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Determination of silicon carbide fiber electrical resistivity at elevated temperature

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Abstract

The resistance of Sylramic-iBN and Hi-Nicalon SiC fiber tows was measured at elevated temperature in air. Resistivity could not be directly measured, since the fibers passed through a furnace with varying temperature along the length. The resistivity of the isothermal section of the fiber tow was modeled by a series circuit of finite elements. Existing data for Hi-Nicalon resistivity vs temperature was used to verify the model and then extend it to Sylramic-iBN, for which there is no literature data readily available. The model matched experimental values with low overall error ($\pm 14\%$). Fiber resistivity decreased by more than two orders of magnitude when heating from 25°C to 1400°C. Sylramic-iBN tow resistance was also measured during a 500 hour hold at 1315°C. The resistance increased by more than 140% during heat treatment. The resistance change correlated well with the decrease in SiC fiber diameter that resulted from oxidation.

Keywords

fibers; electrical conductivity; silicon carbide

1 | INTRODUCTION

Electrical resistance (ER) monitoring of SiC fiber-reinforced ceramic matrix composites (CMC) has been explored over the past several years as a means of observing material damage. Many papers have shown that the changes in ER are highly sensitive to crack formation at room temperature.^{1,2} Data has also shown that ER is sensitive to damage formed at elevated temperatures (up to 1315°C),³ which is the ultimate interest for applying the method to turbine engine components.

Comprehensive analysis and modeling of composite ER data obtained from experiments at elevated temperatures requires knowledge of the constituent resistivity as a function of temperature. For an un-cracked composite, the matrix will likely carry the bulk of the current (especially for melt infiltrated composites with a silicon-rich matrix). The matrix resistivity (consisting of more than one phase for melt infiltrated composites) is difficult to measure directly and, therefore, must be estimated from knowing the fiber resistivity. For the

case of a cracked CMC, all electrical current must be passed through the bridging fibers in the region of a crack (assuming a through thickness crack). Modeling of in situ resistance measurements will require knowledge of the fiber resistivity as a function of temperature. If the constituent resistivity in each crack region can be well understood, then the transfer of current in the crack region can be more accurately modeled. SiC fiber resistivity data are also useful for applications such as radar absorbing materials, where electrical conductivity has an effect on electromagnetic interference shielding.⁴ In addition, SiC electrical properties are of interest for nuclear applications.⁵

Fiber resistivity is typically only reported at room temperature,⁶ likely due to complexities in elevated temperature measurement. Resistivity data has been reported by Scholz et al.⁵ for Hi-Nicalon and other Nicalon type fibers, for temperatures up to 1000°C. The data from Scholz required an inert chamber to measure the resistivity under isothermal conditions. However, isothermal measurement of higher temperature resistivity is not possible due to difficulty in lead attachment.

Therefore, the determination of electrical resistivity for ceramic fibers was pursued for two different fiber types, up to 1400°C. Tows of SiC fibers (Sylramic-iBN and Hi-Nicalon) were heated in a furnace in air while measuring the total resistance outside the furnace. Since the fibers passed through a furnace with a varying thermal profile, the measured resistance was a summation of the cold region resistance of the tow plus the hot regions. The results were modeled in order to estimate the isothermal resistivity of the hot zone of the fiber tow. The estimated relationship of resistivity vs temperature was then compared to the known literature data.

2 | MATERIAL AND EXPERIMENTS

The fibers measured in this study were Sylramic-iBN (produced by COI ceramics, San Diego, CA) and Hi-Nicalon (produced by Nippon Carbon, Japan). Fiber tows were tested instead of individual fibers because of concerns that the high resistance of long individual fibers would decrease the accuracy of the voltage measurement. The tows were manufactured with a thin polymer sizing to prevent the fibers from contacting each other. The sizing also acted as an electrical insulator, preventing electrical contact with the fibers. Therefore, a method of sizing removal was needed. For Hi-Nicalon, the sizing was removed by passing the tow through a flame for several seconds. The Sylramic-iBN tow had been heat treated in an Argon environment for 1 hour at 1315°C to remove the sizing on the fibers. Both methods adequately removed the sizing without modifying the fibers. Initially, the Sylramic-iBN and Hi-Nicalon tows consisted of approximately 800 and 500 fibers, respectively. However, removing the sizing caused the fibers to become loose and separate. The exact number of fibers remaining in the tow was unknown.

