FILLERS FOR IMPROVED GRAPHITE FIBER RETENTION BY POLYMER MATRIX COMPOSITES

By E. E. House and C. H. Sheppard

ENGINEERING TECHNOLOGY ORGANIZATION
THE BOEING AEROSPACE COMPANY
SEATTLE, WASHINGTON 98124

PREPARED FOR:
NASA-LEWIS RESEARCH CENTER
CLEVELAND, OHIO 44135

CONTRACT NAS3-22175

D180-26685-1 NASA CR-165437

FILLERS FOR IMPROVED GRAPHITE FIBER RETENTION BY POLYMER MATRIX COMPOSITES

By E. E. House and C. H. Sheppard

ENGINEERING TECHNOLOGY ORGANIZATION
THE BOEING AEROSPACE COMPANY
SEATTLE, WASHINGTON 98124

PREPARED FOR:
NASA-LEWIS RESEARCH CENTER
CLEVELAND, OHIO 44135

CONTRACT NAS3-22175

FOREWORD

This final report describes the work accomplished in NASA Contract NAS3-22175, "Fillers for Improved Graphite Fiber Retention by Polymer Matrix Composites," from November 29, 1979 to May 15, 1981.

The program was sponsored by NASA-Lewis Research Center, Cleveland, Ohio, with Dr. T. T. Serafini as the NASA Project Manager.

Performance of this contract was under the direction of the Engineering Technology Organization of the Boeing Aerospace Company, Seattle, Washington. Mr. E. E. House was the program manager and Mr. C. H. Sheppard the technical leader. Key personnel contributing to the program and their areas of responsibility are:

E. E. House	Program Manager
C. H. Sheppard	Technical Leader
V. Monroe	Materials and Processes
H. Rathvon	Testing/Flammability and Fiber Release
O. Davis	Materials and Processes
J. Jaquish	Materials and Processes
F. Simpson	Materials and Processes
T. Basu	Materials and Processes

CONTENTS

TABLES

FIGURES

FIGURES (Continued)

1.0 SUMMARY

This report describes the results of a program to determine the extent to which the addition of elemental boron and boron-containing fillers to the matrix resin prevented the release of graphite fibers from resin matrix composites exposed to fire and impact conditions. The conditions evaluated were laboratory simulations of those that could exist in the event of an aircraft crash and burn situation. The baseline (i.e., unfilled) laminates evaluated were prepared from commercially available graphite/ epoxy. The baseline and filled laminates' mechanical properties, before and after isothermal and humidity aging, also were compared.

It was found that a small amount of graphite fiber was released from the baseline graphite/ epoxy laminates during the burn and impact conditions used in this program. However, the extent to which the fibers were released is not considered a severe enough problem to preclude the use of graphite-reinforced composites in civil aircraft structures. It also was found that the boron and boron-containing filler concepts eliminated this fiber release. Isothermal and humidity aging did not appear to alter the fiber release tendencies.

Mechanical properties of laminates containing the boron and boron-containing fillers were slightly lower than those of the baseline laminates. This could probably be attributed to using nonoptimized processing procedures.

2.0 INTRODUCTION

A potential problem has been identified (ref. 1) concerning the accidental release of graphite fibers from polymeric matrix composites during, for example, an aircraft crash/burn situation. The concern is that the electrically conductive fibers would short-circuit electrical equipment that they contact. Should a fire result from the short-circuit, the ensuing damage would affect not only the equipment contacted, but also the surrounding property. A risk analysis, directed by NASA, concluded that these risks were small (ref. 2).

Concurrent with the risk assessment, various programs were conducted to determine effective methods for containing the graphite fibers. One such program, performed by Boeing (ref. 3), showed that several hybrid concepts are effective in preventing fiber release. Also, work at NASA-Lewis (ref. 4) demonstrated that addition of elemental boron filler to the epoxy matrix also prevents the release of graphite fibers under the conditions tested. In addition to improved fiber retention on burning, it is felt that the addition or boron or boron-containing fillers could result in other benefits such as meeting FAA burnthrough requirements for nacelle structure areas.

The objective of this program was to develop technology for fabrication of graphite/epoxy composites containing boron and selected boron-containing fillers, determine the effects of the fillers on physical and mechanical properties of composites, and evaluate the effectiveness of the fillers in retaining the graphite fibers when the composites are exposed to fire conditions followed by impact. The program was conducted in three separate tasks, as shown in the Figure 1 flow diagram. The essential elements of the program were:

1. Task I—Select and characterize the epoxy resin, graphite fiber, and fillers: boron, boron carbide, and aluminum boride.
2. Task II—Prepare laminates from the candidate materials and perform screening tests consisting of mechanical properties, thermal and humidity stability, and fiber retention after 1089K (1500°F) exposure.
3. Task III—Perform additional fire, impact, mechanical properties, and thermal and humidity aging studies on three systems selected from Task II.

This document is the final report on a program performed by the Boeing Aerospace Company for the National Aeronautics and Space Administration, Lewis Research Center, to meet the aforestated objectives. The work was performed under Contract NAS3-22175, "Fillers for Improved Graphite Fiber Retention by Polymer Matrix Composites."

3.0 TECHNICAL DISCUSSION

The program was performed in three separate tasks. Task I, Selection and Characterization of Materials, was devoted to selecting and characterizing fillers, resins, and graphite fibers used in the program. Fillers included elemental boron, boron carbide, and aluminum boride. A commercially available, 450K (350°F) curing epoxy resin (Narmco 5208) and high-tensile-strength graphite fibers were used. Characterization procedures for determining the chemical composition and physical and mechanical properties were used on all materials.

In Task II, Fabrication and Screening of Composites, the materials characterized in Task I were processed into laminates and evaluated. The laminates fabricated in these studies were characterized for porosity, uniformity of dimension, and uniformity of graphite and filler particle distribution. Laminate processing parameters and quality standards were stablished for the remainder of the program. The selected fillers were dispersed into the epoxy resin, processed into laminates, and fire tested. The effectiveness of these hybridized laminates to prevent graphite fiber release or burning were compared to unhybridized laminates tested under the same conditions. Mechanical properties and thermal and humidity stability of the hybridized composites were determined and compared to unmodified laminates. Based on results from Task I and Task II, three hybridized composites concepts were selected for evaluation in Task III.

In Task III, Testing and Evaluation of Selected Composites, comprehensive fire and impact testing, physical and mechanical properties determinations, and laminate stability to thermal and humidity conditioning were performed. The three hybrid concepts selected in Task II were evaluated along with baseline, unhybridized laminates. Fire tests were performed on the laminates under different temperature-time conditions. The laminates were then subjected to impact testing. The capability of the hybridized laminates to prevent graphite fiber release was compared to unhybridized laminates tested under the same conditions. Thermal aging for 1000 hours at 450K (350°F), followed by 1000 hours of humidity aging, was performed. The effects of the thermal and humidity aging on both hybridized and unhybridized laminates were determined.

3.1 TASK I--SELECTION AND CHARACTERIZATION OF MATERIALS

This task was devoted to selecting and characterizing fillers, resins, and graphite fibers to be used in the program. Fillers included elemental boron, boron carbide, and aluminum boride. A commercially available, 450K (350°F) curing epoxy resin (Narmco 5208), and high-tensile-strength graphite fibers also were selected (T300 fabric and Celion 6000).

The criteria used to select a 450K (350°F) epoxy matrix are summarized as follows:

- Resin availability
- Baseline data availability for quality assurance
  - Chemical characterization data
  - Graphite prepreg data
  - Composite data
- Industry usage of material

The candidate resin systems were all MY 720 epoxy based with diaminodiphenylsulfone (DDS) hardener. Systems considered were Fiberite 934, Narmco 5208, Hexcel F263, and Hercules 3501-5A. The results of the evaluation indicated that the Narmco 5208 system was the system for which Boeing had the most available data, and it is currently being used extensively in flight hardware. Quality assurance data with respect to chemical characteristics also are being studied in detail on a current NASA contract. See Table 1 for available data. Boeing also has qualified this system to a current Boeing specification, BMS 8-212 (see Table 2 for typical qualification data). Hence, the Narmco 5208 system was selected as the resin matrix for the program.

The boron and boron-containing fillers were characterized using the following procedures:

- Neutron activation analysis for boron and oxygen
- Energy-dispersive X-ray and vacuum spectroscopy for trace elements
- X-ray diffraction for crystalline form
- Fisher subsieve size number
- Scanning electron microscopy (SEM) for morphology both before and after water boil
- Thermal stability
- Particle size distribution using HIAC Particle Size Counter

The particle sizes determined by the Fisher subsieve method are shown in Table 3. The fillers were boiled in water for an hour to check their stability. In all cases, except elemental boron powders of the 5-micron particle size, there were no appreciable weight changes. In the case of elemental boron (5-micron), there was a 2% weight gain. Boron carbide (100-mesh, 325-mesh) and boron powders (5-micron) were found to form white substances on the surface after exposure to boiling water. This was probably due to formation of boric acid. The presence of boric acid was confirmed by the methyl borate test. Aluminum boride showed very little activity (almost inert to boiling water).

The morphologies of all the powders, both as-received and after exposure to boiling water, were determined using the scanning electron microscope. Results of this analysis showed that in the case of boron metal (100-mesh), very small particles disappeared after exposure to boiling water. These particles are responsible for the formation of the granular deposit of boric acid. Otherwise, in all powders, SEM showed no change in morphology between the as-received material and after its exposure to boiling water.

Next, the thermal stabilities of the various fillers were determined (table 4). Note that all of the fillers were stable at 450K (350°F). However, when the two boron fillers were heated to 1256K (1800°F), the powder oxidized, with the oxidation rate for the smaller particle size (5 micron) being about twice as great as for the 100-mesh (approximately 10-micron) material. It is felt that this weight increase is the key to the mechanism by which boron retains the graphite fibers on burning. Apparently, the weight increase signifies that oxidation has occurred. The oxides produced could melt in a fire and coat the fibers, causing them to adhere to one another and thus prevent the release of free fibers.

The five boron-containing compounds also were characterized by energy-dispersive X-ray and vacuum spectroscopy to identify trace elements, by neutron activation for oxygen content, and by X-ray diffraction for crystalline structure. Results obtained from the energy-dispersive X-ray, vacuum spectroscopy, and neutron activation analyses are shown in Tables 5 and 6. The data shows excellent correlation qualitatively, but the data vary quantitatively with respect to Si, Cu, and Ca. With respect to these elements, the vacuum spectroscopy determination is probably the most reliable indicator of the quantitative amounts. The results of the X-ray diffraction

testing showed the boron carbide and aluminum boride to match ASTM standards. With respect to the amorphous boron (5-micron) and the crystalline boron (100-mesh), the same general pattern was obtained. However, the 5-micron boron gave some evidence (estimated at 50%) of being amorphous.

The desired particle size distribution for coarse powder that required grinding was accomplished in the following manner. A slurry starting with a high concentration of coarse powder suspended in diluted Narmco 5208 resin carrier was ball milled. Milling both reduced the particle size of the powder and ensured uniform mixing. Samples of the grind were extracted on a periodic basis to obtain mixtures with progressively finer particles. These samples were added to a predetermined amount of resin to produce test samples. Small quantities of graphite fabric prepreg were prepared, laminated into composites, and tested as follows to provide the basis for selection of the desired grind:

- o Particle size determination by Hegman gage
- o Resin filler handling characteristics
- o Burning in 1073K (1472°F) air
- o Exposure in boiling water

The milling operation consisted of mixing 100 grams of filler with 10 grams of resin that had been dissolved in about 60 cm^3 of methyl ethyl ketone (MEK) and ball milling for different periods of time. A Hegman gage was used to determine average particle size (table 7). The data demonstrate that it takes approximately 8 hours to reduce the fillers to a particulate size (3 to 4 microns) that can be mixed with the epoxy resin to yield an acceptable mix viscosity. It should be noted that after 8 hours, the average particle size of any of the fillers was 3 to 4 microns. The smooth surfaces of the particles and their small size should preclude cutting of the graphite filaments by the fillers.

Each filler (table 7) was blended with Narmco 5208 resin/MEK solution and applied to 178-micron (7-mil), epoxy compatible, sized graphite (T300) fabric. The level of filler on the graphite prepreg was calculated at 10% of the fiber weight (62% fiber weight and 38% resin/ filler weight). The prepreg was dried at 339K (150°F) until the volatile content was less than 1%. Then, the prepreg was cured into graphite composites. The weight loss and fiber retention characteristics of each composite were obtained by heating duplicate samples in quartz crucibles for 2 minutes in a 1089K (1500°F) muffle

furnace. This treatment removed the resin, leaving the graphite reinforcement and filler (see table 8 for data summary). Each hybrid concept so studied, except those containing coarse-grained aluminum boride (milled 1/2 or 8 hours), provided acceptable fiber retention characteristics, with no fraying at ply edges. The coarse-grained aluminum boride fillers permitted considerable fraying at ply edges. In all but the aluminum boride, weight loss was less with finer-grained fillers.

To determine the overall effects of the various particle sizes of the five fillers on mechanical properties, ±45-degree tensile coupons were machined from the cured laminates and tested at room temperature (see table 9). Upon analysis, the trend that becomes apparent is that as the particle size decreases, the tensile strength generally increases. These data, in addition to the results of the weight loss determination (table 8), were used as criteria for selecting five systems for more detailed testing in composite form in Task II of the program. These were: boron (5-micron), boron (100-mesh) milled 48 hours, boron carbide (325-mesh) milled 48 hours, boron carbide (100-mesh) milled 48 hours, and aluminum boride (325-mesh) milled 16 hours.

3.2 TASK II—FABRICATION AND SCREENING OF COMPOSITES

In Task II, the materials in Task I were processed into laminates and evaluated for porosity, uniformity of dimension, and uniformity of graphite and filler particle distribution. Laminate processing parameters and quality standards were established for the remainder of the program. The effectiveness of these hybridized laminates in preventing graphite fiber release on burning was compared to unhybridized laminates tested under the same conditions. Mechanical properties and thermal and humidity stability of the hybridized composites were determined and compared to unmodified laminates. Based on results from Tasks I and II, three hybridized composite concepts were selected for evaluation in Task III.

Based on the Task I study, the five boron-containing filler compounds selected for evaluation in Task II were: boron (5-micron), boron (100-mesh) milled 48 hours, boron carbide (325-mesh) milled 48 hours, boron carbide (100-mesh) milled 48 hours, and aluminum boride (325-mesh) milled 16 hours. After milling the fillers in a dilute solution of Narmco 5208 resin/ MEK, the resin content of the filler/resin solution was determined (see table 10). Additional resin and MEK were added to make the 20% resin solids solution necessary for preparing prepreg. The resin solids portion of

the solution contained Narmco 5208 and the selected filler at filler loadings of 10, 5, and 2.5%, based on the weight of the graphite fiber.

