#### **RESEARCH PAPER**



# **Wide gap brazing of NIMONIC 105 superalloy using BNi‑2 fller and the efect of post braze heat treatment on joint properties**

**Reza Barazandeh1 · Mohammad Ammar Mofd<sup>1</sup>  [·](http://orcid.org/0000-0003-1687-7440) Mostafa Jafarzadegan2 · Hadi Nasiri Vatan1**

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#### **Abstract**

Wide gap brazing is a critical, cost-efective, and widely used technique for repairing superalloy components. However, even with careful control of the braze fller and brazing variables, formation of intermetallic phases cannot be completely avoided. In this work, wide gap brazing of NIMONIC 105 superalloy was performed in a vacuum chamber using BNi-2 alloy as fller at two diferent brazing temperatures of 1050 °C and 1170 °C. Then, the innovative approach of post braze heat treatment (PBHT) of difusion-brazed bonds performed at 1000 °C to improve the joints' microstructural characteristics and mechanical properties. The phases formed in the difusion-afected zone (DAZ), isothermally solidifed zone (ISZ), and athermally solidifed zone (ASZ) were comparatively studied. The investigation confrms PBHT led to a more uniform hardness profle across the joint and a more homogenous interface region via dissolution of intermetallic phases that, in turn, increases the resistance to oxidation. The shear strength of joints made at brazing temperature of 1170 °C remarkably increased from 40 to 83.8 MPa and fnally reached the maximum value of 92.2 MPa via PBHT. The ductility of this specimen was as high as 19.0%, which is approximately 75% of the ductility of the base metal.

**Keywords** Wide gap brazing · NIMONIC 105 · Superalloy · Post braze heat treatment · Microstructure · Shear strength

# **1 Introduction**

Nickel superalloys are known to have excellent corrosion resistance for high-temperature applications. NIMONIC alloy 105 (W. Nr. 2.4634) is a wrought nickel–cobalt-chromium-based alloy strengthened by molybdenum, aluminum, and titanium additions. It has been developed for service up to 950 °C and combines the high strength of the agehardening nickel-based alloys with good creep resistance. The alloy is used for turbine blades, discs, forgings, ring sections, bolts, and fasteners [\[1](#page-10-0)]. However, the high cost of processing and fabrication of nickel superalloy components

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 $\boxtimes$  Mohammad Ammar Mofid moh.ammar\_mofd@iauctb.ac.ir

Faculty of Engineering, Imam Khomeini International University (IKIU), Qazvin 3414896818, Iran

has limited their use so far. Thus, the problem of repairing the components must be faced  $[2]$  $[2]$ . Cost efficiency has biased the industry toward repairing as the best solution instead of replacing the component with a new one. Various attempts have been made to join nickel-based superalloys [[3–](#page-10-2)[6](#page-10-3)]. Fusion welding repair processes would result in inconvenient microstructural changes to the joint region, hence was not a suitable approach [[7](#page-10-4)]. Braze repair is a proven approach for reliable, cost-efective life extension of gas turbine components [[8,](#page-10-5) [9\]](#page-10-6). Wide gap brazing (WGB) is an efficient approach that has found a unique position in joining superalloys and repairing superalloy parts in recent years [\[10\]](#page-10-7). Conventional brazing, or narrow gap brazing, relies on capillary attraction, and as such, the joint gaps are limited. Since boron (B) and silicon (Si) are often used as melting point depressants in the nickel- and cobalt-based braze alloys, a small clearance of 50–100 µm is required to prevent the formation of brittle intermetallic phases in the form of borides and silicides [\[8\]](#page-10-5). The process is conducted in a vacuum chamber or inert atmosphere. A wide gap is considered to be larger than 500 µm, and during brazing, base metals and the braze fller are held at the brazing temperature for a short time to ensure wetting of faying surfaces

<sup>&</sup>lt;sup>1</sup> Department of Petroleum, Mining and Material Engineering, Faculty of Civil and Earth Resources Engineering, Central Tehran Branch, Islamic Azad University, Tehran, Iran

with the braze fller melt. The specimen is then cooled from the brazing temperature, during which the braze alloy solidifes. Since the cooling occurs under non-equilibrium conditions, solidifcation products contain eutectic and intermetallic phases. Even with careful control of the braze fller and brazing variables, the formation of intermetallic phases cannot be wholly avoided [\[8](#page-10-5)]. Hadibeyk et al. declared that at low temperatures, isothermal solidifcation could not be completed, and a eutectic structure was formed in the bonding zone, while at high temperatures, the hindering efect of substrate elements prevented the completion of isothermal solidification [[11\]](#page-11-0). Khakian et al. concluded that because of incomplete isothermal solidifcation, the remained liquid transforms to gamma-eutectic, and subsequently, Cr-rich boride and Ni-rich boride will form in the interface of dis-similar IN738LC and NIMONIC 75 joint [\[6](#page-10-3)].

