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Behavior of electrical resistance of SiC_{CVD} fiber and development of micro-heater with SiC_{CVD} fiber

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Abstract Behavior of electrical resistance was examined in room temperature and elevated temperatures up to 1000 °C for two types of SiC_{CVD} fibers with diameters of 140 and 70 μm, respectively. The results showed that electrical resistance showed a good linear relationship with the length of fibers. Electrical resistance decreased as temperature increased, besides, temperature coefficient of electrical resistance was a minus constant, -5.2×10^{-4} °C^{-1} except that in the first heating. In the first heating, electrical resistance and temperature coefficient increased and had a peak in the range of 550–700 °C owing to the burning of the carbon-rich layer on the fiber surface. It suggested that behavior of electrical resistance of the fibers depended mainly on the carbon core and the carbon-rich layer. It was confirmed that SiC_{CVD} fiber could be used as heating elements for micro-heater and finally a micro-heater using SiC_{CVD} fiber as heating elements was developed.

Introduction

SiC_{CVD} fiber, produced by Textron Specialty Materials Co. Ltd., is often used as one of the typical reinforcements of fiber-reinforced composites such as SiC/Al, SiC/Ti, and SiC/SiC composites [1–7]. Furthermore, the fiber, produced by CVD (Chemical Vapor Deposition), is a continuous ceramic fiber with a diameter of 140 or 70 μm; besides, it has a carbon (graphite) core of about 30 μm in diameter at the center and a carbon-rich layer of about 1 μm in thickness on the external surface. The microstructure and composition of SiC_{CVD} fiber have been studied and characterized by Ref. [8–10]. The authors have studied the gauge length dependence of the strength for SiC_{CVD} fiber [11] and the strength reliability of SiC_{CVD} fiber bundle [12]. Also, the strength reliability of fiber-reinforced composites was investigated by studying SiC/Al composites with SiC_{CVD} fiber, referring to fiber volume fraction [13], interfacial shear strength [2], and the size effect of the strength [14]. Up to now, the main attention has been paid to the high performance of mechanical properties of SiC_{CVD} fiber-reinforced composites.

It is well known that SiC ceramic and carbon do not only have high performance in mechanical properties but also electrical and thermal properties. Therefore, other applications were developed including as semiconductor or electrical heater [15]. However, there is no relevant research on electrical and thermal properties of SiC_{CVD} fiber, except for the coefficient of thermal expansion offered by the manufacturer.

In this work, behavior of electrical resistance of SiC_{CVD} fibers was examined at room temperature and elevated temperatures up to 1000 °C, and the change of the carbon-rich layer on the external surface was also discussed simultaneously. Finally, a micro-heater using SiC_{CVD} fiber as electrical heating elements was developed.

Experimental

SiC_{CVD} fibers used in the study

Continuous SiC_{CVD} fibers with diameters of 140 μm (SCS-2) and 70 μm (SCS-9) (produced by Textron Specialty

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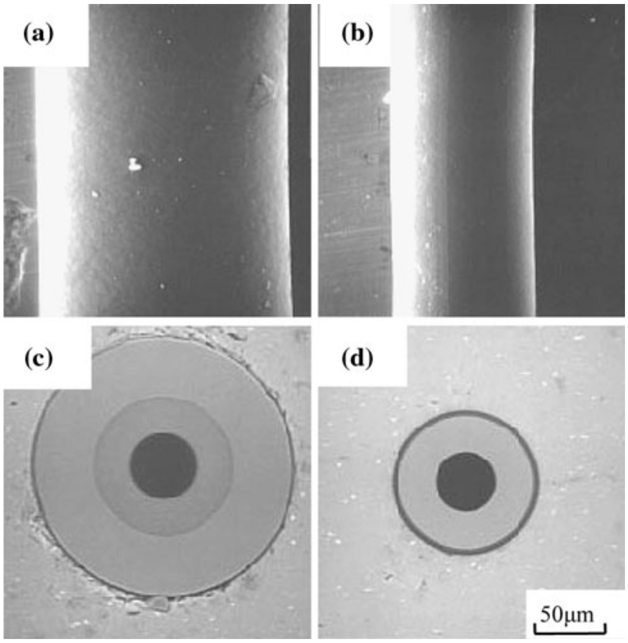

