ADVANCES IN BRAZING: HIGH TEMPERATURE BRAZING OF MONOLITHIC 430 TO POROUS 430L STAINLESS STEEL FOR HYDROGEN SEPARATION APPLICATIONS

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ABSTRACT

The feasibility of high-temperature vacuum brazing of monolithic 430 to porous 430L SS for hydrogen separation applications has been investigated. Nickel (Ni) braze interlayers are prone to form secondary phases that affect joint strength, and porous substrates have the potential to further diminish the joint mechanical performance. The mechanical strength, braze microstructure, and phase chemistry were evaluated for three different Ni-Cr based filler alloys (Si-B, Si, and B-containing type). Brazing using a Ni-Cr-Si alloy resulted in extremely low braze strengths due to brittle phases formed along the joint centerlines in a Si-rich matrix which assisted crack nucleation and propagation during solidification. Higher joint strengths were obtained using a Ni-Cr-B alloy but the strength was limited by significant braze alloy infiltration into the porous body resulting in unwanted secondary phase formation. High-quality joints were achieved using a Ni-Cr-Si-B braze alloy regardless of the intermetallic compounds formed during brazing.

INTRODUCTION

The development of porous inorganic membranes for high-temperature hydrogen separation (from natural gas) is currently being investigated by the Oak Ridge National Laboratory (ORNL) and Pacific Northwest National Laboratory (PNNL) for coal gasification applications [1]. For this purpose, a system that serves to filter hydrogen and contains a joint between a monolithic tube with a porous section has been proposed. A schematic of this system for high-temperature hydrogen separation from natural gas is illustrated in Figure 1. The porous substrate has a 430L stainless steel support structure with a thickness greater than 400 µm (and a pore size ranging between 0.5 to 50.0 µm). The adjoining braze must provide a hermetic seal to ensure that H_2 extraction is efficient. In this application, the natural gas from which hydrogen would be extracted has been heated in the previous water gas shift reaction to temperatures ranging between 700 and 800ºC.

Ni-based filler metals have been successfully used for brazing ferritic stainless steels and a review of potential nickel based brazing alloys for this application has been performed by Malave [2]. Nickel-chromium-based (Ni-Cr) have been used as filler metals in joining iron-chromium alloys. However, the high concentrations of metalloid elements (i.e. silicon (Si) and boron (B)) and phosphorous (P) that act as melting-temperature depressant elements also form intermetallic compounds with chromium (Cr) and nickel (Ni) [3-5]. These phases are extremely hard and very brittle. They commonly agglomerate along the centerline of the joint, thereby decreasing the load-carrying capacity of the brazed joint. This brittle phase formation is a function of braze alloy chemistry, brazing time, brazing temperature, and joint thickness [3,5,6] so careful braze process control is necessary to obtain optimal joint strength.

Brazing of porous metals further complicates the brazing process. When porous materials are brazed, the filler metal alloy tends to flow by capillary action into the porous material depleting joint of braze material which can in turn result in weak braze joints [7]. Efforts have been made to produce reliable brazed joints in porous bodies, including methods that introduce additional materials into the structure. Several of the methods to produce reliable brazed joints in porous bodies have been reviewed by Malave [2].

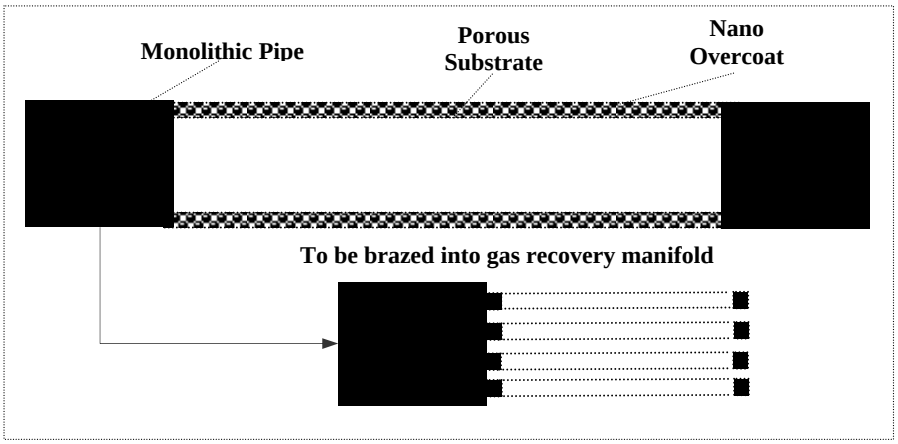

Figure 1 Schematic of system for brazing monolithic pipe to porous membrane for hydrogen separation applications.

