Effect of bonding variables on TLP bonding of oxide dispersion strengthened superalloy

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Received: 7 June 2006 / Accepted: 6 June 2007 / Published online: 27 July 2007 Springer Science+Business Media, LLC 2007

Abstract Transient liquid phase (TLP) bonding has evolved as a successful alternative joining technique for high service temperature components (e.g., vanes and blades for aircraft gas turbine engines) made from superalloys when neither fusion welding nor solid-state bonding techniques are successful. However, study shows that the optimization of bonding variables is critical to achieve a metallurgically sound joint free from deleterious intermetallic constituents in the joint region. In this study, the influence of bonding pressure and interlayer thickness on microstructural developments at the joint region of TLP bonded oxide dispersion strengthened (ODS) superalloy MA758 was examined. A commercial interlayer based on the Ni–Cr–B (MBF-80) system was used and results showed that bonding pressure and interlayer thickness affected the final width of the joints. A theoretical study revealed that the TLP bonding time can also be reduced when there is an increase in bonding pressure.

Introduction

Transient liquid phase bonding has been used widely to join oxide dispersion strengthened superalloys that are difficult to bond by conventional fusion welding. The high heat input employed in fusion welding disrupts the grain structure and produces welds with poor mechanical

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strength at high temperature [[1,](#page-6-0) [2\]](#page-6-0). In TLP bonding, an interlayer alloy of specific composition which has a melting point lower than that of the parent alloy is placed between the bonding surfaces and the assembly is heated to the bonding temperature. At the bonding temperature, the interlayer melts filing the gaps between the surfaces to be joined with a liquid phase. This liquid phase is gradually removed through interdiffusion of alloying elements between the interlayer and the parent alloy. The change in composition of the joint causes the joint to isothermally solidify. A complete isothermal solidification of the liquid interlayer results in the formation of a solid solution phase. In the case of pure metals, homogenization can result in a joint that has a microstructure and mechanical properties similar to those of the base alloy. However, a post-bond heat treatment is normally employed to fully homogenize complex alloy systems such as ODS alloys. Earlier research has shown that the brittle intermetallic constituents, which have been demonstrated to be detrimental to the mechanical properties of joints, are often formed across the joint region in the TLP bonding process [[3\]](#page-6-0). Furthermore, the commercial application of TLP bonding would require a reasonable length of time to complete the bonding process. This suggested that the optimization of bonding variables was critical for TLP bonding technique.

Hitherto most of the research work has focused on the effect of bonding temperature, bonding time, interlayer composition and parent metal grain size on the microstructural development in TLP joints [[4,](#page-6-0) [5\]](#page-6-0). Applying a compressive load during TLP bonding has been reported beneficial. Rabinkin and Pounds [[6\]](#page-6-0) found that for brazing Cu with Cu–P filler metal under an applied pressure of 9.8 MPa, its Charpy impact energy was increased. An ejection model had been proposed to explain the increase in brazement strength. However, the scientific literature

shows that very little has been done on how bonding pressure can affect the microstructural behavior and isothermal solidification during TLP bonding of ODS alloys. Therefore, the objective of this research work is to investigate the effect of bonding pressure on microstructural developments and on isothermal solidification rate of TLP bonded MA 758 ODS nickel alloy using MBF-80 (Ni–Cr–B) interlayer. The influence of interlayer thickness on the bonding behavior is also reported.

Experimental procedure

The fine-grained $(1-2 \mu m)$ nickel based ODS alloy, Inconel MA 758, was joined in the as received condition. The nominal composition of the alloy in wt% was Ni-bal., 30- Cr, 0.3-Al, 0.5-Ti, 0.6- Y_2O_3 , 1.0-Fe, and 0.05-C. All specimens were cut to a dimension of $10 \times 10 \times 5$ mm. Surfaces to be joined were prepared by grinding down to 1000-grit SiC paper finish and cleaned in acetone before bonding. A commercially available Ni based interlayer MBF 80 in the form of thin foils was used for TLP bonding. The composition of the interlayer in wt% was Ni-bal., 15.2-Cr, 0.06-C and 4.0-B. Two different interlayer thicknesses of 40 and 80 µm were employed for TLP bonding.