The graphite prepreg was made using the solvent impregnation method. The Celion 6000 graphite fibers were wound on a 305-mm (12-in.) diameter drum using a 0.076-mm (3-mil) piece of FEP film for release purposes (fig. 2). The filaments were wound with a tow count of 14 per 25 mm (in.). Resin solution was metered onto the fiber so that the end product would contain 62% by weight fiber and 38% by weight resin/filler. Some of the solvent was removed from the prepreg while on the drum to ensure that the prepreg "body" was sufficiently strong to permit handling of the material. After removing the prepreg from the drum winder, the solvent content was reduced by drying in an air-circulating oven at 339K (150°F) for 1 hour.

Since the addition of fillers changes the viscosity of the Narmco 5208 resin, rheometric testing was performed to determine if the established laminate cure cycle would have to be adjusted. In rheometrics testing the objectives could be summarized as follows:

- To identify quantitatively the effects of altering the resin (i.e. by addition of fillers) on processibility.
- To determine proper time-temperature-pressure profiles for optimum fabrication of composite materials.
- To detect any material batches that may have poor processibility.

In all samples tested, the resulting viscosity profiles amply demonstrated that the Boeing cure cycle for Narmco 5208 resin (i.e. BMS 8-212) would yield excellent quality composites. (See figs. 3 through 6 for representative rheometric curves.) Therefore, the BMS 8-212 cure cycle was used to fabricate composites from the previously prepared prepreg. The cured composites successfully passed nondestructive inspection (NDI) ("C" scan) testing (see fig. 7 for a typical "C" scan of a filled composite).

Mechanical and physical property data were obtained on the graphite/5208 epoxy composite panels containing 10, 5, and 2.5% boron-containing filler. Table 11 gives a summary of properties obtained on the as-fabricated control laminates, and Table 12 presents data obtained after isothermal aging at 450K (350°F) for 500 hours followed by 24 hours immersion in boiling water. Also summarized in Tables 11 and 12 are the physical properties of the composites (resin content, specific gravity, and fiber

volume). The resin content, obtained by acid digestion, includes the resin matrix (Narmco 5208) and an unknown amount of the boron-containing filler because the boron and aluminum boride fillers were soluble in the acid and the particle size of the boron carbide was sufficiently small to pass through the filter crucible. Consequently, the void volume of the composite could not be obtained because that calculation requires the resin content and filler content. Evaluation of the data indicates a reduction in mechanical properties when boron-containing fillers are used and that the highest filler content (10%) caused the largest reduction.

The fire testing of the composites was accomplished in the Ohio State University (OSU) release-rate apparatus (fig. 8), which had been successfully used on a previous NASA-sponsored program (ref. 3). The OSU apparatus permits a definitive examination of smoke, particle, and heat release during burning. The laminates were burned using the following conditions:

- 10 W/cm^2
- Temperature 839K (1050°F)
- The nonburn (back) side of each panel was covered with 127 -micron (5-mil) aluminum foil
- Each panel was left in the chamber for 10 minutes

One set of photographs was taken that compared the panels before and after burning (figs. 9 through 13). In addition, for two panels, pictures were taken during testing (figs. 14 and 15). Observations made during the fire test are presented in the appendix, along with observations of the panel conditions after burnout. The boron- and boron carbide-containing laminates prevented fiber release, whereas the aluminum boride allowed some fiber release. In addition, the higher the filler loading, the better the fiber retention characteristics.

Based on results of the burn tests and mechanical properties determinations (tables 11 and 12), the three systems selected for evaluation in Task III were:

1. Boron (5-micron) at 2.5% filler loading
2. Boron (5-micron) at 5.0% filler loading
3. Boron carbide (100 mesh) at 5% filler loading

3.3 TASK III—TESTING AND EVALUATION OF SELECTED COMPOSITES

In Task III, comprehensive fire and impact testing, physical and mechanical properties determinations, and laminate stability to thermal and humidity conditioning were performed. The three hybrid concepts selected in Task II were evaluated along with baseline, unhybridized laminates. Fire tests were performed on the laminates in the OSU release-rate apparatus under different temperature-time conditions and were followed by impact testing. The capability of the hybridized laminates to prevent graphite fiber release was compared to unhybridized laminates tested under the same conditions. Thermal aging for 1000 hours at 450K (350°F), followed by 1000 hours of humidity aging at 95% relative humidity and 322K (140°F), was performed. The effect of the thermal and humidity aging on both hybridized and unhybridized laminates was determined.

3.3.1 Environmental Exposure Evaluation

Celion 6000/Narmco 5208 (filled and unfilled) prepreg was prepared by drum winding as described in Task II. Sufficient prepreg was prepared to conduct burn tests in the OSU release-rate apparatus (sec. 3.3.2) and to perform the properties evaluations shown in Table 13.

The "as-fabricated" properties are presented in Table 14. The "wet strength" properties obtained after environmental aging for 1000 hours at 450K (350°F) plus 1000 hours at 95% relative humidity and 322K (140°F) are presented in Table 15.

To better display the effects of environmental exposure on the systems evaluated in Task III, the data from Tables 14 and 15 are presented graphically in Figures 16, 17, and 18. Also included in these figures are data from the Task II specimens (table 12) conditioned for 500 hours at 450K (350°F) followed by 24 hours water boil. Thus, the data presented are: flexural strength (fig. 16), flexural modulus (fig. 17), and interlaminar shear strength (fig. 18) of specimens as fabricated; after 500 hours exposure at 450K (350°F) followed by 24 hours water boil; and after 1000 hours exposure at 450K (350°F) followed by 1000 hours at 322K (140°F) and 95% RH.

These limited data indicate that:

1. Mechanical properties of the filled systems are lower than those of the control (unfilled) system for the majority of conditions evaluated.
2. It appears that the addition of boron carbide (100-mesh) at 5% filler loading is unsuitable because of reduced mechanical properties.
3. While some mechanical property degradation was incurred by addition of boron (5 microns at 2.5 and 5.0% filler loading), the degradation does not appear severe enough to preclude use of these fillers in structural composites.
4. The moisture pickup of composite specimens containing boron and boron carbide is significantly greater (table 15) than for unfilled specimens after humidity conditioning, with the boron carbide (100-mesh, 5% loading) showing the greatest weight gain; i.e., approximately three times greater than for the unfilled control specimens.

3.3.2 Fire and Impact Testing

The Ohio State University (OSU) release-rate apparatus (fig. 8) was used for the fire exposures of the filled and control (no filler) laminates. The procedures used were the same as in Task II. Two time/temperature burn conditions were used: (1) 10 W/cm^2 for 10 inutes as in Task II, and (2) 7.5 W/cm^2 for 12 minutes. The results obtained agreed with those of Task II: graphite fibers were released from the control laminates but not from the laminates containing boron or boron carbide fillers. This was the case for both fire conditions (10 and 7.5 W/cm^2) evaluated. The effectiveness of the boron and boron carbide to prevent release of graphite fiber on burning can be seen in Figures 19 through 27. A picture of a typical specimen prior to being exposed in the OSU is shown in Figure 19. The control (unfilled) specimens after fire teting are shown in Figures 20 and 21 for exposures of 10 and 7.5 W/cm^2, respectively. In Figures 20 and 21, it is apparent that (1) the integrity of the unfilled matrix has been completely destroyed by the fire exposure, and (2) the graphite fibers could be easily dispersed by mechanical agitation or air currents. However, the addition of boron or boron carbide fillers results in the graphite fibers being "trapped" by the matrix residue after fire exposure, as shown in Figures 22 through 27.

After the specimens had been exposed in the OSU release-rate apparatus, they were subjected to impact and air flow exposure in a laboratory test chamber (fig. 28) used on a previous NASA program (ref. 3). An impact load of 6.80 N·m (5 ft-lb) was

imposed on each specimen, using a 19-mm (0.75-in.) diameter indenter. During the impact, a 56-km/hr (35 mph) flow of air was maintained across the specimens. The residue of each panel was collected on adhesive-coated film placed in the bottom of the test chamber; these specimens are shown in Figures 29 through 35. The graphite fibers in the control (no filler) specimens were widely dispersed by the impact and air flow conditioning (figs. 29 and 30) compared to the boron and boron carbide specimens (figs. 31 through 35). While these laboratory tests are limited in scope, the results clearly indicate that the addition of boron or boron carbide fillers to the epoxy matrix is an effective method of preventing dispersement of fibers from laminates exposed to fire, impact, and air flow.

4.0 CONCLUSIONS AND RECOMMENDATIONS

The conclusions reached from studies conducted during this program are presented in Section 4.1. Recommendations for further work to be considered regarding use of boron and boron-containing fillers in polymer matrix composites are presented in Section 4.2.

4.1 CONCLUSIONS

1. The addition of boron and boron-containing fillers to the epoxy resin matrix effectively prevents dispersal of graphite fibers under the fire, impact, and air flow conditions evaluated.
2. Mechanical properties of graphite/epoxy laminates were reduced by the addition of boron and boron-containing fibers. These property degradations for two systems—boron (5-micron) at 2.5% filler loading and boron (5-micron) at 5.0% filler loading—do not appear severe enough to preclude their use in structural composite applications. However, more testing is needed to verify this.
3. Moisture pickup for laminates containing boron and boron carbide fillers was greater than that of graphite/epoxy specimens that did not contain fillers. However, this did not affect hot-wet properties.

4.2 RECOMMENDATIONS

Based on observations made during this program, the following areas are recommended for future study:

1. The use of boron or boron-containing fillers in the matrix of composite structure, such as engine nacelles, that have "burn-through" requirements (i.e., act as fire barriers).
2. The use of boron fillers in seats to restrict the spread of fire from one seat to another.

APPENDIX

OBSERVATION OF LAMINATES DURING AND AFTER BURN TESTS IN THE OSU RELEASE-RATE APPARATUS

DURING BURNOUT

1. #8B Boron 5-micron, 2.5%
   a. Rough (bag) side burnt
   b. Smoke and fine black particles visible
   c. No fibers released
2. #6B Boron (-100M), 2.5%
   a. Rough (bag) side burnt
   b. Smoke and fine black particles visible
3. #10B B_4C (-100M) 48 hrs, 2.5%
   a. Rough (bag) side burnt
   b. Smoke and fine black particles visible
4. #2B B_4C (-325) 38 hrs, 2.5%
   a. Rough (bag) side burnt
   b. Smoke and fine black particles visible
5. #4B AlB_{12} (-325) 16 hrs, 2.5%
   a. Rough (bag) side burnt
   b. Small amount of gas released during burnout
   c. Smoke and black particles visible

AFTER BURNOUT

1. #8B Boron 5-micron, 2.5%
   a. No fiber breakdown
   b. Looked good
   c. Fiber uniformity
2. #6B Boron (-100M), 2.5%
   a. Not good
   b. Fibers not uniform
   c. Delamination and separation visible
   d. Panel broke during removal from jig

3. #10B B_4C (-100M) 48 hrs, 2.5%
   a. Not good
   b. Fiber delamination and separation visible
   c. Panel broke into pieces
4. #2B B_4C (-325) 48 hrs, 2.5%
   a. Not good
   b. Fiber delamination and separation
   c. Panel separated after burnout
5. #4B AlB_{12} (-325) 16 hrs, 2.5%
   a. Not good
   b. Panel separation
   c. Fiber delamination and separation

DURING BURNOUT

6. C-1 B_4C 100, 5%
   a. Smooth (tool) side burnout
   b. No fibers released into air from panel
   c. Smoke and small black particles visible
7. Boron 5-micron, 5%
   a. Rough (bag) side burnt
   b. No fibers released into air
   c. Smoke and fine black particles visible
   d. Aluminum foil melted
8. AlB_{12} -325, 5%
   a. Smooth (tool) side burnt
   b. No fibers released
   c. Smoke and black particles visible
9. D-1 Boron 100, 5%
   a. Rough (bag) side burnt
   b. No fibers released
   c. Smoke and black particles visible (possible released fibers)
10. B-1 B_4C 325, 5%
   a. Smooth (tool) side burnt
   b. No fibers released from panel
   c. Smoke and fine black particles visible

AFTER BURNOUT

6. C-1 B_4C 100, 5%
   a. Fiber separation
   b. Panel very fragile
7. Boron 5-micron, 5%
   a. Panel looked good
   b. Fibers remained intact
   c. Photo taken approx. 30 sec. after panel was inserted into chamber
8. AlB_{12} -325, 5%
   a. Photo taken approx. 30 sec. of panel insertion into chamber
   b. Panel delaminated after removal from chamber
   c. Fibers separated pretty much
   d. Back side damaged more than front
9. D-1 Boron, 5%
   a. Photo taken 1-1/2 min. after panel was inserted into chamber
   b. Delamination and separation of fibers
   c. Looked good before cool down and removal from jig
10. B-1 B_4C 325, 5%
    a. Fibers tight and uniform
    b. Looked very good
    c. No separation of fibers

DURING BURNOUT

11. #3 5-micron,10%
    a. Smooth (tool) side burnt
    b. Smoke and black particles visible
12. #6 (-100M), 10%
    a. Smooth (tool) side burnt
    b. Small amount of gas released
    c. Smoke and particles released
13. #7 B_4C (-100M) 10%
    a. Smooth (tool) side burnt
    b. Smoke and black particles released

14. #10 B_4C (-325), 10%
    a. Smooth (tool) side burnt
    b. Smoke and black particles visible
15. #16 AlB_{12} (-325) 10%
    a. Smooth (tool) side burnt
    b. No fibers released
    c. Smoke and small black particles airborne

AFTER BURNOUT

11. #3 5-micron, 10%
    a. Looked good
    b. Fibers uniform
    c. Panel separated during handling process
12. #6 (-100M), 10%
    a. Looked good
    b. Fiber uniformity good
    c. No separation of fibers
13. #7 B_4C (-100M), 10%
    a. Looked bad
    b. Fibers not uniform
    c. Delamination and fiber separation visible
    d. Broke into pieces during handling
14. #10 B_4C (-325), 10%
    a. Panel looked good after burnout
    b. Fibers maintained very good uniformity
15. #16 AlB_{12} (-325), 10%
    a. Panel reduced in size during burnout
    b. Looked good
    c. Maintained fiber uniformity

REFERENCES

1. "A Report of Observed Effects on Electrical Systems of Airborne Carbon/ Graphite Fibers," NASA Technical Memorandum 78652.
2. "Assessment of Carbon Fiber Electrical Effects," NASA Conference Publication 2119.
3. "Hybridized Polymer Matrix Composites," NASA Contract NAS3-21383.
4. R. E. Gluyas and K. J. Bowles, "Improved Fiber Retention by the Use of Fillers in Graphite Fiber/ Resin Matrix Composites," presented at the 35th Annual Technical Conference, 1980, Reinforced Plastics/Composites Institute of The Society of the Plastics Industries, Inc (NASA TM 79283).