EXPERIMENTAL

The feasibility of joining SS 430/porous-430L (monolithic-to-porous forms) has been carried out by means of the investigation of this material's brazing behavior with two different joint-types: a) a monolithic-to-monolithic joint (SS 430) and b) a monolithic-to-porous joint (SS 430/porous-430L), with the latter sandwiched between monolithic sections.

Materials

The base metal used for all monolithic brazing, joint characterization, and mechanical property studies was cold-rolled commercial grade 430 SS plates with a thickness of 4.8 mm. The nominal chemical composition for this alloy is Fe with 16.00-18.00 Cr, 1.00 Si, 1.00Mn, 0.12 C, 0.04 P, and 0.03 S (wt.%) [8]. The mechanical properties of the as-received cold rolled and annealed 430 SS samples were determined via tensile tests. The UTS for the annealed samples was between 330 and 366 MPa. Since the brazing temperatures in this investigation exceeded the 430 SS annealing temperature, the minimum strength obtained from the annealed tensile tests was used as a baseline.

The base metal was porous 430L SS fabricated by the Oak Ridge National Laboratories (ORNL, TN). The as-received material was tube-shaped and approximately 55.0 mm-length x 11.5 mm-OD x 0.7 mm-thickness. The as-received porous tubes were cut and flattened for brazing purposes. Chemical quantification from an EDX analysis of the as-received porous-430L tubes yielded the following chemistry: 73.7 Fe, 17.8 Cr, 2.4 Si, 5.4 O, and 0.7 C (wt%). Using mercury porosimetry, it was determined that the porous tubes exhibited a pore-volume of 0.0375 ml/g which is equivalent to approximately 29% porosity. Three Ni-based braze alloys, produced by Wall Colmonoy, Co. (Madison Heights, MI), were selected: 1) Nicrobraz L.M.® (BNi-2), a Ni-Cr-Si-B alloy , 2) Nicrobraz 30® (BNi-5), a Ni-Cr-Si alloy, and 3) Nicrobraz 150®, a commercial grade Ni-Cr-B alloy. The braze alloys were supplied in transfer-tape form, containing a controlled quantity of braze metal approximately 127 µm thick. The chemical compositions and brazing ranges for these braze alloys are given in Table 1.

Brazing Procedure

Prior to the brazing operations, the joining surfaces of the SS 430 specimens were ground to produce the desired surface finish (either a 15-µm or 1 µm polished surface). After grinding, the base metals (430 and porous 430L) were ultrasonically cleaned using acetone and then rapidly dried with hot air. Subsequently, the porous 430L substrates were baked at 100°C for about 30 minutes. The Ni-based transfer tapes were then placed on the ground and cleaned parent metals' surfaces. A porous metal layer was placed between them for the monolithic/porous sandwich configuration samples. Figure 2 shows a schematic of the butt joint-type specimen used for the monolithic-to-porous sandwich joint configuration. The specimens were vertically positioned in a graphite fixture and held in place using alumina wedges in order to maintain joint alignment and to avoid direct contact between the

SS plate surfaces and the graphite fixture. The following thermal cycle was performed under a vacuum of approximately 10^{-1}-10^{-2} Pa (10^{-4}-10^{-5} torr):

1. Heat to 950ºC at a rate of 20ºC per minute.
2. Hold at 950ºC for 1.2 ks (20 minutes).
3. Heat to desired brazing temperature at 20ºC/minute.
4. Hold at the brazing temperature for the desired time.
5. Cool to 800ºC at 1ºC/minute.
6. Cool to room temperature at 8ºC/minute.

Table 1 Composition and Brazing Temperature Range of Braze Alloys [9]

Ni-Based Braze Alloy AWS BNi-Series	Chemical Composition, wt.%						Melting Temperature (°C)		Brazing Temperature (°C)
	Cr	Fe	Si	C[a]	B[a]	Ni	Solidus	Liquidus	
BNi-2 Nicrobraz L.M.	7.0	3.0	4.5	0.06	3.10	Bal	970	1000	1010-1175
BNi-5 Nicrobraz 30	19.0	-	10.2	0.06	0.03	Bal	1080	1135	1150-1205
Commercial Nicrobraz 150	15.0	-	-	0.06	3.50	Bal	1055	1055	1065-1205

a. Maximum values

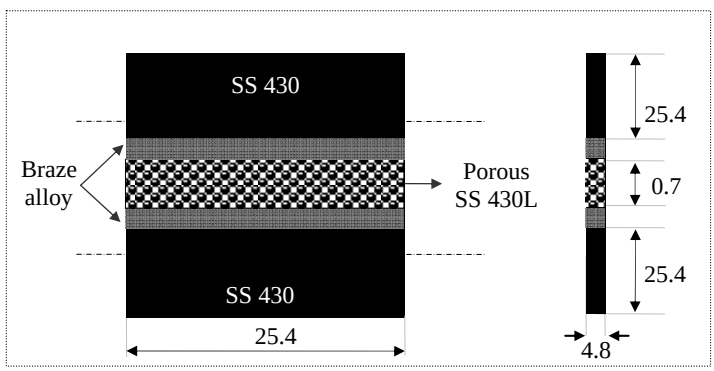

Figure 2 Schematic of SS 430/porous-430L brazed joint sandwich configuration (dimensions in mm).

