Thermal Properties of Ni–Cr–Si–B–Fe Based Interlayer Material and Its Application in TLP Bonding of IN 718 Superalloy

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Abstract

A new Ni–Cr–Si–B–Fe filler material is prepared for transient liquid-phase (TLP) bonding of Inconel 718 superalloy by mechanical alloying technique. The melting temperature range of the filler material and its activation energy of melting are determined by differential scanning calorimetry technique. The activation energy and melting temperature of the alloy powder decrease with increasing milling time. Inconel 718 alloy was joined via TLP by using the newly developed filler material. The effect of TLP bonding temperature and time on microstructural evolution and mechanical properties of the joint was investigated. Three distinct microstructural regions were observed in the bonding area: isothermal solidification zone consisting of a single-phase solid solution, diffusion affected zone consisting of extensive diffusion-induced precipitates of metallic boride, and unaffected base material. The ultimate shear strength and microhardness of the TLP-bonded joint increase with bonding time and temperature.

Keywords Transient liquid-phase (TLP) bonding · Kissinger plot · Mechanical alloying · Inconel 718

1 Introduction

Inconel 718 (IN 718) is precipitation-strengthened nickel-based superalloy suitable for service at temperatures from 650 to 760 °C [1]. As IN 718 is a high-strength alloy (ultimate tensile strength (UTS) up to 1310 MPa) with sluggish aging characteristics, it was originally designed for gas turbine applications [2]. The slow aging characteristics of IN 718 provides a significant amount of metallurgical stability during mechanical fabrication [3]. The excellent mechanical strength is attributed to the strengthening effect due to the precipitation of a unique Ni₃(NbAlTi) phase [5]. In addition to this, the alloy has excellent oxidation and corrosion resistance at elevated temperatures. Unlike other nickel-based superalloys, this alloy has a high resistance to strain age cracking during welding or post-weld heat treatment [4]. However, considering the welding aspects, IN 718 has disadvantages mainly: (1) poor weld penetration

(2) micro-fissuring in the heat-affected zone (HAZ) and (3) poor ductility and impact resistance at the weld fusion zone [6]. The difficulty of obtaining good penetration with root pass during welding of IN 718 is attributable to the poor fluidity of the molten IN 718 superalloy. The root penetration can be improved by any or combination of the following factors: (a) proper shielding gas like helium, (b) preparing U-groove weld geometry, (c) higher root gap and (d) high heat inputs. However, these factors have their own limitations. Using helium as shielding gas increases the manufacturing cost. Preparation of U-groove weld geometry for IN718 is difficult and expensive. High heat input results in burn through problems.

The second disadvantage during welding of IN 718 is the formation of micro-fissures, which are fine cracks, usually around 1 or 2 grains in length, that form in the HAZ of the weld adjacent to the fusion zone. These cracks are inter-granular and seem to form along the boundaries of the partially melted grains close to the fusion zone. The HAZ cracking has been primarily attributed to the constitutional liquation of NbC, and the formation of a low melting laves eutectic in the inter-dendritic regions. These eutectics solidify at a much lower temperature than the solidus temperature of the bulk alloy [7]. Though the micro-fissures can be decreased to a great extent by solution heat treatment

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and low heat inputs, they cannot be completely eliminated. The poor impact and ductility properties of the IN 718 weldments are due to the presence of Lave phases in the inter-dendritic region of the weld metals. The Lave phase is considered to be the main factor for the embrittlement of weldments. This can be overcome to a great extent by high-temperature post-weld solution treatment [8]. However, the full dissolution of the Lave phase is not achieved yet. The facts mentioned above are considered as serious drawbacks of IN 718 components joined by conventional fusion welding techniques, especially for use in critical applications. In order to overcome these problems, the transient liquid-phase bonding process was introduced by a few researchers.

