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Fabrication and Properties of SiCpc Fiber Reinforced Nickel

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Saving energy and saving resources are important themes for advanced technology. Reducing weight and improving heat-resistance of materials are the most effective methods to solve the problems. It is well known that the SiCpc (SiC fiber from poly-carbosilane) has high specific strength and high heat-resistance. Nickel is one of the most important metals for super alloys. Then a composite from these materials is expected to have high specific strength and high heat-resistance. HI-NICALON^{TM} was used as the reinforcement SiCpc. Nickel was deposited on the SiCpc fibers by electroless deposition at room temperature. Commercially available electrolyte was used for the deposition. The surface of the deposited nickel was smooth and compact after the deposition. Volume fraction of the SiCpc fiber was controlled by deposition time. The strength of the as-deposited monofilament composite was the same as the theoretical value by the rule of mixtures. Interfacial reaction between SiCpc and nickel was hardly observed and the strength did not change after holding at an elevated temperature up to 600 ℃ for 9 hours. On the other hand, the strength of the heat treated fiber decreased after heat treatment at 700℃ for 1 hour. Strange reaction was observed at a higher temperature, and the fiber lost the strength.
Key words: SiCpc Fiber, Nickel, Composite, Interfacial reaction, Heat resistance.

1. INTRODUCTION

Saving energy and saving resources are serious problem in advanced industries. Light and heat-resistant materials are required in these fields. Fiber Reinforced Metals(FRM) is one of the solution for the problem.

SiCpc fiber is one of the promising reinforcements because of its high specific strength and high heat-resistance. Ni is an important element for heat-resistant alloys. Therefore the combination of these materials is expected to have high performance for an ecological conscious material.

2. EXPERIMENTAL PROCEDURE

SiCpc fiber of Nicalon, provided by Nippon Carbon Co. Ltd., was used for the reinforcement. The diameter of a fiber is 14 μm. 500 SiCpc fibers are sized to form a yarn. The size was removed by regular method of JIS R7601. The yarn was widened to 30 mm in width for homogeneous Ni plating.

Ni was plated on the fiber by electroless deposition to form a monofilament composite. Pre-treatment of conditioning, sensitizing and activating were performed before the Ni plating. Conditioning was performed to remove stain on the fibers and sensitizing and activating were to form nuclei for Ni deposition. The yarn was cleansed by pure water before the Ni plating.

Commercially available electrolyte,

TOPNICALON-LPH(OKUNO CHEMICAL INDUSTRIES CO.,LTD.) for semiconductor was adopted, as contamination of P or B was little. The concentration of the electrolyte was 250 ml/l. The pH of the electrolyte was controlled at 6.5 and the temperature was kept at 90 ℃ in an oil bath. Fiber volume fraction, hereafter V_f, in the monofilament composite was controlled by the plating time between 5-100 min, i.e. thickness of the plated Ni. V_f was determined by the weight change before and after Ni plating. The surface and the cross section of the monofilament composite were observed by SEM and EDX.

The Ni plated filaments were heat treated at 600-1000 ℃ for 1-3 hours to examine the thermal stability. The strength of the monofilament composites before the heat treatment and after heat treatment was determined. The interfacial reaction was also observed by SEM and EDX.

3. RESULTS AND DISCUSSION

The fiber volume fraction of the monofilament composites was obtained as a function of the plating time as shown in Fig.1. The V_f was precisely controlled by this method.

