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Bonding and Interfacial Structures of SiC/Zr Joint[†]

T. Fukai*, M. Naka** and J. C. Schuster***

Abstract

Phase reactions and compounds between SiC and Zr were investigated at bonding temperatures of 1473 and 1573 K. Zr reacts with SiC, and forms ZrC next to the Zr and $Zr_5Si_3C_x$ accompanying Zr_2Si beside the SiC. The diffusion of silicon and carbon continue from SiC, and ZrC between SiC and $Zr_5Si_3C_x$ were formed during bonding. At bonding time of 1.8 ks or more at 1573 K the complete diffusion path was established between SiC and Zr as, $SiC/ZrC/Zr_5Si_3C_x/Zr_2Si/ZrC_x/Zr$. This diffusion path is correlated with the corresponding Si-Zr-C phase diagram.

KEY WORDS: (Ceramic-metal Joining) (Interfacial Structure) (Ceramic) (Metal) (Diffusion) (SiC) (Zr) (Zirconium Carbide) (Zirconium Silicide)

1. Introduction

The joining of ceramics and metals compensates for the brittleness and poor workability of ceramics, and expands the engineering application of the ceramics. Control of ceramic/metal interface structure is necessary to realize a high mechanically reliable joint. SiC, which possesses superior high temperature mechanical properties, is a candidate for structural applications. Since Ti is also often used as a structural component, several reports on the SiC/Ti system have been reported. The formation of Ti_5Si_3 , TiC and Ti_3SiC_2 has been reported in SiC fiber reinforced Ti composites ^{1),2)} and SiC/Ti joints ³⁻⁵⁾. The phase reaction and diffusion path was discussed using Ti-Si-C phase diagram. Zirconium is also a heat-resistant material, and a carbide former like Ti. The present paper aims to clarify the phase reactions in the SiC/Zr joint, and to relate them to the Zr-Si-C phase diagrams.

2. Experimental procedure

The materials used were cylindrical SiC rods of 6 mm diameter and 4 mm thickness, containing a few percent

alumina as sintering aid, and Zr foils of 25 μm thickness. The bonding was done in vacuum below 1.33 mPa, using a high frequency heater equipped with a graphite tube. The bonding condition were bonding temperatures from 1473 K to 1673 K, and bonding times from 1.8 to 43.2 ks under a bonding pressure of 7.26 MPa. The microstructures and phases at interfaces were investigated by x-ray diffraction ($CuK\alpha$ diffraction) and electron probe microanalysis.

3. Results and discussion

3.1 Microstructures in Zr/SiC joint

The bonding of SiC to SiC using Zr foil of 25 μm was carried at a bonding temperature of 1473 K for a bonding time of 28.8 ks. Figs. 1 and 2 show the interface microstructure and the quantitative elemental compositions respectively. Granular ZrC phases are formed adjacent to Zr, and $Zr_5Si_3C_x$ layer zones accompanying Zr_2Si are formed adjacent to SiC. The quantitative analysis in Fig. 2 indicates a depleted zone of silicon between SiC and $Zr_5Si_3C_x$ which corresponds to ZrC. The x-ray diffraction analysis of the interface revealed by successive polishing also shows ZrC at the

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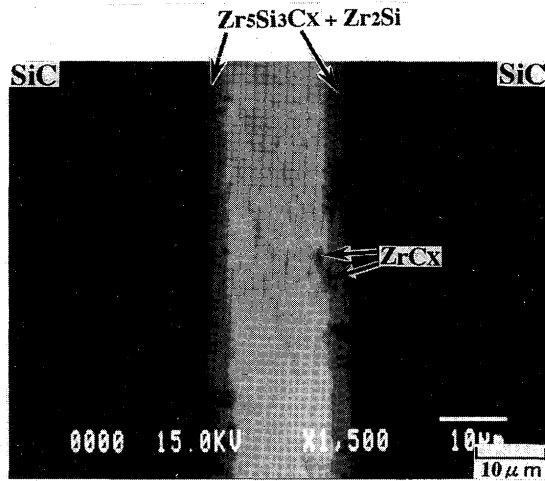


Fig.1 Microphotograph of SiC/Zr/SiC joint bonded at 1473 K for 28.8 ks.