Transient liquid-phase (TLP) bonding is a bonding technique, which eliminates the conventional limitations of the fusion welding process [9]. TLP bonding incorporates the advantageous features of both brazing and diffusion bonding techniques. This bonding process can be applied for the fabrication of robust joints in parts comprising multi-functional structures [10]. This process can be applied to joining a broad spectrum of material viz. superalloy, alloys, ceramics, composites and refractory materials [11]. This technique is highly tolerant to the presence of faying surface oxide layers as a result of which it does not require high pressure [11, 12]. Researchers preferred TLP bonding as a potential joining process for high-temperature gas turbine components, especially superalloys due to its similar bond properties [13], homogeneous microstructure at the bonding area [14], produces robust joint by eliminating centerline eutectics [15] and bond with uniform composition profile. In this bonding technique, a thin filler material constituting at least one melting point depressant (MPD) is sandwiched between the base materials. The bonding process is accomplished at a temperature 20–40 °C above the liquidus point of filler alloy under slight pressure [17]. Bonding occurs by thermally activated inter-diffusion between the base material and the melted filler material. The bond region solidifies isothermally due to a change in concentration of the melt zone [18]. In order to achieve excellent bond quality, the workpiece is required to be maintained at the bonding temperature for sufficient duration for the completion of isothermal solidification (IS) and to eliminate the formation of intermetallic phases [19]. The TLP-bonded sample is held for sufficient time at elevated temperature (below the bonding temperature) in order to achieve homogeneity in chemical composition and microstructure and is generally referred as a homogeneous step in the TLP bonding process [20].

In recent past, several studies have been performed on dissimilar and similar TLP bonding of a wide range of nickel-based superalloys, such as DZ483 [21], Inconel 600 [22], Inconel 617 [9], Inconel 625 [23], Inconel 713C, Inconel 718 [19, 24], Inconel 738 [25] and Inconel 939 [26]. Recently, results of combined analytical [27] and numerical

simulation [28] and experimental investigation on IN 718 and other Ni-based superalloys have also been reported [16]. The reported investigations related to the IN 718 TLP bonding were mainly focused on the microstructure of the bonds made by only BNi-2 or BNi-3 as interlayer materials [15, 16, 26]. There is a need to develop new filler materials for joining, thereby facilitating the components to withstand still higher temperatures. In the current research, for TLP bonding of IN 718, a Ni–Cr–B–Si–Fe nanoparticle-based filler metal has been developed and studied. The effects of bonding temperature and isothermal holding time on the microstructure, shear strength and microhardness of the joints have been investigated and discussed.

2 Experimental

2.1 Synthesis of Powder Interlayer

For better bond quality, the interlayer material should contain major alloying elements of the base alloy and at least one MPD element to lower the melting temperature of the interlayer material. In case of IN 718 superalloy Ni, Cr, and Fe are the major alloying elements and B and Si are taken as MPD material as they have high diffusivity in Ni matrix. Therefore, in this study Ni, Cr, Fe, B and Si are chosen as the elements of the interlayer material. Initial materials for the current investigation were elemental powders of Ni (purity-99.8%), Cr (purity- 99%), B (purity- 99%), Si (purity- 98.5%) and Fe (purity- 95%), obtained from LOBA CHEMIE. The compositions of the powder mixture, in weight percentage, are 80.8%Ni, 10%Cr, 1.5%Fe, 3.2%B and 4.5%Si. The powder mixture was milled using a ball-mill; the details are listed in Table 1.

Milling was carried out up to 60 h. A sampling of powder mixture was carried out after 2, 5, 10, 20, 35, 50 and 60 h of milling. 2 g of powder samples were taken out for each sampling. Thermal analysis of the 60-h milled powder was performed using a differential scanning calorimeter (DSC, Netzsch, STA449F3A00). DSC was carried out from

Table 1 Parameters of ball-milling process

Make/model	Insmart system/PBM07
Vial material	Hardened steel vial
Milling balls	Hardened steel ball
Ball diameter	7.9 mm
Ball: powder weight ratio	10:1
Milling condition	Dry milling
Total weight of the powder mixture	1100 g
Vial speed	300 rpm
Relaxation time after every 45 min	15 min

room temperature to 1200 °C at three different heating rates: 10 °C/min, 15 °C/min and 20 °C/min, in an argon atmosphere. The heat flow vs. temperature data were obtained, from which the activation energy of the melting of the alloy powder was determined using the Kissinger equation. The particle size of the powder was also measured by using a field emission transmission electron microscope (FETEM, JEOL, 2100F).

2.2 Transient Liquid-Phase Bonding

The TLP bonding experiments were carried using an in-house developed TLP setup. Figure 1 shows the schematic of the TLP setup. The setup consists of: (1) a heating chamber with a controlled atmosphere, (2) an impedance heater for heating the samples and isothermal holding at the required bonding temperature, (3) axial loading system and (4) temperature measurement system. The heating chamber made of mild steel tube insulated at the inside with ceramic wool. The output terminal from the transformer of the impedance

heater was connected to the sample using 20-mm-diameter water-jacketed copper electrodes. During the experiment, a continuous argon gas supply was maintained to obtain an inert atmosphere, thereby preventing oxidation of the samples. The samples were heated to the required temperature by passing 300–450 A alternative current. The workpiece temperature was continuously monitored using a K-type thermocouple attached to a digital temperature indicator. The constant axial loading was facilitated using a compression spring and lead screw-nut system. To carry out TLP bonding, an equivalent weight of 1.5 MPa was applied to the sandwiched assembly by the loading system.

