Powder Metallurgy and Metal Ceramics, Vol. 35, Nos. 7-8, 1996

CONTACT INTERACTION, STRUCTURE, AND PROPERTIES OF W(Mo, Cr)–Cu COMPOSITES WITH ADDITIVES

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UDC 621.3:669.018:669"27"28"26:669.3

The results of research on the effect of additives on the adhesive characteristics and phase-boundary formation in W(Mo, Cr)–Cu systems are summarized. The distinctive features of structure formation and the properties of powder composites compacted in the presence of a liquid phase have been studied. Composites with optimal properties (high-temperature hardness, electrical conductivity, high adhesiveness, and stable structure) have been identified.

In their quest for ways of solving problems of modern materials science researchers often turn to the potentialities of composites. By choosing the necessary components, determining their content, and determining the conditions of formation, we can obtain materials and products made of them with the required properties. This is how pseudoalloys of the W–Cu system, which are well known as electrical and structural materials, were produced. The present-day requirements imposed on those materials and the scarcity of tungsten propose either finding new materials based on elements of a different physicochemical nature or improving the properties of composite powder W–Cu pseudoalloys. The most effective method of strengthening the components and boundaries in such composites is by doping [1, 2]. When the above is taken into account, it is possible to define a number of other problems, the solution of which will contribute to the successful development of W(Mo, Cr)–Cu composites and will ensure the functional properties of high-current contacts and electrodes. Such materials are obtained primarily by impregnation [3]. Their properties depend on the volume fraction of the strong phase, the ratio of the properties of the high-melting and low-melting components, and the bond strength at their boundary [1]. A positive spreading coefficient is a necessary condition for attaining a perfect metal contact and maximum bond strength at the phase boundary in technological operations of impregnation and sintering with the participation of a liquid phase [4]. In the presence of oxides and other impurities the properties of the boundary deteriorate and the strength decreases. For example, the adhesion strength under shear at the boundary between tungsten and spectrally pure copper decreases from 196 to 32 MPa when a layer of oxides is present [5]. The decrease in bond strength at the phase boundary entails a drop in electrical conductivity, strength characteristics, etc.

This paper reports the results of studies on the physicochemical conditions of contact interaction of condensed phases in W(Mo, Cr)–Cu systems with various additives. The data concerning the salient features of structure formation and some properties of powder composites of the same systems were obtained with the participation of a liquid phase. The content of the high-melting and low-melting components was determined in accordance with the conditions under which the strongest adhesion bond arose between the particles and the disperse system; the capillary forces were estimated by the method of [6] on a computer.

Methods that can give complementary results were used to solve the problems formulated here. A variation of the quiescent drop method (with separate heating of the objects [4]) was used to study the influence that additives introduced into the low-melting component of the contact couple have on the spreading kinetics, the adhesion characteristics, and the formation of a phase boundary between the high-melting component and the melt. Methods of optical and scanning electron microscopy, Auger spectroscopy, x-ray phase and x-ray structural analyses, and x-ray spectral microanalysis were used to detect distinctive features in the formation of the phase boundary and the structure of the composites as a whole and to identify and determine the composition of the phases at the phase boundary and inside the components of the contact couple.

Institute of Problems in Material Science, National Academy of Sciences of Ukraine, Kiev. Translated from Poroshkovaya Metallurgiya, Nos. 7-8, pp. 46-57, July-August, 1996.

The mechanical properties (microhardness and high-temperature hardness [7, 8], short-term high-temperature tensile strength, and bending strength and ductility in a three-point loading scheme [7] in the temperature range from −196 to 800°C) and the physical properties (electrical resistivity [9]) were measured. The oxidation resistance was studied by the procedure described in [10].

