



## The recovery of tensile ductility in diffusion-bonded Ni-base alloys by post-bond heat treatments

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### ABSTRACT

The effects of post-bond heat treatments on the microstructure and tensile properties of diffusion-bonded Ni-base superalloys, Alloy 617 and Haynes 230, were investigated. In the as-bonded condition, precipitates including carbides and oxides were extensively observed along the bond-line. To remove the precipitates, post-bond heat treatments were applied at temperatures ranging from 1000 to 1200 °C for up to 100 h. In the as-bonded condition, a significant loss of ductility was observed, especially at 900 °C, for both alloys. Also, the final fracture occurred at the bond-line in the form of a brittle fracture in the as-bonded condition. As the post-bond heat treatment temperature was increased, the tensile ductility at 900 °C improved significantly while the strength decreased to a certain extent. The recovery of the tensile ductility was closely related to the dissolution of precipitates during the post-bond heat treatments. Eventually, the tensile ductility of diffusion-bonded Ni-base alloys at 900 °C was increased to half that of the base metal by the post-bond heat treatments.

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### 1. Introduction

A very high temperature gas-cooled reactor (VHTR) is one of leading reactor concepts for generation-IV nuclear reactor systems. As the operating temperature will be greater than 850 °C with a coolant gas pressure up to 9 MPa, Ni-base superalloys such as Alloy 617 and Haynes 230 are considered as candidate materials for intermediate heat exchangers (IHXs) for VHTRs [1]. Micro-channel heat exchanger designs are likely to be adopted for the IHX to utilize the benefits of high heat transfer efficiency and relatively low stress during the operation [2]. The manufacturing process of micro-channel heat exchangers requires the joining of thin metal sheets, a few millimeters thick, with flow passages that are either machined or photochemically etched. To join such thin sheets, brazing, transient liquid phase bonding and diffusion bonding processes are expected to be an essential part of the processes.

Recently, Jiang et al. [3] discussed the hold time effect on brazing for stainless steel 304 for the application of micro-channel type heat exchangers. They identified the developed phases in the brazed joint and evaluated the tensile strength depending on the hold time to avoid the evolution of brittle phases. For the brazed Ni-base alloys [4], stress–rupture tests were conducted up to 1000 h for various brazing alloys in the temperature ranges of

982–1093 °C. Also, many researchers have reported notable results involving the transient liquid phase (TLP) bonding of Ni-base alloys. For example, Jalilian et al. [5] joined Alloy 617 using a different thickness of the filler metals to figure out the effect of holding time on the eutectic width. Arafim et al. [6] suggested the isothermal solidification completion time during TLP bonding of Inconel 718 and Inconel 625 with the experimental results. They showed that the amount of brittle phase, such as intermetallics, carbides, and borides, at the filler metals decreased under a proper isothermal solidification condition, and a high-quality microstructure similar to that of the base metal could be achieved. However, though a sound bond could be developed by brazing or TLP bonding, the complex flow passages of IHX would make the bonding methods less feasible for practical applications.

There have been a few studies of the diffusion bonding of solid solution strengthened Ni-base alloys. Takeda et al. [2] successfully manufactured small-scale compact heat exchangers by the diffusion bonding of Hastelloy XR and evaluated the leakage and thermal performance of the bonds. Based on comprehensive studies (temperature, duration time, surface roughness and compression stress) and mechanical test (tensile and impact) results, Basuki et al. [7] suggested optimized diffusion bonding conditions for Hastelloy C-22. For the precipitation strengthened Ni-base superalloy, Zhang et al. [8] achieved the tensile strengths joint which is comparable to that of the parent metal for Inconel 718. For the intermetallics, Torun and Celikyurek [9] investigated the effect of the holding time on the bond quality of nickel aluminide with a thin Ni interlayer between the base metals. However, these studies

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were mainly focused on microstructural characterizations at the bond-line, while extensive mechanical property evaluation such as creep and tensile tests were not conducted, especially at high temperatures.

Preliminary tests by the authors showed that the tensile ductility of diffusion-bonded Alloy 617 was much lower than that of the base metal. Meanwhile, the possibility of improving the mechanical properties of diffusion-bonded Alloy 617 by a heat treatment was recently reported [10]. However, details of the heat treatment and subsequent analysis results were not provided. Thus, the authors initiated an effort to identify proper post-bond heat treatment conditions to achieve feasible mechanical properties for diffusion-bonded Ni-base alloys for VHTR applications. In this study, the effects of post-bond heat treatments on the microstructure and mechanical properties, especially the tensile properties at a high temperature, were investigated for two commercial Ni-base superalloys, Alloy 617 and Haynes 230.

It could be said that bonding quality is determined by carefully controlled precipitates (oxides or carbides) at the bond-line. From that point of view, the high-temperature oxidation behaviors and mechanical properties of these alloys were extensively studied by the authors [11,12] such that baseline data are readily available for comparison with those of diffusion-bonded specimens. Also, the carbides, which further consideration is given to in the paper, were identified in various environment [12] and temperatures [13–15]. Therefore, based on the test results and the microstructure analysis, the optimum post-bond heat treatment conditions to recover the tensile ductility were proposed.

