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THE EFFECT OF SELECTED PROCESS CONDITIONS ON MICROSTRUCTURE EVOLUTION OF THE VACUUM BRAZED JOINTS OF HASTELLOY X NICKEL SUPERALLOY SHEETS

High temperature vacuum brazing is a well-known and commonly used method for joining of nickel based elements and subassemblies of gas turbines, both for stationary and aviation applications. Despite the fact that currently used brazing filler metals meet stringent requirements of aviation and energetic industries, a lot of effort is spent on improving operational properties of the joints through modification of chemical composition or brazing process parameters. This paper aims for both of these aspects – its purpose is evaluation of the impact of filler metal composition, brazing gap width and process conditions on the microstructure of joints between sheet metal elements made of Hastelloy X nickel superalloy. Two different Ni-based filler materials (BNi-2 and Amdry 915) were investigated, based on the results of light and scanning electron microscopy evaluations, energy dispersive X-ray spectroscopy and hardness measurements.

Keywords: Ni-based superalloy; vacuum brazing; brazing filler metal; microstructure; SEM/EDS analysis

1. Introduction

Hastelloy X is a nickel-based superalloy introduced to industrial use in 1953. Its first application was the combustion chamber in Pratt & Whitney JT3 jet engine [1]. Due to its unique set of properties, this superalloy is widely used in aerospace industry and power engineering to this day, still being the material of first choice for many elements in hot section of turbine engines. The most important characteristics of Hastelloy X are the exceptional oxidation, creep and thermal fatigue resistance, operating temperature reaching 1200°C [2] and very good technological properties [3,4]. Apart from choosing proper construction material, another significant aspect of structural components of the modern turbines is selection of appropriate joining method. In industrial practice, welding and brazing are one of the most important and commonly used processes in that field, both at the stage of manufacturing the parts, as well as during their repairs in further life cycle maintenance [5-8]. Among many available brazing techniques, furnace brazing is still a frequently used method in various industry branches [9-11]. In joining of the sheet metal components process, its main advantage is the possibility of joining complex assemblies, made of multiple elements of different shapes and materials, in one operation. In most cases, for joining nickel-based alloys dedi-

cated for elevated temperature applications, high temperature vacuum brazing is used, with the brazing filler metal (BFM) based on nickel, gold or palladium [12]. In this group, Ni-based alloys are often an optimal choice, as a compromise between design and financial requirements [13]. Such fillers have been used in gas turbine construction for many years and are known to offer good corrosion and heat resistance, which makes them able to withstand the detrimental environment of combustion gases [14-16]. However, addition of melting point depressants (MPD) such as boron, silicon or phosphorus can lead to formation of hard, brittle phases concentrated in the centreline area of the joint, which might result in reduction of its strength and durability [17,18]. Industrial popularity of this BFM group provides an incentive for continuous search for improvement, focused either on replacement of currently used MPD in filler alloy [19-21] or investigation of the brazing process parameters impact on microstructure and properties of the joint [22-24].

Tung et al. [25] investigated joints of commercially pure Ni parts brazed with Ni-based BFMs and observed two zones in their microstructure: interfacial layer (adjacent to the base material) and the braze main body. Authors found that the interfacial layer is composed of a γ -phase, i.e. nickel solid solution, whereas the main body is multiphase and can contain binary or ternary eutectics with γ -phase and intermetallic compounds like nickel

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borides, nickel silicides and chromium borides – depending on the chemical composition of BFM used.

Ghasemi and Pouranvari analyzed microstructural evolution mechanisms during brazing of Hastelloy X superalloy using Ni-Si-B [17] and Ni-Cr-Si-Fe-B [26] filler alloys. Three specific zones of joints were distinguished:

- Diffusion affected zone (DAZ) – base material adjacent to the braze, in which grain growth and diffusion-driven boride precipitations can be observed.
- Isothermal solidification zone (ISZ) – single phase, γ solid solution at the joint interface, formed through isothermal solidification occurring as a result of chemical composition changes due to base metal dissolution and MPD diffusion into the base metal,
- Athermal solidification zone (ASZ) – multi-phase area in the centreline of the joint formed at the cooling stage, consisting of eutectic reaction products with intermetallic compounds, such as borides and silicides.

Due to specific phase composition, ASZ is characterized by significantly higher hardness compared to other zones, and its size increases with the increase of the clearance between joined elements.

In present study, Hastelloy X superalloy in a form of sheet was brazed with two Ni-based filler alloys – BNi-2 (Ni-4Si-7Cr-3Fe-3B) and Amdry 915 (Ni-4Si-13Cr-4Fe-2.7B) using three different clearances. Microstructure evolution was investigated using light (LM) and scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) analysis. Moreover, configuration Hastelloy X/Amdry 915 was subjected to additional tests. This BFM is characterized by a wide solidus-liquidus range (960-1127°C) [27]. Interference in the brazing process above solidus temperature, resulting in disruption of the thermal cycle, such as furnace malfunction or power supply break, may be especially problematic for that kind of filler alloy. Therefore, the influence of interrupting the process on various stages of heating within solidus-liquidus range on microstructure of the joints brazed with Amdry 915 was also examined.

2. Materials and experiment

The Hastelloy X superalloy in a form of sheet (AMS5536) was brazed using two Ni-based brazing filler alloys: BNi-2/AMS4777 (Ni-4Si-7Cr-3Fe-3B) and Amdry 915 (Ni-4Si-13Cr-4Fe-2.7B) (TABLE 1). Both analysed filler alloys are typically used for joining stainless steel, Ni- and Co-based superalloys for jet engine turbines and nuclear applications. Amdry 915 filler metal is also reported to be capable of filling gaps up to 0.5 mm [27] (TABLE 2), which makes it suitable for so-called wide gap brazing applications and makes it particularly useful in process of sheet metal parts brazing, where achieving satisfactory fit is often difficult.

Samples with dimensions of 25.4×76.2×1 mm were prepared by washing in ultrasound cleaner, subsequently degreased with ethyl alcohol and fixed in a manner to create lap joint with overlap length of 8 mm (Fig. 1). For each BFM three different gap sizes were used: 0.05, 0.1 and 0.15 mm. Ball-tack welding using 1 mm stainless steel balls was used as a positioning method. Filler alloy was applied in the form of a paste at one edge of the joint. Brazing processes (Fig. 2) were conducted in Seco/Warwick VP-4050/72HV furnace using vacuum protective atmosphere of 0.1 Pa or lower.

