Development of nickel-based filler metal for producing high-strength joints in critical products from heat-resistant materials*

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Manufacturers of nozzles for liquid rocket engines from heat-resistant austenitic and austenitic-ferritic steels faced the problem of brittle boron nitride formation in the joints. Joints are obtained using high-temperature diffusion brazing in vacuum by filler metal based on Ni – Cr – Fe – Si – B system. This technology is based on the isothermal solidification of the filler metal melt, occurring due to the diffusion of boron into the base material.

The developed boron-free nanocrystalline thin film filler metal Ni – 8Si – 5Nb wt.% was proposed to solve the brittle boron nitride problem. The melting interval 1103–1120 °C of the filler metal is determined by the method of differential thermal analysis. It’s structural and phase state was revealed by *X*-ray methods. Technological modes of high-temperature diffusion brazing of austenitic and austenitic-ferritic steels have been developed in the temperature range of 1150–1200 °C. The metallographic studies of the brazed joint were carried out. To determine the joint strength characteristics, the microhardness was investigated in the brazed zone and mechanical uniaxial tension tests were carried out according to GOST (Russian State Standard) 28830–90.

The best result on the tensile strength 450±30 MPa was achieved on samples obtained at an isothermal holding temperature of 1150 °C for 30 min.

As a result of this work, the possibility of using boron-free nanocrystalline thin film filler metal for brazing heat-resistant nitrogen-containing steels has been demonstrated. High-strength joints were obtained.

Key words: joint, diffusion brazing, filler metal, rocket engine, nozzle, heat-resistant steel, nickel, boron.

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Introduction

Aerospace technology and the creation of modern space and aircraft structures are constantly developing and the production technology is very important. A special place in the improvement and creation of new constructions and materials belongs to engine-building. In the rocket engine industry the introduction of the new materials and technologies is occurring at high rates.

The manufacture of energy-intensive space technology nodes is a complex task. The methods widely used in mechanical engineering is welding and brazing. They have been significantly developed. However, in some cases, welding is not technologically feasible. For example, in the manufacture of liquid rocket nozzles designers use the method of high-temperature diffusion brazing in vacuum. It is implemented at temperatures significantly lower than during welding, and many connections can be obtained in one production cycle, which makes this process the most technologically advanced [1]. This technology is based on the isothermal solidification of the filler metals melt, which occurs due to the diffusion of depressant elements, such as boron, into the base material.

The main problem is the connection of the thin-walled outer part of the cooled nozzle with the inner part without disturbing the structure of the cooling channels. The temperature of the combustion products in the liquid propellant rocket reaches values of 3000–3500 K and above with a pressure in the chamber of 10–20 MPa. This significantly exceeds the melting point of the inner walls of the nozzle. The temperature of the nozzle walls made from heat-resistant steel reaches 700–900 °C.

Among the filler metals used to join thin-walled steel structures, it is convenient to use alloys in the form of thin film filler metal, which provide accurate dosage and minimal degradation of the structure of the base material in the brazing process [2]. In this regard, rapidly quenched amorphous and nanocrystalline filler metals in the form of films with a thickness of 20...80 μm have been widely used [3]. Such filler metals are obtained by the technology of ultrafast quenching from the melt to a rotating refrigerator

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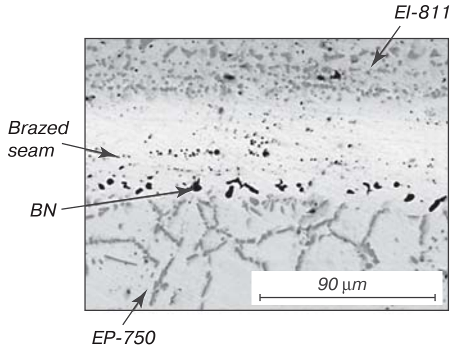

Fig.1. Formation of boron nitride phases in a brazed joint

disk with a speed of $10^4...10^6$ K/s. High cooling rate allows at room temperature to obtain alloys with the structure of a supercooled liquid, in which elements are evenly distributed throughout the volume, which gives certain advantages to the filler metal in the process of melting and interaction of the melt with the base materials. The use of such technology allows to obtain flexible thin film filler metals from hard-to-deform ingots, which have a whole set of advantages over their crystalline counterparts obtained by traditional methods: they have a homogeneous phase state, are characterized by narrow melting and solidification intervals, high adhesion and capillary activity. All this allows improving the quality of brazing, reducing the number of defects of brazed joints, reducing the degree of formation of intermetallic compounds in the seams [4].

