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Features of the Process Obtaining of Mg-Zn-Y Master Alloy by the Metallothermic Recovery Method of Yttrium Fluoride Melt

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Abstract: At present, magnesium master alloys with such rare earth metals (REM) as yttrium are used in the production of alloys of magnesium and aluminum. These alloys especially the system Mg-6Zn-1Y-0,5Zr are commonly used in the aircraft and automotive industries. The article is devoted to the exploration of the synthesis process features for ternary magnesium master alloys with yttrium and zinc. The authors used X-ray fluorescence analysis (XRF), differential thermal analysis (DTA), and X-ray spectral analysis (XRD). Optical microscopy was used to conduct microstructural studies. The thermal effects that occur during metallothermic reactions of yttrium reduction from the YF_3-NaCl-KCl-$CaCl_2$ salt mixture with a melt of magnesium and zinc were investigated, and the temperatures of these effects were determined. It has been confirmed that the metallothermic reaction of yttrium reduction proceeds from the precursors of the composition: $Na_{1.5}Y_{2.5}F_9$, $NaYF_4$, $Na_5Y_9F_{32}$, and KY_7F_{22}, and starts at a temperature of 471 °C. The results of experimental studies of the process of metallothermic reduction of yttrium from the salt mixture YF_3-NaCl-KCl-$CaCl_2$ are presented in detail. These experiments were carried out in a pit furnace at temperatures ranging from 650 to 700 °C, and it was found that, at a synthesis temperature of 700 °C, the yttrium yield is up to 99.1–99.8%. The paper establishes rational technological regimes for the synthesis (temperature 700 °C, exposure for 25 min, the ratio of chlorides to yttrium fluoride 6:1, periodic stirring of the molten metal) at which the yttrium yield reaches up to 99.8%. The structure of the master alloy samples obtained during the experiments was studied. That structure can be distinguished by a uniform distribution of ternary intermetallic compounds (Mg_3YZn_6) in the bulk of the double magnesium–zinc eutectic. Studies have been carried out on testing the obtained ternary master alloy as an alloying material in the production of alloys of the Mg-6Zn-1Y-0.5Zr system, while the digestibility of yttrium ranged from 91 to 95%.

Keywords: rare earth metals; alloy synthesis; master alloy; magnesium alloy; magnesium–yttrium alloy; heat-resistant alloys; magnesium; metallothermic reduction

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1. Introduction

Yttrium has an atomic radius of 0.18012 nm, with a maximum solubility in solid magnesium of 3.6 wt. %. It is actively used as an alloyage supplement in cast and wrought magnesium alloys, in particular, in alloys of grades ML-19 and IMV7-1, as well as in Mg-Zn-Y, Mg-Sn-Zn-Y, Mg-Y-Mn, Mg-Y-Gd-Ca, and other mixtures [1–6]. Previously, it was found that the alloyage of magnesium alloys with yttrium affects the grain refinement; moreover, the supplement of yttrium into the magnesium alloy in the amount of up to 5.0 wt. % reduces the average grain size of magnesium to 33.62 microns.

The efficiency of using REM for the alloyage of magnesium and aluminum alloys [7–11], yttrium in particular, has been proven for a long time. However, the research aimed at improving the properties of magnesium–yttrium alloys continues to this day by scientists from various countries of the world [12–19]. Moreover, zinc is one of the widely used alloying additions in magnesium alloys and is introduced to increase strength in both cast and wrought magnesium alloys. Zinc with REM is usually used in magnesium alloys of various grades. Such additives are introduced to increase the efficiency of heat treatments; due to this, the yield strength and creep resistance of the alloy are each significantly increased. [20,21].

Obtaining light metal-based alloys with improved mechanical properties and performance characteristics depends to a greater extent on the type and quality of the master alloys used [22–26], such that their supplement, and subsequent assimilation, determines the microstructural structure of both alloys and cast products and ingots [27–29]. The relevance of the use of such alloys lies in the low rate of dissolution and assimilation of pure refractory components in the magnesium melt. It also lies in the high degree of assimilation of easily oxidized alloyage components [30]. It was determined that the main method for the production of foundry alloys is the direct alloying method, which is characterized by high temperatures which, accordingly, lead to high irretrievable losses of alloying elements. In this regard, the authors carry out research on obtaining double and ternary master alloys from their compounds by the method of the metallothermic reduction of REM. Ultimately, the presence of zinc in the composition of ternary master alloys provides a decrease in melting points, and also increases the solubility of refractory alloying elements.

