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61.43.Gt, 64.75.Nx,
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81.40.Rs**THERMAL STABILITY OF SOLID SOLUTIONS
FORMED BY ULTRASONIC MILLING OF Cu–Co
AND Cu–Fe POWDER MIXTURES**

The structure and magnetic properties of coarse-grained copper-cobalt and copper-iron powder mixtures produced by the ultrasonic milling and annealed at 600 or 800 °C have been studied, by using the X-ray diffraction and magnetometry methods. The treatment of the mixtures in an ultrasonic ball mill is shown to result in the mutual dissolution of metals and the solid solution formation, as well as in a considerably finer mixture structure. The following annealing of those powder mixtures at 600 °C did not change their phase compositions, but substantially affected the size of coherently scattering domains. In the both cases, the latter grew by almost an order of magnitude in agreement with the Lifshitz–Slyosov–Wagner theory. The annealing of the specimens at 800 °C resulted in the absolute decomposition of supersaturated solid solutions formed under the ultrasonic treatment.

Keywords: Cu–Co and Cu–Fe powder mixtures, ultrasonic milling, supersaturated solid solution, structure, magnetic properties.

1. Introduction

Copper-based systems with a restricted solubility of their components – in particular, Cu–Co, Cu–Fe, Cu–Ni–Fe, and others – have unique electric and magnetic properties. They attract a particular interest of physicists and engineers due to a possibility of their application in various domains of science and engineering.

One of the main difficulties for such systems consists in obtaining their supersaturated solid solutions. This task is often solved, by using the methods that include an intensive mechanical action on the treated material, for example, the mechanical alloying or mechanically activated treatment (MAT) in ball mills of various types. Binary Cu–Co and Cu–Fe systems have been an object of numerous researches [1–6], because they are model ones in many aspects [3].

A few following issues are important from the viewpoint of the possibility of the solid solution formation in those systems. First, since the mixture energy for the indicated components is positive, they are char-

acterized by an extremely low mutual solubility even at high temperatures [7]. Second, the initial components do not form chemical compounds with one another. Furthermore, the difference between the plasticity of components does not reach values, at which a “soft” metal could play the role of a “lubricant” and diminish the MAT effect. In the majority of works, their authors analyzed the influence of MAT conditions on the kinetics and the mechanisms of supersaturated solid solution formation. A considerably lower attention was paid to the structure and physical properties of the obtained powders, as well as to their thermal stability.

In work [8], it was shown that when the binary coarse-grained powder mixtures (PMs) of metals Cu–Co and Cu–Fe, which contain elements with weak mutual solubility, are ultrasonically treated in a ball mill, supersaturated solid solutions of copper in iron or cobalt (and iron or cobalt in copper) are formed. It was also found that the ultrasonic milling (USM) of Cu–Co and Cu–Fe powder mixtures results in a considerable fine-graining of the structure and a growth of the dislocation concentration in metal particles. The structure-phase state of powder particles treated by USM is nonequilibrium at room temperatures. Howe-

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ver, it can be made equilibrium by heating the powder, which can also bring about more intensive diffusion processes, structural relaxation, stratification of solid solutions, and phase transformations.

The aim of this work is to determine the temperature interval of stability for the solid solutions formed in Cu–Co and Cu–Fe systems under the conditions of ultrasonic milling. The results of researches concerning the structural and magnetic characteristics of the PMs indicated above, which were treated in an ultrasonic mill and afterward annealed at temperatures of 600 and 800 °C, are reported.

2. Experimental Materials and Methods

In this work, coarse-grained powder mixtures of copper with iron and cobalt were studied. They were prepared from mechanically crushed electrolytic powders of copper (the average particle size $D_{av} \approx 50 \mu\text{m}$), cobalt ($D_{av} \approx 40 \mu\text{m}$), and carbonyl iron ($D_{av} \approx 30 \mu\text{m}$), intensively stirred in distilled water with the help of an ultrasonic disperser UZDN-2T, and dried at room temperature. The mass ratio between the initial cobalt and copper powders in the Cu–Co powder mixture was taken to be 1:2, and between iron and copper in the Cu–Fe powder mixture to be 1:1. The indicated ratios were chosen to make the intensities of lines obtained from element powders in the diffraction patterns identical or close by amplitude to each other, which makes the monitoring of the processes of phase formation and, at the following PM annealing, the stratification more exact and illustrative.