After removing the sizing, 5 mm of each end of the 180 mm long fiber tow was dipped in conductive silver paint to reduce the contact resistance. Before the silver paint dried, the tow was laid straight on a piece of paper with the ends resting on small (4.42 g) steel tabs, as shown in Figure 1. The distance between the steel tabs was 127 mm. After allowing the paint to dry, the tow ends were then covered in epoxy to secure the tow to the steel tabs.

Conductive metal tape was then placed across the tow and tab, at the inside edge of the tab. A drop of silver paint was applied to the metal tape and fiber tow to improve the electrical contact. The conductive tape served as the electrical lead for the voltage measurement.

The mounted tow was suspended from a hook, directly above a 73 mm tall furnace. The 127 mm long tow ran through the furnace with 38 mm extended below the furnace and 16 mm above. Figure 2 shows a picture of the experimental setup. The furnace was resistance heated, with MoSi₂ heating elements. Ceramic fiber board insulation was stacked on top of the furnace, up to the steel tab. Ceramic wool insulation was used to pack the front and bottom of the furnace. Prior to testing the fibers, the thermal profile of the furnace was measured by passing a thermocouple through the hot zone incrementally. The temperature of the furnace along the length is shown in Figure 3 for seven different test temperatures. When heating the fiber tow, the furnace temperature was raised to the set point at a rate of 30°C per minute and held for 20 minute at each set point to reach equilibrium prior to measuring the resistance.

The resistance of the tow was measured similar to the way previous studies have measured individual fiber resistance.^{6,7} Wires were clipped to the outer ends of the steel tabs and connected to a power supply. A resistor (1 MΩ±1%) was placed in series with the power supply. A voltmeter was used to measure the voltage across the resistor, which was then used to calculate the current in the circuit by Ohm's law. Another channel on the voltmeter was used to measure the voltage across the fiber tow (with wires clipped to the conductive metal tape). The supply voltage was swept through a range of voltages, from positive to negative. By knowing the current in the circuit, a plot of voltage vs current could be formed. The resistance of the tow was then the slope of the V-I plot, according to Ohm's law.

3 | RESULTS

The V-I plots generated from the total tow resistance for Sylramic-iBN and Hi-Nicalon are shown in Figure 4. One specimen was tested for both fiber types. The slope decreased with increasing temperature for both fiber types. All the curves were also linear over the measured range. Hi-Nicalon maintained an intercept near zero for an applied current of zero, while Sylramic-iBN had a shift in the intercept with increasing temperature. This may be due to the thermoelectric effect, but it is unclear why the two fibers exhibited different behavior. There are several differences in microstructure between the two fiber types. Most notably, Sylramic-iBN contains large grains (~100 nm) and a surface layer of BN, whereas Hi-Nicalon contains smaller grains (~5 nm) and a surface layer of carbon.⁸ Sintering aids and lower oxygen content are also present in Sylramic-iBN. The difference in microstructures may have led to a higher Seebeck coefficient for Sylramic-iBN. Table 1 lists the experimental resistances determined from the slope of the V-I plots.

The Sylramic-iBN tow that was measured in Figure 4 was also held at a constant temperature of 1315°C for 500 hour at zero applied load. The purpose of this test was to determine the time-dependent electrical resistance changes for the fibers. Figure 5 shows the resulting tow resistance, normalized by the initial resistance measured at 1315°C. The resistance reached a linear, steady-state relationship after a short initial period of higher

slope. After approximately 450 hour, the slope appeared to increase. The furnace was then turned off and the test was ended. The in situ resistance measured at 1315°C increased by more than 140% after 500 hour, compared to the initial resistance at 1315°C.