However, doping of boron is necessary to obtain nickel filler metals with a lower melting point in the amorphous or nanocrystalline state using fast quenching [5–6]. For example, Ni – Cr – Fe – Si – B alloys with high content of silicon and boron are used to connect austenitic nitrogen-containing Russian steel EP-750 (ЭП-750) with austenitic-ferritic Russian steel EI-811 (ЭИ-811) of nozzles. In this case, brittle boron nitride particles are formed in the brazed seam, which generate into the stitch structure along the seam [7] and under certain conditions can greatly reduce the mechanical characteristics of the joints, which in turn leads to abnormal situations until the engine fails (Fig.1).

Therefore, a current task is to develop new high-temperature filler metal that do not contain boron, suitable for brazing steel combustion chambers of rocket and space technology engines, which are characterized by a lower brazing temperature and enhanced mechanical properties. Since the extreme conditions of rocket engine nozzle operation: nozzle will be subjected to intense thermal effects, — serious requirements are imposed on joints made by brazing and, accordingly, filler metals. Diffusion brazing of steels by developed filler metals should be carried out at temperatures not higher than 1200 °C for 15–30 minutes. As a result, the tensile strength of the joint under uniaxial tension should be at least 400 MPa.

Experimental

The compositions of heat-resistant Russian steels EP-750 and EI-811 are presented in Table 1.

For the ingot manufacture of future filler metals based on Ni – Si –Nb system, the following materials were used as initial materials: nickel of grade N-0 (GOST 849–70 or Russian State Standard 849–70), semiconductor silicon (GOST 19658–74) and niobium of technical purity. The ingots were obtained by the method of induction re-melting. To ensure a homogeneous chemical composition, silicon and niobium were introduced in the form of Ni – 30Si wt.% and Ni – 23Nb wt.%. Further in the work, the percentage of elements will be indicated in weight. The ligatures were melted in a well arc vacuum furnace with a non-consumable tungsten electrode in an argon atmosphere. Zirconium iodide was used as a getter. The mixture was subjected to five-time remelting by turning the obtained ingots.

Rapidly quenched thin film was obtained by melt spinning on the special installation. The method consists in the following: ingots 700 g smelted by the method of induction re-melting were placed in a quartz crucible, which, in turn, was fixed in a special holder in the working chamber. The initial material is heated and melted using a high frequency inductor. The heating process is controlled with a pyrometer. The material is melted in a crucible and under a pressure of gas a jet of molten metal is squeezed out through a hole. Then it gets on the outer surface of a rapidly rotating copper cooling disk, where it quenches into a thin film filler metal, which is then separated from the disk by centrifugal force. The achieved cooling rate can be 10^4–10^6 °C/s.

The STZ 409 CD installation of the company Netzsch (Germany) was used for differential thermal analysis of the experimental alloys. The rate of heating and cooling during the experiments was 20 °C/min. The working environment in the chamber is argon.

X-ray studies of the samples were carried out on a DRON-3.0 diffractometer using the Bragg-Brentano focusing scheme. The characteristic radiation (K_α) of the copper anode was used. To increase the signal/background ratio and to completely eliminate K_α lines, a monochromator from pyrolytic graphite was installed in front of the counter. For phase analysis, the survey was carried out in continuous mode with recording of the diffraction spectrum. The angular velocity of the detector is 1 degree per minute. The obtained data were compared (taking into account the chemical composition of the sample) with the

Table 1

Composition of brazed steels

Steel	Composition								
	Fe	Ni	Cr	Si	Mn	C	S	P	Other elements
EP-750	Base	14–18	23–26	<0.6	5–7	<0.07	<0.02	<0.04	V = 0.2–0.5, N = 0.3–0.45
EI-811	Base	4–6	20–22	<0.8	<0.8	0.09–0.14	<0.02	<0.04	Ti = 0.25–0.5

spectra of the JCPDS database. The search for possible phases was carried out using the XRAYAN program.

