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Fabrication Conditions of SiC Fiber Reiforced TiAl Intermetallics by Hot Press

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Although TiAl based SiC fiber reinfoced composite has been expected to be one of the best combination for the high temperature structural material, a fabrication of SiC_{CVD} / TiAl composite has not been fully success until now. Formation of a brittle reaction layer between fiber and matrix TiAl at the high fabrication temperature and the lack of room temperature ductility of TiAl cause many difficulties. In order to solve these problems, SiC_{CVD} fiber and alloy designed γ-TiAl which has a capability of the super plastic deformation were consolidated by the vacuum hot pressing process. It was found that the most appropriate condition of the consolidation temperature was very narrow temperature range (1423-1523K) based on the observations of the fiber/matrix interface. A unidirectional SiC/TiAl composite having a lower fiber volume fraction appeared to the designed mechanical properties. Fabrication condition of SiC_{CVD}/TiAl composite will be discussed in detail.

Keywords: *SiC fiber, Titanium Aluminide (TiAl), composite material, hot press, mechanical property, elastic constant, rule of mixture, fiber/matirix interface*

1. Introduction

Recently applications of SiC fiber reinforced SiC ceramic composite to a newly designed commercial jet engine components have been reported[1]. Since the boron fiber reinforced aluminum composite was developed for the structural material in the cargo bay of the space shuttle, metal matrix composites for the high temperature structural material have been investigated extensively[2,3]. 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 because both SiC fiber and TiAl are heat resistant and low density materials. The chemical vapor deposited SiC fiber has been manufactured and commercially available. However, a sheet of TiAl intermetallic compound has not been manufactured yet even in small laboratory scale. Lack of the affordability of TiAl sheet has prevented the development of the TiAl base composite materials. Among several approaches[4,5], SiC fiber reinforced TiAl has been fabricated by Nakatani et.al[6,7]. There is no announcement of the success in fabrication of the SiC/TiAl composites since then. In this study, a new method of the sheet fabrication process of TiAl has been proposed and the consolidation conditions of SiC fiber and TiAl by the hot pressing in vacuum have been examined. The purposes of this research are to clarify 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 on the fiber surface for blocking reaction with titanium. Using SCS-6 and titanium alloys, SiC/Ti composites have been fabricated and demonstrated their properties[8].

In this study matrix TiAl is Cr doped TiAl, its chemical compositions are listed in Table 2. Cr doped TiAl ingot was produced by a plasma arc remelting (PAM) process, and then it received HIP treatment at 1323K for the homogenizing treatment in order to eliminate casting defects.

Table 1 Properties of SiC fiber (SCS-2)

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

Table 2 Chemical composition of Cr doped TiAl

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

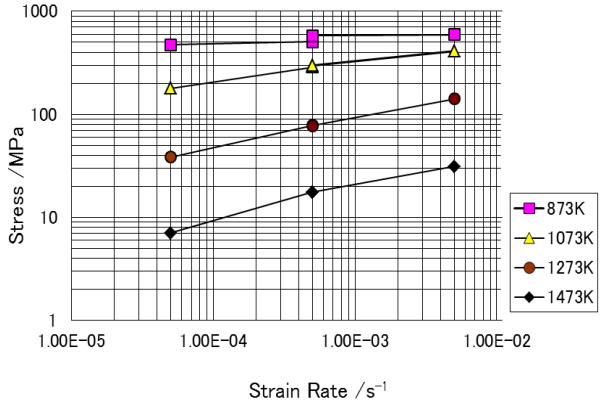

Fig.1 Strain rate dependence on flow stresses of Cr doped TiAl at high temperature tensile tests.

