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THE STUDY OF THE IMPACT OF SURFACE PREPARATION METHODS OF INCONEL 625 AND 718 NICKEL-BASE ALLOYS ON WETTABILITY BY BNi-2 AND BNi-3 BRAZING FILLER METALS

BADANIE WPŁYWU PRZYGOTOWANIA POWIERZCHNI STOPÓW NIKLU INCONEL 625 I 718 NA ZWILŻALNOŚĆ POWIERZCHNI LUTAMI BNi-2 I BNi-3

The article discusses the impact of surface preparation method of Inconel 625 and 718 nickel-base alloys in the form of sheets on wettability of the surface. The results of the investigations of surface preparation method (such as nicro-blasting, nickel plating, etching, degreasing, abrasive blasting with grit 120 and 220 and manually grinding with grit 120 and 240) on spreading of BNi-2 and BNi-3 brazing filler metals, widely used in the aerospace industry in high temperature vacuum brazing processes, are presented. Technological parameters of vacuum brazing process are shown. The macro- and microscopic analysis have shown that nicro-blasting does not bring any benefits of wettability of the alloys investigated.

Keywords: Vacuum brazing, Surface wetting, Inconel 625 and 718, BNi-2 and BNi-3 brazing filler metals

W artykule omówiono wpływ metody przygotowania powierzchni na zwilżalność blach ze stopów niklu Inconel 625 oraz 718. Przedstawiono wyniki badań wpływu metod przygotowania powierzchni (technologii nicro-blasting, niklowania galwanicznego, trawienia, odtłuszczania, obróbki strumieniowo ściernej ścierniwem o granulacji 120 oraz 220, a także ręcznego szlifowania papierem ściernym o ziarnistości 120 oraz 240) na rozpływność dwóch gatunków lutu BNi-2 oraz BNi-3, które są powszechnie stosowane w przemyśle lotniczym podczas procesów wysokotemperaturowego lutowania w próżni. Podano parametry technologiczne procesu lutowania próżniowego. Wyniki badań makro- i mikroskopowych wykazały, że nicro-blasting nie wpływa korzystnie na zwilżalność badanych stopów.

1. Introduction

High-temperature brazing technology in vacuum environment has been widely used in the aerospace industry primarily in the production of modern aircraft engines. Brazing is a bonding process of elements using filler metal, which has a lower melting point and different chemical composition than the brazed materials (BM). After heating of brazing filler metal (BFM) above its melting temperature, liquid BFM wets a parts surface in the solid state creating durable, tight and tough joint. High tightness and mechanical strength are critical aspects for the production of aircraft engines, which components are working in extreme conditions in terms of high temperature, load and corrosion environment [1, 2].

In the era of rapidly developing technology for the production of aircraft engines, modern materials and technologies are sought. High quality steels are replaced by nickel alloys. These alloys are often classified as corrosion-resistant and high temperature resistant materials. These properties cause that nickel base alloys are one of the most widely used materials for the manufacture of aircraft engines. From this group the most popular are Inconel alloys, namely Inconel 600, 625 and 718. These alloys are characterized by excellent strength at both low and high temperature and high corrosion resistance.

The main technological parameters of brazing process are temperature and time. Temperature of the process activates the wetting processes and the diffusion. Basic process time consists of heating time to the process temperature, holding time at process temperature (annealing time) and the cooling time. Longer holding time provides a thicker layer of diffusion, what increases the strength, but also the cost of production and decreases efficiency of the brazing process.

During the formation of joint, adhesion and diffusion phenomena occur. Adhesion phenomenon is related to the chemical and physical contact which occurs between the BM and BFM. In this phenomenon, very important role is played by wettability, surface tension, BFM spreading and capillarity.

Wettability is defined as the ability to cover the material surface with a thin, uniform and continuous layer of BFM. The measure of wettability is wetting angle φ, which is formed by the surface of the liquid BFM and the surface of the solid BM. It is important to note that the value of wetting angle depends on the balance of the surface energy at the interface of the three phases: liquid BFM with flux or gas phase, solid BM with flux or gas phase and solid BM with liquid BFM. With the decrease of the surface energy, the value of wetting angle φ decreases, what means that the wettability increases. In general, the wettability depends on the metal alloy, as well

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as on the type of BFM, the process temperature, the surface condition and its roughness.