To determine the optimal gap between brazed plates of heat-resistant steels (EP-750 and EI-811), studies were carried out on the filler metal`s melt flow into a wedge-shaped specimen. The tests were conducted in accordance with GOST 20485–75 "Brazing. Methods for determining the gap filling by filler metal". The size of the gap varied in the range of 0–100 µm. A schematic representation of the assembly is shown in Fig.2.

Molybdenum foil 100 µm thick was used as a spacer, which was clamped between brazed plates with geometrical parameters 25×20×2 mm. Plates were mounted directly on the filler metal. The assembly was heated to a temperature of 1175 °C at a rate of 30 °C/min in a vacuum of $1.3{\cdot}10^{-2}$ Pa. in the electric furnace with a tungsten heater and screen insulation. The vacuum furnace with resistive heating allows braze samples at temperatures up to 2100 °C. The holding time of isothermal solidification was 30 minutes.

The brazing of the samples was also carried out in vacuum in the same furnace. For the experiments, cylindrical and rectangular samples from EI-811 and EP-750 steels were used. Cylindrical specimens with a diameter of 10 mm and a height of 76 mm. Rectangular samples up to (20...30) mm wide and (7...10) mm high. Immediately before brazing, the surfaces of samples from EI-811 steel were cleaned with emery paper and washed with alcohol. A 50 µm thick filler metal was attached to the surface of the sample using spot capacitor welding. Brazing alloy was used in one layer and placed directly in the brazing gap before brazing. Brazing of rectangular samples was carried out in clamps or specially made conductors.

In the process, a series of experiments was carried out. The temperature varied from 1150 to 1200 °C with a step of 25 °C and a holding time from 10 to 30 minutes with a step of 10 minutes. The rate of heating to an isothermal holding temperature at brazing was 30 °C/min.

To study the possibility of brazing the technological cooling channels, mock-up samples were obtained. The mock-up samples of liquid rocket engine nozzles with dimensions of 100×100×4 mm are two welded plates, on one of which milling paths for coolant are made after brazing. Heating was carried out under the conditions described above to a temperature of 1150 °C with an holding time for 30 minutes.

Metallographic studies and EDX analysis were carried out in an EVO 50 (Carl Zeiss) scanning electron microscope. The elemental composition of filler metals and brazed joints was studied using an INCA 350 x-act energy dispersive spectrometer (Oxford Instruments).

The mechanical properties under uniaxial tension of brazed joints were determined using a PP100/1 testing machine (Germany). For tests of brazed joints, cylindrical samples with a diameter of 10 mm and a height of 76 mm were manufactured according to GOST 28830–90.

Vickers microhardness values were determined on a microhardness tester model HVS-1000. The measurement was performed by indentation of a diamond pyramid into the surface of the test sample under the action of a load of 100 g, which was applied for 15 seconds. After removal of the load, the microhardness value is determined in accordance with the diagonal length of the print.

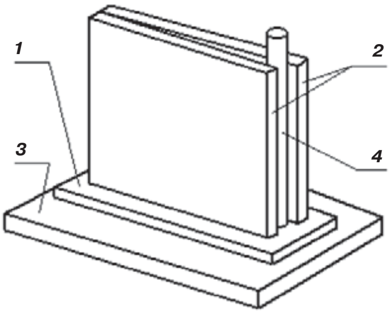

Fig. 2. Assembly scheme for studying filler metal`s flowing into a vertical wedge-shaped gap:
1 — filler metal; *2* — plates of brazed material; *3* — base (quartz); *4* — spacer (Mo foil)

