DISSIMILAR VACUUM BRAZING OF ADDITIVE MANUFACTURED 17-4PH STAINLESS STEEL TO CONVENTIONAL 304 AND 17-4 STAINLESS STEEL BY BNI-2 BRAZING FILLER MATERIAL T.Göynük^{*,***}, O.U. Onem^{*}, E.Yasa^{**}, İ. Karakaya ^{***}

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<u>Abstract</u>

Vacuum brazing process of 17-4PH stainless steel, which was manufactured using additive manufacturing, as well as conventional AISI304 and 17-4PH stainless steel alloys was investigated. The brazing process was conducted at 1050°C for 20 minutes under 10⁻⁶ Torr, using BNi-2 filler material. Various aspects of the vacuum brazed parts were analyzed, including their microstructure, wetting behavior and mechanical strength. To evaluate the wetting behavior, the contact angle and wetted area were measured using optical microscopy. The microstructures were examined using Scanning Electron Microscopy and Energy Dispersive Spectroscopy techniques. Additionally, tensile testing was performed on the joints to assess the influence of surface roughness and brazing of different materials on the strength of the brazed parts.

Introduction

Stainless steel alloys are of utmost importance in various industries because of their exceptional mechanical properties, corrosion resistance, and versatility [1]. Two widely recognized alloys within this category are 17-4PH stainless steel and AISI 304 stainless steel, which are highly regarded for their superior performance and diverse applications in aerospace. 17-4PH stainless steel is a precipitation-hardening alloy known for its remarkable strength, excellent corrosion resistance, and favorable mechanical properties even at elevated temperatures [2]. On the other hand, AISI 304 stainless steel belongs to the austenitic family, offering high corrosion resistance, good formability, and excellent weldability [3].

In recent times, additive manufacturing has emerged as a disruptive technology that has revolutionized the manufacturing industry. This innovative manufacturing technique enables the creation of intricate designs and customized components while minimizing material waste and reducing lead times. It provides unparalleled freedom in design and opens up new possibilities for optimizing the performance of stainless-steel alloys [4,5].

Additionally, vacuum brazing has gained significant importance in the process of joining stainless steel components, particularly in environments with high temperatures and vacuum conditions [6]. Vacuum brazing ensures precise and clean bonding by eliminating oxides and impurities, resulting in strong and hermetic joints. It allows for the assembly of complex structures, reduces distortion, and improves the mechanical properties of the brazed parts. When performing vacuum brazing, the choice of filler material is critical to achieve reliable and successful joints. BNi-2, a nickel-based filler material, has become prominent due to its advantageous characteristics, including a high melting point, excellent wetting properties, and compatibility with stainless steel alloys. The use of BNi-2 filler material facilitates the formation of robust metallurgical bonds, ensuring the integrity and durability of the brazed joints [7,8].

Given the significance of these materials and processes, this study aims to investigate the vacuum brazing process of additive manufactured 17-4PH stainless steel, conventional AISI 304 stainless steel, and 17-4PH stainless steel alloys. The examination will focus on various aspects, including microstructure analysis, wetting behavior, permeability, pressure strength, and shear strength of the brazed parts. Through a comprehensive evaluation, this research intends to shed light on the influence of surface roughness, brazing techniques, and the effect of filler material on the performance of brazed components.

Materials and Methods

In this study 20*40*3 mm test coupons were used for wetting and microstructure analysis. Additive manufactured 17-4PH steel test coupons were produced by pulsed mode selective laser melting method. Renishaw AM400 Selective Laser Melting (SLM) machine, which has a maximum size capacity of $250 \times 250 \times 300$ mm was used. This SLM equipment utilizes a Yb-Fiber pulsed-wave laser with a wavelength of 1064 nm and a maximum power of 400 W. The experiments were conducted in an argon environment. To support the structures, a 15 mm thick baseplate made of AISI 1040 steel was used. The production parameters of 17-4 materials manufactured by SLM are shared in Table 1.

Layer Thickness(µm)	Power (W)	Hatching (µm)	Base Plate Temperature (°C)
30	200	110	20

Table 1 Process parameters for 17-4PH stainless steel by Renishaw.

The chemical compositions, surface roughness values, and mechanical properties of all the materials used in this study are provided in the Table 2. The properties of the filler material in paste form are also given in Table 3.