BFM spreading on the surface of BM is closely connected with the wettability. The characters of both phenomena are similar and depend on the same factors.

Capillary determines the penetration of BFM into the gap which is formed between BM. The height of BFM rise increases with increase of wettability and with decrease of the gap width and specific weight.

Diffusion is phenomenon activated by heat which relies on each other, two-way movement of the components of the BFM and BM. Diffusion occurs extensively when the BFM is in a liquid phase and less intensive during the initial stage of cooling. The diffusion does not always occur during the brazing process. When the liquid BFM does not chemically affect the brazed materials, then there is no mixing of the components of the two phases. It is obvious that adhesion joints are characterized by lower strength properties, than diffusion joints [1].

Use of vacuum during brazing process results in surface deoxidation of BM and BFM. It is important that a high level of vacuum must be maintained throughout the whole brazing process [3].

Institute of Precision Mechanics in cooperation with the WSK "PZL Rzeszów" S.A. and the Warsaw University of Technology performed numerous projects for brazing [2, 4-6]. The aim of the present work was investigation of the impact of surface preparation method on its wettability.

2. Methodology and materials

Studies of the kinetics of surface wetting were conducted by the use of drops lying method and Contact Heating procedure. It consists in common heating, annealing and cooling of substrate and applied BFM drop. Substrate and applied BFM are in continuous physical contact during the entire cycle.

As a material of the substrate, specimens of Inconel 625 and Inconel 718 have been used in the form of rectangular plates with dimensions of 25×25 mm and a thickness of 0.61 mm. Chemical compositions of both alloys investigated are given in Table 1.

TABLE 1
Chemical composition of Inconel 625 and Inconel 718 [7]

[wt%]	Ni	Fe	Cr	Si	Mo	Mn	C
625	58.00	5.00	23.00	0.50	10.00	0.50	0.10
718	50.00	bal	21.00	0.35	3.30	0.35	0.08

Surfaces of specimens, on which BFM in the form of a paste was imposed, have been subjected to nicro-blasting, nickel plating, etching, degreasing, abrasive blasting with grit 120 and 220 and manually grinding with grit 120 and 240. The surfaces of the specimens were subjected to only one of the above mentioned surface preparation methods (except for degreasing prior to etching of specimens).

Nicro-blasting treatment was applied prior to brazing to improve the surface wettability. This technology is similar to abrasive blasting. Grain with suitable shape and size is scattered by compressed air at high pressure and strikes the treated surface. The grain has especially designed chemical composition, with nickel as the main component. This surface treatment causes increase of roughness, but also the grain material remains on the surface, protecting the surface from oxidation during the brazing process [8]. Another source [9] indicates that the grain material remains in only a small amount on the treated surface and does not affect the brazing process.

Nickel-plating was another variant used to prepare the surface for brazing. This treatment was intended to cover the brazed surfaces with a thin layer of nickel. Nickel-plating of Inconel alloys is used in the aviation industry as one of the preparation stages of materials for vacuum brazing. The main purpose of this technology is to increase the wettability of the BM surface. In addition, the use of nickel layer protects the surface from oxidation. For components made by casting, nickel plating operation is necessary before brazing. Without this treatment, brazing can be very difficult and in some case even impossible to realize [10].

Etching was carried out with a solution of hydrochloric acid HCl and preceded by an electrochemical degreasing in liquid alkaline solution heated to temperature 353.15 K (80°C) and intensified with electric current with value about 2 A. The degreasing and etching processes were used to obtain high-purity material surface by removing any remaining chips of cutting tools and base material, residual oils and any preservative formulation materials.

Abrasive-blasting was carried out with the use of two grits of silicon carbide SiC, namely 120 and 220. Abrasive-blasting was carried out in order to extend area of the treated surface, by increase of its roughness.

Grinding with sandpaper was done manually without the use of mechanical grinding equipment. Sandpaper with grit 120 and 240 was used. The manually grinding process was carried out in a such way, that the individual scratches on the treated surfaces were parallel.

