

## MICROSTRUCTURAL EVOLUTION DURING THE TRANSIENT LIQUID-PHASE BONDING OF DISSIMILAR NICKEL-BASED SUPERALLOYS OF IN738LC AND NIMONIC 75

### RAZVOJ MIKROSTRUKTURE MED SPAJANJEM S PREHODNO TEKOČO FAZO NEENAKIH SUPERZLITIN NA OSNOVI NIKLJA IN738LC IN NIMONIC 75

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*Prejem rokopisa – received: 2015-04-01; sprejem za objavo – accepted for publication: 2015-05-08*

doi:10.17222/mit.2015.072

The joining of dissimilar nickel-based superalloys, i.e., IN738LC to Nimonic 75, using transient liquid-phase bonding with a Ni-15Cr-3.5B interlayer (MBF-80) was carried out at temperatures of 1080 °C to 1180 °C for different bonding times of 30–150 min. A joint cross-section was surveyed using optical and scanning electron microscopy. Microstructural examinations showed that after short bonding times, the joint microstructure consists of continuous eutectic intermetallic phases and longer bonding times lead to a eutectic-free microstructure. It was clear that for all the bonding times and temperatures, boride phases were precipitated at the interface of the base metal and the interlayer due to boron diffusion in to the base metals. The results also showed that the morphology of the precipitates in the diffusion-affected zone (DAZ) varies from globular to acicular by increasing the bonding time. Completion of the isothermal solidification, which prevents the formation of continuous intermetallic phases at the joint centerline, was studied at different temperatures.

Keywords: IN738LC, Nimonic 75 superalloy, TLP bonding, isothermal solidification, microstructure

Spajanje različnih superzlitin na osnovi niklja IN738LC na Nimonic 75, z uporabo prehodno tekoče faze z vmesnim slojem Ni-15Cr-3,5B (MBF-80), je bilo izvedeno pri temperaturah od 1080 °C do 1180 °C in pri različnih časih spajanja od 30 min do 150 min. Prečni prerez spoja je bil pregledan s svetlobno in z vrstično elektronsko mikroskopijo. Mikrostrukturne preiskave so pokazale, da pri kratkih časih spajanja mikrostruktura sestoji iz zvezne evtektične intermetalne faze, dolgi časi spajanja pa povzročijo mikrostrukturo brez evtektika. Izkazalo se je, da se pri vseh časih in temperaturah spajanja, boridna faza zaradi difuzije bora izloča na meji z osnovnim materialom v osnovni material. Rezultati so tudi pokazali, da morfologija izločkov v difuzijsko vplivani coni (DAZ) z naraščanjem časa spajanja varira, od globularne do acikularne. Dokončanje izotermnega strjevanja, ki prepreči nastanek zvezne intermetalne faze na liniji spajanja, je bilo proučevano pri različnih temperaturah.

Ključne besede: IN738LC, superzlitina Nimonic 75, TLP spajanje, izotermno strjevanje, mikrostruktura

## 1 INTRODUCTION

IN738LC is one of the most practical casting nickel-based superalloys. IN738LC is strengthened by both solid-solution and precipitation-hardening mechanisms.<sup>1</sup> Due to the presence of coherent  $\gamma'$  precipitates in the alloy with a complex chemical composition and a high stability at high temperatures, the alloy can keep its excellent strength in high-temperature operations and severe conditions, such as gas turbine components. Because of the mentioned properties, the alloy is a good choice for utilization in the first rows of gas-turbine hot sections. Also, this alloy has outstanding strength accompanied by excellent creep, fatigue, oxidation and corrosion resistance at high temperatures.<sup>2-4</sup>

Nimonic 75 is an 80/20 nickel-chromium alloy with controlled additions of titanium and carbon. Nimonic 75 first introduced in the 1940s for turbine blades as sealing components and is now mostly used for sheet applications calling for oxidation and scaling resistance coupled with medium strength at high operating temperatures. It

is still used in gas-turbine engineering and also for industrial thermal processing, furnace components and heat-treatment equipment.<sup>5</sup>

