

NONDESTRUCTIVE EVALUATION OF THE THERMAL CYCLING  
EFFECTS ON GRAPHITE/ALUMINUM PRECURSOR WIRE

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ABSTRACT

The objective of the work was to nondestructively examine the effects of thermal cycling on properties (e.g., torsional velocity and electrical resistance) of graphite/aluminum (Gr/Al) precursor wire. Precursor wires were periodically heated at selected temperatures for various periods of time; the exact heating period being dependent on the test temperature. Wires were tested in the as-received condition and after each thermal excursion. Changes occurred in both the torsional velocity and the resistance; although at different rates and to different extents. Subsequent longitudinal velocity tests run on the heat treated sections of the wires showed little if any changes in the longitudinal velocities.

INTRODUCTION

Graphite/aluminum (Gr/Al) precursor wire has been produced for some time.<sup>1,2</sup> The wire itself has no end use but is used to make intermediates and end item products such as plates, angles, and tubes. In both the fabrication procedures and in some of the potential end item applications, the material may be exposed to elevated temperatures.

There has been very little work reported on the effects of thermal cycling on the mechanical properties of Gr/Al wire. Harrigan<sup>3</sup> reported that the room temperature longitudinal modulus is not affected by extended cycling between room temperature and 485°C and that the tensile strength decreases only slightly under the same treatment. The retention of strength after other thermal cycling treatments has also been reported by Pepper, et. al.<sup>4</sup> Although the latter group found no evidence of matrix-fiber interface failure; they, as well as numerous others, have pointed out the tremendous thermal expansion mismatch occurring between the graphite fiber and the aluminum matrix, and the possibility of this contributing to the failure of this fiber-matrix interface.

Nondestructive testing (NDT) methods of inspecting precursor wire have been under development as a part of the metal matrix composites program. Two of the possible NDT techniques being considered - ultrasonic testing using electromagnetic acoustic transducers (EMAT's) and electrical resistance testing - were used to examine the effects of heat treatment on Gr/Al wires.

## EXPERIMENTAL

The ultrasonic setup (Fig. 1) uses a high current pulser, inducing a signal to excite the transmitting transducer and produce an acoustic signal in the wire. The output from this non-contacting transducer then produces a twisting or torsional mode acoustic wave in the wire. As this wave travels down the wire it is successively picked up by two receiving transducers. The signals from these transducers are amplified and the time interval between them is determined by an automatic dual channel gated peak detector.<sup>5</sup> The output from the gated peak detector is plotted as the Y-axis on an X-Y recorder as the wire is pulled through the transducer configuration by the carriage arm of the recorder. More recent configurations permit continuous scanning of long lengths of wire. (An oscilloscope is used for a visual display of the signals.)

Some data have also been taken on a similar setup only using longitudinal transducers and hence obtaining a longitudinal velocity rather than a torsional velocity. Further details concerning the apparatus are given elsewhere.<sup>6,7,8,9</sup>

In some more recent work (with different transducer spacings), both torsional and longitudinal velocity scans were simultaneously made on Gr/Al wires made from a graphite fiber with a manufacturer's nominal longitudinal modulus value of 55 million psi (Union Carbide designation VSB 32) and another one made with a nominal longitudinal modulus value of 100 million psi (Union Carbide designation VS0054).

