

Investigation of the ytterbium reduction process in the synthesis of Al – Yb master alloys for the modification of aluminum alloys

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This work investigates the synthesis process and characterization of a novel type of aluminum-ytterbium master alloy in molten salts. The significance of the obtained results for the research conducted in the field so far is due to the previously proven effectiveness of the influence of ytterbium on the structure and properties of aluminum alloys. During experimental work, the thermodynamic analysis of the aluminothermic process of ytterbium fluoride reduction from molten salts was performed and revealed that the process might occur and develop in the presence of sodium fluoride and potassium chloride if the NaYbF₄ precursor is formed. Subsequently, the temperature intervals of thermal effects during the process of reduction of ytterbium from its fluoride in molten salts (NaF – KCl) by aluminum were determined as a result of thermal analysis. During Al – Yb master alloys synthesis experiments, the maximal value of ytterbium extraction to master alloy of 82.5% was achieved by changing the process parameters, such as temperature, reaction time, and molten salts/aluminium ratios. Besides, master alloys containing ytterbium from 1.1 to 5.1 wt.% were synthesized using the optimized technological process conditions and examined with metallographic analysis. The analysis revealed that the microstructure of the synthesized master alloys is presented by eutectic colonies of Al + Al₃Yb composition located along the boundaries of α -Al dendritic cells. It was found that about 85.5% of the area of the studied samples with ytterbium content of 5.1 wt.% consist of aluminum dendrites with an average cell size of 25 μ m. The presented in this research results are of significant importance for the development of cheap and high-quality aluminum alloys.

Key words: aluminium alloys, ytterbium, master alloy, aluminothermic reduction, Al₃Yb, fluoride-chloride alloys, intermetallic compounds.

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Introduction

To date, aluminum alloys modified with rare earth metals are increasingly used in the aircraft industry, automotive industry, civil engineering, and other economy-forming industries, due to their superior mechanical, physical and corrosion resistance properties [1–9].

Based on the previous research, it is possible to conclude that scandium showed itself as one of the best modification compounds from the rare earth metal group for improving the properties of aluminum alloys [10–12]. Previous works clearly indicate that introducing even small amounts of scandium to aluminum alloy leads to the formation of secondary dispersed particles of Al₃Sc type coherent with aluminum matrix [13, 14] and results in grain size reduction [15, 16]. Obtained alloys exhibited increased mechanical properties, improved weldability, and increased corrosion resistance [17].

Despite all the advantages, full-scale use of scandium as an alloying element is limited due to the high price, low

production rates, and lack of aluminium-scandium master alloys [18]. This problem motivates the research community to search for alternative modification agents for aluminum alloys capable of fully or partially substituting the use of expensive Sc and providing similar properties, stability of the crystal lattice, and low price of the resulting alloy [19–22].

It was previously discovered [21] that a decrease in the atomic radius in the series of rare earth metals strongly affects the stability, structure, and composition of intermetallic compounds Al₃REMs. Moreover, REMs with the smallest atomic radius such as erbium, thulium, ytterbium, and lutetium have face-centered cubic (FCC) lattice structure. Thus, the aforementioned REMs might become potential Sc substitutes as they tend to form thermodynamically stable intermetallic compounds and have a smaller diffusion coefficient in aluminum compared to Sc. These unique properties allow erbium, thulium, ytterbium, and lutetium to prevent volumetric coarsening of particles making it possible to slow down the movement

of dislocations at elevated temperatures and to demonstrate greater solubility in the Al_3Sc strengthening phase [24].