After preparing the specimens surfaces, roughness was measured for Inconel 718 using a Mitutoyo SJ-201 device with test section 5×0.8 mm. Parameters R_a (arithmetical average deviation of the profile from the average line) and R_{y5} (R_m; the height of roughness profile by ten points) were measured. Results of the roughness measurements are given in Figure 1 and in Table 2.

TABLE 2
Roughness R_a and R_{y5} of Inconel 718 specimens after different surface treatment processes

Method of surface preparation	R_a [μm]		R_{y5} [μm]	
Nicro-blasting	0.64		4.22	
Nickel-plating	-		-	
Etching	-		-	
Degreasing	0.12		1.34	
Abrasive-blasting 120	0.96		7.54	
Abrasive-blasting 220	0.98		7.85	
Sandpaper 120	= 0.46	⊢ 0.55	= 2.91	⊢ 5.11
Sandpaper 240	= 0.14	⊢ 0.22	= 1.22	⊢ 2.35

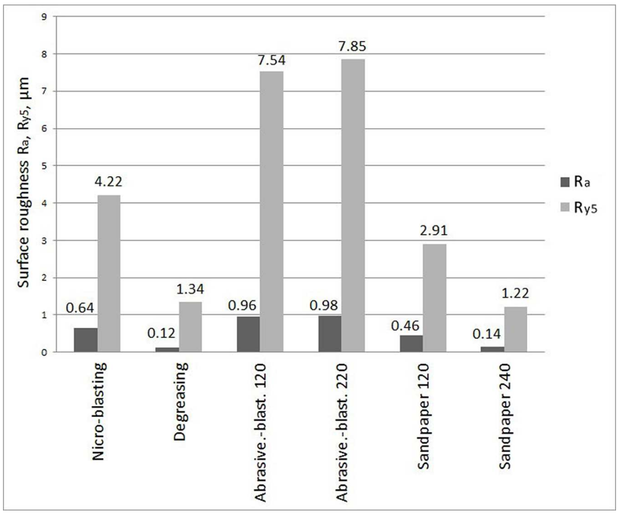

Fig. 1. Results of surface roughness measurements of the Inconel 718 specimens, R_a – arithmetical average deviation of the profile from the average line, R_{y5} (R_m) – the height of roughness profile by ten points

The highest surface roughness was achieved for the specimens treated with abrasive-blasting. Irrespective of the grain size, the measurement results were similar. Almost twice lower roughness was achieved for specimens treated by nicro-blasting. Manually grinded specimens with sandpaper grit 240 had a similar roughness to the base material, while the use of sandpaper with greater granularity (grit 120) caused twice increase of the surface roughness.

Application of BFM was performed by a pneumatic paste dispenser, which provided constant pressure and time of dispensing. Additionally the use of tripod provided constant height of dispenser nozzle above the surface of specimens. These factors allowed to obtain the same amount of BFM and geometry of drops for each specimens. For each of the prepared plates, only one drop of BFM was applied. Figure 2 presents the station to apply the BFM (in bottom right corner the example of a BFM drop on the prepared surface is shown).

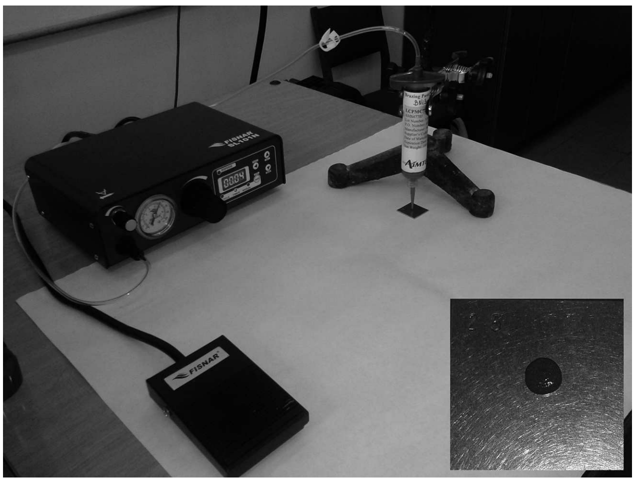

Fig. 2. View of the station used for applying of a BFM drop, an example of a BFM drop applied to the prepared surface (bottom right corner)

In the present study two types of BFM have been used: BNi-2 (AMS4777) and BNi-3 (AMS4778). BNi-2 BFM was applied to Inconel 625, while BNi-3 was applied to Inconel 718. The use of selected BFMs for specific grades of Inconel alloys was justified by aviation industry recommendations for aircraft components brazing technology. The chemical compositions of the BNi-2 and BNi-3 BFMs are given in Table 3.