A rectangular section of size 14 mm × 5 mm × 5 mm was cut from Inconel 718 alloy sheet using a wire cut electro-discharge machining (W-EDM). The bonding surfaces of the base materials were ground to a surface roughness of 0.6 µm by using 800 grit-sized SiC polishing paper. The polished samples were ultrasonically cleaned in acetone. The nano-sized interlayer alloy powder is mixed with ethanol and made in the form of a paste. This paste is spread on the bonding surface of the samples. The composition of the base metal and filler material is given in Table 2. The prepared samples were carefully assembled and kept on the lower base of the TLP setup. The detail experimental conditions are listed in Table 3. Subsequent to the TLP bonding, the joints were sliced by W-EDM for the preparation of samples for microstructural analysis and mechanical property evaluation. Samples for microstructure studies were prepared, following the standard practice of metallographic specimen preparation. Microstructures of the bonded joints have been examined by field emission scanning electronic microscopy (FESEM, Sigma) attached with an energy-dispersive X-ray

Fig. 1 TLP bonding setup

Table 2 Composition of base metal and interlayer (wt%)

Materials	Ni	Fe	Cr	Nb	Mo	Al	Ti	Ta	B	Si	P	S	Cu	Mn	Co
IN 718	55.7	17.7	16	5.1	2.0	0.5	0.9	0.5	—	0.2	0.05	0.2	0.8	0.2	0.2
Interlayer	80.8	1.5	10	—	—	—	—	—	3.2	4.5	—	—	—	—	—

Table 3 Experimental conditions

Exp. No	Temperature (°C)	Bonding time (min)
01	1050	60
02	1050	90
03	1050	120
04	1100	60
05	1100	90
06	1100	120
07	1150	60
08	1150	90
09	1150	120

Fig. 2 Shear test sample holder

Fig. 3 XRD pattern of initial blended powder and 60-h milled powder

analysis (EDS) system. The bond strength was determined from shear tests conducted by using a dynamic universal testing machine (BISS, Median 250). For carrying the shear test, a sample holder setup, similar to the setup reported by Pouranvari et al. [15], was designed and fabricated. Figure 2 depicts the sample holder setup. Shear testing has been carried out at a constant crosshead velocity of 2 mm/min as per ASTM: D1002 standard. The hardness distribution across the TLP-bonded zones was measured by using a micro-hardness tester (Omni Tech.) with a load of 500 g for 15 s. At each zone, the hardness of five points was determined, and the distribution of hardness is evaluated.

3 Results and Discussion

3.1 XRD Result

X-ray diffraction (XRD) spectra of the Ni–Cr–Si–B–Fe alloyed powders before milling and after milling for 60 h are depicted in Fig. 3. The XRD pattern of the blended powder mixture before milling reveals sharp peaks corresponding to reflections from planes of the constituent elements viz. Ni, Cr, Si, B and Fe. The XRD pattern after 60-h milled powder reveals: (1) complete disappearance of the peaks corresponding to Cr, Si, B and Fe, (2) broadening of the peaks corresponding to the nickel element and (3) peak shifts. Comparison with the data of JCPDS file, the peak at a 2θ value of $\sim 44.5^\circ$ corresponds to the reflections from (111) planes of Ni, Cr and Fe. In the present

powder mixture, since Fe content is very low (1.5 wt%), the contribution of intensity from reflections from (111) plane of Fe is marginal. The sharp peak observed at the 2θ value of $\sim 44.5^\circ$ in the XRD pattern for the initial powder mixture can be the sum of the intensities from the planes of Ni and Cr. The XRD peaks corresponding to reflections from Cr, B and Si were not observed for the 60-h milled powder. The complete disappearance of these peaks indicates the possible dissolution of Cr, Fe, Si and B in Ni powder. The dissolution of these elements in nickel leads to the formation of non-equilibrium, meta-stable FCC solid solution of Ni (Cr, Fe, Si, B). An in-depth analysis of 60-h milled powder revealed peak broadening and peak shift corresponding to the reflections from planes of nickel. The peak broadening observed is a consequence of dislocation density generated in the particle and mean crystallite size reduction on account of the high-energy mechanical alloying process [29, 30]. The peak shift observed after 60 h of milling indicates the lattice strains due to the dissolution of the Cr, Si, B and Fe in the Ni matrix, resulting in the formation of an FCC solid solution of Ni (Cr, Si, B, Fe).