The supplement of REM in the form of ternary master alloys with magnesium and zinc can be effectively implemented in the production of alloys. Particularly, it can be used for alloys based on magnesium and aluminum, since a large number of magnesium–yttrium alloy systems include zinc, introduced to increase durability [31–36]. In addition, its presence in the composition of ternary master alloys ensures a decrease in their melting points, and also provides a higher solubility of refractory elements in them [36]. It should be noted that magnesium and its alloys ignite when heated in air at temperatures above 500 °C, while the combustion of magnesium is accompanied by the release of a large amount of heat. Fluxes of various chemical compositions are used in industry to prevent the ignition of magnesium in the synthesis of master alloys. It is known that the composition of the technological salt mixture (flux), namely, its state of aggregation and thermal stability, have a direct impact on the technological parameters of the process of obtaining master alloys based on magnesium [37,38]. In this work, based on the known data [37], the base of the salt mixture of the composition 35KCl-35NaCl-30$CaCl_2$ was chosen. A new component, yttrium fluoride, was added to this flux in order to form complex compounds that are precursors in the production of Mg-Zn-Y ternary master alloys. Based on previous studies [36–38], it is important to emphasize that the use of fluxes in the melting of magnesium alloys does not reduce their corrosion resistance in further operations.

Due to the approval of the Strategy for the Development of the Russian Metallurgical Industry for the period up to 2030 [39], the task of obtaining magnesium master alloys of a new composition for the domestic magnesium industry has an increased priority. According to this document, it is necessary to increase the production of high added-value metallurgical products. Moreover, to increase the efficiency of mineral raw materials' processing, it is highly important for the state to stimulate Russian metallurgical companies to make improvements in the technical level of production [40–47]. In this regard, the research was aimed at studying the features of the process for obtaining ternary master alloys (Mg-Zn-Y), with the identification of technological conditions for their preparation.

2. Materials and Methods

Thermal analysis was performed on the STA 429 CD (NETZSCH, Selb, Germany) setup in an alundum melting pot with lids, in an argon flow, and at a heating rate of 10 °C/min to a temperature of 780 °C.

Studies on the magnesium thermal reduction of yttrium fluoride from molten salts in the presence of zinc were carried out at a variable synthesis process time, from 15 to 25 min, and with a synthesis temperature ranging from 650 to 700 °C. The constant ratio of magnesium to zinc (Mg:Zn) was 1:2, and the variable ratio of yttrium fluoride to chlorides was 1:6. The following methodology was developed for conducting experimental studies. At the start, a flux of composition YF_3+(35NaCl-35KCl-30$CaCl_2$ wt. %) was prepared. Then, it was mixed in a laboratory mixer and loaded into the melting pot, together with a magnesium ingot and granulated zinc. It was installed in a muffle furnace, heated to a predetermined temperature. The synthesis process was carried out by holding the melting pot in a furnace at specified temperatures and times in two modes: with periodic stirring of the melt and without stirring. After the end of the reaction of the exothermic reduction of yttrium, the melt was settled. Next, the spent salt melt, mostly its surface part, was poured off. Then, molds were filled with the master alloy. During the experiments, the mass ratio of the components of the 35NaCl-35KCl-30$CaCl_2$ salt mixture was maintained constant. Yttrium fluoride was added while observing the mass ratio of chlorides to YF_3 in the mixture 6:1. The required amount of yttrium in the alloy was achieved by changing the amount of magnesium and zinc during the recovery of yttrium fluoride from the salt melt. Three parallel experiments have been conducted, and results show the average values of the degree of the yttrium extraction.

An XRF-1800 spectrometer (Shimadzu, Kyoto, Japan) was used to perform an elemental analysis of the master alloys′ samples that were obtained during the experiments. The phases of the spent salt mixture were identified using the XRD-7000 diffractometer (Shimadzu, Kyoto, Japan). The metallographic examination of the samples of the obtained master alloys was conducted using an Axiovert 40 MAT, an optical microscope made by the Carl Zeiss company (Oberkochen, Germany).

