



Silicon carbide coating of the aluminum joined boron carbide by using polycarbosilane



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ARTICLE INFO

Article history:

Received 16 July 2013

Accepted 10 August 2013

Available online 25 August 2013

Keywords:

Silicon carbide

Boron carbide

Polycarbosilane

Coating

Diffusion

Crystal growth

ABSTRACT

Silicon carbide (SiC) coatings on a joined Boron carbide (B₄C), aimed at improving the corrosion resistance, were prepared by using polycarbosilane (PCS) in order to increase its corrosion resistance. Some B₄C was diffused to PCS membrane in the curing process and the PCS membrane was made into SiC including crystal one in spite of 1473 K pyrolysis. The 4-point bending test of the samples after corrosion test shows that the sample without any coating was close to 0 MPa, while the samples with SiC coating maintained more than 300 MPa.

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1. Introduction

Boron carbide (B₄C) is a promising material in the field of engineering because it has high hardness (after diamond and cubic boron nitride), good abrasion resistance, light, and chemical stability that most acid or alkali solvents cannot dissolve it at room temperature [1,2]. Taking advantage of these characters, B₄C ceramics with large sizes and complex shapes such as a stage for semiconductor processing machines are required because of changing requirements and energy saving, and the method of manufacturing such B₄C production by assembly of small B₄C units was proposed [3]. Sekine et al. invented a joining method for B₄C that uses metal aluminum as the adhesive [4,5]. In this process, molten aluminum can intrude into any tiny cracks on B₄C and joined B₄C together.

Although the average tensile strength of these joined B₄C samples is around 400 MPa which is almost the same tensile strength as bulk B₄C, these joined B₄C samples cannot be used in a corrosive environment because of the use of pure aluminum for joining. Preservative treatment is essential when using the joined B₄C in a corrosive condition.

Ceramic coating is a common method for a preservative treatment for metal, ceramic, plastics, and then there are some methods for ceramic coatings. Taking our purpose into consideration, we chose the ceramic coating method by using precursor

polymer. The method is suitable for covering the surface of materials with large surface area and complex shape. Although there are some reports of ceramic coating by using precursor polymer [6–9], the case that the polymer was applied to the ceramics joined by aluminum has never been reported.

We attempted to coat the joined B₄C with polycarbosilane (PCS). It is well known that PCS can be transformed into silicon carbide (SiC), and Si–H group in PCS is very active when heating [10,11]. It seems that PCS is converted into SiC membrane that prevents the corrosion of Al-rich joining layer. In addition, the active Si–H groups can force ceramic and the membrane to join together without any exfoliation [12,13]. Therefore, the coating method is considered as good.

In this study, we tried to coat the B₄C products joined by pure aluminum with PCS solvent. Our results indicate this coating method to be useful in protecting the joining area from a corrosive atmosphere. After the ceramization of PCS, the products were dunked in an alkali solvent to validate effectiveness of the suggested corrosion-proofing method.

2. Experimental procedure

Fig. 1 shows the outline of this experiment. B₄C samples joined by aluminum were prepared and the thickness of aluminum layer in the samples was less than 2 μm. In our experience, PCS can be dissolved in toluene to a concentration of 0.1 mol/L. The samples were dipped in toluene solvent and cured by heating at 473 K for 1 h under air flow with a heating rate of 14 K/h up to 473 K during

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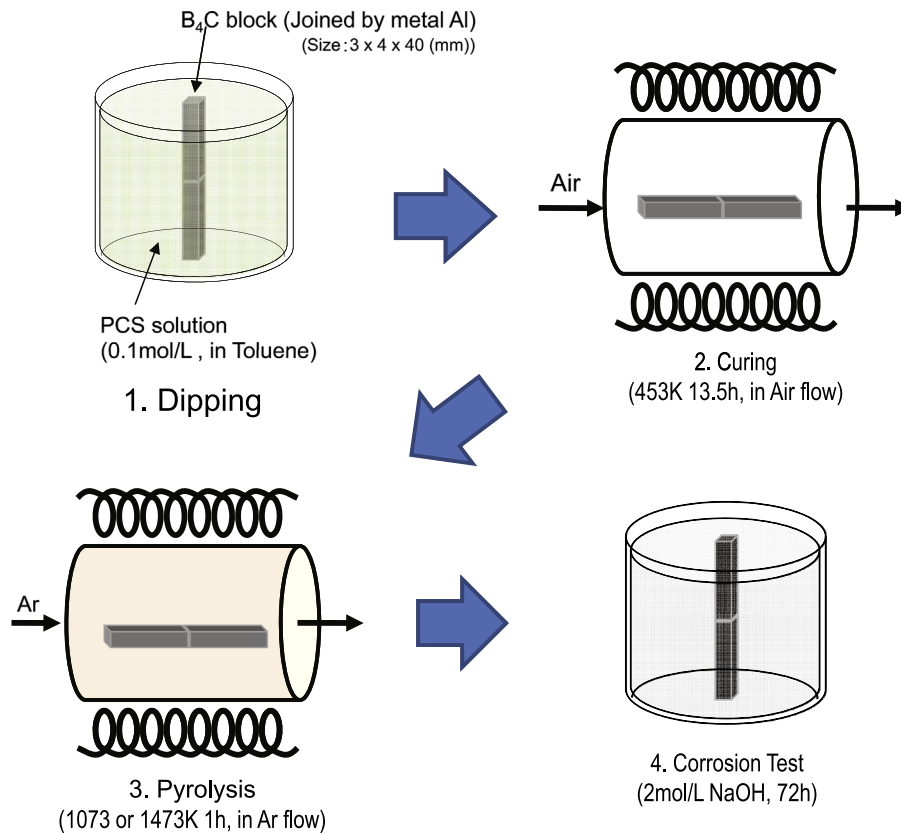


