Microstructural and mechanical investigation of brazing 304L Stainless Steel with corner joint using a Ni-based shim and wire

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Abstract
This study investigated a suitable filler metal, diffusion depth of the elements of this filler metal in the base metal, and its effect on the mechanical properties of brazing bonding of sheets with different thicknesses of 304L stainless steel. For this purpose, brazing was performed with thermal induction under a vacuum of $10^{-5}$ mbar and filler metal BNi-2 at the corner structures. First, the microstructure of different joint regions including base metals, Ni affected areas, and interfaces were examined by scanning electron microscopy and then the relationships between microstructure and microhardness were compared. In sheets with different thicknesses, the differences in diffusion depth were observed. Moreover, the microhardness of different areas of the braze was revealed to be completely affected by the diffusion of nickel. Finally, it was found that, for the bonding between 304L sheets with different thicknesses, BNi-2 filler metal had reasonable strength, diffusion, and filling for vacuum applications.

1. Introduction
Brazing offers various exclusive features in comparison with other joining techniques, for instance adhesives, welding or riveting; hence it is considered as a very versatile technique [1–3]. The major benefit of brazing in comparison with the alternative joining methods is its wide compatibility to join dissimilar materials, in addition to performing this process without the necessity of modifying the materials being joined [4, 5]. Due to the requirements of brazed joints as permanent connections in a number of industries such as chambers and vacuum valves, choosing the right material in terms of brazability is an obvious need. On the other hand, the choice of filler metal and brazing method of each material will have a special effect on the joint. Among metals and alloys, stainless steels may be agreeable for such reasons as wettability, proper melting range and suitable fluidity for diffusion into the joints, sufficient homogeneity of the chemical composition, good stability in minimizing the detachment of components by liquation, well-suited mechanical and physical properties to be used in vacuum valves applications, and the capability to avoid the formation of brittle intermetallic compounds or extreme erosion [6, 7]. Moreover, they have excellent brazability, which can be poured in different methods of brazing and in different thicknesses.

Filler metals with high melting points are frequently among one three categories: palladium, nickel or gold-based alloys. No alloy, as listed in ISO: 1762:2016 standard of any of the three filler metal categories, has a solidus temperature lower than 800°C, hence they can be the candidates for joining by brazing in applications, where high service and processing temperatures are required, e.g. in gas turbines and jet engines. Moreover, Pd and Au-based alloys are also widely used in vacuum tubes and for joining metal/ceramic components. Turbine blades are exposed to very high temperatures during service and the filler metals which are used to repair them must be capable of offering high operating temperatures [4, 6, 8].

Au- and Pa-based fillers are not as expensive as nickel-based fillers due to their very high price and not being used in industrial applications. In addition, the atomic radius of nickel is less than those of silver and copper ($r_{\text{Ni}} = 0.125$, $r_{\text{Ag}} = 0.144$ and $r_{\text{Cu}} = 0.128$ nm). Therefore, diffusion of nickel in steel at the same brazing
Materials and method

2.1. Materials

304L steel sheets in dimensions of 3 × 40 × 300 and 5 × 60 × 300 mm³ were used as the base metals and nickel base filler metal as the shim (width being 3 and thickness 0.05 mm) and wire (diameter 1 mm), whose chemical compositions are brought in table 1. To achieve the highest wettability, obtaining the proper overlap, sufficient diffusion and high strength for the brazed joint based on an earlier study [6], the surface roughness of 1 μm was selected. For this purpose, multi-stage polishing was carried out and after each stage the surface roughness was measured using a roughness gauge (Mitutoyo, Japan).

