

KINETICS OF INTERACTION OF Co–Cu MELTS WITH GRAPHITE AND MICROSTRUCTURE OF FORMING METAL-CARBON COMPOSITIONS

© 2025 O. A. Chikova^{a,*}, I. G. Shirinkina^b, V. S. Tsepelev^a,
N. I. Sinitsin^a, and V. V. Vyukhin^a

^aUral Federal University named after the first President of Russia B.N. Yeltsin,
Ekaterinburg, Russia

^bMikheev Institute of Metal Physics, Ural Branch of the RAS,
Ekaterinburg, Russia

*e-mail: O.A.Chikova@urfu.ru

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Abstract. The time dependences of the contact angle and the wetted surface spot diameter were measured during the interaction of Co–Cu melts with copper contents of 20, 40 and 60 at. % with graphite at temperatures of 1390, 1440, 1490, 1540 and 1590°C. Wetting of graphite by Co–Cu melts does not occur under these conditions: the final contact angle for Co₈₀Cu₂₀ is 95°, Co₆₀Cu₄₀ is 110°, Co₄₀Cu₆₀ is 100°. The final value of the diameter of the wetted surface spot increases somewhat. Metallographic analysis of the microstructure of Co–Cu–C composite materials obtained by contact alloying of Co–Cu melts with carbon showed the dependence of the morphology of the structural components and the phase composition of the samples on the copper content. Composite materials (Co–27%C–10%Cu) + (Co–32%C–62%Cu) + C and (Co–19%C–15%Cu) + (Co–25%C–72%Cu) + C, obtained by the interaction of Co–Cu melts with a copper content of 20, 40 at. % with graphite, have a macro-homogeneous structure.

Keywords: Co–Cu melt, graphite, contact alloying, metal-carbon materials Co–Cu–C, microstructure

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INTRODUCTION

The study of the kinetics of interaction of Co–Cu melts with graphite and microstructure of crystallized samples of Co–Cu–C composite materials is relevant both from the fundamental point of view and from the point of view of practical application. The peculiarity of the phase diagram of the Cu–Co system is the presence of a metastable mixing zone: under sufficiently deep supercooling, the Co–Cu melt splits into two liquids: cobalt-rich and copper-rich [1]. Phase separation occurs due to simultaneous nucleation, coalescence and diffusion [2] and determines the crystallization conditions of Cu–Co alloys [3–7]. The microstructure of crystallized Co–Cu samples contains spherulites of one phase embedded in the matrix of another; spherulites in alloys containing more than ~60 at. % Cu solidify from the Co-based melt, and in alloys containing less than ~40 at. % Cu – from the Cu-based melt [8]. The microstructure of crystallized samples is determined by the morphology, segregation

pattern and size distribution of Co-rich droplets, with droplet migration controlled by the Marangoni force and Stokes force [9–10]. The presence of carbon in the melt, as suggested by the authors, can change the crystallization conditions and lead to the formation of a specific microstructure of crystallized Co–Cu–C samples. The technology of contact alloying of melts Co–Cu with carbon allows to obtain bulk samples of metal-carbon materials Co–Cu–C and is simple enough in technical realization [11]. Earlier the method of contact alloying of Fe–Cu melt with carbon allowed to obtain a unique composite material Fe–60%Cu–C, where iron-based phase is stratified into alloys of pre-eutectic and eutectic compositions (gray and white cast iron) [12–13].

Metal-carbon Co–Cu–C compositions are primarily of interest as materials that absorb microwave radiation [14–15]. A thin film catalyst made of Co–Cu bimetallic alloy has also been developed, which allows the growth of single layer high quality graphene (SLG) [16]. Cobalt electrodes coated with SLG exhibit high magnetic anisotropy [17],

remain resistant to oxidation in air and are promising as spin filtration electrodes [18]. Known methods of obtaining metal-carbon Co–Cu–C compositions: the electrodeposition method [19]; methods when carbide-forming metals are removed from metal carbides by acid etching [20] or chlorination [21], the method of thermal decomposition of the Co–Cu double salicylate complex in argon atmosphere [22], do not allow obtaining bulk samples. In this work another method of obtaining metal-carbon Co–Cu–C compositions – contact carbon doping of Co–Cu melts with copper content of 20, 40 and 60 at. % was used. For the first time temperature and time dependences of the contact angle, diameter of the wetted surface spot were measured, metallographic analysis of the obtained Co–Cu–C samples was carried out. In the future, the results of the study are useful for working out the technology of obtaining granulated Co–Cu–C compositions. Granulated Co–Cu alloys have been intensively investigated since the discovery of the effect of giant magnetoresistance in these materials [23], which determines the ability of Co–Cu–C compositions to absorb microwave radiation [14]. Co–Cu alloys are known to be paramagnetic at Co concentrations less than 25 at. % [24]. Therefore, the interaction of Co–Cu melts with Co content of 20, 40 and 60 at. % with graphite was studied in this work. Obtaining metal-carbon Co–Cu–C compositions by contact alloying is associated with a number of difficulties, since the copper-rich phase is displaced to the surface during separation [25] and prevents the melt from wetting the graphite substrate. Copper in solid and liquid states is in equilibrium with graphite [26]. Liquid copper does not wet carbon substrates [27–30], and graphite is impregnated with copper melts at high temperatures ($>1200^{\circ}\text{C}$) and high overpressure (10–100 MPa) [31]. A slight decrease in overpressure can be achieved by alloying copper melts with surface-active elements (Ti, Mn, Cr, W, Nb, Mo, Co in the amount of 1–2 wt%) [31–33]. Co interacts eutectically with carbon $\text{L} \leftrightarrow (\sigma\text{-Co}) + (\text{C})$ at 1319°C and 11.9 at. % C [26, 34]. Although cobalt melt wets graphite – contact angle decreases from 82 to 35° when heated from 1425 to 1495°C , Co–2.7% C melt at 1495°C does not wet the graphite surface – contact angle 130° , Cu–5% Co melt also does not wet graphite at 1300°C – contact angle 138° [28]. Measurements of the contact angle at interaction of Co–Cu melts with copper content of 20, 40 and 60 at. % with graphite have not been carried out before.

