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The influence of silicon content on the structure of Cu55Ni6Mn4Zn brazing alloy and on the structure and properties of brazed joints

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Abstract: Cu55Ni6Mn4Zn (MNMts55-6-4) copper-zinc alloy is widely used for brazing hard-alloy tools and steels. However, the presence of silicon in the alloy (0.1–0.4 wt. %) can lead to the formation of brittle silicides of iron, nickel, and manganese, which negatively influences the strength of brazed joints. The purpose of the study was to determine the influence of the quantitative content of silicon in copper-zinc brazing alloy doped jointly with nickel and manganese on the structure of brazing alloy blanks before brazing and the structure and properties of brazed joints. In the work, to study the distribution of silicides in ingots, tapes, and brazed seams, the authors used microstructural analysis methods, including electron microscopy and X-ray spectral microanalysis. The results showed that with a silicon content of up to 0.2 wt. %, silicides form finely dispersed inclusions uniformly distributed throughout the seam. However, with an increase in the silicon content to 0.4 wt. %, the formation of continuous layers of iron silicides along the brazing alloy – steel boundary is observed, which leads to brittle failure of the joints under mechanical loads. The influence of small gaps turned out to be especially critical during brazing, where the formation of large crystals of iron silicides significantly reduces the strength of the joints. The scientific novelty of the work lies in identifying the optimal silicon content in the alloy (no more than 0.2 wt. %) to minimize the negative effect of silicides on the properties of brazed joints. The results obtained can be used to develop process recommendations for the production of brazing alloys and brazing of steels, which will allow improving the reliability and durability of brazed joints under production-line conditions.

Keywords: brazing of hard-alloy tools; Cu55Ni6Mn4Zn (MNMts55-6-4); influence of silicon on brazed joints; iron silicides in brazed seams; brazed seam microstructure; embrittlement of brazed joints; optimization of brazing alloy composition.

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INTRODUCTION

One of the most common methods of joining hard alloys to steel is brazing [1]. The Cu55Ni6Mn4Zn alloy (MNMts55-6-4) is nickel silver with a high manganese content and is successfully used in Russia and abroad for brazing hard-alloy tools [2; 3] along with the Cu49Ni9Zn (LNMts49-9-0.2) brazing alloy [4; 5]. The composition and melting point of these alloys are presented in Table 1. The high melting and brazing temperature allows, after the brazing alloy has solidified, to carry out heat treatment of the steel tool bodies using quenching from 860–900 °C in oil or emulsion, followed by tempering [6].

The influence of alloying elements on the properties of copper-zinc alloys has been studied in sufficient detail. Additions of aluminum, manganese, iron, and nickel improve the mechanical properties of brasses and their heat resistance [7; 8]. Alloying with manganese improves the wetting of steel and hard alloy with molten brazing alloy [9].

***Table 1.** Compositions of Cu55Ni6Mn4Zn35 and Cu49Ni9Zn42 alloys and their melting and brazing temperatures*
***Таблица 1.** Состав сплавов Cu55Ni6Mn4Zn35 и Cu49Ni9Zn42, а также их температуры плавления и пайки*

Alloy	Chemical composition, % wt.					$T_{solidus}$, °C	$T_{liquidus}$, °C	$T_{brazing}$, °C
	Cu	Zn	Ni	Mn	Si			
Cu55Ni6Mn4Zn35	54–56	the rest	5.5–6.5	3.5–4.5	0.1–0.4	890	920	920–1040
Cu49Ni9Zn42	48–50	the rest	9–11	0.1–0.2	0.1–0.25	915	930	930–980

Alloying with nickel significantly improves the mechanical properties of brass [10]. Alloying with tin can slightly increase the shear strength of steel-hard alloy joints [11]. The Cu49Ni9Zn42 alloy, due to alloying with nickel, allows producing significantly stronger joints than those brazed with two-component brass [12–14]. Comparative studies of joints made using brass brazing alloy and Cu49Ni9Zn42 nickel silver confirm the thesis that the addition of nickel significantly increases the strength of the joints [15; 16]. Brazing alloys based on copper-zinc alloys for brazing hard alloy are usually doped together with nickel and manganese. The reference literature and standards for brazing alloys also indicate the presence of silicon in an amount of 0.1–0.4 wt. %. However, there is practically no mentioning of the influence of the quantitative silicon content on the formation of the brazed seam and the properties of brazed joints made using brazing alloys doped with nickel and manganese.

It is considered that silicon neutralizes the negative effect of iron impurities on the corrosion resistance of brass due to the formation of iron silicides [17]. It improves the technological efficiency of brazing and welding processes due to the fact that the silicon oxide film reduces zinc oxidation at high temperatures. Brasses containing silicon additives have better technological properties and provide higher density and tightness of the seam.

