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Mechanical Milling of Ni-Cr-B-W Braze Filler Powder for Joining of Aero Engine Materials

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Abstract:

Experiments were carried out in order to investigate the effect of mechanically milled Ni-Cr-B-W braze filler powder on the braze joint microstructure & its properties. The commercially available Ni-Cr-B-W braze filler powder is milled in high energy ball mill system for 10, 20 & 30 minutes with the aim of obtaining less detrimental eutectic phases and enhanced properties of the braze joint. Reduction in powder particle size was analyzed using particle size analyzer followed by brazing of Ni base super alloy with milled braze filler powder. The transient liquid phase bonding of Ni superalloys was conducted at 1100°C for 20 minutes for both the alloys i.e. for GTM- SU-718 & GTM-SU-263 alloy respectively. After joining, the specimens were subjected to post braze heat treatment followed by braze joint analysis by means of OM, SEM and EDS in order to understand microstructure evolution during brazing process. The isothermal solidification process of TLP bonded nickel-based superalloy GTM-SU-718 & GTM-SU-263 alloy was studied. Dendrite formation and solute partitioning governed the microstructure development. Ni-rich solid solution, Ni-rich boride W-rich boride and other carbo-borides were formed in the joint area. The width & micro hardness of isothermal solidified zone, centerline eutectic zone & diffusion zone is analyzed and compared w.r.t. to milling time. Distribution of micro hardness across the joints was studied. Enrichment of refractory elements in the Ni-Cr-W-B interlayer was detrimental to the process of isothermal solidification. Shear strength test was conducted on post braze heat treated specimens. Relationship between milling time, powder particle size, width of the braze zone, micro-hardness & shear strength is established.

Key words: Braze joint, Isothermal solidified zone, Diffusion zone

1. **Introduction**

The development of newer efficient materials for the gas turbines has lead to the need to join the parts of complex geometry with a joint that has homogeneous composition and properties across the joint. Joining of the turbine hot end components can be achieved by welding and brazing. For the components containing substantial gamma prime (γ') or gamma double prime (γ''), welding can cause excessive cracking in the heat affected zone and fusion zone in addition to distortion. Therefore, brazing is the adopted joining technique in the cases where difficult to weld materials, inaccessible areas of welding and batch production and repeatability of results is important [1, 2, 3].

GTM-SU-718 is a precipitation-hardenable nickel-chromium alloy containing significant amounts of iron, niobium, and molybdenum, widely used in liquid fueled rockets, land-based gas turbines, nuclear reactors, pumps, and cryogenic tankage [4]. It is designed to bear exceptionally high yield, tensile and creep-rupture properties at temperatures up to 704 °C [5-7]. GTM-SU-718 can be strengthened by solid-solution strengthening, precipitation hardening, or dispersion hardening mechanism.

GTM-SU-263 is a precipitation hardenable, solution treated, nickel-chromium alloy and 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 [8]. 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 [9, 10]. An attempt has been made to establish the brazing in GTM-SU-718 and GTM-SU-263 alloys.

For repair and regeneration of superalloy components, the conventional high temperature brazing using nickel based braze alloy is used extensively [11-14]. Melting point depressants (MPDs) such as phosphorus, silicon and boron are added to the braze alloy to lower its liquidus temperature and increase the fluidity of the braze filler material. But

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during the cooling stage of brazing process, these elements are incorporated as borides, silicides and phosphides [11, 14]. The presence of intermetallic phases in the centerline of the joint makes the brazed joint hard and brittle and hence reduction in the mechanical properties and creep strength of the joint is observed [14-19]. Also the segregation of melting point depressants into the solidification products reduces the remelting temperature of the joint. The formation of intermetallic phases which tie up the elements that provide corrosion and oxidation resistance such as Cr, Al and Ti makes the brazed joint prone to corrosion and oxidation [11-17].

In the present study an attempt has been made to reduce the width of brazed zone and eliminate the centreline eutectics by using the braze filler metal of different sizes. The braze powders are reduced to different sizes by mechanical alloying.