3. Research Results and Discussion

To understand the stages of the synthesis process for the ternary master alloy from a chloride–fluoride melt of the composition YF_3:(NaCl-KCl-$CaCl_2$) (1:6), the phases of a pre-melted salt mixture of a given composition were identified. This showed that during the melting of a salt mixture, YF_3 interacts with potassium and sodium chlorides, forming complex compounds of the composition: KY_7F_{22}, $NaYF_4$, $Na_{1.5}Y_{2.5}F_9$, and $Na_5Y_9F_{32}$, which, in turn, are precursors in the production of a ternary master alloy with the formation of $Mg_xY_yZn_z$ intermetallic compounds. Figure 1 shows the radiograph of the salt mixture after melting, and Figure 2 shows the radiograph of the salt mixture after the separation of water-soluble chlorides.

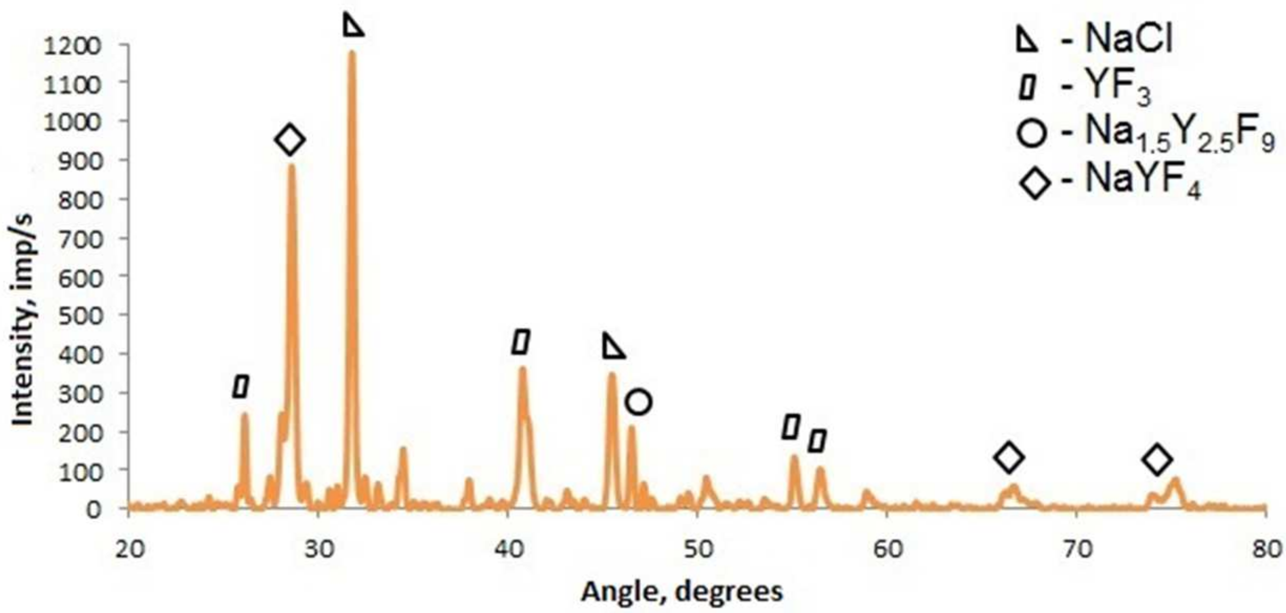


Figure 1. X-ray pattern of melted salt mixture YF_3–NaCl–KCl–$CaCl_2$ after melting.

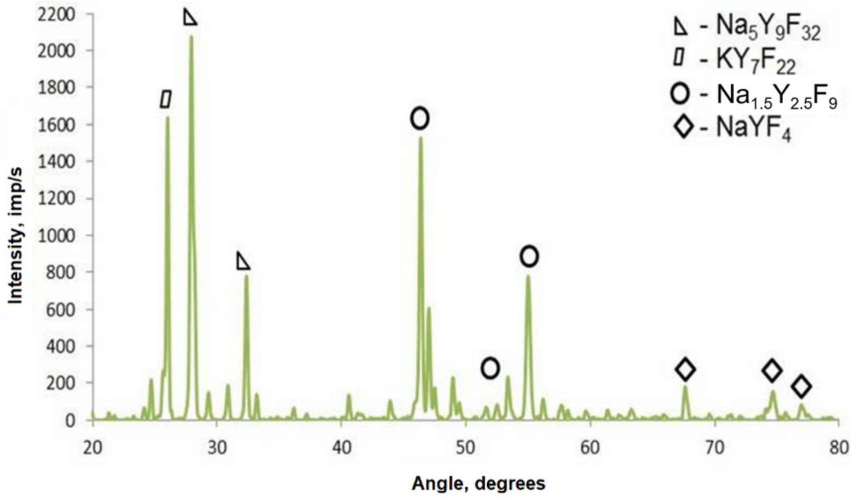


Figure 2. X-ray pattern of melted salt mixture YF_3-NaCl-KCl-$CaCl_2$ after the separation of chlorides.

