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FINAL REPORT

PYROLYTIC BORON NITRIDE COATINGS ON CERAMIC YARNS AND FABRICATION OF INSULATIONS

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ABSTRACT

Pyrolytic boron nitride (PBN) was deposited on Nicalon NL 202 silicon carbide yarns at 1000-1200°C with the goal of improving the resistance of the Nicalon to deterioration in an aerodynamic environment at temperatures up to 1000°C. For continuous coating, the yarns were fed through the deposition chamber of a pilot-plant-sized CVD furnace at a rate of about 2 feet per minute. PBN coatings were obtained by reacting boron trichloride and ammonia gases inside the deposition chamber. Most of the PBN coatings were made at around 1080°C to minimize thermal degradation of the Nicalon. Pressures were typically below 0.1 Torr.

The coated yarns were characterized by weight per unit length, tensile strength and modulus, scanning electron microscopy (SEM), and scanning Auger microscopy (SAM). The PBN coating thicknesses ranged from 0.1-0.7 microns depending on coating conditions, and the coatings were fairly uniform along the length of the yarn snf between fiber bundles. Nicalon coated with 0.1-0.2 microns of PBN was as strong as the as-received Nicalon. The PBN-coated Nicalon showed good resistance to oxidation in air up to 800°C, but the properties were degraded after air oxidation at 1000°C.

The PBN-coated Nicalon was woven into cloth at Fabric Development, Inc. Although the coated Nicalon was easier to handle than oxidized or vacuum-heat-treated yarn, the PBN coating was not entirely satisfactory as a high-temperature sizing. To control loose filaments during weaving into cloth and construction of sewing thread, the PBN-coated Nicalon was wrapped with 30-denier rayon. Quantities of plain-weave and 12-satin harness-weave cloths were prepared from the PBN-coated yarn.

Several 13 in. square pieces of Nicalon cloth were coated with PBN in a batch process in a factory-sized deposition furnace. Temperature, pressure, and time were 1080°C, 0.2 Torr, and 4 minutes, respectively. The batch PBN coatings averaged about 0.3 microns thick, but the coating thickness was more variable than that achieved by continuous yarn coating.

Samples of cloth made from the PBN-coated Nicalon were sewn into thermal insulation panels at Hi-Temp Insulation, Inc., Camarillo, California. The high-temperature performance of these panels is being compared with that of panels made using uncoated Nicalon.

Thicker PBN coatings and coatings with a more oxidation resistant outer layer will be required to minimize degradation of the Nicalon in an aerodynamic environment at 1000°C. An alloyed PBN coating and/or more moisture stable outer coating may be needed to improve the resistance of the PBN coating to reaction with atmospheric moisture.

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SUMMARY

The goal of the work described in this report is to coat ceramic yarns and cloths, in particular Nicalon Type NL 202 silicon carbide yarns, with pyrolytic boron nitride (PBN) so that thermal insulation panels made from these yarns exhibit enhanced resistance to physical damage in an aerodynamic or aeroacoustic environment. It was anticipated that the PBN coating would be beneficial for at least two reasons (1) as a high-temperature sizing to reduce abrasion and (2) as a coating to prevent oxidation of free carbon contained within the Nicalon fibers. Loss of this free carbon leads to surface pitting causing strength loss and also results in growth of the SiC crystallites in the Nicalon which causes strength loss and variability due to embrittlement of the fibers.

The Nicalon yarn and cloth for this program were purchased from Dow Corning. Type NL 202 is ceramic grade Nicalon having a density of 2.55 g/cc and containing 500 filaments totaling 1,800 denier. Each spool contained 100 grams (500 meters) of yarn. The sizing is polyvinyl acetate.

Strand tensile strength data were collected on 20 samples of yarn taken from 80-foot lengths of a 500-meter spool of Nicalon. The average tensile strength ranged from 353-402 ksi, and the tensile modulus was 28-29 Msi. One-minute heat treatments in low-pressure argon at 1000-1100°C caused about 20% loss in tensile strength. Heat treatments at 1200 and 1350°C led to much greater tensile strength losses.

PBN was deposited on Nicalon NL 202 yarn while continuously feeding it through a furnace and on Nicalon cloth using a batch coating process. Apparatus for continuous coating of the yarn was added to an existing pilot-plant-sized CVD furnace. The system is capable of temperatures up to 2000°C, pressures as low as 0.05-0.1 Torr during CVD, and yarn speeds up to 10 feet per minute.

For continuous coating, a 500-meter spool of Nicalon NL 202 was wound onto the feed spool of the coater, and the end of the yarn was drawn through the 6 in. diameter, 12 in. long deposition chamber and attached to the collecting spool. The feed and collecting components of the CVD furnace were then sealed with transparent vacuum covers and the furnace evacuated and heated. The coatings were obtained by reacting boron trichloride and ammonia gases inside the deposition chamber. Typically, the yarns were fed once through the deposition chamber at about two feet per minute. Most of the coatings were made at around 1080°C to minimize the thermal degradation of the Nicalon. Pressures were typically below 1 Torr.

The PBN-coated yarns were characterized by weight per unit length, tensile strength and modulus, scanning electron microscopy (SEM), and scanning Auger microscopy (SAM). The PBN coating thicknesses ranged from 0.1-0.7 microns depending on coating conditions, and the coatings were fairly uniform along the length of the yarn and between fiber bundles but more variable around the circumference of the filaments. Grooved pulleys were used in an attempt to spread the filaments for more uniform coating. Strand tensile strengths of the PBN-coated Nicalon ranged from 342-390 ksi, which is quite close to the range of strengths obtained on the as-received Nicalon.

The PBN-coated Nicalon showed good resistance to oxidation in air at temperatures up to 800°C, but little or no improvement was observed after air oxidation at 1000°C. PBN oxidation data indicate that most of a 0.1-0.2 micron thick coating can be converted into boric oxide after a 30-minute heat treatment in air at 1000°C.

Spools of Nicalon yarn with 0.1-0.2 micron thick PBN coatings were woven into cloth at Fabric Development, Inc., Quakertown, Pennsylvania. The PBN-coated Nicalon was easier to handle than air-oxidized or vacuum heat-treated Nicalon, but the PBN coating was not entirely satisfactory as a 'high-temperature' sizing. To control loose filaments during weaving into cloth, the PBN-coated Nicalon was wrapped with 30-denier rayon. A 90-inch length of 13 in. wide plain-weave cloth and a 108 in. length of 13 in. wide 12-satin harness-weave cloth were prepared from the PBN-coated Nicalon yarn. This required 12 spools (~5,600 meters) of PBN-coated yarn. Another three spools were used to make the 500 meters of sewing thread needed for sewing together the three types of insulation panels required in this program. To facilitate handling, the Nicalon yarn used to make the sewing thread was also cross-wrapped with 30-denier rayon.

Several 13 in. square pieces of Nicalon cloth were coated with PBN in a batch process in a factory-sized deposition furnace. Temperature, pressure, and time were 1080°C, 0.2 Torr, and 4 minutes, respectively. These PBN coatings averaged about 0.3 microns thick, but the coating thickness was more variable than that achieved by continuous yarn coating.

Samples of cloth made from the PBN-coated Nicalon yarn were sewn into thermal insulation panels at Hi-Temp Insulation, Inc., Camarillo, California. The performance of these panels will be compared with that of panels made using uncoated Nicalon.

The results of the present study show that the PBN coatings on Nicalon need to be improved in order to maximize benefits as a high-temperature sizing and for oxidation protection. Thicker coatings, on the order of 0.5 microns and/or coatings with a more oxidation resistant outer coating, such as SiC, will be required to improve the air oxidation resistance. An alloyed PBN coating and/or more moisture stable outer coating may also be needed to improve the resistance of PBN to reaction with atmospheric moisture.

CONCLUSIONS

1. Nicalon NL 202 silicon carbide yarn is susceptible to degradation from heat treatment in air or oxygen at above 800°C because of oxidation of free carbon within the filaments.

2. Continuous coating of Nicalon NL 202 yarn with PBN at 1100°C and 0.1 Torr or less yields PBN coatings which are uniform along the yarn length and between fiber bundles. Coating thickness around the circumference of individual fibers is more variable.

3. PBN coatings up to 0.7 microns thick can be deposited on Nicalon yarn at 1100°C without causing loss of room-temperature strength.

4. PBN coatings 0.1-0.2 microns thick provide improved oxidation resistance in air at temperatures up to 800°C. The coatings provide little or no improvement in oxidation resistance after air oxidation at 1000°C because of conversion to boric oxide.

5. PBN coatings made at temperatures below about 1300°C are moisture sensitive and can deteriorate with exposure to moist air.

6. Thicker PBN coatings and coatings with a more oxidation resistant outer coating, such as SiC, will be required to improve the air oxidation resistance of Nicalon at 1000°C.

7. Batch PBN coatings on Nicalon cloth are more variable in thickness than those obtained by continuous coating.

8. The PBN coating on Nicalon yarn is not entirely satisfactory as a 'high-temperature' sizing. It is necessary to cross wrap the PBN-coated Nicalon with small-denier rayon or some similar material to facilitate weaving cloth and sewing of insulation panels. The cross-wrap material can then be burned away.

RECOMMENDATION

1. In order to make Nicalon more resistant to oxidation at 1000°C, thicker PBN coatings, alloyed PBN coatings, or PBN coatings which are coated over with more oxidation resistant materials, such as SiC, should be explored.

INTRODUCTION

The emerging technology of ceramic textiles presents an attractive opportunity to improve thermal management techniques for aerospace vehicles. Work at NASA Ames Research Center and elsewhere[1] has shown that thermal conductivity of appropriately constructed multilayer ceramic textile insulation can be considerably lower than that of monolithic low-density ceramic tiles. Spectral emissivity characteristics of ceramic textile quilts can also be superior to those of tiles because multilayer quilts have highly anisotropic properties.

Experience with ceramic textile insulation has also revealed limitations, removal of which would permit greatly improved performance. Many ceramic fibers undergo thermal and mechanical degradation at temperatures of interest. Aerodynamic forces tend to fray fibers and degrade textile structures so that protective coatings matched to textile requirements can improve performance.[2] For some applications and constructions, it is desirable to select a fiber exhibiting high thermal emissivity, i.e., a dark fiber rather than a light one. In such a case, the spectral emissivity of the protective coating must be appropriately machined with that of the substrate.

Because its properties bear on all the issues just mentioned, silicon carbide is a material of exceptional interest for aerospace textiles. This hard, refractory ceramic possesses excellent resistance to oxidation and exhibits high values of spectral emissivity (>0.7 at all temperatures) and of thermal conductivity (40 W/mK at 1200°C.[3] It is commercially available as continuous fiber yarn spun from a polymer precursor under the trade name Nicalon™.[4,5] Nicalon silicon carbide fiber exhibits tensile strengths exceeding 2500 MPa (360,000 psi) and Young's modulus approximately 200 GPa (29×10^{-6} psi) in pristine form.

The excellent mechanical properties of Nicalon can be degraded by surface damage induced by normal textile operations, such as twisting and weaving,[6] and also by exposure to temperatures in excess of 800°C, especially in air.

Although the marked sensitivity of silicon carbide yarn to handling can be ameliorated in normal tensile operations by using textile sizes and finishes, the problem recurs if multilayer quilts containing silicon carbide fibers are used in the aerospace environment. Aerodynamic forces can be expected to abrade fibers, especially at crossover points of woven fabrics, and fray the textiles. Since reentry conditions, in particular, are characterized by high temperatures, conventional sizes offer no protection. A protective lubricant coating of each individual filament which can withstand high temperatures and aerodynamic environments is required.

Another problem specific to Nicalon fiber is that its mechanical properties are degraded, and its composition is altered by exposure to elevated temperatures, somewhat more readily in air than in an inert atmosphere.[6-9] As-manufactured Nicalon consists of beta-SiC with about 30Å particle size with a substantial concentration of free carbon of very small particle size, and an amorphous matrix of composition SiO_xC_y.[10] Recently, a molecular composition of $SiC:0.38SiO_{1.2}C_{0.4}:0.54C$ was determined from XPS analyses.[11] Air oxidation will burn away the sizing, and especially at temperatures above 800°C will react with free carbon to leave surface pits which lower the strength of the fibers.[10] Therefore, coatings that are oxidation resistant as well as lubricious are expected to be of value in maximizing the strength of Nicalon in a high-temperature aerodynamic environment.

In this project, boron nitride was chosen as the protective coating for silicon carbide yarn. Boron nitride is a hexagonal, lamellar material that has been used as a lubricant and friction modifier at elevated temperatures.[12] It resists oxidation to temperatures at least as high as 1000°C. Also, thin layers of BN are transparent so that emissivity at elevated temperatures is a good match to that of silicon carbide. A pyrolytic boron nitride coating should prevent air oxidation of the free carbon in the Nicalon at least until the temperature and time are sufficiently severe to convert all of the PBN to boric oxide. The PBN should, therefore, prevent formation of surface pits that cause strength loss and, also, by preventing loss of free carbon should delay the crystal growth of SiC which would cause embrittlement and strength loss to the Nicalon. The value of a PBN coating will, therefore, depend on how long it will survive in an air oxidizing environment at 1000°C.

The goal of this program is to coat ceramic yarns and cloths, in particular, Nicalon Type NLM 202 silicon carbide yarns, with pyrolytic boron nitride (PBN) so that thermal insulation panels made from these yarns exhibit enhanced resistance to physical damage in an aerodynamic or aeroacoustic environment. The Statement of Work for this program is given in NASA Ames RFP2-33431(LMV), pp. C-2 to C-7, December, 1987. The tasks and approaches, proposed staff assignment, and description of facilities and equipment are given in Union Carbide's Technical Proposal, Parts A, B, and C, dated August 3, 1988.

OUTLINE OF PROGRAM TASKS

The Work Statement for this program requires Nicalon NL 202 silicon carbide yarn to be coated with pyrolytic boron nitride (PBN), woven into cloth of specified construction, and the cloths fabricated into insulating panels using uncoated and also PBN-coated sewing threads constructed of yarn. The principal tasks required to complete this project are as follows:

1. Testing the as-received Nicalon yarn (tensile strength, modulus, density).

2. Heat-treatment of Nicalon yarn and testing the heat-treated yarn.

3. Construction of improved continuous yarn coater (for pilot plant furnace).

4. Establishing optimum conditions for coating Nicalon with PBN in the continuous coater (temperature, yarn speed, gas flow rates, etc.)

5. Characterization of the PBN-coated Nicalon (coating thickness, tensile strength, SEM and EDX, scanning Auger microscopy).

6. Preparation of sufficient quantities of PBN-coated Nicalon to yield the cloth needed for fabrication of thermal insulation panels.

7. Preparation of cloth samples from as-received and from PBN-coated Nicalon yarn (by Fabric Development, Inc.)

8. PBN batch coatings on Nicalon cloth samples (in factory-size furnace).

9. Fabrication of insulation panels (by Hi-Temp Insulation Company).

10. Final report.

The specific tasks required to complete the program steps as outlined in the Work Statement Proposal are given in Table I.

<u>Task 03, Subtask 1</u>

Ten spools of Nicalon Type NLM 202 (now referred to as NL 202) and one square meter of Nicalon 202 cloth were purchased from Dow Corning in September 1989. Type NL 202 is ceramic grade Nicalon having a density of 2.55 g/cc and containing 500 filaments totalling 1,800 denier. Each spool contains 100 grams (500 m) of yarn. The sizing is polyvinylacetate.

Tasks 03, Sub-Tasks 1 and 2 were performed first. Using lab-scale yarn winding equipment, we wound 80-foot samples of yarn on separate spools from a single spool of Nicalon NL 202. Strand tests were performed in our Fiber Test Laboratory using ASTM method D-4018. In this test, the yarn is impregnated with Epoxy which is then cured in an oven. For each of the twenty spool samples, 11-13 individual test specimens were prepared for measurement of tensile strength and modulus. The density of each sample was determined by immersion in orthodichlorobenzene.

Task 03, Subtask 2

Ten 160-foot samples of yarn were wound off a second spool of Nicalon NL 202 and five of the samples were heat-cleaned using the procedure recommended in Page C-6 of RFP2-33431(LMV), i.e., the samples were placed in an air recirculating furnace and heated from room temperature to 550°C in 2 hours, held at 550°C for 3 hours, and cooled to 30°C in 16 hours. Such heat-treatments caused a 1.1% weight loss which is due to removal of the polyvinylacetate sizing.

Samples of the heat-cleaned Nicalon yarn were wound on graphite spools and heated in the graphite induction furnace that was used to coat the yarns with PBN. The purpose of these experiments was to determine the effects of heat-treatments on the degradation of Nicalon yarn properties to establish an upper temperature limit for the PBN coating experiments. All samples were heated in an argon atmosphere at approximately 0.5 Torr pressure. Samples were heated for one-minute periods at 1000°C, 1100°C, 1200°C, and 1350°C. Heat-up rates were typically 20-30°C per minute near the final temperature, and cool-down rates were 10-15°C per minute. One sample was heated at 1100°C for 10 minutes, with similar heat-up and cool-down rates as for the other samples.

The heat-treated Nicalon NL 202 yarns were strand tested in the Fiber Test Laboratory using the same procedures as for the as-received Nicalon.

Task 03, Subtask 3

A pilot-plant-sized inductively heated vacuum furnace (Figure 1) for CVD, having a hot zone 6 in. in diameter by 12 in. long, was available at UCAR Carbon Company Inc., Parma Center, for coating SiC yarn with PBN. However, the equipment installed in 1989 for continuous yarn coating in this furnace involved feeding the yarn into and out of the CVD furnace over graphite pulleys located inside the hot zone. We found that a buildup of deposits on the pulleys caused excessive friction and damage to the yarn when long lengths were coated. Therefore, the yarn-coating arrangement was redesigned and rebuilt so that we were able to feed the yarn in a single pass through the hot zone, with the feed and collection spools, windup and tensioning apparatus all located outside the furnace (but not outside the vacuum system). The improved equipment is shown schematically in Figure 2 and the yarn feeding and collecting equipment is shown in more detail in Figures 3 and 4. The parts were vacuum tight, and only minor problems were encountered in feeding and collecting the yarn.

