytical transmission electron microscope from Japan. The microscope had a 200 kV accelerating voltage and an *INCA* attachment for *EDX* analysis from Oxford Instruments in the UK. To achieve precision positioning of the foil specimen, a 2-axes rotation holder was used, allowing it to be tilted ±30° along each axis.

For particle transmission electron microscopy (*TEM*) analysis, standard procedures were followed. This included preparing two 500 µm thick foil specimens, further thinning it mechanically to 120 µm, and electrolytic thinning [29]. A total of five thin foil specimens were prepared for *TEM* analysis. For the examination of Al_3Sc particles, a specimen was placed in the zone axis, and an electron diffraction pattern was taken. During the examination, a weak superstructure reflection from the (011) α plane was detected. This method allowed the acquisition of dark-field images (*DF*), which helped count the visible particles. To determine the particle size and density, we used a *Digitizer* software module. We assessed the average particle size and its fraction by studying five different fields of view for each condition.

Optical microscopy

Optical microscopy was performed using an *Axiovert* microscope, and the average grain size after recrystallization was determined using the secant method.

Mechanical properties

The alloys underwent uniaxial tensile tests at room temperature using a *Zwick/Roell* universal testing machine in accordance with *ISO 6892-1*, *GOST 1497-84*, and *GOST 11150-84* standards. At least 5 tenfold round specimens with a 10 mm diameter were tested for each analyzed state. The data from the tests performed after the specific specimens production process stage is presented in Table 3 below.

Table 3

Technological chain of the specimens' research

Process chain stage	
Homogenized material	Cold rolled material
Test	
TEM Mechanical properties	Mechanical properties Optical microscopy

Results and discussion

The structure of the as-cast material after homogenization annealing for 4 hours at 440 °C is shown in figure 1.

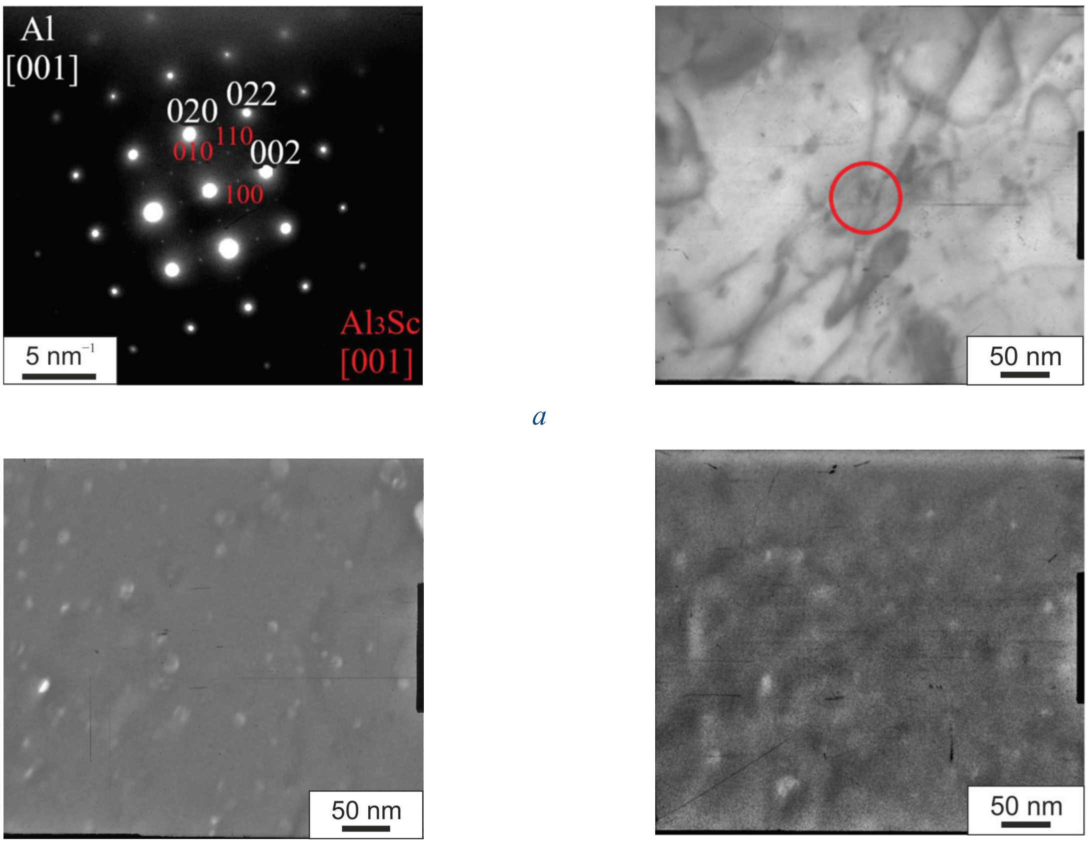
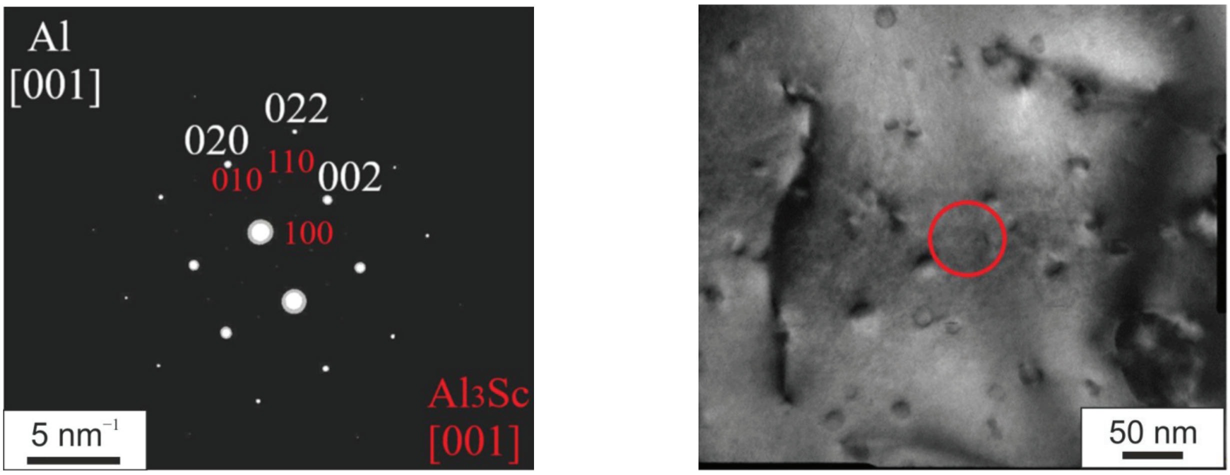