Experimental Parameters

Testing was divided into three phases. The first phase was an evaluation of the effect of hold time, braze alloy, and joint thickness. For the second and third phases, additional variables were added for the most promising braze alloys from the first phase in an effort to determine the optimum brazing conditions for these systems. A total of 42 test conditions were evaluated. The overall brazing variables were:

- Joint thickness: 127, 254, and 381 µm.
- Isothermal hold: 0.6, 1.8, and 3.6 ks.
- Type of brazed joints: monolithic-to-monolithic components (SS 430) and monolithic-to-porous components (SS 430/porous-430L).
- Ni-Cr-based braze alloy: Si-B, Si, and B-type (as previously specified in Table 1).

- Brazing temperature: 1040, 1175 and 1205ºC.
- Monolithic surface finish: 1 µm and 15 µm.

Characterization

After the brazing operation, sections of the brazed samples were machined for joint strength testing. Uniaxial tensile tests were performed at room temperature using an Instron-mechanical testing frame with Series IX Automated software. Testing was performed at a constant displacement rate of 2 mm/min. The brazed specimens had a gage length of approximately 25 mm. A cross-section of each brazed joint was mounted in a phenolic mold and successively down to a final 1 µm finish with diamond paste. The microstructure of the joints was revealed by etching with Villellas reagent for about 20 seconds. The samples were then coated with gold-palladium (Au-Pd) for SEM and EPMA characterization. Braze cross-sections were examined using a Polyvar Met optical microscope. Optical observations were supported by further characterization using an environmental SEM (model: FEI Quanta 200F). Additional investigations were performed to determine joint quality and microstructural evolution (including intermetallic compound formation). An EPMA (model: JEOL JXA-8200 WD/ED) was employed to achieve accurate quantitative and qualitative (through compositional x-ray maps) local chemical composition of brazed joints by means of a wavelength-dispersive spectrometer (WDS). Analysis of B was performed in the Ni interlayer and in the segregation products of the joints obtained, having approximately 10% and 2% of quantitative estimate error, respectively [10]. The capabilities of this analysis technique and its experimental accuracy are discussed in greater detail elsewhere [2].

RESULTS AND DISCUSSION

Brazed Joint Tensile Strengths

A summary of the experimentally determined monolithic and monolithic-to-porous brazed-joint maximum strengths is presented in Table 2 (blank entries indicate that no brazed joint was fabricated under the specified brazing conditions). The minimum values and the individual test results are reported elsewhere [2]. The tensile test results corresponding to the different monolithic-to-porous SS 430/porous-430L workpieces brazed for 1.8 ks are also summarized in Figure 3.

For the Ni-Cr-Si-B braze alloy, joint strengths (316 to 380 MPa) on the order of the annealed SS base metal UTS were obtained for the SS 430/porous-430L joints brazed at 1175ºC for 1.8 ks. The presence of the porous substrate did not adversely effect the joint strength (the SS 430 joint strength ranged between 287 and 322 MPa under the same brazing conditions). The SS braze strength decreased when joined under the same brazing conditions for either 0.6 ks (303 to 308 MPa) or 3.6 ks (242 to 249 MPa). The brazing temperature was another factor that determined the joint strength. A detrimental effect on the joint mechanical strength (28 to 53 MPa) occurred when the temperature was decreased to 1040ºC, particularly when thick-interlayer joints were brazed. Thus, the maximum strength was directly proportional to maximum brazing temperature of the braze alloy regardless the joint thickness. For brazes fabricated at 1175ºC for 1.8 ks with a 254 µm thick-interlayer; the use of a monolithic surface polished to 1 µm resulted in an increased joint strength (366 MPa) relative to the 15 µm surface. The 381 µm (extra-thick) interlayer joint exhibited the highest strength obtained in this study (380 MPa).