The weldability of superalloys is very much dependent on the amount of Al and Ti in their chemical composition. The high precipitation rate of  $\gamma'$  in alloys with a large amount of Al and Ti make them susceptible to crack propagation during welding.<sup>6-8</sup> Fusion welding, diffusion bonding and brazing are the three main methods of joining and repairing superalloys that are widely used in power plants and aerospace industries. Solid-state diffusion bonding needs high pressure, time and temperature. Therefore, the process encounters a lot of practical and economic limitations, like growth of grains and precipitates during bonding and lengthy processing causes it to be uneconomic.<sup>9</sup> Brazing is the other method of joining that uses an interlayer, containing a melting-point depressant like B, Si and P, as the joining media. The joining temperature must be precisely arranged to melt only the interlayer. Brittle boride, silicide and phosphate continuous phases may be developed in the joint

**Table 1:** Chemical composition of base materials and interlayer in mass fractions, (w%)**Tabela 1:** Kemijska sestava osnovnih materialov in vmesne plasti, v masnih odstotkih (w%)

Alloys	Cr	Co	Al	Ti	W	Mo	Ta	Nb	Fe	C	B	Ni
IN738LC	16.23	8.56	3.41	3.45	3.05	1.73	1.57	0.67	0.08	0.09	0.0063	Bal.
Nimonic 75	20.50	–	0.29	0.55	–	–	–	–	4.78	0.10	–	Bal.
MBF-80	14.89	–	–	–	–	–	–	–	–	–	3.72	Bal.

area during the brazing process and can degrade the joint's properties.<sup>10,11</sup>

Transient liquid-phase (TLP) bonding is a hybrid process of brazing and diffusion bonding.<sup>1</sup> This method possesses the advantages of brazing and diffusion bonding and makes the joint structure very similar to the base material. In contrast to diffusion bonding, the process does not need any pressure due to the use of a liquid interlayer.<sup>12,13</sup> The superiority of TLP in comparison with brazing is the completion of isothermal solidification in the bonding temperature.<sup>10</sup> Also, TLP is widely used in the joining of metals and ceramics.<sup>14</sup>

In the standard models of TLP, there are three individual stages: dissolution, isothermal solidification and solid-state homogenization. Because of the short-range diffusion of melting-point depressant elements in the base metal, the dissolution of the base metal usually needs several minutes. In the isothermal solidification stage, long-term diffusion occurs and therefore the process needs much more time, depending on the bonding temperature and the interlayer type and the time varies from minutes to hours.<sup>15</sup> By optimizing the time and temperature of the bonding, a joint microstructure that is very similar to the base metal with no discontinuity in the microstructure can be achieved.<sup>16</sup>

In this paper, the effect of bonding time and temperature on the microstructure evolution of the transient liquid-phase bonding of IN 738LC and Nimonic 75 using MBF-80 filler metal was investigated.

## 2 MATERIALS AND EXPERIMENTAL PROCEDURES

IN738LC and Nimonic 75 nickel-based superalloys were used as the base metals in this study. Both superalloys were used in the standard solution heat-treated condition. Commercial Ni-Cr-B in the form of amorphous foil (MBF-80) with a 75- $\mu$ m thickness was used as the interlayer. The chemical compositions of the base materials and the interlayer are given in **Table 1**.

TLP bonding test coupons were cut using EMD wirecut to a size of 10 mm  $\times$  10 mm  $\times$  5 mm, then they were grounded with a pendulum grinding machine, in order to remove the recast oxide layer from the faying surface of the coupons, and finally cleaned ultrasonically in a acetone bath. The interlayer was cut with a size of 11 mm  $\times$  6 mm and placed between two faying surfaces of the base alloys. In order to prevent the movement of the samples, the assembly was placed in a fixture without any pressure on it. Green stop-off type 1 was used to

prevent the flowing out of the molten interlayer. The solidus and liquidus temperatures of the interlayer are 1050 °C and 1090 °C, respectively.<sup>17</sup> Therefore, the bonding temperatures were selected above 1050 °C. TLP bonding was performed in a furnace under a vacuum of 10<sup>-5</sup> mbar pressure at different temperatures of (1080, 1120, 1150 and 1180) °C and various holding times of (30, 60, 120 and 150) min. In order to investigate the phases formed during the athermal solidification, one test coupon was bonded at 1120 °C for 5 min. The heat-treatment cycle of the bonding is illustrated in **Figure 1**. The heating rate from 950 °C to the bonding temperature was set to 20 °C/min. In order to show the effect of time on the bond strength, a shear test was carried out on all the samples TLP bonded at 1080 °C.

