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Formation of Ti_3SiC_2 Interphase of SiC Fiber by Electrophoretic Deposition Method

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ABSTRACT

Due to its stability at high temperature and its layered structure, Ti_3SiC_2 MAX phase was considered to the interphase of SiC_f/SiC composite. In this study, Ti_3SiC_2 MAX phase powder was deposited on SiC fiber via the electrophoretic deposition (EPD) method. The Zeta potential of the Ti_3SiC_2 suspension with and without polyethyleneimine as a dispersant was measured to determine the conditions of the EPD experiments. Using a suspension with 0.03 wt.% ball milled Ti_3SiC_2 powder and 0.3 wt.% PEI, Ti_3SiC_2 MAX phase was successfully coated on SiC fiber with an EPD voltage of 10 V for 2 h. Most of the coated Ti_3SiC_2 powders are composed of spherical particles. Part of the Ti_3SiC_2 powders that are platelet shaped are oriented parallel to the SiC fiber surface. From these results we expect that Ti_3SiC_2 can be applied to the interphase of SiC_f/SiC composites.

Key words : Interphase, MAX phase, SiC fiber, EPD, Ti_3SiC_2

1. Introduction

Since SiC_f/SiC composites have a high strength, an excellent fracture toughness, corrosion resistance, chemical stability, etc. at elevated temperatures as well as an excellent irradiation resistance, many studies are being conducted concerning applications to high-temperature structures such as gas turbine, heat exchanger and next-generation nuclear reactor parts, etc..[1-3] An interface between SiC fiber and SiC matrix within a composite cause the produced cracks to be deflected along the interface, playing an important role for improving fracture toughness of the SiC_f/SiC composites.[4-6] For an interphase coated onto SiC fibers, PyC and h-BN with a lamellar structure are being most widely applied, displaying excellent mechanical characteristics upon application as the interphase.[7,8] However, PyC and h-BN have a disadvantage of being vulnerable to oxidation at high temperatures so that there is a need for improvement in oxidation resistance of the interphase for application of the SiC_f/SiC composites as a high-temperature structural material.[9,10] Also, in the case of application as a component for nuclear reactor, the interphase should exhibit a high stability and an excellent resistance to high-temperature oxidation in neutron irradiation environments.[11]

Ti_3SiC_2 MAX phase is a metal carbide ternary system ceramic showing a low thermal expansion coefficient, excellent thermal and electrical properties, an excellent high-temperature strength along with an elastically stiff character.[12-15] Meanwhile, however, MAX phase exhibits properties similar to those of metals, such as being very stable for high-temperature oxidation and thermal shock, easily fabricated, with characteristics of a strong impact resistance. MAX phase has characteristics of being easily bent due to formation of kink bands and delamination cracks, and shows a non-isotropic structure by forming a lamellar structure on the (001) basal plane as a hexagonal structure.[16,17] Therefore, when the Ti_3SiC_2 MAX phase is applied as an interphase, cracks are deflected at the interphase accompanied by easy pull-out of the fiber phase from inside of the composite so that ceramic-matrix composites having excellent mechanical characteristics such as improved fracture toughness, etc. along with oxidation resistance may be manufactured. For coating of MAX phase, diversified studies have been performed, of which the studies of using CVD were conducted primarily.[18-20] However, in the case of coating of MAX phase by CVD, the basal plane was formed in a direction perpendicular to the substrate so as not to be able to contribute to an improvement of fracture toughness for the composite when applied as an interphase for the composite.[21] Although there have been attempts with several CVD methods, there is no study yet with vapor deposition to produce the lamellar structure in a direction parallel to fibers.