Fig. 1. Outline of this experiment.

curing. After curing, the samples were pyrolyzed at 1073 K or 1473 K in inert atmosphere. This method of dipping, curing, and pyrolysis was repeated thrice. After repetitions of the above-described process, the coated samples and uncoated sample were dipped in a 3 mol/L NaOH solution for 72 h for corrosion testing. Hereafter, the uncoated sample is referred to as the “bare sample,” the sample with coating by pyrolysis at 1073 K is referred to as “BP08,” and the sample with coating by pyrolysis at 1473 K is referred to as “BP12.”

These samples were investigated with scanning electronic microscopy (SEM; JEM-5600, JEOL, Japan), energy dispersive X-ray spectroscopy (EDS; JEM-5600, JEOL, Japan), X-ray diffraction (XRD; RINT2500, Hitachi Ltd., Japan), and a 4-point bending test along with JIS R1601:2008. For XRD, the sample containing 70 wt% of PCS powder and 30 wt% of B₄C powder after curing and pyrolysis at 1073 K or 1473 K was also prepared and carried out as a replication of the PCS–B₄C interface. The mixed powder pyrolyzed at 1073 K was referred to as “BP08 powder” and that pyrolyzed at 1473 K was referred to as “BP12 powder”.

3. Result and discussion

Fig. 2 shows the SEM image and EDS mappings of the cross sections of PCS coating on the joining area of BP12. In all images, the left side of the image shows B₄C and the right side shows the SiC membrane derived from PCS. In the SEM image, the 5/8 on the left of the image shows B₄C, the 1/8 on the right of the image shows atmosphere, and the remainder shows the PCS-derived membrane. No definitive cracking was observed between B₄C and the membrane. Therefore, it appears that there was no exfoliation between B₄C and the membrane. In the EDS mappings, the boron atom (B) of the mapping shows that boron atoms existed in the

area of the membrane and that B₄C-derived boron diffused in the PCS-derived membrane. Aluminum atoms (Al) resulting from the B₄C joining did not reach the membrane surface, and the EDS mapping also reveals that the membrane completely covered the aluminum. Sekine et al. shows that some Al became into Al₃BC and AlB₂ after joining and these are considered to be increased by heating. Therefore, aluminum would be hard to flow out [4,5]. Silicon atoms (Si) and carbon atoms (C) mainly existed in the membrane, suggesting that the membrane was derived from PCS and changed to SiC.

XRD analysis was carried out and the result was shown in Fig. 3. The membrane on the samples was very thin so that it was hard to investigate by using the samples. Consequently, the mixed powder containing PCS and B₄C was prepared as the substitution of the membrane and these powders were cured and pyrolyzed at the same condition of the joined B₄C samples. Many peaks could be observed in these XRD patterns. The peaks of 26.6° and 44.4° indicate the existence of boron oxide. The boron oxide was derived from the cured PCS. In this curing condition, the weight gain by this pre-heating is approximately 8 wt% [14], which is considered to be due to the oxidation. B₄C can be oxidized by heating at more than 873 K and it was considered that some oxygen in polymer reacted with B₄C during pyrolysis. The peaks of 35.6° and 59.9° indicate the existence of crystal SiC, and any other peaks indicate the existence of B₄C.

The loose curve around 35° in a XRD pattern suggests the existence of amorphous SiC. The patterns of the SiC fibers pyrolyzed at 1473 K show the same loose curves [14,15]. It reveals that PCS pyrolyzed at 1473 K usually become amorphous SiC. Besides, the sharp peaks of SiC crystal can be observed from the PCS pyrolyzed at 1773 K [16].

In these XRD patterns, it is interesting to note that the remarkable SiC crystal peaks could be observed in the pattern of BP12 powder,

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