Alloying-dependent microstructure influence on corrosion resistance of AISI 321 cell joints brazed by Ni-based filler metals

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Cellular structures that are widely used as filters and heat exchangers usually operate under high loads and aggressive environments. Corrosion attack can lead to the destruction of the most vulnerable elements of the structure and, as a result, to the failure of the device. This study is devoted to the influence of the elemental composition of nickel-based brazing alloys on the corrosion resistance of brazed joints. Nickel-based brazing alloys based on Ni – Cr – Si – B (BNi-2, BNi-5a) and Ni – Cr – P (BNi-7) systems, and experimental compositions, were selected for the study. The brazing modes were selected according to differential thermal analysis (DTA). The microstructure of the joints was studied using energy-dispersive *X*-ray spectroscopy (EDS) on a scanning electron microscope (SEM). The effect of brazing temperature and holding time on grain size and corrosion resistance were evaluated. Corrosion tests were performed in a boiling mixture of $CuSO_4$ and H_2SO_4 solutions for 8 hours. The obtained microstructures of the brazed joints with different filler metals and different braze modes, before and after corrosion tests, were compared. The erosion activity of brazing alloys was evaluated, and it was found that an increase in the amount of chromium reduces the damages caused by erosion. The relationships between the chemical composition of the filler metals and the brazed joint, the structural-phase state of the joint and the level of corrosion damage are revealed. The influence of elements such as boron, silicon, molybdenum, phosphorous and chromium on the corrosion resistance of the brazed joint is shown. It was found that BNi-5a, BNi-7 and FM04 show the best corrosion resistance. In brazed joints obtained with low-chromium filler metals, a strong dissolution of the zone adjacent to the base material was detected. The purpose of the study is to determine the influence of elements often used in brazing alloys, as well as the structural-phase state, on the corrosion resistance of the brazed joint.

Key words: Nickel based filler metal, transient liquid phase bonding, TLP-bonding, brazing, microstructure, fillet, austenitic steel, grain growth, temperature characteristic, corrosion test

DOI: 10.17580/nfm.2020.01.07

Introduction

Brazing and welding are the most popular methods in engineering for creating permanent joints. However, in some cases, welding is not possible due to technological problems. For producing cell and thin-walled structures, high temperature brazing is often used as the only possible technology. Nowadays diffusion bonding is widely used for joining materials. Using diffusion brazing or transient liquid phase bonding (TLP-bonding) [1], enables the production of joints with a high resistance to high temperature, mechanical stress and influence of aggressive environments for a long time, without significant property degradations [2]. This technology is based on the process of isothermal solidification of melted filler metal due to elemental diffusion. Nanocrystalline nickel-based foils, produced by the rapid solidification of the melt, are perspective for TLP-bonding of stainless steels and alloys [3]. Such filler metals with a thickness of 30–70 μm enable the control of the amount of liquid in the seam — an important factor for TLP-bonding.

Brazed joints are usually part of high stressed constructions, which are exploited under high pressure, temperature, and in aggressive environmental and friction conditions. Researching the properties of brazed joints and the possibilities of producing them is an actual problem. It is important to establish the alloying-dependent microstructure influence of the filler metal and brazing mode influence on the mechanical properties and corrosion resistance of brazed AISI 321 joints. These influences on corrosion resistance are considered in this work. An investigation was undertaken using typical for brazing type 300 (AISI 304, 316, 321) stainless steel nickel-based filler metals based on the Ni – Cr – Si – B (BNi-2, BNi-5a) and Ni – Cr – P systems (BNi-7), and experimental ones [4].

Materials and methods

Pure Ni (Russian State Standard 849–70), electrochemical Cr (Russian State Standard 5905–2004), pure Mo (Russian State Standard 14316–91) and carbonyl Fe (Russian State Standard 13610–79) were used as initial materials for the production of crystal ingots of alloys based on the system Ni – Cr – Si – B. B and Si were alloyed as ligature Ni – 15%B and Ni – 30%Si wt.%.

The original ingots of the alloys were obtained by melting in an arc vacuum furnace with a non-consumable tungsten electrode with five-time re-melting in an argon atmosphere.

To prepare cell samples for brazing, plates of AISI 321 steel (0.5 mm thickness) with thickness of grooves (where other plates are inserted) ensuring a gap <50 μm, were used. For a satisfactory brazing result, the gap between the joints should not exceed 100 μm [4]. The filler metal was placed on the outside so filler metal melt would flow into the capillaries. A uniform amount of foil was used.