Table 1. Typical Chemical Characteristics of Narmco 5208

Chemical Test	Values	Proposed Limits
High-Pressure Liquid Chromatograph (HPLC)		
Hardener, percent	30.6	± 5.0
Advancement, percent	5.9	± 1.0
Resin, percent	42.0	± 5.0
Gel Permation Chromatography (GPC)		
Hardener, percent	31.1	± 4.0
Advancement, percent	17.7	± 3.0
Resin, percent	36.1	± 3.0
Ion Chromatography		
Hardener, percent	22.0	± 4.0
Differential Scanning Calorimetry (DSC)		
Reaction Temperature, K (°F)		
Onset	204 (-93)	—
Middle	272 (29)	—

Source: NASA Contract NAS1-15222, Development of Quality Assurance Methods for Graphite Epoxy Prepreg.

Table 2. Typical Composite Properties T300/Narmco 5208

Properties	Values
Prepreg Properties	
Resin Content, percent	38.7
Flow, percent	20.1
Volatile Content, percent	0.3
Gel Time, minutes	30.2
Composite Properties	
Tensile, 0-degree, Ultimate, MPa (ksi)	
331K (-65°F)	1442.2 (209.2)
RT	1612.5 (233.9)
406K (270°F)	1657.3 (240.4)
Compression, 0-degree, Ultimate, MPa (ksi)	
331K (-65°F)	1429.1 (207.3)
RT	1520.8 (220.6)
406K (270°F)	1105.8 (160.4)
Short Beam Shear, MPa (ksi)	
331K (-65°F)	113.8 (16.5)
RT	97.2 (14.1)
406K (270°F)	59.3 (8.6)

Table 3. Fisher Subsieve Size Number of Boron and Boron-Containing Fillers

	Sample	Procured as	Measured Average Particle Diameter (microns)
1.	Boron metal B	5 microns	1.0
2.	Boron metal B	100 mesh	10.5
3.	Boron carbide	325 mesh	9.5
4.	Boron carbide	100 mesh	23.0
5.	Aluminum boride	325 mesh	3.5

Table 4. Thermal Stability of Fillers at 450K (350°F)

Material	Weight Loss (percent) after 6 hours at 450K (350°F)	Weight Gain (percent) after Exposure to 1273K (1832°F)
Aluminum boride	0.3	—
Boron carbide (325 mesh)	2.6	—
Boron (5 microns)	3.7	70.8
Boron (100 mesh)	0.2	36.7
Boron carbide (100 mesh)	0.2	—

Table 5. Vacuum Spectroscopy for Trace Elements in Boron-Containing Fillers

Filler Powder	Greater than 10 percent	1 to 10 percent	0.1 to 1 percent	Less than 0.1 percent
Boron (5 microns)	B	Mg, Si	Mn, Fe, Al, Cu	Ca
Boron (100 mesh)	B	Si, Al	Mg, Mn, Fe, Cu	Ca, Pb, Sn
Boron carbide (325 mesh)	B	Si	Fe, Cu	Mg, Mn, Al, Ca 1/
Boron carbide (100 mesh)	B	Si, Fe	Ni, Al, Cu	Mg, Mn, Ca, Cr 1/
Aluminum boride (325 mesh)	B, Al	Si	Fe, Cu	Mg, Mn, Ca

1/ Used "C" electrode.

Table 6. Trace Element Content of Boron-Containing Compounds by Energy-Dispersive X-Ray and Neutron Activation

Filler	Oxygen percent 1/	Less than 0.1 percent	0.01-0.1 percent	0.1-0.5 percent	0.5-1.0 percent	1.0-5.0 percent	Major
Boron metal B (5 microns)	6.0	Ti, Ni, Zn, As, Se, Br, Pb, Sr, Zr, Nb, Mo	Cu	K, C, Mn, Cl, Fe	Cl	—	—
Boron metal B (100 mesh)	4.2	Cl, W, Ga, Ni, Cu, As, Cr, Se, Pb, Rb, Sr, Y, Zn, Nb, Mo	—	K, Ca, Ti,	—	Si	—
Boron carbide (325 mesh)	0.14	Cu, Sr, Zr	Cl	Ca, Ti, Fe	—	—	—
Boron carbide (100 mesh)	2.9	V, Ni, Cu	Si, Ti	Ca	—	Fe	—
Aluminum boride	0.84	V, Ni, Cu, Se, Sr, Y	Ca, T, W, Cr, Mn, Zr, Nb, Mo	Fe	—	—	Al

1/ Oxygen content determinated by neutron activation.

Table 7. Summary of Boron Fillers Average Particle Size After Milling

Powder	Milling Time (hours)	Average Particle Size (microns)	Epoxy Sizing (percent) 1/
Boron (5 microns)	Without grinding	Too small 2/	6.9
Aluminum boride (325 mesh)	1/2	6	6.9
	8	3	5.1
	16	1	2.9, 3.3
Boron carbide (325 mesh)	3/4	Too small 2/	4.2
	8	Too small 2/	2.8
Boron (100 mesh)	8	3	4.8
	16	1.5	2.0
	48	Too small 2/	2.0
Boron carbide (100 mesh)	8	4.5	5.2
	16	3	3.0
	48	Too small 2/	3.6

1/ Measured amount of epoxy resin deposited on surface of filler during milling operation. Resin used during milling operation to enhance adhesion of fillers in composites.

2/ Exceeds the Hegman gage limit.

Table 8. Composite Weight Loss and Fiber Retention

Filler Powder	Milling Time (hours)	Percent Weight Loss at 1089K (1500°F) 1/	Observation of Fiber Retention
Boron* (5 microns)	Without grinding	27.1	Plies intact, can be separated, edges do not fray
Boron (100 mesh)	8	29.1	Plies intact, can be separated, edges do not fray
	16	27.2	
	48*	23.6	
Boron carbide* (325 mesh)	48		
Boron carbide (100 mesh)	3/4	27.8	Plies intact, can be separated, edges do not fray
	8	26.6	
	16	21.4	
	48*	24.3	
Aluminum boride (325 mesh)	1/2	24.9	Plies intact, separate easily, edge fray
	8	27.5	Plies intact, can be separated, edges do not fray
	16*	27.7	

*Optimum filler size.

1/ Average of two samples exposed for 2 minutes in a muffle furnace.

2/ Selected on basis of boron carbide (100 mesh) data.

Table 9. Tensile Strength, Graphite Fabric (±45-deg)/Filled Narmco 5208

Filler (Milling Time, hours)	Tension Ultimate Stress, MPa (ksi)
Boron (5 microns)	51.3 (7.45)
Boron (100 mesh)	
(8)	49.3 (7.16)
(16)	59.3 (8.60)
(48)	104.0 (15.10)
Boron carbide (325 mesh)	
(0.75)	88.7 (12.87)
(8)	100.7 (14.62)
Boron carbide (100 mesh)	
(8)	89.0 (12.92)
(16)	72.4 (10.51)
(48)	100.5 (14.59)
Aluminum boride (325 mesh)	
(0.5)	36.0 (4.36)
(8)	57.5 (8.35)
(16)	77.6 (11.26)

Table 10. Filler and Resin Content of Milled Solution

Filler	Filler Content (percent) 1/	Resin Content (percent) 1/
Boron (5 microns)	20.5	3.9
Boron (100 mesh), 48 hours	41.7	1.2
Boron carbide (325 mesh), 48 hours	26.4	1.0
Boron carbide (100 mesh), 48 hours	34.6	0.8
Aluminum boride (325 mesh), 16 hours	18.7	1.1

1/ Values are averages of three determinations.

Table 11. Summary of Test Data on Control Specimens

Properties 1/	Level of Filler														
	10 percent					5 percent					2.5 percent				
	Boron 5-micron	Boron (-100) 2/	B_4C (-100)	B_4C (-325)	AlB_{12} (-325)	Boron 5-micron	Boron (-100)	B_4C (-100)	B_4C (-325)	AlB_{12} (-325)	Boron 5-micron	Boron (-100)	B_4C (-100)	B_4C (-325)	Al (-3
Composite Physical Properties															
Resin Content, percent 3/	46.1	38.7	31.6	39.4	—	36.9	33.1	29.9	34.0	38.4	37.4	30.6	28.8	31.7	32
Specific Gravity, g/cc	1.534	1.593	1.603	1.592	1.587	1.588	1.598	1.598	1.593	1.575	1.57	1.58	1.59	1.59	1.5
Fiber Volume, percent	47.6	55.5	62.3	54.8	—	57.0	60.7	63.7	59.8	55.1	56.2	62.8	64.6	61.9	60
Composite Mechanical Properties															
Flexural Stress Ultimate, MPa (ksi) 4/															
RT	1199 (174)	1013 (147)	1089 (158)	992 (144)	737 (107) 5/	1109 (161)	992 (144)	1082 (157)	971 (141)	489 (71)	950 (138)	1288 (187)	1268 (184)	1371 (199)	95 (1
450K (350°F)	—	896 (130)	710 (103)	586 (85)	593 (86) 5/	834 (121)	655 (95)	723 (105)	785 (114)	544 (79)	937 (136)	516 (75)	531 (77)	524 (76)	56 (8
Flexural Modulus, GPa (Msi) 4/															
RT	113 (16.4)	112 (16.2)	105 (15.2)	93 (13.5)	83 (12.1) 5/	96 (14.0)	94 (13.7)	107 (15.6)	107 (15.6)	103 (14.9)	129 (18.7)	131 (19.0)	139 (20.2)	147 (21.4)	15 (2
450K (350°F)	—	101 (14.7)	108 (15.7)	92 (13.3)	80 (11.6) 5/	96 (14.0)	90 (13.0)	103 (15.0)	104 (15.1)	96 (13.9)	117 (17.0)	141 (20.5)	142 (20.6)	141 (20.5)	12 (1
Interlaminar Shear Ultimate, MPa (ksi) 5/															
RT	83 (12.0)	83 (12.1)	86 (12.5)	91 (13.2)	84 (12.2)	88 (12.8)	90 (13.1)	89 (12.9)	87 (12.6)	85 (12.3)	96 (13.9)	70 (10.2)	67 (9.7)	82 (11.9)	70 (1
450K (350°F)	34 (4.9)	32 (4.6)	34 (4.9)	25 (3.6)	33 (4.8)	47 (6.8)	35 (5.1)	43 (6.2)	40 (5.8)	36 (5.2)	52 (7.5)	32 (4.6)	37 (5.3)	—	31 (4.

1/ All specimens 0-degree orientation.
2/ (-xxx) indicates screen size.
3/ Resin content determined by acid digestion and includes resin + filler.
4/ Values normalized to 60% fiber volume.
5/ Values not normalized to 60% fiber volume.

NOTE: Typical properties Narmco 5208/T300, flexural values normalized to 60% fiber volume.

Flexural Stress Ultimate	MPa (ksi)
RT	1791 (260)
450K (350°F)	1350 (196)
Flexural Modulus	GPa (Msi)
RT	138 (20.0)
450K (350°F)	124 (18.0)
Interlaminar Shear	MPa (ksi)
RT	104 (15.1)
450K (350°F)	61 (8.8)

Table 12. Summary of Test Data on Environmental Specimens
(500 hours at 450K (350°F) plus 24 hours water boil)

	Level of Filler														
	10 percent					5 percent					2.5 percent				
perties 1/	Boron 5-micron	Boron (-100) 2/	B_4C (-100)	B_4C (-325)	AlB_{12} (-325)	Boron 5-micron	Boron (-100)	B_4C (-100)	B_4C (-325)	AlB_{12} (-325)	Boron 5-micron	Boron (-100)	B_4C (-100)	B_4C (-325)	AlB_{12} (-325)
nposite Physical perties															
in Content, percent 3/	46.1	38.7	31.6	39.4	—	36.9	33.1	29.9	34.0	38.4	37.4	30.6	28.8	31.7	32.9
cific Gravity, g/cc	1.554	1.593	1.603	1.592	1.587	1.588	1.598	1.598	1.593	1.575	1.57	1.58	1.59	1.59	1.58
er Volume, percent	47.6	55.5	62.3	54.8	—	57.0	60.7	63.7	59.8	55.1	56.2	62.8	64.6	61.9	60.4
nposite Mechanical perties															
ural Stress Ultimate, a (ksi) 4/															
RT	1123 (163)	1116 (162)	1192 (173)	1034 (150)	737 (107) 5/	1137 (165)	1054 (153)	1123 (163)	1006 (146)	889 (129)	1186 (172)	474 (68.8)	627 (90.0)	862 (125)	590 (85.5)
450K (350°F)	310 (45)	620 (90)	556 (81)	599 (87)	496 (72) 5/	723 (105)	668 (97)	737 (107)	710 (103)	558 (81)	525 (76.1)	265 (38.5)	258 (37.4)	321 (46.5)	299 (43.3)
xural Modulus, a (Msi) 4/															
RT	104 (15.1)	105 (15.3)	108 (15.7)	102 (14.8)	88 (12.8) 5/	123 (17.9)	105 (15.3)	94 (13.7)	108 (15.7)	111 (16.1)	83.9 (12.2)	55.3 (8.0)	68.5 (9.9)	71.5 (10.6)	63.7 (9.2)
450K (350°F)	65 (9.4)	99 (14.4)	97 (14.1)	97 (14.1)	781 (11.3) 5/	101 (14.6)	101 (14.7)	108 (15.7)	108 (15.7)	109 (14.4)	45.6 (6.6)	38.9 (5.6)	32.7 (4.7)	38.1 (5.5)	37.0 (5.4)
erlaminar Shear imate, MPa (ksi)															
RT	68 (9.8)	64 (9.3)	74 (10.8)	70 (10.1)	76 (11.0)	71 (10.3)	77 (11.2)	72 (10.4)	76 (11.0)	68 (9.8)	70.3 (10.2)	41.4 (6.0)	44.1 (6.4)	64.8 (9.4)	45.5 (6.6)
450K (350°F)	20 (2.9)	26 (3.8)	28 (4.1)	25 (3.7)	32 (4.6)	30 (4.4)	36 (5.2)	39 (5.7)	34 (4.9)	32 (4.7)	47.6 (6.9)	33.8 (4.9)	58.6 (8.5)	40.7 (5.9)	31.0 (4.5)

1/ All specimens 0-degree orientation.
2/ (-xxx) indicates screen size.
3/ Resin content determined by acid digestion and includes resin + filler.
4/ Values normalized to 60% fiber volume.
5/ Values not normalized to 60% fiber volume.

NOTE: Typical properties Narmco 5208/T300, flexural values normalized to 60% fiber volume.

