Structural investigation of 70Cu/30Fe based cast alloy obtained by combined use of centrifugal casting-SHS process and forging

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Abstract. This research is to test a combined chemical-technological chain of material production. The tests are made on a 70Cu/30Fe system. The technology consists of: 1- SHS of a cast material of a specific composition and 2- Mechanical and heat treatment, including: continuous swaging, where the workpiece is heated to 850⁰ C and then drawn cold. In this study first used SHS-metallurgy method for producing, in the molten pseudoalloy Cu-Fe system (Cu- 70%, Fe- 30 wt.%). It was found that the high temperatures melt the SHS provides increased solubility of Cu in Fe. Then, in the crystallization, by decreasing the melt temperature and reduce the solubility limit of copper is released in the form of small dispersed particles. The observed structure is typical only for SHS alloy.

1 Introduction

There exists a well-known broad class of binary and multi-component metallic materials with limited solubility when liquid or solid. In some cases, such materials have unique characteristics.

Alloys with limited solubility (LS) or pseudoalloys (PA) have long been impossible to obtain with traditional methods of metallurgy. When trying to produce such alloys, metallurgists face a number of challenges, which are mostly down to a considerable difference in specific masses and melting points as well as a strong tendency to stratification in the liquid or in the solid state in a wide range of temperatures and concentrations.

According to the literature, Cu-Fe alloys are becoming of even greater interest [5,7,8,9]. Alloys of the Cu-Fe system with a Cu-based matrix (i.e. an increased concentration of Cu) are promising for creating high performance hard magnetic materials. [5,7] The system has an interesting structural peculiarity which is that its component phases actually do not interact, which enables selective effects on such structural components to cause targeted changes in the properties of the material. After necessary dispersion [5] of ferromagnetic Fe particles is achieved, and their form becomes isotropic, one can expect high magnetic energy values as it is known [5,7] that extended ferromagnetic particles of critical size do not prevent magnetic reversal in a non-ferromagnetic matrix.

This is why developing low-cost and simple technology to produce such alloys and materials based thereon and to adjust their physico-mechanical properties is still a relevant problem when it comes to the production of such materials.

This research is to test a combined chemical-technological chain of material production; the tests are made on a 70Cu/30Fe system. The technology consists of:

- (I)- SHS of a cast material of a specific composition,
- (II)- Mechanical and heat treatment, including: continuous swaging, where the workpiece is heated to 850⁰C and then drawn cold.

2 Research Methodology

2.1 Synthesis of a cast alloy by means of SHS

To synthesize the alloy, we used SHS [3,4,10]. This method is about using chemical energy produced in the reaction of highly exothermic thermite alloys (combustion), making this a very energy-efficient method for production of cast materials. Synthesis does not take long (dozens of seconds), and the top surface of ingots is protected from oxidation with an oxide melt (Al₂O₃), which enables synthesis in atmospheric conditions.

To synthesize a cast Cu-Fe alloy with 70% Cu and 30% Fe, we used powder Cu and Fe oxides as well as Al for reduction. Source powder particles did not exceed 100 μm in size. Exothermic compositions were prepared as follows: components were dosed, then mixed in a planetary mixer for 15 minutes, then charge was put into graphite 40- or 80-mm forms.

The general chemical synthesis reaction can be written as follows:

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where the weight of components was adjusted based on thermodynamic calculations and actual analysis of alloy composition.

To intensify the gravitational separation of the alloy and aluminum oxide as well as to induce convective mixing of the melt components, the synthesis was done with a centrifugal SHS machine [5] with 2 to 50a/g overloads, where a is centrifugal acceleration, g is gravitational acceleration.

Fig. 1. (a) preparing the mixture, (b) SHS combustion, (c) gravitational separation of phases, (d) crystallization and cooling of the melt.

Figure 1 presents the main stages of the SHS method, i.e. component dosage, mix preparation, and putting the powder mixture into graphite forms. Then the form is placed on the centrifugal rotor; upon the necessary RPM is reached, the mixture is ignited, and combustion synthesis begins. The combustion temperature exceeds the melting point of both source and final reaction components, which enables one to get cast products.

Due to the mutual insolubility and significant difference in specific weights, gravity causes reciprocal separation of the metallic (target) and the oxide (Al_2O_3) phase. As a result, an ingot of the alloy is formed at the bottom of the form, whereas oxide solution is generated in its upper part. High gravity generated in the centrifugal machine inhibits the dispersion of combustion products during the synthesis, intensifies the separation of metallic (alloy) phase and the oxide (corundum) phase, and homogenizes the composition of the alloy, which is important for producing the PA and form a highly dispersive structure of the final product.

2.2 Continuous swaging (forging)

Continuous swaging was done using a C41-250 pneumatic hammer. This model has the following characteristics: falling parts weigh 250 kg, maximum impact energy equals 5.6 kJ, 140 hits per minute-1; the drive is Y180L-4-B3. Continuous swaging consists in consecutively hitting and moving the workpiece, whereby only a part of the workpiece is located between the dies as it sustains the impact. After each compression, the workpiece is moved by a distance that less than the length of the die. To make forgings quickly, we used rolling-impression dies, as they prevent axial fractures thanks to no intensive lateral flow of the metal.

2.3 Drawing

Drawing was done using an MB1 drawing machine. The machine consists of the main drawing unit, a bath, a wire

spool, a wire-pointing machine, and a mechanism to handle finished products. When passing the drawing hole, rods become thinner and longer while unchanged in volume. Drawing was done using a multiple-drawing machine at a rate of 4.5 m/s with a die step of 0.2 mm.

