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The influence of hafnium on high-magnesium alloys doped with transition metals during heat treatment

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Abstract: The purpose of the work is to study the influence of hafnium additives on the mechanical properties and thermal stability of particles at elevated temperature during heat treatment of aluminum alloys with a high magnesium content. Two modifications of 1570 alloy were chosen for the study: without hafnium content and with its addition of 0.5 % by weight. Both alloys were subjected to homogenizing annealing at a temperature of 440 °C with different exposure modes, which ranged from 2 to 100 h. Microhardness was studied for various heat treatment modes, and the fine microstructure was studied as well using transmission microscopy. As a result, it was possible to identify that during annealing at a short exposure time (2–8 h), the alloy with the hafnium addition has higher microhardness values exceeding those of 1570 alloy by an average of 20 HV units. This is associated with the fact that in 1570 alloy with hafnium additives, during heat treatment, the number of precipitated particles increases while their average size decreases compared to the base alloy. At the same time, in 1570 alloy without hafnium content, when annealed at a temperature of 440 °C, there is no increase in microhardness. This is caused by the fact that in 1570 alloy without hafnium content, when cooled after casting, discontinuous decomposition occurs, which resulted in the fact that most of the scandium precipitates from the supersaturated solid solution in the form of dispersoids. This phenomenon is not observed in the alloy with hafnium additives, which indicates its ability to stop discontinuous decomposition during cooling the ingot after casting.

Keywords: aluminum alloys; transition metals; scandium; hafnium; heat treatment; strengthening nanoparticles.

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INTRODUCTION

Aluminum alloys are currently among the most desirable materials in many industries. One of the most popular additives to aluminum alloys is magnesium, alloying with which leads to a significant increase in strength properties due to solid solution strengthening. Another common additive, that increases the mechanical properties of aluminum alloys is scandium [1]. Its use leads to a significant refinement of the grain structure during casting [2–4]. Moreover, it promotes an increase in strength, due to the formation of coherent strengthening Al_3Sc nanoparticles with the $L1_2$ structure [5–7]. Considering the above, it is not surprising that magnesium-scandium joint doping is very common in modern industry.

At the same time, the use of scandium as an alloying element has certain disadvantages: firstly, it is very expensive, and secondly, due to the high rate of its diffusion in aluminum, Al_3Sc nanoparticles have low thermal stability and quickly coagulate when heated [8; 9]. To increase their thermal stability, zirconium is added to the alloys, which creates a shell around Al_3Sc preventing the transition of $L1_2$ to $D0_{23}$ and the decomposition of the supersaturated scandium solution [10; 11]. Moreover, zirconium reduces the amount of scandium required for effective modification of the cast structure [7; 12].

It is the principle of joint scandium-zirconium doping that was used to develop a number of aluminum alloys, with a high magnesium content. One of them is 1570 alloy, which is very popular in modern industry. Hafnium additives could further increase the effectiveness of strengthening particles. This element is also an effective grain refiner [13]. Moreover, like zirconium, it creates a shell around Al_3Sc particles preventing further diffusion of scandium, i. e., thermally stabilizes them [10; 14].

Recent studies [15; 16] showed that hafnium additions to 1570 alloy significantly slow down the decomposition of the supersaturated solid solution. Thus, in [16] it was found that when adding 0.5 % of hafnium to 1570 alloy, the discontinuous decomposition of the supersaturated solid solution in it completely stops. The study [15] showed that adding hafnium to 1570 alloy reduces the number of nanoparticles precipitated from it during annealing at a temperature of 370 °C and an exposure time of 4 h. This also indicates a slowdown in the decomposition of the supersaturated solid solution. It is worth noting that inhibiting the decomposition of a supersaturated solid solution at a temperature of 370 °C will not provide advantages, since due to a decrease in the number of nanoparticles, the strength characteristics of the alloy will also decrease. However, slowing down the rate of decomposition of a supersaturated solid solution of scandium in aluminum can be useful at higher temperatures, when the particles in the 1570 alloy begin to coagulate and coalesce, thereby losing their strengthening effect. At the same time, hafnium additives allow thermally stabilizing nanoparticles, thereby increasing their strength properties. Note, that increasing the thermal stability of particles is very important, since it will allow increasing the hot deformation temperature. This, in turn, will increase the ductility of the material and improve the energy efficiency of hot rolling [17].