Fig. 1. Fine structure of a cast billet made of *1570* alloy after its homogenization annealing at 440 °C for 4 hours:
a) microdiffraction in the zone of axis [001] α; *b*) *DF*

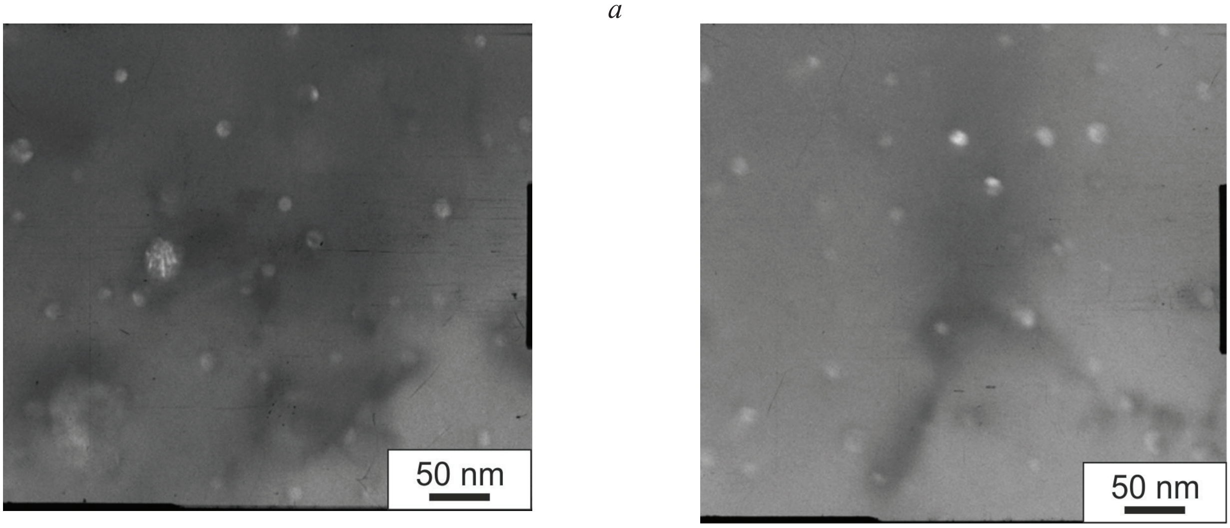
The average size of the particles in the specimen is 11.4 nm with a density of $f = 2.2 \times 10^{10}$ cm^{-2} as analyzed through transmission electron microscopy (*DF*) in fig. 1. The homogenization annealing process resulted in particles mostly ranging from 1.6 nm to 13.3 nm, indicating a prevalence of fine phases in the specimen. However, there are also some coarse particles exceeding 25 nm in size. In figure 1 a one can clearly observe superstructural reflections $L1_2$; this fact, according to [32], indicates the presence of Al_3Sc particles in aluminum alloys containing scandium.