Hastelloy X/Amdry 915 joint was also subjected to investigation of the impact of brazing cycle disruption on the microstructure evolution. The same lap joint samples were used in this test (Fig. 1), with brazing gap width of 0.05 mm. Two samples were used for each batch. Original brazing process was interrupted in two points of the heating stage: one within solidus-liquidus range of the BFM (1050°C) and the other at

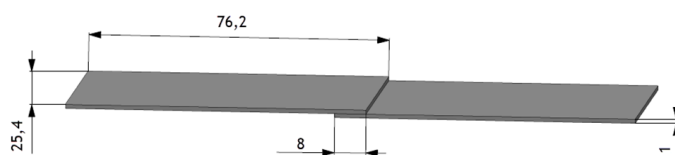


Fig. 1. Geometry of brazed specimens (all dimensions given in mm)

TABLE 1

Chemical composition of investigated alloys [27,28]

Alloy	Chemical composition (% wt.)									
	Ni	Cr	Fe	Mo	Co	W	C	Mn	Si	B
Hastelloy X	bal.	20.5-23	17-20	8-10	0.5-2.5	0.2-1	0.05-0.15	max. 1	max. 1	max. 0.008
Amdry 915	bal.	12-14	4-5	—	—	—	max. 0.06	—	4-5	2.5-2.9
BNi-2	bal.	6-8	2.5-3.5	—	—	—	max. 0.06	—	4-5	2.75-3.5

TABLE 2

Key processing parameters of selected brazing filler metals [27,28]

Processing parameter	BNi-2 (AMS4777)		Amdry 915	
Available forms	foil, powder, paste		foil, powder, paste	
Melting range	Solidus	Liquidus	Solidus	Liquidus
	971°C	999°C	960°C	1127°C
Recommended brazing range	1026-1054°C		1135-1205°C	
Recommended gap size	0.02-0.1 mm		0.05-0.5 mm	

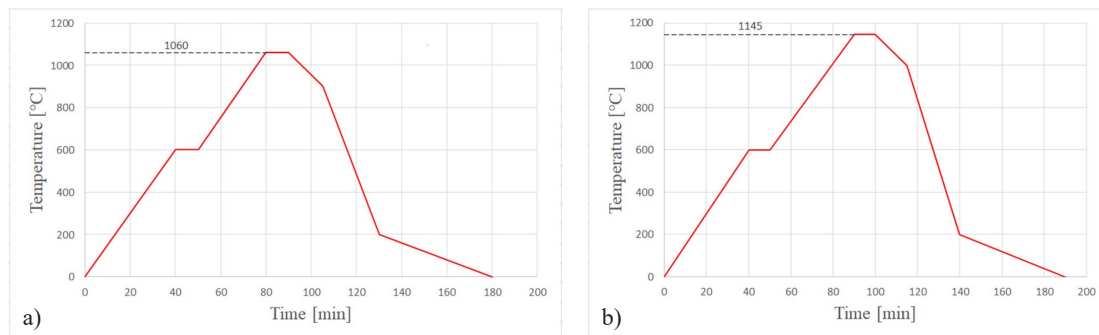


Fig. 2. Brazing cycles for a) BNi-2, b) Amdry 915 filler alloys

the brazing temperature setpoint – 1145°C. Microstructure of the joint was analysed on the first sample directly after cycle disruption. Subsequently, a rebrazing process was carried out on the second sample according to parameters dedicated for the Amdry 915 filler alloy (Fig. 2b) and the microstructure changes were examined.

Specimens for metallographic examinations were prepared using standard methods and etched with a solution of 15 ml HNO₃, 15 ml CH₃COOH, 60 ml HCl and 15 ml distilled water. Microstructure observations were carried out with Leica DM2000 light microscope and Hitachi S-3400N scanning electron microscope using secondary electron (SE) and back-scattered electron (BSE) detectors. Chemical composition in selected areas of the joint was investigated using energy dispersive X-ray spectroscopy. Microhardness measurements were done on Innovatest Nexus 4303 Vickers hardness tester using 0.3 kG load.

3. Results

3.1. Hastelloy X brazed with BNi-2

3.1.1. Microstructure and chemical composition

Obtained joints show a very good fit, with even gap width at the entire length of mating elements (Fig. 3a). Base material microstructure (Fig. 3b,d) consist of equiaxed grains of austenitic γ -phase with twins and precipitations, which based on Hastelloy X precipitation characteristics are believed to be predominantly M₆C carbides [29-30]. Adjacent to the braze, the DAZ was observed (Fig. 3b-d), with the width of 30-40 μ m, independently on the gap size. Multiple, (Cr, Mo)-rich, needle-like and blocky borides precipitation (areas designated as 2 in Fig. 4, TABLE 3) can be observed in that zone. Microstructure of brazed joints is

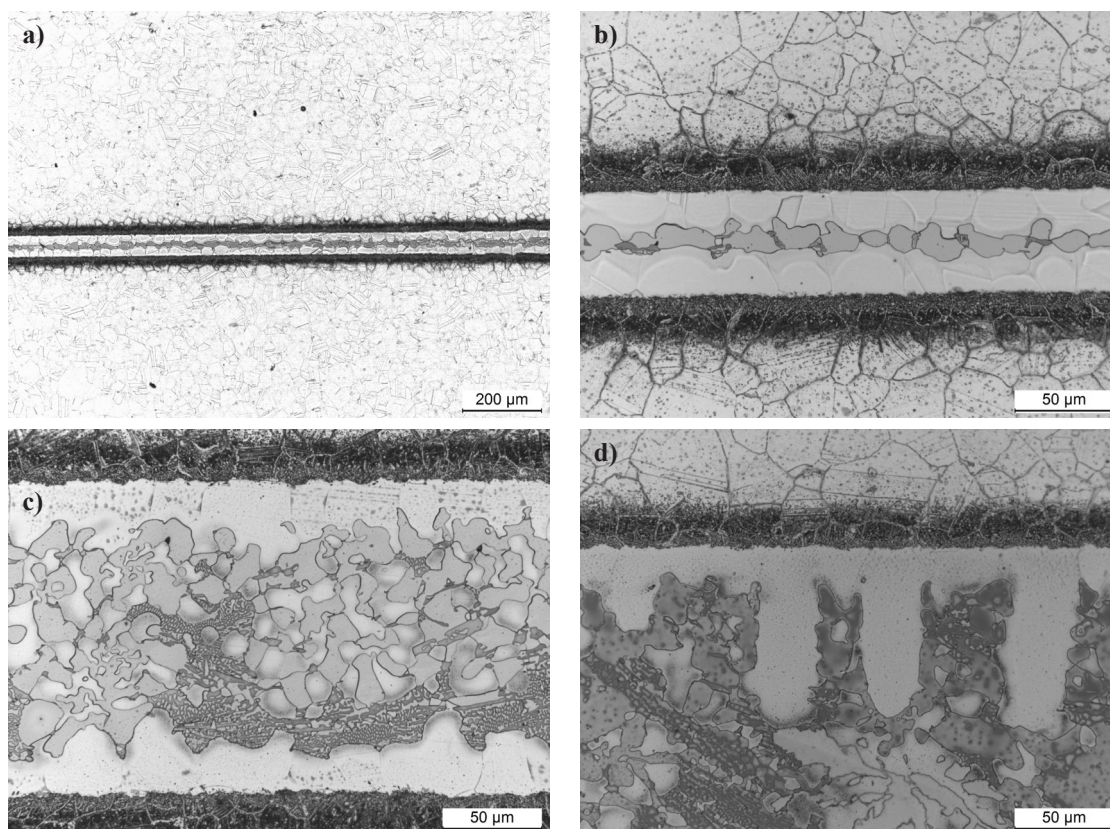


Fig. 3. Microstructure (LM) of Hastelloy/BNi-2 joints with gap width: a,b) 0.05, c), 0.1 and d) 0.15 mm