This paper presents relevant data on the kinetics of the interaction of Co–Cu melts with graphite and a description of the microstructure of crystallized samples of Co–Cu–C composites.

1. KINETICS OF INTERACTION OF Co–Cu GRAPHYTIC ARMOSTS WITH GRAPHITE

Determination of graphite wetting angles with Co–Cu melts was carried out according to the standard lying drop technique under conditions of joint heating of

the sample and substrate. Samples for the experiment were preliminary alloyed from pure components in a closed muffle resistance furnace at 1530°C for 60 min. The starting materials were metals of high purity: copper grade B-3 (99.98%) and cobalt grade K1Au (99.35%). The mass of the sample was 4 g. As substrates we used polycrystalline graphite of spectral purity. The porosity of graphite (13.5%) was determined by the technique of measuring the volume of open pores [35]. Substrates from such graphite were carefully polished and ground, then calcined in vacuum (at a pressure below 1 Pa) at 1500°C for 1 h. The surface roughness of the substrate was of the order of $10\text{ }\mu\text{m}$. Fusion of initial samples and measurement of edge angles were carried out in an atmosphere of high-purity helium. The working chamber was pre-vacuumed to 0.001 Pa. Helium was then run to a pressure of $\sim 10^5$ Pa. Wetting angles were measured in the mode of successive step heating from 1420 to 1620°C with holding at each temperature for 20 min. The sufficiency of such holding time is confirmed both by earlier studies [28, 36] and by our studies of the flow velocity of Fe–Cu melts on the graphite surface [13]. The profile of the investigated drop was recorded every minute using a digital camera. The images were recorded in computer memory and analyzed using SIAMS700 software. The wetting angle was determined with an accuracy of $0.3\text{--}0.6^{\circ}$. Signs of melt evaporation, drop volume reduction were not observed. The random error in determining the edge angle did not exceed 3% at a confidence level of 0.95. The method of measuring the angle of wetting of the graphite surface by metal melt (sitting drop method) and the setup for its realization have been previously described in [13, 28, 36–37].

The results of measuring the wetting angles θ of graphite with Co–Cu melts are presented in Fig. 1. Upon heating from 1390 to 1590°C , the final contact angle decreases from 125 to 95° (by 24%) in the experiment with Co80–Cu20 melt; from 138 to 110° (by 20%) in the experiment with Co60–Cu40 melt and from 110 to 100° (by 9%) in the experiment with Co40–Cu60 melt. Thus, the more Cu in the melt, the worse it wets the graphite surface. Note that the value of the contact angle depends little on time and decreases weakly with temperature: in the experiments with the Co80–Cu20 and Co40–Cu60 melts from about 110 to 100° , in the experiment with the Co60–Cu40 melt – from about 130 to 110° .

The results of measuring the diameter d of the spot diameter of the graphite surface wetted with Co–Cu melts are presented in Fig. 2. The spot diameter of the graphite surface wetted by Co–Cu melts significantly increases at the beginning of the interaction at 1390°C , and in the experiments with Co80–Cu20 (by 2%) and Co60–Cu40 (by 7%) melts – during the first 5 minutes, in the experiment with Co40–Cu60 melt (by 3%) during 15 minutes (Fig. 2). The spreading rate of Co–Cu melts at higher temperatures is weakly time dependent and does not significantly increase with temperature. The final value of the spot diameter of the graphite surface

