DIFFUSION BRAZING OF A NICKEL BASED superalloy
PART 3 – EFFECT OF POST BOND HEAT TREATMENT

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Abstract
Microstructure of an isothermally solidified transient liquid phase (TLP) bonded joints of GTD-111 superalloy almost consists of nickel rich solid solution in joint centerline plus significant Cr-boride precipitates in the diffusion affected zone. Considering lack of sufficient γ' precipitation which is vital for high temperature performance of superalloy and the presence of the large amount of Cr-rich borides in DAZ which reduce local corrosion resistance of the base alloy, there is a need to design a proper post bond heat treatment (PBHT) to homogenize the bond. In this part of the work the effect of PBHT on the microstructure and mechanical properties of an isothermally solidified TLP bonded GTD-111 was studied. Joints with complete isothermally solidified microstructure were homogenized at 1150°C for 240 min. Shear strength of bonds after PBHT exhibited a microstructure almost free of undesired boride precipitates with significant presence of γ' phase across the joint. Also, shear strength of bonds after PBHT was close to that of the base metal.

Key words: Transient liquid phase bonding; Nickel-base superalloy; Post bond heat treatment

Introduction
Transient liquid phase (TLP) bonding is considered as a preferred repairing/joining process for nickel base superalloys due to its ability to produce near-ideal joints [1]. A typical microstructure of TLP bonded joint of a nickel based precipitation hardened superalloy such as GTD-111 using a boron containing interlayer, consists of three distinct microstructural zones, before completion of isothermal solidification [2]:

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Athermally Solidified Zone (ASZ) which usually consists of eutectic microconstituents. This zone is formed due to insufficient time for isothermal solidification completion. Cooling is the main driving force for athermal solidification (i.e. non-isothermal solidification).

Isothermally Solidified Zone (ISZ) which usually consists of a solid solution phase. Compositional change induced by interdiffusion between substrate and interlayer during holding at a constant bonding temperature is the driving force for isothermal solidification. As a result of the absence of solute rejection at the solid/liquid interface during isothermal solidification under equilibrium, formation of second phase is basically prevented [3].

Diffusion Affected Zone (DAZ) which consists of boride precipitates due to B diffusion into the base metal (BM) during TLP bonding.

The presence of different zones of TLP bonded joint is schematically shown in Fig. 1.

Fig. 1 Schematic representation of various microstructural zones in a TLP bonded γ′ strengthened nickel based superalloys before isothermal solidification completion

Although some researchers [3-6] worked on TLP bonding of γ′ strengthened nickel base superalloys, but their works was focused on microstructural control of joint centerline via control of process parameter (e.g. bonding time, bonding temperature and joint gap) to achieve a eutectic free joint. There is a little emphasis on the effect of bonding variables on DAZ precipitates. Also, the attempts to clarify the effect of post bond heat treatment (PBHT) on the microstructure and properties of TLP bonded nickel based superalloys were rare. The aim of this paper is to investigate and analyze the effects of PBHT on microstructural features and mechanical properties of TLP bonded GTD-111 nickel base superalloy.

Experimental Procedure

Standard heat treated GTD-111 nickel based superalloy was used as the base metal in this investigation. A commercial Ni-4.5Si-3.2B alloy (MBF30), in the form of amorphous foil with 25.4 µm thickness was used as the interlayer. Details of bonding procedure are given in Part 1 [7]. As stated in this paper, isothermal solidification was completed after 75 min holding at 1100°C. To investigate the effect of PBHT, joints with complete isothermally solidified microstructure were homogenized at 1150°C for 240 min in an argon gas atmosphere (%99.999 Ar) using a tunnel furnace.
Microstructure of the joints was studied using optical microscope and scanning electron microscope (SEM). An etchant consisting of 0.5g MoO$_3$, 50ml HCl, 50ml HNO$_3$, 200ml H$_2$O, which preferentially etches $\gamma'$ phase, was used to indicate $\gamma$-$\gamma'$ microstructure of the joints, in addition to joint centerline microstructure. Thereafter, hardness characteristics and shear strength of the joint were measured. The details of hardness test and shear test were previously described in Part 2 [8].