Electrophoretic Deposition (EPD) is a method for depositing powders charged in a suspension into a preform through an electric field, and employed for manufacturing of composites by deposition of the matrix phase in a SiC_f/SiC composite.[22,23] In addition to deposition of the matrix phase, ceramic powders may be coated onto substrates or fibers, and studies on coating of PyC interphase onto SiC fibers through EPD have been conducted.[24-26] Yoshida et al have

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coated the PyC interphase onto SiC fibers within a SiC fabric by using graphite suspension, and affirmed improvement in fracture toughness characteristics by preparation of SiC_f/SiC composites using manufactured preforms.[26] When MAX phase is coated through EPD, coating may be realized on the basis of non-isotropy of the MAX phase powder so that lamellar structures within the particle are arranged in a direction parallel to the SiC fiber. Filbert-Demut et al have deposited the MAX phase powder onto large-diameter SiC fibers through EPD, and Mishra et al maintained crystal directions of the MAX phase powder particles to be constant by using a magnetic field over a flat plate substrate.[27,28]

In the present study, Ti_3SiC_2 MAX phase was coated onto Tyrano-SA3 SiC fibers through EPD method. High-energy ball milling was employed to reduce the particle sizes of the MAX phase powder, and PEI was used as a dispersant for coating. For uniform coating onto SiC fibers, a study was made on the variables of EPD, and the Ti_3SiC_2 MAX phase could be uniformly coated onto SiC fibers through appropriate conditions. The case could be affirmed for the MAX phase coated onto SiC fibers where the particles having non-isotropy were coated in a direction with the lamellar structure being parallel to the fibers. When SiC_f/SiC composites are manufactured through this method, it is considered that the composites with excellent resistance to high-temperature oxidation while displaying excellent mechanical characteristics may be manufactured.

2. Experimental Procedure

To coat MAX phase onto SiC fibers, Ti_3SiC_2 powder (MAXTHAL 312, Sandvik AB, Hallstahammar, Sweden) was employed. Ti_3SiC_2 powder was subjected to ball milling for 30 min by using a planetary ball-mill. Shapes of the powders before and after ball milling were analyzed by using a scanning electron microscope (SEM, FE-SEM; S-4800, Hitachi, Tokyo, Japan), and sizes were analyzed by using MALVERN Zetasizer (Malvern, Worchestershire, UK). As a dispersant of the suspension for EPD, branched Polyethyleneimine (PEI; Sigma-Aldrich Co. LLC., Saint Louis, USA) with a molecular weight of 10,000 was used. PEI consists of a structure where the molecular structures of [-CH2-CH2-NH-]n are connected. Ti_3SiC_2 MAX phase powder together with PEI was dispersed in the suspension by ball milling with SiC balls for 24 h using ethanol as a medium. To determine optimum suspension conditions, Ti_3SiC_2 powder suspension and the suspension with addition of Ti_3SiC_2 powder and PEI were prepared and zeta potentials were measured. Zeta potential was measured with ethanol and water as solvent by using MALVERN Zetasizer, with HNO_3 and NaOH being added to adjust pH.

For Tyranno-SA3 (Ube Industries, LTD., Japan) fiber fabric woven at 0/90° · EPD was conducted by using Ti_3SiC_2 powder suspension. A given size of SiC fabric together with the electrodes were mounted to the center of EPD apparatus and then impregnated in the suspension, while the electrodes were installed at an interval of 20 mm on both sides. EPD experiments were conducted for 1 ~ 2 h under applied voltages of 5 ~ 20 V, and 10 W of ultrasonic wave was applied for a given time. For the SiC fabric after EPD experiments, microstructures were checked by using a scanning electron microscope following drying in an oven at 60°C for 24 h.