The transformation of the initial powder mixture into a single-phase FCC Ni (Cr, Si, B, Fe) is the consequence of the sequence of the milling process. During the high-energy ball milling, some amount of powder particles is trapped in between the colliding hardened steel balls. The ductile powder particles undergo severe plastic deformation, whereas the brittle particles undergo fracturing. Due to the severe plastic deformation, the ductile particle strain hardens and becomes brittle. The brittle particle during the impact either gets embedded inside the ductile particles and forms a composite powder or remains in the fragmented form. The ductile particles can also get cold-welded. During subsequent milling, powder particles are repeatedly plastically deformed, cold-welded, fractured and re-welded. This stage is marked by a sharp reduction in the particle size [31]. After some period, the powder mixture can no longer be plastically deformed as it loses its ductility because of the prior plastic deformation. The processes dominating during this stage are fracturing and cold welding. A stage will be attained when equilibrium is achieved between the rate of fracturing and cold welding. At this stage, the particle size almost remains constant. The continuous cold welding and fracturing processes result in uniform distribution of the elements at the atomic level, and thereby mechanically alloyed powder is formed. 60 h of milling of the present mixture powder resulted in an FCC structured single-phase Ni (Cr, Fe, Si, B) alloy powder having an average crystallite size and lattice parameters of 40 Å and 3.53 Å, respectively. The obtained X-ray peak profile could be approximated to a Gaussian curve. Multiple Gaussian curves were used to analyze the overlapping peaks. Figure 4 shows a typical Gaussian curve fit in which the black solid line corresponds

Fig. 4 Gaussian curve fitting and peak separation for 60-h milled powder **a** peak 1, **b** peak 2

to the XRD data, the blue solid line corresponds to the sum of the multiple Gaussian profiles, and the violet, green and red solid lines represent the individual Gaussian fits for the multiple peaks. The peak analysis of the individual Gaussian profile confirms small intensity peaks with corresponding 2θ values of 44.3°, 44.4°, 51°, 51.3° and 51.7° as shown in Fig. 4. Comparing these 2θ values with JCPDS files shows formation of Ni, B and Si type eutectic compounds during MA process. BNi_2 , B_3Ni_4 and Ni_2Si type of eutectic constituents are formed during the MA processes.

3.2 TEM Result

Figure 5 illustrates the TEM bright-field image and selective area electron diffraction (SAED) pattern of 60-h milled alloy powders. Analysis of the bright field image revealed an average particle size of 7.3 nm. The ring pattern of the SAED depicts the polycrystalline nature of the alloy. Investigation of the SAED pattern using ImageJ software revealed the d_{hkl} spacing of the alloy powder as 2.05, 1.79, 1.26 and 1.064 Å corresponding to reflection of plane (111), (200), (220) and (311), respectively. As the indices of the different reflective planes are all even or all odd, the diffraction rings are in accordance with the FCC lattice, thereby confirming that the alloy powder is face-centered cubic structure. Analysis of the SAED pattern revealed the lattice parameter of the alloy powder as 3.54 Å, which is in agreement with the XRD data.

3.3 DSC Results

The DSC was carried out to study the thermal properties of the milled alloy powder. The DSC thermograms of alloy powders milled for different time periods are shown in Fig. 6a, b. Up to 20 h of milling, no peaks were observed. The result indicates a gradual exothermic nature till 1016 °C

Fig. 5 Bright-field TEM micrographs and SAED patterns of 60-h milled alloy powder

in the milled powder. This is attributed to the crystallization of the amorphous milled powder during heating. The extensive plastic deformation induced during mechanical alloying produces amorphization of many crystalline materials by introducing a large number of defects [32]. As a result of this, exothermic nature of thermogram is observed until 1016 °C. Figure 6b shows the DSC heat flow versus temperature plot of powder milled for 35 h, 50 h and 60 h. The DSC curves reveal the endothermic peak for powders milled for more than 35 h, approximately at 1026 °C. In order to obtain the peak start, peak maximum and peak end temperatures, the first derivative of the DSC curve was