TABLE 3
Chemical composition of BNi-2 and BNi-3 BFMs [11]

[wt%]	Ni	Cr	Si	B	P	C	Fe
BNi-2	Bal	7.00	4.50	3.13	0.01	0.03	3.00
BNi-3	Bal	-	4.50	3.13	0.01	0.03	0.25

The main difference in the chemical compositions of these BFMs is in chromium and iron content. BNi-2 (AMS4777) has increased content of chromium (7%) and iron (3%). BNi-3 (AMS4778) does not contain chromium and the content of the iron is restricted only to 0.25%.

Heating, annealing and cooling of specimens with applied BFM drops were carried out in a laboratory vacuum furnace. Heating rate 15 K/min (15°C/min) was set to a temperature of 1233.15 K (960°C). At temperature above 1233.15 K (960°C), heating rate 8 K/min (8°C/min) was applied. After reaching the process temperature of 1338.15 K (1065°C), holding stage was conducted for three other times: 5, 15 and 60 minutes. Afterwards slowly cooling with the furnace to the room temperature was performed. Vacuum, which was achieved during the process, was approximately 0.03 Pa ($3\cdot10^{-4}$ mbar). Different time of holding (annealing) at a temperature of 1338.15 K (1065°C) was applied to investigate its effect on the spreading of BFM.

Specimens of Inconel 625 and Inconel 718 after vacuum brazing were subjected to macroscopic and microscopic observations.

Macroscopic observations of specimens were carried out using the stereoscopic microscope Olympus SZX9. The analysis were performed using the analySIS software. Captured images have been saved in digital format. Observations and measurements were conducted for all specimens by using images with magnification x6.3 in order to determine the surface area and the diameters of BFM drops.

After macroscopic observations, the specimens were cut, mounted, grinded, polished and etched in order to conduct light microscopy analysis. In order to determine the wetting angles φ between BFM drops and surfaces of BM, measurements were carried out using the metallographic microscope Olympus IX 70 with magnification x100 and analySIS software.

3. Results and discussion

3.1. Macroscopic observations

Images of the selected specimens, after the spreading test of BNi-2 BFM on Inconel 625 alloy are presented in Figure 3.

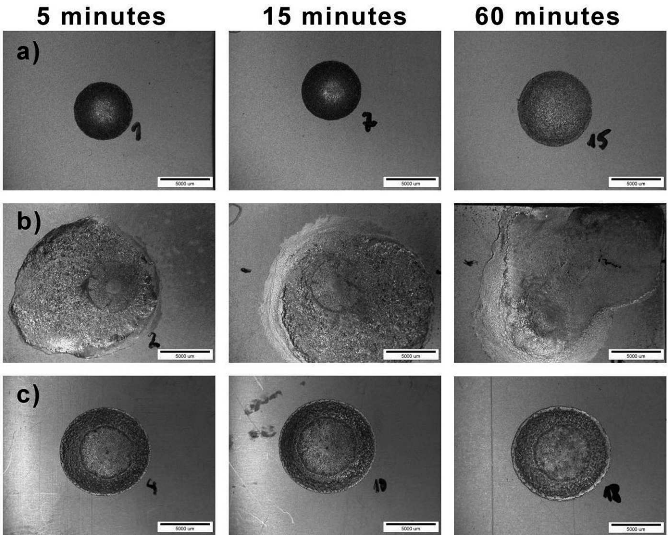

Fig. 3. Macroscopic views of selected specimens after spreading tests of BNi-2 BFM on the Inconel 625 alloy surface; a) specimens subjected to nicro-blasting technology, b) specimens nickel-plated, c) specimens degreased; results shown for the annealing time 5, 15 and 60 minutes, vacuum furnace

The results of surface area and diameter measurements of the BFM drops are shown in Table 4, as well as in Figures 4 and 5.