	Chemical Composition (%)								Surface Roughness Ra (um)	Yield Strength (MPa)	Tensile Strength (MPa)		
Materials	Fe	Cr	Ni	Cu	Mn	Si	Nb	С	Р	S	Itu (µIII)	(1011 u)	(1011 u)
Additive Manufactured 17-4 PH, As Built (AM174)	76	15	4	3.9	0.24	0.29	0.33	0.01	0.01	0.02	5.8	871	931
Conventional 17-4PH, Solution Treated (C174)	73	16	4	4	1	1	0.45	0.07	0.04	0.03	1.0	1000	1105
Conventional AISI 304 (C304)	70	19	9	-	1	0.75	-	0.07	0.04	0.03	0.6	205	515

 Table 2 Properties of materials used in study.

 Table 3 Properties of the brazing filler material.

	Chemical Composition (%)										Recommended Brazing			
	Ni	Cr	В	Si	Fe	Р	С	S	Ti	Al	Zr	Со	Temperature (°C)	
BNi-2	82	7	3.2	4.5	3	0.01	0.05	0.01	0.05	0.04	0.03	0.09	1026-1054	

The temperature profile utilized for the brazing process is depicted in Figure 1. The profile can be divided into five distinct steps. Initially, the furnace was gradually heated to approximately 200°C and maintained at that temperature for approximately 15 minutes. This step aimed to eliminate any water-based contaminants and humidity from the system without causing any damage to the filler material. In the second step, the temperature

was raised to 540°C and held for ten minutes to remove organic substances from the surface. The third step involved heating the furnace to 950°C to ensure a uniform temperature distribution across the sample. At the fourth step, the filler material was melted. The final step involved cooling the furnace under a vacuum environment.



Figure 1 Heating cycle of vacuum brazing process for BNi-2.

Wetting analysis was done by optical microscope, while Scanning Electron Microscopy (SEM) together with Energy Dispersive Spectrometry (EDS) was used to evaluate microstructure analysis. The geometry of the butt joint specimen used for mechanical tests and the geometry of the fixture used to align these specimens are shared in Figure 2.



Figure 2 Butt joint and fixture geometry.

Results and Discussion

Wetting Analysis

Wetting, an important criterion in evaluating the quality of brazing, has been examined by observing the spreading behavior of the same amount of BNi-2 paste on AM174, C174, and C304 coupons. The flow characteristics of brazing are influenced by several factors, including interalloying between the braze material and the substrate, diffusion of the braze material, dissolution of grain boundaries, and the formation of intermetallic compounds. These effects can be reduced by careful selection of the braze material, lowering the temperature, shortening the brazing duration, and increasing the cooling rate. Conversely, they can be intensified by choosing a braze material with a high solubility limit in the substrate material or by raising the braze temperature and extending the brazing time [9].

Braze wetting was assessed by measuring the contact angle and wetted area using optical microscopy. Surface flashing, blushing and wetting, were observed by visual inspection as shown in Figure 3. Braze alloys

that exhibit good wetting characteristics spread across the surface with a sharp contact angle at the edge of the molten pool, whereas braze alloys that do not wet tend to form spherical shapes on the surface. Ideally, when a braze wets the surface perfectly, it flashes the surface and forms a thin film with a contact angle close to zero. Blushing, characterized by the separation of braze components, occurs due to a process called liquation, where elements with lower melting temperatures in non-eutectic alloys melt and separate from the braze. These separated elements flow across the surface, leaving behind the higher melting temperature elements. The occurrence of liquation observed in brazing filler material is dependent on whether the alloy is eutectic or not. Despite using the same brazing filler material, it exhibits different spreading characteristics on different materials in this study. This difference can be attributed to various factors such as the metallurgical bond formed with the surface, the presence of contaminants or oxide layers on the surface, surface roughness, and other similar factors.



Figure 3 Wetting characteristics of BNi-2 brazing filler on different materials.

The analysis of braze wetting on AM174, C174 and C304 conducted using a parameter called the Wetting Index (WI). The WI is determined by multiplying the coupon area covered by braze flow with the cosine of the contact angle, as shown in equation (1), where A is the wetting area and θ is denoted for wetting angle. It is an empirical value that depends on the amount of braze material used in wetting tests and serves as a useful metric for comparing the wettability of brazes under identical test conditions. All the results were tabulated in Table 4 including surface roughness, wetting index and wetting characteristics (melting, flashing, blush). While wetting angle was measured during optical microscope, wetting area is calculated by using Image J program.

$$WI = \begin{cases} 0, \ \theta \ge 90^{\circ} \\ A \times \cos \theta, \ \theta < 90^{\circ} \end{cases}$$
(1)

2	Base Metal	Surface Roughness, Ra (µm)	Contact Angle, θ (deg)	Wetting Area (mm ²)	Wetting Index, Area*Cos (θ)	Wetting	Flashing	Blush
3Ni-2	AM174	5.8	0.82	975.2	975.10	\checkmark	\checkmark	
Ι	C174	1.0	2.55	313.5	313.19	\checkmark		
	C304	0.6	1.56	396.0	395.85	\checkmark		\checkmark

Table 4 Wetting analysis of BNi-2 filler material on AM174, C174 and C304.