Fig. 1 SEM images of the surface and cross-section of SiC_{CVD} fibers. **a** and **c** 140 μm in diameter, **b** and **d** 70 μm in diameter

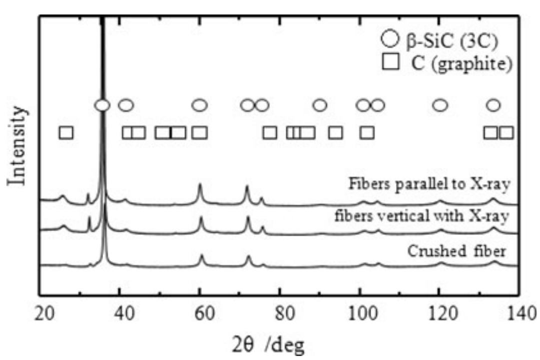

Fig. 2 X-ray diffraction patterns of SiC_{CVD} fiber with 140 μm in diameter

Materials Co. Ltd.) were used as experimental materials. SEM images of the surface and cross-section of the SiC_{CVD} fibers are given in Fig. 1. It can be seen (Fig. 1c, d) that there is a carbon core at the center. To confirm crystal structures of the SiC_{CVD} fiber, X-ray diffraction was carried out for those with a diameter of 140 μm through making the direction of the X-ray parallel to and vertical with the fiber direction as well as the crushed fiber. The results are shown in Fig. 2. From the figure, it can be concluded that SiC_{CVD} fiber is composed of two phases including *β*-SiC (3C) and carbon (graphite), and no anisotropy exists in the crystal. Table 1 lists some parameters of the electrical and thermal properties of the SiC_{CVD} fiber, carbon fiber and SiC fiber from different manufacturers, which will be compared with those of the present results. Noticeable difference can be found in the corresponding parameters between carbon fiber and SiC fiber.

Table 1 Parameters of SiC_{CVD} fiber, carbon and SiC fibers

Fiber	Density (g/cm^3)	Electrical resistivity (Ω m)	CTE ($\times 10^{-6}$/K)	Heat capacity (J/g K)
SiCCVD	3.05	–	1.5	–
Carbon	1.77	1.5×10^{-4}	−0.7	0.71
SiC	2.55	10^2–10^3	3.1	1.14

SiC_{CVD} fiber, Textron Specialty Materials Co., Ltd, *Carbon fiber* Toho Rayon CO., Ltd, *SiC fiber* Nippon Carbon Co., Ltd

Measurement of electrical resistance

Electrical resistance of SiC_{CVD} fibers was measured by the 4-terminal method [16] by using a digital multi-meter and a direct current power supply. At room temperature, the measurement was carried out by changing the length of the fibers, which were cut into 1000 mm in length. To ensure good electrical contact, the ends of the clips were wrapped up with aluminum foil and the fiber was cleansed by acetone before the measurement. Also, at elevated temperatures up to 1000 °C, electrical resistance of the SiC_{CVD} fiber with a diameter of 140 μm was examined by using the 2-terminal method as follows. The two ends of the SiC_{CVD} fiber, which was cut into 720 mm in length, passed through a small hole at the top of the annealing oven, and were partly kept outside of the annealing oven. In the annealing oven, the length of SiC_{CVD} fiber was about 400 mm and formed into a loop. After heating to the specific temperature and keeping for 5 min, measurements were performed through contacting the two ends of the SiC_{CVD} fiber outside.

Results and discussion

Electrical resistance at room temperature

Figure 3 shows the relationship between fiber length and electrical resistance for the studied fibers at room temperature. It can be seen that the electrical resistance of the fibers, despite of their different diameters, remains almost the same and shows a good linear relationship with the fiber length. Relationships between fiber length, *L* (unit: mm) and electrical resistance, *R* (unit: Ω) are given by Eqs. 1 and 2 from the linear regression analysis for the two fibers as follows:

$$R = 12.4L + 32.09 \quad \text{when } d_{\text{SiCCVD}} = 140\,\mu\text{m} \tag{1}$$

$$R = 11.8L + 175.90 \quad \text{when } d_{\text{SiCCVD}} = 70\,\mu\text{m} \tag{2}$$