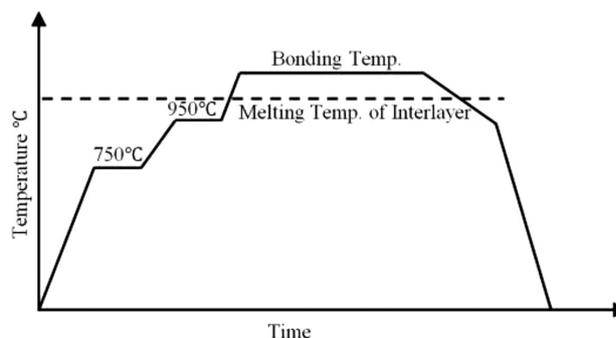
The TLP-bonded test coupons were cut perpendicular to the bonding surface, due to brittleness of the intermetallic phases formed in the bonding centerline, by EDM wirecut and then prepared for metallographic examinations. In order to reveal the microstructure constituent, the samples were etched with Kalling's solution (5 gr CuCl<sub>2</sub> + 100 cc HCl + 100 cc Ethyl alcohol).<sup>18</sup> Metallographic examinations were carried out using optical and scanning electron microscopy.

## 3 RESULTS AND DISCUSSION

### 3.1 Microstructure of the joint

The microstructure of the TLP-bonded specimen at 1120 °C and 5 minutes is shown in **Figure 2**. As can be seen, the bonding area can be divided into six distinct zones:

- Base metal - Nimonic side
- Isothermally Solidified Zone – Nimonic 75 Side (ISZ – Nimonic Side)

**Figure 1:** Heat-treatment cycle of the TLP bonding test coupons**Slika 1:** Potek cikla toplotne obdelave pri TLP spajanju preizkušancev

**Table 2:** EDS analysis (x/%) of phase formed in bond line shown in **Figure 2**

**Tabela 2:** EDS analiza (x/%) faze, ki je nastala na liniji stika, prikazani na **Sliki 2**

	Al	Ti	Cr	Fe	Co	Ni	Nb	Mo	Ta	W
ISZ - Inconel Side	3.12	0.94	19.41	1.23	3.01	70.43	0.64	0.52	0.28	0.41
ISZ - Nimonic Side	0.57	0.46	22.12	4.26	0.35	71.21	0.34	0.40	0.0	0.28
A (Ni-rich Boride)	1.08	3.83	9.18	0.71	2.28	80.70	0.85	0.56	0.80	0.0
B ( $\gamma$ Eutectic)	0.97	0.53	16.27	1.64	2.47	75.84	0.60	0.66	0.30	0.71
C (Cr-rich Boride)	0.41	0.08	89.72	0.15	0.47	5.37	0.46	2.02	0.0	1.31
D (Cr-rich Boride)	0.04	0.05	87.50	0.40	0.69	5.36	1.37	1.97	1.72	0.89
E	0.75	0.36	73.05	2.34	0.0	22.04	0.63	0.44	0.13	0.25
F	0.56	0.84	40.06	4.11	0.03	52.66	0.66	0.52	0.25	0.30

- Athermally Solidified Zone (ASZ)
- Isothermally Solidified Zone – Inconel 738 Side (ISZ – Inconel Side)
- Diffusion Affected Zone – Inconel 738 Side (DAZ – Inconel Side)
- Base metal – Inconel side

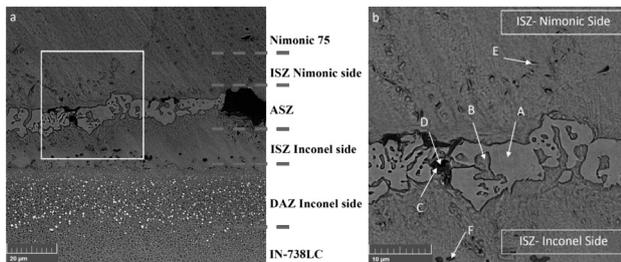
As can be seen in **Figure 2a**, the ISZ zone on both sides of the ASZ contains a thin layer of  $\gamma$  solid solution that formed at the solid/liquid interface during isothermal solidification from the base metal to the joint centerline. The chemical composition of the solid solutions formed on the ISZ – Inconel Side and ISZ - Nimonic Side are shown in **Table 2**. A significant difference between these two compositions is related to the chemical compositions of the Nimonic 75 and the IN738LC. Also, in **Figure 2b**, the different phases that are formed during bonding are specified with A to F.