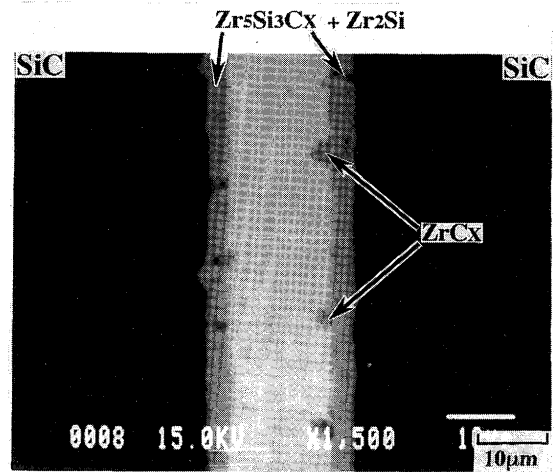


Fig.3 Microphotograph of SiC/Zr/SiC joint bonded at 1573 K for 1.8 ks.

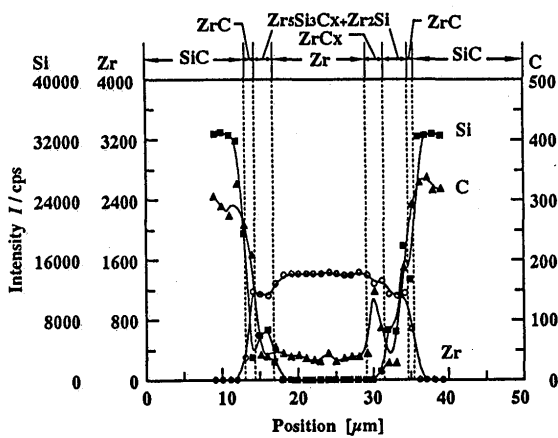


Fig.2 Elemental distribution in SiC/Zr/SiC joint bonded at 1473 K for 28.8 ks.

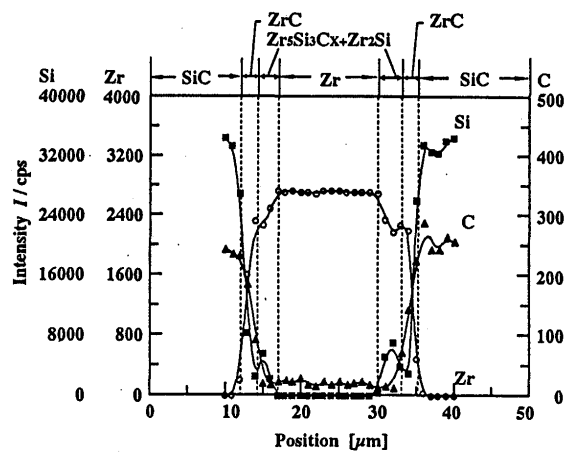


Fig.4 Elemental distribution in SiC/Zr/SiC joint for 1.8 ks.

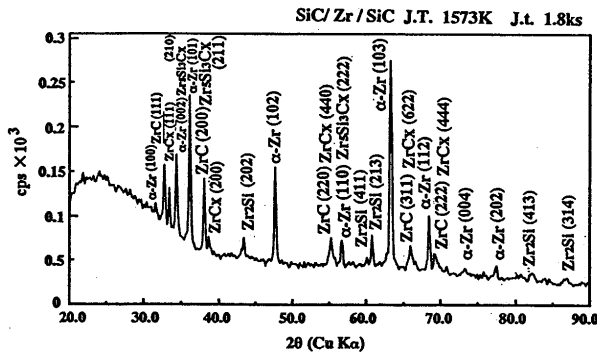


Fig. 5 X-ray diffraction pattern of the revealed interface of a SiC/Zr/SiC joint bonded at 1573 K for 1.8 ks.

