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Microstructure Studies of Nickel Base Super Alloy Braze Joints

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***Abstract*: Nickel base super alloys are used for fabrication of hot end components of power plants, automobiles, aero engines etc. These components are realized by welding & brazing techniques. Welding of super alloys becomes difficult when aluminium & titanium content in the alloys is more than 4.5 % as it may lead formation of deleterious brittle phase Ni3 (Al ,Ti) resulting in weld joint cracking. Therefore, brazing operation is well adopted technique wherein difficult to weld material, inaccessible areas for welding and for batch production & repeatability of the result is important. In this work, Nickel base superalloy GTM SU 263 was brazed in a high vacuum furnace using BNi-9 filler material. Specimens were brazed at 1100 °C for 20 minutes and then heat treated at 1080 °C and 800°C for the duration of 3 hours and 8 hours respectively. The effects of heat treatments were investigated on the microstructure and properties of GTM SU263 braze joint. The resultant microstructures were examined using optical and scanning electron microscope. EDAX analysis is performed to understand the diffusion of elements occurring during brazing process. Micro-hardness reading across the brazed joint is studied to correlate the same with braze joint strength.**

***Keywords*: Aero-engines, EDAX, Joint Cracking, Microstructure, Microhardness, Nickel-base superalloy, Vacuum brazing**

1. Introduction

Brazing is a highly attractive form of joining in the aerospace industry, specifically for high-temperature applications in engines. Brazing is a metallic joining process that allows for the joining of both similar and dissimilar metals as well as metals to ceramics and ceramics to ceramics. Brazing is similar to soldering and welding, but there are key differences that set three joining processes apart. Soldering is a mechanical joining process while as brazing and welding are both metallurgical joining processes. Unlike welding, the base metal does not melt during brazing and it occurs at a higher temperature than soldering, above 450 °C. Brazing uses a braze alloy with a lower melting point than the base material that flows into the joint via capillary action when heated to the melting temperature of the braze alloy. The assembly can either be heated locally at the joint or it can be placed in a furnace and heated as a whole [1].

Liquid fuel rocket engines are powered by gas turbines that ingest air from their surrounding environment and compress it. Fuel is added to the air and consecutively burned to produce hot gases, above 750°C, that drive the turbine. These turbines are prone to oxidation, corrosion, creep, high cycle fatigue, and thermal fatigue (2). By using materials that can withstand higher temperatures and the stresses associated with this process, more air is made available for propulsion rather than cooling, resulting in increased efficiency of the engine. Such materials include super alloys joined with advanced braze alloys, providing a superior thermal barrier. One particular joining process is done through active metal brazing (AMB), which removes the need for a metal coating to join dissimilar metals. The manufacturers employ brazing processes on engineering alloys used for the aero engine nozzles and combustion chambers.

There are many methods to heat the assembly to brazing temperatures. The main types include torch, exothermic, induction, and furnace brazing. Torch brazing uses a gas flame as the heat source, typically fuelled by acetylene, propane, or methane. This process is useful for local heating of assemblies but often leaves residual oxides on the surface on the joint. Exothermic brazing uses the heat produced from a solid-state chemical reaction to heat the assembly. This is a quick heating method, but experimentation is required to get the correct feed ratio. Vacuum furnaces are most commonly used in the aerospace industry because they can be used for mass production. The heating rates, temperatures, and times can all be easily controlled, and the brazing can be performed in an inert or vacuum environment [2].

2. Experiment

A. *Base and Braze Materials*

1) *Base-material*

Nickel base super alloys are mainly used in hot end components of aero engines because they can be used at high temperatures while maintaining a significant amount of strength. They also have excellent oxidation and corrosion resistance at elevated temperatures. Nickel and cobalt super alloys can be used up to 1100 °C; however, nickel-base super alloys tend to be higher in strength than cobalt-base super alloys [3]. The base-material used in our study is GTM SU 263 that is a precipitation hardenable, solution treated, nickel-chromium alloy. It has density of 0.297 lb/inch^3 and a melting range of 2160-2449°F. GTM SU 263 is extensively used in aircraft parts and aero-engines for the manufacture of combustor chamber, cone components, jet pipes, ignitor branch, pipe inter-connector, flame tube flange, adopter, deflector, etc. The major problems of GTM SU 263 alloy are micro-fissuring during fusion welding and strain age cracking during post welding heat treatment. Also the mechanical properties and corrosion and oxidation resistance are diminished in the weld and/or heat affected zone because of structural changes. It is precisely because of these difficulties in conventional welding of superalloys; brazing has become very attractive for applications in aerospace industry [4].