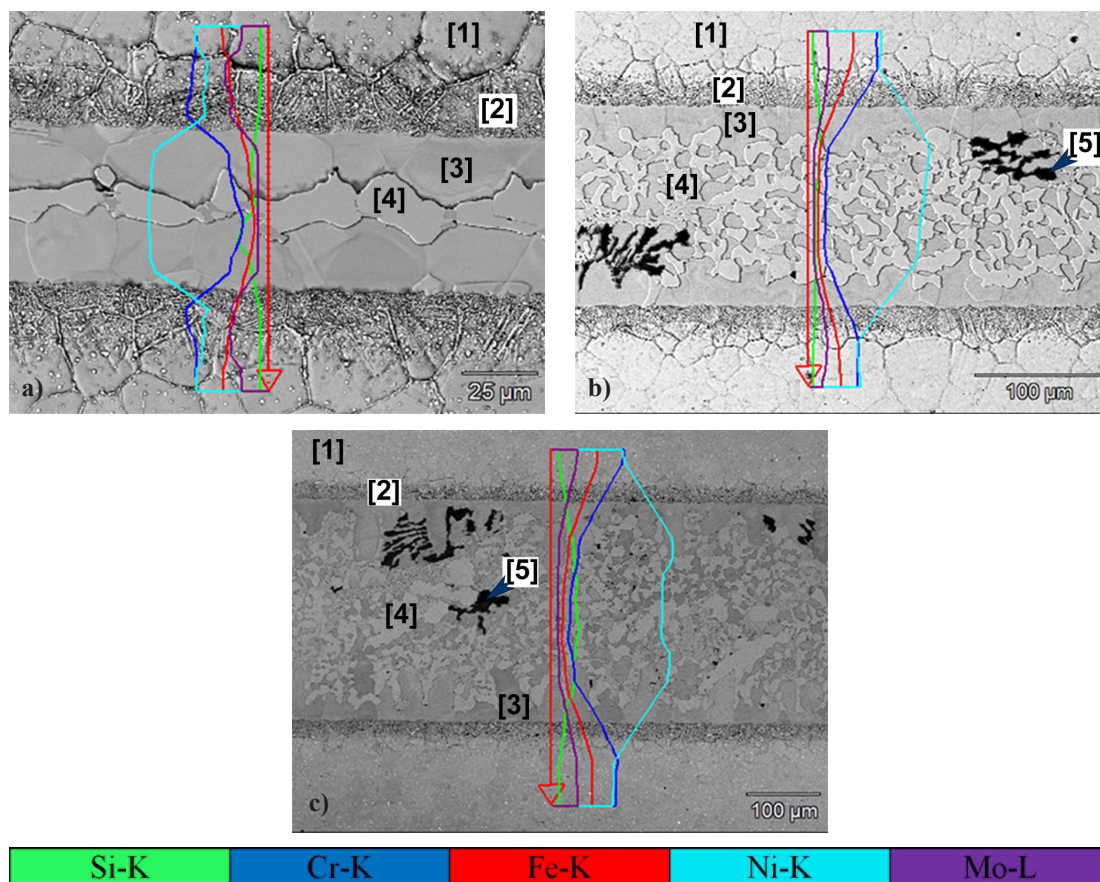


Fig. 4. Microstructure (SEM) and linear elements distribution for Hastelloy X/BNi-2 joints with gap width: a) 0.05 mm, b) 0.1 mm, c) 0.15 mm [1] – γ -phase solid solution and carbides in base material [2] – DAZ – borides in the matrix of γ -phase solid solution, [3] – BOZ – isothermally solidified γ -phase grains, [4] – BIZ – (Ni, Cr) borides (a) and eutectic-type mixture (b, c), [5] – Cr-rich borides in BIZ

composed of clearly distinguishable zones (Fig. 3b-d): braze outer zones (BOZs) – symmetrically arranged in contact with the sheets – and braze inner zone (BIZ) in the centre of the joint. These particular areas can also be referred to as the isothermal and athermal solidification zones, respectively, depending on the crystallization mechanism. Significant impact of the gap size on phase morphology in the braze microstructure was noticed. BOZ is composed of single phase γ solid solution grains: equiaxed in the case of 0.05 and 0.1 mm gaps (Fig. 3b,c) and columnar for 0.15 mm gap (Fig. 3d). The formation of this zone is explained by diffusion-induced isothermal solidification phenomenon, causing changes in liquid phase chemical composition and resulting in the liquidus temperature increase [17]. Such conditions favour the epitaxial growth of γ solid solution grains, even to the columnar form in the case of wide gaps (Fig. 3d). Inner zone of the braze is multiphase, containing the eutectic-like mixture of γ -Ni solid solution and intermetallic compounds. Width of this zone is increasing along with the increase of the gap size, constituting a major part of the entire braze volume in 0.1 and 0.15 mm joints (Fig. 3c,d). BIZ is formed from the remaining, liquid portion of filler metal during cooling, which indicates that holding time was not sufficient enough for isothermal solidification to take place in the entire volume of the joint [17,26].

SEM/EDS linear distribution analysis of the alloying elements (Fig. 4) has proved their diffusion between base material

and BFM. Abrupt changes in concentration of Ni, Cr and Fe can be observed at the interfaces of particular zones. BOZ is composed of Ni-rich γ solid solution (areas designated as 3 in Fig. 4, TABLE 3), with increased amount of Cr and Fe, compared to filler metal nominal composition, which confirms that dissolution of base material by liquid filler metal occurred. Increased content of silicon was noticed in the BIZ, especially in 0.1 and 0.15 joints (Fig. 4b,c; TABLE 3). Cr-rich boride phases (area 5 in Fig. 4b and Fig. 4c, TABLE 3), visible as the black areas on SEM images, were also identified in this region of the same joints (Fig. 4b,c). This allows to conclude that crystallization of silicides and Cr-rich borides occurred in this area, which is typical for braze alloys containing these elements as MPDs [17,22,25,31].

3.1.2. Hardness

Hardness measurements, made along a straight line perpendicular to the cross-section of the joint, indicated noticeably lower hardness of the Hastelloy X comparing to the joint area (Fig. 5). It can be seen that hardness is increasing in the DAZ of base material, which is a consequence of microstructural changes, caused mostly by diffusion of boron from the BFM, reaching the highest value in the BIZ. Maximum hardness of