3. Results and Discussion

To coat Ti_3SiC_2 MAX phase powder onto Tyranno-SA3 SiC fibers of about 8 mm in diameter, the sizes of the powder were reduced through ball milling. SEM pictures for the initial Ti_3SiC_2 powder and the powder subjected to high-energy ball milling for 30 min are shown in Fig. 1. Initial Ti_3SiC_2 particles have a wide plate shape with a high non-isotropy due to the lamellar structure. Although the particle sizes of Ti_3SiC_2 powder were greatly reduced through ball milling, it was affirmed that the particles which had showed the plate shape due to the lamellar structure of MAX phase were pulverized mostly into spherical particles as the non-isotropy disappeared by ball milling. While some plate-shaped particles were observed, the particle sizes were not sufficiently small so that the plate shape did not appear to be exhibited when the particles were pulverized into a submicron size through milling. According to the measurement results of particle sizes by using a particle size analyzer to determine particle sizes, the average size of particles was affirmed to have been reduced from 5.665 mm of the existing powder to 0.843 mm after ball milling. Coating onto SiC fibers having a small diameter is considered possible by reduction of particle sizes through ball milling.

For coating of Ti_3SiC_2 onto SiC fibers by EPD method, the suspension was produced by using the pulverized powder after ball milling. For the dispersion of Ti_3SiC_2 powder, PEI was employed as a dispersant. The measurement results of zeta potential as a function of pH for the suspensions used in the experiments are shown in Fig. 2. Fig. 2(a) shows zeta potential values as a function of pH for the suspension consisting of Ti_3SiC_2.in ethanol as a solvent. IEP was measured to be pH 7.5, and the zeta potential showed the higher positive value, the lower the pH, exhibiting a high positive voltage of more than 80 mV. Fig. 2(b) shows zeta potential values as a function of pH when 0.3 wt.% of PEI as a dispersant was added to Ti_3SiC_2 powder suspension. PEI is a polymer-based dispersant having a long branch, easily attached

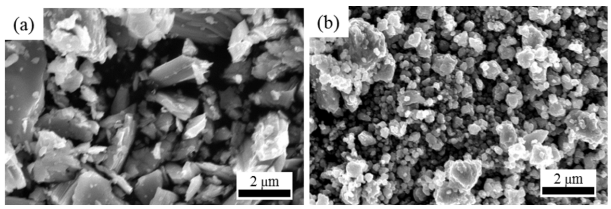

Fig. 1. SEM micrographs of Ti_3SiC_2 powder: (a) as-received and (b) after ball-milling for 30 min.

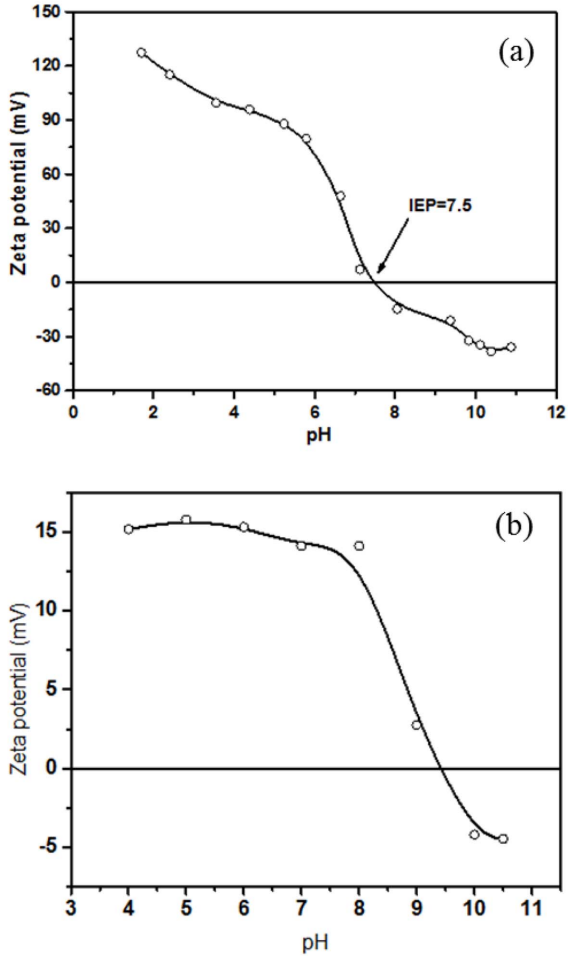

Fig. 2. Zeta potential versus pH of (a) Ti_3SiC_2 suspension and (b) Ti_3SiC_2 suspension with 0.3 wt.% PEI.

