DIFFUSION BRAZING OF A NICKEL BASED SUPERALLOY PART 4: EFFECT OF BONDING TEMPERATURE

Majid Pouranvari*

Department of Materials and Metallurgical Engineering, Dezful Branch, Islamic Azad University, Dezful, Iran

Received 20.05.2011
Accepted 15.09.2011

Abstract
The effect of bonding temperature on microstructure of a transient liquid phase (TLP) bonded GTD-111 nickel base superalloy, using a Ni-Si-B interlayer, was investigated. Bonding was carried out at 1100, 1150 and 1180°C for various bonding times. At bonding temperatures of 1100 and 1150°C, the microstructure of the joint centerline is controlled by B diffusion. However, at bonding temperature of 1180°C, the effect of base metal alloying elements on the joint microstructure development was more pronounced. It was found that contrary to general expectation, above a critical temperature, the time required for isothermal solidification completion was increased.

Key words: TLP bonding; Superalloy; Microstructure; Isothermal solidification

Introduction
The development of more efficient gas turbine engines has led to the need to join geometrically complex parts with a joint that has a homogeneous composition profile and, thus, homogeneous mechanical properties across the joint. 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].

In series of papers [2-4], the behavior of GTD-111 nickel based superalloy during TLP bonding or so called diffusion brazing was reported. In consideration of TLP bonding for commercial applications an important bonding parameter is the holding time required to complete isothermal solidification ($t_{IS}$). Isothermal solidification is a prerequisite for obtaining a proper bond microstructure during TLP bonding. If holding time is lower than $t_{IS}$, athermal solidification of residual liquid phase will lead to intermetallic formation which, in turn, degrades shear strength of bond. In Part 1 [2], the solidification behavior of liquid phase formed during TLP bonding of

* Corresponding author: Majid Pouranvari, mpouranvari@yahoo.com
GTD-111/Ni-4.5Si-3.2B/GTD-111 at 1100°C was detailed. It was observed that the eutectic width decreases linearly with square root of bonding time. In Part 2 [3], the correlation between microstructure and mechanical properties of the joints was studied. High hardness of non-isothermally solidified eutectic products coupled with the fact that nickel boride phase forms interlinked network provide a metallurgical notch which significantly decreases the load carrying capacity of the joint. It was concluded that in bonding condition in which isothermal solidification is not completed, the extent of athermal solidified zone (ASZ) product, eutectic constituents, is the controlling factor of the joint strength. Therefore, it is necessary to eliminate the eutectic products in order to improve the strength of joints [3]. 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 diffusion affected zone (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 [4]. In Part 3, a PBHT was proposed to achieve bonds with improved mechanical properties.

In the final part of this work, the effects of bonding temperature on microstructural features and the time required for isothermal solidification completion \((t_{IS})\) during TLP bonding of GTD-111 nickel base superalloy are investigated. Isothermal solidification is controlled by the solid state diffusion of melting point depressant (MPD) element in the base metal [5-8]. Therefore, it is expected that by using higher temperature, the isothermal solidification will be completed in shorter time. However, as it is discussed in this paper, there is an optimum bonding temperature to minimize the time required to obtain a eutectic free joint.

**Experimental procedure**

The chemical composition (in wt.%) of the base metal, GTD-111 superalloy, was Ni-13.5Cr-9.5Co-4.75Ti-3.8W-1.53Mo-2.7Ta-0.23Fe-0.09C-0.01B.

A commercial Ni-4.5Si-3.2B alloy (MBF30), in the form of an amorphous foil with 25.4 μm thickness was used as the interlayer. The surfaces to be bonded were ground by using 600 grade SiC paper and cleaned in acetone before bonding. Bonding was carried out at 1100, 1150 and 1180°C for various bonding times under a vacuum of approximately 10⁻⁴ torr in a vacuum furnace.

