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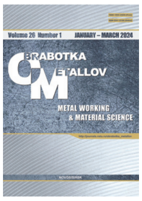

Study of the effect of hafnium and erbium content on the formation of microstructure in aluminium alloy 1590 cast into a copper chill mold

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ABSTRACT

Introduction. High-magnesium aluminum alloys are widely used in the automotive, building and aerospace industries due to its low specific gravity and high strength. The characteristics of such alloys can be improved by small additions of scandium and zirconium. However, scandium is very expensive, so in new generation alloys its amount is tended to be reduced. In the recently developed *1590* aluminum alloy, this was achieved by addition of erbium and hafnium. **The objective of the paper** is to study the effect of erbium and hafnium concentrations on the modification of the cast structure in *1590* aluminum alloy at high solidification rates. **Research Methods.** The paper investigates the microstructure, chemical composition and size of intermetallic compounds in specimens from ten alloy 1590 modifications with different hafnium and erbium contents cast into a copper chill mold with a solidification rate of 10 °C/sec. The grain structure was studied using an optical microscope. The chemical composition and size of the intermetallic phases were studied using a *Tescan Vega 3* scanning electron microscope. **Results and discussion.** It is established that as the amount of hafnium and erbium increases, the cast structure is modified. In general, grain refinement with the addition of hafnium and erbium can be explained by a higher degree of supercooling between the solid and liquid phases. At a hafnium content of 0.16 %, the dendritic structure begins to transform into an equiaxed grain structure. This transformation can be explained by the appearance of primary intermetallic compounds of the Al_3Sc type in the liquid phase. Such intermetallic compounds are identified at a concentration of erbium and hafnium equal to 0.16 %. Moreover, in all alloys eutectic intermetallic compounds are identified that contained manganese and iron and had no effect on the cast structure. Comparison with previously obtained results on the grain size of specimens cast into a steel mold shows that with higher solidification rate, the structure modification in *1590* alloy is getting less efficient. This is explained by an increase in the concentration of transition elements in the solid solution, primarily scandium, necessary for the formation of primary intermetallic particles.

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Introduction

Aluminum alloys, due to its low weight, high strength, and corrosion resistance, are widely used in modern industries. Magnesium is added to aluminum alloys to further enhance its properties. Aluminum-magnesium alloys are in high demand by aviation and aerospace technologies due to its high strength. Scandium is added to improve the strength of this group of alloys, and a scandium concentration

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of 0.5 % is ideal for refining the finished aluminum alloy structure. Scandium is an expensive metal, so attempts are continuously made to reduce its content by adding zirconium and hafnium. These elements stimulate the thermal stability of Al_3Sc particles, and erbium precipitates as Al_3Er nano-particles, increasing its total amount. The *1570* alloy is a conventional commercial high-magnesium aluminum alloy with combined scandium-zirconium additions. However, attempts are being made to further reduce the expensive scandium content in the alloys. For example, in the recently designed *1590* alloy, the scandium content varies in the range 0.06–0.16 %. Hafnium and erbium are added along with zirconium to reduce the scandium content in the alloy. The study aims to investigate the effect of erbium and hafnium concentrations on the modification of the cast structure in *1590* aluminum alloy under fast crystallization conditions. The study will evaluate the grain structure formation when casting *1590* alloy into a copper chill mold and assess the effect of hafnium and erbium content on its size and type. It will also study the effect of erbium and hafnium on the formation of intermetallic particles, emerging during crystallization of *1590* alloy cast into a copper chill mold, and the relationship between intermetallic particles and grain structure size and type.

Methods

For the purpose of the study, ten melts were cast into a copper chill mold with different chemical compositions as shown in Table 1. The melts contained *Er* and *Hf* in the range of 0.03–0.16 wt. % and 0.05–0.16 wt. %, respectively. These ranges are close to the minimal and maximal allowed concentrations of these elements in the *1590* alloy. The chemical compositions were previously analyzed in [21] as part of the study of 1590 alloy casting into a steel mold. Using the same compositions enables the comparison of the effects of crystallization rate on grain size in alloys with identical chemical compositions. Other elements in the melts corresponded to the chemical composition of the *1590* alloy, which was studied in [15, 16] from the perspective of heat treatment effect on microstructure and mechanical properties. Thus, this chemical composition allows the investigation of the effect of changes in *Er* and *Hf* concentrations on microstructure formation during casting and heat treatment. The charge stock used for experimental alloy design consisted of primary aluminum *A85* grade, primary magnesium *Mg90* grade, primary zinc *TS1*, and alloying compounds *Al-Mn10*, *Al-Zr5*, *Al-Sc2*, *Al-Er5*, and *Al-Hf2*. All materials were weighed using high-precision measurement devices before being charged into the furnace. The "*MECHELECTRON-M VR4900*" electronic scales, with a 5 g error, were used for materials weighing up to 15 kg, while the "*MIDLENA 251*" electronic scales were used for materials weighing up to 500 g, ensuring an accuracy level of plus/minus 0.1 g. The furnace was charged manually following the sequence below:

1. Primary aluminum was charged and melted first.
2. Once the temperature reached 730 °C, slag was removed from the molten metal surface.
3. The molten metal was then heated to a temperature range of 770–790 °C.
4. Portions of 300 g of *Al-Sc2*, *Al-Hf2*, *Al-Zr5*, *Al-Mn10* alloying compounds were sequentially introduced.
5. After each component was introduced, the molten metal was carefully stirred and soaked for 5 minutes.
6. After introducing all the calculated alloying components, the molten metal was cooled down to 740 °C.
7. Magnesium and zinc were then added to the molten metal.
8. The molten metal was stirred for 3 minutes using a titanium spoon.
9. The molten metal was reheated to 740°C.

After casting, the chemical composition of all aluminum alloys was comprehensively studied using atomic emission spectroscopy with an *ARL 3460* detector. The detector operates in the 0–10 keV energy range and has an energy resolution of 122 eV, which ensures accurate analysis. The analysis was carried out in accordance with the standards established by *GOST 25086*. Permissible concentration limits for key elements have been strictly established as follows:

– *Sc* and *Zn*: 0.009 %
– *Hf*, *Zr*, *Er*, *Si*, and *Fe*: 0.0053 %

– *Mn*: 0.018 %

– *Mg*: 0.15 %.

The chemical composition of all experimental melts was established experimentally and is presented in Table 1. The ingots were cast into a copper mold with a crystallization rate of 10 °C /s.

Table 1

Chemical composition of the studied alloys

Alloy	Element weight fraction, %									
	Al	*Si*	*Fe*	*Mn*	*Mg*	*Zn*	*Zr*	*Sc*	*Er*	*Hf*
1590 basic	base	0.03	0.06	0.42	5.54	0.19	0.1	0.14	–	–
*1590 Er*0.03-*Hf*0.05	base	0.04	0.07	0.41	5.54	0.21	0.1	0.14	0.03	0.05
*1590 Er*0.03-*Hf*0.1	base	0.04	0.07	0.41	5.58	0.2	0.1	0.14	0.03	0.1
*1590 Er*0.03-*Hf*0.16	base	0.05	0.08	0.41	5.58	0.2	0.1	0.14	0.03	0.16
*1590 Er*0.1-*Hf*0.05	base	0.04	0.07	0.41	5.57	0.21	0.1	0.14	0.1	0.05
*1590 Er*0.1-*Hf*0.1	base	0.05	0.08	0.41	5.53	0.21	0.1	0.14	0.1	0.1
*1590 Er*0.1-*Hf*0.16	base	0.05	0.08	0.41	5.57	0.19	0.1	0.14	0.1	0.16
*1590 Er*0.16-*Hf*0.05	base	0.04	0.07	0.41	5.55	0.21	0.1	0.14	0.16	0.05
*1590 Er*0.16-*Hf*0.1	base	0.05	0.08	0.42	5.56	0.2	0.1	0.14	0.16	0.1
*1590 Er*0.16-*Hf*0.16	base	0.05	0.09	0.41	5.58	0.2	0.1	0.14	0.16	0.16

The grain structure of the specimens was analyzed using a complex optical microscope *Carl Zeiss Axiovert-40 MAT*. The average size of the grains was determined for each specimen using the secant method, which is described in *GOST 21073.2*. Furthermore, a *Tescan Vega 3* scanning electron microscope was used to study the dimensions of intermetallic compounds in its cast state. The exact chemical composition of the structural elements in aluminum alloys 1590 *Er*0.03-*Hf*0.05, 1590 *Er*0.03-*Hf*0.16, 1590 *Er*0.16-*Hf*0.05, and 1590 *Er*0.16-*Hf*0.16 were determined using energy-dispersive X-ray spectroscopy (*EDS*). The analysis was carried out using a *Max 80T* X-detector that operates in the energy range of 0–10 keV and has an energy resolution of 122 eV. The specimen preparation process involved several steps, including mechanical grinding, precision polishing, and electropolishing. Electropolishing was performed under controlled conditions, maintaining a temperature range of 85–110 °C and applying a voltage of 10–30 V. The electrolyte solution used for electropolishing comprised H_3PO_4 (500 ml), H_2SO_4 (300 ml), CrO_3 (50 g), and H_2O (50 ml).