The ultrasonic milling of powder mixtures was performed in an ultrasonic ball mill. The working chamber of a mill was filled with a powder mixture and steel balls. The chamber content was subjected to ultrasonic vibrations. The process of ultrasonic disintegration was carried out together with the imposing of an ac magnetic field 40 kA/m in strength [9, 10]. In works [10–14], it was shown that the combined action of ultrasound and magnetic fields substantially accelerates the kinetics of diffusion processes and solid-phase reactions in treated materials. As a result, the time of the mechanic activation can be substantially reduced in comparison with other kinds of mills. In this work, the ultrasonic milling of Cu–Co and Cu–Fe powder mixtures in ethanol was continued for ten hours at room temperature. The further annealing

was performed in the argon atmosphere at temperatures of 200–500, 600, or 800 °C.

X-ray diffraction researches were carried out on an X-ray diffractometer DRON-3.0, by using the cobalt irradiation. The dimensions of coherently scattering domains (CSDs) were determined from the broadening of X-ray lines in the diffraction patterns with the help of the Selyakov–Scherrer formula [15]. The phase analysis was carried on the basis of the intensity ratio between the most intensive lines of available crystalline phases in the diffraction patterns. The lattice parameters were calculated according to the Wolf–Bragg formula with an error of $\pm 0.00005 \text{ nm}$. Magnetic measurements of the specific saturation magnetizations were carried out with the help of a ballistic magnetometer in a field interval of up to 800 kA/m at room temperature with an error of $\pm 1 \text{ A m}^2/\text{kg}$.

The concentrations of components were calculated from the values of the lattice parameter and the specific saturation magnetizations of solid solutions, proceeding from the fact that, as the first approximation, those parameters for binary solid solutions of metals often depend linearly on the concentrations of their components [16–20]. For instance, the concentrations of components in solid solutions can be calculated by the following formulas, by using the tabulated values for lattice parameters of pure metals and experimentally determined lattice parameters for solid solutions [7, 17]:

$$C_{\text{Co}} = \frac{a - a_{\text{Cu}}}{a_{\text{Co}} - a_{\text{Cu}}} = 1.429 \times 10^4 (a - a_{\text{Cu}}), \quad (1)$$

$$C_{\text{Fe}} = \frac{a - a_{\text{Cu}}}{a_{\text{Fe}} - a_{\text{Cu}}} = 3.155 \times 10^4 (a - a_{\text{Cu}}), \quad (2)$$

where a , a_{Co} , a_{Cu} , and a_{Fe} are the lattice parameters of the alloy, cobalt, copper, and iron, respectively; and C_{Co} and C_{Fe} are the concentrations of cobalt and iron (in at.%) in the solid solutions Cu–Co and Cu–Fe, respectively. The values of lattice parameters were taken from handbook [7].

The process of solid solution formation in ferromagnetic alloys can be monitored by measuring the saturation magnetization σ of the alloys. It is known that, for binary iron, cobalt, and nickel alloys with non-magnetic metals, the parameter σ decreases with increase in the concentration of non-magnetic components. For many alloys—e.g., alloys of nickel with cop-

per, aluminum, silicon, and titanium– σ diminishes linearly [17, 18].

According to the Friedel–Slater–Pauling model [19, 20] for the dependence of the saturation magnetic moment in ferromagnetic metals Co, Fe, or Ni on the concentration of such non-magnetic metals as Al, Si, Ti, V, Zn, and Cu, the specific saturation magnetization σ of iron- or cobalt-based alloys can be described by the formulas:

$$\sigma = \sigma_{\text{Fe}} - 2.2\sigma_{\text{Fe}}C, \quad (3)$$

$$\sigma = \sigma_{\text{Co}} - 2.0\sigma_{\text{Co}}C. \quad (4)$$

Here, C is the alloy concentration; and σ , σ_{Fe} , and σ_{Co} are the specific saturation magnetizations for the alloy, iron, and cobalt, respectively. From formulas (3) and (4), it is easy to determine the concentrations of Fe–Cu alloy,

$$C = 45.5 \frac{\sigma_{\text{Fe}} - \sigma}{\sigma_{\text{Fe}}}, \quad (5)$$

and Co–Cu alloy,

$$C = 50 \frac{\sigma_{\text{Co}} - \sigma}{\sigma_{\text{Co}}}, \quad (6)$$

in at.% units.