The addition of silicon to brass brazing alloys can reduce the ductility and strength of the brazed joint made of steel through the formation of a brittle Fe-Si intermetallic layer at the brazing alloy – steel interface. Therefore, when brazing steels with brass, it is recommended to limit the alloying of the latter with silicon – no more than 0.3 wt. %. Nickel has a greater chemical affinity for silicon than for iron. When Cu–Zn is introduced into a brazing alloy containing 0.5 % of Si and 2 % of Ni, silicon binds with nickel into a chemical compound and does not form an intermetallic compound with iron along the seam boundary[1]. The same effect is observed during laser brazing of steels using a copper-based brazing alloy with 3 wt. % of silicon [18]. Despite the short time of heating to high temperatures, a layer of iron silicides is formed at the brazing alloy – steel boundary [19], which are also present as small inclusions in the weld volume. Mechanical tests of such joints show that destruction mainly occurs precisely along the brazing alloy – steel boundary due to the presence of such compounds [20].

Iron, nickel and manganese silicides have a high melting point: NiSi – 992 °C, FeSi – 1410 °C, and MnSi – 1280 °C[2,3]. The enthalpies of formation of various silicides have similar values, so that the complex formation of manganese and nickel silicides is also possible, and in the presence of iron in the brazing alloy – of iron silicides. It is known that some iron silicides have a lower standard enthalpy of formation than nickel silicides. This may lead to the fact that even in brazed seams produced using nickel-alloyed brazing alloys, iron silicides may form [21].

Therefore, the effect of the silicon quantitative content in copper-zinc brazing alloys with joint alloying with nickel and manganese on the structure and properties of brazed joints has not been sufficiently studied.

The purpose of the study is to determine the influence of the quantitative content of silicon in copper-zinc brazing alloy doped jointly with nickel and manganese on the structure of brazing alloy blanks before brazing and the structure and properties of brazed joints.

METHODS

To study the structure of ingots and pressed bands made of Cu55Ni6Mn4Zn35 (MNMts55-6-4) alloy, industrial samples produced by AO ALARM (Russia) were selected, cast in a metal chill mold. The samples were melted in an induction furnace in an AH 200 clay-graphite crucible, where 200 is the crucible capacity (for copper) of 200 kg. After melting copper and nickel, copper was deoxidized with phosphorus and lump silicon was introduced. Silicon was introduced at the rate of 0.35 wt. %. Mn985 electrolytic manganese was used (manganese content of at least 98.5 %). During melting, 30–40 % of own production waste was used. The resulting 50 mm diameter ingots were hot pressed in a hydraulic press with a force of 300 t through a 10×1 mm matrix. The ingot temperature during pressing was 700 °C. The pressed band had dimensions of 10×1 mm.

To study the influence of silicon content in the brazed seams, small-volume alloy samples with different silicon content were separately melted. The melting differed by the fact that, unlike industrial melting, the copper melt

[1] *Lashko S.V., Lashko N.F. Payka metallov [Brazing of metals]. Moscow, Mashinostroenie Publ., 1988. 376 p.*

[2] *Lyakishev N.P., ed. Diagrammy sostoyaniya dvoynykh metallicheskikh system [State diagrams of two-component metal systems]. Moscow, Mashinostroenie Publ., 2001. Vol. 3, kn. 1, 972 p.*

[3] *Lyakishev N.P., ed. Diagrammy sostoyaniya dvoynykh metallicheskikh system [State diagrams of two-component metal systems]. Moscow, Metallurgiya Publ., 1997. Vol. 2, 1024 p.*

was not deoxidized with phosphorus and silicon was introduced in the form of a Cu – 10 wt. % Si alloy. The samples were melted in an induction furnace in an AH 5 clay-graphite crucible of the OAO Luga Abrasives Plant (Russia). The blending was carried on the basis of producing 2.5 kg of alloy from pure components, M0 oxygen-free copper (copper content of at least 99.95 %), NP-1 nickel (high-purity nickel, nickel content of at least 99.9 %), Mn985 manganese, Ts0 zinc (high-purity zinc, zinc content of at least 99.9 %). In molten copper under a Probat Fluss layer, nickel and manganese were dissolved one after another, after which the Cu–Si alloy and zinc were added. The calculated silicon content in the alloy was 0.1, 0.2, and 0.4 wt. %. The resulting melt was poured into a water-cooled casting form with a diameter of 50 mm. The resulting ingots were pressed on a hydraulic press at a temperature of 700 °C to form a brazing alloy tape measuring 10×1 mm.

The produced samples of ingots and bands were used to make sections for examination using a TESCAN VEGA SBH 3 scanning electron microscope (Czech Republic) with an Oxford Instruments attachment (UK) for X-ray spectral microanalysis (XSMA). The probe diameter was 1 µm.