As the interlayer melts, because the liquid and solid phases are not in the equilibrium condition, base-metal dissolution commences. The diffusion of boron into the base metal and the diffusion of base-metal elements like Cr, Fe, Co, Al and Ti into the molten interlayer leads to the equilibrium condition between the liquid and the solid. A chemical analysis of ISZ shows the diffusion of the base-alloy elements that were not present in the primary interlayer. The alloying elements' diffusion from the base metal to the interlayer and also boron diffusion from the interlayer to the base metal increases the liquidus temperature of the interlayer. The dissolution stage continues until the liquidus temperature of the molten interlayer reaches the bonding temperature, and

then the isothermal solidification starts from the solid/liquid interface to the joint centerline.<sup>2, 19</sup>

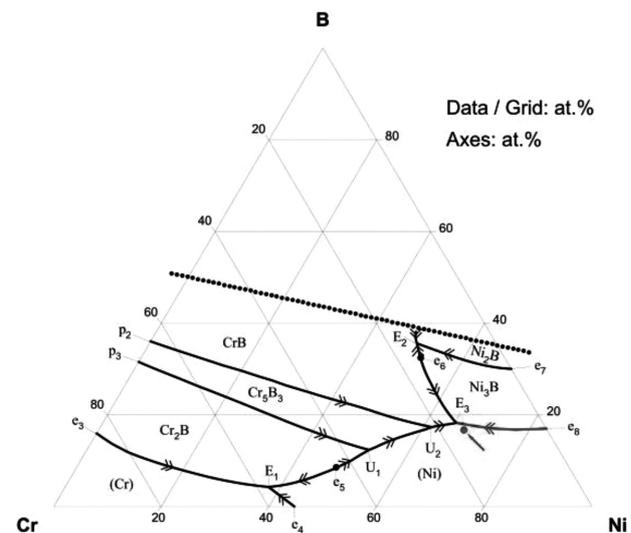
As seen in **Table 2**, the chemical composition of the ISZ regions adjacent to the base alloys (Inconel Side and Nimonic Side) are not similar; therefore, we can conclude that the time required to reach equilibrium on each side are not equal. In contrast with the TLP bonding of two similar alloys, in which the isothermal solidification starts simultaneously and the eutectic centerline forms symmetrically, in dissimilar alloys the TLP bonding eutectic centerline lies closer to the alloy so that its equilibrium starts earlier.

A variation of the chemical composition that is due to the interdiffusion of elements of the base alloy and the interlayer is the main driving force of solidification for the ISZ.<sup>19</sup> Since the solid and liquid are in an equilibrium condition during isothermal solidification, the secondary phases cannot form at this stage.<sup>3,20</sup> The solidification behavior of the remaining liquid during the TLP bonding of pure nickel using the Ni-Cr-B interlayer, before completion of the isothermal solidification, was modeled by K. Ohsasa et al.<sup>21</sup> The ternary phase diagram of Ni-Cr-B, in which the chemical composition of the interlayer is specified, is shown in **Figure 3**.<sup>22</sup> During the cooling of the remaining liquid, which is the main



**Figure 2:** a), b) SEM micrograph of sample bonded at 1120 °C for 5 min

**Slika 2:** a), b) SEM-posnetek vzorca, spajaneja pri temperaturi 1120 °C, 5 min



**Figure 3:** Liquidus projection of ternary phase diagram Ni-Cr-B

**Slika 3:** Projekcija likvidusa v ternarnem faznem diagramu Ni-Cr-B

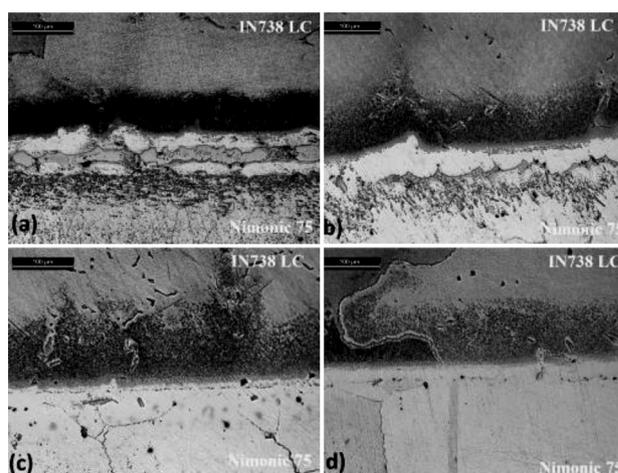
driving force for ASZ solidification,  $\gamma$  dendrite is the first phase that forms at 1100 °C<sup>21</sup> and grows from the solid/liquid interface towards the joint centerline. Solute elements with a partition coefficient less than unity ( $k < 1$ ) are rejected into the liquid. Continuous enrichment of the liquid from the solute elements leads to the solute concentration becoming more than the solubility limit of the solute in the  $\gamma$  and then the secondary solidification constituents are formed between the dendrites (Phases E and F).<sup>20</sup> O. Ojo et al.<sup>23</sup> and A. Egbewande et al.<sup>10</sup> observed the formation of these phases in the TLP bonding of IN738 and IN600, by use of a Ni-Cr-B interlayer, respectively.

