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Letter to the Editor

Interface characteristics of Al_2O_3-TiC/Cr18-Ni8 diffusion-bonded joint using a Ti/Cu/Ti multi-interlayer

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Abstract

Diffusion bonding Al_2O_3-TiC ceramic matrix composites with Cr18-Ni8 austenitic stainless steel has been performed with a Ti/Cu/Ti multi-interlayer. The Al_2O_3-TiC/Cr18-Ni8 joints were characterized by means of scanning electron microscope (SEM) with energy-disperse spectrometry (EDS), electron probe microanalysis (EPMA) and X-ray diffraction (XRD). The results indicate that the Al_2O_3-TiC/Cr18-Ni8 diffusion-bonded joint with shear strength of 106 MPa was obtained by using the Ti/Cu/Ti multi-interlayer. An interfacial transition zone was formed by diffusion reaction between Ti, Cu and the elements from the substrates, Al_2O_3-TiC and Cr18-Ni8. TiO, TiC, α-Cu, CuTi, FeTi and Cr_2Ti are produced near the interface of the Al_2O_3-TiC/Cr18-Ni8 joint.

K e y w o r d s: diffusion bonding, Al_2O_3-TiC ceramic matrix composites, Ti/Cu/Ti multi-interlayer, microstructure, XRD

1. Introduction

Al_2O_3-TiC ceramic matrix composites are potential substitutes for more traditional materials due to their high hardness, excellent chemical and mechanical stability under a broad range of temperatures [1, 2]. However, Al_2O_3-TiC ceramic matrix composites are brittle and difficult to machine which prevents their introduction as monolithic parts in engineering structures [3, 4]. In order to improve their reliability and to make up for their low workability, Al_2O_3-TiC ceramic matrix composites are most often utilized in combination with ductile metallic materials for structural applications. The diffusion bonding process has been one of the technological tools used in order to increase the use of ceramic materials [5–7]. However, less attention has been given to the joining of Al_2O_3-TiC ceramic matrix composites.

In previous study, diffusion bonding of Al_2O_3-TiC ceramic matrix composites to Q235 low carbon steel was successfully achieved [8]. In this study, diffusion bonding of Al_2O_3-TiC ceramic matrix composites with Cr18-Ni8 stainless steel was performed with the help of a Ti/Cu/Ti multi-interlayer in a vacuum. The shear strength of the Al_2O_3-TiC/Cr18-Ni8 joint was determined. The fracture morphology was observed and the compositions on the fractured face were further analyzed by electron probe microanalysis (EPMA). The interfacial microstructure was characterized by means of scanning electron microscope (SEM) with energy-disperse spectrometry (EDS). The phase constitution near the Al_2O_3-TiC/Cr18-Ni8 interface was analyzed by means of X-ray diffraction (XRD). The preparation of ceramic-metal joints and knowledge of their properties may broaden the application of ceramic-matrix composites.

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2. Experimental

The materials used were Al_2O_3-TiC ceramic matrix composites and Cr18-Ni8 austenitic stainless steel. Al_2O_3-TiC ceramic was made by hot pressure sintering (HPS) to a final circle plate having dimensions ø 52 × 3.5 mm^2. The size of Cr18-Ni8 circle plate specimen is ø 52 × 1.2 mm^2. Al_2O_3-TiC ceramic consists of Al_2O_3 matrix with TiC particles. Chemical composition (wt.%) of Al_2O_3-TiC is 65.6 % Al_2O_3 and 34.4 % TiC. Chemical composition (wt.%) of Cr18-Ni8 steel is C $\leq$ 0.12 %, Si $\leq$ 1.00 %, Mn $\leq$ 2.00 %, P $\leq$ 0.035 %, S $\leq$ 0.030 %, Ni 8.00–11.00 %, Cr 17–19 % and Fe balance. The multi-interlayer of Ti/Cu/Ti at 60 μm thickness was employed to promote the joining between Al_2O_3-TiC ceramic and Cr18-Ni8 stainless steel and obtain tight metallurgic joint.

