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MODIFIED CARBON FIBERS TO IMPROVE COMPOSITE PROPERTIES

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FOREWORD

This document represents the final report of the work accomplished between March, 1978, and December, 1978, by Union Carbide Corporation, Parma Technical Center, for the National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia, under Contract NAS1-15283 entitled "Modified Carbon Fibers to Improve Composite Properties." This program was conducted under the technical direction of Mr. C. M. Pittman, Technical Representative, NASA Langley Research Center, Hampton, Virginia.

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1.0 SUMMARY

Thin coatings, 5 to 10 wt.%, were applied to PAN-based carbon fibers. These coatings were intended to make the carbon fibers less electrically conductive or to cause fibers to stick together when a carbon fiber/epoxy composite burned. The effectiveness of the coatings in these regards was evaluated in burn tests with a test rig designed to simulate burning, impact and wind conditions which might release carbon fibers. The effect of the coatings on fiber and composite properties and handling was also investigated.

Attempts at coating carbon fibers with silicon dioxide, silicon carbide and boron nitride meet with varying degrees of success. Still, none of these coatings provided an electrically nonconductive coating as had been hoped.

Coatings intended to stick carbon fibers together after a composite burned were sodium silicate, silica gel, ethyl silicate, boric acid and ammonium borate. Of these, only the sodium silicate and silica gel provided any sticking together of fibers. The amount of sticking was insufficient to achieve the objectives of this program. In all cases the amount of material present in the coatings was too small to cause the massive amount of sticking needed.

The coatings studied under this project gave reasonable translation of fiber properties into composite properties considering that no attempt was made to optimize this translation with the use of fiber sizings. One exception was the silicone fluid coatings which gave very poor translation of fiber properties.

Because of the lack of success of coatings and coating methods in this feasibility study, no further effort on these materials is recommended. Future effort might be better spent looking at matrix modifications or combinations of matrix modifications and fiber coatings.

2.0 INTRODUCTION

Fibrous reinforced composites have been in existence in one form or another for a long time. The most common occurrence in nature is in the form of wood. Man-made fibrous composites have come into abundant use only in recent years. Fiberglass reinforced plastics are the best known of the high performance man-made composites. More recently the so-called advanced composite materials, consisting mainly of carbon or boron fiber composites, have been used in aerospace applications as well as in the sporting goods industry. In the future, it is expected that carbon fibers will see increasing use and represent a significant proportion of the total amount of man-made fibers.

The projected increase in use of carbon fibers raises the question about possible electrical hazards posed by airborne electrically conductive carbon fibers. Carbon fibers have very small diameters, on the order of 7 microns, and thus, individual fibers or small clumps of fibers are easily airborne with a relatively small amount of air movement. Carbon fibers are also highly electrically conductive, especially in the axial direction of the fiber. Airborne fibers can easily settle on unprotected electrical equipment causing resistive loads and thus short circuiting.

The most common uses of carbon fibers are in plastic matrices. Compared with the carbon fibers, these plastic matrices are easily burned leaving the carbon fibers free to become airborne if conditions permit. Plastics with a low char value could especially release large amounts of fibers. Even small components can release large amounts of fibers since individual fibers are about 7 microns in diameter and typically occupy about 60% by volume in a reinforced plastic.

There are three basic ways the possible electrical hazards associated with burning carbon fiber composites might be circumvented: fibers made electrically nonconductive, plastic matrices modified to give large char values to hold fibers together, or all electrical equipment protected to prevent fiber interaction. The last method, while representing an impossibly large task when all the electrical devices and equipment in use by man today are considered, is the method used by producers of carbon fibers and fabricators of fiber-containing plastic compounds.

New or existing electrical equipment can be protected from airborne carbon fibers using materials and technology readily available today. While this can represent a modest effort by a producer or fabricator, it is an impractical approach for general protection.

That leaves the other two methods, modifying the fibers or modifying the matrices, as the only viable methods of circumventing the electrical hazard. Since much effort has gone into both fiber and matrix development, neither of these two methods offers an obvious, quick or straightforward solution. Both fibers and matrix materials have specific physical properties which are utilized to their fullest and to modify any aspect of these systems generally will mean an extended redevelopment effort.

The work reported on here is a feasibility study into a modification of carbon fibers which would not change present production procedures and not alter composite properties significantly. The modification consists of coatings on carbon fibers. These are thin coatings, being only 1-10 wt.% of the fiber, so the fibers can maintain their integrity and normal interactions with the matrix material. In order to limit the effort of this feasibility study so as to obtain some results in a limited amount of time, one fiber and one epoxy matrix system were used throughout. The fiber is "Thornel"* 300 grade WYP 30 1/0 and the epoxy is a proprietary Union Carbide material essentially the same as other aerospace-quality high-temperature curing epoxies.

The objective of this study was to apply coatings to the carbon fibers which would not significantly affect composite properties but would either form a high electrically resistive coating when the epoxy burned or cause fibers to stick together when the epoxy burned. Five tasks were proposed for this study to evaluate the feasibility of fiber coatings.

Task I was to evaluate silicone coatings. When a composite containing silicone coated fibers burned, the silicone would be converted to silicon dioxide. This silicon dioxide coating might either render the fibers less electrically conductive or cause fibers to stick and clump together.

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*"Thornel" is a registered trademark of Union Carbide Corporation.

Task II was to evaluate boron nitride coatings. Boron nitride possesses many properties similar to graphite except it is electrically nonconductive. Boron nitride coatings should not significantly alter the behavior of carbon fibers but might effectively make the fibers nonconductive.

Task III was to evaluate silicate coatings. When a composite containing silicate coated fibers burned, the silicate would be converted to silicon dioxide. As in Task I, the silicon dioxide might either render the fiber less electrically conductive or cause fibers to stick and clump together.

Task IV was to evaluate silicon carbide coatings. Like the boron nitride in Task II, it might make the carbon fibers effectively nonconductive.

Task V was to build a burn test chamber to evaluate the amount of fiber released when a composite burns and collect released fibers for examination. To simulate hostile conditions, the test chamber was to burn composite samples under various temperature, wind, and impact conditions.

3.0 EQUIPMENT AND PROCEDURES

3.1 Carbon Fiber

The carbon fiber yarn used in this study was "Thornel" 300 grade WYP 30 1/0. This is a PAN-based carbon fiber having 3000 filaments per strand. All yarn was water-sized during processing (no finish applied, only water for handling purposes) and came from the same lot number.

3.2 Coating Application

All coatings were applied from solutions by running the yarn, via a pulley, through the solution then into a drying tower. Weights per unit length of the water-sized starting material and the dried coated yarn were used to calculate weight percentages of coatings applied.