Results and Discussion

Need for PBHT

As previously described in Parts 1 and 2 [7, 8], isothermal solidification which prevent formation of hard eutectic structure in the joint centerline, was completed after 75 min of holding at 1100$^\circ$C (Fig. 2). As documented [8] in isothermally solidified bonds, shear strength of about 68% of that of the base metal was obtained. In this condition, there are two dissimilarities between microstructure of the isothermally solidified zone and base metal.

(i) Lack of significant amount of $\gamma'$ phase in ISZ: As can be seen from Fig.2, there is no significant amount of $\gamma'$ phase within the ISZ of bond made at 1100$^\circ$C for 75 min, but the presence of some fine $\gamma'$ phase at the joint/base metal interface may be detected. This can be related to the low content of Al + Ti in the ISZ which could affect the $\gamma'$ phase stability. Presence of these elements is a key important factor for high temperature performance of the joint. According to EDS analysis the average contents of Al and Ti in the ISZ of isothermally solidified bond are 3.7 and 3.65 at.%. Since the
high temperature performance of precipitation hardened nickel based superalloy widely depends on the γ’ volume fraction; there is a need to design a proper PBHT to formation sufficient γ’ in bond region.

(ii) Presence of borides in DAZ: As mentioned in the previous sections, isothermal solidification prevents the formation of eutectic structure in the bond region; however significant Cr-rich borides were still present in the DAZ. Despite the fact that the precipitates in DAZ have less detrimental effect on the joint shear strength due to their non-continuously distributed fashion along the joint/base metal interface, the high chromium content of DAZ precipitates, can lead to a significant depletion of chromium around this region, which may result in a decrease in the corrosion resistance.

Therefore, considering the lack of sufficient γ’ precipitation in ISZ and the presence of the large amount precipitates in DAZ, there is a need to design a proper PBHT to homogenize the bond. To pursue this purpose, isothermally solidified joints were homogenized at 1150°C in an argon atmosphere using a tunnel furnace. The holding time was 240 min.

**Effect of PBHT**

Microstructure of bond made at 1100 °C for 75 min and then homogenized at 1150 °C for 240 min is shown in Fig.3. As can be seen, volume fraction of secondary phase precipitates in the DAZ has been significantly reduced. Also, γ’ phase was formed in the bond region (Fig.3). This can be related to more diffusion of Ti and Al from base metal into the bond region.

![Fig.3 a) Optical micrograph showing microstructure of isothermally solidified bond after PBHT, b) SEM micrograph showing an enlarged area of a.](image)

The hardness profile of bonds made at this bonding condition is shown in Fig.4a. As discussed in part 2 [8], the hardness profile of an unhomogenized isothermally solidified bond exhibits a soft region in joint centerline due to lack of significant amount of γ’ phase in ISZ and a peak in hardness in DAZ due to carboride formation. In comparison to unhomogenized isothermally solidified bond, the hardness profile across the bond region is significantly affected by PBHT. The hardness of the post bond heat treated isothermally solidified (i. e. homogenized) bond centerline is greater than the unhomogenized isothermally solidified bond. The hardness of bond region depends on the size and volume fraction of γ’ precipitates and the concentration of solid solution.
elements. This can be related to the formation of significant γ’ at the bond region due to the diffusion of Al and Ti from the base metal into the bond region during PBHT. Average contents of Al, Ti and Co in the joint region of homogenized bond measured by EDS, were 6.1, 6.4 and 5.3at% respectively which are higher than the corresponding values of, 3.7, 3.65 and 4.3at%, for unhomogenized isothermally solidified bonds.

![Fig.4 a) Hardness profile and b) SEM microstructure of bonds made at 1100°C for 75 min and subjected to PBHT (i.e. homogenized)](image)

The applied PBHT removed carbo-boride precipitates from DAZ causing significant decrease in the hardness of this region. However, there is a narrow soft zone within the base metal adjacent to the joint interface (see Fig.4a). As can be seen from Fig.4b, a thin layer of γ-γ’ microstructure exists in the base metal adjacent to the interface which differs from base metal microstructure with respect to the γ’ size. Lower hardness of this zone can be attributed to γ’ coarsening.