The assembly was fixed in a special jig, so that during the heating/cooling cycle of the brazing a uniform pressure was ensured over the entire surface. The scheme of the brazing assembly is shown in Fig. 1.

The compositions of steel and filler metals used in the work are presented in Table 1.

The compositions of the filler metals were selected in such a way that it is possible to consider the alloying-dependent influence on the properties of the joint. BNi-2 is compared with FM02 to investigate the influence of silicon increasing and boron decreasing. BNi-7 is compared with FM03 to investigate influence of replacing boron and silicon by phosphorus. FM01 is compared with FM02 to investigate the influence of iron. BNi-5a is compared with FM04 to investigate the influence of molybdenum. FM02, FM03 and FM04 are compared with each other to investigate the influence of the chromium content. BNi-2 and BNi-7 were also chosen as reference filler metals. In the work [5], AISI 316L joints brazed by Ni – 7Cr – 5Si – 3Fe – 3B (BNi-2), Ni – 18Cr – 7Si – B, Ni – 2Cr – 0.5Si – 0.5 – B – 8P, and Ni – 25Cr – 1.5 Si – 0.5B – 6P wt.% were investigated after corrosion tests. It was established that samples brazed with BNi-2 show the worst corrosion resistance against filler metals with phosphorus. Based on this, BNi-2 was selected to be the reference filler metal with the worst corrosion resistance and BNi-7 was selected to be the reference filler metal with the best corrosion resistance.

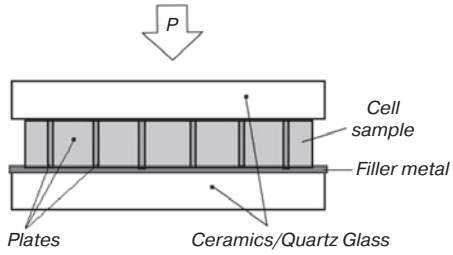

Fig. 1. Scheme of the assembly for brazing

Brazing foils were obtained from the ingots by the rapid solidification of the melt in a special installation. For further experiments, foils in the form of a ribbon 10 ± 2 mm wide and 45 ± 5 μm thick were selected.

The critical temperatures of phase transformations in the experimental filler metals (FM01-FM04) were determined using differential thermal analysis (DTA) with a SDTQ600 thermal analyser. A fixed heating and cooling rate of 10 °C/min was used for all samples. The brazing modes were selected based on the results of the DTA. Recommended brazing temperatures were chosen for typical filler metals according different previous investigations [4, 6–7]. For the low temperature eutectic filler metals BNi-2 and BNi-7, lower temperature and shorter holding times were chosen, 1100 °C for 15 min and 1020 °C for 15 min, respectively. For the high temperature filler metal BNi-5, a higher temperature and longer holding time were chosen, 1160 °C for 40 min.

For the brazing, a vacuum furnace with resistive heating was used which provided a vacuum of up to $1.3 \cdot 10^{-3}$ Pa. The brazing mode depends on the composition of the filler metal since the composition affects the melting range. To equalize the temperature field of the jig, an additional heating stage was used at a temperature of 900 °C for 15 minutes. The heating rate was 20 °C/min, and the cooling occurred within the furnace. The temperature was controlled by a tungsten-rhenium thermocouple.

Before optical microscopy, the samples were polished and etched with acids HNO_3 + HCl, in a ratio 1:3, to

Table 1.
The chemical composition of used materials

Alloy name	Analogue	Purpose	Alloy composition, wt.%									
			Fe	Ni	Cr	C	Si	Mn	Mo	B	Ti	P
Fe18Cr10NiTi	AISI 321	Base metal	bal.	10	18	0.02	0.4	0.5	–	–	0.1	–
–	BNi-7	Filler metal	0.2	bal.	14	–	0.1	–	–	–	–	10
–	BNi-2	Filler metal	3.5	bal.	7	–	4.5	–	–	3	–	–
FM01	–	Filler metal	–	bal.	7	–	7.5	–	–	1.5	–	–
FM02	–	Filler metal	4	bal.	7	–	7.5	–	–	1.5	–	–
FM03	–	Filler metal	4	bal.	15	–	7.5	–	4	1.5	–	–
FM04	–	Filler metal	4	bal.	20	–	7.5	–	4	1.5	–	–
–	BNi-5a	Filler metal	4	bal.	20	–	7.5	–	–	1.5	–	–

reveal the structure and grain size before and after brazing. Microstructural studies of the brazed seam were carried out by the metallographic method using an optical microscope METAM PB-21-1. The average grain size was determined using the program Structure 5.2 (according State Standard 5639-82, [8]).

