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THE INFLUENCE OF IRON ON THE STRUCTURE AND TECHNOLOGICAL CHARACTERISTICS OF Cu–Mn–Co–Fe BRAZING FILLER METAL

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ABSTRACT

The results of complex studies on brazing filler metals of Cu–Mn–Co–Fe system alloyed with iron in the range of 1–5 wt.% are presented. The melting point was determined through calculation, and it was shown that increasing the concentration of iron from 1 to 5 wt.% leads to a slight (from 912 °C to 923 °C) increase in the solidus temperature and a significant increase in the liquidus temperature (from 931 °C to 1027 °C). At the same time, the melting temperature range expands to 104 °C. The results of experimental studies on spreading of brazing filler metals over Kovar and corrosion-resistant steel have established that an increase in the iron concentration from 1 to 5 % contributes not only to an increase in the liquidus temperature, but also to an increase in the spreading area, which is due to the melting temperature of a copper-based solid solution. Local X-ray microanalysis established a discrete distribution of constituent elements and showed that the brazing filler metal contains two solid solutions after spreading: copper-based and manganese-based. The research results on overlap brazed joints of Kovar - corrosion-resistant steel have demonstrated that addition of iron to Cu–Mn–Co–Fe alloy leads to an increase in microhardness and shear strength.

KEYWORDS: copper–manganese–cobalt–iron brazing filler metal, melting temperature range, structure, microhardness, wetting angle, solid solution

INTRODUCTION

Joining dissimilar materials (Kovar–corrosion-resistant steel) is one of the most complicated tasks, faced by modern manufacturers. There exist different technologies of producing joints. In particular, brazing, diffusion, electron beam, laser welding, etc. have become the most widely applied [1–6]. Dissimilar joints are usually used in instrument-making, electrical engineering, chemical, petrochemical and nuclear industries, where it is necessary to ensure shape and dimensional stability at temperature change [7–11].

One of the main methods of producing this type of joints is brazing. Brazing technologies allow joining parts, if required, not around the contour, but simultaneously over the entire surface, while providing a high efficiency of the process, possibility of automation with preservation of the initial structure of the base metal, under the condition of correct selection of chemical composition of the brazing filler metal and melting temperature range [12].

Alloys based on binary copper-manganese system, characterized by solid solution structure, are widely used as filler metals for brazing dissimilar materials [13]. One can see on the state diagram of copper-manganese system the presence of a minimum on the liquidus curve, which corresponds to melting temperature of 870 °C and manganese concentration of approximately 37 at.% [14]. At lowering of heating temperature, the processes of solid solution ordering run in the binary system. At the same time, it should be noted that two-component brazing filler metals are seldom used. One of the important factors is the possibility of manganese evaporation during heating that influences the chemical composition of the brazing filler metal and melting temperature. Additional doping of alloys of this system by other chemical elements is used to improve the technological characteristics of brazed joints [12]. The most common of them are nickel, iron, and cobalt, which improve the mechanical properties of not only the brazing filler metals, but also of the brazed joints.

THE OBJECTIVE

of this work is investigation of iron influence on the technological properties of brazing filler metals of Cu–Mn–Co–Fe system, their melting temperature range, chemical heterogeneity, microhardness and wetting angle, which forms at Kovar spreading over corrosion-resistant steel.

INVESTIGATION METHODS AND MATERIALS

Experimental alloys based on Cu–Mn–4.5Co–Fe system were produced by nonconsumable tungsten electrode argon-arc melting on a cold copper substrate in high purity argon (argon volume fraction of not less than 99.993 %). The uniformity of alloying element distribution through the ingot volume was achieved by fivefold remelting with ingot turning over.

Table 2. Composition and calculated solidus and liquidus temperatures of experimental brazing filler metals

Brazing filler metal No.	Composition, wt.%	Temperature, °C	
		T_s	T_l
1	Cu–Mn–4.5Co–1Fe	912	931
2	Cu–Mn–4.5Co–2Fe	915	946
3	Cu–Mn–4.5Co–2.5Fe	917	957
4	Cu–Mn–4.5Co–5Fe	923	1027

Used as the base material were plate samples of 20×20×2 mm size from Kovar and corrosion-resistant 12Kh18N10T steel (Figure 1, Table 1). The brazing filler metal was used in the cast condition.

