EDDY CURRENT TESTING OF CARBON-CARBON COMPOSITES

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INTRODUCTION

The objective of this project is the evaluation of eddy current methods for monitoring the electrical conductivity of carbon-carbon composite materials during high-temperature pyrolytic processing. During the processing changes in conductivity of the order of 100% have been reported [1]. Changes in conductivity due to temperature are also of this order of magnitude. The principal results of this investigation will be briefly outlined now.

To measure the electrical conductivity of carbon-carbon composites during high temperature pyrolytic processing, we have constructed apparatus for performing in situ eddy current measurements of conductivity. For verification, we have constructed a four-probe dc system suitable for room temperature measurements on carbon-carbon samples and for elevated temperature measurements on metallic sheets.

Frequency dependent impedance measurements on a 12"x10.6"x0.2" sheet of preprocessed C-Composite were carried out at room temperature by using spiral pancake coils. The theoretical model of through-transmission was used to infer the electrical conductivity of the sample. An independent measurement of the conductivity was also performed by the van der Pauw method [2], which uses four DC contact probes.

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Stainless steel sheets offered the possibility of making measurements on a substitute material which would have similar conductance (conductivity x thickness) to that of carbon-carbon. Measurements were made on foils at room temperature by the eddy current and the van der Pauw methods, concurrently.

Empty coil measurements were made at elevated temperatures. To check on the stability of the eddy current measuring system, the empty sensor was subjected to the same heating and cooling cycle as would later be used for the material measurements. The gain of the coil system changed 2.5% between room temperature and 860°C, compared with the sample resistivity change of 100%. The phase varied an insignificant amount.

A 6"x6"x0.1" sheet of carbon-carbon, coated with silicon carbide was put through four heating cycles to 350, 575, 850, and lastly 800°C, with eddy current measurements carried out during the heating and cooling cycles. The results indicate that the precision of the conductivity measurements was approximately ±2.5% over the entire temperature range, where this value represents the extreme variation of the data.

EXPERIMENTAL SYSTEM

The eddy current measuring system [3,4] is shown in Figs 1, and 2. The analyzer measures the ratio \( V_2/V_1 \) (which we call the gain) and the phase difference \( \phi_2 - \phi_1 \), (which we refer to as the phase). From the circuit diagram we see that \( V_1 \) is proportional to the primary current. We report measurements in a normalized form [5]. The real and imaginary parts of the normalized impedance, \( Z_N \), are given by the formulas

\[
\text{Re}(Z_N) = \frac{V_2}{V_1} \left[ \frac{V_1}{V_0} \right] \sin \left[ (\phi_2 - \phi_1) - (\phi_2^0 - \phi_1^0) \right], \quad (1)
\]

\[
\text{Im}(Z_N) = -\frac{V_2}{V_1} \cos \left[ (\phi_2 - \phi_1) - (\phi_2^0 - \phi_1^0) \right], \quad (2)
\]

where the \( V \)'s are voltages of the primary and secondary circuits and the superscript 0’s indicate the values for the empty coil condition. We have programmed the analyzer to calculate these quantities automatically during on-line operation.

For a given thickness of sample, the \( Z_N \) curve is a universal function of the quantity (frequency x conductivity). The unknown conductivity can be expressed as a simple proportion

\[
\sigma_1 = \sigma_0 \frac{f_0^\text{max}}{f_1^\text{max}}, \quad (3)
\]
Fig. 1 Block diagram of eddy current measuring system.

Fig. 2 Circuit of eddy current measuring system. Voltage ratios and phase differences are measured by the network analyzer.

Fig. 3 Real part of the normalized impedance of coated carbon-carbon sample at varied temperatures.

Fig. 4 Imaginary part of the normalized impedance of coated carbon-carbon sample at varied temperatures.
where the 0 sub- or superscripts indicate the values where \( \text{Re}(Z) \) is a maximum in the reference calculation and the 1 sub- or superscripts refer to the measurement results. The value of \( f'_{\text{max}} \) is reported by the analyzer and the conductivity is calculated and recorded in a personal computer.

Fig. 3 shows the real part of the normalized impedance as a function of frequency for the carbon-carbon sample described in the introduction of this report. The five curves were obtained at temperatures 22.4, 202, 400, 601, and 849°C. The only physical difference in the sample measurements is in the variation of electrical resistivity. It is seen that the shift of the peak frequency with temperature offers a method of monitoring resistivity. The variation of the imaginary part of the impedance is shown in Fig. 4. This displays the modification in the inductive coupling of the coils caused by the resistivity changes. In this work, the frequency of the maximum dissipation is used for the resistivity measurement.

