

DEPENDENCE OF THE ELECTRICAL RESISTANCE OF INTERCALATED GRAPHITE FIBERS ON ELECTRIC POWER

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Abstract—The electrical resistivity of graphite fibers intercalated with iodine monochloride was found to vary with the applied electric power, such that the electrical resistivity increased reversibly by up to 90% upon the application of electric power up to 7×10^{-2} W/cm of a single fiber. The increase was most abrupt at a power of $\sim 4 \times 10^{-2}$ W/cm.

Key Words—Carbon fibers, intercalation, electrical resistance, iodine monochloride.

1. INTRODUCTION

The low electrical resistivity of intercalated graphite fibers along the fiber axis (*a* axis) promises the application of intercalated graphite fibers as lightweight and high temperature electrical conductors.

Oshima *et al.*[1] reported that the electrical resistivity of copper chloride intercalated pitch-based carbon fibers increased irreversibly by 140% after heating at 475 K. At 450 K the resistivity was higher than the value at 300 K by 17%, but decreased back to the original value after cooling to 300 K.

The dependence of the resistivity on temperature is metallic[1]. The resistivity of copper chloride intercalated pitch-based fibers increased by 43% upon heating from 100 to 400 K[1]. The electrical resistance of first stage potassium intercalated pitch-based fibers increased by 270% upon heating from 100 to 400 K[2].

The reported variation of the electrical resistivity with temperature suggests that the electrical resistivity also varies with the electric power applied to the fiber. The dependence of the resistivity on the electric power is relevant to the application of intercalated graphite fibers as electrical conductors. It is therefore the subject of this paper.

2. EXPERIMENTAL

2.1 Sample preparation

The pristine graphite fibers used were pitch-based (Union Carbide Thornel P-100-4), with a microstructure such that the graphite planes were in the radial directions. They were a production-type, non-standard material which differed from the commercially available Thornel P-100 fibers by being cracked along the length of the filaments. The cross-sectional shape of these fibers is shown in Fig. 3 of [3]. The cross-sectional area of a fiber is 5.2×10^{-7} cm².

Intercalation with ICI was performed by immersion in liquid ICI at 100°C for a day. That this resulted in superlattice formation had been previously shown by X-ray diffraction[4]. After intercalation, the fibers were

allowed to desorb in air at room temperature for a few days. The desorbed intercalated fibers were similar to the pristine fibers in terms of the cross-sectional shape and area.

2.2 Experimental technique

Measurement of the electrical resistivity along the fiber axis was performed on single fibers by using the four-probe method, with the fibers under a vacuum of 200 μ m Hg. A DC 24 V, 12 A variable power supply was used. Electrical contacts were made by using silver conducting paint. The separation of the voltage probes was typically 3–4 min.

The temperature for the fibers to glow with an orange color and that for the fibers to burn was measured by using an optical pyrometer, which was sensitive to temperatures above about 760°C. Because a single fiber did not give out enough light to be detected by the pyrometer, the temperature measurement was made on a bundle of Thornel P-100 graphite fibers heated under a similar vacuum by the passage of an electric current. It was thus found that the average temperature for the fibers to glow with an orange color was about 850°C, whereas the temperature for the fibers to burn was about 1100°C. Furthermore the temperature variation along the length of the fibers was less than 20°C when the fibers glowed.

2.3 Experimental results

Figures 1–3 show the results obtained on the same single intercalated graphite fiber in various power cycles. Similar results were also obtained in other single fibers. In any cycle, the average power required for the fiber to glow with an orange color was about 7×10^{-2} W/cm. Hence, this power level corresponds to a temperature of about 850°C.

Figures 1 and 2 show the variation of the electrical resistance per unit length with the electric power per unit length for Power Cycles No. 2 and 3. In each power cycle, the power was increased and then decreased. In general, the resistance increased with increasing power and then decreased with subsequent power decrease. The

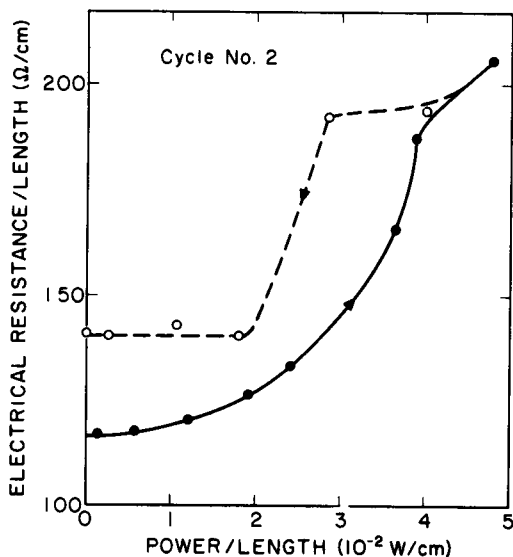


Fig. 1. Variation of the electrical resistance per unit length with the electric power per unit length during power increase (full line and solid circles) and power decrease (dashed line and open circles) in Power Cycle No. 2.

resistance change was partially reversible in Cycles No. 1 and 2, but was completely reversible in subsequent cycles. Hysteresis was observed in all cycles. The dimensions and morphology of the fibers were not affected by such power cycling, as revealed by scanning electron microscopy (SEM).