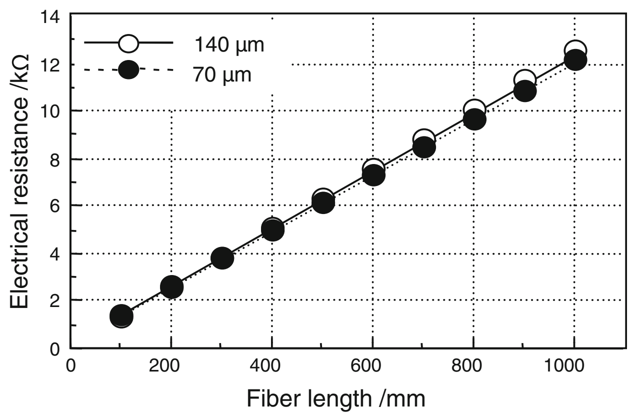

Fig. 3 Relationship between fiber length and electrical resistance of SiC_{CVD} fibers

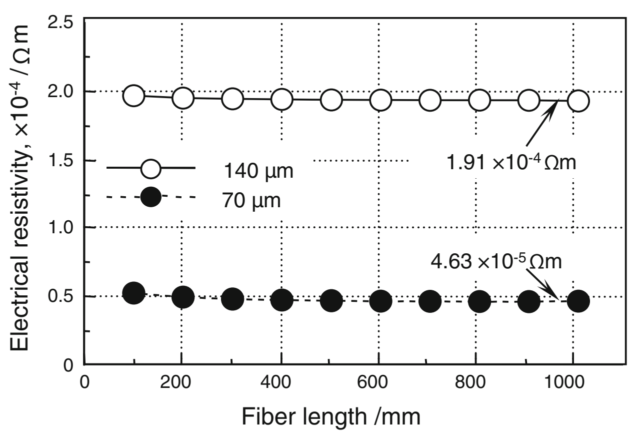

Fig. 4 Relationship between fiber length and electrical resistivity of SiC_{CVD} fibers

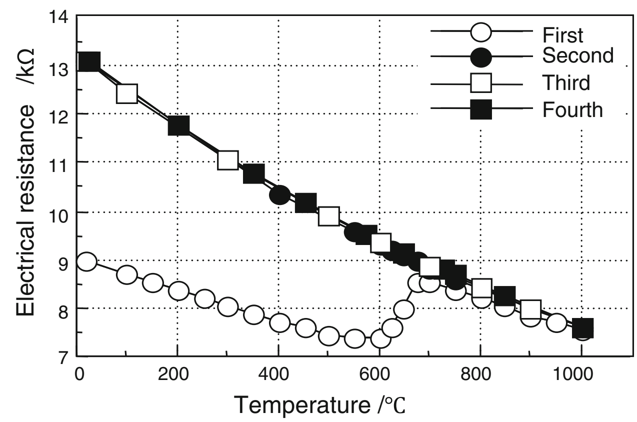

Fig. 5 Electrical resistance of the SiC_{CVD} fiber with a diameter of 140 µm at elevated temperature

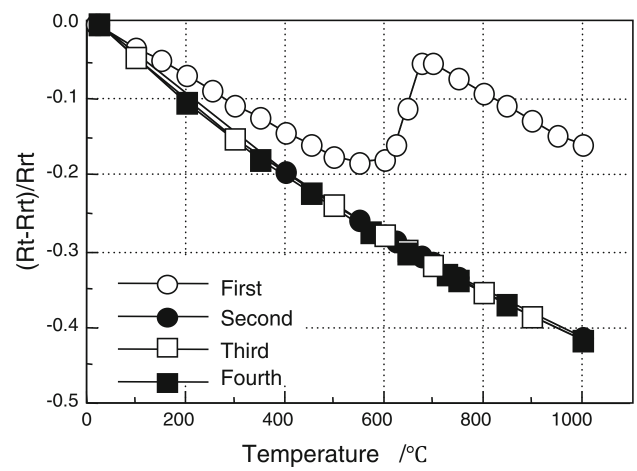

Fig. 6 Relationship between ratio of electrical resistance variation and temperature for the case of 140 µm in diameter

It can be seen from Eqs 1 and 2, even when fiber lengths reach zero, electrical resistances cannot be zero, but get 32.09 and 175.90 Ω, respectively. That is possibly related to the contact electrical resistance during the measurement.