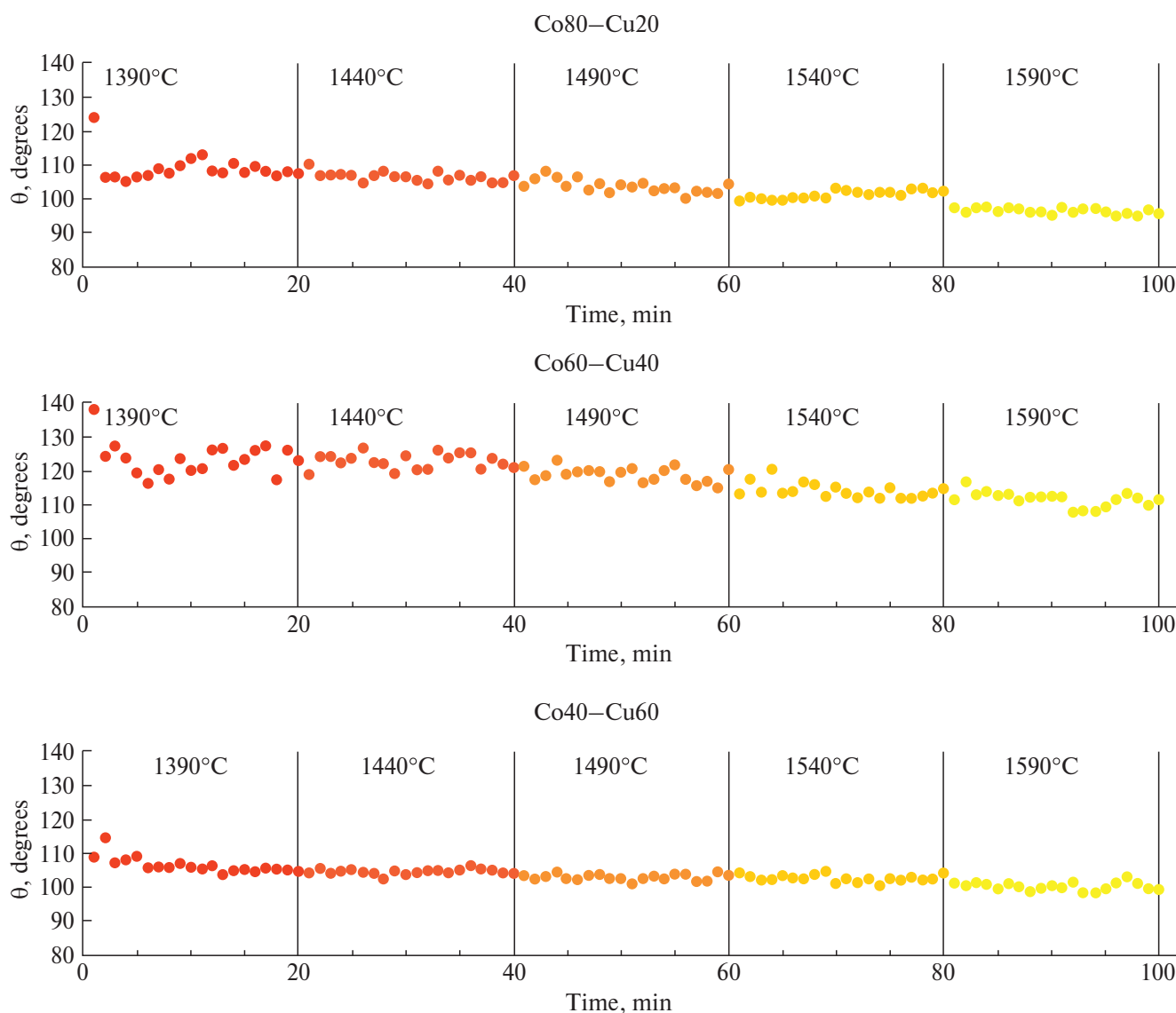


Fig. 1. Time dependences of graphite wetting angles θ with Co–Cu melts with Cu content of 20, 40 and 60 at. % at 1390, 1440, 1490, 1540 and 1590°C. The method of measuring θ is shown in Fig. 2a.

wetted by Co–Cu melts when heated from 1390 to 1590°C increases from 10.4 to 12.0 mm (by 15%) in the Co80–Cu20 melt experiment; from 11.2 to 12.4 mm (by 11%) in the Co60–Cu40 melt experiment from 11.8 to 12.4 mm (by 5%) in the Co40–Cu60 melt experiment (Fig. 2). Thus, the more copper in the melt, the worse it sinks on the graphite surface.

The results of measuring the height h of Co–Cu melt droplet with Cu content of 20, 40 and 60 at. % above the graphite surface are presented in Fig. 3. The change of height h of Co–Cu melt drop above the graphite surface reflects the change of its geometrical dimensions, including due to the formation of a depression on the graphite surface as a result of its interaction with the melt. The height of Co–Cu melt drop above the graphite surface

significantly decreases at the beginning of interaction at 1390°C in the experiments with Co80–Cu20 (by 6%) and Co60–Cu40 melt (by 16%), in the experiment with Co40–Cu60 melt (by 6%) within 15 minutes (Fig. 3). The height h of the Co–Cu melt droplet above the graphite surface at higher temperatures is weakly time dependent and does not significantly decrease with temperature. The final value of the height h of the Co–Cu melt droplet above the graphite surface when heated from 1390 to 1590°C increases from 4.8 to 4.5 mm (by 7%) in the Co80–Cu20 melt experiment; from 5.9 to 5.7 mm (by 4%) in the Co60–Cu40 melt experiment and from 5.1 to 4.9 mm (by 4%) in the Co40–Cu60 melt experiment (Fig. 3). Thus, the more cobalt in the melt, the better it dissolves the graphite surface.