The γ’ coarsening adjacent to the bond region is also reported by Schnell [9] in TLP bonding of single crystal nickel base superalloy CMSX-4 using a Ni-Cr-Co-Al-Ta-B filler alloy, after aging of an isothermally solidified bond at 870°C for 20 h. It is interesting to note that γ’ coarsening is not only due to the PBHT. Coarsened γ’ was also observed in DAZ of unhomogenized joint before completion of isothermal solidification. For example, Fig.4a shows coarsened γ’ in DAZ of bonds made at 1100°C for 30 min. This diagram indicates that Cr-rich carboborides in DAZ are encased by coarse γ’ precipitates. It should be noted that the γ’ coarsening was only observed in the region near joint region but they are not observed in the regions further away from the joint interface. Furthermore, γ’ coarsening was not observed when a GTD-111 superalloy specimen was subjected to the same heat treatment schedule that was used for the TLP bonding operation, but without the presence of the interlayer. Therefore, it can be deduced that coarsening of γ’ occurs during TLP bonding by a mechanism which is differs from isothermal coarsening (i.e. long thermal exposure). Comparison of γ’ in DAZ of Fig.5a with γ’ in the base metal of Fig.4b indicates that boron diffusion induced precipitates in DAZ can significantly change the morphology and size of γ’. This figure indicates that Cr-rich carbo boride in DAZ are encased by
coarse $\gamma'$. Results of Iduwu et al. [10] and Liu et al. [11] also show that size and morphology of $\gamma'$ phases surrounding the boride precipitates in diffusion zone are changed. Fig. 5b shows microstructure of $\gamma'$ coarsening zone of PBHT joint.

Fig. 5 SEM micrographs showing microstructure of base superalloy region near the bond region for bonds made at a) 1100°C for 30 min b) 1100°C for 75 min followed by PBHT at 1150°C for 240 min

As can be seen from Fig. 5b after thermal cycle of PBHT, the carborubide phases were dissolved and $\gamma'$ precipitates again undergo a change in their morphology and size resulting observed coarsened $\gamma'$ precipitates at the base metal adjacent to the joint region. According to these observations, $\gamma'$ coarsening can be related partly to the formation of carborubides in DAZ and partly to the experienced thermal cycle of PBHT. Despite the explanations given in this section, further study is needed in order to establish a better understanding of exact mechanisms for this $\gamma'$ coarsening during TLP bonding.

The shear test results of bonds made at 1100°C for 75 min (before and after PBHT) and the base metal are shown in Fig. 6. These results show that PBHT joints have higher shear strength (702 MPa) than isothermally solidified bond, which is about 90% of that of the base metal shear strength. This can be attributed to the increase in $\gamma'$ volume fraction of bond region during PBHT. Considering the softening effect of $\gamma'$ coarsening zone, there is a need to design a proper solution/aging treatment for the homogenization of the microstructure across the joint. Further study is needed for achieving a joint with the similar microstructure to that of the base metal.
Conclusion

The effects of post bond heat treatment on the microstructure and mechanical properties of an isothermally solidified bonded GTD-111 nickel based superalloy were investigated. From this research the following conclusions can be drawn:

1- Homogenization of isothermally solidified bond at 1150°C for 240 min was successful in (i) significant reduction in boride precipitates within the diffusion affected zone and (ii) formation of significant γ' precipitates in the bond region.

2- After PBHT, significant γ' phase, formed within the bond region, increased the bond shear strength. The shear strength of the homogenized bond was about 90% of that of the base metal.

3- The hardness profile of TLP joint became more uniform after post bond heat treatment, due to the formation of significant amount of γ' precipitates in the bond region and the removal of carboboride precipitates from DAZ. A soft region was observed adjacent to the joint interface, due to γ' coarsening in this region, which may contribute to the reduction of joint shear strength.

References

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