3. Experimental Results and Their Discussion

The X-ray diffraction patterns obtained for PMs subjected to USM for ten hours demonstrate no diffraction lines of pure metals Cu, Co, and Fe (Figs. 1, *a* and 2, *a*). Only the weak ($\approx 5\%$) diffraction lines of Co(HDP) and the remnants of the most intensive lines of CoO oxide in the Cu–Co powder mixture (Table 1) and the lines of magnetite Fe_3O_4 in the Cu–Fe powder mixture (Table 2) are observed. The appearance of diffraction lines typical of alloys testifies that the ultrasonic mechanical activation gives rise to the formation of solid metal solutions, which demonstrate a weak mutual dissolution under metallurgical alloying conditions.

The absence of changes in the structure-phase state of solid solutions formed at USM and annealed at 200–500 °C evidences their thermal stability in this temperature interval. The annealing at a temperature of 600 °C for 5, 25, and 125 min also did not result in appreciable modifications of the phase composition

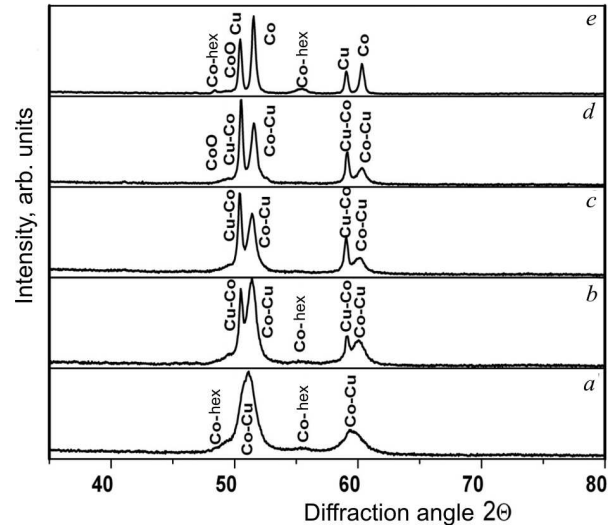


Fig. 1. X-ray diffraction patterns of the Cu–Co powder mixture after USM for 10 h (*a*) and the subsequent annealing at 600 °C for 5 (*b*), 25 (*c*), and 125 min (*d*), and the annealing at 800 °C for 15 min (*e*)

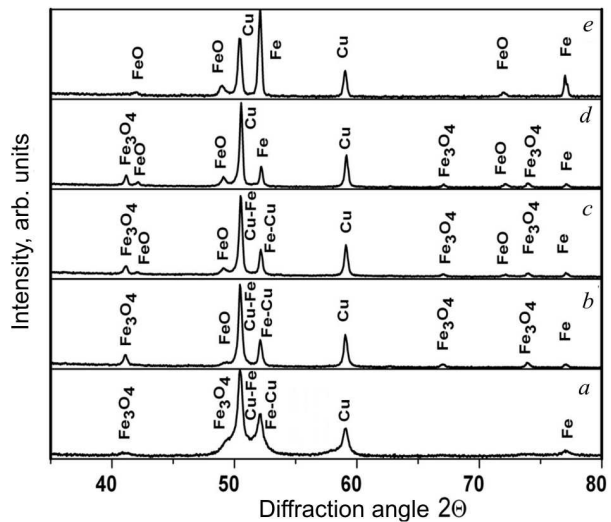


Fig. 2. X-ray diffraction patterns of the Cu–Fe powder mixture after USM for 10 h (*a*) and the subsequent annealing at 600 °C for 5 (*b*), 25 (*c*), and 125 min (*d*), and the annealing at 800 °C for 15 min (*e*)

of the mechanically activated PM (Figs. 1, *b–1*, *d* and 2, *b–2*, *d*), although an insignificant amount of wustite FeO does appear in the Cu–Fe powder mixture (Table 2). At the same time, the annealing at a temperature of 600 °C has much stronger influence on the CSD dimensions. For instance, as the annealing

duration grows, the dimensions of CSDs of particles increase by a factor of nine in the Cu–Co solid solution (Fig. 3), and they become almost eight times as large in the Cu–Fe solid solution (Fig. 4). The parameters of Cu–Co, Co–Cu, and Fe–Cu crystal lattices decrease as the time of annealing increases, whereas the lattice parameter of the Cu–Fe solid solution remains constant (Table 2). Those facts testify to an insignificant reduction in the concentrations of copper, iron, and cobalt impurities in the corresponding solid solutions.