Results and discussion

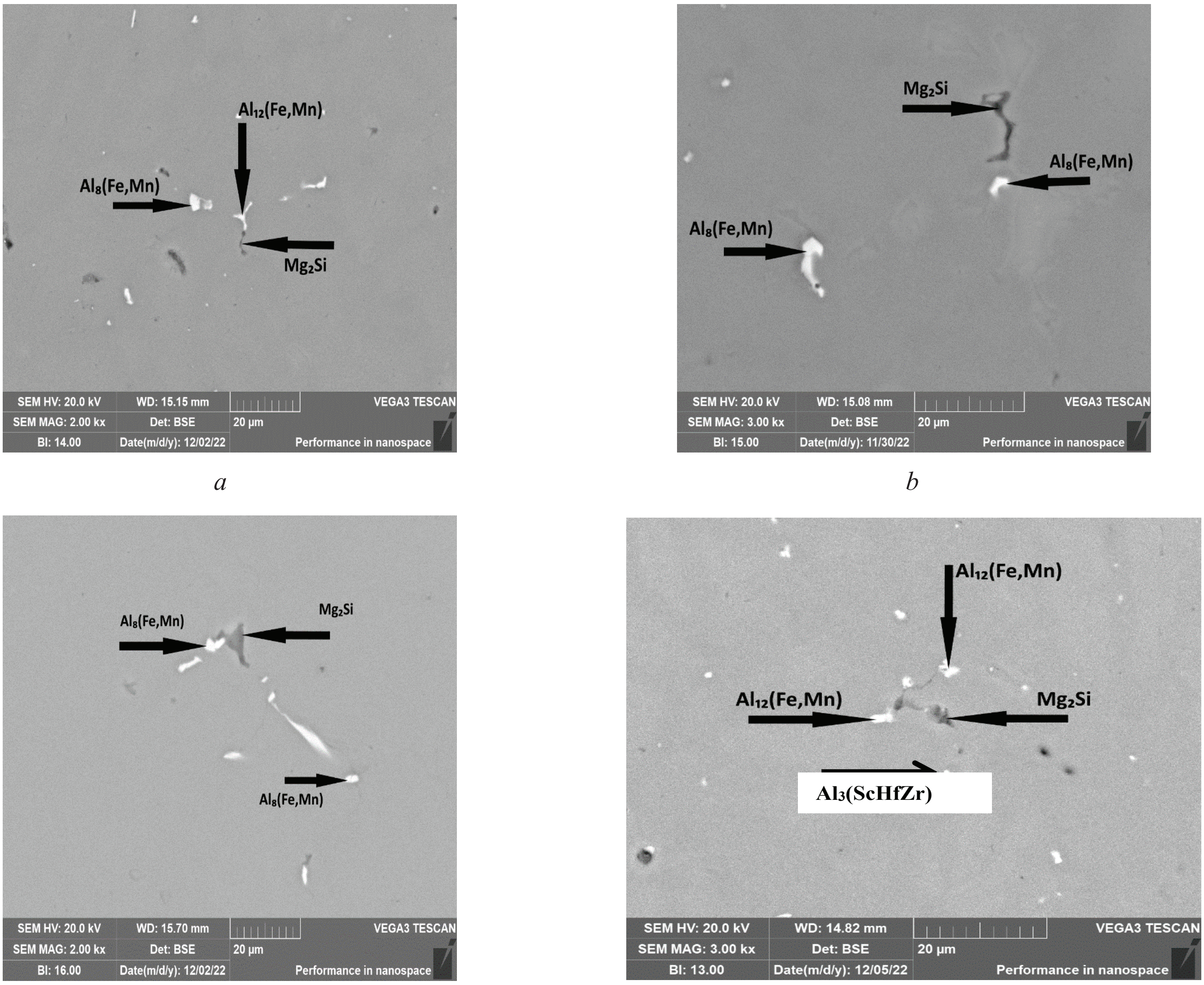
The analysis of the microstructure revealed that when cast into a copper chill mold, intermetallic compounds are formed by the process of eutectic reaction, and these compounds contain manganese and iron. The chemical composition of these compounds (as demonstrated in Table 2 and figures 1, 2) is similar to Al_8 (*FeMn*), Al_{12} (*FeMn*), and $MgSi_2$ [27]. It should be noted that to accurately determine the crystal structure of the phases, X-ray phase analysis is necessary. In this study, *EDS* analysis is conducted, which can determine the approximate chemical composition of intermetallic compounds and establish its correlation with the previously described phases that have similar compositions [28]. This study has successfully accomplished this.

