

In-Situ Evaluation of High Temperature Oxidation of Carbon-Carbon Composites

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I. Objectives of Research

A key element to exploiting the performance advantages of carbon-carbon composites is the use of design strategies that accommodate the oxidation characteristics of the material. The objective of this research is to develop methods to determine residual properties of carbon-carbon and other materials that degrade in the high-temperature environments in which they are utilized. For the specific material considered, the variation of the material from a nominal material symmetry is measured. Currently the tests are performed using an ultrasonic buffer rod system with extensive signal processing of the received signal. A new system for in-situ sensing is being developed that will make use of a novel time-reversal mirror sensor for the determination of properties of the carbon-carbon material in the oxidizing atmosphere.

II. Materials

The carbon-carbon samples were produced by Fiber Materials Incorporated in Biddeford Maine. The specimens used in the research were cut from larger samples that are consolidated using multiple impregnation and carbonization cycles to obtain the final matrix density. The preform is a 3-dimensional weave of a nominally orthogonal fiber orientation. To obtain the full material and damping properties of the sample, measurements are most effectively performed in the principal material axes. When an elastic wave is propagated in the sample, orientation of the sample in a principal material axis reduces the complexity of the received signal. It is also expected that the diffusion of oxygen that occurs as a part of the oxidation of the matrix will occur preferentially along a lower density material axis. Because of the manufacturing process used with this material, the material axes are not necessarily coincident with the geometric axes of the samples. The extent to which this misorientation exists in commercially produced materials is not known. The material axis variation can also result from variability in the material lay-up as well as from variation in the consolidation of the samples.

To measure the material axis misorientation, flat plates are removed from the larger specimens (See Figure 1). It is assumed that the symmetry class of the sample is unknown since the sample orientation is not known a-priori. The full 21 elastic constants are measured; it is then possible to find the planes of symmetry for the material and the reduced set of elastic constants Cowin [1987]. Results from the initial work show that the symmetry of the material can be found for at least simple misorientations [Sun et. al., to appear].

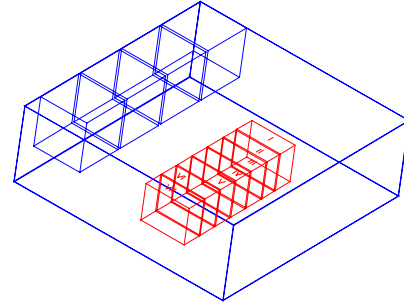


Figure 1: Sketch of composite block and locations of specimens.

III. Material Symmetry

Ultrasonic velocity measurement are often used to determine the elastic constants of a general anisotropic material [Hosten, 2001]. To determine the elastic constants, C_{ijkl} , the Christoffel equation is solved

$$(C_{ijkl}n_i n_j - \delta_{ik} V^2 \rho) P_m = 0 \quad (1)$$

where V , the phase velocity of the ultrasonic wave, is known from experiments. In general, for any given propagation direction, three wave modes are generated. The phase velocities corresponding to each of these wave modes are known as functions of the elastic properties of the material [Musgrave, 1970].

In this technique, the phase velocities of the ultrasonic wave that is obliquely incident from liquid onto the testing sample is determined. Water immersion is used in order to have good coupling between the sample and the ultrasonic transducers [Hosten, 2001]. The experiment data are collected from four incident planes (x_1, ϕ), where

azimuthal angles of $\varphi=0^{\circ}, 45^{\circ}, 90^{\circ}, 135^{\circ}$ are used [Aristegui, 1997]. The elastic constants can be reconstructed, from the velocity data, by performing a Newton-Raphson nonlinear optimization.

The identification of elastic symmetry in a general anisotropic material from the elastic constants was developed by Cowin [1987] and Norris [1988]. Two tensors A_{ij} and B_{ij} are required to determine the elastic symmetry planes of the material.

$$A_{ij} = C_{ijkk}, \quad B_{ij} = C_{ikjk} \quad (2)$$

A vector is normal to a symmetry plane of the material if and only if the tensor is an eigenvector of the A and B tensors [Cowin, 1989]. This procedure has been demonstrated for both two example materials. Unit normals, the eigenvectors, to the plane of symmetry are obtained after rotation of a known elastic tensor. The direction of symmetry that was assumed based on rotation of an initially symmetric data set is recreated after rotation out of the plane of symmetry as shown in Figure 2.