2.2. Brazing

Corner joint brazing is carried out with a thermal source of induction, an energy source of electrical and vacuum shielding. A vacuum of ~10⁻⁵ mbar was used to protect the braze. For the aim of fixing the sheets, TIG spot welding with a distance of every 2 cm was used, whose schematic of the created assembly is shown in figure 1(a). Furthermore, fixers were used to prevent the sheets from warping during brazing. A seam thickness of 0.05 mm was selected. Brazing was performed at 1100 °C for 2 min according to figure 1(b). It should be noted that the sample was preheated to 950 °C for 3 min before the brazing so as to prevent any thermal shock and higher filling quality. Braze heating was performed at a 45° angle. This angle was chosen for a better wire feeding and proper
| Material | C  | Si  | Mn  | Cr  | Cu  | Mo  | S   | P  | Al  | V  | Ti  | Co  | B  | W  | Fe  | Ni  |
|----------|----|-----|-----|-----|-----|-----|-----|----|-----|----|-----|-----|----|----|-----|-----|
| 304L     | 0.02 | 0.06 | 0.57 | 0.93 | 18.41 | 0.08 | 0.07 | 0.022 | 0.038 | <0.03 | <0.03 | 0.18 | 2.7 | 8.58 | Bal. |
| BNi-2    | 0.06 | 4.6  | 0.93 | 18.41 | 0.08 | 0.07 | 0.022 | 0.038 | <0.03 | <0.03 | 0.05 | 0.25 | 2.85 | 2.7  | Bal. |
adhesion strength on two of the steel sheets in the same way. Additionally, a concavity was created for the aim of increasing the bond strength of the braze due to the capillary nature of BNi-2 melt with 304L sheets and 45° angle of brazing operation (figure 2).

2.3. Mechanical test
Butt joint was used to perform the tensile test. For this purpose, 6 sheets of 304L steel with dimensions of $100 \times 30 \times 3$ mm$^3$ and 6 sheets of 304L steel with dimensions of $100 \times 30 \times 5$ mm$^3$ were used. Sheets of the same thickness in the sections of $20 \times 3$ and $20 \times 5$ mm$^2$ were brazed by shim with a thickness of 0.05 mm and exactly the same brazing conditions in the corner structure. They were then subjected to a tensile test according to ASTM A370 (figure 3). Tensile tests were performed by Santam STM-150 at a crosshead speed of 2 mm min$^{-1}$. To increase the accuracy of the test results, these experiments were repeated three times for each case and average of the obtained data was reported. Next, Vickers microhardness was used to determine the hardness of the layers in the braze microstructure. The QV-1000 DAT from Qualitest USA was used and the experiments were performed with a load of 100 g and dwell time of 10 s.

2.4. Microstructure characterization
Ferrite percentage was measured according to ANSI/AWS A4.2M/A 4.2 standard at room temperature (According to ASTM A799) by Fisher FMP 30 American Fisher Scope. The error percentage of the 304L steel
ferrite and Ni diffusion zone with the ferrite scope machine was about 0.1%. In addition, microstructural and chemical composition evolution was carried out using the Philips scanning electron microscopy and EDS.

3. Results and discussion

Figure 4 shows microstructure of the braze joint and it is evident that there is no porosity and crack. Absence of cracks and porosity in the filler metal can be attributed to three factors. The first factor concerns structure design in a way that, at the corner connection, one side of the geometry is considered open to allow gases to escape in order to prevent porosity (figure 2). The second is related to the presence of feed at the top of the braze. Finally, the third factor relates to the vapor pressure of the elements at the brazing temperature which are calculated by equation (1) [25, 26]. Because the vapor pressure of the nickel base is low at the brazing temperature (2.3 × 10⁻⁹ atm at 1100 °C), the final porosity of the filler is low.

\[ \log P \text{ (atm)} = A + B.T^{-1} + C \cdot \log T + D.T.10^{-3} \] (1)

Where P is the vapor pressure (atm), T is the brazing temperature (K), and A, B, C, and D are the equation constants for each element.

