Vacuum 85 (2010) 327–331

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Element distribution and phase constitution of Al_2O_3–TiC/W18Cr4V vacuum diffusion bonded joint

Huang Wanqun [a,b,*], Li Yajiang [a], Wang Juan [a], Jiang Qinglei [a]

[a] Key Laboratory for Liquid-Solid Structural Evolution and Processing of Materials (Ministry of Education), Shandong University, Jinan 250061, PR China
[b] College of Mechanical and Electronic Engineering, China University of Petroleum (East China), Dongying 257061, PR China

ARTICLE INFO

Article history:
Received 11 August 2009
Received in revised form
15 June 2010
Accepted 8 July 2010

Keywords:
Diffusion bonding
Electron probe microanalysis
X-ray analysis
Al_2O_3–TiC ceramic composites

ABSTRACT

Vacuum diffusion bonding between Al_2O_3–TiC ceramic composites and W18Cr4V tungsten-based tool alloy has been carried out by using Ti/Cu/Ti multi-interlayer. Element distribution near the Al_2O_3–TiC/W18Cr4V interface was discussed and fracture morphology was analyzed using electron probe microanalysis. Additionally, phase constitutions of the Al_2O_3–TiC/W18Cr4V joint were determined by X-ray diffraction. The results indicate that Ti-rich layers are formed near both Al_2O_3–TiC and W18Cr4V. The Ti-rich layer near Al_2O_3–TiC helps to wet the Al_2O_3–TiC surface. The Ti-rich layer near W18Cr4V can restrain the formation of Fe–Ti intermetallic compounds in the diffusion transition zone. Residual Cu in the diffusion transition zone can act as a stress releasing zone. The structures of interfacial phases are identified as follows: Al_2O_3–TiC/TiO + Ti_3Al/Cu + CuTi/TiC layer/mixed layer of Fe_3W_3C, $Cr_{23}C_6$ and α-Fe/W18Cr4V. The fracture morphology of Al_2O_3–TiC/W18Cr4V joint appears brittle features and the failure occurs within the Al_2O_3–TiC ceramic.

1. Introduction

Al_2O_3–TiC ceramic composites have been widely used in the field of engineering with high strength and hardness, chemical stability and excellent wear resistance [1]. Al_2O_3–TiC ceramic composites are frequently used to be one of the most suitable tool materials for machining hard materials [2]. Due to inherent brittleness of ceramics, Al_2O_3–TiC presents poor machinability, which limits its extensive application. If the joining of Al_2O_3–TiC ceramic composites and W18Cr4V tungsten-based tool alloy can be successfully realized, it will be important to expand the utilization of Al_2O_3–TiC ceramic composites in machining industry.

Diffusion bonding process has been one of the technological tools to increase the use of ceramic materials. In order to form reliable diffusion bonded joint, a multi-interlayer is sandwiched between the components to be joined. Joining of Al_2O_3–TiC ceramic composites with W18Cr4V tool alloy was investigated by using a Ti/Cu/Ti multi-interlayer. The Ti/Cu/Ti system was selected for the following reasons: (1) Ti shows excellent behavior in promoting the wettability of Al_2O_3 as the most extensively studied active element [3,4]; (2) Cu can form a low-melting eutectic with Ti and the activity coefficient of Ti in Cu is close to unity. This high activity makes Ti attractive as the active element in Cu-based filler metals for joining ceramics [5]; (3) it has been well established that Al_2O_3, when in contact with Cu–Ti alloys, develops strong chemical bonds at the interface [6].

In previous work, the residual stress distribution in Al_2O_3–TiC/W18Cr4V diffusion bonded joints was calculated using finite element method (FEM) and preliminary study on the formation process of the transition zone and phase constitutions was also done [7–9]. However, details about the role of reaction layers are still largely unresolved issues. The objective of the present work is to begin to address some of these problems by reexamining element distribution and phase constitutions of the reaction layers formed during diffusion bonding of Al_2O_3–TiC and W18Cr4V with a Ti/Cu/Ti multi-interlayer.

2. Experimental procedure

2.1. Equipment

The vacuum diffusion bonding equipment (Workhorse-II manufactured by Vacuum Industry Company) was used in the joining

---

* Corresponding author. Key Laboratory for Liquid-Solid Structural Evolution and Processing of Materials (Ministry of Education), Shandong University, Jinan 250061, Jing Shi Road 17923, Shandong, PR China. Tel.: +86 0531 88392924; fax: +86 0531 82609496.

E-mail address: hwanqun@gmail.com (H. Wanqun).

0042-207X/$ – see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.vacuum.2010.07.005

process. The main parameters of the equipment are: size of vacuum chamber $305 \times 305 \times 457$ mm^3, heating power 45 kVA, highest heating temperature 1623 K, maximum pressure 30 tons applied through hydraulic pressure and maximum vacuum degree 1.5×10^{-5} Pa. The vacuum system is composed of the mechanical pump and the diffusion pump.

2.2. Materials

Materials used are Al_2O_3–TiC ceramic composites, W18Cr4 V tool alloy and Ti/Cu/Ti multi-interlayer. Al_2O_3–TiC ceramic was made by hot pressure sintering (HPS) to a final circle plate having a dimension $\phi 52 \times 3.5$ mm^2. The size of W18Cr4 V circle plate specimen is $\phi 52 \times 2$ mm^2. Al_2O_3–TiC ceramic consists of Al_2O_3 matrix with TiC particles. The chemical composition (wt%) of W18Cr4V alloy is: C 0.70–0.80%, Mn 0.10–0.40%, Si 0.20–0.40%, $S \leq 0.030\%$, $P \leq 0.030\%$, Cr 3.80–4.40%, V 1.00–1.40%, W 17.50–19.00%, Mo $\leq$0.30% and Fe balance. A multi-interlayer of Ti/Cu/Ti (20 μm Ti/60 μm Cu/20 μm Ti) is employed to promote the joining between Al_2O_3–TiC ceramic and W18Cr4V alloy and obtain tight metallurgic joint. Table 1 gives the chemical compositions and the thermo-physical properties of Ti and Cu.