Flexural Stress Ultimate	MPa (ksi)
RT	1791 (260)
450K (350°F)	1350 (196)
Flexural Modulus	GPa (Msi)
RT	138 (20.0)
450K (350°F)	124 (18.0)
Interlaminar Shear	MPa (ksi)
RT	104 (15.1)
450K (350°F)	61 (8.8)

Table 13. Task III Physical and Mechanical Properties Tests

Test (Test Method)	Specimen Conditioning	Test Temperature (No. of Specimens) RT	450K (350°F)
Flexural Stress Ultimate (ASTM D790, 4-point loading)	No conditioning	3	3
	1000 hours, air, at 450K (350°F) plus 1000 hours, 95% RH, 322K (140°F)	3	3
Flexural Modulus Ultimate (ASTM D790, 4-point loading)	No conditioning	3	3
	1000 hours, air, at 450K (350°F) plus 1000 hours, 95% RH, 322K (140°F)	3	3
Interlaminar Shear (ASTM D2344)	No conditioning	3	3
	1000 hours, air, at 450K (350°F) plus 1000 hours, 95% RH, 322K (140°F)	3	3
Weight Change	1000 hours, air, at 450K (350°F) plus 1000 hours, 95% RH, 322K (140°F)	12	-
Resin Content	No conditioning	3	-
Void Volume	No conditioning	3	-
Fiber Volume	No conditioning	3	-

Table 14. Task III Composite Properties—"As Fabricated"

Boron Filler/Level	Flexural Stress Ultimate, MPa (ksi)		Flexural Modulus, GPa (Msi)		ILS Ultimate, MPa (ksi)		Resin Content, percent	Specific Gravity, g/cc	Fiber Volume, percent
	RT	405K (350°F)	RT	405K (350°F)	RT	405K (350°F)			
None	1502 (218)	1330 (164)	137 (19.9)	136 (19.8)	95.6 (13.9)	51.0 (7.4)	28.3	1.590	65.1
Boron (5M)/2.5%	1254 (182)*	882 (128)	124 (18.0)	124 (18.0)	92.3 (13.4)	42.7 (6.2)	35.9	1.590	58.2
Boron (5M)/5.0%	1316 (191)*	909 (132)	128 (18.6)	115 (16.7)	82.0 (11.9)	36.5 (5.3)	37.3	1.585	56.8
Boron Carbide (100)/ 5.0%	1345 (195)*	779 (113)	114 (16.6)	11 1 (16.1)	64.1 (9.3)	35.8 (5.2)	30.1	1.578	63.0

*Flexural values were normalized to 65 percent fiber volume for comparative purposes.

Table 15. Summary Property Data—Environmentally Conditioned Specimens 1/

	Control	Boron 5M 2.5%	Boron 5M 5.0%	Boron Carbide -100M 5%
Composite Physical Properties				
Resin Content, percent	28.3	35.9	37.3	30.1
Fiber Volume, percent	65.1	58.2	56.8	63.0
Specific Gravity, percent	1.590	1.590	1.585	1.578
Composite Mechanical Properties 2/				
Flexural Ultimate, MPa (ksi) 3/				
295K (RT)	1350 (196)	1096 (159)	1089 (158)	331 (48)
450K (350°F)	579 (84)	593 (86)	503 (73)	303 (44)
Flexural Modulus, GPa (Msi)				
295K (RT)	127 (18.4)	130 (18.8)	110 (15.9)	105 (15.2)
450K (350°F)	101 (14.6)	104 (15.1)	110 (16.0)	93 (13.5)
Short Beam Shear, MPa (ksi)				
295K (RT)	61.3 (8.9)	50.3 (7.3)	47.5 (6.9)	30.3 (4.4)
450K (350°F)	30.3 (4.4)	20.7 (3.0)	20.7 (3.0)	26.9 (3.9)
Weight Gain After Conditioning, %				
Flexural Specimens	1.14	1.69	1.79	3.06
Short Beam Shear Specimens	1.23	1.76	2.00	4.29

1/ Specimens aged 1000 hours at 450K (350°F), then 1000 hours at 95% relative humidity and 322K (140°F).

2/ Test specimens in wet condition.

3/ Flexural values normalized to 60 percent fiber volume.

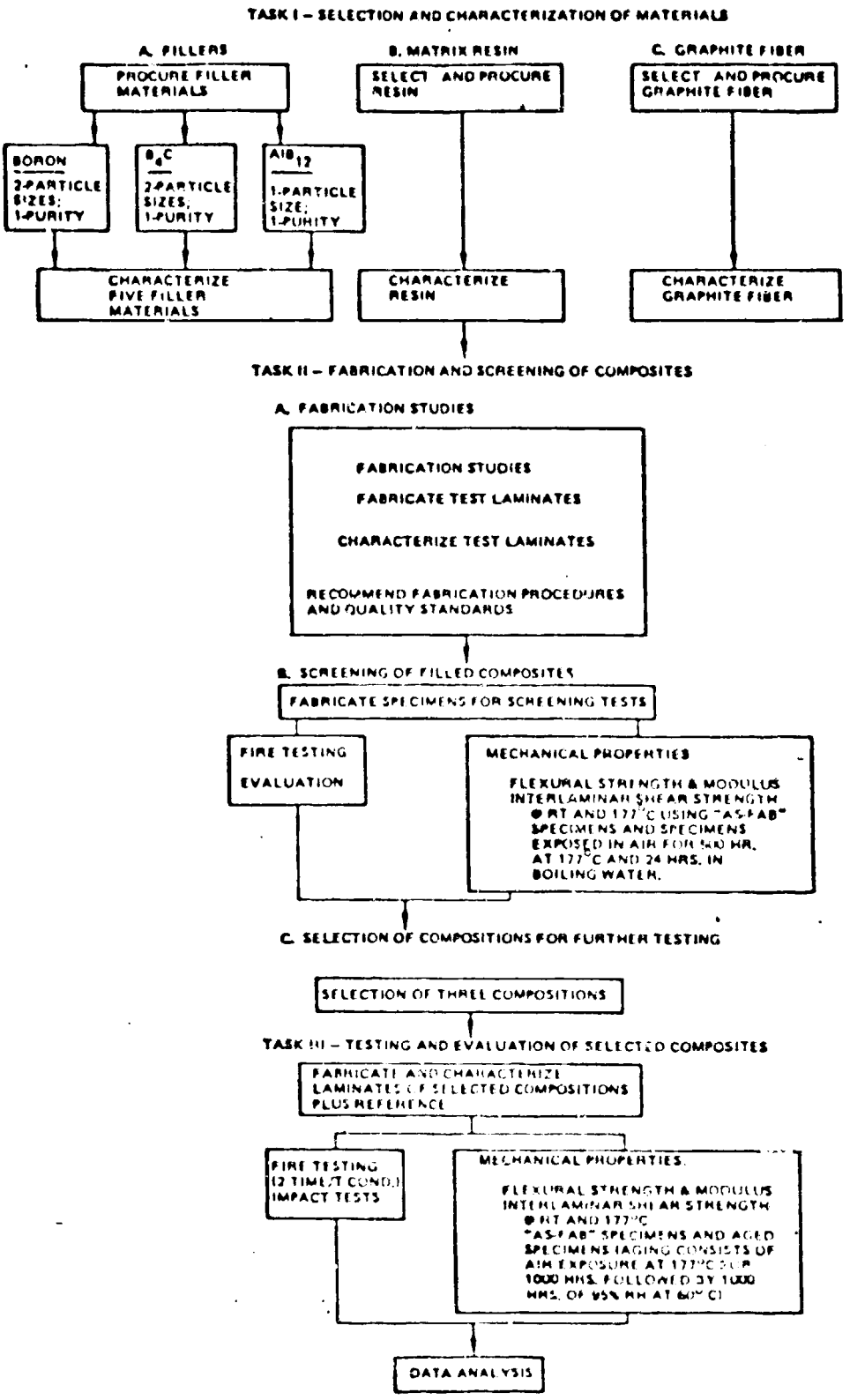

Figure 1. Program Flow Diagram

Figure 2: Preparation of Celion 600/Narmco 5208 Prepreg

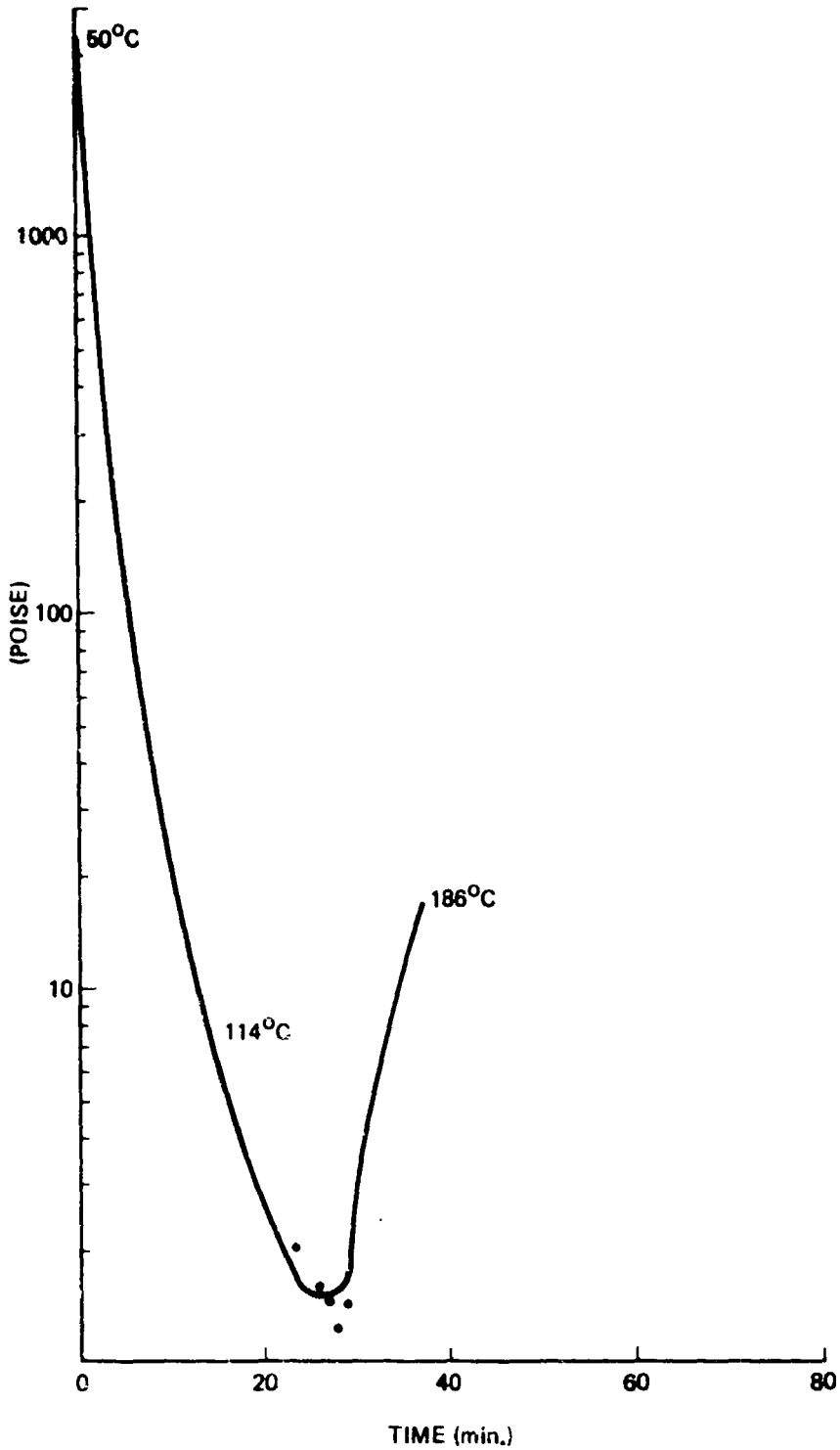

Figure 3: Rheometric Curve: Control, Narmco 5208 Resin

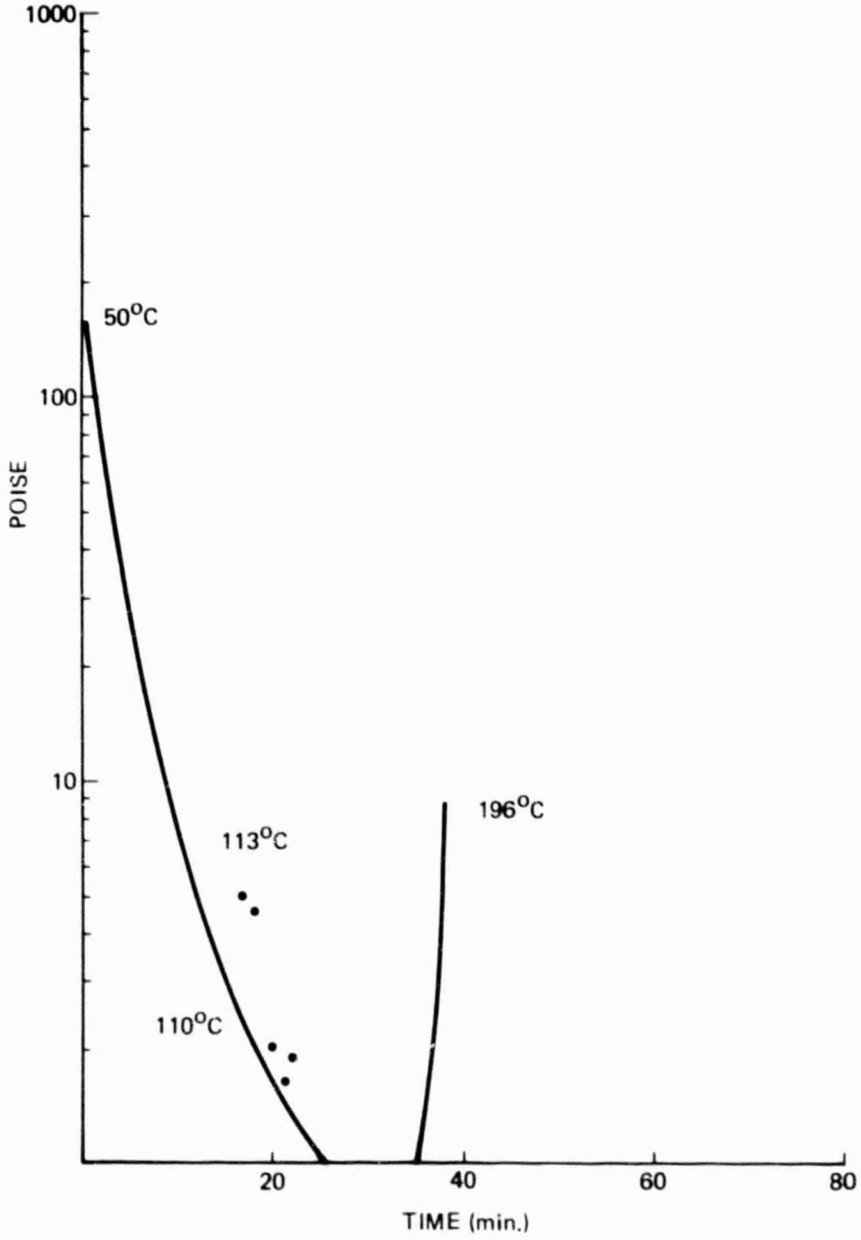

Figure 4: Rheometric Curve: Boron (-100 Mesh), Milled 48 Hours at 10 Percent Solids Loading

