Investigations of Reaction Layers between $_{CVD}$SiC Fiber and TiAl Matrix

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Abstract

Metal matrix composites have been investigated extensively for many years. Many fabrication methods have been developed such as a powder metallurgy process, a casting process, HIP and Hot press processes. The hot press process has been often selected because of the availabilities of the many kinds of metal sheet and the easiness of control of the fiber direction. SiC fiber reinforced TiAl intermetallic compound is one of the most promising candidate composite among the heat resistant materials. In the case of $_{CVD}$SiC fiber reinforced gamma TiAl, high process temperatures above 1373K are necessary to consolidate SiC/TiAl composite. Reaction layers between SiC fiber and TiAl matrix have been formed due to interatomic diffusion of Ti, Al, Si, C and other atoms at high temperatures. It is confirmed that more than two reaction layers of intermetallic compounds have been formed. Based on the analyses of SEM-EDS and the X-ray diffraction, it is clarified that inner layer is Ti_5Si_3, TiC and outer one is consisting of Ti_3SiC_2, respectively. In order to improve the mechanical properties of SiC/TiAl composite, a specially designed interface structure between SiC fiber and TiAl matrix will be necessary.

Keywords
SiC fiber, titanium aluminide (TiAl), composite material,

1. Introduction

Although metal matrix composites for the high temperature structural material have been investigated extensively[1,2], applications of MMC have been limited. Recently applications of SiC fiber reinforced SiC ceramic composite to a newly designed commercial jet engine components have been announced[3]. Among many combinations between the ceramic fibers and matrix materials, SiC fiber and TiAl based intermetallic compounds have been expected to be one of the best combination since both SiC fiber and TiAl are heat resistant as well as low density materials. The chemical vaper deposited SiC fiber has been manufactured and commercially available. However, a sheet of gamma-TiAl intermetallic compound has not been fabricated yet even in the small laboratory scale. Lack of the affordability of TiAl sheet has been inhibited the development of the TiAl base composite materials. Among several approaches[4,5], SiC fiber reinforced TiAl has been successfully fabricated by Kawasaki Heavy Industry (KHI) -Nippon Steel Corp. (NSC) group in 1997[6,7]. After studies of KHI-NSC group, any investigations has not been hardly found to follow the results of their SiC/TiAl composites. In this study, the consolidations of SiC fiber and TiAl by the hot press in a vacuum have been examined. The purpose of this research is to understand the reaction layers between fiber and TiAl matrix. Results would lead to establish the most appropriate fabrication conditions of SiC/TiAl composite.

2. Experimental Procedure

2.1 SiC fiber and matrix TiAl

The properties of chemical vapor deposited SiC fiber (SCS-2) are listed in Table 1. Another type of SiC fiber is available, SCS-6 has carbon coating at the fiber surface for preventing reaction with titanium. Using SCS-6 and titanium alloys, SiC/Ti composites have been produced and published their properties[8]. In order to depress reaction between SiC fiber and TiAl matrix, physical vapor deposition of Pt-20%Pd coating have been applied. The coating thickness was approximately 20 nm.

Table 1 Physical properties of SCS-2

Properties	
Diameter /μm	140
Density /g・cm^{-3}	3.05
Tensile strength / MPa	3500
Elastic constant /GPa	400

Table 2 Chemical composition of TiAlCr

atomic %			*weight %*		
Ti	Al	Cr	O	H	C
50.7	44.8	4.5	0.020	0.0008	0.009

In this study, Cr doped γ-TiAl was selected as the matrix, its chemical compositions are listed in Table 2. TiAl ingots were fabricated by the plasma arc melting (PAM) facility. Then they received HIP process at 1323K for homogenizing treatment in order to eliminate casting defects. Ingots were compressed up to 80% at 1473K, in a vacuum atmosphere. This thermo-mechanical treatment produces a controlled microstructure having uniform fine grains consisting of α+β+γ three phases[5]. Figure 1 shows the strain rate dependence of the flow stress of γ -TiAl at 873, 1073, 1273 and 1473K based on the high temperature tensile tests. Slope of each lines is indicating the stress sensitivity factor *n*. If *n* value is greater than 0.3, the criterion of super plasticity is satisfied. Cr doped TiAl shows that *n* value was 0.34 at 1473K, thus it has the capability of the superplastic deformation. The sheets of γ-TiAl specimens whose thickness were 0.2mm were cut by the diamond multi wire saw machine. The wire speed was 600mm/min and cutting speed was 0.2mm/min. This process is a relatively slow process without mechanical damages on the TiAl surface. Since both cutting and polishing process have been finished simultaneously by the fine diamond powders, the surface quality of TiAl was enough to consolidation. Using a specially designed jig, five TiAl sheets (20x20x0.2mm^3) and properly chopped SiC fibers were laminated in layer by layer as shown in Fig.2. Matrix and fiber were fixed by the instant glue.

