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# INTERFACIAL STUDIES OF CHEMICAL VAPOR INFILTRATED (CVI) CERAMIC MATRIX COMPOSITES

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Prepared by  
J. J. Brennan

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The objective of this program was to investigate the fiber/matrix interfacial chemistry in CVI SiC matrix composites utilizing Nicalon SiC and Nextel 440 mullite fibers and to determine how this interface influences composite properties such as strength, toughness, and environmental stability. The SiC matrix was deposited using three different reactants; methylchlorosilane (MDS), methyltrichlorosilane (MTS), and dimethylchlorosilane (DMDS). The fiber/matrix interface was tailored by means of introducing a carbon, BN, or carbon rich SiC interfacial layer.

It was found that applying a carbon interfacial layer to either NICALON or Nextel 440 fibers prior to deposition of SiC resulted in a weakly bonded interface that imparted toughness to a CVI matrix composite through its ability to deflect matrix cracks. This carbon layer can be applied either deliberately by the decomposition of methane or by utilizing an argon flushing gas in the reactor that apparently interrupted the normal deposition of SiC from silane precursors and instead allowed carbon to deposit. It was found that this carbon interfacial layer, no matter how it was deposited, was not oxidatively stable at elevated temperatures, leading to severe degradation of composite properties. *JK*

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13. Abstract (cont.)

Attempts to produce an interfacial layer of carbon rich SiC (actually a mixture of carbon and SiC) that, from earlier work, appeared to act as a relatively oxidatively stable yet weakly bonded interface, were not successful. While this type of material could be obtained in bulk samples utilizing MDS plus methane additions, it could not be reproducibly obtained when deposited in thin coating form on fibers. However, studies that utilized BN coated NICALON fibers with relatively thin (~1 mm) CVD SiC overcoatings indicated that the BN layer remained intact during the SiC deposition, resulting in relatively weak bonding between the fiber and the deposited SiC. The RT flexural strength of CVI SiC/NICALON fiber composites with BN interfacial layers was quite high [66 ksi (455 MPa)] with typical composite non-linear fracture behavior. Tests were conducted on these composites in flowing oxygen at temperatures from 550° to 1200°C and showed that no degradation in composite strength occurred up to 1000°C with only moderate strength loss after 1200°C exposure. This was in stark contrast to CVI SiC matrix/NICALON fiber composites with carbon interfacial layers that exhibited severe strength loss after similar exposures.

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FINAL REPORT

Contract N00014-87-C-0699

REPORTED BY

  
J. J. Brennan

APPROVED BY

  
K. M. Prewo, Manager of  
Materials Sciences

DATE 3/31/90

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## SUMMARY

The main objective of this program was to investigate the fiber/matrix interface in chemically vapor infiltrated (CVI) composites and to understand how the microstructure and chemistry of this interface influence composite properties such as strength, toughness, and environmental stability. The long range goal of this investigation was to develop a CVI ceramic matrix composite that combined toughness and strength and could operate under stress in oxidizing conditions to temperatures approaching 1300°C. The CVI matrix system studied during the first year of this contract consisted of SiC deposited from three different reactant gases; methylchlorosilane (MDS), methyltrichlorosilane (MTS), and dimethylchlorosilane (DMDS). The fibers utilized were Nicalon SiC and Nextel 440 mullite. During the second year of the program, the emphasis was concentrated on the study of utilizing carbon rich SiC and BN fiber coatings as oxidatively stable yet weakly bonded interfacial layers. The chemistry and microstructure of the fiber/matrix interfacial region were studied through the use of scanning electron microscopy (SEM) of fracture surfaces, scanning Auger multiprobe (SAM) analysis of the elemental composition of the interface from depth profiles of fiber surfaces and matrix troughs that lie on composite fracture surfaces, and transmission electron microscopy (TEM) of polished surface replicas and ion beam thinned foils.

A number of CVI SiC matrix composites were fabricated and analyzed. It was found that composites utilizing Nicalon or Nextel 440 fibers that contained a thin carbon interfacial layer, whether deliberately or inadvertently deposited, exhibited high toughness and strength due to the weakly bonded carbon interface's ability to deflect matrix cracks. However, this type of interface was not oxidatively stable and, when oxidized, lead to severe degradation of composite properties.

From studies of deposited SiC layers on Nicalon and Nextel 440 fibers utilizing MDS and MTS precursors, it was found that the use of an argon flushing gas in the reactor prior to the introduction of the reactant gases initially disrupted the deposition of SiC such that a thin carbon layer formed on the surface of the fibers prior to SiC deposition. While this situation resulted in a tough composite structure, the lack of oxidative stability, as mentioned previously, was undesirable. Overcoating this type of composite structure with CVD SiC to seal the fiber/matrix interface is practiced, but the likelihood of cracking in the coating is high, especially under stress, so that this approach is not considered to be a long term solution.

When hydrogen was used as the reactor flushing gas, no carbon interfacial layer was formed and the deposited SiC bonded very strongly to both Nicalon and Nextel 440 fibers, resulting in weak and brittle composites. However, the deposited SiC appeared to be more consistent in composition and contain less oxygen.

From studies of the bulk composition of SiC deposited on carbon plates utilizing MDS, MTS, and DMDS precursors, it was found that both MTS and DMDS could controllably yield compositions that ranged from very silicon rich to essentially stoichiometric SiC, depending on the hydrogen carrier gas to silane flow rate ratios. The utilization of MDS as a precursor to SiC appeared to yield more consistent depositions with compositions ranging from stoichiometric to slightly carbon rich SiC. The addition of large amounts of methane (CH<sub>4</sub>) during the MDS deposition appeared to yield a product that was even more carbon rich (56-60 at %). Previous results on a different program indicated that a carbon rich SiC coating on Nicalon fibers (most likely a mixture of SiC + C) did not appear to bond very well to the fibers. It is likely that a coating of this composition would exhibit relatively good oxidative stability, especially when compared to the carbon interfacial layers.

Thus, the emphasis for the second year of this investigation was focused on the feasibility of utilizing a carbon rich SiC interfacial layer for both Nicalon and Nextel 440 fiber composites fabricated by CVI. In addition to carbon rich SiC as a weakly bonded, crack deflecting, oxidatively stable interface, the application of a CVD BN coating on the fiber surfaces prior to deposition of the SiC matrix was studied.

Attempts to produce an interfacial layer of carbon rich SiC (actually a mixture of carbon and SiC) that, from earlier work, appeared to act as a relatively oxidatively stable yet weakly bonded interface, were not successful. While this type of material could be obtained in bulk samples utilizing MDS plus methane additions, it could not be reproducibly obtained when deposited in thin coating form on fibers. The reasons for this probably have more to do with the type of gas management system utilized for this work than with the inherent thermodynamics and kinetics of the deposition process. If better deposition equipment with accurate flow controls and shorter lines from the evaporator to the reaction chamber were utilized, this approach to interface control in these types of composites could result in improved properties, especially inherent high temperature oxidation resistance.

The other approach to obtaining an oxidatively stable yet weakly bonded interface in CVI SiC matrix composites that was investigated consisted of applying BN coatings to the fibers prior to deposition of the SiC. Initial studies that utilized BN coated NICALON fibers from Synterials with

relatively thin (~1  $\mu\text{m}$ ) CVD SiC overcoatings indicated that the BN layer remained intact during the SiC deposition, although some diffusion of Si and possibly C into the BN appeared to occur, resulting in relatively weak bonding between the fiber and the deposited SiC. Additional NICALON fibers that were coated with BN at UTRC were then utilized to produce actual small CVI SiC composite rings. The RT flexural strength of the resultant composites was quite high [66 ksi (455 MPa)] with typical composite non-linear fracture behavior. Tests were conducted on these composites in flowing oxygen at temperatures from 550° to 1200°C and showed that no degradation in composite strength occurred up to 1000°C with only moderate strength loss after 1200°C exposure. This is in stark contrast to CVI SiC matrix/NICALON fiber composites with carbon interfacial layers that exhibited severe strength loss after similar exposures. Humidity testing also indicated that the BN interfacial layers were stable under this type of environment.

Although the results obtained on this program utilizing the BN interfacial layers can only be considered preliminary, they are sufficiently promising to warrant additional investigation. In order to fully explore the potential of this system, it is obvious that further processing studies must be done to scale up the size of the fabricated composites, as well as comprehensive elevated temperature mechanical and thermal property measurements.

## I. INTRODUCTION

During the past decade, the interest in ceramic matrix composites for high temperature structural applications, especially for use in heat engines, has increased to the point that a large number of industrial organizations as well as universities and government laboratories throughout the world are actively performing research into a myriad of different systems and different processing procedures for these materials. Among the types of ceramic matrix composites under investigation are whisker reinforced glasses and glass-ceramics<sup>1</sup> as well as whisker reinforced crystalline ceramics<sup>2-18</sup>, and continuous fiber reinforced ceramics produced by methods that include hot-pressing of glasses and glass-ceramics<sup>19-33</sup>, sol-gel infiltration and pyrolysis of ceramics<sup>34</sup>, polymer precursor infiltration and pyrolysis<sup>35</sup>, reactive oxidation of metals<sup>36</sup>, reactive sintering<sup>37</sup>, and chemical vapor infiltration (CVI) of silicon based ceramics<sup>38-49</sup>.

It has been found in all of the above-mentioned ceramic composites that in order to achieve high strength and, in particular, high toughness, the bonding at the fiber/matrix interface must be controlled such that bonding is strong enough to allow load transfer from the matrix to the fibers under stress but weak enough so that an advancing matrix crack can be deflected by the fibers. In addition, the nature of the fiber/matrix interface must include resistance to oxidation at elevated temperature as well as resistance to other environmental effects.

For the past decade, research at United Technologies Research Center (UTRC) in the area of ceramic matrix composites has centered on systems based on the reinforcement of glass and glass-ceramic matrices with Nicalon polymer derived SiC fibers. In the past few years, this research has concentrated on the study of the fiber/matrix interface and the relationship of the interfacial chemistry and morphology to the composite mechanical and thermal properties<sup>28,31</sup>. The characterization of the interfaces in these composites has been accomplished primarily by a combination of scanning electron microscope (SEM) observations of composite fracture surfaces, transmission electron microscope (TEM) replica and thin foil analysis, and scanning Auger microprobe (SAM) analysis of composite fracture surfaces. This work has enabled a greater understanding to be reached of the reactions that occur and the phases formed in these systems and has led to the successful development of strong, tough, and oxidatively stable glass-ceramic matrix/Nicalon fiber composite systems for use to temperatures approaching 1000°C. While the attainment of much higher use temperature glass-ceramic matrices has been demonstrated, the inherent formation of a carbon rich interfacial layer between the Nicalon fibers and the glass-ceramic matrices during fabrication make the oxidative stability of these composites difficult to

achieve in the temperature range of 1000-1300°C. Work in this area is being carried out at UTRC, primarily through the application of fiber coatings prior to composite fabrication. However, with the recent successful development of CVD coatings for carbon/carbon composites at UTRC<sup>50</sup>, it was decided to use the knowledge gained in this area with the previous experience in fiber/matrix interfacial analysis of glass-ceramic matrix composites and apply them to a somewhat different class of ceramic matrix composites.

One of the most promising routes to the development of ceramic matrix composites that could potentially exhibit use temperatures to 1300°C or higher is that of chemical vapor infiltration (CVI) of silicon carbide or silicon nitride into fibrous preforms. Work in this type of ceramic matrix composite, pioneered by researchers at SEP in France<sup>43</sup>, is now being carried out at a number of institutions<sup>38-49</sup>. Most of this work has concentrated on SiC matrices, since the deposition of crystalline silicon nitride can only be obtained at temperatures that are higher than the maximum stability temperature of fibers such as Nicalon. While CVI SiC reinforced with carbon or Nicalon fibers is now a commercially available ceramic composite from SEP in France and its licensee, DuPont in the U. S., very little work has been reported on the fiber/matrix interfacial chemistry in these systems, with the exception of some recent research from Oak Ridge National Lab (ORNL)<sup>51,52</sup>. Since the nature of the fiber/matrix interface plays such an important role in the strength, toughness, and oxidative stability of these composites, it was decided to investigate the interfacial chemistry in the CVI SiC matrix composite systems reinforced with both Nicalon SiC and Nextel 440 mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) fibers, with and without fiber coatings. TEM and SAM analyses of the fiber/matrix interfacial regions in these systems constituted a major portion of this work.

## II. BACKGROUND

The use of gaseous precursors as a means of depositing a silicon carbide or silicon nitride ceramic matrix around arrays of oxide, carbide, and carbon fibers has been under investigation at UTRC at a rather low level of effort for several years. From the results of this preliminary work, it became obvious that control of the deposition parameters and an understanding of the role of the fiber/matrix interface chemistry would be necessary in order to fabricate in a reproducible manner composites that exhibited high strength and toughness. It was with this basic goal that the current ONR program was initiated. The first year's work under this program (9/87-9/88) was reported in detail in an annual report<sup>53</sup>. The highlights from this report are described below.

The CVI matrix system studied during the first year of this contract consisted of SiC deposited from three different reactant gases: methylchlorosilane (MDS), methyltrichlorosilane (MTS), and dimethylchlorosilane (DMDS). The fibers utilized were Nicalon SiC and Nextel 440 mullite. The chemistry and microstructure of the fiber/matrix interfacial area were studied through the use of scanning electron microscopy (SEM) of fracture surfaces, scanning Auger multiprobe (SAM) analysis of the elemental composition of the interface from depth profiles of fiber surfaces and matrix troughs that lie on composite fracture surfaces, and transmission electron microscopy (TEM) of polished surface replicas and ion beam thinned foils.

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When hydrogen was used as the reactor flushing gas, no carbon interfacial layer was formed and the deposited SiC bonded very strongly to both Nicalon and Nextel 440 fibers, resulting in weak and brittle composites. However, the deposited SiC appeared to be more consistent in composition and contain less oxygen.

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Thus, the emphasis for the continuation of this investigation was focused on the feasibility of utilizing a carbon rich SiC interfacial layer for both NICALON and Nextel 440 fiber composites fabricated by CVI. In addition to carbon rich SiC as a weakly bonded, crack deflecting, oxidatively stable interface, the application of a CVD BN coating on the fiber surfaces prior to deposition of the SiC matrix was also studied. BN coatings on Nicalon fibers have been found recently to yield tough, strong, and relatively oxidatively stable glass-ceramic matrix composites at UTRC.

### III. TECHNICAL DISCUSSION

#### A. Materials

The SiC fiber utilized for this program is that produced by Nippon Carbon Co. in Japan and distributed in the U. S. by Dow Corning Corp., Midland, Mich., under the trade name "Nicalon". The fiber used for this program is the lower oxygen level "Ceramic Grade". The fibers were obtained on spools of continuous length (500 m) tows of 500 fibers/tow with an average fiber diameter of 14  $\mu\text{m}$ . The average tensile strength and elastic modulus of this fiber, as measured at UTRC, is 2400 MPa (350 ksi) and 193 GPa ( $28 \times 10^6$  psi), respectively. The mullite fiber (Nextel 440) was obtained from 3M Corp., Minneapolis, Minn. on spools of continuous length tows of 780 fibers/tow. According to 3M data, this fiber has a diameter of 7-11  $\mu\text{m}$  (somewhat oval shaped), a tensile strength of 1400-2000 MPa (200-300 ksi), and an elastic modulus of 205-240 GPa ( $30\text{-}35 \times 10^6$  psi). Some of the deposition runs utilized ATJ carbon plates as the substrate material.

The BN fiber coatings were either deposited in-house or by Synterials, Inc., Herndon, VA. Both sources of BN coatings utilized the decomposition of boron trichloride plus ammonia at temperatures of  $\sim 1000^\circ\text{C}$  for the deposition of BN on the fibers.

Three different silane precursors were utilized during the first year of the program to deposit SiC. Methylchlorosilane (MDS),  $\text{CH}_3\text{SiHCl}_2$ , has long been used at UTRC to deposit SiC since its high vapor pressure (B.P. =  $41.5^\circ\text{C}$ ) has allowed the transference of the saturated carrier to the reactor over long distances without heated lines. Methyltrichlorosilane (MTS),  $\text{CH}_3\text{SiCl}_3$  (B.P. =  $66.4^\circ\text{C}$ ), is used extensively by other researchers to deposit SiC and was also used in this program along with dimethyldichlorosilane (DMDS),  $(\text{CH}_3)_2\text{SiCl}_2$  (B.P. =  $70.5^\circ\text{C}$ ). All three of the silane precursors were obtained from Alfa Products, Danvers, Mass. During the last phase of the program as reported here, only MDS was utilized as the SiC precursor, sometimes in combination with methane ( $\text{CH}_4$ ) as a source of excess carbon.

#### B. Sample Fabrication

The reactor utilized to deposit the CVD SiC coatings and to form the CVI SiC matrix composites was described in detail in the previous annual report on this program<sup>53</sup>. Basically, the reactant gases are introduced into a hollow quartz reaction cylinder at one end and the exhaust gases are removed at the other end. If the reaction is being run at reduced pressure, a vacuum pump is attached to the exhaust line. The reactor has removable water cooled copper plates at

each end. Pieces of carbon placed on the bottom of the reactor hold a hollow carbon cylindrical susceptor which is heated by an RF coil. When CVD coating either flat carbon plates or tows of fibers, carbon rods are inserted into the center of the susceptor to hold the samples being coated. When CVI matrix composites are being fabricated, the fiber tows are wound around a solid carbon cylinder that is placed in the center of the reactor without utilizing the normal hollow carbon susceptor. Thus, the wound carbon rod is heated directly producing a temperature gradient from the inside of the fiber winding to the outside. This allows deposition of the SiC to occur on the inside of the windings first so that the wound tows are not sealed off on the outside. The water cooled RF coil around the reactor also adds to the temperature gradient.

### C. CVD SiC on Carbon Substrate Studies (MDS + CH<sub>4</sub>)

During the first year of the program, CVD SiC layers were applied to small flat carbon substrates utilizing MDS, MTS, and DMDS precursors. After deposition, all samples were depth profiled in the SAM from the coating surface inward to a depth of 6000Å. The analysis of the bulk composition of each coating was taken at a depth where surface effects such as oxide formation would be negligible, normally 5000Å. From the results of these experiments, it appeared that MDS was more controllable than the others as far as obtaining a deposit that was somewhat carbon rich. Thus, additional experiments were performed utilizing MDS with a hydrogen carrier plus methane (CH<sub>4</sub>) additions in order to attempt to reproducibly obtain a coating with a composition of ~60%C, 40%Si, which, as mentioned earlier, may be useful as a relatively weakly bonded but oxidation resistant fiber coating.

