Abstract. The effect of high pressure torsion (HPT) on the microstructure evolution of Cu-Fe 36% wt alloy has been studied. The initial Cu-Fe alloy has a dendritic structure, the length of dendrites is up to 100 µm. As a result of HPT (20 anvil rotations at 400 ºC) the refinement of α-Fe dendrites occurs, and a microstructure with Fe inclusions with a size from 0.1 to 5 µm uniformly distributed in the copper matrix forms. Subsequent annealing at 700 ºC for 1 hour results in some coarsening of α-Fe particles, as compared to the state after HPT. However, the dendritic structure typical of the cast alloy does not recover; it remains dispersed with a size of α-Fe particles less than 20 µm. As a result of HPT the alloy microhardness increased from 1800 to 4000 MPa. The subsequent annealing at T = 700 ºC decreased the microhardness to 2700 MPa, but this value is 1.5 times higher than that in the initial as-cast state.

1. Introduction

The alloy properties are determined, aside from the composition, by its microstructure. The efficient method of microstructure transformation and refinement is severe plastic deformation (SPD) [1]. Currently, there are two most well-known SPD techniques. They are equal channel angular pressing (ECAP) and high pressure torsion (HPT), which enable achieving the highest strain degrees (e > 2-4) without failure of billets under quasi-hydrostatic pressure [1]. Previously, homogeneous ultrafine-grained (UFG) and nanostructured (NS) states were produced via HPT in such metall materials as Cu, Ni [2-4], Fe [5, 6] steel [7, 8], magnesium [9], aluminum and titanium alloys [10-13].

The object of study in this paper is Cu-Fe alloys. The interest in these alloys is explained by their possible application as low-consumable anodes for aluminum production [14, 15]. Copper and iron are slightly soluble in each other; a two-phase region can be seen in a wide range of concentrations in the phase diagram of Cu-Fe (Fig. 1). When the melt is cooled, these alloys crystallize, and a coarse dendrite structure (fcc copper matrix and α-Fe dendrites) forms. The dendritic microstructure of these alloys formed during casting reduces the corrosion resistance of these alloys during electrolysis in aluminum oxide.

The scientific idea of applying SPD to Cu-Fe alloys is based on the results of study on the regularities of structural and phase transformations in various metals and alloys during SPD [1]. In particular, during high pressure torsion of metals and alloys, in addition to UFG and nanostructured state formation, the metastability increases, the phase composition changes, the phases dissolve, the supersaturated solid solutions form, or disordering takes place [1]. Since the SPD will transform the initial dendritic microstructure of the Cu-Fe alloy, one may assume that this would enhance the corrosion resistance of alloys and effectiveness...
of their application as anodes. The aim of this work is to analyze the changes in the microstructure and phase composition of the Cu-36 % Fe alloy during HPT.

![Phase diagram of the iron-copper system](image)

**Fig. 1.** Phase diagram of the iron-copper system [16].

### 2. Experimental techniques

In this work the samples of the cast alloy Cu-36 % Fe (wt.) of 10 mm in diameter and 1.5 mm in thickness were subjected to HPT on grooved anvils with a number of rotations 10 and 20 at 400 °C, at a pressure 6 GPa. The shear strain at the edge of the sample was $\gamma \sim 450$ and 960, respectively.

Heat treatment of the samples after HPT was performed in a vacuum furnace at 700 °C for 1 hour (this temperature is the lowest possible temperature for operation of the electrolytic cell and anodes in the aluminum production). The sample surface was polished and then etched in the acid mixture (50 % of HNO$_3$ and 50 % of HCl) for structural studies. The structure was investigated by the scanning electron microscope (SEM) JSM-6490LV Jeol in the mode of back-reflected electrons in the edge parts of the samples after HPT. The average sizes of structural elements and the phase fraction were estimated by 60-200 measurements.

### 3. Results and discussion

The microstructure of the cast Cu-Fe alloy is heterogeneous: it is represented by the Cu matrix and columnar Fe crystals with a distinct dendritic relief distributed in it (Fig. 2). Such parameters as the average length and width of major $\alpha$-Fe dendrites, the average length and width of dendrite “branches”, the average sizes of $\alpha$-Fe equiaxed grains were measured to describe the dendritic structure of the original cast alloy. The average length of the major dendrite axes was about 60 µm and the width was about 10 µm. The major axes of the dendrites branch off up to 30 µm in length, 7-10 µm in width. Other than the basic Fe dendrites of “skeletal” shape equiaxed inclusions of 5-10 µm are observed.
High pressure torsion led to significant structural changes in the Cu-Fe alloy. As a result of HPT, the dendrites get crushed and refined. In the copper matrix only separate α-Fe inclusions of a globular shape are observed. According to the SEM image taken at a magnification of 1000 times, the size of the α-Fe inclusions observed after 10 HPT rotations was about 5-10 µm (Fig. 3, a) and after 20 HPT rotations — no more than 5 µm (Fig. 3, b). According to the SEM image at a magnification of 1000 the α-Fe volume fraction markedly decreased as compared to the initial one (compare Fig. 2 and Fig. 3, b). SEM study of the structure with high magnification (20 000) shows that the copper matrix structure after HPT (20 rotations) is refined to a grain size of about 250 nm, and the Fe inclusions of 100-200 nm are uniformly distributed in it (Fig. 4, a).