Microstructures of joints were examined by optical microscopy and scanning electron microscopy (SEM) equipped with a beryllium window energy dispersive spectrometer (EDS) system using INCA software. For microstructural examinations, specimens were etched using two etchants. The Murakami etchant (10g KOH, 10g K₃[Fe(CN)₆], 100 ml H₂O) preferentially etches Cr-rich phases and can therefore be used to reveal precipitates adjacent to the joint/base metal interface. Molybdenum-acid etchant (0.5g MoO₃, 50ml HCl, 50ml HNO₃, 200ml H₂O), which preferentially etches γ′ phase, was used to indicate γ-γ′ microstructure of the joints, in addition to joint centerline microstructure.
Results and discussion

Effect of bonding temperature on joint microstructure

Fig. 1 shows SEM image of joint made at 1100°C for 30 min. A typical microstructure of TLP bonded joint of a nickel base precipitation hardened superalloy such as GTD-111 using a B containing interlayer, consists of three distinct microstructural zones, before completion of isothermal solidification [2]:

i) Athermally Solidified Zone (ASZ): The microstructure of this region consists of microconstituents with eutectic-like morphology, and is made of two distinct phases: a nickel-rich boride intermetallic phase and a nickel rich γ-solid solution [2]. 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).

ii) Isothermally Solidified Zone (ISZ): The microstructure of this zone consists of proeutectic nickel rich γ solid solution phase and is almost free from γ' precipitates [2]. 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 [9].

iii) Diffusion Affected Zone (DAZ): The microstructure of this region consists of second phase particles with two different morphologies: blocky particles and
particles with needle-like morphology. EDS analysis of metallic elements suggested that both particles are Cr rich carbo-boride [2].

![Figure 2](image_url)

**Fig. 2 Microstructure of bonds made at a) 1150°C and b) 1180°C for 30 min**

Fig. 2a shows a typical microstructure of joint made at 1150°C for 30 min illustrating some eutectic type structure. EDS analysis of the eutectic microconstituent showed that microstructure constituents are similar to those of at 1100°C.

Fig. 2b shows microstructure of joint made at 1180°C for 30 min. Detail of joint centerline microstructure is shown in Fig. 3 indicating that the joint centerline comprised of $\gamma$-$\gamma'$ eutectic and an intermetallic phase which was formed adjacent to the non-equilibrium $\gamma$-$\gamma'$ eutectic. EDS analysis of the intermetallic phase showed that this
Poranvari - Diffusion brazing of a nickel based superalloy Part 4...

One interesting feature of bonds made at 1180°C was the lack of secondary precipitates, Cr-borides, in the DAZ (see Fig. 2b). The lack of these precipitates was confirmed by Murukami etchant. However, some liquated regions were observed in the substrate region.

Effect of bonding temperature on solidification behavior

Solidification behavior of ASZ is governed by formation of γ solid solution and subsequent solute partitioning. Microstructure of ASZ in bonds made at 1100 and 1150°C is governed by segregation of MPD elements during non-equilibrium solidification. Very low solubility of B in Ni and partition coefficient of B in Ni [10] lead to rejection of B into the adjacent melt shifting composition of melt towards the eutectic composition; thus, with progressing of solidification, binary eutectic of γ solid solution and nickel boride is formed. The EDS analysis of ASZ showed no silicide
phase in this zone. Indeed, low solubility and low partition coefficient of B in Ni base substrates resulted in formation of intermetallic phases within eutectic structure of ASZ. Therefore, it can be deduced that joint microstructure and isothermal solidification rate at 1100 and 1150°C is dominated by diffusion of B.

According to the microstructural investigation, at bonding temperature of 1180°C, the ASZ microstructure is controlled by segregation of base metal alloying elements particularly Ti and Cr. Indeed, greater dissolution of base metal alloying element and higher diffusion of Ti and Cr from base metal during isothermal solidification at bonding temperature of 1180°C in comparison to lower bonding temperatures of 1100 and 1150°C govern the microstructure development in the ASZ. It has been reported that $\gamma$-$\gamma'$ eutectic was formed due to the segregation of some alloying elements particularly Ti during none-equilibrium solidification of nickel base superalloy [11]. Cr, Mo, and W have low concentrations in $\gamma'$ phase, the main phase in $\gamma$-$\gamma'$ eutectic; therefore have low concentrations in $\gamma$-$\gamma'$ eutectic [12]. Therefore, residual liquid is enriched with these boride forming elements (e.g. Cr, Mo and W). It has also been reported that Cr and Mo exhibit positive segregation into the residual liquid phase during $\gamma$-$\gamma'$ eutectic transformation in a nickel base superalloy [13]. Therefore, formation of Cr-rich borides in the ASZ can explain enrichment of liquid with boride forming elements and presence of boron in the liquid phase.