Immediately after coating, the yarns could be visually inspected for coating uniformity, broken filaments (fuzzy yarn), and bundle integrity (tightness of the strand bundle).

3.3 Strand Testing

Strand testing is done on the coated yarn to determine if the strength or modulus of the yarn has been affected by the coating. Strands are pulled in tension to get tensile strengths and tensile moduli. Small rods, see Figure 3.1, are made to get a torsional shear strength. The torsional shear test measures not only the shear strength of the fibers themselves but also the bond strength between the fiber and the epoxy matrix. A low torsional shear strength usually indicates that a low shear strength can be expected from short beam shear testing.

3.4 Plate Fabrication

Flat unidirectional composite plates were made from which specimens could be cut for composite physical property measurements. It is important that the fiber properties can be translated to the composite otherwise the idea of a thin coating which does not significantly alter fiber and matrix properties is no longer valid. Plates were made by drum winding epoxy impregnated yarn, hand lay-up of the resultant prepreg, and autoclaving to cure the final plate. From the unidirectional composite plates, burn test specimens were cut as well as the physical property specimens.

3.5 Composite Properties

Four sample geometries were used to obtain composite physical properties, see Figure 3.2. Tensile strengths and moduli were obtained from the tensile test, flexural strength from a four point flex test, compressive strength from a Texaco compression test, and shear strength from a short beam shear test. All results as reported in this study, except the short beam shear, are normalized to 60 vol.% carbon fiber.

Resin contents were determined by an acid ingestion method which at times suffered from a lack of reproducibility. Results reported here were determined by the more reproducible optical image analyzing computer from a polished composite cross section. It was important to be able to determine resin content with a degree of confidence since these data were used to normalize the physical property data.

3.6 Microscopy

Optical microscopy was used to examine fibers and composites. It aids in the visual inspection of coating and composite quality. The SEM (scanning electron microscope) was also used in examining coated yarns, composites and burn test remains. Specimens for use in the SEM were gold-coated to enhance image contrast and insure charge dissipation in the microscope. An energy dispersive elemental analyzer (Kevex) on the SEM was used to identify burn test residues. The Kevex analyzer is capable of identifying sodium and all elements with atomic numbers higher than sodium.

Some other methods employed in the analysis of coatings and burn test residues were X-ray diffraction, X-ray fluorescence, atomic absorption spectrometry, and standard wet chemistry analysis.

3.7 Electrical Resistance

Electrical resistance of carbon fibers were measured, before and after coating and on both single filaments and fiber bundles. Two and four probe methods of resistance measurement were used to separate contact resistance from carbon fiber resistance. Since the coatings studied here do not affect the resistance of the carbon fibers themselves, only the contact resistance can really be changed. Silver paint was used to make the electrical contacts for the resistance measurements.

The electrical circuit made by a coated carbon fiber is schematically represented in Figure 3-3. This representation is valid so long as the fiber length is much larger than the diameter which is the case in the electrical measurements done in this study.

The total electrical resistance of a coated fiber between points A and B is

$$R = 2\,R_3 + \frac{R_2\,(R_1 + 2\,R_4 + 2\,R_5)}{R_2 + R_1 + 2\,R_4 + 2\,R_5}$$

where
R_1 = resistance of fiber
R_2 = resistance of coating
R_3 = contact resistance between probe and coating
R_4 = contact resistance between coating and fiber
R_5 = radial resistance through coating.

Since $R_2 \gg R_5$ then R reduces to

$$R = 2\,R_3 + \frac{R_2\,(R_1 + 2\,R_4 + 2\,R_5)}{R_2 + R_1 + 2\,R_4}\,;$$

A four probe resistance measurement on an uncoated fiber gives R_1. A four probe resistance measurement on a coated fiber gives $\frac{R_1\,R_2}{R_1 + R_2}$, so R_2 can be calculated. A two probe measurement on a coated fiber gives R. For $R_2 \gg R_1$, which should be the case for a nonconductive coating, and $R_2 \gg R_4$, which should also be the case for a fiber of reasonable length, the expression for R reduces to

$$R = R_1 + 2\,R_3 + 2\,R_4 + 2\,R_5\,.$$

In order to increase R (by say an order of magnitude or more) over R_1, either the contact resistances, R_3 and R_4, have to be high or the resistance through the coating, R_5, has to be high.

3.8 Burn Testing

A chamber was built to burn test composite plates made with the coated carbon fibers, see Figure 3-4. The chamber consists of a stand to hold the composite plate, a gas fired torch to heat the plate, a device to drop weights on the plate, a fan to blow air across the plate, and a vented exhaust system with a filtered collector to gather airborne debris from the burn test. Conditions in the chamber were

variable to maxima of 850°C temperature and 12 m/sec wind velocity. Impact energies used in testing did not exceed 1.5×10^6 dyne • cm. Sample size was 37 mm by 62 mm and 8 plys (~ 1.5 mm) thick.

The chamber was made of stainless steel to make cleaning between tests easier and an access door in the front and a hinged top also facilitated cleaning. It was necessary to clean the chamber before testing each new sample so debris collected could be assured to be from the sample under test. A large window made it easy to view the sample under test while a smaller quartz glass window was used in sighting on the sample with a pyrometer.

Burn testing done in the above described chamber is designated dynamic burn testing to differentiate from the testing to be described below.

Burn testing was also done in an electric furnace at 1000°C. This is termed static burn testing because no impact or wind conditions were introduced. Both composites and coated yarn strands were tested this way. No specific sample size was needed for static burn testing since samples simply lay in a porcelain crucible.

4.0 RESULTS AND DISCUSSION

Results will be discussed initially by task number since the different tasks designate different coating systems. The control samples frequently referred to and appearing in tables for comparison were fabricated using uncoated water-sized "Thornel" 300. Control samples were checked at various points throughout this work to assure the carbon fiber yarn did not change significantly from spool to spool and to check on reproducibility of the data being generated.

4.1 Task I - Silicone Coatings

Under this task, water-sized "Thornel" 300 carbon yarn was coated with silicones. The coatings ranged from 1 to 14 wt.% of silicones over the carbon fibers. The silicones were from the class of dimethyl-silicone fluids (Union Carbide Corporation Grade L-45) and reactive

silicone fluids (Union Carbide Corporation Grade L-31). Coatings were applied from solvent based solutions. The coatings appeared very uniform, see Figure 4-1, and gave good bundle integrity to the yarn, see Figure 4-2. The uncoated control yarns can be seen in Figures 4-3 and 4-4. The coated yarn felt oily but handled well in the equipment. The coated yarns were static burn tested, see Figure 4-5. Figure 4-6 shows the uncoated control yarn after static burn testing. The silicones did not significantly affect fiber tensile strength or modulus but drastically reduced torsional shear strengths, see Table 4-1. The decrease in torsional shear strength results because the epoxy matrix material does not wet the silicone coated yarns. Changes in solvents and coating drying conditions did not change the nonwetting aspects of these systems. Since useful composites could not be fabricated using epoxies, no further work was done with silicone coatings.