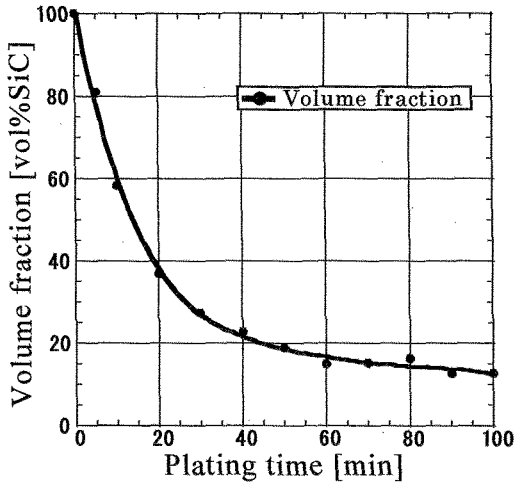
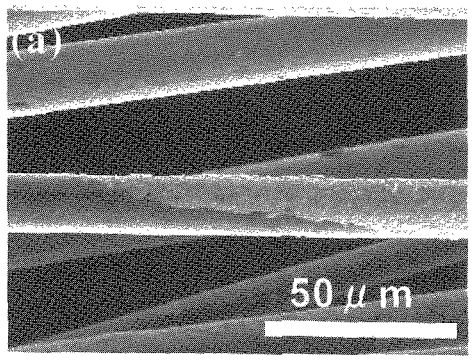
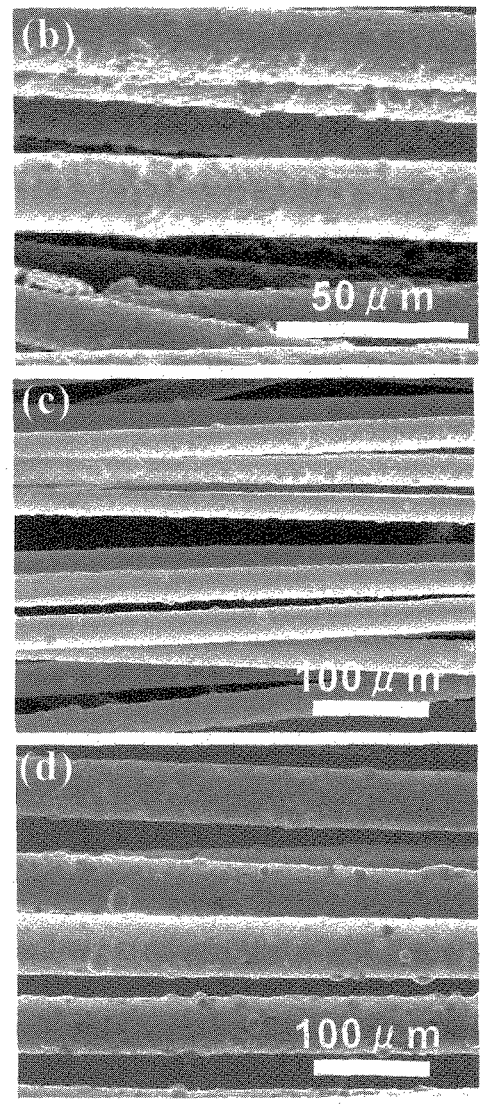


Fig.1 Fiber volume fraction of the monofilament composites as the function of plating time.

The surfaces of the Ni plated filaments are shown in Fig. 2. Some part of the filament was not covered with Ni, when the volume fraction was higher than 80 % as shown in the left lower side of the middle fiber. On the other hand, whole surface of the filaments was covered by Ni, when the V_f was lower than 60 %, though the surface of the Ni was slightly uneven. The unevenness of the Ni is not harmful for fabricating a bulk composite, because the bumps are squeezed to be flattened during consolidation by hot-pressing.





(a)V_f:80 [vol%SiC] (b)V_f:60 [vol%SiC]
(c)V_f:35 [vol%SiC] (d)V_f:15[vol%SiC]

Fig.2 SEM images of monofilament composites.

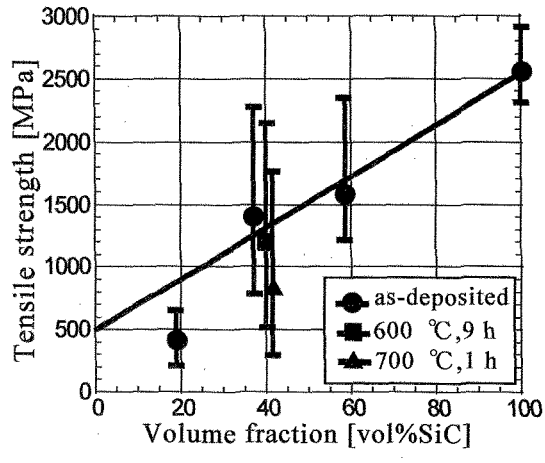


Fig.3 Observed tensile strength of monofilament composite and theoretical value as the function of volume fraction.

