# MAGNETIC PROPERTIES OF IRON-BEARING GRAPHITE FIBERS

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## INTRODUCTION

Carbon fibers containing ferromagnetically-ordered iron or other transition metals could be used in a variety of lightweight magnetic composites. Intercalation of bulk graphite with $CoCl_2$[1] or $FeCl_3$[2], followed by reduction with butyl lithium, did indeed produce magnetic samples; however, the observed room temperature permeabilities ($\mu$) were < 2 G/Oe. (In these units, $\mu$ of vacuum is 1.00).

In this paper we present magnetic data on carbon fibers, containing large amounts of elemental iron, which were prepared by a new method, described elsewhere at this conference [3]. We observe room temperature permeabilities as large as 40 G/Oe.

## SAMPLE PREPARATION AND CHARACTERIZATION

Amoco P-75 fibers were intercalated with $Br_2$ and $I_2$ followed by fluorination, forming $CF_{0.75}$. This product was then intercalated with $FeCl_3$. subsequent heat treatments in oxydizing and reducing atmospheres converted most of the iron to the pure $\alpha$ phase [3]. Weight analysis of samples before and after decomposition showed Fe:C atomic ratios as large as ~ 1:2.

Fibers were examined by optical and scanning electron microscopy to detect the presence of surface deposits of iron. Some samples were found to be coated with a metallic deposit and were excluded from this report. The remaining fibers appeared to have small metallic inclusions on their surfaces or in cracks. Thus we cannot eliminate the possibility that the observed permeability arises from these surface deposits. On the other hand, the amount of iron visible on the surface appeared to be much smaller than the total iron content indicated by weight analysis.

X-ray diffraction studies showed mainly peaks from the $\alpha$ (metallic) phase of iron and from graphite [3], In samples with the highest permeability, the graphite peaks were severely broadened and weakened. This effect is not yet understood.

## EXPERIMENTAL METHODS

Permeabilities were measured at room temperature in an ac susceptometer using a magnetic field of ~ 10 Oe peak at 1 kHz. Calibration of the susceptometer was done with Pt wires obtained from two different sources and was checked by direct calculation based on the coil geometry. Fibers, cut to a length of 6-8 mm, were measured individually or in bundles of up to 50 depending on their permeability. The fibers were oriented roughly parallel to the magnetic field - thus the error due to demagnetizing effects was negligible.

## RESULTS AND DISCUSSION

Permeability ($\mu$) and other data on fibers prepared by four variants of the method described in Ref. [3] are shown in Table I. The four preparation techniques involved variation of the heating rate and sample holder used for the final reduction process (which was carried

out at 1100 degrees C).

The data of Table I indicate that slow heating during reduction is essential to achievement of a high Fe:C ratio. For fibers heated slowly (SNQ and SNC), the use of a quartz, rather than carbon, sample holder caused a dramatic increase in $\mu$. Since the SNQ and SNC fibers proved to have similar Fe:C ratios, this finding suggests that other factors, in addition to the Fe:C ratio, have a major effect on magnetic properties.

Despite the substantial iron concentrations in some of our fibers, the largest values of $\mu$ observed in this experiment are several orders of magnitude smaller than those of pure $\alpha$-Fe and various permanent-magnet alloys. This suggests that the iron either does not order ferromagnetically or has an extremely "hard" ferromagnetic ordering (perhaps within small iron clusters) that is unaffected by the small (10 Oe peak) susceptometer measuring field. Magnetization and susceptibility measurements as a function of temperature and magnetic field are planned to investigate this problem.

## CONCLUSIONS

Carbon fibers containing large amounts of iron have been shown to have magnetic permeabilities of ~ 30-40 G/Oe. X-ray diffraction data suggest that much of the iron is in the $\alpha$ (metallic) phase; however, the measured permeability is much smaller than expected for $\alpha$-Fe. X-ray and resistivity data suggest considerable disorder in the graphite lattice.

## ACKNOWLEDGEMENT

Work at Cleveland State University was supported by NASA Lewis Research Center under Cooperative Agreements NCC3-19 and NCC3-339.

## REFERENCES

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2. R. Yazami, C. Meyer, and G. Chouteau, ibid, p. 93.

3. Ching-cheh Hung, Extended Abstracts and Program, 22nd Biennial Conference on Carbon, July 16-21, 1995.

TABLE I; RESULTS

| FIBER | $\mu$ (G/Oe) | HEATING RATE | SAMPLE HOLDER | Fe:C (ATOMIC) |
|---|---|---|---|---|
| SNQ | 26 - 43 | SLOW | QUARTZ | 1:3 |
| SNC | 2.8 | SLOW | CARBON | 1:2-1:3 |
| FNQ | 1.2 | FAST | QUARTZ | 1:10 |
| FNC | 1.05 | FAST | CARBON | 1:100 |