A typical yarn coating experiment was carried out as follows. A 500 m spool of Nicalon was wound onto the graphite feed spool as shown in Figure 3(a) and then strung through the furnace over the appropriate pulleys and attached to the collecting spool as shown in Figure 3(b). The yarn was then run for a few minutes through the furnace to make sure all the machinery was working properly. The plexiglass covers were then installed, the system was pumped down, and was then induction heated to the desired coating temperature. The yarn was fed through the furnace at a slow speed (1 ft/min) during the later stages of heat up. The reactant gases (BCl_3 and NH_3) were then fed through the water-cooled injector to start PBN deposition and the yarn speed adjusted to achieve the desired coating thickness.

At the end of the run, the gases were shut off and a sample of yarn was run at the deposition temperature but with no coating so that the weight per unit length of this vacuum heat-treated sample could be compared with that of the PBN-coated material. Based on the measured yarn weight gain/unit length and an assumed coating density of 1.40 g/cc, the estimated coating thicknesses on Nicalon were about 0.1 micron, assuming that all filaments were coated identically. This was calculated as follows:

Typical weight of 1 meter of Nicalon NL 202 = 0.215 grams.

Therefore, weight of 9,000 meters = 1,935 grams = 1,935 denier.

One meter of single filament = 0.215/500 = 430×10^{-6} grams.

Density = 2.50 g/cc. Volume of one meter single filament = 172×10^{-6} cc
= 172×10^{6} microns^3.

$$\text{Cross-sectional area} = \frac{172 \times 10^6 \text{ microns}^3}{1\text{m} = 10^6 \text{ microns}} = 172 \text{ microns}^2 = \frac{\pi}{4}\ d^2.$$

Therefore, average diameter = 14.8 microns (round filaments).

Assume PBN coating at 1100°C has a density of 1.40 g/cc. Then we can calculate coating thickness from yarn weight gain/unit length as follows, assuming all filaments are coated equally.

Weight Gain mg/ft	Weight Gain mg/Meter	Volume Increase Micron^3/Meter	$d_o^2 - d_i^2$ Micron^2	Thickness Microns
1.0	3.28	2.343×10^6	2.983	0.05
5.0	16.4	11.72×10^6	14.915	0.25
10.0	32.8	23.43×10^6	29.83	0.49

Thus, for a PBN coating density of 1.40 g/cc, the coating thickness in microns is 0.05 x the yarn weight gain in milligrams per foot.

Task 03, Subtask 4

Nicalon NL-202 yarns were coated with PBN at various temperatures and feed rates, etc., to yield PBN-coated yarns with differing coating thicknesses. Some of these coated yarns were examined for thickness uniformity and composition using scanning electron microscopy (SEM) and scanning Auger microscopy (SAM). Most of this testing was done at United Technologies Research Center using well-established procedures. Samples of PBN-coated Nicalon from two lots were sent to an outside laboratory for measurement of strand strength, modulus, and strain-to-failure using the same techniques as those used for the as-received and heat-treated Nicalon in Task 03, Subtask 1. Several samples were sent to NASA Ames Laboratory for tests of the effects of air oxidation up to 1000°C on the short-strand tensile strength.

Task 03, Subtask 5

Fifteen spools (7,500 meters) of PBN-coated Nicalon were prepared and sent to Fabric Development for preparing the plain-weave cloth, harness-weave cloth, and sewing thread needed to make the insulation panels.

Tasks 04 and 06

The spools of PBN-coated Nicalon NL 202 yarn from our continuous CVD process were woven into a 90-inch length of 13 in. wide plain-weave cloth (18 x 18 yarns per inch = 709 x 709 per meter) and into a 108-inch length of 13 in. wide 12-satin harness-weave cloth (26 x 26 yarns per inch = 1,024 x 1,024 per meter) at Fabric Development. After some small-scale testing, Fabric Development concluded that a sizing was needed to control loose filaments during weaving. However, the polyvinyl acetate sizing was avoided because of the water base which we felt could cause degradation of the PBN coating. Instead, the loose filaments were controlled by wrapping the yarn in 30-denier rayon thread which can be burned away after the insulation panels are fabricated. The cross-wrapping with rayon greatly facilitated the weaving operation. Twelve spools (~5,000 meters) of PBN-coated Nicalon were used in making the two kinds of cloth.

The cloth samples prepared by Fabric Development were sent to Hi-Temp Insulation Company for fabrication into Types A, B, and C panels according to Tasks 08, 09, and 10 of the Proposal Work Statement.

Task 05

Fabric Development converted the PBN-coated Nicalon NL 202 into a sewing thread similar to Astroquartz Q-24. Approximately 550 meters of sewing thread were prepared with a weight per unit length twice that of the Nicalon yarn. The thread was then cross-wrapped with rayon to facilitate stitching the insulation panels by Hi-Temp Insulation Company. About three spools of PBN-coated Nicalon (~1,400 meters) were needed to prepare this thread, which included a small sample that was delivered directly to NASA Ames.

Task 07

In work on this statement, Fabric Development, Inc., was able to prepare plain-weave cloth from Nicalon NL 202 yarn to a density of 305 g/m^2 compared with 280 g/m^2 for plain-weave cloth available commercially from Dow Corning. Test samples of the commercially available Nicalon cloth were batch coated at 1050°C and 1150°C in a factory-sized CVD furnace at Union Carbide's Lakewood facility. Cloth samples were mounted flat in graphite racks each holding a 12 in. x 5 in. section of cloth, and the racks were rotated about an axis along the long dimension during the deposition. The 1050°C and 1150°C coatings were deposited for five minutes and three minutes, respectively. Test samples were examined using SEM and SAM, and ultimate tensile strengths were determined on filaments taken from both deposits.

To coat larger samples of cloth, graphite frames were machined large enough to hold a 33 cm wide piece of Nicalon flat while batch coating. This frame was also rotated around its long axis. Four samples of Nicalon NL 202 cloth were PBN-coated at a deposition temperature of 1080°C for four minutes. Two 13-in. square samples of commercial Nicalon plain-weave cloth (16 x 16 yarns per inch, 277 g/m^2) and two 12 in. square samples of denser plain-weave cloth (18 x 18 yarns per inch, 312 g/m^2) prepared by Fabric Development, Inc., were coated with PBN in four separate runs. Three of these larger PBN-coated Nicalon cloth samples were sent to NASA Ames for evaluation.

Tasks 08, 09, and 10

The PBN-coated cloth prepared as described in Tasks 04 and 06 and the sewing thread prepared as described in Task 05 were sent to Hi-Temp Insulation, Inc., Camarillo, California, where they were sewn into insulation panels Types A, B, and C as prescribed in the Work Statement. The completed panels were sent directly to NASA Ames for evaluation.

EXPERIMENTAL RESULTS

Task 03, Subtask 1

The average properties of each sample of as-received Nicalon NL 202 yarn are given in Table II. The density (2.48-2.50 g/cc) is less than that reported by the manufacturer for ceramic grade Nicalon (2.55 g/cc), but the true densities may be about 1% higher than the measured values if the polyvinyl acetate sizing dissolved im the orthodichlorobenzene. The average tensile strength ranged from 353-402 ksi with standard deviations (N = 11-13) of 13-58 ksi. These strengths are 82-93% of the manufacturer's reported value of 430 ksi. The tensile modulus of 28-29 Msi is in very good agreement with the manufacturer's listed value of 28 Msi.

Complete test results for the as-received Nicalon yarn are given in the Appendix, Part I.

Task 03, Subtask 2

Test results for samples of heat-treated Nicalon NL 202 yarn are given in Table III. Complete test results are given in the Appendix, Part II. Heat treatments in the range of 1000-1200°C increased the density to 2.52-2.53 g/cc. The 1350°C treatment caused a larger increase in density, to 2.605 g/cc. One-minute heat treatments at 1000-1100°C caused just over 20% loss in tensile strength compared with the as-received Nicalon yarn. A ten-minute heating at 1100°C resulted in more than 35% loss in tensile strength. The one-minute heat treatments at 1200°C and 1350°C caused severe degradation of the Nicalon resulting in very low tensile strengths. The modulus of the heat-treated Nicalon yarns was 29-30 Msi, slightly higher than that of the as-received yarns (28-29 Msi).

Table III also shows the weight losses of the Nicalon yarns as a result of the heat treatments. These weight losses are due to the release of CO and SiO and are comparable to those reported for 12-hour heat treatments in flowing argon,[9] although the strength loss was much less in our heat-treated samples. The comparable weight losses in much shorter heating times in our experiments are probably due to the low pressure of argon used (0.5 Torr). However, the low-pressure heat treatment should give more realistic results because in order to obtain clean PBN deposits, it is necessary to use deposition pressures of 1 Torr or less. The strength degradation results suggest that the upper temperature limit for making PBN deposits on Nicalon should be set at about 1100°C.

Task 03, Subtask 3

In initial tests, PBN coatings were applied to Nicalon NL 202 yarn which was fed through the deposition chamber at about 2 feet per minute (30-second residence time). In these early trials, the reactant gases (BCl_3 and NH_3) were directed straight towards the moving yarn as shown in Figure 2. Visual inspection indicated variations in coating thickness when the yarn was coated this way. Visually improved coatings were obtained by using methods to prevent the gases from directly impinging on the yarn. Using these methods, PBN-coated Nicalon was prepared at temperatures in the range 1075-1200°C, pressures of 40-80 microns, yarn speeds of 0.4-5.0 feet per minute (coating times 155-12 seconds), and at NH_3:BCl_3 ratios of 2.0-3.6. Results are given in Table IV. A "muzzled" injector was used in Runs 9024-9029 as a first method to prevent direct impingement of the gases onto the yarn. The muzzle attached to the end of the injector consisted of a tapered PBN crucible in which holes were drilled through the lower wall but not the bottom. Later, the muzzle was removed, and a 30 mm square graphite deflector plate was mounted between the injector and the yarn in Runs 9030-9036.

As stated earlier, yarn weight gains due to coating with PBN were determined by comparing the weight of a given length of coated yarn with a sample of vacuum heat-treated yarn obtained at the beginning and/or end of each run by shutting off the reactant gases but holding the furnace at temperature and continuing to feed the yarn. Based on the assumptions given earlier, the estimated coating thicknesses varied from about 0.1 micron at the highest yarn speed (5.0 ft/min in part of Run 9028) to about 0.7 micron at the lowest speed (0.4 ft/min in Run 9035). The yarns with the heaviest coatings (9035 and 9036B) were noticeably stiffer than the others.

In Run 9029, a sample of 600-denier Nicalon was successfully coated at 1075°C by minimizing all sources of friction from feed to windup. For this run, it was necessary to remove the brake on the feed spool to prevent yarn breakage. The brake was found to be unnecessary for coating the 1,800-denier NL 202 yarn as well.

Removal of the polyvinyl acetate sizing from the Nicalon NL 202 by heating in flowing air at 600°C for 30-60 seconds greatly increased the number of loose filaments. Such 'heat-cleaned' yarn was used in only two coating runs (9033 and 9034). Although both coating runs were successful in that there was no yarn breakage during processing, the yarn strength must have been reduced in proportion to the number of loose filaments. The vacuum heat-treatment that occurred as the yarn was fed into the coating furnace appeared to remove the size before coating. The vacuum heat-treated yarn obtained by feeding the Nicalon through the furnace without coating looked very much like the air-oxidized yarn.

Some of the PBN-coated Nicalon yarns listed in Table III were examined for thickness uniformity and composition using SEM and SAM. Results on Samples 9024-1 and 9024-2 indicated fairly uniform coatings of near-stoichiometric BN with a thickness of about 0.1 micron, less than that estimated from the weight gain. Data for coated yarn Sample 9028 show stoichiometric BN with a thickness somewhat less than 0.1 micron (see Figure 5). Typical thicknesses determined by SAM were in quite good agreement with the estimates based on yarn weight uptake for material from Runs 9032, 9034, and 9035. The 1200°C coating (9032) consisted of stoichiometric BN with relatively small concentrations of oxygen and carbon (see Figure 6), but the tensile strength of the coated fiber was poor (187 ksi) because of the degradation caused by the high-deposition temperature. This sample showed many rough and peeled-away coating areas. Coating 9034, made on Nicalon in which the polyvinyl acetate sizing was first removed by air oxidation, also consisted of stoichiometric BN with small amounts of oxygen and carbon (see Figure 7).

The 0.7 micron thick coating from Run 9035 was relatively smooth (Figure 8) but was somewhat boron rich with very little carbon but relatively high oxygen (20-30 at%). The average tensile strength after coating was very high (345 ksi) indicating that the coating did not degrade the Nicalon.

Coating thicknesses by SAM for Samples 9036A and 9036B were higher than the values estimated from yarn weight uptake. These coatings were found to be very rich in boron, but oxygen and carbon contents were relatively low. This result shows that a $NH_3:BCl_3$ ratio of at least 3.0 may be needed to produce stoichiometric PBN coatings at 1100°C. The boron-rich coatings were rougher than the stoichiometric PBN coatings.

Samples of PBN-coated Nicalon from Runs 9028, 9029, 9034, and 9036 and two PBN-coated Nicalon cloth samples were sent to the NASA Ames Laboratory for examination. Also included were samples of as-received Nicalon NL 202 yarn, 600-denier yarn, and air-oxidized and vacuum heat-treated samples of the NL 202. The results of the testing done at NASA Ames is summarized in the following.

Figure 9 compares the short-strand tensile strength of as-received Nicalon NL 202 yarn with that of several PBN-coated samples (9028, 9034, and 9036A) after 30 minutes of oxidation in air at temperatures up to 800°C. At the lower oxidation temperatures, the as-received yarn was about 20% stronger than the coated yarn. After oxidation at 600-800°C, the coated yarn showed tensile strengths equal to or sometimes greater than that of the as-received Nicalon. However, the strength of a coated yarn sample fell almost to zero following air oxidation at 1000°C.

Similar results were obtained for PBN-coated 600-denier Nicalon yarn, as shown in Figure 10. In this case, the PBN-coated Nicalon showed superior strength after the 600°C and 700°C oxidations, but yarn strengths decreased to low values following oxidations at 800°C and 1000°C.

The tensile strength results for PBN-coated NL 202 1,800-denier yarns following oxidation tests at up to 800°C were considered sufficiently good to proceed with the program to prepare several spools of PBN-coated Nicalon under conditions similar to those of Lots 9028 or 9034 so that the amounts required for cloth and insulation panel production could be realized.

<u>Task 03, Subtask 4</u>

Several 500 meter spools of NL 202 were coated with PBN under conditions nearly identical to those used in Runs 9028, 9034, and 9036A. Run conditions and results are given in Table V. Run 9110 was divided into two parts because of slippage of the take-up spool drive coupling after about 30%

of the yarn had been run through the furnace. Complete spools were successfully processed in Runs 9111-9113. Coated sample weight gains compared with vacuum heat-treated Nicalon indicated average PBN coating thicknesses in the desired range of 0.1-0.2 microns.

Five samples each of PBN-coated Nicalon from Runs 9028 and 9110 were tested according to ASTM Method D-4018 as was used in testing the as-received and heat-treated yarns. For each sample, 10-12 individual test specimens were prepared for measurement of tensile strength and modulus. Results of the testing are given in Table VI. Complete test data are given in the Appendix, Part III. The average tensile strengths of coated yarn from Lots 9028 and 9110 were 342 ksi and 390 ksi, respectively. These strengths are 15-30% greater than those of the 1000-1100°C heated Nicalon NL 202 and are close to the upper and lower limits of strengths measured on the as-received Nicalon (see Table II for comparison).

Task 03, Subtask 5

Thirteen more 500 meter spools of Nicalon NL 202 yarn were coated with PBN at 1080°C in the pilot plant continuous coater to provide the quantities needed for preparation of both plain-weave and harness-weave cloth for subsequent fabrication into insulation panels. Experimental details and results are given in Table VII. PBN coating thicknesses for all lots were within the specified range of 0.1-0.2 microns based on yarn weight uptake. Run times varied from 720-848 minutes. Reactant gas flow rates were increased by about 10% after Run 9118. This change yielded about the same coating thickness with about a 10% decrease in run time. Yarn weight change during coating varied from a low of 1.8% loss in Run 9118 to 3.2% gain in Run 9128 although most values were less than ±1%.

After collecting samples for weight uptake and other measurements and characterization, the remaining coated yarn was rewound onto a cardboard spool for sending to Fabric Development for cloth fabrication. The coated yarn lengths ranged from 460-480 meters. Sample 9114 was shorter because the yarn broke after about 85% of the full spool had passed through the continuous coater. Thirteen spools of PBN-coated Nicalon were sent to Fabric Development to make the plain-weave cloth, harness-weave cloth, and sewing thread. Three remaining coated spools were retained for any additional testing that may be required. One of these was Sample 9129, the first run in which the yarn was passed through the furnace three times and in which the desired coating thickness was obtained at three times the yarn speed used in the single-pass experiments. Some testing must be done before this yarn can be considered equivalent to the slower speed single-pass coated yarn because in the three-pass arrangement the yarn leaves and reenters the deposition zone after each pass.