Fig. 6 DSC thermogram of powders **a** milled up to 20 h showing no peaks and **b** more than 20 h showing endothermic peaks, **c** first derivative of 35-h milled powder, **d** variation of peak temperature with milling time

carried out. The first derivative of the 35-h milled powder DSC thermogram is shown in Fig. 6c. From the first derivative versus temperature curve, the peak start, peak maximum and peak end temperatures for 35-h milled powder were 1019 °C, 1026.3 °C and 1032.3 °C, respectively. Similarly, the peak start, peak maximum and peak end temperatures of 50-h and 60-h milled powder were obtained to be 1014 °C, 1025.495 °C and 1032 °C and 1008 °C, 1023.974 °C and 1031.4 °C, respectively. The endothermic peaks observed after 35 h of milling is due to the melting of the alloyed powders. After 35 h of milling, nano-crystalline non-equilibrium solid solutions form. At elevated temperature, these solid solution melts to form Ni–B–Si ternary eutectic. This results in a lowering of the alloy melting temperature. The points corresponding to the peak start and peak ends are the solidus and liquidus temperatures of the respective alloy mixtures. Increasing milling time increases the amount of eutectic formed during milling, thereby further lowering the melting temperature. The variation of the solidus, peak maximum and liquidus temperature is shown in Fig. 6d. The graph shows that with increasing milling time, the solidus, peak maximum and liquidus temperature of the alloy powder decreases. However, for powders milled for a period below 20 h, the melting temperature is high since proper alloying

of the powder mixture has not taken place. Data obtained from Fig. 7 indicate that as the heating rate increases, the endothermic peak shifts toward higher temperatures. The variation of the peak maximum ($P_{\max}$) with a heating rate is shown in Fig. 7b. This higher side shift is attributed to less time availability at a specific temperature [33].

The activation energy for the powders milled for 35, 50 and 60 h has been calculated using the Kissinger equation [34, 35]:

$$\ln\left(\frac{\varnothing}{T_p^2}\right) = \left(\frac{-E_a}{RT_p}\right) + \ln K_0 \quad (1)$$

where $\varnothing$ is heating rate, T_p is peak transformation temperature, E_a is activation energy of the process, R is universal gas constant and K_0 is constant. From the Kissinger plot, as shown in Fig. 8, the activation energy ($-E_a$) was determined from the slope of the $\ln\left(\frac{\varnothing}{T_p^2}\right)$ and $1/T_p$ plot. The activation energy for the melting of the milled powder reduces with increasing milling time, which is mainly due to the introduction of a large number of imperfections like grain boundaries, sub-grain boundaries, free surfaces, dislocations, etc. during mechanical alloying [36]. Lowering of the activation

Fig. 7 **a** DSC curve of 60-h milled powder heated different heating rates **b** variation of peak maximum with heating temperature rate

Fig. 8 Kissinger plot of **a** 35 h, **b** 50 h, **c** 60-h milled powder **d** variation of activation energy with milling time

energy facilitates inter-diffusion of the atoms between inter-layer material and bond zone, thereby reducing the time required for the bonding of IN 718 alloy.

3.4 FESEM and EDS Results

Microstructures of IN 718 superalloy are shown in Fig. 9. The microstructure comprises a matrix of austenitic “ γ ” solid solution containing a distribution of fine MC carbides, (M refers to Nb and Ti) and precipitates of delta (δ) phase

along the grain boundaries. As the IN 718 alloy consists of 0.02 wt% carbon, finely dispersed of Nb and Ti carbides are noticed in the microstructure as shown in enlarged view (Fig. 9b). The grain size of the base material was determined as per ASTM-E112 standard using the line intercept method. The average grain size determined for IN 718 superalloy was approximately 54.3 μm .

Figure 10 shows the FESEM micrograph of TLP-bonded sample made at 1100 °C and bonding time of 60 min. The microstructure shows three zones, viz., (1) unaffected base

Fig. 9 Micrograph of **a** IN 718 **b** enlarged view of “A” marked part

Fig. 10 TLP-bonded sample made at 1100 °C, 60 min

metal zone (BM), (2) diffusion affected zone (DAZ), and (3) isothermally solidified zone (ISZ). During the bonding process, in the BM zone, the composition remains the same as the base material. Elements of the MPD have not diffused up to this region. In the DAZ zone, metallic precipitates are found inside the grain and along grain boundaries. In the ISZ, different types of second phase labeled as “B”, “C”, “D”, “E” and “F” are observed. Along with second phase, some voids are also evident in the ISZ. The voids are mainly due to air entrapped between the powder particles during the application of the interlayer between base material. The micrograph of different bonding conditions is depicted in Fig. 11. From the micrograph, it was found that with increasing bonding time and temperature, more amount of boron precipitates was observed along the grain boundaries than inside the grain. At lower temperatures and time, bigger second-phase particles were single-phase in the ISZ. This is attributed to the lower diffusivity of boron at a lower temperature in comparison with high temperatures.