For the Ni-Cr-Si braze alloy, extremely weak SS 430/porous-430L joints (10 to 125 MPa) were obtained when joining under all brazing conditions studied. The presence of the porous substrate in the sandwich configuration resulted in a decrease in strength relative to the brazed monolithic SS components (e.g. 241 versus 29 MPa for SS 430 and SS 430/porous-430L brazes respectively when joined with a 127 µm-thick interlayers at 1205ºC for 3.6 ks). Increasing the brazing temperature resulted in a decrease in strength for the SS 430/porous-430L joints (e.g. 29 versus 241 MPa respectively when raising the brazing temperature from 1175 to 1205ºC for 3.6 ks).

For the Ni-Cr-B braze alloys, intermediate to relatively high joint strengths were obtained for the SS 430 (156 to 279 MPa) and SS 430/porous-430L (157 to 224 MPa) joints under the different brazing conditions evaluated. The SS 430 joints exhibited slightly higher strength than the SS 430/porous-430L joints. Increasing the brazing temperature did not result in a significant strength improvement in the SS 430 and SS 430/porous-430L joints.

Table 2 Monolithic SS 430- and Monolithic-to-Porous SS 430/Porous-430L Brazed Joint Maximum Tensile Strengths (All Strengths in MPa)

Braze Alloy (Condition)	Hold Time=0.6 ks		Hold Time=1.8 ks		Hold Time=3.6 ks	
	SS 430	SS 430/ Porous-430L	SS 430	SS 430/ Porous-430L	SS 430	SS 430/ Porous-430L
Ni-Cr-Si-B alloy (Joints brazed at 1175ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint (a)	300	303	308	329	238	242
Thick-interlayer joint (b)	320	308	287	327	347	249
Extra-thick-interlayer joint (c)	-	-	-	380	-	-
Ni-Cr-Si-B alloy (Joints brazed at 1040ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint	-	-	53	229	-	-
Thick-interlayer joint	-	-	28	40	-	-
Ni-Cr-Si-B alloy (Joints brazed at 1175ºC using 1-µm diamond polished monolithic surface)						
Thin-interlayer joint	-	-	289	316	-	-
Thick-interlayer joint	-	-	322	365	-	-
Ni-Cr-Si alloy (Joints brazed at 1175ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint	-	-	164	82	178	125
Thick-interlayer joint	-	-	50	19	177	35
Ni-Cr-Si alloy (Joints brazed at 1205ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint	-	-	-	-	241	29
Thick-interlayer joint	-	-	-	-	-	-
Ni-Cr-B alloy (Joints brazed at 1175ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint	-	-	239	184	156	194
Thick-interlayer joint	-	-	279	191	206	157
Ni-Cr-B alloy (Joints brazed at 1205ºC using 15-µm grit SiC polished monolithic surface)						
Thin-interlayer joint	-	-	227	224	-	-
Thick-interlayer joint	-	-	269	198	-	-

(a) Thin interlayer=127 µm. (b) Thick interlayer=254 µm. (c) Extra-thick interlayer=381 µm.

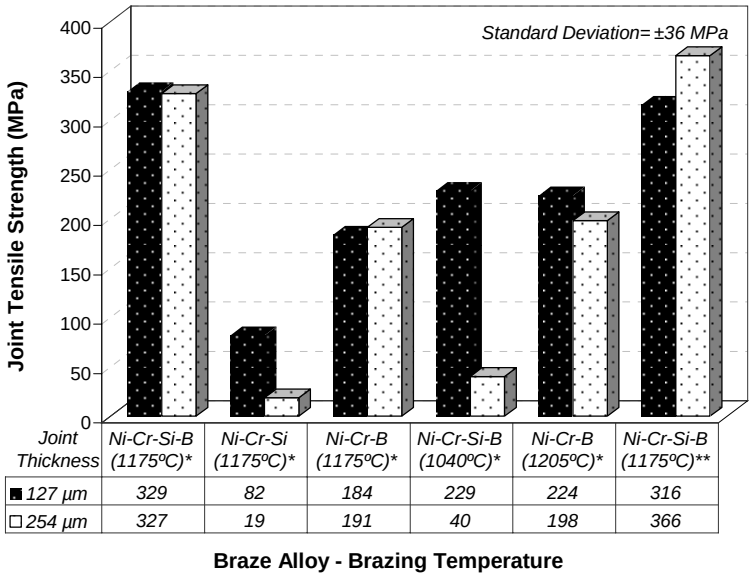

Figure 3 Tensile strength of monolithic-to-porous, SS 430/porous-430L, joints brazed for 1.8ks. (*) 15-µm grit SiC polished surface. (**) 1-µm diamond polished surface.