Chemical composition in selected areas shown in Fig. 4

Gap width [mm]	Area	Chemical composition											
		[% wt]						[% at.]					
		B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L	B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L
0.05	1	—	0.6	23.7	19.6	46.8	8.3	—	1.3	26.4	20.3	46.1	5.0
	2	2.4	0.8	26.6	16.8	40.8	11.9	11.9	1.5	27.0	15.8	36.6	6.5
	3	1.0	3.4	10.7	6.7	76.2	1.9	4.9	6.6	11.0	6.4	69.8	1.1
	4	1.5	0.5	8.2	4.7	83	1.8	7.6	1.0	8.6	4.6	77.0	1.0
0.1	1	—	0.7	24.0	20.7	48.1	5.4	—	1.3	6.4	21.2	46.7	3.2
	2	2.5	0.8	25.1	19.3	43.1	8.5	12.0	1.5	25.1	18.0	38.2	4.6
	3	2.4	3.8	9.2	5.6	79.0	—	11.1	6.9	8.9	5.1	68.0	—
	4	2.8	1.7	5.3	2.8	87.3	—	13.3	3.2	5.2	2.5	75.8	—
	5	2.9	2.7	44.2	3.1	47.0	—	13.1	4.7	40.9	2.7	38.6	—
0.15	1	—	0.6	23.9	20.3	46.3	7.9	—	1.2	26.5	20.9	45.5	4.7
	2	2.2	0.6	24.5	18.9	42.8	10.1	10.7	1.2	25.0	17.9	38.7	5.6
	3	1.8	4.0	8.1	5.7	80.4	—	8.5	7.3	8.1	5.3	70.9	—
	4	2.6	—	5.2	2.9	89.2	—	12.6	—	5.3	2.7	79.4	—
	5	3.2	—	80.8	1.4	4.0	10.6	14.6	—	75.5	1.2	3.3	5.4

that area rises with the increase of the gap between joined elements – from 362 HV0.3 in case of 0.05 mm gap (Fig. 5a), to significantly higher values of 790 and 683 HV0.3 for 0.1 and 0.15 mm joints (Fig. 5b,c), respectively. Hardness distribution on the joints cross-sections confirms previous observations about the phase composition, involving presence of intermetallic phases, which are reported to be very hard and brittle [17,22,31]. The relationship between hardness and gap size indicates that solidification conditions in wider joints favours hard intermetallic phases formation.

3.2. Hastelloy X brazed with Amdry 915

3.2.1. Microstructure and chemical composition

LM observation of Hastelloy X/Amdry 915 joints revealed microstructure (Fig. 6) similar to joints obtained with BNi-2 alloy (Fig. 3), with a slightly deeper diffusion zone of approximately 50 μm (Fig. 6b). Microstructure of the joints was also changing significantly depending on the distance between brazed elements. Joint with a gap of 0.05 mm brazed using Amdry 915

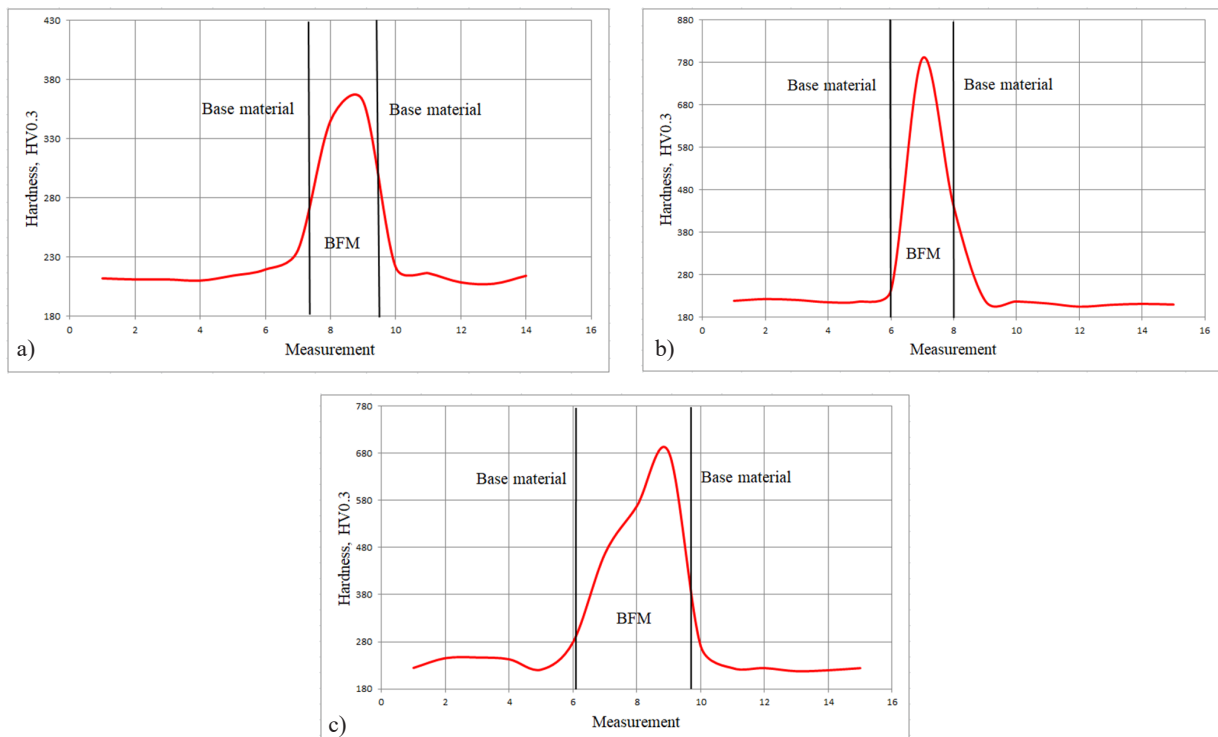


Fig. 5. Hardness distribution on the cross-section of the Hastelloy X/BNi-2 joints for gap width: a) 0.05 mm, b) 0.1 mm and c) 0.15 mm