4.2 Task II - Boron Nitride Coatings

Boron nitride coatings were applied using two different techniques. In one technique, an ammonium borate-water solution was applied to the yarn. After the yarn was dried, it was run through a nitriding furnace at temperatures from 1200 to 1500°C. The furnace atmosphere was nitrogen. The boron nitride coating, thus produced, caused excessive sticking between carbon filaments and resulted in many breaks in the otherwise continuous yarn. Processing parameters were changed to reduce yarn breakage so the coating system could be evaluated, but many broken filaments (fuzzy yarn) and much fiber sticking still existed in the yarn. The yarn was difficult to handle but composite plates were made for further testing.

The other boron nitride coating technique consisted of applying a boric acid-water solution to the yarn, then heat treating the yarn to 900 to 1000°C in an ammonia atmosphere. The coated yarn looked and performed very similar to that coated using the ammonium borate technique; it contained many stuck together and broken filaments. Thus, boron nitride from boric acid coated yarn was only evaluated for strand and torsional shear properties. These tests and yarn inspection indicated

that it should perform essentially the same as the other boron nitride coated yarn so no further work on this coating was done.

Both boron nitride coatings gave the yarn good bundle integrity but poor handleability. The coatings were nonuniform as can be seen in Figure 4-7. The sticking of individual filaments can also be seen in Figure 4-7. The nonuniformity of the coatings was not expected after viewing micrographs of the ammonium borate and boric acid coated yarns, Figure 4-8. It can be seen that at this initial stage of processing the coatings are relatively uniform. Any handling of the ammonium borate yarn at this intermediate stage resulted in a flaking or dusting of the coating which contributed to its nonuniformity.

Static burn testing of the coated yarn at 1000°C in air showed little fiber oxidation, Figure 4-9, which would be expected since the oxidation resistance of boron nitride is generally much better than carbon.

Strand and torsional shear testing of the boron nitride coated yarns indicates the basic properties of the carbon yarn are changed only slightly as can be seen in Table 4-2.

Electrical resistance as measured on single filaments and yarn strands is shown in Table 4-3. The boron nitride coating does not change the resistance relative to the uncoated fibers to any appreciable degree. Most changes indicated in Table 4-3 could be attributed to experimental fluctuations or changes in yarn density. Since the boron nitride coatings are nonuniform and the silver paint used to establish electrical contact in this test covers a large portion of the fiber surface under test, electrical contact is most certainly always made on areas of the fiber unprotected by the coating. Thus, no changes in the electrical resistance due to the boron nitride coating would be expected.

An attempt was made to verify that the coating on the fibers really was boron nitride and not a boron oxide or boron carbide.

Elemental analysis could detect boron near expected levels but X-ray diffraction analysis could not detect either boron nitride, boron oxides, or boron carbide over the large background count of the graphite. An electron microprobe was also used to try and map the concentration of boron over the surface of a carbon fiber to determine whether or not a continuous coating existed along with the apparent noncontinuous one. Due to the small amounts of boron present and the fact that boron was at the very edge of the instrument's detection limits, the desired mapping could not be obtained.

Burn testing of composite materials made with boron nitride coated fibers was done in both the static and dynamic atmospheres. Composites burned at 1000°C in a static atmosphere oxidized slightly, see Figure 4-10. Composites made with water-sized fibers oxidized severely as can be seen in Figure 4-11. Composites burn tested dynamically also behaved similar to uncoated control samples. At these temperatures the boron nitride coatings protected the carbon fibers from oxidation but not to the extent seen in some of the other coatings. The dynamic burn test residues shown in Figure 4-12 contain epoxy char and the cooler the area of the sample examined, the more epoxy char was present. Figure 4-13 shows results of dynamic burn testing on a composite made with control yarn. In areas of the sample where temperatures were the hottest, very little epoxy char remained and the fiber oxidation was the most severe. The epoxy char holds carbon fibers together when the composite is burned at lower temperatures. At higher temperatures, when the epoxy char becomes minimal, the fibers themselves oxidize, so very little resultant fiber fly occurs. The amount of fibers released in the air and trapped by the filtration/collection system of the dynamic burn test chamber was so small that it could not be measured in the experiments reported on here.

4.3 Task III - Silicate and Boron Nitride Precursor Coatings

The coatings studied under this task included sodium silicate, a silica gel suspension ("Ludox"* AM), ethyl silicate and boric acid and

*"Ludox" is a registered trademark of E. I. DuPont de Nemours Corporation.

ammonium borate (the first stages of the boron nitride coating process). Originally, it was thought that some of these coatings might act to increase the electrical resistance of a coated carbon fiber. After preliminary examinations it was obvious that some of these coatings were very discontinuous and could only be of help if they fused fibers together from a burned composite.

All the coatings studied under this task improved the bundle integrity to some extent over the water-sized yarn but not as well as the standard "Thornel" 300 yarn with UC-309 size, see Figure 4-14. Some fuzzing of the "Thornel" 300 yarn occurred with these coatings, but they could all be handled with normal carbon fiber handling techniques. The ethyl silicate coating appeared quite uniform as can be seen in Figure 4-15. The boric acid and ammonium borate coatings were uniform as was shown in Figure 4-8. The sodium silicate and especially the silica gel coatings were nonuniform as is evident from Figure 4-16. The sodium silicate, silica gel, and ammonium borate coatings dusted and flaked off some with normal yarn handling which led to further coating nonuniformity.

The strand and torsional shear properties of carbon fibers with the coatings under Task III are shown in Table 4-4. Ethyl silicate and boric acid coated fibers still retained good physical properties. The silica gel coated fibers showed some degradation and the sodium silicate coatings were rather poor. The ammonium borate coatings gave the worst translation of fiber properties.

The coated fibers were static burn tested at 1000°C. As can be seen in Figure 4-17, most all of these coatings protected the fibers from oxidation except the ethyl silicate and to a smaller extent the silica gel. Since the sodium silicate coating protected the carbon fibers from oxidizing so well, the coating must be somewhat uniform but with regions of much thicker material, i.e., lumps, see Figure 4-16. The ethyl silicate coating must vaporize before it can form silica residues because the static burn tested fibers do not look significantly different than the uncoated control, Figure 4-6.

