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Interfacial microstructure and mechanical characterization of silicon nitride/nickel-base superalloy joints by partial transient liquid phase bonding

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Abstract

Si_3N_4 ceramic has been joined successfully to nickel-based superalloy by partial transient liquid phase (PTLP) bonding using Ni/Cu/Ti multi-interlayers. The interfacial microstructure and strength of the joints were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and a three-point bending test. According to the TEM analysis, it was found that a TiN reaction layer was formed at the Si_3N_4/filler alloy interface. The TiN reaction layer was composed of two zones: one next to the Si_3N_4 ceramic with a thickness of about 0.4 μm and the other zone with grains of about 0.8 μm. The microstructure of the joint between the Si_3N_4 and the Cu interlayer can be described as: TiN layer with fine grains/TiN layer with coarse grains/Ti_2Ni layer. Ni_3Ti and $CuTi_2$ phases were produced at the interface between the Cu interlayer and the DZ483 superalloy. The room-temperature average strength of the joint was 147 MPa, and 96 MPa was achieved when the joints were tested at 1073 K.
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1. Introduction

Ceramics, especially silicon nitride, have been widely used in the field of the aerospace because of their excellent thermal and oxidation resistance as well as outstanding high-temperature mechanical properties. Low fracture toughness and poor machinability of Si_3N_4, however, are limiting its massive practical engineering applications [1–4]. A promising method to overcome the limitation is combining the ceramics with metallic component, taking advantage of their superior properties [5,6]. Therefore, the production of such component requires the development of joining technologies.

In the last few decades, various techniques have been developed to join ceramics to themselves or to metals. Among all the joining methods, those involving liquid phase formations such as active brazing and partial transient liquid phase (PTLP) bonding are receiving more attention [7,8]. For joining Si_3N_4 to superalloy, active brazing, particularly with Ag–Cu–Ti active brazing alloy has been widely used and considered as one of the most effective methods. However, the maximum service temperature of the joint brazed with Ag–Cu–Ti active brazing alloy is lower than 773 K [9].

Partial transient liquid phase bonding, which is combination of brazing and diffusion bonding, can be used to fabricate complex shape parts at lower temperature than operational temperature [10–14]. Thus, it has received more attention to join nonmetallic materials and dissimilar materials. A considerable research work has already been focused on the PTLP bonding of Si_3N_4 ceramic. Iino [15] first proposed partial transient liquid phase bonding technique of ceramic. PTLP bonding of Si_3N_4 ceramic with NiCr/Au, NiCr/Cu [16], and Au/Ni–22Cr [17] interlayer have also been investigated. Ti/Cu/Ni [18], CuTi/Pd, and Ti/Ni/Ti multilayer [19,20] have been used as interlayer in PTLP bonding of Si_3N_4 ceramic. However, few researches concerning the high-temperature properties of Si_3N_4

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ceramic–superalloy joints bonded through the partial transient liquid phase process were reported up to now.

In addition, deeply understanding of the interfacial microstructure is a key issue since the properties of the joints depend on the microstructure. Nevertheless, most of the microstructure analysis in ceramic–metal joints bonded by PTLP bonding method has been limited to SEM analysis in conjunction with electron probe microanalysis techniques [18]. To identify the reactive phase formation at the interface and to clarify its formation mechanism, TEM analysis of the reaction layers is important.

The present study is aimed at analyzing the interfacial microstructure of the joint and evaluating the high-temperature properties of bonding silicon nitride to a nickel-based superalloy by PTLP bonding process with Ni/Cu/Ti multi-interlayer. The interfacial microstructure of the joint was investigated in detail by SEM and TEM. The bending strength of the joint was tested at room temperature and elevated temperatures.

2. Experimental procedure

Hot pressed Si_3N_4 ceramic (Institute of Ceramics, Shanghai, China) containing alumina and yttria as sintering additive was used in this study. The bending strength of Si_3N_4 ceramics at room temperature was 750–850 MPa. The nickel-base superalloy used was DZ483 (12.10 wt% Cr, 9.18 wt% Co, 5.06 wt% Ta, 4.03 wt% Ti, 3.76 wt% W, 3.52 wt% Al, 1.91 wt% Mo, 0.026 wt% B, Ni bal). The coefficient of thermal expansion (CTE) of Si_3N_4 is 3.1×10^{-6}/°C and the CTE of nickel-base superalloy was 12.8×10^{-6}/°C. The ceramic and superalloy partners were cut into rectangular specimens 3 mm × 4 mm × 19 mm in dimension. Joining was performed on the 3 mm × 4 mm face. The bonding surfaces of the samples were coarsely ground on SiC sandpapers and then polished using 1 μm diamond paste. A 20 μm thick Ti foil and a 200 μm thick Cu foil were used as the outer layer. A 0.5 mm thick Ni sheet was used as the core material. Prior to joining, all the materials were cleaned in acetone and ethanol using ultrasonic vibration for about 15 min, respectively.