2. **Experimental procedures**

2.1 Selection of Materials

Brazing can be performed virtually on any metal if the appropriate braze alloy is chosen. Each material system requires specific brazing alloys and brazing procedures to ensure proper joint strength [9]. The base materials chosen for our study are GTM-SU-718 and GTM-SU-263, considering their usage atomizer body & swirler assemblies respectively of combustor chamber of a gas turbine engine [5, 6]. These superalloys contain a variety of alloying elements shown in table 1 and table 2.

Table 1– Nominal Compositions of GTM-SU-263 Expressed in Weight Percent [8]

Elements	Cr	Co	Mo	Ta	Al	Ti	S	W
(Wt %)	19-21	18.5-21	5.6-6.1	1.3	0.3-0.6	1.9-2.4	0.007	0.2
	Fe	Mn	Si	Cu	Ni	C	Nb	
	0.8	0.6	0.4	0.2	Bal	0.04-0.08	0.25	

Table 2– Nominal Compositions of GTM-SU 718 Expressed in Weight Percent [4]

Elements	C	Mn	Si	P	S	Cr	Ni	Nb
(Wt %)	0.02-0.08	0.35	0.35	0.015	0.015	17-21	50-55	4.75-5.50
	Al	Co	Ta	B	Cu	Fe	Mo	Ti
	0.3-0.7	1.0	0.10	0.0028	0.30	Bal	2.80-3.30	0.75-1.15

The filler material used to braze the similar combinations of GTM-SU-718 and GTM-SU-263 was commercially available Ni-Cr-B-W Braze Filler Powder i.e AWS 5.8 BNi-9 powder [28] ; which is a nickel base superalloy with the nominal compositions given in the table 3. AWS 5.8 BNi-9 is a eutectic filler material having liquids temperature of 1050°C.

Table 3– Chemical Composition of Filler Material AWS 5.8 BNi-9 Expressed in Weight Percent

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

2.2 Mechanical milling of braze filler material:

The commercially available braze filler material AWS 5.8 BNi-9 was mechanically milled in high energy ball mill machine (Retsch Make; model-Emax). The braze filler material AWS BNi-9 of weight 100 grams was mechanically

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milled using zirconia balls of 3 mm diameter as grinding medium for 10, 20 and 30 minutes at 1000 rpm . After milling, the powders were analysed in particle size analyser to determine the size reduction in powder particles.

2.3 Brazing of test specimens

The samples were extracted from the cylindrical bars of GTM-SU-718 and GTM-SU-263 of 15 mm diameter. The material was first visually inspected and then taken for non-destructive testing. After being cleared by NDT, the test coupons were cut to the dimensions of 15 mm diameter and 30 mm length. Test specimens were cleaned in ultrasonic cleaner prior to brazing. Mechanically grinded braze filler powders were used to braze the samples of GTM-SU-718 and GTM-SU-263 in high temperature high vacuum furnace (Therelek Make). Brazing for both the alloys was done at 1100 °C for 20 minutes. However, the post braze heat treatment (PBHT) cycle were different. Ageing heat treatment cycle of GTM-SU-263 was given as: Hold at 800 °C for 8 hours (Ramp rate of 10°C/min) then GFQ, while ageing cycle of GTM-SU-718 was given as: Hold at 720 °C for 8 hrs (Ramp rate of 10°C/min) then FC to 620°C (@55 C/hr) and hold at 620°C for 8 hrs then GFQ [8,4] .

Fluorescent penetration inspection was carried on the samples after completion of brazing and PBHT cycles to ensure defect free brazed joints. For metallographic examination samples were sectioned normal to the brazed surface and then mounted. The joints are ground, polished, etched, and examined with optical microscope, scanning electron microscope (SEM) equipped with energy descriptive X-ray analysis. To determine micro-hardness profile of the joint region, the micro-hardness test was carried by using Wilson's hardness testing machine (Model BH 1202), according to ASTM E384. The test was conducted on samples across sections using the load of 100 grams. Room temperature shear test was performed according to the ASTM D1002 to determine the shear strength of the bonds made under different conditions. Shear tests were done on a 50T closed loop servo hydraulic UTM machine.