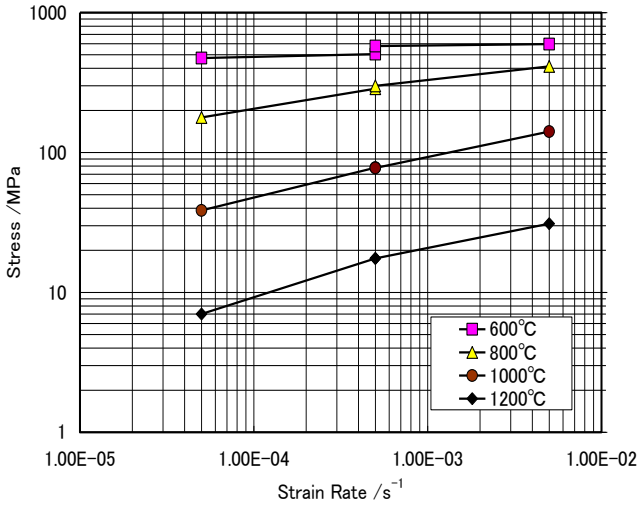

Fig.1 Strain rate dependence on flow stresses of Cr doped TiAl at high temperature tensile tests.

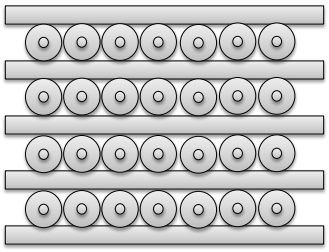

Fig.2 Schematic drawing of SiC/TiAl Preform.

2.2 Fabrication process

Consolidations of the preform were carried out by means of the hot press facility at various temperatures in a vacuum. After the preform was set in the chamber, temperature was raised with heating rate 15K/min, and kept for 40min. Preform was hot pressed under the condition of 7.0MPa for 10min. After consolidations, the pressure was released then specimen was cooled down in the furnace. SiC/TiAl specimens were cut from composites perpendicular to the fiber direction by the diamond wheel saw, and then polished by emery paper. Cross section of the specimens were observed by SEM-EDS operated at 15keV. Using imaging plate X-ray diffraction (IPXRD) with collimator of 0.1mm diameter, diffraction patterns from the fiber/matrix interface were studied.

3. Results and Discussion

3.1 Conditions of Hot Press Process

Determination of an adequate hot press condition for SiC/TiAl composite is the most important purpose of this research. Figure 3 shows the cross sections of the composites at various hot pressing temperatures under the condition of 7.0MPa for 10min. The volume fraction of fiber in matrix is examined from the SEM photographs and listing in Fig.3. Based on the SEM observations, composites

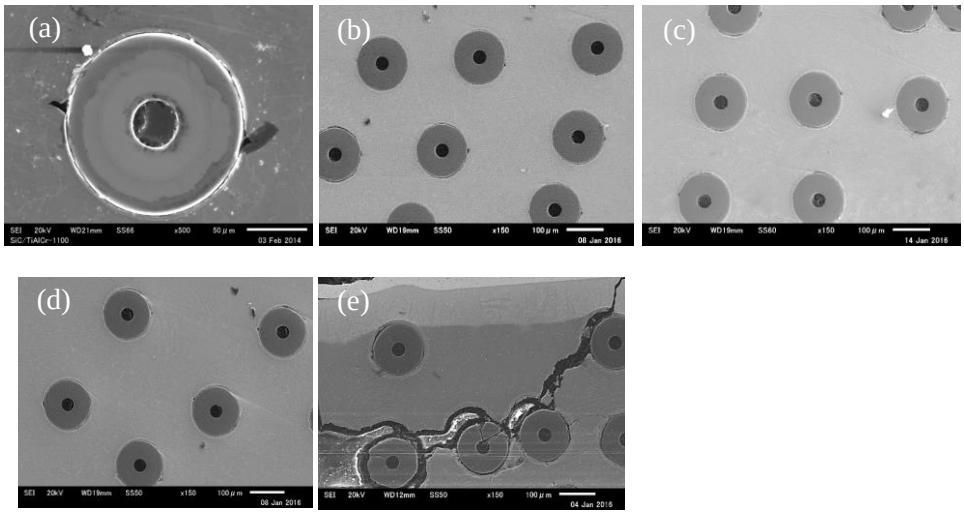

Fig.3 Microstructure of SiC/TiAl composites.
(a)1273K,Vf=9.5%, (b)1473K,Vf=14.3%, (c)1498K,Vf=18.3%, (d)1523K,Vf=13%, and (e)1573K,Vf=13.7%