Task 07

The thickness and composition of the 1050°C and 1150°C PBN coatings on Nicalon NL 202 cloth, as determined using SEM and SAM showed large variations in coating thickness between top and bottom of the fiber weave. SAM depth profiles showed that the top of the weave in the 1050°C deposition is coated with about 7,000Å of PBN with an average composition of 44% B, 37% N, 17% O, and 2% C except near the coating surface where the carbon content is higher and the oxygen is lower (see Figure 11). The underside of the weave exhibited a coating thickness of 1,000-3,000Å and more variable composition (see Figure 12). The fibers coated at 1150°C showed a coating thickness of 7,000Å on the top of the fiber weave and less than 1,000Å on the underside. The average composition of the thicker coating was 40% B, 35% N, 20% C, and 5% O. The 100°C increase in deposition temperature thus appears to yield a PBN with less oxygen but more carbon impurity. The ultimate tensile strengths of filaments taken from the 1050°C and 1150°C deposits were 260 ksi and 250 ksi, respectively.

Based on weight uptake, the average PBN coating thicknesses in the 1050°C and 1150°C deposits were 0.15 micron and 0.25 micron, respectively, if each filament in the cloth was coated equally.

Details of coating conditions for PBN deposits on the large cloth samples are given in Table VIII. Based on weight of strands taken from the edge of cloth samples from Runs 6955 and 6964, an average coating thickness of about 0.3 microns was calculated if it is assumed that all filaments were coated equally.

DISCUSSION

The results of continuous coating of Nicalon NL 202 yarn with PBN using the equipment and procedures described in this report show that the PBN coatings were fairly uniform along the yarn length and between fiber bundles, but more variable around the circumference of the individual fibers. The PBN-coated yarn had fewer loose and broken filaments than either the air oxidized or vacuum-heat-treated "heat-cleaned" yarn. However, the PBN was still not entirely satisfactory as a "high-temperature" sizing. To facilitate weaving of cloth and sewing of insulation panels, it was necessary to cross-wrap the PBN-coated Nicalon with small-denier rayon or some similar material to control loose filaments. The cross-wrapped material is then burned away after the panels are prepared.

Although the Nicalon NL 202 exhibited strength losses on the order of 20% following short-time heat treatments to 1000-1100°C, the Nicalon which was coated with PBN at 1100°C showed virtually no room-temperature strength loss. This was observed for coating thicknesses in the range 0.1-0.7 microns. The PBN-coated Nicalon also showed good properties after air oxidation up to 800°C for 30-minute periods. However, the 0.1-0.2 micron thick coatings appeared to be ineffective following air oxidation treatments at 800-1000°C.

The loss of coating effectiveness following oxidation treatments in air at 800-1000°C is probably due to oxidation of the PBN itself. A paper by Lavrenko and Alexeev[13] quotes a linear oxidation rate of 0.15×10^{-7} g/m^2/s for PBN in pure oxygen at 900°C. For PBN of density 1.4 g/cc and 30 minutes of oxidation, this corresponds to a 0.2 micron layer which is equal to or greater than the PBN coating thickness. The oxidation rates in air may be less than those reported, but the low-temperature glassy-PBN deposits may be more readily oxidized than the high-temperature more crystalline deposits described in the Lavrenko and Alexeev paper.

The loss of coating effectiveness following high-temperature heat treatments in air may also be due to the reaction of low-temperature glassy PBN (typical of a 1100°C PBN deposit) with atmospheric moisture. The coated yarns were stored in a desiccator to minimize exposure to atmospheric moisture, but were subsequently exposed to atmospheric moisture during weaving and then stored in plastic wrap without any desiccant. Low-temperature glassy PBN can react with atmospheric moisture according to the reaction:

$$BN + 3H_2O \rightarrow H_3BO_3 + NH_3.$$

This reaction proceeds with a free-energy change of -18 Kcal/mole at room temperature, and the free energy change is negative at temperatures up to 190°C. As an example, a 1300°C PBN deposit showed a weight gain of 35% following two days exposure to flowing saturated air at room temperature.[14] More recently, Matsuda[15] reported on the moisture stability of low-temperature PBN deposits and attributed their reactivity to unstable species or sites in the PBN which were converted to ammonium borate hydrates by reaction with moisture. The presence of these reaction products on the surface of the PBN will cause loss of coating material when it is heated in air (or other environments) to 800°C or higher.

The effects of air oxidation at above 800°C on Nicalon yarns with thicker PBN coatings, up to 0.7 micron, and on PBN coatings which are overcoated with more oxidation-resistant materials, such as SiC or Si_3N_4, should be determined because the results should give guidance towards developing a better coated Nicalon for application in an aerodynamic environment. Thicker PBN coatings made by us have been used to prepare Nicalon-reinforced ceramic composites with good results[16] showing that moisture attack is not a problem for coated yarns exposed to the environment for several hours to several days.

ACKNOWLEDGMENTS

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A. W. Moore
January 27, 1992
6989d

Table I

Pyrolytic Boron Nitride Coatings on Ceramic Yarns and Fabrication of Insulations

Task No.	Description of Work	Done By	Amount of Material	Date Completed
01	Make silicon carbide thread from Nicalon NL 202 SiC yarn.	Fabric Development, Inc.	20 Feet	June, 1991
02	Provide sample of Nicalon NL 202 yarn to NASA Ames.	Purchased from Dow Corning	50 Feet	November, 1990
03-1	Determine baseline properties of NL 202 yarn.	Union Carbide Corporation (UCC) Parma	One Spool 500 Meters 20 Samples	January, 1990
03-2	Determine properties of heat-treated Nicalon NL 202 yarn.	UCC Parma	Half Spool 250 Meters	January, 1990
03-3	Coating NL 202 with PBN using different temperatures, residence times, etc.	UCC Parma	Three Spools 1,500 Meters	October, 1990
03-4	Evaluation of coated yarns from Task 03-3.	UCC Parma, NASA Ames, and Other Labs		January, 1991
03-5	Prepare enough PBN-coated NL 202 to meet requirements of Tasks 03, 04, 05, 06, 08, 09, and 10.	UCC Parma	15 Spools 7,500 Meters	May, 1991
04	Fabricate 12-satin harness-weave cloth from PBN-coated NL 202.	Fabric Development, Inc.	10 Sq. Ft.	May, 1991
05	Fabricate sewing thread from PBN-coated Nicalon.	Fabric Development, Inc.	500 Meters	July, 1991
06	Fabricate plain-weave cloth from PBN-coated Nicalon.	Fabric Development, Inc.	8 Sq. Ft.	May, 1991
07	Batch coating of plain-weave Nicalon NL 202 cloth.	UCC Lakewood	3 Sq. Ft.	March, 1991
08	Construction of Type A insulation panels.	Hi-Temp Insulation, Inc.	Four Panels Ea. 12 In. Sq.	September, 1991
09	Construction of Type B insulation panels.	Hi-Temp Insulation, Inc.	Four Panels Ea. 12 In. Sq.	September, 1991
10	Construction of Type C insulation panels.	Hi-Temp Insulation, Inc.	Four Panels Ea. 12 In. Sq.	September, 1991
11	Quarterly Progress Reports	UCC Parma	Six	May, 1991
12	Final Technical Report	UCC Parma		

Table II

Strand Tensile Strength and Modulus of
As-Received Nicalon NL-202 Yarn Spool #1

Sample	Density g/cc	Tensile Strength Ksi				Modulus Msi	
		Ave	Min	Max	S.D.	Ave	S.D.
1	2.494	379	323	419	29	28.4	0.4
2	2.498	377	321	410	31	29.1	0.2
3	2.494	386	327	417	29	29.0	0.3
4	2.494	396	333	431	36	28.9	0.4
5	2.494	399	369	430	20	28.6	0.4
6	2.491	386	327	466	44	28.3	0.4
7	2.494	402	385	423	13	28.3	0.3
8	2.480	386	338	426	34	28.4	0.3
9	2.483	402	360	439	24	28.1	0.5
10	2.476	383	276	446	49	28.2	0.5
11	2.483	363	271	404	37	28.3	0.3
12	2.486	378	332	415	29	28.0	0.3
13	2.468	393	313	436	42	28.2	0.5
14	2.490	378	318	427	28	28.3	0.3
15	2.487	370	297	409	31	28.7	0.4
16	2.486	364	215	431	58	28.4	0.3
17	2.483	390	361	419	22	27.9	0.6
18	2.486	353	305	401	30	28.5	0.5
19	2.487	369	281	436	49	28.5	1.1
20	2.479	356	296	422	43	27.8	0.5

N = 13 for Sample 15

N = 11 for Sample 17

N = 12 for all other Samples

Sample 1 is outermost part of as-received Nicalon Spool #1

Sample 20 is innermost part of as-received Nicalon Spool #1

Table III

Strand Tensile Strength and Modulus of
Heat-Treated Nicalon NL-202 Yarn

Sample	Heat Treatment Temperature °C	Time Min	Wt Loss %	Density g/cc	Tensile Strength, Ksi				Modulus Msi	
					Ave	Min	Max	S.D.	Ave	S.D.
3	1000	1	4	2.531	296	213	353	38	28.8	0.5
2	1100	1	4	2.523	293	196	363	45	29.1	0.8
7	1100	10	9	2.530	238	184	272	24	30.4	0.8
4	1200	1	11	2.523	108	41	165	46	29.4	1.0
5	1350	1	9	2.605	178	118	201	29	30.1	0.6

Sample 1 is outermost part of as-received Nicalon Spool #2

Sample 10 is innermost part of as-received Nicalon Spool #2

Sample 3 is 3rd from outside, etc.

Table IV

PBN Coatings on Nicalon Yarn That Was Continuously Fed Through the CVD Furnace

Run Number	Deposition Temp., °C	Pressure Microns	Time Minutes	Yarn Speed Ft/Min	Approximate Coating Time Seconds	Gas Flow Rates, lpm BCl_3	Gas Flow Rates, lpm NH_3	Estimated Coating Thickness Microns(4)
9024-1	1075	48	120	2.0	30	0.55	1.95	0.2
9024-2	1140	48	120	2.0	30	0.55	1.95	0.3
9027	1075	60	120	2.0	30	0.45(1)	1.28(1)	0.3
9028-1	1075	45	230	2.0	30	0.42	1.53	0.2
9028-2	1075	35	300	2.0	30	0.42	1.53	0.2
9028-3	1075	43	20	3.2	19	0.42	1.53	0.15
9028-4	1075	43	20	5.0	12	0.42	1.53	0.1
9028-5	1075	43	45	2.0	30	0.42	1.53	0.2
9029(2)	1075	55	120	2.0	30	0.43	1.39	0.2
9030	1075	50	150	2.0	30	0.43	1.39	0.1
9031-1	1075	55	120	2.0	30	0.45	1.56	0.18
9031-2	1075	50	240	0.9	66	0.45	1.56	0.27
9031-3	1100	58	90	3.5	17	0.45	1.56	0.12
9032	1200	78	120	2.0	30	0.45	1.56	0.3
9033(3)	1075	75	120	2.0	30	0.45	1.56	0.15
9034(3)	1100	80	420	2.0	30	0.43	1.55	0.17
9035	1100	47	540	0.42	155	0.43	1.41	0.7
9036A	1100	79	180	2.0	30	0.69(1)	1.41	0.2
9036B	1100	79	330	0.75	80	0.69(1)	1.41	0.4

Notes:
(A) 'Muzzled' injector used in Runs 9024-9029.
(B) Graphite deflector plate between injector and yarn in Runs 9030-9036.
(C) All coatings on as-received 1800 denier Nicalon 202 except where stated otherwise.
(D) BCl_3 fed through outer (annular) tube and NH_3 fed through center tube of injector in Runs 9024-9030. BCl_3 through inner tube and NH_3 through outer tube in Runs 9031-9036.

(1) Estimated values.
(2) Coating on 600 denier Nicalon.
(3) Polyvinyl acetate sizing removed before coating by heating yarn in flowing air at 600°C for 30-60 seconds.
(4) Coating thicknesses estimated from weight-gain measurements assuming a density of 1.4 g/cc and identical coating of all the filaments in the yarn.

Table V

PBN Coatings on Nicalon SiC Yarns Using Continuous Coater

Run Number	Deposition Temp. °C	Pressure Microns	Time Minutes	Speed Ft/Min	Gas Flow Rate, ℓpm BCl_3	Gas Flow Rate, ℓpm NH_3	Estimated Coating Thickness, μ
9110A	1090	40	255	1.93	0.44	1.38	0.10
9110B	1080	35	480	1.95	0.44	1.38	0.10
9111	1080	37	815	1.85	0.44	1.35	0.10
9112	1080	38	870	1.80	0.44	1.40	0.12
9113	1080	35	830	1.90	0.44	1.36	0.15

NM - Not Measured

Table VI

Tensile Strength of PBN-Coated Nicalon NL 202 Yarns
(Strand Tests)

Samples	Density g/cc	Tensile Strength ksi	Tensile Strength S.D.	Modulus ksi	Modulus S.D.
9028-1	2.485	319.4	17.7	29.8	0.6
9028-2	2.516	328.9	20.4	30.4	0.2
9028-3	2.527	343.4	27.0	31.3	0.8
9028-4	2.504	244.9	28.6	31.2	0.6
9028-5	2.515	371.4	22.3	31.0	0.3
Averages	2.509	342		30.7	
9110-2	2.495	368.2	22.4	30.3	0.7
9110-3	2.495	377.7	18.7	30.0	0.4
9110-5	2.528	374.9	14.4	31.1	1.2
9110-9	2.528	406.1	33.4	30.4	0.4
9110-10	2.539	424.8	25.1	31.5	0.7
Averages	2.517	390		30.7	

Table VII

PBN Coatings on Nicalon NL 202 Yarn Using Continuous Coater

Run Number	Deposition Temp. °C	Pressure Microns	Run Time Minutes	Yarn Speed ft/min	Gas Flow Rate, ℓpm BCl_3	NH_3	Yarn Weight Change %	Estimated Avg. PBN Thickness Microns	Coated Yarn Weight Grams	Coated Yarn Length Meters
9114	1080	40	688	1.98	0.48	1.39	-0.7	0.16	89.8	409(1)
9115	1080	38	783	2.05	0.49	1.60	+0.5	0.10	103.8	472
9116	1080	50	763	2.14	0.49	1.45	+1.0	0.19	108.4	486
9118	1080	50	848	1.83	0.47	1.44	-1.8	0.10	105.0	476
9119	1080	45	720	2.12	0.55	1.86	-0.9	0.11	103.0	475
9120	1080	50	732	2.12	0.55	1.87	-0.3	0.10	102.5	480
9121	1080	55	775	1.97	0.55	1.65	+1.1	0.11	105.0	480
9122	1080	60	805	1.97	0.55	1.59	-0.7	0.15	104.2	475
9123	1080	45	760	2.04	0.54	1.77	+1.2	0.13	102.4	473
9124	1080	55	760	1.97	0.60	1.61	+1.2	0.18	98.5	460
9127	1080	70	785	1.97	0.56	1.59	+1.8	0.20	103.7	475
9128	1080	65	760	2.12	0.56	2.00	+3.2	0.15	105.7	477
9129	1080	55	260	5.54	0.58	1.70	-1.3	0.18	90.3	421(2)

(1) Yarn broke after about 85% of full spool fed through continuous coater.
(2) Yarn passed through furnace three times.

Table VIII

PBN Batch Coatings on Nicalon NL 202 Plain-Weave Cloth

Run Number	Type of Plain Weave Nicalon Cloth	Yarns per Inch	Size of Coated Sample	Approximate Average Coating Thickness, Microns
6937	Commercial	16 x 16	13" x 13"	N.M.
6938	Commercial	16 x 16	13" x 13"	N.M.
6955	Fabric Development	18 x 18	12" x 12"	0.3
6964	Fabric Development	18 x 18	12" x 12"	0.3

Coating Conditions

Temperature	1080°C
Pressure	200 μ
Time	4 Minutes
NH_3	4.4 ℓpm
BCl_3	1.75 ℓpm
N_2	0.80 ℓpm
N.M.	Not Measured