4 | DISCUSSION

4.1 | Long-term resistance and oxidation

The most likely explanation for the resistance change in the SiC fiber tow during heat treatment was that oxidation caused the SiC radius to decrease over time. SiC is known to react with oxygen in air to form SiO₂ glass and CO gas, according to Equation 1.⁹

As the reaction proceeds, the SiC fiber will undergo recession as the SiO₂ glass is formed on the surface. In this case, recession is used to describe the decrease in SiC diameter as it reacts with oxygen. In reality, Sylramic-iBN fibers have a thin initial layer of BN on the surface, which alters the oxidation reaction. The BN will react with oxygen and form liquid B₂O₃ and eventually react with SiO₂ to form borosilicate glass.¹⁰ The resulting glass layer is a high-resistivity phase and is assumed to not carry current. The SiC fiber is, therefore, assumed to carry all current. The decreasing SiC diameter is expected to result in increased resistance according to Equation 2; where ρ is resistivity, A is cross-sectional area, and L is length.

$$R = \frac{\rho L}{A} \quad (2)$$

The oxidation reaction in Equation 1 is passive unless the partial pressure of oxygen is very low, meaning that a protective oxide scale will form on the surface and slow the reaction.⁹ Equation 3 can be used to determine the oxide thickness, x , as a function of time, t . At 1315°C, the parabolic rate constant (k_p) for SiO₂ on SiC, from Jacobson et al.,¹¹ was approximately $1.29910^{-5} \mu\text{m}^2/\text{s}$.

$$x^2 = k_p t \quad (3)$$

The oxide thickness is related to the remaining SiC fiber radius (r_f) according to Equation 4; where r_{f0} is the initial fiber radius, and $V_{m(\text{SiC})}$ and $V_{m(\text{SiO}_2)}$ are the molar volume of SiC and SiO₂, respectively.

$$r_f = r_{f0} - x \left(\frac{V_{m(\text{SiC})}}{V_{m(\text{SiO}_2)}} \right) \quad (4)$$

If the fiber resistance increase over time was assumed to be only a function of changing cross-section (due to SiC recession), then according to Equation 2 a 1.8 μm recession would be needed to cause the observed resistance increase after 500 hour. This assumes a uniform recession throughout the length of the tow. In reality, the SiC recession would be greater in the hotter parts of the furnace. According to the rate constants from Jacobson et al.,¹¹ the SiC would have recessed by approximately 3.2 μm after 500 hour at 1315°C. At a lower temperature of 800°C (near the bottom of the furnace), the recession would be 0.56 μm , based on k_p of $3.97910^{-7} \mu\text{m}^2/\text{s}$.¹¹ These recession numbers provide an estimate of the upper and lower bounds of SiC recession along the tow. The 1.8 μm average recession, which would be needed to cause the observed resistance change, fell within the likely range of values. This indicates that the changing area of the SiC could have caused the resistance change over time.

4.2 | Modeling isothermal fiber resistivity

For all resistance measurements above 25°C, the temperature was not constant along the measured tow length. Therefore, the measured values in Figure 4 and Table 1 do not accurately represent the fiber resistance as a function of temperature. The resistivity of the isothermal gage section must have been lower than the average resistivity measured for the total tow since the average temperature of the total tow was lower than the furnace set point. In order to more closely estimate the isothermal resistivity, the tow resistance was modeled as a series circuit of multiple resistors. To do so, the furnace temperature profile was used to estimate the temperature along the length of the sample. Outside the furnace, the temperature of the tow was estimated to decrease linearly with distance. At the bottom of the tow, the temperature was assumed to be 25°C since the fibers were far from the heat. At the top of the tow, the temperature was assumed to be similar to that measured near the bottom of the furnace in the temperature profile (starting at 75°C and increasing by approximately 25°C for each test temperature).

Once the tow temperature along the length was estimated, the tow was broken into finite elements (3.175 mm long). Each finite element was assigned a finite temperature by calculating the average temperature of the region and rounding to the nearest finite temperature. The 16 finite temperatures used in the model are listed in Table 1. The temperatures were originally broken into 100 degree increments, but the much higher resistance of the low temperature sections made it important to sub-divide the first 100 degrees into finite regions of 25, 50, and 100°C. Other temperatures (815, 1204, and 1315°C) were chosen because they corresponded to common test temperatures for CMCs on the Fahrenheit scale (1500, 2200, and 2400°F). Figure 6 shows the measured temperature along the length at set points of 400, 815, and 1315°C, as well as the breakdown of the tow into finite temperature regions.