SS 430/Porous-430L Brazed Joint Microstructure and Chemical Composition

The braze microstructural development for the brazed alloys was relatively complicated. Each of the brazing parameters evaluated in this work influenced the nature of the secondary phases formed. In the systems studied, there was an interchange of elements between the Ni-based braze alloy and the monolithic 430- and porous 430L base metals. While the Ni interlayer was increasing in Fe and Cr composition by dissolution and infiltration from the SS components, the filler-metal elements were entering the base metals (Si and/or B when applicable, and to some extent Ni and Cr) during the brazing process. This resulted in a dilution of the Ni-interlayer alloy. Typical microstructures are presented and the major features are described in order to introduce the complexity of these microstructures.

The EPMA compositional x-ray maps used for the quantitative analysis of the bond Ni-interlayer and secondary-phase products are archived elsewhere [2]. Basically, these x-ray maps were used to analyze the Fe, Cr, Ni, Si, B, and C present in the bond region to qualitatively estimate: a) the bond elemental distribution and b) the chemical composition of the different joint zones (dissolution, base metal outside of the braze affected zone, infiltration, Ni-based solid solution, and segregation-products formation zones). Only the major results are presented and discussed. The emphasis of the chemical analysis is on the secondary phases as they were identified as the major factor in determining the strength of the joints.

(1) ***Ni-Cr-Si-B Braze Alloy (BNi-2):*** A typical microstructure for a Ni-Cr-Si-B braze alloys is shown in Figure 4. The typical structures exhibited by the brazes joined with Ni-Cr-Si-B alloy, can be divided morphologically into seven segments [2]. It is postulated that the flow of the braze alloy into the SS base metals was stopped during the joining operation, forming well-defined dissolution and infiltration zones in the joints brazed at 1175ºC. Good fillets were formed from the brazing operation, demonstrating adequate wetting when brazing at 1175ºC for 1.8 ks. In these joints, it was observed that: a) reduced zones of brittle structures formed only at some outer edges of the joints, b) shrinkage voids formed through intermetallic compounds at outer edges, and c) porosity formed in the central zone of the fillet surfaces and dissolution zones, as shown in Figure 4. Based on the EPMA analysis, a large number of intermetallic compounds (borides: Cr_xB_y and/or $Fe_xCr_yB_z$ and silicides: Ni_xSi_y, and $Ni_xCr_ySi_z$) formed in the braze layer during solidification. The degree of dissolution varied from 81 to 151 µm in the central segment of the joint. Meanwhile, the maximum degree of infiltration in the central segment of the joint varied from 109 to 192 µm. High-quality joints were obtained when brazing at 1175ºC for 1.8 ks regardless of the different secondary phases formed in the braze region during solidification. In this respect, neither the formation of borides nor the capillary flow of the braze material into the porous substrate affected the joint strength. This is an unexpected result since the formation of brittle phases has been stated in previous reports to have a detrimental effect on the joint strength [3,4,11].

The joints having thick interlayers exhibited large zones of intermetallic compound formation. These zones were generally concentrated in the outer section of the joints for the brazing temperature as illustrated in Figure 5. The secondary phase formation increased with brazing time and these hard phases were highly concentrated in the joint central region, as shown in Figure 6a. The EPMA results indicate that the joint strength was not affected by the segregation products formed during the brazing process with the exception of: a) Ni_xB_y-rich structures (possible compounds: Ni_3B and/or Ni_2B), [12] and b) clusters of Si and/or B segregation products (the latter formed along the bond centerline) [13,14]. The joint brazed with a 1-µm diamond polished monolithic surface produced a sound, nearly intermetallic-compound free, and outer edge-voidless joint (Figure 6.b). For these brazes, the Cr depletion from the base metal into the Ni solid solution was also reduced which could be related to the high boron content remaining in the Ni-Cr-Si-B braze alloy (0.53 wt.%).

(2) Ni-Cr-Si Braze Alloy (BNi-5): A typical microstructure for the Ni-Cr-S braze alloys is shown in Figure 7. Six different segments were identified in the joints [2]. Extremely weak joints were obtained for this alloy system. In these brazes, it was observed that: a) large zones of brittle structures formed along the joint centerline causing crack nucleation and propagation from the bond outer-to-central region during solidification (as shown in Figure 7); b) porosity formed along the Ni interlayer; and c) non-uniform dissolution zones formed. The bond interlayer consisted of Ni-Cr-Si-Fe solid solution and segregation products distributed along the braze centerline. The secondary phases consisted of Cr-rich, Ni_xSi_y, and $Ni_xCr_ySi_z$. It is proposed that the latter two compounds assisted the section cracking due to thermal stresses developed during solidification and that the Cr_xSi_y compounds probably did not severely affect the joint integrity. It is also hypothesized that the high Si content in the braze solid solution was the major factor that weakened the braze (as the Si content in the solid solution increased, the joint strength decreased). The

degree of dissolution ranged from 35 to 79 µm. and the highest strengths exhibited by the joints brazed with the Ni-Cr-Si braze alloys were related to the minimum penetration depth of the braze material. The degree of infiltration into the porous substrates was difficult to estimate since in most brazes there was a complete infiltration of Ni and Cr from the braze alloy into the porous 430L occurred.