Spreading of experimental brazing filler metals was conducted in a vacuum furnace (SGV 2.4-2/15-13) with radiation heating (working space rarefaction of $1.33 \cdot 10^{-3}$ Pa) with the heating rate of 18–20 °C/min, and cooling rate of (10–15) ± 5 °C/min. Heating temperature (T_l + 20 °C) was controlled using a thermocouple, which was fastened on the sample (soaking for 3 min).

For thermodynamic calculations of the melting temperature range of the investigated alloys, a specialized program, JMatPro v.7.0, developed by "Sente Software" company, was used. This software package enables thermodynamic calculations for multi-component systems. These calculations are based on evaluation of Gibbs' energy functions for each phase at the specified temperature [16–18].

Metallographic investigations and X-ray microanalysis of experimental brazing filler metals were conducted on cast samples of a polycrystalline structure (after melting). Here, all the alloys were cooled to room temperature at the same rate. A standard procedure was used to prepare microsections and conduct their microstructural studies with application of scanning electron microscope TescanMira 3 LMU. Local distribution of elements in individual phases was determined by X-ray microanalysis using energy-dispersive spectrometer X-max 80 of Oxford Instruments. The microsections were studied without chemical etching in BSE mode. Locality of measurements was up to 1 μm.

Vickers microhardness (*HV*) of brazing filler metals in the initial state was studied using a stationary hardness meter NOVOTEST TC-MKB-1M with

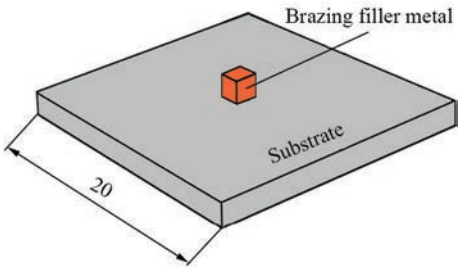

Figure 1. Schematic image of substrate–brazing filler metal

0.05 N load (τ = 15 s), measurement error was 5 %. Image-Pro Plus 6.0 program was used to measure the spreading area. Application of this software allowed conducting accurate measurements and analysis of the obtained data on spreading. Moreover, the software provides a visualization of the results in a format convenient for further use. The shear strength of brazed overlap plate joints was determined at room temperature with application of testing machine ZDM 10 Zwick-1488.

INVESTIGATION RESULTS AND DISCUSSION

Thermodynamic calculations determined the solidus and liquidus temperatures of the studied brazing filler metals of Cu–Mn–4.5Co–Fe system (Table 2).

Performed calculations showed that increase of iron concentration from 1 up to 5 % in Cu–Mn–Co–Fe system alloy leads to increase of the liquidus temperature from 931 up to 1027 °C. Solidus temperature changes only slightly (from 912 to 923 °C). Determined temperature results were used to plot a graph, which demonstrates iron influence on the alloy melting range, and shows that with increase of iron concentration the melting temperature range becomes much wider (Figure 2).

Further processing of the data by statistical analysis methods yielded the dependencies of the liquid phase on heating temperature for each alloy, which is indicative of a smooth increase of the quantity of the liquid phase at increase of iron concentration from 1 up to 2.5 % (Figure 3, *a–c*).

At heating of alloys containing 5 % of iron, the quantity of the liquid phase rapidly increases with temperature increase from 925 up to 937 °C (Figure 3, *d*). Further temperature rise (from 937 to 1027 °C) only slightly influences the increase in the liquid

Table 1. Base material chemical composition [15]

Material grade	Chemical element, wt.%									
	Fe	Ni	Co	C	Si	Mn	Cr	Ti	Al	Cu
29NK Kovar	51.14–54.5	28.5–29.5	17–18	0.03	0.3	0.4	0.1	0.1	0.2	0.2
12Kh18N10T	67	9–1	–	0.12	0.8	2	17–19	0.4–1	–	0.3

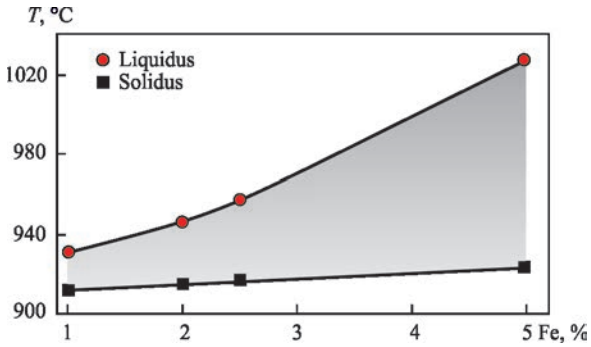

Figure 2. Influence of iron on melting range of alloys of Cu–Mn–4.5Co–Fe system

phase quantity (within 83–100 %). Obtained data agree well with the results of experiments on brazing filler metal spreading over the base metal (Kovar and corrosion-resistant steel), which will be given below.