The alloys *1590 Er*0.03-*Hf*0.05, *1590 Er*0.03-*Hf*0.16 and *1590 Er*0.16-*Hf*0.05, which were cast into a copper chill mold, contain manganese and iron and produce intermetallic compounds due to the eutectic

Table 2

Chemical composition of coarse intermetallic particles

	Mg	Al	Sc	Hf	Zr	Mn	Si	Zn	*Fe*	*Ti*	*Er*
$Al_8(FeMn)$	4.48	74.32	0.12	0.61	0.2	8.11	0.1	0.37	10.54	0	0.06
$Al_{12}(FeMn)$	7.06	83.93	0.18	0.3	0.21	3.25	0.58	0.32	3.95	0.02	0.15
$MgSi_2$	23.39	60.95	0.08	0	0.1	0.13	14.88	0.42	0	0.06	0
Al_3ScHf	6.36	72.82	5.78	6.86	6.13	0.47	0.39	0.17	0.02	0.08	0.19

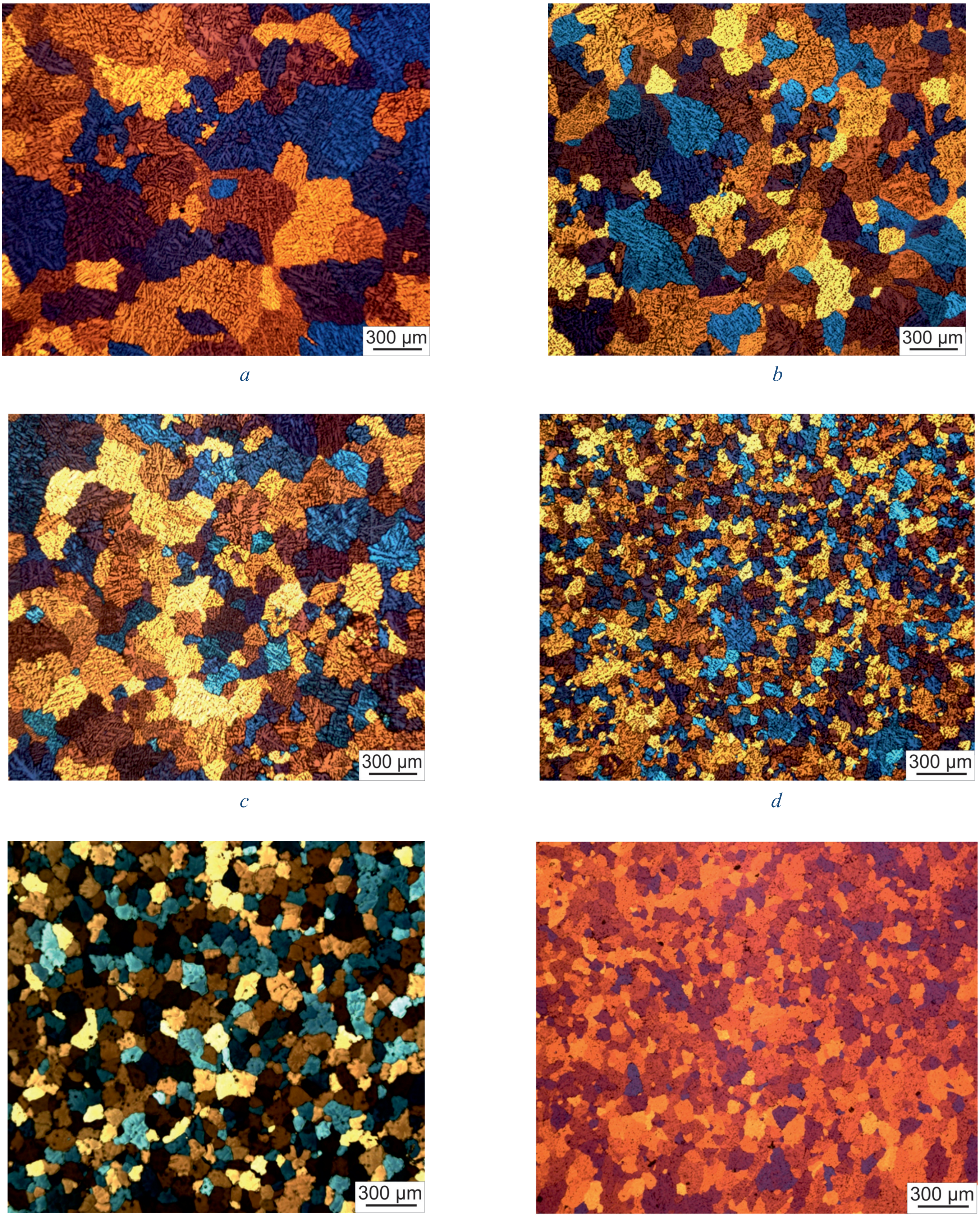


a *b*

c *d*

Fig. 1. Chemical composition of large intermetallic particles in specimens cast into a copper chill mold:
a) *1590 Er*0.03-*Hf*0.05; *b*) *1590 Er*0.03-*Hf*0.16; *c*) *1590 Er*0.16-*Hf*0.05; *d*) *1590 Er*0.16-*Hf*0.16

reaction. These compounds cannot modify the structure. However, particles of the Al_3Sc-, Al_3Hf-, and Al_3Zr-type, modifying the cast structure, were not found in these alloys. The alloy *1590 Er*0.16-*Hf*0.16 with a maximum content of hafnium and erbium at 0.16 % contains Al_3Sc-type intermetallic compounds. The presence of zirconium and hafnium is explained by the fact that it can dissolve in the Al_3Sc phase up to 35 % and 36 %, respectively. These compounds are primary intermetallic ones, i.e. are formed directly in