The size range of particles in the *1570* 0.5*Hf* alloy is dominated by particles that are between 5.2 and 14.5 nm in size (as shown in figure 2, *b*). However, particles larger than 25 nm are also identified in *DF* images. The average particle size in this alloy is 10.5 nm, with a particle distribution density of 2.6×10^{10} cm^{-2}. It is observed that the non-uniformity of particle distribution inside the grain volume was reduced when compared to the *1570* alloy. Although superstructure reflections are present (as shown in figure 2, *a*), it is relatively weak compared to the original *1570* alloy. This means that a smaller amount of dispersed phases is formed.

It is important to note that previous studies showed that in the as-cast *1570* alloy, discontinuous decomposition resulted in the formation of some 7–10 nm Al_3Sc nanoparticles. However, discontinuous decomposition is not observed in the alloy with hafnium addition, and Al_3Sc particle formation does not occur. By comparing the study results for the as-cast state and after homogenization with the data presented in the paper [29], it can be concluded that heating at 440 °C for 4 hours increases the general number of nanopar-



a



b

Fig. 2. Fine structure of the cast billet made of alloy 1570 0.5*Hf* after its homogenization annealing at 440 °C for 4 hours:

a) microdiffraction in the zone of axis [001] α; *b*) *DF* ticles in the *1570* alloy. In an alloy with hafnium additives, all particles are formed during heat treatment. However, finally, after thermal treatment, Al_3Sc nanoparticles in both alloys generally have approximately equal size and number.

The *1570* aluminum alloy and its modified version with 0.5 % hafnium addition exhibit almost identical strength in a homogenized state (figure 3). After analyzing post-heat treatment strength properties and as-cast state data from the paper [33], it is evident that heating has minimal effect on ultimate strength (figure 3), showing only a slight increase. The influence of particles on the increase in yield stress is explained primarily by the degree of influence of dispersed phases. The greater the number of particles of the dispersed phase released, the more difficult it is for dislocations to move along planes and, as a consequence, when the movement of dislocations is difficult, the yield strength increases. On the other hand, several factors such as metal porosity, presence of coarse intermetallic compounds, etc., affect the ultimate strength. Therefore, the ultimate strength remains at the same level.

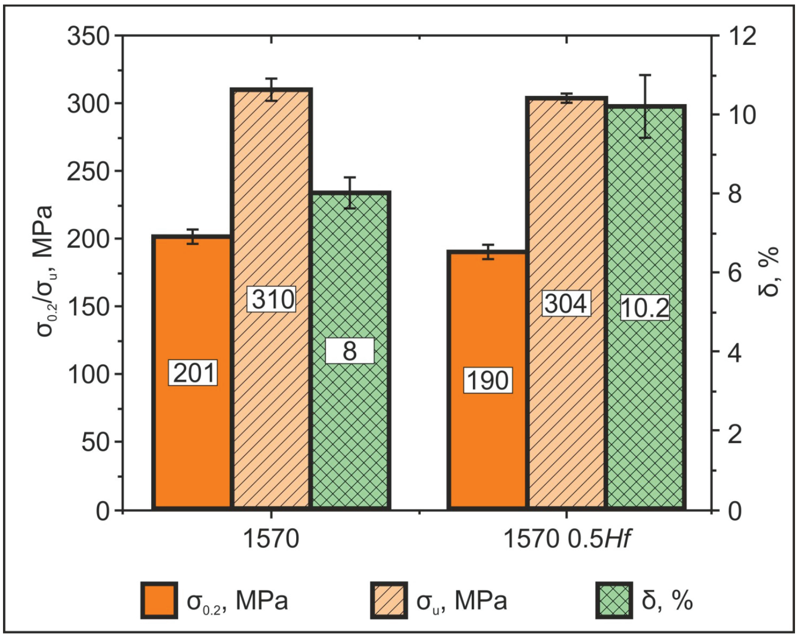


Fig. 3. Mechanical properties of studied as-cast alloys after homogenization annealing at 440°C – 4 hours