Due to the nature of the additive manufacturing process, the surface roughness on parts is generally higher compared to conventional materials, despite efforts to reduce it through various process parameters. This difference in surface roughness has been observed to affect the wetting characteristics of brazing filler materials. Excluding conventional 17-4 material from the discussion, it can be observed that as surface roughness increases,

the wetting index also increases. However, it is also considered that at a certain point, excessive surface roughness may have a detrimental effect on the wetting behavior. Generally, roughening a flat surface was found to decrease the ability of a liquid drop to spread and wet the surface, unless the liquid had high inherent wettability, or the surface texture was extremely rough. Because surface roughness created energy barriers that a liquid had to overcome to wet the surface, and wetting was more likely to occur when these barriers were small compared to the enthalpy of the liquid. Surface roughness amplifies the influence of surface energy at the interface between the liquid, atmosphere, and the surface. A rough surface has a higher ratio of actual surface area to apparent surface area, which can enhance wetting when it is energetically favorable, or decrease wetting when it is energetically unfavorable [10]. Even though all materials have undergone the same cleaning procedure, it is considered that the surface of conventional 17-4 PH stainless steel may still retain an oxide layer from the solution treatment process, which is not completely removed. Therefore, it is evaluated that despite having higher surface roughness than AISI 304, the contact angle is higher, and the wetting index is lower for conventional 17-4 PH stainless steel.

The ability of a braze material to flow should not be the sole criterion when selecting a braze alloy because some combinations of base metals and braze alloys do not promote free flow along the surface. When the braze material and base metal undergo interalloying, it can increase the melting temperature of the filler above the brazing temperature, causing the alloy to solidify and impede further flow.

Microstructure Analysis

Brazing is a high-temperature process, when this joining technique is applied between two materials, the microstructure, diffusion, and interalloying between parent metal and filler metal should be thoroughly examined. To examine these properties the following brazing couples have been formed.

- C174 C174
- C174 AM174
- C304 C174
- AM174 AM174
- C304 AM174
- C304 C304

The microstructures of the brazed joints were analyzed using optical and SEM micrographs, with EDX mapping used to assess the elemental composition and measure the segregation of braze elements, agglomeration of precipitates, as well as the diffusion of brazing constituents within the base metals. The optical microscope images and SEM micrographs of these brazing couples have been shared in Figure 4. Optical microscope images, it was observed that darker-colored intermetallics formed within the brazing paste. These intermetallics are also visible in SEM images, exhibiting different distributions and morphologies across different brazing couples. It was determined that the diffusion and the resulting intermetallic formation within the base metal were more prominent in AISI 304 compared to 17-4 PH stainless steel. This enhanced diffusion facilitated stronger metallurgical bonding between the brazing material and the base metal. The diffusion depth in AISI 304 reached up to 30 microns, while it was measured at approximately 15 microns in 17-4 PH material. Both conventionally and additively manufactured 17-4 PH materials exhibited similar behavior in terms of diffusion and microstructure. However, unlike AISI 304, voids were observed at the interface between the additively manufactured 17-4 PH material and the brazing material, which was attributed to surface roughness.

Material Pair	Optical Microscope	Scanning Electron Microscope,600x	Scanning Electron Microscope,1200x
C174-C174		VVD rese MVT 25 mm 0000-1.2025V/ 25 mm METEMANE	3 2.5 mm 1.50 m 1.50 m
C174-AM174			20 100 mm 1 000 100 100 100 100 100 100 1
C304-C174		WG [150] W [157] Sommed de la constance de part Sommed de la constance de la con	₩0 mag 19/ 19/ 19/ 19/ 10 mm
AM174- AM174		1900 reagi MW reagi re	1 100 may 100 100 m 100
C304- AM174		WD [sag] [W] [SW] Sogn 20 yn WT Met	2 97 m 120 200 100 100 100 100 100 100 100 100
C304-C304			

Figure 4 Optical and SEM micrographs of brazing couples.