The ratio of ρ/ρ_0 (fiber resistivity relative to the initial resistivity at 25°C) was assumed to be exponential based on literature data for chemical vapor infiltrated (CVI) SiC composites with Sylramic-iBN fibers,³ sintered SiC,¹² biomorphic SiC,¹³ and Hi-Nicalon fibers⁵ (all of which are shown in Figure 7). Note that the experimental data from CVI SiC/SiC³ and this study were both obtained under non-isothermal conditions. The biomorphic SiC was

processed from a fiberboard precursor, resulting in a porous, fibrous microstructure. Excess silicon was removed by etching. The sintered SiC had the lowest porosity and most uniform microstructure. The sintered SiC also had the highest initial resistivity and the largest decrease with temperature. The CVI SiC/SiC composite consisted of multiple phases (fibers, BN interphase, CVI SiC, and porosity). The pores in these composites were larger than for the other samples, but the SiC phase was continuous along the length.

The procedure for estimating the isothermal fiber tow resistivity was as follows. First, the trend of ρ/ρ_0 as a function of temperature was assumed for both Sylramic-iBN and Hi-Nicalon fiber types. The trend was assumed to follow the literature data shown in Figure 7, particularly that of Scholz et al. Next, by knowing the temperature profile along the fiber tow, the resistivity of each finite element could be calculated by assuming a value for ρ_0 (room temperature resistivity). The values assumed for ρ_0 (0.23 Ω -cm for Sylramic-iBN and 3.5 Ω -cm for Hi-Nicalon) were taken from previous work as well as literature data.^{5,6,14} The total resistance of the non-isothermal tow could then be determined and compared to the experimental data in Table 1.

When the relationship of ρ/ρ_0 vs temperature from Scholz et al. was assumed for the Sylramic-iBN and Hi-Nicalon fiber tows in this study, the total (non-isothermal) tow resistance predicted from the finite element model was greater than the experimentally observed total tow resistances at all furnace peak temperatures. Figure 8A shows the difference between measured and predicted tow resistance, when assuming the same relationship as Scholz. In order to best fit the experimental data, the ρ/ρ_0 as a function of temperature relationship from Scholz in Figure 7 was decreased. The final model values used for the fibers in this study are shown in Figure 7. Figure 8B shows the difference between the best-fit and experimental tow resistance for each furnace set point, based on the final assumed isothermal resistivity relationship. The average error (from all furnace set points) is also included in Figure 8. The final relationship of ρ/ρ_0 for both fiber types was selected in order to minimize the average error between the experimental and model non-isothermal tow resistance for all temperatures.

When calculating the resistance of each element of the model, Equation 2 was used. Cross-sectional area depended on the number of fibers and the average fiber radius. Therefore, A and ρ were both unknown since the number of fibers in the tow was unknown. Based on 800 fibers per tow (5 μ m radius) for Sylramic-iBN and 500 fibers (6.75 μ m radius) for Hi-Nicalon, the initial room temperature resistivity would have been 1.09 and 3.2 Ω -cm, respectively. The previously measured room temperature resistivity of individual fibers was used to estimate the number of fibers in the tows. For Hi-Nicalon, Scholz reported an initial resistivity of approximately 4 Ω -cm and COI Ceramics reported a resistivity of 3 Ω -cm.^{5,14} Therefore, assuming a tow of approximately 500 fibers, the room temperature resistivity of Hi-Nicalon closely matched the literature data. Sylramic-iBN fibers were previously measured as 0.23 Ω -cm, with a SD of 0.21, a maximum of 0.79 Ω -cm and a minimum of 0.06 Ω -cm.⁵ The Sylramic-iBN tow measured in this study had higher resistivity (1.09 Ω -cm) than the literature value (0.23 $\pm$ 0.21 Ω -cm). This meant that either the individual fibers in the tow had higher initial resistivity than 0.23 Ω -cm, or that there were fewer than 800

fibers in the tow carrying current. Assuming that the average fiber resistivity was in fact 0.23 Ω -cm, the Sylramic-iBN tow would have consisted of approximately 170 fibers.