Recently published works [25, 26] indicate that there is a trend to partially replace scandium with ytterbium as an alloying and modifying additive in the production of aluminum alloys. In research published by [25, 26], it was stated that ytterbium can have some effects on diminishing grain size during deformation or cast processing of aluminum alloys due to release of dispersoids $L12$. Experimental studies by Hong, et. al. [27] showed that the addition of ytterbium and lanthanum to an aluminum alloy provides the formation of $\text{Al}_{11}\text{La}_3$ and Al_3Yb compounds, which are nucleation centers that contribute to grain refinement. Wang W et. al. [28] also observed effects of grain size reduction after addition of ytterbium up to 0.5 wt.% in aluminum alloy system (Al – 40Zn – Yb). In works of [29–31], the simultaneous addition of ytterbium, chromium, and zirconium to the alloy of the Al – Zn – Mg – Cu system resulted in the increase of the strength and ductility of the aluminum alloy, and also contributed to a significant increase in corrosion resistance. Furthermore, the addition of ytterbium has a positive effect on the hardness increase of aluminoscandium alloys with increasing temperature [32–34]. Research published by Wen et. al. [35] shows that the combined additions of Yb and Si in the Al – Zr alloy system lead to hardness enhancement and cause secondary crystallization of the system. Curiously, the hardness results for the Al – Yb – Si – 0.08Zr alloy during isochronous and isothermal aging are 510 MPa and 515 MPa, which is much more than for Al – Yb – Zr (390 MPa, 400 MPa) and for Al – Si – Zr (300 MPa, 310 MPa) alloys.

Typically, the production of light metal alloys is carried out by dissolution in the melt of aluminum-based or magnesium-based master alloys with rare earth metals. In turn, typical methods for master alloys production are (i) fusion of pure components, (ii) electrolysis, or (iii) reduction of the alloying metal from its compounds [36–46]. It is worth emphasizing that no research works have been found in the domestic and foreign literature that reveal the technological features of the synthesis of Al – Yb master alloys. Taking into consideration the aforementioned aspects and the urgent need to search for alternative compounds to fully or partially replace scandium in aluminum alloys, it is possible to emphasize the importance of this work for the research field. The present study targets substantiating and developing scientific and methodological approaches to the synthesis of Al – Yb master alloys during the metallothermic reduction of ytterbium compounds from molten salts. Furthermore, it is important to note that the task of obtaining aluminum master alloys of a new composition for the domestic aluminum industry has an increased priority in connection with the approval of the Strategy for the Development of the Russian Metallurgical Industry for the period up to 2030 [47]. According to this document, it is necessary to increase the production of

metallurgical products with high added value. Moreover, the priority of the state is to stimulate the improvement of the technical level of production of Russian companies in order to increase the efficiency of processing mineral raw materials [48–54].

In this regard, the purpose of the present work is to study the features of obtaining Al–Yb master alloys with the determination of rational technological conditions for their production.

Materials and methods

Laboratory experimental studies of Al – Yb master alloy synthesis were carried out in a muffle furnace (LF-9/13-V2).

Potassium chloride and sodium fluoride were used as a technological salt mixture in this study. The feedstock containing ytterbium was YbF_3 with a purity of 99.99% (chemically pure), corresponding to TC 6-09-4677-83. Granulated aluminum with a granule size of up to 10 mm was used as a reducing agent.

The experimental procedure consisted of the following stages. A technological salt mixture was prepared in advance, consisting of potassium chloride, sodium and ytterbium fluorides at a given ratio of flux to aluminum from 0.21 to 0.94. Then the salts were thoroughly mixed, followed by placing the prepared mixture with aluminum granules in a graphite crucible so that the flux was evenly distributed over the entire surface of the metal. The crucible was installed in an electric heating furnace, with further exposure of the melt at a temperature of 750–900 °C for 10–60 minutes. At the end of the experimental time, the crucible was removed from the furnace and settled in air in order to separate the reaction products: the spent flux melt and the Al–Yb master alloy. Next, the spent salts were drained and the resulting master alloy was poured.

Thermal processes occurring during Al – Yb synthesis were determined by differential thermal analysis using STA 429CD of “NETZSCH”, Germany. Thermocouples of type “S” (Pt–PtRh10) were used in aluminum crucibles with lids in an air stream. Typically, a 200 mg sample consisting of an aluminum and salt mixture of NaF – KCl – YbF_3 was used for conducting differential thermal measurements. Heating and cooling were carried out in two cycles at a rate of 10 °C per minute; in the first cycle, the sample was heated to a temperature of 900 °C, then cooled to 300 °C; in the second successive cycle, the sample was heated to 900 °C and cooled again.

The chemical compositions of the obtained master alloy samples were determined using mass spectrometry (XRF-1800 Shimadzu spectrometer). The identification of crystalline phases was carried out by X-ray diffractometry using an XRD-6000 Shimadzu diffractometer equipped with a high-temperature camera HA1001 (CuK_α radiation, angle range $2\theta = 10\text{--}80^\circ$, shooting speed $2^\circ/\text{min}$.). The ASTM data library was used to interpret the obtained results.