The X-ray mapping of TLP-bonded sample is shown in Fig. 12. The mapping indicates the presence of the high amount of Al and Ti along with the interface between the ISZ and the DAZ. This signifies the dissolution of the base metal during TLP bonding. Trace of O and Si was observed at the ISZ. During ball milling, Si fractures into nano-level, which increases the surface energy of the Si particles. These nanoparticles are very active, reacts with oxygen to form silicon oxides, which are very stable particles. As a result of which silicon oxides are observed in the ISZ, and very less amount of Si participates in the diffusion during TLP bonding of IN 718 superalloy. A small amount of Ta, Nb and Al were also observed at ISZ, which confirms the inter-diffusion of the atoms during TLP bonding.

Elemental line scanning for various elements across a TLP bond processed at 1050 °C and holding time of 120 min is shown in Fig. 13. The result indicates significant amounts of B along with Nb and Mo at the grain boundaries in the DAZ. During IS, B diffuses from the interlayer into the DAZ and forms borides. Higher amounts of Al and Ti are evident at the ISZ–DAZ interface regions, as observed by the sharp peaks for these elements. Similarly, the line scan depicts the depletion of Ni, Cr and Fe at ISZ–DAZ interface regions, indicating diffusion of these atoms from the base material to the interlayer. The result also confirms the presence of the higher amount of Si at ISZ, revealing the negligible amount of diffusion of Si from the filler material into the IN 718. This is attributed to the very low diffusivity of Si in Ni compared to B [37].

Composition variation of various elements at different zones of the TLP bond region is illustrated in Fig. 14. The results reveal the high atomic percentage of B at the DAZ and a very low percentage of B at the ISZ, which confirms the diffusion of B from the melted filler material into DAZ. The results also reveal the presence of Ti, Nb and Al at the ISZ. From the results obtained, one can conclude

Fig. 11 FESEM micrographs of TLB-bonded samples made at **a** 1050 °C, 60 min, **b** 1050 °C, 90 min, **c** 1050 °C, 120 min, **d** 1100 °C, 60 min, **e** 1100 °C, 90 min, **f** 1100 °C, 120 min, **g** 1150 °C, 60 min, **h** 1150 °C, 90 min, **i** 1150 °C, 120 min

inter-diffusion of Ti, Nb, Al and B across interlayer and IN 718 alloy during TLP bonding. Figure 15 shows the distribution of different elements obtained by elemental X-ray mapping at DAZ. The figure shows a uniform distribution of elements except Nb, Mo and B in the DAZ. The figure also reveals enrichment of grain boundaries by Nb, Mo and B with concomitant depletion of elements Ni and Fe. From the figure, it is evident that Mo, Nb and B are present at the precipitates in high concentration, which confirms the formation of borides of Mo and Nb. The presence of Si in the DAZ was negligible. As the diffusivity of B ($1.67 \times 10^{-10} \text{ m}^2/\text{s}$) is four orders higher than the diffusivity of Si ($2.11 \times 10^{-14} \text{ m}^2/\text{s}$), B diffused from the interlayer area into the IN 718 whereas the Si particle remained in the ISZ. It appears that silicon can diffuse from the interlayer to the base material only with prolonged exposure time, which is much beyond that required for TLP bonding.

The EDS spectrum of grain boundary phase marked as “A” is also shown in Fig. 15. The analysis revealed the grain boundary phase has 87% B (in at.%) with small amounts of Ni, Nb, Fe, Mo and Cr. It is also observed that the Nb and Mo in the grain are lower than that in the grain

boundary phase. The results confirm the presence borides of Nb and Mo at the boundaries of grains in the DAZ.

The ISZ is formed at the center of the joint due to the compositional variation during the isothermal holding. The ISZ consists of the γ -Ni matrix, along with the presence of Ti, Al, Nb, Mo and Si elements. The presence of some amount of voids (marked as “A” in Fig. 10) has also been observed. Though the initial interlayer material did not contain Ti, Al, Nb and Mo, these elements diffuse from IN 718 superalloy into the interlayer region during TLP bonding. Concomitantly, B and Si diffuse into the IN 718 superalloy. This inter-diffusion of elements is the primary mechanism of IS in TLP bonding. The bond formed at higher bonding time and temperature has a more uniform microstructure than the bond made at lower bonding time and temperature.