Effect of bonding temperature on the time required for isothermal solidification completion

Investigation of the influence of bonding temperature on tIS bonding was carried out for different bonding times at 1100, 1150 and 1180°C. The average ASZ width was measured using SEM micrographs and plotted against bonding time for various bonding temperature (Fig. 4). As can be seen, the ASZ decreases with increasing bonding time at a given bonding temperature. Isothermal solidification, which prevents the formation of centerline eutectic constituent, completes at the bonding time of 75 min at 1100°C. Increase in bonding temperature is expected to significantly reduce the required time to attain a eutectic free joint due to the increase in MPD diffusion coefficient. Sakamoto et al. [14] proposed a linear relation between maximum clearance free from eutectic and a brazing parameter, $(T+20 \log t)^{1/2}$, where T is bonding temperature and t is bonding time. Therefore it was suggested that the increasing bonding temperature results in shorter tIS.

Although, increasing the bonding temperature to 1150°C led to decreasing of tIS to 45 min (see inset of Fig. 4) but, a eutectic free joint was not attained even after 120 min holding time at 1180°C (see inset of Fig.4). Therefore, isothermal solidification behavior at 1180°C is abnormal with regard to conventional expectation. This is in agreement with the previous works on TLP bonding of In738LC using Ni-Cr-B interlayer by Idowu et al. [15] and Wikstrom et al. [16] who reported that isothermal solidification rate decreases at high temperatures.
Isothermal solidification requires diffusion of MPD element out of liquid phase into the base metal until liquidus temperature of the liquid phase reaches the bonding temperature. When liquidus temperature of liquid phase reaches to bonding temperature isothermal solidification starts. Isothermal solidification is completely accomplished when liquidus temperature of residual liquid at the center of the joint reaches the bonding temperature. In this situation MPD concentration at the center of the joint is reduced to the solid solubility of MPD at the bonding temperature [5]. Therefore, tIS depends on the MPD elements flux from the liquid phase into the BM and phase relationships in BM/interlayer system. In binary TLP system, a critical bonding temperature is also expected due to interplay between increase in diffusivity of MPD elements and increase in maximum widening of the liquid [17]. However, in multicomponents systems such as present system, GTD-111/MBF30, this cannot be solely explanation. The observed behavior can be explained as follows:

i) as previously mentioned, B controls isothermal solidification process at 1100 and 1150°C, while at bonding temperature of 1180°C isothermal solidification is controlled by Ti. Diffusion coefficient of Ti in Ni base alloys is much lower than that of B in Ni. Ti is a substitutional solute and B is interstitial solute in Ni.

ii) The solidification partition coefficient of Ti in nickel is less than unity [18]; therefore an increase in its concentration in the liquid would result in a depression of residual liquid solidification temperature. Isothermal solidification is completely accomplished when liquidus temperature of residual liquid at the joint centerline reaches the bonding temperature, as a result of diffusion of MPD elements from the liquid to the adjacent solid. Therefore, considering low diffusion coefficient of Ti in Ni and high difference between bonding temperature and liquidus temperature of

![Fig. 4 Variation of ASZ size with bonding time at various bonding temperature](image-url)
residual liquid can explain lower isothermal solidification rate at 1180°C compared to the isothermal solidification rate at 1100 and 1150°C.

Isothermal solidification kinetics during TLP bonding depends not only on diffusivity of MPD elements, but also on the MPD solubility in the base metal. Indeed in multicomponents system such as nickel base superalloy interplay between these factors is the controlling factor for the required time to attain a eutectic free TLP joint. Therefore, developing of multicomponents TLP models is necessary for contribution of better understanding of isothermal solidification during TLP bonding of complex alloy systems.

Conclusions
From this research the following conclusions can be drawn:
1. Bonding temperature significantly affects athermally solidified zone microstructure; at lower temperature ASZ microstructure is governed by B diffusion, while at high bonding temperature ASZ microstructure is significantly affected by dissolution and diffusion of base metal alloying element into the joint region, particularly Ti and Cr.
2. DAZ precipitates formation is significantly affected by bonding temperature. At bonding temperatures of 1100 and 1150°C extensive Cr-rich carbo-boride was observed in DAZ, while at bonding temperature of 1180°C joint/base metal interface was almost precipitates free.
3. There is a critical bonding temperature to minimize the required time for isothermal solidification and hence, to attain a eutectic free TLP joint. Reduction of isothermal solidification rate at bonding temperature of 1180°C can be attributed to the enrichment of residual liquid with some base metal alloying element particularly Ti and Cr, during dissolution and isothermal solidification, in addition to increasing joint width.

References