The model was based on the trend of ρ/ρ_0 , rather than the absolute values of resistivity. Thus, the assumed number of fibers did not affect the resulting ρ/ρ_0 vs temperature. If, however, the initial room temperature resistivity of the tow were different from the assumed value, then the absolute resistivity of the fibers (plotted in Figure 10) would have been affected.

The model indicated that the resistivity of both fiber types decreased by approximately two orders of magnitude when heating from 25 to 1400°C. Figure 9 shows each modeled tow temperature for Sylramic-iBN and the amount it was influenced by each finite temperature. 41.7% of the total resistance for the 400°C tow model was due to 25°C regions, while the 400°C region only influenced the total resistance by 4.0% for that furnace set point. The same trend can be seen for all model temperatures. Since the lower temperature sections had higher resistance, they had greater influence on the tow resistance than highest temperature sections. The implication is that the thermal profile will have an effect on measuring the gage resistivity. If the thermal profile had a steeper temperature drop outside the gage section, the sensitivity to the gage resistivity would be increased. The resulting ρ/ρ_0 along the length of the tow is plotted in Figure 6 for test temperatures of 400, 815, and 1315°C.

The absolute resistivity of the fibers, obtained from the model, is plotted in Figure 10 vs temperature. For comparison, SiC resistivity from previous work is also included. The SiC/SiC composite data were from a CMC with a CVI matrix and Sylramic-iBN fibers.³ However, that data were obtained under non-isothermal conditions (measuring across a large thermal gradient). Therefore, the resistivity of the CMC is greater than the isothermal resistivity. Sintered SiC exhibited high initial resistivity but decreased rapidly with temperature.¹² At temperatures above 800°C, the sintered SiC resistivity became comparable with the CMC data. The resistivity of biomorphic SiC closely matched the Sylramic-iBN fibers up to approximately 600°C.¹³ Beyond that temperature, the biomorphic SiC did not decrease in resistivity as much as the model estimated for the fibers. The porosity of the biomorphic SiC may have contributed to the higher resistivity at elevated temperature. There was also greater uncertainty in the predicted resistivity of the fibers at the highest temperatures, since the fiber resistance model was dominated by the lower temperature sections of the fiber. The Hi-Nicalon resistivity from this study closely matched that reported by Scholz et al.⁵ The comparison of the Hi-Nicalon data with the literature value provided added confidence that the model resistivity for Sylramic-iBN in this study was reasonable.

Figure 8B shows the error of the total tow resistance obtained from the model at each furnace set point, compared to the experiments. The total tow resistance of the model varied by a maximum of -13.3% for Sylramic-iBN (at 400°C set point) and 12% for Hi-Nicalon (at 815°C set point), compared to the measured tow resistance. The resulting average error among all furnace set points was -0.17% for Sylramic-iBN and 0.001% for Hi-Nicalon. The average temperature of the model, using the finite temperature regions, was within 3% of the measured average sample temperature from the furnace profile.

After completing the experiment, the Sylramic-iBN tow was mounted in epoxy, polished and examined to determine the number of fibers. Four hundred and thirtynine fibers were counted, which does not account for fibers that may have separated from the tow after the experiment. The observed number of fibers was less than the approximately 800 fibers originally in the tow, but greater than the 170 fibers estimated to be in the tow based on the nominal resistivity. It is likely that not all of the fibers in the tow were electrically contacted during the experiment. Also, it is possible that the individual fibers in the tow had a higher average initial resistivity than those previously measured. Regardless of the exact number of fibers in the tow, the relative change in resistivity was based on the assumption that the number of fibers remained constant throughout the test.