To study the influence of silicon on the structure of brazed seams, experimental models of joints were made in size and shape close to brazed mining cutters. The only difference was that instead of hard alloy, an insert made of 30HGSA steel (GOST 4543-71) (chemical composition: 0.28–0.34 % C, 0.9–1.2 % Cr, 0.8–1.1 % Mn, 0.8–1.1 % Si, the rest is Fe) with a diameter of 18 mm was used, and the outer casing was made of low-carbon steel 1010 (Fig. 1). Heating of the models for brazing was carried out on the SELT-001-15/66-T induction unit (Russia) with the frequency of 40–70 kHz in the cylindrical inductor. The brazing temperature was 940–950 °C. The brazing time was 90 s. The model of the 30HGSA steel insert was dipped in the FP2 flux paste based on potassium borax, borates and fluorides of alkali metals (TU 48-17228138/OPP-004-2001) and placed in the housing bore, on the bottom of which the brazing alloy plates with the total weight of 3.6 g were placed to ensure filling of the entire gap. During the heating process, the brazing alloy melted, and the insert was lowered to the bottom of the housing. The time and power on the brazing unit were selected so that a solid fillet was formed at the end of the process. This ensured the same holding time for all samples during brazing.

After brazing, the samples were cut along the axis and metallographic sections were prepared for examination using a TESCAN VEGA SBH 3 electron microscope (Czech Republic). The elemental composition was determined using an Oxford Instruments energy dispersive microanalyzer (UK).

RESULTS

Structure of Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy ingots and bands depending on silicon content

Fig. 2 shows images of microstructure of cast samples produced during industrial melting using deoxidation with phosphorus and the introduction of 0.35 wt. % lump silicon in comparison with a sample manufactured without copper deoxidation with phosphorus and with the introduction of 0.2 wt. % silicon in the form of Cu10Si alloy. The structure contains dark areas located along the boundaries of dendritic cells; the results of their X-ray spectral microanalysis are presented in Table 2.

The structure of industrial brazing alloy bands after hot pressing is shown in Fig. 3. One can see that the dark areas look like dispersed inclusions 2–10 µm in size and are uniformly distributed over the area of the section. All samples contain iron in an amount of 0.2 wt. %. One should note that with decreasing silicon content, the dark phase becomes more dispersed. The composition of the dark areas is similar to the phases in the cast structure and contains silicon in an amount of about 10 wt. % and phosphorus in an amount of 1.7–3 wt. %.

Structure of brazed joints produced with brazing alloy with different silicon content

When brazing steel models of mining cutters, different structures were obtained in the bottom and side parts of the joint. In the structure of the brazed joints, finely dispersed inclusions are observed, mainly iron, as well as nickel and manganese in the central part of the joint and along the interface of the brazing alloy and base material, which is confirmed by the results of X-ray microanalysis (Fig. 4). Due to the small size of the phase, it was not possible to identify its exact composition;

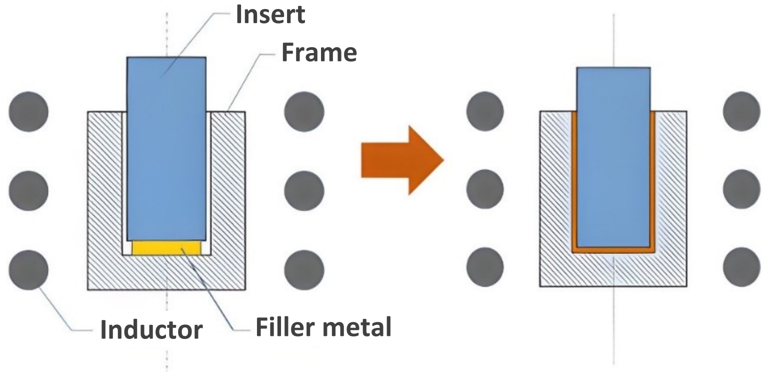


Fig. 1. *Scheme of induction brazing of a mining cutter model with the Cu55Ni6Mn4Zn35 brazing alloy*
Рис. 1. *Схема индукционной пайки макета горного резца припоем Cu55Ni6Mn4Zn35*