The BNi fller metals commonly are used on stainless steels for oxidation resistance at temperatures up to 980 to 1095 °C [\[2](#page-10-1)]. BNi-2 braze alloys that contain boron and silicon as melting point depressants are the most common fllers for brazing Ni superalloys. However, the problem is that this fller develops a brittle eutectic constituent that is the source for crack initiation and propagation [\[12](#page-11-1), [13\]](#page-11-2). Up to now, two approaches have been proposed to overcome these problems. In the frst approach, composite fllers, which are prepared by introducing tiny ceramic particles or fbers into traditional active brazing fllers, have been employed to retard atomic difusion and control the formation of brittle intermetallics [\[14](#page-11-3)]. Much research has been devoted to overcoming or at least moderating the unfavorable impact of such brittle phases by introducing silicon nanoparticles, graphene,  $ZrO<sub>2</sub>$  nanoparticles, TiO<sub>2</sub> nanoparticles, and tungsten powder into the brazing fller [[14–](#page-11-3)[16\]](#page-11-4). García et al. used silicon nanoparticles and claimed that this addition diminished the eutectic structure's size and promoted a uniform distribution in the bonding area [[15\]](#page-11-5). Shen et al. added  $ZrO<sub>2</sub>$  nanoparticles into an Sn-Ag filler and showed that this addition controlled the formation of intermetallic particles during solidifcation [\[16\]](#page-11-4). Liu et al. concluded that adding graphene signifcantly improved the shear strength of the GH99 superalloy brazed joints. They claimed that graphene efectively retarded atomic difusion and controlled the formation of brittle boride precipitates [[14\]](#page-11-3).

Another approach to overcoming or at least moderating the unfavorable impact of such eutectic and intermetallic phases is applying subsequent homogenization treatment to the diffusion-brazed bonds. Post braze heat treating (PBHT) can help to achieve a more uniform microstructure that, in turn, can minimize stress concentration in the interface and lead to stress release. As mentioned earlier, the phases formed in the centerline eutectic are deleterious to the mechanical properties. The centerline eutectic phases are avoided by completing isothermal solidifcation. Thought, if isothermal solidifcation is incomplete, the microstruc-ture can be improved with a diffusion heat treatment [\[17](#page-11-6)]. According to the literature, difusion brazing can be divided into three separate stages, including base metal dissolution, isothermal solidifcation, and bond region homogenization. Duvall et al. [[17\]](#page-11-6) have stated that isothermal solidifcation has a critical infuence on the joint quality of the brazed joints. Pouranvari et al. stated that if isothermal solidifcation is completed, there would be no trace of detrimental eutectic constituents along the joint centerline. A joint almost identical to the base metal in terms of microstructure and mechanical properties can be achieved by applying subsequent homogenization treatment to the difusion-brazed bonds [[18](#page-11-7)]. However, applying PBHT to NIMONIC 105 difusion-brazed bonds to improve the microstructural characteristics and comparison of obtained mechanical properties has not been reported.

In this study, wide gap brazing of NIMONIC 105 superalloy was performed in a vacuum chamber using BNi-2 alloy as fller at two diferent brazing temperatures, and then, PBHT of the specimens was performed in an inert atmosphere for two diferent times. Microstructural evolution, microhardness, and shear strength points of view were compared. Some remarkable results were obtained through applying PBHT. The formation of intermetallic phases was investigated by hardness test and scanning electron microscope (SEM)-energy-dispersive X-ray spectroscopy (EDS) analysis. The novelty of this study is the investigation of the infuence of bonding temperature and homogenization heat treatment on the microstructure and mechanical properties of difusion-brazed NIMONIC 105 superalloy using a BNi-2 interlayer. This innovative method is hoped to help arrive at a practical solution to applications concerning difficult to weld superalloys which are prone to the formation of deleterious intermetallic compounds.

## **2 Experimental procedure**

NIMONIC 105 superalloy and commercially obtained BNi-2 fller were received as the experimental materials in this study. The compositions of the base metal and BNi-2 fller were analyzed experimentally. The result is listed in Table [1](#page-2-0). BNi-2 contains boron and silicon as melting point depressants. According to ASM International, BNi-2 fller is an appropriate brazing fller metal for high-temperature, high-stress moving engine components and heavy, nonmoving structures (variable gaps). It is appropriate for jet engine difuser components [[2\]](#page-10-1). Two specimens have been prepared by cutting for metallography and shear strength test with the dimensions of 20 mm  $\times$  20 mm  $\times$  2.5 mm and  $40$  mm  $\times$  20 mm  $\times$  2.5 mm, respectively. Figure [1](#page-2-1) demonstrates the sample specimens as well as the experimental

<span id="page-2-0"></span>



<span id="page-2-1"></span>**Fig. 1** Dimensions of the base metals, metallography, and shear strength test specimens and the experimental setup for difusion brazing

setup. The specimens were ultrasonically cleaned in an acetone bath to remove adhered contaminants and then dried in air. For preparing the shear strength test specimens, the samples were cut into dimensions of  $40 \times 20 \times 2.5$  mm. Then, the BNi-2 fller in the form of paste with a thickness of 1 mm was applied to join NIMONIC 105 superalloy. Solidus and liquidus temperatures of the brazing fller are 970 °C and 1000 °C, respectively. So, brazing temperature was selected at temperatures above 970 °C. Experiments were performed in an electrical furnace with a vacuum of  $1 \times 10^{-4}$  Pa. The sample assembly was first heated to 750 °C and held for 30 min. Then, the temperature was increased to 950 °C and held for 30 min. Finally, the temperature was increased to a specified brazing temperature (1050 °C and 1170 °C). The holding time was 8 min. In order to investigate the effect of PBHT on microstructure and mechanical properties of the joints, homogenization of the specimens was performed at 1000 °C for 1 h and 4 h. The assemblies were cooled in the processing chamber under a vacuum.