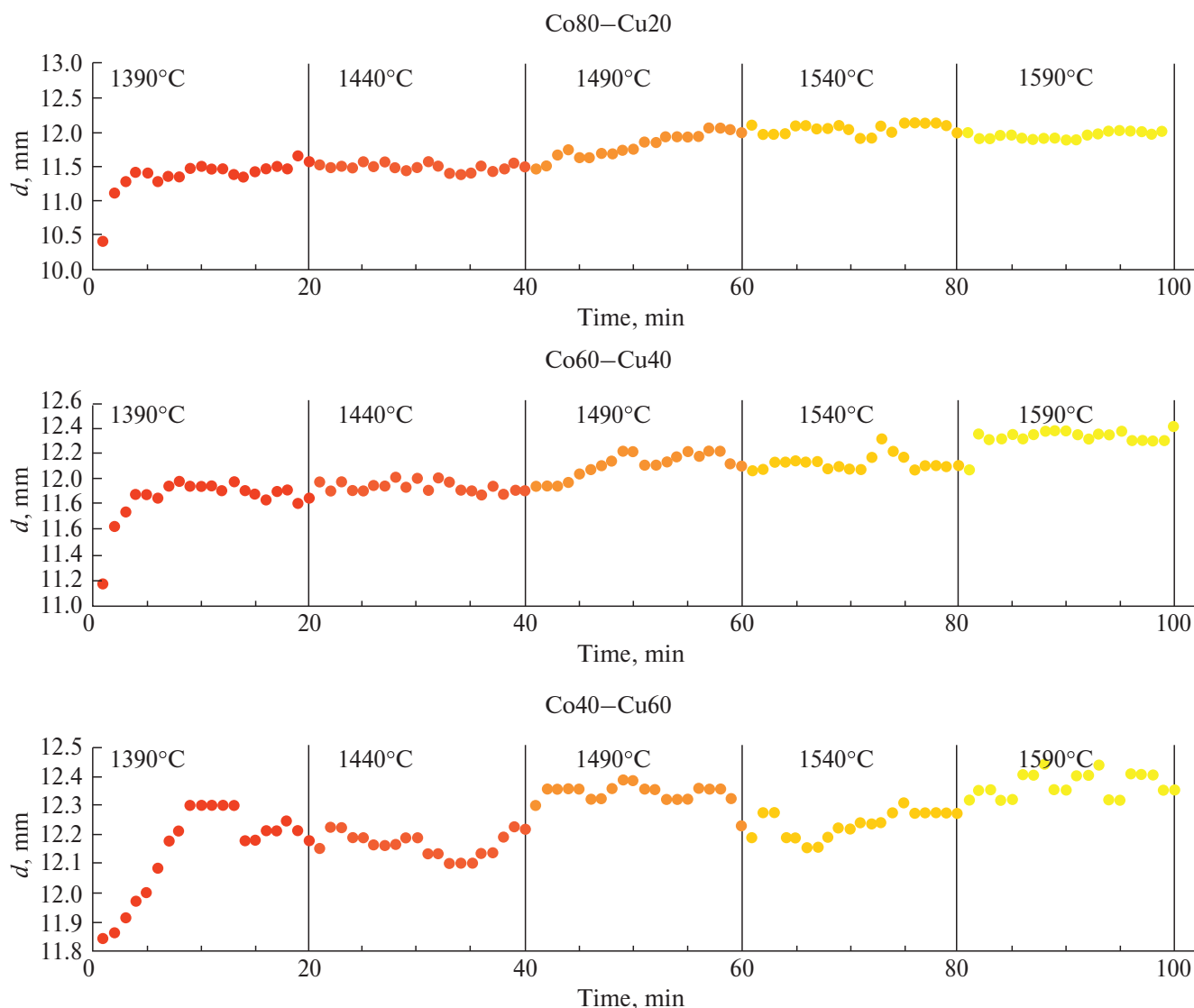


Fig. 2. Time dependences of spot diameter of the graphite surface wetted with Co–Cu melts with 20, 40 and 60 at. % Cu content at 1390, 1440, 1490, 1540 and 1590°C.

Thus, it was possible to continue studies of the kinetics of interaction of Fe–Cu melts delaminating in the liquid state with graphite [13] for the system Co–Cu with a similar type of phase diagram, the peculiarity of which is the presence of a metastable mixing zone; under sufficiently deep supercooling, the Co–Cu melt splits into two liquids: cobalt-rich and copper-rich [1]. It was found that, as for Fe–Cu [13], so for Co–Cu the contact angle and diameter of the wetted surface spot depend weakly on temperature, but there is a significant difference – wetting of graphite by Co–Cu melts with copper content of 20, 40 and 60 at. % at temperatures of 1390, 1440, 1490, 1540 and 1590°C does not occur. Earlier it was noted that pre carburized cobalt Co–2.7%C melt does not wet graphite at 1495°C, contact angle 130° in contrast to Fe–C melts [28].

2. MICROSTRUCTURES OF Co–Cu–C SAMPLES OBTAINED BY CONTACT LAGGING OF Co–Cu ALLOYS WITH HYDROCARBON

Metallographic study of macro and microstructures of Co–Cu–C samples obtained after wetting experiments at cooling rates of 1–10°C/s was performed using a Neophot-32 light microscope. Fine structure studies and local microanalysis were performed on a Tescan MIRA scanning electron microscope with an energy dispersive analysis (EDS) attachment. The samples cut out of the alloy in longitudinal section were ground on grinding paper with different grits, from P80 to P2500. Then polishing was carried out on aqueous chromium oxide solution and colloidal silicon suspension.