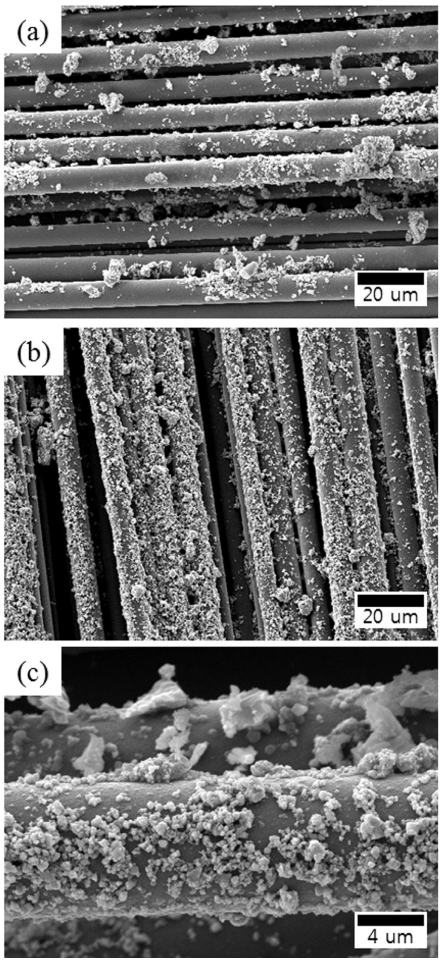

Fig. 3. SEM micrographs of the SiC fibers coated with Ti_3SiC_2 suspension (a) without PEI, and (b) and (c) with PEI by electrophoretic deposition method.

to the surfaces of the powder having a negative zeta potential value, showing a positive zeta potential value in itself.[28,29] Thus, EPD experiments were conducted under the condition of pH 8 where Ti_3SiC_2 powder suspension had a negative zeta potential value while the suspension with addition of PEI dispersant showed a positive zeta potential value. IEP of the suspension with addition of PEI to Ti_3SiC_2 powder showed pH 9.4, while the zeta potential had a positive charge below pH 9 and continued to maintain a positive charge value of about 15 mV as the pH was lowered below 8. In general, the suspension is known to be stable for a zeta potential higher than 30 mV. In the present study, although the zeta potential for the suspension with addition of PEI dispersant to Ti_3SiC_2 powder suspension was too low at about 15 mV to realize a stable colloidal suspension, fibers could be coated by application of EPD method in a short term. EPD mobility is proportional to intensity of electric field, dielectric constant of liquid, viscosity of liquid, and zeta potential values. To perform MAX phase coating using a suspension with a low zeta potential value, experiments were conducted with the intensity of electric field as a variable.

Shown in Fig. 3 are the results for coating onto SiC fibers of the suspension with 0.03 wt.% of Ti_3SiC_2 powder suspension and the suspension with 0.03 wt.% of MAX phase powder and addition of 0.3 wt.% PEI as a dispersant using EPD. For the Ti_3SiC_2 powder suspension, coating was conducted after adjustment to pH 3 where the zeta potential showed a value higher than 90 mV, and EPD voltage for coating was maintained at 10 V for the coating time of 1 h. Fig. 3(a) shows the result of coating Ti_3SiC_2 powder suspension, where SiC fibers were not uniformly coated and large particles formed by agglomeration of small powders were shown to be unevenly attached to SiC fibers when the dispersant was not added. Fig. 3(b), (c) show SEM results for the specimens coated after addition of a dispersant to the suspension. When the dispersant was added, the cases where small particles were agglomerated for non-uniform attachment were hardly observed, and small particles could be seen to be attached relatively uniformly to SiC fibers instead. Although the zeta potential for the suspension with addition of PEI used as a dispersant showed too low a value of about 15 mV for formation of a stable suspension, it was affirmed