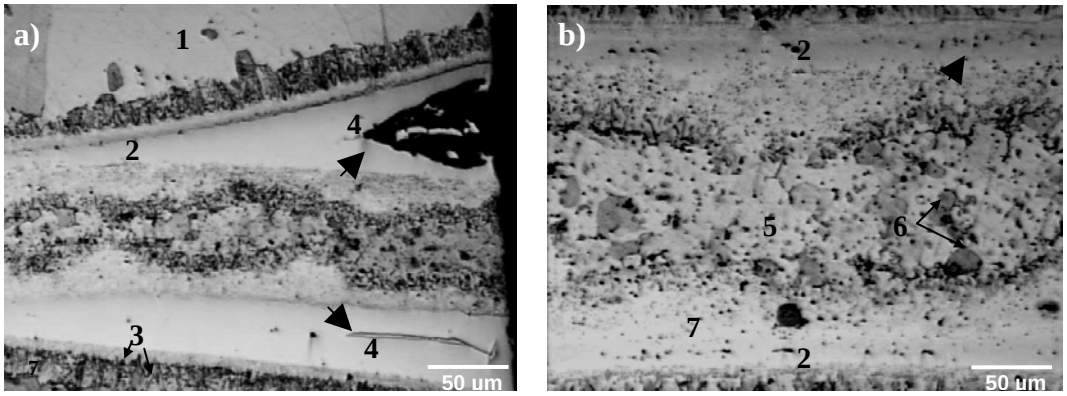

Figure 4 Typical microstructure of the SS 430/porous-430L joint brazed at 1175ºC for 1.8 ks with a 127 µm-thick Ni-Cr-Si-B interlayer (optical micrographs): a) joint outer section: arrow indicates Cr_xB_y segregation products and voids formed through them and b) joint central segment: arrow indicates porosity in the interlayer.

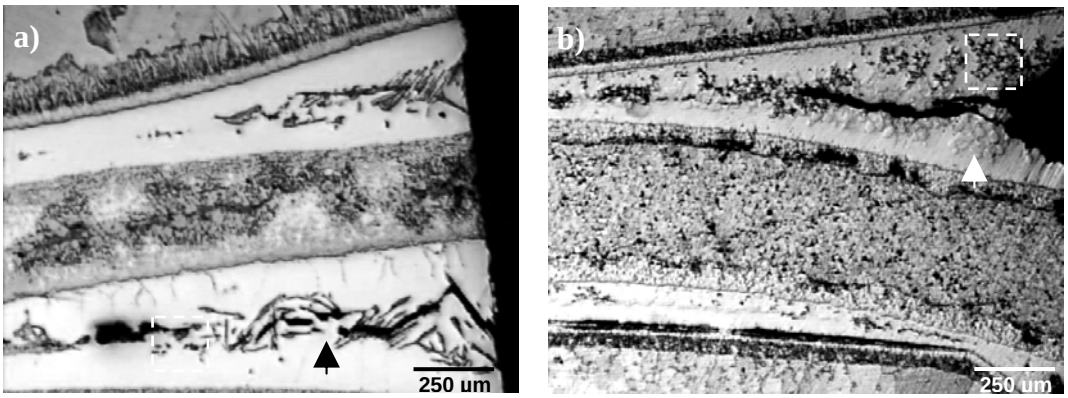

Figure 5 Typical microstructure of SS 430/porous-430L joints outer edge brazed with a 254 µm-thick Ni-Cr-Si-B interlayer (optical micrographs): a) 1175ºC for 1.8 ks: arrow indicates platelike Cr_xB_y precipitates and outlined square encloses B-rich phases and b) 1040ºC for 1.8 ks: arrow indicates globular Si-rich phases and outlined square encloses Cr_xB_y.