The purpose of the work is to study the influence of hafnium additives on the mechanical properties and thermal stability of Al_3Sc particles at elevated temperature of heat treatment of high-magnesium aluminum alloys.

METHODS

To study the influence of hafnium on the formation of microstructure and mechanical properties during high-temperature annealing of rolled samples from aluminum alloys with a high magnesium content, 1570 and 1570 (+0.5 wt. % Hf) alloys were cast. To cast ingots of the studied alloys, a medium-frequency induction furnace was used; ingots with dimensions of 20×40×400 mm and a mass of 5 kg were cast into a steel mold, followed by cooling in water.

The following materials were used as charging materials for the alloy: aluminum of A85 grade, magnesium of MG90 grade, alloying composition of $Al–Sc_2$, $Al–Zr_5$, $Al–Hf_2$ grades, and alloying tablets of $Mn_{90}Al_{10}$ grade. First, aluminum was loaded and melted. After the aluminum melted and the temperature reached 730 °C, slag was removed from the melt surface. Next, the melt was heated to a temperature of 770–790 °C and $AlSc_2$, $AlZr_5$, $Al–Hf_2$ alloying compositions were added in portions weighing no more than 300 g, followed by stirring and holding the melt for 5 min. After introducing the above-mentioned alloying compositions, the melt was cooled to a temperature of 750 °C, after which new alloying components (Mg, Mn) were added. Next, the melt was stirred for 3 min, followed by heating the melt to a temperature of 740 °C and taking a sample for express analysis of the melt chemical composition. The chemical composition of the alloys (Table 1) was determined by the spectral method on an ARL 3460 atomic emission spectrometer (GOST 25086, GOST 7727, GOST 3221, ASTM E 716, ASTM E 1251). The Hf content was determined by calculation due to the absence of standard samples. Before pouring the molten metal into the mold, it was refined with carnallite flux introduced at the rate of 5 g per 1 kg of charging material. After this, slag was removed from the surface of the molten metal, and the metal was poured into a steel casting mold at a uniform pouring time of 20–30 s at a melt temperature of 730–750 °C. After solidification, the ingot was removed from the chill mold and cooled in water.

The ingots were annealed in an electric muffle furnace at a temperature of 440 °C and held for 2, 4, 8, 16, 24, 48, 72, and 100 h, followed by quenching in water to fix the supersaturated solid solution.

The microhardness of the studied alloy was measured using a Wolpert 402MVD automatic microhardness tester in accordance with GOST 9450–76 at a load of 0.2 N and a holding time of 10 s. Before testing began, one of the surfaces of the plane-parallel sample was ground and polished.

Using transmission microscopy on a JEM-2100 microscope (JEOL, Japan) equipped with an INCA energy dispersive analysis attachment (Oxford Instruments, UK), samples for both alloys considered in the work were studied after 4 h of holding at temperatures of 370 and 440 °C. Sample preparation for transmission electron microscopy was carried out in several stages. At the first stage, using a Sodick electroerosion machine (Sodick Co., Ltd, Japan),

Table 1. *Chemical composition of the studied alloys*
Таблица 1. *Химический состав исследуемых сплавов*

Alloy	Al	Si	Fe	Mn	Mg	Ti	Zr	Sc	Hf
1570	Base	0.13	0.21	0.44	6.25	0.02	0.06	0.25	–
1570–0.5Hf	Base	0.12	0.22	0.45	6.29	0.04	0.06	0.25	0.5

two blanks were cut for foils with a thickness of 500 μm. The indicated thickness is determined by possible deformation and bending of the foils during cutting, due to the possible presence of internal stresses in the samples. Next, the resulting blanks were mechanically thinned to a thickness of ~120 μm using Grid 2000 abrasive wheels (Struers, Denmark). Using a special punch, disks with a diameter of 3 mm were extruded from the resulting blanks and placed in a TenuPol-5 electrolytic thinning installation (Struers, Denmark). Thinning was carried out at a temperature of −30 °C in an electrolyte of the following composition: 75 % of CH_3OH, 25 % of HNO_3. As a result, at least 5 foil samples for TEM were produced from each of the 6 states. The fine structure of the samples was studied on a JEM-2100 TEM transmission electron microscope (JEOL, Japan), with an accelerating voltage of 200 kV equipped with an INCA EDX-analysis attachment (Oxford Instruments, UK). The resulting foils were immediately placed in a bi-inclined TEM holder with the ability to incline by ±30° along two axes. Due to the small size of the particles (5–10 nm), the shooting was carried out at a magnification of ×200,000 and a long exposure time (about 1 min), which made it possible to reliably record even such small coherent particles. In the resulting images, the number of particles and their chemical composition were considered using an EDX-detector.