The vacuum diffusion bonding equipment (Workhorse-II manufactured by Vacuum Industry Company) was used in the test. The heating power was 45 kW. Before diffusion bonding, the surfaces to be joined were ground with abrasive paper, and cleaned by immersing in acetone. After that, the diffusion bonding plates were overlapped and placed into a vacuum chamber. Technological parameters used were: the heating temperature T = 1323–1433 K, the pressure P = 12–18 MPa, the holding time t = 45–90 min and the vacuum degree of 1.33 × 10^{-4}–10^{-5} Pa. The cooling process was conducted in a vacuum chamber that was cooled by circuit water. When the chamber temperature was cooled to 373 K, the Al_2O_3-TiC/Cr18-Ni8 diffusion bonded joints were taken out from the chamber.

The interfacial strength was measured by mechanical (shear) testing using a special device. The nominal strain rate was 5 × 10^{-3} min^{-1}. The shear strength was calculated by the load at the fracture divided by the nominal area of the joint. Fracture morphology was observed and the compositions on the fractured face were analyzed by JXA-8800R EPMA. For metallographic examination, the bonded specimens were cut transversely through the bond and mounted in bakelite. The Al_2O_3-TiC/Cr18-Ni8 joints were etched by a 3 : 1 solution of HCl and HNO_3 for 10 s. Interfacial microstructure was observed by JXA-840 SEM and the chemical compositions across the joint were determined by EDS. The phases formed in the joint were further determined with D/MAX-RC diffractometer.

3. Results and discussion

3.1. Shear strength and fracture

In order to evaluate the mechanical properties for the Al_2O_3-TiC/Cr18-Ni8 joint, the interfacial shear strength was measured with a WEW-600E test machine. The relationship between load and displacement is shown in Fig. 1. As can be seen from Fig. 1, when the maximum load is 10.6 kN, the Al_2O_3-TiC/Cr18-Ni8 joint is destroyed. The shear area of the specimen measured is 100 mm^2. So the maximum load 10.6 kN, divided by the shear area 100 mm^2, results in shear strength of 106 MPa. When the joint is destroyed, the load decreases suddenly. This suggests that fracture of the Al_2O_3-TiC/Cr18-Ni8 joint is a brittle fracture.

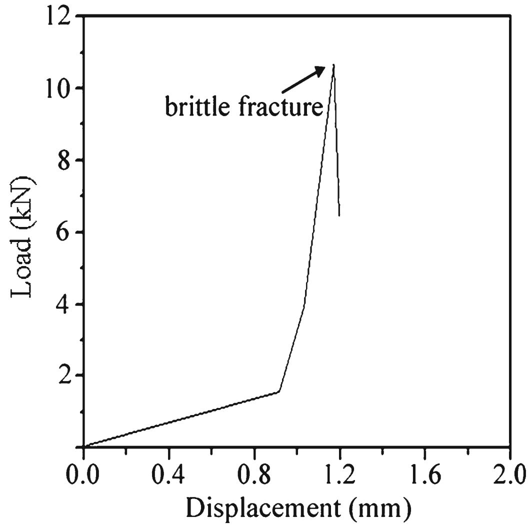

Fig. 1. Relationship between load and displacement in shear strength measurement.

After strength testing, the fracture faces of joints were analyzed by EPMA. The results of fracture morphology and composition analysis are shown in Fig. 2. According to Fig. 2a, the fracture surface shows cleavage steps. The fracture surface does not show metallic shine and fracture color is consistent with that of Al_2O_3-TiC ceramic composites. According to Fig. 2b, the fracture face (the black circle zone in Fig. 2a) consists of Al, O and Ti. It reveals that the Al_2O_3-TiC/Cr18-Ni8 joint fractured within the Al_2O_3-TiC ceramic. It suggests that the Ti/Cu/Ti multi-interlayer tightly combines with the substrates, Al_2O_3-TiC and Cr18-Ni8, producing the strong metallurgical joint.