Conducted metallographic investigations and results of X-ray microanalysis showed that Cu–Mn–4.5Co–(1–5)Fe alloys are characterized by a two-phase structure, which is formed by two solid solutions. Local X-ray microanalysis determined a discrete distribution of the constituent elements in individual phases.

In the case of investigations of Cu–Mn–Co–Fe system alloy with 5 % Fe it is shown that its structure is two-phase, and it consists of two solid solutions,

Table 3. Chemical composition of individual phases of Cu–Mn–4.5Co–5Fe alloy

Spectrum	Chemical elements, wt.%			
	Mn	Fe	Co	Cu
1	39.41	22.59	16.73	21.27
2	32.97	3.37	4.48	59.18

which are characterized by a pronounced segregation, inherent to alloys of Cu–Mn system [19]. The main phase is copper-based solid solution (α-Cu) (Figure 4, Table 3, spectrum 2), along the grain boundaries of which dendrites of a manganese-based solid solution (γ-Mn) precipitate, which are enriched in iron, cobalt and copper (Figure 4, Table 3, spectrum 1).

Electronic images of the microstructure of Cu–Mn–Co–(1–5)Fe alloy (in the initial condition) are indicative of certain morphological changes, which are due to the influence of the alloying element — iron (Figure 5). Analysis of these structures leads to the conclusion that increase of iron concentration from 1 up to 5 % in alloys of Cu–Mn–Co–Fe system leads to refinement of the structural components and to formation of fine-crystalline structures (Figure 5).

X-ray microanalysis allowed establishing a connection between iron concentration in the brazing fill-

