Corrosion of SiC Ceramics Fabricated by Different Methods in NaCl-MgCl$_2$ Molten Salt

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1. Introduction

Silicon carbide (SiC) ceramics and composites have a great potential for critical components of fusion and advanced fission energy systems due to their excellent high temperature properties, irradiation tolerance, inherent low activation and other superior physical/chemical properties [1]. In the fluoride salt-cooled high-temperature reactor (FHR), SiC ceramics and composites have been considered as a coating layer for graphite moderator and TRISO particle fuel and as fuel claddings in some designs [2]. In addition, SiC ceramics are also being considered as out-of-core components such as pump bearings in the liquid-fuel chloride salt-cooled reactor [3]. Although SiC ceramics are well-known for their high corrosion resistance, the corrosion behavior is largely dependent on fabrication methods that lead to different purity, phase composition, and microstructure in the final product.

In this study, we investigated the corrosion behavior of various SiC ceramics fabricated by different methods, reaction-bonded SiC (RBSC), solid-state sintered SiC (SSiC) and chemically vapor deposited SiC (CVD SiC), in NaCl-MgCl$_2$ molten salt. Weight changes after corrosion tests were compared between different SiC ceramics. Microstructures, phase composition, and surface chemistry before and after corrosion tests were analyzed using various tools.

2. Experimental Methods

Corrosion test samples were machined from plates of three kinds of SiC ceramics, RBSC, SSiC, and CVD SiC. Two specimens for each material were used in each corrosion test and the dimensions of the specimens were 20x10x3 mm. The specimens were put into alumina crucibles filled with chloride salts and the crucibles were placed in a furnace installed in a glove box maintaining an Ar atmosphere. The corrosion tests were performed at 650°C for 200 and 500 h in a molten salt composition of NaCl:MgCl$_2$ = 57mol%:43mol%. The molten salt was heat treated at 300°C for 24 h and at 550°C for 48 h for the purpose of purification, and an additional salt purification was performed using Mg metal flakes. Oxygen and moisture contents in the glove box were maintained less than 20 and 2 ppm, respectively.

The weight of specimens before and after corrosion tests was measured using an electronic balance with an accuracy of 0.01mg. The surface microstructure was analyzed using a scanning electron microscope (SEM) and the surface roughness measured using an atomic force microscope (AFM). Any phase formation on the surface after the corrosion test was characterized using a grazing incidence X-ray diffractometry (GIXRD).

3. Results and Discussion

Fig. 1 shows the weight change after the corrosion test for three types of SiC specimens, with Hastelloy N material being tested together as a reference specimen. The greater weight loss in the 200 h test specimen is likely to be due to the difference in the content of metallic impurities such as Fe and Cr after refining. The weight loss after the 500 h corrosion test was 0.22 mg/cm^2 for RBSC, 0.19 mg/cm^2 for SSiC, and 0.047 mg/cm^2 for CVD SiC, which is a very low value compared to 1.16 mg/cm^2 for Hastelloy N.

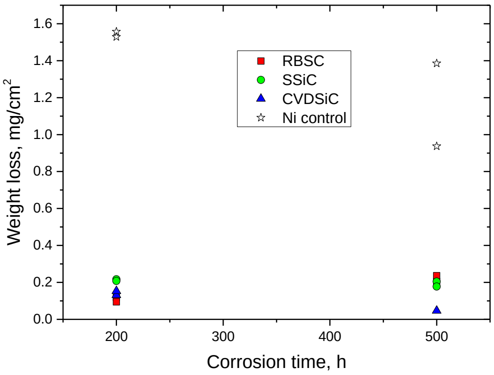

Fig. 1. Weight change of various types of SiC ceramics after corrosion tests in molten chloride salts. Data for Hastelloy N were also included as a reference.

The GIXRD analysis results before and after the corrosion test are depicted in Fig. 2. The GIXRD results show that no phase change is observed in the SSiC and CVD SiC specimens, whereas a Mg$_2$SiO$_4$ (forsterite) phase is formed on the surface of the RBSC specimens after the corrosion test. It is thought to be due to selective corrosion of free Si present in the RBSC specimens and a reaction with MgO in the molten salt, leading to the higher weight loss as shown in Fig. 1.