Error estimation was carried out according to the formulas 1 and 2, where σ — standard deviation of the grain size x from the average grain size $<x>$, n — number of measurements.

$$\sigma = \sqrt{\frac{1}{n(n-1)} \Sigma_{i=1}^{n} (x_i - <x>)^2} \quad (1)$$

$$x = <x> \pm \sigma \quad (2)$$

The change in grain size Δd was calculated according to formulas 3 and 4, where d — size after brazing, d_0 — initial grain size, ε — relative change in grain size.

$$\Delta d = d - d_0 \quad (3)$$

$$\varepsilon = \Delta d / d_0 \quad (4)$$

For a more accurate study, a scanning electron microscope (SEM) EVO 50 (Carl Zeiss) was used (images were obtained by backscattered electrons (BSE) and by secondary electrons (SE)). The elemental composition of the filler metals and brazed joints was studied using an energy dispersive spectrometer (EDS) INCA 350 x-act (Oxford Instruments).

Corrosion tests were carried out according to the State Standard 6032–2003 in a mixture of 5% $CuSO_4$ solution and 25% H_2SO_4 solution. The volume of the resulting solution was calculated in the proportion of 4–8 cm^3 per 1 cm^2 of the total surface of the samples. The tests were carried out in the presence of copper chips. The samples were in close contact with copper but did not come into contact with each other [9]. The tests were carried out in a boiling solution for 8 hours. The boiling point measured using a thermocouple was 110 °C. At the end of the tests, the samples were washed in running water to remove corrosion products on the surface. For microstructure SEM investigations of the fillet after corrosion, a test layer 100–120 μm thick was removed.

The corrosion damage on the samples was determined according to the scheme shown in Fig. 2.

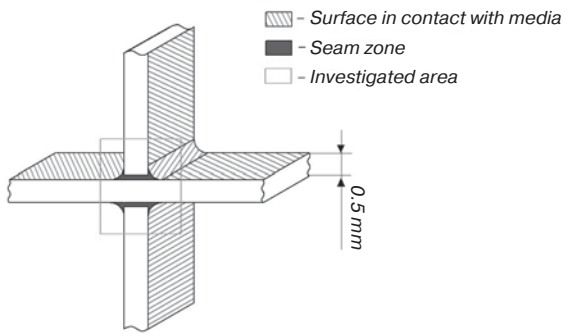

Fig. 2. Scheme of cell sample for metallographic investigation after the corrosion test

Results and discussion

Temperature characteristics of filler metals and brazing modes

The experimental results of the determination of the phase transformations temperatures for the filler metals FM01-FM04 are shown in Table 2.

The temperature mode of brazing was selected based on the obtained data. Heating should be carried out to a temperature of 1160 °C, which is higher than the liquidus temperatures for all the experimental filler metals by up to 30–50 °C. Holding times of 40 minutes during brazing were used to ensure total completion of the TLP-bonding [4]. Since silicon has a low diffusion coefficient in steel compared to boron, when using filler metals with a high silicon content a longer exposure time is required otherwise, brittle, silicide phases form in the soldered joints [10].

The holding time and overheating above the liquidus temperature strongly affect the properties of brazed steel joints produced using nickel filler metals and have been well reported in the literature [11–13].

The selected modes are shown in Table 3.

Grain growth

During the brazing process, recrystallization takes place in the base material. As the holding temperature increases, the final grain size grows exponentially. An increase in holding time also leads to an increase in grain size. Growth occurs according to the logarithmic law [14]. Experimental data on grain growth are shown in Fig. 3.

In steel AISI 321 strong grain growth begins above a temperature of 1080 °C [15]. The initial state (grain size, degree of deformation) affects the processes of recrystallization that occurs during brazing. Both brazing modes

Table 2.
Temperature characteristics of used filler metals*

Filler metal	$T_{solidus}$, °C	$T_{liquidus}$, °C	ΔT, °C
FM01	971	1128	157
FM02	958	1111	153
FM03	1049	1115	66
FM04	1078	1125	47

* Error is ± 0.5

Table 3.
Brazing modes

Alloy name	Brazing mode					
	I step			II step		
	Temperature	Time	Heating rate	Temperature	Time	Heating rate
BNi-7	900 °C	15 min	20 °C/min	1020 °C	15 min	40 °C/min
BNi-2				1100 °C	15 min	
FM01				1160 °C	40 min	
FM02						
FM03						
FM04						
BNi-5a						