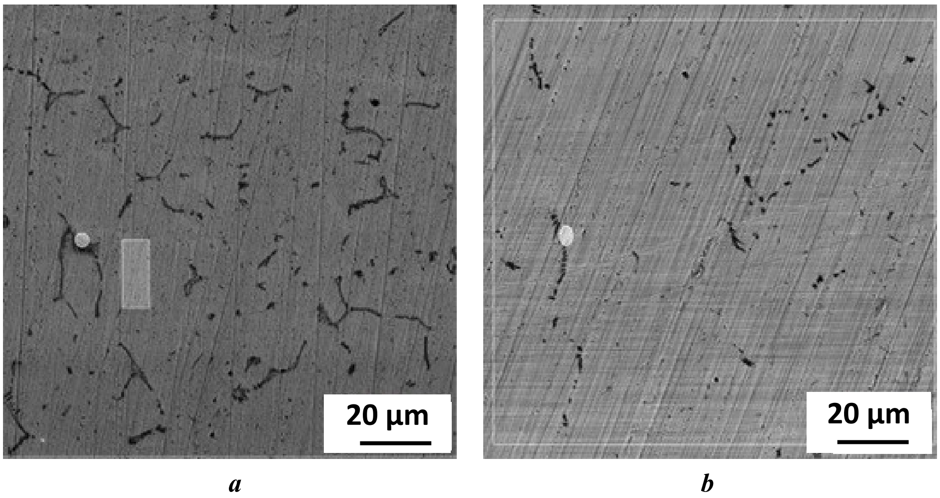


Fig. 2. Microstructure of Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy ingots: ***a*** – industrial melting; ***b*** – experimental melting without deoxidation with phosphorous and with the introduction of silicon by doping material
Рис. 2. Микроструктура слитков припоя Cu55Ni6Mn4Zn35 (МНМц55-6-4): ***a*** – промышленная плавка; ***b*** – экспериментальная плавка без раскисления фосфором и с введением кремния лигатурой

Table 2. *Results of X-ray spectral microanalysis of ingots in as-cast condition, % wt.*
Таблица 2. *Результаты микрорентгеноспектрального анализа слитков в литом состоянии, % мас.*

Area of analysis	Si	P	Cr	Mn	Fe	Ni	Cu	Zn
Industrial sample								
Bright area	0.01	–	0.87	3.37	–	5.75	54.2	33.42
Dark area	9.1	2.43	–	16.96	1.12	44.13	17.0	9.26
Analysis of alloy	0.4	–	–	3.92	0.18	5.6	52.01	37.89
Experimental sample								
Dark area	10.56	–	–	18.23	0.05	52.35	13.48	5.33
Analysis of alloy	0.26	–	–	4.26	0.02	5.98	54.78	34.7

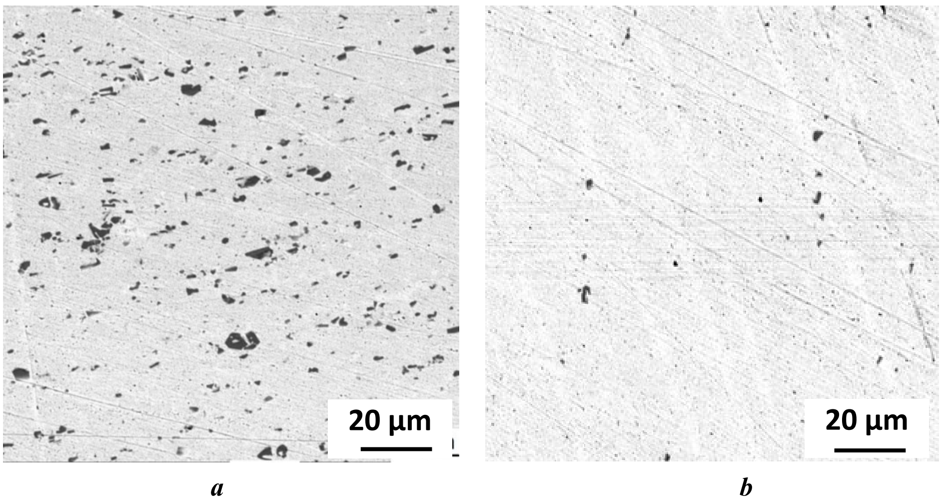
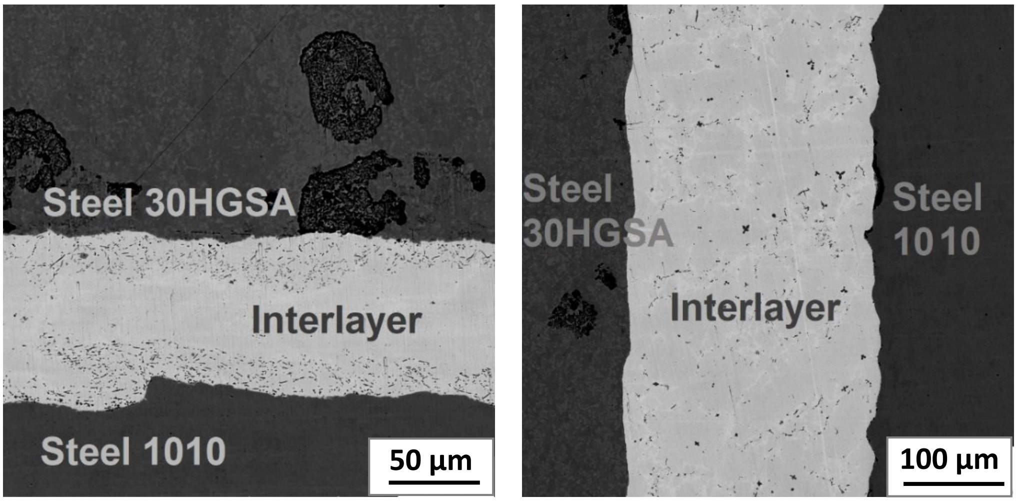