Results and discussion

Based on the composition (see Table 1) of EP-750 and EI-811 steels, Ni was chosen as the base for the filler metal. This is due to the fact that the following requirements are made to the developed brazing alloys: brazing in the temperature range of 1100–1200 °C, the possibility of obtaining a thin film filler metal 40–100 µm thick, which should ensure the absence of brazing effect of technological cooling channels, physicochemical affinity with brazed steels, low cost. Brazing joints, in turn, must withstand high working temperatures (not less than 450 °C for a long time) and have high strength characteristics (temporary resistance to fracture not lower than 400 MPa). The use of Cr, Mn, Pd, Fe as a base cannot meet the requirements for the filler metals and brazing joints. Analysis of the literature data and the experience of obtaining flexible technological thin film filler metals showed that nickel is the most optimal as the base material of the filler metal [8–11]. Moreover, nickel is already contained in brazed steels in the amount of 4–18%, so its use will reduce the erosion of the base material during brazing. The base of brazed steels is Fe with Cr. Consider their interaction with Ni, which is selected as the base for the developed filler metal.

γ-Fe and nickel form between themselves a continuous series of solid solutions at the temperature of brazing (presumably 1150 °C). In the Cr – Ni system, the solubility of Ni in Cr at an eutectic temperature 1345 °C is 32 at.%, at 1000 °C — 10 at.% and at 500 °C — 2 at.%. The solubility of Cr in Ni at an eutectic temperature is 50 at.% and at 700 °C — 36 at.%. At the brazing temperature, a solid solution based on nickel will be formed, since the Cr content in steels does not exceed 26 wt.%.

The next step in the development of the alloy was the choice of the alloying complex. To reduce the melting point, B, C, Si can serve as the most accessible eutectic-forming additives for depressant elements. Of these three elements, the eutectic with nickel with the lowest melting point gives boron — 1100 °C. But in this case, boron is not suitable, since it gives a very refractory BN compound with nitrogen contained in EP-750 steel. Carbon with iron gives resistant carbides. Its additives, like boron additives, will adversely affect the mechanical properties of the brazed joint. Therefore, the second component of the filler metal was selected, it`s silicon — an element that gives the eutectic from the side of nickel, the melting point of which is 1143 °C. In addition, silicon is already included in the steel in appreciable quantities (0.6 wt.%). The concentration of silicon is most appropriate to choose in the range from 8 to 12 wt.%, which corresponds to the hypoeutectic and eutectic composition (see Fig. 3).

The melting points of these alloys are 1300 °C (8 wt.% Si) and 1140 °C (12 wt.% Si). But this is too high a temperature. Brazing temperature of 1150 °C requires that the liquidus temperature of the alloy is no higher than 1115–1120 °C. Therefore, the addition of the third component is necessary. The iron, chromium and vanadium contained in the steels being joined do not give a noticeable effect on lowering the liquidus temperature of nickel alloys. Forming eutectics with nickel, their small additives (up to 5 at.%) practically do not affect the melting point of the alloy. Manganese also does not give a noticeable decrease in the melting points of alloys, both with nickel and silicon. The best additives in binary systems with Me – Si and Me – Ni are Ti and Nb. But titanium contained in steel EI-811, gives with nitrogen much more refractory nitrides than niobium. Therefore, preference should be given to niobium, although it is not part of the steel EP-750 and EI-811.

The amount of Nb selected 5 wt.%, based on its maximum solubility at the proposed temperature of the brazing. The addition of niobium shifts the melting point of the Ni – Si alloy to lower temperatures. Based on the known double phase diagrams of Ni – Si, Ni – Nb, Nb – Si, we can offer the following composition for the desired low-melting filler: Ni – base, 8–12% Si, 5% Nb.

To determine the optimal amount of silicon, studies have been carried out on its variation in the Ni – Si – Nb system alloys. The ingots of various compositions were obtained: Ni – 5Nb – 8Si, Ni – 5Nb – 10Si, Ni – 5Nb – 12Si wt.%. Differential-thermal analysis of these samples showed that the lowest liquidus temperature has an alloy with 8 wt.% Si (see Table 2).