Table 1
Nominal Compositions of GTM SU 263 Expressed in Weight Percent

Alloy	*C*r	Co	Mo	Ta	Al	Ti	Ni	*F*e	*M*n	Si	Cu
GTM SU 263	20	20	6.0	1.3	0.6	2.4	Bal	0.7	0.6	0.4	0.2

2) *Braze filler material*

There are many different brazing alloys in production that can be chosen using selection criteria. One criteria is the form of the braze alloy. Braze alloys are found as paste, powder, wires, strips, rods, foils, tapes, or pre-forms in any desired shape. The braze material used in our study is nickel base alloy namely BNi-9, with the nominal compositions given in table 2. Boron is added because it can lower the melting point of the alloy. Additionally, if in low concentrations, boron can improve the wetting of nickel. Boron's distinctive trait is that it diffuses quickly into base metals making it popular for diffusion brazing. Chromium has excellent corrosion and oxidation resistance, which is why it is often added to braze alloys that will be used in high temperature applications [5].

Table 2
% elemental composition of braze alloy BNi9

C	B	Cr	S	P	Al	Fe	Ti	Co	Zr	Se	Ni
0.06	3.25-4.0	13.5-16.5	0.02	0.02	0.05	1.5	0.05	0.10	0.05	0.005	Bal

B. *Brazing of materials*

1) *Brazing operation*

The base material GTM SU 263 is taken as a cylindrical block with 10 mm diameter & 15 mm length in solution treated condition. Brazed specimens were cleaned and braze filler alloy BNi-9 was applied to cover the joining surfaces of the base metal blocks, with a slight overhang. The extra braze alloy helps to ensure full wetting of the joint. All of the test coupons were fixtured and placed in the vacuum brazing furnace. The brazing heat cycles were performed at Thermovac Aerospace Private Limited Bengaluru. During the entirety of the braze cycle, the furnace was kept at low pressure, approximately 10^{-5} torr. After the heating was completed the vacuum was removed and the samples were fan cooled to room temperature. The brazing is performed at 1100°C by using eutectic filler material BNi-9 having liquids temperature of 1050°C. In as-brazed condition the GTM SU 263 is in soft condition.

2) *Heat treatment of brazed specimens*

The brazed specimens are subjected to two heat treatment cycles (i) Heat treatment cycle 1 (HT1): 1080°C for 3 hrs is given to the brazed specimen to give strength to the brazed zone. (ii) Heat treatment cycle 2 (HT2) : Since the base materials GTM Su 263 has become soft after brazing, therefore another set of brazed specimens are subjected to 800°C for 8 hrs heat treatment cycle that corresponds to aging heat treatment cycle of GTM SU 263 to provide strength to GTM SU 263 and also to make comparable study on the microstructures of the braze joints with diffusion heat treated brazed joint.

Visual inspection was conducted after completion of brazing cycles. Indication of the brazing alloy observed around the joint periphery of all the samples, suggesting sufficient flow of the alloy during brazing. For metallographic examination, samples were sectioned normal to the brazed joint. To reveal the joint and base material microstructures, cross sections of the joints are ground, polished, etched with marbles reagent, and examined with optical microscope, scanning electron microscope (SEM) equipped with energy descriptive X-ray analysis. The percentage of key alloying elements in the brazed region of the samples was measured by EDAX analysis. Lastly, the micro hardness values of various phases were measured using Vickers hardness tester, with 25g impression weight.

3. Results and discussion

A. *Optical microscopy*

Fig. 1 illustrates the typical microstructure of the GTM SU 263 braze joint to evaluate the effect of heat treatments given at temperatures of 1080 °C and 800°C for 3hrs and 8 hrs respectively, after the completion of brazing at 1100 °C for 20 minutes. Three zones are observed across the brazed joint: (1) solid solution zone along the base metal; (2) Intermetallics and centreline eutectic constituents in the centre of brazing clearance; (3) diffusion layer in the base metal near the brazing clearance. From fig 1 a, only two zones can be seen in the brazed area. It is because of the less time in brazing, for the elements to activate and form the centreline eutectic zone. However, when the heat treatment of 1080 °C for 3 hours was given, the key elements got activated and separated as eutectic zone shown in fig 1b. When the next heat treatment was carried at 800 °C for 8 hours, the constituents of centreline eutectic got completely diffused into the base metal, as shown in fig 1c.

From fig 2, base material GTM SU 263 can be seen as per vacuum brazed condition, heat treatments 1 & 2. The base metal GTM SU 263 is exhibiting equiaxed structure with well-defined grain boundaries. From figure 2b, twinning in base material GTM SU 263 is observed at heat treatment of 1080 °C for three hours. The twinning decreases (figure 2c) after giving the aging cycle of GTM SU 263.