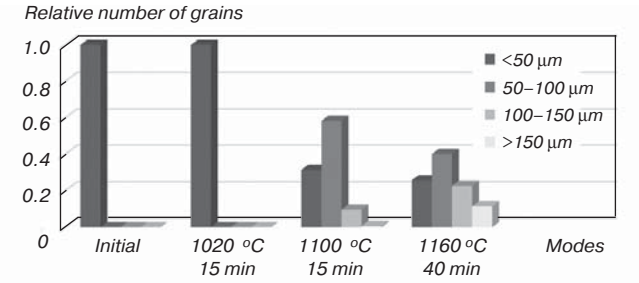

Fig. 3. Grain size distribution diagram for the steel AISI 321 based on experimental data

1100 °C for 15 min and 1160 °C for 40 min cause strong grain growth; there is an abnormal growth of individual grains of steel. The effect of grain size (different brazing temperature) on corrosion resistance will be discussed below.

Microstructure of brazed joints

Fig. 4 shows the microstructure of the fillet for cell samples brazed with different filler metals. Fillets of regular shape were observed in joints obtained with filler metals BNi-2, BNi-7, FM01 and FM02. The fillet area is different due to the different wetting angles of the filler metals.

In the case of filler metals with high chromium content (>15 wt.%): BNi-5a, FM03, FM04 — fillets of an irregular shape are formed due to the weak surface tension of the melt (Fig. 4). The results are consistent with other investigations [16–17]. In the work [17] high chromium nickel-based filler metals (Ni – 19Cr – 10.2Si, Ni – 22Cr – 4.5P – 6.5Si, and Ni –30Cr – 6P – 4Si wt.%), produced by Nicrobraze®, have a bigger flow area than Ni – 7Cr – 4.5Si – 3Fe – 3.1B wt.% (analogue BNi-2). At the same time, a noticeable steel erosion in the area adjacent to the fillet is observed on these cell samples. This effect can be serious and unsuitable for brazing thinner cellular structures than used in this work.

The microstructure of the formed fillets can be divided into three zones (Fig. 5):