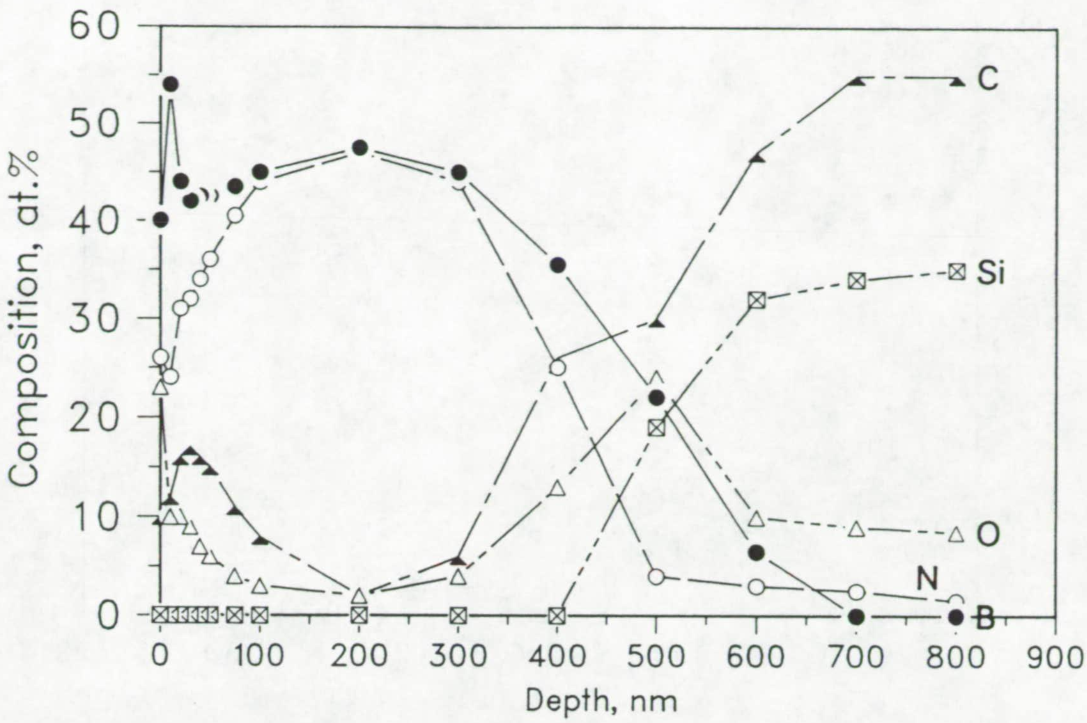

Figure 6(a) SAM depth profile of PBN-coated Nicalon NL 202 sample 9032

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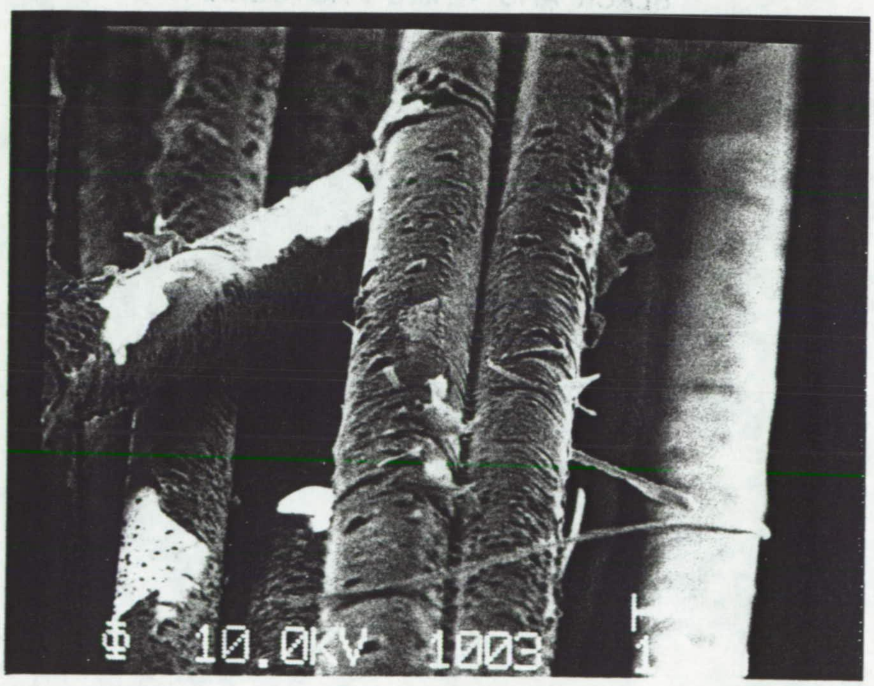

Figure 6(b). SEM photo of PBN-coated Nicalon NL 202 Sample 9032

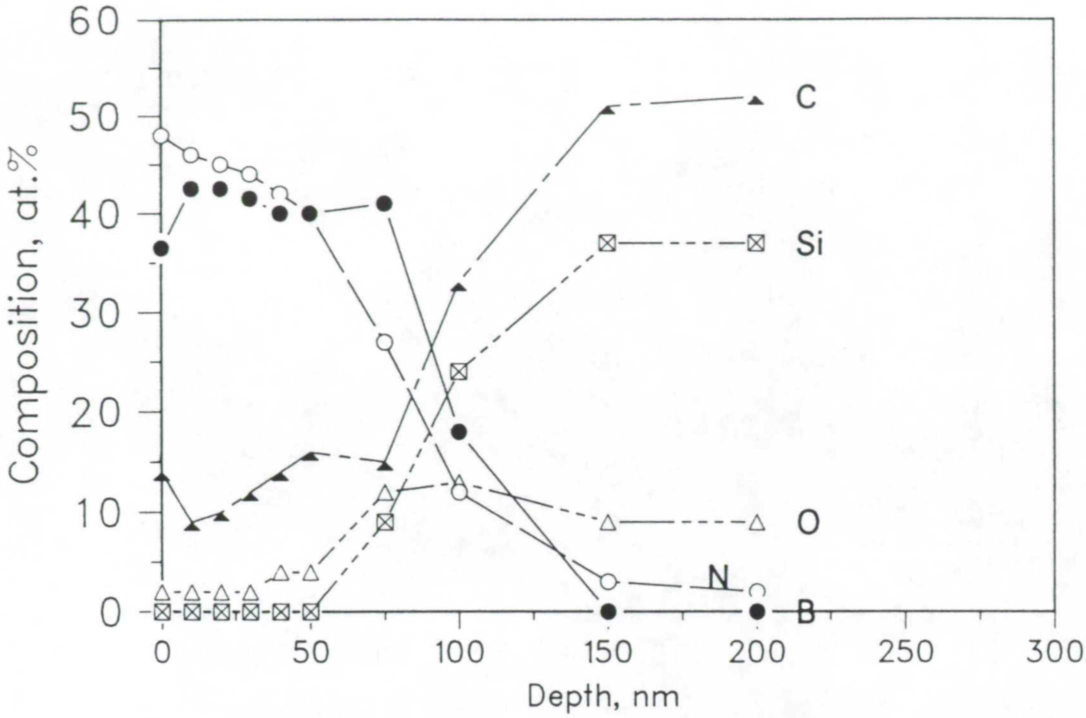

Figure 7(a). SAM depth profile of PBN-coated Nicalon NL 202 sample 9034

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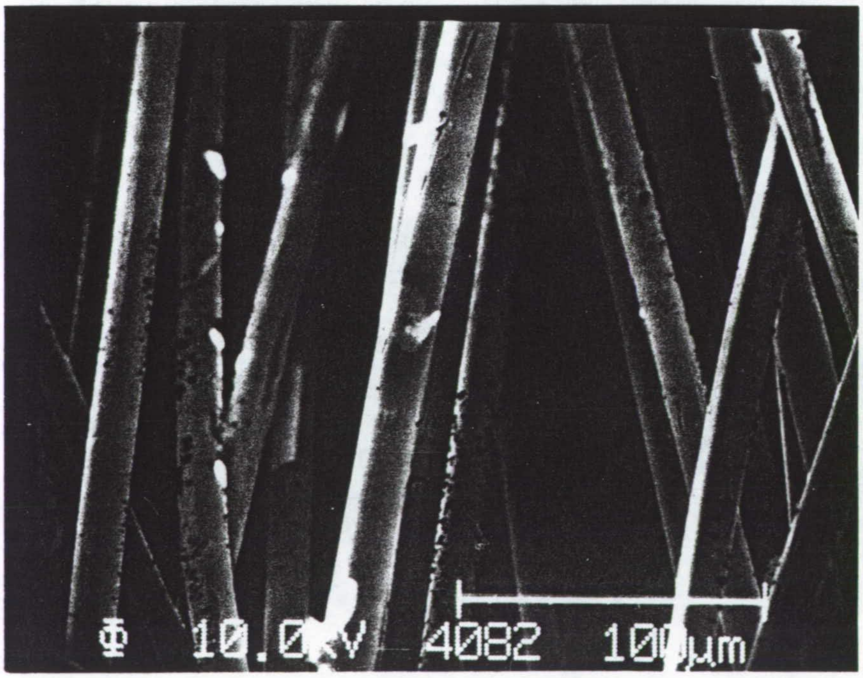

Figure 7(b). SEM photo of PBN-coated Nicalon NL 202 sample 9034

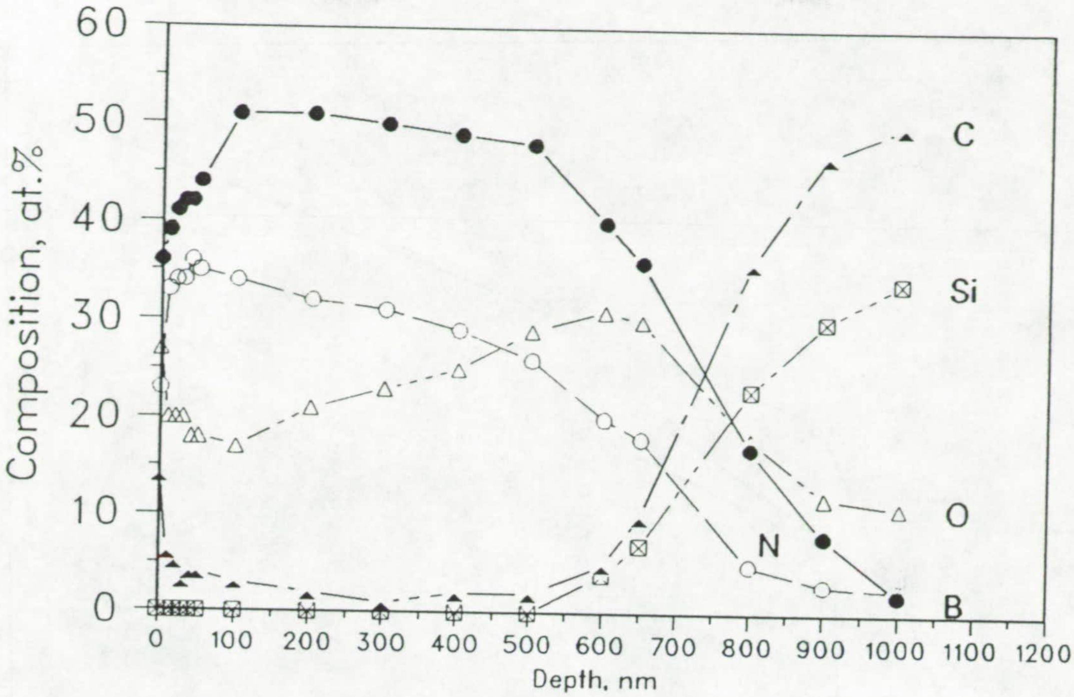

Figure 8(a) SAM depth profile for PBN-coated Nicalon NL 202 sample 9035

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

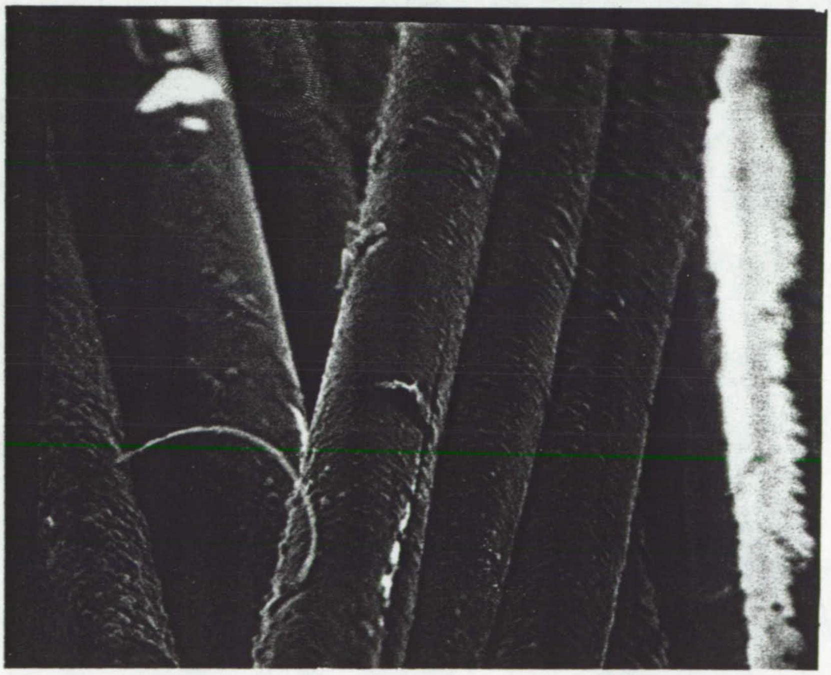

Figure 8(b). SEM photo of PBN-coated Nicalon NL 202 sample 9035

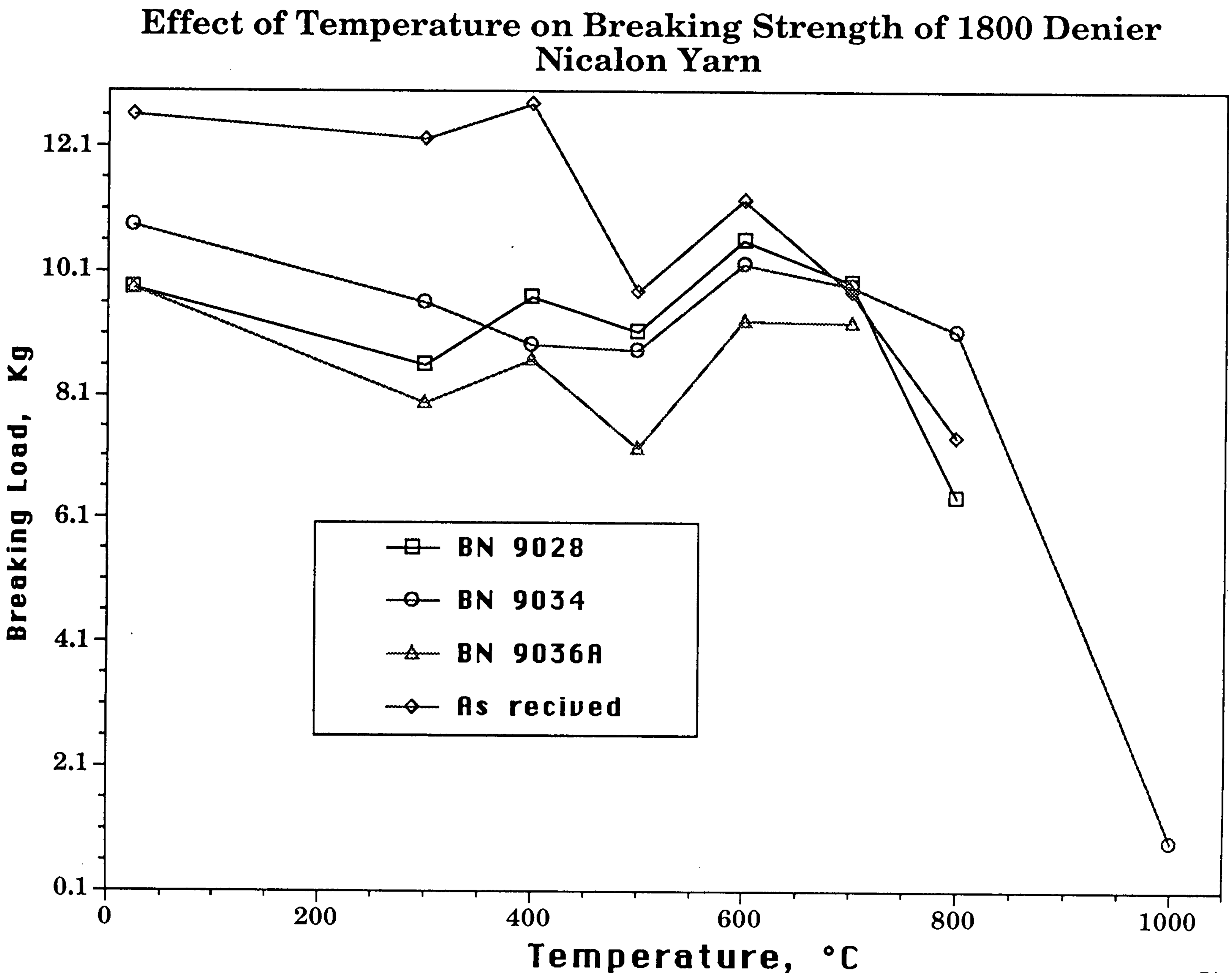
Effect of Temperature on Breaking Strength of 1800 Denier Nicalon Yarn
Breaking Load, Kg
Temperature, °C
12.1
10.1
8.1
6.1
4.1
2.1
0.1
0
200
400
600
800
1000
BN 9028
BN 9034
BN 9036A
As recived

Figure 9

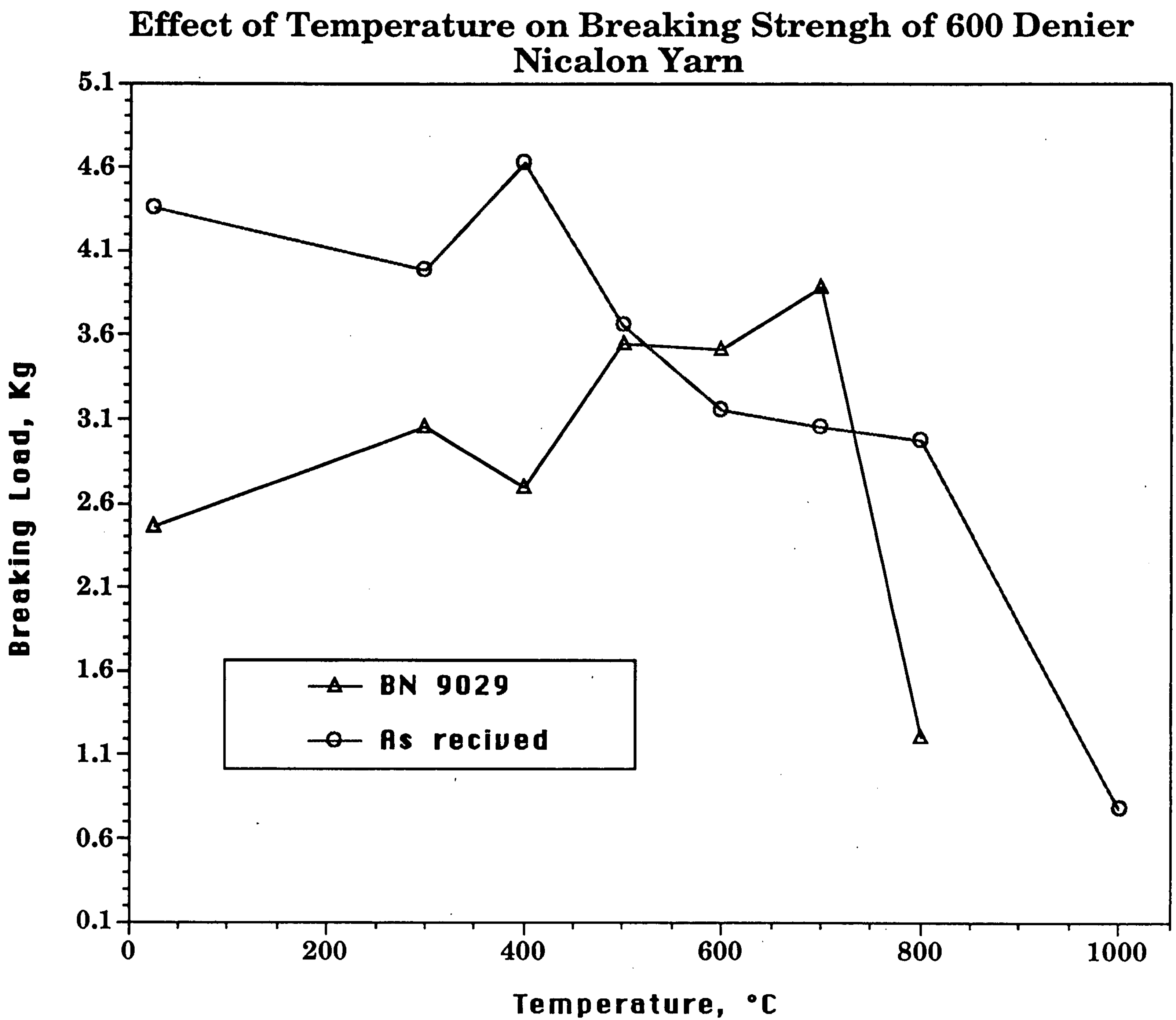
Effect of Temperature on Breaking Strengh of 600 Denier
Nicalon Yarn
Breaking Load, Kg
5.1
4.6
4.1
3.6
3.1
2.6
2.1
1.6
1.1
0.6
0.1
0
200
400
600
800
1000
Temperature, °C
BN 9029
As recived