2.4 Microstructure Research

The microstructure of the SHS-made cast alloy was studied using an Ultra-55 based Zeiss Ultra plus auto-emission ultra-high resolution scanning electron microscope. This microscope is able to perform point analysis, analysis along an arbitrary line, analysis over an arbitrary area, analysis of an array of points; it is also capable of mapping the distribution of elements over the surface of the sample as well as to identify phases. Magnification is 12 to 1,000,000. Acceleration voltage is 0.02V to 30kV. Probe current is 4 pA to 20 nA.

The chemical composition of the melt was tested using an OBLF emission spectrometer with CCD detectors.

This structure of the SHS alloy was studied by transmission electron microscopy (TEM) using a JEM-2100 machine made by JEOL, Japan.

3 Experiment Results and Discussion

We first conducted a series of experiments to SHS-synthesize a 70Cu/30Fe alloy and optimized the synthesis condition; we researched how overloads could affect the parameters of the process. The first stage is about using chemical energy produced in the reaction of highly exothermic thermite alloys (combustion), making this a very energy-efficient method for production of cast materials. Synthesis does not take long (dozens of seconds), and the top surface of ingots is protected from oxidation with an oxide melt (Al_2O_3), which enables synthesis in atmospheric conditions. To ensure the uniform distribution of iron particles in the copper matrix, experiments were done using a centrifugal SHS machine [10] where centrifugal forces cause overloads within a range of 2 to 50a/g, where a is the centrifugal acceleration, g is the gravitational acceleration. The main process parameters as a function of a/g are presented in Figure 2. Experimental data revealed a considerable increase in the combustion rate from 4 to 9 cm/s depending on the SHS a/g value. The most intensive increase in rate (U) (more than 20 times) was observed within overloads of 2 to 20 a/g.

Microanalysis of the 70Cu/30Fe SHS alloy (Figure 3a) and the distribution of elements in structural components (Figure 3) confirmed the presence of dispersive precipitations varying in level, both in the Cu matrix and in the dispersed Fe particles.

Fig.2. Effect of overloads (a/g) on the combustion rate (u), mixture dispersion (η_1), and the extent to which the metal phase transformed into an ingot (η_2 .)

Note that microanalysis of local alloy areas always identified the presence of both components in the area analyzed. Given the low mutual solubility of the alloy components, this result is very interesting.

Fig. 3. EDS microanalysis of 70Cu/30Fe SHS alloy (a); microphotograph of the SHS alloy structure.

SHS alloy microstructure was studied at a greater magnification (Figure 3b) to find out that the SHS-made alloy has a multi-level hierarchical structure. Analysis of these structures led to a conclusion that a tri-level structure had been formed. The first level (Figure 3a) is characterized by uniform distribution of 10-30 μm Fe particles across the ingot. The second level is formed by uniformly distributed 2-0.5 μm Cu particles (Figure 3a) in the precipitated Fe particles of the 1st structural level. Greater-magnification TEM revealed a third level formed by uniformly distributed sub-micron 20-50 nm Fe particles (Figure 3b) in the copper particles of the 2nd level. We have to note that this multi-level microstructure was observed not only in the Fe particles; the first-level copper matrix contained dispersive precipitations of Fe particles (Figure 3b'). This is perhaps tied to the specifics of the SHS method. SHS mixture combustion temperature exceeds 2500 K. This ensures increased solubility of Cu in Fe. At the crystallization stage, as the temperature of the melt and the solubility limits are lowered, copper is precipitated in the form of small dispersed particles.

The next technological stage is mechanical and heat treatment to achieve necessary dispersion and ensure isotropy of ferromagnetic Fe particles in the Cu matrix.

SHS is first heated in a muffle furnace to 850°; then it is continuously swaged. At this stage, SHS workpieces are transformed into 10-mm rods, while the quality of the final material is improved. For the microstructure of a longitudinal cut of our sample, see Figure 4b. Then we wanted to retrieve longitudinal structural components from the 8-mm cylindrical rod cleansed of scales; for that purpose, we would draw it. The long workpiece was subjected to plastic deformation by drawing it through a hole in the die; the diameter of the hole was less than the cross-section of the workpiece. Thus we obtained samples that were reduced from 8 to 4.5 mm (deformation degree $\varepsilon=67\%$, see Figure 4c) and from 8 to 3 mm ($\varepsilon=86\%$, see Figure 4). Figure 4d presents the microstructure of the samples

Fig.4. Microstructure of SHS samples (a) after continuous swaging (b) and being drawn to $\varnothing 4.5$ mm (c) or $\varnothing 4.5$ mm (d).

4 Conclusion

The paper is the first to present, test, and discuss a combined method to make limitedly-soluble alloys. With evidence from a 70Cu/30Fe alloy, we demonstrate that this SHS alloy features multi-level distribution of mutually unmixable components, whereby a pseudoalloy with uniformly distributed structural components is produced.

Based on the microstructure and chemical analysis of alloys made by SHS and mechanical and heat treatment, one can say that combustion-produced alloys have a homogeneous structure with uniform distribution of all structural components in the sample, which is of great practical interest for making isotropic and anisotropic hard-magnetic materials with great magnetic energy.

SHS-produced Cu-Fe alloys can be readily used to regulate their structure and to fabricate bulk materials with longitudinally oriented structural constituents.

Data analysis led to conclusion that these materials and combined SHS could be promising for making and generating dimensional nano-structure materials. In the future, studying these alloys to learn how to control their structure and properties could be of interest.

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