As mentioned in the previous annual report on this program, the addition of methane to MDS at low flow rates (100 cc/min) did not affect the composition of the SiC deposited by a significant amount. All of the conditions explored resulted in a bulk SiC composition of from 53-57% C. Increasing the flow rate of the added methane gas from 100 cc/min to 600 cc/min did appear to slightly increase the carbon content but also lead to some inconsistent deposition, as shown in Fig. 1 for run #13-8-3A for a methane flow rate of 200 cc/min. Figure 2 shows the SAM depth profile and surface morphology for the 600 cc/min methane addition run. In general, while some variations in composition were noted, the coating was extremely low in oxygen and approached the 60% C content that has been set as an initial goal for composite fabrication.

It was thus decided to explore in greater detail the MDS plus methane addition system, with emphasis on lowering the hydrogen carrier gas flow rate while keeping the methane flow rate at 600 cc/min in order to attempt to increase the amount of co-deposited carbon in the SiC. From the results of these tests, as shown in Table I, it is apparent that this approach did not yield higher

carbon containing SiC coatings. Lowering the H<sub>2</sub>/MDS flow rate from 100cc/min to 50 cc/min actually reduced the amount of excess carbon in the coating, as shown in the SAM depth profile for run #13-10-17 in Fig. 3. Reducing the H<sub>2</sub>/MDS flow rate even further, to 25 cc/min, resulted in even more stoichiometric SiC being deposited, except for an abrupt change in composition near the end of the run to almost pure carbon (Fig. 4). The reason for this carbon surface layer formation is not clear, but may have been caused by the inadvertent closure of the H<sub>2</sub>/MDS flow prior to shutting off the methane flow. In any case, it appears that although the composition varies somewhat, the addition of methane at 600 cc/min to MDS with a hydrogen carrier gas flow rate of 100 cc/min produces the optimum amount of excess carbon in the deposited SiC. It was thus decided to perform additional deposition runs utilizing these conditions on NICALON and Nextel fiber tows.

#### **D. CVD SiC Coated Fiber Studies (MDS + CH<sub>4</sub>)**

The initial experiment in depositing carbon rich SiC on fibers was done utilizing Nextel 440 mullite fibers, with a run time of 1 hr (Run #13-10-12A). Typical fractured ends of the coated fibers are shown in Fig. 5. From this figure, it can be seen that the coating deposition was very nonuniform, with most of the fibers exhibiting a very thick SiC deposit on one side and a very thin deposit on the opposite side. It is obvious that as the coating deposition became a certain thickness, the paths for the reactant gases between fibers in close proximity to one another became blocked by the SiC deposit. There appeared to be some type of interaction between the coating and the Nextel fibers, however, no SAM analysis of this run was done due to the thickness of the coating. In general, the bonding between the coating and Nextel fiber appeared to be quite strong.

An identical coating run (#13-10-13) was performed on NICALON fibers, with rather similar results, as shown in Fig. 6. In this case, the coating was smoother in appearance and more uniform in thickness, but still appeared very strongly bonded to the fibers. In order to obtain a SiC coating that was thin enough to perform SAM analysis on, the above run was repeated on NICALON fibers for a total run time of 8 minutes, with the other coating parameters supposedly held constant. The results of this run (#15-12-9A) are shown in Fig. 7 and tabulated in Table II, along with other runs utilizing fiber substrates. From Fig. 7, it can be seen that the coating thickness for this run was ~2 $\mu$ m, with quite weak bonding between the coating and fibers. From the high magnification SEM in Fig. 7, it appears that a separate very thin coating exists at the SiC coating/NICALON fiber interface. Scanning Auger analysis of a coated fiber from this run was performed, as shown in Fig. 8, and indicated that, while the surface region of the coating (~500 $\text{Å}$ ) was somewhat carbon rich, the bulk of the SiC coating appeared to be very close to stoichiometric SiC. No indication of a separate thin coating between the SiC coating and the fiber was found,

however, the analysis steps at this depth into the coating were at 5,000Å sputtering intervals, so a thin coating such as that shown in Fig. 7 could easily have been missed.

Another deposition run under the above conditions on NICALON fibers was performed utilizing a run time of only 4 minutes (#15-1-11A). SEM analysis of fractured fiber ends from this run, as shown in Fig. 9, again shows a quite weakly bonded coating with a thin interlayer evident between the SiC layer and the fiber surface. From SAM analysis of a coated fiber from this run, as shown in Fig. 10, a very carbon rich (95%) layer of ~1000-2000Å in thickness exists between the NICALON fiber and a slightly carbon rich SiC layer of ~1 μm thickness that grades to a very carbon rich surface. This almost pure carbon interlayer undoubtedly also existed in the previous run #15-12-9A and accounts for the weak coating/fiber interfacial bonding observed for these two runs.

An additional deposition run was done utilizing the exact conditions of run #15-1-11A but attempting to make sure that the MDS/H<sub>2</sub> and CH<sub>4</sub> flows were introduced into the reaction chamber at the same time and that they were shut off at the same time. In addition, although all of the previous runs utilized a hydrogen flush prior to the introduction of the reactant gases, this procedure was monitored more closely. The result of this run was that the carbon rich surface layer found on the deposited SiC of run #15-1-11A was eliminated, but the thin carbon interlayer between the NICALON fiber and the SiC was still present.

At this point, it was decided that with the SiC deposition equipment available to this program, the controlled deposition of thin carbon rich SiC coatings on small diameter fibers such as NICALON was not possible utilizing the procedure that had been successful in depositing these types of coatings on flat carbon plates. The system of flow control valves was just not accurate or sensitive enough to ensure the proper introduction and mixing of the various precursor gases in the reaction chamber. It was thus decided to concentrate efforts under this program to the creation of a weakly bonded but oxidatively stable interface in fiber reinforced CVI SiC matrix composites through the use of BN fiber coatings.

#### **E. CVD SiC Over BN Coated Fibers**

A series of experiments were performed utilizing BN coated Nextel and NICALON fibers, overcoated with CVD SiC from H<sub>2</sub>/MDS with no added CH<sub>4</sub>. The results of these experiments are given in Table III.

The first experiment was conducted on BN coated Nextel 440 mullite fibers, with the BN coating being deposited at UTRC. From the work conducted previously<sup>53</sup>, it was found that CVD SiC deposited directly on Nextel fibers using a hydrogen flush resulted in a very well bonded SiC/Nextel fiber interface (Fig. 11). When the BN coated Nextel fibers were utilized, the bonding appeared to range from relatively well bonded to rather weakly bonded, as shown in Fig. 12 for run #13-10-11A. There also appears to be a reaction of some sort at the CVI SiC coating/Nextel fiber interface that may be a result of interaction between the deposited SiC and the BN coating. It is not clear from Fig. 12 exactly where the BN coating resides.

Figure 13 shows the SAM depth profile obtained from a BN coated Nextel fiber from run #BN-106. It can be seen that the fiber coating is not particularly smooth. The depth profile was obtained from the relatively smooth region in the center of the pictured fiber and indicates that the ~1  $\mu\text{m}$  thick coating is quite boron rich, with a significant amount of carbon in the near-surface region plus from 7-15% oxygen throughout the coating. As will be shown later, BN coatings from Synterials are also boron rich with significant oxygen content. Figure 14 shows SEM views of the same BN coated fiber bundle as that utilized for the SAM analysis in Fig. 13, taken from a different location. From this figure, it can be seen that many of the fibers are covered with large globs of deposited BN. These large uneven deposits of BN could very well be responsible for the apparent reaction zone seen in Fig. 12 between the fiber and CVD SiC coating. In any case, it was apparent that the UTRC process to fabricate CVD BN coated fibers was not sufficiently developed to produce the uniformly coated fibers necessary to carry out CVD SiC coating experiments. It was thus decided to utilize BN coated fibers from Synterials that had previously been used for glass-ceramic matrix composites and found to be quite uniform in coating thickness and morphology, at least until the UTRC BN coating process became better developed and capable of producing coated fibers of higher quality.

The SAM depth profile and coated fiber appearance for BN coated NICALON fiber lot #88-5-5 from Synterials is presented in Fig. 15. This coated fiber lot, which exhibited a measured UTS of 360 ksi (2480 MPa), consisted of a quite boron rich coating of ~2000Å in thickness that contained a significant amount of oxygen (15-20 at%), but essentially no carbon. A small amount of Al impurity was detected in the BN coating near the fiber surface. The coating thickness was very uniform with a very smooth surface morphology.

Four CVD SiC coating runs were done utilizing the Synterials BN coated NICALON fibers, as detailed in Table III. The first two runs, #13-10-11 and 13-11-7, were done at a CVD SiC run time of 1 hour utilizing MDS precursor from two different sources. The results of both of these runs were quite similar, as shown in the SAM depth profiles in Figs. 16 and 17, except that the SiC

coating morphology of run #13-11-7, which used an old tank of MDS, was quite rough and bumpy. All additional runs were done with the MDS source of run #13-10-11, which produced a smooth SiC fiber coating. The chemistry of the SiC coatings for both of the above runs consisted of relatively stoichiometric SiC with ~2% oxygen. Both coatings were on the order of 2-2.5  $\mu\text{m}$  in thickness. From these SAM depth profiles, it appears that the BN interlayers are present at the SiC/NICALON fiber interface, as evidenced by the relatively weakly bonded nature of the SiC to the fibers as can be seen in Fig. 16, but that some interdiffusion of Si and C into the BN has occurred during the SiC deposition run.

Samples of the CVD SiC over BN coated NICALON fiber (#13-10-11) were subjected to ion beam thinning and analyzed in the transmission electron microscope (TEM). Figure 18 shows a thinned region of the CVD SiC/BN/NICALON fiber interface and associated energy dispersive X-ray spectroscopy (EDX) patterns. From this figure, it can be seen that area A, the deposited SiC, is extremely fine grained or amorphous and gives an EDX pattern showing primarily Si with small amounts of O and C. The small amount of O detected correlates well with the SAM analysis, but the small amount of C detected is an artifact of this analysis method, in that carbon in the presence of Si is difficult to detect due to absorption of the carbon X-rays by the Si. This effect is noticed in area C, the NICALON fiber, as well. Area B, the BN interlayer, indicates the presence of B, N, O, Si, and also Al. All of these elements (in addition to C) were detected in the BN layer from the SAM analysis except for the Al. Aluminum was detected from the SAM analysis of the BN coated NICALON fiber (Fig. 15), however, and, as will be shown later, was detected from the SAM analysis of another CVD SiC/BN/NICALON run (#15-3-22).

Figure 19 shows another thinned region from sample #13-10-11 where it can be seen that the deposited SiC initially is very fine grained but then transitions to a somewhat coarser grained columnar structure at a distance of ~3000-4000 $\text{\AA}$  from the BN interlayer. This is visually very evident from the dark field micrograph taken from a region somewhat to the right of the bright field micrograph shown in Fig. 19A. It is also evident from the selected area electron diffraction (SAED) patterns shown in Fig. 19, as is the extremely fine grained to amorphous nature of the NICALON fiber and the BN interlayer.

Two additional CVD SiC deposition runs were performed on Synterials BN coated NICALON fiber. The first run (#15-1-18) was done under supposedly identical conditions as run #13-10-11, except the time of deposition was shortened to ~3 min in order to obtain a relatively thin SiC coating. As can be seen from the SAM depth profile of this run (Fig. 20), the thickness of the SiC coating was indeed thinner (~7000 $\text{\AA}$ ), but the deposited SiC was very carbon rich, especially at the beginning and end of the deposition run. The average tensile strength of 20 coated fibers

from this run was 243 ksi (1680 MPa), indicating some degradation in tensile strength due to the SiC deposition. The second run (#15-3-22) was also done with a deposition time of ~3 min but at a reduced hydrogen carrier gas flow rate of 25 cc/min. This yielded a thinner SiC coating, as anticipated, but the composition of this coating was even more carbon rich (Fig. 21) than the previous run. A small amount of Al was detected in the BN interlayer, as was also found for the as BN-coated sample (Fig. 15).

It is apparent that with the reactor and gas flow controls being used to deposit CVD SiC on fibers, attempting to deposit thin SiC coatings on fibers by reacting for very short periods of time results in uncontrolled stoichiometry of the coating. The reasons for this are not particularly apparent at this time, but it is obvious that this phenomenon does not occur during longer deposition runs, as seen from Figs. 16 and 17. No evidence of a carbon rich layer between the BN interlayer and the SiC coating could be seen in either the SAM depth profile or the TEM thin foil characterization of run #13-10-11 (Figs. 16, 18, and 19). The sputter rate for the SAM depth profiling in Fig. 16 was slowed down to 250Å increments as the BN layer was approached and no indication of high carbon content was found, which it surely would have been if indeed the carbon rich layer was present. It was thus decided to fabricate actual small CVI SiC composites utilizing BN coated NICALON fibers such that some mechanical property and oxidative stability data could be obtained and compared to other CVI SiC/NICALON fiber composites with carbon interfacial layers.

#### F. CVI SiC Matrix Composites

Two CVI SiC matrix composite rings were fabricated utilizing BN coated NICALON fibers. The method used to fabricate these rings was described previously, but basically consists of winding the BN coated NICALON tows around a carbon mandrel. The wound tows were then infiltrated utilizing the normal H<sub>2</sub>/MDS ratios and flow rates at a temperature of 1080°C for a total run time of 100 hrs. The infiltrated composites were then removed from the carbon mandrels, sliced into 0.2" (0.51 cm) wide rings and then cut into 90° ring segments.

The BN coated NICALON fibers were processed at UTRC in a batch reactor utilizing BC<sub>3</sub> + NH<sub>3</sub> at 1050°C in a 0.2 torr vacuum for various periods of time. Figure 22 shows examples of two of these runs (BN 255 and BN 257) that were processed for 10 min and 15 min, respectively. The BN coatings are quite smooth, with the BN 257 coating being obviously thicker than the BN 255 coating, but some coating thickness variation can be seen in the BN 255 coating. From SEM evaluation of a number of coated fibers from these and other runs, it was obvious that the BN coating thickness varied considerably from fiber to fiber within each run and even around the periphery of a single coated fiber. A continuous BN fiber coater, which is currently under development at UTRC, should result in much more uniform coating thicknesses.

The composition of the UTRC BN coatings, while somewhat variable from run to run, was found to be significantly closer to stoichiometric BN than the previously utilized Synterials BN coated fibers, although carbon and oxygen contamination was found to exist. The SAM depth profile for a BN coated NICALON fiber from run BN 255 is shown in Fig. 23. The rather thick (~1  $\mu\text{m}$ ) coating on this fiber consists of ~40-45% B and N, with ~5% O and from 5-12% C. Later BN deposition runs with a liquid nitrogen cold trap installed between the reaction chamber and the vacuum pump exhibited much reduced levels of carbon.

Both the BN 255 and BN 257 coated NICALON fibers were utilized to fabricate CVI SiC composite rings. A polished cross-section parallel to the fiber winding direction for composite #255 is shown in Fig. 24. From this figure, it can be seen that the infiltration of SiC between the wound fiber tows is quite good. It is also obvious that the BN coating on the fibers varies considerably in thickness, as previously discussed. Composite #257 was quite similar in appearance except that the average BN interlayer thickness was somewhat greater, although variations in thickness were also present.

Samples of composite #255 were prepared in the form of thin foils and examined in the TEM. Figure 25 shows the interfacial region of this composite and the associated selected area electron diffraction (SAED) patterns for the three constituents; NICALON fiber, BN interfacial layer, and CVI SiC matrix. The apparent gap between the BN and the NICALON fiber appears to be an effect of preferential thinning. It was noted that the BN layer and the NICALON fiber thinned much faster than the CVI SiC, consequently the SiC matrix was very seldom thin enough for adequate transmission of electrons. From the SAED patterns and the bright field/dark field pairs shown in Fig. 26, it is apparent that the NICALON fiber consists of extremely fine SiC crystallites while the BN layer consists of slightly larger, but still very fine, crystallites of BN. From the EDX patterns of Fig. 26, it is obvious that some Si has diffused into the BN layer, since the BN analysis was taken from the center of the quite thick (~1  $\mu\text{m}$ ) layer. Again, some carbon is also present in the BN layer and also, of course, in the CVI SiC and the NICALON fiber, but is next to impossible to detect by this method in the presence of Si. The transition of the deposited SiC from rather amorphous next to the BN layer to a fine grained submicron columnar nature farther out into the matrix is evident in the TEM micrographs of Fig. 27.

As mentioned previously, 90° segments of these composite rings were cut so that mechanical property measurements could be obtained. The 3-pt flexural strength of these cut segments was obtained from the equations for curved beams presented in Ref. 55. Figure 28 shows the RT fracture surface of a cut segment of composite #257 that exhibited a flexural strength of 66 ksi (455 MPa). While not a "brushy" type of fracture surface that is often obtained in glass-ceramic

matrix/NICALON fiber composites, a significant amount of fiber pullout and fiber/matrix debonding can be seen, with the BN interlayer obviously contributing to this fracture behavior and visible in the high magnification SEM of Fig. 28. The fracture behavior of this composite is similar to, but not quite as fibrous as, that observed previously<sup>53</sup> for carbon coated NICALON fiber/CVI SiC matrix composites obtained from Oak Ridge National Lab (ORNL)<sup>56</sup>, as shown in Fig. 29. Both of these composites with either BN or carbon interfacial layers are very different in strength and fracture behavior when compared to a CVI matrix/NICALON fiber composite from ORNL with no interfacial layer between the CVI SiC and the NICALON fiber (Fig. 30). It is very apparent that the presence of a weakly bonded carbon interface or a relatively weakly bonded BN interface drastically alters the fracture behavior and increases the strength and toughness of these types of composites.

As was discussed in a previous report on this program<sup>53</sup>, carbon interfaces have also been found to result in strong and tough composites in glass-ceramic matrices reinforced with NICALON and other fibers (Dow Corning's HPZ and MPDZ)<sup>57</sup>. However, the presence of this type of interface can lead to severe degradation of composite properties when the composite is exposed to oxidizing environments at elevated temperatures. This has also been found to occur for CVI SiC matrix composites with carbon interfaces, as shown for an ORNL composite with a carbon interfacial layer that has been exposed to flowing oxygen at 1000°C for 70 hrs (Fig. 31). From this figure, it can be seen that while the fracture surface of this composite is still quite fibrous in appearance, the strength has been reduced drastically to 11.2 ksi (77 MPa) from the previous 55 ksi (380 MPa). At high magnification, it is apparent that the carbon layer has oxidized away leaving a gap between the fibers and matrix. While this gap results in a "tough" fracture appearance, load transfer from the matrix to the fibers cannot occur, thus resulting in a very weak composite.