## 2. Experimental procedure

The chemical compositions of the commercial alloys used in this study are summarized in Table 1. Diffusion bonding was done by a contractor, Weltec Korea Corporation, and the post-bond heat treatment was done by the authors. Though the authors suggested a possible diffusion bonding condition for Alloy 617 and Haynes 230 based on the published results on other Ni-base alloys [2,7,16], the contractor used its own proprietary condition. Therefore, the details of the diffusion bonding conditions, such as the temperature, pressure, holding time, and surface roughness, are not available. However, we can estimate that the diffusion bonding temperature did not differ much from the suggested temperature of 1150 °C based on the microstructure and tensile test results (see Sections 3.1 and 3.3).

After diffusion bonding, post-bond heat treatments were carried out in a high-temperature vacuum furnace ( $\sim 10^{-6}$  Torr). The details of the post-bond heat treatment conditions used in this study are summarized in Table 2, as determined based on a review of published research results for Ni-base alloys [10,17]. A scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) was used to investigate the shape and distribution of precipitates along the bond-line and surrounding region before and after the post-bond heat treatments. To evaluate the effects of heat treatment on the local property variation around the bond-line, local hardness values were measured with a micro-Vickers hardness tester with a load of 0.1 kg following ASTM: E384-11e1.

**Table 1**  
Chemical compositions of the Ni-base superalloys (wt.%).

Alloy	Ni	Cr	Co	Mo	W	Al	Fe	Mn	Ti	Si	C
Alloy 617	Bal.	22.1	11.6	9.57	0.07	1.41	1.8	0.12	0.35	0.42	0.09
Haynes 230	Bal.	20.05	5.12	1.66	14.0	0.64	1.03	0.63	0.18	0.02	0.10

**Table 2**

Post-bond heat treatment conditions for superalloys (DB: diffusion bonding, HT: heat treatment, WQ: water quenching, AC: air cooling).

Materials	HT condition	Temperature (°C)	Dwell time (h)	Cooling method
Alloy 617	DB/HT-A	1177	20	WQ
	DB/HT-B	1150	4	WQ
	DB/HT-C	1135	24	AC
	DB/HT-G	1123	24	AC
	DB/HT-H	1000	100	AC
Haynes 230	DB/HT-D	1200	100	AC
	DB/HT-E	1180	46	AC
	DB/HT-F	1150	23	AC

Miniature tensile specimens were machined after the post-bond heat treatments. Fig. 1 shows the schematics of a diffusion-bonded block. Also shown is the miniature tensile specimen machined from the block following ASTM: E8/E8M-11. Tensile tests were conducted both at room temperature and at 900 °C with the strain rate of  $3.33 \times 10^{-4} \text{ s}^{-1}$ . Finally, the fracture surfaces of tensile tested specimens were observed under SEM to characterize the fracture mode.

## 3. Results and discussion

### 3.1. Evolution of microstructure

SEM micrographs of Alloy 617 and Haynes 230 after various heat treatments are shown in Figs. 2 and 3. In the as-received condition, Cr-rich  $\text{M}_{23}\text{C}_6$ -type carbides developed along the grain boundary in both alloys (Figs. 2a and 3a). In addition, while stringers of carbides are aligned in the rolling direction for Alloy 617 (Fig. 2a), bulky W-rich  $\text{M}_6\text{C}$ -type carbides are sporadically distributed in Haynes 230 (Fig. 3a). The morphology and distribution of the carbides are in good agreement with previously reported results of Alloy 617 [13–15] and Haynes 230 [18]. In the as-bonded condition (Figs. 2b and 3b), the morphology and distribution of the carbides away from the bond-line are nearly identical to those of the as-received condition for both alloys. This may be evidence that diffusion bonding was performed at a temperature lower than the final annealing temperature of the alloys. More specifically, in the case of Alloy 617, the as-bonded microstructure was clearly altered after the DB/HT-A heat treatment but it was scarcely changed after the DB/HT-B and DB/HT-C heat treatments. It can therefore be estimated that the diffusion bonding of Alloy 617 was performed in the temperature range of 1135–1150 °C. The temperature range performed in this study is similar to previously reported bond condition [16]; they obtained sound bond with that condition and the microstructure near the bond-line is similar to that of our result (Fig. 2b). In the case of Haynes 230, the as-bonded microstructure (Fig. 3b) was intact after the DB/HT-F heat treatment, suggesting that the diffusion bonding of Haynes 230 was carried out at a temperature of approximately 1150 °C.

In the as-bonded condition, fine precipitates are extensively present along the bond-line, as shown in Figs. 2b and 3b. Previous studies of Ni-base alloys indicated that they may be intermetallics [4–6], oxides (Ni and Cr) [7,16,17] and/or carbides (Ni, Mo and Cr)

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