The microstructure of the synthesized Al – Yb master alloy samples was studied by optical microscopy (light microscope Axio Vert.A1 of “Carl Zeiss”, Germany). All samples for microstructure analysis were prepared in accordance with standard metallographic grinding procedures using a Tegamin-25 of “Struers”, Denmark. Grinding was carried out with water on three types of sandpaper with a decrease in the dispersion of abrasive particles – 40, 26 and 14 microns. Initial polishing was performed with 3 micron Mol diamond slurry and 1 micron Nap slurry. For final polishing, a 0.05 μm Eposil suspension was used. The resulting microsections were etched with Keller’s reagent. Quantitative processing of the obtained metallographic images was carried out using Image J V 1.8.0 software. The original image of the sample section was uploaded to the software, then the scale was set, and colour correction was carried out. Then each object with the assigned number was converted into an ellipse for further calculations of granulometric characteristics.

The studies were carried out using laboratory facilities of the Scientific Center “Problems of processing mineral and technogenic resources” and the Center for Collective Use of the Mining University.

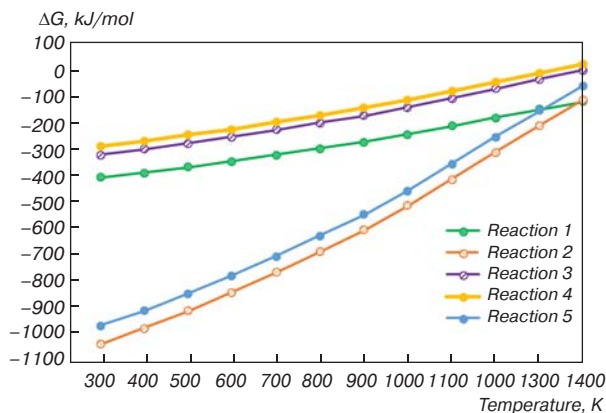


Fig. 1. Temperature dependences of changes in the isobaric-isothermal potential for reactions (1)–(5)

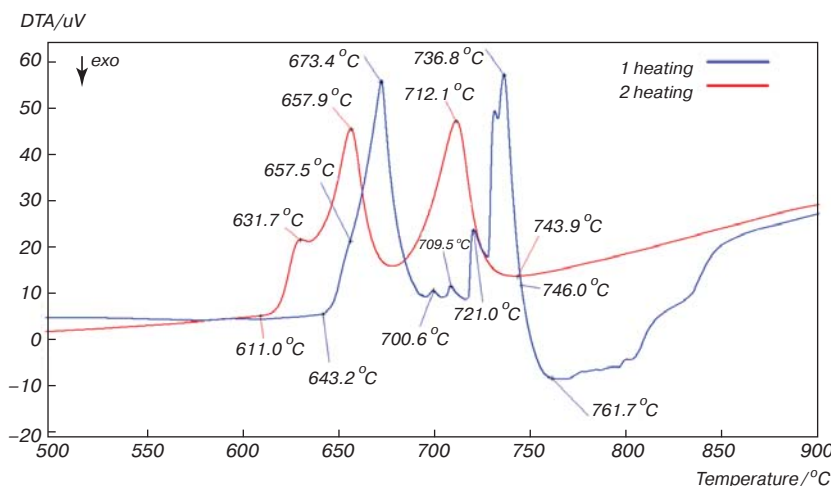
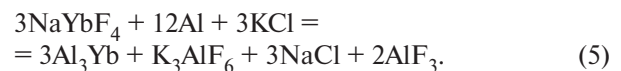
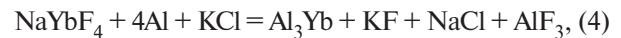
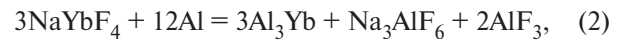


Fig. 2. T and DTA curves of the 1st and 2nd heating process of the NaF – KCl – YbF₃ – Al sample to 900 °C

Results and discussions

At the first stage, the thermodynamic probability of aluminothermal reduction of ytterbium compounds in chloride-fluoride melts was analyzed. The following reactions (1)-(5) describe the process of synthesis of the Al – Yb master alloy during the aluminothermal reduction of YbF₃ in the melt of NaF and KCl salts while considering the formation of Al₃Yb intermetallic compounds.