In contrast to the above ORNL composites and previous CVI SiC/NICALON fiber composites fabricated at UTRC<sup>53</sup> with carbon interfacial layers, the CVI SiC matrix composites (#255 and 257) fabricated with BN interfacial layers exhibited excellent oxidative stability at high temperatures. Cut samples of these composites with no SiC overcoating were subjected to flowing oxygen exposure for 100 hrs at temperatures of 550°, 1000°, and 1200°C, and then tested at RT in flexure. In addition, a sample was also subjected to humidity testing at 120°F, 100% RH, for 70 hrs. This is the standard humidity test for inhibited C/C composites that contain boron additives. From the results of these tests, as shown in Table IV along with the oxidation results from previous composites with carbon interfacial layers, no degradation in flexural strength for the BN interlayer composites occurs as a result of humidity testing or oxidative exposure to 1000°C. A relatively small decrease in strength (~20%) was observed after oxidative exposure to 1200°C. These

results for the BN interlayer composites are in direct contrast to the very poor results obtained for the carbon interlayer composites as a result of high temperature oxidative degradation.

Figure 32 compares the crosshead displacement vs stress curves for the CVI SiC matrix/BN coated NICALON fiber composite #257 in the as-fabricated condition and after exposure to flowing oxygen at 1000°C for 100 hrs. From these curves it can be seen that both composites exhibit linear behavior to a stress of ~200-250 MPa, followed by a non-linear region to ultimate failure. Even after failure, the composites retain significant load carrying capability. Figure 33 shows the fracture surface of the sample exposed to 1000°C in flowing oxygen and then tested at RT. The fracture surface is quite similar to that shown in Fig. 28 for the as-fabricated sample in that it is not particularly fibrous in nature, but from the high magnification views in Fig. 33 does exhibit fiber pullout and a considerable amount of fiber/matrix debonding. Evidence of the BN interfacial layer can be clearly seen at the fiber/matrix interface and is undoubtedly contributing to the toughness and strength of the composite. In the SEM view of the edge region of the composite in Fig. 33, it appears that some of the BN interfacial layer has oxidized slightly and is now sticking to the fibers upon fracture. This was only observed on fibers right next to a cut edge of the composite and would be expected to contribute to the oxidative stability of this type of composite through "plugging" of the fiber/matrix interface with a borosilicate glass, thus inhibiting further oxidation along the interface.

#### IV. CONCLUSIONS AND RECOMMENDATIONS

From the results of the studies conducted under this program, certain conclusions can be made. From the first year's efforts, it was found that applying a carbon interfacial layer to either NICALON or Nextel 440 fibers prior to deposition of SiC results in a weakly bonded interface that imparts toughness to a CVI matrix composite through its ability to deflect matrix cracks. This carbon layer can be applied either deliberately by the decomposition of methane or by utilizing an argon flushing gas in the reactor that apparently interrupts the normal deposition of SiC from silane precursors and instead allows carbon to deposit. It was found that this carbon interfacial layer, no matter how it was deposited, is not oxidatively stable at elevated temperatures, leading to severe degradation of composite properties.

Attempts to produce an interfacial layer of carbon rich SiC (actually a mixture of carbon and SiC) that, from earlier work, appeared to act as a relatively oxidatively stable yet weakly bonded interface, were not successful. While this type of material could be obtained in bulk samples utilizing MDS plus methane additions, it could not be reproducibly obtained when deposited in thin coating form on fibers. The reasons for this probably have more to do with the type of gas management system utilized for this work than with the inherent thermodynamics and kinetics of the deposition process. If better deposition equipment with accurate flow controls and shorter lines from the evaporator to the reaction chamber were utilized, this approach to interface control in these types of composites could result in improved properties, especially inherent high temperature oxidation resistance.

The other approach to obtaining an oxidatively stable yet weakly bonded interface in CVI SiC matrix composites that was investigated consisted of applying BN coatings to the fibers prior to deposition of the SiC. Initial studies that utilized BN coated NICALON fibers from Synterials with relatively thin (~1  $\mu\text{m}$ ) CVD SiC overcoatings indicated that the BN layer remained intact during the SiC deposition, although some diffusion of Si and possibly C into the BN appeared to occur, resulting in relatively weak bonding between the fiber and the deposited SiC. Additional NICALON fibers that were coated with BN at UTRC were then utilized to produce actual small CVI SiC composite rings. The RT flexural strength of the resultant composites was quite high [66 ksi (455 MPa)] with typical composite non-linear fracture behavior. Tests were conducted on these composites in flowing oxygen at temperatures from 550° to 1200°C and showed that no degradation in composite strength occurred up to 1000°C with only moderate strength loss after 1200°C exposure. This is in stark contrast to CVI SiC matrix/NICALON fiber composites with carbon interfacial layers that exhibited severe strength loss after similar exposures. Humidity testing also indicated that the BN interfacial layers were stable under this type of environment.

All of the CVI SiC matrix composites fabricated at UTRC with BN (and C) interfacial layers were tested with diamond saw cut surfaces on two sides of the segmented ring samples and a ground surface on the tensile (concave) surface, so that fiber/matrix interfaces were exposed to the atmosphere. The ORNL samples also were tested with cut surfaces. Although commercial CVI SiC matrix/NICALON fiber composites with carbon interfacial layers plus a final CVI SiC overcoat for oxidation protection are being utilized in prototype gas turbine applications, it is apparent that if the SiC oxidation protection layer is damaged from impact events, attachment contact stresses, or overstressing of the part, severe strength degradation could result due to the inherent oxidative instability of the carbon interfacial layer. From the results obtained on this program utilizing BN interfacial layers in CVI SiC matrix/NICALON fiber composites, the catastrophic degradation in composite properties due to failure of an oxidation protection coating would not occur. Although the results obtained on this program utilizing the BN interfacial layers can only be considered preliminary, they are sufficiently promising to warrant additional investigation. In order to fully explore the potential of this system, it is obvious that further processing studies must be done to scale up the size of the fabricated composites, as well as comprehensive elevated temperature mechanical and thermal property measurements.

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TABLE I

**BULK COMPOSITION OF CVD SIC FROM MDS WITH METHANE ADDITIONS**  
 ( $H_2$  carrier gas/MDS ratio = 3.28/1, 1080°C, 30 min run time)

<u>Run Number</u>	<u>Gas Flow Rates (cc/min)</u>		<u>Bulk Composition - at%</u>		
	<u><math>H_2</math>/MDS</u>	<u><math>CH_4</math></u>	<u>C</u>	<u>Si</u>	<u>O</u>
12-6-27	100	none	55	43	2
12-7-5A	100	100	57	41	2
13-8-3A	100	200	58	39	3
13-8-15	100	400	51-59	37-45	3
13-8-16	100	600	56-60	40-44	0
13-10-17	50	600	51	45	4
15-1-16	25	600	50	47	3

TABLE II

**CVD SIC COATED FIBERS (MDS + CH<sub>4</sub>)**  
 -- (H<sub>2</sub>/MDS = 3.28/1, 1080°C, Evap. at 1028 mm, Cond. at 11°C)  
 (100 cc/min H<sub>2</sub>, 600 cc/min CH<sub>4</sub>)

<u>Run #</u>	<u>Fiber</u>	<u>Run Time</u>	<u>Coating Thickness and Morphology</u>	<u>Coating Comp - at%</u>		
				<u>C</u>	<u>Si</u>	<u>O</u>
13-10-12A	Nextel 440	1 hr	0-20 μm, extremely non-uniform rough, well adhered	-	-	-
13-10-13	NICALON	1 hr	5-10 μm, somewhat non-uniform quite smooth, very well adhered	-	-	-
15-12-9A	NICALON	8 min	2-3 μm, uniform, coating not well adhered	50-52	46-50	0-3
15-1-11A	NICALON	4 min	~1 μm, uniform, coating not well adhered, carbon layer between fiber and coating, plus carbon rich surface	51-90	8-48	0-1

TABLE III

## CVD SIC OVER BN COATED FIBERS

. (H<sub>2</sub>/MDS = 3.28/1, 1080°C, 100 cc/min H<sub>2</sub>)

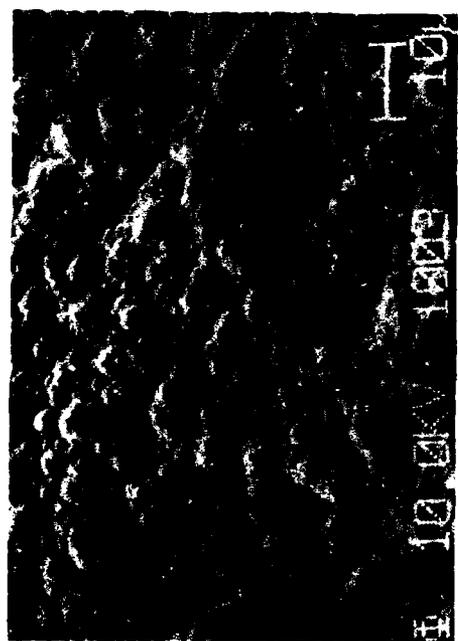
<u>Run #</u> <u>(time)</u>	<u>Fiber</u>	<u>BN Coating</u>	<u>SIC Coating Thickness</u> <u>and Morphology</u>	<u>SIC Coating Comp - at%</u>		
				<u>C</u>	<u>Si</u>	<u>O</u>
13-10-11A (1 hr)	Nextel 480	UTRC (BN-106)	3-6 μm, non-uniform, smooth to rough surf., quite well adhered	-	-	-
13-10-11 (1 hr)	NICALON	Synterials (88-5-5)	2-3 μm, quite smooth and uniform, not well adhered	48-50	45-50	2
13-11-7 (1 hr)	"	"	2 μm, uniform thickness, bumpy surface	48	51	1
15-1-18 (3 min)	"	"	7000Å, uniform thickness, smooth surface, moderate adherence, carbon rich interlayer and surface	54-92	10-40	2-6
15-3-22 (3 min)	"	"	2000-3000Å, moderate adherence, high carbon content, H <sub>2</sub> - 25 cc/min	66-85	10-30	1-3

TABLE IV

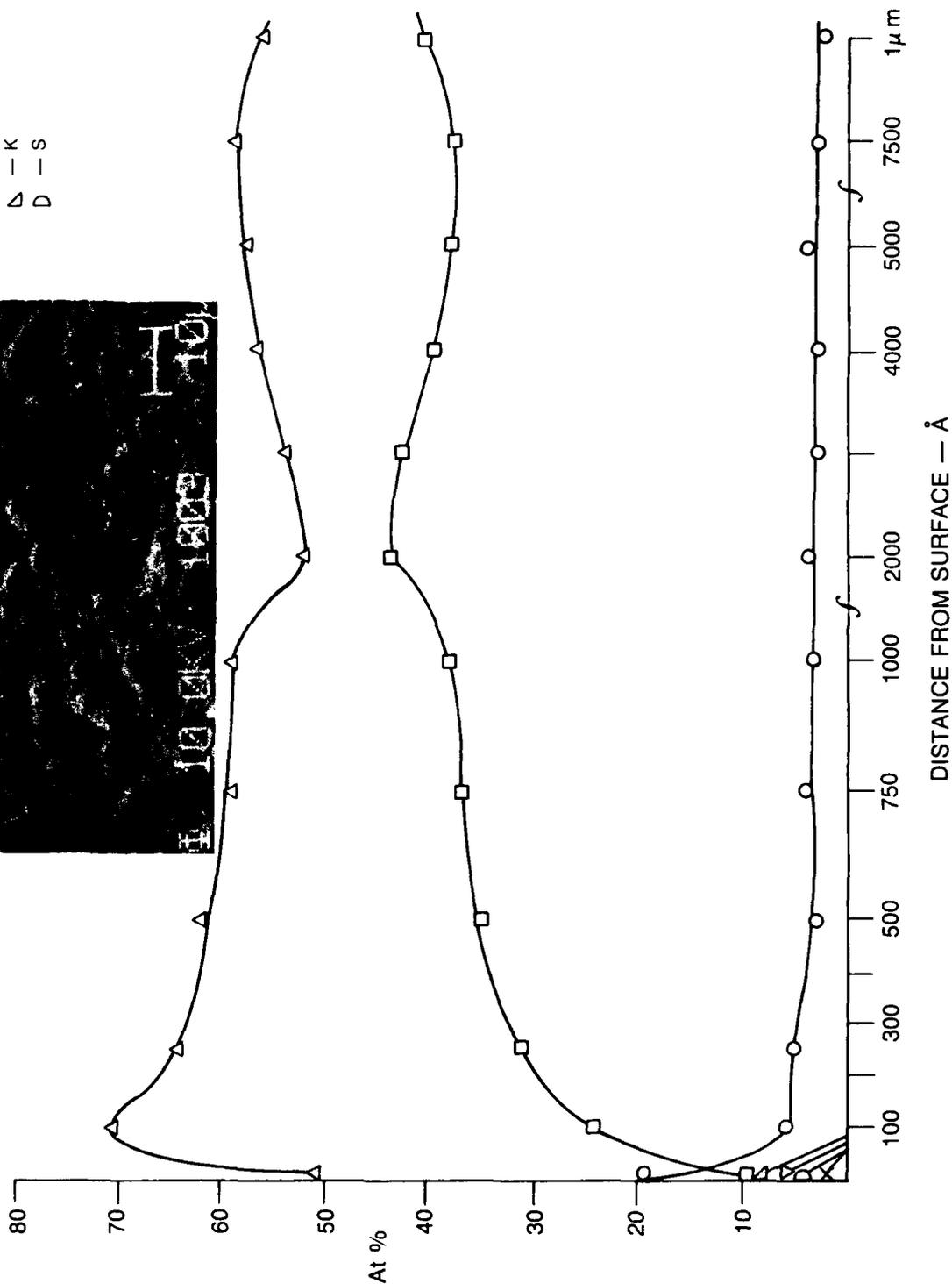
**EFFECT OF FIBER COATINGS AND OXIDATION ON THE RT FLEXURAL  
STRENGTH OF CVI SIC MATRIX/NICALON FIBER COMPOSITES**

<u>Source</u>	<u>Fiber Layup</u>	<u>Fiber Coating</u>	<u>Heat-Treatment</u>	<u>RT <math>\sigma</math> - ksi (MPa)</u>
ORNL	plain weave	none	none	13 (90)
"	"	carbon	none	55 (380)
"	"	"	1000°C, 70 hrs, O <sub>2</sub>	11 (76)
UTRC	wound tow	carbon	none	30 (207)
"	"	"	1000°C, 120 hrs, O <sub>2</sub>	16 (110)
UTRC	wound tow	BN	none	66 (455)
"	"	"	120°F, 100% RH, 70 hrs	64 (441)
"	"	"	550°C, 100 hrs, O <sub>2</sub>	64 (441)
"	"	"	1000°C, 100 hrs, O <sub>2</sub>	68 (469)
"	"	"	1200°C, 100 hrs, O <sub>2</sub>	52 (358)

CVD SiC ON CARBON PLATE (#13-8-3A), H<sub>2</sub> CARRIER, H<sub>2</sub> FLUSH, CH<sub>4</sub> ADDED (200 cc/min)  
 SAM DEPTH PROFILE

