Achieving high-strength joining of TiAl- and Ni-based alloys at room temperature and 750 °C via utilizing a quinary FeCoNi-based amorphous filler

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High content of single element in TiAl- or Ni-based brazing filler metal always causes the formation of bulk, directional and brittle intermetallics in the TiAl/Ni brazed joints, which deteriorates the joint mechanical properties. In this study, a multicomponent FeCoNi-based amorphous filler was applied as the filler metal to vacuum braze γ-TiAl and Ni-based superalloy. The high mixing entropy derived from the multi-principle elements was envisaged to retard the atomic diffusion and slower grain growth. The experimental results showed that three different zones were formed in the TiAl/Fe$_{30}$Co$_{20}$Ni$_{30}$Si$_{15}$B$_{15}$/Ni joint: A diffusion zone I composed of fine γ-(Fe, Ni) phase; a crystallization seam II containing dendritic α'- (Fe, Co) phase and nanoscale (Fe, Co, Ni)$_{33}$B$_6$, Fe$_2$B in the inter-dendritic regions; a diffusion and reaction zone III consisting of blocky γ$_3$ and mixed (Fe, Co, Ni)$_{33}$B$_6$, γ, TiCo phases. The relationship between the microstructure and the mechanical property both at room temperature and high temperatures were discussed. The shear tests showed that the maximum shear strength at 30 °C and at 750 °C were 363 MPa and 252 MPa respectively when brazed at 1020 °C for 15 min.

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1. Introduction

Owing to the attractive properties of excellent high-temperature property, high fatigue resistance and low density, the TiAl-based intermetallics have been regarded as the most potential materials to substitute the Ni-based superalloys in the temperature range of 650 °C–750 °C, with the benefit of a weigh saving of 50% [1-5]. Among the numerous manufacturing technologies, joining has been verified as the most innovative and feasible method that can make the most use of the advantages of the two alloys [6-10]. Unfortunately, the joining of TiAl-based intermetallics ad Ni-based superalloys has always been a challenging work due to the big difference between the two alloys and the poor weldability of themselves. For TiAl-based intermetallics, the room-temperature brittleness and high crack sensitivity make it hard to get a high-strength joint utilizing the conventional fusion joining methods such as arc welding [7,8]. As to the Ni-based superalloys, the high alloying content and combined effect of Al and Ti in Ni-based superalloys seriously decrease their weldability. Multiple defects including solidification cracking, gain boundary liquation cracking and so on may occur during joining.

Brazing has been verified as the most feasible and economical bonding technique to join the two alloys [7,8,11-15]. The selection of filler metal plays a key role in obtain-
ing a satisfactory joint. Oliveira et al. have reported that the dissimilar welding difficulties such as the difference in thermo-physical compatibility, the formation of bulky brittle intermetallic compounds can be mitigated through selecting an appropriate filler metal [16]. Recently, the amorphous brazing filler metals (BFMs) have attracted more and more researcher’s attention for the outstanding mechanical properties of amorphous alloys and their eutectic or near eutectic composition [13,17–21]. Shujie et al. have reported that a high strength joining of titanium alloys was achieved through using a multicomponent TiZr-based amorphous BFM [17]. Jung et al. investigated the low-temperature brazing of titanium by the application of a Zr–Ti–Ni–Cu–Be bulk metallic glass (BMG) alloy as a filler [22]. In our previous work, we have achieved a high strength joining of TiAl and Ni-based alloys through utilizing a Ti–Zr–Be–Co amorphous ribbon [23]. In addition, from a practical point of view, the high temperature property of TiAl/Ni joint is also essential. The author has reported that a TiAl/Ni brazed joint with a high strength at 600° was obtained by using a Zr-Al-Ni-Co amorphous BFM, whereas the serving temperature of TiAl-based intermetallics and Ni-based alloys are always higher than 600° [24].