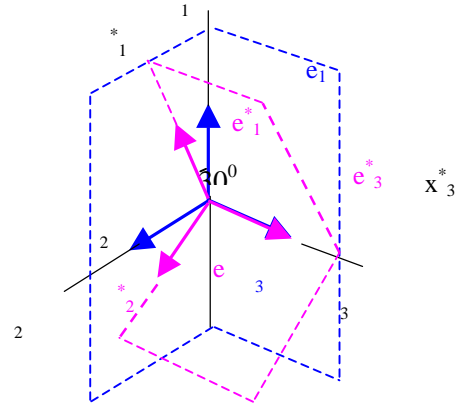


Figure 2: Illustration of the unit normals of the symmetry planes. e_1, e_2 and e_3 indicate the initial unit normals of the symmetry planes, e_1^*, e_2^* , and e_3^* indicate the unit normal calculated after rotation of 30° with respect to X_3

IV. In-Situ Measurements

After the carbon-carbon samples have been cut so that the wave propagates in a direction along a unit-normal to a plane of symmetry, measurements of the change in material properties in an oxidizing environment over time can be performed [Peterson et. al, to appear]. In the case of many applications for carbon-carbon materials, a finite life is assumed due to oxidation. The oxidation can either occur on the surface of the material, or will occur selectively in areas where the protective oxidation coating is lost. Initial high temperature testing is focussed on the measurement of the residual strength of carbon-carbon samples tested in an oxidizing environment. Figure 3 shows the general configuration of the test apparatus. The apparatus consists of a high temperature tube type furnace, an ultrasonic square wave generator, transmitting and receiving ultrasonic transducers, elastic waveguides, the required cooling system and pneumatic actuators. Waveguides are used in order to isolate ultrasonic transducers from a carbon composite that is tested in an 1100°C atmosphere.



Figure 3: Furnace used for ultrasonic monitoring of oxidation of samples

A. Signal Processing

Six signals must be recorded at each temperature increment. The six signals are used to separate the effects of the waveguide and the changes in the properties of the other elements of the apparatus. The six signals are shown in figure 4. The six signals are then deconvolved to eliminate the remainder of the setup and thus to separate the effects of a signal caused by propagation only through the carbon composite sample. Most importantly, the

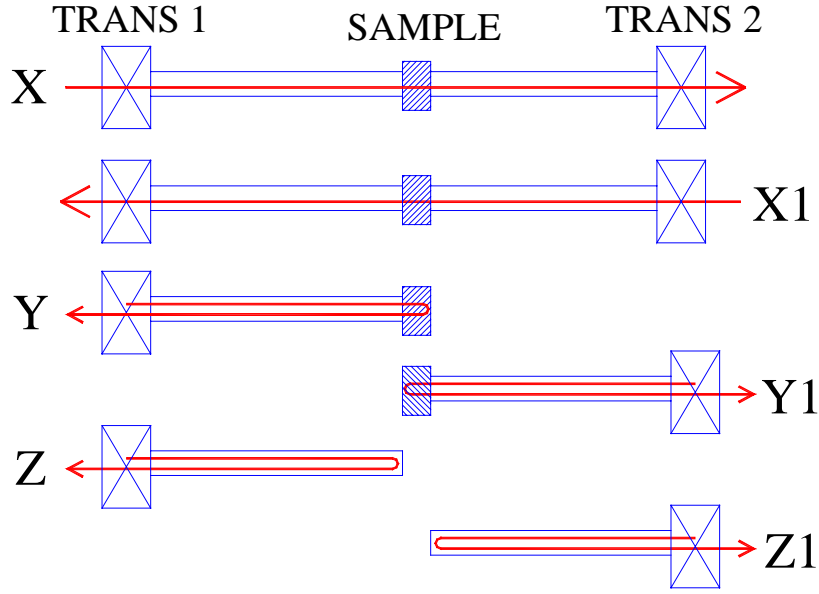


Figure 4: Six signals acquired for use in the deconvolution.

deconvolution removes the effects of coupling between transducers and waveguides and the coupling between waveguides and C/C sample. The six signals then are deconvolved as:

$$H_s(\omega) = \sqrt{\frac{\left[\frac{X(\omega) \cdot X1(\omega)}{Z(\omega) \cdot Z1(\omega)} \right]}{\left[1 - \left(\frac{Y(\omega)}{Z(\omega)} \right)^2 \right] \left[1 - \left(\frac{Y1(\omega)}{Z1(\omega)} \right)^2 \right]}} \quad (3)$$

where $H_s(\omega)$ is the response of the carbon sample, and the remaining variables are the correspond to the Fourier transforms of the signals shown in the figure.