of 1473, 1498 and 1523K are well formed and show relatively good interfaces. Matrix TiAl has deformed significantly around the fibers and the initial TiAl sheets have bonded together perfectly as like a bulk TiAl specimen. Although, 1373K composite shows defects such as voids at the interfaces, 1573K composite shows severe cracks in either interface or matrix. More detailed observations have revealed the chemical reaction layer between SiC fiber and TiAl matrix.

3.2 Analysis of reaction layer

Although SEM micrographs show relatively well formed SiC/TiAl interfaces in the composites, the thickness of the interface increases with increasing the consolidation temperatures. Figure 4 has demonstrated the EDS compositional mapping of Si, C, Ti, Al and Cr at the interface of SiC/TiAl that

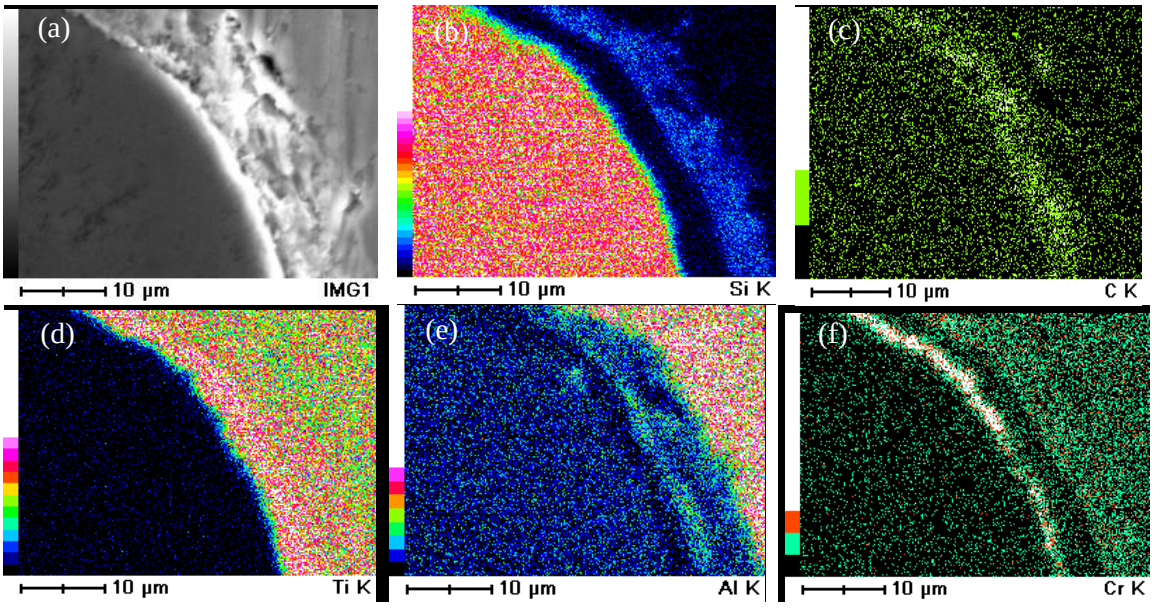

Fig.4 EDS compositional mapping of Si, C, Ti and Al in the SiC/TiAl interface region. (a) SEM image, (b) Silicone, (c) Carbon, (d) Titanium (e)Aluminum and (f)Chromium

was fabricated at 1573K. SEM image is indicating 8.6 μm reaction layer in Fig.4(a), Si is segregated outer layer of the interface (Fig.4(b)), although the both C, Ti and Cr show more segregation at the vicinity of the SiC fiber interface (Fig.4(c) and (d)). This observation is suggesting that there are at least two layers inside reaction layer between SiC fiber and TiAl matrix. Interface layer is consisting of inner titanium and carbon rich layer, then outer layer is consisting of silicone and titanium rich layer.