The structure and phase composition of the transition zone at the phase boundary that forms as a drop of melt spreads at the temperature of the experiment and during subsequent cooling of the contact couple were studied. At the same we studied the structure and phase composition of the boundaries of the components of the composites, which were made by impregnating porous tungsten, molybdenum, or chromium compacts with copper or a melt of copper and an alloying element (the content of additives was determined with allowance for the results of experiments on spreading) by the method described in [11-13]. The content of the low-melting component in the composite ranged from 30 to 60 vol.%. Silver, titanium, zirconium, and cobalt, i.e., elements with limited solubility in copper, were used as the alloying elements and nickel, which forms an unlimited series of solid solutions with copper, was used for comparison [14].

The System W(Mo) – Cu – Alloying Element. In spreading and wetting experiments it was established that additives improve the adhesion characteristics in the system consisting of a high-melting (W, Mo) substrate and copper [15]. The introduction of nickel and cobalt into the melt increases the work of adhesion (lowers the contact wetting angle) in the Mo–Cu system [16]. The quasi-equilibrium angle is smallest when molybdenum is wetted with a Cu–Co melt containing more than 2 mass% Co and with a Cu–Ni melt containing more than 7 mass% Ni. This was the content of the given additives in the low-melting component of the Mo–(Cu + Ni(Co)) composite.

The results of qualitative x-ray spectral microanalysis of the transition zone at the phase boundary, which forms when a drop of melt spreads at 1200°C and cools to room temperature, showed that nickel migrates in molybdenum by diffusion along grain boundaries while under the same conditions cobalt concentrates at the phase boundary, forming a new phase. After prolonged holding this phase corresponds to the intermetallic compound Mo_6Co_7 [14]. Under the conditions of the experiment, a layer with a variable cobalt content is formed when the isothermal holding time is 1 min.

The additives, first, improve the adhesion characteristics of a system consisting of the high-melting component of a pseudoalloy and an alloy and, second, increase the heat resistance and other properties of the composite [15, 16]. After an analysis of the variation of the interfacial energies during spreading and the nature of the interaction of the additives introduced along with the tungsten and molybdenum [17] it was found that the additives (with the exception of silver) are interfacially active at the interface between the melt and the high-melting substrate of the contact couple. The results of x-ray spectral microanalysis confirmed that the boundary regions are enriched with those elements [11]. For example, the silver enrichment of the boundaries of the main components in the W–(Cu + Ag) composite [17] evidently can be attributed to processes similar in nature to chemical adsorption. This explanation is in accord with the known information about internal intercrystalline adsorption of silver in silver–copper binary alloys [18]. As was to be expected, by increasing the work of adhesion at the indicated boundaries by dissolution in copper and the high-melting component of the composite, the additives affect the nature of the distribution of elements in the main components, the structure, and the properties.

The structure of pseudoalloys is formed during all the operations of the technological process. In the finishing stage (during sintering in the presence of the liquid phase) the type of structure of the composite, the grain size of the components, their distribution, etc. are due to the processes of zonal liberation, recrystallization, coalescence, and liquid-phase recrystallization. A characteristic feature of those processes is that conglomerates of large single-crystal grains form and grains of the high-melting component are spheroidized (this can occur both with and without alloying elements being present).

Alloying with nickel somewhat increases the grain size of the high-melting component. The possibility of such growth, obviously, is determined by the increase in the degree of interaction in the W–Ni–Cu system, the acceleration of mass transfer in the solid phase and in the presence of the liquid phase, and the attendant recrystallization, coalescence, and recrystallization through the liquid phase. The grain size is not observed to change appreciably when silver is introduced. Like nickel, silver is redistributed in the copper, forming a solid solution. The phase composition of the composite alloyed with titanium and zirconium depends on the content of those elements in the copper. For example, the low-melting component of a composite with 3 mass% Zr consists of a solid solution and individual eutectic colonies, the number of which increases with the zirconium concentration [16]. In a composite containing 7 mass% Zr the low-melting component has a eutectic structure. Microdurometric analysis and x-ray spectral microanalysis showed that an α-solid solution of zirconium and tungsten in copper as well as the intermetallic compound Cu_5Zr, which crystallizes as needles, enters into the composition of the eutectic [11]. In the case of alloying with titanium the low-melting component of the composite contains an α-solid solution of titanium and tungsten in copper as well as the intermetallic compound Cu_4Ti, which precipitates at the phase boundary [11, 16].