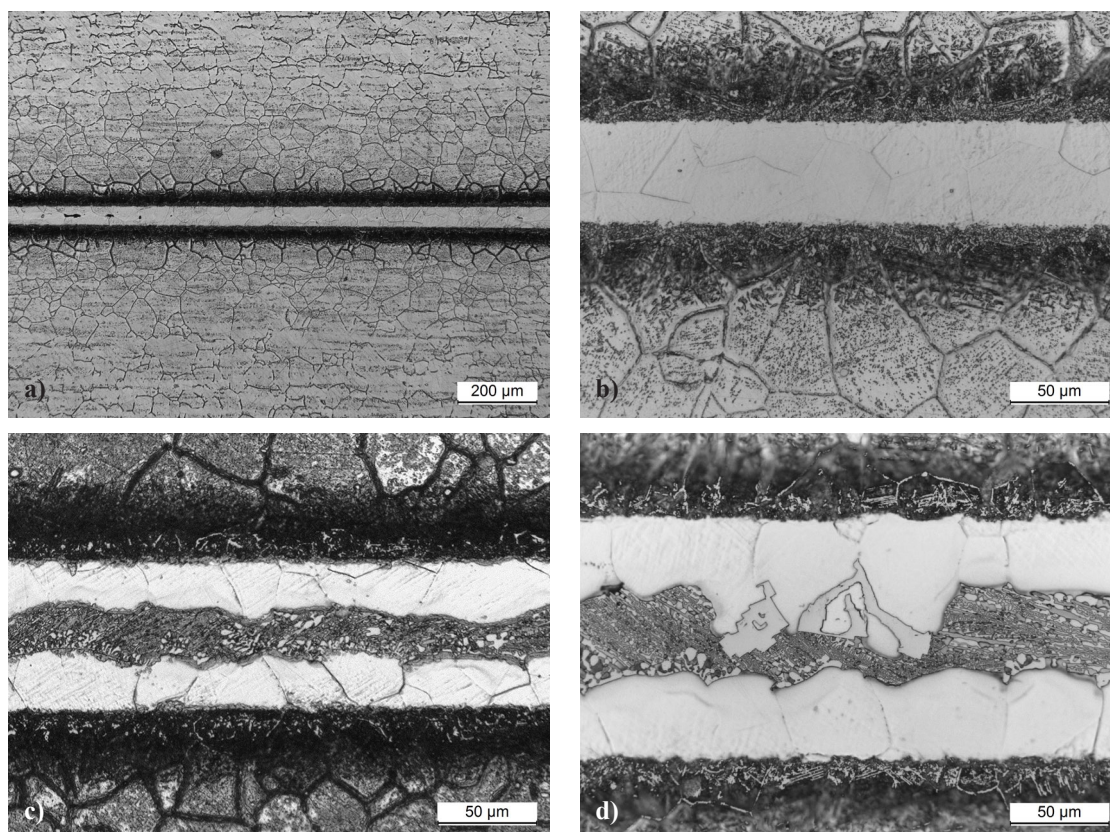


Fig. 6. Microstructure (LM) of Hastelloy/Amdry 915 joints with gap width: a,b) 0.05, c), 0.1 and d) 0.15 mm

was completely uniform (Fig. 6b), without ASZ, which means that in case of this BFM selected holding time (10 minutes) was sufficient for the liquid filler to crystallize isothermally in the entire volume of the joint. Two-zone braze was observed in joints with wider gaps – symmetrically arranged BOZ's and BIZ in the centre of the braze (Fig. 6c,d). However, contrary to BNi-2 alloy, no significant increase in the ASZ width was observed between 0.15 mm and 0.1 joints. No major differences between BOZ in these joints were noticed – both consisted of equiaxed grains of γ -Ni solid solution, without signs of columnar growth.

SEM/EDS analysis revealed that the chemical composition of DAZ in base material is similar for all gap sizes and BFM's

used – BNi-2 (areas designated as 2 in Fig. 4, TABLE 3): B 2,4 Si 0.6-0.8, Cr 24.5-26.6, Fe 16.8-19.3 % wt. and Amdry 915 (areas designated as 1 in Fig. 7, TABLE 4): B 1.7-2.8, Si 0.4-0.6, Cr 23.8-26.9, Fe 16.9-18.5 % wt. Diffusion of boron is a main factor determining DAZ depth and morphology. Content of this element in both filler alloys is comparable. In consequence, DAZ's phase composition were also comparable in both BM/BFM combinations, containing mostly needle-like and blocky (Cr, Mo)-rich borides (TABLE 3, TABLE 4). Chemical composition of isothermally solidified braze in the 0.05 mm joint (area 2 in Fig. 7a, TABLE 4) seems to correspond to the BOZ in Hastelloy X/BNi-2 joints (areas 3 in Fig. 4, TABLE 3).

TABLE 4

Chemical composition in selected areas shown in Fig. 7

Gap width [mm]	Area	Chemical composition											
		[% wt]						[% at.]					
		B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L	B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L
0.05	1	1.8	0.6	23.8	18.2	43.9	11.7	9.3	1.1	24.8	17.7	40.5	6.6
	2	1.6	4.2	10.1	6.5	77.7	—	7.5	7.8	10.1	6.0	68.7	—
0.1	1	1.7	0.4	24.1	18.5	46.2	9.2	8.5	0.7	25.1	17.9	42.6	5.2
	2	2.1	4.5	9.8	6.7	76.8	—	10.0	8.1	9.6	6.1	66.3	—
	3	3.1	5.7	4.7	2.9	83.5	—	13.9	9.9	4.4	2.6	69.1	—
	5	1.8	0.4	30.3	15.7	41.9	9.8	9.1	0.8	31.2	15.1	38.2	5.5
0.15	1	2.8	0.6	26.9	16.9	40.6	12.3	13.4	1.0	27.0	15.8	36.1	6.7
	2	1.4	4.5	10.8	7.2	76.1	—	6.7	8.4	10.8	6.8	67.4	—
	3	3.2	9.5	3.7	2.5	81.1	—	13.7	15.9	3.3	2.1	64.9	—
	4	3.3	—	84.6	—	2.8	9.4	14.5	—	78.5	—	2.3	4.7

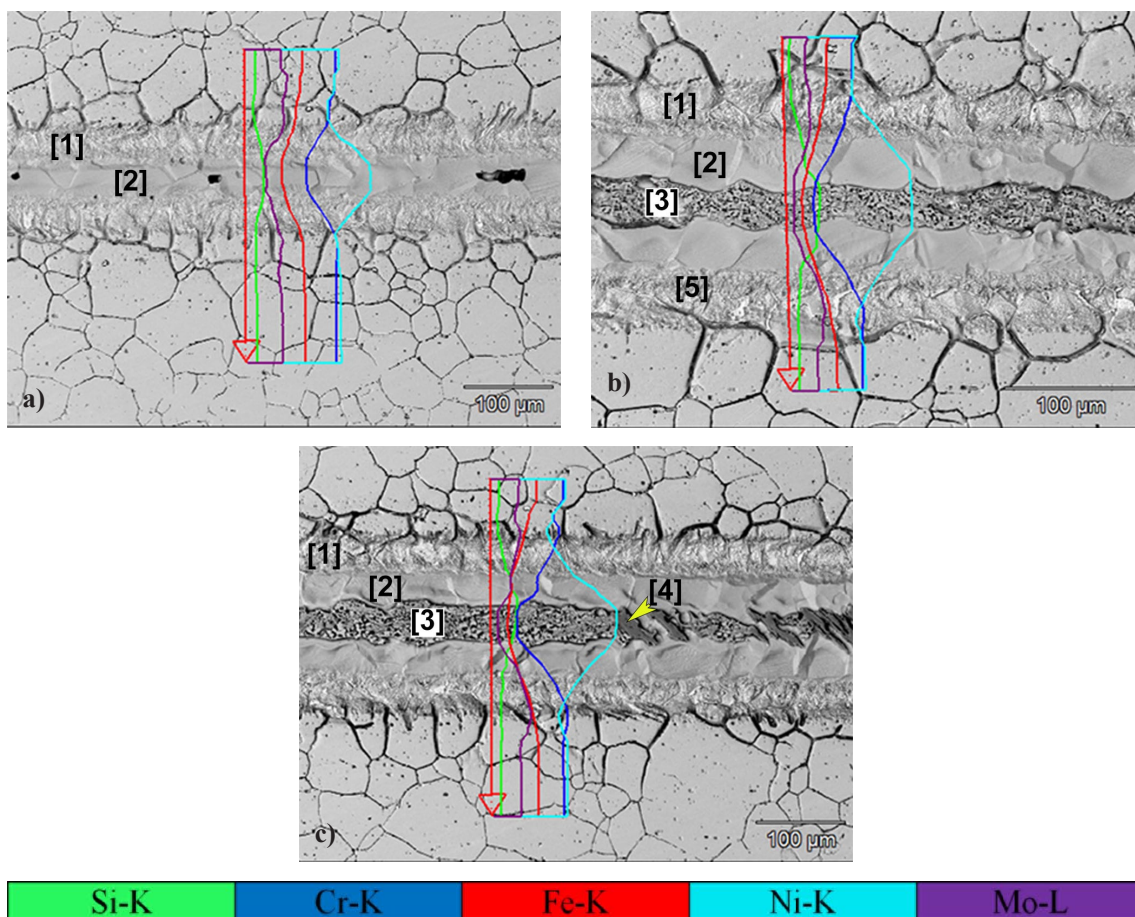


Fig. 7. Microstructure (SEM) and linear elements distribution for Hastelloy X/Amdry 915 joints with gap width: a) 0.05 mm, b) 0.1 mm, c) 0.15 mm; [1] and [5] – DAZ – borides in the matrix of γ -phase solid solution, [2] – BOZ – isothermally solidified γ -phase grains, [3] – BIZ – eutectic-type mixture, [4] – (Cr, Mo) boride in BIZ