Fig. 1 shows a schematic illustration of the multiple interlayer joint investigated. A sandwich-like bonding couple of Si_3N_4/Ti/Cu/Ni/Cu/Ti/DZ483 was placed in a graphite die of coating boron nitride. A uniaxial pressure was applied to maintain the intimate contact of the samples during the thermal cycle. The bonding process was carried out at 1323 K for 60 min in a furnace with a vacuum of about 1.0×10^{-2} Pa. The heating rate was 8 °C min^{-1} and cooling of the joint was performed at 4 °C min^{-1}

The microstructure of the joint was investigated using a SEM equipped for electron dispersive spectroscopy (EDS).

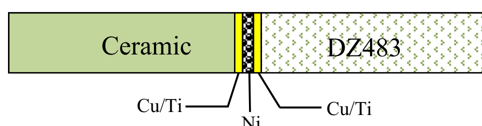

Fig. 1. Schematic illustration of the joint used in the present study.

Sample preparation from metal–ceramic joint for TEM by using conventional techniques is difficult. To overcome this problem, TEM samples were prepared by using a focus ion beam (FIB) technique. FIB prepared electron transparent samples were characterized by using 200 kV field emission gun-TEM attached with high angle annular dark field scanning transmission electron microscope (STEM-HAADF) detector. Chemical elements of the interfacial reaction layers were analyzed by STEM combined with EDS. The morphology and crystal structure of the interfacial reaction layer in the joint were analyzed by TEM.

After PTLP bonding, the specimens with dimension of 3 mm × 4 mm × 38 mm were obtained, which were used for bending tests. The flexural testing of the joints were carried out, both at room temperature and elevated temperatures, under a three-point-bend loading conditions using a cross-head speed of 0.5 mm min^{-1}. The data collected was plotted using Weibull statistics, an accepted procedure used in the evaluation of strength of ceramic materials [21,22]. Eleven samples were used to determine the bending strength of the joints for each joining condition in order to obtain Weibull data.

3. Results and discussions

Fig. 2 shows the interfacial microstructure of the Si_3N_4–DZ483 superalloy joint bonded using Ni/Cu/Ti multi-interlayer at 1323 K for 60 min. In Fig. 2a, the joint is well-bonded and voids are not observed along the interlayer and in the ceramic part. Fig. 2b shows the detailed microstructure between Si_3N_4 ceramic and Cu interlayer. In Fig. 2b, the dark region on the left is Si_3N_4 ceramic and the reaction layer are observed adjacent to Si_3N_4 ceramic. It can be seen that the region between Si_3N_4 ceramic and Cu interlayer consists of two different reaction layers: one is a thin reaction layer with a thickness of about 1.5 μm labeled A, and the other is a thick reaction layer labeled B in Fig. 2b. The compositions of positions A and B labeled in Fig. 2b were measured by EDS to identify these phases in the joint, as shown in Table 1. The composition analysis results shown in Table 1 indicate that the reaction layer A is mainly a compound of Ti and N, which cannot be confirmed exactly because the content of N cannot be determined accurately by means of the EDS; the reaction layer B contains 52.52 at% Ti, 24.87 at% Ni, 13.84 at% Si, 8.52 at% Cu. However, SEM equipped for EDS technique is not good enough to detect very small reaction layers and to quantify their compositions at the interfaces [23]. Therefore, the reaction layers need to be further confirmed.

Fig. 3 shows STEM-HAADF image and elemental line-scanning patterns in the active reaction region between the Si_3N_4 ceramic and Cu interlayer. The elemental distribution of Si, Ti, Ni, Cu and Y of the joint is measured along the yellow line in the Fig. 3a, and the results are shown in Fig. 3b. As seen from Fig. 3a, the two different reaction layers exist near to the Si_3N_4. One contains mainly Ti and N, the other is composed of Ti, Ni, Si and Cu. In Si_3N_4 ceramic zone, only Si, N and Y exist and no other element in the filler alloy can diffuse into the ceramic. Nevertheless, Fig. 3b reveals the diffusion

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