The homogenized state of both alloys exhibits similar strength parameters primarily associated with similar particle numbers and sizes.

Figure 4 shows the effects of cold rolling and annealing at different temperatures on the alloys. During cold rolling, the alloys form a fiber structure (as shown in figure 4), and black dots visible in the grain structure depict coarse intermetallic compounds. The size, chemical composition and morphology of these alloys have been already studied [32, 33]; therefore, its analysis by scanning microscopy methods was not carried out in the current paper.

At temperatures up to 440 °C, cold-rolled sheets maintain a non-recrystallized structure. This shows that the Al_3Sc particles efficiently inhibit the recrystallization process [37]. In the *1570* alloy, new grain nuclei only appear during annealing at 500 °C for 3 hours, and only with an increase in annealing temperature to 530 °C, a mixed structure with an approximate ratio of 1:1 can be observed. The alloy with 0.5 % hafnium addition is more susceptible to recrystallization. After annealing at 500 °C and soaking for 3 hours, a mixed structure with predominantly recrystallized grains is observed in the *1570* alloy with 0.5 wt% *Hf*. If the annealing temperature is further increased to 530 °C, a fully recrystallized structure is observed in the alloy containing 0.5 % hafnium. It is worth noting that in hafnium-containing alloys, the resultant microstructure has a smaller grain size due to recrystallization than the as-cast state. The causes of accelerated recrystallization in the *1570* alloy with hafnium addition require further investigation.