Epoxy composite plates were made with sodium silicate, silica gel, ethyl silicate and boric acid coated carbon fibers. Physical property data for these plates are shown in Table 4-5. Plates were not made with the ammonium borate coating because of the poor torsional shear strengths measured during strand testing. The composite properties of the plates made did not differ significantly from those obtained on the control plates except for a noticeable decrease in the shear strength for the sodium silicate coating.

Samples of these plates were burn tested in the dynamic burn test chamber under different temperature, wind velocity and impact conditions. Since very few airborne fibers were released during testing and the epoxy matrix char held fibers together quite well except at the highest temperatures, only the highest temperatures and largest wind velocity results will be discussed in detail.

The sodium silicate coated fiber composite is shown in Figure 4-18 after dynamic burn testing. Upon impact, the composite separated into individual yarn strands as shown. There was virtually no fiber fly, but the yarn strands were quite easily fuzzed if worked by rubbing between the fingers. Any fuzzing visible in Figure 4-18 is due to the handling of the specimen in removing it from the burn test chamber and all subsequent handling.

The sodium silicate coating apparently protected the carbon fiber from burning because no fiber burning was noticed during or after the burn test; and SEM examination, see Figure 4-18, showed no evidence of burned fibers. The residue from the coating can be seen sticking some fibers together but, in general, does not wet the individual fibers. These clumps of material show a high sodium and silicon content when probed with the Kevex analyzer.

The burned composite made with silica gel coated carbon fibers is shown in Figure 4-19. The remains of the composite itself do not look much different than those of Figure 4-18. A small amount of fiber burning was noticed during testing, and the evidence of individual

filament burning can be seen in the SEM micrographs of Figure 4-19. The coating residue contains large amounts of silicon as determined by Kevex analysis. The residue appears in lumps which do not wet the fiber surface and cause only minimal sticking together of filaments.

The ethyl silicate samples behaved essentially the same as the water-sized control samples during burn testing. Extensive carbon fiber burning took place during the burn test. As can be seen in Figure 4-20, SEM analysis showed evidence of burned fibers. There is little indication of any of the coating material remaining after burn testing. The only parts of the composite where filament-to-filament sticking took place were in cooler regions of the sample where significant epoxy char remained after the burn test.

Figure 4-21 shows the composite made with boric acid coated carbon fibers after burn testing. The boric acid afforded oxidation protection to the fibers because only a small amount of fiber burning was noticed during the burn test, and SEM analysis showed evidence of fiber burning only in the hottest regions of the composite sample. There was very little sticking together of filaments due to the applied coating. Most of the sticking observed could be attributed to the epoxy char.

4.4 Task IV - Silicon Carbide Coatings

Silicon carbide coatings for the "Thornel" PAN-based carbon fibers were applied in a two-step process. The first step involved applying coatings of dimethyl-silicone fluids and reactive-silicone fluids as in Task I of this project. The same silicone fluids used for Task I were used in Task IV. The second step of the process sent the silicone coated carbon fibers through a 1500°C furnace under an inert atmosphere to convert the silicone fluids to a silicon carbide coating.

The silicon carbide coating process led to breaking of individual filaments so the yarn was fuzzy and required some extra care in handling. Even though the yarn contained broken ends, the bundle integrity was improved over the control specimens, see Figure 4-22. The coatings appeared quite uniform when examined with the SEM as can be seen in Figure 4-23.

Attempts to determine how much of the coating was really silicon carbide and how much might be silicon dioxide were not successful. Standard X-ray diffraction analysis could not detect either silicon carbide or silicon dioxide peaks over the large graphite background. Likewise, attempts at mapping the silicon concentration on the fiber surfaces using an electron microprobe were not successful.

Strand data for the silicon carbide coatings are given in Table 4-6. A slight drop in the torsional shear strength did occur for some samples. The weight percents of the silicon carbide coatings given in Table 4-6 may be low due to broken and lost filaments, yet ash analysis of these fibers indicates that the amount of silicon carbide may be even smaller than that given in Table 4-6, more on the order of 0.5 wt.%. The coating thickness applied here is about the maximum attainable using these techniques.

Static burn testing at 1000°C of the silicon carbide coated carbon fibers indicated that the coatings did not protect the carbon fibers from oxidation, see Figure 4-24. This complements the composite burn testing to be discussed below which produced extensive fiber burning.

The electrical resistance of single filaments and yarn strands is shown in Table 4-3. As with the boron nitride coatings, no significant increase in the electrical resistance of coated fibers was achieved. In fact, the electrical resistance of the silicon carbide samples from the reactive silicone fluid (L-31) appears to have actually decreased the electrical resistance of the coated carbon fiber.

Physical property data obtained from epoxy composite plates made with the silicon carbide coated yarns are given in Table 4-7. A significant drop in compressive strength is seen along with a noticeable decrease in short beam shear strength. Since some minor problems were encountered in wetting the coated fibers with the epoxy, it is expected that an epoxy compatible finish on these fibers might result in better translation of fiber properties, especially the compressive strength.

Samples of these plates were burn tested in the dynamic burn test chamber. They behaved essentially the same as the uncoated control samples. Extensive fiber burning took place; the samples continued to burn for approximately five minutes after the burn test rig torch was turned off. No difference in performance between the two silicon carbide coatings was discernible. Figures 4-25 and 4-26 show the composites after burn testing and SEM micrographs of the burn test residues. SEM examination indicated that, just as with the control samples, when temperatures got high enough to reduce the epoxy char so fiber release might take place, fiber burning took place. Enough fiber burning took place so that only a few fibers were found in the exhaust collection screens even though only about 25% of the fibers remained in the sample holder after some burn tests.

4.5 Control Fibers

Throughout this study, the water-sized "Thornel" 300 yarn has been used as the control. Physical properties of this yarn and plates made with it have appeared in Tables 4-1, 4-3, and 4-5. Photographs and micrographs have appeared in Figures 4-3, 4-4, 4-6, 4-11, and 4-13. This yarn was picked for the control because the same yarn could be coated with the different materials under consideration without introducing the complicating factor of another coating already on the yarn. But water-sized yarn is not, in general, used to make composites. Most yarns have some kind of a surface finish to aid in handling and to promote good bonding between the carbon fiber and the matrix material in a composite. "Thornel" 300 yarn is normally used with an epoxy compatible finish designated as UC-309 (a Union Carbide Corporation proprietary material).