Figure 10

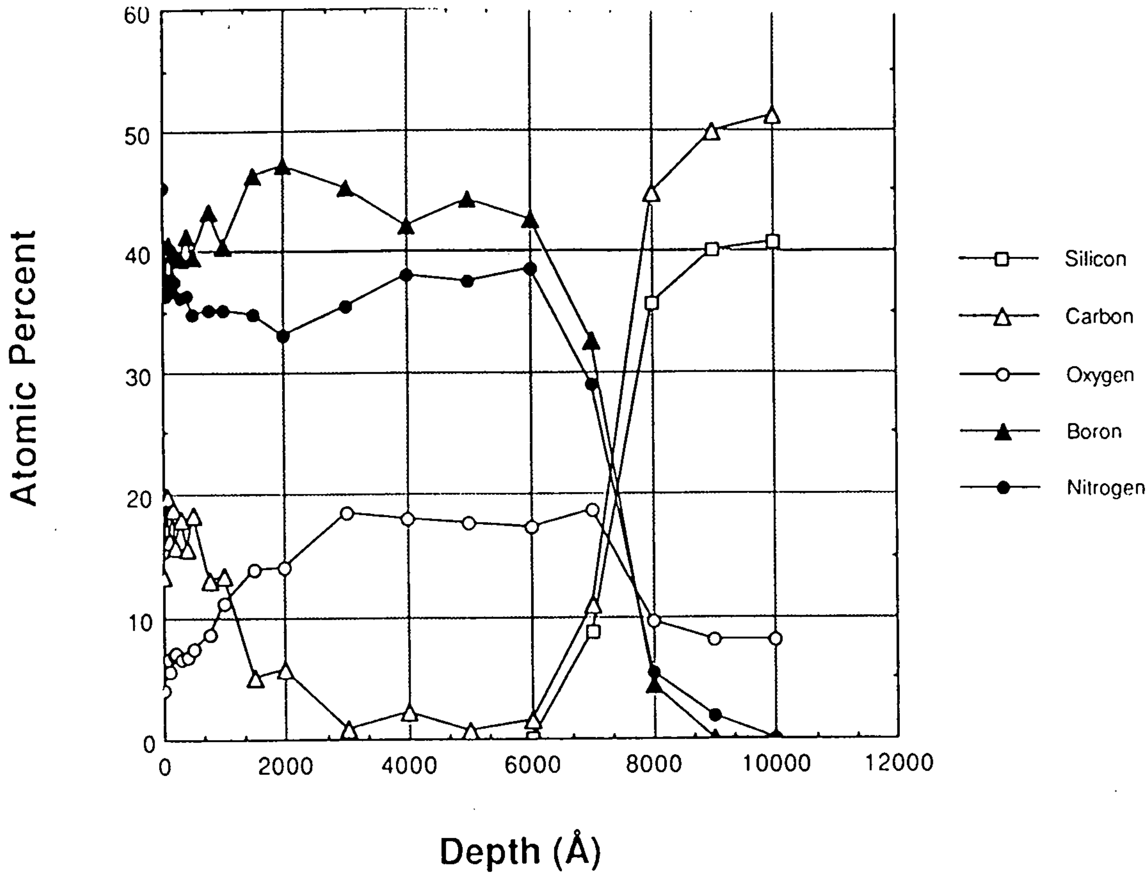

Figure 11. PBN coating 6518 on Nicalon NL 202 cloth (1050°C, 5 minutes) (Coating on outer filament of fiber weave)

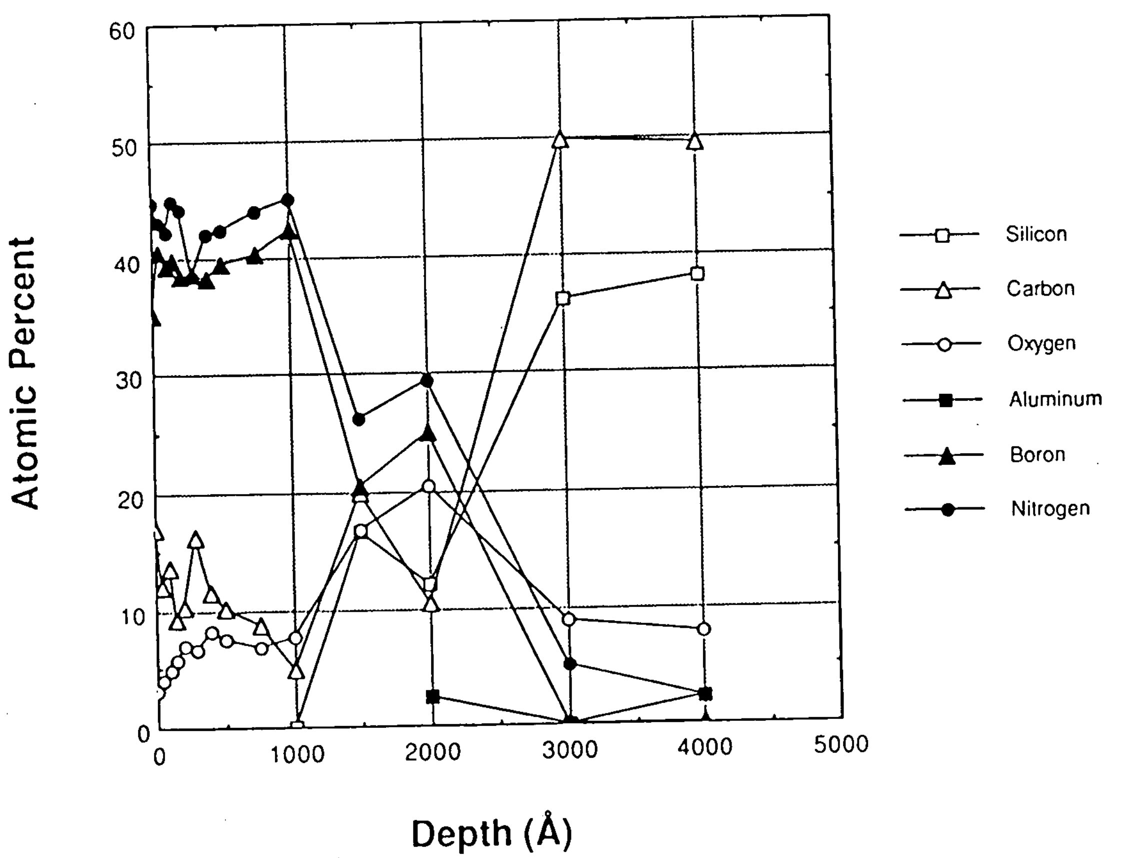

Figure 12. PBN coating 6518 on Nicalon cloth (1050°C, 5 minutes) (Coating on inner filament of fiber weave)

APPENDIX, Part I

Strand Tensile Strength of as-received Nicalon NL 202

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 1 I.D. N1 NICALON FIBERS
Tested as: P25 1.0 k Trace Number: N/A Time: 10:15
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 μm² (.000135 in²) Density: 2.494 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		401.4	0.0*	28.5	29.51	-128.9	1.463	0.99999
2		322.5	28.3	28.5	29.03	-94.5	1.170	0.99998
3		418.6	29.0	29.2	29.94	-114.2	1.495	0.99999
4		394.5	28.2	28.6	29.01	-93.1	1.428	1.00000
5		340.5	28.2	28.4	29.28	-137.9	1.239	0.99998
6		386.0						
7		385.7						
8		387.9						
9		371.0						
10		414.8						
11		374.5						
12		352.5						
N		12	4	5	5	5	5	5
AVG.		379.1	28.4	28.7	29.35	-113.7	1.359	0.99999
S.D.		29.0	0.4	0.3	0.39	20.0	0.145	0.00034
C.V.		7.6	1.4	1.1	1.32	-17.6	10.678	0.03450
Min.		322.5	28.2	28.4	29.01	-137.9	1.170	0.99998
Max.		418.6	29.0	29.2	29.94	-93.1	1.495	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 2 I.D. N2 NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 11:02
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 μm² (.000135 in²) Density: 2.498 g/cc Yield: 0.218 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		394.8	29.0	28.3	29.63	-76.7	1.379	1.00000
2		329.4	28.8	29.0	29.18	-56.2	1.156	1.00000
3		338.2	29.5	0.0*	30.14	-77.8	1.161	1.00000
4		403.3	28.9	28.2	29.86	-113.2	1.439	0.99999
5		401.7	29.1	29.5	29.76	-71.4	1.400	1.00000
6		395.7						
7		385.5						
8		409.9						
9		367.2						
10		371.1						
11		400.7						
12		321.1						
N		12	5	4	5	5	5	5
AVG.		376.6	29.1	28.8	29.71	-79.1	1.307	1.00000
S.D.		31.2	0.2	0.6	0.35	20.9	0.137	0.00035
C.V.		8.3	0.9	2.1	1.18	-26.5	10.486	0.03452
Min.		321.1	28.8	28.2	29.18	-113.2	1.156	0.99999
Max.		409.9	29.5	29.5	30.14	-56.2	1.439	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 3 I.D. N3
Tested as: SiC 1.0 k Trace Number: N/A Time: 11:14
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 μm² (.000135 in²) Density: 2.494 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		409.0	29.0	29.3	29.88	-109.2	1.450	0.99999
2		415.1	29.3	28.5	30.34	-115.3	1.454	0.99999
3		380.1	0.0*	30.2	29.93	-86.5	1.321	1.00000
4		389.6	28.8	27.6	29.34	-65.3	1.373	1.00000
5		407.2	0.0*	0.0*	30.42	-113.6	1.425	0.99998
6		364.9						
7		339.4						
8		327.1						
9		416.6						
10		386.5						
11		398.2						
12		402.2						
N		12	3	4	5	5	5	5
AVG.		386.3	29.0	28.9	29.98	-98.0	1.404	0.99999
S.D.		29.1	0.3	1.1	0.43	21.6	0.057	0.00035
C.V.		7.5	1.0	3.9	1.44	-22.0	4.046	0.03452
Min.		327.1	28.8	27.6	29.34	-115.3	1.321	0.99998
Max.		416.6	29.3	30.2	30.42	-65.3	1.454	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 4 I.D. N4
Tested as: SiC 1.0 k Trace Number: N/A Time: 11:53
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 μm² (.000135 in²) Density: 2.494 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		408.7	28.5	0.0*	29.21	-75.5	1.464	0.99999
2		348.8	28.8	28.4	29.54	-88.7	1.229	1.00000
3		418.3	29.4	0.0*	30.27	-97.7	1.464	0.99999
4		341.1	28.6	28.8	28.96	-45.8	1.200	1.00000
5		423.1	29.3	29.4	30.54	-145.8	1.512	0.99998
6		410.0						
7		379.1						
8		426.3						
9		431.1						
10		402.2						
11		426.2						
12		332.5						
N		12	5	3	5	5	5	5
AVG.		395.6	28.9	28.9	29.70	-90.7	1.374	0.99999
S.D.		36.0	0.4	0.5	0.68	36.5	0.147	0.00001
C.V.		9.1	1.4	1.8	2.28	-40.2	10.702	0.00101
Min.		332.5	28.5	28.4	28.96	-145.8	1.200	0.99998
Max.		431.1	29.4	29.4	30.54	-45.8	1.512	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 5 I.D. N5
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:03
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 µm² (.000135 in²) Density: 2.494 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		423.8	28.3	28.8	29.76	-106.1	1.520	0.99997
2		397.6	29.3	0.0*	29.86	-73.0	1.376	1.00000
3		400.0	28.8	0.0*	29.59	-102.9	1.422	1.00000
4		392.9	28.5	0.0*	29.01	-42.9	1.400	0.99999
5		369.0	28.2	0.0*	28.62	-65.1	1.315	0.99999
6		370.5						
7		409.3						
8		375.3						
9		406.8						
10		396.9						
11		419.8						
12		430.4						
N		12	5	1	5	5	5	5
AVG.		399.4	28.6	28.8	29.37	-78.0	1.407	0.99999
S.D.		20.3	0.4	0.0	0.53	26.6	0.075	0.00001
C.V.		5.1	1.5	0.0	1.82	-34.1	5.321	0.00133
Min.		369.0	28.2	28.8	28.62	-106.1	1.315	0.99997
Max.		430.4	29.3	28.8	29.86	-42.9	1.520	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 6 I.D. N6
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:12
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 µm² (.000135 in²) Density: 2.491 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		406.7	27.8	27.0	28.35	-63.7	1.492	0.99999
2		335.9	27.9	0.0*	28.24	-52.7	1.214	1.00000
3		333.6	28.9	29.4	29.21	-43.8	1.165	1.00000
4		422.4	28.4	0.0*	29.65	-132.3	1.542	0.99999
5		376.8	28.5	28.8	29.22	-84.1	1.349	0.99999
6		410.1						
7		413.9						
8		465.8						
9		360.0						
10		426.4						
11		326.5						
12		353.5						
N		12	5	3	5	5	5	5
AVG.		385.9	28.3	28.4	28.93	-75.3	1.352	0.99999
S.D.		44.4	0.4	1.2	0.61	35.2	0.166	0.00035
C.V.		11.5	1.6	4.3	2.11	-46.7	12.249	0.03452
Min.		326.5	27.8	27.0	28.24	-132.3	1.165	0.99999
Max.		465.8	28.9	29.4	29.65	-43.8	1.542	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 7 I.D. N7
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:26
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 µm² (.000135 in²) Density: 2.494 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		395.5	27.9	26.9	28.70	-75.1	1.437	0.99999
2		411.5	28.6	28.0	29.47	-112.4	1.481	0.99999
3		390.2	28.4	28.2	29.26	-96.9	1.403	0.99999
4		409.3	28.1	28.5	29.19	-115.8	1.505	0.99998
5		421.0	28.4	0.0*	29.73	-129.9	1.525	0.99998
6		423.3	28.5	29.4	29.39	-94.0	1.517	0.99999
7		401.2						
8		387.0						
9		397.9						
10		392.5						
11		385.3						
12		405.6						
N		12	6	5	6	6	6	6
AVG.		401.7	28.3	28.2	29.29	-104.0	1.478	0.99999
S.D.		12.7	0.3	0.9	0.35	19.3	0.049	0.00044
C.V.		3.2	1.0	3.2	1.18	-18.6	3.288	0.04365
Min.		385.3	27.9	26.9	28.70	-129.9	1.403	0.99998
Max.		423.3	28.6	29.4	29.73	-75.1	1.525	0.99999

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 8 I.D. N8
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:41
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87639 µm² (.000136 in²) Density: 2.480 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		343.3	28.7	0.0*	29.38	-97.5	1.217	0.99999
2		414.8	28.3	27.6	29.69	-146.1	1.516	0.99998
3		397.2	28.1	0.0*	29.10	-112.0	1.461	0.99999
4		410.4	28.7	0.0*	29.39	-71.5	1.455	0.99999
5		348.0	28.3	0.0*	28.55	-39.8	1.230	1.00000
6		425.7						
7		372.6						
8		337.5						
9		425.0						
10		424.0						
11		354.6						
12		375.3						
N		12	5	1	5	5	5	5
AVG.		385.7	28.4	27.6	29.22	-93.4	1.376	0.99999
S.D.		34.4	0.3	0.0	0.43	40.3	0.141	0.00035
C.V.		8.9	0.9	0.0	1.46	-43.1	10.257	0.03451
Min.		337.5	28.1	27.6	28.55	-146.1	1.217	0.99998
Max.		425.7	28.7	27.6	29.69	-39.8	1.516	1.00000