At the following stage, the thermal analysis of the master alloy synthesis for a given composition, in the presence of magnesium and zinc, from a technological salt mixture YF_3:(NaCl-KCl-$CaCl_2$) (6:1), was carried out. Figure 3 shows the thermogram obtained by heating a mixture of YF_3-NaCl-KCl-$CaCl_2$ with magnesium and zinc.

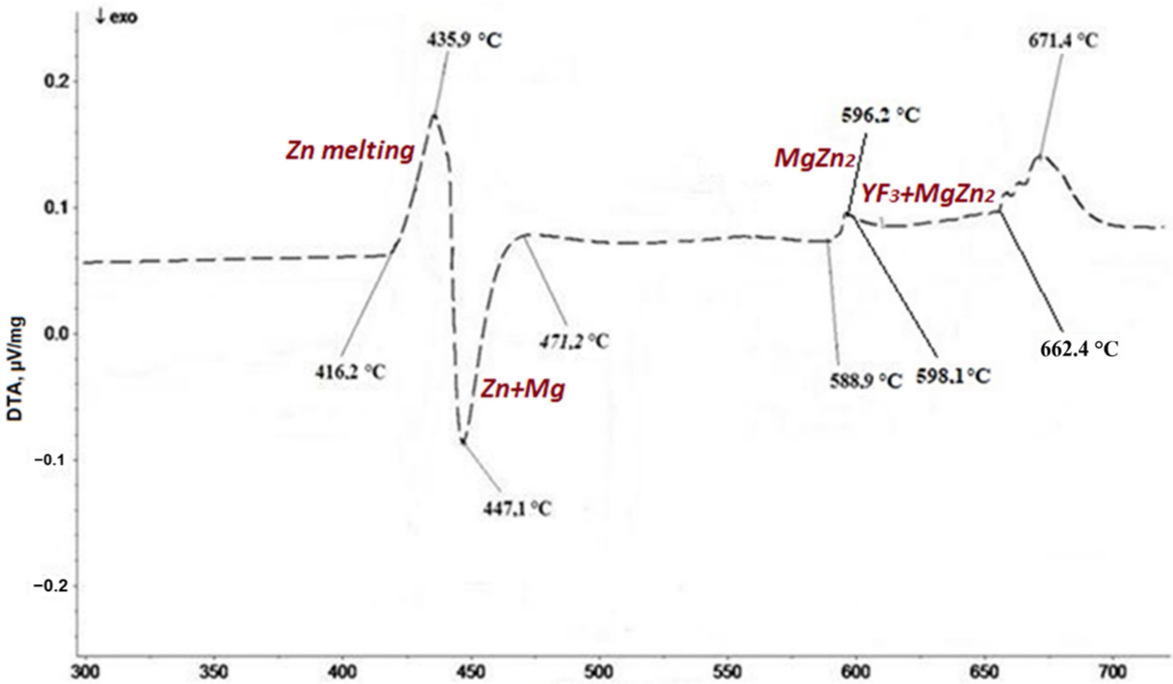


Figure 3. DTA curve for the melting of a YF_3-NaCl-KCl-$CaCl_2$ mixture with magnesium and zinc when heated to 780 °C.

It was determined that the endothermic effect, formed at a temperature of 416.2 °C and with the highest point at 435.9 °C, coincides with the melting of the most low-melting element in a given system, namely, granulated zinc, which has a melting point of 419.6 °C. After that, the molten zinc begins to actively interact with the magnesium ingot (2Zn + Mg = $MgZn_2$); this can be confirmed by the exothermic effect, with a minimum at the temperature of 447.1 °C, which ends at 471.2 °C. When the temperature reaches 471.2 °C, an exothermic effect with a slight inflection is detected on the thermogram; it ends at the temperature of 588.9 °C, and is interrupted by the melting, most likely, of the double intermetallic compound $MgZn_2$, with an endothermic effect and maximum at the temperature of 596.2 °C [48]. After that, in the range from 598.1 to 662.2 °C, another exothermic effect is detected. The revealed exothermic effects indicate the occurrence of an exothermic reduction reaction of YF_3 ($YF_3 + MgZn_2 = Mg_xZn_yY_z + MgF_2$) from the salt mixture with a magnesium–zinc melt, which is confirmed by the Mg-Zn-Y composition obtained after the analysis of the metal bead.