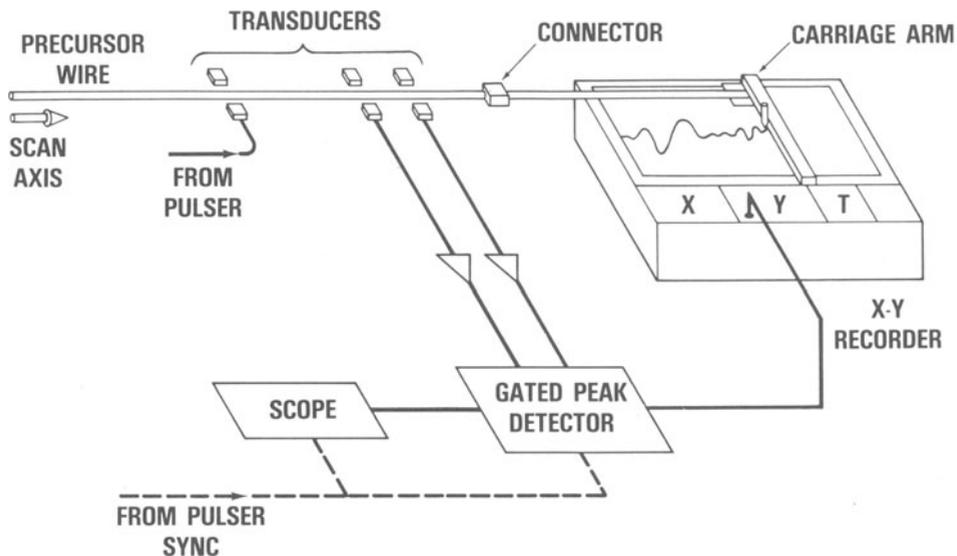


Fig. 1. Schematic of the electromagnetic acoustic transducer (EMAT) velocity apparatus.

The shear modulus values of the two fibers are not greatly different.<sup>10</sup> As shown in Fig. 2, there was little difference between the two torsional velocities but there was a significant difference between the two longitudinal velocities.

The resistance scanning apparatus (Fig. 3) consists of a constant current source which provides one ampere of current through the Gr/Al wire. A set of contacts with a spacing of one centimeter is used to measure the voltage drop (and hence resistance) as the wire was pulled by the carriage arm of the recorder. The voltage drop is then plotted on the X-Y recorder as a function of wire position.

All of the measurements were made at room temperature. For the velocity measurements, the time delay of the wire was measured, then the wire was heated in a laboratory tube furnace for a specified length of time. After removal from the furnace, the wire was cooled and the time delay was again measured. This sequence was repeated as indicated by the cumulative heating time. A typical curve of a composite of time delay plots is shown in Fig. 4. This plot is more reasonable than might first appear if one examines the temperature profiles which were measured in the furnace (Fig. 5). It was determined that a 10 cm section of the wire in the center of the furnace was within a  $\pm 10^{\circ}\text{C}$  range. Therefore, a total of 11 points (the center and at each centimeter for five cm on each side of the center) was taken and averaged for each set of measurements.

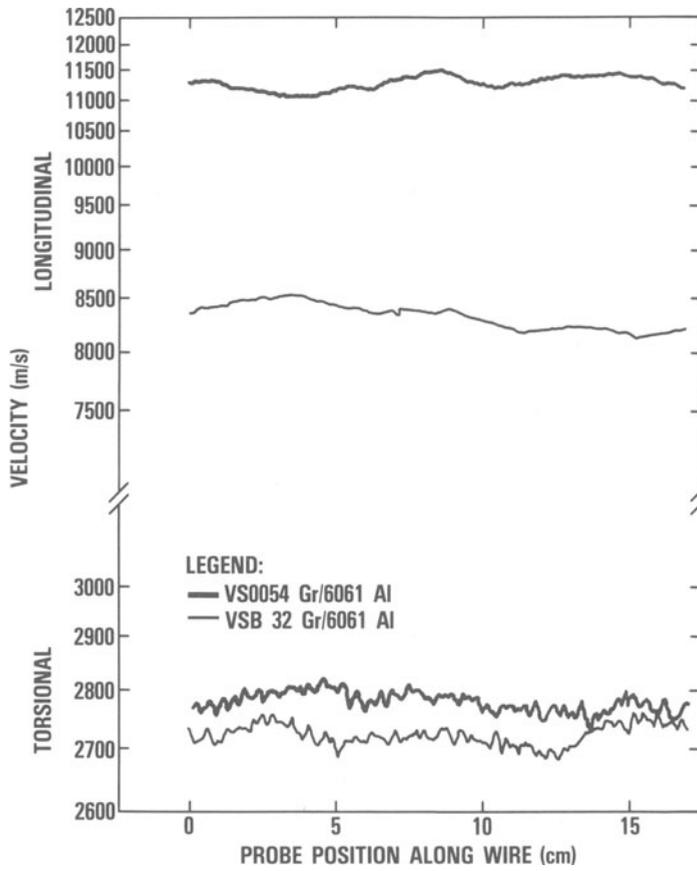


Fig. 2. Comparison of velocities of wires made with VS0054 and VSB32 graphite fibers.