Following brazing and PBHT, transverse cross sections were prepared for microstructural observations with an optical microscope (OM) and scanning electron microscope (SEM). The etchant is composed of a "15 ml HCl, 10 ml  $HNO<sub>3</sub>$ , 10 ml acetic acid, and 5 ml glycerol" solution. The etching time was 5 s. SEM (TESCAN MIRA3) equipped with energy-dispersive spectroscopy (EDS) analysis system was employed to analyze the chemical composition of the present phases at the interface. For the microhardness test, a WOLPERT machine was used that was able to measure microhardness at a microscale with 50 gf load weight according to ASTM standard E384 (2017). The indenter was the diamond-based pyramid. The roomtemperature shear strength of the joints was measured by a tensile test machine (INSTRON-5500R) according to ASTM standard D1002-99 at a crosshead speed of 1 mm/ min.

The cyclic oxidation test was carried out in an automatic test bed designed and built for this purpose. Cyclic conditions in this device are provided through the reciprocating movement of the samples in the vertical direction into the furnace. Test samples in the form of rectangular cubes with dimensions of  $12\times7\times2$  mm were prepared according to the standard from the available raw materials, which were mainly the roots of the blades, by wire-cut method [[19](#page-11-8)]. Six samples were prepared and used in the cyclic oxidation test. Considering that the surface of the samples became completely dark due to wire cutting, these oxides and other surface contaminants were removed through mechanical polishing with sandpaper no. 180, 500, and 1000, and all the corners and sharp edges of the samples were also polished. The initial weight of the samples was determined and recorded with the help of a digital scale with an accuracy of 0.0001 g after partial sanding into round shape and acetone washing of the samples.

The test consisted of 1-h cycles at 1100 °C, the details of which are as follows:

- Step 1: Place the weighed samples in special places on the sample stand.
- Step 2: Heating the furnace thermal chamber to  $1100^{\circ}$ C.
- Step 3: As soon as the temperature reaches  $1100^{\circ}$ C, the arm of the samples starts moving upwards, and within 5 s, the samples are placed in the center of the thermal zone.
- Step 4: Keeping the samples at 1100 °C for 1 h.
- Step 5: Moving the sample arm down and leaving the thermal chamber and the sample temperature reaching 300 °C.

Steps 1 to 5 in this experiment were repeated for 50 cycles.

In between the cycles where the weighing of the samples is not considered, the system is allowed to repeat steps 1 to 5, but if it is necessary to weigh the samples, they are removed from their place in the device and then cooled down to the temperature of the weighing environment. Following the weighing process, the samples are returned to the test cycle.

# **3 Results and discussion**

## **3.1 Microstructural characterization of the joints**

### **3.1.1 As‑brazed specimens**

From the Arrhenius formula, temperature is the most important parameter in any difusion bonding process [[20](#page-11-9), [21](#page-11-10)].

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Therefore, the macroscopic images and macrostructures of the joints made at 1050 and 1170 °C were studied. Figure [2](#page-3-0) shows a typical overview of joint regions. It is obvious from Fig. [2](#page-3-0) that with the rise of brazing temperature from 1050 (Fig. [2a](#page-3-0)) to 1170 °C (Fig. [2b\)](#page-3-0), the thicknesses of dissolution and widening zones increase signifcantly from 470 to 850 µm, respectively. The reason is that atomic diffusion occurs evenly and thoroughly at a higher temperature. This leads to the better dissolution of the base metal in the joint area, leading to the wider joint region [[22](#page-11-11)]. On the other hand, the increasing temperature raises the base metal grain size, which in turn deteriorates the mechanical properties of the base metal.

In order to better characterize the joint interface, higher magnifcation OM images of the zone were taken and are shown in Fig. [3.](#page-4-0) As can be seen, in addition to NIMONIC base metal (BM), three zones are recognizable: (i) the difusion-afected zone (DAZ), (ii) the isothermally solidifed zone (ISZ), and (iii) athermally solidifed zone (ASZ). According to the literature [\[6](#page-10-3)] in DAZ, the base metal interacts with the melting point depressant (MPD) elements diffused from the fller metal, such as boron and silicon. It is apparent in Fig. [3](#page-4-0) that the DAZ consists of globular/cuboidal and needle-like precipitates distributed in the interface. Figure [3](#page-4-0) suggests that the morphology and distribution of these secondary phases depend on the brazing temperature.

