

Oxidation Resistance Behavior of Silicon Hexaboride and Silicon Hexaboride Covered C/C Composite

Jun-ichi Matsushita, Samon Tanaka, Takashi Akatsu*, Koichi Niihara** and Eiichi Yasuda*
Tokai University, 1117 Kitakaname, Hiratsuka 259-1292, Japan

*Tokyo Institute of Technology, 4259 Nagatsuta, Midori-Ku, Yokohama 226-0047, Japan

**Osaka University, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan

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Introduction

Silicon chemical compound of oxide, carbide and nitride is the useful industrial materials. Several silicon boride phases such as SiB_4 , SiB_6 , SiB_{6+x} , SiB_{6-x} , SiB_{14} , $\text{Si}_{8.44}\text{B}_{305.51}$, and SiB_{31} , were previously reported. Among them, SiB_6 has proved to be a potentially useful material because of its excellent chemical stability¹⁻³. Carbon Fiber reinforced Carbon composite (C/C composite) has excellent specific strength in high temperature exceeding 1750 K. its material has shown promise for the application to the rocket components with the advantage of lightweight. However, all constituent material of the fiber and matrix in the C/C composite consists of carbon, which causes its material to oxidation-exhaust intensely under the presence of oxidation atmosphere at temperature of more than 800 K³. We made C/C composite that SiB_6 covered the surface of C/C composite. In this study, the oxidation behavior at high temperature of the SiB_6 powder, SiB_6 sintered body and SiB_6 covered C/C Composite were investigated in order to determine its high-temperature utility.

Experimental

In this present work, SiB_6 powder manufactured by Cerac Co. was used as the starting material. A SiB_6 sintered body was prepared by hot pressing in a VHP gr 18/15 from Shimadzu Mectem Co., Ltd. Table 1 shows sintering condition. SiB_6 was prepared by hot pressing using carbon jigs. SiB_6 specimens were prepared by hot pressing and smaller specimens were cut out for measurement of the oxidation behavior (5×5×10 mm).

Table 1 Sintering condition of hot pressing

Pressure (MPa)	25
Atmosphere (Pa)	1×10^{-4}
Heating rate (K/s)	0.17
Sintering temperature (K)	1823 ~ 1923
Sintering time (ks)	3.6
Cooling condition	Furnace cooling

SiB_6 powder was dispersed to triethyleneglicol. The C/C composite (Nippon Carbon Co., Ltd.) was dipped by it. The sample was prepared by the defatting process to evaporate triethyleneglicol completely from sample. The C/C composite was obtained with SiB_6 mass equal to about 2.5 % of the total sample mass, and surface of its sample was coated with SiB_6

powder. The sample was measured under these following conditions in the analysis of mass change by way of TGA was undertaken; from the room temperature to 1273 K and in the rising rate of 10 K/min. All specimens were subjected to X-ray diffraction (XRD) analysis for phase characterization using an X-ray diffraction meter with an APD1700 goniometer provided with a monochromator and using $\text{CuK}\alpha$ from Philips Ltd. The relative density of the sintered body was determined using the Archimedean method and scanning electron microscope (SEM) observations.

Results and Discussion

The oxidative weight gain of the SiB_6 sintered body at 673 to 1273 K versus oxidation time is shown Fig. 1. The specimen oxidized at 873 to 1273 K for 25 h exhibited weight gain with increasing oxidation temperature. The weight gain of the specimen oxidized at 1273 K for 25 h was approximately 1.5 %. Figure 2 shows TGA curve of the sample covered with SiB_6 by dipping process and the as-received C/C composite. When the sample oxidized from a room temperature up to 1273 K rising by 10 K per minute under the oxidation atmosphere, the starting oxidation temperature given by the TGA curve was observed to be shifted to the higher temperature side.

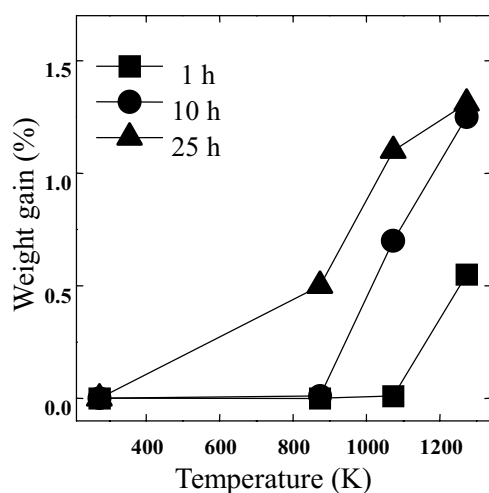


Fig. 1 Weight gain with oxidation as a function of temperature and time

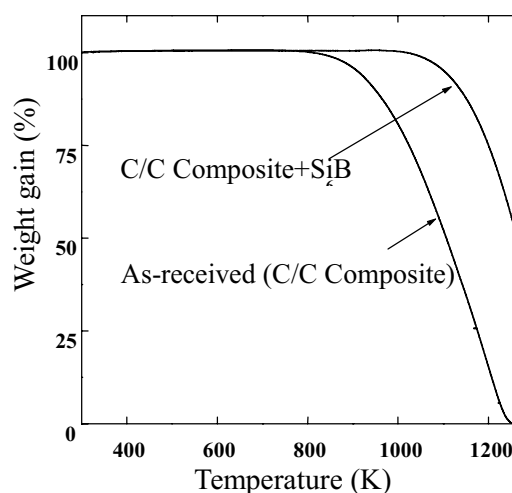


Fig. 2 TGA curve of the sample covered with SiB_6 by dipping process and the as-received C/C composite

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