The strength of the monofilament composites is shown as the function of volume fraction as circles in Fig.3. The results of heat treated composite noted by ▲and■ will be described later. The thick line shows the theoretical value by ROM. The strength of the SiCpc fiber was determined as 2547 MPa by experiment.

The strengths of the monofilament composites are almost same as the theoretical value except the low V_f as 20 %. The observed values for low V_f composite should be slightly lower than the real ones, because thickness of the plated Ni for low V_f was very thick and some of the filaments were slightly curled. Then the applied stress was not exactly uni-axial, as the curled fiber was rigid and stiff.

A cross section of the monofilament composite after heat treatment at 600 ℃ for 9 hours is shown in Fig.4. Particular morphological change was not observed in the plated Ni, SiCpc fiber nor the interface between them.

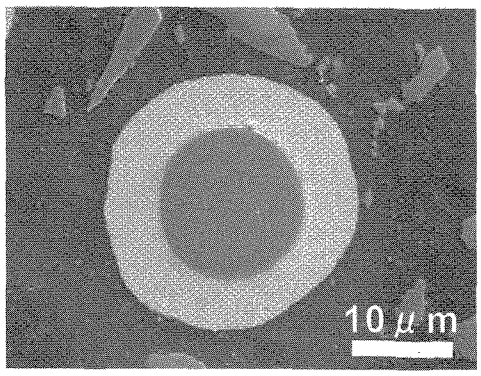


Fig.4 SEM image of the cross section of a monofilament composite after heat treatment at 600℃ for 9 hours.

Elemental analysis was carried out at the interface between the fiber and the Ni by EDX. Heat treatment was performed at 600 ℃ for 9 hours. The results are shown in Fig.5.

No obvious diffusion was observed in these results, though slight slopes are observed at the interface due to the resolution power of the EDX.

As shown in Fig.3, the strength change of the monofilament composite with 40 volume % of fiber was very little after heat treatment at 600 ℃ for 9 hours, even though the average strength showed slightly lower values. The strength of the fiber may not be changed by the heat treatment, even the observed strength was lower than the original values. Because handling of the fiber was difficult and slight damage may be caused, as fibers were curled and sticked each other by sintering during heat treatment.

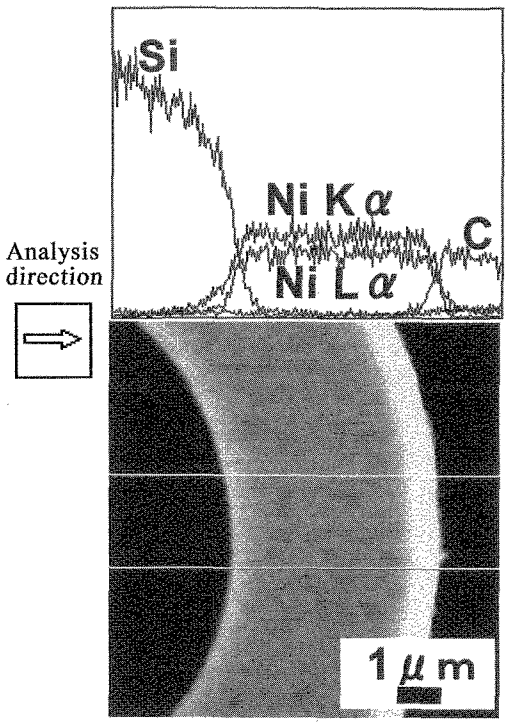


Fig.5 Elemental analysis at the interface by EDX after heat treatment at 600 ℃ for 9 hours.