The joining process was performed in an induction heating furnace with the vacuum of 0.053 Pa $(10^{-4}$ Torr) using four different bonding pressures of 0.2, 1, 2, and 3 MPa. A hold time of 30 min at the bonding temperature was used. Depending on the melting temperature of interlayer the bonding temperature of $1,120$ °C was selected. The specimens were furnace cooled to room temperature in vacuum once the holding time was completed. For metallographic examination, the bonded samples were sectioned through the joint region and one half of each bonded sample was then heat treated using a temperature of 1,360 \degree C for 2 h. All specimens were prepared using standard methods of metallographic preparation which includes grinding surfaces down to a 1000-grit SiC paper finish, followed by polishing with diamond suspension to a 1 µm finish. The chemical enchant was prepared by mixing one part of H_2O_2 , two parts of concentrated HCl, and two parts of distilled water just before etching. The joint region was examined using optical and scanning electron microscopy (Jeol JXA-8200). The changes in the composition of the bond interface were analyzed using energy dispersive X-ray spectroscopy (EDS). Micro-hardness testing was done to assess the homogeneity of the bond region using a Leitz Mini-load micro-hardness tester with a load of 50 g. Each value of the Vicker's micro-hardness is an average of five measurements.

Results and discussion

Joint microstructure

The effect of holding time at the bonding temperature on TLP bonding behavior of MA 758 using Ni–Cr–Co–Fe–B–Si and Ni–P interlayers has been reported previously [\[5](#page-6-0)]. It was suggested that a hold time of 30 min at the bonding temperature could complete isothermal solidification and produce a joint free from residual eutectic constituents. Therefore, in this study a 30 min hold time was employed for all bonds. Figure [1a](#page-2-0)–d shows SEM micrographs of joints made using MBF 80 interlayer for 30 min hold time with various bonding pressures of 0.2–3 MPa. The absence of the residual eutectic structure within the joint indicated that a significant amount of interdiffusion between the interlayer and the parent alloy had taken place resulting in isothermally solidified joint. However, inclusions were detected at the joint interface and close examination revealed that the amount of the inclusion was reduced as the bonding pressure increased (see Fig. [1\)](#page-2-0). As shown in Fig. [2](#page-2-0), the EDS spectrum obtained from these inclusions suggests that they are oxides of aluminium and yttrium. When pressure was applied to the TLP bonds, a liquid phase enriched with Al and Y was thought to be ejected from the joint region resulting in reduction of inclusions along the bond interface. Figure [3](#page-3-0) shows the ejected liquid at the joint edge of a TLP bond made under an applied pressure of 2 MPa. Both Al and Y_2O_3 are constituents of the ODS parent alloy and could enter the liquid during parent metal dissolution during the early stages of bondings. Although the amount of ejected liquid increased with the increase of bonding pressure, the amount of Al and Y_2O_3 constituents remain same for bonds made using all four bonding pressures. Therefore, like all other ejected elements, higher bonding pressure would result higher amount of Al and Y to be squeezed out.

The width of the liquid produced by the melting of the interlayer during TLP bonding markedly affects the joint width which in turns affects the bond quality [\[7](#page-6-0), [8](#page-6-0)]. As mentioned earlier, when TLP bonds are made under a bonding pressure, the liquid interlayer is ejected out of the joint region and the joint width changes at the same time. The influence of bonding pressure on the final joint width is shown in Fig. [4](#page-3-0). Each value of the final joint width is an average of 10 measurements made along the bonded interface. The joint width decreased with an increase in bonding pressure up to a value of 2 MPa. The decrease in joint width is related to the amount of ejected liquid interlayer which increases as the bonding pressure rises. However, a further increase of bonding pressure to 3 MPa had no further effect on the final joint width. An equal value of $17 \mu m$ of the final joint width was measured for Fig. 1 SEM micrographs of joints made using a bonding pressure of: (a) 0.2 MPa (b) 1 MPa (c) 2 MPa (d) 3 MPa

the TLP bonds made under the bonding pressure of both 2 and 3 MPa. A similar finding was reported by Li et al. [[8\]](#page-6-0) when TLP bonding of ASM 6061 T6 using copper insert under various applied load. They introduced a new variable, Wc (minimum liquid width), and suggested that the liquid is expelled until Wc is attained during TLP bonding under an applied load.

To study the effects of interlayer thickness on the microstructural development the parent alloy was bonded using an 80-µm thick interlayer with a bonding pressure of 0.2 MPa. A holding time of 30 min and the bonding temperature of $1,120$ °C were employed for this bond. Figure [5](#page-3-0) shows the microstructure of such a joint. In general, the microstructures of the joints made using both 40 and 80 µm thick interlayers look similar with the exception in the final joint width. A final joint width of 25 and $46 \mu m$ was measured for joints made using 40 and 80 µm thick interlayers, respectively.

In order to induce recrystallization and grain growth throughout the alloy, the bonded samples were subjected to a post-bond heat treatment at $1,360$ $1,360$ $1,360$ °C for 2 h. Figure 6 shows a continuous microstructure across the joint region for the bond made under a bonding pressure of 1 MPa followed by a post-bond heat treatment.