The following research [20, 38, 33–58] served as the basis for the thermodynamic assessment of the reaction mechanism of ytterbium reduction reactions, taking into account the formation of intermetallic compounds (IMCs) – Al₃Yb (Fig. 1).

The obtained temperature dependences show that the synthesis of Al – Yb master alloys by the method of aluminothermal reduction according to reactions (1)-(5) has a high thermodynamic probability, which is confirmed by the negative values of the Gibbs energy over the entire temperature range. A differential thermal analysis of the salt mixture with aluminum was performed to study the thermal processes occurring during the synthesis of aluminum-ytterbium master alloys. Fig. 2 shows the T and DTA curves, with NaF – KCl – YbF₃ – Al up to 900 °C.

During the first heating (blue color of the curve), the beginning of the melting of the salt mixture was recorded at 643.2 °C. This event is followed by the melting of aluminum, accompanied by an endothermic effect with a maximum at 673.4 °C. The next three minor thermal effects observed at 700.6 °C, 709.5 °C, 721.0 °C are associated with phase transformations of the salt components NaF, KCl, YbF₃, which are absent during the second heating. Subsequently, an endothermic effect was recorded with a maximum at 736.8 °C, apparently indicating the formation of precursors of the reduced metal which can be described by reaction (6):



With a further increase in temperature, an exothermic effect was recorded with a minimum at 761.7 °C, characterized by a large release of energy in a fairly narrow range. Presumably, the detected peak indicates the occurrence of the reaction of aluminothermal reduction of ytterbium since it is absent during the second melting.

The second heating (red color of the curve) is characterized by the start of melting process, obtained on the first melting of the Al – Yb alloy, at a temperature of 611°C, with a concomitant endothermic effect at 657.9 °C. At this phase of the process, the eutectic transformation of Al + Al₃Yb at 631.7 °C is observed, which can be confirmed by the phase diagram of the Al – Yb system [57, 59]. Afterward, a thermal effect with a maximum at 712.1°C, corresponding to the melting of the reacted technological salt mixture, is observed. Fig. 3 shows the thermograms obtained during the first and second cooling of the sample to a temperature of 500 °C.

During cooling process (curves 1 and 2), the beginning of crystallization of the resulting Al – Yb alloy is recorded at a temperature of 708 °C, with the first exothermic effect at 685.9–683 °C. Probably, the first exothermic effect indicates the crystallization of Al₃Yb intermetallic compounds, which correspond to the base phase for the Al + Al₃Yb eutectic. Apparent exothermic effects of crystallization of aluminum and reacted technological salt mixture are observed at minimums of 636.1–635.2 °C and 608.7–609.3 °C.

The next stage of the present research included experiments targeted at finding a rational ratio between the flux components, which ensures the maximum extraction of ytterbium from the salt melt. The compositions of salt mixtures (flux) and technological parameters for the first melting experiments were chosen based on the analysis of previous studies [20, 38]. Table 1 presents the results of exploratory experiments on the aluminothermic reduction of ytterbium from fluoride–chloride melts.

Based on the conducted study, it was found that the optimal composition of the technological salt mixture is 9.6YbF₃ + 6.5NaF + 6.5KCl (composition No. 2), with the ratio of the technological salt mixture to aluminum equal to 0.71. This composition of the technological salt mixture (flux) was used for further experiments on the aluminothermic production of master alloys.