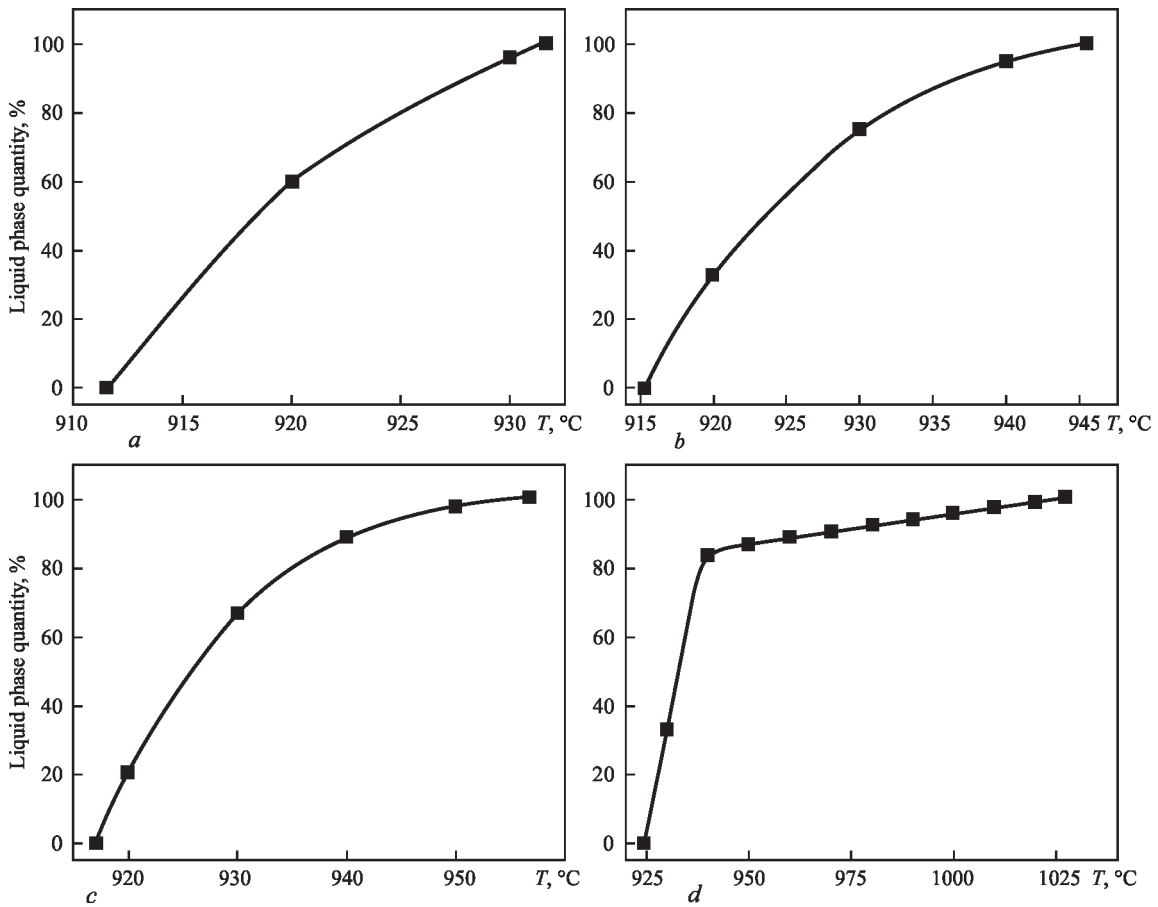

Figure 3. Quantity of the liquid phase in alloys of Cu–Mn–Co–Fe system, depending on temperature and Fe content in %: 1 (*a*); 2 (*b*); 2.5 (*c*); 5 (*d*)

er metal and in individual phases. So, increase of iron concentration from 1 to 5 % in experimental brazing filler metals leads to a slight increase in its quantity from 0.3 to 3.37 % in copper-based α-solid solution. Here, a slight increase of iron concentration up to 22.59 wt.% (at alloying with iron in the quantity of 5 %) is found in γ-phase (based on manganese) (Figure 6). Such a structural heterogeneity is responsible for increase of brazing filler metal microhardness (Figure 7).

Results of experiments on spreading of experimental brazing filler metals over Kovar and corrosion-resistant steel (in keeping with the calculated melting temperatures) are indicative of satisfactory wetting of base metals under vacuum. Here, small wetting angles are formed: within 4–22° over Kovar and 6–10° over the corrosion-resistant steel. It should be noted that spreading is accompanied by certain kinetic peculiarities. The first to melt is the copper-based solid solution, which spreads over the base metal as a plane front to form a halo around the perimeter of the refractory component of the brazing filler metal, which is concentrated in the central zone of the plate sample (Figure 8).

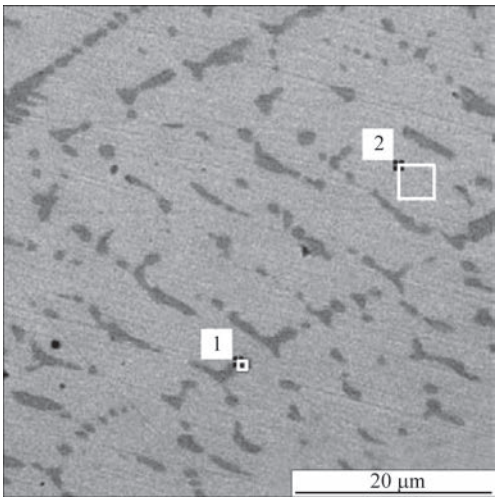

Figure 4. Microstructure of Cu–Mn–4.5Co–5Fe alloy

This is the result of partial evaporation of manganese taking place during heating [20], which is due to the high vapour pressure of the latter that is visually confirmed by the brazing filler metal colour after its spreading (Figure 8). Moreover, the concentration of refractory elements, namely iron and cobalt, increases considerably in γ-phase (manganese-based) that is confirmed by the results of X-ray microanalysis. In