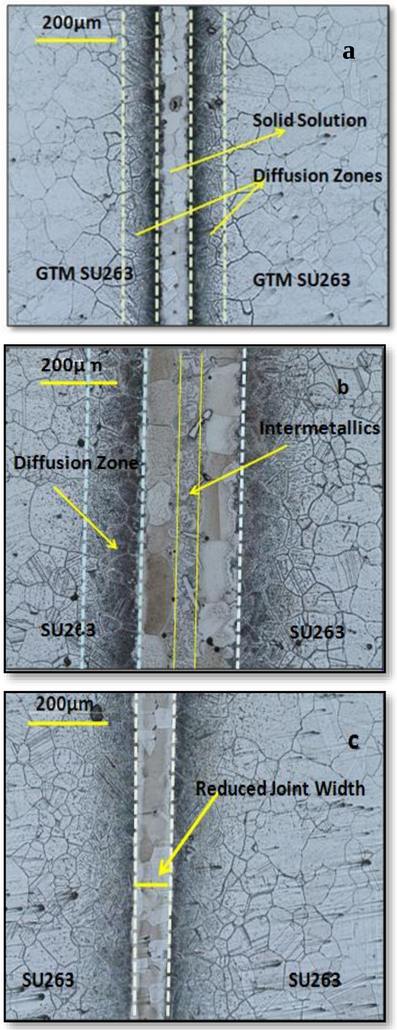


Fig. 1. Interfacial microstructure of the GTM SU 263 joints as per the heat treatments (a) As-brazed (b) Heat Treatment-1 (c) Heat treatment-2

B. *Scanning Electron Microscopy*

The elemental distribution across the joint is analysed on the joint cross-sections with EDAX line scan. It can be seen that main elements appearing in line scan are Ni, Cr, Co, and W derived from the base metal and braze metal. The microstructures of brazed joints are also obtained by SEM operated in secondary electron mode.

Figure 3 shows the SEM microstructure of GTM SU 263 joints for different heat treatments. Microstructure shown by SEM reveals only two zones i.e. solid solution zone and diffusion zone for all the samples, except for heat treatment cycle 1, where centreline eutectic is also found in addition to solid solution zone and diffusion zone.

Table 3 gives the elemental distribution across GTM SU 263 joints of the above three samples taken at different points as marked in the figures. From the EDAX scan, it can be noticed that the content of nickel is higher than any other elements due to nickel base filler and base metal. At the same time, it can be found that the content of nickel in solid solution is higher than

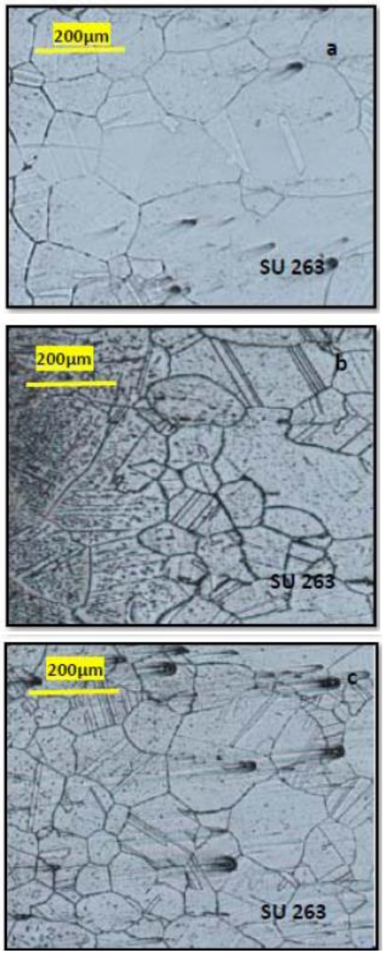


Fig. 2. Interfacial microstructure of the base material GTM SU 263 joints as per the heat treatments (a) As-brazed (b) Heat Treatment-1 (c) Heat treatment-2

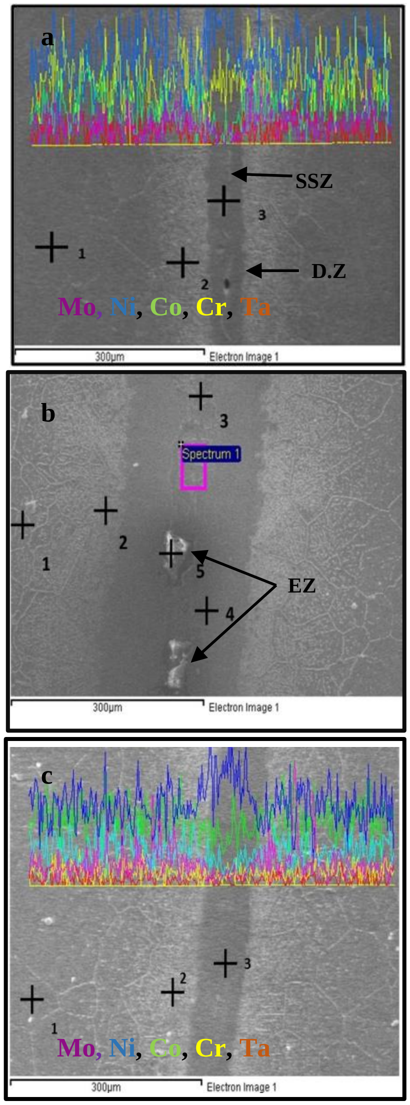
Table 3
Elemental distribution across the SU 263 joints corresponding to the SEM images (a) As Brazed (b) HT-I (c) HT-II