Fig. 3. Microstructure of brazing alloy bands with different silicon content (not etched): ***a*** – 0.57 % of Si; ***b*** – 0.31 % of Si
Рис. 3. Микроструктура лент припоя при различном содержании кремния (не травлено): ***a*** – 0,57 % Si; ***b*** – 0,31 % Si

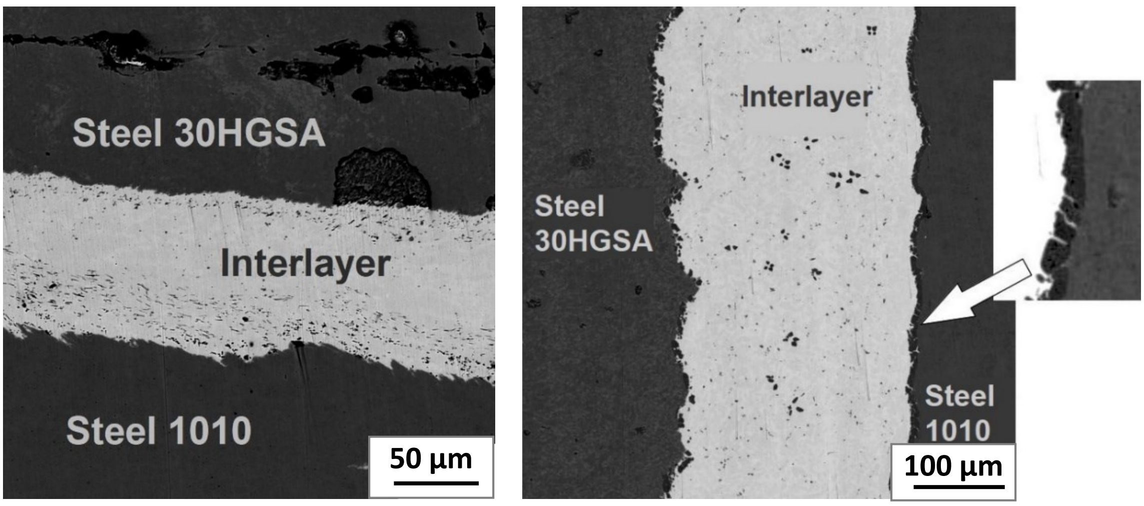


a *b*

Fig. 4. *Microstructure of the model of the joints of mining cutters made with Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy with a silicon content of 0.2 wt. %:* ***a*** *– bottom part;* ***b*** *– side part*
Рис. 4. *Микроструктура макета соединений горных резцов, выполненных припоем Cu55Ni6Mn4Zn35 (МНМц55-6-4) с содержанием кремния 0,2 % мас.:* ***a*** *– донная часть;* ***b*** *– боковая часть*

presumably, these are iron silicides. At low silicon contents of 0.1 and 0.2 wt. %, dispersed inclusions are located in the volume of the joint and do not form large formations that could become stress concentrators for crack formation when the joints are loaded. The structure of the seams with a silicon content of 0.1 and 0.2 wt. % is identical, therefore Fig. 4 shows the structure of the joint with 0.2 wt. % of silicon. The structure of the seams with a silicon content of 0.4 wt. % is shown in Fig. 5.

With a silicon content of 0.4 wt. %, the picture changes radically. At the boundary of the steel with the brazing alloy, especially on the side of 30HGSA steel containing silicon (0.8 wt. % according to X-ray micro-analysis), the formation of large regular-shape crystals is observed, forming a continuous layer at the steel – brazing alloy boundary. Such a pattern is observed with a normal brazing gap size (50–100 µm). The structure in small gaps (less than 50 µm) is formed completely differently. During brazing, a tortuous seam boundary is formed (Fig. 6). Large crystals based on iron and nickel compounds with silicon are formed at the brazing alloy – steel interface, which in some places practically cover



a *b*

Fig. 5. *Microstructure of the model of the joints of mining cutters made with Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy with a silicon content of 0.4 wt. %:* ***a*** *– bottom part;* ***b*** *– side part*
Рис. 5. *Микроструктура макета соединений горных резцов, выполненных припоем Cu55Ni6Mn4Zn35 (МНМц55-6-4) с содержанием кремния 0,4 % мас.:* ***a*** *– донная часть;* ***b*** *– боковая часть*