A typical curve obtained in the experiments is shown in Fig. 4. The solidus and liquidus temperature of the alloys was determined from the heating curve, since the degree of superheat is lower than the degree of supercooling.

For the low-melting filler metal being developed, the following composition was chosen: Ni – base, 8% Si, 5% Nb.

In the process of manufacturing rapidly quenched microcrystalline thin film filler metals a number of technological factors were taken into account and controlled at the same time, such as process temperature, overpressure of the gas in the crucible during melt extrusion, rotation speed of the quenching disk, crucible nozzle-disk distance the surface of the quenching disk, the width of the crucible nozzle, the mass of the initial melted ingot. For brazing experiments, a filler metal film was selected with a width of 20 ± 2 mm and a thickness of 0.05 ± 0.01 mm.

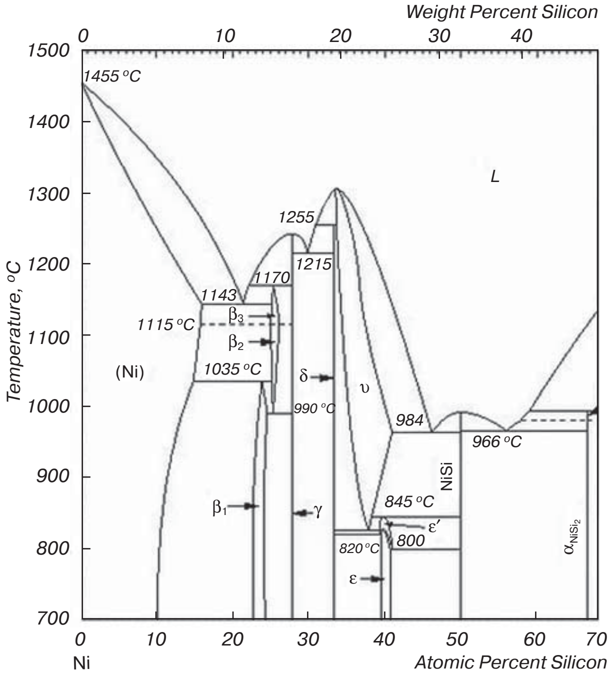

Fig. 3. Part of the state diagram of the system of Ni – Si alloys [12]

Table 2

Liquidus and solidus temperatures of Ni – 5Nb – 8Si, Ni – 5Nb – 10Si, Ni – 5Nb – 12Si alloys

Alloy	Ts, °C	Tl, °C
Ni – 5Nb – 8Si	1087	1120
Ni – 5Nb – 10Si	1096	1135
Ni – 5Nb – 12Si	1120	1140

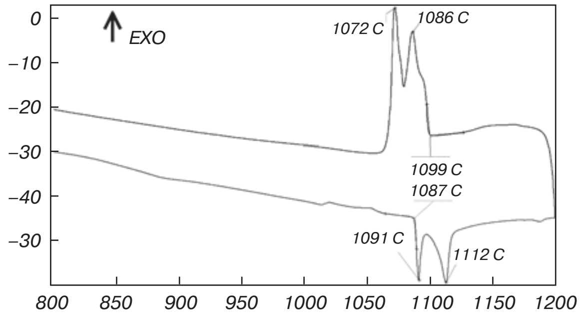

Fig. 4. DTA curve for the initial ingot of the Ni – 8Si – 5Nb alloy

The obtained filler metal was studied using DTA to determine the temperature of phase transformations. The measurement results are shown in Fig. 5. As follows from the data obtained, the rapidly quenched thin film filler metal has a non-amorphous structure (there is no crystallization peak), however, high homogeneity is confirmed by a narrow melting interval. The Ni – 8Si – 5Nb ingot alloy is less homogeneous, as can be judged by the longer melting interval compared to the rapidly quenched thin film filler metal (Fig. 4).