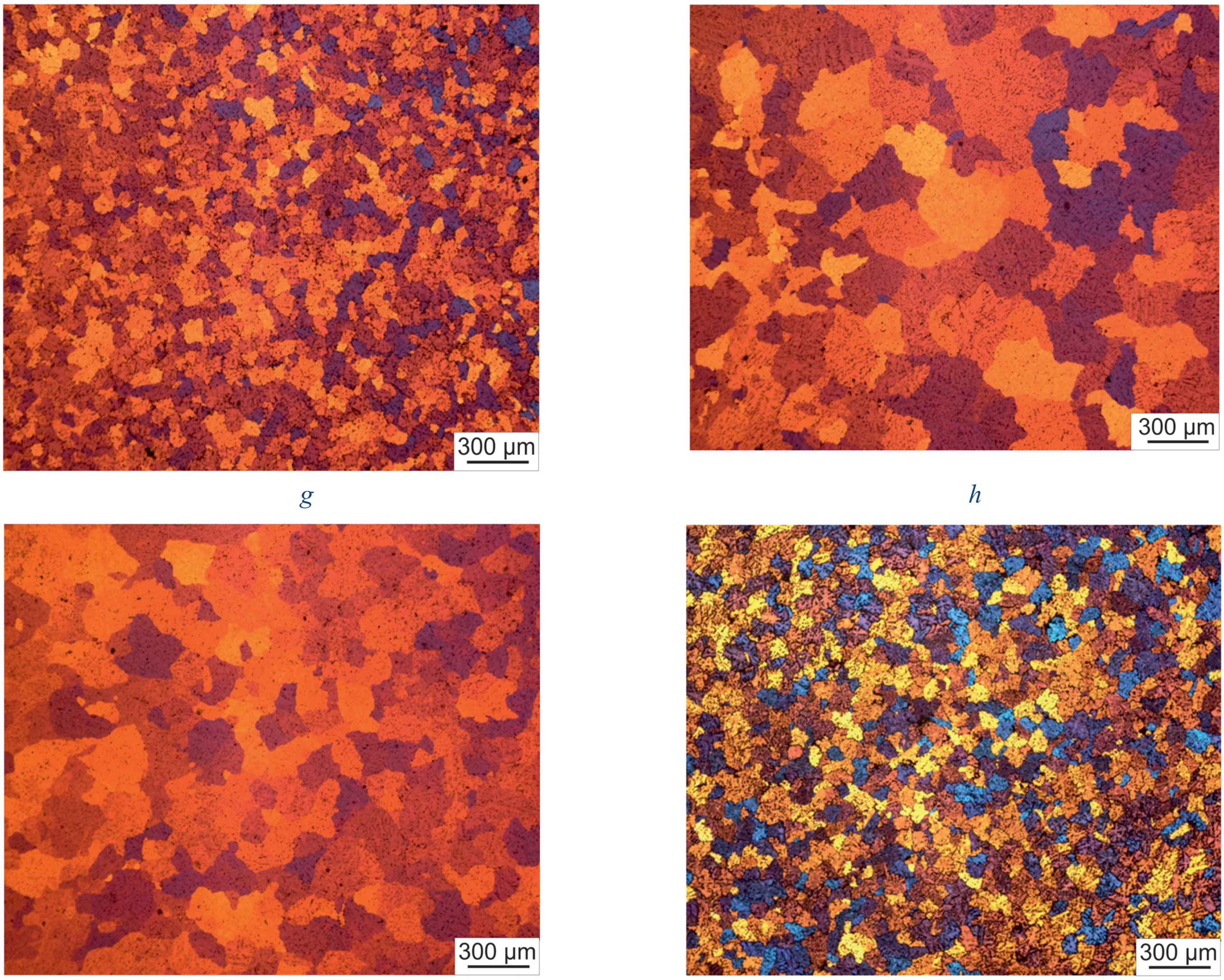
a *b*

c *d*

e *f*

Fig. 2. Microstructure of the studied alloy specimens:

a) *1590*; *b*) *1590 Er*0.03-*Hf*0.05; *c*) *1590 Er*0.03-*Hf*0.1; *d*) *1590 Er*0.03-*Hf*0.16; *e*) *1590 Er*0.1-*Hf*0.05; *f*) *1590 Er*0.1-*Hf*0.1; *g*) *1590 Er*0.1-*Hf*0.16; *h*) *1590 Er*0.16-*Hf*0.05; *i*) *1590 Er*0.16-*Hf*0.1; *j*) *1590 Er*0.16-*Hf*0.16 (see next page)



g *h*

i *j*

Fig. 2. The End

the liquid phase before solid solution crystallization. It's worth noting that some intermetallic compounds contain titanium, which is a result of using a titanium spoon during molten metal handling.

The microstructure of specimens cast in a copper chill mold is illustrated in figure 2, while figure 3 shows the variation in grain size depending on the concentration of the element. It is worth noting that for comparison, figure 3 contains the results of the dependence of grain sizes on the concentration of chemical elements during casting into a steel mold, taken from [21].