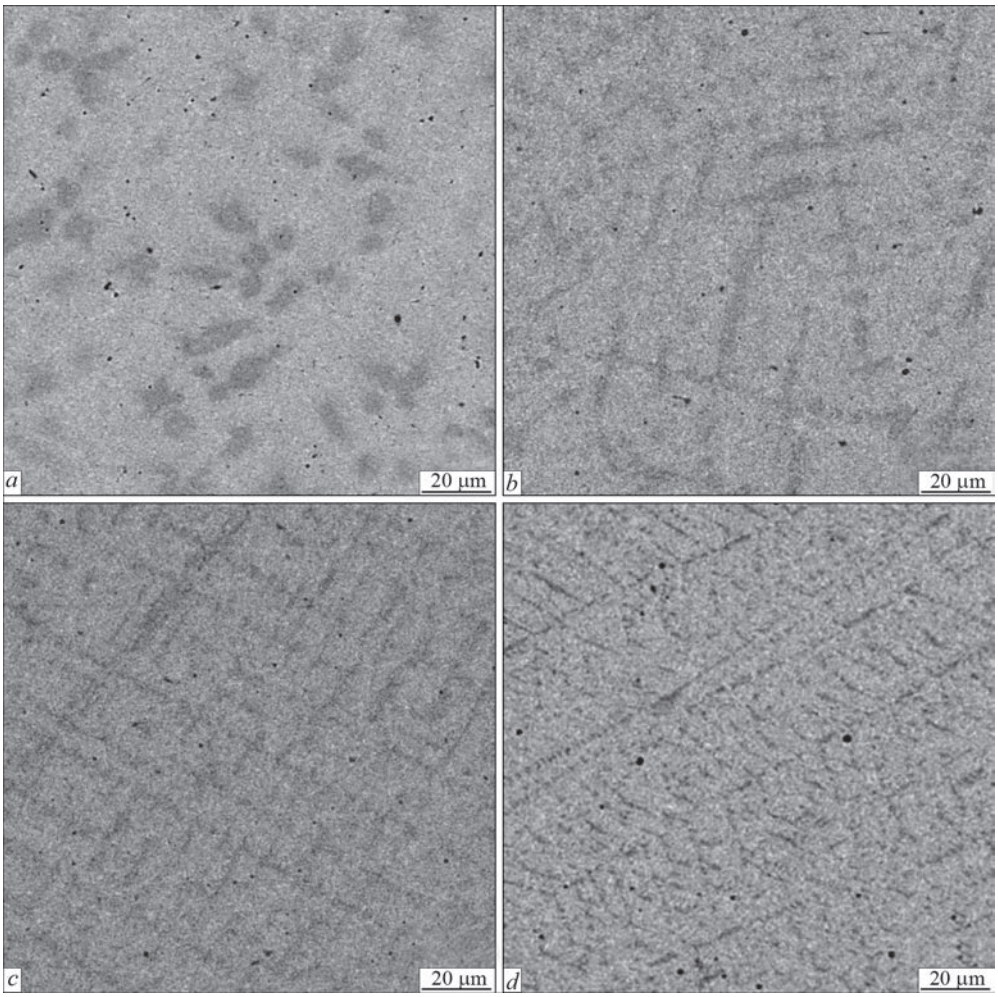

Figure 5. Microstructure of alloys of Cu–Mn–Co–Fe system with different Fe concentration in %: 1 (*a*); 2 (*b*); 2.5 (*c*); 5 (*d*)

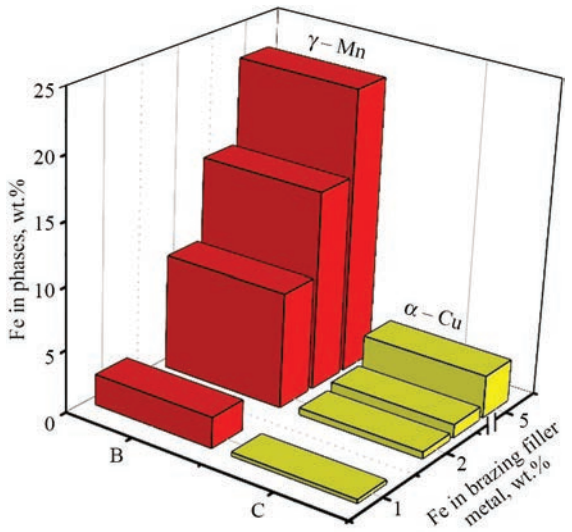

Figure 6. Dependence of Fe concentration in individual phase on its content in the initial brazing filler metal

the copper-based solid solution, the iron quantity also increases, but only slightly, which was established by X-ray microanalysis of a drop of brazing filler metal after spreading. A significant heterogeneity of the brazing filler metal drop as to chemical composition (at spreading) leads to an increase in the difference between the melting temperatures of both the components. As the two solid solutions are characterized by different melting temperature, at base metal wetting the copper-manganese solid solution is the first to spread, which is exactly what forms a halo around the perimeter of the brazing filler metal drop. A schematic representation of the brazing filler metal after spreading is given in Figure 9.