Figure 4 shows the relationship between fiber length and electrical resistivity of the two fibers at room temperature. Electrical resistivity went down with fiber length till to about 600 mm and then reached a constant, 1.91×10^{-4} Ω m and 4.63×10^{-5} Ω m, respectively. The change of electrical resistivity is considered to originate in the contact resistance as electrical resistivity should be independent of fiber length. Therefore, it can be concluded that electrical resistivity are 1.91×10^{-4} Ω m and 4.63×10^{-5} Ω m for the two fibers, respectively. Interestingly, electrical resistivities of the two fibers are low values, and more close to that of carbon fiber than SiC fiber as shown in Table 1. Based on the results above, electrical resistance of the SiC_{CVD} fibers should depend mainly on the carbon core and the carbon-rich layer.

Electrical resistance at elevated temperatures

Figure 5 shows electrical resistance of the SiC_{CVD} fiber with a diameter of 140 µm at elevated temperatures up to 1000 °C during repeated heating for four times. In the first heating, electrical resistances went down as temperature increased but rose in the range of 550–700 °C. In the following heating, electrical resistance increased (from 9.0 to 13.1 kΩ) comparing with that of the original fiber at room temperature, but then the same evolution and decreased with the increase of temperature. To remove the electrical resistance outside the furnace and discuss the change of the electrical resistance at elevated temperatures, the ratio of electrical resistance variation was calculated with $(R_t - R_{rt})/R_{rt}$. Here, R_{rt} and R_t are the electrical resistance at room temperature and elevated temperature, respectively. The results are shown in Fig. 6. It can be seen that the ratio of electrical resistance variation increased in the range of 550–700 °C in the first heating, but then the same evolution as temperature increased in the

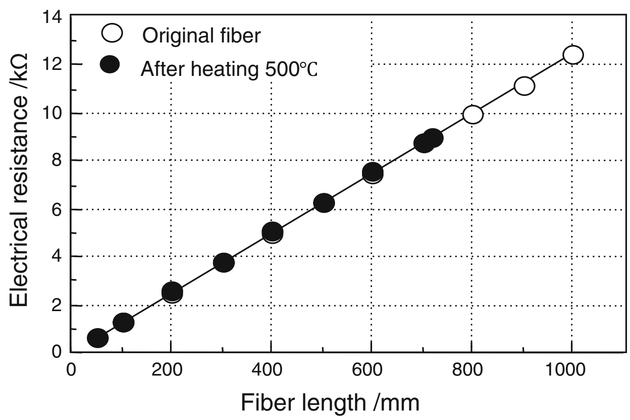

Fig. 7 Comparison of electrical resistance after heating to 500 °C with that of original SiC_{CVD} fiber with 140 μm in diameter

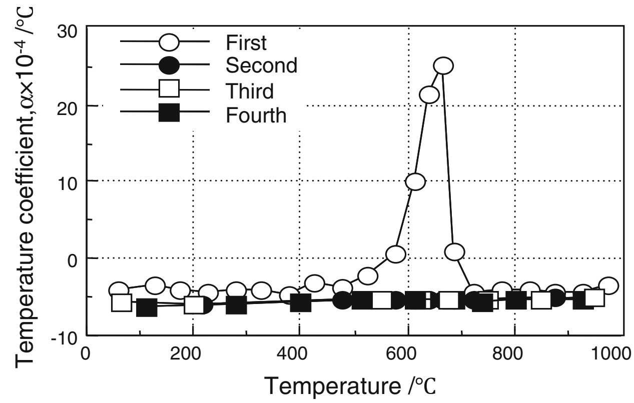

Fig. 8 Relationship between temperature coefficient and elevated temperature for the case of 140 μm in diameter

following heating and measurements. Comparing Figs. 5 and 6, it can be found that electrical resistance and ratio of resistance variation have a similar evolution pattern during the range of 550–700 °C in the first heating. Therefore, it can be understood that the change of electrical resistance at elevated temperature is only related to the part of the fiber inside the furnace.

To discuss the increase of electrical resistance at the first heating, electrical resistance was measured at room temperature after heating the fiber with a diameter of 140 μm to 500 °C, compared with the original fiber as shown in Fig. 7. It can be found that heating had no influence on electrical resistance. The results above suggest that the change of the electrical resistance in the temperature range of 550–700 °C is due to some change of the carbon-rich layer on the surface in the first heating.