The general view of the samples is shown in Fig. 4. By appearance they can be divided into two groups:

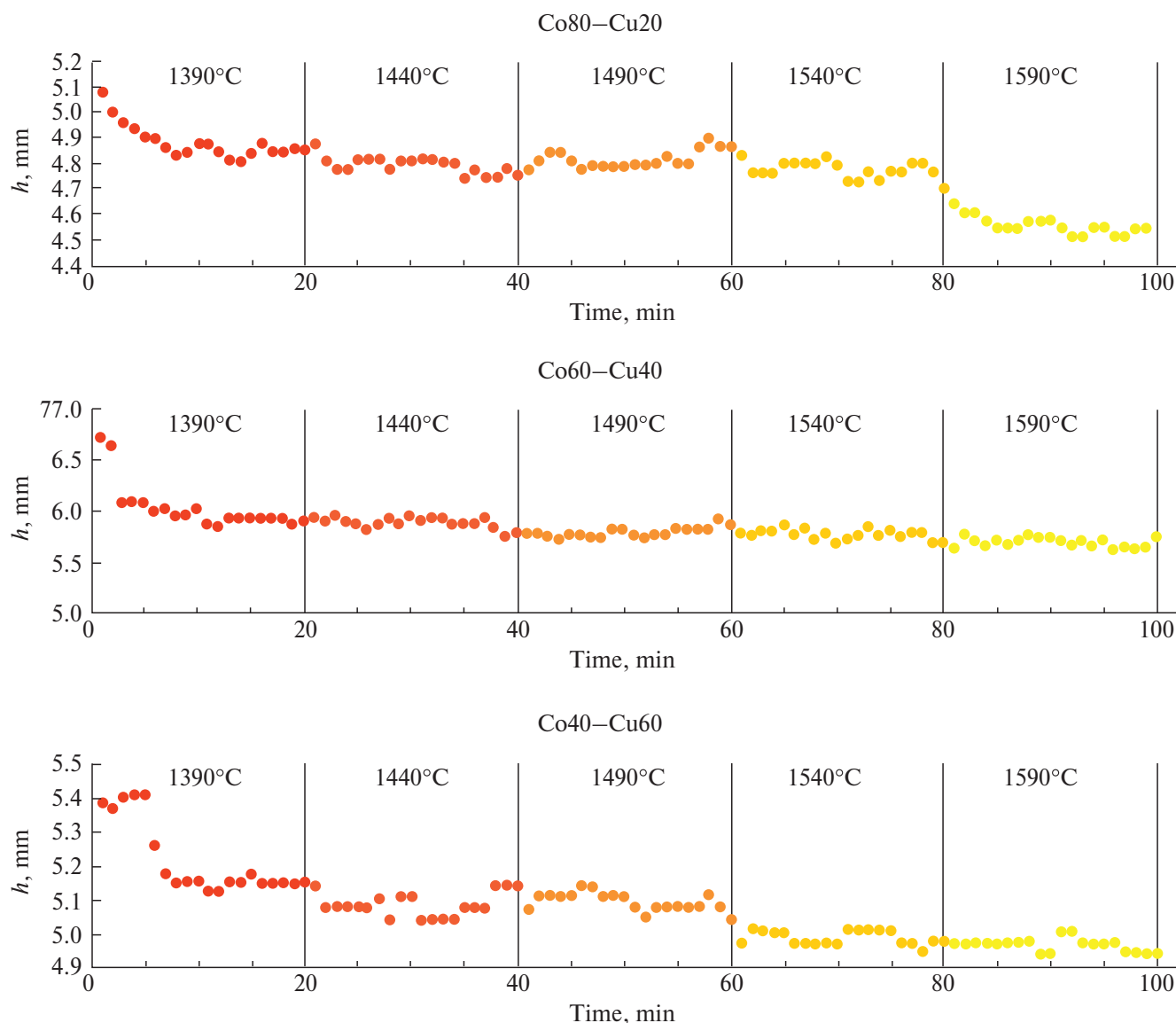


Fig. 3. Time dependences of the drop height h of Co–Cu melt with Cu content of 20, 40 and 60 at. % at 1390, 1440, 1490, 1490, 1540 and 1590°C relative to the graphite surface.

rich in cobalt (Co–20 at. % Cu–C and Co–40 at. % Cu–C) and copper (Co–60 at. % Cu–C). Both groups are characterized by melt stratification with displacement of the copper phase to the periphery of the sample. For samples of the first group there is isolation of the copper phase, and for samples of the second group – the phase rich in cobalt. Previously, phase reversal at the transition through equiatomic composition has been reported for Fe–Cu melts during graphite wetting [12–13]. The stratification of Co–60 at. % Cu–C sample occurs in the whole volume (Fig. 4c). The alloys Co–20 at. % Cu–C and Co–40 at. % Cu–C are characterized by a rather homogeneous structure. Earlier by the method of contact alloying of Fe–Cu melt with carbon a similar result was obtained: delamination of Fe–60%Cu–C sample

occurred in the whole volume [12–13]. Consequently, obtaining bulk macro homogeneous samples of metal–carbon materials Co–Cu–C by the technology of contact alloying of Co–Cu melts with carbon at low cooling rates (1–10°C/s) is possible with excess of cobalt in the melt.

As follows from the structural analysis, the increase of copper content from 20 to 60% leads to significant changes in the morphology of structural components and phase composition of samples after wetting experiments. The interaction of carbon with cobalt and cobalt with copper according to the state diagrams, Co–C and Co–Cu [1, 26, 34], causes the formation of complex multiphase structures.

The main structural component in the metal–carbon material Co–20%Cu–C is Co–C eutectic and primary

carbon crystals, i.e. in the process of carbon diffusion from the substrate into the Co phase, its concentration becomes higher than the eutectic one, and the alloy solidifies according to the hypereutectic type. Co–C eutectic structure belongs to the category of disordered eutectics of plate-needle morphology. Primary carbon crystals have the form of thin plates, the length of which reaches 100–300 μm (Fig. 5a). Rounded copper zones of rounded shape and ~ 10 –50 μm in size are formed throughout the sample volume (to a greater extent along the periphery). Most often they are located near the carbon plates (Fig. 5a, b).