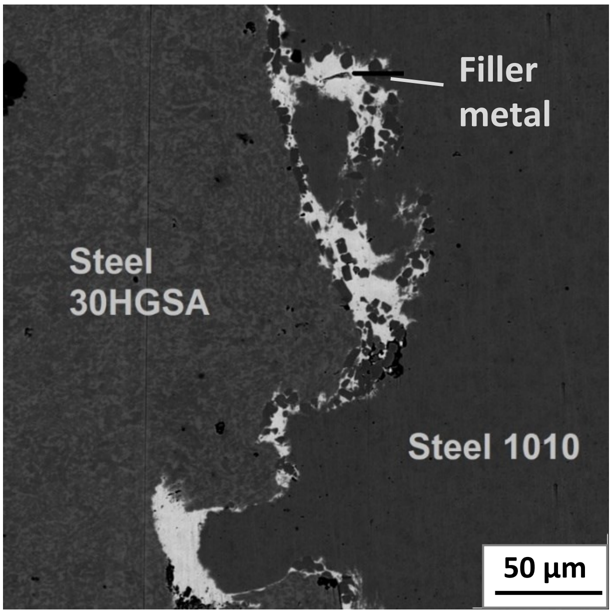


Fig. 6. *The structure of the brazed seam at its thickness less than 50 µm*
Рис. 6. *Структура паяного шва при его толщине менее 50 мкм*

the entire seam. Analysis of the element distribution profiles indicates that these are iron and nickel silicides (Fig. 7). The size of the inclusions reaches 10 µm.

The formation of a continuous layer of iron and nickel silicides led to a four-fold decrease in shear strength compared to the sample where the silicon content was 0.2 wt. %. The fracture surface runs along the brazing alloy – steel interface along this layer (Fig. 8).

DISCUSSION

Influence of silicon on the structure of ingots and pressed bands

The cast structure of the Cu55Ni6Mn4Zn35 (MNMts55-6-4) alloy of industrial production is characterized by the presence of inclusions at the grain boundaries. These inclusions contain both silicon and phosphorus. It is obvious that the alloy deoxidation with phosphorus is excessive and even a small content of it provokes the formation of phases at the grain boundaries. The cast structure of the experimental alloy, which does not contain phosphorus, differs from the structure of the industrial sample. The presence of dark areas rich in silicon, manganese and nickel is observed, but they have a less extended shape and are more dispersed, which generally corresponds to the literature data [21].

The use of hot pressing of ingots to produce bands leads to a noticeable crushing of inclusions. The crushing of inclusions occurs when pressing a band measuring 10×1 mm from an ingot with a diameter of 50 mm. The result of pressing the alloy into such a form is a high degree of compression (about 200). With a silicon content of over 0.3 wt. %, inclusions become larger – up to 10 µm or more, compared to 2–4 µm with a low silicon content. The amount of silicon-containing phase increases with increasing silicon content in the alloy. The influence of iron content of approximately 0.2 wt. % on the alloy structure was not revealed.

Influence of silicon on the structure of brazed seams

In brazed seams, except for silicon contained in the brazing alloy, silicon from 30HGSA steel can participate in diffusion processes. In the structure of the seams, the formation of small inclusions of iron and nickel compounds with silicon is observed that are mainly located along the brazing alloy – base metal boundary, which corresponds to the data on laser brazing of steels with Cu–Si brazing alloy [18]. Their concentration increases in the areas of small gaps, however, no continuous layers capable of acting as stress concentrators were detected with a silicon content of 0.1–0.2 wt. %, in contrast to 0.4 wt. % of silicon in capillary brazing and 3 wt. % of silicon in laser brazing. It is obvious that these inclusions are formed due to the dissolution of iron in the brazing alloy and its interaction with silicon, which leads to the formation of silicides in the melt volume located along the steel boundaries. Thus, silicon in the brazing alloy is bound with iron into stable compounds, which can be located both in the volume of the copper-zinc alloy [17] and along the brazing alloy – steel interface without forming a continuous layer [19].

With an increase in the silicon content in the brazing alloy up to 0.4 wt. %, the formation of large clearly defined crystals in the form of a continuous layer along the steel – brazing alloy boundary is observed. In this case, their sizes and concentration are higher on the side of steel alloyed with silicon (30HGSA), which is more consistent with brazed seams with Cu–3 % Si brazing alloy [20]. This confirms the statements about the danger of

