Microstructure and phase analysis of brazing bonds for stainless steel (AISI 316L and 431) to carbon steel (A516 G70) using a Ni–Si–B filler metal alloy

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Abstract: Investigation the mechanism of brazing stainless steels (Austenitic stainless steel type 316L and martensitic stainless steel type 431) similar and dissimilar and also brazing them with carbon steel type A516 grade 70 to see the difference in bonding phases, the five specimen were brazed with Ni base filler metal with (4.5% Si and 3.2% B) using a furnace with protected gas (pure Argon) at constant holding time 10 min. The optical and scanning electron microscopy (SEM) are used to study the joint microstructure, also the interdiffusion of the elements was examined by energy dispersive spectroscopy analysis (EDS). While the reaction phases are identified by x-ray diffraction (XRD). The brazed joints showed continuous bonding between the filler alloy and the substrate, indicating to good wetting between the surfaces. EDS line and mapping scan shows The Nickel element which it’s at the filler metal alloy will be goes into the interface almost, because the stainless steels sides can handle it, also Cr element. While Si element will concentrate at the bond zone, finally Fe element Transfer through brazing zones easy. XRD test reveals that the bonding zone of the five specimen shows more the six types of phases which are Ni-Cr-Fe, Cr-Ni-Fe-C, SiC, Fe2B, Cr2B, and Cr-Fe-Ni-Mo.

Keywords: Brazing, Microstructure, XRD, Ni base filler metal, Ni-Si-B, Stainless steel.

1. Introduction

A significant type of stainless steel has been developed, and many applications required them such as kitchenware and automobiles, electrical applications, and construction. When it comes to joining pieces of stainless steel via heat treating, there are important reasons to use brazing instead of welding, in brazing only the filler metal is melted not the base metals which means lower temperature is required in brazing and hence lower distortion, when workpieces are joined together across a wide point of contact, brazing is preferable [1]. To accomplish a good brazing joint with furnace method, the specimen must be properly cleaned and also needs protection during heating process using either flux or shielding gas to avoid excessive oxidation. To ensure the capillary action for the filler metal can be afforded the specimens must properly aligned.

During the bonding process, the filler metal which is containing melting point depressants such as Si, P, and B is placed between the base metal surfaces and the then the specimen is heated to the brazing temperature. Interdiffusion of the alloying element occurs between the base metal and the inserted filler metal, especially the diffusion of melting point depressants (B, P, and Si) to the base metal, resulting in change in the composition and isothermal solidification in the brazed joints. Unlike conventional welding process, holding time must be acquired to ensure the isothermal solidification of the liquid in transient liquid phase bonding. Transient liquid phase
bonding for such systems as pure nickel, nickel-based superalloy, carbon steel and stainless steel with Ni- and Fe-based filler metals have been widely investigated [5-11]. In 2009 C. Yun Kang and his colleagues studied brazing of duplex stainless-steel using Ni- based filler metal alloy. The microstructure of the brazed specimen was tested using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and layer by layer XRD analysis. The results show that before isothermal solidification to take place, BN, Ni3B, and Ni5Si phases precipitate at the interface. After completion of isothermal solidification only γ-nickel phase exists in filler metal zone [2].

In 2018 Y. Chen, H. Cui investigated brazing of martensitic stainless steel (12Cr-1Mo) using nickel base filler alloy (BNi-2) in a protected atmosphere of argon inert gas. The effect of brazing temperature (1050, 1120) °C and holding time of 10, 30, 50, 90 seconds was studied. The results show the element diffusion of filler metal is more active with increasing brazing temperature and holding time [3]. A. Ghasemi, M. Pouranvari (2017); their study was brazing of Hastelloy X nickel super alloy using (Ni-4.5%Si-3.2%B) filler metal. The aim of the study was to understand the solidification phenomena and solid solution precipitation. The isothermal solidification of the joint is characterized by formation of nickel rich solid solution, while the athermally solidified zone featured borides/silicide formation, on cooling precipitation of fine nickel silicide at filler metal zone, and diffusion affected zone contain Mo-Cr rich borides [4].