The basic alloy, containing no erbium and hafnium, has a dendritic structure with an average grain size of 372 μm. It is worth noting that some grain sizes vary from 600 to 800 μm, while others stay within the range of 100–200 μm, as illustrated in figure 2, *a*. With the addition of 0.03 % *Er* and 0.05 % *Hf* to the basic alloy, the average grain size decreases to 181 μm. The number of grains with sizes of 600–800 μm decreases, while the number of grains with sizes of 100–200 μm increases, as shown in figure 2 b. With 0.03 % *Er* and 0.1 % *Hf*, the average grain size continues to decrease and reaches 175 μm. Most grains have sizes of 300–400 μm or 100 μm. At the same time, the first 50 μm equiaxed grains appear, as seen in figure 2, *c*. With 0.03 % *Er* and 0.16 % *Hf*, the average grain size decreases to 86 μm, and almost all grains become equiaxed, as shown in figure 2, *d*. At 0.1 % *Er* and 0.05 % *Hf*, the average grain size reaches 113 μm, and two types of grains are observed: sufficiently coarse 300–400 μm and finer 100–200 μm grains, maintaining overall dendritic structure (figure 2, *e*). In the alloy containing 0.1 % *Er* and 0.1 % *Hf*, the average grain size is 105 μm, and the overall pattern doesn't differ much from the previous case