Regarding Fig. [3](#page-4-0), brazing temperature increment will increase the width of this DAZ from 12 (Fig.  $3a$ ) to 35  $\mu$ m (Fig. [3b\)](#page-4-0). On the other hand, it is apparent from Fig. [3a](#page-4-0) that the morphology of these precipitates changes from globular to needle-like, with getting away from the interface. This is in accordance with the study of Li and Liu  $[4, 5]$  $[4, 5]$  $[4, 5]$ , who reported that the morphology of these secondary phases depends on shrinkage stresses arising from isothermal

<span id="page-3-0"></span>

<span id="page-4-0"></span>**Fig. 3** Optical microscope images of the joint region for the specimens brazed at bonding temperature of **a** 1050 °C and **b** 1170 °C



solidification. At the vicinity of NIMONIC/filler interface, more shrinkage stresses developed. As a result, precipitates become spherical adjacent to this interface and acicular beyond it. Nucleation of these phases occurs at the frst stages of brazing in NIMONIC 105 superalloy in short times, and as Fig. [3b](#page-4-0) shows, they will dissolve at higher temperatures.

The precipitation-free zone in Fig. [3a](#page-4-0) and [b](#page-4-0) is relevant to ISZ, which is formed near NIMONIC 105/braze alloy interface. During the isothermal solidifcation stage, the Nirich gamma phase formed with solid solution strengtheners, including boron and silicon, which solidifes inward. At brazing temperature, fller melts and wets the joint faying surfaces. Because of the establishment of perfect contact between solid and liquid, NIMONIC 105 begins to dissolute. At this time, MPD difusion from fller to NIMONIC and difusion of alloying elements from NIMONIC to fller occur. The result is an extension of liquid formation. The volume of liquid continues to increase up to the establishment of local equilibrium between solid and liquid. Once the molten fller reaches brazing temperature, the dissolution stage ceases, and at this time, isothermal solidifcation commences from the liquid/solid interface inward to the joint centerline. According to the microstructure shown in Fig. [3,](#page-4-0) it can be concluded that after the completion of isothermal solidifcation, there would be a single phase with a uniform microstructure in the bond line. The reason is that the liquid/ solid interface does not experience any super cooling during isothermal solidifcation. Therewith, non-equilibrium segregation of solute elements is resolvable, and no secondary phases can form. Therefore, in correspondence with the literature, ISZ microstructure is a solid solution single phase [\[23,](#page-11-12) [24\]](#page-11-13).

If insufficient time is given, isothermal solidification is unable to be complete, and the residual liquid is rejected to the center of the joint [[4\]](#page-10-8). The liquid in the center becomes enriched in melting point depressants, shifting the concentration to a eutectic composition resulting in eutectic-like solidifcation referred to as the ASZ, as shown in Fig. [3](#page-4-0). It is known that ISZ thickness is controlled by MPD element diffusion [\[6](#page-10-3)]. According to the microstructural studies in Fig. [3,](#page-4-0) it can be concluded that with the rise of brazing temperature from 1050 (Fig. [3a\)](#page-4-0) to 1170 °C (Fig. [3b\)](#page-4-0), the thicknesses of ISZ increase signifcantly from 15 to 30 µm, respectively. The reason is that the rate of MPD difusion into base metal is higher at a higher temperature.

In order to acquire a more precise investigation of microstructural evolutions at the interface, backscattered electron (BSE) micrograph of the interface and concentration profle of some important elements (Cr, Fe, and Si) across the bonding region were taken and are shown in Fig. [4](#page-4-1). The EDS technique is applied to identify each phase individually. Regions in a joint created at a brazing temperature of 1050 °C have been analyzed using EDS at some selected regions marked as 1, 2, and 3 in Fig. [4.](#page-4-1) The concentration of Ni, Co, Cr, Mo, Al, Si, Fe, Ti, and Cu in the aforementioned regions analyzed by EDS is listed in Table [2](#page-5-0). Based on the compositional analysis, regions 1 and 2 (acicular and cuboidal precipitates in DAZ) contain higher chromium content in comparison with BNi-2 fller. On the other hand, boron



<span id="page-4-1"></span>**Fig. 4** BSE micrograph of the interface and concentration profle of some important elements across the bonding region for the specimens brazed at a bonding temperature of 1050 °C

solubility in nickel is very low (0.3 at.% at a temperature range of 1060 to 1120 °C [\[25\]](#page-11-14)). As a result, when the concentration of boron exceeds from solubility limit in the base metal, secondary precipitates can form. Boron combines with chromium or molybdenum to form chromium borides within the gamma-grain boundaries. The precipitates in the DAZ were mainly composed of Ni–Cr-Mo-rich borides. This is in agreement with the results of Wu et al. [\[9](#page-10-6)]. During the brazing process, silicon and boron atoms difused to the base metal from molten fller. Here (in DAZ), boron reacted with elements from base metal to form Ni–Cr-Mo-rich borides.