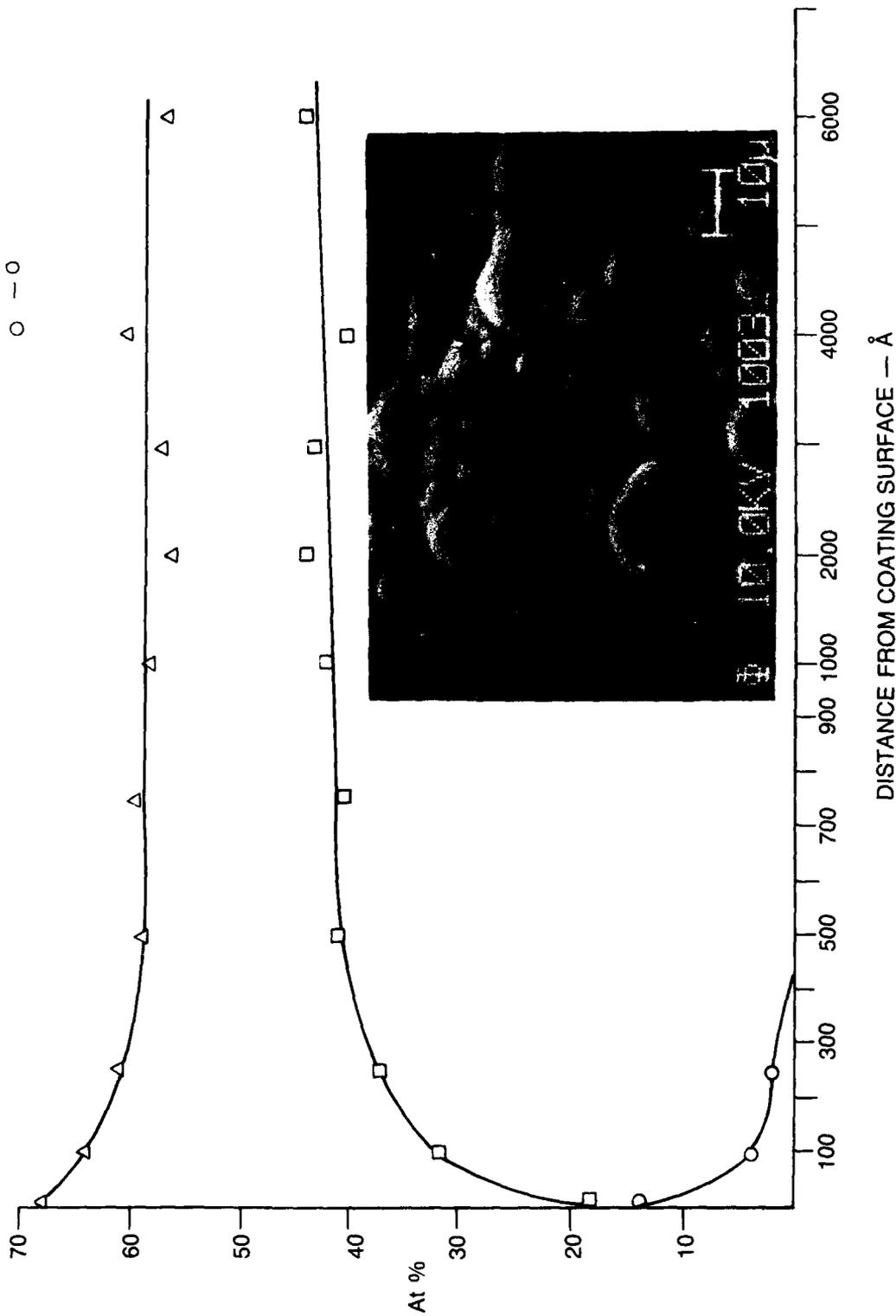


- C
- Si
- O
- Na
- Cl
- K
- S

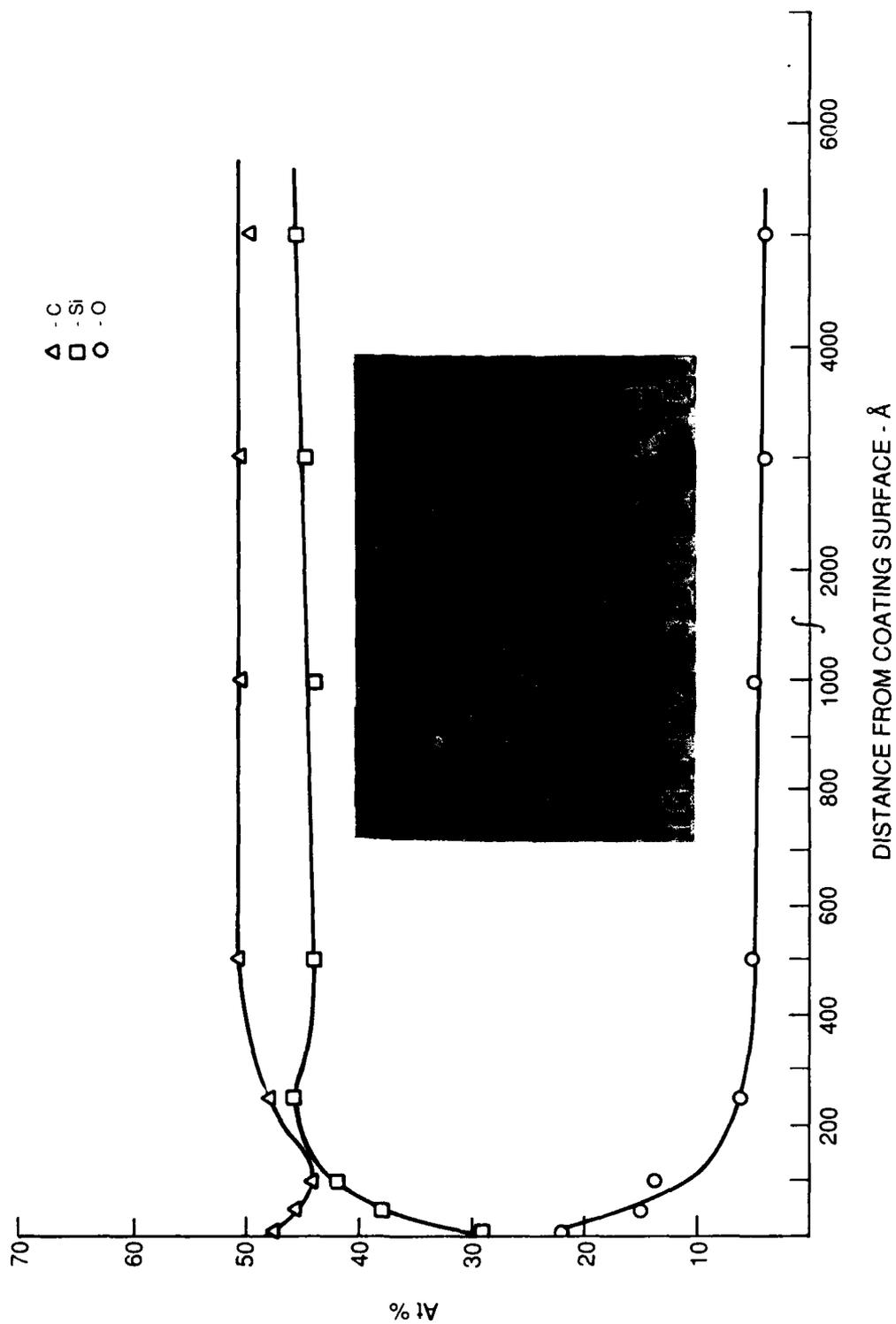


**SAM DEPTH PROFILE**  
**CVD SiC ON CARBON PLATE (#13-8-16) H<sub>2</sub> CARRIER, CH<sub>4</sub> ADDED (600 cc/min)**

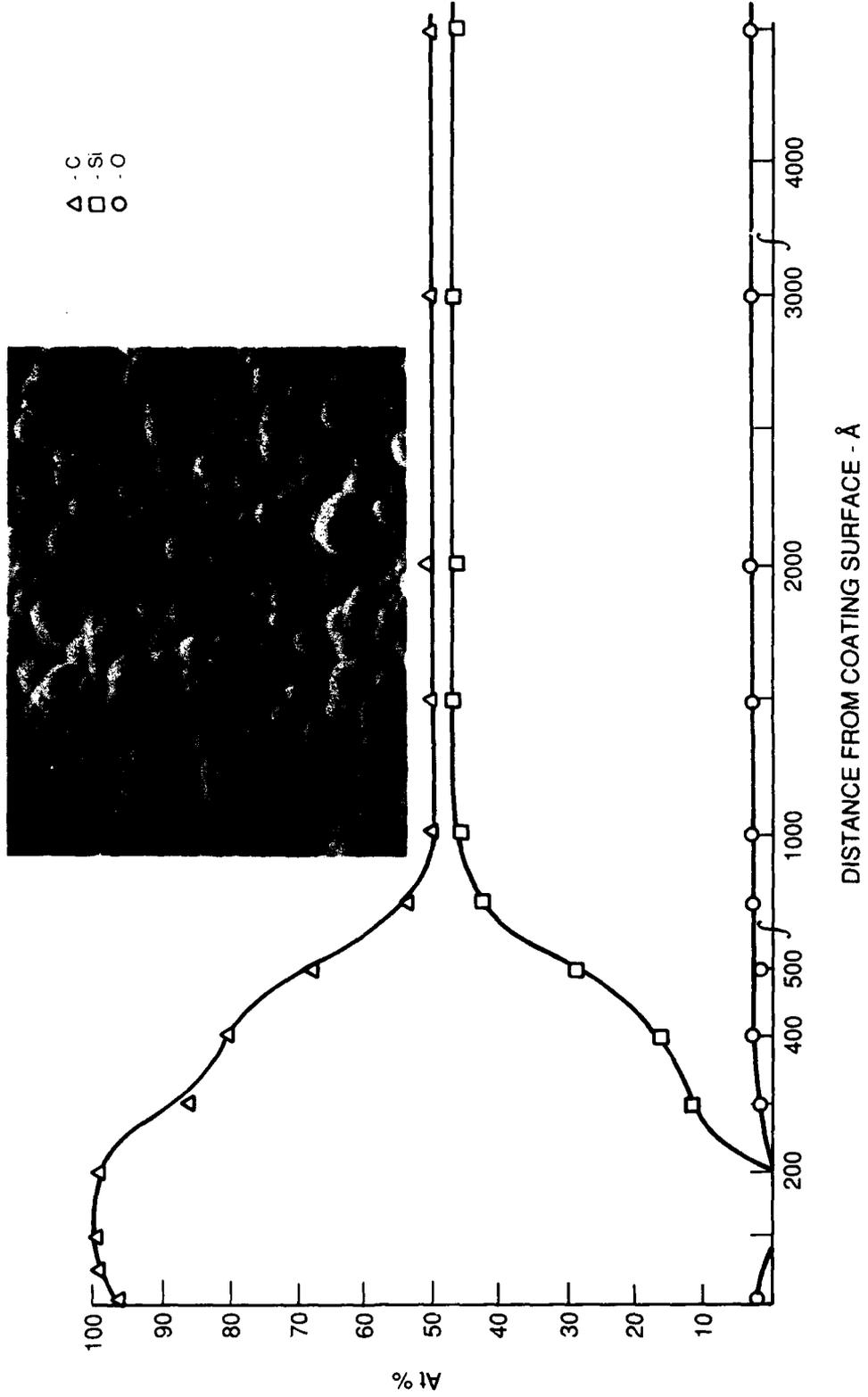
△ — C  
□ — Si  
○ — O



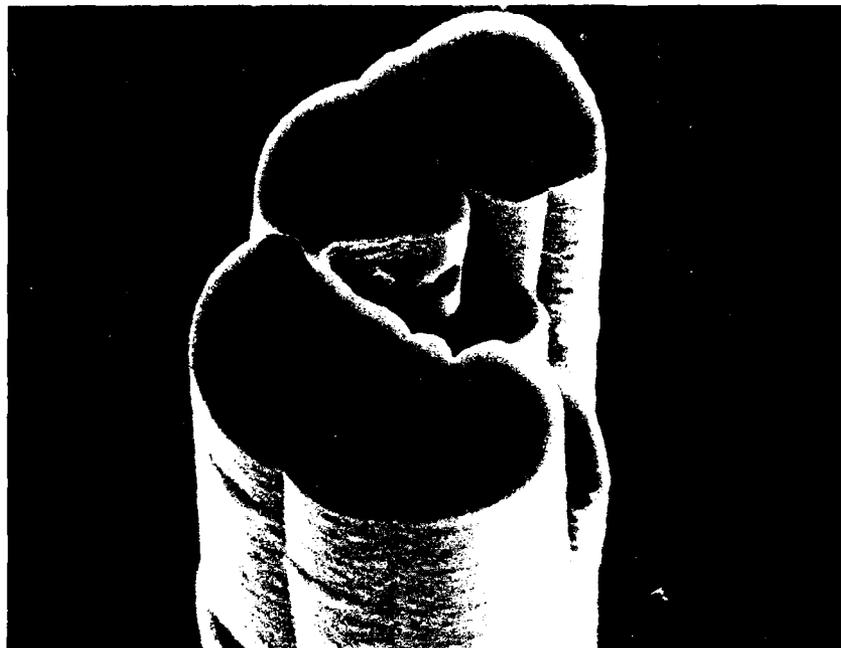
**SAM DEPTH PROFILE**  
**CVD SiC ON CARBON PLATE (#13-10-17) MDS, H<sub>2</sub>-50 cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 1 hr**



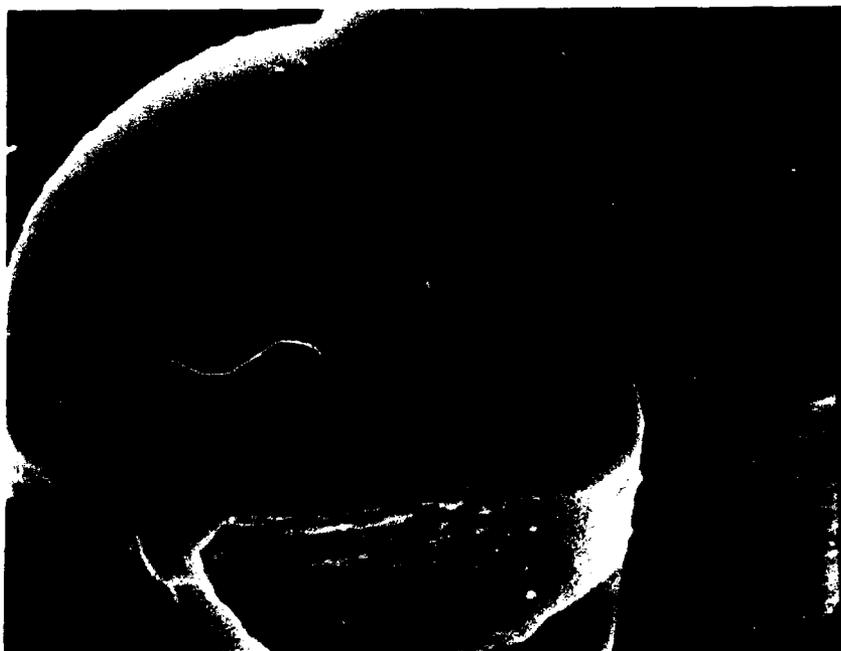
**SAM DEPTH PROFILE**  
**CVD SiC ON CARBON PLATE (#15-1-16), MDS, H<sub>2</sub>-25 cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 1/2 hr**



SEM OF CVD SiC ON NEXTEL 440 FIBERS (RUN #13-10-12A)  
(MDS, H<sub>2</sub>-100 cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 1 hr)

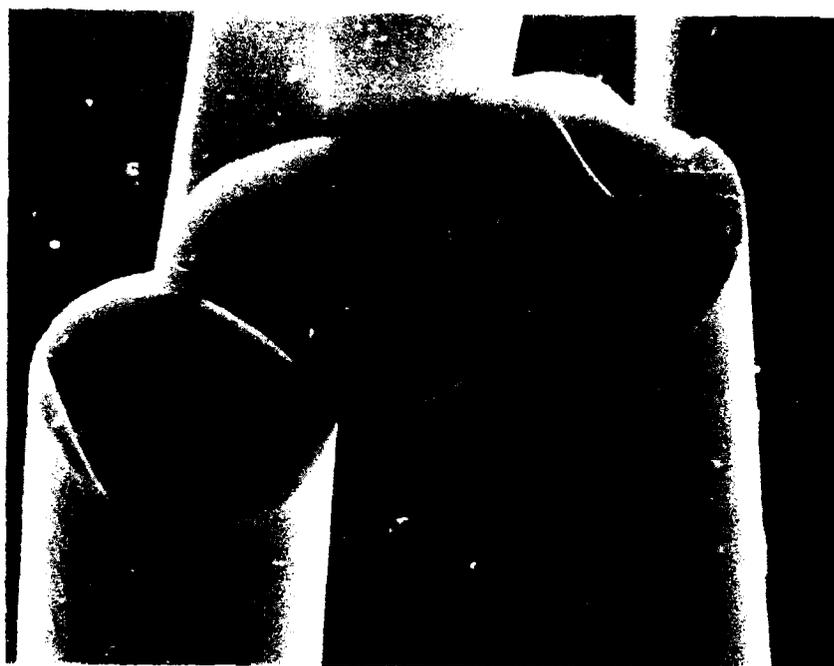


10μm



4μm

SEM OF CVD SIC ON NICALON FIBERS (RUN #13-10-13)  
(MDS, H<sub>2</sub>-cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 1 hr)



10μm

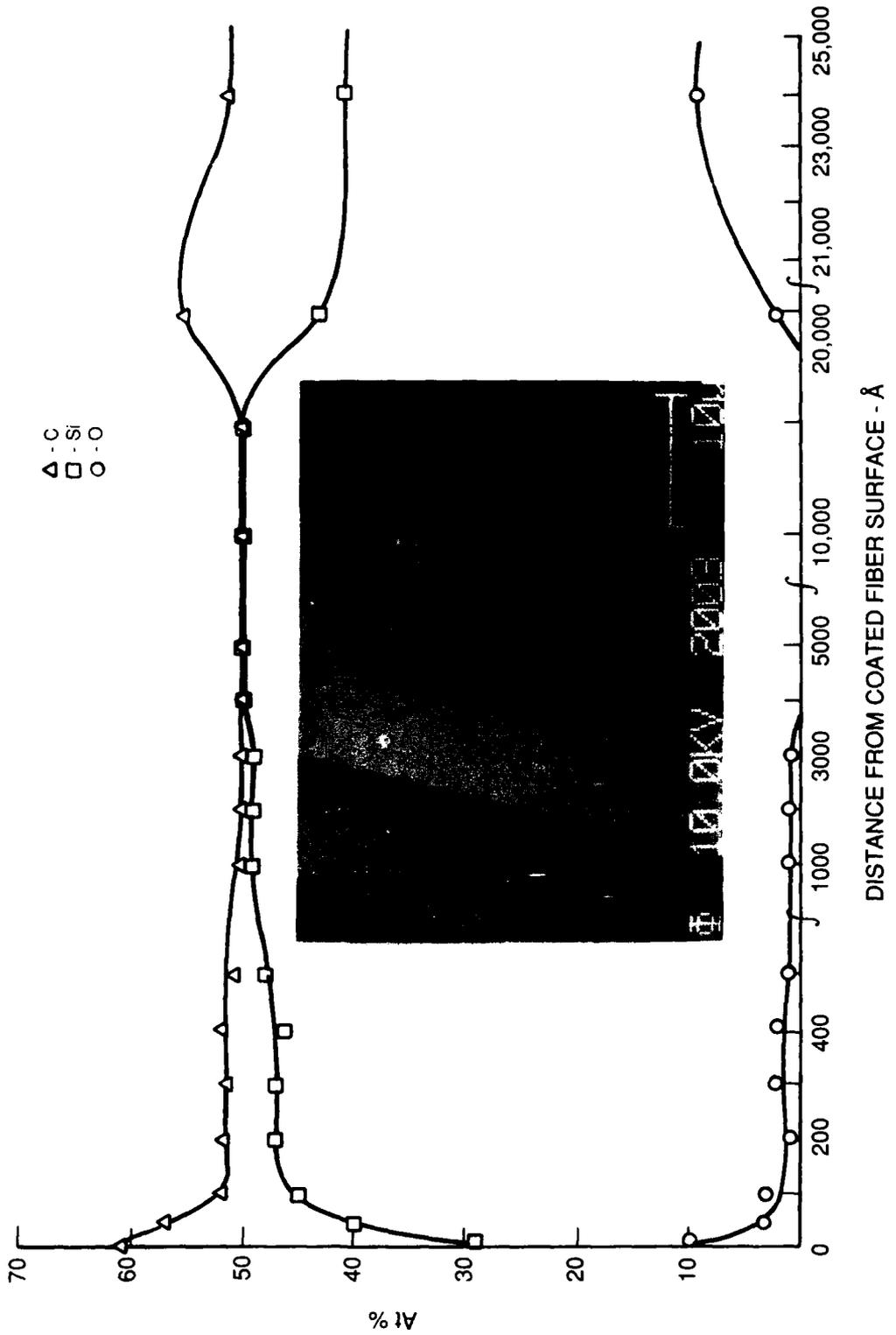


1μm

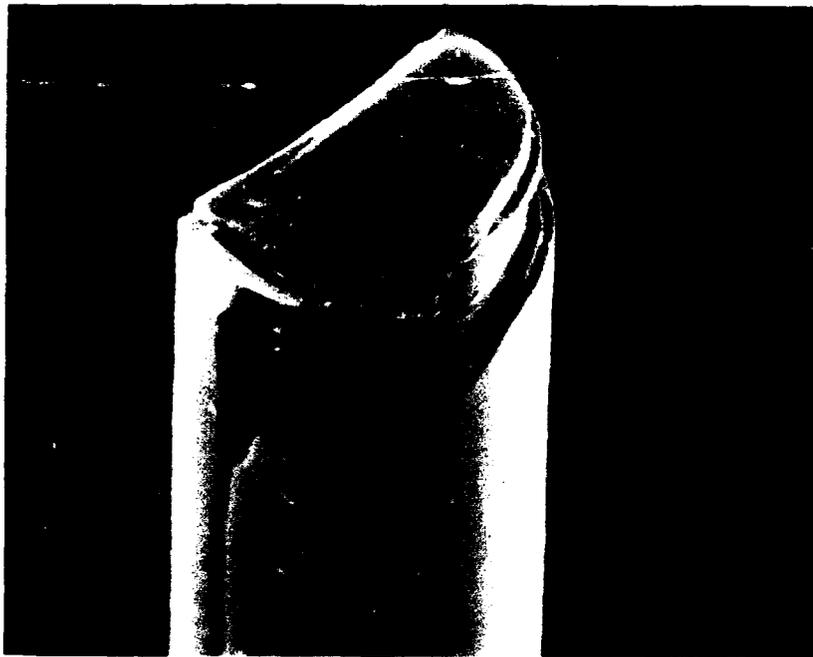
**SEM OF CVD SiC ON NICALON FIBERS (RUN #15-12-9A)**  
(MDS, H<sub>2</sub>-100 cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 8 min)