The annealing at a temperature of 800 °C turned out more fruitful. The lines of solid solutions on the basis of copper, iron, and cobalt disappeared from the diffraction patterns. Instead, there appeared the lines of pure metals (Figs. 1 and 2), which testifies to the total decomposition of supersaturated solid solutions in accordance with the diagrams of state for those systems. The indicated processes illustrate the results of calculations of the metal concentration in the solid solutions (Figs. 3 and 4). They were calculated in the framework of a technique that is based on experimentally determined changes in the lattice

parameters of solid solutions and the saturation magnetization by formulas (1)–(6). The results of calculations are quoted in Tables 1 and 2.

It is worth noting that, for the Cu–Fe powder mixture, the lattice parameters of solid solutions, both copper- and iron-based, increase, when iron or copper, respectively, are dissolved in them (Table 2), which agrees with tabulated data [7]. The same situation takes place for the Cu–Co powder mixture: the lattice parameters of solid solutions increase both if copper is dissolved in cobalt and if cobalt is dissolved in copper (Table 1). Evidently, similarly to the case of Cu–Fe powder mixture, this phenomenon is a result of the deviation of lattice parameters from the Vegard rule [7, 15] when the solid solutions are formed.

The results of calculations based on experimentally determined changes of lattice parameters in solid solutions testify that the concentration of metals strongly changes at the annealing of the powder mixtures at a temperature of 800 °C. For instance, according to calculations, the cobalt concentration in the Cu–Co solid solution at its annealing decreases from 14% to 1.5% (Fig. 3), and the copper concen-

Table 1. Phase composition, lattice parameters a , and magnetization σ_S for the Cu–Co powder mixture after USM and the subsequent annealings

Treatment	Components	%	a , nm	σ_S , A m ² /kg
Initial mixture	Co	66.6	0.3545	115
	Cu	33.3	0.3615	–
USM 10 g	Co–Cu	55	0.3573	106
	Cu–Co	40	0.3625	–
	Co(HDP)	5	–	–
	CoO	Remnants	–	–
600 °C 5 min	Co–Cu	53	0.3569	101
	Cu–Co	45	0.3620	–
	Co(HDP)	2	–	–
	CoO	Remnants	–	–
600 °C 25 min	Co–Cu	53	0.3568	100
	Cu–Co	45	0.3618	–
	Co(HDP)	2	–	–
	CoO	Remnants	–	–
600 °C 125 min		53	0.3564	98
	Cu–Co	45	0.3617	–
	CoO	2	–	–
800 °C 15 min	Co	58	0.3547	114
	Cu	37	0.3616	–
	Co(HDP)	3	–	–
	CoO	2	–	–

Table 2. Phase composition, lattice parameters a , and magnetization σ_S for the Cu–Fe powder mixture after USM and the subsequent annealing

Treatment	Components	%	a , nm	σ_S , A m ² /kg
Initial mixture	Cu	52	0.3615	132
	Fe	48	0.2866	–
USM 10 g	Cu–Fe	60	0.3614	52
	Fe–Cu	30	0.2876	–
	Fe ₃ O ₄	10	–	–
600 °C 5 min	Cu–Fe	60	0.3615	68
	Fe–Cu	25	0.2872	–
	Fe ₃ O ₄	10	–	–
	FeO	5	–	–
600 °C 25 min	Cu–Fe	60	0.3616	97
	Fe–Cu	20	0.2870	–
	Fe ₃ O ₄	8	–	–
	FeO	12	–	–
600 °C 125 min	Cu–Fe	60	0.3614	126
	Fe–Cu	20	0.2868	–
	Fe ₃ O ₄	5	–	–
	FeO	15	–	–
800 °C 15 min	Cu	40	0.3615	131
	Fe	50	0.2866	–
	FeO	10	–	–

tration in the Fe–Cu solid solution from 10% to 0% (Fig. 4). At the same time, the copper concentration in the Cu–Co powder mixture, which was determined by magnetic measurements of σ_S , grows from 6% in the initial mixture subjected to the USM to 7.4% after its annealing at 600 °C for 125 s (Fig. 3). On the contrary, when the Cu–Fe powder mixture is annealed, the copper concentration decreases in time from 27.5% to 0.7% (Fig. 4).

