GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA, GEORGIA 30332

December 30, 1965

Dr. Daniel T. Meloon, Jr.
Senior Chemist
Research Branch
The Carborundum Company
P. O. Box 337
Niagara Falls, New York 14302

CENTRAL FILES

Re: Project A-232-292

Dear Dr. Meloon:

Electron micrographs and a report on the analysis of thin sections of BN fibers are enclosed. The microprobe and replica work is still in progress and should be completed by January 15.

If you need further information, please let me know.

Very truly yours,

John L. Brown, Head
Analytical Instrumentation Labs

JLB/jwb

ELECTRON MICROSCOPY OF MICROTOMED SECTIONS OF BORON NITRIDE FIBERS

I. Sample Preparation

Portions of each fiber sample were placed in No. 2 gelatin capsules. The capsules were filled with resin and allowed to harden. The resins, maraglas, epon, and araldite were used; best results were obtained with the latter. Sectioning was done on the Leitz ultramicrotome using a diamond knife. Sections were collected from a water-filled trough behind the knife.

II. Electron Microscopy

Micrographs and selected area diffraction (S. A. D.) patterns were made from a number of thin sections. Typical examples are included with this report.

Micrograph (GT-1)-1-B shows a typical thin section. (GT-1)-1-C shows a S. A. D. pattern from the light rectangular area in (GT-1)-1-D. Other views are similarly arranged.

For analysis of the diffraction patterns the relation $d = K/R$ may be used with $K = 40.5$ and R the ring radius in millimeters. A casual measurement fits d values for the 3 prominent lines of BN within 5%. No oxide is indicated. The change in outer ring intensity from edge to center may be due to changing crystallite orientation.

No differences were evident between samples GT-1 and GT-2.

High magnification views of each sample were made to show crystallite structure. The dark particles represent crystallites suitably oriented for diffraction contrast, i.e. their Bragg reflections do not enter the objective aperture. This was determined by tilting the specimen while under observation to change the contrast from dark to light.

Micrograph (GT-1)-1-O shows a folded-over section. The dark area at the apex of the fold indicates a section thickness of 600-700 angstroms.

Micrograph (GT-2)-1-J shows 3 adjacent fibers. There may be some indication of bonding between 2 of these.

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February 11, 1966

A232-292
Central Files

Dr. Daniel T. Meloon, Jr.
Senior Chemist
Research Branch
The Carborundum Co.
P. O. Box 337
Niagara Falls, New York

Dear Dr. Meloon:

Electron micrographs and a report on the microprobe analysis of samples GT-1 and GT-2 are enclosed.

The P. O. for this work was $500.00. Total charges were $516.75. Let me know if this is not o.k. Also let me know if you want to continue replica work on GT-2 with time charged to the new P. O. I believe a suitable technique has finally been developed.

Cordially,

John L. Brown, Head
Analytical Instrumentation Labs

COPY

Reports
300. A-232-292

ELECTRON MICROSCOPY AND MICROPROBE ANALYSIS

I. Electron Microscopy

A number of methods were attempted for replication of the fiber fracture face. The final successful method involved the construction of a polyethylene mold from 1/8 inch sheet stock. BN fibers were suspended across the interior of the mold and the cavity was filled with araldite epoxy. After curing, the epoxy block was fractured in shear. The resin fracture face was replicated by a water soluble plastic (polyvinyl alcohol) and evaporated platinum carbon films.

The enclosed micrographs show typical views of sample GT-1. Sample techniques for GT-2 were not successful. Micrograph (GT-1)-R-1-A shows two adjacent fibers fractured at an angle to the fiber axis. The fracture has a different appearance from the perpendicular fracture shown in the other views. This may indicate crystallite orientation.

To aid in replica interpretation two stereoscopic views are enclosed. Slide (GT-1)-RS-1 shows a fiber end projecting from the resin fracture face. Both fracture and surface structure of the fiber may be seen. The fuzzy surface may result from pulling free of the opposite resin face during fracture. The same fiber is shown in micrograph (GT-1)-R-1-F.

To use the enclosed stereo viewer insert the slide, look toward a bright light, and squeeze sides to focus. It may be necessary to tilt the slide a little to fuse the images. In the stereo slide the shadows produced by a hill on the sample surface are black; in the prints it is white.

II. Microprobe Analysis

Thin sections of BN fibers GT-1 and GT-2 were examined with the electron-microprobe analyzer. To get a sufficient signal to noise ratio it was necessary to cut 1 micron sections. Sections thin enough for the electron microscope were not suitable.

The sections were scanned with the smallest available beam spot (about 1.5 microns) at a speed of 12.5 microns per minute. The beam voltage was 20 KV and chart speed 1 inch per minute. Each section was scanned in forward and reverse directions. There was no noticeable difference in peak shapes.

All of the GT-1 sections were uniformly low in oxygen as shown in GT-1-1.

Samples of GT-2 seemed to vary as in GT-2-1 and GT-2-6; also the count rate for B and N was not always uniform as shown by the traces.

The block containing BN fibers from which the sections were cut was also examined in the microprobe. No differences were noted from data obtained from the section.