During the cooling of the liquid and solid-solution growth additional boron is rejected to the liquid, as a result of poor solubility and the low partition coefficient of boron in Ni (0.3 % of amount fractions and  $\sim 0.008$ ),<sup>24</sup> and then at 1042 °C the solidification path coincides with the eutectic line that separates the  $\gamma$  phase stability region from  $\text{Ni}_3\text{B}$  ( $e_8$  line in **Figure 3**) and the  $\gamma$  solid solution and nickel boride eutectic phases are formed simultaneously through a binary eutectic transformation ( $L \rightarrow \gamma + \text{Ni}_3\text{B}$ ).

More cooling leads to the growth of the  $\gamma$  solid solution and the rejection of boron and the formation of nickel borides lead to a reduction of the Ni concentration and an increment in the Cr in the remaining liquid (Cr solubility in Ni boride is relatively low (10.11 % of amount fractions)).<sup>19</sup> Also, the Cr content of the liquid increases by temperature decrement until at 997 °C the liquid transforms into three phases of  $\gamma$  solid solution, nickel boride and chromium boride through a ternary eutectic transformation ( $L \rightarrow \gamma + \text{Ni}_3\text{B} + \text{CrB}$ ).<sup>21</sup> These phases are shown in **Figure 2b**, and its chemical compositions are shown in **Table 2**.

### 3.2 Effect of time and temperature on the microstructure of the TLP bonds

In order to investigate the effect of time on the microstructure of the TLP bonds, bonding was performed at 1080 °C for 30, 60, 120 and 150 min. It was clear that the thickness of the intermetallic phases decreases with the increasing time (**Figure 4**). The extent of isothermal solidification during TLP bonding depends on the amount and rate of boron diffusion into the base alloys. The thickness of remaining interlayer, which can transform into a continuous eutectic centerline during cooling, decreases for longer bonding times. It was clear that with an increasing time from 30 to 60 min, the thickness of the centerline eutectic decreases. After 120 min there is no continuous eutectic and after a longer time (150 min) there is a negligible amount of intermetallic phases. The results showed that 150 min is not enough for the completion of isothermal solidification at 1080 °C. **Figure 5** shows the ASZ thickness and the shear strength of the bond at different times. It is clear that a dominant

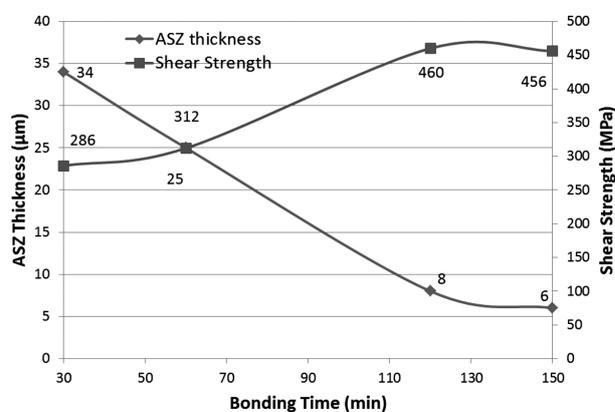


**Figure 4:** Light micrograph of TLP-bonded specimen at 1080 °C: a) 30 min, b) 60 min, c) 120 min, d) 150 min

**Slika 4:** Svetlobni posnetek vzorcev, spajanih s TLP, na temperaturi 1080 °C: a) 30 min, b) 60 min, c) 120 min, d) 150 min