Thus, at increase of iron concentration in brazing filler metals of Cu–Mn–Co–Fe system the liquidus temperature rises, requiring a corresponding increase of the heating temperature, when conducting the spreading experiments. It promotes better spreading of the low-melting component — the copper-based solid solution [21].

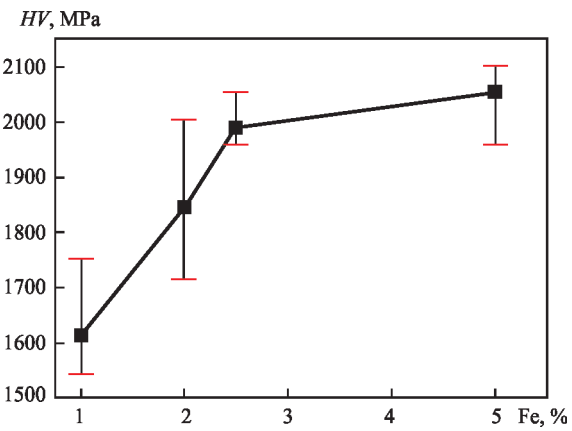

Figure 7. Influence of iron concentration on microhardness of brazing filler metals of Cu–Mn–Co–Fe system

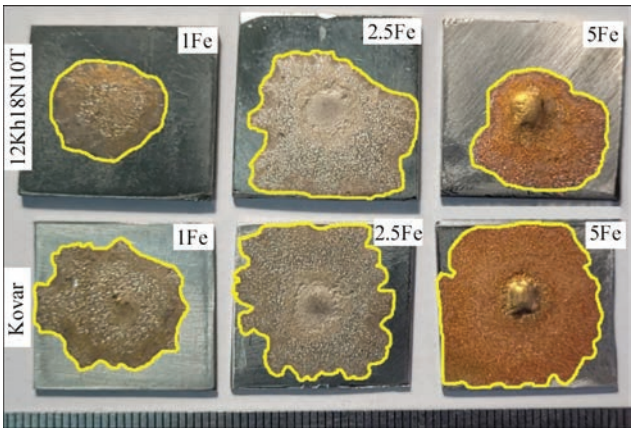

Figure 8. Appearance of samples after spreading of brazing filler metals with different quantity of iron over corrosion-resistant steel and Kovar

Analysis of the obtained results of X-ray microanalysis and local distribution of elements in a drop of brazing filler metal No. 1 (after spreading) points to a similar tendency of structure formation (as in the initial brazing filler metal). It was proved empirically that iron concentration in the copper-based and manganese-based solid solutions becomes higher with increase of its quantity in the brazing filler metal. It was determined, in particular, that the phase based on manganese-copper system is also enriched in iron up to 13.47 % and in cobalt up to 27.85 % (Figure 10, Table 4, spectrum 2). At the same time, manganese-based dendrites (86.06 % Mn) were revealed during investigations (Figure 10, *a*, Table 4, Spectrum 1).

Copper-based and manganese-based solid solutions also form at spreading of this brazing filler metal over corrosion-resistant steel (Figure 10, *b*). Iron concentration in grains of manganese- and copper-based phase practically does not change (Figure 10, *b*, Table 5).

After spreading of brazing filler metal No. 1 over corrosion-resistant steel a similar structure appears, which is also formed by the two solid solutions and manganese-based phase inclusions (Figure 10, *b*). Local X-ray microanalysis revealed that manganese-based grains, which solidify against the background of α-Cu solid solution, contain the same quantity of iron (13.37 %), similar to spreading over Kovar. Iron concentration in the solid solution does not change, either.