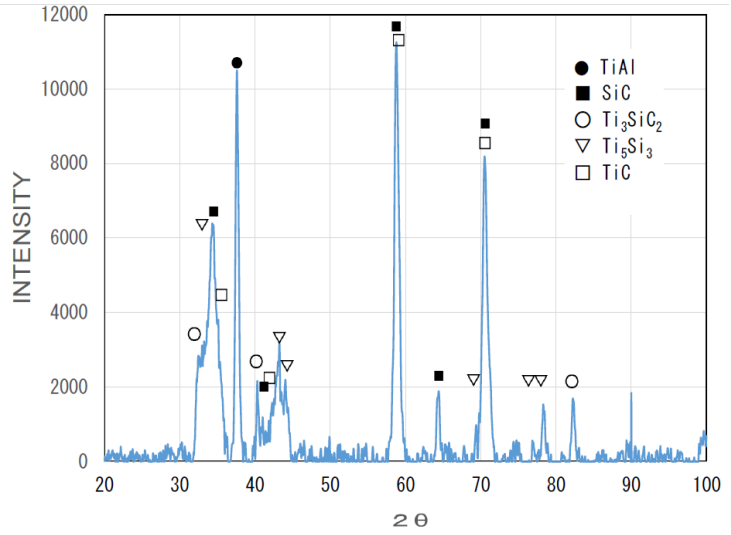

Fig.5 XRD pattern from the reaction layer of SiC/TiAl composite using collimator of 0.1mm diameter

In order to clarify the formed compounds in the interface by inter atomic diffusion between the SiC fiber and TiAl matrix, IPXRD was carried out. Figure 5 shows the XRD pattern from the interface region. According to accumulated XRD database, TiAl, SiC, Ti_3SiC_2, Ti_5Si_3 and TiC have been identified. Based on the XRD and EDS analyses, Ti atom in the matrix diffuses fast and reacts with carbon atom in SiC fiber to create TiC compound layer at the interface. Very complex chemical reactions have been taken place between Ti, C and Si atoms in the reaction zone[9].

Figure 6 shows the effects of the reciprocal of the consolidation temperatures on the reaction layers. According to Fig.6, log scale of the thickness of the reaction layer tends to decrease linearly with increasing the reciprocal of the temperatures as coincidence with the temperature dependence on the diffusion rates. Since the other process conditions are fixed except consolidation temperatures, the thickness of interface layer is directly related with the diffusion constant value at the given temperatures.

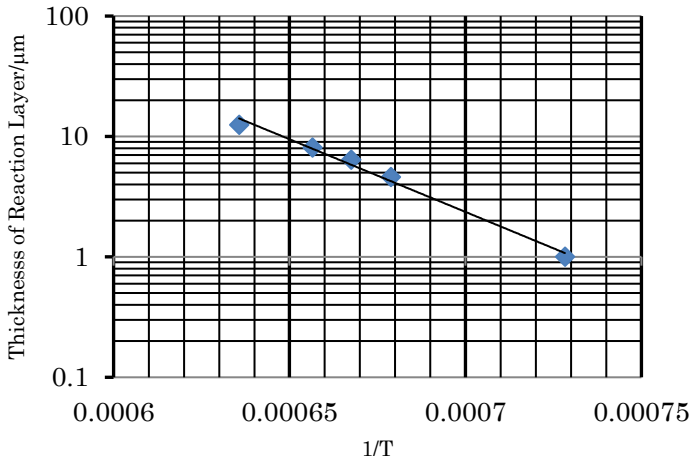

Fig.6 Effects of the reciprocal of the consolidation temperatures on the thickness of reaction layers.

3.3 Three point bending tests

Three point bending tests were carried out to examine the mechanical properties of SiC/TiAl composites. These unidirectional composites have to obey the rule of mixture about elastic constant. Bending strength of SiC/TiAl composites were measured from the load-deflection curves. Values of the bending strength were well below the strength value that expecting the rule of mixture about strength. The reason why they show lower strength is possibly quite large number of defects such as

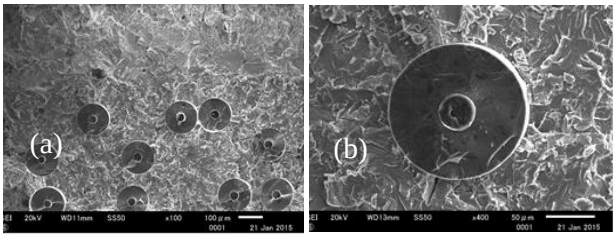

Fig.7 Fracture surface of 1423K specimen after bending test. (a) low magnification, (b) high magnification

voids would be existing in the composite. Therefore, crack would easily nucleate at the stress concentrated point during three points bending tests. Exceptionally, 1423K specimen showed 702MPa (88% of ROM strength). Fracture surface of 1423K specimen after the three points bending test has been observed. Figure 8 shows the fracture surface which have a relatively high elastic constant. TiAl matrix shows quasi-cleavage fracture and SiC fiber shows brittle fracture surface. There is an evidence that fiber and matrix do not fractured simultaneously and pull out of the fiber has been observed in Fig.7(b). This observation is suggesting that the interface of this specimen has almost ideal character. Stress distribution of the fiber is rather higher than that of matrix and reaction layer between SiC and TiAl does not degraded the strength of SiC fiber. Design of SiC/TiAl composite interface is the most important to fabricate the SiC/TiAl composite that has both high elastic modulus and high strength.