Metallographic studies of the source ingot Ni – 8Si – 5Nb were carried out on a scanning electron microscope. It is established that the alloy has a two-phase structure. *X*-ray microanalysis of these phases showed that one of them is enriched with niobium and has a composition of 67Ni – 22Nb – 11Si, while the second contains silicon and niobium at the level of solubility in nickel and has a composition of 88Ni – 8Si – 4Nb.

Structural phase analysis was performed by using *X*-ray methods on different samples. An ingot, a rapidly quenched thin film filler metal and same filler metal after annealing at 800 °C for 10 minutes were investigated. The main phase in all samples is a Ni-based solid solution. The main differences between the spectra are, first of all, the different correlation between the intensities of the same pairs of diffraction lines that indicates changes in the orientation of the grains in the samples (crystallographic texture). There is much more difference between the thin film filler metal (before and after annealing) and the ingot than between the thin film filler metal before and after annealing. In addition, after annealing the thin film filler metal, the broadening of *X*-ray lines has significantly changed (decreased). This is due both to a decrease in the degree of microdistortions in the sample, and to an increase in the size of coherent scattering regions (CSR). A rough estimate, which assumes that grain size is equal to the CSR value, gives the grain size about 90 nm (in the direction perpendicular to the sample surface). The evaluation was carried out using the Scherrer formula. It was assumed that the shape of the diffraction line is described by the Gaussian distribution. It should also be noted that grains of a certain volume involved in the formation of a diffraction pattern and their size distribution, generally speaking, can have any value. Volume is depending on the area of the irradiated surface and the depth of the effective absorption of *X*-rays. This means that the observed broadening of diffraction lines characterizes average grain size.

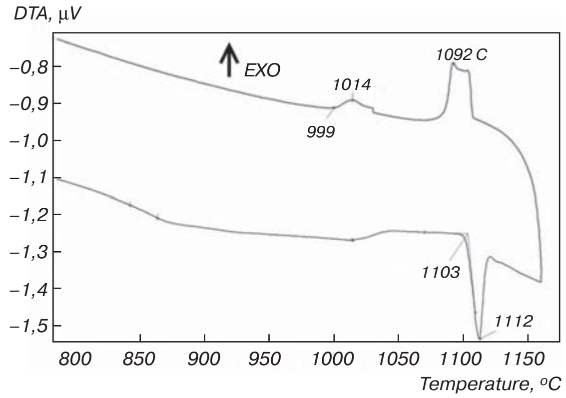

Fig. 5. Heating-cooling curves for filler metal:

The results of metallographic analysis of wedge-shaped samples (Fig. 6) showed that the optimal gap when brazing steel EP-750 and EI-811 with Ni – 5Nb – 8Si filler metal (wt.%) is 50 µm, which corresponds to the minimum content of excess (white) phase in the seam . The structure of the joint with this gap and brazing mode is the most uniform.

The results of metallographic studies of brazed joints showed that during the brazing process, a structure consisting of two phases is formed in the brazed joint zone. The first phase (further phase 1) is a nickel-based compound with a high content of niobium up to 30% and silicon up to 12% (see Table 3). The concentration of elements included in the composition of the steel in phase 1

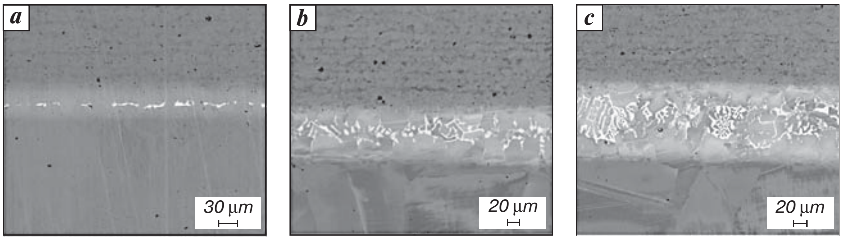

Fig. 6. Microstructure of a brazed joint depending on the thickness of the gap: *a* — 50 µm; *b* — 70 µm; *c* — 100 µm