The described variations of the iron, cobalt, and copper concentrations in the powder mixture at its annealing qualitatively agree with the results of X-ray diffraction researches, although there is some disagreement between the concentrations determined from X-ray and magnetic data. As was mentioned earlier, this is a result of the approximation character of calculations of the concentration on the basis of the lattice parameter and the solid solution magnetization values. Moreover, a reduction of the specific magnetization in PMs occurs not only owing to the dissolution of copper in iron and cobalt, but also owing to the formation of iron and cobalt oxides (Table 2), which have smaller values of σ_S than pure iron or cobalt.

The interesting information concerning the growth mechanisms for Cu, Co, and Fe particles, when supersaturated solid solutions Cu–Co and Cu–Fe decay, can be obtained, by analyzing the kinetics of variations of CSD dimensions at the PM annealing. From Figs. 3 and 4, one can see that those dimensions grow with the annealing time. According to the Lifshits–Slyozov theory, if the particles have medium sizes, the following relation is obeyed for various mechanisms of mass transfer [21, 22]:

$$R_t^n - R_0^n = Kt, \quad (7)$$

where R_t is the current average radius of particles, R_0 the critical average radius of particles at the given coalescence stage, K the coalescence rate constant, and t the decay time. The quantities n and K depend on the specific diffusion mechanism. For example, $n = 2$ in the case of surface diffusion, $n = 3$ for diffusion in bulk, and $n = 4$ for diffusion over grain boundaries [21, 22].

Figure 5 exhibits the cubic dependences of the average CSD size (R^3) on the annealing time t for the Cu–Fe and Cu–Co powder mixtures. One can see that those dependences have a linear character for both

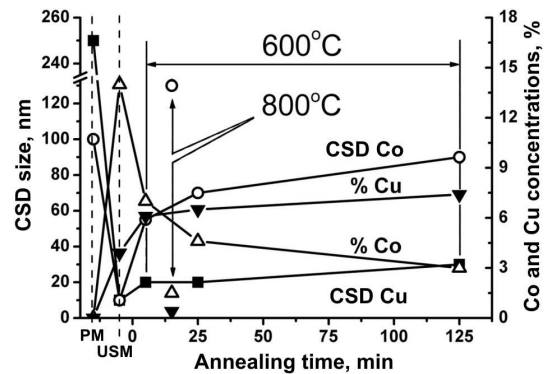


Fig. 3. CSD dimensions and the Co and Cu concentrations in the Cu–Co solid solution after USM and the further annealing at 600 or 800 °C. Vertical dashed lines mark the values for the powder mixture before and after USM

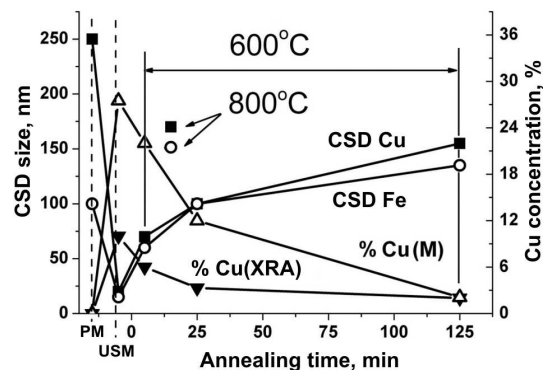


Fig. 4. CSD dimensions and the Cu concentration in the Cu–Fe solid solution after USM and the further annealing at 600 or 800 °C according to magnetic measurements (M) and X-ray analysis (XRA). Vertical dashed lines mark the values for the powder mixture before and after USM

systems: points in the plots corresponding to the CSD dimensions measured at definite annealing times form almost straight lines. This character of the dependences of the average CSD dimension on the annealing time testifies to the bulk diffusion mechanism governing the particle growth. This conclusion agrees with the theory of particle coalescence at the last decay stage of supersaturated solid solutions developed by I.M. Lifshits and V.V. Slyozov [21, 22].