From Table [2,](#page-5-0) it can be found that the nickel content in the ISZ (region 3) was higher (74.45 at.%), followed by silicon (10.11 at.%) and chromium (6.37 at.%). Based on the Ni–Cr binary phase diagram, the ISZ mainly consisted of a gamma solid solution [[25](#page-11-14)]. At the frst stage of brazing, when the temperature of the chamber reached the melting point of the fller, BNi-2 melted gradually, and the liquid phase formed. Then, the liquid phase infltrated into the NIMONIC 105 superalloy substrate, and boron and silicon atoms difused to the NIMONIC 105 substrate. During the isothermal solidifcation stage, the difusion process of atoms was accelerated. Boron and silicon atoms further diffused to NIMONIC substrate, and high-melting elements chromium and molybdenum of base metal dissolved and diffused into the molten fller at the same time. The presence of  $Ni<sub>3</sub>(Si, B)$  reported in the literature [\[10](#page-10-7)] is in good agreement with the results of this study. This phase formed with the other elements in solid solution. The EDS line scan of elements perpendicular to the interface (Fig. [4](#page-4-1)) shows a severe decrease in chromium profle and a signifcant increase in silicon and iron inside the ISZ. Fe and Ni can dissolve each other infnitely during bonding. Therefore, a large number of Fe atoms can difuse and traverse the interlayer, leading to increased Fe content (5.06 at.%) at region 3. The issue surrounding boron as an MPD is its tendency to form borides. Chromium boride often forms, which reduces the chromium content in the braze and decreases corrosion resistance. The formation of mentioned chromium boride precipitates is responsible for localized depletion in the concentration of Cr and some other refractory elements such as Mo in ISZ. These phases reduce the chromium and molybdenum concentrations from the gamma-matrix. They would have a detrimental effect on the high-temperature performance and make these regions prone to selective corrosion or oxidation. According to Table [2](#page-5-0) in region 3 (ISZ), the main constituent is nickel (74.45 at.%) which suggests that the matrix is gamma-nickel with solid solution strengthening elements chromium (6.37 at.%), cobalt (1.29 at.%), and silicon (10.11 at.%). Analyses of region 3 showed that elements like Co, Al, and Ti, which were not present in the initial chemical composition of fller, have difused into the joint zone. This fact confrms the transportation of solute elements into the liquid phase during the brazing and the occurrence of solid state difusion.

The BSE micrograph of the ASZ for the sample brazed at 1170  $\degree$ C is shown in Fig. [5](#page-6-0). The formation of the eutectic that contains fne precipitates is visible in the fgure. The presence of some eutectic islands and intermetallics at the centerline of the joint suggests that the isothermal solidifcation of the liquid phase was not completed in 8 min. As a result, the remained liquid undergoes supercooling. Under such circumstances, microstructural evolution is driven by the dissolution and difusion of the base metal alloying elements into the joint  $[18]$  $[18]$ . Due to the low solubility of boron in nickel (0.3 at.% [\[25](#page-11-14)]), extra boron will be rejected into the molten metal.

Table [2](#page-5-0) indicates that the ASZ in Fig. [5](#page-6-0) contains the matrix solid solution of gamma-nickel (region 4 in Fig. [5](#page-6-0)), Ni-rich boride (region 5), and two Cr-rich phases (regions 6, 7). Gamma solid solution (region 4) is the frst phase that forms and grows during cooling. Since there is a large amount of silicon (6.08 at.%), the phase that forms with the other elements in the solid solution is most likely  $Ni<sub>3</sub>(Si,$ B). There was a signifcant amount of aluminum in region 5, which would most likely contribute to the gamma-prime precipitation. The ongoing formation of gamma-nickel and Ni-rich gamma-prime dendrites leads to the rejection of alloying elements such as Cr, Mo, Al, and B to liquid [\[26](#page-11-15)]. As a result, the remained liquid will become enriched by these elements. Phases 6 and 7 in Fig. [5](#page-6-0) are Cr-rich boride and Cr-rich intermetallic compounds, respectively. These intermetallics would not be ideal phases to form because that would mean that chromium is being depleted from the

<span id="page-5-0"></span>





**Fig. 5** BSE micrograph of the ASZ for the sample brazed at a bonding temperature of 1170 °C

<span id="page-6-0"></span>matrix, thereby decreasing the corrosion resistance of the material. Arhami et al. [[3\]](#page-10-2) have also reported the formation of the same phases in the ASZ of difusion-brazed IN-939 using a Ni–Cr-B interlayer.

An issue with the specimen brazed at 1170 °C was the large amount of void-like defects observed in the BSE micrograph that formed within the joint in ASZ adjacent to the Cr-rich phases (Fig. [6a](#page-6-1)). In order to determine whether these features are real voids or not, secondary electron (SE) micrograph of the same zone was taken and is shown in Fig. [6b](#page-6-1). It is apparent from the SE image that electron number effects make the edges of surface voids brighter than the rest of the surface. It can be concluded that Fig. [6](#page-6-1) shows many voids in ASZ, indicating a brittle braze owing to inter-metallics. This agrees with the results of Kim et al. [[10](#page-10-7)]. The voids formed due to solidifcation shrinkage in ASZ can reduce the strength.