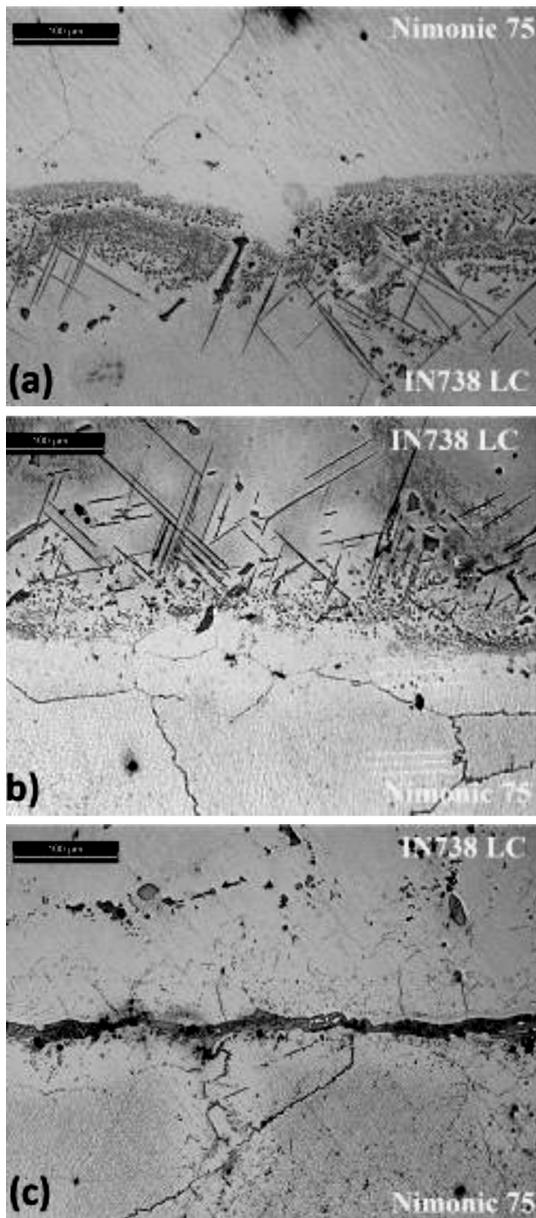
increment in strength can be associated with a decrement of the ASZ thickness. The shear strengths of Nimonic 75 and IN738 at room temperature are 650 MPa and 860 MPa, respectively. The results showed that the strength of the TLP-bonded specimen at 1080 °C is less than the strength of the base metals.

**Figure 6** shows the optical microstructure of TLP bonding at 1120 °C, 1150 °C after 120 min and 1180 °C after 150 min. It was clear that at bonding temperatures of 1120 °C and 1150 °C there was no centerline eutectic micro-constituents, and isothermal solidification was complete. It can be seen that with increasing bonding temperatures from 1120 °C to 1150 °C, significant microstructural changes occurred in the DAZ of IN738, but no changes happened in the bond line. It is expected that increasing the bonding temperature and consequently increasing the diffusion rate of boron, decreases the time required for completing the isothermal solidification. But as shown in **Figure 6c**, at 1180 °C



**Figure 5:** Shear strength and ASZ thickness versus bonding time at 1080 °C

**Slika 5:** Odvisnost strižne trdnosti in debeline ASZ od časa spajanja na temperaturi 1080 °C



**Figure 6:** Light micrograph of TLP-bonded specimens at: a) 1120 °C for 120 min, b) 1150 °C for 120 min, c) 1180 °C for 150 min

**Slika 6:** Svetlobni posnetek vzorcev, spajanih s TLP: a) 1120 °C – 120 min, b) 1150 °C – 120 min, c) 1180 °C – 150 min

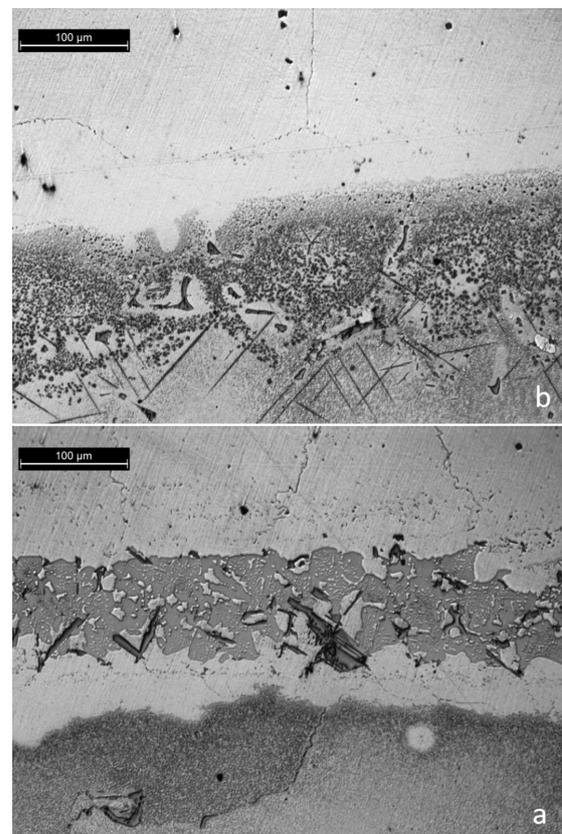
there are significant amounts of centerline eutectic in the bonding after 150 min and isothermal solidification was not completed yet. O. Idouw et al.<sup>25</sup> reported that in TLP bonding of IN738, increasing the bonding temperature from 1170 °C leads to the diffusion of aluminum and titanium into the molten interlayer and a reduction of the isothermal solidification rate may happen. They reported that the diffusion of titanium to the molten layer leads to the formation of a Ni-Ti rich phase, M<sub>2</sub>SC-type sulphocarbide, chromium-rich boride and a  $\gamma$ - $\gamma'$  centerline eutectic. These phases are very stable and hard to solute. Therefore, they can decrease the rate of isothermal