RESULTS

The changes in the microhardness of the studied alloys are given below. According to Fig. 1, the mechanical characteristics of 1570 alloy generally remain unchanged and are in the range of 82–89 HV units. This points to the fact that all strengthening $Al_3(Sc,Zr)$ particles, which have a coherent $L1_2$ structure, precipitate during discontinuous decomposition, and their formation does not occur at subsequent stages of heat treatment.

At the same time, the alloy doped with hafnium shows the greatest increase in microhardness in the range of 2–8 h. This is associated with the fact that in the indicated time intervals, the active decomposition of the supersaturated solid solution begins and the precipitation of strengthening particles of the Al_3Sc type begins. After 16 h of holding, the alloy with the hafnium addition exhibits a decrease in microhardness, which is likely associated with the onset of loss of coherence and coalescence of particles of the Al_3Sc type.

Using transmission microscopy after annealing at 440 °C for 4 h, the fine structure in both alloys was studied. This annealing mode was chosen because, according to microhardness data (Fig. 1), its greatest increase was observed in the range from 2 to 8 h. Therefore, it is under these heat treatment modes, that the precipitation of the largest amount of strengthening nanoparticles is expected.

Fig. 2 presents the results of transmission electron microscopy for 1570 alloy after annealing at 440 °C for 4 h.

Superstructure $L1_2$ reflections in 1570 alloy are visible quite clearly, which indicates the presence of Al_3Sc nanoparticles coherent with the aluminum matrix. Shown in Fig. 2 b data indicate the presence of large (about 1 μm) particles precipitated in the alloy structure. These particles are close in their chemical composition to $Al_6(Mn,Fe)$ (Fig. 3) and, like Al_3Sc, appear during the decomposition of a supersaturated solid solution, since this alloy contains Mn and Fe. One should note that iron in aluminum alloys is an unavoidable impurity.

Al_3Sc nanoparticles are also observed in 1570 alloy, when it is heated to a temperature of 440 °C. Fig. 2 b shows a predominance of particles with sizes in the range from 1.6 to 13.3 nm in 1570 alloy. This indicates a predominantly finely dispersed phase in this sample, however, bigger particles, larger than 25 nm, are also observed. In general, the average particle size is 11.4 nm, and their average density is $2.2\cdot10^{10}$ cm^{-2}. It should be noted that the particles in the grain volume are distributed very unevenly, which is observed in Fig. 2 c, 2 d. One can assume that this is a consequence of discontinuous decomposition in this alloy during cooling of the cast blank.

In the presented state (Fig. 4), superstructural reflections are not visible so clearly, but they are present, which is associated with a decrease in the coefficient of diffusion of scandium in aluminum when doping the alloys with hafnium. A rather large number of relatively coarse particles can be observed (Fig. 4 b). These particles are also close in chemical composition to $Al_6(Mn,Fe)$ (Fig. 3 and Table 2) and are explained by the presence of manganese and iron in the alloy.

In the 1570–0.5Hf alloy, particles with sizes ranging from 5.2 to 14.5 nm prevail (Fig. 4 b). At the same time, dark-field images also show the precipitation of particles larger than 25 nm. The average particle size in this alloy is 10.5 nm, and the distribution density is 2.6×10^{10} cm^{-2}. In this case, the uneven distribution of particles in the grain volume is somewhat reduced.