TABLE 4
The results of surface area (SA) and diameter (DIA) measurements of the BNi-2 BFM drops on Inconel 625 specimens

Method of surface preparation	Annealing time					
	5 minutes		15 minutes		60 minutes	
	SA [mm^2]	DIA [mm]	SA [mm^2]	DIA [mm]	SA [mm^2]	DIA [mm]
Nicro-blasting	27.34	5.92	28.70	6.03	45.54	7.61
Nickel-plating	163.20	-	234.84	-	254.94	-
Etching	65.91	9.18	75.23	9.90	70.48	9.54
Degreasing	64.90	9.02	73.97	9.80	76.13	9.61
Abrasive-blasting 120	-	-	24.65	5.49	-	-
Abrasive-blasting 220	-	-	27.34	5.84	-	-
Sandpaper 120	54.70	8.26	78.84	9.88	89.61	-
Sandpaper 240	48.30	7.93	62.00	8.92	61.45	8.85

Macroscopic observations have shown that the surface preparation method of the Inconel 625 alloy has a big impact on BFM spreading during high temperature vacuum brazing. The best spreading, and thus the wettability of the surface, was achieved for specimens subjected to an electrochemical nickel-plating operation. Surface areas and diameters of drops were more than twice bigger than for specimens subjected to degreasing (or etched specimens). The diameters of the BFM drops on the surfaces subjected to nickel-plating were not measured because BFM covered almost completely the entire surface of specimens. The main purpose of nickel-plating technology as a treatment used before brazing is to increase the wettability of the surface what was confirmed in this study.

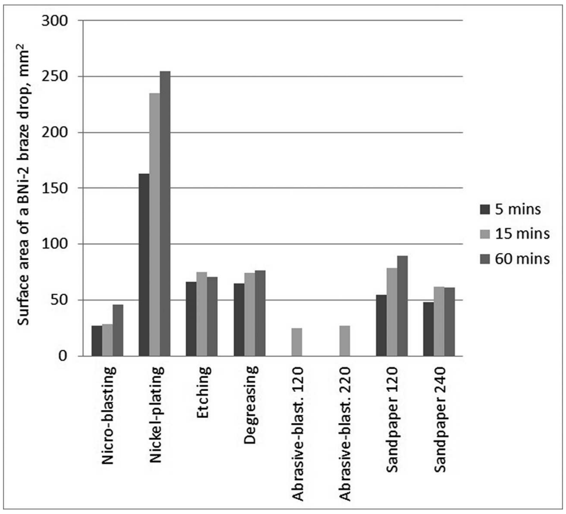

Fig. 4. Effect of surface preparation method of Inconel 625 alloy on the surface area of a BNi-2 BFM drop

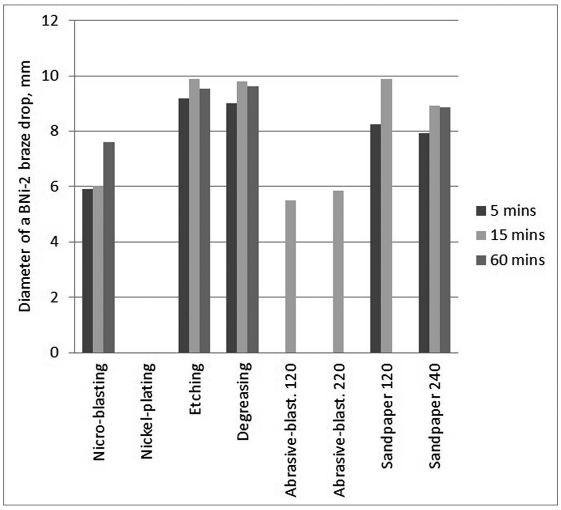

Fig. 5. Effect of surface preparation method of Inconel 625 alloy on the diameter of a BNi-2 BFM drop

Inconel 625 specimens subjected to nicro-blasting treatment were characterized by almost twice worse wettability than specimens, which were degreased only. This fact is contradictory with information given in [8, 9] that nicro-blasting technology should improve wettability or left it unchanged. Therefore, it should be considered whether nicro-blasting treatment was carried out properly. It is necessary to analyze process parameters used, verify them and new attempts should be made.