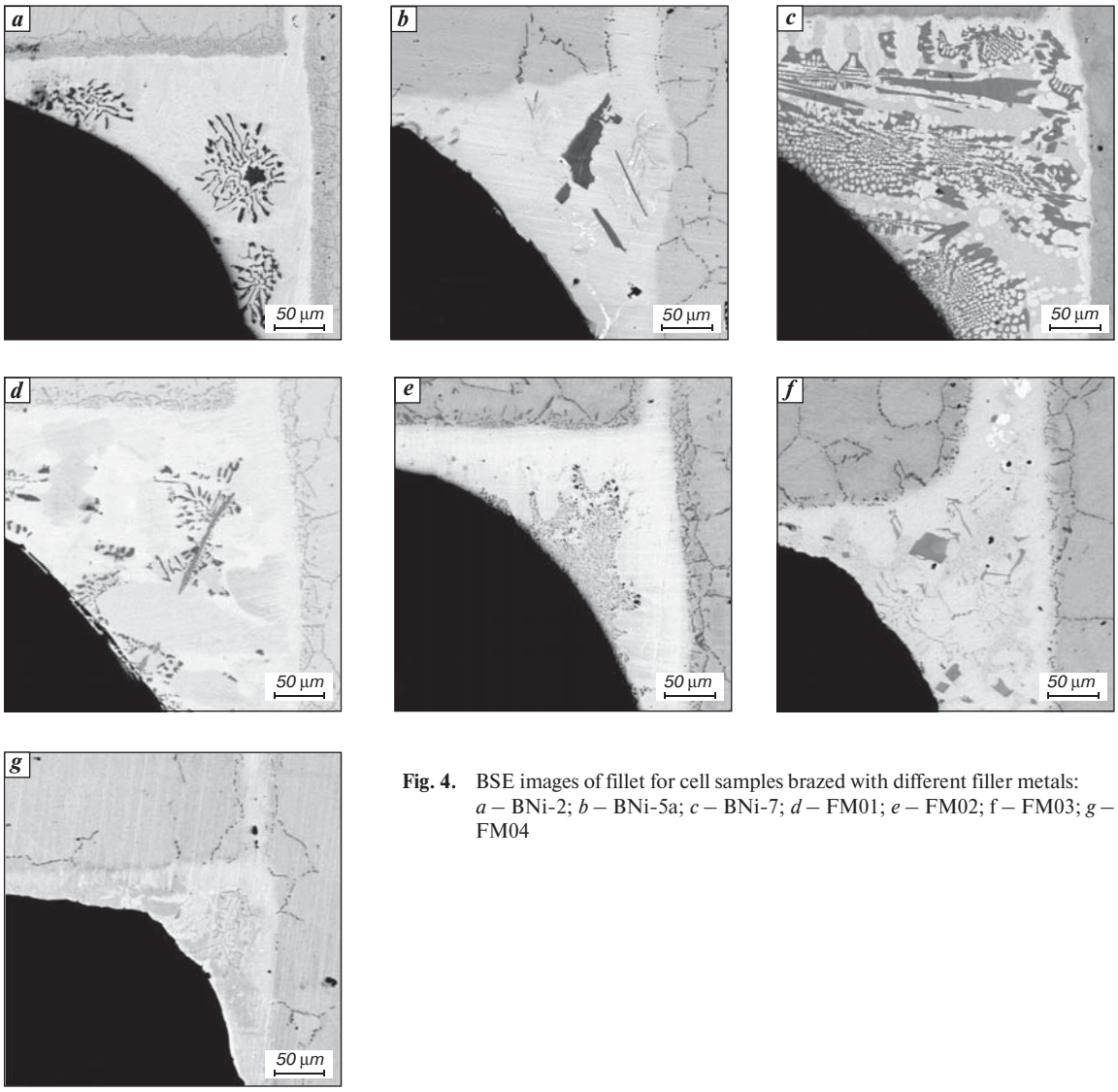

Fig. 4. BSE images of fillet for cell samples brazed with different filler metals: *a* – BNi-2; *b* – BNi-5a; *c* – BNi-7; *d* – FM01; *e* – FM02; f – FM03; *g* – FM04

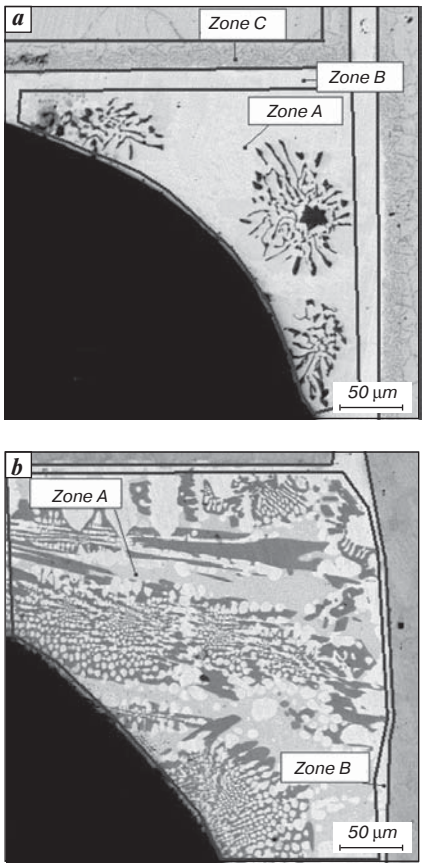

Fig. 5. The microstructure of the formed fillets divided into zones *a* – BNi-2; *b* – BNi-7

– Zone A: the central zone, which contains a large amount of eutectic (a mixture of γ-Ni solid solution and intermediate phases), this zone solidified athermally;

– Zone B: the peripheral zone, which consists mainly of γ-Ni solid solution, this zone solidified isothermally (very small for BNi-7).

– Zone C: the diffusion zone, which contains a large amount of chromium boride phases along steel grain boundaries (not for BNi-7).

In the work [18], similar schemes of fillet zones are given for ultrathin-walled structures from the nickel-based superalloy Inconel 718 brazed with BNi-5 (Ni – 19Cr – 10.2Si, wt.%). Their fillet also consisted of a eutectic island (athermal solidification zone), an isothermal solidification zone which consisted of primary γ adjoined to the base material, and a diffusion affected zone.

In the fillet of samples brazed by BNi-5a and FM04 (a difference in the presence of molybdenum) the amount of borides and silicides are larger than in the fillets of the other samples. Presumably, this is due to the high chromium content and the formation of a degenerate eutectic. A fine boride network in the diffusion zone is noticeable in samples BNi-2, FM01 and FM02 which is due to the low chromium content (7 wt.%) and in the case of BNi-2, also the high boron content.

Compositions of phases formed during brazing by typical nickel-based filler metals have been well-studied in many works, for example [5, 18–20]. In work [18] the phase composition is well described for a fillet obtained from BNi-5 (Ni – 19Cr – 10.2Si, wt.%). In work [19] the phase composition is well described for a fillet obtained from BNi-2. In work [20] the phase composition is well described for a fillet obtained from BNi-1a (Ni – 14Cr – 4.5Si – 4.5Fe – 3B, wt.%). However, studies for experimental alloys containing molybdenum are poor.