#### **3.1.2 Homogenization of specimens**

In order to investigate the efect of PBHT on microstructure and mechanical properties of the joints, homogenization of the specimens was performed at 1000 °C for 1 h and 4 h. The aim of this PBHT was as follows:

- Achieving a more uniform microstructure
- Achieving a more uniform hardness profle across the joint
- Minimizing stress concentration
- Stress releasing

Figure [7](#page-7-0) shows the efect of PBHT time (1 h and 4 h) on the microstructure of the joints brazed at 1050 °C (Fig. [7a,](#page-7-0) [b](#page-7-0)) and 1170 °C (Fig. [7c, d](#page-7-0)). The thickness of ASZ increased with increasing PBHT time for the joint made at the brazing temperature of 1050 °C, and decreased with the increase of PBHT time for the joint made at the brazing temperature of 1170 °C. This is because difusion is a thermally activated process and MPD atoms (boron and silicon) difused insuffciently from liquid fller to solid base metal in the specimen brazed at a relatively low temperature of 1050 °C. As a result, regarding Fig. [7a](#page-7-0) and [b](#page-7-0) with increasing PBHT time from 1 to 4 h, the ASZ width increased from 290 to 340  $\mu$ m, respectively. In the case of the joint made at higher brazing temperature, the difusion rate of MPD atoms into the base metal was higher during brazing. As a result, regarding Fig. [7c](#page-7-0) and [d](#page-7-0) with increasing PBHT time from 1 to 4 h, the ASZ width decreased from 630 to 540  $\mu$ m, respectively. This is because, in longer PBHT times, a higher number of MPD atoms have the opportunity to difuse out of the interlayer. Therefore, by increasing the bonding time, the isothermal solidifcation front can progress further toward the centerline, and this fact will disturb ASZ formation.

Another interesting matter about Fig. [7d](#page-7-0) is the dissolution of intermetallic phases and more uniform microstructure in comparison with the microstructure in Fig. [7c](#page-7-0). This issue

<span id="page-6-1"></span>**Fig. 6** Void-like defects observed in the SEM micrograph of the ASZ for the sample brazed at a bonding temperature of 1170 °C, **a** BSE image and **b** SE image



<span id="page-7-0"></span>**Fig. 7** The effect of PBHT at 1000 °C, on the microstructure of the joints **a** brazed at 1050 °C and homogenized for 1 h, **b** brazed at 1050 °C and homogenized for 4 h, **c** brazed at 1170 °C and homogenized for 1 h, and **d** brazed at 1170 °C and homogenized for 4 h



is better observed in Fig. [8](#page-7-1) which indicates the EDS line scan of elements across the red dashed line for joint made at 1170 °C and homogenized at 1000 °C for 4 h. The variation of Ni, Cr, Mo, and MPD elements across the interface is continuous and smooth compared to the as-brazed specimen (Fig. [4](#page-4-1)). Additionally, the absence of intermetallic compounds in the difusion zone is deduced by the smoothly varying nature of the profle curves. This indicates that PBHT leads to a more homogenous interface region via the dissolution of intermetallic phases. This is because the brazing process is a difusion control phenomenon [[27\]](#page-11-16).

## **3.2 Mechanical properties of the joints**

## **3.2.1 Microhardness**

Figure [9](#page-8-0) exhibits the variation of microhardness in the joint region as a function of distance from the center of interface for joints created at brazing temperatures of 1050 °C (stretch line curves) and 1170 °C (dashed line curves) with diferent PBHT times of 0, 1, and 4 h. It can be seen that substantial changes can be observed at the joint interface, which is directly related to the composition and thickness of DAZ, ISZ, and ASZ generated. Fairly high hardness values (866 and 770 VHN for the specimens created at brazing temperatures of 1050 and 1170 °C, respectively) are observed in the ASZ of specimens that were not undergone PBHT. These values are much greater than those for the NIMONIC 105 base metal. Khakian et al. [\[6\]](#page-10-3) have reported that intermetallic phases that form during non-isothermal solidifcation possess very high hardness (750HVN). These brittle phases are suitable sites for crack initiation and therefore reduce the mechanical properties of the joint. These high hardness values are not seen in the joint region of the specimens that have undergone PBHT. This approves that PBHT leads to a more homogenous interface region via the dissolution of intermetallic phases. Figure [9](#page-8-0) indicates that PBHT resulted in more uniform hardness profles across the joint

<span id="page-7-1"></span>**Fig. 8** BSE micrograph of the interface and respective EDS line scan of elements across the red dashed line for joint made at 1170 °C and homogenized at 1000 °C for 4 h



**50 µm**





<span id="page-8-0"></span>**Fig. 9** Microhardness profles across the joint region as a function of distance from the center of interface for joints created at brazing temperatures of 1050 °C (stretch line curves) and 1170 °C (dashed line curves) with diferent PBHT times of 0, 1, and 4 h

that, in turn, led to less stress concentration. Remarkably, the hardness between 450 and 550 HVN tended to correspond with indents on the eutectic constituent and matrix. The high hardness of about 800 HVN suggests the probability for the precipitation of Cr-rich intermetallics discussed in Fig. [5.](#page-6-0) Aside from these two observations, little diference in the hardness values at diferent brazing temperatures was noticed indicating brazing temperature did not have much efect on hardness. Instead, PBHT time has a considerable efect on phase formation and distribution. Thus, the microstructure and microhardness are consistent with the formation of brittle intermetallic compounds in ASZ. To minimize the extent of these detrimental phases, it is necessary to perform PBHT. On the other hand, Fig. [9](#page-8-0) indicates that PBHT lowers the base metal hardness because of grain growth, which, in turn, can deteriorate the mechanical properties of the base metal.