Abrasive-blasting, which uses SiC grit, significantly reduced the wettability of the surface, reaching values of the surface area and diameter of the BFM drop similar to nicro-blasting. Both technologies are technically similar to each other, because they used devices with the same construction. The only difference is the type and geometry (shape of the grain) of abrasive material. Use of finer grit contributed slightly to increase BFM spreading than for larger abrasive grain.

The use of manually grinding with sandpaper resulted in a slight change in surface wettability. An interesting fact is, that for sandpaper with higher size of grit, BFM wettability was higher than for grinding with smaller grain size. Nevertheless, treatment with sandpaper only slightly influenced the wettability results in comparison to base material. It was also observed that created BFM drop had elliptical shape elongated in the direction of scratches formed during grinding.

It was observed that with the increase of annealing time, degree of BFM spreading on the specimens surfaces increases. For surfaces treated with nicro-blasting, the biggest difference in results is noticeable for annealing for 15 and 60 minutes. For specimens treated with nickel-plating, etching, degreasing and grinding with sandpaper the biggest difference in the surface areas and the drop diameter of BFM is noticeable for annealing for 5 and 15 minutes. Thus, it can be concluded that the annealing longer than 15 minutes is economically unreasonable.

Comparison of the results of roughness measurements and BFM spreading revealed an interesting dependence. Although the surface wettability after nicro-blasting and abrasive-blasting technologies is similar, the roughness after nicro-blasting technology is approximately 1.5 times lower than for specimens subjected to abrasive-blasting. This dependence can result from the difference in the shape of grains, because used grain for nicro-blasting technology has a regular circular shape.

Similarly as for Inconel 625, macroscopic observations of the Inconel 718 alloy specimens were performed. Figure 6 shows selected pictures of specimens after spreading test of BNi-3 BFM.

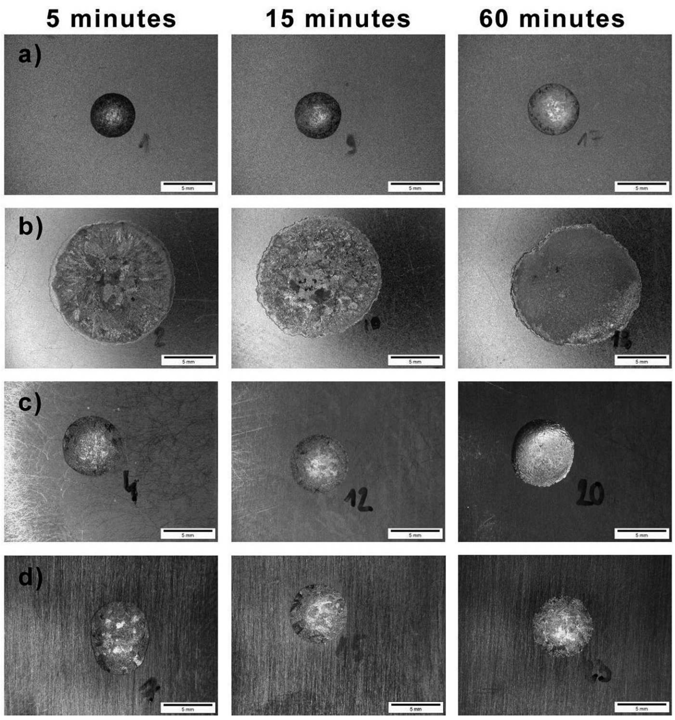

Fig. 6. Macroscopic views of selected specimens after spreading test of BNi-3 BFM on the Inconel 718 alloy surface; a) specimens subjected to nicro-blasting technology, b) specimens subjected to nickel-plating treatment, c) specimens degreased, d) specimens subjected to manually grinding by sandpaper 120; results shown for annealing time 5, 15 and 60 minutes, vacuum furnace

The results of surface areas and diameters of the BNi-3 BFM drop measurements on the Inconel 718 specimens are given in Figures 7 and 8, as well as in Table 5.