that the powder coated through EPD had been sufficiently dispersed for coating onto SiC fibers. However, Ti_3SiC_2 particles were observed to have failed to uniformly cover the entire SiC fibers, leaving many unfilled areas. Also, whereas SiC fibers on the outer surfaces of SiC fabric were relatively uniformly coated, Ti_3SiC_2 particles could be seen to be scarcely attached onto the SiC fibers inside. To use Ti_3SiC_2 coating layer as the interphase of SiC_f/SiC composites, all SiC fibers within the SiC fabric should be uniformly coated. Therefore, experiments for optimization of the variables for EPD were conducted. In the SEM image of Fig. 3(c), a wide face can be affirmed to be attached to SiC fibers in the case of some plate-shaped particles showing non-isotropy among Ti_3SiC_2 particles attached to the SiC fibers. Therefore, when Ti_3SiC_2 particles are pulverized into small sizes for preparation of plate-shaped particles, SiC fibers can be easily pulled out to improve fracture toughness of the composites when the lamellar structure was made to be parallel to SiC fibers so as to be applied to interfaces of the SiC_f/SiC composite.[30]

Based on the basic experimental results, EPD voltages and coating times were changed for uniform coating of Ti_3SiC_2 particles onto SiC fibers, and ultrasonic wave was applied for a given time to allow a sufficient amount of Ti_3SiC_2 particles to be put into the SiC fabric. In Table 1, EPD coating conditions are given. The results of coating after application of ultrasonic wave for a given time in the experiments (Experiment Nos. 5, 6) and the results without application of ultrasonic wave (Experiment Nos. 3, 4) under the same EPD voltage and time did not show a large difference. When the powder used as SiC matrix phase is deposited in SiC fabric for production of SiC_f/SiC composites, the suspension concentration of the powder for deposition is generally made to be very high at more than 10 wt.%. In such case as above, a study was conducted where the use of ultrasonic wave prevented thick coating of SiC matrix phase powder onto the outside of SiC fabric so as to promote infiltration of SiC matrix phase particles into the fabric in the early stages of EPD.[31] In the case of the present study, however, since a thin coating was applied to SiC fibers and the concentration of 0.03 wt. for the suspension was very low in comparison with the slurry for composite production resulting in prevention of a thick coating of particles onto the outside of fabric in the early stages of EPD experiment, application status of the ultrasonic wave is not considered to have greatly affected the coating onto SiC fibers inside SiC fabric.

Table 1. Electrophoretic Deposition Experiments Condition

Experiments	Voltage	Time	Ultrasonic
1	10 V	1 h	
2	20 V	1 h	
3	5 V	2 h	
4	10 V	2 h	
5	5 V	2 h	1 h
6	10 V	2 h	1 h

In Fig. 4, SEM results for Experiment No. 2, 3, 4 are shown. Fig. 4(a) and (b) show SEM micrographs for the specimens with application of 20 V, 1 h and 5 V, 2 h for EPD voltage and time, respectively. When the EPD voltages were increased, uncoated SiC fibers were frequently observed as Ti_3SiC_2 particles were distributed unevenly and in a large thickness on the fibers outside SiC fabric. When the EPD voltage was lowered further to 5 V, overall coating could be observed to have been poorly implemented as Ti_3SiC_2 particles were scarcely attached, failing to cover the entire SiC fibers. Fig. 4(c) and (d) show SEM micrographs of the specimen with the EPD voltage maintained at 10 V and the time at 2 h. As shown by the SEM picture, most SiC fibers can be affirmed to have been uniformly coated with Ti_3SiC_2 particles. In the SEM micrograph (Fig. 4(d)) magnifying one of the SiC fibers, Ti_3SiC_2 particles can also be affirmed to have been uniformly coated. Most of Ti_3SiC_2 particles used in the present study showed a spherical shape, and mechanical characteristics of the composite may not be greatly improved when applied as the interphase, since the basal plane can be randomly arranged for coating in the case of spherical particles. To apply Ti_3SiC_2 powder as the interphase for SiC_f/SiC composites, there should be more particles having non-isotropy in the initial powder. However, since Ti_3SiC_2 MAX phase is shock-resistant and shows the characteristics of being easily deflected under the influence of kink bands, the effects on the characteristics of the composite need to be checked when applied as the interphase.[16] As some SiC fibers were observed to have areas without uniform coating of Ti_3SiC_2 particles while some SiC fibers inside SiC fabric had uniform coating of the Ti_3SiC_2 particles, further studies on EPD coating conditions for optimization are needed to ensure uniform coating of all SiC fibers inside SiC fabric. Consequently, although optimization studies for some conditions are required, Ti_3SiC_2 MAX phase powder could be coated onto SiC fibers inside SiC fabric to a given thickness by using the EPD method, and the