After HIP treatment, cylindrical specimens were cut and isothermally forged at 1473K up to 70% reduction in a vacuum atmosphere. This thermo-mechanical treatment produces a controlled microstructure having fine uniform grains consisting of α+β+γ three phases[5]. The capability of the super plastic deformation is confirmed from Fig. 1. Figure 1 shows the strain rate dependence on the flow stress of Cr doped TiAl at 873, 1073, 1273 and 1473K, respectively, based on the high temperature tensile tests. Slope of each lines is indicating the strain rate

sensitivity factor *n*. When *n* value becomes greater than 0.3, the criterion of the 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 TiAl specimens whose thickness were 0.2mm were sliced 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 but there is no mechanical damage on the TiAl surface since cutting and polishing process have been taken place simultaneously by fine diamond powders. As illustrated in Fig.2, five TiAl sheets ($20x20x0.2mm^3$) and properly chopped SiC fibers were laminated in layer by layer using a specially designed jig. Matrix and fiber were instantly glued by Aron alpha.

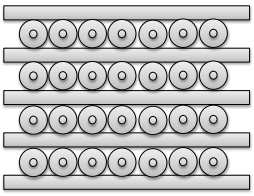

Fig.2 Schematic drawing of SiC/TiAl Preform.

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. The preforms were hot pressed under the condition of 7.0MPa for 10min. After consolidation, the pressure was released then specimen was cooled down in the furnace. SiC/TiAl specimens were cut from the composites perpendicular to the fiber direction by the diamond wheel saw, and then polished by emery paper. Cross section of the specimens was observed by SEM-EDS operated at 15keV. Using imaging plate X-ray diffraction (IPXRD) with the collimator of 0.1mm diameter, diffraction patterns from the fiber/matrix interface were analyzed.

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 microstructures of the composite cross sections at various hot pressing temperatures under the compressive stress of 7.0MPa for 10min. The volume fraction of fiber V_f is examined from the SEM photographs and listed in Fig.3. Based on the SEM observations, the composites 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. 1373K composite shows some defects such as voids at the interfaces. 1573K composite shows severe cracks in either interface and 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

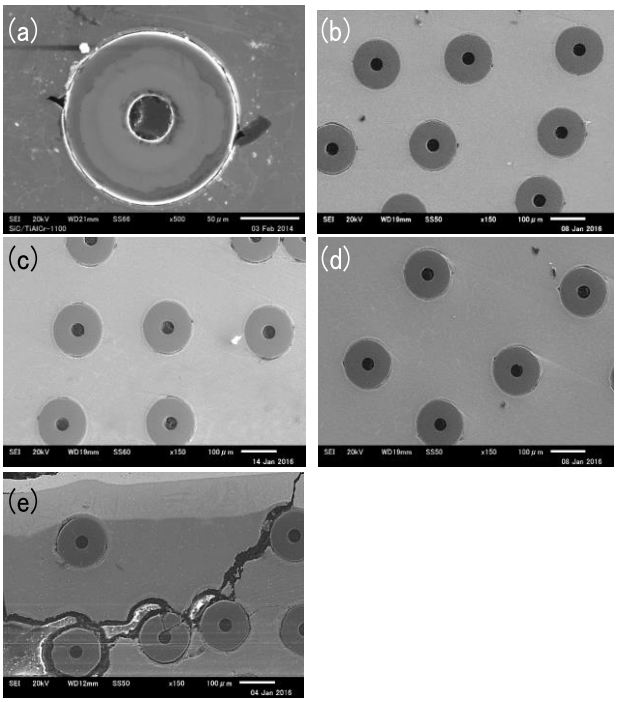

Fig.3 Microstructures of SiC/TiAl composites.
(a)1373K, V_f=9.5%, (b)1473K, V_f=14.3%, (c)1498K, V_f=18.3%, (d)1523K, V_f=13%, and (e)1573K, V_f=13.7%

increases with increasing the consolidation temperatures. Figure 4 shows the effects of the reciprocal of the consolidation temperatures on the thickness of the reaction layers. According to Fig.4, logarithm 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 at the given temperatures.

