

A Gravimetric Study of Intercalation and Deintercalation of Various Quantities of Graphite Fibers

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Introduction

Bromine intercalation and deintercalation of Union Carbide P-100 graphite fibers of different lengths were studied gravimetrically. The bromine content of these fibers at room temperature was determined. Also, these fibers were compared to intercalated and deintercalated HOPG.¹

Methods and Results

Bromine Consumption of a Strand of Fibers

A pre-weighed strand of P-100 fibers (2000 fibers per strand), about 11 m long, were wound on a mandrel (diam 5.5 cm) such that the fiber spool was 1 mm thick and less than 0.75 cm wide. It was placed in saturated bromine vapor for 2 days and then taken out of the bromine environment. The brominated product was 19.3% heavier than its parent pristine fiber after 1 day of air exposure. The same weight was also observed after 14 days of air exposure. Five 30 cm long sections were cut out of the strand of fibers including both ends and three middle sections at 2.74, 5.49, and 8.23 m from the outside end of the spool. Knowing the pristine P-100 is 0.090 g/ft of fiber strand, measurement on these five sections showed an increase in mass by: (from outside end to inside end of the spool) 18.3, 18.9, 20.8, 17.8, and 20.8%.

Bromine Consumption of Long, Short, and Ground Intercalated Fibers

Seven samples of different fiber length were intercalated for 60 hr in bromine vapor. These samples were: One 168 m fiber spool (the spool was 10 cm wide, less than 1 mm thick and was wrapped on a mandrel 10 cm in diam), two 7.6 cm samples (both in test tubes), two 0.36 cm samples (in either a beaker or a test tube) and two ground samples (in either a flask or a test tube). The ground fibers were obtained by grinding a strand of fibers in a test tube with a glass rod for 15 min. Microscopic inspection of the ground fibers showed that 75% were less than 90 μ m in length. All samples were intercalated in saturated bromine vapor except the 168 m fiber spool, which absorbed all available bromine in the reactor. The mass of all samples was measured several times during the 10 days after the intercalation period. Figure 1 presents the results of two samples after 100 min of desorption and table 1 summarizes the results from all seven samples after

240 hr of desorption.

Discussion

The increases in mass measured at different locations along the 11 m long fibers were from 17.8 to 20.8%. This suggested that bromine penetrated through and reacted with the whole spool of fibers, but the distribution of bromine was not uniform. These results are significant because they show that large quantities of bromine intercalated P-100 fibers can be produced in spool form. However, further work is needed to improve the uniformity of intercalation.

Figure 1 shows the mass of the 168 m fiber sample and a ground fiber sample for the first 105 min of bromine desorption. Table 1 summarizes the mass values of all seven samples over a 10 day period. These results were examined as follows:

(1) The increase in mass due to bromine intercalation plus bromine condensation on the fiber surface at the end of the intercalation period was 50% for long fibers (168 m) and more than 61% for ground fibers. The increase in mass due to intercalation alone was believed to be the same for both samples because the observed mass increase after 10 days of desorption were the same² (18-19%). Thus the difference in mass increase observed initially between these two samples is believed to be due to different degrees of bromine condensation on fiber surface.

(2) By examining the mass data presented in fig. 1 and table 1, it can be observed that the mass of bromine remaining in the strand of fibers asymptotically approached a value in or slightly below the range of 16.7 to 19.5%. This range of values compares favorably with the range of values reported from the analysis of the 11 m strand. The average value of 18.3% corresponds to an atomic ratio of C₇₂Br₂.

(3) Bardhan et al.¹ reported that the bromine content in HOPG residue compound could stay at 43.3% at room temperature, reached 19% at 50-90 °C and dropped below 18% at 110-140 °C. In the present report, debromination of P-100 at room temperature reached completion at about 18.3%. It seemed that the bromine intercalated P-100 was to some degree similar to the intercalated HOPG, but has a much more active desorption area. Therefore high

temperature was needed to drive out bromine trapped in the HOPG used by Bardhan et al.

References

1. K.K. Bardhan, J.C. Wu, J.S. Culik, S.H. Anderson, and D.D.L. Chung, Synthetic Metals, Vol. 2, pp. 57-84, 1984.
2. G. Hennig, The Journal of Chemical Physics, Vol. 20, No. 9, pp. 1438-1442, September 1952.

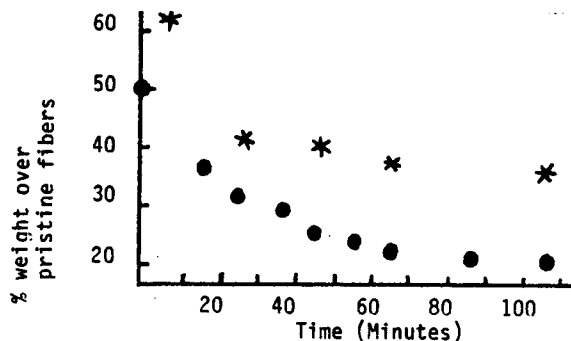


Figure 1. Weight of intercalated fibers during the first 105 minutes of bromine desorption. *ground fibers in flask •168m fiber sample.

Table 1. Weight of Intercalated Fibers in Percent Over the Parent Pristine Fibers During a 10-Day Period of Desorption.

Time, hr	Approximate lengths of fiber samples						
	168 m	7.6 cm	0.36 cm	Ground			
	Approximate weights of fiber samples, gm						
	49.5	0.28	0.28	0.23	0.16	0.61	0.61
	M	T	T	B	T	F	T
1.66	20	---	---	---	---	36.3	---
4.25	---	---	---	18.5	27.4	---	---
5.25	20	---	---	---	---	---	---
8	---	---	---	---	---	22.4	---
20	---	19.4	19.7	---	---	19.7	19.9
23	---	---	---	18.3	16.7	---	---
48	---	19.5	19.7	---	---	19.4	19.0
168	18.3	---	---	17.1	16.7	---	---
240	18.2	19.1	19.5	---	---	18.8	18.7

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