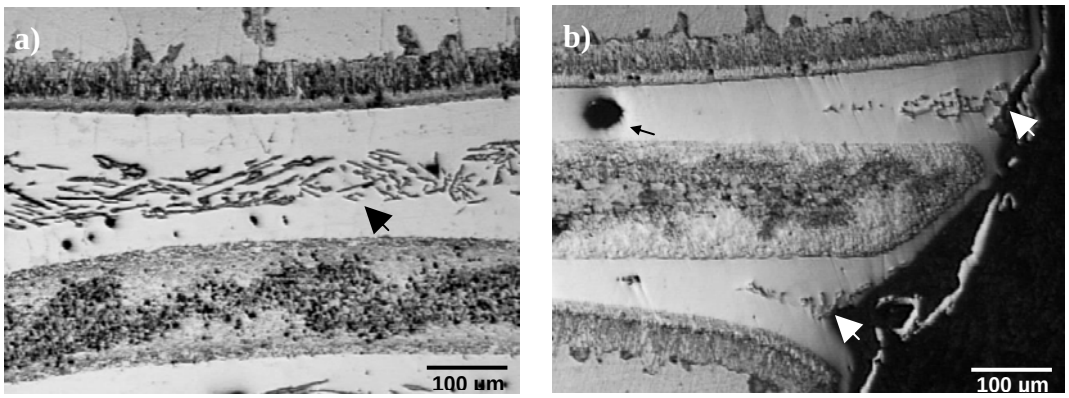

Figure 6 Typical microstructure of SS 430/porous-430L joints brazed with a 254 µm-thick Ni-Cr-Si-B interlayer at: a) 1175ºC for 3.6 ks: arrow indicates platelike segregation products along central joint and b) 1175ºC for 1.8 ks with 1-µm diamond polished SS surface: arrows indicate a few needles-shaped segregation products in the joint outer edge, slender arrow points to void formation (optical micrographs).

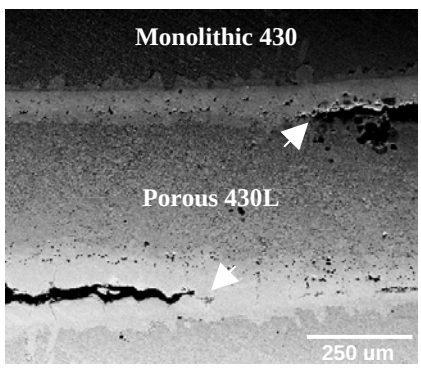

Figure 7 Typical microstructure of SS 430/porous-430L joint central region brazed with a 127 µm-thick Ni-Cr-Si interlayer at 1175ºC for 1.8 ks: arrows indicate crack propagation through Ni_xSi_y or $Ni_xCr_ySi_z$ secondary phases (SEM/BSE image).

(3) Ni-Cr-B Braze Alloy (NB 150): A typical microstructure for the Ni-Cr-B braze alloys is shown in Figure 8. The corresponding typical microstructure for a thicker interlayer and a longer hold time is shown in Figure 9. The typical microstructure of these joints was characterized by: a) void formation and void coalescence at the joint side edges subsequent to secondary phase precipitation (as shown in Figure 8); b) large infiltration of the porous substrate (as shown in Figure 8); c) distortion in the porous substrate that assisted the formation of detrimental thick sections in the bond, causing secondary phases to form (as illustrated in Figure 8b); d) cracking through intermetallic compounds during solidification (as shown in Figure 9); and e) porosity formed along the interlayer. Moderate-to-complete infiltration in the porous body was exhibited by the brazes under the different brazing scenarios evaluated. It is hypothesized that the braze alloy did not stop flowing into SS porous substrate and therefore boron remained in the liquid braze alloy when isothermal solidification began. Intermediate to relatively high joint strengths were obtained under the different brazing conditions evaluated in this investigation. It is suggested that aggressive infiltration caused distortion in the porous substrate, which assisted the formation of unusual thick sections in the bond, where secondary phases formed. The secondary phases formed in the bond region (complex compounds, Cr_xB_y, $Fe_xCr_yB_z$, and $Ni_xCr_yB_z$) did not exhibit a significant correlation with the joint strength. The minimum

strength (157 MPa) was obtained for the braze exhibiting the formation of $Ni_xCr_yB_z$. Platelike Cr_xB_y phases were found to be similar to those formed in the Ni-Cr-Si-B-interlayer joints. The degree of dissolution of the SS base metal in the joint central region varied from 107 to 143 µm. All of the Ni-Cr-B-interlayer-brazes analyzed in this study exhibited large poorly-defined infiltration zones in the porous substrates.