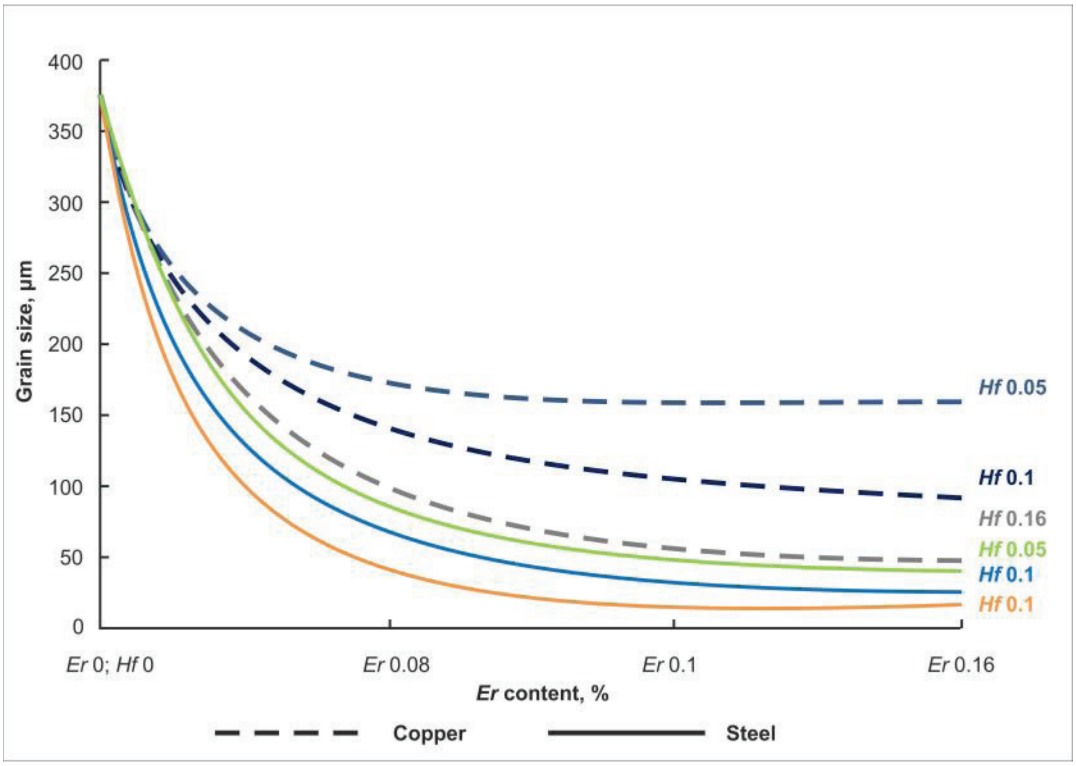


Fig. 3. Dependence of grain size on the amount of alloying components (on specimens cast into a steel and copper chill molds)

(figure 2, *f*). With the addition of 0.1 % *Er* and 0.16 % *Hf*, the grain sharply refines to 69 µm; most of the observed grains become equiaxed (although individual dendrites remain) (figure 2, *g*). With 0.16 % *Er* and 0.05 % *Hf*, the average grain size is 172 µm (figure 2, *h*). In general, in this case, the grain structure pattern corresponds to the pattern observed in *1590 Er*0.03-*Hf*0.05 alloys. With 0.16 % *Er* and 0.1 % *Hf*, the grain size is 168 µm (figure 2, *i*), and the grain structure resembles the structure observed in *1590 Er*0.03-*Hf*0.05 and *1590 Er*0.16-*Hf*0.05 alloys. The alloy *1590 Er*0.16-*Hf*0.16 demonstrates a notable average grain size drop to 64 µm, and the structure acquires the equiaxed shape, as shown in figure 2, *j*.

The analysis of the results leads to the conclusion that hafnium primarily presents the primary grain modifier. For instance, when alloy contains 0.05 %, 0.1 %, and 0.16 % *Hf,* increasing the *Er* content from 0.03 % to 0.16 % reduces the grain size by only 191, 76, and 36 µm, respectively. At the same time, *Hf* content growth from 0.05 % to 0.16 % enables decreasing the average grain size from 181 to 64 µm. However, most importantly, hafnium transforms the grain structure into an equiaxed type. This is the effect of Al_3Sc intermetallic compounds, containing both zirconium and hafnium, which are capable of modifying the as-cast structure (fig. 1d). The following factors explain its capability to refine grain: firstly, unlike other detected intermetallic compounds, it is formed in the liquid phase prior to aluminum solid solution crystallization; secondly, it has crystalline lattice parameters close to aluminum matrix parameters. This ultimately gives it the opportunity to act as nuclei of new grains consisting of an aluminum solid solution.

Note that such intermetallic compounds were detected only in alloy *1590 Er*0.16-*Hf*0.16, due to its reasonably small size (about 1 µm). Therefore, it is rather difficult to detect and identify it using *SEM*. Thus, only indirect evidence of its presence is used, i.e., dendritic structure becomes equiaxed. It should also be noted that grain refinement without dendritic structure conversion to equiaxed structure occurs as hafnium concentration increases from 0.05 to 0.1 % and erbium concentration increases from 0.03 to 0.16 %. This can be explained by the fact that an increase in the concentration of transition elements, especially hafnium, can also promote grain refinement due to increased supercooling between the liquid and solid phase nuclei.