3. **Results and discussion**

3.1 Particle size analysis of braze filler material

After milling of the braze filler powders in high energy ball mill, the powders were analysed in CAMSIZER X2 (Retsch make) particle size analyser and the results are shown in the table 4 below:

Table 4 Particle size analysis

Q3 AWS 5.8 BNi-9	Initial Powder Size (µm)	10 Min (µm)	20 Min (µm)	30 Min (µm)
10 %	14.5	41.1	13.9	11.8
50%	28.6	21.9	25.4	22.2
90%	215.4	106.3	207.7	51.6
% Reduction		50.69 %	3 %	76.2 %

During high-energy milling, the powder particles are repeatedly flattened, fractured and rejoined (or cold worked or agglomerated) and fractured again with milling time. After milling for a certain length of time, the mechanical milling process reaches the stage where no further reduction in particle size occurs [21]. The average size of the as received commercially available powder was found to be 215.4 µm. After the mechanical milling for 10, 20 and 30 minutes, the average size of 90% of the powder was reduced to 106.3 µm, 207.7 µm and 51.6 µm respectively which is about 50 %, 3% & 76.2 % reduction from the initial size of the powder. The 3 % reduction in 20 minute milling time is attributed to the cold working stage of mechanical milling. It has been observed that with the increase in alloying time, the average size of the particles has reduced substantially.

3.2 Microstructural Evolution

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An ideal brazement shall exhibit the following characteristics: (i) free from intermetallic phases, (ii) free from porosity, (iii) compositional, microstructural and mechanical properties as similar as possible to that of the base metal [1, 22].

The optical microstructures of GTM-SU-263 and GTM-SU-718 brazed with the braze filler powder grinded for 10, 20 and 30 minutes is shown in fig. 1 (a , b & c) and 2 (a , b & c). Three distinct zones are observed across the brazed joint: (i) Solid Solution Zone (SSZ) found along the base metal and contains ɣ-solid solution, (ii) Centerline Eutectic Zone (CEZ) and (iv) Diffusion Zone in the base metal near the brazing clearance. The formation of these regions is attributed to two distinct transformation mechanisms: solidification and solid state precipitation. The solidification occurs by two different modes: isothermal solidification (mode I) and athermal solidification (mode II).

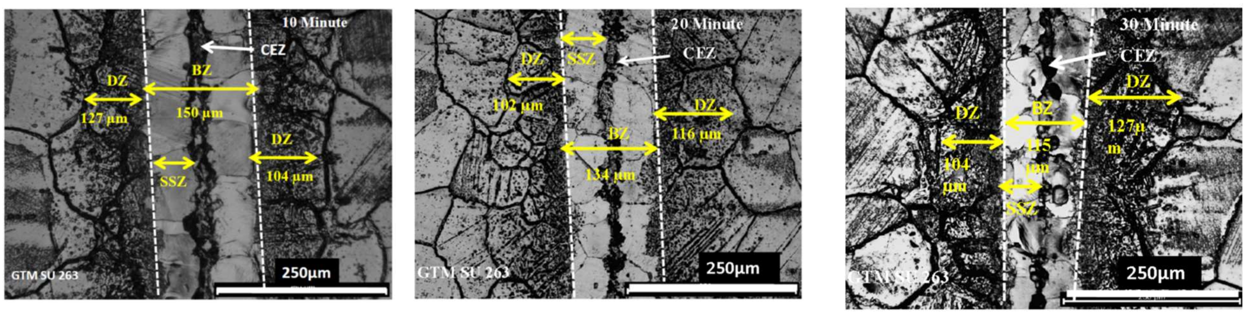

Fig 1. : Microstructure of GTM-SU-263 brazed joints using AWS 5.8 BNi-9 filler metal grinded for a) 10 Min b) 20 Min c) 30 Min