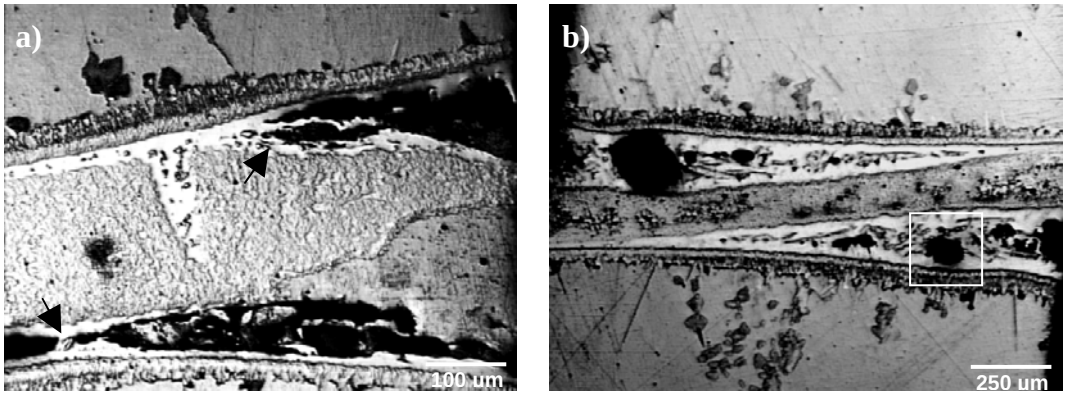

Figure 8 Typical microstructure of SS 430/porous-430L joints brazed with Ni-Cr-B interlayers at: a) 1175ºC for 1.8 ks with a 127 µm-thick interlayer exhibiting complete infiltration and voids at joint outer edges: arrow indicates complex intermetallic compounds; b) 1205ºC for 1.8 ks with a 254 µm-thick interlayer exhibiting detrimental thick bond's sections and porous substrate distortion: outlined square indicates voids formed within borides matrix (optical micrographs).

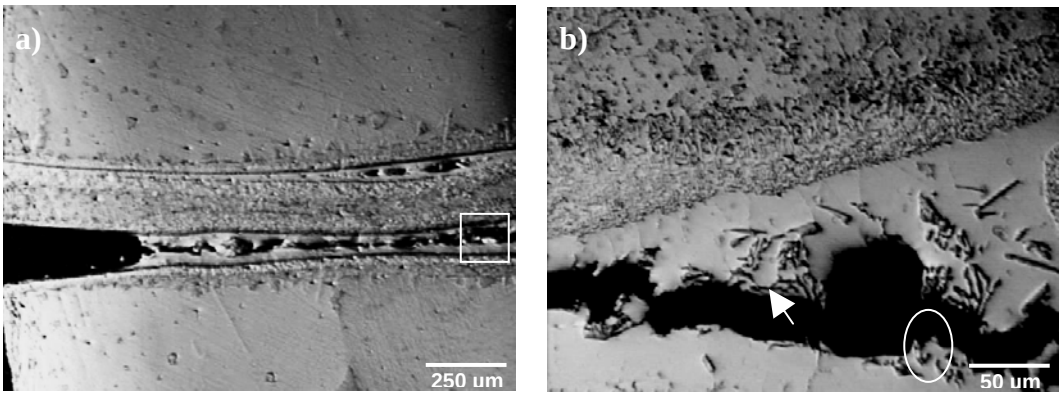

Figure 9 Typical microstructure of SS 430/porous-430L joints brazed at 1175ºC for 3.6 ks with a 254 µm-thick Ni-Cr-B interlayer (optical micrographs): a) braze exhibiting cracking along interlayer centerline during solidification and b) outlined square indicates cracking through secondary phases: arrow points to $Ni_xCr_yB_z$ compound and area outlined by oval indicates Cr_xB_y compounds.

CONCLUSIONS

Joint strengths on the order of the ultimate tensile strength of annealed 430 SS demonstrate the feasibility of brazing monolithic SS 430 to SS porous-430L. The highest quality joints were obtained when brazing with a Ni-Cr-Si-B braze alloy (BNi-2) at 1175ºC for 1.8 ks specifically for the 254 µm-thick interlayer joints with SS surfaces polished to 1 µm. It is postulated that the latter variable assisted in the formation of nearly free segregation-formation and

outer-edge voids. For thick-interlayer brazes, this brazing condition prevented excess Cr depletion from the monolithic base metal to the braze alloy. Conversely, the SS 430/porous-430L joints brazed with the Ni-Cr-Si interlayers were the weakest joints in this study exhibiting extremely low strengths (19 to 125 MPa) under the different brazing conditions evaluated in this study. This weakness was particularly apparent when brazed with thick interlayers. Intermediate to relatively high strength (157 to 224 MPa) was exhibited by the SS 430/porous-430L brazes joined using the Ni-Cr-B braze alloy under the different brazing variables examined. Generally, the low-strength joints exhibited large unbrazed zones and the formation of detrimental secondary phases that provided sites where cracks originated and/or propagated.

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