Annealing of the sample subjected to HPT at 700 °C for 1 hour results in growth of α-Fe inclusions to 3-10 µm, although there are larger α-Fe precipitations reaching 20 µm in the structure (Fig. 4, b). But the dendritic structure, typical of the cast state after HPT and annealing, does not recover, it remains dispersed, and α-Fe inclusions have an equiaxed shape.
Microhardness (H<sub>v</sub>) measurements showed that H<sub>v</sub> after HPT increases to 4000 MPa from the initial value of 1800 MPa. Subsequent annealing at 700 °C (1 hour) reduces H<sub>v</sub> to 2700 MPa, however, this value is 1.5 times higher than H<sub>v</sub> in the initial as-cast condition. Thus, the effect of HPT and subsequent annealing at a relatively high temperature (700 °C) has not only substantially refined the cast Cu-Fe structure, but also enhanced the alloy hardness.

![Fig. 4. SEM-image of the Cu-Fe structure in the state after HPT at T = 400 ºC, the edge of the sample: a) HPT n = 20 rotations, magnification x 20 000; b) HPT n = 20 rotations and subsequent annealing at 700 ºC for 1 hour, magnification x 1000.](image)

4. Conclusions:

1. It is established that during HPT the initial dendritic structure of the α-Fe cast alloy gets refined. The size of α-Fe particles after HPT ranges from 0.2 to 5 µm. It is shown that the α-Fe phase refinement degree is proportional to the HPT strain degree.
2. Annealing after HPT at 700 °C for one hour resulted in the growth of α-Fe particles. However, the initial dendritic structure does not recover and is represented mainly by fine precipitates of 3-10 µm uniformly distributed in the Cu matrix.
3. As a result of HPT and structure refinement, the microhardness increases from 1800 to 4000 MPa. Subsequent annealing at 700 °C (1 hour) reduces H<sub>v</sub> to 2700 MPa, but this value is higher than in the initial as-cast condition.

The study was carried out as part of the RFBR Project № 12-08-00971 “Structural and phase transitions in nanostructured metal anodes for electrolysis of cryolite-alumina melts”.

5. References:

[1] Valiev R Z, Zhilyaev A P, Langdon T G 2014 Bulk Nanostructured Materials: Fundamentals and Applications by John Wiley & Sons Inc. 456 p.
[2] Zhilyaev A et al 2001 Microhardness and microstructural evolution in pure nickel during high-pressure torsion Scripta Mater. 44 pp 2753-2758.
[3] Gertsman V, Birringer R, Valiev R, Gleiter H 1994 On the structure and strength ultrafine-grained copper produced by severe plastic deformation Scripta Met. 30 pp 229-234.
[4] Islamgaliev R, Chmelik F, Kuzel R 1997 Thermal stability of submicron grained copper and nickel Mat. Sci. Eng. A 237 pp 43-49.

[5] Ivanisenko Yu, Sergueeva A, Minkow A, Valiev R and Fecht H.-J 2002 Nanomaterials by Severe Plastic Deformation J. Wiley, VCH Weinheim pp 453–458.

[6] Valiev R, Ivanisenko Yu, Rauch E, Baudelet B 1996 Structure and deformation behavior of armko iron subjected to severe plastic deformation Acta Mater. 44 pp 4705–4712.

[7] Ivanisenko Yu, MacLaren I, Sauvage X, Valiev R, Fecht H.-J 2006 Structure, phase composition, and microhardness of carbon steels after high-pressure torsion Acta Mater. 54: 1659.

[8] Korznikov A, Ivanisenko Yu, Laptionok D, Safarov I, Pilyugin V & Valiev R 1994 Influence of Severe Plastic Deformation on Structure and Phase Composition of Carbon Steel Nanostruct. Mater. 4 pp 159-67.

[9] Abdulov R, Valiev R, Krasilnikov N 1990 Formation of submicrometer-grained structure in magnesium alloy due to high plastic strains J. Mater. Sci. Lett. 9 pp 1445–1501.

[10] Furukawa M, Horita Z, Nemoto M, Valiev R, Langdon T 1998 Microhardness measurements and the hall-petch relationship in an Al-Mg alloy with submicrometer grain size Philos Mag. A 78:203.

[11] Valiev R, Krasilnikov N, Tsenev N 1991 Plastic deformation of alloys with submicron-grained structure Mater. Sci. Eng. A 137 pp 35–40.

[12] Zhilyaev A, Langdon T 2008 Using high-pressure torsion for metal processing: Fundamentals and applications Progress in Materials Science 53 pp 893–979.

[13] Stolyarov V, Latysh V, Shundalov V, Islamgaliev R, Valiev R 1997 Influence of severe plastic deformation on aging effect of Al-Zn-Mg-Cu-Zr alloy Mat. Sci. Eng. A 234-236 pp 339-342.

[14] Antipov E, Borzenko A, Denisov F, Filatov A, Ivanov V, Kazakov S, Mazin P, Mazin V, Shtanov V, Simakov D, Tsirlina G, Vasiliev S., Velikodny Yu 2006 Electrochemical behavior of metals and binary alloys in cryolite-alumina melts Light metals 403-408.

[15] Filatov A, Antipov E, Borzenko M, Vasiliev S, Denisov V, Ivanov V, Kazakov S, Kuzminova Z, Laurinavichyute V, Lunin V, Simakov D, Shtanov V 2008 Determination of the integral dissolution rate of the two-phase alloys in cryolite-alumina melts Physical chemistry of surfaces and protection of metals 44 pp 664-668 (in Russian).

[16] Shukhardina S V 1979 Two-component and multi-component systems based on copper ed. Science (in Russian).