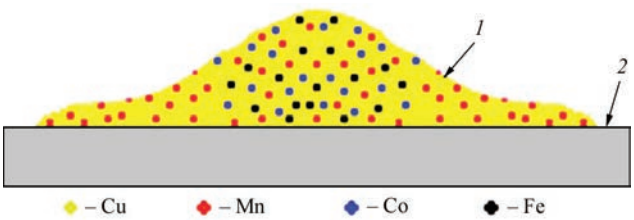

Figure 9. Schematic representation of brazing filler metal after spreading of brazing filler metal drop (*1*) and base metal substrate (*2*)

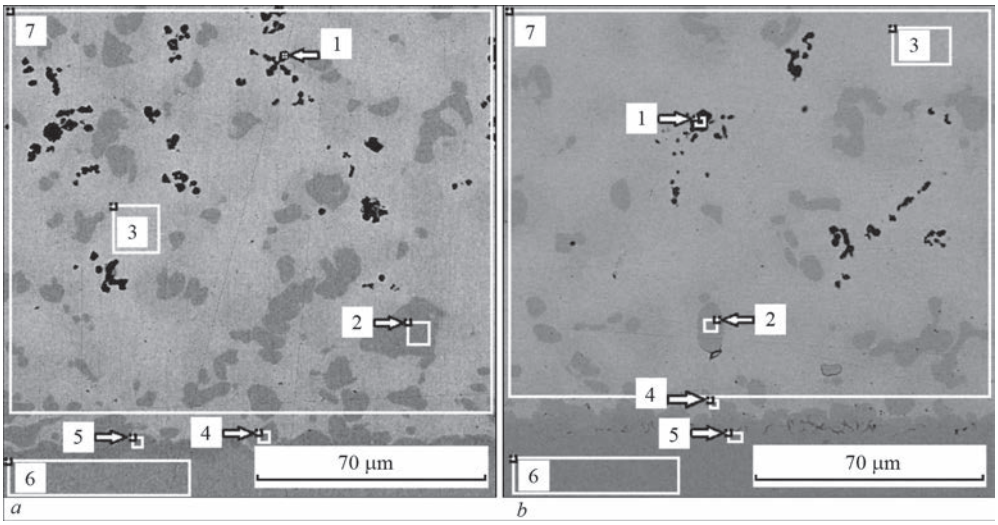

Figure 10. Microstructure of brazing filler metal No.1 after spreading over Kovar (*a*) and corrosion-resistant steel (*b*)

A somewhat higher concentration of alloying elements in individual phases after spreading of the brazing filler metal over the base metal is attributable to non-equilibrium thermokinetic conditions of brazing filler metal solidification, and presence of a gradient of constituent element concentration on the interface of the brazing filler metal with the base metal, leading to running of the diffusion processes and structural heterogeneity.

The derived investigation results proved that increase of iron quantity in the initial brazing filler metal from 1 up to 5 % leads to increase in its concentration in the structural components. So, in Cu–Mn solid solution (matrix) a slight increase in iron concentration from 1.1 to 3.0 % at spreading over Kovar and from 0.36 to 2.6 % over corrosion-resistant steel is observed (Figure 11, *a*).

It should be noted that in γ-Mn based solid solution a rapid increase in iron concentration (from 13.47 to 42.65 %) is observed after spreading of brazing filler metal No. 4 over Kovar (Figure 11, *b*). A similar increase in iron quantity (13.37 % to 44.82 %) is found also at spreading over corrosion-resistant steel

Table 4. Chemical composition of base metal and individual phases at spreading over Kovar

Spectrum	Chemical elements, wt.%				
	Mn	Fe	Co	Ni	Cu
1	86.06	0.83	1.15	0.27	11.70
2	40.71	13.47	27.85	0.52	17.47
3	27.15	1.10	3.92	0.39	67.44
4	39.51	14.91	27.97	0.78	16.73
5	34.50	24.52	24.94	3.34	12.60
6	0.51	52.97	17.58	28.61	0.33
7	3..93	3.66	9.76	0.50	54.15

(Figure 11, *b*). Results of electron microscopy and X-ray microanalysis correlated well with those of mechanical tests.