**DC FOUR PROBE VAN DER PAUW METHOD**

Four small electrical contacts are attached to the perimeter of the sheet at arbitrarily located points labelled A, B, C, D. Let current \( i \) flow from A to B. Then the resistance \( R_{AB,CD} \) is defined as the ratio \( (V_B - V_C)/i \). In a similar way other such resistances are defined by permutation. It has been shown by van der Pauw that the resistivity, \( \rho \), of the sheet can be determined by the relation

$$
\rho = \frac{\pi t}{\ln(2)} \cdot \frac{(R_{AB,CD} + R_{BC,DA})}{2} \cdot f\left(\frac{R_{AB,CD}}{R_{BC,DA}}\right),
$$

where \( t \) is the thickness of the sample, and where the function \( f \) has been plotted by van der Pauw; \( f \) ranges from 0.2 to 1.0 in practical cases. When the temperature is not uniform, thermal emfs will contribute to the voltages used to evaluate the resistivity in Eq.(4). Thermal emfs were eliminated by averaging the \( R \) values under reversals of current.

Fig. 5 shows the resistivity vs temperature for S-S 304 by two methods: Eddy-current and DC four probe. There is good agreement between the experimental data, up to 700°C at which point the stainless steel sample began to oxidize, producing a magnetic oxide which influenced the eddy current readings.

**COMPARISON BETWEEN THEORY AND EDDY CURRENT MEASUREMENTS**

We have programmed the Dodd-Deeds analysis [6], assuming an infinite sheet of arbitrary thickness, approximating the flat spiral coils as a set of concentric circles.

Fig. 6 shows the comparison of theoretical and measured values of the real part of the normalized impedance of a 12"x10.6"x0.2" sheet of carbon-carbon composite at room temperature. The difference in magnitude between the theoretical curve and the experimental measurements may be associated with a small amount of leakage of magnetic field around the edges of the finite sized sample.
Fig. 5 Resistivity vs. temperature for S/S 304 by eddy current and DC methods.

Fig. 6 Comparison of experimental data and theoretical calculations for a carbon-carbon sample at room temperature.

Fig. 7 Real part of the impedance vs. frequency for carbon-carbon plate, a stainless steel sheet of comparable conductance as a simulation, and piles of stainless steel sheets.
SIMULATION WITH STAINLESS STEEL SHEETS

The theoretical analysis has shown that the eddy current measurement in the materials we are using determines the conductance, i.e., the product of conductivity and thickness. In the approximation of a thin sheet, the product

$$\sigma \cdot t \cdot f_{\text{max}} = \text{const},$$

(5)

where $\sigma$ is the conductivity ($1/\rho$), $t$ is the thickness, and $f_{\text{max}}$ is the frequency of the maximum dissipation (the peak of the real part of the impedance). This allows the use of thin sheets of higher conductivity stainless steel (shim stock) to simulate at room temperature the behavior of heated carbon-carbon. By welding contacts to the edges of a sheet, it was possible to run eddy current and DC measurements concurrently, thereby testing the measuring apparatus. The eddy current data are shown in Fig. 7. Table 1 demonstrates the approximate constancy of the product of Eq. (5), supporting the applicability of the theoretical model. (See Fig. 6.)

CARBON-CARBON SAMPLE AT ELEVATED TEMPERATURES

The first run was carried up to 350°C. The results are shown in Fig. 8, where the square markers indicate values observed during heating and the solid circles correspond to cooling. During the heating we observed two regions: the first was from room temperature to 80°C with constant resistivity, and a second region above 80°C with temperature coefficient $\alpha = -0.00077/\circ C$, where the coefficient is defined by

$$\rho = \rho_0 (1 + \alpha T),$$

(6)

with $\rho_0$ the extrapolated value at $T=0$. On cooling, a hysteresis was observed of about 7.5%, but at room temperature the resistivity returned to its original value.

The second run was carried up to 575°C. The results were similar to those of Run 1, but the temperature coefficient changed to $\alpha = -0.00071/\circ C$ and the hysteresis had reduced to 5.5%.

In the third run the maximum temperature was 850°C. On this third cycling the hysteresis disappeared. On heating, shown in Fig. 9, three regions with constant slope were observed. Up to 80°C the resistivity did not change, as before. From 80°C to 530°C a temperature coefficient of $\alpha = -0.00075/\circ C$ was observed, and above 530°C the temperature coefficient was $\alpha = -0.00043/\circ C$. The complete heating and cooling cycle is shown in Fig. 9. The hysteresis had all but disappeared. The coating had changed from a green-yellow color to a grey-black, indicating a deterioration.