Joint composition

Composition profiles, measured as a function of distance from the center of the joint, for bond made under a bonding pressure of 0.2 MPa can be seen in Fig. [7.](#page-4-0) The graph shows that the distribution of Ni and Cr is not homogeneous within the joint. The concentration of Ni decreased

Fig. 3 Ejected liquid at the joint edge of the TLP bond (2 MPa)

Fig. 4 Effect of bonding pressure on final joint width

(the concentration of Cr increased) with distance away from the center of the joint until it became constant within the parent alloy. There is a high concentration of Al near the joint interface (liquid/solid interface); this can be attributed to the segregation of Al to the bonding interface and also to the dissolution and break-up of surface oxides during the bonding process. A lower content of yttrium within the joint region suggested the inhomogeneous distribution of this strengthening particle across the joint region. The composition profiles across the bonds made using higher bonding pressures were, in general, similar to that for bond made under a bonding pressure of 0.2 MPa. When a post-bond heat treatment was applied to the as

Fig. 5 SEM micrograph of joint made using 80 µm thick interlayer

Fig. 6 Microstructure of joint made under a bonding pressure of 1 MPa followed by heat treatment

bonded specimens it allowed some compositional homogenization of the joint region. Figure [8](#page-4-0) shows the composition profiles for the bond made using MBF 80 interlayer followed by a post-bond heat treatment.

Isothermal solidification

The time required to complete the bonding process is largely determined by the completion time for isothermal solidification [[9\]](#page-6-0). However, some information on the isothermal solidification process can be obtained by monitoring changes in the joint composition with respect

Fig. 8 Composition profiles of the bond made under a bonding pressure of 0.2 MPa followed by post-bond heat treatment

to the concentration of melting point depressants remaining in the interlayer. An experimental value for the completion of isothermal solidification is more difficult to obtain. In reality, it would be necessary to monitor the dynamic changes in composition of the joint during the bonding process in order to determine the exact time for isothermal solidification. These limitations have therefore, led to the use of analytical models to predict a value for the bonding time. Research work on the modeling of TLP bonding process by Tuah-Poku et al. [\[9](#page-6-0)] suggested that the time for completion of isothermal solidification strongly depends on the liquid width at the bonding temperature and is given by:

$$
t_{is} = \frac{W_{\text{max}}^2}{16K^2D} \tag{1}
$$

where K is a constant and D is the diffusion coefficient of boron in nickel. Although both the base metal and the interlayer contain a number of elements, nickel and boron are thought to be the key elements for the TLP bonding process. Therefore, the diffusivity of the solute in the base metal is approximated as a binary case of B in Ni.

TLP models developed by Tuah-Poku et al. [\[9](#page-6-0)] using the Ag/Cu/Ag system, have shown that it is possible to calculate the maximum liquid width produced during

the bonding process by applying the mass conservation equation:

$$
W_{\text{max}} = W_o \left[\left(1 + \frac{C_o - C_{L\alpha}}{C_{L\alpha}} \right) \frac{\rho_y}{\rho_x} \right]
$$
 (2)

where W_o is the initial interlayer width, W_{max} is the maximum liquid interlayer width, C_o is the initial concentration of boron in the interlayer, $C_{L\alpha}$ is the solute concentration at which the liquid interlayer homogenizes, ρ_{v} and ρ_{x} are the respective densities of interlayer and parent material. In the calculations, the change in volume due to liquefaction is neglected and hence the changes in density. The models developed for TLP bonding process assume that there are no changes in density of the liquid and solid phases.

However, when TLP bonds are made using a bonding pressure the maximum liquid width is restricted because of the expulsion of liquid filler from the joint. Therefore, the isothermal solidification time in the present study was predicted using the value of final joint width (see Fig. [4\)](#page-3-0) which is assumed to be the same as maximum liquid width. The maximum liquid width was measured experimentally by considering the solid/liquid interfaces where the agglomerations/inclusions were detected. In their model on TLP bonding process Tuah-Poku et al. [[9\]](#page-6-0) suggested that the maximum liquid width is attained during the second

stage of TLP bonding process when widening and homogenization of the liquid layer takes place. The parent metal dissolution (also known as melt-back) occurs as a result of the homogenization of the liquid layer and, in the present work, melt-back produced a line of agglomeration of the dispersed phase (which is rich in Al and Y) at the solid/liquid interface. Research work on TLP bonding of ODS alloy reported by Khan and Wallach also observed melt-back phenomenon leading to delineation of Y_2O_3 particles at the solid/liquid interface [\[10](#page-6-0)].