At the brazing temperature, the vapor pressures of other conventional fillers, \( P^{Ag} \) and \( P^{Cu} \), are equal to 4.8 × 10⁻⁷, 7.8 × 10⁻⁷, respectively, indicating that in the brazing temperature, there is always \( P^{Ag} \gg P^{Cu} \gg P^{Ni} \). Therefore, nickel-based filler metals are the cause for the lowest porosity in terms of vapor pressure. On the other hand, in nickel-based filler metals, the vapor pressure of their alloying elements such as chromium and iron is important. The vapor pressure of chromium is 3.3 × 10⁻⁸ atm and iron is 1.1 × 10⁻¹¹ atm, due to the fact that the lower the amount of these elements in the filler, the less porosity occurrence in the joint. Thus, BNi-2 filler metal, which has the lowest amount of iron and chromium in the group of nickel-based filler metals, is suitable for the brazing vacuum joints [6, 25, 26].

Figure 4 depicts the joint design, the thickness of the joint and the seam. According to this figure, 0.05 mm thickness of the seam which is included in the initial design is clearly visible, which is filled with filler metal and no porosity nor filling are evident. Due to high temperature eutectic solidification (1100 °C), AWS BNi-2 brazing created a uniform melt flow in the 0.05 mm seam, which caused fulfilling of the filler metal elements. The BNi-2 melt had good capillarity on the 304L surface. The design of the engineered joint in terms of joint thickness, smoothness of the joint surfaces, the shape of the filler metal such as shim, the use of filler metal backing, as well as the use of vacuum shield, caused proper capillary and no porosity in the joint. Proper capillary caused optimal diffusion (creation of diffusion layers) in the microstructure (figure 5). This diffusion had created the required strength against vacuum pressure. Since by reducing the seam distance, the strength is increased, the lowest possible practical distance of 0.05 mm was selected. Of course, this thickness should be such that the filler metal would have acceptable fluidity and capillarity when brazing and also in a way that it would create the most appropriate interface strength [6, 27]. For the joint, it could create the best shear strength according to the vacuum shield and BNi-2 filler metal and 304L base metal [26].

According to figure 5, it became clear that precipitation was not obtained in the microstructure during the brazing. Formation of carbides (SiC, B₁₁C, B₄C, Fe₃C and Ni₃C), borides (CrB₂, SiB₃ and SiB₆) and nickel
silicates (Ni$_2$Si, NiSi$_2$ and Ni$_3$Si) required high temperatures and time, such as SiC, which, around the melting point of Si (1408 °C), B$_2$C and B$_4$C, required a minimum temperature of 1600 °C and SiB$_3$ and SiB$_6$; these were formed at temperatures above 1220 and 1300 °C, respectively, requiring sufficient concentrations of silicon and boron elements. Also, free energy was not low enough to form compounds. Therefore, the mentioned compounds could not be formed at low temperatures such as brazing temperature [28–32]. On the other hand, lack of brittle phases in the 304L steel structure could be due to short joint length and low brazing clearance, which required short brazing times that reduced the temperature gradient in the sample and did not allow the formation of brittle phases in the 304L steel joint. Furthermore, due to using a low-carbon grade of austenitic stainless steel, plate shape of the work piece, low seam distance and low brazing time (2 min), there was no sufficient carbon content and enough time for the formation of chromium carbides over the temperature range of 450 °C–800 °C during cooling [16, 33–37]. Hence chromium carbide was not observed in the 304L joint.

The amount of 0.5% ferrite was measured by ferrite scope analysis in 304L structure. According to equations (2) and (3), the values of Cr and Ni equivalents, 304L steel was equal to 2.1. On the other hand, if the C$_{req}$/N$_{eq}$ ratio was greater than 1.95 and, according to Scheffler’s modified diagram, the presence of ferrite in the structure was negligible [33, 38]. However, in the Ni diffusion zone of 304L steel, a significant decrease in the C$_{req}$/N$_{eq}$ ratio occurred due to diffusion of nickel in the ferrite phase structure and the enrichment of this layer with nickel. Thus, the transformation of ferrite into austenite due to high temperature of the brazing and increase of nickel have completely been done and the percentage of ferrite in the structure of the braze diffusion layer has significantly decreased so that its insignificant amount could not be measured.