The formation of Cu phase occurs by peritectic reaction from Co-based solid solution and Cu-enriched melt. The presence of Co–C eutectic leads to the fact that the nucleation of copper phase is heterogeneous on the interfacial surface of particles of carbon and Co solid solution, which creates the effect of two-phase composite particles C/Cu. The structure of the Co–40%Cu–C metal-carbon sample, in contrast to the Co–20%Cu sample, is very heterogeneous (Fig. 6b). A ~ 300 –350 μm thick copper layer is located near the substrate. Further up to the middle of the sample and above extends an extensive

zone enriched with cobalt, within which a copper-based phase is located in the form of irregularly shaped zones (grains). Within this phase are cobalt crystals of dendritic morphology. Fig. 6a shows the boundary of Co and Cu zones and Co dendrites in the Cu zone.

The copper layer located near the substrate is also biphasic, the Co alloy dendrites in it grow perpendicular to the graphite substrate (Fig. 6b). Carbon in the form of thin needle-shaped crystals is present throughout the sample volume, however, its amount in the cobalt-based zone is much less than in the copper zone (Fig. 6a).

In the Co–60%Cu–C sample, an opposite set of structures is observed, in which the volume ratio of cobalt and copper zones changes in favor of the latter (Fig. 4c). Most of the sample has a eutectoid-type structure consisting of a copper base and cobalt dendrites (Fig. 7a).

At the substrate (Fig. 7a) and along the periphery of the sample, isolated zones of Co–C eutectic with inclusions of copper phase are formed (Fig. 7b). In these zones, the tendency of joint nucleation and growth of Cu and C phases is clearly manifested.

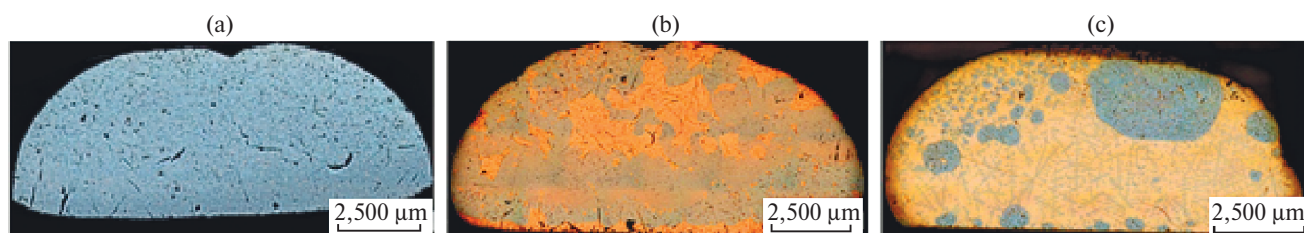


Fig. 4 General view of Co–Cu–C samples: a) Co–20%Cu–C; b) Co–40%Cu–C; c) Co–60%Cu–C.

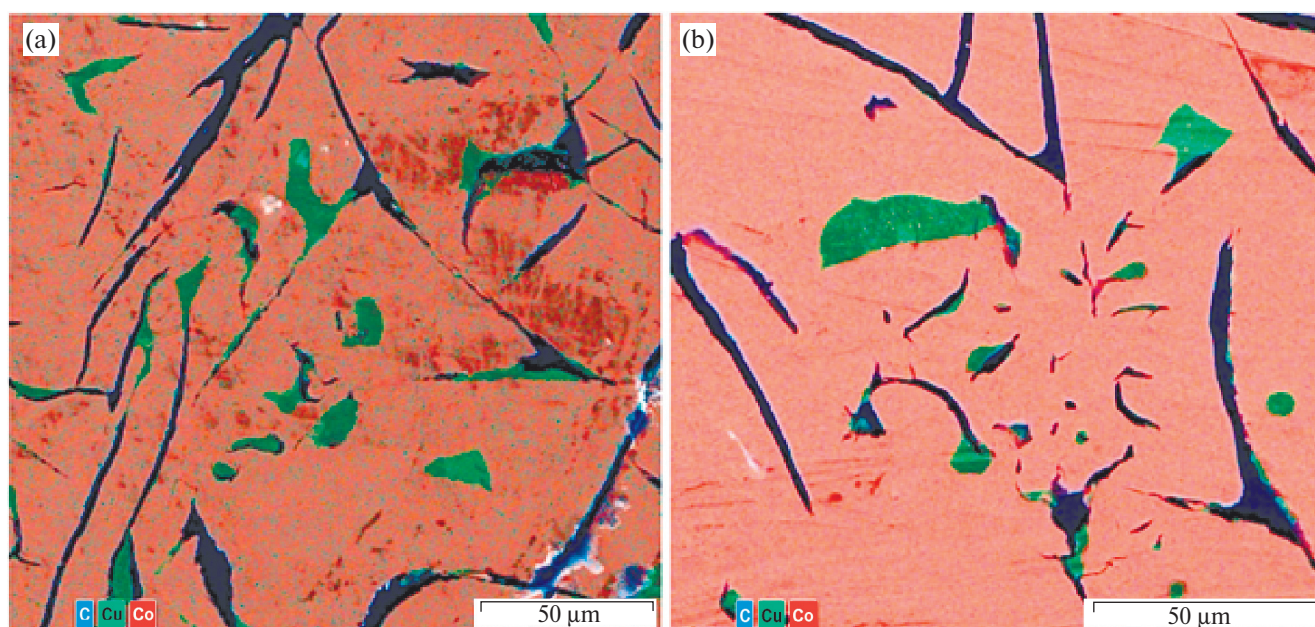


Fig. 5. Structure of Co–20%Cu–C metal-carbon material: a) Co–C eutectic; b) copper zones in Co–C eutectic and EDS map of element distribution (SEM).