To determine the chemical composition of the phases formed during brazing by filler metals containing Mo (FM03 and FM04), energy-dispersive *X*-ray spectroscopy (EDX) was used. It was shown that the structures formed athermally in the centre of the seam are enriched with molybdenum, silicon and boron. They have a complex non-stoichiometric composition. The white phase is a silicide composition: 23.8Si – 14.5Cr – 7.9Fe – 37.0Ni – 16.8Mo, at.%. The dark phase is a boride of the following composition: 40.9B – 48.1Cr – 7.3Fe – 1.2Ni – 2.5Mo, at.%. The formation of such phases is characteristic for joints brazed by nickel-based filler metals containing boron [21–22].

The results of corrosion tests of brazed cell samples

The influence of filler metal composition and brazing modes on the corrosion resistance of brazed joints was investigated. Fig. 6 illustrates the effect of plate dissolution in the area located near to the fillet in alloying-dependence. Fig. 6*b* shows a sample whose surface was not

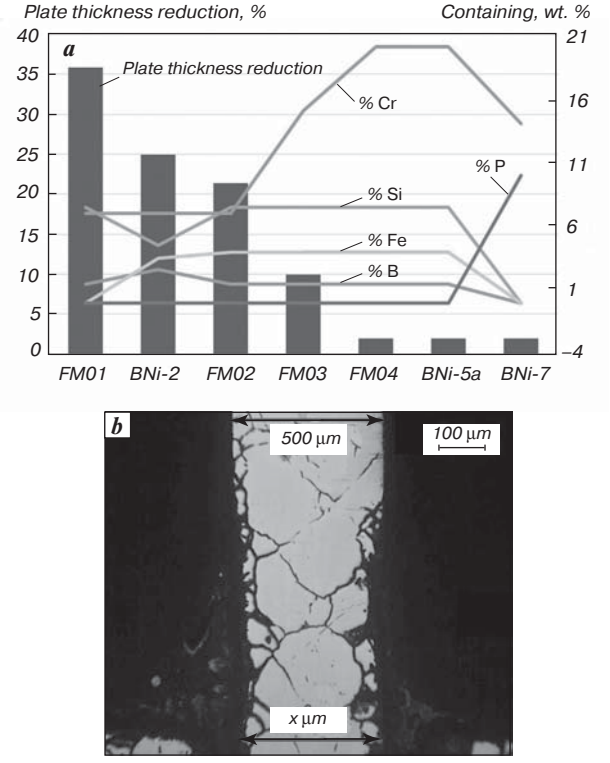

Fig. 6. The effect of plate dissolution in the area located near to the fillet of brazed cell samples after corrosion tests:
a — diagram of the dependence of plate thickness reduction on the elemental composition; *b* – optical image brazed cell sample AISI 321/BNi-2