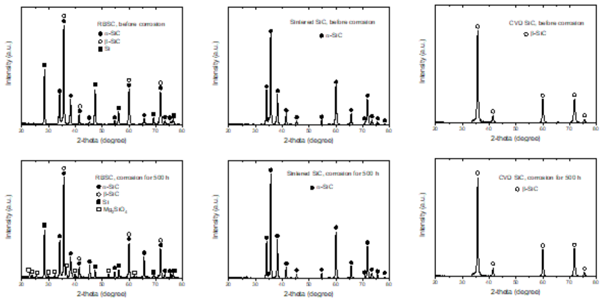

Fig. 2. GIXRD results of three types of SiC ceramics before and after the corrosion test for 500 h in molten chloride salts.

Fig. 3 shows microstructures of various SiC ceramics before and after the corrosion test. In the RBSC specimen, it can be observed that a granular corrosion reaction product is formed especially on the residual Si phase. The reaction product corresponds to the Mg_2SiO_4 phase based on the XRD results. On the other hand, in the SSiC and CVD SiC specimens, slight corrosion traces can be observed along the grain boundaries.

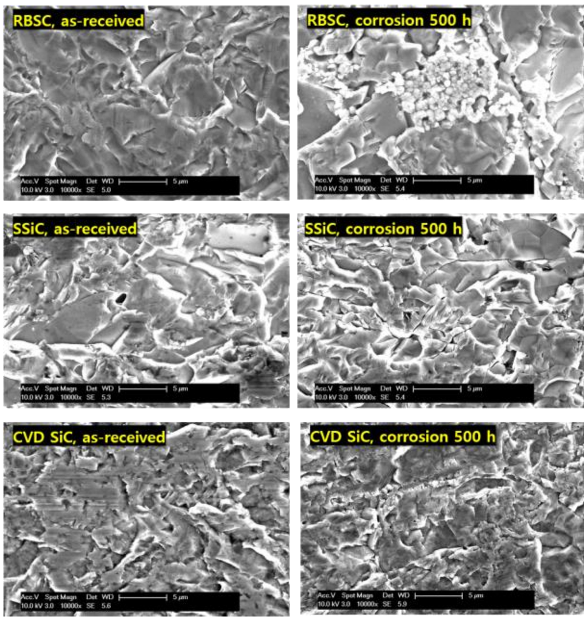

Fig. 3. SEM microstructures for the surfaces of three types of SiC ceramics before and after the corrosion test for 500 h in molten chloride salts.

AFM analysis was performed to evaluate any change in surface roughness before and after the corrosion test as shown in Fig. 4. The surface roughness showed a value in the range of 200-400 nm, and no significant change was observed between different types of SiC specimens or before and after the corrosion test. Additional analysis needs to be performed after a long-term corrosion test.

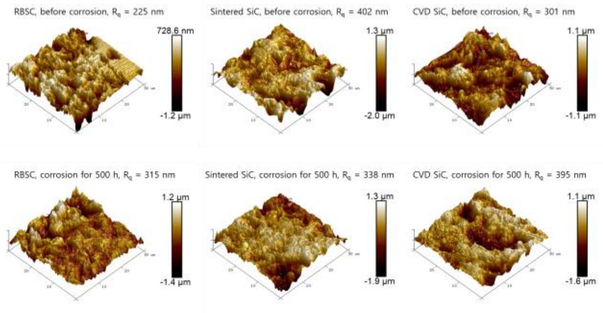

Fig. 4. AFM analyses results of three types of SiC ceramics before and after the corrosion test for 500 h in molten chloride salts.

4. Conclusions

Corrosion tests were carried out at 650°C up to 500 h on three types of SiC ceramics, including RBSC, SSiC, and CVD SiC, in NaCl-$MgCl_2$ molten salt. All SiC materials showed very low corrosion weight loss compared to Hastelloy N. The CVD SiC specimen with the highest purity showed the best corrosion resistance, and the RBSC specimen showed relatively high corrosion weight loss. This is thought to be due to selective corrosion of the residual Si phase presents in the RBSC material.

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