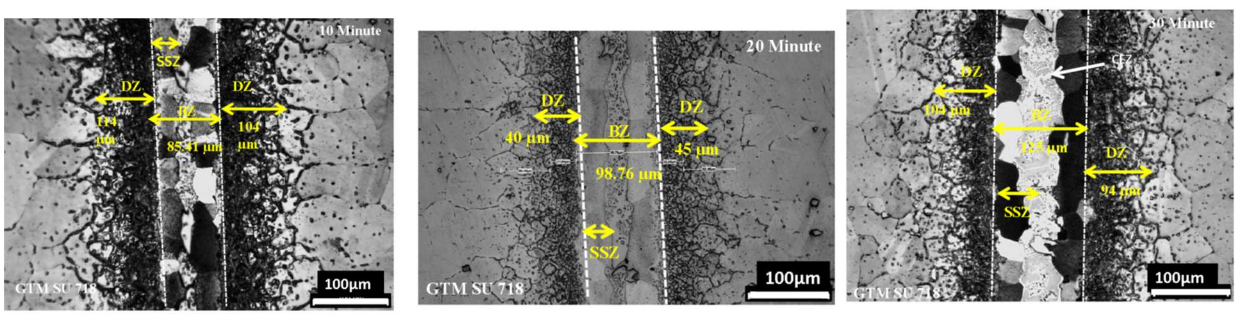

Fig 2. : Microstructure of GTM-SU-718 brazed joints using AWS 5.8 BNi-9 filler metal grinded for i) 10 Min ii) 20 Min iii) 30 Min

The solid solution zone is attributed to mode I of solidification and is formed due to the chemical compositional changes induced by inter-diffusion of alloying elements between the molten interlayer and base metal. This phenomenon increases the liquids temperature of the molten interlayer [23]. The formation of centerline eutectic zone is attributed to mode II of solidification in which the driving force is reduction in temperature. When the bonding time is not sufficient for the completion of isothermal solidification, the residual liquid phase at the centerline of the joint will be solidified athermally during the cooling cycle.

The diffusion zone is formed due to solid state precipitation. The formation of diffusion zone precipitates mainly deals with two interrelated factors: the transportation of boron atoms into the adjacent base metal during the diffusion cycle of brazing and the presence of strong boride former elements such as Cr, W and Ta in the substrate. Idowu et al. [24] have also reported the formation of diffusion zone boride precipitates in a study on the diffusion brazing of IN-738 LC superalloy.

3.3 Effect of braze filler powder milling on width of various zones:

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The effect of milling time on width of the diffusion zone of Su 263 alloy is decreasing & then increasing with milling time of braze filler powder. This is due to increase in braze filler powder particle size at 20 minute milling time. While GT-SU-718 alloy shows continues reverse pattern than GTM-SU-263 in diffusion zone width i.e. first increasing and then decreasing fig. 3. However, centre line eutectic zone (CEZ) is continually decreasing for GTM-SU-263 alloy fig.1. & the same is found to be increasing for GTM-SU-718 alloy fig. 2.

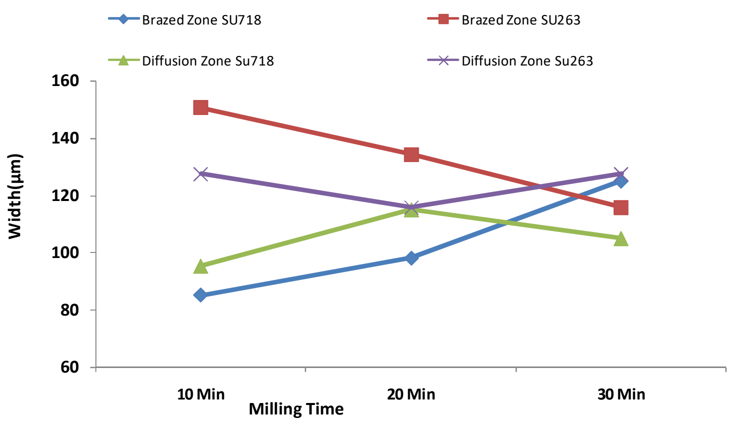
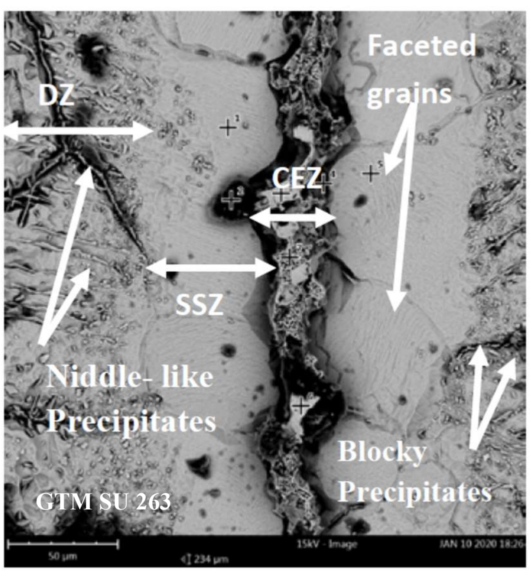