Preliminary studies [22] demonstrated that at brazing dissimilar overlap joints of Kovar-corrosion-resistant steel by Cu–Mn–4.5Co brazing filler metal the shear strength is equal to 434 MPa. Alloying of this brazing filler metal by iron in the quantity of 1 %

Table 5. Chemical composition of base metal and individual phases at spreading of brazing filler metal No.1 over corrosion-resistant steel

Spectrum	Chemical elements, wt.%							
	Si	Ti	Cr	Mn	Fe	Co	Ni	Cu
1	16.73	0.11	0.00	79.72	0.43	0.23	0.32	2.46
2	0.00	0.00	0.68	42.00	13.37	28.10	0.00	15.85
3	0.00	0.00	0.00	28.61	0.36	2.66	0.00	68.36
4	0.00	0.00	0.89	41.84	13.86	27.20	0.00	16.22
5	0.31	0.24	15.28	11.40	60.55	4.28	6.57	1.38
6	0.53	0.28	17.65	1.27	71.21	0.34	8.72	0.00
7	0.00	0.00	0.21	32.16	2.88	7.69	0.00	57.06

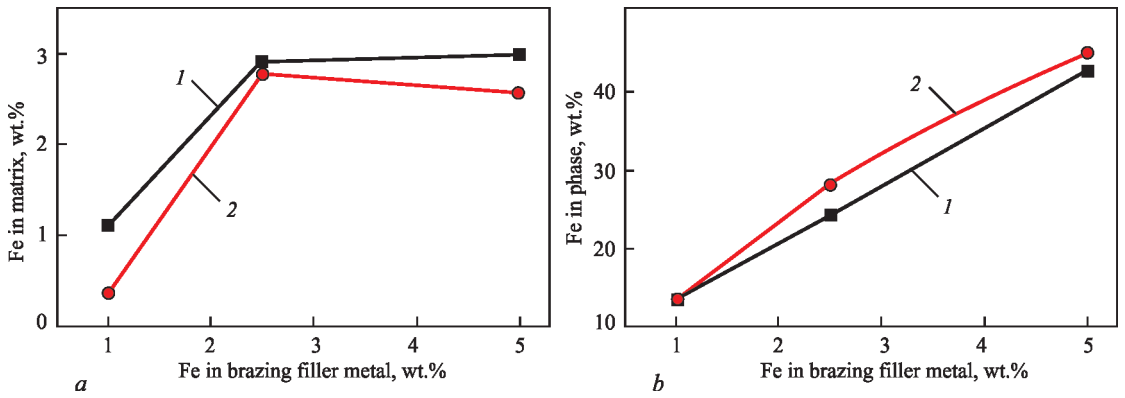

Figure 11. Fe content influence on its concentration in α-Cu based solid solution (*a*) and γ-Mn based solid solution (*b*) after spreading of brazing filler metals over Kovar (*1*) and corrosion-resistant steel (*2*)

leads to strengthening of the two solid solutions by iron, and to increase of shear strength from 434 to 488 MPa, respectively.

Comprehensive results of the conducted studies open up additional possibilities of improving the technological properties of copper-manganese brazing filler metals which are applied in manufacture of brazed components from dissimilar materials.

CONCLUSIONS

It was determined by calculations that increase of Fe content in alloys of Cu–Mn–Co-Fe system from 1 to 5 % leads to increase of liquidus temperature from 931 to 1027 °C, whereas the solidus temperature rises only slightly (from 912 to 923 °C). A significant (104 °C) widening of the melting range of the brazing filler metal is observed here.

Using the results of X-ray microanalysis studies and taking into account the shape of fusibility diagrams, it was proved that alloys of Cu–Mn–4.5Co–(1–5)Fe system are characterized by a two-phase structure, which consists of two solid solutions. The main phase is a copper-based solid solution, which contains dendrites of manganese-based solid solution, enriched in iron, cobalt and copper. Increase of iron concentration in the brazing filler metal promotes a considerable increase in its quantity in the manganese-based solid solution (γ-phase) and increase of its microhardness, respectively.

It is established that increase of iron quantity in the initial brazing filler metal from 1 to 5 % leads to increase of its concentration in the structural components after spreading. So, a slight increase of iron concentration (from 1.1 to 3.0 %) is found in Cu–Mn solid solution (matrix) at spreading over Kovar and from 0.36 to 2.6 % at spreading over the corrosion-resistant steel. In manganese-based solid solution, a rapid increase in iron concentration from 13.47 to 42.65 % is observed after spreading of the brazing filler metals over Kovar and from 13.37 to 44.82 % at spreading over the corrosion-resistant steel.

Mechanical testing of overlap brazed joints of Kovar-corrosion-resistant steel proved that alloying of brazing filler metal of Cu–Mn–Co–Fe system by iron in the quantity of 1 % promotes an increase of shear strength from 434 (basic brazing filler metal) to 488 MPa.

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CONFLICT OF INTEREST
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