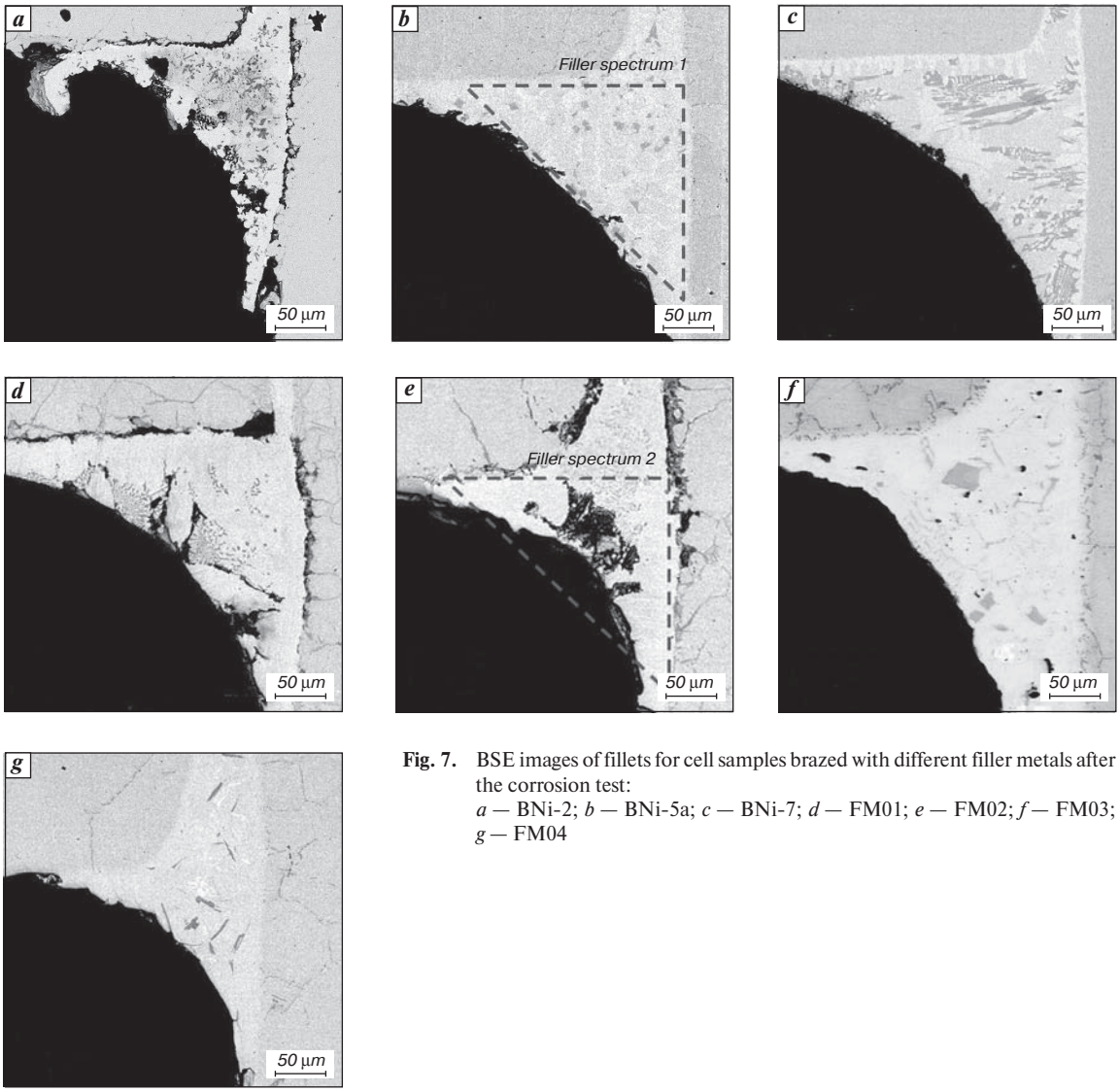

Fig. 7. BSE images of fillets for cell samples brazed with different filler metals after the corrosion test:
a — BNi-2; *b* — BNi-5a; *c* — BNi-7; *d* — FM01; *e* — FM02; *f* — FM03; *g* — FM04

polished after the test. The grain boundaries are etched after boiling in an acidic medium due to the contact of the end face of the sample with a braze ribbon (according to Fig. 1) and the formation of a diffusion region saturated with chromium borides along the steel boundary during brazing. The effect of plate dissolution relates with erosion occurring during brazing. As mentioned above, after the brazing a noticeable steel erosion occurred in the area adjacent to the fillet. Filler metals with high chromium content: BNi-5a, FM03 and FM04, – have stronger steel erosion, but it is established that they have a minimum effect on plate dissolution compared to a filler metal with low chromium content: BNi-2, FM01 and FM02, which have a less noticeable steel erosion in the area adjacent to the fillet.

Comparison of two samples brazed with FM01 and FM02 (a difference in the content of 4 wt.% iron) shows that adding Fe to the filler metal leads to a reduction of the effect of plate dissolution by 11%. The presence of 4% iron leads to the need for less dissolution of iron from the steel to achieve an equilibrium state.

Comparison of two samples brazed with BNi-2 and FM02 (a difference in the content of silicon and boron) shows that replacing boron with silicon in terms of reducingw the effect of plate dissolution, is justified. This can be explained by the brazing temperature and the melting interval.The interval of BNi-2 is ~30 °C compared to FM02 – 153 °C. Also, the holding time, in the case of the second one, is longer.

According to Fig. 8, with an increase in the Cr content of the filler metal, the effect of plate dissolution decreases significantly. Comparison of the two samples brazed with FM03 and BNi-7 (the amount of chromium 14–15 wt.%, the difference in element, which reduce of melting point) shows the effect of plate dissolution is noticeably lower. This can be explained by phosphorus reactivity with iron: P creates a refractory phosphide at the contact boundary and then further diffusion occurs through this layer, excluding dissolution. FM03 has a long melting interval, 66 °C, against BNi-7 – 0 °C (eutectic filler metal). Thereby FM04 is equivalent to BNi-7 and BNi-5a, which is the reference filler metal with the best corrosion resistance.