Fig.3 Width of BZ & DZ with milling time

3.4 Elemental Analysis across the joint:

The braze joint for both the alloys was observed under SEM (Phenom make) fig. 3 A & B. The diffusion zone (DZ) shows blocky & needle like precipitates for both the alloys. The Solid solution zone (SSZ) revealed faceted grains. The elemental analysis of DZ, SSZ & CEZ was performed and the EDS spectrum is shown in Fig 1 C, D,E,F,Z.



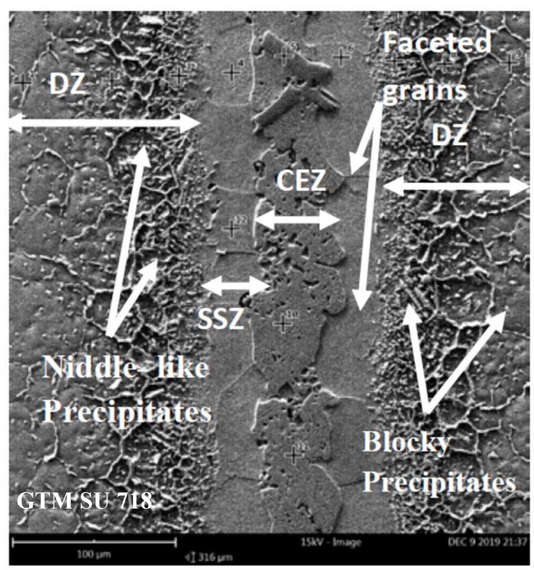


Fig 3. SEM images A) GTM-SU-263 brazed joint using 10 minute milled Braze filler powder & B) GTM-SU-718 brazed joint using 30 Minute milled Braze filler powder

The DZ of GTM-SU-263 reavlead Ni , Cr ,Mo rich prcipitates & Ni, Cr, Fe rich precipaitaes for GTM-SU-718 alloy.Frazam arhami et al [25] found simlilar phases as broton rich precipatates through FE SEM analysis . The ISZ compostion of the both the alloys suggests gamma solid solution.The formation of this phase in SSZ of various Ni base super alloys has been reported by many other researchers as well [26]. While CEZ reavled presence of Cr rich precipitates & Mo rich precicpitaes in GTM-SU-263 alloy and only Cr rich prpecipitates in GTM-SU-718 alloy.