In order to provide a frame of reference for some of the work done here, "Thornel" 300 yarn with UC-309 finish was evaluated in the same way as the other materials in this project. The physical properties of this yarn are given in Table 4-6, and the physical properties of epoxy composite plates made with this yarn are given in Table 4-7. As can be seen, this yarn gives very good translation of fiber properties in the composite. This is to be expected since the UC-309 finish was developed for that purpose. Figure 4-27 shows the excellent bundle integrity of the

UC-309 sized yarn. Burn testing of samples with UC-309 finish gave the same results as burn testing with samples containing the water-sized carbon fiber. Photographs of burn residues from UC-309 sized yarns and water-sized yarns are indistinguishable.

4.6 Electrical Resistance

The electrical resistances of carbon fibers and coated carbon fibers and their measurement deserve additional comment. One of the initial objectives of this program was to coat the electrically conductive carbon fibers with an electrically nonconductive coating. In order for the coating to make the fiber effectively nonconductive, it must be a continuous coating. Discontinous coatings allow electrical contact to be made with the carbon fiber itself at the points of discontinuity and thus defeat the purpose of the nonconducting coating.

Assuming a coating is uniformly continuous, it must have a high enough resistivity to be effective as an electrical insulator. The data in Table 4-3 shows that an uncoated carbon filament has a resistance of approximately 4000 ohms over a 2.5 cm distance. The diameter of the filament is 7 microns which gives a fiber resistivity of 6.2×10^{-4} ohm $\cdot$cm. Neglecting for the moment contact resistances, to increase the resistance of the fiber by one order of magnitude with a coating comprising 5 wt.% of the fiber, which makes the coating on the order of 8×10^{-6} cm thick, the necessary resistivity of the coating depends on total area of surface contact made by the electrical charge carrier. For the contact made by silver paint completely surrounding the 7 micron fiber for a length of 0.2 cm, the area of electrical contact is 4.4×10^{-3} cm^2. This means the coating must have a resistivity of 40,000 $(4.4 \times 10^{-3})/8 \times 10^{-6} = 2 \times 10^{7}$ ohm $\cdot$cm to provide the one order of magnitude increase in resistance. If the area of electrical contact were very small, like from contact with another fiber, it might have an effective contact area on the order of 2.5×10^{-7} cm^2. Then the resistivity of the coating would be on the order of 40,000 $(2.5 \times 10^{-7})/8 \times 10^{-6} = 1 \times 10^{3}$ ohm $\cdot$cm for a one order of magnitude increase in resistance.

To increase the resistance by four orders of magnitude over that of the uncoated fiber would require a coating resistivity of 2×10^{10} ohm • cm for the silver paint case and 1×10^{6} ohm • cm for the small contact area case. Silicon carbide with a resistivity of only 10 ohm • cm would not provide sufficient electrical shielding for any of the cases considered above. Silicon dioxide with a resistivity on the order of 10^{14} ohm • cm and boron nitride with a resistivity of 10^{13} ohm • cm would provide shielding to the extent discussed above.

On the other hand, silicon dioxide has a dielectric breakdown strength on the order of 3×10^{5} volts/cm and boron nitride on the order of 2×10^{5} volts/cm. For a 8.6×10^{-6} cm thick coating, this corresponds to a breakdown voltage of approximately 3 volts for silicon dioxide and 2 volts for boron nitride. These voltages are very low compared to the voltages of many circuits needing protection from airborne carbon fibers.

In the above discussion, contact resistances were neglected. The resistivities of silicon dioxide and boron nitride are so high that contact resistances on the order of those measured in this study can be neglected. For the silicon carbide case, the resistivity is so low that the opposite is true. The contact resistances are probably more significant than the resistance through the silicon carbide coating.

The contact resistances are those described earlier as R_3 and R_4 while the resistance through the coating is that described as R_5.

It is not unreasonable in hindsight to see why the various coatings could not provide the necessary electrical shielding: the silicon dioxide coatings produced when composites burned were very discontinuous; the boron nitride coatings were likewise discontinuous; and the silicon carbide coatings (if continuous) were far too thin. Because of this, more effort in this program was directed toward coatings which would cause the individual carbon fiber filaments to stick together when a composite burns.

4.7 Filament Sticking

Individual carbon fiber filaments must be free to slide over one another during composite part fabrication and all handling prior to this stage. Any sticking filaments in these stages reduce the optimum fiber properties. If carbon fiber filaments are to stick together when a composite burns and prevent fiber fly, then the sticking must take place when the composite matrix burns. In order for filament sticking to prevent airborne carbon fibers, a sufficiently large mass of carbon fibers must be stuck together. The size of this mass will depend on the external conditions contributing to fiber release. In general though, the mass will most certainly have to contain more than a few (tens) of fibers.

The coatings examined under this study which were to promote filament sticking upon composite burning did not provide sufficient sticking to even meet the intuitive minimum discussed above. Only two coatings, sodium silicate and silica gel, really provided any significant filament sticking. These two coatings might have produced more sticking had they wet the carbon fiber surfaces better. Under the conditions presented in this study, the sodium silicate and silica gel coatings were present in insufficient amounts to cause enough carbon fiber sticking to prevent airborne carbon fibers under adverse burning, impact, and wind conditions. To increase the amount of coating beyond that discussed here would degrade the translation of fiber properties to composite properties to the point where carbon fiber benefits could not be realized.

5.0 CONCLUSIONS

The conclusions presented here apply only to the present feasibility study on "Thornel" 300 grade WYP 30 1/0 and the Union Carbide Corporation proprietary high-temperature aerospace quality epoxy matrix materials.

None of the coatings examined here provided significant electrical protection for airborne carbon fibers. In particular, the coatings examined were boron nitride and silicon carbide formed before composite fabrication and silicon dioxide formed *in situ* when a composite burns.

The silicon carbide coating does not have high enough resistivity to be effective as a thin coating. The silicon dioxide and boron nitride, while having a high enough resistivity, were not uniform and continuous coatings and thus could not function effectively.

Two coatings, sodium silicate and silica gel, provided a small amount of carbon fiber sticking after composite burning. The amount of sticking obtained utilizing thin coatings was insufficient to prevent fiber fly under adverse burning, impact, and wind conditions.

Coatings from silicone fluids were found to be impractical because epoxy will not wet the carbon fibers and thus composites cannot be fabricated.

Ammonium borate, boric acid, and ethyl silicate coatings provided no benefits under the objectives of this study.

Burn testing of composites using the burn test chamber built under this study resulted in very little airborne carbon fibers being produced, even for uncoated control samples. The following burn test observations were made: the epoxy char after burn testing was sufficient to hold fibers together at burn temperatures approaching 700°C; above 700°C the carbon fibers burned before they could be released in sufficient quantities, and the filter collection system in the exhaust of the burn test chamber collected such a small amount of carbon fibers that they were obscured by the soot particles. Burn testing took place at temperatures to 850°C, impact energies to 1.5×10^6 dyne • cm, and air velocities to 12 m/sec (27 mi/hr wind).