A separate calculation is used for the ultrasonic velocity. The ultrasonic velocity is obtained from the cross-correlation that is used to compare the time delay between two different signals. The cross-correlation compares a signal with an unknown wave velocity to a signal of known wave velocity. From the phase of the deconvolution relationship, the change in velocity of a signal that propagates through the sample is:

$$t_s = \frac{(X + X1) - (Z + Z1)}{2} \quad (4)$$

Where **X**, **X1**, **Z**, and **Z1** are the signals recorded as shown in Figure 4. In addition, the sample thickness and density must known as well. This is then a simple elastic modulus of the material since the sample has already been oriented in one of the principal materials axes based on the previous section. After calculating the change in signal velocity through the sample at multiple elevated temperatures, the change in modulus of the sample can be obtained.

Multiple specimens will be placed inside an inert atmosphere in the high temperature furnace to obtain modulus change with oxidation and temperature. Experiments with a controlled amount of oxidation will also allow residual strength to be obtained from mechanical testing.

V. Time Reversal Sensor

In order to make the in-situ measurements described, the configuration shown in figure 1 is used. Ultrasonic transducers are coupled to a solid cylindrical waveguide that is used to isolate the transducer from the high temperature sample that is positioned in a controlled environment. However, the waveguide that must be used

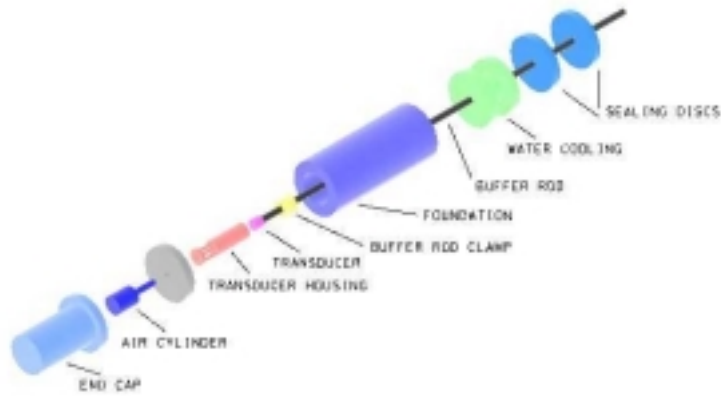


Figure 5: Waveguide sensor with cooling system to isolate transducer from sample.

for this configuration propagates multiple waveguide modes. The complexity of the signal that results from propagation of the wave is shown in the second signal from the top of Figure 6. Making use of a single element time-reversal mirror it is possible to simplify the signal reflected from the specimen and reduce the amount of signal processing required [Puckett and Peterson, in review, Puckett and Peterson, to appear]. The time-reversed input and the received signal are shown in trace 3 and 4 of Figure 6. Thus, a sample oriented in a principal material axis is used with time reversal sensor to reduce the signal complexity.

VI. Overall Objectives And Future Work

The degradation of the elastic and damping properties of the material can be measured in real time. Standard high temperature mechanical testing is also performed to measure the residual strength of the samples at temperatures up to 2500°C. The residual strength testing is performed after the sample has been oxidized to a specified degree based on monitoring sample in-situ. This will then facilitate the determination of the property degradation with temperature for use in the design of these components. Data is also expected to contribute further to the understanding of the mechanisms of degradation of carbon composites and their oxidation resistant coatings. A particularly useful application is for the development of functional grading to increase the longevity of the coatings of the samples.

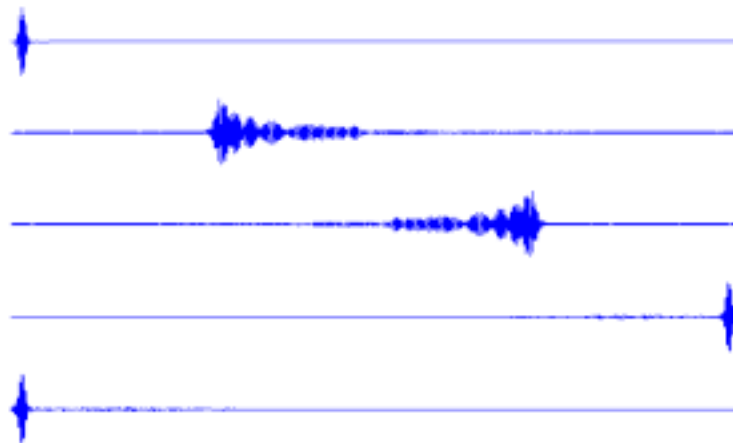


Figure 6: Time reversed signal showing the reduction in complexity.

VII. Acknowledgements

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VIII. References

A. Publications supported by BMDO/ONR

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