The maximum power levels in Figs. 1 and 2 were below that required to exfoliate[3] or burn a fiber. Figure 3 shows the variation of the electrical resistance with power for the case where the maximum power was just sufficient to burn the fiber. The power per unit length necessary for burning was 0.14 W/cm, which thus corresponds to a temperature of about 1100°C. The resistance continued to increase with increasing power until

burning opened the circuit and ended the measurement. SEM revealed that exfoliation had occurred in the fibers which had fractured by burning.

3. DISCUSSION

The electrical resistance increase was not totally reversible in the first two cycles. This is attributed to intercalate desorption, which was most significant in the first two cycles.

The resistance increase was not linear with power. The most abrupt increase occurred at a power of about 0.04 W/cm (Fig. 1 and 2). This behavior was observed in all cycles and suggests that the observed change in resistance is not merely due to the metallic electrical behavior of the intercalated material. We suggest that the observed resistance increase is probably due to the *initiation* of intercalate bubble formation. The actual formation of the bubbles would occur in exfoliation, but was inhibited at the power levels of Fig. 1 and 2 by the imperfect alignment of the graphite layers in the fiber. The initiation of intercalate bubble formation may cause the migration of the intercalate to defects and/or bend the graphite layers slightly, thereby increasing the electrical resistivity along the *a* axis of the fiber axis.

A similar effect of the initiation of intercalate bubble formation on the *a* axis electrical resistivity of graphite-bromine (based on artificial graphite heat-treated at 3000°C) had been previously reported[5]. It should be noted that exfoliation did not occur up to 800°C in [5] due to the relatively low degree of orientation in the graphite material, whereas exfoliation occurs below 200°C in graphite-bromine based on highly oriented pyrolytic graphite or single crystal graphite[3]. Nonetheless, the initiation of intercalate bubble formation caused the increase in the *a* axis electrical resistivity.

This paper and [5] show that even when exfoliation is inhibited by the relatively poor graphite quality, the initiation of intercalate bubble formation still occurs and

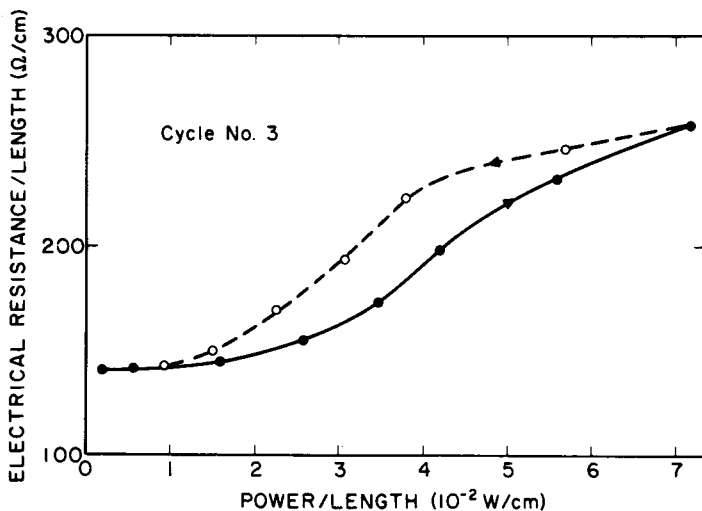


Fig. 2. Variation of the electrical resistance per unit length with the electric power per unit length during power increase (full line and solid circles) and power decrease (dashed line and open circles) in Power Cycle No. 3.

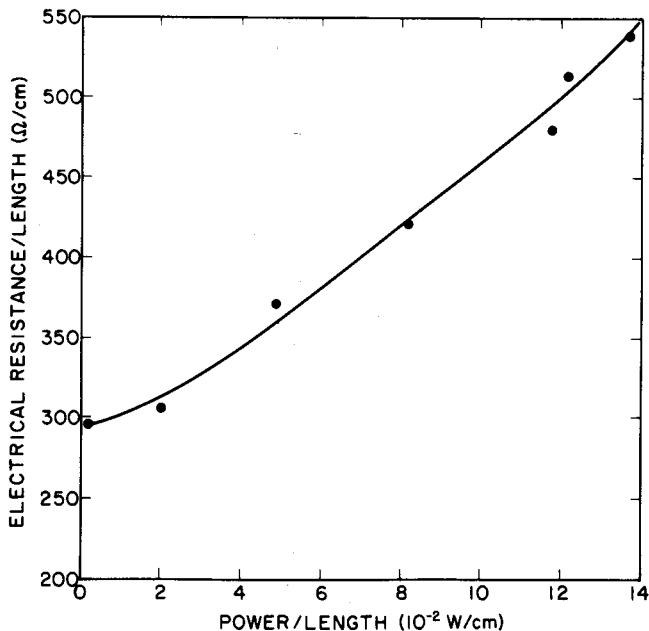


Fig. 3. Variation of the electrical resistance per unit length with the electric power per unit length for the case where the maximum power per unit length (14×10^{-2} W/cm) was just sufficient to burn the fiber.

increases the a axis electrical resistivity. The reversibility of the electrical resistivity change implies that this initiation is a reversible process, especially after the first two cycles.

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