\[
\text{Cr}_{\text{eq}}(\text{wt.\%}) = \text{Cr} + 2\text{Si} + 1.5\text{Mo} + 5\text{V} + 5.5\text{Al} + 1.75\text{Nb} + 1.5\text{Ti} + 0.75\text{W} \quad (2)
\]

\[
\text{Ni}_{\text{eq}}(\text{wt.\%}) = \text{Ni} + \text{Co} + 0.5\text{Mn} + 0.3\text{Cu} + 25\text{N} + 30\text{C} \quad (3)
\]

Austenitic stainless steels, such as 304L, provided an angle of 10–45° for wettability for nickel-based filler metals, which caused the molten nickel filler melt to spread evenly over them [6]. On the other hand, high
brazing temperature, such as 1100 °C in this study, increased the wettability of 304L steel. Finally, increased wettability led to optimal diffusion (figure 5).

Figure 5 illustrates the diffusion layers at the joint, where two diffusion layers have been formed in the 304L steel with BNi-2. The creation of diffusion layers indicates the optimal filling in the overlap joint. The primary diffusion layer is formed by intragranular diffusion (>0.75–0.8 Tm) and the second diffusion layer is formed by granular diffusion (<0.75–0.8 Tm). By calculation of the grain size of the selected sheets via linear intercept method on the micrographs, due to less thickness, the 304L sheet with a thickness of 3 mm, the number of grains per area unit was more than that of the 304L sheet with the thickness of 5 mm. According to figure 5, when brazing (1100 °C), in the sheet with a thickness of 3 mm was compared to the sheet with a thickness of 5 mm, the depth of diffusion in grain was shown to be greater and the depth of diffusion of grain boundary was less. By considering the fact that a volume unit of the sheets consisted of grains and grain boundaries, consequently grain and grain boundary volume percentages affect the diffusion phenomenon. Hence, the thickness of the grain diffusion layer was higher because in a sheet with a thickness of 3 mm, the grain boundary volume percentage was higher compared to that of a sheet with a thickness of 5 mm, and the grain boundary volume percentage was lower. In other terms in a uniform diffusion layer thickness, with a thickness of 3 mm, it required more time to fill its grain boundaries with BNi-2 elements, and 304L sheet with a thickness of 5 mm, required more time to fill its grains with BNi-2 elements. On the other hand, due to the lower volume percentage of grain boundary in the sheet with a thickness of 5 mm, grain diffusion occurred more intensely, leading to the reach of grain boundaries.

According to defect-free lattice diffusion coefficient equation 11 and to compare the diffusion of the main elements BNi-2 in 304L, the diffusion data of Si, Cr and Ni elements in Feγ were used. As it is obvious in table 2, nickel has the lowest permeability of gamma iron at brazing temperature. But according to the first law of Fick equation (4)

$$J_{Ni} = -D_{Ni} \frac{\partial C_{Ni}}{\partial X}$$  

and as figures 5 and 6 show, it is evident that the concentration gradient of the element nickel ($\frac{\partial C_{Ni}}{\partial X}$) has been much higher than the concentration gradient of silicon and chromium. Hence, the diffusion flux of nickel atoms into 304L ($J_{Ni}$) was much higher than the diffusion flux of chromium ($J_{Cr}$) and silicon ($J_{Si}$) [9, 13, 39, 40]. As a result, most part of the surface, grain boundary and volumetric positions diffused by Ni and the microstructure were affected by nickel (figure 6).