DISCUSSION

It should be noted that a comparison of the average particle size observed in 1570 alloy after casting in [18] and obtained in this study after heating at a temperature of 440 °C with a holding time of 4 h indicates that heat treatment practically does not change the number and size of particles. In both cases, their size is around 10 nm. This leads to the fact that the microhardness of this alloy does not change over time. The latter occurs because the major share of scandium precipitates during the continuous decomposition of a supersaturated solid solution when cooling the ingot during casting, as well as during the formation of primary intermetallides at the crystallization of this alloy [16]. Therefore, for the process of continuous decomposition of a supersaturated solid solution when heating a given alloy, there is no longer enough scandium and the number of particles does not change. One can assume that zirconium still quite actively blocks the growth of particles when annealed at 440 °C for 4 h, which does not contradict the data [19].

It is worth noting that the particles formed during decomposition can also be coherent and make a rather large contribution to strengthening [8]. In this case, their strengthening effect is confirmed by the fact that the microhardness after casting is significantly higher in 1570 alloy containing nanoparticles formed as a result of discontinuous decomposition, than in 1570 alloy with a 0.5 % hafnium content, in which they are absent in this state [10].

A sharp increase in microhardness during heat treatment of 1570 alloy with a 0.5 % hafnium content is explained

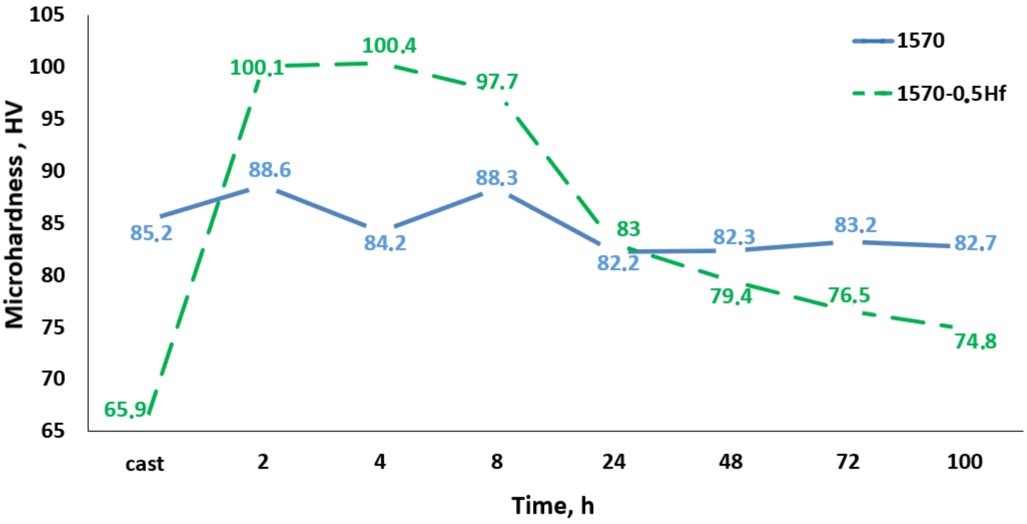
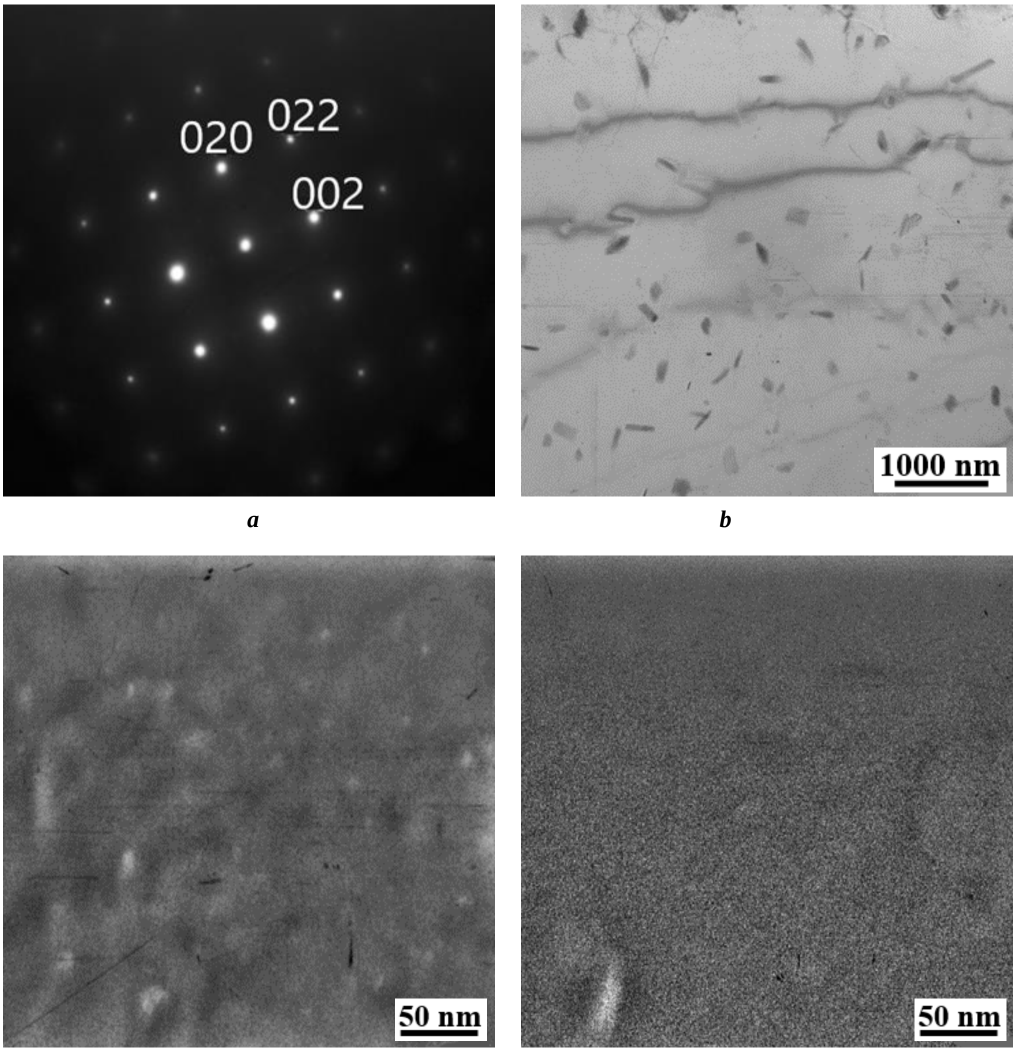