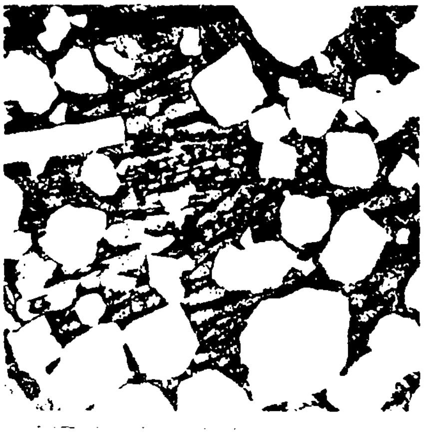

Fig. 1

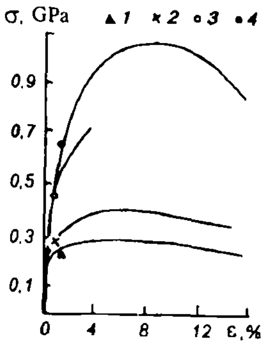

Fig. 2

Fig. 1. Structure of a W–Cu–Zr composite. ×300.

Fig. 2. Stress–strain diagram obtained from bending tests (T = 400°C) on pseudoalloys with additives: W–Cu (1), W–Cu + Ni (2), W–Cu + Ti (3), and W–Cu + Ti + REM (4).

The introduction of zirconium and titanium into the composite results in some tungsten grain growth. Those elements evidently cause a redistribution of the interstitial impurities and cleanse the phase boundaries, thus increasing the probability that grains of the high-melting component in the composite will coalesce. The structure of composites with additions of yttrium and rare-earth metals (REMs) is fine-grained, the grains of the high-melting component are round, and the low-melting component is a solid solution with inclusions of intermetallic compounds and complex oxides [16].

Nickel and cobalt in Mo–Cu composites cause grains of the high-melting component to grow. How the stock is prepared affects the grain size and the grain distribution by size. The structure of composites from stock that consists of a mechanical mixture of powders of pure metals is characterized by a large average grain size (15 μm) and a smeared grain size spectrum. The grain size of the high-melting component in composites obtained from a mixture of oxygen-containing compounds of the components, which are reduced at the same time, decreases to 5 μm.

The structure of the low-melting component was studied by conventional methods of optical and scanning electron microscopy as well by an original method of x-ray spectral analysis [19]. It was found that the powder composites of the W–Cu–Ni system with a tungsten content of 50-70 mass% (40-60 vol.% of the high-melting component based on tungsten), obtained by impregnation and liquid-phase sintering, consist of grain colonies. Those colonies consist of grains of the low-melting component (ageing solid solution) with high-melting component inclusions dispersed in it. The fact that the W–Cu–Zr composite has such a structure is indicated by the direction of crystallization of the acicular precipitates of Cu_5Zr, which changes in the transition from one grain of a colony of the low-melting component to another (Fig. 1).

The size of a grain colony of the copper-based component in the Mo–Cu–Ni(Co) composite, obtained by impregnation, lies in the range 110-250 μm. With an average high-melting component grain size of 5-15 μm (the value depends on how the stock was prepared) the low-melting component grain can be represented as a matrix, in which the grains of the molybdenum-based structural component are dispersed. The phase composition of a Mo–Cu composite alloyed with nickel and cobalt is represented by three solid solutions based on copper and molybdenum and a small content of cobalt-based precipitate phases and low-symmetry phases, molybdates of the type $(Cu, Ni, Co)_xMo_yO_z$.