**SAM DEPTH PROFILE**  
**CVD SIC ON NICALON FIBER (DESIZED) - #15-12-9A, H<sub>2</sub>-100 cc/min, CH<sub>4</sub>-600 cc/min,**  
**1080°C, 8 min**



**SEM OF CVD SiC ON NICALON FIBERS (RUN #15-1-11A)**  
(MDS, H<sub>2</sub>-100 cc/min, CH<sub>4</sub>-600 cc/min, 1080°C, 4 min)  
[UTS=137 ksi (945 MPa)]

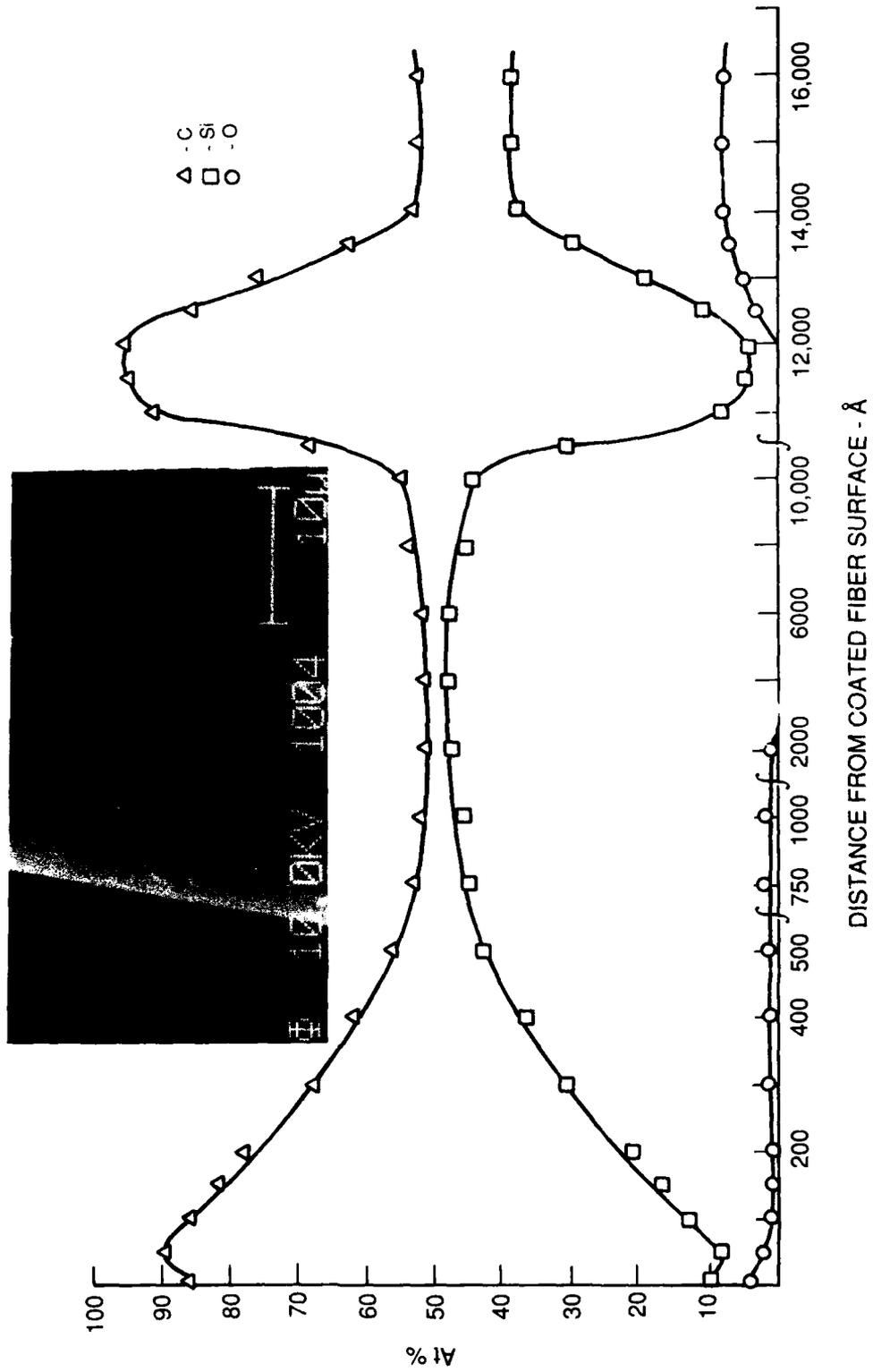


5 $\mu$ m

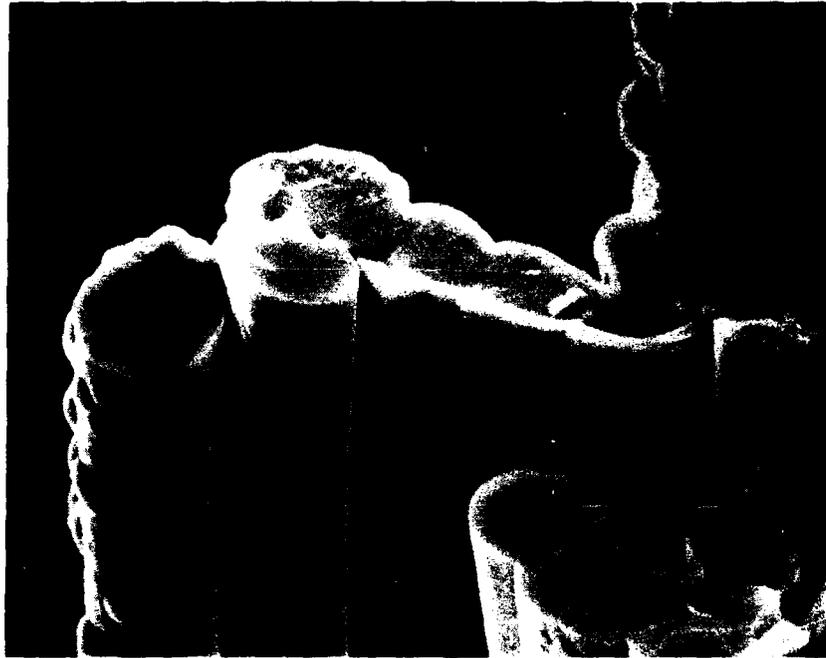


1 $\mu$ m

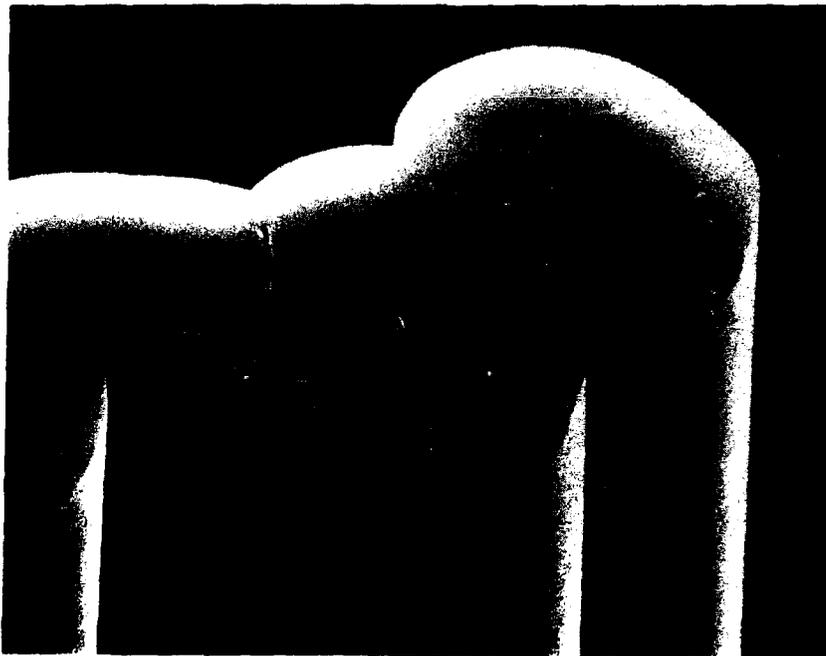
**SAM DEPTH PROFILE**  
**CVD SIC ON NICALON FIBER (RUN #15-1-11A) MDS, H<sub>2</sub>-100 cc/min, CH<sub>4</sub>-600 cc/min,**  
**1080°C, 4 min**



SEM OF CVD SiC ON NEXTEL 440 FIBERS (#7-10-22) (MDS, 2 hrs, H<sub>2</sub> FLUSH)



10  $\mu$ m



5  $\mu$ m

**SEM CVD SiC ON BN COATED NEXTEL 440 MULLITE FIBERS (RUN #13-10-11A)**  
(MDS, H<sub>2</sub>-100 cc/min, 1080°C, 1 hr)

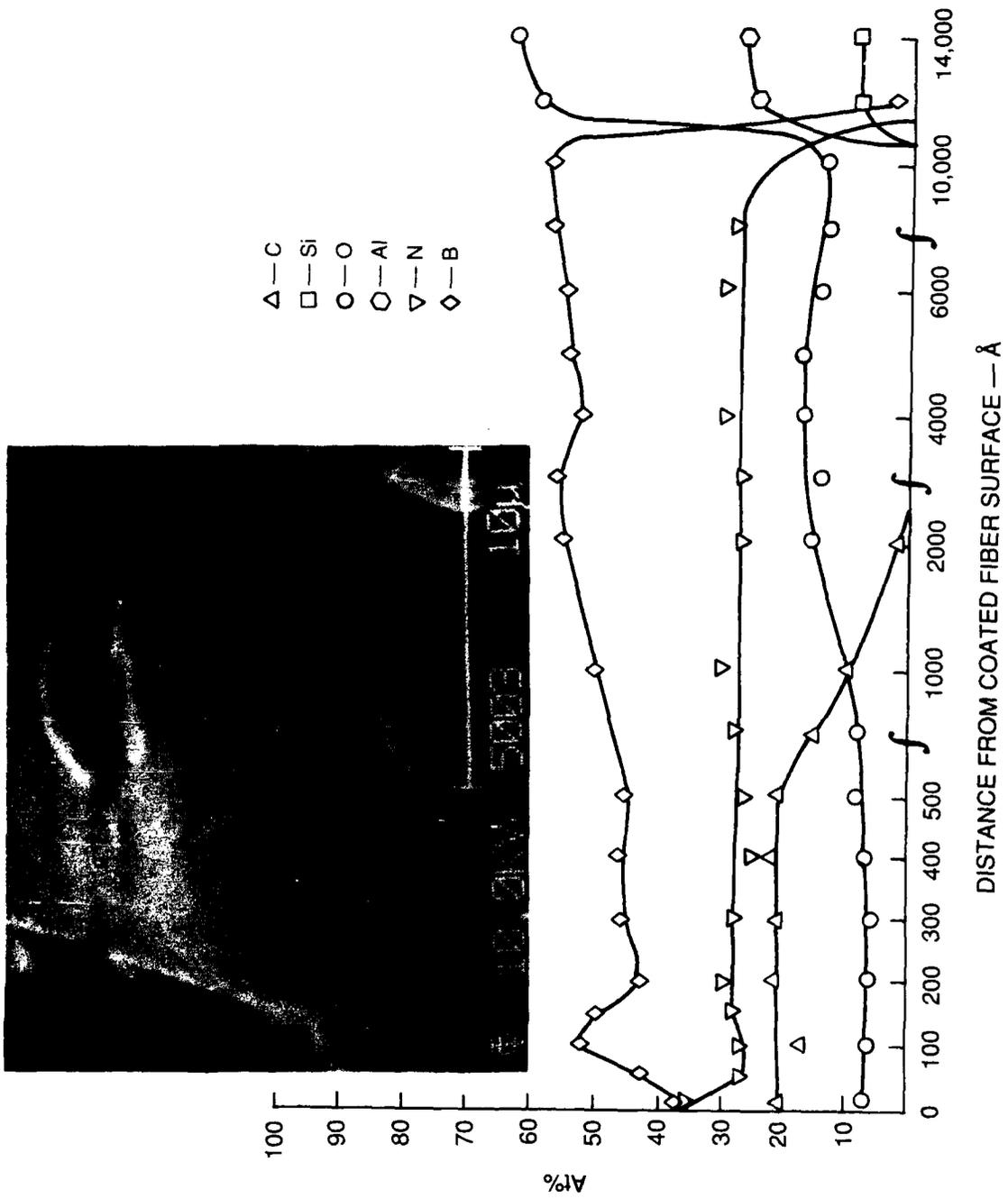


4 μm

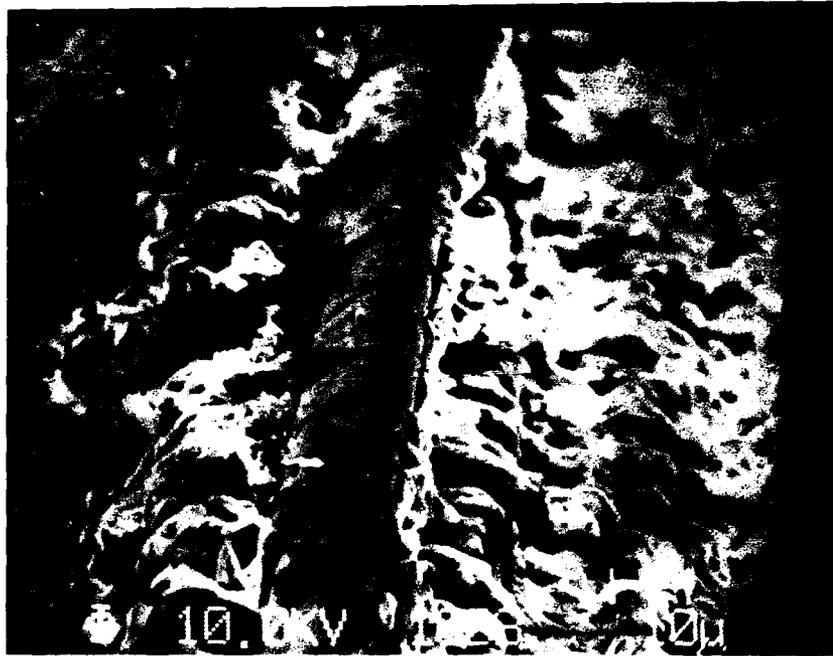


1 μm

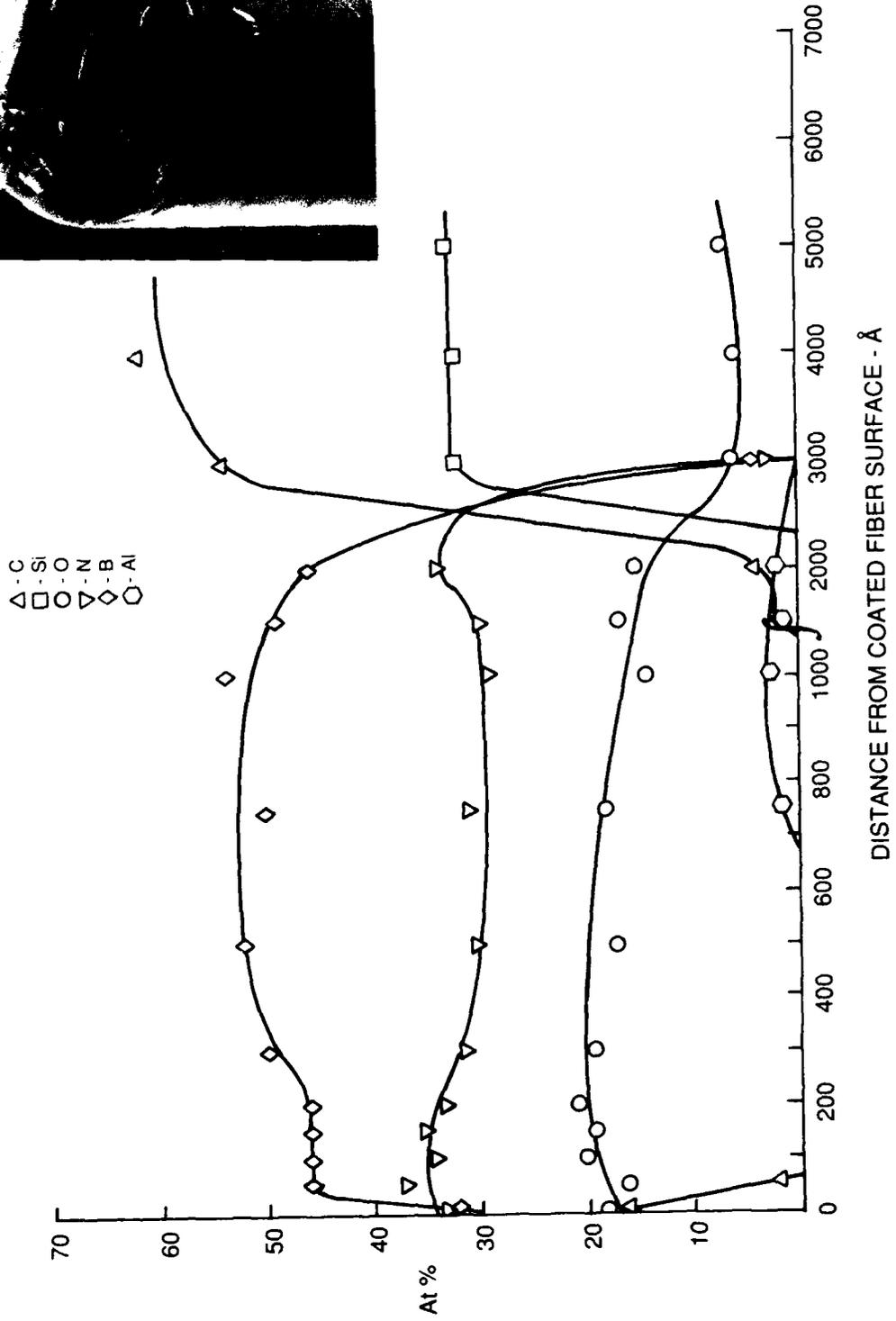
SAM DEPTH PROFILE OF BN-COATED NEXTEL 480 FIBER (RUN BN-106)