Basing on EDS results and literature reports [25, 26], these areas are composed of γ nickel solid solution. The BIZ's in both 0.1 and 0.15 mm joints are composed of eutectic-like mixture, with similar chemical composition (areas 3 in Fig 7b,c, TABLE 4). Content of boron and silicon indicates presence of intermetallic compounds. In microstructure of the BIZ in 0.15 joint a plate-shaped Cr-rich phase with significant amount of boron and molybdenum was detected (area 4 in Fig. 7c. TABLE 4), which corresponds to (Cr, Mo)-rich boride, also identified by other authors [26]. Even keeping in mind limitation of EDS analysis regarding characterization of boron content, it can be concluded that chromium and boron intermetallic compounds are the main phase components in this area.

3.2.2. Hardness

Microhardness measurements of joints brazed with Amdry 915 alloy also showed significant increase in hardness of the braze with increase of the gap size, with evident uptrend – from 280 HV for 0.5 mm (Fig. 8a), through 358 HV for 0.1 mm (Fig. 8b), to 930 HV for 0.15 mm (Fig. 8c). Similarly to BNi-2 filler alloy, an increase in hardness is observable in the diffusion zone and its peak occurs in the centre of the joint.

In case of the narrowest gap, the hardness difference between base material (approx. 200 HV) and joint (280 HV) is the lowest – about 80 HV (Fig. 8a). For analogous BNi-2 joint, where hardness over 360HV was reached, the difference was nearly twice as large. A major difference can be also observed between 0.1 mm joints brazed with particular filler alloys, where BNi-2 joint hardness (800 HV – Fig. 5b) exceeded the hardness of the Amdry 915 joint (360 HV – Fig. 8b) more than twice. Obtained results might be related to ASZ width, as this zone in the Amdry 915 joint is noticeably narrower. In case of the largest analysed gaps, significantly higher hardness of braze was obtained with Amdry 915 alloy (Fig. 8c). Changes in hardness distribution are clearly related to the gap width, which affects the liquid filler solidification conditions and determines the phase composition of the braze.

3.3. Impact of the process interruption on Hastelloy X with Amdry 915 joints

Another part of conducted research addressed the issue of the impact of interruption in the brazing process on joint microstructure. In industrial conditions there are a number of potential factors that may disturb the brazing cycle, such as furnace

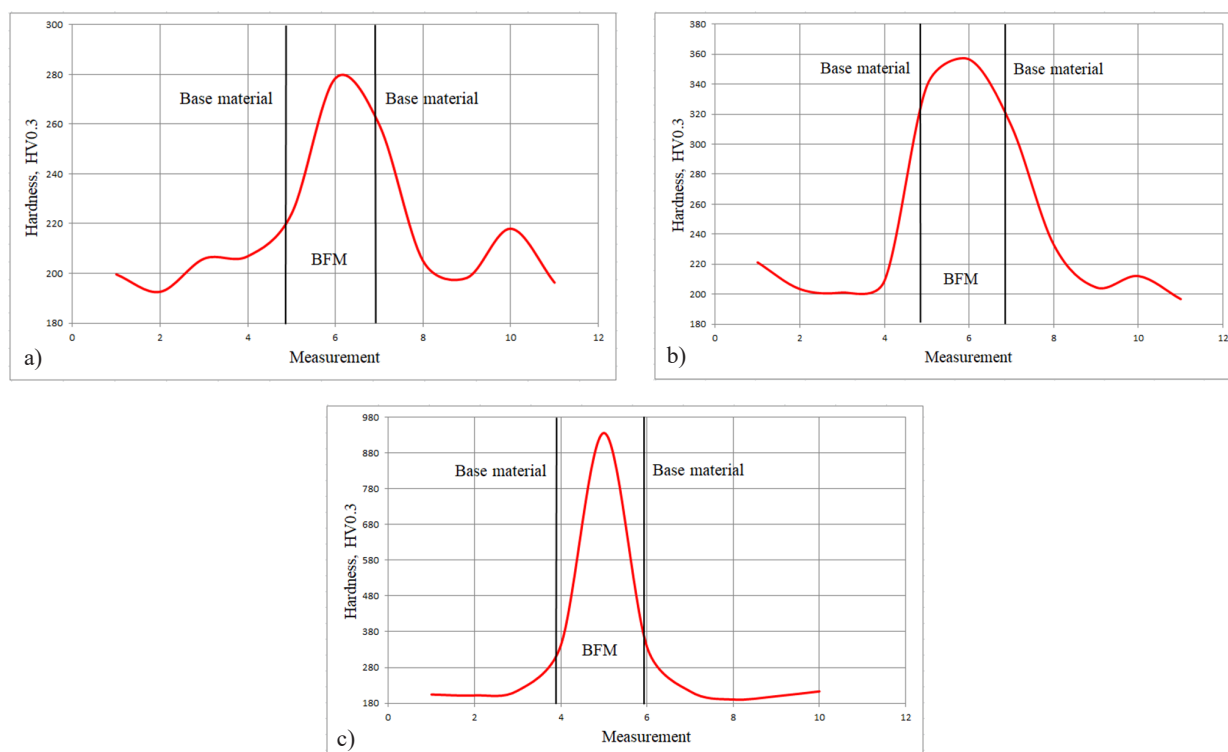


Fig. 8. Hardness distribution on the cross-section of the Hastelloy X/Amdry 915 joints for gap width: a) 0.05 mm, b) 0.1 mm and c) 0.15 mm

malfunction, power supply break or furnace operator error. It is particularly crucial in the case of filler alloys with wide solidus-liquidus range, such as Amdry 915, because breaking the process within that range may lead to forming of a joint that doesn't meet quality standards, inspection of which is often technologically difficult. Thus, better understanding of phenomena that might occur in joints exposed to such conditions is essential in evaluation of the described process.