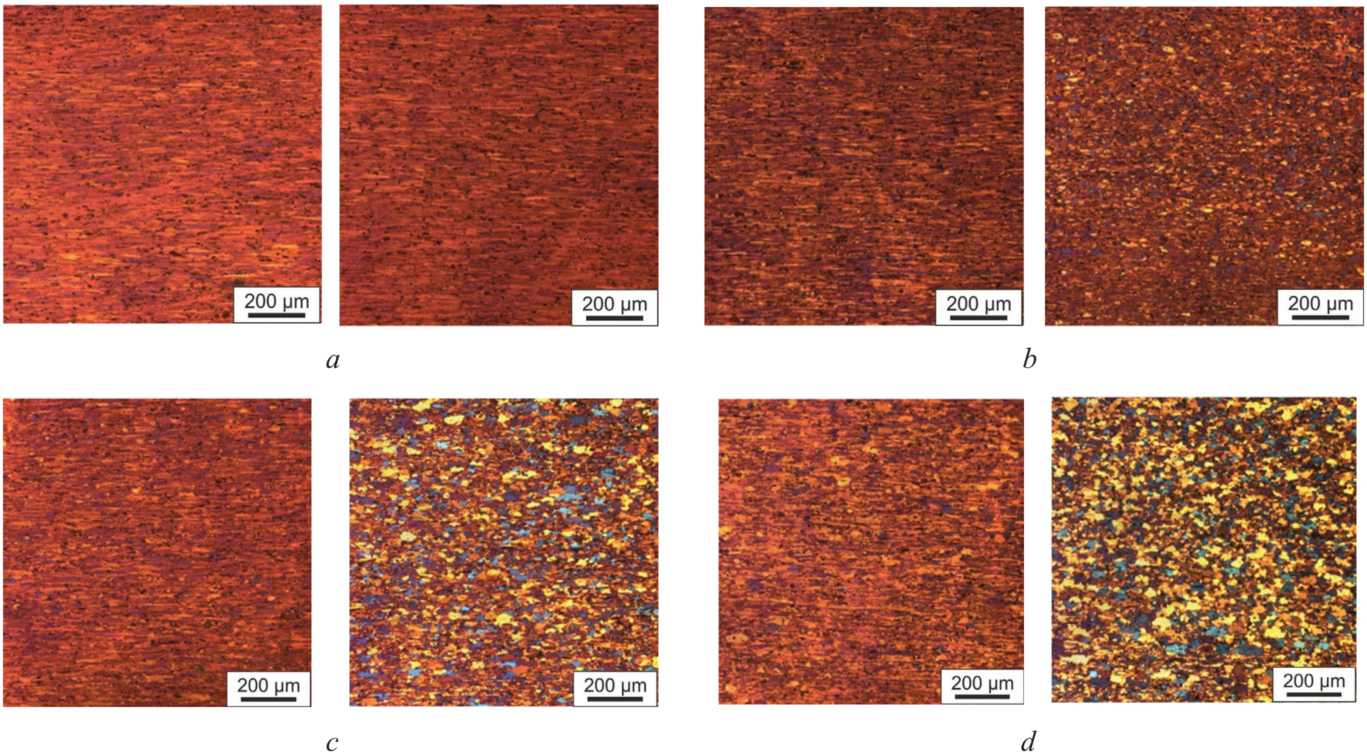


Fig. 4. Microstructure of sheets after cold rolling with 90 % deformation and subsequent annealing of alloys 1570 (left) and 1570-0.5Hf (right):

a) after cold rolling; *b*) after annealing at 470 °C for 3 hours; *c*) after annealing at 500 °C for 3 hours; *d*) after annealing at 530°C for 3 hours

It should also be emphasized that recrystallization in hafnium-containing alloys should not be interpreted as an entirely negative process. The occurrence of a super-fine as-cast structure and the possibility of modification during recrystallization can create conditions for obtaining grain with sufficient sizes for superplastic flow. This is facilitated by the fact that, according to [32], hafnium additives contribute to the modification of the cast structure in *1570* alloy. In the case of recrystallization after annealing at 530 °C for 3 hours, the grain size is 25 μm. Therefore, by increasing the overall degree of cold rolling in an alloy containing hafnium and introducing several intermediate recrystallization annealing, each of which will cause a refinement of the structure, it is possible to achieve an average grain size of up to 8 μm. This grain size is sufficient for superplastic flow in aluminum alloys with high magnesium content [38].

Figure 5 depicts the mechanical properties of the alloys after undergoing different treatment modes, as shown in figure 4. The yield strength values of the studied alloys are generally similar (figure 5, *a*). In both cases, the yield strength of the alloys dropped from 460 MPa in the cold-rolled state to around 150 MPa after being annealed at the highest temperature of 530 °C and soaked for 3 hours. This decrease is due to the recovery and dislocation annihilation that occur during the low-temperature thermal treatment. Consequently, the strength parameters after annealing at 530 °C and 3 hour soaking are almost the same as the parameters observed in the as-cast state [33].

As the temperature and soaking time increase, the ultimate strength of both alloys changes in a manner similar to the yield strength values (figure 5, *b*). The plasticity of the alloys increases, which is related to a decrease in the number of linear defects and hardening degree (figure 5, *c*). In general, the *1570-0.5Hf* alloy has lower plasticity than the original alloy. This is due to the formation of coarse primary Al_3Sc intermetallic compounds caused by the hafnium content in *1570-0.5Hf* [32]. Thus, the hafnium content does not have a significant effect on either the number of Al_3Sc nanoparticles or the increase in strength properties caused by it.

Separately, it is worth saying that the past recrystallization does not have a significant effect on the strength properties. This is explained by the fact that in *1570* alloy the grain remains deformed even during