Thus, on the basis of the conducted studies it is shown that at the transition through the equiatomic composition there is a change in the composition and volume fraction of isolated zones. In the samples of metal-carbon material Co–20%Cu–C and Co–40%Cu–C the isolated zones are enriched with copper, and in the sample of metal-carbon material Co–60%Cu–C – with cobalt. Previously, a similar phase reversal at the transition through the

equiatomic composition was found for melts of the Fe–Cu system [12–13].

The kinetics of diffusion dissolution of carbon depends on the composition of the initial alloy. From the data [28] it is known that carbon does not diffuse through the copper phase, so in experiments on wetting the graphite substrate Co–Cu melts, its dissolution is carried out in the phase rich in cobalt. It is found

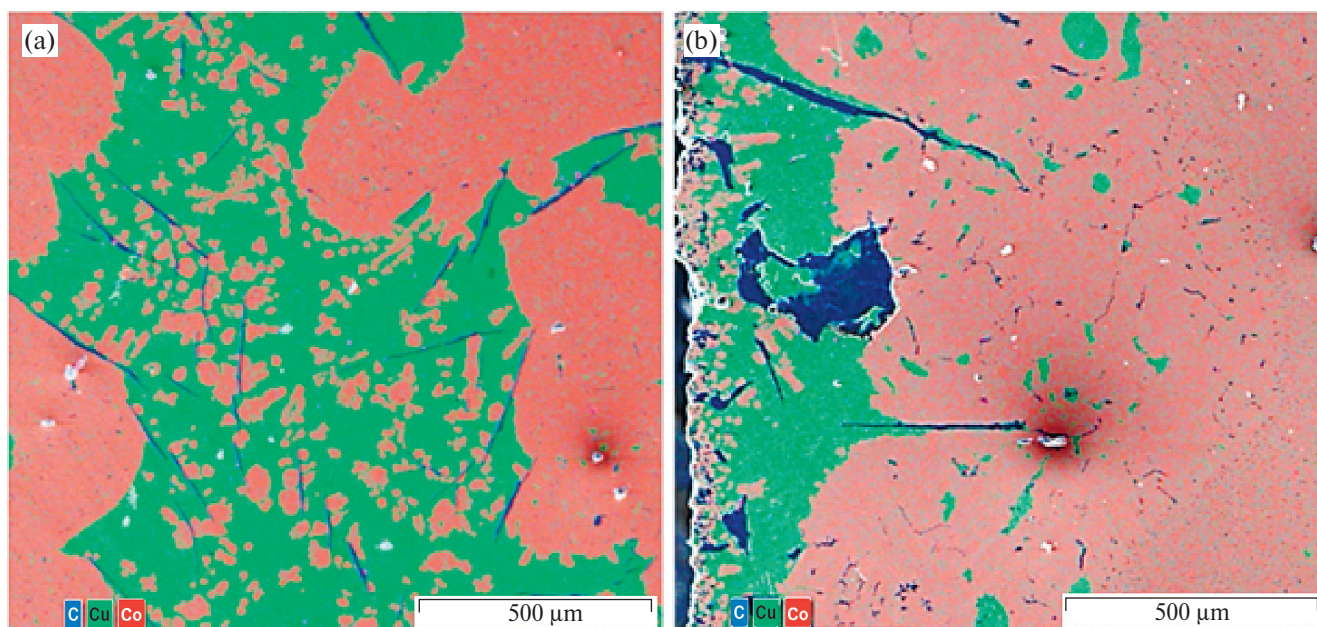


Fig. 6. Structure of metal-carbon Co–40%Cu–C material: a) boundary of Co and Cu zones and Co dendrites in Cu zone; b) area at the contact point of the sample with graphite substrate; EDS maps of element distribution (SEM).