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 9 I.D. N9
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:51
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 86872 µm² (.000135 in²) Density: 2.483 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		408.3	27.6	28.4	28.84	-125.0	1.534	0.99998
2		392.0	28.4	0.0*	29.33	-107.9	1.419	0.99999
3		412.2	28.7	28.1	29.43	-101.3	1.478	0.99999
4		420.7	27.5	0.0*	28.21	-51.9	1.555	0.99998
5		399.7	28.5	29.5	29.37	-125.3	1.455	0.99999
6		360.4						
7		439.1						
8		410.5						
9		375.6						
10		412.2						
11		427.6						
12		370.1						
N		12	5	3	5	5	5	5
AVG.		402.4	28.1	28.7	29.04	-102.3	1.488	0.99999
S.D.		23.8	0.5	0.7	0.52	30.1	0.056	0.00002
C.V.		5.9	1.9	2.5	1.79	-29.4	3.766	0.00155
Min.		360.4	27.5	28.1	28.21	-125.3	1.419	0.99998
Max.		439.1	28.7	29.5	29.43	-51.9	1.555	0.99999

STRAND TEST REPORT

Requisition Number: 19120703 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 10 I.D. N10
Tested as: SiC 1.0 k Trace Number: N/A Time: 13:01
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87383 µm² (.000135 in²) Density: 2.476 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		364.2	27.7	26.8	28.07	-43.8	1.326	1.00000
2		318.0	28.3	27.6	28.84	-70.7	1.136	1.00000
3		360.7	28.8	27.9	29.33	-59.5	1.263	0.99999
4		398.9	27.5	28.0	28.41	-99.4	1.491	0.99999
5		373.8	28.5	0.0*	28.85	-45.0	1.324	0.99999
6		432.2						
7		380.7						
8		425.4						
9		405.0						
10		276.3						
11		445.6						
12		418.0						
N		12	5	4	5	5	5	5
AVG.		383.2	28.2	27.6	28.70	-63.7	1.308	0.99999
S.D.		49.2	0.5	0.6	0.48	22.8	0.128	0.00001
C.V.		12.8	1.9	2.0	1.67	-35.9	9.795	0.00074
Min.		276.3	27.5	26.8	28.07	-99.4	1.136	0.99999
Max.		445.6	28.8	28.0	29.33	-43.8	1.491	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 1 I.D. N11 NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 13:22
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 87128 µm² (.000135 in²) Density: 2.483 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		383.4	28.1	27.3	28.73	-79.6	1.394	1.00000
2		356.4	28.1	0.0*	28.95	-101.8	1.295	0.99999
3		367.4	28.2	0.0*	28.80	-62.8	1.317	1.00000
4		371.1	28.8	0.0*	29.28	-56.7	1.301	1.00000
5		367.6	0.0*	28.0	29.23	-81.8	1.321	0.99999
6		403.7						
7		271.2						
8		400.2						
9		364.2						
10		385.0						
11		375.1						
12		313.9						
N		12	4	2	5	5	5	5
AVG.		363.3	28.3	27.7	29.00	-76.5	1.326	0.99999
S.D.		37.0	0.3	0.5	0.25	17.7	0.040	0.00035
C.V.		10.2	1.1	1.8	0.86	-23.2	3.009	0.03452
Min.		271.2	28.1	27.3	28.73	-101.8	1.295	0.99999
Max.		403.7	28.8	28.0	29.28	-56.7	1.394	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-12-1989

Sample No. 2 I.D. N12
Tested as: SiC 1.0 k Trace Number: N/A Time: 13:35
Notebook Ref. N/A Extensometer: A Frame Operator DEH
Area: 86872 µm² (.000135 in²) Density: 2.486 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		387.4	28.0	0.0*	28.86	-87.1	1.401	0.99999
2		378.3	27.6	0.0*	28.72	-121.3	1.408	0.99998
3		414.8	28.2	27.7	29.17	-102.7	1.512	0.99999
4		335.6	28.4	30.0	29.06	-75.5	1.198	0.99999
5		331.7	27.8	0.0*	28.30	-68.2	1.209	1.00000
6		367.7						
7		411.7						
8		348.3						
9		396.4						
10		397.2						
11		366.5						
12		405.6						
N		12	5	2	5	5	5	5
AVG.		378.4	28.0	28.8	28.82	-90.9	1.346	0.99999
S.D.		28.8	0.3	1.7	0.34	21.4	0.137	0.00001
C.V.		7.6	1.1	5.8	1.19	-23.5	10.197	0.00116
Min.		331.7	27.6	27.7	28.30	-121.3	1.198	0.99998
Max.		414.8	28.4	30.0	29.17	-68.2	1.512	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 3 I.D. N13NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 10:17
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 87639 µm² (.000136 in²) Density: 2.468 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		313.2	28.4	0.0*	29.98	-191.4	1.130	0.99997
2		435.8	27.9	0.0*	28.51	-71.9	1.603	0.99999
3		313.9	27.6	0.0*	28.02	-50.4	1.138	0.99999
4		432.5	27.9	27.3	28.94	-108.5	1.598	0.99999
5		381.7	28.9	0.0*	29.63	-91.6	1.345	1.00000
6		378.2						
7		434.6						
8		404.7						
9		417.5						
10		396.0						
11		415.1						
12		388.5						
N		12	5	1	5	5	5	5
AVG.		392.6	28.2	27.3	29.01	-102.8	1.363	0.99999
S.D.		41.9	0.5	0.0	0.80	54.1	0.234	0.00034
C.V.		10.7	1.8	0.0	2.76	-52.6	17.138	0.03450
Min.		313.2	27.6	27.3	28.02	-191.4	1.130	0.99997
Max.		435.8	28.9	27.3	29.98	-50.4	1.603	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 4 I.D. N14NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 10:34
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 86617 µm² (.000134 in²) Density: 2.490 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		376.5	28.3	0.0*	29.27	-113.3	1.367	0.99999
2		373.7	28.3	27.5	28.76	-55.6	1.335	0.99999
3		387.1	28.5	0.0*	29.41	-104.2	1.392	0.99998
4		392.5	28.7	0.0*	29.45	-96.3	1.406	0.99999
5		370.0	28.0	27.5	0.00*	0.0*	1.384	0.99993*
6		370.6						
7		384.9						
8		318.1						
9		347.0						
10		415.0						
11		378.6						
12		426.9						
N		12	5	2	4	4	5	4
AVG.		378.4	28.3	27.5	29.22	-92.3	1.377	0.99999
S.D.		28.2	0.3	0.0	0.32	25.4	0.027	0.00028
C.V.		7.5	0.9	0.0	1.09	-27.6	1.986	0.02821
Min.		318.1	28.0	27.5	28.76	-113.3	1.335	0.99998
Max.		426.9	28.7	27.5	29.45	-55.6	1.406	0.99999

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 5 I.D. N15NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 10:46
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 87128 µm² (.000135 in²) Density: 2.487 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		408.5	28.5	0.0*	29.30	-91.8	1.466	1.00000
2		371.8	28.8	28.4	29.95	-145.8	1.341	0.99998
3		372.8	29.3	28.4	30.14	-137.0	1.331	1.00000
4		390.7	28.1	0.0*	28.66	-72.3	1.423	1.00000
5		372.1	28.7	0.0*	29.44	-88.0	1.322	0.99999
6		387.5						
7		297.2						
8		392.1						
9		381.5						
10		365.1						
11		385.3						
12		373.6						
13		315.7						
N		13	5	2	5	5	5	5
AVG.		370.3	28.7	28.4	29.50	-107.0	1.377	0.99999
S.D.		30.8	0.4	0.0	0.58	32.4	0.064	0.00035
C.V.		8.3	1.5	0.1	1.98	-30.3	4.669	0.03451
Min.		297.2	28.1	28.4	28.66	-145.8	1.322	0.99998
Max.		408.5	29.3	28.4	30.14	-72.3	1.466	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 6 I.D. N16NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 10:58
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 86617 µm² (.000134 in²) Density: 2.486 g/cc Yield: 0.215 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		391.9	28.3	27.8	29.66	-144.1	1.438	0.99997
2		353.9	28.8	28.4	29.98	-139.1	1.258	0.99999
3		395.9	28.2	28.2	28.81	-82.7	1.436	1.00000
4		386.6	28.7	0.0*	29.66	-112.0	1.376	0.99999
5		290.9	28.2	0.0*	28.64	-52.3	1.033	1.00000
6		377.9						
7		365.1						
8		407.1						
9		214.7						
10		385.2						
11		365.3						
12		430.6						
N		12	5	3	5	5	5	5
AVG.		363.8	28.4	28.1	29.35	-106.0	1.308	0.99999
S.D.		58.0	0.3	0.3	0.59	38.8	0.170	0.00001
C.V.		15.9	1.0	1.1	2.00	-36.6	13.009	0.00129
Min.		214.7	28.2	27.8	28.64	-144.1	1.033	0.99997
Max.		430.6	28.8	28.4	29.98	-52.3	1.438	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 7 I.D. N17NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 11:10
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 86872 µm² (.000135 in²) Density: 2.483 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		366.6	28.5	0.0*	29.54	-116.4	1.321	0.99999
2		411.7	27.9	27.7	28.80	-103.0	1.526	0.99999
3		392.4	26.9	26.5	27.37	-46.9	1.479	1.00000
4		363.0	28.2	27.8	28.92	-97.1	1.322	0.99999
5		396.6	28.2	0.0*	29.00	-97.8	1.441	0.99999
6		415.7						
7		405.5						
8		360.6						
9		375.1						
10		378.5						
11		419.4						
N		11	5	3	5	5	5	5
AVG.		389.5	27.9	27.4	28.73	-92.3	1.418	0.99999
S.D.		21.9	0.6	0.7	0.81	26.5	0.093	0.00001
C.V.		5.6	2.2	2.6	2.82	-28.7	6.548	0.00102
Min.		360.6	26.9	26.5	27.37	-116.4	1.321	0.99999
Max.		419.4	28.5	27.8	29.54	-46.9	1.526	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 8 I.D. N18NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 11:59
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 86872 µm² (.000135 in²) Density: 2.486 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		381.4	28.6	27.9	29.48	-101.7	1.360	0.99999
2		321.1	28.2	0.0*	28.69	-62.3	1.159	0.99999
3		351.7	28.6	28.5	29.16	-80.5	1.249	1.00000
4		358.8	27.9	27.5	28.91	-111.2	1.314	0.99998
5		339.2	29.2	0.0*	29.79	-107.3	1.191	0.99999
6		360.9						
7		349.6						
8		401.0						
9		388.6						
10		305.2						
11		363.1						
12		310.7						
N		12	5	3	5	5	5	5
AVG.		352.6	28.5	28.0	29.21	-92.6	1.255	0.99999
S.D.		30.0	0.5	0.5	0.44	20.7	0.083	0.00001
C.V.		8.5	1.8	1.7	1.51	-22.3	6.647	0.00109
Min.		305.2	27.9	27.5	28.69	-111.2	1.159	0.99998
Max.		401.0	29.2	28.5	29.79	-62.3	1.360	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 9 I.D. N19NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:10
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 87128 µm² (.000135 in²) Density: 2.487 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		305.2	30.3	29.4	30.62	-59.3	1.012	1.00000
2		366.8	27.6	27.4	28.71	-123.3	1.365	0.99998
3		373.3	28.2	0.0*	28.84	-81.0	1.349	1.00000
4		281.1	28.5	0.0*	29.16	-100.2	0.998	1.00000
5		380.9	27.7	27.6	28.31	-56.3	1.396	0.99999
6		410.7						
7		386.2						
8		435.5						
9		305.1						
10		425.3						
11		361.4						
12		397.2						
N		12	5	3	5	5	5	5
AVG.		369.1	28.5	28.2	29.13	-84.0	1.224	0.99999
S.D.		49.1	1.1	1.1	0.89	28.2	0.200	0.00001
C.V.		13.3	3.8	3.9	3.04	-33.6	16.381	0.00086
Min.		281.1	27.6	27.4	28.31	-123.3	0.998	0.99998
Max.		435.5	30.3	29.4	30.62	-56.3	1.396	1.00000

STRAND TEST REPORT

Requisition Number: 19120704 Charge Number: 484-4903

Submitter: MOORE Date: 12-15-1989

Sample No. 10 I.D. N20NICALON FIBERS
Tested as: SiC 1.0 k Trace Number: N/A Time: 12:21
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 87128 µm² (.000135 in²) Density: 2.479 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	COEFFICIENTS F Msi	FAILURE STRAIN %	Coef. of Deter.
1		422.4	27.3	27.4	28.26	-92.8	1.593	0.99999
2		330.4	27.4	0.0*	27.58	-12.7	1.207	0.99999
3		323.7	27.9	27.2	28.10	-25.8	1.164	1.00000
4		383.4	28.5	27.7	29.15	-76.0	1.369	1.00000
5		295.7	28.0	0.0*	28.15	6.0	1.050	0.99996
6		320.3						
7		379.3						
8		351.9						
9		296.6						
10		372.2						
11		411.7						
12		387.8						
N		12	5	3	5	5	5	5
AVG.		356.3	27.8	27.4	28.25	-40.3	1.276	0.99999
S.D.		42.9	0.5	0.3	0.57	42.3	0.211	0.00001
C.V.		12.0	1.7	1.0	2.01	-105.0	16.501	0.00144
Min.		295.7	27.3	27.2	27.58	-92.8	1.050	0.99996
Max.		422.4	28.5	27.7	29.15	6.0	1.593	1.00000

APPENDIX, Part II

Strand Tensile Strength of Heat-Treated Nicalon NL 202

STRAND TEST REPORT

Requisition Number: 19121803 Charge Number: 484-4903

Submitter: MOORE Date: 01-04-1990

Sample No. 1 I.D. NLM-HT-1000 C
Tested as: SiC 1.0 k Trace Number: N/A Time: 15:43
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 80995 µm² (.000126 in²) Density: 2.531 g/cc Yield: 0.205 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		317.0	29.3	29.3	30.65	-166.3	1.113	0.99997
2		269.8	28.9	28.9	29.38	-55.0	0.934	1.00000
3		352.7	29.1	29.1	30.06	-120.1	1.240	0.99998
4		275.0	0.0*	31.6	0.00*	0.0*	0.875	0.99979*
5		331.6	28.1	27.4	29.04	-108.9	1.202	0.99998
6		257.0	28.7	0.0*	28.98	-48.5	0.894	1.00000
7		309.6						
8		331.6						
9		293.4						
10		212.7						
11		306.0						
12		299.6						
N		12	5	5	5	5	6	5
AVG.		296.3	28.8	29.3	29.62	-99.7	1.043	0.99999
S.D.		38.2	0.5	1.5	0.71	48.9	0.162	0.00002
C.V.		12.9	1.6	5.2	2.41	-49.0	15.543	0.00151
Min.		212.7	28.1	27.4	28.98	-166.3	0.875	0.99997
Max.		352.7	29.3	31.6	30.65	-48.5	1.240	1.00000

STRAND TEST REPORT

Requisition Number: 19121803 Charge Number: 484-4903

Submitter: MOORE Date: 01-04-1990

Sample No. 2 I.D. NLM202-HT-1100 C
Tested as: SiC 1.0 k Trace Number: N/A Time: 15:56
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 81506 µm² (.000126 in²) Density: 2.523 g/cc Yield: 0.206 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		307.9	29.5	0.0*	29.83	-71.1	1.052	1.00000
2		313.5	28.9	0.0*	29.13	-24.1	1.080	1.00000
3		332.2	30.2	29.8	30.52	-49.1	1.108	1.00000
4		285.7	28.1	28.5	28.01	13.3	1.018	1.00000
5		324.7	28.7	0.0*	29.45	-96.6	1.154	1.00000
6		269.4						
7		248.4						
8		301.7						
9		196.1						
10		279.8						
11		363.2						
N		11	5	2	5	5	5	5
AVG.		293.0	29.1	29.2	29.39	-45.5	1.082	1.00000
S.D.		45.1	0.8	0.9	0.93	42.4	0.052	0.00000
C.V.		15.4	2.7	3.1	3.15	-93.2	4.809	0.00038
Min.		196.1	28.1	28.5	28.01	-96.6	1.018	1.00000
Max.		363.2	30.2	29.8	30.52	13.3	1.154	1.00000

STRAND TEST REPORT

Requisition Number: 19121803 Charge Number: 484-4903

Submitter: MOORE Date: 01-04-1990

Sample No. 3 I.D. NLM-HT-1200 C
Tested as: SiC 1.0 k Trace Number: N/A Time: 17:09
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 81506 μm² (.000126 in²) Density: 2.523 g/cc Yield: 0.206 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi SECANT .1-.6	* COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		153.2	29.7	29.2	30.14	-67.2	0.509	0.99999
2		67.3	0.0*	29.2	29.49	-119.7	0.230	0.99995
3		41.3						
4		117.6						
5	BGrip	137.3*	30.1	0.0*	30.16	-1.3	0.447	0.99999
6	BKnif	93.4*	28.2	0.0*	0.00*	0.0*	0.348	0.99989*
7	EStop	54.9*						
8		81.8						
9		130.6						
10		165.0						
11	BGrip	33.0*						
12	BGrip	114.4*						
13	BGrip	31.9*						
N		7	3	2	3	3	7	3
AVG.		108.1	29.4	29.2	29.93	-62.7	1.492	0.99998
S.D.		46.0	1.0	0.0	0.38	59.3	1.559	0.00049
C.V.		42.5	3.4	0.2	1.26	-94.5	%104.521	0.04875
Min.		41.3	28.2	29.2	29.49	-119.7	0.230	0.99995
Max.		165.0	30.1	29.2	30.16	-1.3	4.266	0.99999