At the next stage of the experiments, the main task was to determine the optimal technological parameters for melting so that the higher yttrium yield for the master alloy could be achieved. The results of the studies that were carried out, the experiments that were conducted, and the initial data on obtaining a ternary master alloy are shown in Table 1.

Table 1. Results of experiments on obtaining Mg-Zn-Y master alloy.

Melt No.	Ratio YF_3: Chlorides	T, °C	t, min	Stirring	Recovery Y, %
1	1:4	650	15	no	87.1
2	1:6	650	15	no	96.2
3	1:4	700	15	yes	87.8
4	1:6	700	15	yes	99.1
5	1:4	650	25	yes	86.4
6	1:6	650	25	yes	94.2
7	1:4	700	25	no	89.1
8	1:6	700	25	no	98.3
9	1:4	650	15	yes	86.2
10	1:6	650	15	yes	99.6
11	1:4	700	15	no	88.2
12	1:6	700	15	no	94.4
13	1:4	650	25	no	86.3
14	1:6	650	25	no	95.1
15	1:4	700	25	yes	89.1
16	1:6	700	25	yes	99.8

It was determined that the result of the exothermic reactions of the yttrium reduction from the obtained salt mixture (YF_3-NaCl-KCl-$CaCl_2$) by a magnesium–zinc melt is the production of a magnesium–zinc–yttrium ternary master alloy. According to experimental data, it was discovered that with an increase in the ratio of YF_3 to chlorides to 1:6 in the technological salt mixture, the yield of yttrium increases to 99.8%. There were no significant changes to the yttrium yield after increasing the temperature to 700 °C. The main result of the research was obtaining the ternary master alloys, with a 25% Y wt. content and a clean surface without non-metallic inclusions and gas pores. The macro images were taken on a Canon 60D and Canon 100 mm f/2.8 macro (Tokyo, Japan) (Figure 4).

(**a**) (**b**)

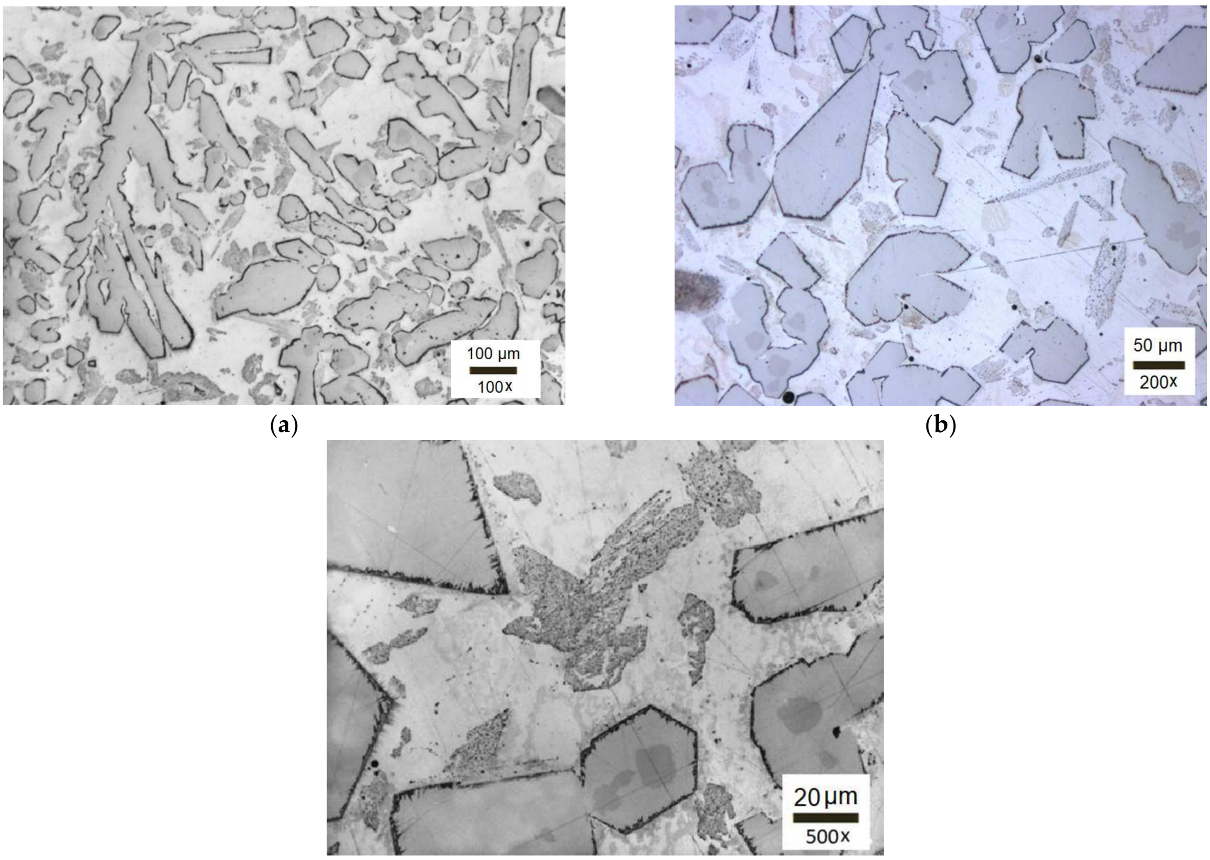
Figure 4. Macrostructure of the Mg-Zn-Y master alloy: (**a**) 5×; (**b**) 5.5×.