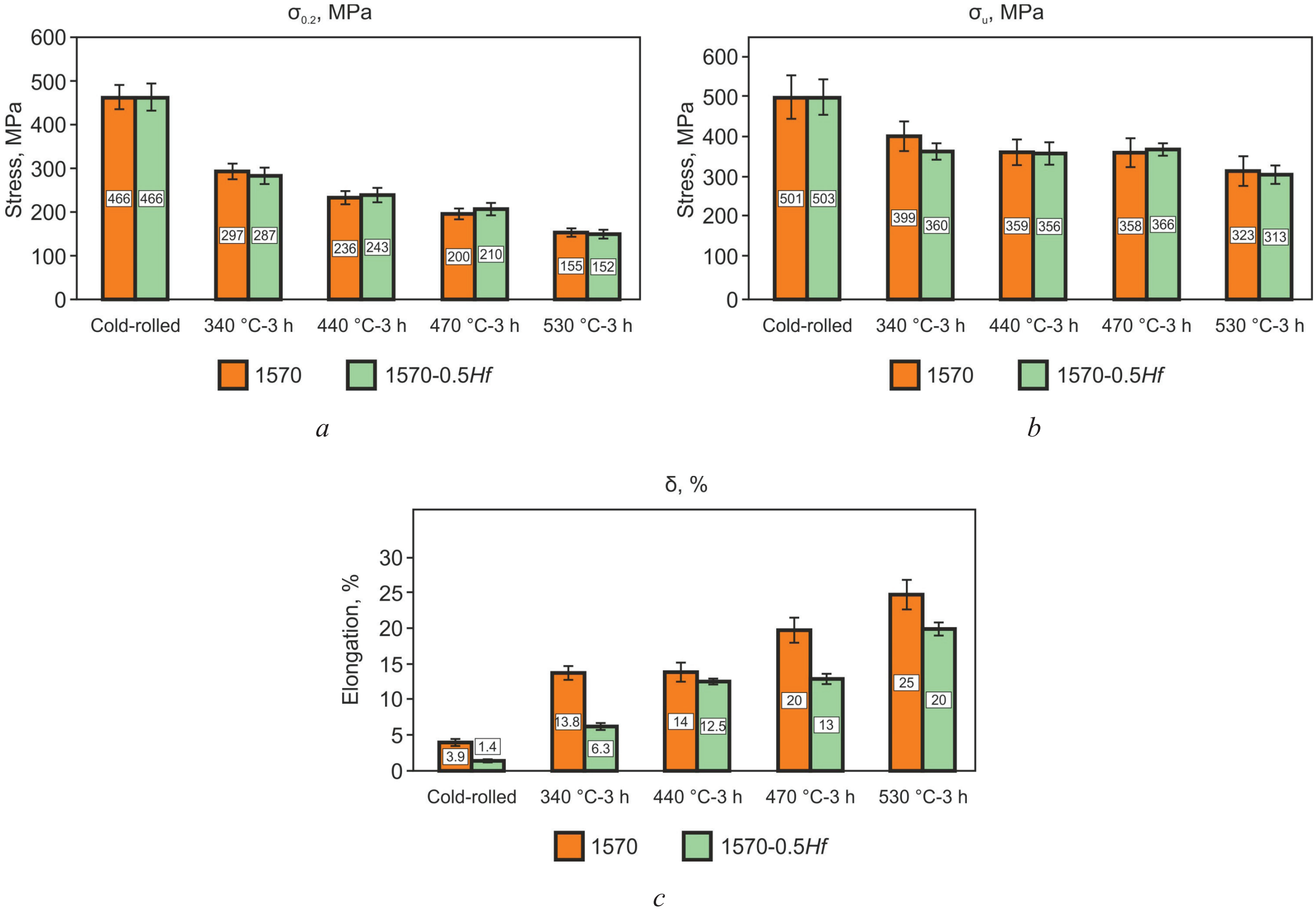


Fig. 5. Mechanical properties of 1570 and 1570–0.5Hf sheets:
a) yield strength, σ0.2, MPa; *b*) ultimate strength, σв, MPa; *c*) relative elongation, δ, %

annealing at a temperature of 530 °C (figure 4), which improves the mechanical properties. On the other hand, in an alloy with hafnium after recrystallization, a decrease in the average grain size is observed, which also has a beneficial effect on strength.

Conclusion

After conducting the study, the following conclusions can be made:

1. The *TEM* results after homogenization showed that annealing in a *1570-0.5Hf* alloy led to a reduction in the average size of nanoparticles and an increase in its overall portion compared to the *1570* alloy. However, this does not have a significant effect on the difference in mechanical properties in homogenized states.

2. The addition of 0.5 wt. % hafnium content increases the tendency of the *1570* alloy to recrystallize during high-temperature treatment. However, further studies are required to understand the causes of this effect. Regardless of whether there is recrystallization in the *1570-0.5Hf* alloy, both studied alloys demonstrate similar strength parameters associated with the average grain size decrease after recrystallization. Recrystallization can also have an additional modifying effect on the size of the cast structure.

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Conflicts of Interest

The authors declare no conflict of interest.