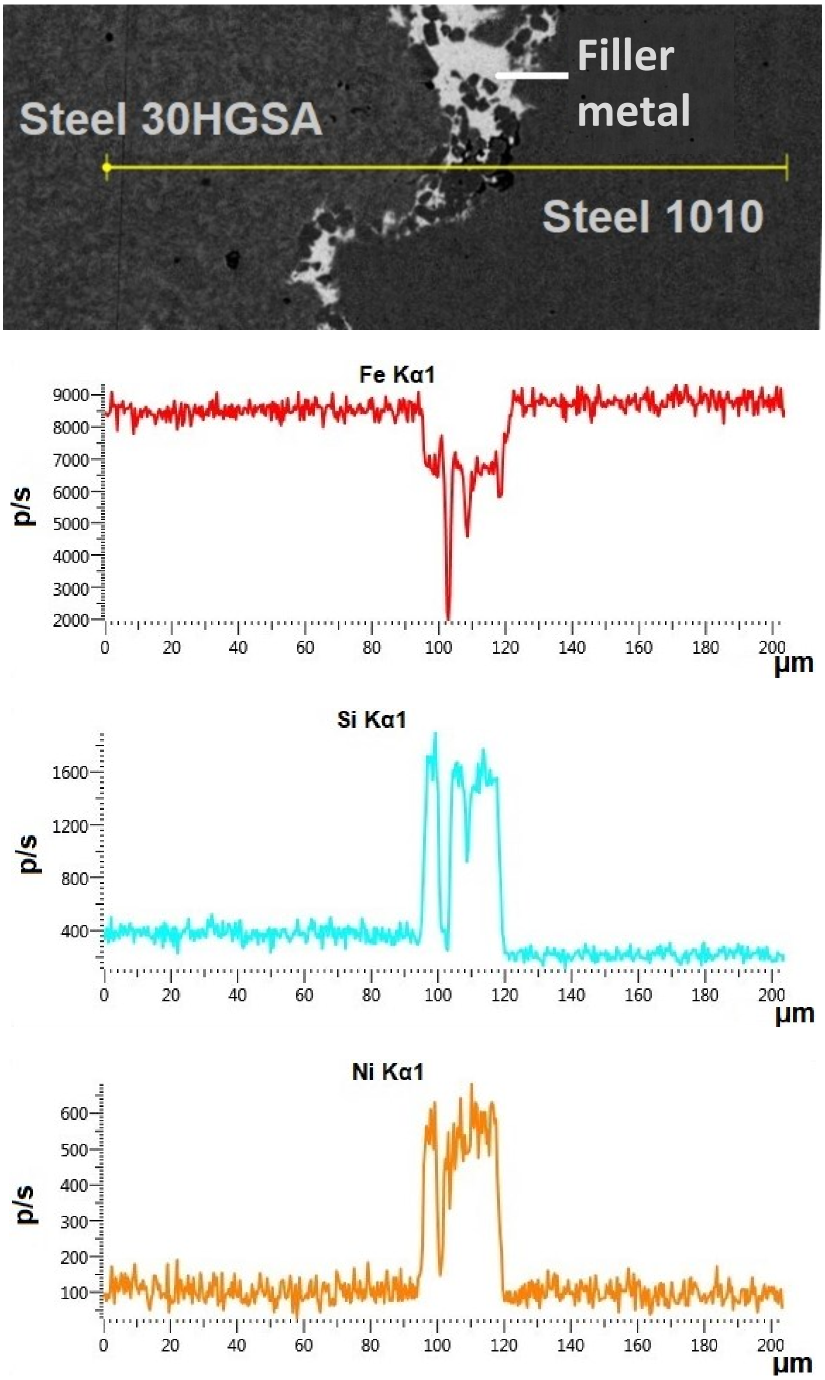


Fig. 7. *Change in the content of iron, silicon and nickel across the brazed seam at a silicon content in the brazing alloy of 0.4 wt. %*
Рис. 7. *Изменение содержания железа, кремния и никеля поперек паяного шва при содержании кремния в припое 0,4 % мас.*

exceeding the silicon content in copper-zinc brazing alloys over 0.3 wt. %. However, for copper-zinc brazing alloys jointly doped with nickel and manganese, we did not find a noticeable influence of nickel on reducing the formation of iron silicides, although it was previously believed that nickel blocks the formation of silicides at the base metal – brazing alloy[4] boundary. Probably, this is because the Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy is not brass, but a nickel silver alloy. Due to the high nickel and manganese content, the solubility of iron in the alloy also changes. It is possible that under given conditions, iron silicides having a lower standard enthalpy of formation than nickel silicides are formed, which is described in [21].