To evaluate the relationship between electrical resistance and temperature, temperature coefficient of electrical resistance was calculated by Eq. 3 [17] and shown in Fig. 8.

$$\alpha = \frac{(R_i - R_j)}{\frac{(R_i + R_j)}{2}(T_i - T_j)} \tag{3}$$

The Eq. 3 can be simplified as the following equation.

$$\alpha = \frac{2(R_i - R_j)}{(R_i + R_j)(T_i - T_j)} \tag{4}$$

where, R_i and R_j are electrical resistances at temperature T_i and T_j. α is the temperature coefficient at a mean temperature $(T_i + T_j)/2$. In the first heating, temperature coefficient almost got a constant, -4.1×10^{-4} °C^{-1}, except for the peak in temperature range of 550–700 °C. Also, after the first heating, temperature coefficient became a constant, -5.2×10^{-4} °C^{-1} as far as 1000 °C. Generally, SiC_{CVD} fiber is a composite obtained by depositing SiC on the carbon fiber core and temperature coefficient of carbon exhibits a constant in range of $(-5 \sim -2) \times 10^{-4}$ °C^{-1} [15]; but for SiC ceramics, it appears minus from room temperature to 600 °C, and becomes plus over 600 °C [15]. Based on the results above of temperature coefficient of carbon and SiC ceramics, it can be safely concluded that electrical resistance of the SiC_{CVD} fibers depends mainly on the carbon core and the carbon-rich layer.

Figure 9 shows line analysis results of EPMA for the original fiber and one after heating to 1000 °C. It can be seen that there is almost no change for carbon core, but the carbon-rich layer seems to disappear after heating. Combining with all the results above, it can be concluded that the changes of electrical resistance and temperature coefficient in the range of 550–700 °C due to the burning of the carbon-rich layer.

Development of micro-heater using SiC_{CVD} fibers as heating elements

A micro-heater, using SiC_{CVD} fibers as heating elements was developed as shown in Fig. 10. Where, SiC_{CVD} fibers (ϕ140 μm × 100 mm) were arranged densely on the outside of an alumina pipe (ϕ2 × 100 mm). The two ends of SiC_{CVD} fibers were fixed by Cu foil and as the contactors. Finally, they were inserted in another alumina pipe with inner diameter of 5 mm as the enclosing part. The electrical resistance of the micro-heater was 30 Ω. Temperature of the micro-heater was measured by inserting a thermocouple into the center as shown in Fig. 10. There is a linear relationship between electrical power and heating temperature till about 700 °C as shown in Fig. 11. When temperature was over 700 °C, the lines deviated from the linear relationship owning to thermal loss of radiation. Also, temperature of the micro-heater reached 1000 °C when it was just given an electrical power of 28 W. Figure 12 shows temperature distribution from the center of the micro-heater. The higher the heating temperature is, the wider the

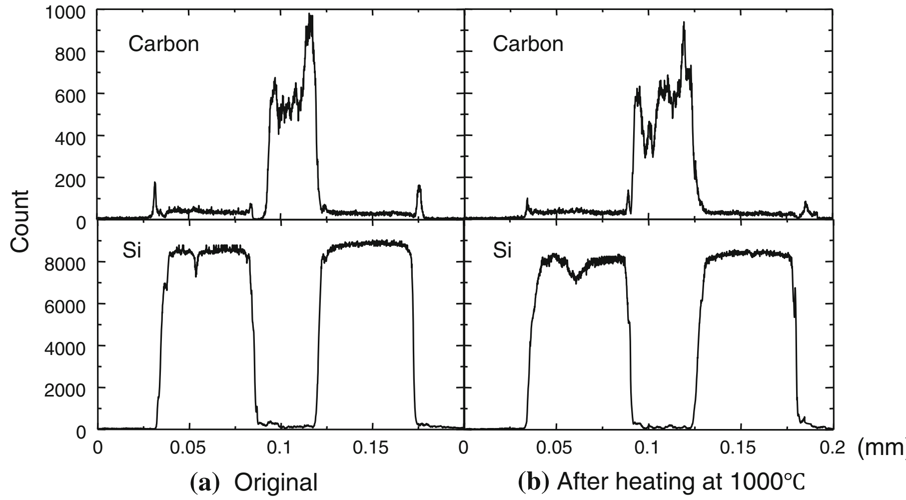

Fig. 9 Distribution of carbon and Si in the cross-section of SiC_{CVD} fiber with 140 μm in diameter by EPMA line analysis