Figure 5 has demonstrated the EDS compositional mapping of Si, C, Ti and Al at the interface of SiC/TiAl composite that was fabricated at 1573K. SEM image is indicating 12.5 μm reaction

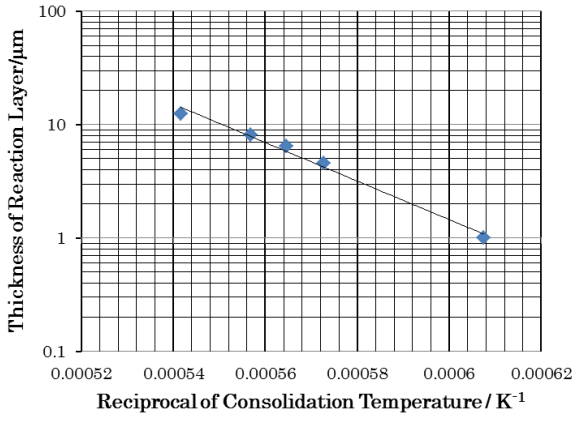

Fig.4 Effects of the reciprocal of the consolidation temperatures on the thickness of reaction layers.

layer in Fig.5(a), Si is segregated outer layer of the interface (Fig.5(b)). Both C and Ti show more segregation at the vicinity of the SiC fiber interface (Fig.5(c) and (d)). These observations are suggesting that there are 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.

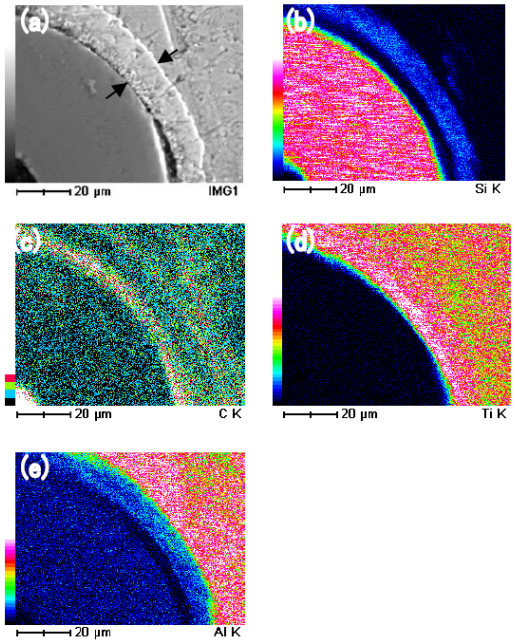

Fig.5 EDS compositional mapping of Si, C, Ti and Al in SiC/TiAl interface region of 1573K composite. Arrows are indicating reaction layer. Concentration increases with color change from blue to red. (a) SEM image, (b) Silicone, (c) Carbon, (d) Titanium and (e)Aluminum

In order to clarify the formed compounds in the interface by the inter atomic diffusion between SiC fiber and TiAl matrix, IPXRD has been carried out. Figure 6 shows the XRD pattern from the interface region of 1573K composite. According to accumulated

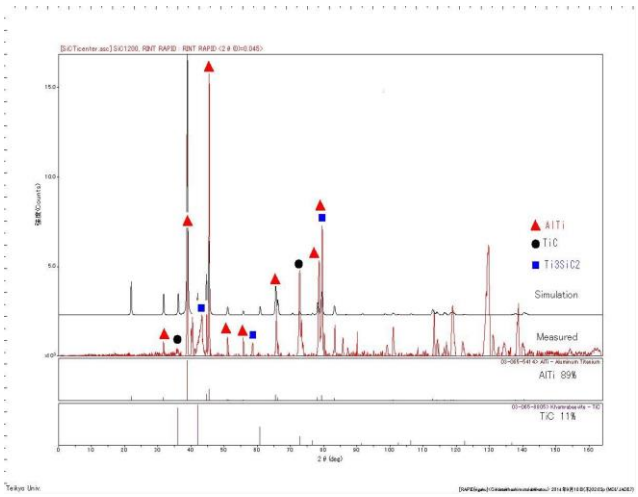

Fig.6 XRD pattern from the reaction layer, Red: measured XRD pattern, ideal AlTi pattern and TiC pattern from database. Top is simulated XRD pattern consisting with 89%AlTi and 11% TiC.