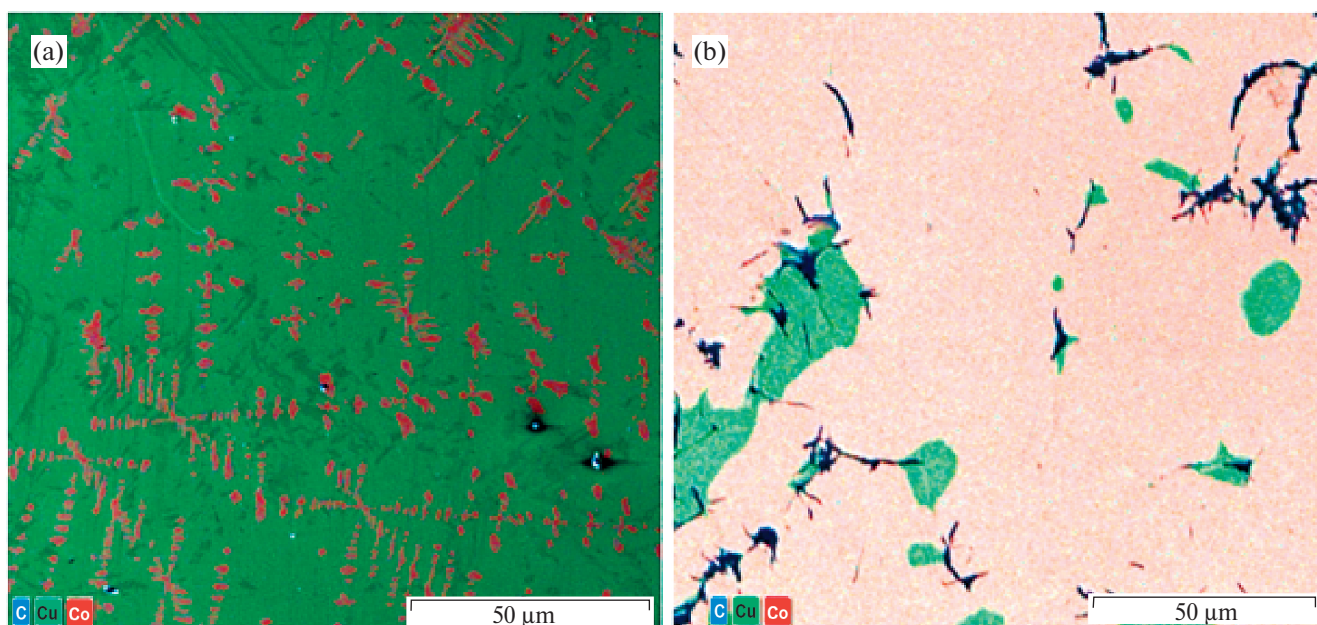


Fig. 7. Structure of Co–20%Cu–C metal-carbon material: a) Cu zone with Co dendrites; b) isolated Co–C eutectic zones with copper phase inclusions, EDS maps of element distribution (SEM).

how the carbon content of metal-carbon Co–Cu–C materials varies with the cobalt content of the initial alloy. According to EDS-analysis data, contact alloying of Co–20%Cu melts with carbon resulted in the composite material (Co–27%C–10%Cu) + (Co–32%C–62%Cu) + C (Fig. 5a), Co–40%Cu – (Co–19%C–15%Cu) + (Co–25%C–72%Cu) + C melt (Fig. 6a), Co–60%Cu – (Co–20%C–15%Cu) + (Co–23%C–74%Cu) + C melt (Fig. 7a).

Despite the above-mentioned differences in the structure formation of metal-carbon Co–Cu–C materials with different copper content, it is possible to note the features common to all compositions. In particular, it is a change in the geometry of the contact zone of the substrate with the melt, which is manifested in the destruction of the graphite substrate. Changes in the geometry of the contact zone of solid and liquid metal – crater formation and dispersion of graphite were found. Crater formation is a sign of intensive dissolution of carbon in the melt. Graphite dispersion accompanies the process of phase infiltration of the melt through the pores. The destruction of the graphite substrate is most pronounced for the samples rich in cobalt: Co–20%Cu–C and Co–40%Cu–C (Fig. 6b), which agrees with the data of measuring the height h of the Co–Cu melt drop with copper content of 20, 40 and 60 at. % above the graphite surface (Fig. 3). The final value of h height at heating from 1390 to 1590°C increases by 7% in the Co80–Cu20 melt experiment; by 4% in the Co60–Cu40 melt experiment and by 4% in the Co40–Cu60 melt experiment (Fig. 2). Thus, the more cobalt in the melt, the better it dissolves the graphite surface.

Thus, by methods of light optics, scanning electron microscopy and local X-ray spectral analysis the following regularities of structure formation of metal-carbon Co–Cu–C compositions obtained as a result of wetting graphite with Co–Cu melts have been established: (1) increase of copper content from 20 to 60 at. % leads to changes in morphology of structural components and phase composition of samples; (2) at transition through equiatomic composition the ratio of volume fraction of phases changes, i.e. in Co–20%Cu–C and Co–40%Cu–C samples the copper phase is isolated, and in Co–60%Cu–C sample – the cobalt-based phase; (3) due to intensive dissolution of carbon in cobalt, the cobalt-rich phase has the structure of Co–C eutectic; (4) interaction of cobalt and copper leads to the formation of a mixture of two solid solutions of eutectoid type, taking into account the weak mutual solubility of the components, it can be considered that the eutectoid consists of pure Cu and pure Co; (5) as a result of interaction of graphite substrate with Co–Cu melts, its destruction is observed.

CONCLUSION

Studies of kinetics of interaction of Co–Cu melts with copper content of 20, 40 and 60 at. % with graphite

were carried out by measuring time dependences of contact angle and diameter of the wetted surface spot at temperatures of 1390, 1440, 1490, 1540 and 1590°C. Wetting of graphite by Co–Cu melts does not occur under these conditions: the final contact angle for Co80–Cu20 is 95°, Co60–Cu40 – 110°, Co40–Cu60 – 100°. The spot diameter of the wetted surface increases significantly only at the beginning of the interaction within 5–10 min. The final value of the spot diameter of the wetted surface increases for Co80–Cu20 by 15%, Co60–Cu40 by 11%, and Co40–Cu60 5% when heated from 1390 to 1590°C.

Metallographic analysis of the microstructure of Co–Cu–C composite materials obtained by contact alloying of Co–Cu melts with carbon showed the dependence of the morphology of structural components and phase composition of samples on the copper content; macrohomogeneous samples were obtained with excess of cobalt melt; as a result of interaction of graphite substrate with Co–Cu melts, its destruction is observed.

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ETHICS DECLARATION

There are no human or animal studies in this paper.

CONFLICT OF INTERESTS

The authors of this paper declare that they have no conflict of interests.

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