At the next stage, a series of melting experiments was carried out by varying 2 parameters: the temperature regime from 750–900 °C and the melt holding time at a given temperature from 10 to 60 minutes. As a result of processing the obtained data, by means of a selective set

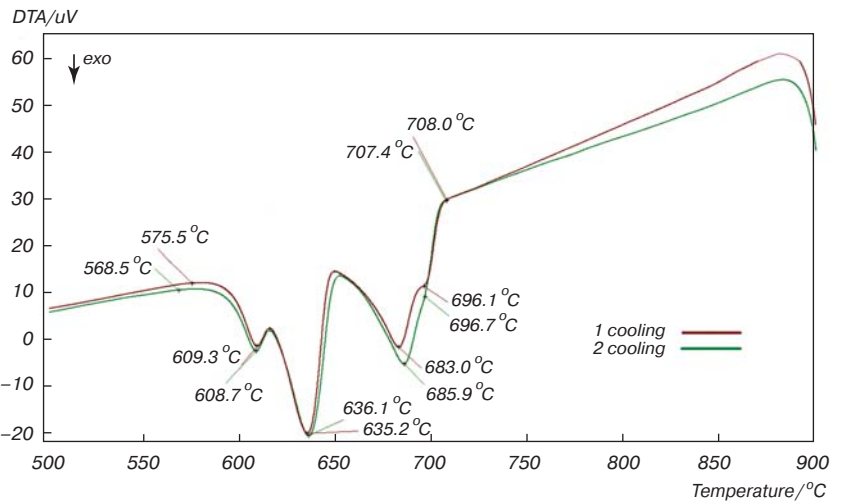


Fig. 3. T and DTA curves of the 1st and 2nd cooling process of the NaF – KCl – YbF₃ – Al sample to 500 °C

of different values of the yield of ytterbium into the master alloy from technological parameters, a graph was created, shown in Fig. 4.

The resulting dependence confirms the high rate of ytterbium reduction reactions from a selected salt mixture (9.6YbF₃ + 6.5NaF + 6.5KCl) with aluminum [55].

The maximum reduction percentage is achieved at a holding time of about 10 minutes at a temperature of 760 °C. When holding time increases, the extraction of ytterbium into the master alloy decreases, which can be explained by the deposition of ytterbium at the bottom of

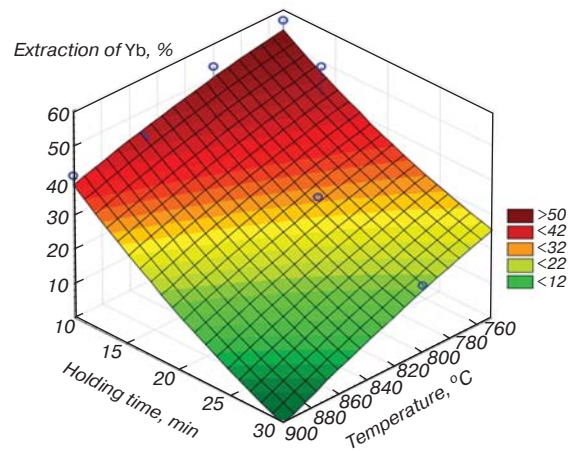


Fig. 4. Dependence of yield of ytterbium into master alloy from holding time and process temperature

Table 1
Indicators and conditions for conducting exploratory experiments to obtain Al – Yb master alloy

No.	The content of components in the mixture, wt. %			Flux ratio: Al	Process parameters		Extraction of Yb, %
	YbF ₃	NaF	KCl		T, °C	t, min	
1	13.6	6	80.4	0.33	750	35	25
2	9.6	6.5	6.5	0.71	750	35	44
3	32.6	21.7	45.7	0.09	750	35	16
4	50	10	40	0.17	750	35	10

Table 2
Results of Al – Yb master alloys synthesis

No.	The composition of the flux, wt. %			Flux ratio: Al	Process parameters		Ytterbium in master alloy, wt. %	Extraction of Yb, %
	YbF ₃	NaF	KCl		T, °C	t, min		
1	9.6	6.5	83.9	0.21	760	10	1,10	71.2
2	9.6	6.5	83.9	0.35	760	10	2,01	82.5
3	9.6	6.5	83.9	0.49	760	10	2,91	81.9
4	9.6	6.5	83.9	0.64	760	10	3.51	74.7
5	9.6	6.5	83.9	0.79	760	10	3.84	67.1
6	9.6	6.5	83.9	0.94	760	10	5.09	73.3

Table 3
Chemical composition of the obtained master alloys

No.	Mass fraction, %						
	Main components		Impurities				
	Al	Yb	Fe	Si	Na	K	Cl
3	88.75	2.91	0.52	0.32	0.34	2.32	4.84
6	93.61	5.09	0.51	0.47	0.20	0.13	–