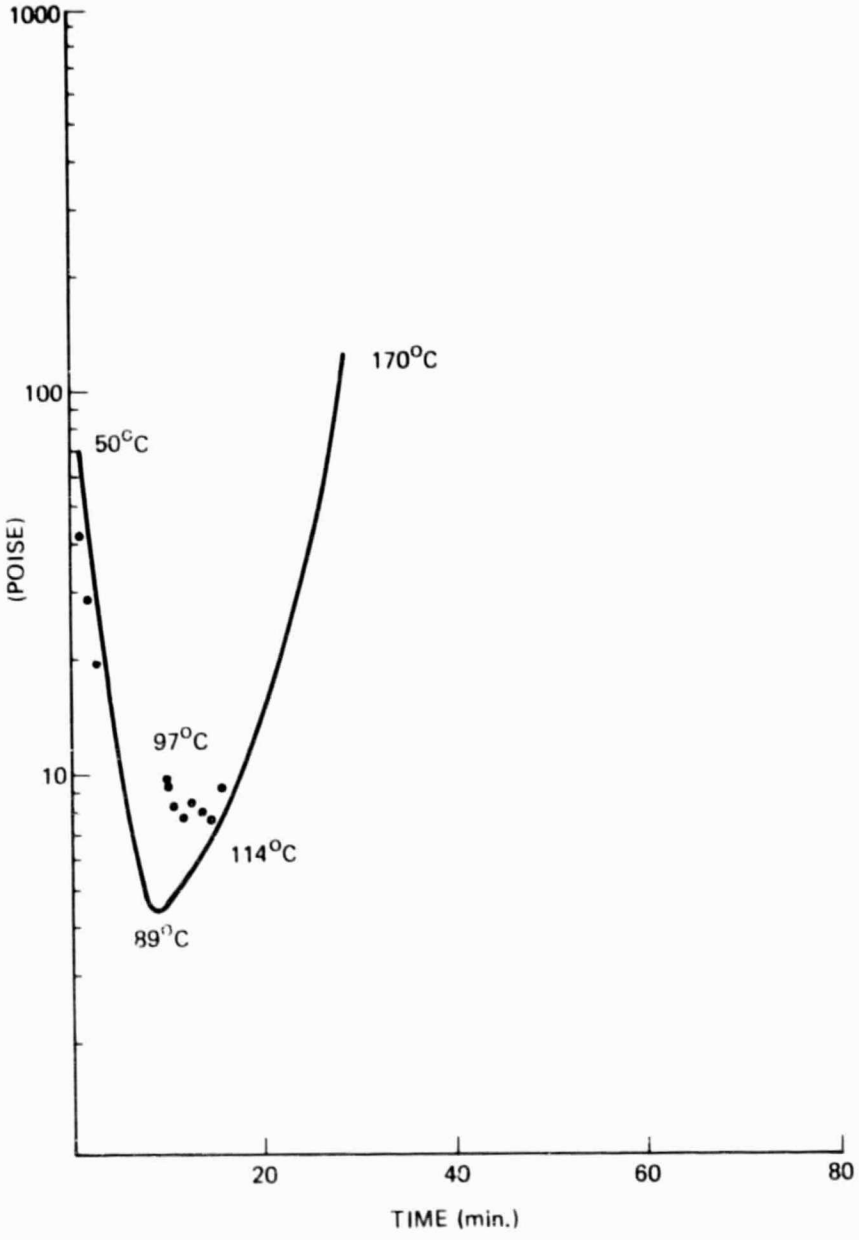

Figure 5: Rheometric Curve: Boron (5 Micron) at 10 Percent Solids Loading

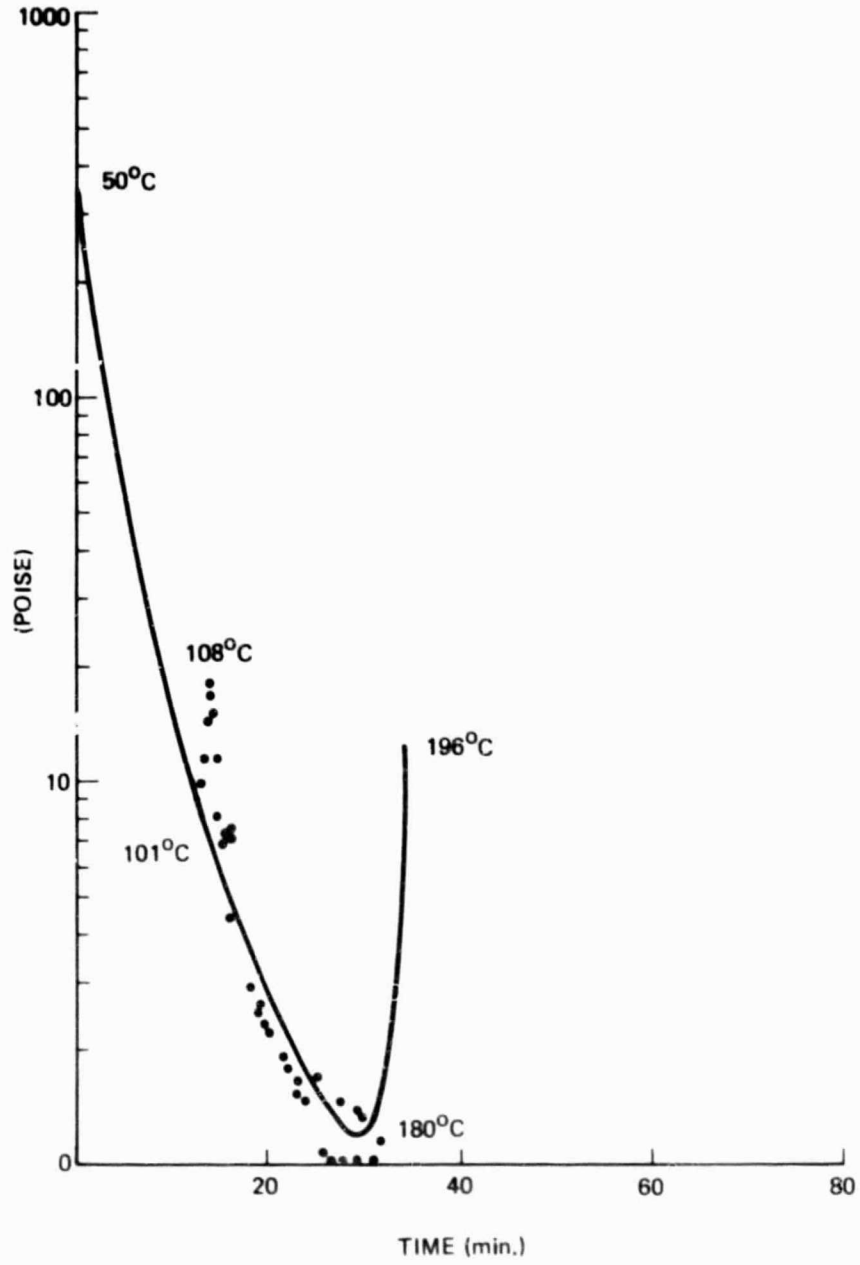

Figure 6: Rheometric Curve: Boron Carbide (-325 Mesh), Milled 48 Hours, at 10 Percent Solids Loading