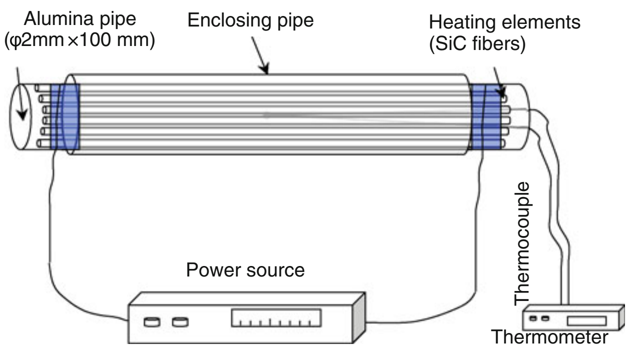

Fig. 10 Schematic diagram of the micro-heater using SiC_{CVD} fiber as heating elements

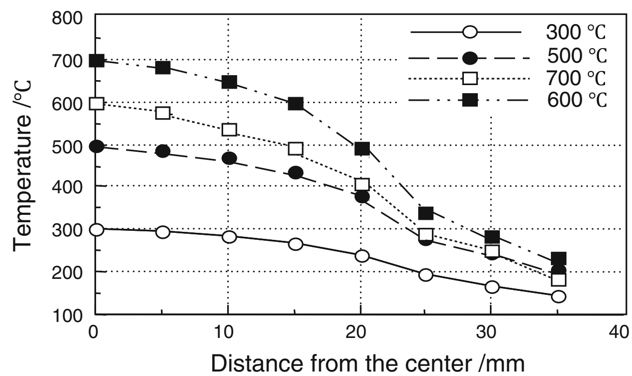

Fig. 12 Temperature distribution from the center of the micro-heater

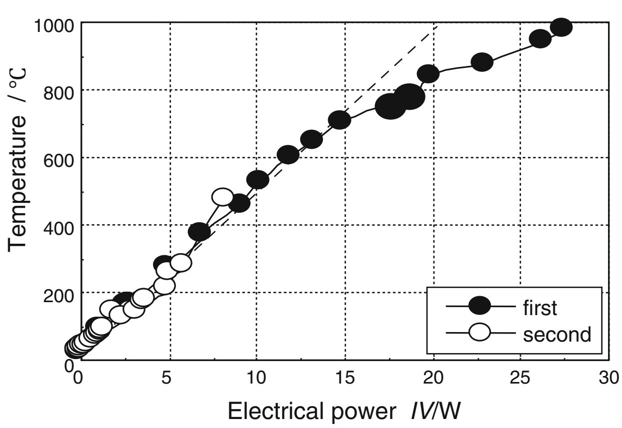

Fig. 11 Relationship between electrical power and heating temperature of the micro-heater

temperature distribution is. From the above results, the SiC_{CVD} fibers can be used good electrical heating elements. Although the contact at the contactors and the temperature distribution of the micro-heater needs to be improved, it is expected to apply the micro-heater to heat some small samples such as wire or powder.

Conclusions

1. At room temperature, electrical resistance of SiC_{CVD} fiber showed a good linear relationship as fiber length increased, besides, electrical resistivity were 1.91×10^{-4} and 4.63×10^{-5} Ω m for the fibers with diameters of 70 and 140 μm, respectively.
2. In the temperature range of 550–700 °C, electrical resistance increased owing to the burning of the carbon-rich layer on the SiC_{CVD} fiber surface in the first heating. In the following heating, electrical resistance decreased monotonously as temperature increased. Temperature coefficient of electrical resistance was a constant, -4.1×10^{-4} °C^{-1}, except for a peak at the temperature range of 550–700 °C in the first heating, but reached a constant, -5.2×10^{-4} °C^{-1} after the first heating. Behavior of electrical resistance at room temperature and elevated temperatures depended mainly on the carbon core and the carbon-rich layer.
3. It was confirmed that SiC_{CVD} fibers could be used as excellent electrical heating elements by developing the micro-heater using SiC_{CVD} fibers as heating elements.

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