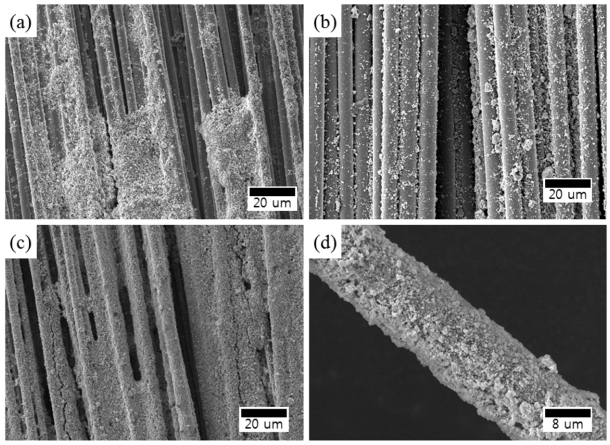

Fig. 4. SEM micrographs of the SiC fibers coated with Ti_3SiC_2 suspension with PEI at different EPD condition. The EPD voltage and time were (a) 20 V and 1 h, (b) 5 V and 2 h, (c), (d) 10 V and 2 h.

possibility for its utilization as the interphase was verified. In the future, when SiC_f/SiC composites are manufactured by using the powder employed as a SiC matrix phase for SiC fabric preform with Ti_3SiC_2 MAX phase powder coated as the interphase by using this method, high-temperature oxidation characteristics of the composites may be expected to be greatly improved.

4. Conclusions

In the present study, coating of Ti_3SiC_2 MAX phase powder onto SiC fiber surfaces was attempted by using electrophoretic deposition (EPD) method. Ti_3SiC_2 powder was reduced in sizes through ball milling, and dispersed in a suspension by using PEI. According to the results of measuring zeta potentials for Ti_3SiC_2 powder suspension, IEP was shown to be 7.5, and the higher positive voltages were observed as the pH was reduced. Zeta potential for the suspension of Ti_3SiC_2 powder with addition of PEI showed a positive voltage of about 15 mV below pH 9. While the Ti_3SiC_2 suspension without using PEI dispersant was not coated properly onto SiC fibers, well-dispersed Ti_3SiC_2 particles were affirmed to be coated onto SiC fibers according to the results of experiments with pH adjusted to 8 for the suspension with addition of 0.3 wt.% of PEI. For uniform coating of Ti_3SiC_2 particles inside SiC fabric, EPD experimental conditions were optimized. When coating was implemented in the suspension with addition of 0.3 wt.% PEI as a dispersant to 0.03 wt.% Ti_3SiC_2 powder under the condition of 10 V in voltage for 2 h, uniform coating onto SiC fibers was affirmed. Although most particles coated onto the SiC fibers were spherical without showing a lamellar structure, some plate-shaped particles were affirmed to be coated in a direction parallel to the SiC fibers. Through EPD method, Ti_3SiC_2 MAX phase powder can be coated onto SiC fibers, and its application as the interphase for SiC_f/SiC composites may be expected in the future.

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