5 | CONCLUSIONS

The isothermal resistivity of Sylramic-iBN and Hi-Nicalon fibers was estimated from non-isothermal data by means of a series circuit finite element model. For both fiber types, fiber resistivity decreased by more than two orders of magnitude when heating from 25 to 1400°C. The total tow resistance of Sylramic-iBN (under non-isothermal conditions) exhibited >140% increase after a 500 hour hold at 1315°C. The resistance change correlated well with the SiC fiber recession that resulted from oxidation. The relationships found in this study will aid in the modeling of electrical resistance data measured for SiC fiber-reinforced composites at elevated temperatures, which may ultimately serve as a means of structural health monitoring.

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FIGURE 1.

Mounted fiber tow for measuring resistance. Note that the actual test was conducted in a vertical orientation. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 2.

Fiber furnace with mounted fiber tow and electrical resistance leads. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 3.

Fiber furnace temperature profile for set points ranging from 400 to 1400°C. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 4.

Measured V-I plots for (A) Sylramic-iBN and (B) Hi-Nicalon fiber tows at temperatures ranging from 25 to 1400°C. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 5. Resistance of a Sylramic-iBN tow during zero-stress heat treatment at 1315°C, relative to the initial resistance at 1315°C. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 6.

Model temperature and relative resistivity along the length of a Sylramic-iBN tow at furnace set points of (A) 400°C, (B) 815°C, and (C) 1315°C. Measured furnace temperatures are included for comparison. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 7.

Resistivity normalized to room temperature resistivity. Also included is literature data for biomorphic SiC, sintered SiC, SiC/SiC composites, and Hi-Nicalon fibers.^{3,5,10,12} Note the SiC/SiC composite shown had Sylramic-iBN fibers but the resistivity was measured under non-isothermal conditions. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 8.

The difference between experimental and modeled resistance of non-isothermal fiber tows (A) assuming the same isothermal relative resistivity change as Scholz⁵ and (B) using the final model trend of isothermal resistivity vs temperature, shown in Figure 7. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 9.

Influence of temperature on model tow resistance for Sylramic-iBN. For each model, the influence of each finite temperature section is listed as a percentage of the total tow resistance. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 10.

Modeled isothermal resistivity of Sylramic-iBN and Hi-Nicalon fiber tows. Also included is literature data for biomorphic SiC, sintered SiC, SiC/SiC composites and Hi-Nicalon fibers.^{3,5,10,12} Note the SiC/SiC composite shown had Sylramic-iBN fibers but the resistivity was measured under non-isothermal conditions. [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 1

Relative resistivity used in the model, along with the resulting isothermal resistivity and total tow resistance. The experimental values are included for comparison

Temp., °C	Sylramic-IBN				Hi-Nicalon			
	Experimental tow resistance, Ω	Model relative resistivity, ρ/ρ_0	Model isothermal resistivity, $\Omega\text{-cm}$	Model tow resistance, Ω	Experimental tow resistance, Ω	Model relative resistivity ρ/ρ_0	Model isothermal resistivity, $\Omega\text{-cm}$	Model tow resistance, Ω
25	22 015	1	0.230	22 015	56 885	1	3.5	56 885
50	—	0.6087	0.140	—	—	0.7429	2.6	—
100	—	0.4348	0.100	—	—	0.6000	2.1	—
200	—	0.3130	0.072	—	—	0.4000	1.4	—
300	—	0.2174	0.050	—	—	0.2857	1.0	—
400	10 653	0.1348	0.031	9234	29 627	0.2000	0.7	28 117
500	—	0.0957	0.022	—	—	0.1429	0.5	—
600	8064	0.0652	0.015	8035	24 526	0.1143	0.4	24 339
700	—	0.0478	0.011	—	—	0.0857	0.3	—
815	6038	0.0348	0.008	6762	20 247	0.0629	0.22	21 023
900	—	0.0261	0.006	—	—	0.0514	0.18	—
1000	5122	0.0196	0.0045	5040	17 393	0.0429	0.15	16 468
1100	—	0.0139	0.0032	—	—	0.0314	0.11	—
1204	4303	0.0096	0.0022	4202	14 855	0.0229	0.08	14 290
1315	4037	0.0065	0.0015	4089	13 471	0.0143	0.05	13 794
1400	3938	0.0048	0.0011	4061	12 524	0.0086	0.03	13 619