2. Experimental Work

2.1. Specimens Preparation:
Austenite and martensite Stainless steel and carbon steel specimens preparation was done by cutting them to rectangular shape with 20mm x 20mm x 10mm dimension. Cutting was done by wire cutting. After cutting specimens were grinding by using emery paper of SiC with 180 grid to 800 grid. After grinding process, specimen cleaned up with alcohol, washed, and dried to remove any oil or contamination on surface.

2.2. Filler Metal Preparation:
In this work a nickel-based filler metal was used containing (4.5% Si, and 3.2 B) in form of past, the paste of filler metals were prepared by weighting a suitable amount of metal powder alloy and mixing together with drops of glycerin to form a filler metal paste which is suitable for one joint [12]. The required filler alloy weight of each sample depended upon the volume of the designed gap for that sample, and the filler alloy density, according to the equation:

\[ \rho=\frac{M}{V} \]

Where M. Filler alloy weight (g), \( \rho \). Filler alloy density (g/cm³) and V. Volume of the joint gap

2.3. Brazing
The Furnace brazing process was carried out about 1100°C. The joints were kept under constant brazing temperature for holding time of 10 minutes with Argon gas at flow rate of (1.5 L/min) as show in figure (1) brazing cycle. Under atmosphere the shielding gas was pumped when the temperature reached 300 °C, with a flow rate about (1.5 L/min) continued through the joining process and turned off at 200°C in cooling modes. The furnace heating rate was about (10 °C/min) and cooling rate to room temperature was about (8°C/min) according to the heating cycle that used. Figure 2 Ni-Si-B ternary phase diagram which shows phases expected at equilibrium for different combinations of nickel content and temperature. The brazing temperature can be determined by (T brazing = 0.8 Tm in K°) of melting point of alloy [13].
3. Results and Discussion

3.1. Microstructure of brazed specimens:
It can be noticed in figure 3 (a,b,c & e), that a good metallurgical bond, continuous connection was found in the joint, indicating good wettability. Where the filler metal spreads by capillary action, fills the joint clearance or gap and eliminate potential voids at the interface between the two parts. The capillary force entails an interaction between the base metal and the filler metal; the dissolution of some base metal does occur due to interdiffusion between the filler metal and base metals [14].
Figure 3. Microstructure of similar and dissimilar brazed joints of all specimen.

It is known that a typical diffusion brazing joint consists of four zones as can be shown in figure (4), (I) isothermally solidified zone, (II) athermally solidified zone, (III) bonding affected zone and (IV) base metal zone respectively. The isothermally solidified zone was formed by isothermal solidification during holding brazing time. The athermally solidified zone was directly solidified from residual liquid upon cooling. Further away from the joint centerline, the liquid/solid transition reaction layer is formed between the filler and the base metal. These reactions result from dissolution and inter-diffusion. Consequently, form what is known as the bonding line and diffusion affected zone (DAZ) denoted by (III).

Figure 4. Optical micrograph of AISI 316L bonded to AISI 316L.
3.2. Energy Dispersive Spectroscopy (EDS) Analysis:
The element’s distribution throughout the substrate and the bond region is shown with EDS scan in the centerline as shown in Figure 5.

![Figure 5. Energy dispersive spectroscopy (EDS) line scan for: (a) AISI 316L to AISI 316L, (b) AISI 431 to AISI 431, (c) AISI 316L to AISI 431, (d) AISI 316L to ASTM A516, and (e) AISI 431 to ASTM A516.](image)

3.3. Discussion of Bonding Mechanism:
The Creation of bonding phases at the bond interface of austenitic to austenitic stainless steel, clearly pointing out the bond zone not exceeding 10 microns on the two sides of brazing joint as in figure (6). Also, the effect of bonding diffusion will be moving out to about 100 microns, and the effect of this process on grain boundaries as shown. Figure (7) which represent austenitic to martensitic stainless-steel joint shows the same picture.