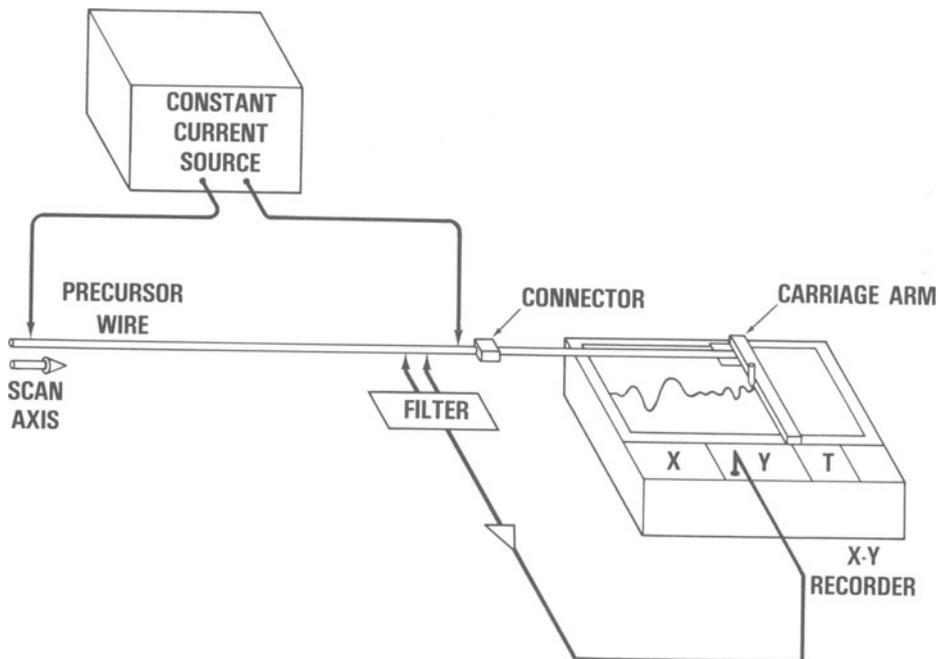


Fig. 3. Schematic of resistance scanning apparatus.

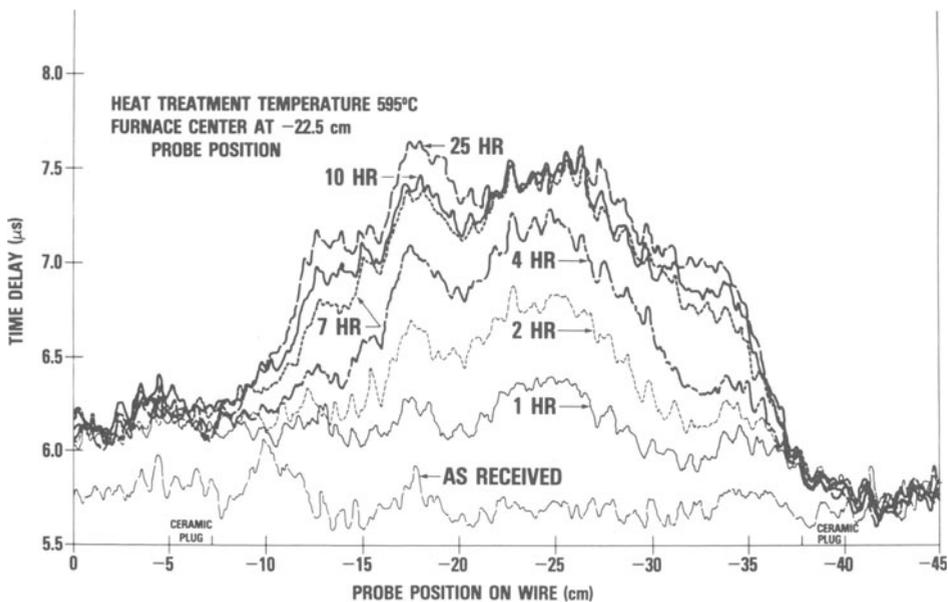


Fig. 4. Composite of typical time delay curves.