A more severe working, i.e., grinding type forces, of burned carbon fiber epoxy composites than that studied here is necessary to release significant amounts of airborne fibers.

6.0 RECOMMENDATIONS

No further work on the coatings studied here is recommended.

Thin coatings alone do not appear to be the solution to airborne carbon fiber electrical problems.

Thin coatings, such as some of those studied here, may be successful at sticking fibers together if an appropriate material were also added to the epoxy matrix. For instance, low melting glass particles added to the matrix might interact with thin coatings to provide a sufficient amount of material to cause massive fiber sticking when a composite burns.

Another technique of applying thin coatings with very high resistivity, silicon dioxide or boron nitride, might make carbon fibers effectively less electrically conductive if the coatings can be continuous and extremely uniform. Such a technique might be chemical vapor deposition.

In general, work toward increasing the char value of composite matrix materials may hold the greatest chance of success for reducing carbon fiber fly and its associated electrical problems.

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Table 4-1

Strand Test Data for Silicone Coatings

Coating	Wt.%	Tensile Strength GPa (ksi)	Young's Modulus GPa (Msi)	Torsional Shear Strength MPa (ksi)
Control[1] A	--	2.68 (389)	219 (31.8)	103 (14.9)
Control[1] B	--	2.47 (358)	219 (31.8)	101 (14.6)
Control[1] C	--	2.65 (384)	254 (36.8)	100 (14.5)
Control[1] D	--	2.81 (407)	189 (27.4)	104 (15.1)
Control[1] E	--	2.71 (393)	224 (32.5)	104 (15.1)
Control[1] F	--	2.66 (385)	191 (27.7)	100 (14.5)
Silicone L-45[2]	8	1.74 (252)	187 (27.1)	26 (3.8)
Silicone L-45[2]	14	1.35 (196)	179 (26.0)	28 (4.1)
Silicone L-45[3]	5	2.22 (322)	227 (32.9)	49 (7.1)
Silicone L-45[3]	5	1.60 (232)	168 (24.4)	could not wet
Silicone L-31[4]	1	2.42 (351)	214 (31.0)	38 (5.5)
Silicone L-31[4]	5	1.84 (267)	197 (28.6)	29 (4.2)
Silicone L-31[4]	13	1.67 (242)	178 (25.8)	46 (6.7)
Silicone L-31[4]	5	2.11 (306)	216 (31.2)	could not wet
Silicone L-31[4]	5	2.29 (332)	181 (26.3)	could not wet

Notes:
1. Controls are water-sized "Thornel" 300.
2. Union Carbide Corporation dimethyl-silane Grade L-45 in kerosene.
3. Union Carbide Corporation dimethyl-silane Grade L-45 in ethyl acetate.
4. Union Carbide Corporation reactive silicone Grade L-31 in isopropanol.

Table 4-2

Strand Test Data for Boron Nitride Coatings

Coating	Wt.%	Tensile Strength GPa (ksi)	Young's Modulus GPa (Msi)	Torsional Shear Strength MPa (ksi)
From ammonium borate	3	2.15 (312)	249 (36.1)	72 (10.4)
From boric acid	1*	1.43 (208)	225 (32.7)	103 (14.9)
" " "	1*	2.26 (328)	199 (28.9)	100 (14.4)
" " "	1*	2.23 (324)	187 (27.1)	98 (14.3)

*These values are low because of broken filaments and fiber loss. Real values are probably in range 3-5 wt.%.

Table 4-3

Electrical Resistance of Single Fibers and Fiber Bundles

Sample	Single Filament (ohms)		Bundle (ohms)	
	2 probe	4 probe	2 probe	4 probe
Control B	4940 ± 180	4070 ± 150	--	--
Control C	4790 ± 330	3930 ± 290	--	--
Control D	5690 ± 520	4430 ± 330	3.60 ± .30	3.80 ± .10
Control E	5670 ± 290	4470 ± 260	3.70 ± .40	3.80 ± .20
Boron nitride (3 wt.%)	4580 ± 350	3640 ± 250	2.63 ± .21	3.03 ± .30
Silicon carbide (L-45)	5330 ± 340	4240 ± 200	4.40 ± .50	3.90 ± .10
Silicon carbide (L-31)	4450 ± 310	3630 ± 190	2.35 ± .10	2.66 ± .09

Table 4-4

Strand Test Data for Silicate Coatings Under Task III

Coating	Wt.%	Tensile Strength GPa (ksi)	Young's Modulus GPa (Msi)	Torsional Shear Strength MPa (ksi)
Sodium silicate	5	2.53 (367)	191 (27.7)	83 (12.0)
" "	8	2.14 (310)	185 (26.8)	78 (11.3)
" "	10	2.06 (299)	186 (27.0)	78 (11.3)
" "	5	2.29 (332)	215 (31.1)	85 (12.4)
Silica gel	6	2.47 (358)	214 (31.0)	71 (10.3)
Silica gel + X-1042*	9	2.37 (344)	206 (29.9)	85 (12.3)
" " " "	9	2.42 (350)	214 (31.1)	102 (14.8)
Ethyl silicate	3.5	2.52 (365)	249 (36.1)	102 (14.8)
" "	3.5	2.51 (364)	240 (34.8)	104 (15.0)
" "	4.5	2.74 (397)	229 (33.2)	105 (15.2)
Ammonium borate	3	2.63 (381)	207 (30.0)	--
" "	6	2.34 (339)	215 (31.2)	69 (10.0)
" "	10	2.26 (328)	225 (32.6)	70 (10.2)
" "	5	2.33 (338)	223 (32.3)	--
Boric acid	5	2.52 (365)	208 (30.2)	102 (14.8)
" "	4	2.78 (403)	214 (31.0)	104 (15.1)
" "	6.5	2.41 (349)	256 (37.1)	98 (14.2)
" "	7	2.08 (302)	233 (33.7)	74 (10.7)

*Union Carbide Corporation Latex X-1042.