 corresponding author;Email:shweta.gtre@gov.in

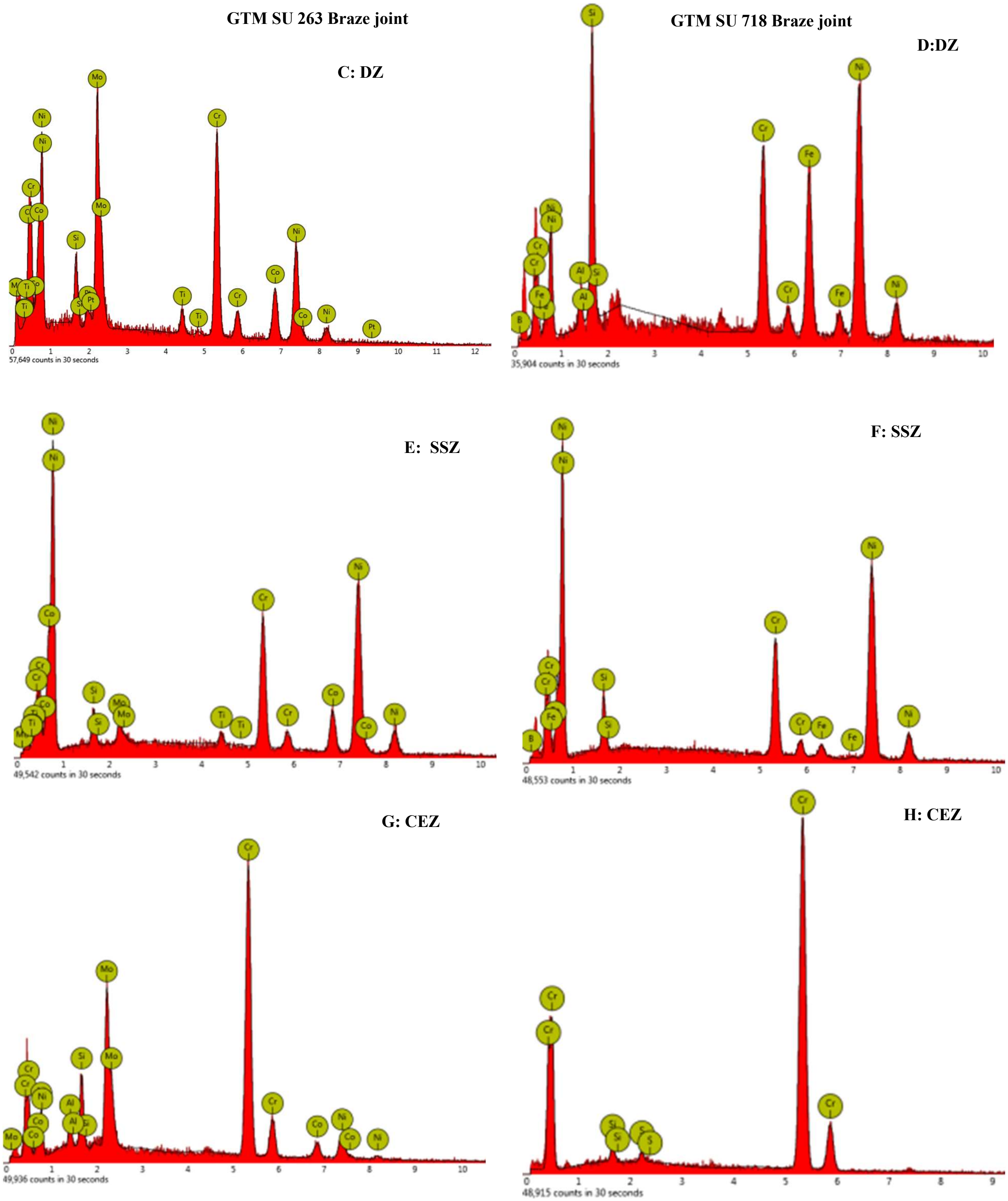


Fig 4. C, E & G are EDS spectrum from image A of   fig.3 for DZ, ISZ & CEZ resp. and D, F, H are EDS spectrum from image B of Fig.3 for DZ, ISZ & CEZ resp.

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Table 5 : SEM EDS analysis of GTM-SU-263 brazed joint using 10 minute milled Braze filler powder

Zone/Element Wt %	Ni	Cr	Co	Mo	Si	Ti	Al
DZ	31.09	24.99	1330	26.10	2.56	1.95	-
SSZ	64.78	17.49	12.05	3.06	1.69	0.93	-
CEZ	11.05	52.88	5.74	25.34	3.59	-	1.40

Table 6: SEM EDS analysis of GTM-SU-718 brazed joint using 30 Minute milled Braze filler powder

Zone / Element Wt %	Ni	Cr	Fe	B	Si	Al
DZ	54.03	13.69	18.89	5.09	7.24	1.07
SSZ	76.10	15.85	2.20	2.96	2.61	-
CEZ	-	97.99	-	-	1.16	Others S=0.85

3.5 Micro hardness study

The micro hardness across the braze joint indicated highest hardness in CEZ and the lowest hardness in SSZ as shown in fig5. The highest hardness in CEZ zone is attributed to the presence of Cr. rich precipitates in CEZ.