There were no requirements or standards for ternary master alloys of the studied composition, so for the purpose of making a comparison of the content of impurities, the list of requirements for the magnesium–neodymium master alloy was taken as a reference. This master alloy is also used in the production of heat-resistant magnesium alloys. According to TU 48-4-271-91, concerning the impurities content, the Mg-Zn-Y master alloy meets the requirements for magnesium master alloys Mg-Nd (grade MN) (Table 2).

Table 2. Elemental composition of the master alloy 25Mg-50Zn-25Y (TU 48-4-271-91).

Composition	Mass Fraction, %							
	Main Components			Impurities, Less				
	Zn	Mg	REM	Fe	Ni	Cu	Al	Sc
Mg-Nd	-	basis	20–35	0.15	0.01	0.1	0.05	0.05
Mg-Zn-Y	basis	25.6	23.9	0.09	-	0.05	0.03	0.04

Microstructural studies show that the obtained samples of the magnesium-zinc-yttrium master alloy are characterized by a structure that includes the double magnesium-zinc eutectic and uniformly distributed intermetallic compounds of the composition Mg_3YZn_6 [49]. Figure 5 shows typical images of the microstructures of the magnesium-zinc–yttrium master alloy obtained by magnesium thermal reduction of a chloride-fluoride melt.



(**a**) (**b**) (**c**)

Figure 5. Microstructure of the master alloy 25Mg-50Zn-25Y; (**a**) 100×, (**b**) 200×, (**c**) 500×.

At the final stage, the resulting ternary master alloy was tested as an alloyage material, which was carried out during the production of the alloy system Mg-6Zn-1Y-0.5Zr in a muffle furnace. The heat treatment of the obtained alloys, as well as further tests, were not carried out at this stage. The assimilation of yttrium in the process of obtaining an alloy ranged from 94 to 98%, while the structure of the obtained alloy is a microstructure typical of magnesium alloys, which is characterized by uniformity and a grain size that equals 52.4 µm on average (Figure 6).