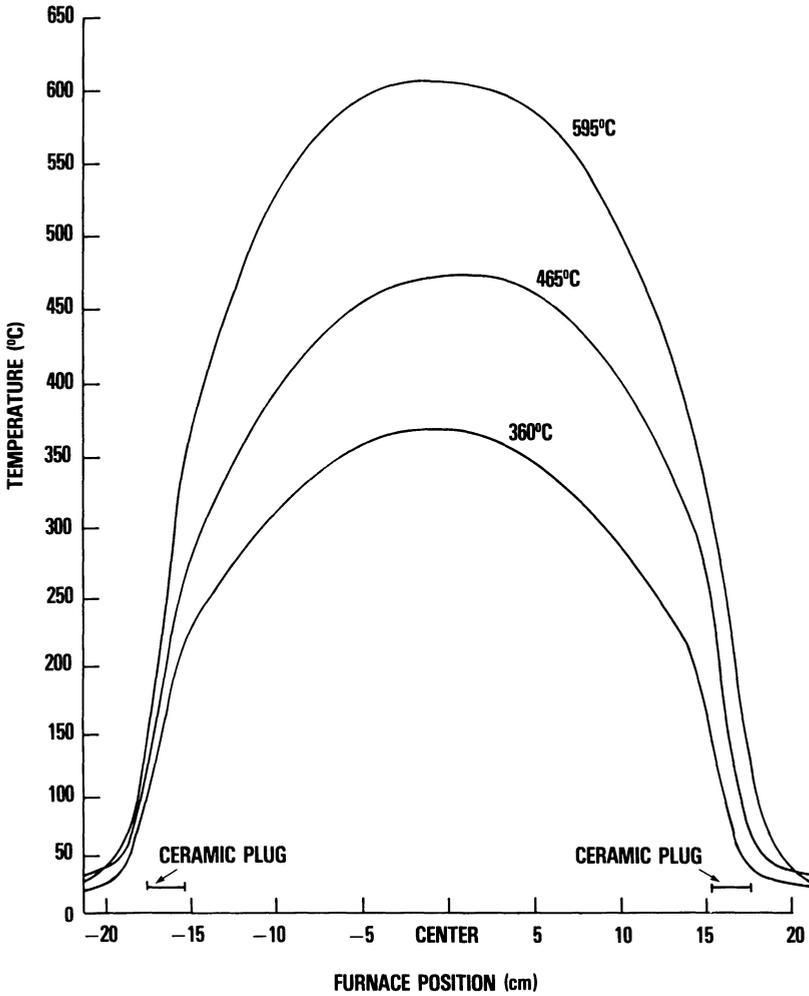


Fig. 5. Temperature profiles in furnace.

Although only a couple of test runs (taken at one week intervals) were made on wires heat treated at 360°C, scans made at this temperature indicate that decreases in the torsional velocity also occur at this temperature.

## RESULTS

All of the wires exhibited decreases in torsional velocity (and hence modulus) on heat treatment (Fig. 6). At 595°C the decreases in velocities ranged from 22.6 to 25.8 percent after a total heating time of 10 hours. Two additional 15 hour heating periods added only small additional velocity decreases. There appeared to be no significant differences between wires made from the VS0054 graphite fibers and the VSB32 graphite fibers.

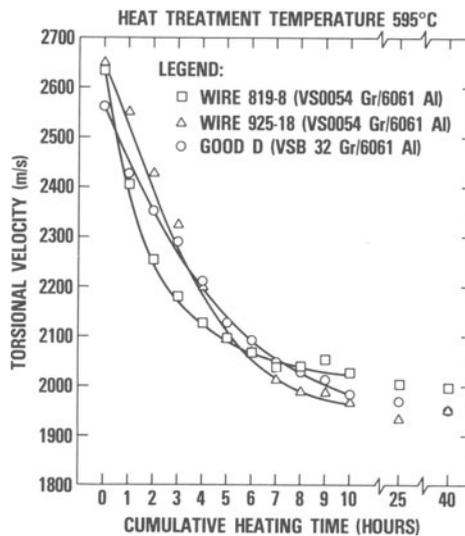


Fig. 6. Decreases of torsional velocity obtained at 595°C with various cumulative heating times.