The magnified view of the ISZ of TLP bond is shown in Fig. 16. The micrograph shows different phases are present at the ISZ. The composition of various constituents seen in the micrograph analyzed by EDS is depicted in Table 4. The result reveals a higher concentration of Nb, Cr, Ti and Fe in ISZ as compared to the matrix. Also, the amount of Si in all phases is more than the initial concentration of

Fig. 12 X-ray mapping of TLP-bonded sample made at 1050 °C, 120 min

interlayer material. Which signifies the formation of Nb, Cr, Ti and Fe silicide during TLP bonding. During the cooling-induced athermal solidification, as the solubility of Si in the solidifying γ phase is low, Si atoms are rejected to the unsolidified liquid interlayer. Si enrichment in the liquid interlayer causes the solute concentration of the residual liquid to surpass the solubility limit of the γ phase. Therefore, the remaining liquid transforms into eutectic-type hard and brittle silicides phases of Nb, Cr and Fe. In TLP bonding, interdiffusion of atoms takes place between the base material and the interlayer material. In the isothermally solidified zone, different phases are formed due to the interdiffusion of elements. Some phases have a very high concentration of Si (81.2 at.%). EDS analysis of some phases shows Ni silicide and precipitates containing Cr, Nb and Ti. Silicon diffuses from the interlayer to the base material during isothermal solidification. If the isothermal holding time is insufficient, diffusion of complete silicon to the base metal will not take place, resulting in the presence of silicide at the bond zone after the isothermal solidification. The EDS analysis revealed the presence of Ni_3Si at the bond zone. The other phases present at the ISZ due to interdiffusion of elements are $\text{Ti}_{33.9}\text{Al}_{36.5}\text{Cr}_{16.5}\text{Fe}_{0.7}\text{Nb}_{3.7}\text{Si}_{6.3}$, $\text{Cr}_{39.1}\text{Ni}_{22.5}\text{Fe}_{2.5}\text{Nb}_{6.2}\text{Ti}_{16.8}\text{Al}_{2.8}\text{Si}_{10}$ and $\text{Ni}_{18.1}\text{Cr}_{7.9}\text{Fe}_{4.0}\text{Si}_{67.9}\text{Ti}_{0.5}\text{Al}_{1.5}$.

Fig. 13 Distribution of elements along the bond zone (bond made at 1050 °C, 120 min)

In DAZ, almost similar types of phases are formed when bonded by BNi-2 interlayer and present interlayer material. A similar kind of borides of Cr, Nb and Mo was also found in the DAZ of TLP-bonded IN718 joint made by using BNi-2 interlayer material [15, 24]. As boron acts as the main diffusing elements in BNi-2 and present interlayer material,

Fig. 14 Variation of elements along the different zones of TLP-bonded joint: **a** TLP bond made at 1150 °C, 120 min, **b** EDS of spot marked as 1, **c** EDS of spot marked as 2, **d** EDS of spot marked as 3

it reacts with the high boride promoter elements like Nb, Cr and Mo to form borides of respective elements, whereas binary eutectic borides of Ni and Cr, ternary eutectic compound of Ni–B–Si and Ni-silicides observed in the ISZ of TLP-bonded sample made by using BNi-2 interlayer material [15, 38]. However, no borides are evident in the ISZ of the TLP-bonded sample made by using present interlayer material. This is mainly due to lower holding time for complete isothermal solidification and low diffusivity of BNi-2 interlayer elements in IN 718 superalloy.

3.5 Shear Strength

The ultimate shear strength variations of the bonded samples processed at different combination of temperature and time are presented in Fig. 17. The shear stress vs. ram displacement at 1050 °C, 1100 °C and 1150 °C for an isothermal holding time of 90 min are shown in Fig. 18a. When the bonding temperature was increased from 1050 °C to 1150 °C, the ultimate shear strength increases from 381 to 460 MPa. This is attributed to the depletion in the eutectic amount formed during isothermal holding [39]. In

TLP bonding, the joint quality depends upon the degree of inter-diffusion of elements [40]. At higher temperatures, the diffusivity of the MPD is high as compared to lower temperatures leading to enhanced inter-diffusion of atoms between the liquid interlayer and base material. This results in the formation of less eutectic at the bonding zone leading to higher strength at the higher bonding temperature. Figure 18b reveals higher shear strength with an increase in bonding time at a constant bonding temperature. Higher bonding time allows more volume of elements to inter-diffuse between the base and the interlayer material. This results in a homogenous distribution of elements at the bond regions, thereby reducing the amount of eutectic formed by athermal solidification.