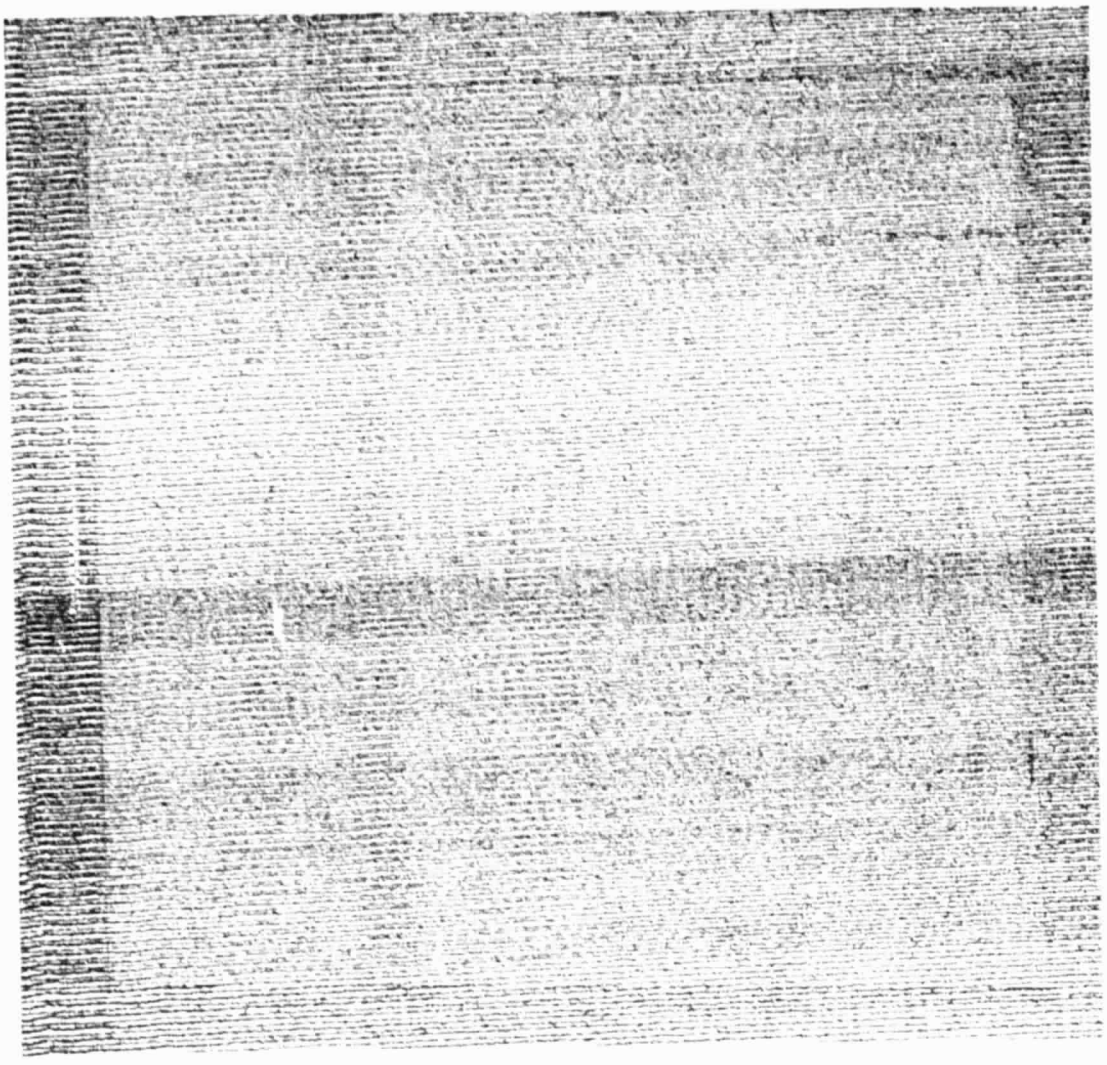

Figure 7: NDI "C" Scan, Boron (5 Micron) at 5 Percent Solids Loading

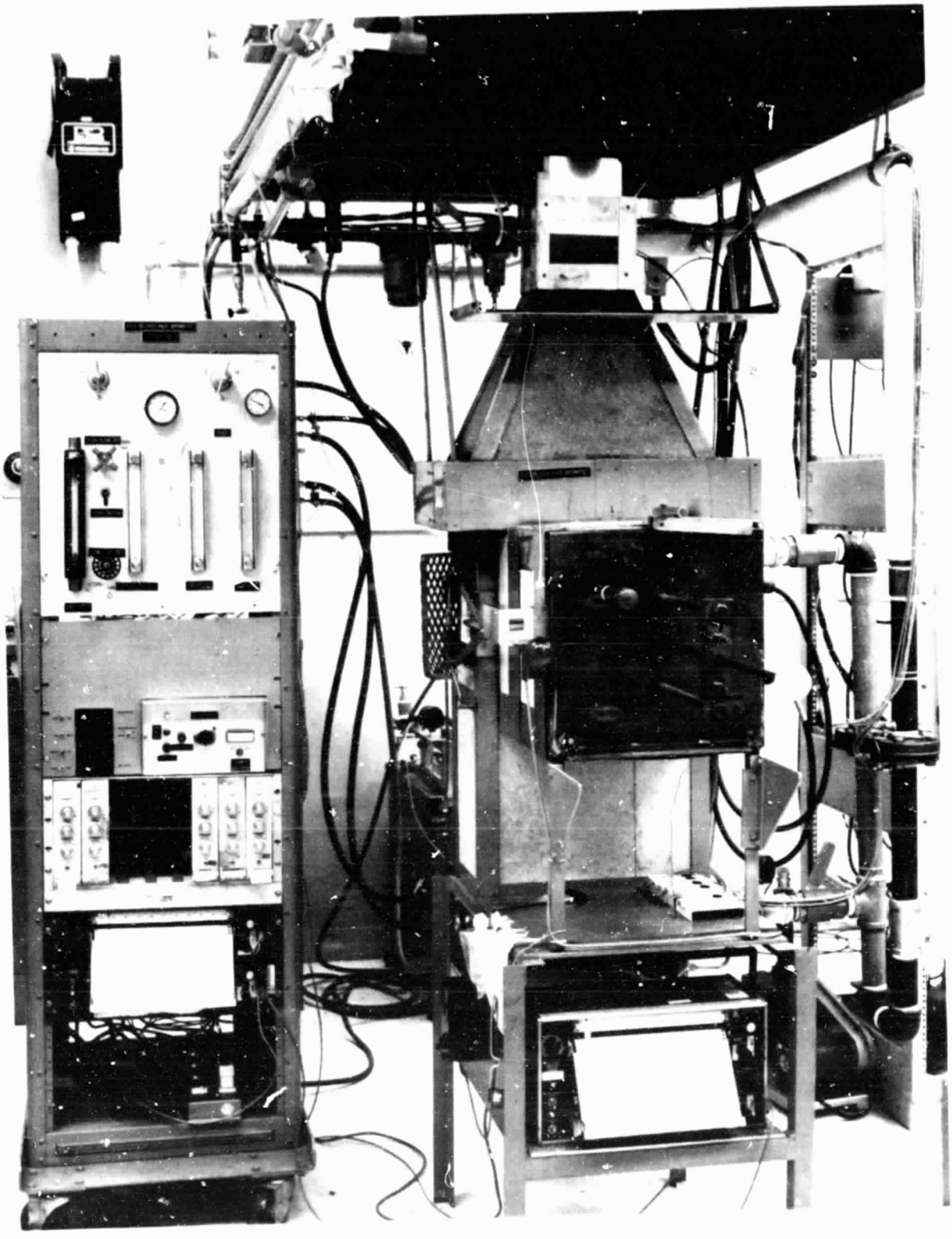

Figure 8: Ohio State University (OSU) Release Rate Apparatus

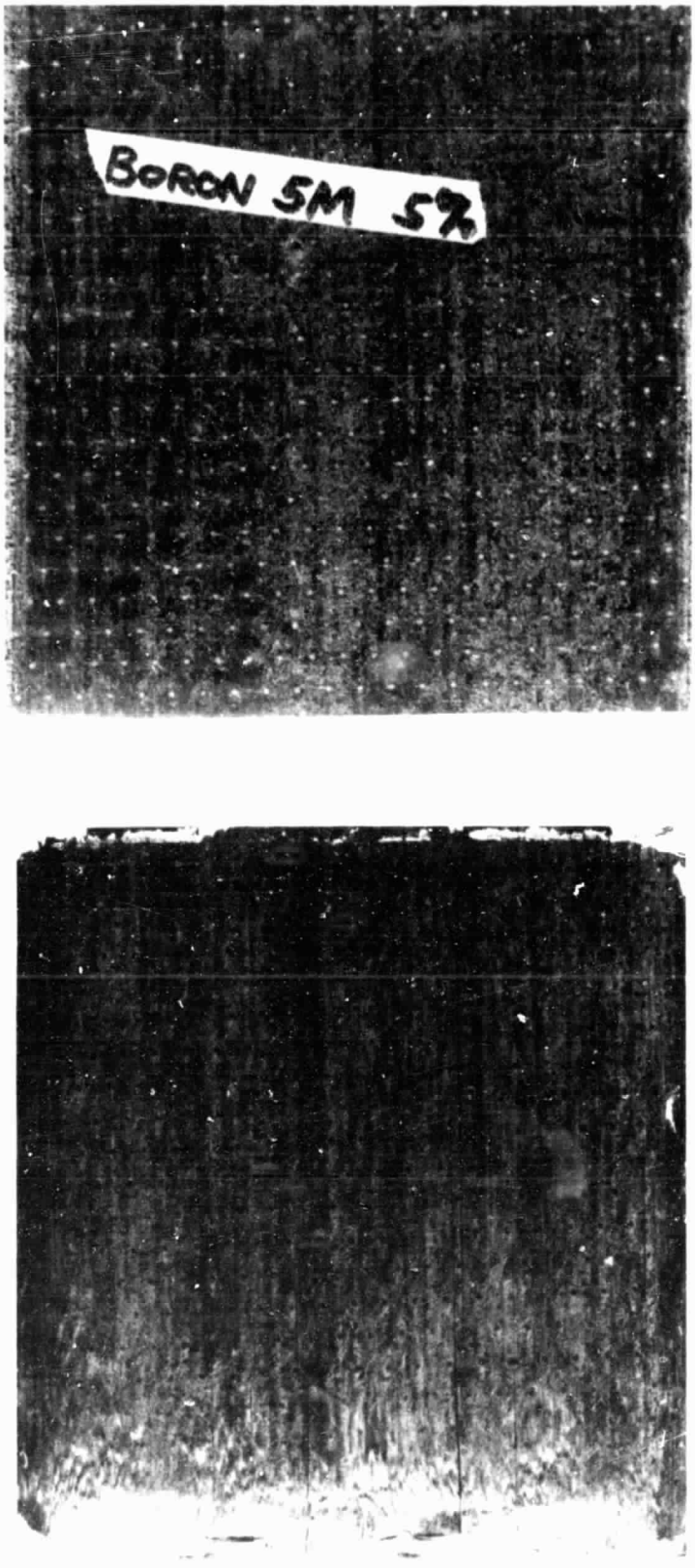

Figure 9 Boron (5-micron) Filled (5%) Laminate, Before and After Burn Test

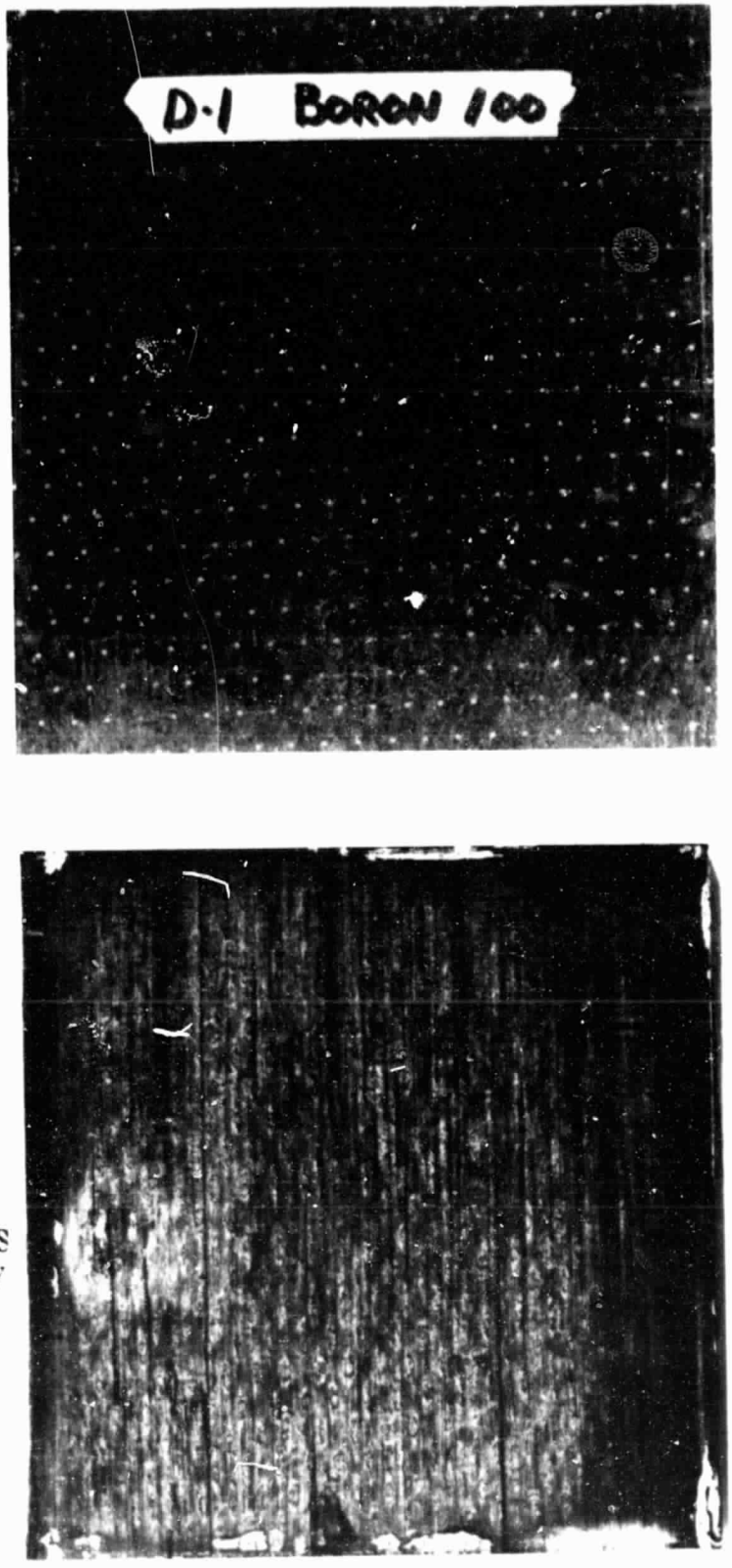

Figure 10: Boron (-100 Mesh) Filled (5%) Laminate, Before and After Burn Test

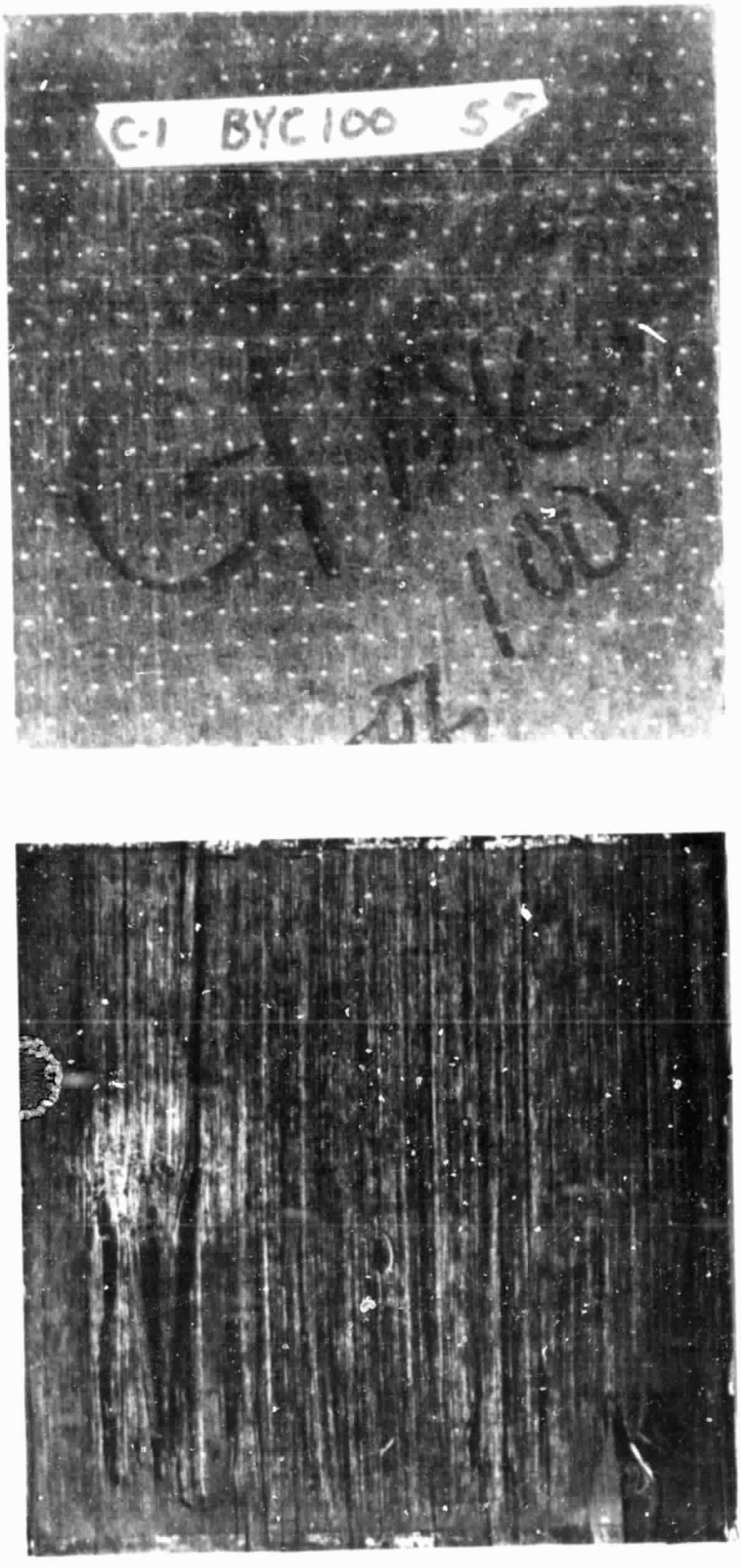

Figure 11: Boron Carbide (100 Mesh) Filled (5%) Laminate, Before and After Burn Test

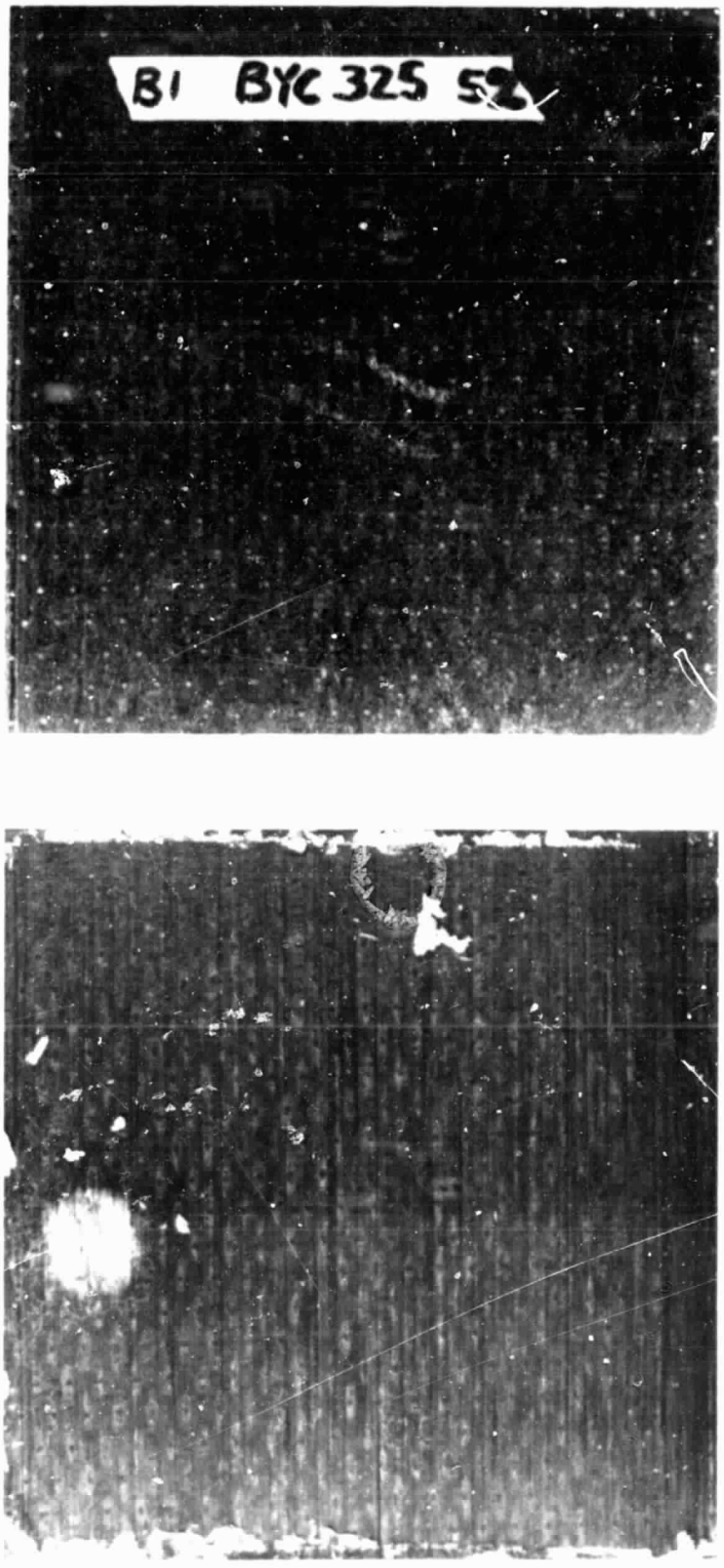

Figure 12: Boron Carbide (-325 Mesh) Filled (5%) Laminate, Before and After Burn Test