A critical influence of the gap was found when brazing with a brazing alloy with a high silicon content (0.4 wt. %). On the one hand, with a small melt layer of brazing alloy, a change in the curvature of the brazing alloy – steel interface is observed, on the other hand, the crystals of silicon,

[4] *Lashko S.V., Lashko N.F. Payka metallov [Brazing of metals]. Moscow, Mashinostroenie Publ., 1988. 376 p.*

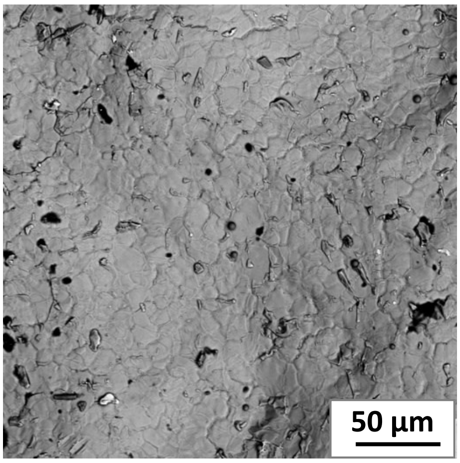


Fig. 8. *Brazed joint destruction along the brazing alloy – steel interface at a silicon content in the brazing alloy of 0.4 wt. %*
Рис. 8. *Разрушение паяного соединения вдоль границы «припой – сталь» при содержании кремния в припое 0,4 % мас.*

iron and nickel compounds become larger and can occupy a significant volume of the seam. These crystals can act as stress concentrators when loading the joints and lead to brittle fracture of the seams similar to the fracture of seams along the steel – brazing alloy boundary in [20]. Previously, this phenomenon of enhanced mass transfer at small gaps has not been described.

CONCLUSIONS

1. The silicon content in the Cu55Ni6Mn4Zn35 (MNMts55-6-4) brazing alloy plays a significant role in the formation of the structure of the brazed seams due to the formation of dispersed inclusions of silicides of complex composition. The presence of residual phosphorus in the alloy leads to the formation of inclusions along the grain boundaries, which can negatively affect the strength properties of the brazing alloy. Deoxidation of this alloy by phosphorus is excessive and has a negative influence on the cast structure.

2. The application of hot pressing of ingots when producing brazing alloy band leads to the crushing of inclusions, but their size and quantity clearly correlate with the silicon content in the alloy. With an increase in the silicon content to 0.4 wt. %, the size of the inclusions and their share in the structure of the brazed seams increase.

3. It was found that the silicon content in the brazing alloy up to 0.2 wt. % leads to the formation of finely dispersed compounds of iron and nickel with silicon due to the dissolution of iron in the brazing alloy melt and the formation of complex silicides in the volume of the seam and along the brazing alloy – base material boundary. These inclusions are distributed unevenly in the seam.

4. When the silicon content reaches 0.4 wt. %, the silicides form a continuous layer along the steel – brazing alloy boundary in the form of larger regular-shape crystals; a high nickel content does not prevent this formation.

5. A critical factor that can affect the properties of the brazed joint is the size of the brazing gap. With a high silicon content, a change in the curvature of the boundaries and the presence of crystals based on iron and nickel silicides in the seams are observed. In this case, a decrease in the strength of the joints and the destruction of the joints along these formations are observed.

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Влияние количественного содержания кремния на структуру припоя Cu55Ni6Mn4Zn и на структуру и свойства паяных соединений

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Аннотация: Медно-цинковый сплав Cu55Ni6Mn4Zn (МНМц55-6-4) широко применяется для пайки твердосплавного инструмента и сталей. Однако наличие кремния в составе сплава (0,1–0,4 % мас.) может приводить к образованию хрупких силицидов железа, никеля и марганца, что негативно влияет на прочность паяных соединений. Цель исследования – определение влияния количественного содержания кремния в медно-цинковом припое, легированном совместно никелем и марганцем, на структуру заготовок припоя перед пайкой и структуру и свойства паяных соединений. В работе использовались методы микроструктурного анализа, включая электронную микроскопию и микрорентгеноспектральный анализ, для изучения распределения силицидов в слитках, лентах и паяных швах. Результаты показали, что при содержании кремния до 0,2 % мас. силициды образуют мелкодисперсные включения, равномерно распределенные в объеме шва. Однако при увеличении содержания кремния до 0,4 % мас. наблюдается формирование сплошных слоев силицидов железа вдоль границы «припой – сталь», что приводит к хрупкому разрушению соединений при механических нагрузках. Особенно критичным оказалось влияние малых зазоров при пайке, где образование крупных кристаллов силицидов железа значительно снижает прочность соединений. Научная новизна работы заключается в установлении оптимального содержания кремния в сплаве (не более 0,2 % мас.) для минимизации негативного влияния силицидов на свойства паяных соединений. Полученные результаты могут быть использованы для разработки технологических рекомендаций при производстве припоев и пайке сталей, что позволит повысить надежность и долговечность паяных соединений в промышленных условиях.

Ключевые слова: пайка твердосплавного инструмента; Cu55Ni6Mn4Zn (МНМц55-6-4); влияние кремния на паяные соединения; силициды железа в паяных швах; микроструктура паяного шва; хрупкость паяных соединений; оптимизация состава припоя.

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