2.3. Technology and preparing samples

Before diffusion bonding, the surfaces of materials to be bonded were prepared by conventional grinding and polishing techniques, resulting in a surface roughness R_a of 0.27–0.35 μm for the ceramic and 0.10–0.20 μm for both steel and interlayer. The faying surfaces were cleaned in acetone, followed by rinsing in distilled water and dried in air. After that, the diffusion bonding plates were overlapped (Fig. 1) and placed into vacuum chamber. Technological parameters used in the joining process were: the heating temperature $T = 1373$–1423 K, the pressure $P = 10$–15 MPa, the holding time $t = 45$–60 min and the vacuum degree of 1.33×10^{-4}–10^{-5} Pa. The cooling process was conducted in the vacuum chamber that was cooled by circuit water. When the chamber temperature was cooled to 373 K, the Al_2O_3–TiC/W18Cr4V diffusion bonded joints were taken out from the chamber.

A photo of the Al_2O_3–TiC/W18Cr4V joint is shown in Fig. 2. The Al_2O_3–TiC/W18Cr4V joints were cut into $12 \times 10 \times 9$ mm^3 specimens (Fig. 1) perpendicular to the bonded interface by the line cutting machine. Microstructural morphology of the Al_2O_3–TiC/W18Cr4V joint was analyzed by means of Nikon AFX-IIA metalloscope. Microhardness was measured via SHIMADZU microsclerometer. Element distribution near the Al_2O_3–TiC/W18Cr4V interface, the fracture morphology and the composition on the fractured face were analyzed by JXA-8800R electron probe microanalysis (EPMA). The reaction products were determined by D/MAX-RC XRD.

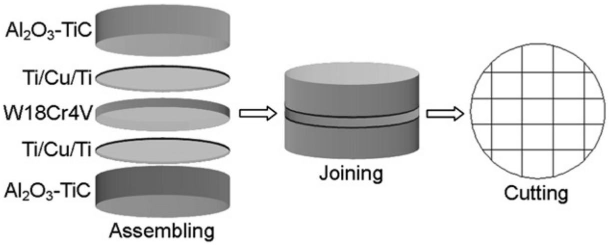

Fig. 1. Assembled and cut schematic diagram of specimen.

3. Results and discussion

3.1. Microstructural characteristics and element distribution

Microstructure near the interface of Al_2O_3–TiC/W18Cr4V joint was observed by optical microscope (Fig. 3). The results indicate that a diffusion transition zone is formed between Al_2O_3–TiC and W18Cr4V. Microstructural morphology of the diffusion transition zone is significantly different from that of the substrates, Al_2O_3–TiC and W18Cr4V. The interface is straight at the Al_2O_3–TiC side and zigzag at the W18Cr4V side.

Microhardness near the Al_2O_3–TiC/W18Cr4V interface was measured via SHIMADZU microsclerometer with a load of 100 g and a load time of 10 s and the results are shown in Fig. 4.

It can be seen from Fig. 4 that the microhardness of diffusion transition zone is far lower than that of Al_2O_3–TiC and slightly lower than that of W18Cr4V. Microhardness distribution, with a softer diffusion transition zone and two harder substrates, can provide sufficient ductility in the diffusion transition zone to avoid cracking.

Composition near the interface is the fundamental part of the joint in determining mechanical behavior. Distribution of alloy elements near the diffusion bonded Al_2O_3–TiC/W18Cr4V interface was measured via EPMA. Fig. 5 presents a backscattered electron (BSE) image (Fig. 5a) and composition variation across the interface (Fig. 5b).

From Fig. 5a and b, it is evident that there are four different layers from Al_2O_3–TiC to W18Cr4V in the diffusion transition zone: (1) a layer near Al_2O_3–TiC, (2) wide central zone, (3) narrow black layer and (4) a layer near W18Cr4V. Fig. 5b shows that the layer near Al_2O_3–TiC mainly contains Ti (22.4–91.7 wt%), Al (6.9–64.3 wt%) and O (1.4–13.3 wt%). According to Ti–Al and Ti–O binary phase diagrams [10], Ti–Al and Ti–O compounds may be formed. Wide center zone consists of three sub-layers and they are Ti, Cu (3.7–82 wt%) and Ti (18–96.3 wt%), and Cu. Based on Cu–Ti binary phase diagram, this layer maybe contain Cu–Ti compounds, Cu and α-Ti. Narrow black layer contains Ti and a small amount of Cu and Fe. Although the EPMA analysis of C was missing, this layer may be TiC. On the base of Ti–C binary phase diagram, Ti combines with C

Table 1
The chemical compositions and the thermo-physical properties of Ti and Cu.

Chemical compositions (wt. %)

Material	H	C	O	N	Bi	S	Fe	Sb	As	Pb	
Ti	0.015	0.10	0.25	0.05	–	–	0.30	–	–	–	Bal.
Cu	–	–	–	–	0.001	0.005	0.005	0.002	0.002	0.005	Bal.

Thermo-physical properties

Material	Melting point (K)	Density at 293 K (g/cm^3)	Crystal structure[a] at 293 K	Coefficient of thermal expansion (10^{-6}/K) 293–373 K	Young's modulus (GPa)
Ti	1913–1943	4.5	h.c.p.	8.2	115
Cu	1338–1355	8.92	f.c.c.	16.92	125

[a] h.c.p. = hexagonal-close-packed; f.c.c. = face-centerd-cubic.