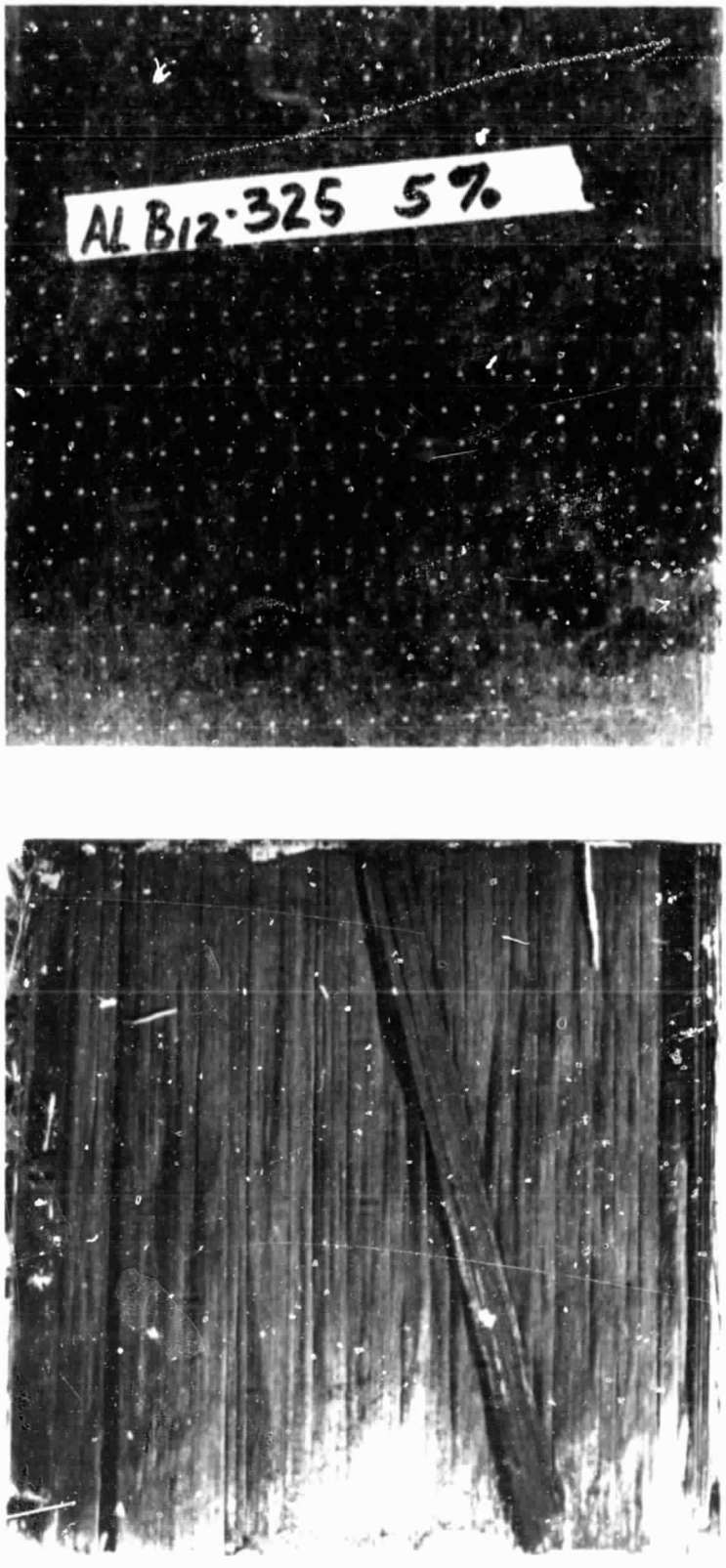

Figure 13: Aluminum Boride (-325 Mesh) Filled (5%) Laminate, Before and After Burn Test

Figure 14: Aluminum Boride (-325 Mesh) Filled (5%) Laminate During Burn Test

Figure 15: Boron (-100 Mesh) Filled (5%) Laminate During Burn Test

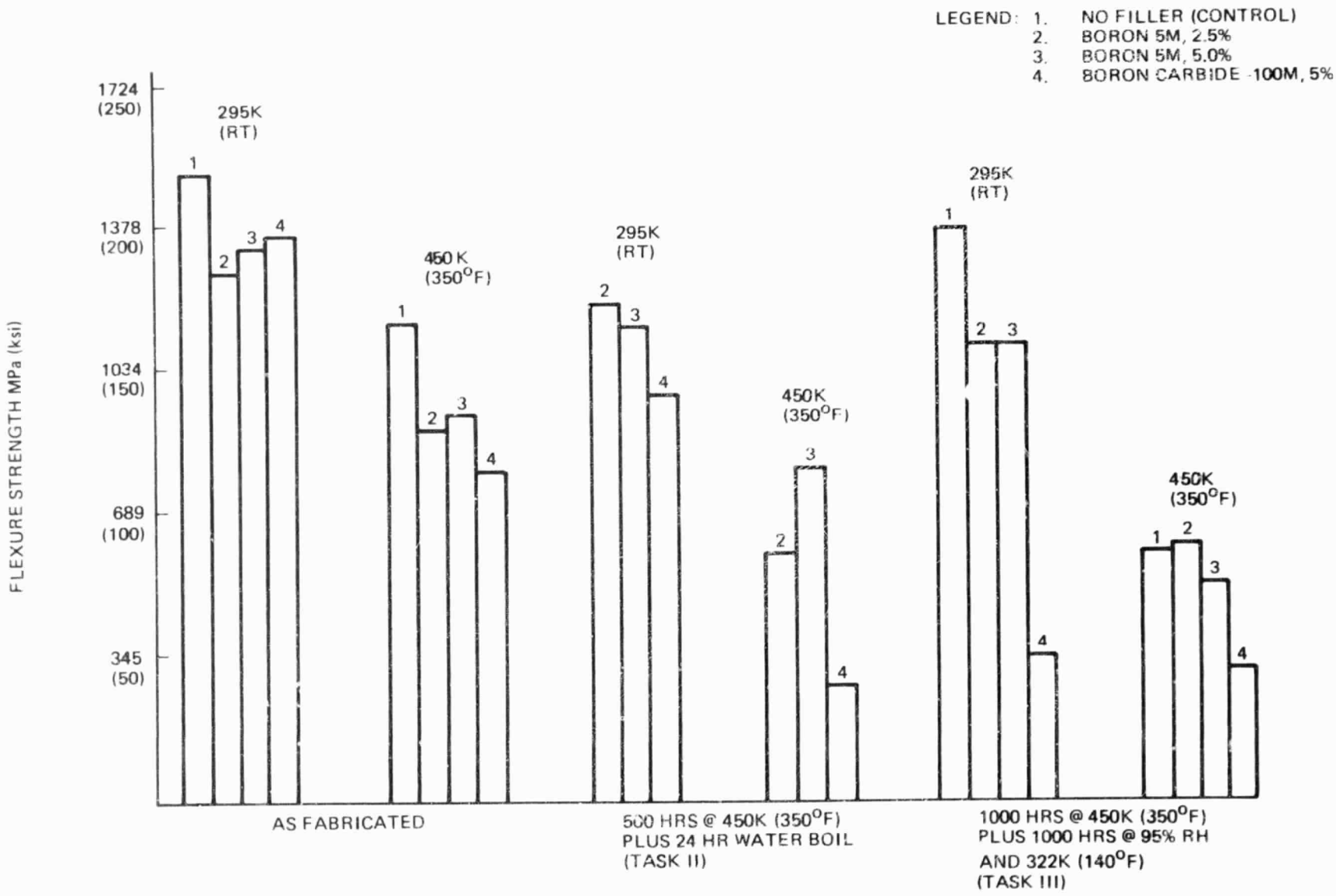

Figure 16: Effect of Environmental Exposure on Flexural Strength

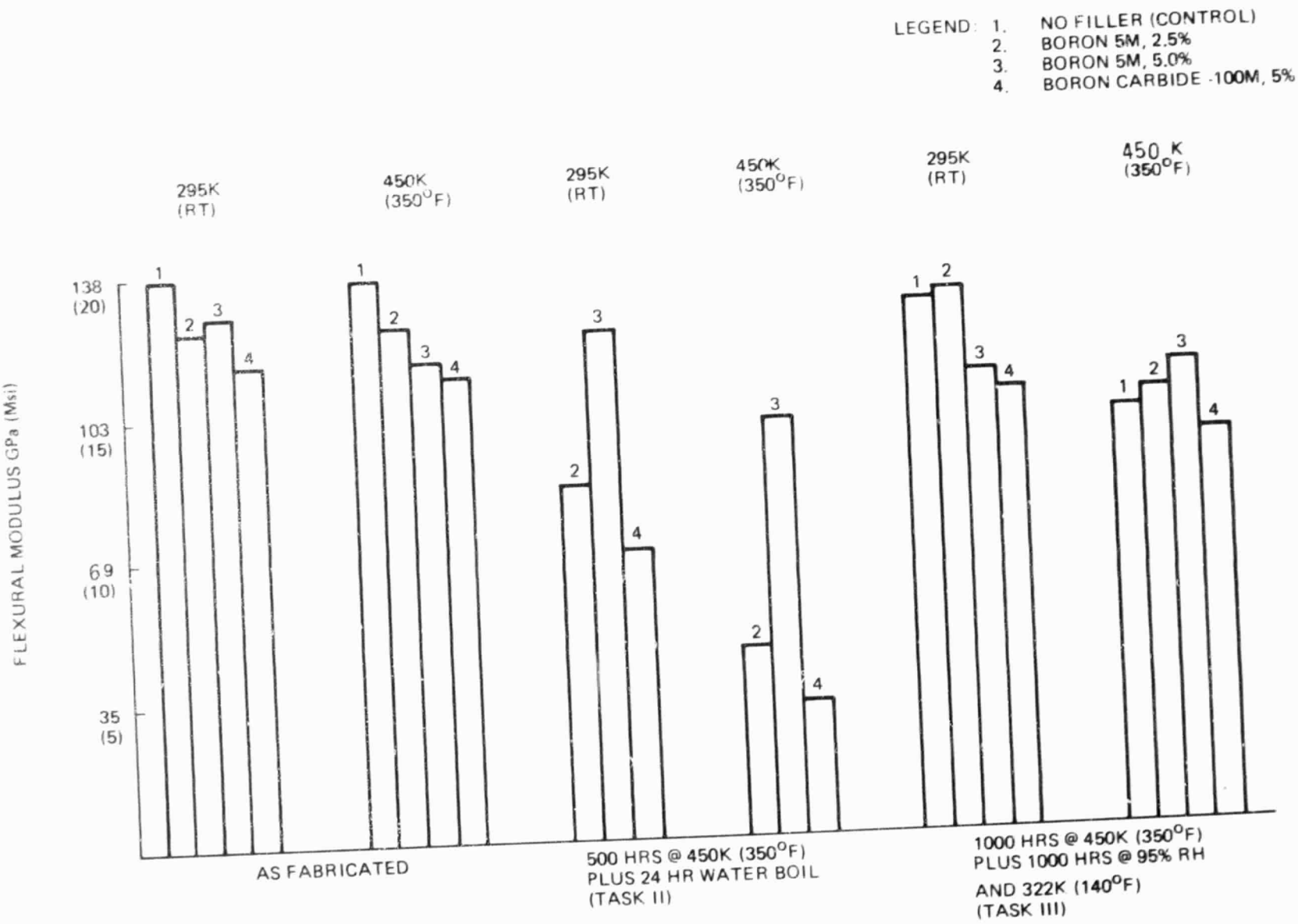

Figure 17: Effect of Environmental Exposure on Flexural Modulus

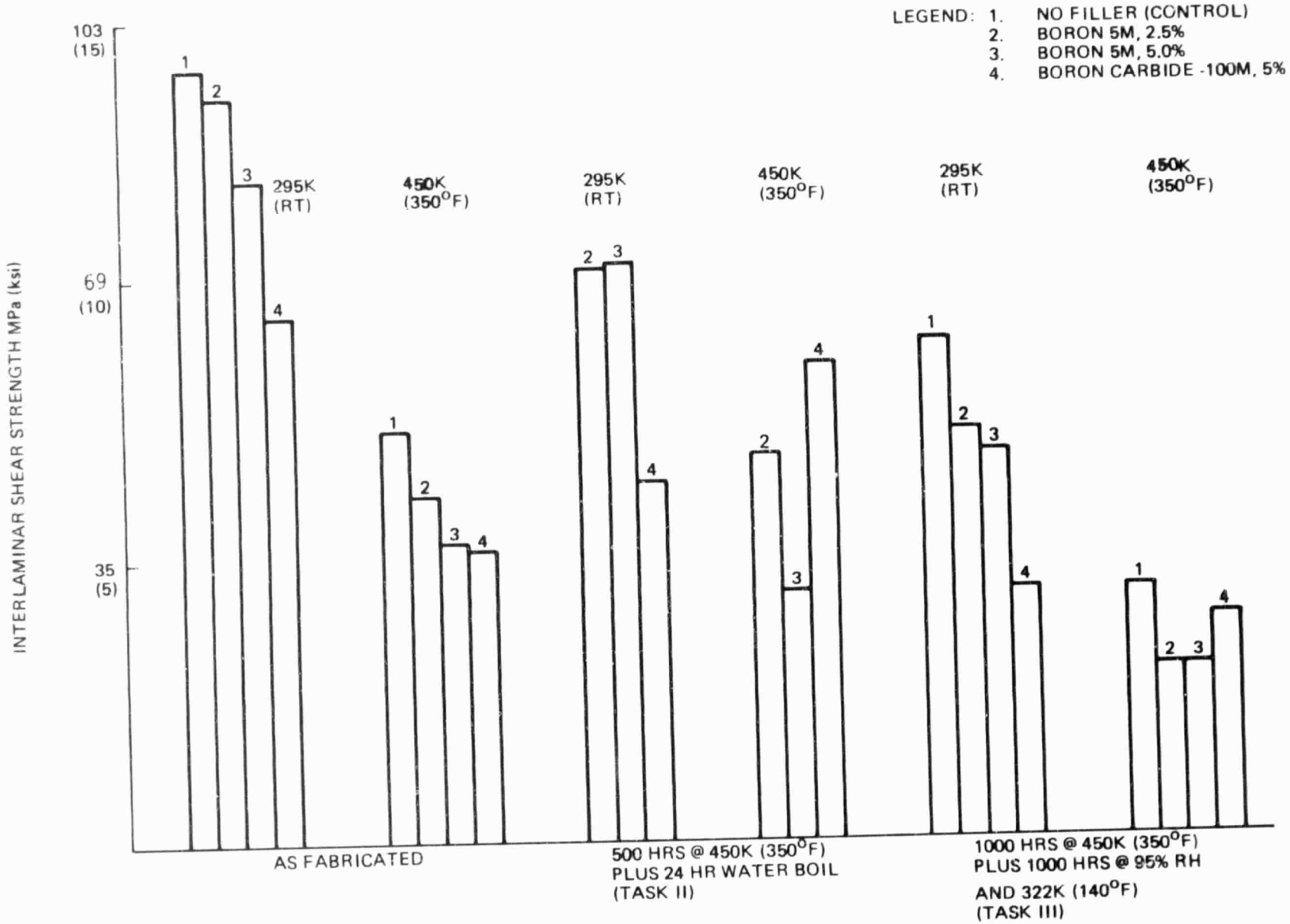

Figure 18: Effect of Environmental Exposure on Interlaminar Shear Strength

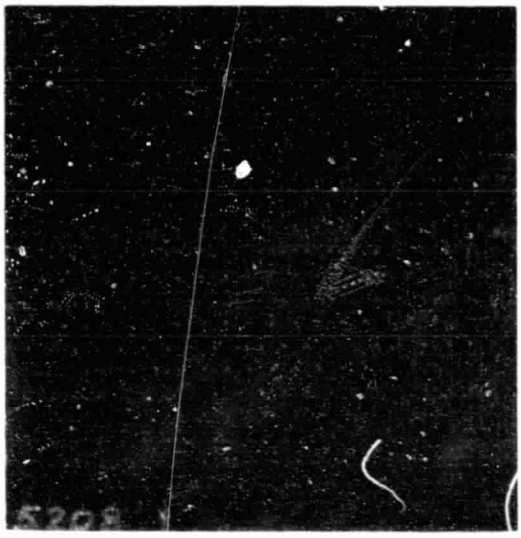

Figure 19: Specimen Prior to OSU Exposure

Figure 20: Unfilled (Control) Specimen After OSU Exposure (10W/cm^2)