STRAND TEST REPORT

Requisition Number: 19121803 Charge Number: 484-4903

Submitter: MOORE Date: 01-04-1990

Sample No. 4 I.D. NLM-HT-1350 C
Tested as: SiC 1.0 k Trace Number: N/A Time: 17:56
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 75119 μm² (.000116 in²) Density: 2.605 g/cc Yield: 0.196 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi SECANT .1-.6	* COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1	BGrip	121.8*	29.9	0.0*	30.84	-179.1	0.396	0.99997
2	BGrip	49.2*						
3		155.4	0.0*	0.0*	30.00	119.2	0.508	0.99996
4		117.6	0.0*	0.0*	0.00*	0.0*	0.369	0.99995*
5	BGrip	72.5*						
6		200.6	31.0	0.0*	31.58	-96.3	0.648	0.99999
7		200.8	30.0	29.2	30.15	-36.7	0.667	0.99999
8		180.6	29.5	0.0*	29.61	-22.8	0.606	0.99999
9		199.3						
10	BGrip	114.1*						
11		172.0						
12		196.3						
N		8	4	1	5	5	8	5
AVG.		177.8	30.1	29.2	30.44	-43.1	1.062	0.99998
S.D.		29.3	0.6	0.0	0.78	109.7	1.135	0.00002
C.V.		16.5	2.2	0.0	2.57	-254.3	%106.886	0.00215
Min.		117.6	29.5	29.2	29.61	-179.1	0.369	0.99996
Max.		200.8	31.0	29.2	31.58	119.2	3.701	0.99999

STRAND TEST REPORT

Requisition Number: 19121803 Charge Number: 484-4903

Submitter: MOORE Date: 01-04-1990

Sample No. 5 I.D. NLM-HT-1100 C 10 Minutes
Tested as: SiC 1.0 k Trace Number: N/A Time: 18:07
Notebook Ref. N/A Extensometer: A Frame Operator DKS
Area: 80229 μm² (.000124 in²) Density: 2.530 g/cc Yield: 0.203 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.6	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		258.9	30.0	0.0*	30.47	-52.5	0.862	0.99999
2		271.9	31.6	30.5	33.16	-239.2	0.881	0.99998
3	TGrip	119.7*	0.0*	28.4	0.00*	0.0*	0.419	0.99994*
4		246.9	29.8	30.4	30.06	-49.9	0.837	1.00000
5		245.8	29.7	29.5	29.97	-47.0	0.834	1.00000
6		240.9	30.8	0.0*	31.11	-82.5	0.789	0.99999
7		230.9						
8		222.4						
9		228.8						
10		184.4						
11		231.0						
12		260.8						
N		11	5	4	5	5	6	5
AVG.		238.4	30.4	29.7	30.96	-94.2	0.771	0.99999
S.D.		23.6	0.8	1.0	1.31	82.3	0.175	0.00049
C.V.		9.9	2.8	3.3	4.24	-87.3	22.705	0.04882
Min.		184.4	29.7	28.4	29.97	-239.2	0.419	0.99998
Max.		271.9	31.6	30.5	33.16	-47.0	0.881	1.00000

APPENDIX, Part III

Strand Tensile Strength of PBN-Coated Nicalon NL 202

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 02-25-1991

Sample No. 1 I.D. PBN-9028-1
Tested as: P25 2.0 k Trace Number: N/A Time: 14:48
Notebook Ref. N/A Extensometer: MTI SST Operator JAZ
Area: 88916 μm² (.000138 in²) Density: 2.485 g/cc Yield: 0.221 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi SECANT .1-.3	* COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		316.9	28.9	28.7	0.00*	0.0*	1.075	0.99986*
2		299.7	30.0	30.8	31.28	-164.6	1.035	0.99996
3		297.5	30.6	30.1	30.79	-97.6	1.012	0.99997
4		334.9	29.7	29.7	31.15	-179.5	1.162	0.99996
5		328.3	29.8	30.0	30.64	-99.4	1.112	0.99999
6		324.3						
7		330.1						
8		344.1						
9		285.0						
10		312.4						
11		325.8						
12		334.1						
N		12	5	5	4	4	5	4
AVG.		319.4	29.8	29.9	30.96	-135.3	1.079	0.99997
S.D.		17.7	0.6	0.8	0.30	42.9	0.060	0.00028
C.V.		5.5	2.1	2.6	0.97	-31.7	5.547	0.02799
Min.		285.0	28.9	28.7	30.64	-179.5	1.012	0.99996
Max.		344.1	30.6	30.8	31.28	-97.6	1.162	0.99999

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 02-25-1991

Sample No. 2 I.D. PBN-9028-2
Tested as: P25 2.0 k Trace Number: N/A Time: 15:01
Notebook Ref. N/A Extensometer: MTI SST Operator JAZ
Area: 85850 μm² (.000133 in²) Density: 2.516 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi SECANT .1-.3	* COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		345.3	30.4	30.9	0.00*	0.0*	1.188	0.99967*
2	Spltl	334.4*	30.1	30.1	31.02	-59.2	1.112	0.99995
3		305.3	30.4	30.4	30.45	1.4	1.002	0.99998
4		307.0	30.8	30.7	31.99	-180.8	1.020	0.99998
5		325.2	30.3	30.6	31.38	-123.6	1.089	0.99998
6		299.2						
7		315.3						
8		347.7						
9		347.7						
10		350.7						
11		322.5						
12		352.2						
N		11	5	5	4	4	5	4
AVG.		328.9	30.4	30.5	31.21	-90.5	1.082	0.99997
S.D.		20.4	0.2	0.3	0.64	78.9	0.075	0.00040
C.V.		6.2	0.8	1.0	2.06	-87.1	6.899	0.03976
Min.		299.2	30.1	30.1	30.45	-180.8	1.002	0.99995
Max.		352.2	30.8	30.9	31.99	1.4	1.188	0.99998

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 | Charge Number: 710-000058

Submitter: SPRAGG | Date: 02-25-1991

Sample No. 3 I.D. PBN-9028-3
Tested as: P25 2.0 k | Trace Number: N/A | Time: 15:32
Notebook Ref. N/A | Extensometer: MTI SST | Operator JAZ
Area: 85084 μm² (.000132 in²) | Density: 2.527 g/cc | Yield: 0.215 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		352.7	32.6	0.0*	33.99	-230.1	1.128	0.99999
2	Spltl	348.7*	30.8	30.3	31.15	-74.3	1.153	0.99999
3		299.4	31.0	31.3	0.00*	0.0*	0.995	0.99994*
4		363.7	31.5	31.5	32.47	-143.0	1.186	0.99999
5		359.2	30.5	30.8	31.54	-116.6	1.208	0.99995
6		336.7						
7		365.5						
8		329.6						
9		359.0						
10		358.2						
11		289.1						
12		364.6						
N		11	5	4	4	4	5	4
AVG.		343.4	31.3	31.0	32.29	-141.0	1.134	0.99998
S.D.		27.0	0.8	0.5	1.26	65.8	0.084	0.00028
C.V.		7.9	2.6	1.7	3.90	-46.7	7.364	0.02828
Min.		289.1	30.5	30.3	31.15	-230.1	0.995	0.99995
Max.		365.5	32.6	31.5	33.99	-74.3	1.208	0.99999

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 | Charge Number: 710-000058

Submitter: SPRAGG | Date: 02-25-1991

Sample No. 4 I.D. PBN-9028-4
Tested as: P25 2.0 k | Trace Number: N/A | Time: 16:03
Notebook Ref. N/A | Extensometer: MTI SST | Operator JAZ
Area: 85850 μm² (.000133 in²) | Density: 2.504 g/cc | Yield: 0.215 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		345.2	30.3	30.8	30.96	-109.6	1.169	0.99999
2		376.1	31.5	31.6	32.07	-119.4	1.234	0.99999
3		347.1	31.7	32.3	33.08	-153.7	1.109	0.99998
4		393.0	30.7	30.9	32.28	-190.0	1.335	0.99996
5		335.2	31.6	31.5	32.32	-130.9	1.090	0.99999
6		315.1						
7		378.8						
8		312.1						
9		302.6						
10		322.7						
11		358.4						
12		352.1						
N		12	5	5	5	5	5	5
AVG.		344.9	31.2	31.4	32.14	-140.7	1.187	0.99998
S.D.		28.6	0.6	0.6	0.76	32.1	0.100	0.00002
C.V.		8.3	1.9	2.0	2.38	-22.8	8.413	0.00215
Min.		302.6	30.3	30.8	30.96	-190.0	1.090	0.99996
Max.		393.0	31.7	32.3	33.08	-109.6	1.335	0.99999

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 02-25-1991

Sample No. 5 I.D. PBN-9028-5
Tested as: P25 2.0 k Trace Number: N/A Time: 16:17
Notebook Ref. N/A Extensometer: MTI SST Operator JAZ
Area: 85084 μm² (.000132 in²) Density: 2.515 g/cc Yield: 0.214 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		330.0	31.0	30.8	31.57	-37.6	1.065	0.99998
2		359.4	30.4	30.3	0.00*	0.0*	1.226	0.99992*
3		369.7	31.0	31.2	32.25	-150.2	1.219	0.99998
4		387.9	31.1	0.0*	0.00*	0.0*	1.256	0.99995*
5		394.7	31.4	31.6	0.00*	0.0*	1.303	0.99992*
6		372.2						
7		364.8						
8		364.1						
9		388.2						
10		374.2						
11		341.2						
12		410.3						
N		12	5	4	2	2	5	2
AVG.		371.4	31.0	31.0	31.91	-93.9	1.214	0.99998
S.D.		22.3	0.3	0.6	0.48	79.6	0.090	0.00002
C.V.		6.0	1.1	1.8	1.50	-84.8	7.382	0.00246
Min.		330.0	30.4	30.3	31.57	-150.2	1.065	0.99998
Max.		410.3	31.4	31.6	32.25	-37.6	1.303	0.99998

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 03-13-1991

Sample No. 10 I.D. PBN-9110-2
Tested as: P25 2.0 k Trace Number: N/A Time: 15:11
Notebook Ref. N/A Extensometer: MTI SST Operator MAS
Area: 87383 μm² (.000135 in²) Density: 2.495 g/cc Yield: 0.218 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .10-.30	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		379.4	30.3	30.3	31.32	-144.2	1.295	1.00000
2		394.7	30.6	31.5	0.00*	0.0*	0.650	0.88819*
3		366.4	31.0	31.6	32.11	-179.7	1.231	0.99998
4		355.0	30.6	30.9	31.33	-108.0	1.201	0.99999
5		357.5	29.1	29.1	30.11	-130.4	1.259	0.99999
6		361.4						
7		396.4						
8		365.9						
9		312.5						
10		372.2						
11		388.6						
12		368.0						
N		12	5	5	4	4	5	4
AVG.		368.2	30.3	30.7	31.22	-140.6	1.127	0.99999
S.D.		22.4	0.7	1.0	0.83	30.0	0.269	0.00028
C.V.		6.1	2.4	3.3	2.65	-21.4	23.866	0.02816
Min.		312.5	29.1	29.1	30.11	-179.7	0.650	0.99998
Max.		396.4	31.0	31.6	32.11	-108.0	1.295	1.00000

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 02-25-1991

Sample No. 6 I.D. PBN-9110-3
Tested as: P25 2.0 k Trace Number: N/A Time: 17:18
Notebook Ref. N/A Extensometer: MTI SST Operator JAZ
Area: 87383 μm² (.000135 in²) Density: 2.495 g/cc Yield: 0.218 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		372.0	29.7	30.4	0.00*	0.0*	1.273	0.99994*
2		410.7	30.0	29.9	0.00*	0.0*	1.388	0.99985*
3		394.7	30.2	30.2	0.00*	0.0*	1.355	0.99994*
4		393.2	30.5	30.0	31.11	-98.4	1.327	0.99998
5		385.7	29.4	0.0*	30.57	-104.2	1.334	0.99998
6		371.8						
7		374.6						
8		335.0						
9		371.7						
10		365.1						
11		374.2						
12		384.2						
N		12	5	4	2	2	5	2
AVG.		377.7	30.0	30.1	30.84	-101.3	1.336	0.99998
S.D.		18.7	0.4	0.2	0.39	4.1	0.042	0.00003
C.V.		4.9	1.4	0.7	1.25	-4.1	3.150	0.00312
Min.		335.0	29.4	29.9	30.57	-104.2	1.273	0.99998
Max.		410.7	30.5	30.4	31.11	-98.4	1.388	0.99998

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 02-25-1991

Sample No. 7 I.D. PBN-9110-5
Tested as: P25 2.0 k Trace Number: N/A Time: 17:36
Notebook Ref. N/A Extensometer: MTI SST Operator JAZ
Area: 85850 μm² (.000133 in²) Density: 2.528 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .1-.3	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		378.6	32.6	33.0	33.83	-161.2	1.198	0.99997
2		389.4	30.2	30.5	31.72	-102.5	1.294	0.99995
3		394.7	29.6	30.2	30.63	-90.6	1.353	0.99997
4		371.8	31.7	31.3	32.88	-183.6	1.217	0.99999
5		366.1	31.3	29.9	31.57	-14.4	1.168	0.99996
6		396.9						
7		361.4						
8		367.5						
9		355.2						
10		367.3						
N		10	5	5	5	5	5	5
AVG.		374.9	31.1	31.0	32.13	-110.5	1.246	0.99997
S.D.		14.4	1.2	1.2	1.24	66.4	0.076	0.00003
C.V.		3.8	3.8	4.0	3.86	-60.1	6.068	0.00343
Min.		355.2	29.6	29.9	30.63	-183.6	1.168	0.99995
Max.		396.9	32.6	33.0	33.83	-14.4	1.353	0.99999

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 03-13-1991

Sample No. 8 I.D. PBN-9110-9
Tested as: P25 2.0 k Trace Number: N/A Time: 14:36
Notebook Ref. N/A Extensometer: MTI SST Operator MAS
Area: 85850 μm² (.000133 in²) Density: 2.528 g/cc Yield: 0.217 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .10-.30	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		422.8	30.3	30.4	0.00*	0.0*	1.472	0.99994*
2		367.3	29.8	0.0*	0.00*	0.0*	1.242	0.99983*
3		410.8	30.9	31.8	0.00*	0.0*	1.392	0.99990*
4		421.6	30.2	30.4	31.59	-169.3	1.458	0.99997
5		420.8	30.5	32.0	0.00*	0.0*	1.434	0.99992*
6		403.9	30.4	30.8	31.39	-126.5	1.368	0.99999
7		403.5						
8		448.0						
9		436.4						
10		371.1						
11		433.9						
12		333.2						
N		12	6	5	2	2	6	2
AVG.		406.1	30.4	31.1	31.49	-147.9	1.394	0.99998
S.D.		33.4	0.4	0.8	0.14	30.3	0.084	0.00035
C.V.		8.2	1.2	2.5	0.43	-20.5	6.036	0.03463
Min.		333.2	29.8	30.4	31.39	-169.3	1.242	0.99997
Max.		448.0	30.9	32.0	31.59	-126.5	1.472	0.99999

STRAND EXTENSOMETER TEST REPORT

Requisition Number: 91022001 Charge Number: 710-000058

Submitter: SPRAGG Date: 03-13-1991

Sample No. 9 I.D. PBN-9110-10
Tested as: P25 2.0 k Trace Number: N/A Time: 14:57
Notebook Ref. N/A Extensometer: MTI SST Operator MAS
Area: 85084 μm² (.000132 in²) Density: 2.539 g/cc Yield: 0.216 g/m

SPEC No.	BREAK TYPE	TENSILE STRENGTH Ksi	* MODULUS GREENV'L 60-120	Msi * SECANT .10-.30	COEFFICIENTS Eo Msi	F Msi	FAILURE STRAIN %	Coef. of Deter.
1		366.9	31.9	0.0*	0.00*	0.0*	1.129	0.99951*
2		412.3	30.5	31.1	0.00*	0.0*	1.380	0.99987*
3		440.3						
4		429.3	32.3	0.0*	0.00*	0.0*	1.375	0.99964*
5		437.2	31.6	31.7	0.00*	0.0*	1.469	0.99993*
6		442.7	31.1	31.7	0.00*	0.0*	1.516	0.99987*
7		429.4						
8		441.8						
9		455.1						
10		387.0						
11		423.1						
12		431.9						
N		12	5	3	0	0	5	0
AVG.		424.8	31.5	31.5	31.49	-147.9	1.374	0.99998
S.D.		25.1	0.7	0.3	0.14	30.3	0.149	0.00035
C.V.		5.9	2.3	1.1	0.43	-20.5	10.865	0.03463
Min.		366.9	30.5	31.1	0.00	0.0	1.129	0.00000
Max.		455.1	32.3	31.7	0.00	0.0	1.516	0.00000

NASA

Report Documentation Page

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15. Supplementary Notes

16. Abstract Pyrolytic boron nitride (PBN) was deposited on Nicalon NL 202 silicon carbide yarns at 1000-1200°C with the goal of improving the resistance of the Nicalon to deterioration in an aerodynamic environment at temperatures up to 1000°C. Continuous PBN coatings were obtained by reacting boron trichloride and ammonia on the Nicalon as it was passed through a pilot-plant-sized CVD furnace. Most of the coatings were made at 1080°C to minimize thermal degradation of the Nicalon.

The coated yarns were characterized by weight per unit length, tensile strength and modulus, scanning electron microscopy (SEM), and scanning Auger microscopy (SAM). PBN coating thicknesses ranged from 0.1-0.7 micron, and the coatings were fairly uniform along the length of the yarn and between fiber bundles. The PBN-coated Nicalon was as strong as the as-received Nicalon and showed good resistance to oxidation in air up to 800°C, but the properties were degraded after air oxidation at 1000°C.

To control loose filaments during weaving, the PBN-coated Nicalon was wrapped with 30-denier rayon. Quantities of plain-weave and 12-satin harness-weave cloths were prepared from the PBN-coated yarn. Samples of cloth made from the PBN-coated Nicalon were sewn into thermal insulation panels for tests of high-temperature performance.

Thicker PBN coatings and coatings with a more oxidation resistant outer layer will be required to minimize degradation of the Nicalon in an aerodynamic environment at 1000°C. An alloyed PBN coating and/or more moisture stable outer coating may also be needed to improve the resistance of the PBN coating to reaction with atmospheric moisture.