Fig. 1. *Change in microhardness during 440 °C annealing*
Рис. 1. *Изменение микротвердости при отжиге 440 °C*



a *b*

c *d*

Fig. 2. *Fine structure of 1570 alloy during 440 °C annealing with a duration of 4 h:*
a *– microdifraction in the zone axis [001]*$_\alpha$*;* ***b*** *– bright field, ×20,000;* ***c, d*** *– dark field, ×200,000*
Рис. 2. *Тонкая структура сплава 1570 при отжиге при 440 °C длительностью 4 ч:*
a *– микродифракция в оси зоны [001]*$_\alpha$*;* ***b*** *– светлое поле, ×20 000;* ***c, d*** *– темное поле ×200 000*

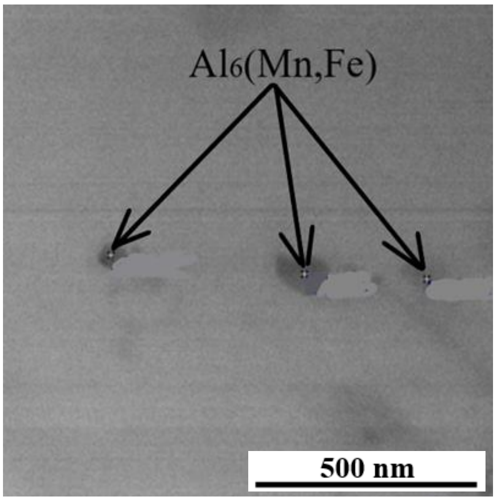
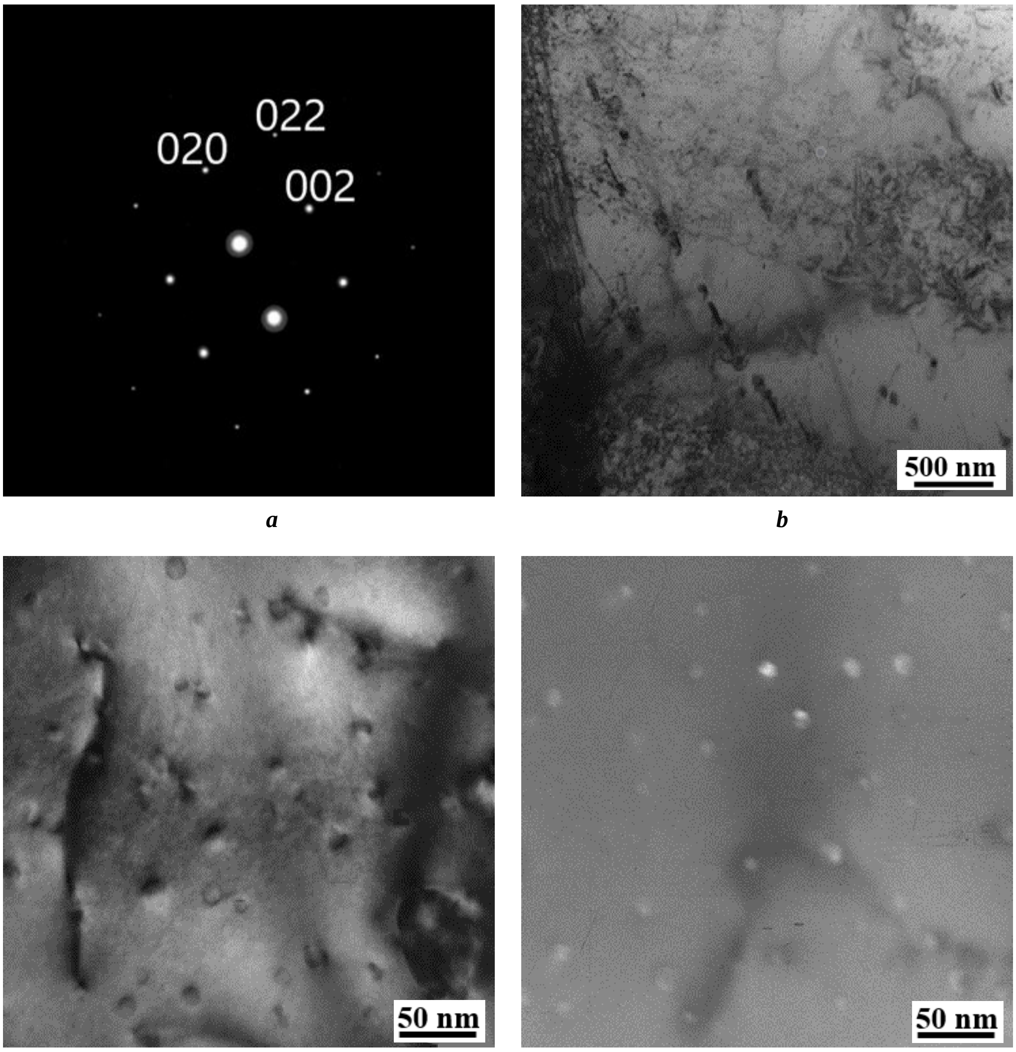


Fig. 3. *Areas of analysis of local chemical composition in a sample of 1570 alloy during 440 °C annealing with a duration of 4 h*
Рис. 3. *Участки анализа локального химического состава в образце сплава 1570 при отжиге 440 °C длительностью 4 ч*



a ***b***

c ***d***

Fig. 4. *Fine structure of 1570–0.5Hf alloy during 440 °C annealing with a duration of 4 h:*
a *– microdifraction in the zone axis* $[001]_a$*;* ***b*** *– bright field, ×20,000;* ***c, d*** *– dark field, ×200,000*
Рис. 4. *Тонкая структура сплава 1570–0,5Hf при отжиге при 440 °C длительностью 4 ч:*
a *– микродифракция в оси зоны* $[001]_a$*;* ***b*** *– светлое поле, ×20 000;* ***c, d*** *– темное поле, ×200 000*

Table 2. *Local chemical composition of the particles in the 1570 alloy sample during 440 °C annealing with a duration of 4 h, at. %*
Таблица 2. *Локальный химический состав частиц в образце сплава 1570 при отжиге 440 °С длительностью 4 ч, ат. %*