3.2. Microstructural characteristics

Microstructure characteristics of the diffusion bonded Al_2O_3-TiC/Cr18-Ni8 joint are shown in Fig. 3. An obvious interfacial transition zone was formed between Al_2O_3-TiC ceramic composites and Cr18-Ni8 steel, see Fig. 3a. In the process of diffusion bonding, complex diffusion reactions must have occurred between the Ti/Cu/Ti multi-interlayer and the base materials, Al_2O_3-TiC and Cr18-Ni8, to produce new mi-

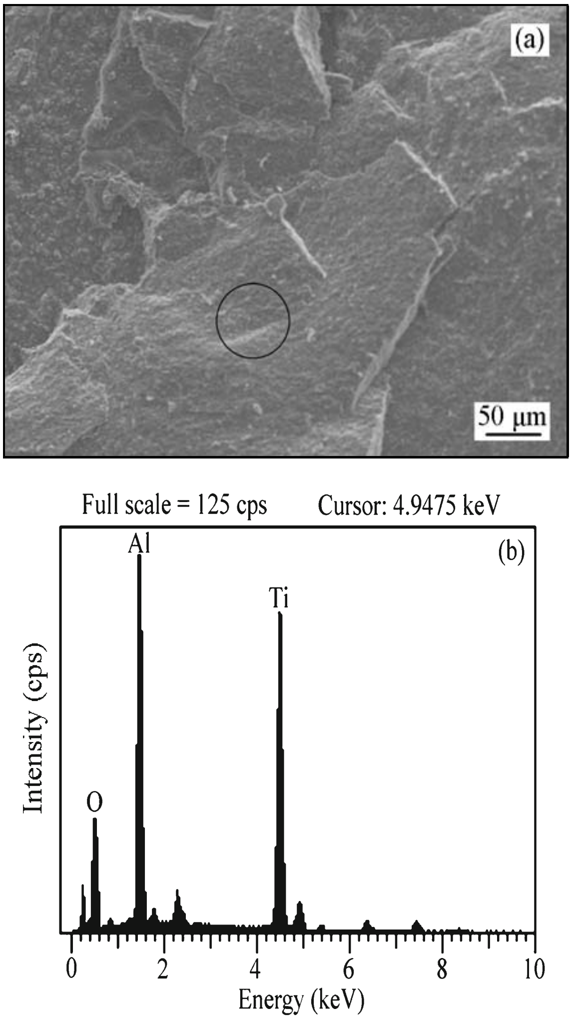

Fig. 2. Fracture morphology and composition analysis of Al_2O_3-TiC/Cr18-Ni8 joint: (a) fracture morphology, (b) composition analysis (the black circle in Fig. 2a).