The results of tensile tests for 304L sheets in as-received, preheat treated at 950 °C and butt joint are given in table 3. The UTS of the sheets with a thickness of 3 mm was higher than the sheets with a thickness of 5 mm in the received samples and the preheat treated at 950 °C, because the sheets with a lower thickness were finer and
had more volume percentage grain boundaries. However, in brazed butt joint samples at 1100 °C, the connection in the 5 mm thick sheet was wider than in the 3 mm thick sheet with higher mechanical properties. Figure 7 shows the microhardness of different braze regions. Based on this figure, the microhardness of the initial diffusion layer (Lattice diffusion, h₂) and the secondary diffusion layer (grain boundary diffusion, t₂) in the 3 mm sheet has been higher than that of the 5 mm sheet. On the other hand, hardness of the initial diffusion layer was always higher than the second diffusion layer and the hardness of the second diffusion layer was higher than that of the base steel sheet, because in the initial diffusion layer, nickel diffused in the grains and grain boundaries and in the second diffusion layer only diffused in the grain boundaries.

4. Conclusions

According to the studies which have so far been carried out on the selection of BNi-2 filler metal for 304L steel brazing for vacuum applications, the results of the diffusion of BNi-2 base elements in 304L steel sheets and the effect of diffusion on the microstructure and mechanical properties can be presented as following.

Table 2 Data needed to calculate the influence of Si, Cr and Ni on Feγ

| Element (in Feγ) | D₀ (cm²/s) | Q (J/mol) | D (cm²/s) | Brazing temperature (K) | R (J/K mol) |
|-----------------|-----------|-----------|-----------|------------------------|------------|
| Si              | 0.4       | 201000    | 9 × 10⁻⁹  | 1573                   | 8.314      |
| Cr              | 10.8      | 291800    | 8.57 × 10⁻¹¹ | 11373                 | 3.39 × 10⁻¹² |
| Ni              | 3         | 314000    | 3.39 × 10⁻¹² | 1239                 |            |

Table 3 Strength and hardness of 304L sheets at as received and preheat treated at 950 °C

| Heat treat                      | Thickness (mm) | UTS (MPa) | Y₀.2% offset (MPa) | Hardness (HV) |
|---------------------------------|----------------|-----------|--------------------|---------------|
| As received                     | 2.9            | 710       | 486                | 220           |
|                                 | 4.9            | 636       | 421                | 162           |
| Preheat treated in 950 °C       | 2.9            | 535       | 288                | 158           |
|                                 | 4.9            | 510       | 229                | 155           |
| Brazed butt joint in 1100 °C    | 2.9            | 216       | —                  | —             |
|                                 | 4.9            | 236       | —                  | —             |

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1. Due to the need for high temperature and time to form carbides, borides and nickel silicates, use of low carbon grade and the low free energy of their formation were required to be managed so that the compounds at brazing temperatures might not form.

2. Creation of two diffusion layers that were created due to lattice diffusion and grain boundaries diffusion.

3. In the sheet with less thickness, the number of grains and the depth of intra-grain diffusion was more than the thicker sheet, but the grain boundary diffusion depth was less because, in the sheet with less thickness, the grain volume percentage was higher and the grain volume percentage was lower.

4. Nickel has the lowest diffusion coefficient in gamma iron at brazing temperature, but the concentration gradient of nickel was much higher than the concentration gradient of silicon and chromium. Hence, the diffusion flux of nickel atoms into 304L was much higher than the diffusion flux of chromium and silicon. Therefore, most of the surface, boundary and volumetric diffusion sites were affected by nickel.

5. Tensile strength of the braze joint was less than the tensile strength of the base sheets. Also, due to the wider connection in the 5 mm sheet, a higher tensile strength was created than the 3 mm sheet.

6. Microhardness of the initial diffusion layer and the second diffusion layer of 3 mm sheet, were higher than that of the 5 mm sheet. On the other hand, the hardness of the initial diffusion layer was always higher than the hardness of the second diffusion layer and the hardness of the second diffusion layer was higher than the base steel sheet. This was because, in the initial diffusion layer, the element nickel diffused into the grains and grain boundaries, but in secondary, it was diffused only in grain boundaries.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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