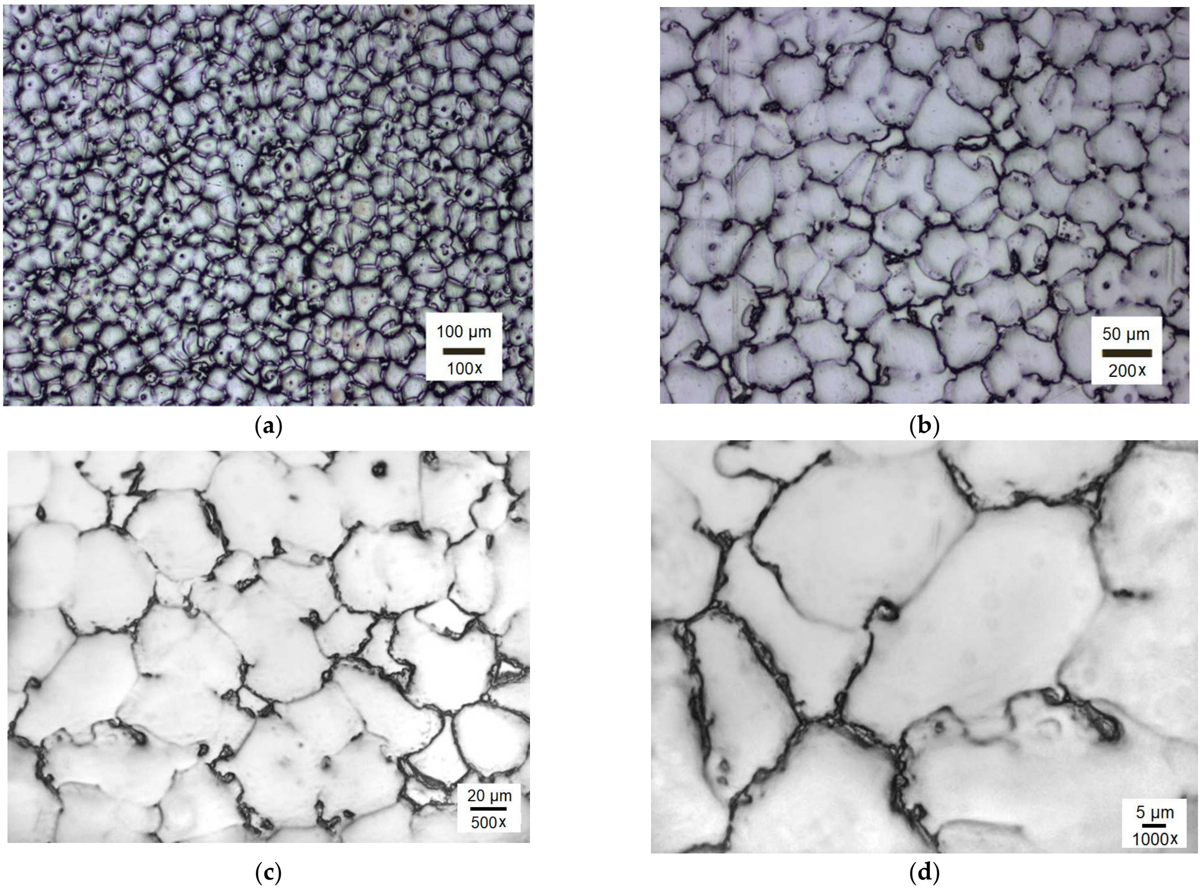


Figure 6. Microstructure of the Mg-6Zn-1Y-0.5Zr alloy wt. %; (**a**) 100×, (**b**) 200×, (**c**) 500×, (**d**) 1000×.

4. Conclusions

Experimental studies were carried out, and as a result, a magnesium-zinc-yttrium ternary master alloy was obtained, and technological solutions were developed that ensure a high extraction of yttrium (up to 99.8%) into the Mg-Zn-Y ternary master alloy from fluoride during metallothermic reduction. Rational technological regimes for the synthesis (temperature 700 °C, exposure time 25 min, ratio of yttrium fluoride to chlorides 1:6, periodic stirring of the melt) were revealed, at which a high degree of yttrium extraction is achieved.

Using differential thermal analysis, the starting and ending points of the reduction reaction of yttrium with a magnesium–zinc melt from its fluoride in a chloride melt were determined: it occurs at the temperature of 471 °C, and is then further confirmed by an exothermic effect that ends at the temperature of 588.9 °C.

The structure of the obtained samples can be distinguished by the uniform distribution of ternary intermetallic compounds (Mg_3YZn_6) in the bulk of a double magnesium–zinc eutectic.

The detailed study was carried out on testing the obtained ternary master alloy as an alloying material in the production of alloys of the system Mg-6Zn-1Y-0.5Zr, while the digestibility of yttrium ranged from 94 to 98%.

The data obtained can be used as the basis for the development of an industrial technology for the production of magnesium–zinc–yttrium alloys, as well as for the development of standards for their use in non-ferrous and ferrous metallurgy.

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