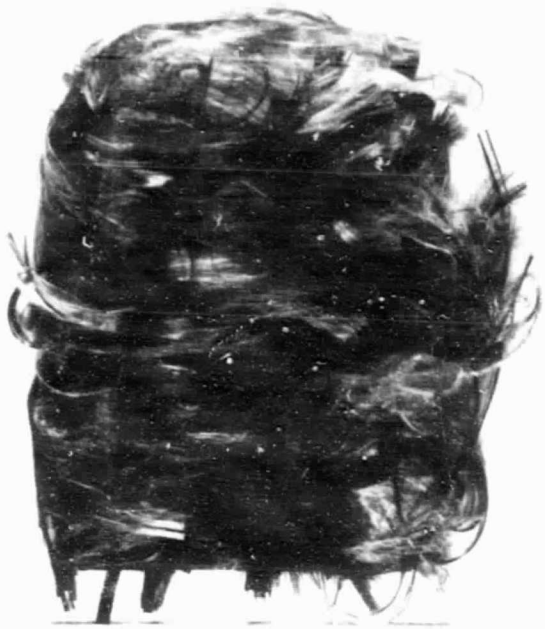

Figure 21: Unfilled (Control) Specimen After OSU Exposure (7.5/cm^2)

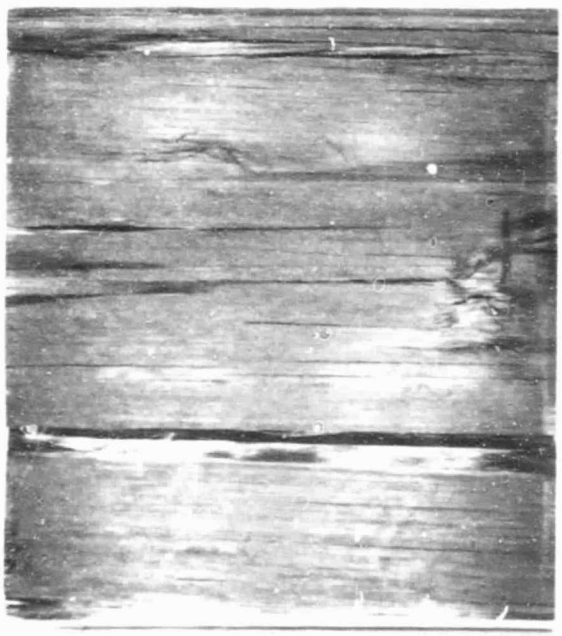

***Figure 22**: Boron Carbide (5%) Specimen After OSU Exposure (10W/cm^2)*

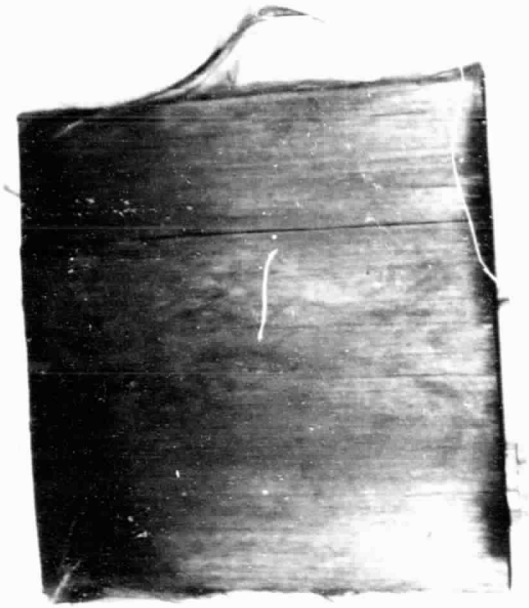

Figure 23: Boron Carbide (5%) Specimen After OSU Exposure (7.5W/cm^2)

Figure 24: Boron (5%) Specimen after OSU Exposure (10W/cm^2)

Figure 25: Boron (5%) Specimen after OSU Exposure (7.5W/cm^2)

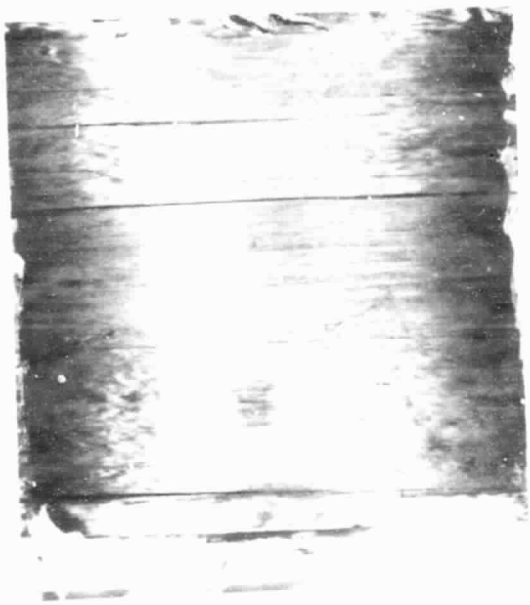

Figure 26: Boron (2.5%) Specimen after OSU Exposure (10W/cm^2)

Figure 27: Boron (2.5%) Specimen after OSU Exposure (7.5W/cm^2)

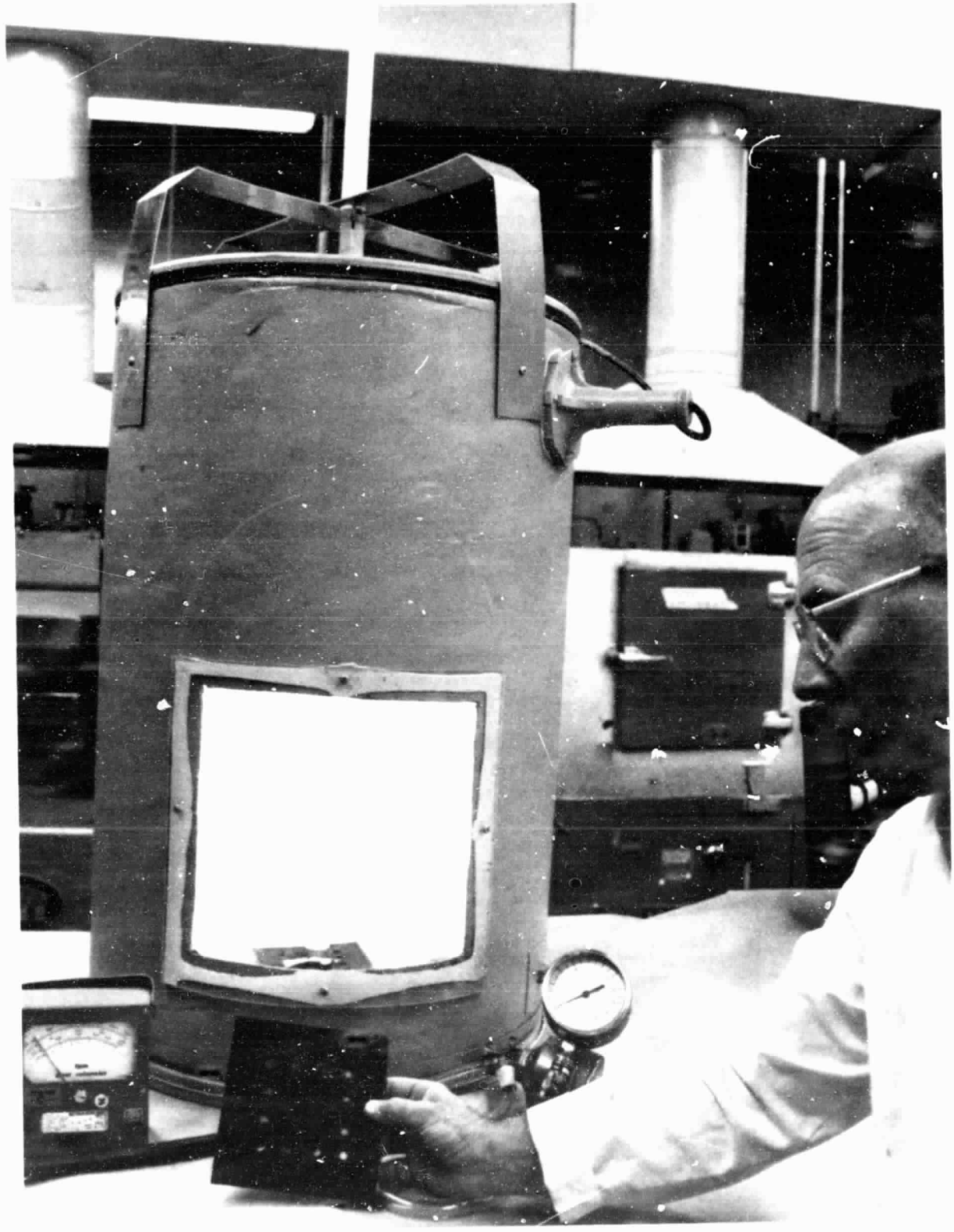

Figure 28: Impact and Air Flow Test Chamber

Figure 29: Unfilled (Control) Specimen after OSU Exposure (10W/cm^2) and Impact Testing

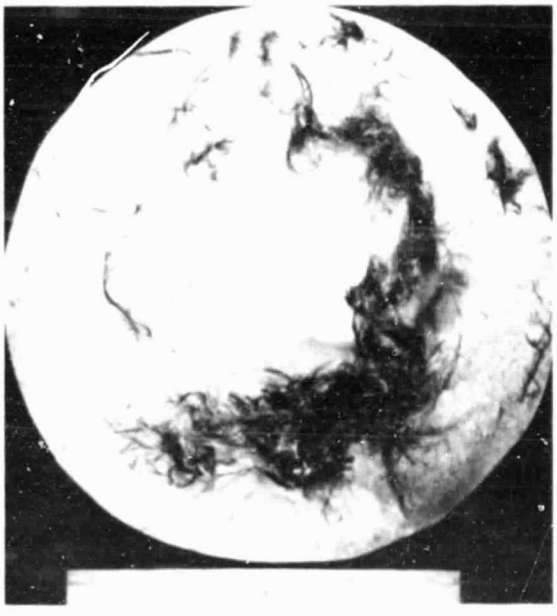

Figure 30: Unfilled (Control) Specimen after OSU Exposure (7.5W/cm^2) and Impact Testing

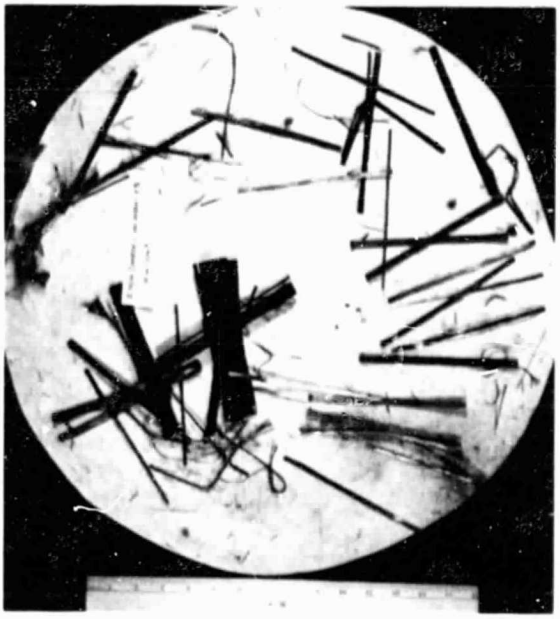

Figure 31: Boron Carbide (5%) Specimen after OSU Exposure (10W/cm^2) and Impact Testing

Figure 32: Boron (5%) Specimen after OSU Exposure (10W/cm^2) and Impact Testing

Figure 33: Boron (5%) Specimen after OSU Exposure (7.5W/cm^2) and Impact Testing

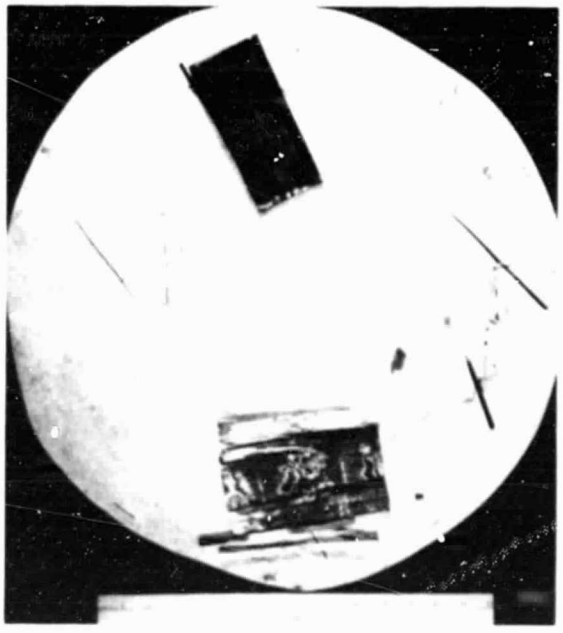

Figure 34: Boron (2.5%) Specimen after OSU Exposure (10W/cm^2) and Impact Testing

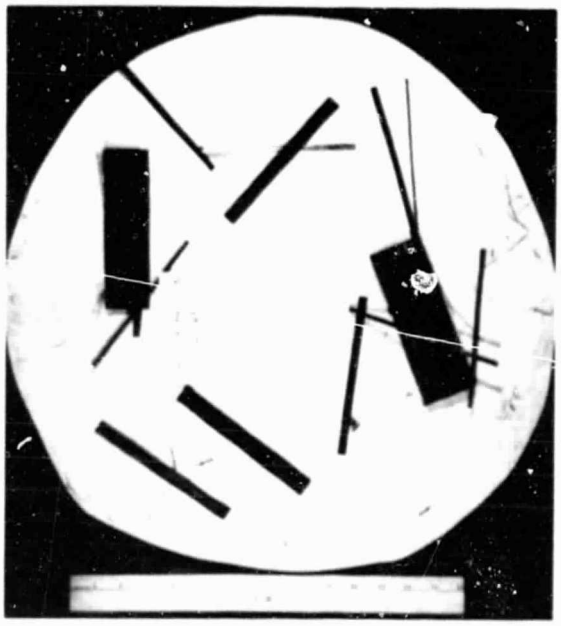

Figure 35: Boron (2.5%) Specimen after OSU Exposure (7.5W/cm^2) and Impact Testing