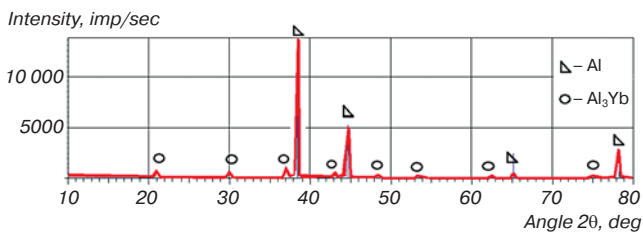


Fig. 5. X-ray pattern of Al – Yb master alloy with ytterbium content of 5.09 wt.%

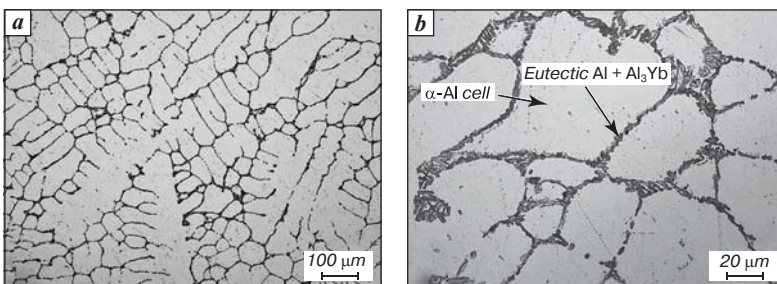


Fig. 6. The microstructure of the master alloy with Yb content of 2.91 wt. %: a – ×100, b – ×500

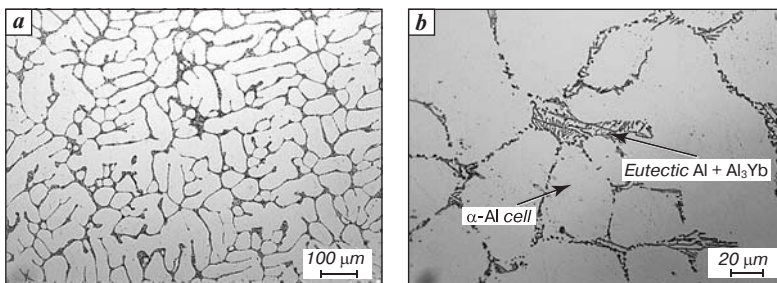


Fig. 7. Microstructure of master alloy with Yb content of 5.09 wt. %: a – ×100, b – ×500

the crucible. This deposition associates with the formation of a dense layer of IMC – Al₃Yb, which hinders their further diffusion into the alloy melt volume. Table 2 shows the results of these experiments.

As a main result of the described earlier experiments, master alloys containing ytterbium from 1 to 5 wt.% were synthesized using optimized technological parameters: technological salt mixture of the following composition 9.6% YbF₃ + 6.5% NaF + 83.9% KCl, mass ratio of salt mixture to aluminum equal to 0.7, process temperature of 760 °C, and holding time of 10 minutes. The highest achieved extraction of ytterbium into the master alloy was 82.5%, which is at a technologically acceptable level. Table 3 shows the chemical composition of castings No. 3 and No. 6.

A two-phase structure of the obtained Al – Yb master alloys was established based on the results of X-ray phase analysis. Fig. 5 shows an X-ray pattern for a master alloy sample with an ytterbium content of 5.09 wt.%, on which reflections with an interplanar spacing corresponding to Al and Al₃Yb were recorded [60].

Metallographic analysis of the synthesized master alloys confirmed the data of X-ray phase analysis. The microstructure of the Al – 2.91%Yb master alloy (Fig. 6) represents a colony of the Al + Al₃Yb eutectic composition (dark areas) located along the boundaries of enlarged α-Al dendritic cells (light areas). The microstructure can be characterized by a fragmented and discontinuous distribution of phase components. Most likely, the crystallization of the aluminum-ytterbium alloy with the formation of a closed framework of the aluminum matrix is achieved at a higher ytterbium content in the alloy. This assumption was confirmed by microstructural analysis of the master alloy with a Yb content of 5.09 wt.% (Fig. 7).