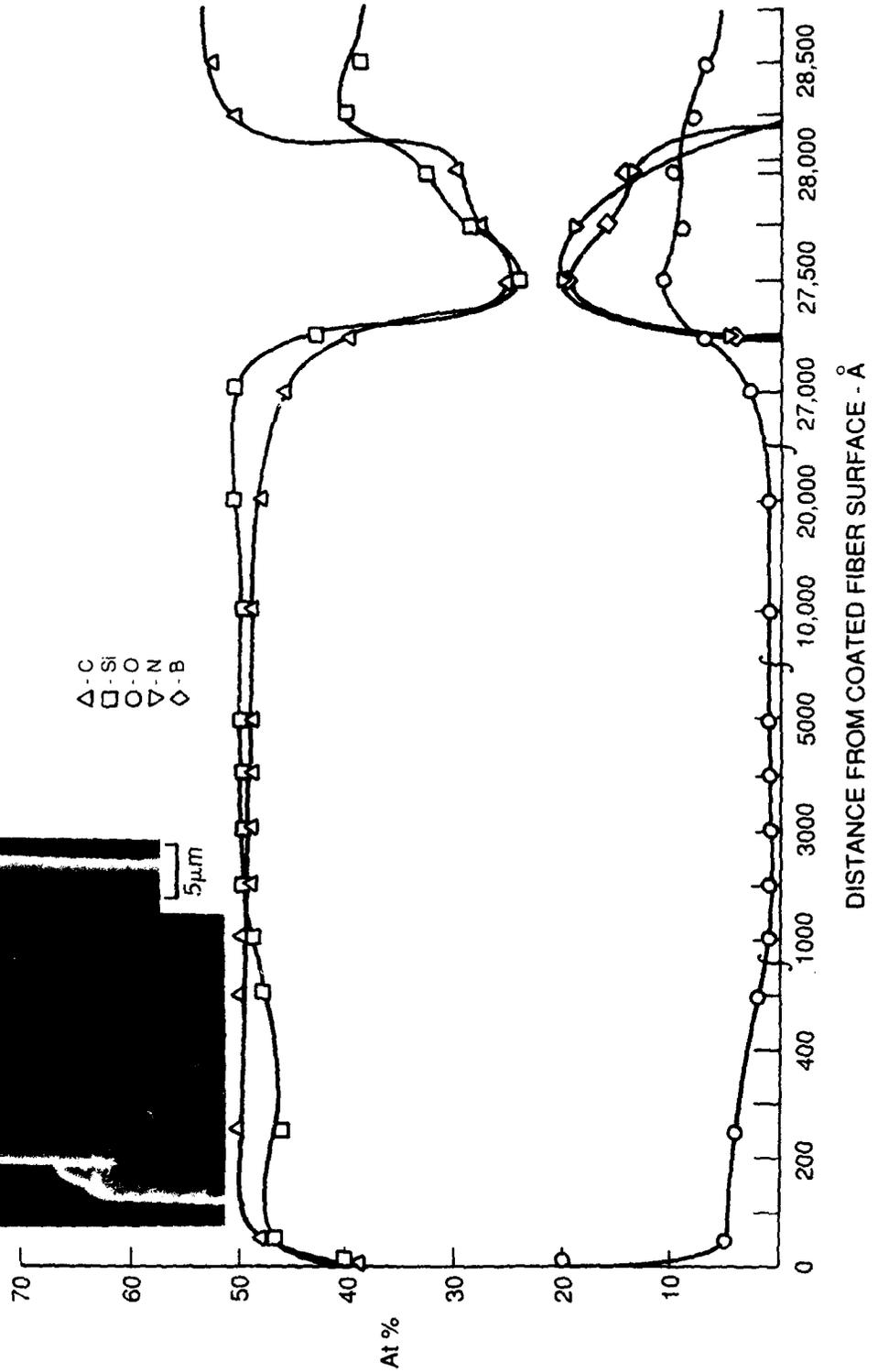
SEM OF BN COATED NEXTEL 480 FIBERS (UTRC RUN #BN-106)



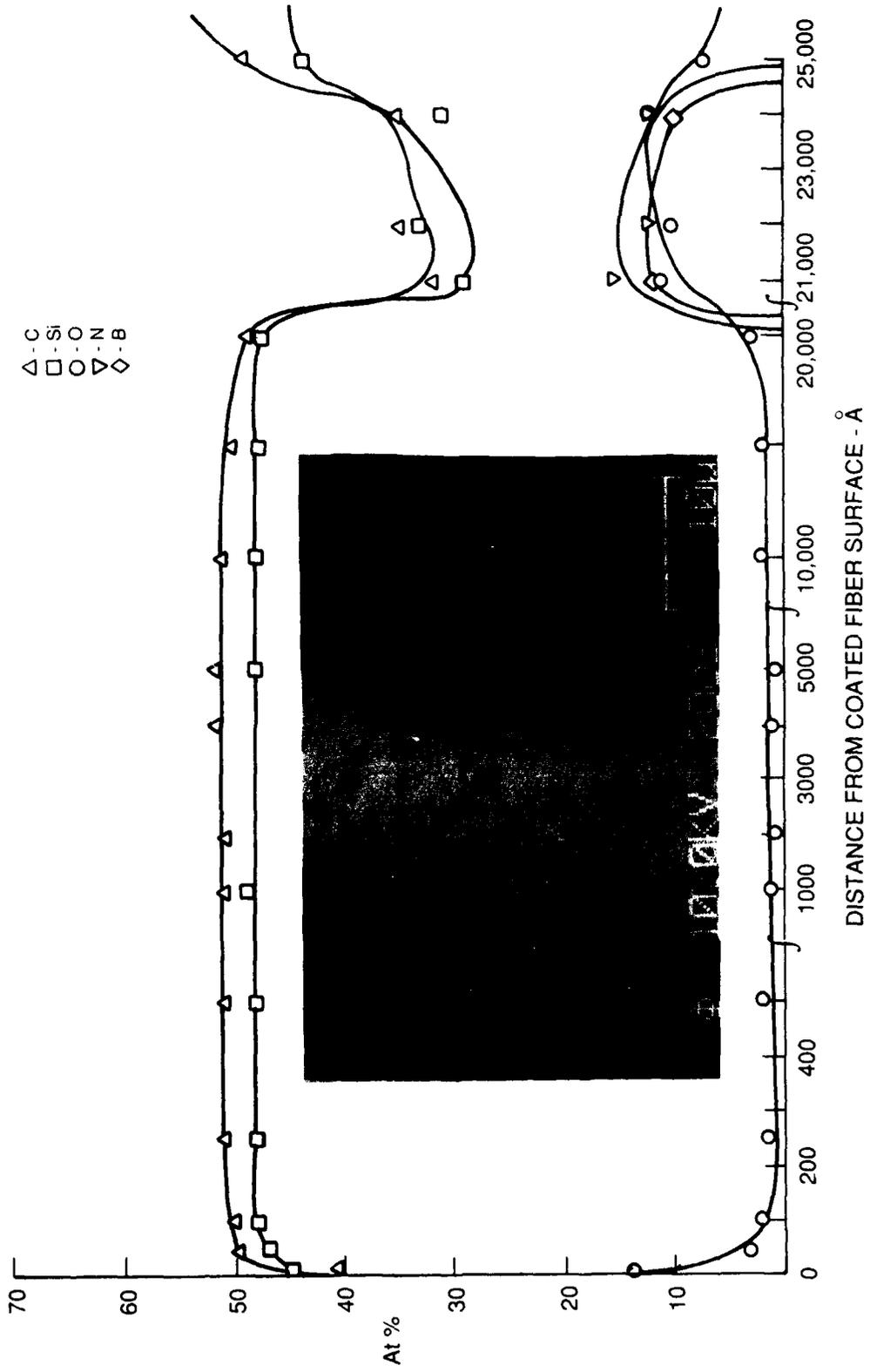
**SAM DEPTH PROFILE**  
**SYNTERIALS BN COATED NICALON FIBER, LOT #88-5-5 - UTS=360 ksi (2480 MPa)**



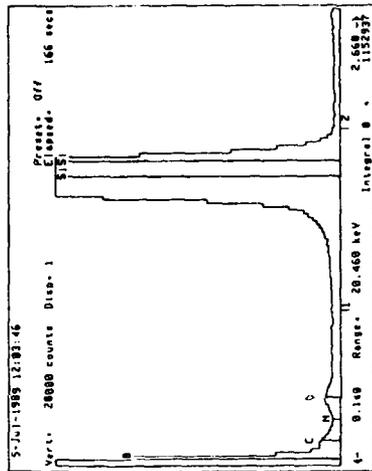
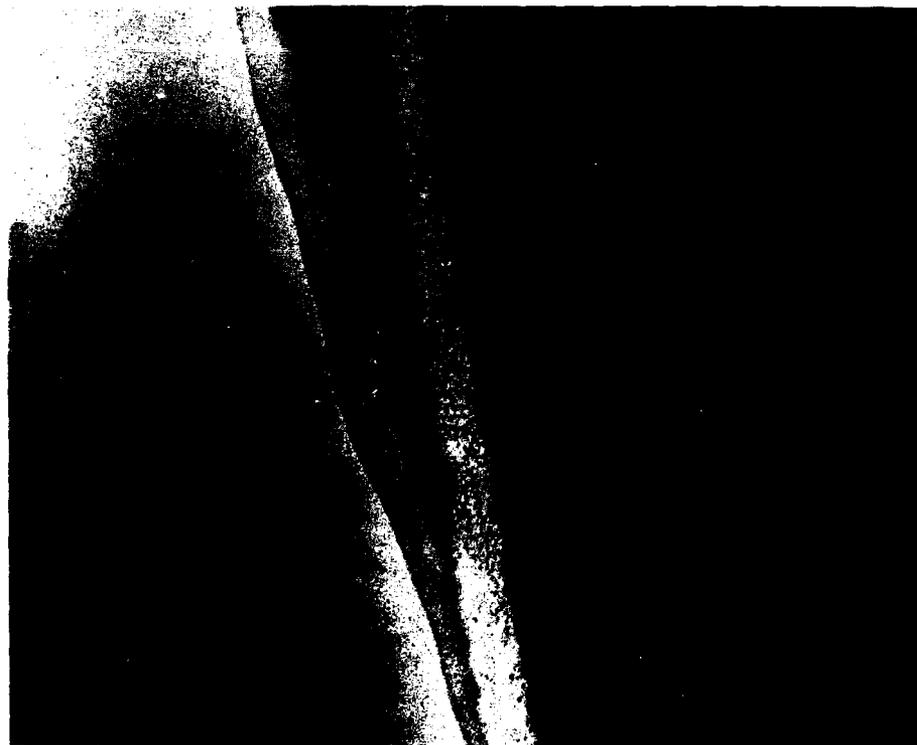
**SAM DEPTH PROFILE**  
**CVD SiC (MDS, H<sub>2</sub>-100 cc/min, 1080°C, 1 hr) ON SYNTHERIALS**  
**BN COATED NICALON FIBER (88-5-5), RUN #13-10-11**



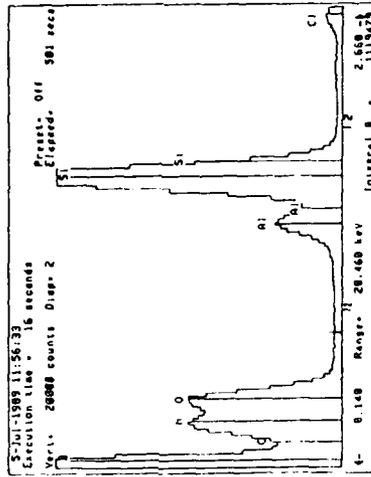
**SAM DEPTH PROFILE**  
**CVD SiC (MDS, H<sub>2</sub> FLUSH, VAC, 1 hr, OLD GAS) ON SYNTHERIALS BN COATED NICALON**  
**FIBER (88-5-5), RUN #13-11-7**



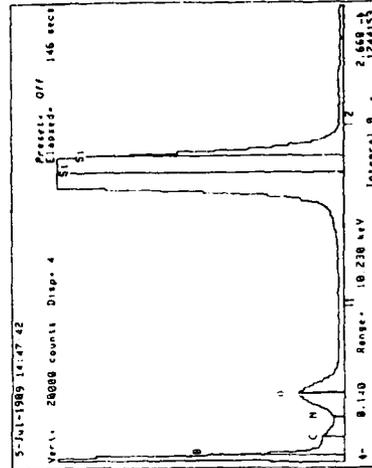
TEM/EDX THIN FOIL ANALYSES OF CVD SIC/CVD BN COATED NICALON FIBER - (RUN #13-10-11)



AREA A



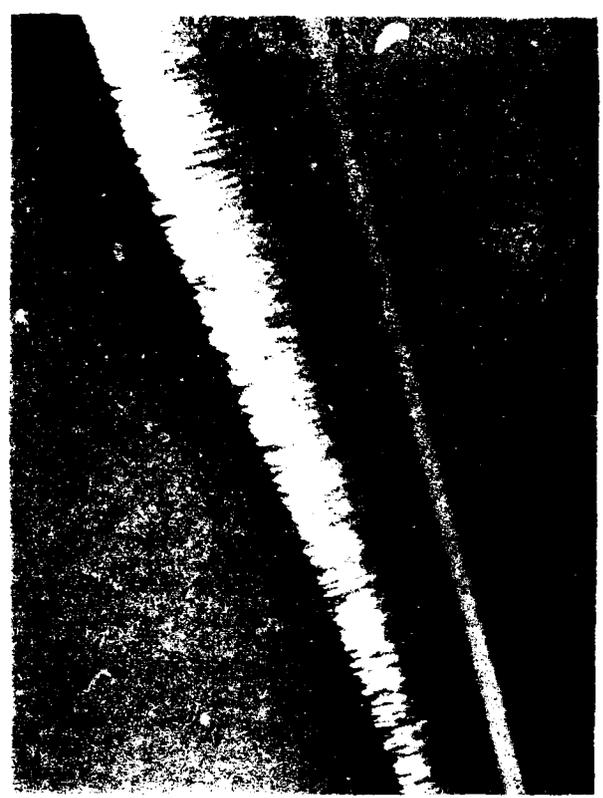
AREA C



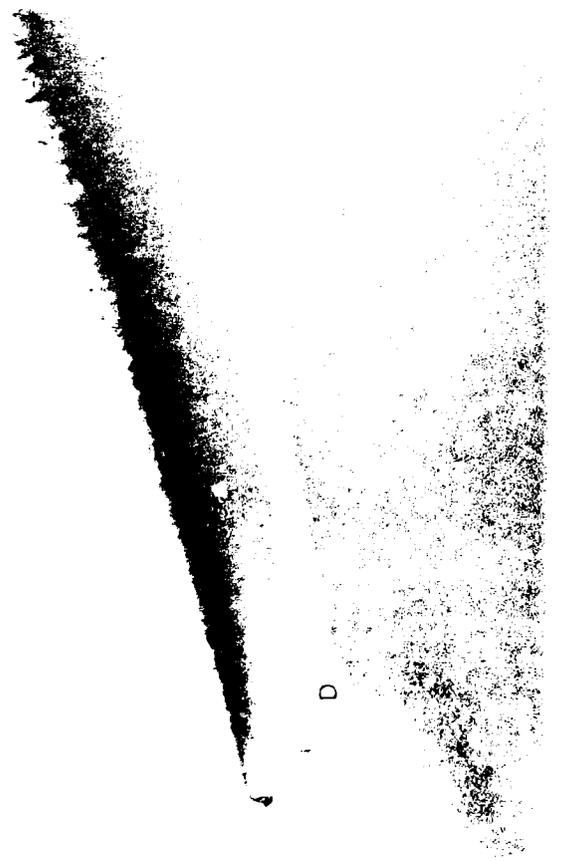
AREA B

FIG. 18

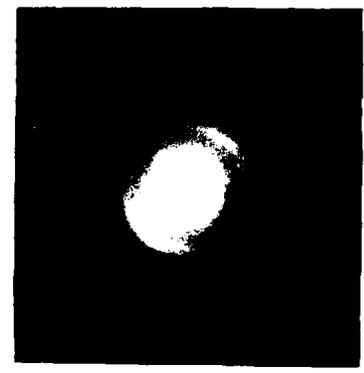
TEM/SAED THIN FOIL MICROGRAPHS OF CVD SiC/CVD BN COATED NICALON FIBER - (RUN #13-10-11)



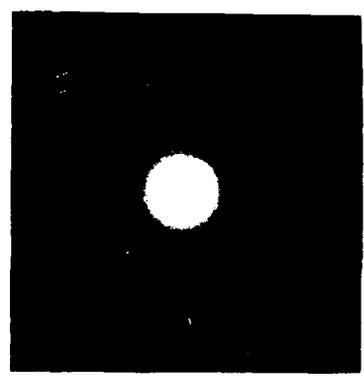
B. DARK FIELD



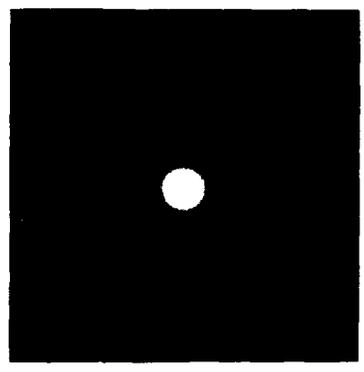
A BRIGHT FIELD



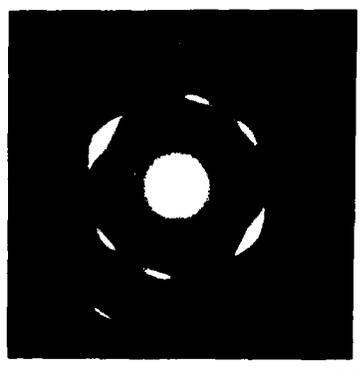
REGION D - BN (CVD)



REGION C - SiC (NICALON)