TABLE 5
The results of surface area (SA) and diameter (DIA) measurements of the BNi-3 BFM drops on Inconel 718 specimens

Method of surface preparation	**Annealing time**					
	5 minutes		**15 minutes**		**60 minutes**	
	SA [mm^2]	**DIA [mm]**	**SA [mm^2]**	**DIA [mm]**	**SA [mm^2]**	**DIA [mm]**
Nicro-blasting	16.74	4.55	18.06	4.74	22.22	5.30
Nickel-plating	129.89	12.90	124.88	12.80	128.07	13.20
Etching	24.86	5.56	26.17	5.73	29.02	6.00
Degreasing	30.25	6.13	29.20	5.98	36.17	6.72
Abrasive-blasting 120	13.43	4.12	11.83	3.76	14.00	4.20
Abrasive-blasting 220	14.58	4.26	13.66	4.23	15.02	4.24
Sandpaper 120	37.46	-	28.75	5.97	30.91	6.19
Sandpaper 240	32.71	6.18	27.33	5.91	30.06	5.74

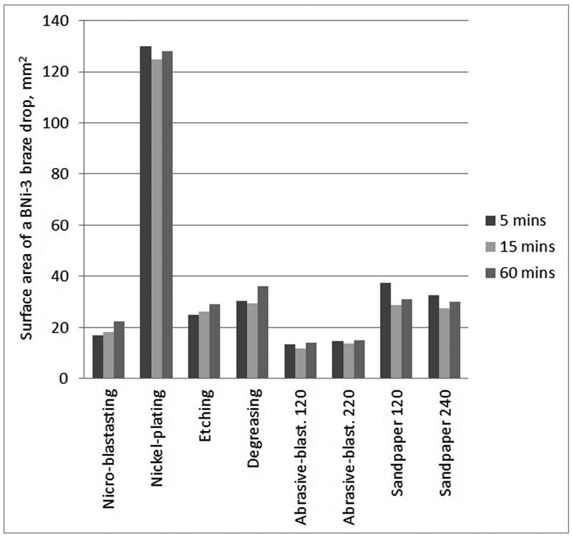

Fig. 7. Effect of surface preparation method of Inconel 718 alloy on the surface area of a BNi-3 BFM drop

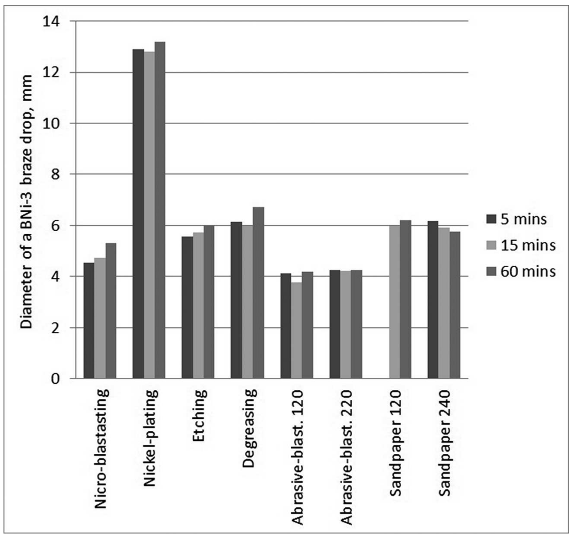

Fig. 8. Effect of surface preparation method of Inconel 718 on the diameter of a BNi-3 BFM drop

Observations of Inconel 718 alloy specimens with BNi-3 BFM led to similar conclusion as the Inconel 625. First of all it is noticeable that the surface areas and diameters of the drops are significantly (almost twice) smaller than the values obtained for Inconel 625. Moreover, the results obtained with Inconel 718 alloy are far more uniform. The use of electrochemical nickel-plating has allowed to increase the BFM drops surface area more than four times and its diameter more than three times. Nicro-blasting operations greatly reduced the degree of BFM spreading. Similar result as for nicro-blasting were obtained for abrasive-blasting.