The formation of a matrix-type structure (lack of a rigid framework of the molybdenum-based high-melting component) does not contradict the observed distribution of high-melting grains in colony grains and does not rule out their forming chains and conglomerates in the moving disperse system. Chains and conglomerates can form and exist, breaking apart and forming again under the fluctuations of the capillary forces, which are caused by the variation of the principal parameters of the system during the formation of the material [12]. The parameters studied (i.e., the particle size and shape, the amount of liquid phase, the ratio of the surface energies at the phase boundaries, the wetting angle, etc.) vary between wide limits, depending on the conditions of the technological processing and the quality and history of the initial materials.

TABLE 1. Temperature Dependence of the Short-Term Strength of Composites under Tension (MPa)

Material	Temperature, °C		
	400	600	800
Mo—Cu—2Ni	351	302	216
Mo—Cu—6Ni	400	380	240
Mo—Cu—2Co	516	472	380
Mo—Cu—6Co	520	512	312

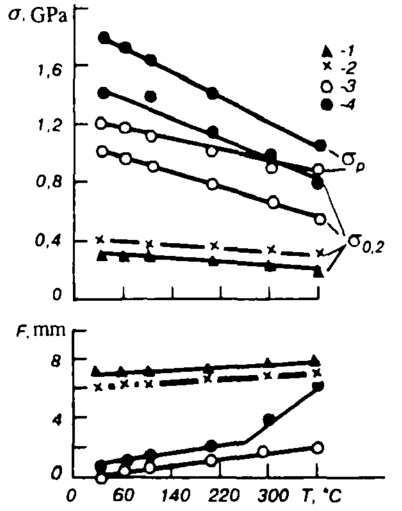

Fig. 3

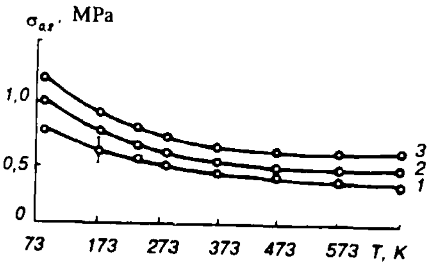

Fig. 4

Fig. 3. Temperature dependence of mechanical properties of pseudoalloys with additives W–Cu (1), W–Cu + Ni (2), W–Cu + Ti (3), and W–Cu + Ti + REM (4) in bending tests.

Fig. 4. Temperature dependence of the yield stress of the composites Mo–Cu (1), Mo–Cu–Ni (2), and Mo–Cu–Co (3) in bending tests.

The change in the phase composition and the structure of the composite as the result of alloying correlates with the level of the mechanical properties and the distinctive features of the fracture in bending tests in the temperature range 77-673 K. Analysis of the stress–strain diagrams (Fig. 2) obtained in bending tests showed that the introduction of additives to the W–Cu composite increases the ordinates of the entire curve, causes its slope to grow in the elastic region, raises the yield stress, and increases the strain hardening to fracture. The influence of the alloying elements intensifies on passage from nickel (which forms a series of solid solutions with copper) to titanium, zirconium, and rare-earth metals (which have limited solubility in copper) [14]. The conditional yield stress of the W–Cu and W–Cu–Ni composites is almost independent of the temperature (Fig. 3). The ordinates of the stress-strain curves were also observed to increase when nickel and cobalt were added to Mo–Cu composites [12]. The degree of influence that the alloying elements have intensifies on going from nickel to cobalt (Fig. 4). The experimental finding that the yield stress $\sigma_{0.2}$ of composites based on the W(Mo)–Cu systems has only a weak temperature dependence becomes understandable if we consider that the temperature of the tests has the weakest influence on the yield stress of copper [20]. The yield stress of the other fcc and hcp metals increases by 20% when the temperature of the tests is lowered from room temperature to 77 K, while under the same conditions the $\sigma_{0.2}$ of bcc metals increases 4-10 times [21]. The mechanical properties of W(Mo)–Cu composites, therefore, are determined to a considerable degree by the component with the fcc lattice, as is confirmed by the existence of a matrix-type structure and the lack of a rigid framework of the high-melting component based on tungsten (molybdenum).