Table 4-5

Composite Plate Properties for Task III and Controls

Sample	Vol.% Fiber	Tensile Strength* GPa (ksi)	Tensile Modulus* GPa (Msi)	Flexural Strength* GPa (ksi)	Texaco Compressive Strength* GPa (ksi)	Short Beam Shear Strength MPa (ksi)
Control A	58	1.34 (194)	124 (18.0)	1.55 (224)		
" "	57				1.52 (219)	95 (13.7)
Control B	62	1.35 (196)	131 (18.9)	1.55 (225)		
" "	63				1.34 (194)	89 (12.9)
Control D	62	1.39 (201)	126 (18.3)	1.49 (216)		
" "	57				1.60 (232)	94 (13.7)
Control E	60	1.39 (201)	131 (18.9)	1.58 (230)		
" "	62				1.54 (224)	99 (14.4)
Sodium silicate	51	1.53 (222)	132 (19.1)	1.61 (235)		
" "	57				1.23 (179)	75 (10.9)
Silica gel	45	1.49 (217)	141 (20.6)	1.64 (238)		
" "	52				1.27 (185)	93 (13.4)
Ethyl silicate	59	1.42 (206)	140 (20.2)	1.59 (231)		
" "	56				1.67 (243)	104 (15.1)
Boric acid	47	1.73 (250)	155 (22.4)	1.91 (276)		
" "	55				1.58 (229)	94 (13.7)

*These values normalized to 60 vol.% fiber loading.

Table 4-6

Strand Test Data for Silicon Carbide Coatings

Coating	Wt.%	Tensile Strength GPa (ksi)	Young's Modulus GPa (Msi)	Torsional Shear MPa (ksi)
From L-45	0.7	2.48 (260)	264 (38.3)	95 (13.8)
From L-31	0.6	2.56 (371)	257 (37.3)	94 (13.6)
From L-45	0.8	2.48 (360)	227 (32.9)	77 (11.2)
From L-31	0.7	2.55 (370)	229 (33.2)	99 (14.3)
UC 309	1	3.11 (451)	238 (34.5)	106 (15.3)

Table 4-7

Composite Plate Properties for Task IV

Sample	Vol.% Fiber	Tensile Strength* GPa (ksi)	Tensile Modulus* GPa (Msi)	Flexural Strength* GPa (ksi)	Texaco Compressive Strength* GPa (ksi)	Short Beam Shear Strength MPa (ksi)
Silicon carbide (L-45)	64	1.34 (195)	124 (18.0)	1.37 (199)		
" " "	65				1.04 (150)	72 (10.4)
Silicon carbide (L-31)	71	1.27 (184)	125 (18.1)	1.60 (232)		
" " "	72				0.96 (140)	78 (11.3)
T-300 w/UC 309	60	1.72 (249)	143 (20.7)	1.76 (255)		
" " "	57				1.46 (211)	101 (14.6)

*These values normalized to 60 vol.% fiber loading .

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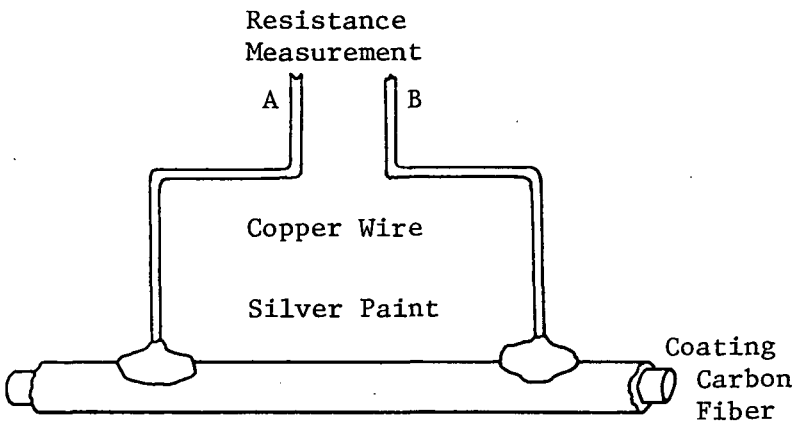

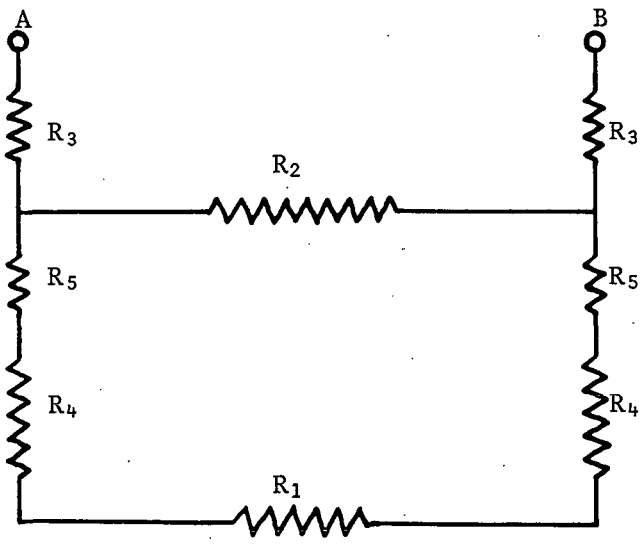

Figure 3-3. The Measurement of the Electrical Resistivity of a Coated Carbon Fiber can be Schematically Represented as shown where:

R_1 = resistance of fiber,

R_2 = resistance of coating,

R_3 = contact resistance between probe and coating,

R_4 = contact resistance between coating and fiber,

R_5 = resistance radially through coating.

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FIGURE CAPTIONS

Figure 3-1. Torsion Rod Specimen. Scale in Inches.

Figure 3-2. Physical Property Sample Geometrics. Scale in Inches.
a) Tensile Specimen with Fiberglass Tabs.
b) Texaco Compression Sample.
c) Flexural Specimen.
d) Short Beam Shear Sample.

Figure 3-3. The Measurement of the Electrical Resistivity of a Coated Carbon Fiber can be Schematically Represented as Shown Where:

R_1 = resistance of fiber
R_2 = resistance of coating
R_3 = contact resistance between probe and coating
R_4 = contact resistance between coating and fiber
R_5 = resistance radially through coating.

Figure 3-4. Dynamic Burn Test Chamber.
a) Chamber Showing Collection System.
b) Temperature Measurement.
c) Sample Under Test.

Figure 4-1. SEM Micrographs of Carbon Fibers Coated with Silicone Fluids.
a) UCC L-45 Silicone Fluid, 8 wt.%, Applied from Kerosene Solution. 2000X
b) UCC L-31 Silicone Fluid, 5 wt.%, Applied from Isopropanol Solution. 2000X

Figure 4-2. Silicone Coatings, Showing Bundle Integrity, Scale is Centimeters.
a) UCC L-45 Silicone Fluid, 8.4 wt.%, Applied from Kerosene Solution.
b) UCC L-31 Silicone Fluid, 4.6 wt.%, Applied from Isopropanol Solution.

Figure 4-3. SEM Micrograph of Uncoated Water-Sized "Thornel" 300. 2000X

Figure 4-4. Uncoated Water-Sized "Thornel" 300 Showing Bundle Integrity, Sacle is Centimeters.