SPOT (a)	Ni	Cr	Co	Al	Ti	Mo	Si	Mn	Fe
1	49.12	20.13	19.34	0.72	2.43	7.39	0.07	0.73	0.13
2	46.69	22.54	17.99	0.5	2.11	8.92	0.16	0.48	0.03
3	74.68	16.58	5.12		0.92	1.17	0.01	0.47	0.18

SPOT (b)	Ni	Cr	Co	Al	Ti	Mo	Si	Mn
1	48.8	20.4	18.13		2.8	8.47	0.3	0.27
2	45.24	22.6	17.65	0.46	2.23	11.3		0.43
3	82.80	12.5	2.95		1.70			
4	74.93	18.1	2.93		1			
5	50.6	25.4		4.08	6.90		13.55	

SPOT (c)	Ni	Cr	Co	Al	Ti	Mo	Si	Mn	Cu
1	48.35	20.68	20.24	0.40	2.33	6.45	0.5	0.66	0.20
2	46.37	20.60	19	0.66	2.17	10.04	0.15	0.44	0.38
3	71.84	16.83	7.28	0.27	0.96	2.20	0.39	0.12	

The adjacent zones. It has been proved that elements both in filler metal and base metal diffuse sufficiently in narrow brazing clearance. Little Intermetallics phases have been formed and much nickel base solid solution has been formed. A chromium runway is seen along the solid solution. This has been also observed by Chunwei MA, Kun SHI, Zhishui YU et al., in their study [6], [7].



SSZ: Solid Solution Zone; DZ: Diffusion Zone; EZ: Eutectic Zone

Fig. 3. SEM Microstructure of GTM SU 263 joints for different heat treatments (a) As-brazed (b) HT-1 (c) HT-2

C. Micro-hardness

Hardness profile is a good indicator of bond microstructure and can be used to assess the effect of secondary phase precipitates on mechanical properties. The Vickers micro hardness (Hv) of each specimen is measured for as-brazed condition and for heat treatment cycles 1 & 2. Micro-hardness values are taken at load of 25 grams for 10 seconds.

Figure 4, shows the micro hardness of GTM SU 263 joints for various heat treatments as samples 1, 2, and 3. The figure exhibits the variation of micro-hardness values in the joint region as a function of distance from the centre. The maximum hardness is found in the eutectics, then diffusion zone followed by base metal GTM SU 263 and least hardness is observed in solid solution zone. The sample underwent heat treatment cycle 1 i.e. 1080 °C for 3 hours shows very hard eutectic precipitates. It is also noticed that the hardness of base material GTM SU 263 got increased by giving heat treatment cycle 2.

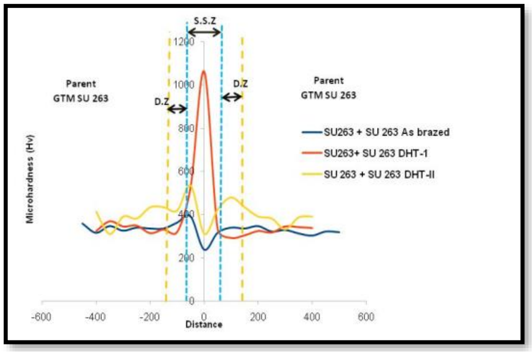


Fig. 4. Variation of micro-hardness for GTM SU 263 brazed joints for sample 1, 2 & 3

4. Conclusion

The heat treatment cycle 1 on the brazed specimen has revealed some hard precipitates of the order of 1024-1074 Hv present in the brazed zone, as the complete diffusion of elements could not occur at this temperature, leaving the brazed joint brittle in nature. However, the heat treatment cycle 2 has yielded eutectic free brazed zone, making joint ductile when compared with the HT cycle 1.

The micro-hardness of brazed samples also confirms the above findings. Micro-hardness of brazed joint from HT1 yields very hard precipitates in the range of 1024-2024 Hv, while as micro-hardness of brazed joint from HT2 yields eutectic free brazed zone with hardness of solid solution zone of the order of 230-250 Hv.

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