The increase in $\sigma_{0.2}$ with the temperature, as observed in the region of low values, indicates that the additives introduced affect the properties of the matrix, a copper-based solid solution. The interrelation between the temperature, the

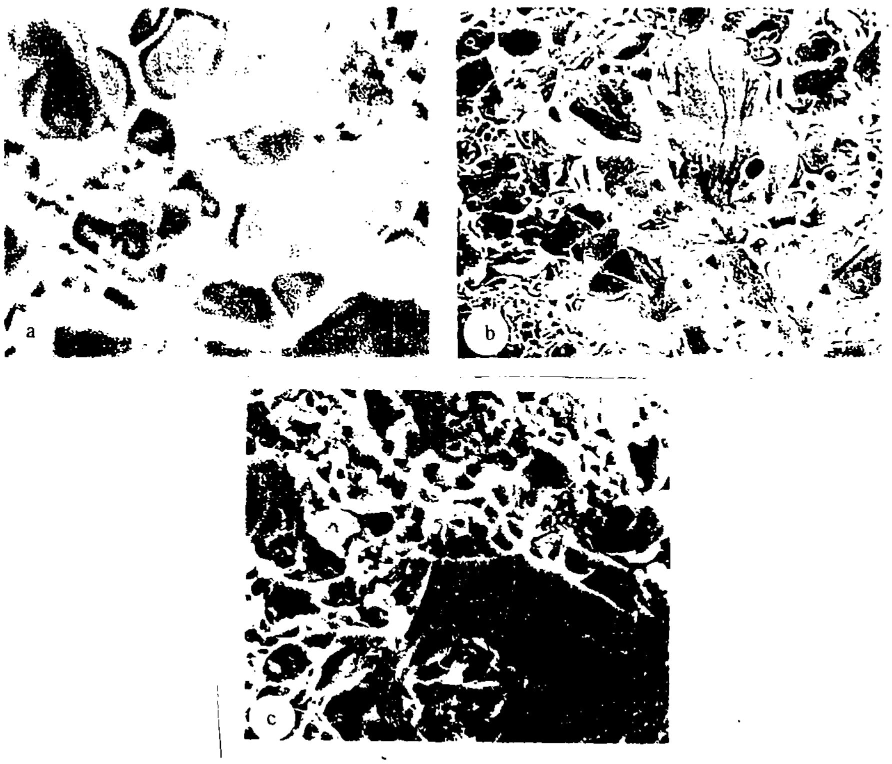

Fig. 5. Types of fracture of composites. Transcrystalline fracture through a grain-colony (a); transcrystalline fracture with a brittle break along the Mo – Mo boundary (b); intercrystalline along the Cu – Cu boundary (c). t = 20°C (a, c), –196°C (b).

short-term strength, and the content of additive in the composite is reflected in Table 1. As the nickel content rises the long-term strength σ_b increases over the entire temperature range (400-800°C). The large effect of strain hardening of the composites when cobalt is added is clearly due to dispersion hardening of the matrix (ageing copper-based solution [14]), the precipitation of intermetallic compounds at the boundary between the high-melting and low-melting components of the composite under contact interaction (under the conditions of the interfacial activity of the cobalt), and an increase in the role of the high-melting component in the plastic deformation and fracture. Dispersion hardening of the low-melting component of the W – Cu composite occurs when titanium, cerium, lanthanum, or yttrium is introduced. The resulting increase and the temperature sensitivity of the strength of the materials (Fig. 3) and the change in the ductile-brittle transition point apparently can be explained by the increase in the temperature sensitivity of the strength of the alloyed copper, the increase in the strength of the phase boundaries (under the conditions of interfacial activity of the additive), and the greater role of the high-melting component of the composite in the deformation and fracture. The results of studies on the salient features of fracture of the composites support [11, 22] those assumptions.