To data, no research on the brazing of TiAl and Ni-based alloys utilizing the Fe-, Co- or Ni-based high-temperature amorphous fillers has been reported. In this work, a quinary FeCoNi-based amorphous alloy with a nominal composition of Fe_{30}Co_{30}Ni_{15}Si_{8}B_{17} was fabricated and applied as the filler metal to join γ-TiAl-based alloy and Ni-based superalloy K24. It is well known that the elements of Fe, Co and Ni are all the base metals of the high-temperature alloys, therefore the Fe-, Co-, Ni-based amorphous alloys should possess excellent mechanical properties at elevated temperatures. In addition, Compared with the crystalline filler metals which are based on single Fe, Co or Ni, the amorphous BFMs have more uniform chemical composition, lower impurity content, and higher purity. The multicomponent composition was reported that it can enhance the mixing entropy of the joint, which will bring a sluggish diffusion effect and hence, slower grain growth, increased recrystallization temperature and lead to exceptional high-temperature strength and structural stability [25–27]. The joining was performed in a vacuum furnace with an atmosphere of 3 × 10^{-3} Pa and in order to investigate the effect of the joining temperature on the evolution of microstructure of the joint, the experiments were conducted at different temperatures. The relationship between microstructure and mechanical properties was also discussed and to evaluate the high-temperature serving property of the joint, the microstructure of the joint after holding at 750° for 1 h was also analyzed.

2. Materials and experiments

2.1. Materials

The nominal compositions of the TiAl- and Ni-based alloys tested by energy-dispersive spectrometer (EDS) are listed in Table 1. Before brazing, the TiAl- and Ni-based alloys were cut into 10 mm × 4 mm × 4 mm and 10 mm × 10 mm × 4 mm in length, width and height respectively. All contacting surfaces were polished with SiC paper up to 1000-grit and then cleaned in the ultrasonic bath using the acetone.

An ingot of Fe_{30}Co_{30}Ni_{15}Si_{8}B_{17} was prepared by arc-melting a mixture of the pure elements with purities of 99.95 percent under the protection of an argon atmosphere. For homogeneity, the ingot was remelted at least 4 times. The ribbon-shaped filler metal was fabricated by remelting the ingot in a quartz tube and then ejecting to a high rotating copper wheel with a speed of 1800 r/min. The width and thickness of the obtained ribbons were 10 mm and 80 μm, respectively. The structure of the obtained ribbons was examined and analyzed by Bruker-AXS D8 Advance X-ray diffraction (XRD) with Cu Kα and Jade software. The scanning speed of the XRD was 2 deg. /min, with a step time of 0.02° from 20° to 80°. The thermal behaviors of the ribbon were examined by using the NETZSCH STA-409PC differential scanning calorimeter (DSC) at a heating rate of 20 °C/min. The XRD result was shown in Fig. 1(a). From the picture, it can be found that only one broad peak exists in the pattern, indicating an amorphous structure. From the DSC curve shown in Fig. 1(b), it can be detected that the glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m) and liquid temperature (T_l) of the Fe_{30}Co_{30}Ni_{15}Si_{8}B_{17} are 507 °C, 561 °C, 927 °C, and 980 °C, respectively.

2.2. Bonding process

The samples were sandwiched with the amorphous ribbon as the interlayer. The joining experiments were performed in a vacuum hot-pressing furnace with a vacuum of 3 × 10^{-3} Pa and a pressure of 5 MPa, respectively. The assembled samples were first heated to 300 °C at a rate of 5 °C/min from room temperature and held for 10 min for homogeneity. Then, in order to avoid the influence of crystallization on the brittleness of the amorphous ribbon, the samples were heated to 1020 °C, 1050 °C, and 1080 °C at a rate of 10 °C/min. Three specimens were prepared for each condition to evaluate the repeatability of the experiments. Schematic diagrams of the assembled samples and heating processes were shown in Fig. 2(a) and (b).

After brazing, the interfacial microstructures and elemental distributions in the joints processed with different parameters were analyzed using the FEI quanta 250 FEG SEM and EDS. XRD and TEM equipped with a selected-area electron diffraction (SAED) were also used to examine the phases in the joint. The room temperature shear strength was tested at 30° with a loading rate of 0.15 mm/min. The high-temperature shearing strength was tested at 750° and before testing, the samples were firstly heated to that temperature and held for 1 h. The fracture morphologies were also analyzed using the SEM and EDS.