XRD database (JADE program), TiC, TiAl and complex compound (Ti_3SiC_2) 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 taken place between Ti, C and Si atoms in the reaction zone[9].

3.3 Three Point Bending Tests

Three point bending tests have been carried out to examine the mechanical properties of SiC/TiAl composites. These unidirectional composites have to obey the rule of mixture about elastic constant. Figure 7 shows the measured elastic constant values from the data of bending stress and strain which was measured by the strain gages on the specimens. Values of the measured elastic constant have scattered widely depending on the consolidation temperatures, although SEM observations have shown little differences in microstructures. The mechanical properties of 1423, 1473 and 1523K composite specimens have demonstrated relatively high elastic constant values than the other consolidation temperatures. Furthermore data scattered even in the specimens having the same consolidation condition. These results suggest that the most appropriate process condition of SiC/TiAl composite would be a very narrow process window about the consolidation temperatures. Composite having a higher fiber volume fraction becomes more difficult to get ideal mechanical properties, since inter distance of fibers becomes very narrow and matrix TiAl would not be squeezed into them. The elastic constant of SiC/TiAl composite increases with increasing the fiber volume fraction theoretically, because the elastic constant of SiC fiber is greater than that of TiAl. In this research, we have not reached the most appropriate fabrication condition of SiC/TiAl composite.

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

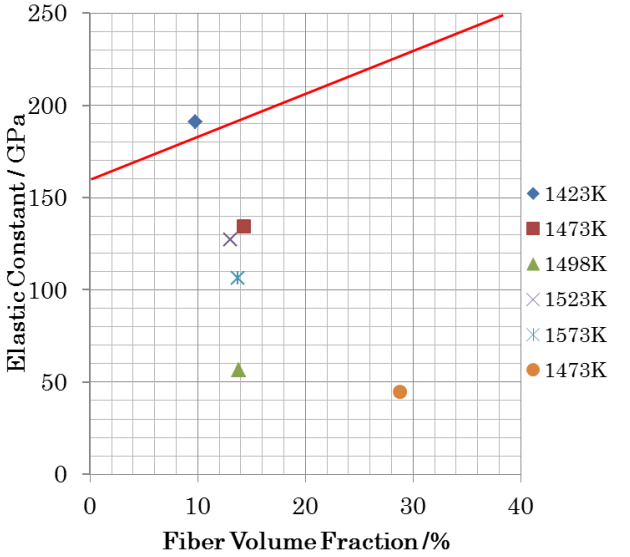

Fig.7 Measured elastic constant values as a function of fiber volume fraction(%) in SiC/TiAl composites, Red line is indicating calculated ideal elastic constant based on the Rule of Mixture (ROM).

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

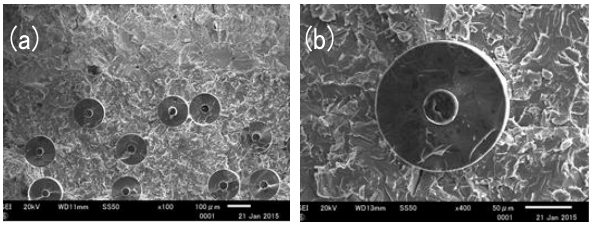

Fig.8 Fracture surfaces of 1423K specimen after bending test.
(a) low magnification, (b) Pull out of fiber is observed in high magnification

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. Specimen having a low fiber volume fraction and a lower process temperature shows an excellent mechanical properties.
5. Process conditions of specimen having a high fiber volume fraction have not been optimized, further investigation would be necessarily.

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