Bull. Mater. Sci., Vol. 29, No. 2, April 2006, pp. 155–158. © Indian Academy of Sciences.

Microstructural characterization in diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint with Ti interlayer

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MS received 10 October 2005; revised 26 December 2005

Abstract. Ceramic matrix composite, TiC–Al_2O_3, and stainless steel, Cr18–Ni8, were joined at 1400 K by solid state diffusion bonding, making use of a Ti foil acting as thermal stress relief interlayer. The microstructure of the joint was thus formed. The diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint was investigated by a variety of characterization techniques such as scanning electron microscope (SEM) with energy dispersion spectroscopy (EDS) and X-ray diffraction (XRD). The results indicate that Ti foil is fully fused to react with elements from substrates and Ti_3Al, TiC and α-Ti are formed in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint. The interfacial shear strength is up to 99 MPa and the shear fracture occurs close to the ceramic matrix composite due to the application of Ti foil acting as thermal stress relief interlayer.

Keywords. Microstructural characterization; TiC–Al_2O_3/Cr18–Ni8 joint; diffusion bonding; Ti interlayer.

1. Introduction

Ceramic matrix composite, TiC–Al_2O_3, has unique characteristics such as high hardness, chemical stability, good strength and toughness at elevated temperature and excellent wear resistance (Atong *et al* 2004; Brochu *et al* 2004). Especially, it is most suitable for high speed cutting tool used in difficult machinable materials. Metal/ceramic joining technologies play very important role in various aspects of the electronics and high-temperature component industries (Krugers *et al* 1992; Deng *et al* 2005). If the joining of ceramic matrix composite, TiC–Al_2O_3, and stainless steel can be successfully realized, it will open a new way to broaden the application of TiC–Al_2O_3 in machine manufacturing. But there has not been any report on the joining of ceramic matrix composite, TiC–Al_2O_3, even with stainless steel.

Diffusion bonding is an advantageous technique for producing metal/ceramic joint since high-temperature mechanical resistance and defect-free interfaces can thus be achieved (Passerone *et al* 2000; Li *et al* 2002). However, one of the shortcomings of diffusion bonding is the high temperature needed for the bonding operation, which causes the development of high thermal stresses across the interface and fast growth of often undesirable reaction products. Usually, the residual stress that originates from different thermal contractions of the ceramics and the metal are minimized by the addition of interlayer, which must be ductile and suitably reactive to both metal and ceramics in order to efficiently relieve the above-mentioned thermal stresses (Kliauga *et al* 2001; Rice *et al* 2002).

In the present work, ceramic matrix composite, TiC–Al_2O_3, and stainless steel, Cr18–Ni8, were joined at 1400 K by solid state diffusion bonding using a Ti foil as stress relief interlayer. The microstructure in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint was observed by means of scanning electron microscope (SEM) with energy dispersion spectroscopy (EDS) and X-ray diffraction (XRD). The interfacial shear strength and the fracture morphology were investigated by test machine and the compositions in fractured face were determined by EDS.

2. Experimental

Ceramic matrix composite, TiC–Al_2O_3, made by hot pressure sintering (HPS) to a final circle plate was diffusion bonded to stainless steel, Cr18–Ni8, both having dimensions Φ 52 × 3·5 mm. A Ti foil with 50 μm thickness was employed as thermal stress relief interlayer. Bonding was performed at 1400 K for 1h with a pressure of 15 MPa and a vacuum 10^{-5} Pa. The cycle of diffusion bonding is shown in figure 1.

Metallographic cross-sections were prepared and etched with a solution consisting of 95% nitric acid and 5% alcohol for 150 s. The microstructural observations were performed by optical microscope (OM) and JXA-840 scanning electron microscope (SEM). The chemical composition across the joint was determined by energy dispersion spectroscopy (EDS). Further, the phases formed in the

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joint were studied with D/MAX-RC diffractometer. Finally, interfacial shear strength was measured using CMT 5105 test machine and the schematic graph in shearing is shown in figure 2.

3. Results and discussion

3.1 *Microstructure*

Figure 3 shows the microstructural characterization in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint. There is an obvious interfacial layer formed between composite, TiC–Al_2O_3, and steel, Cr18–Ni8, by optical microscope (OM), seen in figure 3a. The total thickness of the interfacial layer is measured to be about 90 μm. The microstructure in the interfacial layer is shown in figure 3b.