3. Results and discussion

Fig. 3(a) shows the microstructure and corresponding EDS mapping of the joint brazed at 1020° for 15 min. It can be seen from the picture that a sound joint with no micro-cracks and holes was obtained after brazing. The width of the brazing seam was approximately 180 μm, which was thicker than that of the initial thickness of the glass ribbon (80 μm). Based
The chemical compositions of the TiAl alloy and Ni-based superalloy (wt. %). 

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Ni</th>
<th>Cr</th>
<th>Nb</th>
<th>Ti</th>
<th>V</th>
<th>Co</th>
<th>Mo</th>
<th>Rest</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiAl</td>
<td>50.3</td>
<td>–</td>
<td>1.8</td>
<td>1.9</td>
<td>44.9</td>
<td>1.1</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>K24</td>
<td>5.3</td>
<td>57.5</td>
<td>9.5</td>
<td>–</td>
<td>4.5</td>
<td>–</td>
<td>13.5</td>
<td>3.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Fig. 1 – (a) XRD pattern and (b) DSC curve of the Fe_{30}Co_{30}Ni_{15}Si_{15}B_{17} amorphous ribbon.

Fig. 2 – (a) Schematic of the assembled samples and (b) brazing process.

on the difference in micromorphology, the joint was divided into three different regions: Ni-based diffusion zone (marked with I), intermediate crystallization zone (marked with II) and TiAl-based diffusion and reaction zone (marked with III). In addition, since the phases formed in the interdendritic regions were different, the intermediate region II was then divided into two different part marked with i, ii. From the EDS compositional maps, it can be found that enrichments of Ti and Al were displayed in region IV while Co, Si displayed in the intermediate region. Ni and Fe were mainly distributed in region I. Because of the small radius of boron, its diffusion rate is high, as a result, the boron was uniformly distributed in the joint and the rapid diffusion of boron into base metals allows the filler metal and the parent metals to be quickly homogenized. The atomic diffusion distance of Ti and Al elements was about 30 μm. The element of Fe mainly diffused towards the Ni-based alloy and segregated in the interfacial region while the rest elements stayed in the intermediate region.

High magnification images of zone I, i, ii, III marked in Fig. 3(a) were shown in Fig. 4 and corresponding composition of each spot in Fig. 4 is listed in Table 2. It can be seen from Fig. 4(a) that some white smallish strip-like and little blocky phases (marked with A and B) were formed in the interfacial region of Ni-based alloy. According to the Hall-Petch equation, the finer of the phases, and the stronger of the materials. The EDS results show that the two phases were enriched in Fe, Ni, elements. Based on the analysis of XRD pattern of region I in Fig. 5 and Fe-Ni binary phase diagram [28–32], the two phases were determined to be γ-(Fe, Ni) phases. The phases in the intermediate region were mainly derived from the crystallization of the amorphous. Under the effect of constitutional supercooling, the dendritic phases were firstly formed and as the temperature decreased, two different fine phases grey dendritic phase and white smallish phases were then formed in the interdendritic regions as shown in Fig. 4(b) and (c). According to the EDS results of region C and D, the contents of Fe and Co were high. Based on XRD pattern of the intermediate region and Fe-Co binary phase diagram [33,34], the two different phases were detected to be (Fe, Co, Ni)_{23}B_{6} and α’-Fe (Co Ni) respectively [35]. The EDS result of region E exhibited that the phase was the same with D. To identify the phase in interdendritic region marked with white oval circle in Fig. 4(c), TEM and SAED analysis were performed. Fig. 6 shows the high-angle annular dark field (HAADF) transmission electron microscopy image and the SAED of three different region marked with H, I and J. According to the SAED patterns, the three phases
Fig. 3 – (a) Microstructure of the joint brazed at 1020 °C for 15 min; corresponding EDS mapping of the joint.

Table 2 – EDS compositional analysis (at. %) of different positions in Fig. 4.