#### **3.2.2 Shear strength**

The shear strength of the brazed specimens was determined by the single lap shear test. An illustration of the joint geometry prepared for performing the shear strength tests is shown in Fig. [1.](#page-2-1) Figure [10](#page-8-1) shows room-temperature shear strength test results of the brazed joints with respect to diferent brazing temperatures and PBHT times discussed in this study. The strength of all joints is lower than that of NIMONIC 105 base material, and in all samples, failure occurred across the bonded interface except one. From Fig. [10](#page-8-1), it can be seen that the highest shear strength of the brazed joint made at 1170 °C followed by 4-h PBHT at 1000 °C case can be up to 92.2 MPa. The ductility of this specimen was as high as 19.0%, which is approximately 75% of the ductility of NIMONIC 105. This sample failed away from the base metal, as seen in Fig. [10.](#page-8-1) The maximum



<span id="page-8-1"></span>**Fig. 10** Stress–strain curves obtained by a room-temperature shear strength test of the brazed joints with respect to diferent brazing temperatures and PBHT times discussed in this study

ductility recorded in the present study is higher than those obtained in all similar previous works with diferent variables [[3–](#page-10-2)[6,](#page-10-3) [10,](#page-10-7) [27](#page-11-16)].

It is obvious that the shear strength of brazed joints is directly associated with that of joint microstructure. According to the literature  $[27-29]$  $[27-29]$  $[27-29]$ , the shear strength and ductility of brazed joints depend on diferent complicated factors that are summarized in the following:

• Both the quantity and the distribution of intermetallic compounds in the joint region

As shown in Fig. [3](#page-4-0), brazing temperature increment will increase the width of DAZ (quantity of precipitates) and change the morphology of precipitates, thus decreasing the shear strength of brazed joints from 62.4 to 40 MPa. The shear strength of joints made at a brazing temperature of 1170 °C remarkably increased from 40 to 83.8 MPa and fnally reached the maximum value of 92.2 MPa via PBHT. This is because PBHT resulted in the formation of a more uniform microstructure and distribution of intermetallic compounds in the joint region. With extending PBHT time, the elements difused deep into the substrates and gradually became uniform. Thus, the number of brittle intermetallic compounds decreased, and the corresponding shear strength increased. Moreover, according to Zhang et al. [\[29](#page-11-17)], the ductility of the brazed joints depended on the quantity of the boride precipitation in the DAZ, largely. High ductility of homogenized joints made at a brazing temperature of 1170 °C (13.5% and 19.0% for PBHT time of 1h and 4h, respectively) can be attributed to further difusion of MPDs and intermetallic dissolution. In other words, it can be said that large stress concentration caused by borides was relieved during PBHT.

#### • The base metal grain growth

This factor was signifcant when the brazing temperature was 1170°C (Fig. [2b\)](#page-3-0). The higher brazing temperature deteriorated the performance of base metal, resulting in the decrease of shear strength of the brazed joint to 40 MPa. On the other hand, in the case of the joint made at a brazing temperature of 1050 °C, a higher PBHT time of 4h led to lower shear strength (62.7 MPa) in comparison with a lower PBHT time of 1h (81.8 MPa). This fnding can be attributed to the same factor.

• ASZ thickness

As mentioned earlier, the phases formed in the centerline eutectic (ASZ) are deleterious to the mechanical properties. The centerline eutectic phases are avoided by completing isothermal solidification. However, if isothermal solidification is incomplete, the microstructure can be improved with a diffusion heat treatment [[17\]](#page-11-6). It is evident that for the joint made at brazing temperature of 1050 °C, homogenization of the specimen for 1h can increase the shear strength from 62.4 to 81.8 MPa. This is in accordance with the study of Khakian et al. [[6](#page-10-3)] that there is a reverse relationship between bonding strength and ASZ thickness. As it is shown in Fig. [2a](#page-3-0) and Fig. [7a,](#page-7-0) PBHT at 1000 °C will extenuate the ASZ width from 470 to 290  $\mu$ m. As the same way, regarding Fig. [7a](#page-7-0) and [b](#page-7-0) with increasing PBHT time from 1 to 4h, the ASZ width increased from 290 to 340 µm. As a result, the shear strength of the joint decreased to 62.67 MPa for higher PBHT time of 4h.

#### • Brazing time and temperature

Kim et al. [[10](#page-10-7)] found that increasing the brazing temperature increased the wettability of the filler metal and, therefore, the fracture strength increased. However, in the present study, increasing the brazing temperature from 1050 to 1170 °C resulted in a shear strength decline from 62.7 to 40.0 MPa. It can be concluded that blindly increasing the brazing temperature will not improve the joint performance. However, it will increase the possibility of defects (the voids observed in Fig. [6](#page-6-1)). Another mechanism that improves fracture strength is the diffusion of solid solution strengthening elements such as chromium, cobalt, aluminum, and titanium that diffused from the filler metal powder into the joint. Thus, by increasing the PBHT time (from 0 to 4h), the shear strength of joints made at brazing temperature of 1170 °C remarkably increased from 40 to 83.8 MPa and finally reached the maximum value of 92.2 MPa, because the microstructure becomes more homogeneous.