The wires heated at 465°C showed similar decreases in velocity (Fig. 7). Both a sample heated for regular 15 hour increments and one heated for irregular increments showed similar trends. After 150 hours both samples showed somewhat lesser velocities ( $\sim 1850$  m/s) than the lowest velocities obtained from the samples heated at the higher temperature. The reason for this is not known.

Several wires were examined from a lot that was sold as production material. Two distinctly different kinds of wires were found in this lot - those with nominal and constant torsional velocities (i.e., agree with theory for the volume fraction of graphite) and those with abnormally low and varying torsional velocities. The former wires are termed "good" and the latter "bad". There was little difference in the longitudinal velocities of the two types of wires. Samples of each kind of wire from this

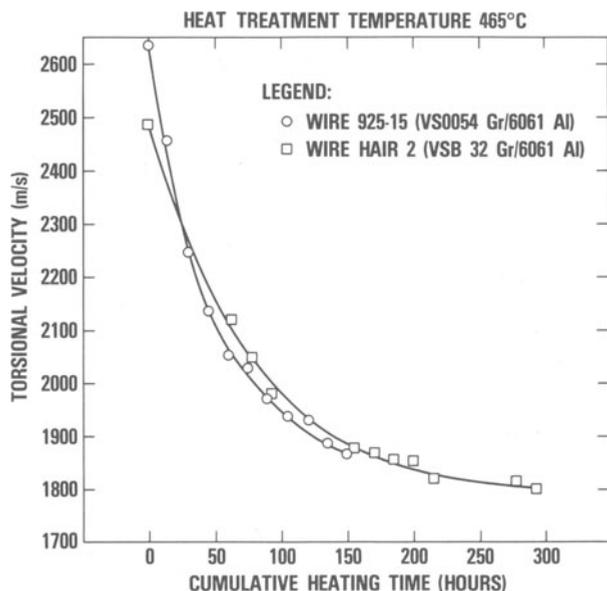


Fig. 7. Decreases of torsional velocity obtained at 465°C with various cumulative heating times.

lot were then heat treated for successive one hour periods at 595°C. The results (shown in Fig. 8) show that the torsional velocity in the wire designated as "bad" deteriorated about twice as rapidly as in the wire designated "good". These results may be quite significant since precursor wires used to make end items undergo significant heat treatment while they are hot pressed in the consolidation process.

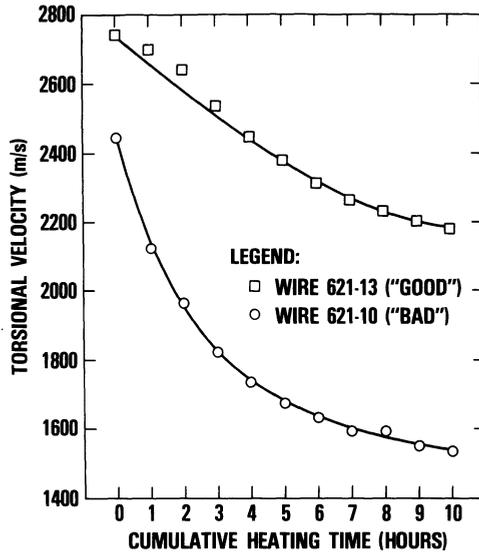


Fig. 8. Differences in torsional velocity decreases between a "good" precursor wire and a "bad" precursor wire.

The longitudinal velocity of the "good" wire showed a decrease of approximately four percent after a cumulative heating time of 10 hours at a temperature of 595°C.