<table>
<thead>
<tr>
<th>Region</th>
<th>Ti</th>
<th>Al</th>
<th>Ni</th>
<th>Fe</th>
<th>Si</th>
<th>B</th>
<th>Co</th>
<th>Probable phases</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.09</td>
<td>0.45</td>
<td>9.32</td>
<td>86.31</td>
<td>1.75</td>
<td>0.09</td>
<td>1.21</td>
<td>γ' (Fe, Ni)</td>
</tr>
<tr>
<td>B</td>
<td>0.21</td>
<td>0.4</td>
<td>9.84</td>
<td>43.51</td>
<td>0.58</td>
<td>4.95</td>
<td>39.92</td>
<td>γ' (Fe, Co Ni)</td>
</tr>
<tr>
<td>C</td>
<td>0.02</td>
<td>2.41</td>
<td>20.81</td>
<td>27.61</td>
<td>4.4</td>
<td>16.15</td>
<td>28.69</td>
<td>(Fe, Co, Ni)\textsubscript{2}B\textsubscript{6}</td>
</tr>
<tr>
<td>D</td>
<td>0.31</td>
<td>0.89</td>
<td>9.84</td>
<td>43.51</td>
<td>0.58</td>
<td>4.95</td>
<td>39.92</td>
<td>γ' (Fe, Co Ni)</td>
</tr>
<tr>
<td>E</td>
<td>37.52</td>
<td>10.5</td>
<td>12.96</td>
<td>13.34</td>
<td>5.25</td>
<td>23.38</td>
<td>17.06</td>
<td>(Fe, Co, Ni)\textsubscript{2}B\textsubscript{6}, γ, TiCo</td>
</tr>
<tr>
<td>F</td>
<td>17.36</td>
<td>36.38</td>
<td>15.64</td>
<td>8.23</td>
<td>0.04</td>
<td>0</td>
<td>7.75</td>
<td>γ\textsubscript{2}-Al\textsubscript{3}NiTi\textsubscript{2}</td>
</tr>
</tbody>
</table>
were determined to be face-centered cubic (Fe,Co,Ni)$_{23}$B$_6$, α' and tetragonal Fe$_2$B respectively. The phases in zone III came from two different modes: diffusion and reaction. It can be seen from Fig. 4(d) that the diffusion of Ti and Al into the filler caused the formation of a dark grey layer, in which a series of orientated bulky phases formed. According to A.Takeuchi, the mixing enthalpy between Ti-Fe, Ti-Co, Ti-Ni are −17 KJ/mol, −28 KJ/mol, −35 KJ/mol [36]. The mixing enthalpy between Al-Fe, Al-Co, and Al-Ni are −35 KJ/mol, −38 KJ/mol, and −40 KJ/mol respectively. Hence, the Ti and Al will firstly react with Ni forming $\gamma_3$ phase. From the EDS result, it can be seen that region F contained almost every elements in the filler metal and base materials and the content of each element was no less than 5at%. The mixing entropy can be calculated by [37]:

$$S_{mix} = -R \sum_{i=1}^{n} c_i \ln c_i$$

where $c_i$ is the concentration $\sum_{i=1}^{n} c_i = 1$, R is the gas constant. According to the EDS results, the mixing entropy in region F was calculated to be 14.308 J$^{-1}$·mol$^{-1}$ while the mixing entropy in the base metals was 3.921 J$^{-1}$·mol$^{-1}$. Under the effect of the high mixing entropy, the atomic diffusion during the solidification was slow, as a result, the phases in the reaction layer was fine. Combining the XRD pattern III, the F was detected to be mixed to be (Fe, Co, Ni)$_{23}$B$_6$, τ, TiCo and G was determined to be $\gamma'_3$.