In case of a process interrupted in 1050°C (Fig. 9a), despite the fact that liquidus temperature has not been reached yet, it can be seen that melting of filler alloy occurred and it has been drawn by capillary action between mating sheets. However, the BFM melted only partially, and the joint wasn't completely filled. SEM/EDS analysis of that joint didn't show any significant differences between chemical composition of parent material and joint area (TABLE 5), with the elements content corresponding to nominal composition of Hastelloy X. Taking into considera-

tion incomplete penetration of the joint, this analysis is given for indicative purposes only, and cannot be treated as a reliable measurement. Microstructure of the same joint after rebrazing (Fig. 9b) is typical for brazed joint and can be considered as correct. Except for occasional porosity, the BFM zone is uniform in terms of phase morphology, composed entirely of γ -Ni solid solution (area designated as 3 in Fig. 9b, TABLE 5).

In the brazing process interrupted in 1145°C (setpoint temperature) filler alloy was completely melted, full penetration of the gap occurred and filler metal created proper meniscus on both sides of the joint. Regardless of disrupting the process directly after achieving setpoint temperature, the obtained joint complied with visual inspection standards. Nevertheless, it was found that joint microstructure is heterogeneous (Fig. 9c), containing centreline eutectic reaction products formed during cooling – the BIZ. Linear elements distribution showed that the content of chromium, nickel and iron varies significantly in

TABLE 5

Chemical composition in selected areas shown in Fig. 9a,b

Process step	Area	Chemical composition											
		[% wt]						[% at.]					
		B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L	B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L
After interruption at 1050°C	1	—	0.5	24.1	20.3	46.2	7.8	—	1.0	26.7	21.0	45.3	4.7
	2	—	0.5	24.4	20.4	46.1	7.4	—	1.1	27.0	21.0	45.1	4.4
	3	—	0.6	23.7	20.1	46.2	8	—	1.3	26.1	20.7	45.2	4.8
After rebrazing	1	—	—	23.8	19.4	45.3	8.4	—	—	27.0	20.5	45.6	5.2
	2	—	0.4	26.2	16.2	38.1	16.8	—	0.9	30.4	17.6	39.3	10.6
	3	1.1	4.4	8.9	6.1	79.4	—	5.5	8.3	9.1	5.8	71.4	—

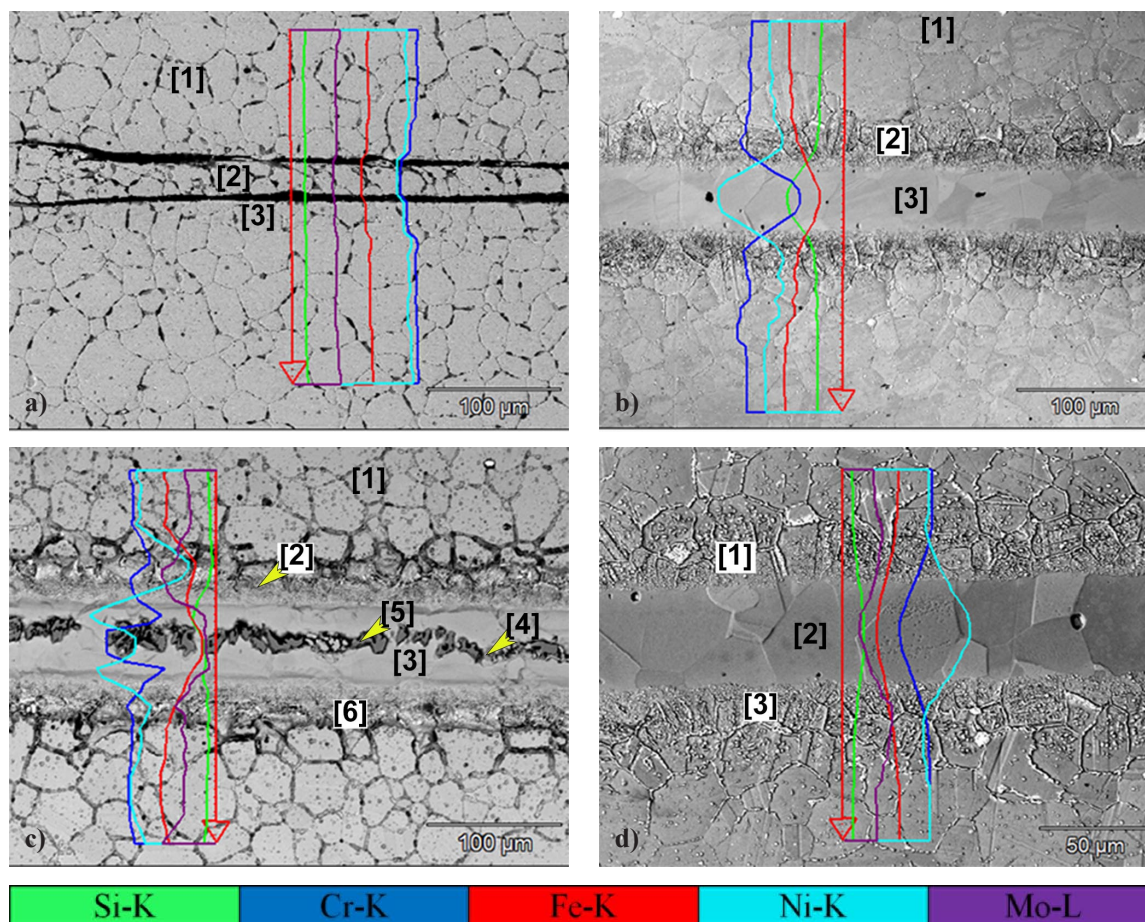


Fig. 9. Microstructure (SEM) and linear elements distribution for Hastelloy X/Amdry 915 joints for brazing processes interrupted at: a) 1050°C, c) 1145°C and corresponding microstructures after re-brazing: b) and d), respectively

TABLE 6

Chemical composition in selected areas shown on Fig. 9c,d

Process step	Area	Chemical composition											
		[% wt]						[% at.]					
		B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L	B-K	Si-K	Cr-K	Fe-K	Ni-K	Mo-L
After interruption at 1145°C	1	—	0.5	24.3	20.2	46.7	7.3	—	1.1	26.9	20.8	45.8	4.4
	2	2.2	1.3	25.5	14.2	46.3	10	10.9	2.5	25.8	13.4	41.5	5.5
	3	1	3.9	14.5	8.4	70.7	1.4	4.9	7.4	14.8	8.0	64.0	0.8
	4	1.8	0.9	53.3	6.5	34	3.4	8.5	1.7	52.4	6.0	29.6	1.8
	5	2.9	1.7	59	5.3	27.3	3.7	13.2	2.9	55.0	4.6	22.5	1.9
	6	2.2	0.7	26.1	17.6	42.3	10.1	10.8	1.3	26.5	16.7	38.1	5.6
After re-brazing	1	2.4	0.7	23.4	17.9	42.2	12.5	11.9	1.4	23.8	17.0	38.2	6.9
	2	0.9	4.2	12	—	81.2	0.6	4.6	8.1	12.3	—	73.7	0.3
	3	2.3	0.8	22.8	17.6	43.7	12.1	11.4	1.5	23.3	16.8	39.6	6.7

this area (Fig. 9c) and basing on chemical composition analysis (TABLE 6), Cr-rich phases are main components. It also can be seen that a diffusion zone has been formed, but due to process interruption its depth is limited to approximately 10-15 μm. Carrying out the re-brazing process led to full homogenization of the joint microstructure (Fig. 9d). Comparison of linear distribution between both joints (Fig. 9c,d) and investigation of chemical composition and depth of diffusion zones (Fig. 9d, TABLE 6) clearly shows that diffusion driven homogenization of the braze phase composition occurred.