Resistances in the precursor wires not only showed very sharp initial decreases but showed them in very short times. In the typical case of a VSB 32 Gr/6061 Al wire (Fig. 9), there was a large decrease shown in a manner of seconds. The drop continued, amounting to 4.6 percent in 10 minutes and continuing to about 6.5 percent in two hours.

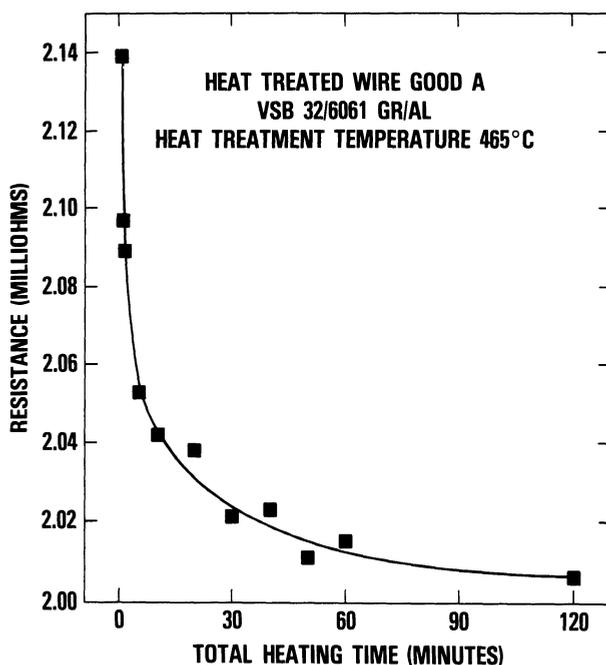


Fig. 9. Decrease of resistance with total heating time at 465°C.

Wires heat treated at 360°C and 465°C had very similar resistance curves. The resistances in the wires heated at 595°C also exhibited sharp decreases on initial heating but after a total heating time of approximately two hours, the resistance then increased on further heating. The decrease of resistance of a graphite aluminum made with alloy 5083 aluminum showed over twice the decrease of one made with alloy 6061. (Fig. 10).

Perryman<sup>11</sup> has proposed that recovery after cold working could be reasonably fitted to an equation of the type