Ti foil was both fused fully and bonded to the composite, TiC–Al_2O_3, and stainless steel**,** Cr18–Ni8. The different regions can be recognized and labeled as I, II and III in figure 3b, starting from the TiC–Al_2O_3/Ti interface and moving to the right, i.e. into the Ti and the Ti/Cr18–Ni8 interface: I is a reaction layer at the TiC–Al_2O_3/Ti interface; II is a non-transformed region of *a*-Ti; III is a reaction layer at the Ti/Cr18–Ni8 interface and its width is about 50 μm. The microstructures in the above mentioned three regions were different due to the fusion of Ti foil and the diffusion of elements from TiC–Al_2O_3 and Cr18–Ni8.

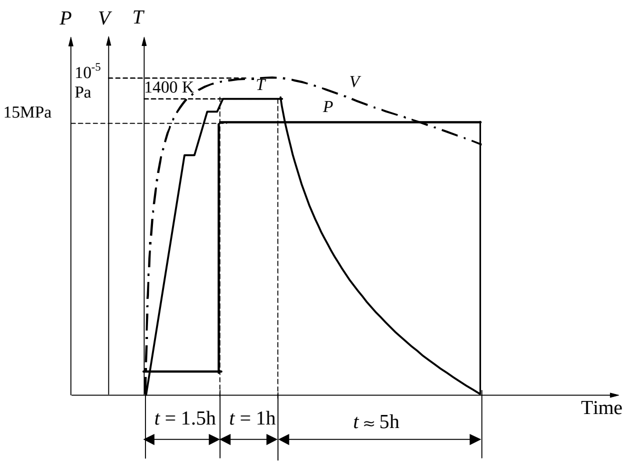

Figure 1. Cycle of diffusion bonding of TiC–Al_2O_3 with Cr18–Ni8.

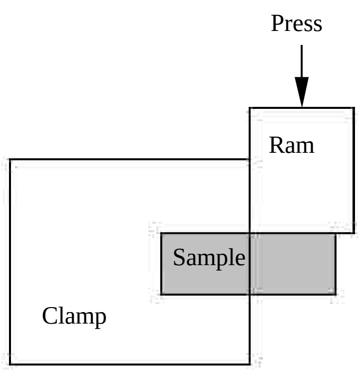

Figure 2. Schematic graph in shearing.

The backscattered electron image of the bonded TiC–Al_2O_3/Cr18–Ni8 joint is shown in figure 4. The microstructures in the joint are represented by A, B, C, D and E and distribute continuously with black block on TiC–Al_2O_3 side and branch structure on Cr18–Ni8 side. In order to clarify the microstructural compositions in the bonded joint, chemical compositions were carried out on microsections square by energy dispersion spectroscopy (EDS). The results are listed in table 1.

There are Ti (42·3%), O (32·9%) and Al (21·6%) in region A, which are determined to be ceramic matrix composite, TiC–Al_2O_3. Region B contains Ti_3Al precipitates in an *a*-Ti matrix. In this matrix, close to the particles, an Al content of 30·2% was measured. Region C appears to be *a*-Ti, stabilized up to the bonding temperature by Al and oxygen diffusion. Moving towards the Ti/steel interface, Fe content increases to 50·8% and Cr accumulates at the diffusion front resulting in its content of 24·8% in region D. *g*-Fe is identified in region E and the compositions are nearly the same as steel, Cr18–Ni8.

3.2 *XRD analysis*

X-ray diffraction studies were carried out under the same measuring conditions (radiation: Cu, working current: 45 mA, working voltage: 40 kV). The diffractometer measurements were made on two sides, one near the TiC–Al_2O_3 and the other near the Cr18–Ni8. The X-ray patterns are shown in figure 5.

Near TiC–Al_2O_3 side, there are Al_2O_3, TiC and Ti_3Al, in which a portion of Al_2O_3 and TiC are probably from composite, TiC–Al_2O_3. Ti_3Al is produced due to the diffusion reaction of Al and Ti from Ti foil. Near Cr18–Ni8 side, TiC and *a*-Ti were formed. This is the result of diffusion reaction of C from Cr18–Ni8 and Ti from Ti foil. A few of Ti foil was fused during heating and then cooled into *a*-Ti. The diffraction examinations show the existence of Ti_3Al, TiC and *a*-Ti in the interfacial layer of the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint. Correspondingly, the phases in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint are TiC+Al_2O_3/Ti_3Al+TiC/α-Ti/TiC/*g*-Fe.

3.3 *Interfacial shear strength*

The nature and structure of the interfacial layer also determines the properties of the diffusion bonded joint. The interfacial shear strength was measured with CMT 5105 machine and shearing speed, 0·5 mm/min in test. The

Table 1. Compositions of phases in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint analysed by EDS (wt.%).