Figure (8) explains that the carbon steel side which austenitic differs the other side will differ in structure from the joint that martensitic. The bonding width will be about 10 um while with martensitic it’s about 50 um, because the existence of carbon element will help to present that zone. Figure (9) shows exactly that evidence.

If that can be monitoring the elements movement through brazing zones like, Ni, Cr, Si, B, C, and Fe as in figures (10), (11), (12), (13), and (14), it’s may be revels: The nickel element which it’s at the
filler metal alloy will be goes into the interface almost, because the stainless steels sides can handle it, also Cr element. While Si element will concentrate at the bond zone. Finally, Fe element transfer through brazing zones easy. While carbon and boron participating in limit manner.

Figure 6. SEM of: (a) Austenitic to Austenitic st.st (b) Martensitic to Martensitic st.st.

Figure 7. SEM of austenitic to martensitic stainless steel.
Figure 8. SEM of: (a) Carbon steel to Austenitic st.st (b) Carbon steel to Martensitic st.st.

Figure 9. Bonding phases (BP) for (a) Austenitic to Austenitic st.st, (b) Martensitic to Martensitic st.st, (c) Austenitic to martensitic st.st, and (d) carbon steel to Martensitic st.st.
Figure 10. EDS mapping analyses for AISI 316L to AISI 316L specimen.

Figure 11. EDS mapping analyses for AISI 431 to AISI 431.

Figure 12. EDS mapping analyses for AISI 316L to AISI 431 specimen

Figure 13. EDS mapping analyses for AISI 316L to ASTM A516.
3.4. Layers-by-Layer XRD Characterization of Microstructure:
Because of the limitations of EDS for quantitative analysis, layer-by-layer XRD analysis was performed in order to identify the reaction phases formed on the joint. One Specimen for each experiment after grinding and polishing was prepared for Three XRD inspection; the first along a direction parallel with the interface with 50 um above the joint, denoted by layer I, the second along a direction parallel with the interface between the base metal and bonded zone and 25 um above the joint, denoted by layer II, and the third along a direction parallel to the bonded zone, denoted by III respectively as shown in Figure (15).

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**Figure 14.** EDS mapping analyses for AISI 431 to ASTM A516.

**Figure 15.** Specimen preparation method for XRD inspection.
The x-ray diffraction (XRD) inspections were explained that there are six types of phases that were shown at the bonding zone. These phases are; Ni-Cr-Fe, Cr-Ni-Fe-C, SiC, $Fe_2B$, $Cr_2B$, and Cr-Fe-Ni-Mo.

First layer for the five joints are contain the Ni-Cr-Fe and Cr-Ni-Fe-C as shown in figure (16). The second layer also contain the phase of Ni-Cr-Fe as shown in figure (17), while the bonding phase for austenitic stainless steel bonded to austenitic stainless steel contain five phases Ni-Cr-Fe(Austenite), Cr-Ni-Fe-C(Martensite), SiC, $Cr_2B$, and $Fe_2B$ which means that silicon carbide, boron reacted with chromium and iron to form $Fe_2B$ and $Cr_2B$, as in figure (18a). For joint of martensitic stainless steel bonded to martensitic stainless steel the finale bonding phase is Cr-Fe-Ni-Mo as in figure (18b). For the Austenitic stainless steel bonded to martensitic stainless steel the final bonding phase is containing the $Cr_2B$ B as in figure (18c). The bonding phases for Carbon steel bonded to austenitic stainless steel and carbon steel bonded to martensitic stainless steel containing SiC, and $Fe_2B$ respectively as in figures (18d) and (18e).