The use of manually grinding with sandpaper had no effect on the surface wettability. However, it was observed, similar to Inconel 625 alloy, that BFM drops had elliptical shape elongated in the direction of scratches formed during grinding. On surfaces subjected to other technologies circular shape drops were created.

In summary, dependences verified during macroscopic observation for both Inconel 625 and 718 alloys were similar. The legitimacy of nicro-blasting technology as one of the variants of surface preparation method for brazing process was not confirmed in this study. However, nickel-plating technology is the most recommended, as the best way to prepare the surface by increasing their wettability and degree of BFM spreading for brazing processes.

3.2. Microscopic observations

Images of the selected specimens, after the spreading test of BNi-3 BFM on Inconel 718 alloy are presented in Figure 9.

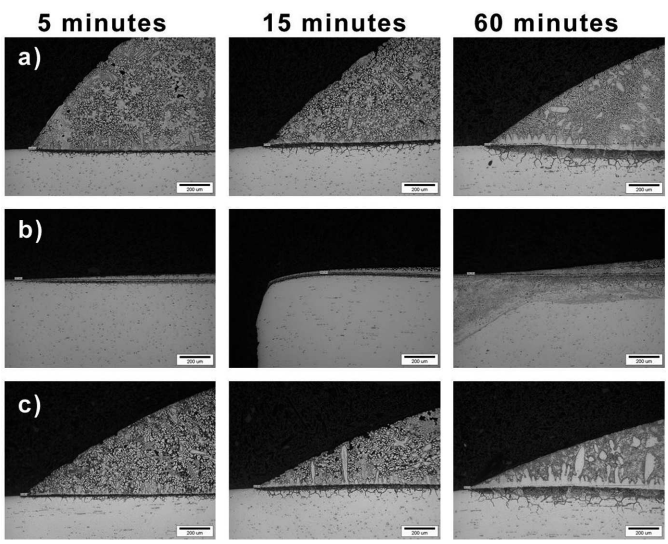

Fig. 9. Macroscopic views of selected specimens after the wetting angle measurements for BNi-3 BFM applied on Inconel 718 surface; a): specimens subjected to a nicro-blasting technology, b) specimens subjected to nickel-plating treatment, c) specimens etched and degreased

The results of the wetting angle for BNi-3 and Inconel 718 specimens are presented in Table 6.

Nickel-plated specimens were characterized by the lowest values of wetting angles, which is associated with the greatest degree of BFM spreading on the surface. Specimens subjected to nicro-blasting treatment were characterized by greater wetting angle than for the other methods applied. With the increase of a brazing time, wetting angle decreased. The attention was paid for measurement results of wetting angle obtained for specimens subjected to degreasing and etching and also for specimens subjected to degreasing only. For brazing time 5 minutes, the difference between above methods of surface preparation was almost three times higher for wetting angle values for etched specimens. However, for annealing time 15 minutes the wetting angles were almost identical.

TABLE 6

The results of wetting angle (WA) measurements of BNi-3 BFM drop applied on the surface of Inconel 718 alloy

Method of surface preparation	Annealing time		
	5 minutes	15 minutes	60 minutes
	WA [°]	WA [°]	WA [°]
Nicro-blasting	53	42	34
Nickel-plating	3	2	5
Etching	32	24	20
Degreasing	13	23	-

Based on both macroscopic and microscopic observations it can be concluded that the use of nicro-blasting technology does not bring any benefits for high temperature brazing in vacuum furnace. This treatment does not improve the wettability and the spreading of BFM, as suggested in [7], but also significantly worsens them. Thus, the information given in [8] that the nicro-blasting operation is recommended to lead before the brazing process has not been confirmed in this study.

4. Conclusions

1. The surfaces of Inconel 625 and Inconel 718 alloys subjected to nickel-plating treatment were characterized by a very good wettability, far better than surfaces subjected to other preparation methods.
2. Nicro-blasting technology, contrary to expectations, used as a variant of surface preparation method of Inconel 625 and Inconel 718 alloys, does not bring any benefits.
3. The use of other mechanical methods of surface preparation decreases only the surface wettability of the alloys investigated.
4. Annealing time longer than 15 minutes is economically unprofitable, because desired results were achieved after 5 and 15 minutes.

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