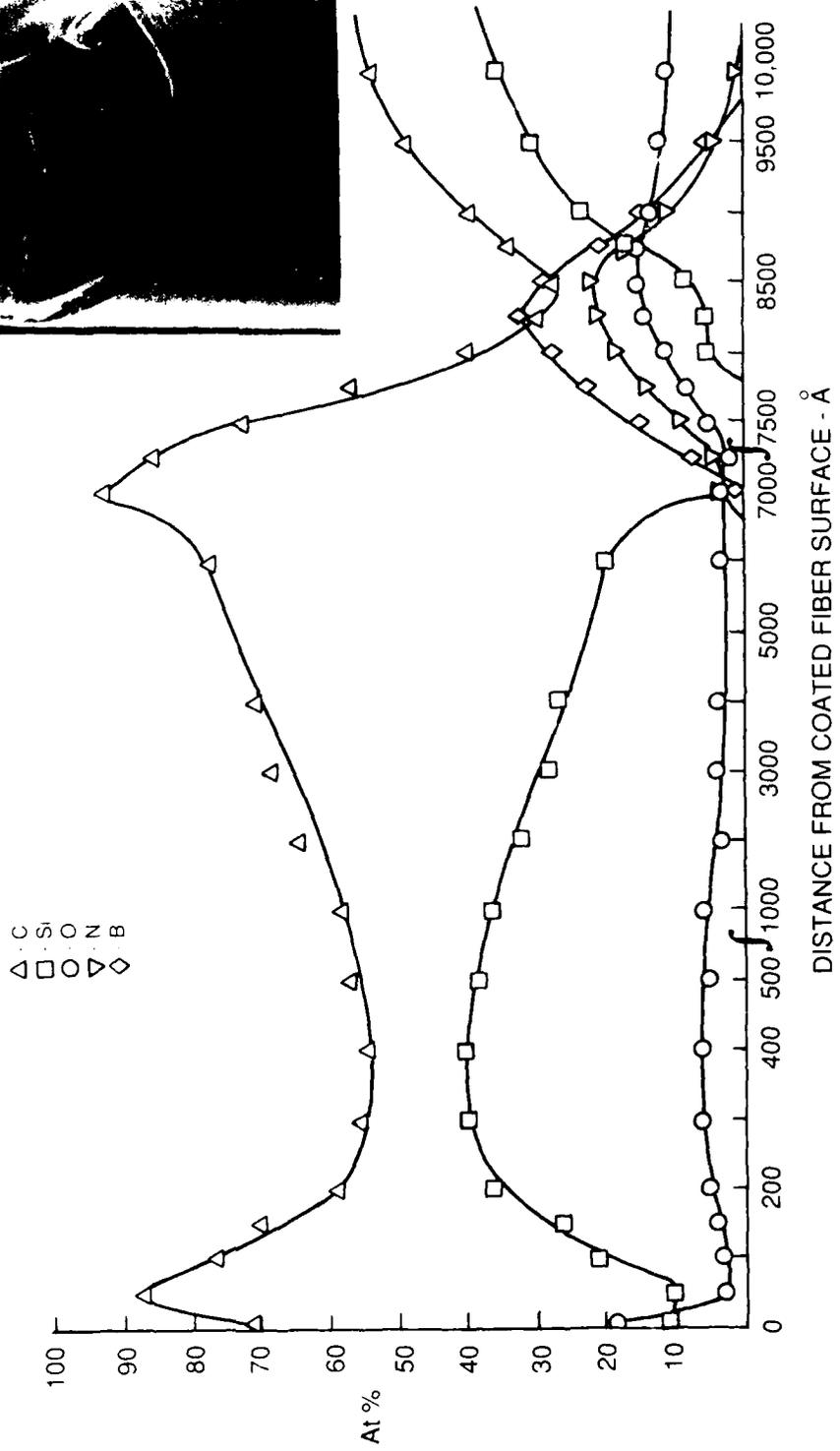
REGION B - SiC (CVD)



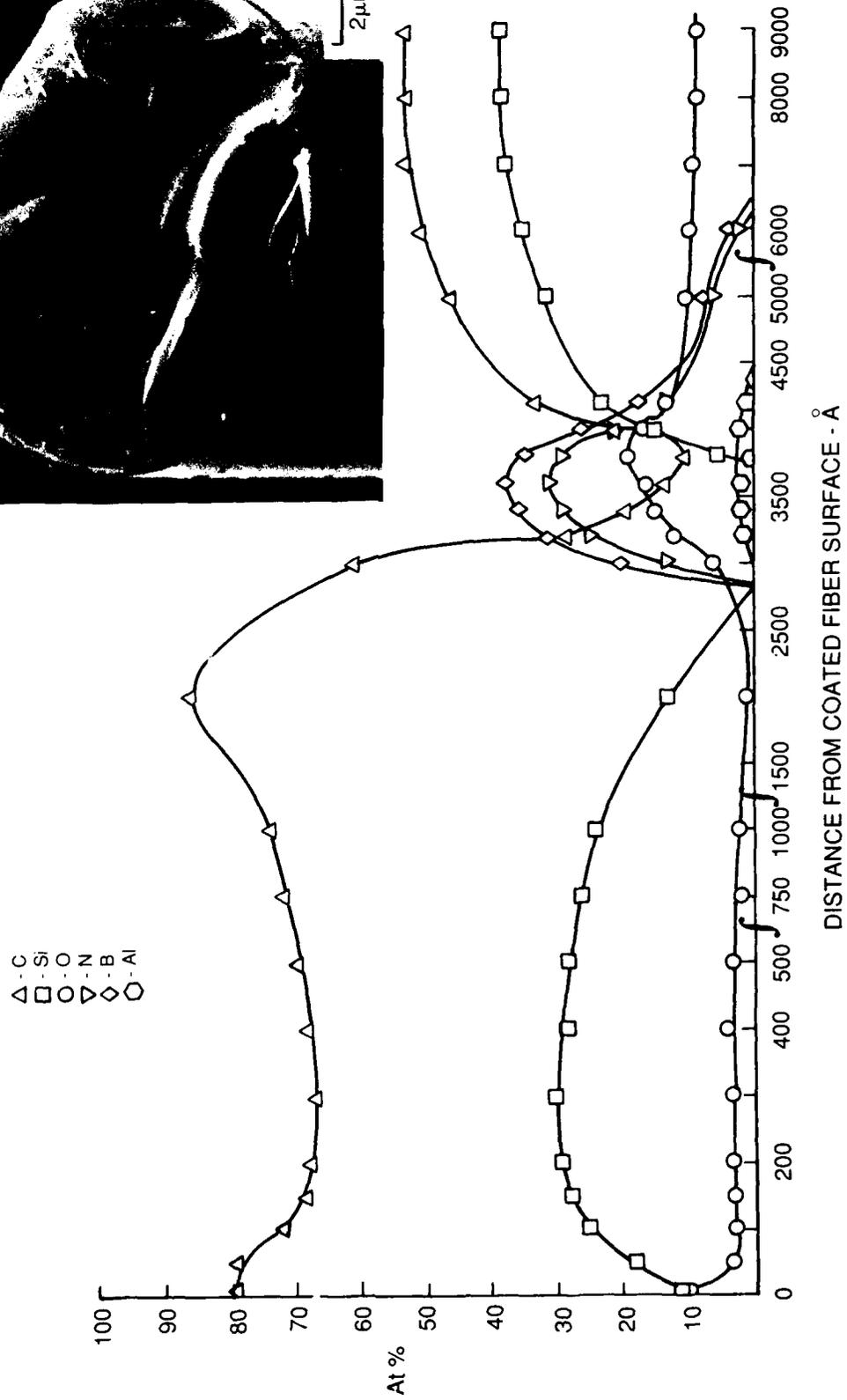
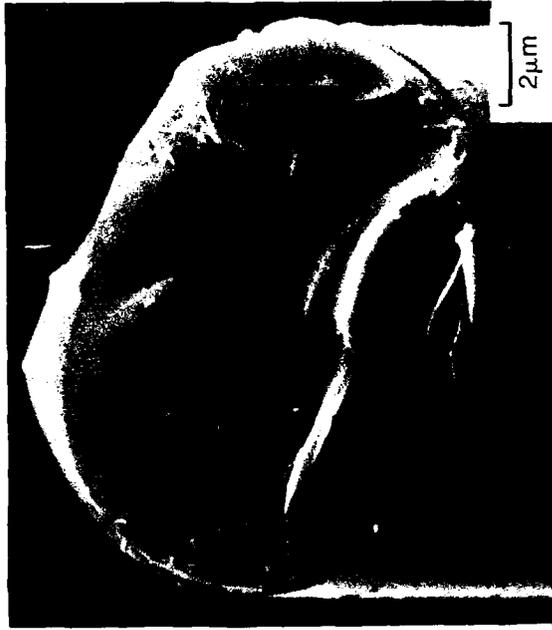
REGION A - SiC (CVD)

FIG. 19

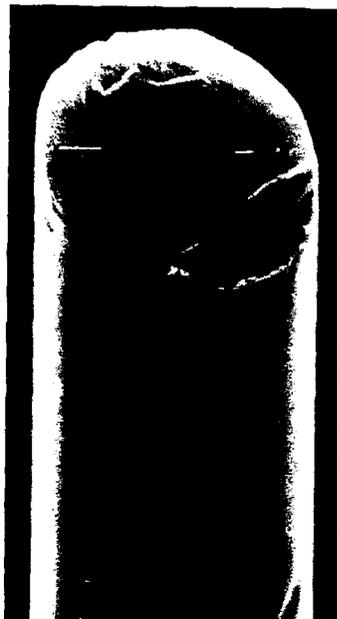
**SAM DEPTH PROFILE**  
**CVD SiC OVER BN (SYNTERIALS 88-5-5) COATED NICALON FIBER (#15-1-18) MDS,**  
**H<sub>2</sub>-100 cc/min, 1080°C, ~3 min**  
**[UTS=243 ksi (1680 MPa)]**



**SAM DEPTH PROFILE**  
**CVD SiC OVER BN (SYNTERIALS 88-5-5) COATED NICALON FIBERS (#15-3-22) MDS,**  
**H<sub>2</sub>-25 cc/min, 1080°C, 3 min**



SEM OF CVD BN COATED NICALON FIBERS (UTRC RUNS BN 255 + 257)



4 $\mu$ m

a) BN 255 ( $\text{BCl}_3 + \text{NH}_3$ , 1050°C, 10 min)



1 $\mu$ m



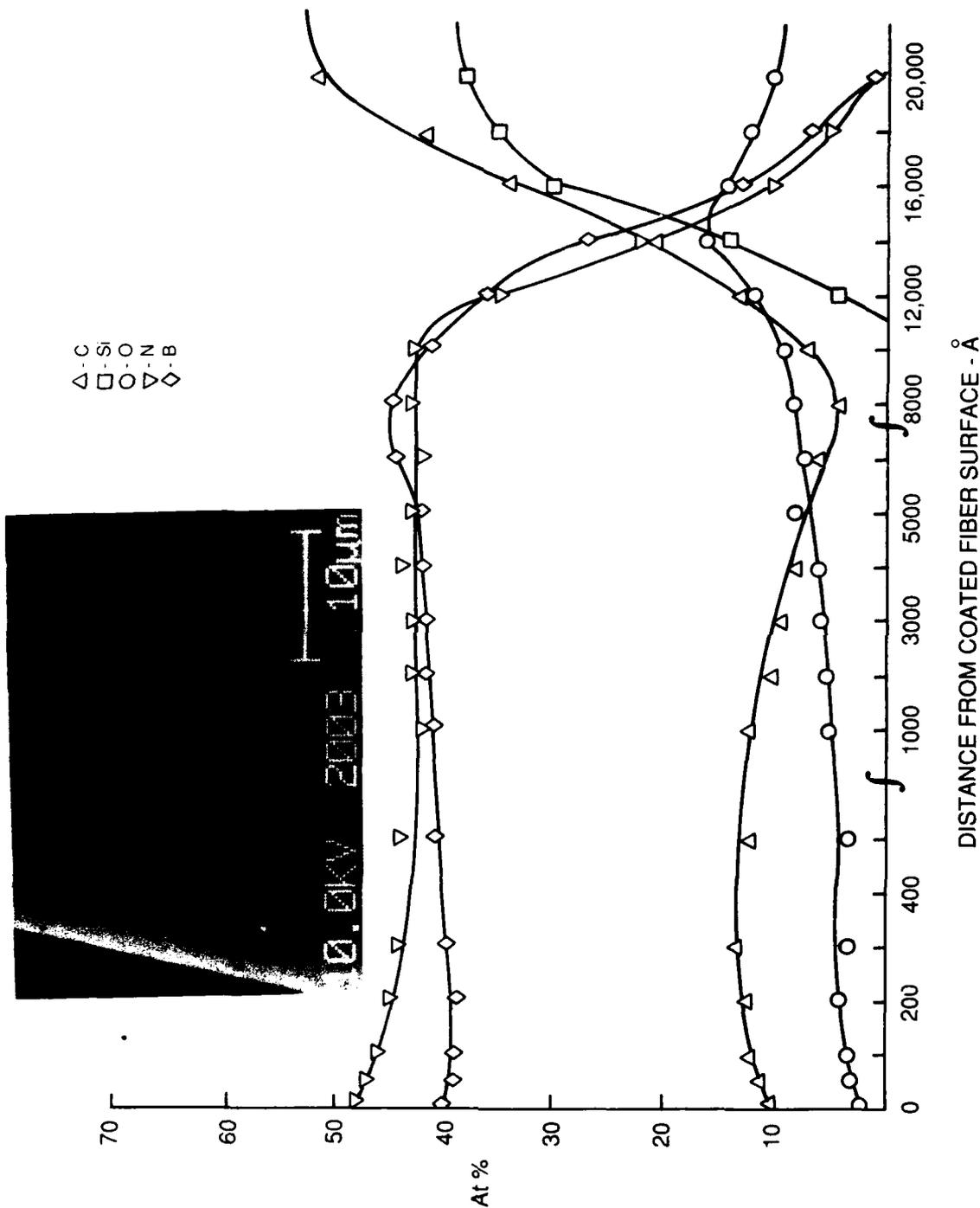
4 $\mu$ m

b) BN 257 ( $\text{BCl}_3 + \text{NH}_3$ , 1050°C, 15 min)



1 $\mu$ m

SAM DEPTH PROFILE  
CVD BN ON NICALON FIBERS (BN 255) - 1050°C, 10 min



CROSS-SECTIONS OF CVI SiC MATRIX/BN COATED NICALON  
FIBER COMPOSITE #255-2



100 $\mu$ m

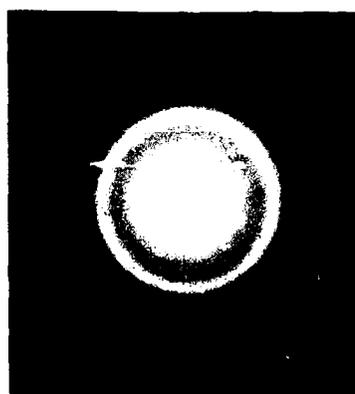
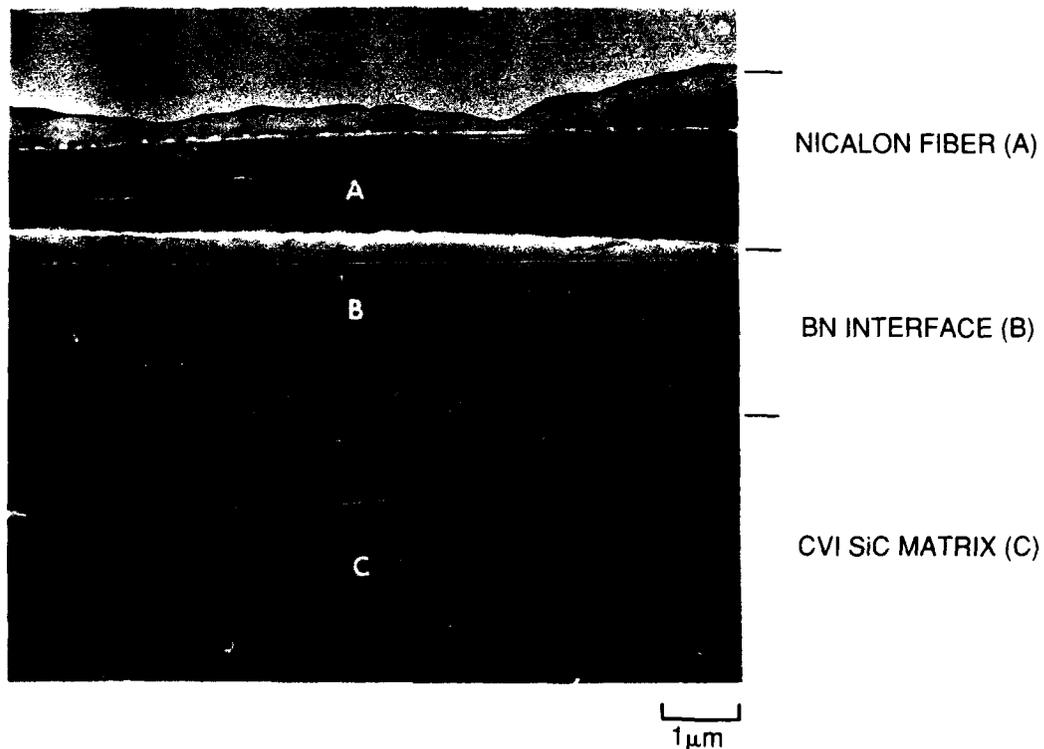


50 $\mu$ m

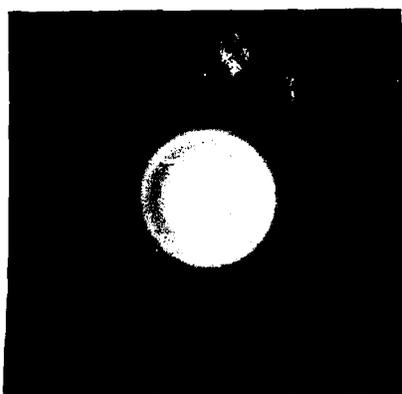


20 $\mu$ m

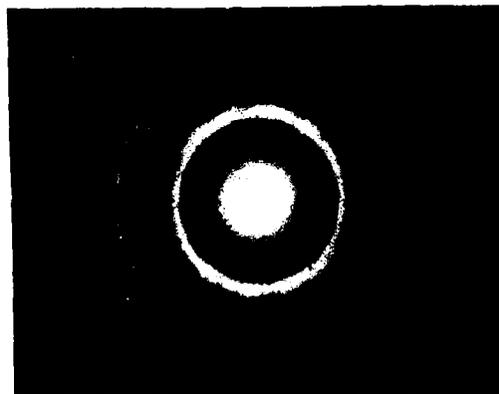
TEM/SAED THIN FOIL ANALYSES OF CVI SiC OVER BN COATED NICALON FIBER COMPOSITE #255 (AS-FABRICATED)



NICALON FIBER (A)



BN INTERFACE (B)



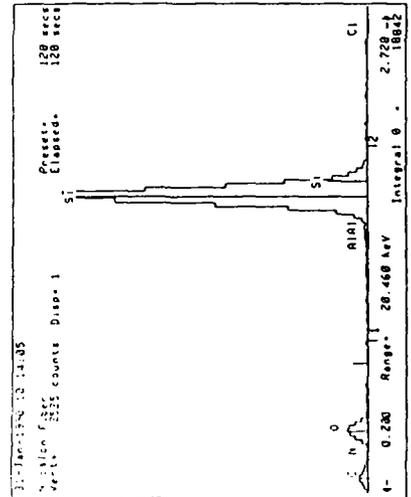
CVI SiC MATRIX (C)