Zhou $[11]$ $[11]$ indicated that K in Eq. 1 has to be numerically calculated using the following equation:

$$
\frac{K(1+erf(K)\sqrt{\pi})}{\exp(-K^2)} = \frac{C_{\alpha L} - C_M}{C_{L\alpha} - C_M} \tag{3}
$$

where, C_M is the initial boron concentration in the parent alloy (0 wt%), $C_{\alpha L}$ was taken to be 0.1 wt%, [[12\]](#page-6-0) which is the average concentration of boron in Ni–B binary solidus over the bonding temperature range $1,070-1,130$ °C and $C_{L\alpha}$ was taken from the phase diagram [\[13](#page-6-0)] to be 3.6 wt% at the bonding temperature of $1,100$ °C.

The value of 6.22×10^{-11} m²/s for boron diffusivity in the parent alloy was used in the present work. This diffusivity was determined for the diffusion of boron out of a Ni–Cr–Si–B interlayer into nickel based substrate by Kucera et al. [[14\]](#page-6-0). When substituting all the respective values in to Eq. 1, the time required to complete isothermal solidification, t_{is} , was found to be 40, 28, and 18.5 min for 0.2, 1, and 2 MPa, respectively. When comparing these values with the experimental values for completing isothermal solidification time, both calculated and experimental time (30 min hold time) are in reasonable agreement, although, the predicted value of 40 min in the 0.2 MPa case is greater than the experimental isothermal hold time of 30 min. In general, the theoretical results suggested that the applied bonding pressure can reduce the TLP bonding time. This is attributed to the decrease of volume fraction of liquid phase from the joint region resulting in a shorter time to complete isothermal solidification. It follows that expulsion of liquid from the joint region is particularly beneficial in productivity term. This result is in consistent with the previous finding by Li et al. [\[8](#page-6-0)] when TLP bonding of aluminium based metal matrix composite (ASM 6061 T6) using a copper insert.

Micro-hardness measurements

The changes in the micro-hardness across a joint region can be used to assess the degree of homogeneity of the bond region compared to the parent alloy. The uniform hardness values across the joint region would mean a good homogeneity. In this study, micro-hardness profiles were

Fig. 9 Hardness profiles for bonds made under a bonding pressure of 0.2 and 3 MPa

measured as a function of distance from the center of the joint region. Figure 9 shows the micro-hardness profiles for bonds made using two different bonding pressures of 0.2 and 3 MPa. The hardness value measured at the center of the TLP bond is low (246 VHN) compared to that of the parent alloy (407 VHN) for the bond made using a bonding pressure of 0.2 MPa. When micro-hardness measurements were taken across the bond made with a bonding pressure of 3 MPa, a similar hardness profile was recorded except that the value of increasing zone of micro-hardness was narrower. This corresponds to the difference in joint width measured for bonds made using the bonding pressure of 0.2 and 3 MPa. A variation in micro-hardness values at center of the bonds between 0.2 and 3 MPa was measured and this was thought to be related to the difference in grain size within the joint region between these two bonds. The size of the grains at joint region of as bonded samples is restricted by the joint interface and therefore relatively larger grain size can be expected for the bond with higher

450 400 Vicker's hardness, VHN 350 300 250 200 150 Before H.T. 100 After H.T. 50 0 $\bf{0}$ $20\quad 40$ 60 80 100 120 140 160 180 200 220 Distance from the centre of the bonds, um

Fig. 10 Hardness profiles for bonds made under a bonding pressure of 0.2 MPa (before and after heat treatment)

joint width. However, approximately 40-um distance away from the joint center the hardness values became uniform within the parent alloy in both cases. Figure [10](#page-5-0) shows the differences in hardness profiles before and after post-bond heat treatment. Lower hardness values are obtained after the use of a post-bond heat treatment. This decrease in hardness was attributed to an increase in grain size which is expected because of the well-known Hall-Petch relationship, which shows that the hardness is proportional to the square root of the diameter of a grain. However, the profiles indicated that the difference in hardness between the joint region and parent alloy was still significant and the bonded region would not qualify as a fully homogenized region. This suggested that the joint region will probably have lower mechanical strength compare to the parent ODS alloy.

Conclusions

The transient liquid phase diffusing bonding of nickel based ODS alloy, Inconel MA758, was performed using four different bonding pressures of 0.2, 1, 2, and 3 MPa. Theoretical and experimental approaches were conducted to study the bonding behavior. The results from this studies showed that the use of bonding pressure during TLP bonding reduced the final width of the joint as well as the amount of inclusions adjacent to the joint interface. However, no difference in final joint width between the bonds made using the bonding pressure of 2 and 3 MPa was observed. The final joint width increased when TLP bonds

were made using a higher interlayer thickness of 80 um. The theoretical study suggested that the TLP bonding time can be reduced with an increase in bonding pressure. The post-bond heat treatment at $1,360$ °C for 2 h helped to homogenize the composition of the joint region.

Acknowledgments The authors wish to thank Natural Sciences and Engineering Research of Canada for financial support for this research.

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