Figure 3 demonstrates that when casting into a steel chill mold, the grain size is half as large as when casting into a copper chill mold. This is explained by the fact that with an increase in the crystallization rate, transition metals do not have time to precipitate in the form of primary intermetallic compounds and remain in a supersaturated solid solution, which is confirmed in [31]. It shows that Al_3Zr-type primary intermetallic compounds are not formed at a higher crystallization rate of aluminum alloy with zirconium addition. At the same time, zirconium itself remains in a supersaturated solid solution. In our case, the same effect is identified in the alloys containing hafnium and erbium, except for *1590 Er*0.16-*Hf*0.16, where close to $Al_3(Zr,Sc,Hf)$ primary intermetallic compounds [21].

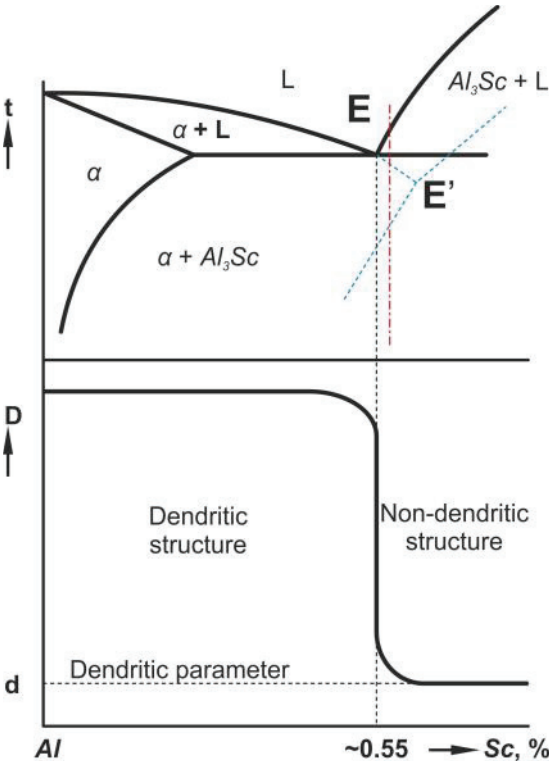


Fig. 4. Aluminum-scandium phase diagram [32]

In order to understand the phenomenon of transition elements migrating to the supersaturated solid solution at high casting rates, let us take the example of the aluminum-scandium phase diagram illustrated in figure 4. The figure shows the eutectic interaction in the aluminum-rich zone, which is identified by point ***E***. At a temperature of 655 °C and a scandium content of 0.55 wt % scandium, the equilibrium state $Zh \leftrightarrow ((Al) + Al_3Sc)$ is achieved.

When the cooling rate of the aluminum-based alloy increases to 10 °C/s, a marked shift in the eutectic interaction temperature from equilibrium conditions becomes apparent. The *Sc* content increases to 0.8 wt%, which enables the formation of primary intermetallic compounds, identified as *E’* in the diagram.

Thus, with an increased rate of crystallization in alloys where a dendritic structure is observed, the concentration of scandium, zirconium, hafnium and erbium required for the formation of primary intermetallic compounds increases, so the content of these transition elements for the appearance of such particles becomes insufficient.

Conclusions

An increase in the content of erbium and, mainly, hafnium helps to refine the grain structure. However, only when the content of hafnium reaches 0.16 %, the dendritic structure is replaced by an equiaxed one. This is because the refinement process begins due to supercooling between the nuclei of solid and liquid phases. When the hafnium content reaches 0.16 %, primary intermetallic compounds emerge in the liquid phase, thus facilitating refinement and causing a modification of the as-cast structure.

Intermetallic compounds, which have no effect on the modification of the as-cast structure, are identified in all alloys close to $Al_8(Fe,Mn)$, $Al_{12}(Fe,Mn)$, and $MgSi_2$ eutectic origin. Al_3Sc-type primary intermetallic compounds were found only in the *1590 Er*0.16-*Hf*0.16, and its presence in other alloys containing 0.16 % hafnium can be indicated only by indirect indicators, such as as-cast structure refinement. The absence of these intermetallic compound traces can be explained by its relatively small sizes in such alloys, making its identification and detection difficult using scanning microscopy.

An increase in the crystallization rate of the *1590* alloy leads to grain size growth at any content of erbium and hafnium. This is mainly due to the fact that an increase in the casting rate leads to an increase in the concentration of transition elements required for the formation of primary intermetallic compounds that act as grain modifiers in the liquid phase.

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Conflicts of Interest

The authors declare no conflict of interest.

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