3.6 Micro-hardness

Hardness profile in TLP joint is an excellent indicator to evaluate the degree of homogenization achieved during the bonding process. The variation of micro-hardness at the three distinct zones of TLP-bonded samples is shown in Fig. 19a–c. Hardness at ISZ is lower as compared to the

Fig. 15 X-ray mapping of DAZ of TLP bond made at 1100 °C, 120 min

base material hardness. The hardness of ISZ is determined by inter-diffusion of alloying elements between the base material and bond region. The low hardness value of ISZ is attributed to the inadequate diffusion of alloying elements [14, 41] during the bonding process due to the unavailability of sufficient time for completion of IS. The hardness of ISZ, in bonds produced at elevated temperature, is high as compared to bonds formed at a lower temperature, as shown in Fig. 19d. As mentioned in the earlier section, this

is attributed to the higher rate of inter-diffusion of Nb, Mo, Al and Ti, at a higher temperature. The hardness of DAZ is higher than the hardness of BM, due to the enrichment of Mo–Nb borides in this region. During the TLP bonding, the hardness of the BM region is not changed much by the diffusion of MPD elements. The BM zone hardness obtained at higher bonding temperature is slightly lower than that achieved at lower temperatures. The grain size of the BM increases with increasing bonding temperature. The decrease

Fig. 16 Magnified view of ISZ of TLP joint (bond made at 1050 °C, 120 min)

in hardness of BM with an increase in bond temperature is attributed to the grain growth at elevated temperature.

Inconel 718 is precipitation-strengthened nickel-based superalloy, the hardness of the material is influenced by its alloying elements like Cr, Fe, Nb, Ti and Al. The Cr and Fe act as solid solution strengthening elements, and the Nb, Ti and Al act as the precipitation strengthening elements. During TLP bonding Nb, Ti and Al diffuse from the base material into the bond zone. Due to insufficient isothermal solidification time, the amount of elements which has diffused into the ISZ is low compared to base material. As a result, less γ' and γ'' precipitates form, and the hardness of the ISZ decreases.

4 Conclusions

Ni–10%Cr–1.5%Fe–4.5%Si–3.2%B (in wt%)-based interlayer alloy powder synthesized by mechanical alloying process and was used for TLP bonding of IN 718 superalloy. The thermal properties and microstructural characterization of the interlayer material were carried out. The effects of bonding temperature and IS time in the TLP bonding of IN 718 similar joints were studied. The major conclusions of the current research are as follows:

Fig. 17 Ultimate shear strength of the different experimental conditions

1. A non-equilibrium single-phase FCC Ni (Cr, B, Si, Fe) solid solution alloy powder was synthesized by MA of elemental powders for 60 h.

2. The activation energy of milled powder decreases with increasing milling time, which enhances the inter-diffusion rate.

3. The TLP-bonded IN 718 joints exhibited three different zones: DAZ, ISZ and unaffected base material. Due to inter-diffusion of MPD, borides of Nb and Mo were formed in the DAZ.

4. The ISZ hardness was lower than the hardness of BM due to the incomplete diffusion of elements from the BM to ISZ for the time duration under the present investigation.

5. The hardness of the DAZ is higher than the base material due to the formation of Nb, Ti and Mo borides during IS of IN 718 joints.

6. Increasing the bonding time and temperature increases the shear strength and hardness of the TLP-bonded IN 718 joint. At higher temperatures, the diffusivity of B is high, which enhances the diffusion rate, hence a more homogenized bond is formed. With increasing the isothermal holding time, the shear strength and hardness of the joints increase due to the greater progression of IS during this time.

Table 4 EDS analysis results of different phases present in the ISZ (at.%)

Precipitate	Ni	Cr	Fe	Nb	Ti	Al	Si	Mo
B	5.5	7.6	0.8	–	2.5	2.3	81.2	–
C	2.4	16.5	0.7	3.7	33.9	36.5	6.3	–
D	22.5	39.1	2.5	6.2	16.8	2.8	10	–
E	18.1	7.9	4.0	–	0.5	1.5	67.9	–
F	53.4	8.8	5.8	14.3	0.1	0.2	16.8	0.5

Fig. 18 Shear stress variation with **a** temperature and **b** time

Fig. 19 Microhardness variation at different zones **a** Exp. No. 1–3, **b** Exp. No. 4–6, **c** Exp. No. 7–9 **d** variation of hardness with bonding temperature

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