Chemical elements	Al	Mn	Fe
Studied spectra	77.84	10.6	11.4

by the fact that active processes of precipitation of Al_3Sc particles from a supersaturated solid solution begin in it. This occurs because, unlike 1570 alloy, it retains enough scandium for continuous decomposition. Note that there are several possible reasons for suppressing the discontinuous decomposition of a supersaturated solid solution with the help of hafnium [16]. Not dwelling on the hypotheses in detail, one can state that the absence of discontinuous decomposition in alloys when doped with hafnium allows activating the formation of Al_3Sc nanoparticles, which as a result leads to an increase in microhardness. The higher properties of the 1570–0.5Hf alloy are generally explained by a larger number of nanoparticles and their more even precipitation. A more finely dispersed distribution of nanoparticles, in principle, distinguishes a solid solution with continuous decomposition from a discontinuous one [8]. With further heating for several hours, the microhardness remains at the same level, which means that the nanoparticles retain their size and number. However, after holding for more than 8 hours, the microhardness begins to decrease, which indicates a coagulation process. Then its decrease slows down significantly, which indicates the inhibition of coagulation processes. The lower microhardness values in 1570–0.5Hf alloy during long holding can be explained by the fact that a larger number of particles are available for the coalescence process in it than in 1570 alloy. Thus, hafnium additives make it possible to provide a significant advantage in strength properties during the first 8–10 h of heating, however, and then it is lost. It should also be noted that further study of the influence of hafnium additions on the thermal stability of Al_3Sc particles should be carried out in low-alloyed aluminum alloys (possibly with a lower scandium and zirconium content), which allow dissolving the discontinuous decomposition products. This will make it possible to distinguish the influence of an increase in strength properties resulting from the inhibition of discontinuous decomposition during the recrystallization process from the effect obtained by increasing the thermal stability of Al_3Sc nanoparticles.

CONCLUSIONS

1. Hafnium additives have a positive effect on the properties of 1570 alloy during heat treatment. Hafnium prevents the process of discontinuous decomposition of a supersaturated solid solution both during the ingot cooling after crystallization and during subsequent heat treatment.

2. It was found that in 1570 alloy with hafnium additives, during heat treatment, the total fraction of particles increases while their average size decreases in comparison with the original 1570 alloy.

3. Time intervals of 2–8 h are the most successful annealing mode for alloys with hafnium additives at a temperature of 440 °C. This is associated with the precipitation of strengthening Al_3Sc particles from the supersaturated solid solution; while for 1570 alloy, microhardness indicators remain unchanged, due to the fact that all nanosized dispersoids precipitate during the discontinuous decomposition of the solid solution.

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Влияние гафния на высокомагниевые сплавы, легированные переходными металлами, при термической обработке

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Аннотация: Целью работы является изучение влияния добавок гафния на механические свойства и термостабильность частиц при повышенной температуре термической обработки алюминиевых сплавов с высоким содержанием магния. Для изучения был выбран сплав 1570 в двух модификациях: без содержания гафния и с его добавкой 0,5 % по массе. Оба сплава были подвергнуты гомогенизационному отжигу при температуре 440 °С с различными режимами выдержки, которые составили от 2 до 100 ч. Для различных режимов термической обработки изучалась микротвердость, а также с помощью просвечивающей микроскопии исследовалась тонкая микроструктура. В результате удалось установить, что в процессе отжига при малом времени выдержки (2–8 ч) сплав с добавкой гафния имеет более высокие показатели микротвердости, превосходя показатели сплава 1570 в среднем на 20 HV. Это связано с тем, что в сплаве 1570 с добавками гафния при термообработке увеличивается количество выделяющихся частиц при одновременном уменьшении их среднего размера по сравнению с базовым сплавом. В то же время в сплаве 1570 без содержания гафния при его отжиге при температуре 440 °С роста микротвердости не происходит. Это обусловлено тем, что в сплаве 1570 без содержания гафния при остывании после литья происходит прерывистый распад, в результате которого большая часть скандия выделяется из пересыщенного твердого раствора в виде дисперсоидов. В сплаве с добавками гафния такого явления не наблюдается, что свидетельствует о его способности останавливать прерывистый распад в процессе охлаждения слитка после литья.

Ключевые слова: алюминиевые сплавы; переходные металлы; скандий; гафний; термообработка; упрочняющие наночастицы.

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