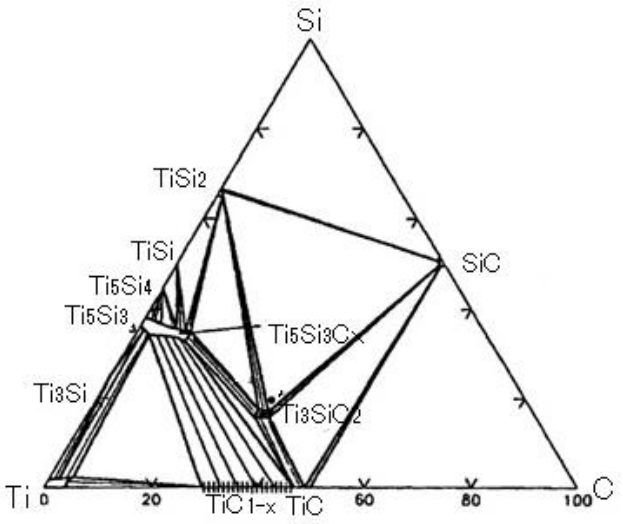

Fig.8 Isothermal cross section of Ti-Si-C ternary phase diagram at 1373K[x)]

3.3 Ternary phase diagram

Reaction between SiC and TiAl intermetallic would be elucidated by the Ti-Si-C ternary phase diagram. Figure 8 show the isothermal cross section of Ti-Si-C ternary phase diagram at 1373K. Titanium react with Si and form intermetallic compound Ti_3Si,Ti_5Si_3, Ti_5Si_4,TiSi and $TiSi_2$. Titanium also react with Carbon and form stable TiC compound. Ternary phase diagram indicate the possibility of ternary compound Ti_3SiC_2. According to XRD results, TiC, Ti_5Si_3 and Ti_3SiC_2 have been identified. EDS analyses have revealed that there are several compounds layers such as (SiC) / Ti_5Si_3 / TiC / Ti_3SiC_2 / (TiAl). Figure 9 have demonstrated schematic drawing of the reaction layers. Chromium atom segregated Ti_5Si_3 intermetallic compound since it has more solubility than the other compounds such as TiC and Ti_3SiC_2. These intermetallic compounds would be very brittle because of their covalent bonding nature rather than metallic bonding. In order to design the interface between SiC fiber and γ-TiAl, formation of brittle intermetallic compounds could be prevented and form an interface layer having the nature of metallic bonding. Several candidate materials have been coated on the SiC fiber and consolidation process have been investigating.

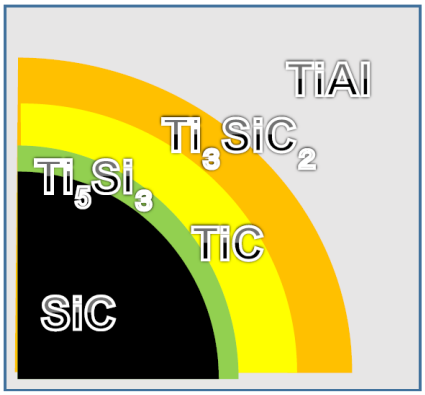

Fig.9 Schematic drawing of the identified reaction layers of SiC fiber reinforced TiAl

4. Conclusions

SiC_{CVD} fiber and sheet of Cr doped TiAl were consolidated at high temperatures in a vacuum using hot press method. Following process conditions have been clarified.

1. For matrix materials, the capability of the super plastic deformation above 1473K is essential.
2. Process temperatures are limited from 1423 to 1523K and have a very narrow range.
3. Thickness of reaction layer increases with increasing process temperatures.
4. Reaction between SiC and TiAl intermetallic would be elucidated by the Ti-Si-C ternary phase diagram Complex reaction products were identified.
5. In order to improve the mechanical properties of SiC/TiAl composite, a specially designed interface structure between SiC fiber and TiAl matrix will be necessary.

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