<table>
<thead>
<tr>
<th>Material</th>
<th>$f_{\text{max}}$ (kHz)</th>
<th>Resistivity ($\mu\Omega \cdot \text{cm}$)</th>
<th>Thickness (in)</th>
<th>Conductance $\sigma t$ ($\Omega^{-1}$)</th>
<th>$\sigma \cdot t \cdot f_{\text{max}}$ ($\Omega^{-1} \cdot \text{kHz}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-C</td>
<td>69</td>
<td>2860</td>
<td>0.2</td>
<td>178</td>
<td>12260</td>
</tr>
<tr>
<td>C-C</td>
<td>129</td>
<td>2780(*)</td>
<td>0.1</td>
<td>91</td>
<td>11790</td>
</tr>
<tr>
<td>S/S 302</td>
<td>69</td>
<td>75</td>
<td>0.005</td>
<td>169</td>
<td>11680</td>
</tr>
<tr>
<td>S/S 302</td>
<td>61</td>
<td>75</td>
<td>3x0.002</td>
<td>203</td>
<td>12400</td>
</tr>
<tr>
<td>S/S 304</td>
<td>10</td>
<td>76</td>
<td>0.0375</td>
<td>1253</td>
<td>12530</td>
</tr>
</tbody>
</table>

(*) Measured by the eddy current method.
Fig. 8 Measured resistivity of carbon-carbon sample during heating to 350°C and subsequent cooling.

Fig. 9 Measured resistivity of carbon-carbon sample during heating to 850°C and subsequent cooling.

Fig. 10 Comparison between fitted model and eddy current conductivity of coated carbon-carbon plate.
The last run to 800°C was similar to Run 3, but the resistivity was increased by approximately 50 $\mu\Omega$cm over the whole temperature range. Again, three regions were observed with similar slopes, as indicated in Fig. 9. The hysteresis had disappeared, as before. It is to be noted, however, that on cooling, the resistivity rose to a room temperature value of 3050 $\mu\Omega$cm, approximately 200 $\mu\Omega$ cm higher than the starting value of Run 1.

CONDUCTIVITY MODEL FOR CARBON-CARBON

A conductivity model based on the superposition of two conduction contributions, follows from a simple rule of mixtures calculation [7]:

$$\sigma = \sigma_f v_f + \sigma_m v_m,$$

where $\sigma$, $\sigma_f$, and $\sigma_m$ are the composite, graphite-fiber and matrix conductivities respectively, and $v_f$ and $v_m$ are the respective fiber and matrix volume fractions. For our material $v_m = 0.4$ and $v_f = 0.6$. It is known that half of the fiber weave is oriented normal to the direction of the electrical conduction, and contributes little to the conductivity [8]. We substitute $v_f = 0.6/2 = 0.3$ in Eq. 7. The contribution of $\sigma_f$ (graphite-fibers) to the composite conductivity, $\sigma$, is approximately independent of temperature [9].

At room temperature $\sigma_f \gg \sigma_m$. Using our experimental data, Fig. 9, and selected data in Refs. [7, 8, and 10] we estimate $\sigma_f = 1154$ $\Omega$cm$^{-1}$ at r.t. On the other hand the contribution of $\sigma_m$ to the total conductivity $\sigma$, is negligible at low temperatures, while comparable to $\sigma_f$ at high temperature. The matrix conductivity, $\sigma_m$, increases with temperature and could arise from activation of carriers and/or activated mobility, as given by

$$\sigma_m = \sigma_m \exp(-\Delta/kT),$$

where $\Delta$ is the activation energy composed from a sum $\Delta = \Delta_c + \Delta_m$ with $\Delta_c$ and $\Delta_m$ the activation energies of the carrier density and carrier mobility respectively. Therefore the conductivity $\sigma$ of the composite material is given by

$$\sigma = 1154 \times 0.3 + [3380 \exp(-0.133/kT)] \times 0.4 \ (\Omega \text{cm}^{-1}),$$

where $\sigma_f = 3380$ $\Omega$cm$^{-1}$ and $\Delta = 0.133$ eV are the best fits to experimental data as shown in Fig. 10. Eq. 9 (Fig. 10) indicates that the three characteristic regions in $\rho$ vs $T$ (Fig. 9) really constitute one smooth curve of $\sigma$ vs $T$ representing conduction by thermal activation. The activation energy $\Delta$, found to be 0.133 eV is equivalent to a temperature $T_a = 1546$ K or $1273$ °C. It would be interesting to test the model by extending the eddy current measurements beyond 850°C to temperatures approaching 3000 °C.

CONCLUSION

We have demonstrated a non-contact electromagnetic measuring system for monitoring the conductivity of carbon-carbon composite and other materials at elevated temperatures. At room temperature the electromagnetic system agrees with standard four-probe DC measurements to within 1.4%, when applied to carbon-carbon plates. In thin sheets of test material, it was verified that the frequency of the peak eddy current dissipation was proportional to the product of the thickness and the
conductivity. Hysteresis on heating and cooling was greatest in the early runs to lower maximum temperatures. After heating to higher temperatures (above 800°C) the hysteresis disappeared. We believe this is most likely due to changes in the matrix; however, the coating did degenerate and may have influenced this change.

A conductivity model based on two mechanisms explain the temperature dependence of the data. A constant conductivity was used for the contribution of the fiber and an activated conductivity was invoked for the matrix. The matrix activation energy, $\Delta = 0.133 \text{eV}$, combines possible contributions from the carrier density and mobility activation.

These experiments indicate the practicality of using the eddy current method for monitoring the pyrolytic process by sensing of conductivity.

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