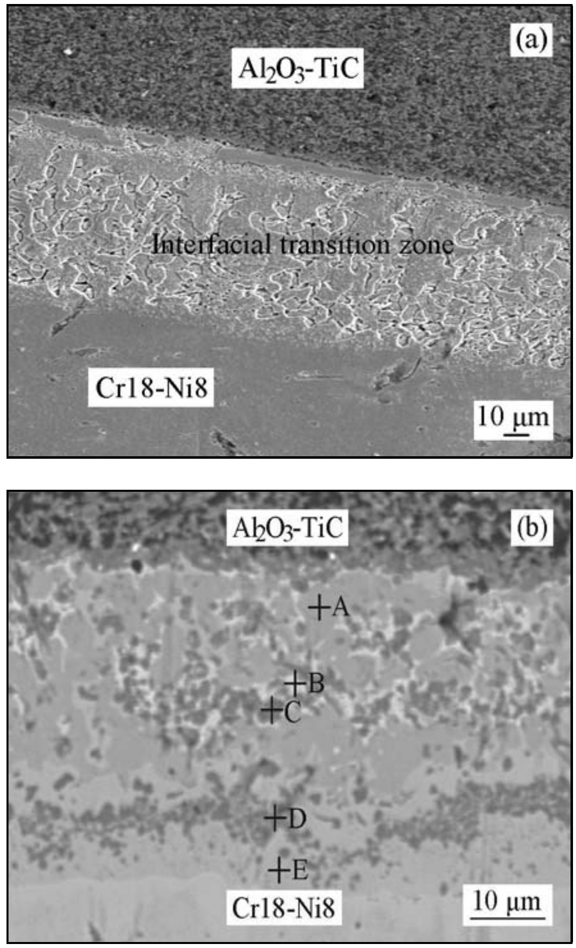

Fig. 3. Microstructure characteristics of diffusion bonded Al_2O_3-TiC/Cr18-Ni8 joint: (a) interfacial transition zone (SEM), (b) backscattered electron image.

crostructure in the interfacial transition zone. Backscattered electron image of the interfacial transition zone is shown in Fig. 3b.

Microstructure in the interfacial transition zone is various and they have clearly different backscattered electron images from that in Al_2O_3-TiC composites and Cr18-Ni8 steel. In order to clarify the microstructural characteristics in the interfacial transition zone, the compositions of various microstructures labeled by A, B, C and D were measured by EDS and the results are shown in Table 1.

Near the Al_2O_3-TiC side, the gray block microstructure labeled by A is enriched with O and Ti with a small quantity of Ni, Cu and Cr; hence, the composition indicates that the phases may be the oxide of titanium. At the middle of the interfacial transition zone, the white reticular microstructure labeled by B consists of Cu, O and Ti in association with Al, Ni and Cr; accordingly, which is possibly residual Cu existed as α-Cu, Cu-Ti intermetallic compounds and other reaction products which can not be identified by composition. The black particles are surrounded by the white microstructure labeled by B, which mainly contain Ti and C and they are presumably TiC. Near the Cr18-Ni8 steel, the black particles labeled by D are aggregated on the bright gray matrix labeled by E and enriched with Ti and C, the ratio of Ti and C is about 1 : 1; therefore, they are TiC. Near the Cr18-Ni8 steel, the bright gray microstructure labeled

Table 1. Composition of phases analyzed by EDS (at.%)

Location	Al	O	Ti	Cu	Ni	Cr	Fe	C
A	0.59	60.82	27.90	2.71	4.40	2.91	–	–
B	4.95	14.34	8.09	68.46	2.34	1.03	–	–
C	–	–	54.88	2.35	–	–	0.36	42.40
D	–	–	48.70	1.30	0.40	0.45	2.57	46.58
E	0.98	–	34.31	–	–	7.83	54.88	–