$$\frac{1}{P-x} = \frac{1}{P_0-x} - \alpha t$$

where P is the value of the property measured at some annealing

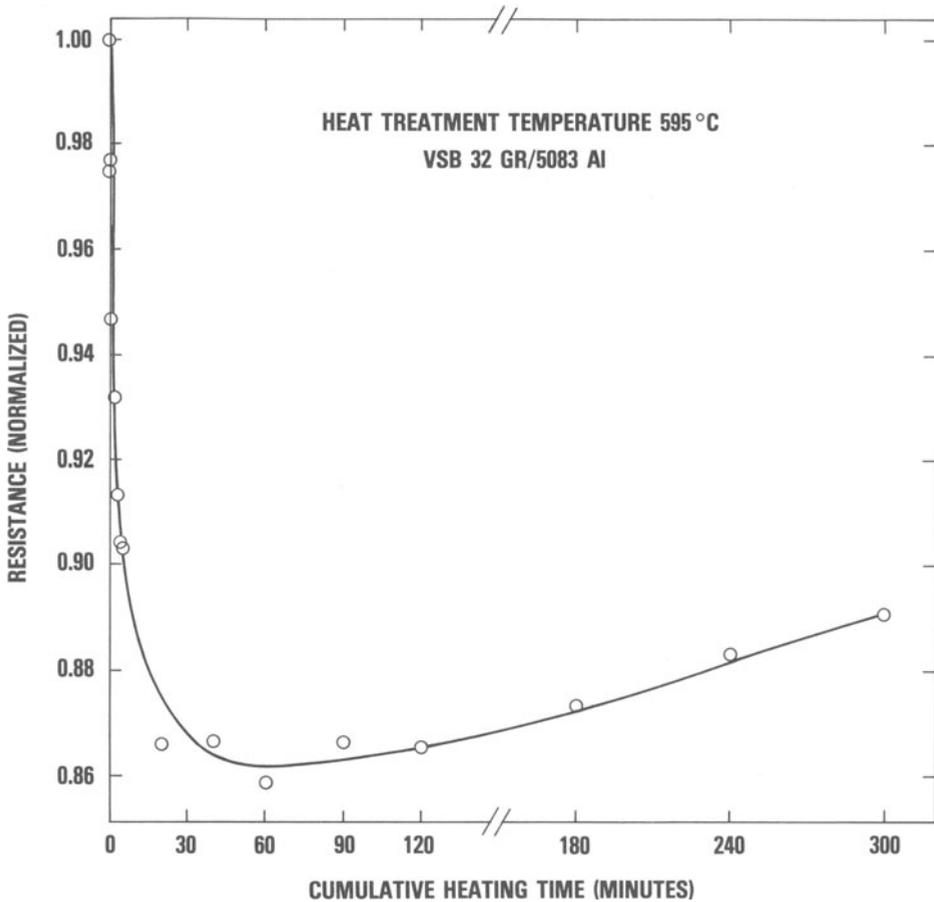


Fig. 10. Change of resistance with total heating time at 595°C.

time  $t$ ,  $P_0$  is the initial value of the property and  $x$  is the steady value of  $P$  after some long annealing time. Betteridge<sup>12</sup> had originally proposed this as a formula to describe the reduction of the number of dislocations in a cold-worked metal. Figure 11 is a fit of the equation (with  $\alpha = 1$ ) to the resistance data obtained for the wire shown in the previous figure.

It is believed that the resistance curves obtained are due to two separate combinations of events. The initial decrease is due to stress relieving and reductions of dislocations in the matrix. Further heating at the higher temperature then causes solutionizing and grain growth (which then causes higher resistance).

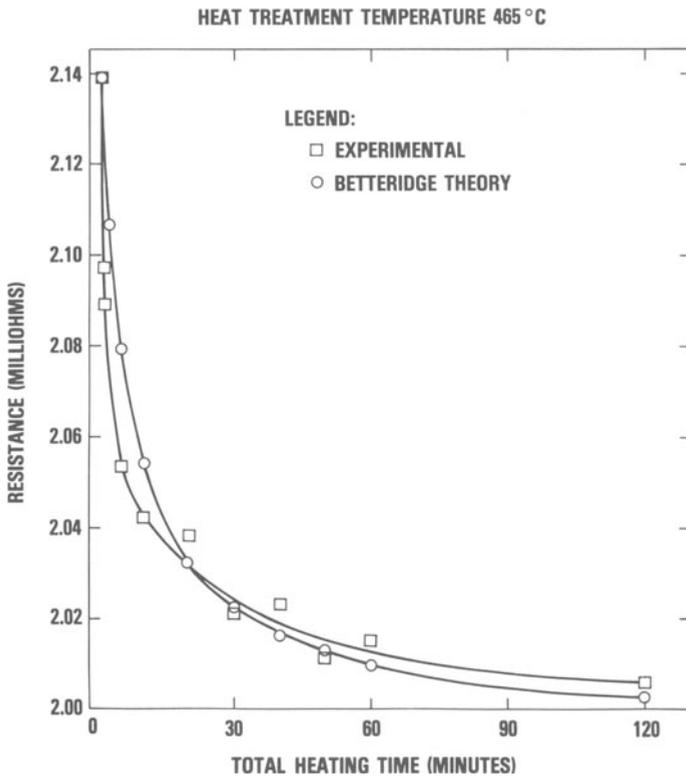


Fig. 11. Comparison of experimental change in resistance with theory.

## CONCLUSIONS

As a result of this work, it can be seen that:

- (a) decreases in the torsional modulus occur when Gr/Al precursor wires are heated at temperatures ranging from near the melting point to as low as 360°C,
- (b) slight decreases in the longitudinal modulus occur when the wires are heated at the higher temperatures, and
- (c) changes (either a decrease or a decrease followed by an increase) in the resistance occur on heat treatment of precursor wire.

It is believed that the heat treatment is affecting the fiber-matrix interface and this is being reflected in the velocity changes. This is particularly true of the torsional velocity which would be more sensitive to this interface. The changes in the resistance probably result from a combination of factors.

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