solidification and the completion of the process will be postponed.

### 3.3 Precipitation in the diffusion-affected zone (DAZ)

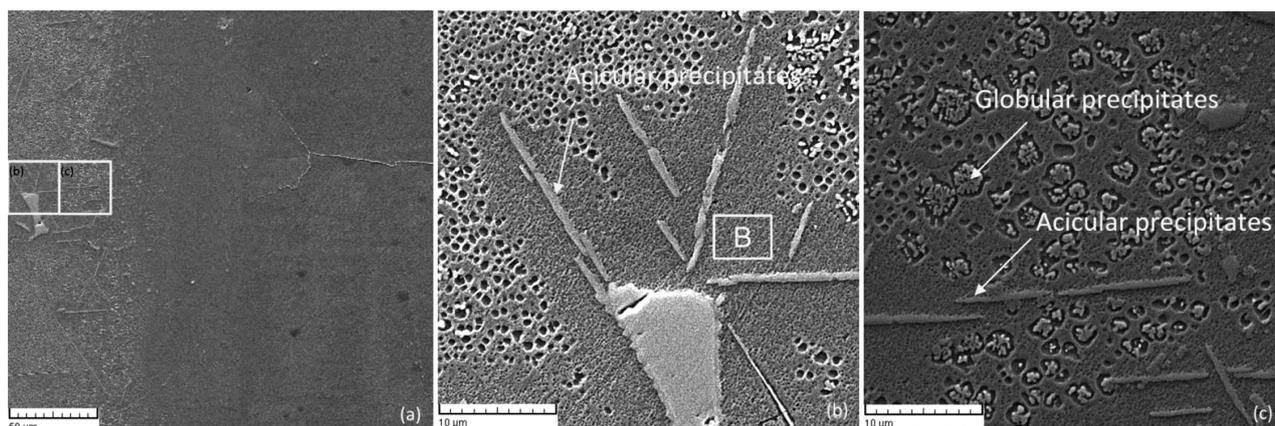
Boride precipitation is expected in the base metal where the concentration of boron is more than its solubility limit in a  $\gamma$  solid solution.<sup>23</sup> The solubility limit of boron in nickel is 0.3 % of amount fractions over the range of 1065–1110 °C, according to the Ni–B binary phase diagram.<sup>26</sup> Therefore, the diffusion of boron into the base metal during the holding at bonding temperature leads to the formation of boride precipitates in the DAZ. These precipitates are shown in **Figures 7** and **8** in two morphologies: globular and acicular.

When the distance between the precipitates and the interface increases the morphology of the precipitates changes from globular to acicular. As mention before, dissolution is the first stage of TLP bonding where the base-alloys solute elements diffuse into the base alloy until the equilibrium condition between solid/liquid is achieved. Therefore, the alloying-elements concentration in the base metal layer adjacent to the bond is poorer than the other layers. As shown in **Table 3**, the amount



**Figure 7:** Variation of precipitates' morphology in DAZ at 1120 °C with time from a) 5 min to b) 120 min. The morphology changes from globular to globular/acicular.

**Slika 7:** Spreminjanje morfologije izločkov v DAZ na temperaturi 1120 °C, v odvisnosti od časa od a) 5 min do b) 120 min. Morfologija se spreminja od globularne do globularno/acikularne.