Table 3

Results of chemical analysis of white formations in the brazed joint EP-750/EI-811, obtained with filler metal Ni-8Si-5Nb under various conditions

Mode	Element, wt.%						
	Si	Ti	Cr	Mn	Fe	Ni	Nb
1150 °C, 10 min	7.96	0.13	1.07	0.26	2.39	59.91	28.25
1150 °C, 20 min	8.21	0.58	1.95	0.32	3.79	56.80	28.23
1175 °C, 30 min	11.09	0.30	1.32	0.32	4.26	51.15	31.54
1200 °C, 10 min	10.72	0.06	3.42	0.75	7.88	50.70	26.45

Table 4

Results of chemical analysis of gray formations in the brazed joint EP-750/EI-811, obtained with filler metal Ni – 8Si – 5Nb under various conditions

Mode	Element, wt.%						
	Si	Ti	Cr	Mn	Fe	Ni	Nb
1150 °C, 10 min	4.00	0.03	8.25	4.01	23.90	59.60	0.19
1150 °C, 20 min	2.79	0.00	10.25	2.24	28.52	55.62	0.46
1175 °C, 30 min	2.71	0.09	12.33	1.71	36.56	45.98	0.58
1200 °C, 10 min	2.95	0.00	11.87	1.61	33.91	48.81	0.81

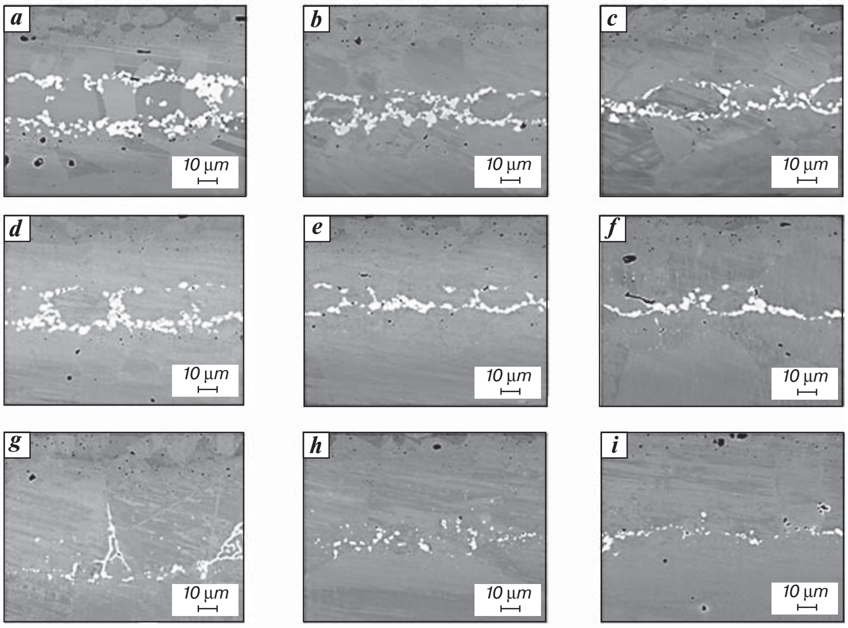

Fig. 7. Microstructure of brazed EP-750/ Ni – 8Si – 5Nb /EI-811 joints for modes: *a* — 1150 °C, 10 min; *b* — 1150 °C, 20 min; *c* — 1150 °C, 30 min; *d* — 1175 °C, 10 min; *e* — 1175 °C, 20 min; *f* — 1175 °C, 30 min; *g* — 1200 °C, 10 min; *h* — 1200 °C, 20 min; *i* — 1200 °C, 30 min

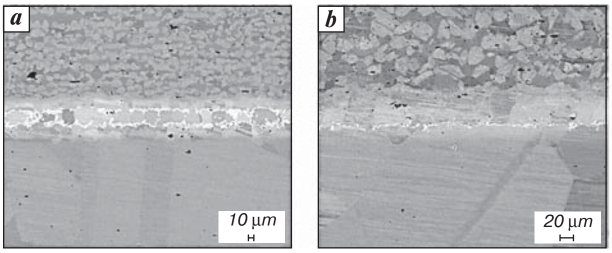

Fig. 8. Microstructure of brazed EP-750/ Ni-8Si-5Nb /EI-811 for modes: *a* — 1150 °C, 10 min; *b* — 1200 °C, 30 min