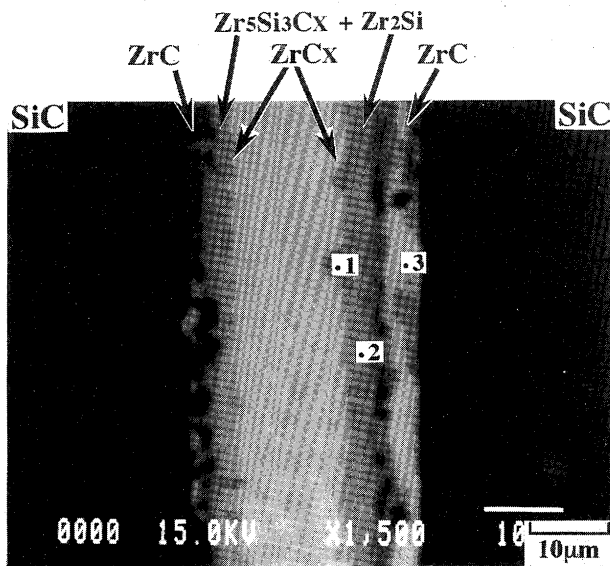


Fig. 6 Microphotograph of SiC/Zr/SiC bonded at 1573 K for 3.6 ks.

interface, and Zr_2Si next to $Zr_5Si_3C_x$. $Zr_5Si_3C_x$ is a ternary compound denoted as Nobotny phase⁶⁾. At a temperature of 1573 K for a bonding time of 1.8 ks the same reaction phases are observed as shown in Fig. 3. The silicon depleted zone corresponding to ZrC adjacent to SiC is clearly seen in Fig. 4. The layer $Zr_5Si_3C_x$ phase accompanying Zr_2Si is also formed. The x-ray diffraction pattern of the revealed interface of the joint bonded at 1573 K for 1.8 ks in Fig. 5 shows α -Zr, ZrC and $Zr_5Si_3C_x$ phases accompanying Zr_2Si formed at the interface between Zr and SiC. The position of this revealed surface is described by A in a later schematic figure. The characteristic of the x-ray diffraction pattern indicates that ZrC with two different

Table 1 Chemical composition of phases formed.

SiC / Zr 1573K, 3.6ks

N.o	Zr (at%)	Si (at%)	C (at%)	Phases formed
1	32.5	0	67.5	ZrC
2	46.8	25.1	28.1	$Zr_5Si_3C_x + Zr_2Si$
3	59.3	0.3	40.4	ZrCx

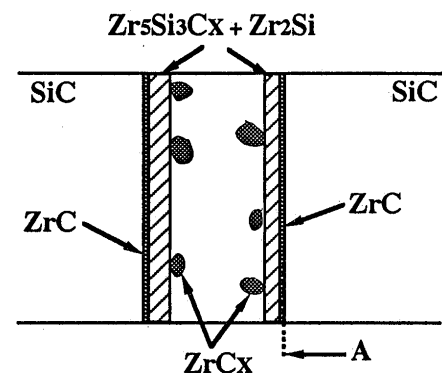


Fig. 7 Schematic structure of the interface between SiC and Zr in the joint bonded at 1573 K for bonding times of 1.8 ks to 3.6 ks.

parameters is present. One, assumed to belong to the granular ZrC adjacent Zr, is rather small ($a = 0.4603\text{nm}$) indicating a lower carbon content. The other lattice parameter, assumed to belong to ZrC adjacent to SiC, is rather large ($a = 0.4688\text{nm}$) indicating a very high carbon content. The ZrC next to SiC is clearly seen in the microstructure of a SiC/Zr/SiC joint bonded at 1573 K for 3.6 ks in Fig. 6. The chemical composition of the reaction phases formed are presented in Table 1. At longer bonding time, cracks between ZrC adjacent SiC and $Zr_5Si_3C_x$ are observed after cutting the joint since the thickness of brittle $Zr_5Si_3C_x$ phase is larger. The growth rate of ZrC in Zr is low because the $Zr_5Si_3C_x$ layer becomes a diffusion barrier to silicon and carbon.

Fig. 7 shows the schematic structure of the interface between SiC and Zr bonded at 1573 K for the bonding time of 1.8 ks to 3.6 ks. The reaction phases formed at the interface between SiC and Zr in the joint bonded at 1573 K are α -Zr, ZrCx, ZrC, Zr_2Si and $Zr_5Si_3C_x$ as shown in Table 2. All these compounds are formed at a bonding time of 1.8 ks or more at the bonding temperature. In other words, the diffusion path between

Table 2 Reaction phases formed at SiC/Zr/SiC joint bonded at 1573 K.