**Figure 16.** Layer 1 of XRD for all joints.
Figure 17. Layer 2 of XRD for all joints.
4. Conclusions
The main conclusions from this work are the following:
1. It can be noticed that a good metallurgical bond, continuous connection was found in the samples joint, indicating good wettability.
2. In Austenitic stainless steel brazed to carbon steel and Martensitic stainless steel brazed to carbon steel specimens it can be noticed that the bonding phases in carbon steel side in Austenitic joint will differs in structure from the joint that martensitic.
3. EDS mapping shows that the nickel transfer to stainless steel side more than carbon steel because stainless steel can handle it especially austenitic stainless steel, silicon and boron will remain at filler metal zone and transfers to base metal as well and form nickel silicate and borides, while iron and chromium elements transfers to filler metal zone freely.
4. XRD inspection showed that the Cr-Ni-Fe-C(Martensite) and Ni-Cr-Fe(Austenite) are the most common phases in all five specimen especially in the first two layers and also in the third layers as well which is in the filler zone and that proving of the Cr and Fe elements crossing the filler zone as mentioned early.
5. The silicon carbide, boron reacted with chromium and iron to form Fe2B and Cr2B in final bonding phases of Austenitic st.st brazed to Austenitic st.st, while the final bonding phase of martensitic st.st brazed to martensitic st.st is Cr-Fe-Ni-Mo which contain molybdenum, Cr2B is the bonding phase of Austenitic st.st brazed to martensitic st.st, while Fe2B is the bonding phase for austenitic st.st brazed to carbon steel.
6. The final conclusion is the Ni base containing (4.5% Si and 3.2% B) filler metal can successfully joint all five specimens.

Reference

[1] https://www.franklinbrazing.com/info/brazing-versus-welding/
[2] Xinjian Yuan, Chung Yun Kang, Myung Bok Kim, 2009 Microstructure and XRD analysis of brazing joint for duplex stainless steel using a Ni–Si–B filler metal, *Materials Characterization* 60: 923-931.
[3] Yunxia Chen, Haichao Cui 2018 Effect of Temperature and Hold Time of Induction Brazing on Microstructure and Shear Strength of Martensitic Stainless Steel, *Materials*, 11: 1586.
[4] Ali Ghasemi & Majid Pouranvari, 2017 Microstructural evolution mechanism during brazing of Hastelloy X superalloy using Ni–Si–B filler metal, *Science and Technology of Welding & Joining*, pp. 3.
[5] Saha RK, Wei S, Khan TI. 2005 A comparison of microstructural developments in TLP diffusion bonds made using ODS Ni alloy. *Mater Sci Eng A*, 406. pp. 319–27.
[6] Pouranvari M, Ekrami A, Kokabi AH. 2008 Microstructure development during transient liquid phase bonding of GTD-111 nickel-based superalloy. *J Alloys Compd*, 461: 641–7.
[7] Araf M, Medraj M, Turner DP, Bocher P. 2007 Transient liquid phase bonding of Inconel 718 and Inconel 625 with BNi-2: modeling and experimental investigations. *Mater Sci Eng A*, 447:125–33.
[8] Jalilian F, Jahazi M, Drew RAL. 2006 Microstructural evolution during transient liquid phase bonding of Inconel 617 using Ni–Si–B filler metal. *Mater Sci Eng A*, 423:269–81.
[9] Luozzo ND, Fontana M, Arcondo B. 2007 Transient liquid phase bonding of steel using an Fe–B interlayer. *J Mater Sci*, 42:4044–50.
[10] Ou CL, Shu RK. 2003 Microstructural evolution of brazing 422 stainless steel using the BNi-3 braze alloy. *J Mater Sci*, 38:2337–46.
[11] Araf M, Medraj M, Turner DP, Bocher P. 2007 Effect of alloying elements on the isothermal solidification during TLP bonding of SS 410 and SS 321 using a BNi-2 interlayer. *Mater Chem Phys*, 106:109–19.
[12] *ASM Handbook*, Volume 6, 10th ed. Metals Park, Ohio: American Society for Metals, 2005.
[13] A.E. M. Pouranvari, A.H. Kokabi, 2013 Solidification and solid-state phenomena during TLP bonding of IN718 superalloy using Ni–Si–B ternary filler alloy, *Journal of Alloys and Compounds* 563: 143-149.
[14] X. Wu, R. Chandel and H. LI, 2001 Evaluation of transient liquid phase bonding between nickel-based superalloys, *Journal of Materials Science*, 36: 1539 – 46.