A similar growth kinetics of the CSD dimensions in the (Cu–Co, Fe) powder mixture after USM and the following annealing can be explained as follows. It is known that the diffusion processes in metallic materials and powder mixtures can be driven by several mechanisms: bulk diffusion, diffusion over grain

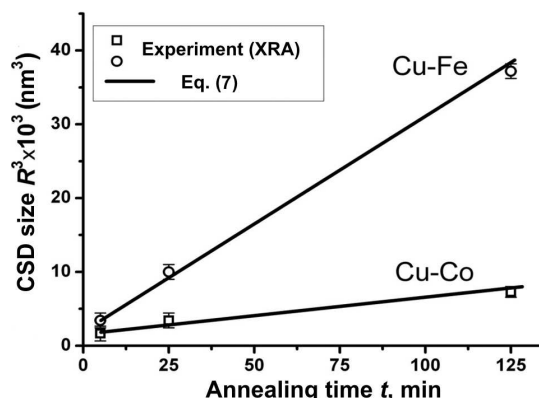


Fig. 5. Dependences of the CSD dimensions (R^3) on the annealing time of the Cu–Fe and Cu–Co powder mixtures at 600 °C

boundaries, surface diffusion, diffusion over dislocations, and so forth. The kinetics of the resulting process will be controlled by the slowest diffusion mechanism [21, 22]. In our case, this is the bulk diffusion of atoms. Therefore, the process of CSD dimension growth (copper, iron, and cobalt particles) can be imagined as follows. At the first stage of the coalescence process, metal atoms move from the internal regions of solid-solution particles to their surface being driven by the bulk diffusion. Afterward, due to the much quicker surface diffusion, they move from smaller particles to bigger ones. As a result, the coalescence process is controlled by the slower bulk diffusion of metal atoms in the particles and, therefore, satisfies the law $R \approx t^{1/3}$ described in works [21, 22].

The plots in Fig. 5 make it possible to determine another important parameter, the critical radius R_0 for particles capable to grow further. Really, from formula (1), one can see that, at $t = 0$, the current radius of particles is equal to the critical radius, i.e. $R_t = R_0$. Hence, the segment intercepted by the plot $R^3(t)$ on the ordinate axis (at $t = 0$) gives the value of the critical radius for a nucleus capable to grow further. Performing this procedure for both alloys, we obtain the following values for the critical nucleus radius: $R_0 = 10$ nm for the Cu–Co alloy and $R_0 = 14$ nm for the Cu–Fe one. As one can see from Figs. 3 and 4, those values coincide with the CSD dimensions in the initial state after the ultrasonic treatment. Hence, the size of particles of the solid solutions remains invariable in the course of USM owing to their intense mechanical crushing. Their structure-

phase state is thermally stable in a temperature interval of 200–600 °C.

4. Conclusions

1. The ultrasonic milling of the Cu–Co and Cu–Fe powder mixtures and the annealing of the obtained solid solutions do not result in the appreciable modification of their phase composition. Only an insignificant content of cobalt and iron oxides was revealed. The annealing has a much larger influence on the CSD dimensions. In particular, the CSD dimensions increase by a factor of nine in the Cu–Co solid solution, as the annealing time grows, and they become almost eight times as large in the case of Cu–Fe solid solution.

2. The CSD dimensions grow together with the annealing time in both Cu–Co and Cu–Fe solid solutions. The growth kinetics is driven by the bulk diffusion of atoms, in accordance with the Lifshitz–Slyozov–Wagner theory.

3. The annealing of supersaturated Cu–Co and Cu–Fe solid solutions, which were obtained with the use of USM, at a temperature of 800 °C gives rise to their total decomposition according to the state diagrams of those systems. After the annealing, the concentrations of metals dissolved in the course of USM vanish.

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ТЕРМІЧНА СТАБІЛЬНІСТЬ ТВЕРДИХ
РОЗЧИНІВ, СФОРМОВАНИХ УЛЬТРАЗВУКОВИМ
РОЗМЕЛЮВАННЯМ ПОРОШКОВИХ
СУМІШЕЙ Cu + Co I Cu + Fe

Резюме

Методами рентгеноструктурного аналізу та магнітометрії досліджено структурні та магнітні характеристики крупнозернистих порошкових сумішей (ПС) міді з залізом або кобальтом, оброблених ультразвуковим розмелюванням і потім віддалених за температур 600 °C та 800 °C. Показано, що механоактиваційна обробка в ультразвуковому кульовому млині спричиняє взаємне розчинення металів і утворення твердих розчинів, а також значне подрібнення структури. Наступний відпал цих ПС Cu + Co та Cu + Fe за температури 600 °C не змінює їх фазового складу, але разом з тим суттєво впливає на розміри ОКР, які в обох сумішах зростають майже на порядок відповідно до теорії Лівшиця–Сльозова–Вагнера. Відпал ПС за температури 800 °C спричиняє повний розпад пересичених твердих розчинів, сформованих ультразвуковою механоактивацією.