Comparative analysis of microstructures of master alloys with ytterbium content of 2.91 wt.% and 5.09 wt.% showed that in the latter case, the microstructure is characterized by a large number of evenly spaced equiaxed dendrites. An increase in the Yb content caused a more uniform “envelopment”

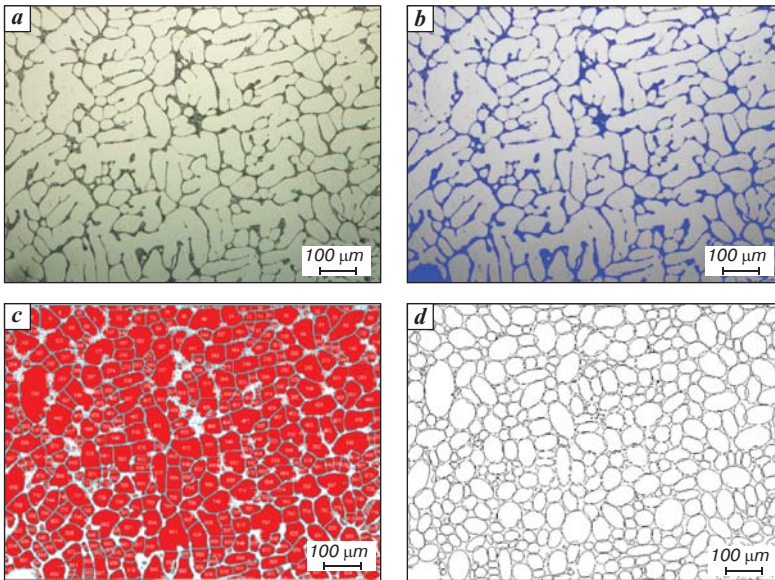


Fig. 8. Images of samples in Image J: *a* – original; *b* – threshold; *c* – contour and numbering of particles; *d* – transformation to an ellipse

of aluminum grains with eutectic interlayers, which makes it possible to more accurately determine the grain size. Also, a change in the morphology of the eutectic from dispersive to needle-shaped with an increase in the ytterbium content is observed. Using the “Image J” image analyzer, the granulometric characteristics of aluminum dendritic cells with an ytterbium content of 5.09 wt.% were calculated (**Fig. 8**).

As a result, the total number of analyzed α -Al dendritic cells was 1123. The calculated area was 1.03 mm^2 while the average area of one cell was $914.6 \mu\text{m}^2$. It was found that about 85.5% of the section area is occupied by aluminum dendrites, while the average size of a dendritic cell was $25 \mu\text{m}$.

Conclusion

The performed thermodynamic calculations of the chemical reactions of the aluminothermic reduction of ytterbium fluoride in the composition of the salt mixture confirmed the theoretical possibility of obtaining the Al – Yb master alloy, since the isobaric-isothermal potentials have negative values in the range of temperatures used.

The temperatures of endothermic and exothermic effects during the interaction of a NaF – KCl – YbF₃ mixture with aluminum were established by the method of differential thermal analysis. The exothermic effect of ytterbium reduction was established in the temperature range of 745–761 °C.

Aluminum-ytterbium master alloys were synthesized by aluminothermic reduction using a mixture of salts (9.8% YbF₃ + 6.8% NaF + 83.4% KCl) as the initial flux with a mass ratio to aluminum of 0.3–0.5. It has been established that at temperatures of 750–760 °C, the minimum time required for the metallothermic reduction of ytterbium to occur is 10 minutes. Using the optimized

technological conditions, the maximum achieved extraction of ytterbium into the master alloy is 82.5%.

The conducted X-ray diffraction analysis revealed the two-phase structure of the obtained samples of Al – Yb master alloys: solid solutions of aluminum (Al) and intermetallic compounds Al₃Yb.

The metallographic analysis showed that the structure of the obtained master alloys is characterized by a large number of aluminum dendrites surrounded by the Al + Al₃Yb eutectic. The calculated average size of the α -Al dendritic cells was $25 \mu\text{m}$.

The data gathered in this work represent an important step in research dedicated to the production of high-quality aluminum-ytterbium master alloys and their subsequent use in non-ferrous metallurgy as modifying and alloying additives for aluminum alloys.

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