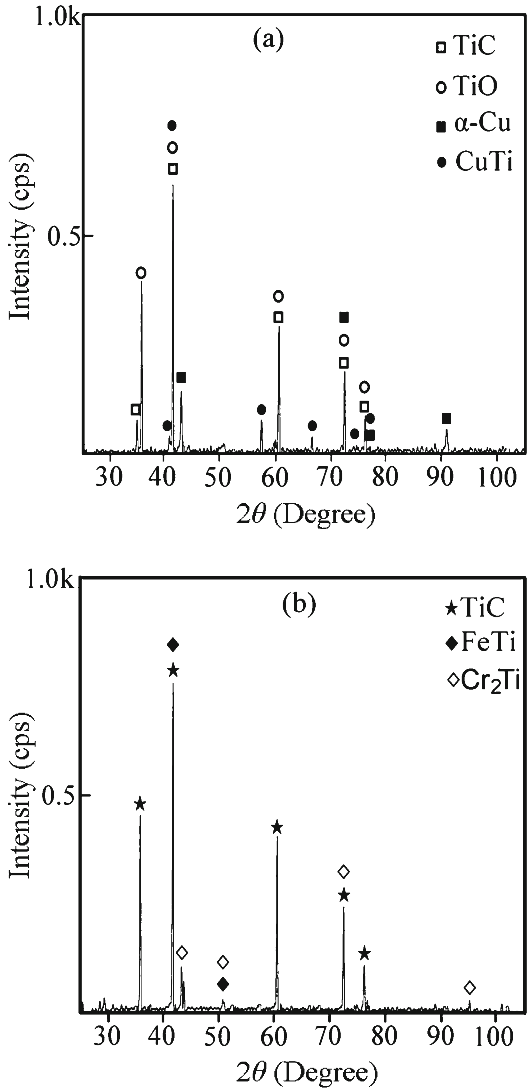

Fig. 4. XRD patterns of Al_2O_3-TiC/Cr18-Ni8 interface: (a) near Al_2O_3-TiC, (b) near Cr18-Ni8.

by E mostly consists of Fe, Ti and Cr; consequently, the composition reveals that they are probably the mixture phases of γ-Fe, Fe-Ti and Cr-Ti intermetallic compounds.

3.3. XRD analysis

The phase constitution near the Al_2O_3-TiC/Cr18--Ni8 interface was further analyzed by means of XRD. The XRD analysis was carried out with copper target under working voltage 60 kV and working current 40 mA. The XRD results near the Al_2O_3-TiC side and near the Cr18-Ni8 side are shown in Fig. 4.

According to Fig. 4a, TiO, TiC, α-Cu and CuTi exist at the interface near the Al_2O_3-TiC side. There was Al_2O_3 existed near the Al_2O_3-TiC side and Al_2O_3 was omitted in Fig. 4a. In the process of diffusion bonding, Cu and Ti in the Ti/Cu/Ti multi-interlayer forms a Cu-Ti liquid phase. When the Cu-Ti liquid phase contacts Al_2O_3-TiC ceramic, Ti segregates from the liquid phase to the Al_2O_3-TiC surface due to its great affinity to O [9]. Ti segregated reacts with Al_2O_3 by redox reaction resulting in local replacement of Al_2O_3 with TiO. This metallic-type oxide TiO [10] layer formed adjacent to Al_2O_3-TiC provides a smoother transition in the chemical bond from an ionocovalent bond in Al_2O_3-TiC, to a metallic bond in Cr18-Ni8. The TiC phase detected by XRD possibly derived from either the Al_2O_3-TiC substrate or the reaction between Ti and C in the liquid phase. In the Cu-Ti liquid phase, some Cu reacts with Ti forming CuTi intermetallic compounds, some Cu remains without any reaction exiting as α-Cu acting as a stress releasing media [11]. According to Fig. 4b, TiC, FeTi and Cr_2Ti exist at the interface near the Cr18-Ni8 side. There is γ-Fe exited near the Cr18-Ni8 side and γ-Fe was omitted in Fig. 4b. Ti in the Cu-Ti liquid phase is also attracted by the Cr18-Ni8 steel due to presence of C and Fe [12]. Ti combines with C, Fe and Cr from Cr18-Ni8 forming TiC, FeTi and Cr_2Ti compounds near the Cr18-Ni8 side. It is found that the XRD results are in agreement with EDS in the interfacial transition zone.

4. Conclusions

1. Diffusion bonding Al_2O_3-TiC ceramic matrix composites with Cr18-Ni8 austenitic stainless steel has been successfully performed with a Ti/Cu/Ti multi--interlayer. The shear strength of the Al_2O_3-TiC/Cr18-Ni8 joint reaches 106 MPa. The shear fracture morphology of the Al_2O_3-TiC/Cr18-Ni8 joint clearly reveals a brittle fracture and the joint fractures locating mostly in the Al_2O_3-TiC ceramic near the interface.

2. During the diffusion bonding, diffusion reaction occurs between Ti, Cu and the elements of the substrates, Al_2O_3-TiC and Cr18-Ni8. A clear interfacial transition zone is formed between Al_2O_3-TiC and Cr18-Ni8. By means of EPMA and XRD analysis, TiO, TiC, α-Cu, CuTi, FeTi and Cr_2Ti are determined in the interfacial transition zone.

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