Drastic decrease in the shear strength of a joint made at the brazing temperature of 1170 °C can be attributed to the voids observed in Fig. [6](#page-6-1). Voids are signifcant stress concentration sites and have a detrimental infuence on the mechanical properties of the joints. As a result, the corresponding shear strength of the joint was drastically reduced to 40.0 MPa.

#### **3.3 Cyclic oxidation results**

In order to investigate the high-temperature performance of the joints, cyclic oxidation test is performed to compare the oxidation resistance of the selected specimens. The mass change with the number of oxidation test cycle curves corresponds to the selected brazements created at brazing temperature of 1170 °C with PBHT times of 0 and 4 h, during cyclic oxidation at  $1100^{\circ}$ C in air which is shown in Fig. [11.](#page-9-0) In fact, the spalling rate is a measure to compare oxidation resistance in cyclic conditions [[30](#page-11-18)]. The results show that the weight changes include an increase in the weight of the sample during the initial cycles (up to the 15th cycle) and then a decrease in the weight of the sample. As mentioned previously, the formation of Cr-rich and Mo-rich intermetallic compounds during the brazing process leads to the discharge of these elements from the center line of the brazements. These brittle phases would have a detrimental efect on the high-temperature performance of the alloy and make it prone to selective corrosion or oxidation. This fnding is in good agreement with other investigations [[3,](#page-10-2) [6,](#page-10-3) [9\]](#page-10-6). As mentioned, performing PBHT will result in a more uniform distribution of Ni, Cr, Mo, and MPD elements across the interface (Fig. [8\)](#page-7-1) and, in turn, increases the resistance to oxidation. Thus, the homogeneous distribution of Mo and Cr



<span id="page-9-0"></span>**Fig. 11** Mass change curves of selected brazements created at brazing temperatures of 1170 °C with PBHT times of 0 and 4 h during cyclic oxidation at 1100 °C in air

in the joint region seems to be a crucial factor about oxidation resistance improvement.

## **4 Conclusions**

The innovative approach of NIMONIC 105 brazing using BNi-2 fller, followed by PBHT, was experimentally investigated in this paper. Considering the results achieved, the following conclusions can be drawn:

- 1. The DAZ consists of globular/cuboidal and needle-like precipitates distributed in the interface. The precipitates in the DAZ were mainly composed of Ni–Cr-Mo-rich borides. The morphology and distribution of these secondary phases depend on the brazing temperature. So, the brazing temperature increment will increase the width of this DAZ from 12 to 35  $\mu$ m.
- 2. The ISZ was a precipitation-free zone, mainly consisting of gamma solid solution. With the rise of brazing temperature from 1050 to 1170 °C, the thicknesses of ISZ increase significantly from 15 to 30  $\mu$ m, respectively. The reason is that the rate of MPD difusion into base metal is higher at a higher temperature.
- 3. Brazing time of 8 min employed in this study was insufficient to complete the isothermal solidifcation process. As a result, the remained liquid undergoes super cooling and ASZ with eutectic microstructure that contains fne precipitates formed. ASZ contains the matrix solid solution of gamma-nickel, Ni-rich boride, and two Cr-rich phases. Cr-rich boride and Cr-rich intermetallic compounds are detrimental phases that formed in this zone. Void formation in ASZ reduced the strength signifcantly.
- 4. Homogenization of the brazements was performed at 1000 °C for 1 h and 4 h in order to achieve a more uniform microstructure. PBHT led to a more uniform hardness profle across the joint and a more homogenous interface region via the dissolution of intermetallic phases. PBHT time had a dual efect on the thickness of ASZ. So, the thickness of ASZ increased with increasing PBHT time for the joint made at the brazing temperature of 1050 °C and decreased with the increase of PBHT time for the joint made at the brazing temperature of 1170 °C.
- 5. The shear strength of joints made at a brazing temperature of 1170 °C remarkably increased from 40 to 83.8 MPa and fnally reached the maximum value of 92.2 MPa via PBHT. This is because PBHT resulted in the formation of a more uniform microstructure and distribution of intermetallic compounds in the joint region.
- 6. Performing PBHT will result in a more uniform distribution of Ni, Cr, Mo, and MPD elements across the interface and, in turn, increases the resistance to oxidation. Thus, the homogeneous distribution of Mo and Cr in the

joint region seems to be a crucial factor about oxidation resistance improvement.

## **5 Proposal for future work**

The quality of the brazement needs to be improved by the further design of interlayer composition and further optimization of the bonding parameters. This will include the insertion of an interlayer containing a high amount of refractory elements such as tungsten that efectively retard atomic difusion and control the formation of brittle boride precipitates. The authors intend to study this approach in subsequent studies.

The high-temperature performance of the repaired joints is another important issue. High-temperature tensile strength and stress-rupture life are suggested to be supplemented in future work.

## **Declarations**

**Conflict of interest** The authors declare no competing interests.

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