As part of the elemental analysis, all brazing couples were analyzed through EDS mapping, and the results are presented below. In all joint interfaces, the formation of Cr and Mn carbides or borides within the brazing material was observed. These carbides and/or borides appeared larger and more angular in the presence of AISI 304 material in the brazing couple, whereas they exhibited a more homogeneous distribution throughout the joint region when 17-4 PH material was present. In AISI 304-conventional or additive manufactured 17-4 PH joints, it was observed that the formed carbides generally concentrated towards the AISI 304 material, which had a higher carbon content.



Figure 5 EDS mapping of C304-C304 brazing pair.



Figure 6 EDS mapping of C174-AM174 brazing pair.





Fe

Figure 7 EDS mapping of C174-C174 brazing pair.





Figure 8 EDS mapping of AM174-C304 brazing pair.



Figure 9 EDS mapping of AM174-AM174 brazing pair.



Figure 10 EDS mapping of C304-C174 brazing pair.

Mechanical Tests

The coupons prepared for mechanical testing were joined with BNi-2 brazing paste, following the brazing couples specified in the microstructure analysis. The use of a fixture ensured a fixed tolerance of 50-100 microns between the butt joint parts. All butt joint assemblies were tested at a speed of 1 mm/minute. The tensile stress-strain graph, derived from the raw data of the mechanical tests for the brazing couples, is provided in Figure 11. Additionally, for easier comparison, the complete data graph of the brazing couples is provided in Table 5.

All test specimens ruptured at the brazing interface. It was observed that the elongation was higher in the brazing couples using AISI 304 stainless steel. Particularly, in the 17-4 PH materials produced through additive manufacturing, the elongation was found to be significantly low. It was observed that the gaps at the brazing interface, as seen in the microstructure analysis, contributed to this result. In the brazing couples using 17-4PH

material, the gaps in the interface acted as crack initiation points, resulting in both lower tensile stress and less elongation.

In brazing couples using AISI304 material, higher strength and elongation values were achieved, along with the observed diffusion and metallurgical bonding. According to the mechanical test results, when comparing additive manufactured 17-4 PH material and conventional 17-4PH material, it was observed that the assemblies with conventional 17-4PH material exhibited greater strength and more elongation. This can be attributed to the gaps present at the interface, formed due to surface roughness.



Figure 11 Tensile stress-strain curve for brazing pairs.

 Table 5 Tensile stress-strain values of brazing pairs.

D	•	D	•
Brg	izino	· P	air

Tensile Stress (Mpa) Tensile Strain (%)

C304- AM174	260	0,2
C174-AM174	225	0,1
C174-C174	269	0,1
C304-C174	289	0,5
C304-C304	290	1
AM174-AM174	210	0,1

Conclusion

The aim of this study was to investigate the vacuum brazing process using additive manufactured 17-4 PH material, conventional 17-4 PH material, and conventional AISI304 material. For this purpose, the vacuum brazing process was performed with these materials and material combinations under the same vacuum brazing thermal cycle. In this study, differences were observed through wetting analysis, microstructure analysis, and mechanical testing, and the study was completed. Based on the obtained data, the following conclusions can be made:

- Wetting analysis showed that the wetting index of parts manufactured by additive manufacturing was significantly higher due to the surface roughness. As the surface roughness increased, the wetting tendency of the brazing filler material also increased up to a certain point.
- Although the wetting of the brazing filler material on the surfaces of materials produced by additive manufacturing was high, it was observed that the diffusion within the material was lower compared to AISI 304.
- Microstructure analysis revealed that the BNi-2 filler material diffused more into the conventional AISI 304 material. The high carbon content in the composition of the AISI 304 material resulted in the formation of carbides both within the base material and in the brazing region. When brazing pair with 17-4 PH material and AISI 304 material, these precipitates were observed to be closer to the AISI304 side. In experiments using the AISI304 material, the precipitates exhibited a larger and heterogeneous distribution.
- Surface roughness led to the observation of voids at the brazing interface between the additive manufactured 17-4 PH material and the brazing filler. These voids were found to reduce tensile stress and strain by acting as stress concentration points.
- The conventional 17-4PH material exhibited similar voids in the microstructure of brazing joints due to the oxide layer remaining from the solution treatment on its surface. These voids also had a negative effect on the mechanical strength.

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