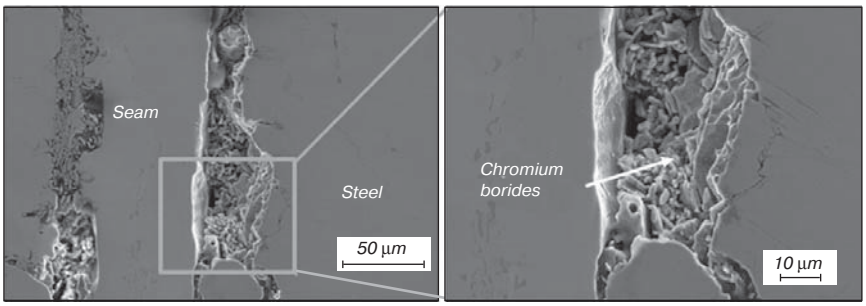

Fig. 8. SE image of fillet for cell sample brazed with FM02 after the corrosion test

FM01 and FM02 are equivalent to BNi-2, which is the reference filler metal with the worst corrosion resistance. FM03 is in the middle.

Fig. 7 shows the microstructure of the fillet for cell samples brazed with different filler metals after the corrosion test. For samples brazed with BNi-2, FM01 and FM02 (7 wt.% of Cr) the corrosion damage is concentrated in the diffusion zone and the central zone along the eutectic, as in the works [23–24]. In the diffusion zone at the junction of chromium boride and a grain of steel, a micro-galvanic pair is formed, and the grain is locally depleted in chromium.

As a result, corrosive action occurs along the grain boundary which leads to its washing out by the medium (Fig. 8). In areas subject to corrosion, particles of chromium borides were found. Their composition is 45.1Cr – 35.2Fe – 2.6Ni – 4.3Si – 7.8B-4.7O wt.%. The oxygen in the oxide film on the surface was also determined. Fillets with a higher chromium content do not show any significant changes.

The samples brazed with the filler metal based on the Ni – Cr – P system (BNi-7) showed the same good corrosion resistance as samples brazed with filler metals based on the Ni – Cr – Si – B system (BNi-5a, FM03, FM04) with high-chromium content (>15 wt.% Cr).

The high corrosion resistance of these samples is explained by the high chromium content in the whole fillets. For example, the chemical composition of the fillets for samples BNi-5a and FM03 is: 59.7Ni – 19.0Fe – 14.9Cr – 6.3Si wt.% (Fillet spectrum 1, Fig. 8) and 57.1Ni – 18.2Fe – 16.0Cr – 5.6Si – 3.1Mo wt.% (Fillet spectrum 2, Fig. 8).

Thereby BNi-5a, BNi-7, FM03 and FM04 show the best corrosion resistance in terms of the microstructure.

According to Fig. 7, it was found that annealing of steel in various modes, leading to recrystallization, does not impair the corrosion resistance of the steel. No corrosion damage was found along the grain boundaries in the steel (excluding the diffusion zone).

Conclusions

The alloying-dependent microstructural influence on the corrosion resistance of a brazed joint was studied.

– Brazing modes 1100 °C for 15 min and 1160 °C for 40 min caused strong grain growth in steel AISI 321 and there was an abnormal growth of individual grains of steel.

– The least effect of plate dissolution in the area located near to the fillet after the corrosion test was observed on high-chromium filler metals based on Ni – Cr – Si – B system (BNi-5a, FM04) and on the Ni – Cr – P system (BNi-7).

– The main factor that reduced the corrosion damage was the concentration of chromium in the body of the brazed joint. At a low content of 7 wt.% Cr, the fillet of the brazed joint degraded in the diffusion zone and the central zone along the eutectic. With increasing concentration of up to 15 wt.% Cr, there were no significant changes in the microstructure of the fillet.

– The presence of intergranular corrosion in the diffusion zone is due to the appearance of a micro-galvanic pair: chromium boride — grain of steel.

– BNi-5a, BNi-7 and FM04 showed the best corrosion resistance in a mixture of 5% $CuSO_4$ solution and 25% H_2SO_4 solution, boiled for a duration of 8 hours.

– Annealing of the steel in various modes, leading to recrystallization, did not impair the corrosion resistance of the steel. No corrosion damage was found along the grain boundaries in the steel.

Acknowledgment

The reported study was funded by RFBR according to the research project №19-32-90092.

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