The scanning electron microscopy images of the joints brazed at 990°−1080° is shown in Fig. 7. Obviously, the reaction between the filler metal and the base metals was not sufficient when the temperature was 990°. It was mainly because in the process of heating, the successive diffusion of Ti and Ni from the base metals into the filler metal led to the increase of melting point of the filler metal. Therefore, the brazing needed a higher temperature. When the temperature was 1020°, the thickness of region III was only about 50 μm. As the brazing temperature increased from 1020° to 1050°, the thickness of region III reached approximately 100 μm. Whereas, there was no obvious change in the interfacial microstructure of Ni-based alloys. The thickness of region I kept almost unchanged as the temperature increased. When the temperature increased to 1080°, the zone III continued to grow into the filler metal and dendritic phases in the intermediate region decreased as shown in Fig. 7(d). Although the joints exhibited
different interfacial microstructure, there was no new phases forming in the joints as the brazing temperature increased.

The joining and phase formation process can be described as follows: In the initial stage, the temperature was low. The oxide films in the base alloys were broken by the intermediate layer under the effect of the applied force. The assembled samples obtained a preliminary contacting from the compression molding and creep of the filler metal. When the temperature increased, the atoms in the base alloys and filler metal were activated and then entered a new equilibrium position through interfacial diffusion and volume diffusion. When the temperature rose to the melting point of the filler metal, the filler metal melt; under the effect of the concentration gradient between the filler metal and the base metals, the elements of Fe, Co, Si, B diffused into the base alloys, causing the formation of γ-(Fe, Ni) phase in the interface of Ni-based alloys; simultaneously, the Ti, Al and Ni elements in the base metals also diffused into the molten filler and metallurgically reacted with it, forming the blocky η phases adhering to the interface of TiAl-based alloy and fine mixed phases next to the blocky phase. Therefore, the joint was further subdivided into five different regions: The Ni-side diffusion layer (DL), the intermediate crystallization layer (CL), the TiAl-side reaction layer (RL) and the TiAl-side diffusion layer (DL), as illustrated in Fig. 8. The fine phases formed in the interfacial regions and interdendritic regions were beneficial to the joint properties.

Fig. 9 shows the growth kinetics curves of the interfaces of TiAl and Ni. It can be found that the thickening of the interfaces obeys parabolic law $H = kt^{\frac{1}{2}} + b$. Where, $k$ is rate constant; $H$ and $b$ are original and final thickness. According to the experimental results, the formulas fitted are $H = 3.55t^{\frac{1}{2}} + 19.3, H = 1.57t^{\frac{1}{2}} + 9.82$ for TiAl and Ni respectively. The atomic diffusion mechanism is different for TiAl And Ni-based alloys. The atomic diffusion in TiAl-based alloy is easier than in the Ni-based alloy for the difference in the atomic radii of Ti and Al will cause lattice distortion of TiAl-based alloy, decreasing the activation barriers for atomic diffusion.

Fig. 10 shows the microstructure of the joint after holding for 1 h at 750°C. It can be seen from the picture that the seam became wider and the Ni-based diffusion layer was thicker than its initial state. But the grain size of the white γ-(Fe, Ni) phases in the Ni-based diffusion layer and (Fe, Co, Ni)$_{23}$B$_6$, γ, TiCo and γ$_3$-Al$_2$NiTi$_2$ phases in the TiAl-based reaction layer and diffusion layer kept almost unchanged, indicating that the
Fig. 7 – Effect of brazing temperature on microstructure of the joints: (a) 990 °; (b) 1020 °; (c) 1050 ° and (d) 1080 ° for 15 min.

Fig. 8 – Schematic of the brazing process.
joint brazed utilizing the Fe₃₀Co₃₀Ni₁₅Si₈B₁₂ can work for a long time at high temperatures without grain coarsening.

Fig. 11 exhibits the hardness distribution in different positions of the joint brazed at 1020◦C for 15 min. As can be seen from the picture that the bulky γ-TiAl and α-Fe (Co, Ni) phase had higher hardness than others while the hardness of the nanoscale phases: γ(Fe, Ni) and τ, τ₃, TiCo in the interfacial regions of Ni- and TiAl-based alloys was higher than the base metals. Cai et al. have reported that the reaction phases with high hardness usually exhibits a low plastic deformation capability [38]. The residual stress could be released through plastic deformation and it was beneficial to the joint property. The formation of the fines phases was envisaged to improve the strength and plasticity of the joint.