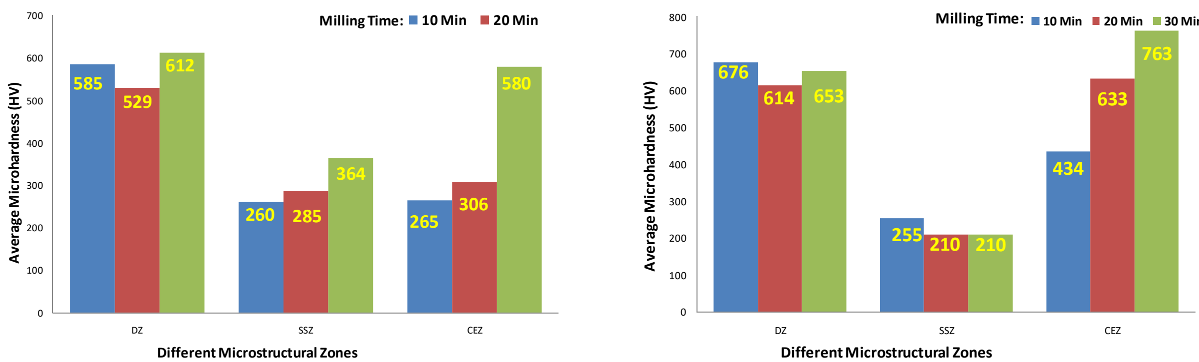

Fig 5: The average micro hardness values (HV) in different microstructural zones for (A) GTM-SU-263 joints & (B) GTM-SU-718 braze joints with different milling time of braze filler powder.

3.6 Shear Strength

The shear strength of all the brazed joints of GTM-SU-718 and GTM-SU 263 accompanied with base metal shear strength is presented in the Figs 5 and 6.

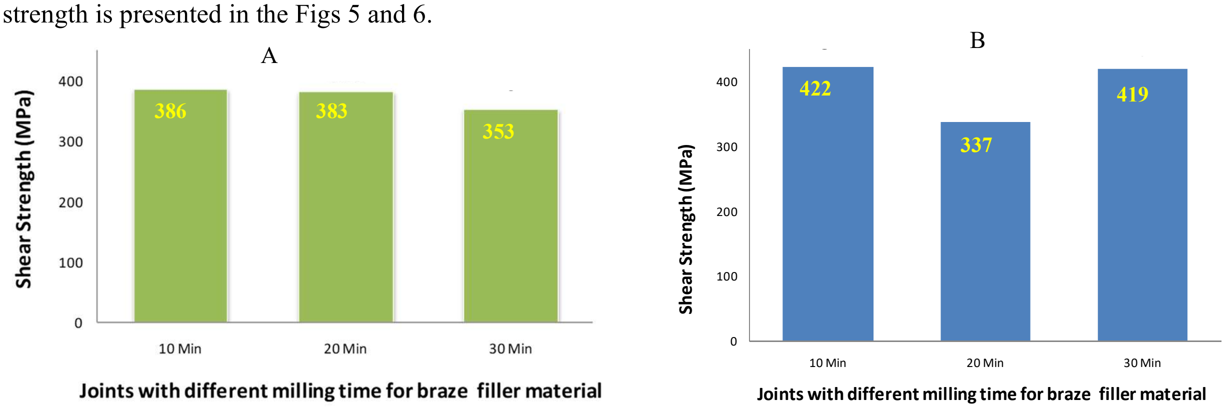

Fig 6: The diagram showing the shear strength of brazed joints of (A) GTM-SU-263 & (B) GTM-SU 718 with different milling time for braze filler material.