Location	Al	O	Ti	Fe	Cr	Ni	Diffusion elements and features
A	21·6	32·9	42·3	1·1	0	2·1	TiC–Al_2O_3 matrix
B	30·2	16·6	50·6	1·4	0·8	0·4	Al and O in Ti, Ti_3Al
C	0·8	0	42·3	43·8	5·8	7·3	Ti in Fe, kirkendall effect to form Fe_2Ti and *a*-Ti
D	0	0·4	18·7	50·8	24·8	5·3	Fe and Cr in Ti, interface moves towards the steel Cr accumulates at the diffusion front, *g*-Fe + TiC
E	0	0	2·7	70·0	18·8	8·5	*g*-Fe

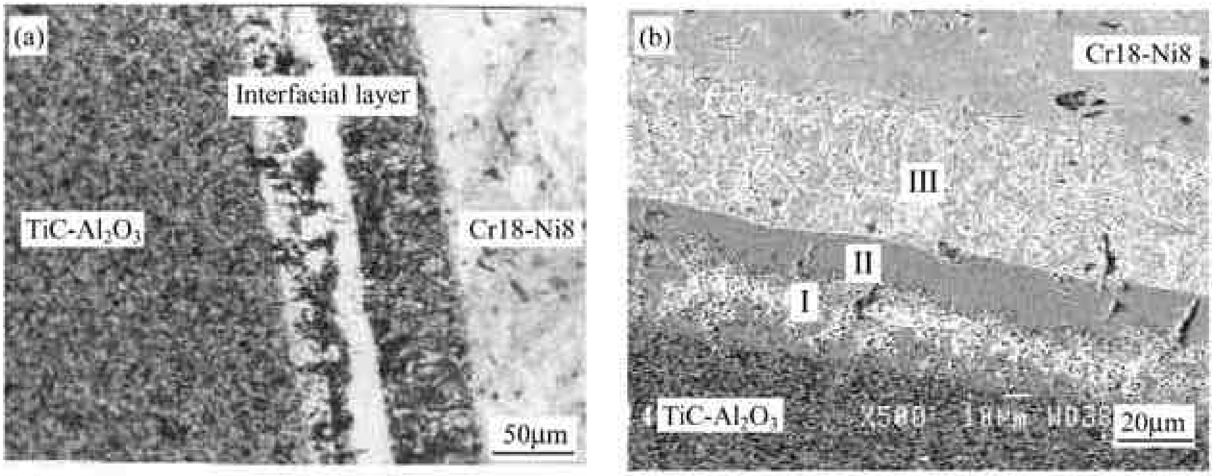

Figure 3. Microstructure in the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint: (**a**) interfacial layer (OM) and (**b**) microstructure in the layer (SEM).

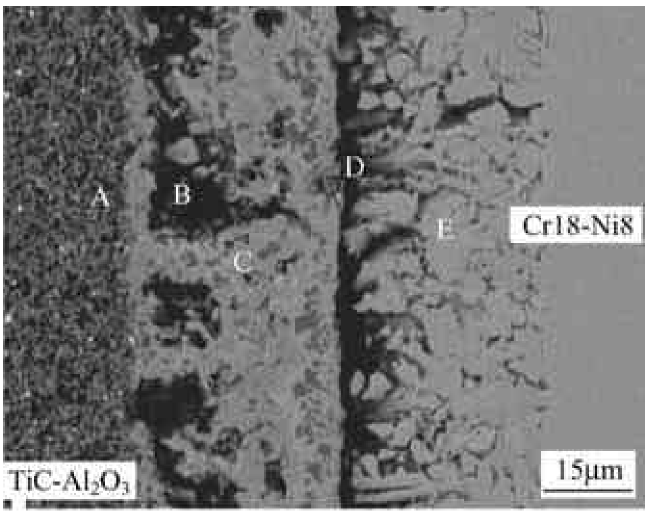

Figure 4. Backscattered electron image of the diffusion bonded TiC–Al_2O_3/Cr18–Ni8 joint.