17. Key Words (Suggested by Author(s)) Pyrolytic Boron Nitride Coatings Nicalon Yarn and Cloth Thermal Insulation Panels Oxidation Resistance and Moisture Stability	18. Distribution Statement UNCLASSIFIED-- UNLIMITED

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price
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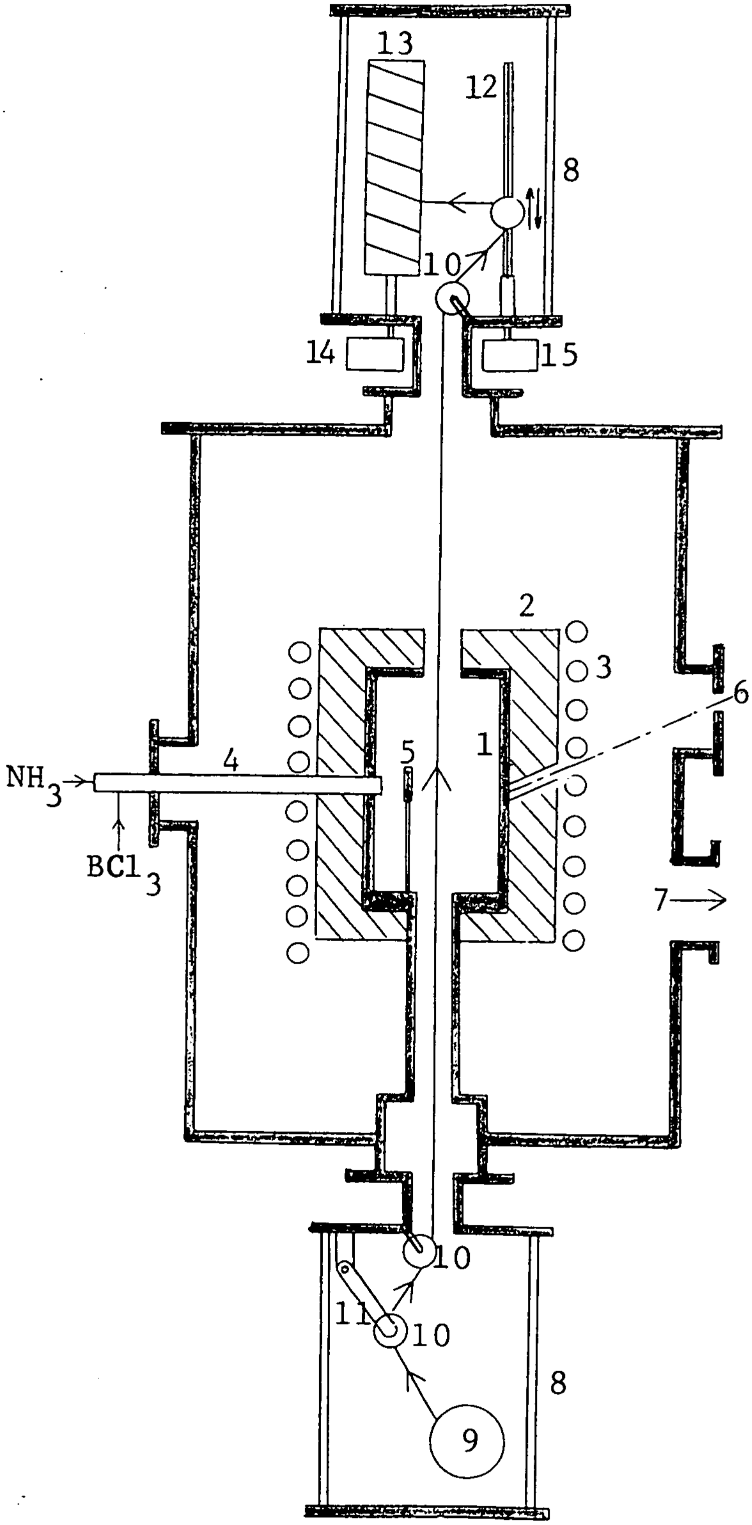
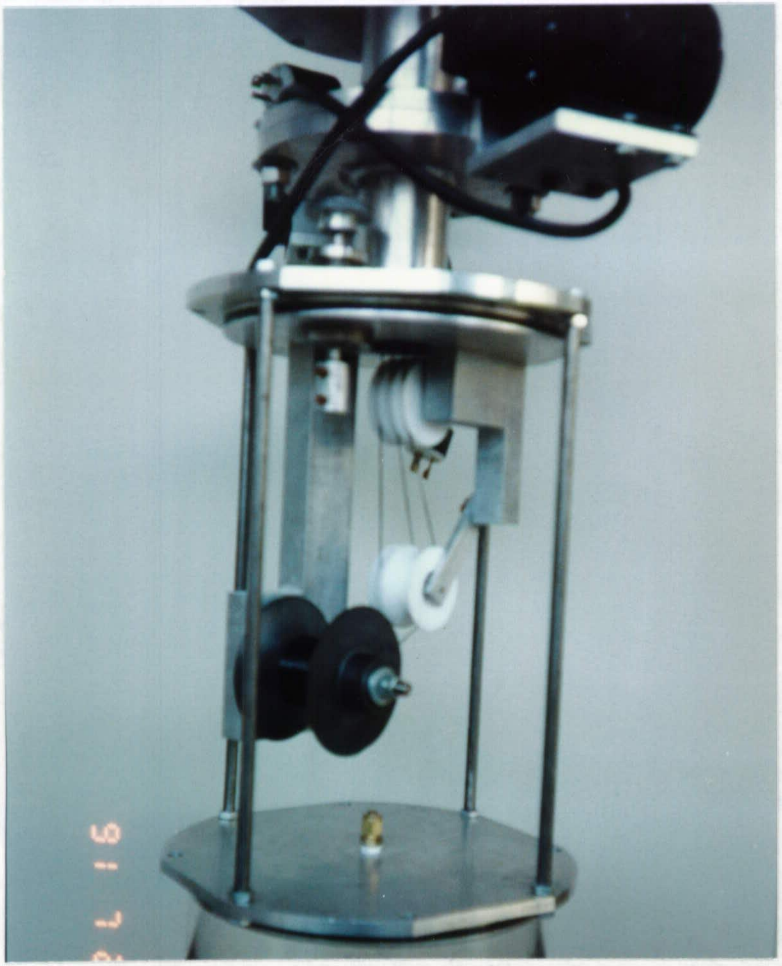
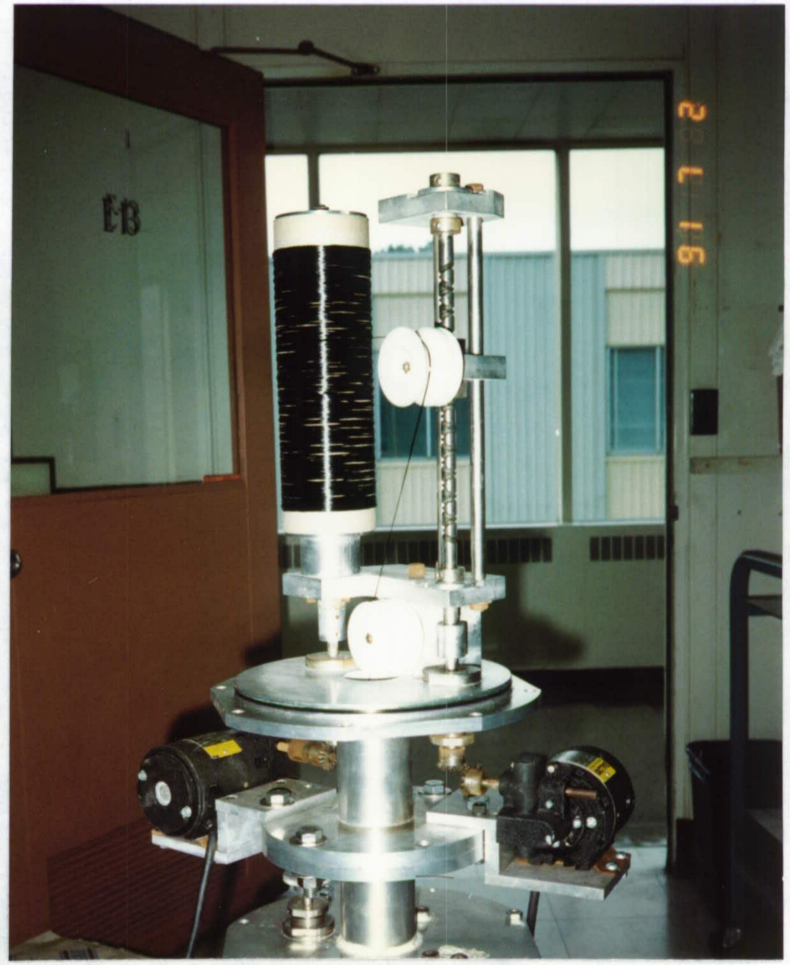


1. Graphite Susceptor Enclosing Deposition Chamber
2. Graphite felt Thermal Insulation
3. Induction Heating Coil
4. Two-Gas Injector
5. Gas Deflector
6. Sight Hole for Optical Pyrometry
7. Exhaust to Vacuum Pumps
8. Vacuum Covers for Yarn Feed and Collection Apparatus
9. Yarn Feed Spool
10. Teflon Pulleys
11. Yarn Tension Indicator
12. Reversing Actuator
13. Yarn Collecting Spool
14. Motor Drive for Yarn Collecting Spool
15. Motor Drive for Reversing Actuator

Figure 2. Arrangement for Continuous Yarn Coating in Pilot Plant CVD Furnace.



(a) Feed Spool and Pulleys
Three Yarn Passes Through Furnace



(b) Collecting Spool and Pulleys
Single Yarn Pass Through Furnace

Figure 3. Arrangement for Continuous Yarn Coating with PBN (Vacuum Covers Removed).

(a) Collecting Spool and Pulleys
Three Yarn Passes Through Furnace

(b) Collecting Spool and Pulleys with
Vacuum Cover in Place

Figure 4. Arrangement for Continuous Coating of Nicalon Yarn with PBN.

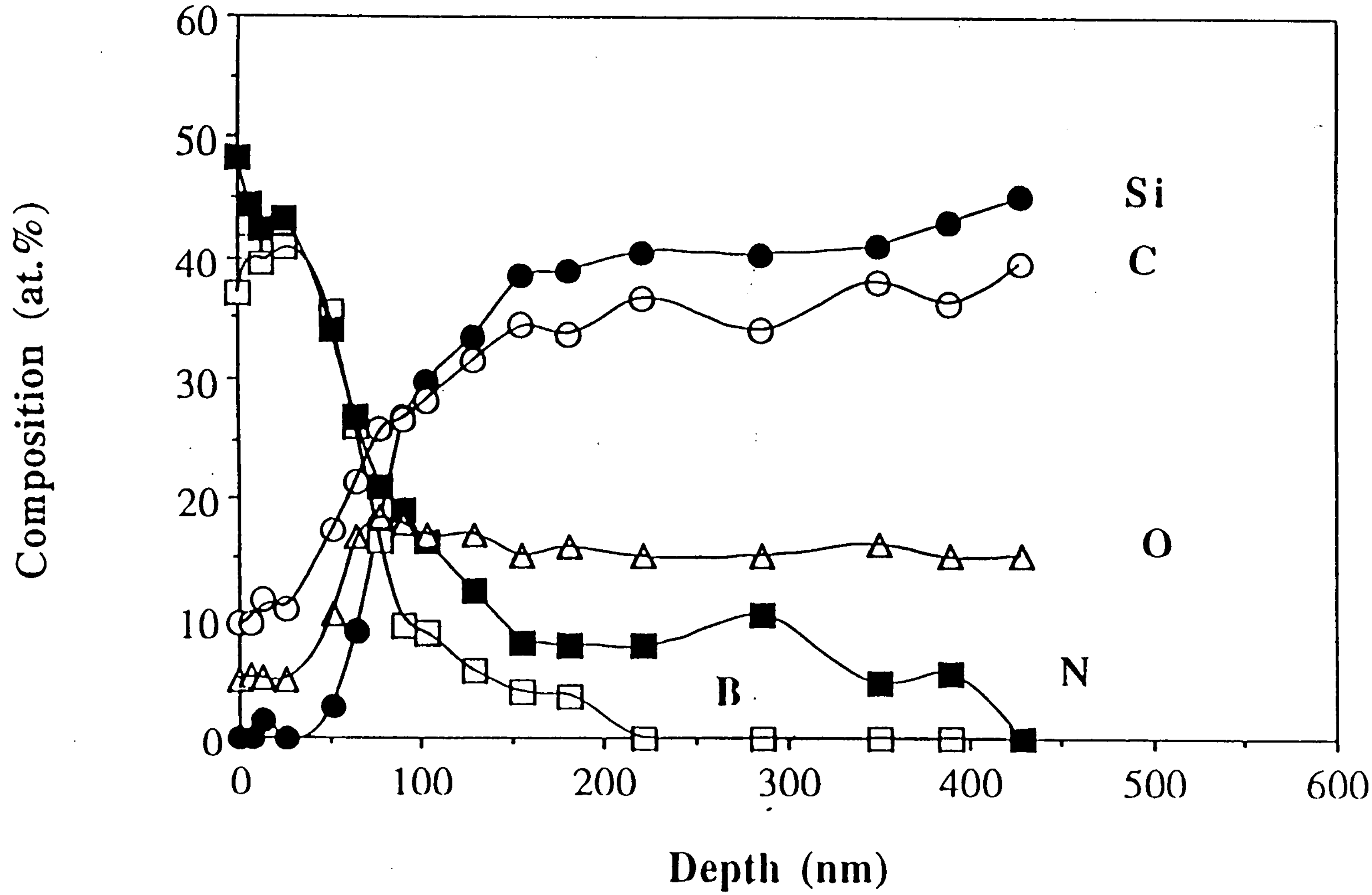


Figure 5. Scanning Auger composition depth profile of PBN-coated Nicalon sample 9028

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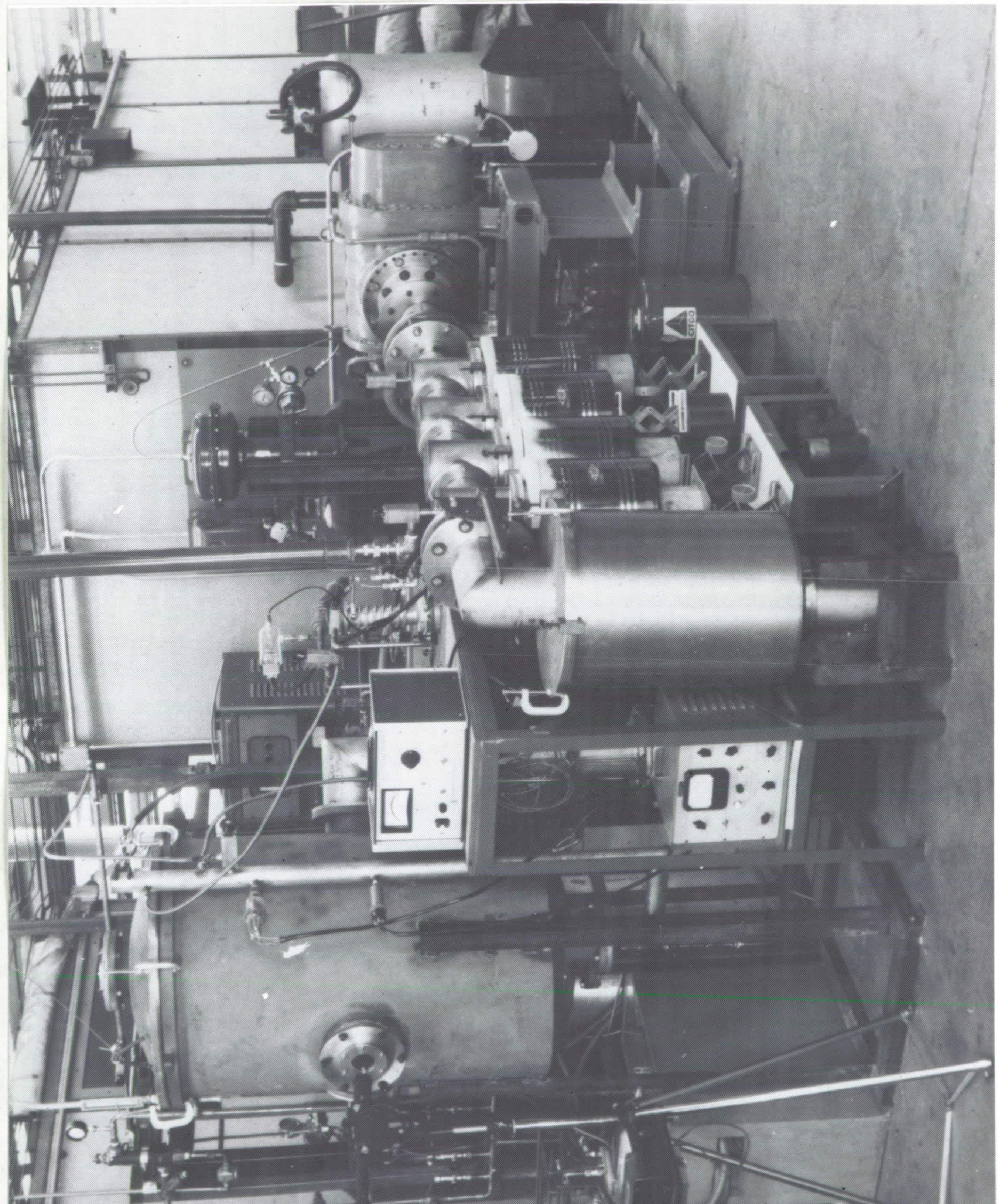

Figure 1. Pilot Plant CVD Facility.