Phases	Structure of crystals	Lattice constant (0.1 nm)
ZrC	Cubic	a = 4.688
ZrC _x	Cubic	a = 4.603
Zr ₂ Si	Tetragonal	a = 6.609 c = 5.298
Zr ₅ Si ₃ C _x	Hexagonal	a = 7.886, c = 5.558
α-Zr	Hexagonal	a = 3.223, c = 4.693
α-SiC (hp4)	Hexagonal	a = 3.067, c = 5.035

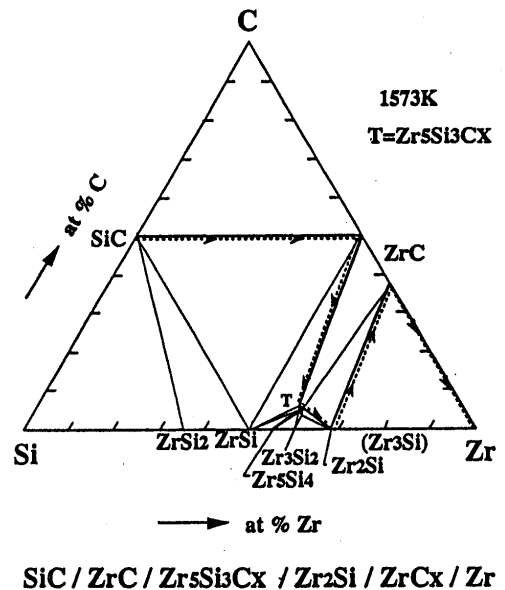
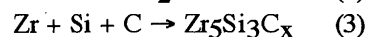
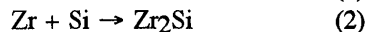
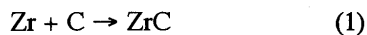


Fig. 8 Diffusion path between SiC and Zr on the Zr-Si-C phase diagram at 1573K.

SiC and Zr is formed and expressed as,
 $\text{SiC/ZrC/Zr}_5\text{Si}_3\text{C}_x/\text{Zr}_2\text{Si/ZrC}_x/\alpha\text{-Zr}$.

This path is represented with a dotted line on the Zr-Si-C ternary phase diagram in Fig. 8 compiled by Schuster⁷⁾.

The reaction mechanism between SiC and Zr is discussed using the present results. First, Zr reacts with SiC at the interface, and forms ZrC, Zr₂Si and Zr₅Si₃C_x by eqs. 1 to 3.



Since carbon diffuses faster than silicon, ZrC is formed at the Zr side, and Zr₅Si₃C_x accompanying Zr₂Si is formed at the SiC side. As Zr remains at the inside, carbon and silicon continue to diffuse from SiC. Then, ZrC adjacent to SiC is formed and the thickness of Zr₅Si₃C_x zone becomes larger. After a bonding time of 1.8 ks at 1573 K the complete diffusion path between SiC and Zr is formed as indicated in Fig. 8. The diffusion path is illustrated using the Zr-Si-C phase diagram.

4. Conclusions

The bonding of SiC and Zr was performed at bonding temperatures of 1473 K and 1573 K. The reaction

mechanism at the interface between SiC and Zr was investigated by observing microstructures of the joint, and x-ray diffraction analysis of the interface revealed mechanically.

At the bonding temperatures Zr reacts with SiC, and ZrC_x beside Zr and Zr₅Si₃C_x accompanying Zr₂Si are formed at the interface between SiC and Zr. Carbon and silicon continue to diffuse from the SiC during bonding, and ZrC is formed adjacent to SiC. Then, the complete diffusion path between SiC and Zr is expressed as, $\text{SiC/ZrC/Zr}_5\text{Si}_3\text{C}_x/\text{Zr}_2\text{Si/ZrC}_x/\alpha\text{-Zr}$. This diffusion path is predicted on a line connecting SiC and Zr in Zr-Si-C phase diagram.

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