**Figure 8:** a) SEM micrograph of specimen TLP-bonded at 1150 °C for 150 min, b) and c) specified area in a) showed by b) and c) respectively  
**Slika 8:** SEM-posnetek vzorcev, spajanih s TLP, na temperaturi 1150 C, 150 min, b) in c) določeno področje a) prikazano na b) oz. c)

**Table 3:** EDS analysis of phases shown in Figure 8 (x/%)

**Tabela 3:** EDS analiza faz prikazanih na Sliki 8 (x/%)

	Al	Ti	Cr	Co	Ni	Nb	Mo	Ta	W
Acicular Precipitates	2.25	39.45	7.99	2.81	18.78	7.49	5.07	11.46	4.69
Globular Precipitates	8.44	3.53	38.70	4.06	34.68	0.57	5.28	0.61	4.12
B	3.71	0.46	25.65	6.32	60.61	0	0.60	1.15	1.49

of alloying elements in the precipitates closer to the bonding interface (globular precipitates) is less than the ones further than the bond line (acicular precipitates). As seen in **Table 3**, the high chromium content of the globular precipitates leads to a significant depletion of the chromium around them. Since the Cr is the main element for resistance to corrosion of the base alloy, the depletion of Cr around these precipitates leads to a reduction of the matrix's corrosion resistance.<sup>27</sup> Moreover, a high concentration of  $\gamma'$ -formers in the precipitates leads to the depletion of Al and Ti in the matrix around the precipitates, and as seen in **Figure 8**, there is no  $\gamma'$  particle around them.

The morphology of the precipitates varies by increasing the bonding temperature from the globular to the acicular morphology, and at 1150 °C the acicular morphology is dominant (**Figures 6a** and **6b**). The reason is probably related to more diffusion of the alloying elements at the higher temperature and greater homogeneity of the chemical composition in the DAZ. The other point that can be gained from **Figure 6c** is that the amount of precipitates in the DAZ decreases by raising the temperature, such that at 1180 °C there is no acicular and globular precipitate. It can be concluded that these precipitates may be dissolved at temperatures lower than 1180 °C.

**Figure 7** shows that at 1120 °C, the morphology of the precipitates varies from globular to globular/acicular by increasing the holding time from 5 to 120 min. This phenomenon is related to more diffusion of boron into the base alloy at longer times and implies that the formation of acicular precipitates need more alloying elements than globular precipitates (**Table 3**).

**Figure 8** shows the bond interface in a specimen TLP bonded at 1120 °C for 150 min. Boron was detected in all precipitates, but their accurate concentration could not be reported quantitatively due to X-ray absorption of the EDS analyzer window. The compositional analyses shown in **Table 3** suggests that globular precipitates are borides rich in Ni, Cr, Mo, W and Al and acicular precipitates are borides rich in Ti, Ta, Mo, Nb and W. O. Ojo et al.<sup>22</sup> and O. Idowu et al.<sup>24</sup> observed boride precipitates rich in Cr, W and Mo in TLP bonding of IN738 by a Ni-Cr-B interlayer at 1130 °C. Also, it can be seen that there are lots of very fine  $\gamma'$  around acicular precipitates. The reason is related to the absorption of Ti by these precipitates so that the concentration of the  $\gamma'$  former element is less than normal. Since the Nimonic 75 does not have any boride formers in its chemical composition, no precipitate was observed in its diffusion-affected zone. **Figures 8b** and **8c** show the different zones specified in **Figure 8a**.

#### 4 CONCLUSION

The effect of time and temperature on the TLP bonding of the dissimilar nickel-based super alloys IN738LC and Nimonic 75 using an MBF-80 interlayer was investigated. The following conclusions can be drawn from this study:

- Before completion of the isothermal solidification, the microstructure of the joint centerline consists of three eutectic phases:  $\gamma$  solid solution, Ni-rich boride and Cr-rich boride.
- DAZ precipitates are formed due to boron diffusion from the interlayer to the base metal during a

solid-state transformation. Boride precipitates were observed in the DAZ at bonding temperatures up to 1150 °C. However, at a bonding temperature of 1180 °C, the boride precipitates were not observed.

- At a given bonding temperature, the morphology of the DAZ precipitates changes from globular to globular-acicular with an increasing bonding time.
- Boride precipitation in Nimonic 75 DAZ did not occur due to a lack of boride-forming elements in its chemical composition.
- A reduction in the isothermal solidification rate was observed when TLP bonding at 1180 °C.

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