It can be seen from the figure 6; the highest shear strength (422 MPa for GTM-SU-718 and 386 MPa for GTM-SU-263) belongs to the joint for which the filler metal is milled for 10 minutes. The other types of joints show relatively lower shear strength when compared with the joint for which the filler metal is milled for 10 minutes.

For GTM-SU-263, the shear strength had decreased slightly with the increase in milling time. However for GTM-SU-718, the shear strength is reduced for only for the joint brazed with filler powder milled for 20 minutes. The joints which are brazed with the filler material milled for 10 and 30 minutes shows almost the same shear strength.

Table7 BNi9 Powder Milling time Vs Shear Strength of GTM –SU-263

Powder Milling time	Powder Particle Size (µm)	Width of DZ(µm)	Width of BZ(µm)	MH at DZ Hv	MH at BZ Hv		Shear Strength(MPa)
					SSZ	CEZ	
10 Min	106	127.31	150.46	585	260	265	386
20 Min	208	115.34	134.25	529	285	306	383
30 Min	52	127.31	115.74	612	364	580	353

GTM-SU-263 alloy braze joint did not show significant change in shear strength when braze filler powder particles are in the size range of 100 to 200 µm. In GTM-SU-263 alloy braze joint high hardness and low strength is observed for low particle size and vice-versa. This can be attributed to harder CEZ which reduces the strength of the joint. It can be noticed that wider BZ has less hard precipitates in the CEZ hence higher shear strength is obtained (Table 7).

Table 8 AWS 5.8 BNi-9 Powder milling time Vs Shear Strength of GTM-SU-718

Powder Milling time	Powder Particle Size (µm)	Width of DZ(µm)	Width of BZ(µm)	MH at DZ	MH at BZ		Shear Strength(MPa)
					SSZ	CEZ	
10 Min	106	95.26	98.76	614	210	633	422
20 Min	208	115	85.41	676	255	464	337
30 Min	52	105	125	653	210	763	419

In GTM-SU-718 alloy braze joint, shear strength & hardness (CEZ of BZ) increases with decrease in powder particle size. At lesser particle size shear strength is high. BZ width & hardness is also high. Wider BZ leads to high hardness & good shear strength property. The width of the brazed zone had increased with the increase in milling time (Table 8). Farzam Arhami et al found increase in shear strength with increase in boding time an a reverse relationship between SSZ width & shear strength for Su-738 alloy [25]. J. Cao et al have found increase in tensile strength properties with increase in holding time during brazing process of Su 718 alloy [27].

4. Conclusions

In this research, a Ni-Cr-B-W interlayer, a nickel based eutectic filler alloy, was used for brazing of superalloys GTM-SU 718 and GTM-SU-263. The braze filler metal was mechanically grinded for 10, 20 and 30 minutes. The brazing was carried out at 1100 °C for 20 minutes for both the superalloys. After the completion of brazing, the bonded samples were post brazing heat treated. Correspondingly, the microstructural evolution during brazing, effect of mechanical grinding of brazes filler superalloy, and the mechanical behavior of the joints was investigated. The following conclusions can be drawn from the above mentioned discussions:

1. Due to inter-diffusion of melting point depressants of the filler alloy and base metal alloying elements, the three main zones formed in the microstructure during the brazing process are solid solution zone, centerline eutectic zone and diffusion zone.
2. Isothermal solidification, athermal solidification and solid state precipitation are the main transformation phenomena involved in formation of the various zones evolved during the brazing process. The solid

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solution zone consisting of single phase gamma solid solution, and diffusion zone, consisting of extensive diffusion induced boride precipitates

3. The hardness of the solid solution zone was lower than that of base metal and centerline eutectic zone due to the insufficient amount of solid solution strengthening alloying elements. The centerline eutectic zone demonstrated the hardest structure in comparison to other microstructural zones.
4. The shear strength of the bonds was inversely governed by the width of brazed joint of all the samples of GTM-SU 263 and GTM-SU 718.

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