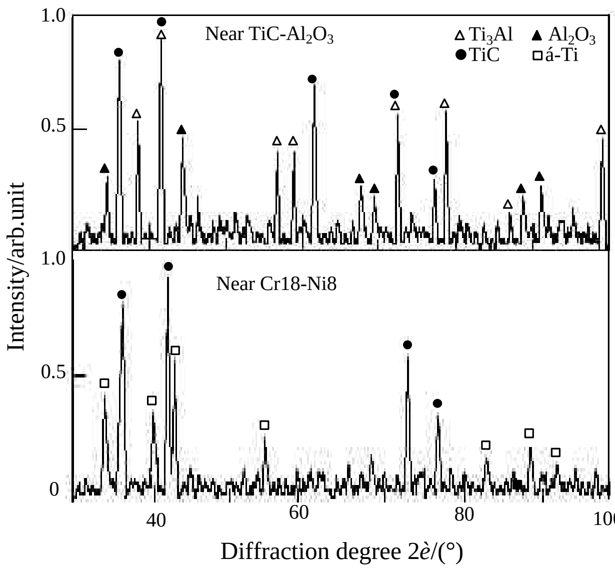

Figure 5. X-ray diffraction patterns.

relation between press and displacement in shear strength measurement is shown in figure 6.

With the increase of the ram displacement, the press increased quickly. When the press was 8·42 kN, the diffusion bonded joint was destroyed. The area of the joint for shearing is 85 mm^2. So the maximum press, 8·42 kN, divided by the area for shearing, 85 mm^2, gives shear strength, 99 MPa. Then by EDS analysis, the compositions in the fractured face are Al, Ti and O (see figure 7).

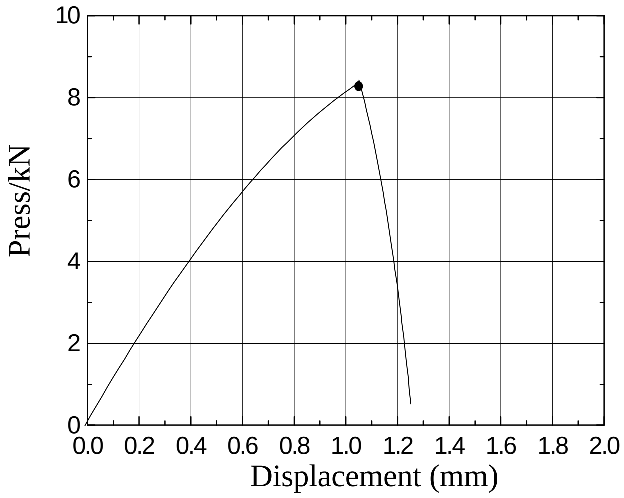

Figure 6. The relation between press and displacement in shear strength measurement.

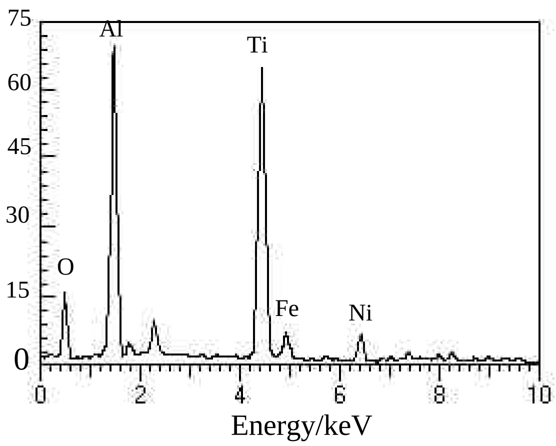

Figure 7. EDS analysis on the fracture face of the bonded Al_2O_3–TiC/Cr18–Ni8 joint.

The EDS result indicates that the fracture location is on Al_2O_3–TiC side of the bonded Al_2O_3–TiC/Cr18–Ni8 joint. This is attributed to the addition of Ti interlayer, which relieves the stress of the joint after diffusion bonding. In addition, the Ti_3Al and a-Ti phase produced has excellent toughness in the joint. So by addition of Ti interlayer, perfect Al_2O_3–TiC/Cr18–Ni8 joint can be obtained successfully using diffusion bonding.

4. Conclusions

Ceramic matrix composite, TiC–Al_2O_3, and stainless steel, Cr18–Ni8, are successfully diffusion bonded at 1400 K for 1h with a pressure of 15 MPa by addition of Ti interlayer. Ti foils fully fused and an obvious interfacial layer with a thickness of 90 μm is formed between TiC–Al_2O_3 composite and Cr18–Ni8 steel. There are the diffusions of Al, Ti and C and Al_2O_3, Ti_3Al, a-Ti and TiC are measured in the interfacial layer by EPMA and XRD examinations. The shear strength is up to 99 MPa and the shear fracture occurs close to the ceramic matrix composite due to the application of Ti foil acting as thermal stress relief interlayer.

Acknowledgements

This project was supported by the Foundation of Key Lab of Liquid Structure and Heredity of Materials and the National Natural Science Foundation of China (50375088). The authors express their heartfelt thanks here.

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