A noteworthy feature of the powder materials studied is the fairly strong adhesion bond at the boundaries of the main structural components. Transcrystalline fracture initiated through a grain-colony of the copper matrix along the W – W (Mo – Mo) boundary is a characteristic form of the fracture of such composites, regardless of the temperature of the test. As the temperature of the test is lowered the fraction of transcrystalline fracture of grains of the high-melting component increases. This is especially clear, e.g., upon the introduction of nickel and cobalt, which promote grain growth in the composite based on the Mo – Cu system (Fig. 5b). In that case, evidently, the grain size exceeds the critical size of a crack that can grow when the stress increases [23]. The matrix undergoes ductile fracture over the entire range of temperatures: slip bands are recorded on the walls of pores and pitted fracture is detected inside a grain-colony (Fig. 5a). Grain-boundary fracture of the matrix (Fig. 5c) occurs as a result of segregational enrichment of the surface of grains by impurity atoms (they can be detected by Auger electron spectroscopy [12]). The high-temperature hardness method makes it possible to compare the influence of alloying elements on the structure, the ratio of the properties of the components, and the adhesion characteristics of the phase boundaries. The high-temperature hardness was chosen as the criterion for assessing whether the material is suitable for making electrodes. The basis for this was the correlation (correlation coefficient r = 0.76-0.83) found between the strength properties and the durability of the electrodes [24].

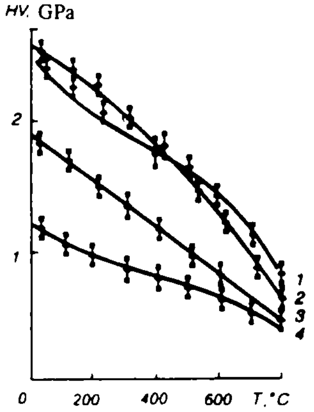

Fig. 6

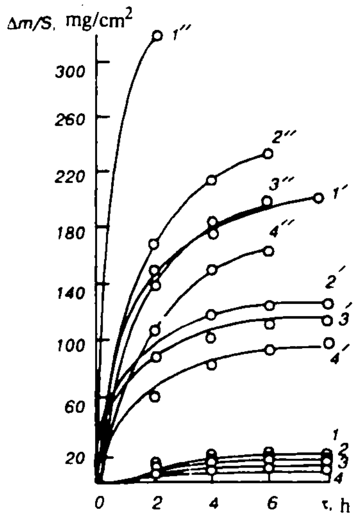

Fig. 7

Fig. 6. High-temperature hardness of the Mo–Cu (1), Mo–Cu–2Ni (2), Mo–Cu–6Ni (3), and Mo–Cu–2Co (4) composites.

Fig. 7. Influence of the composition of Mo–Cu (1), Mo–Cu–2Co (2), Mo–Cu–2Ni (3), and Mo–Cu–6Co (4) composites on the kinetics of their oxidation: T = 400°C (1–4), 500°C (1′-4′), and 600°C (1″-4″).

When the temperature dependence of the hardness of the alloyed composites of the W–Cu system in the range 20-800°C was analyzed it was found [15] that the variation of the properties as a function of the composition in the series (W–Cu) → (W–Cu + Ni) → (W–Cu + Ag) → (W–Cu + REM) → (W–Cu + Ti) → (W–Cu + Zr) is similar in nature to that in bending tests. The wear resistance of the materials increases in this sequence during use, e.g., as electrodes of contact welding machines. The lack of a pronounced temperature dependence of the yield stress of the alloyed composites based on tungsten and copper (which characterizes fcc metals, as already mentioned), the ductile nature of the fracture (which is maintained down to –196°C), and the decrease in the fraction of fracture along phase boundaries upon alloying all indicate that the hardening of the composites is due mainly to the rise in the strength of the low-melting component and the phase boundaries. The different types of hardening of the low-melting component (solid-solution hardening and dispersion hardening of the matrix) in the Mo–Cu system are made possible by additives introduced to an extent ensuring the necessary adhesion properties.