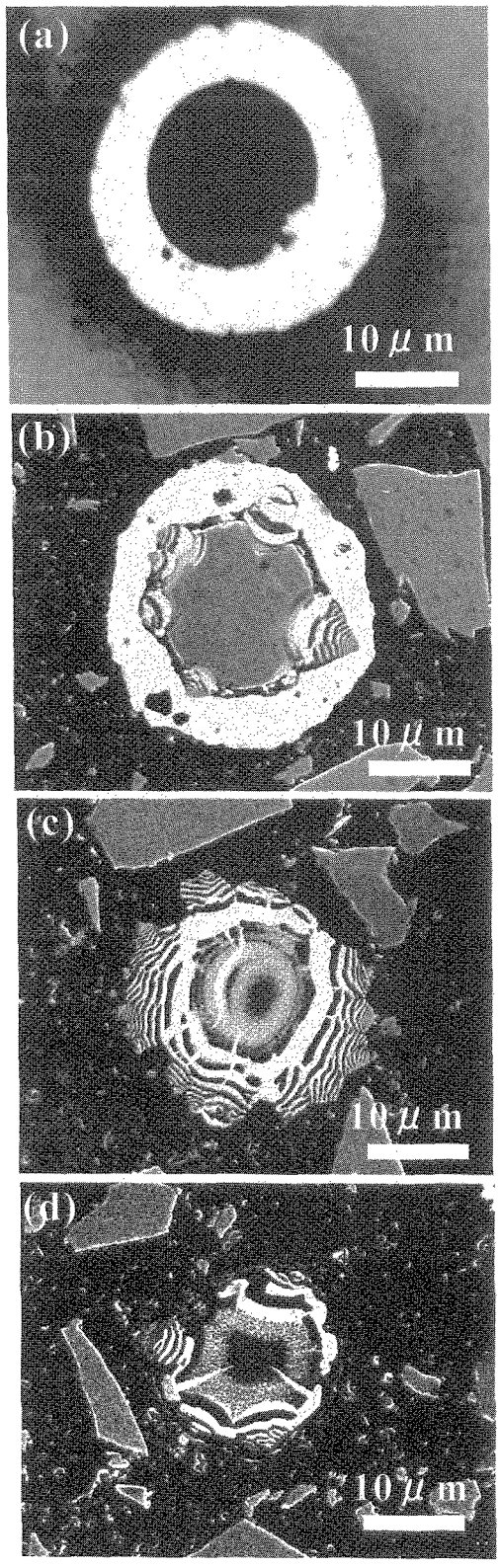
On the other hand, the average strength of the monofilament composite with 40 volume % of fiber is obviously lower after heat treatment at 700 ℃ for 1 hour as shown in Fig.3.

Anyhow, the strength decreased by the heat treatment. Then further heat treatment was carried out to examine the effect of the heat treatment at various temperatures. The heat treatment time was 1 hour for each temperature. The results are shown in Fig.6.

Slight reaction is recognized at the right lower side of the interface when the temperature was 700 ℃ as shown in Fig.6(a).

When the temperature was 800 ℃, several reaction parts are observed at the interface as shown in Fig.6(b). The shape of every reaction part is triangular and it consists of many black and white stripes though the size of each reaction part is different. They look like a cross section of a bamboo shoot. The bottom white stripe of the reaction part is thick and curved inward to the fiber.

The space between the stripes at the top part is narrower than the bottom part. Every top of the reaction part touches the plated Ni, besides the interface without reaction has a gap.



(a) 700 ℃, (b)800 ℃
(c) 900 ℃,(d)1000 ℃

Fig.6 SEM images of cross section of monofilament composite after heat treatment at various temperatures for 1 hour.

When the temperature became higher as 900 ℃, whole periphery of the SiCpc fiber was covered with the stripes as shown in Fig.6(c). The most inner white stripe is 3-5 time thicker than the others. Most reaction parts are connected with each other. A white stripe is mainly connected with other white stripe, and a dark stripe connected with other dark stripe. They form multiple concentric circles. Not only the stripes but also sharp narrow white parts in radial direction were observed in this photograph. They look like a spike. The center of the SiCpc fiber is dark and a light gray circle is observed outside the dark center. The plated Ni already disappeared in this stage.

When the temperature became 1000 ℃, the fiber lost the original shape as shown in Fig.6(d).

The strength of these heat treated fibers was not able to be observed because every fiber was extremely weak and brittle.

If the reaction mechanism is clarified, it will be a clue to prevent the reaction. Some intermediate layer between the fiber and the matrix is necessary to prevent the reaction. Carbon or alumina layer may not be the candidate as they are reactive to Ni(ref)[1],[2]. Stable oxide film, such as magnesia may be effective.

4. CONCLUSION

Developing a heat resistant composite of SiCpc fiber reinforced Ni was failed because of serious reaction at the interface. Some intermediate layer, such as magnesia, between the fiber and the matrix is necessary to prevent the reaction.

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