4. Discussion

Conducted tests undoubtedly show that gap width is a crucial parameter in process of brazed joints microstructure evolution. In both investigated filler alloys, BNi-2 and Amdry 915, formation of complex, multi-phase area localized in the centreline of the joint was observed. Width of ASZ increased with the increase of distance between joined elements. Chemical analysis of this zone using SEM/EDS method and microhardness measurements led to the conclusion that it was an eutectic

mixture of γ -Ni solid solution and intermetallic, boron- and silicon-based compounds. Forming of such microstructure constituents in Ni-B-S filler alloys is reported in literature. Tung et al. [25] investigated microstructure evolution during brazing of nickel with nickel based filler alloys containing boron and silicon as MPD. In joints where BNi-1a brazing filler was used, which chemical composition is similar to Amdry 915, three main intermetallic compounds were identified: nickel silicide, nickel boride and chromium boride. Furthermore, the joint consisted of three eutectic systems: γ -phase and nickel boride binary eutectic, γ -phase and chromium boride binary eutectic and γ -phase, nickel boride and nickel silicide ternary eutectic. Authors also described the solidification mechanism, starting with nucleation of primary γ -phase, which causes the enrichment of remaining liquid in chromium, silicon and boron, leading to subsequent formation of reported systems. Authors of the study on Hastelloy X brazed with Ni-13Cr-4.5Si-4.2Fe-2.8B alloy [26] described the same solidification sequence, taking into account the influence of molybdenum. It was reported that the BIZ consisted of complex binary, ternary and quaternary eutectics with following intermetallic phases: (Cr, Mo) boride in binary systems, (Cr, Mo) boride and (Ni, Cr) boride in ternary systems and Ni boride, Cr boride, Ni borosilicide and fine Ni₃Si in quaternary systems. Due to the same base material and resemblance of BFM used in this study, it can be assumed that obtained eutectic was composed of similar constituents.

Important differences in joints brazed with particular filler alloys should be noted. In case of 0.05 mm gap width, Hastelloy X/Amdry 915 joint microstructure was uniform, without BIZ eutectic transformation products. Even though this area was formed in joints with 0.1 and 0.15 gaps, its width was notably lower than in corresponding joints brazed with BNi-2 alloy. This observation was also confirmed by hardness measurement. BFM zone hardness of 0.1 mm Hastelloy X/Amdry 915 joint was close to hardness of 0.05 mm Hastelloy X/BNi-2 joint. All of the above can lead to general conclusion, that under chosen conditions Amdry 915 alloy shows better tolerance for gap size deviating from the optimum value, which should be taken into consideration at the stage of filler metal selection for particular applications.

Second aspect of this research was evaluation of the brazing process interruption on the joint microstructure. Investigation of the joint from a process disrupted in 1050°C has shown that melting of Amdry 915 filler alloy, to an extent sufficient for occurrence of capillary action, might take place well below liquidus temperature. Microstructure of the same joint after the rebrazing process was found to be typical, without any indications of previous disruption. This observation is particularly important from the perspective of evaluation of similar processes in industrial conditions. Brazing is widely used as a joining method when complex geometry and different material forms are included. If we consider an interrupted process in a context of such assembly, transition of BFM into liquid not only causes filler alloy flow into the areas from which it cannot be removed without disassembling, but also locally affects the surface of

parent material. Therefore, performing the rebrazing operation in such cases appears to be a reasonable approach to achieve proper joint quality.

Process of Hastelloy X/Amdry 915 brazing interrupted in 1145°C led to formation of a joint which macroscopic appearance was conforming to the visual inspection standards. However, microscopic investigation revealed significantly limited diffusion zone and major microstructure heterogeneity. Characteristic ASZ was observed in the braze, consisting of predominantly chromium rich products of the eutectic transformation (Fig. 9c). Diffusion driven homogenization of this joint occurred during the rebrazing process (Fig. 9d). Formation of the brittle intermetallic phases in the joint is an undesirable phenomenon, creating preferred crack propagation paths, which can reduce its durability, especially when subjected to the vibrations. This justifies the conclusion, that in case of process interrupted close to the brazing setpoint, rebrazing operation is necessary despite that the joint may meet visual control requirements after the original process.

Finally, another issue is related to potential differences between joints brazed with Amdry 915 after rebrazing, which might be caused by process interruption in different stages of the cycle. Obtained results allow to compare corresponding areas of both joints. In the diffusion zone of a joint from process interrupted in 1145°C significantly higher content of boron (2.4 % wt.) and silicon (0.7 % wt.) was found, compared to the one interrupted in 1050°C (0.4 % wt. Si, no boron found) along with lower concentration of chromium and molybdenum – 26.2 % wt. Cr and 16.8 % wt. Mo compared to 23.4 % wt. Cr and 12.5 % Mo, respectively. As could be expected, lower content of chromium and molybdenum in the diffusion zone is inevitably related to higher content of these elements in the braze area (TABLES 5 and 6). It appears that even if microstructure of both joints after rebrazing is comparable, the sequence of performed processes is not irrelevant for chemical composition of filler alloy and diffusion zones. Further tests, involving potential effect on strength and fatigue properties should be performed to investigate significance of these differences.

5. Conclusion and summary

In present work the influence of key brazing process parameters, such as gap size and course of brazing cycle, on microstructure evolution of the joint of Hastelloy X nickel superalloy brazed with two different Ni-based filler alloys was investigated. Following conclusions can be made:

- The width of gap between joined elements has a significant impact on microstructure of the joint for both BNi-2 and Amdry 915 filler alloys. Smaller gap allows to produce joints with more uniform microstructure.
- In all joints brazed with BNi-2 alloy, a characteristic centre-line area with complex morphology and phase composition was observed. This area (referred to as ASZ or BIZ) was formed from the remaining portion of liquid filler alloy

during cooling. Width of this zone was increasing with the increase of the clearance.

- In the case of Amdry 915 filler alloy, microstructure of the joint with the gap width of 0.05 mm was completely uniform, entirely composed of γ -Ni solid solution. In other joints centreline ASZ/BIZ was noticed, but its width was generally lower than this in corresponding joints brazed with BNi-2 alloy, and its morphology was different.
- Basing on SEM/EDS analysis, hardness measurements and literature reports, it was determined that ASZ/BIZ in the centre of the joints consisted of various eutectic systems with intermetallic phases such as (Cr, Mo) borides, (Ni, Cr) borides, borosilicides and silicides.
- Interruption of the brazing process with Amdry 915 filler alloy, especially near setpoint temperature, leads to formation of heterogeneous microstructure with intermetallic phase constituents. Rebrazing operation is an effective solution for homogenization of the joint microstructure.
- Comparison of corresponding areas of the Hastelloy X/Amdry 915 joints subjected to process interruption indicates that disrupting the brazing cycle at different stages may lead to dissimilarities in chemical composition of particular zones after rebrazing process.

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