Hardening of the matrix of compositions based on Mo–Cu and intensification of this effect as the additive (nickel and cobalt) content grows promote a change in the other properties of the materials of the Mo–Cu + Ni and Mo–Cu + Co systems, in particular a change in the high-temperature hardness and the resistance to high-temperature oxidation under annealing in air in the range 400-800°C (Figs. 6, 7). Ultimately this increases the composite hardening effect over the entire range of temperatures. The recorded change (increase) in the electrical resistivity of the composites is attributed to the different nature of the influence that the additives introduced have on the properties, primarily of copper and the high-melting component.

The results of the studies have confirmed that the properties of composites based on the W(Mo)–Cu system can be changed in a directed manner: action on the structure, the phase composition, and the properties of the main components and their phase boundary upon the introduction of the appropriate additives.

The Cr–Cu System. As already mentioned, wetting and adhesion in a metal–metal system are due to the nature of the intermetallic interaction at the boundary. A weak intermetallic interaction and oxides on the surface of the solid substrate have a negative effect, which can be reduced by interfacially active additives, as already observed in the W(Mo)–melt (Cu and an alloying element) system. With mutual solubility in the Cr–Cu system the interaction at the contact sites ensures diffusion through the film and destruction of the film. The films, therefore, only delay the wetting. This assumption was confirmed experimentally. The slight surface oxidation (nitration) observed when a chromium substrate undergoes vacuum heat

Fig. 8

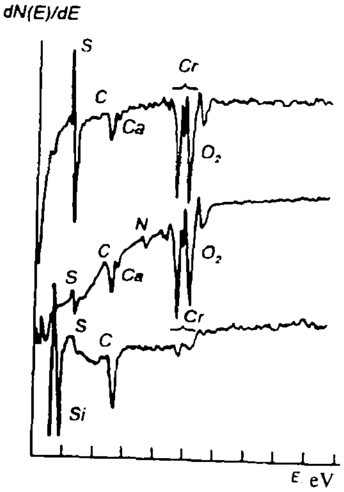

Fig. 9

Fig. 8. Structure of the region of nonwetting of a pore in a break in a sample of Cr–Cu composite. ×1500.

Fig. 9. Elemental composition of the surface of chromium particles in Cr–Cu composite.

Fig. 10

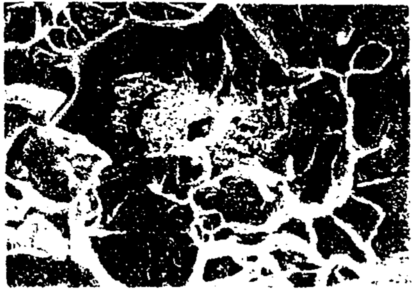

Fig. 11

Fig. 10. Toroidal shape of chromium particles in a powder obtained by calcium hydride reduction of Cr_2O_3. ×4000.

Fig. 11. Transcrystalline fracture of toroidal particles, caused by filling of pores, in a Cr–Cu composite. ×1500.

treatment promoted the formation of a film that was of varying thickness and color but not continuous and, therefore, cannot completely prevent a metal–metal bond from forming.

Measurements of the contact angles in a series of experiments showed that the system studied is characterized by good wettability ($\theta = 5\text{-}38°$), a weak temperature dependence of the wetting angle, and a high work of adhesion. Even samples with part of the substrate surface copper-plated, however, were not wetted completely. A drop of melt with a finite contact wetting angle was found to be clearly localized. When the microstructure of a contact couple in a plane perpendicular to the contact surface was studied on samples after a short hold (60 sec) a sort of channel of drops was observed, indicating intensive dissolution of chromium in the copper melt and penetration of the melt along the boundaries of chromium grains in the substrate.