Fig. 12 shows the shear strength of the joint tested at 30◦C and at 750◦C. When the processed parameters were 990◦C/15 min, both room temperature and high-temperature shear strength were very low only about 150 MPa, 80 MPa respectively. The main reason is that the low brazing temperature resulted in an insufficient reaction between the molten filler metal and the base alloys. When the joining temperature increased to 1020◦C, maximum shear strength approximately 363 MPa at 30◦C and 255 MPa at 750◦C were achieved, which indicated that increasing the brazing temperature can effectively facilitate the diffusion of atoms and enhance the metallurgical reaction. However, further increasing the brazing temperature did not increase the shear strength of the joint as expected. When the joining parameters were 1050◦C/15 min, the room-temperature and high-temperature shear strength decreased to 290 MPa and 225 MPa, respectively.

Fig. 13 illustrated the relationship between the serving temperature and strength. A parameter Tₜw = Tₛ/Tₚ was introduced, where the Tₛ is the applying temperature, Tₚ is the welding temperature and in general Tₚ = Tₛ + 30−50◦C. The
higher of the $T_{aw}$ and strength of the joint denoted the better performance of the joint at elevated temperatures. Fig. 13 compared the high-temperature of previously reported TiAl/Ni joint brazed utilizing Ag-based and Zr-based filler with current work [6,10,24]. As can be seen from the picture that the shear strength of the joint brazed with Ag-based filler decreased with the increasing of $T_{aw}$. The shear strength and $T_{aw}$ value of the joint brazed with the FeCoNiSb filler were far greater than the joint brazed by Ag-based filler metal. This mainly because the high-temperature property of the Fe-based filler metal is better than the Ag- and Zr-based filler metals.

The fracture mechanism was discussed after shearing tests. The fracture morphology of the room temperature and 750$^\circ$ of the joint brazed at 1020$^\circ$ for 15 min were shown in Fig. 14. Fig. 14(a) shows the crack surface tested at room temperature and the fracture exhibited a cleavage facet feature. According to the EDS result of spot A and B listed in Table 3, it revealed that the dendritic $\alpha'$ dominated the fracture. Fig. 14(b) shows the fracture morphology of the joint tested at 750$^\circ$. During testing, the high testing temperature coarsened the $\gamma$-TiAl phases, as a result, the fracture occurred near to the interface of TiAl-based alloy with the filler metal. High-density short and curved tearing lines and big fracture facets can be observed in Fig. 14(b). The EDS analysis of spot C and D indicated that the cracks initiated form the bulky $\gamma$-TiAl phases and expanded across region IV.

### 4. Conclusions

The TiAl-based alloy and Ni-based superalloy were successfully brazed with Fe$_{23}$Co$_{10}$Ni$_{15}$Si$_{5}$B$_{17}$ amorphous ribbon as the filler metal. The relationship between microstructure and the mechanical properties at 30$^\circ$ and 750$^\circ$ of the joints were studied. Some main conclusions were listed as follows:

1. The typical microstructures of the joint from the Ni-based superalloy to the TiAl-based alloy brazed at 1020$^\circ$ for 15 min were determined to be $\gamma$-(Fe, Ni)/ (Fe, Co, Ni)$_{23}$B$_6$, $\alpha'$-(Fe-Co Ni)/ (Fe, Co, Ni)$_{23}$B$_6$, $\gamma$, TiCo /$\gamma_2$.

2. The shear strength tested at 30$^\circ$ and 750$^\circ$ firstly increased and then decreased with the increase of brazing temperature. The maximum shear strength of the joint tested at 30$^\circ$ and at 750$^\circ$ were 363 MPa and 252 MPa when the joint was brazed at 1020$^\circ$ for 15 min.

3. The microstructure of the joint was determined by the interfacial atomic inter-diffusion between the molten filler metal and base metals as well as the crystallizing of the residual amorphous filler. The formation of the fine grains in the interdendritic region and in the interfacial regions was determined to be the main reason for the good mechanical property both at room temperature and at high temperature.

### Conflict of interest

The authors declare no conflict of interest.

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### Data availability

All data included in this study are available upon request by contact with the corresponding author.
Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.jmrt.2019.12.028.

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