TEM/EDX THIN FOIL ANALYSES OF CVI SiC OVER BN COATED NICALON FIBER COMPOSITE #255



BRIGHT FIELD

0.25 μm

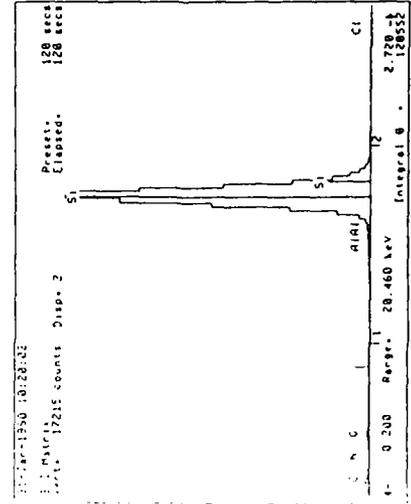


A. NICALON FIBER

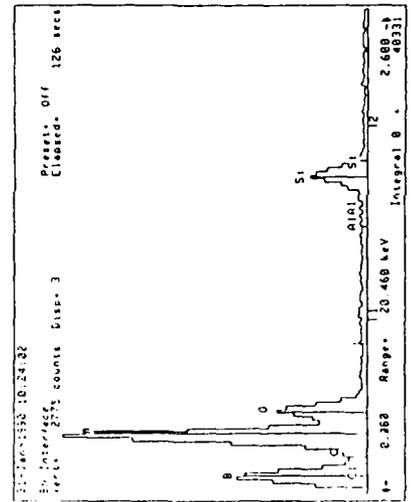


DARK FIELD

0.25 μm

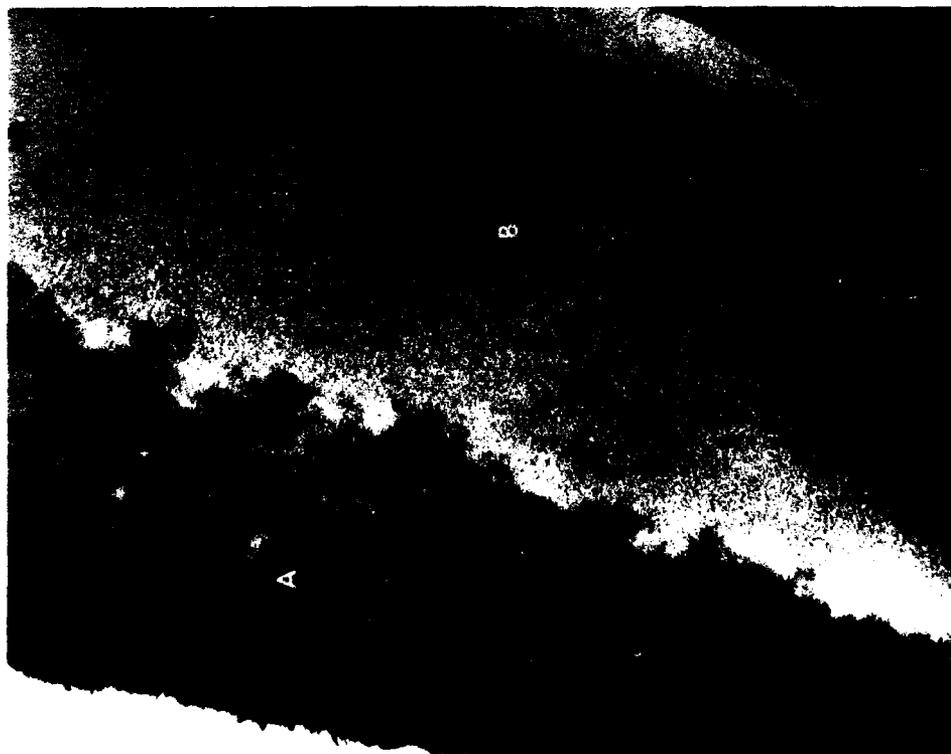


C. CVI SiC MATRIX

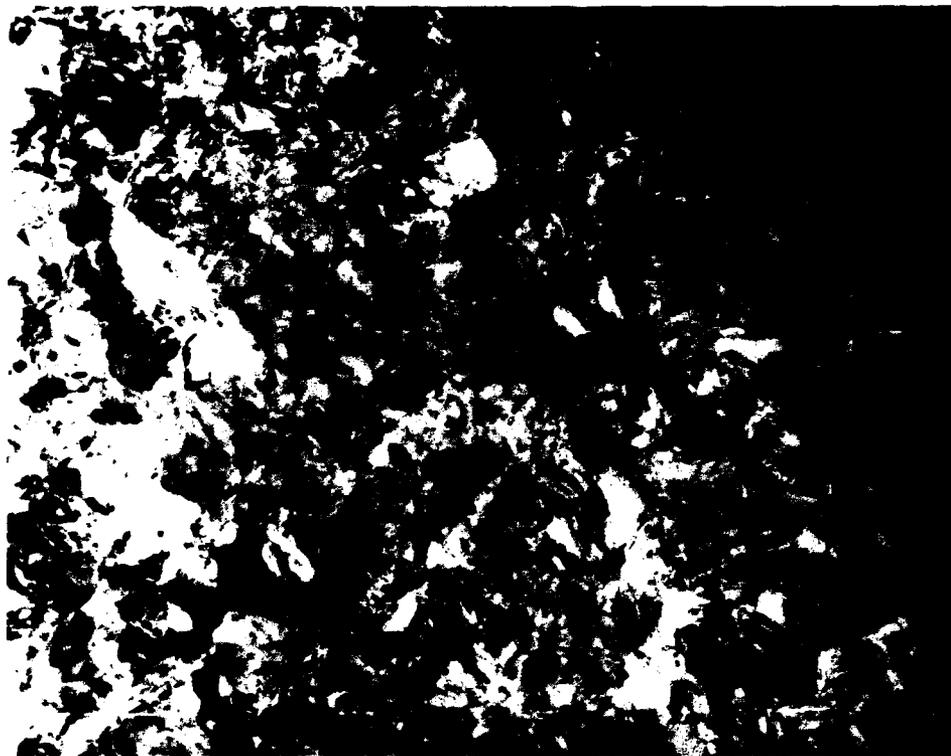


B. BN INTERFACE

TEM THIN FOIL ANALYSIS OF CVI SiC OVER BN-COATED NICALON FIBERS  
COMPOSITE #255 (AS-FABRICATED)



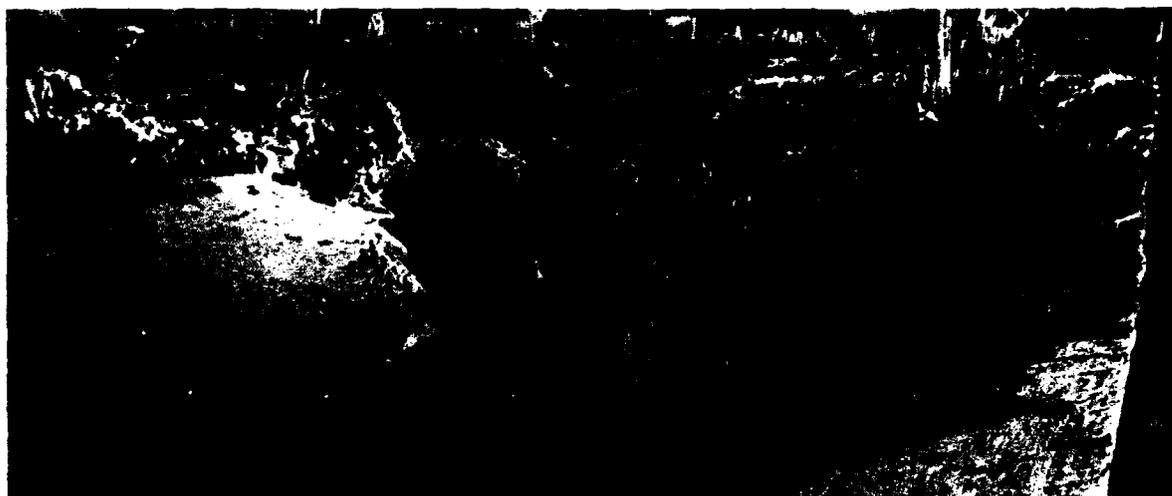
A. SECTION THROUGH CVI SiC (A)/BN INTERFACE (B)



B. SECTION NORMAL TO CVI SiC GROWTH DIRECTION

FIG. 27

**RT FRACTURE SURFACE OF CVI SiC/BN COATED NICALON FIBER COMPOSITE  
#257-7-2 [AS-FABRICATED,  $\sigma=66$  ksi (455 MPa)]**



200 $\mu$ m



5 $\mu$ m

1 $\mu$ m

FRACTURE SURFACE OF ORNL CVI SIC MATRIX/CARBON COATED NICALON FIBER COMPOSITE [RT $\sigma$ ≈55 ksi (380 MPa)]



400 $\mu$ m



100 $\mu$ m

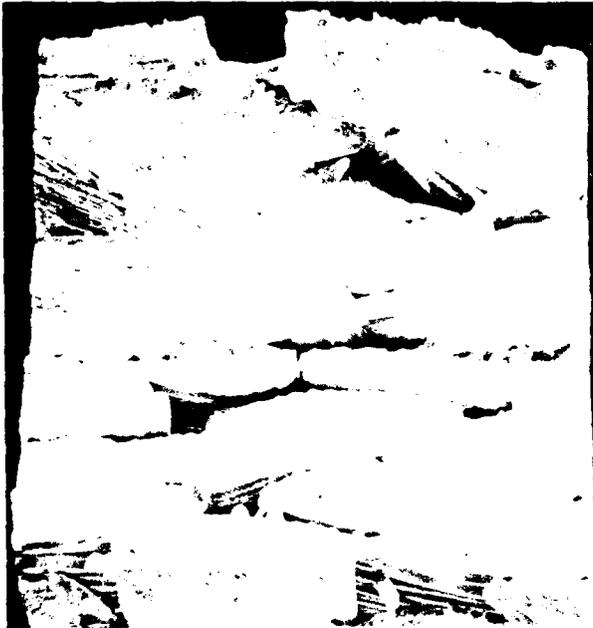


10 $\mu$ m



5 $\mu$ m

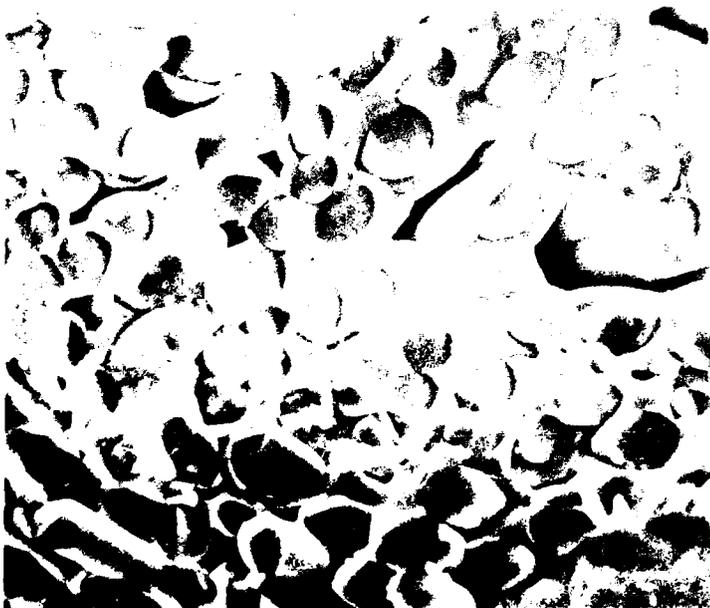
FRACTURE SURFACE OF ORNL CVI SiC MATRIX/NICALON FIBER COMPOSITE  
(RT $\sigma$  = 13 ksi (90 MPa))



400 $\mu$ m



100 $\mu$ m

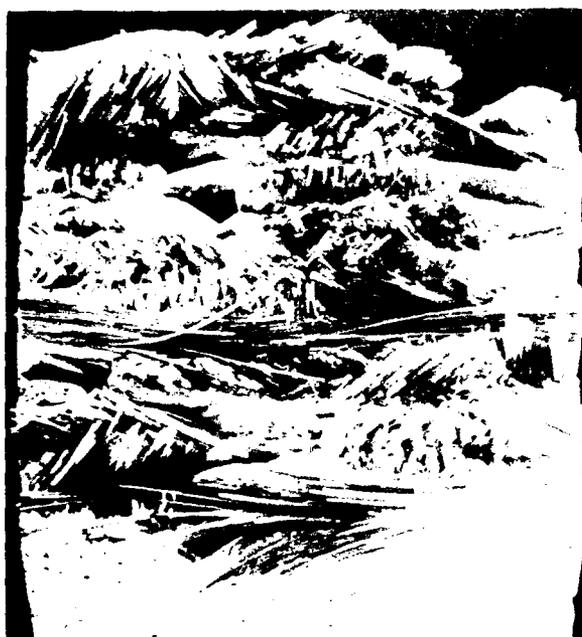


20 $\mu$ m

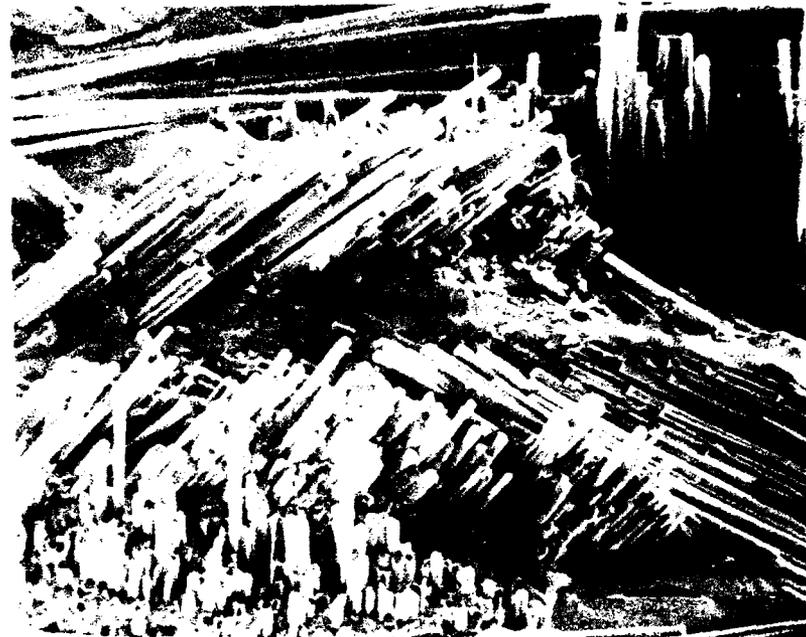


5 $\mu$ m

FRACTURE SURFACE OF A BN-C/SiC MATRIX/CARBON COATED NICALON FIBER COMPOSITE [OXIDIZED 1000 C. 70 hrs — RT = 11.2 ksi (77 MPa)]



100µm



100µm

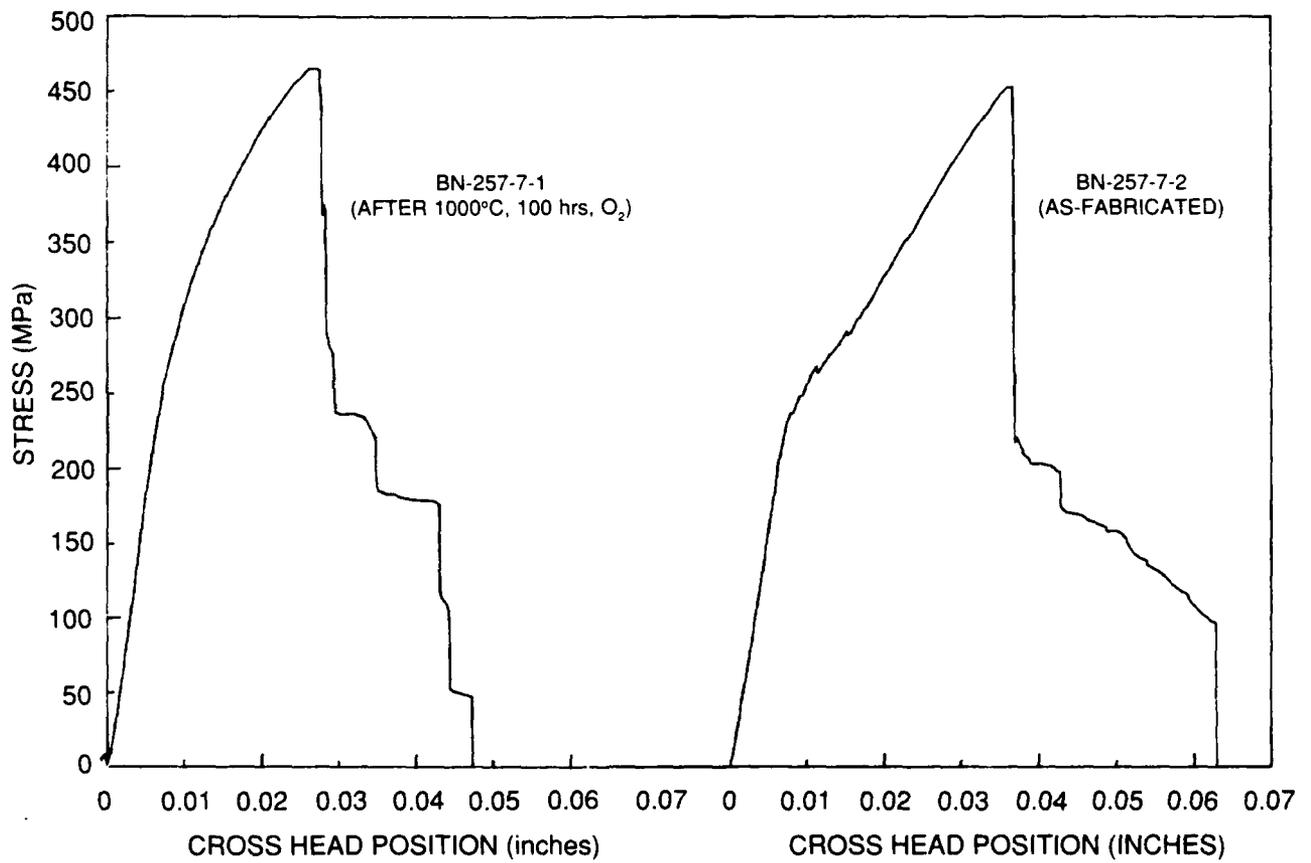


1µm



5µm

**STRESS VS CROSS-HEAD DISPLACEMENT FOR CVI SiC MATRIX/BN COATED NICALON FIBER COMPOSITE #257**



**RT FRACTURE SURFACE OF CVI SiC/BN COATED NICALON FIBER COMPOSITE #257-7-1  
[AFTER 1000°C, 100 hrs, FLOWING O<sub>2</sub> - RT  $\sigma$ =68 ksi (469 MPa)]**



200 $\mu$ m



EDGE OF SAMPLE

5 $\mu$ m



CENTER OF SAMPLE

5 $\mu$ m