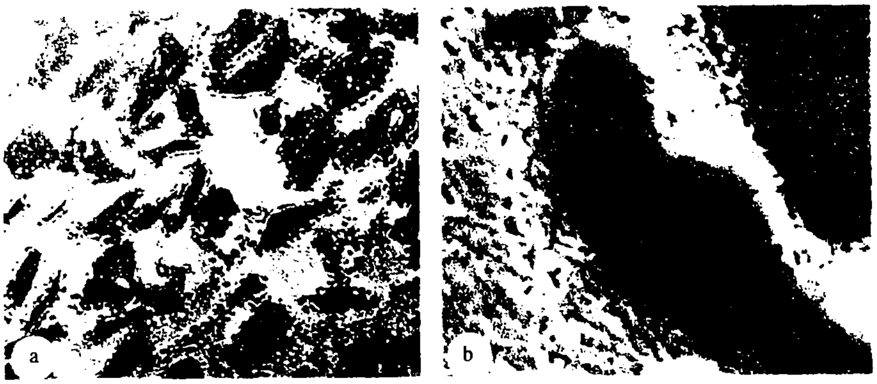

Fig. 12. Structure (in secondary electrons) of sintered Cr–Cu composite, made of electrolytically refined chromium powder. Magnification: 100 (a) and 3000 (b).

Presaturation of the copper with chromium did not change the quasiequilibrium wetting angle in the Cr–Cu system; this evidently indicates that the substrate material dissolves fairly rapidly in the melt and that almost saturated melt spreads. The example of the Cr–Cu system was used to show how the structure and properties of the composites depend on the conditions of the technological processing and on the history and quality of the initial materials. Copper and chromium (in contrast to tungsten and molybdenum) form series of limited solid solutions, with the solubility of chromium in copper dropping abruptly with decreasing temperature [14]. Consequently, chromium-copper melts are in the class of ageing melts; hence, there are broad possibilities for changing their mechanical properties without additives by means of additional treatment.

It is important to optimize the technological conditions in order to obtain practically pore-free material, in which the main structural-phase components, elements, and impurities are uniformly distributed inside the composite. As mentioned above, this is furthered by the method of capillary impregnation [1].

Breaks in Cr–Cu composites, made in commercial hydrogen by copper impregnation of porous compacts of chromium calcium hydride powder, were found to have large pores, regions where wetting did not occur (Fig. 8). The surface of high-melting particles is rich in sulfur, oxygen, carbon, nitrogen, calcium, sodium, and chlorine impurities (Fig. 9). Pores may also be hereditary: in that case they result from the formation of toroidal agglomerates of high-melting particles during calcium hydride reduction (Fig. 10). The residual inherited porosity (its maximum value reaches 2–3%) does not change in Cr–Cu composites made by copper impregnation of the same kind of compacts in a vacuum-hydrogen (from titanium hydride) medium. Such pores cannot be crack nucleators in composites (Fig. 11). The grain-colonies of the copper–chromium matrix are larger in transcrystalline and intercrystalline breaks, however, and no melt-filled pores were detected. Finally, in dense composites of the Cr–Cu system (Fig. 12), which are of interest as a material for electrical engineering purposes, PÉRKh electrolytic chromium powder served as the porous compact, and special-purity copper premelted in a vacuum was the melt. The impregnation was done in a vacuum-hydrogen (from titanium hydride) medium at 1200°C for 5 min. A composite of the Cr–Cu system (50:50 by mass) is characterized by a matrix type of structure with fixed fragmentation of particles (lamellas) of the high-melting component. This is evidently due to local consolidation of grains in chromium lamellas during sintering and the Rebinder effect in the system of polycrystalline chromium and a copper-based melt [12].

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