(white) is insignificant and reaches in the maximum case for iron containing a few percent. It is established that this phase is formed along the grain boundaries of the second phase (further phase 2, gray), which apparently is a nickel-based solid solution that contains Fe up to 24%, Mn up to 4.5%, Cr up to 8% and Si up to 4%. With an increase in the brazing temperature, the nickel content in phase 2 decreases due to an increase in the iron concentration to 35% (see Table 3).

It has been established that with increasing temperature the amount of phase 1 in the seam decreases (Fig. 7); however, the composition of the phase remains almost unchanged. A decrease in the Ni concentration in phase 2 was also detected (see Table 4). The result of such phenomena can be considered diffusion processes occurring in the brazed seam during the brazing process: the filler metal components (Ni, Nb and Si) diffuse into the brazed steel EP-750 and EI-811.

An increase in the holding time from 10 to 30 min at various brazing temperatures leads to a substantial modification of the brazed joint. The amount of white phase decreases. Starting from 1175 °C with an increase in the brazing time, grain growth in the steel EI-811 begins. When brazing at a temperature of 1200 °C for 30 minutes, the grains in steel EI-811 undergo significant growth and their forming takes place (Fig. 8, steel EI-811 is shown above), as a result of which the structure of the steel becomes coarser characteristics.

The microhardness measurements of brazed joints were carried out. The results are presented in Table 5.

The values of microhardness in the brazed seam are slightly higher or almost the same as the value of microhardness in the brazed steels. Abnormally high values of this parameter were not detected in the seam, which means the absence of solid precipitates that can lead to degradation of the properties of the final product during operation.

Table 5

The results of measuring the microhardness of the seams of brazed joints, MPa

T, °C	*t*, min		
	10	20	30
1150	2410±60	2200±20	2300±110
1175	2320±40	2430±20	2700±90
1200	2800±200	2300±110	2060±90

Fig. 9. Microstructure of mock-up samples obtained at 1150 °C for 30 minutes

To determine the mechanical characteristics, tests of cylindrical brazed samples obtained at 1150 °C for 30 minutes were carried out for uniaxial tension. The temporary resistance to the destruction of the joints was (450 ± 30) MPa.

The mock-up samples of nozzle (Fig. 9) showed the absence of sealing of technological cooling channels, which in turn ensures the absence of local overheating. The path geometry has not changed as a result of brazing and filler metal interaction with the connected steels.

Conclusion

The boron-free nanocrystalline thin film filler metal Ni – 8Si – 5Nb wt.% for high-temperature diffusion brazing nozzles for liquid rocket engines from heat-resistant austenitic and austenitic-ferritic steels was developed.

It was determined by *X*-ray analysis that the filler metal has a nanocrystalline structure with a grain size of ~ 90 nm.

It has been established that the melting interval of the developed filler metal is 1103–1120 °C. This is 15 °C less compared with the crystalline alloy of the same composition.

The optimal gap size between the brazed steels, at which the second phase formation (Ni – 30Nb – 10Si wt.%) is minimal, was determined. It was 50 μm.

Technological modes of high-temperature diffusion brazing of austenitic and austenitic-ferritic steels have been developed in the temperature range of 1150–1200 °C. The best result on the tensile strength 450 ± 30 MPa was achieved on samples obtained at an isothermal holding temperature of 1150 °C for 30 min. This is the optimum mode and this isn`t accompanied by grain growth in austenitic-ferritic steel. No overlap of technological cooling channels by filler metal was found on model samples of nozzles.

As a result of this work, the possibility of using boron-free nanocrystalline thin film filler metal for brazing heat-resistant nitrogen-containing steels has been demonstrated. High-strength joints were obtained.

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