Figure 4-5. Silicone Fluid Coated Carbon Fibers After 1000°C Static Burn for 30 Seconds.
a) UCC L-45 Silicone Fluid Coating, 14 wt.%. 2000X
b) UCC L-31 Silicone Fluid Coating, 13 wt.%. 2000X

Figure 4-6. SEM Micrograph, Water-Sized "Thornel" 300 After 1000°C Static Burn for 30 Seconds. 2000X

Figure 4-7. SEM Micrograph of Boron Nitride, from Ammonium Borate, Coating on Carbon Fibers. 5000X

Figure 4-8. SEM Micrographs of Coated Carbon Fibers.
a) Boric Acid Coating, 5 wt.%. 2000X
b) Ammonium Borate Coating, 10 wt.%. 2000X

Figure 4-9. SEM Micrograph of Boron Nitride, from Ammonium Borate, Coated Carbon Fibers After 30 Seconds Static Burn Test at 1000°C. 400X

Figure 4-10. SEM Micrograph of Boron Nitride Coated Fiber Composite After 1000°C Static Burn Test for 30 Seconds. 1350X

Figure 4-11. SEM Micrograph of Water-Sized "Thornel" 300 Fiber Composite After 1000°C Static Burn Test for 30 Seconds. 1200X

Figure 4-12. Boron Nitride, from Ammonium Borate, Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.
b) SEM Micrograph from Cooler Corner of Composite During Burn Test. Epoxy Char is Holding Fibers Together. 180X
c) SEM Micrograph from Hottest Area of Composite Where Fiber Burning was Taking Place. 180X
d) Kevex Trace of Burned Composite Showing Gold Coating and Small Silicon Background.

Figure 4-13. Water-Sized "Thornel" 300 Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 11 M/sec. Air Velocity. Approximately 60% of Fibers Burned Away.
b) SEM Micrograph from Cooler Area of Composite During Burn Test. Epoxy Char Holds Fiber Together. 120X
c) SEM Micrograph of Fibers from Hotter Area of Sample Where Fiber Burning was Taking Place. 2700X
d) Kevex Trace of Burned Composite Showing Gold Coating and Small Silicon Background.

Figure 4-14. Task III Fiber Coatings Showing Bundle Integrity. Scale is Centimeters.
a) Sodium Silicate Coated Fiber, 5 wt.%.
b) Silica Gel Coated Fibers, 6 wt.%.
c) Ethyl Silicate Coated Fibers, 3.5 wt.%.
d) Boric Acid Coated Fibers, 5 wt.%.
e) Ammonium Borate Coated Fibers, 10 wt.%.

Figure 4-15. SEM Micrograph of Ethyl Silicate, from an Isopropanol Solution, Coated Carbon Fibers. 1600X

Figure 4-16. SEM Micrograph of Sodium Silicate and Silica Gel Coated Carbon Fibers.
a) Sodium Silicate, 10 wt.%, Coated Carbon Fiber. 2000X
b) Silica Gel, 6 wt.%, Coated Carbon Fiber. 2000X

Figure 4-17. SEM Micrograph of Task III Coatings on Carbon Fibers After 1000°C Static Burn Test for 30 Seconds.
a) Sodium Silicate, 10 wt.%, Coated Fibers. 2000X
b) Silica Gel, 6 wt.%, Coated Fibers. 2000X
c) Ethyl Silicate, 3.5 wt.%, Coated Fibers. 1600X
d) Boric Acid, 7 wt.%, Coated Fibers. 1800X
e) Ammonium Borate, 10 wt.%, Coated Fibers. 2000X

Figure 4-18. Sodium Silicate Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.
b) SEM Micrograph from One of Larger Pieces of Sample After Burn Testing. 1350X
c) SEM Micrograph from One of the Single Yarn Strands of Sample After Burn Testing. 1350X
d) Kevex Trace of Burned Composite Showing Gold Coating, Silicon, and Sodium.

Figure 4-19. Silica Gel Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.
b) SEM Micrograph from One of the Smaller Fiber Bundles After Burn Testing. 1350X
c) SEM Micrograph from One of the Larger Fiber Bundles After Burn Testing. 1200X
d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon.

Figure 4-20. Ethyl Silicate Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 11 M/sec. Air Velocity. Approximately 50% of Fibers Burned Away. Scale is Centimeters.
b) SEM Micrograph from Cooler Region of the Largest Piece of Sample After Burn Testing. 1350X
c) SEM Micrograph from Small Fiber Strands at Edge of Sample After Burn Testing. 1350X
d) Kevex Trace of Burned Composite Showing Gold Coating and Small Amount of Silicon.

Figure 4-21. Boric Acid Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 11 M/sec. Air Velocity. Scale is Centimeters.
b) SEM Micrograph from Large Piece of Sample Remaining After Burn Testing. 1350X
c) SEM Micrograph from Fuzzy Edge of a Loose Strand After Burn Testing.
d) Kevex Trace of Burned Composite Showing Gold Coating and Background Silicon.

Figure 4-22. Silicon Carbide Coated Carbon Fibers Showing Bundle Integrity.
a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. Scale is Centimeters.
b) Silicon Carbide Coating from UCC L-31 Silicone Fluid. Scale is Centimeters.

Figure 4-23. SEM Micrograph of Silicon Carbide Coated "Thornel" 300 Carbon Fibers.
a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. 2000X
b) Silicon Carbide Coating rrom UCC L-31 Silicone Fluid. 2000X

Figure 4-24. SEM Micrograph of Silicon Carbide Coated Carbon Fibers After 30 Seconds Static Burn at 1000°C.
a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. 1600X
b) Silicon Carbide Coating from UCC L-31 Silicone Fluid. 1350X

Figure 4-25. Silicon Carbide, from UCC L-45 Silicone Fluid, Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 11 M/sec. Air Velocity. Scale is Centimeters.
b) SEM Micrograph from One of the Larger Pieces Remaining After Burn Testing. 1350X
c) SEM Micrograph from Fuzzy Edge of a Smaller Piece Remaining After Burn Testing. 1350X
d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon Levels Similar to Control Samples.

Figure 4-26. Silicon Carbide, from L-31 Silicone Fluid, Coated Fiber Composite After Dynamic Burn Test.
a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne · cm Impact Energy, 11 M/sec. Air Velocity. Approximately 50% of Fibers Burned Away. Scale is Centimeters.
b) SEM Micrograph from One of the Larger Pieces Remaining After Burn Testing. 1350X
c) SEM Micrograph from Edge of a Sample Piece Remaining After Burn Testing. 1350X
d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon Levels Similar to Control Samples.

Figure 4-27. "Thornel" 300 Carbon Fiber with UC-309 Size Showing Bundle Integrity. Scale is Centimeters.

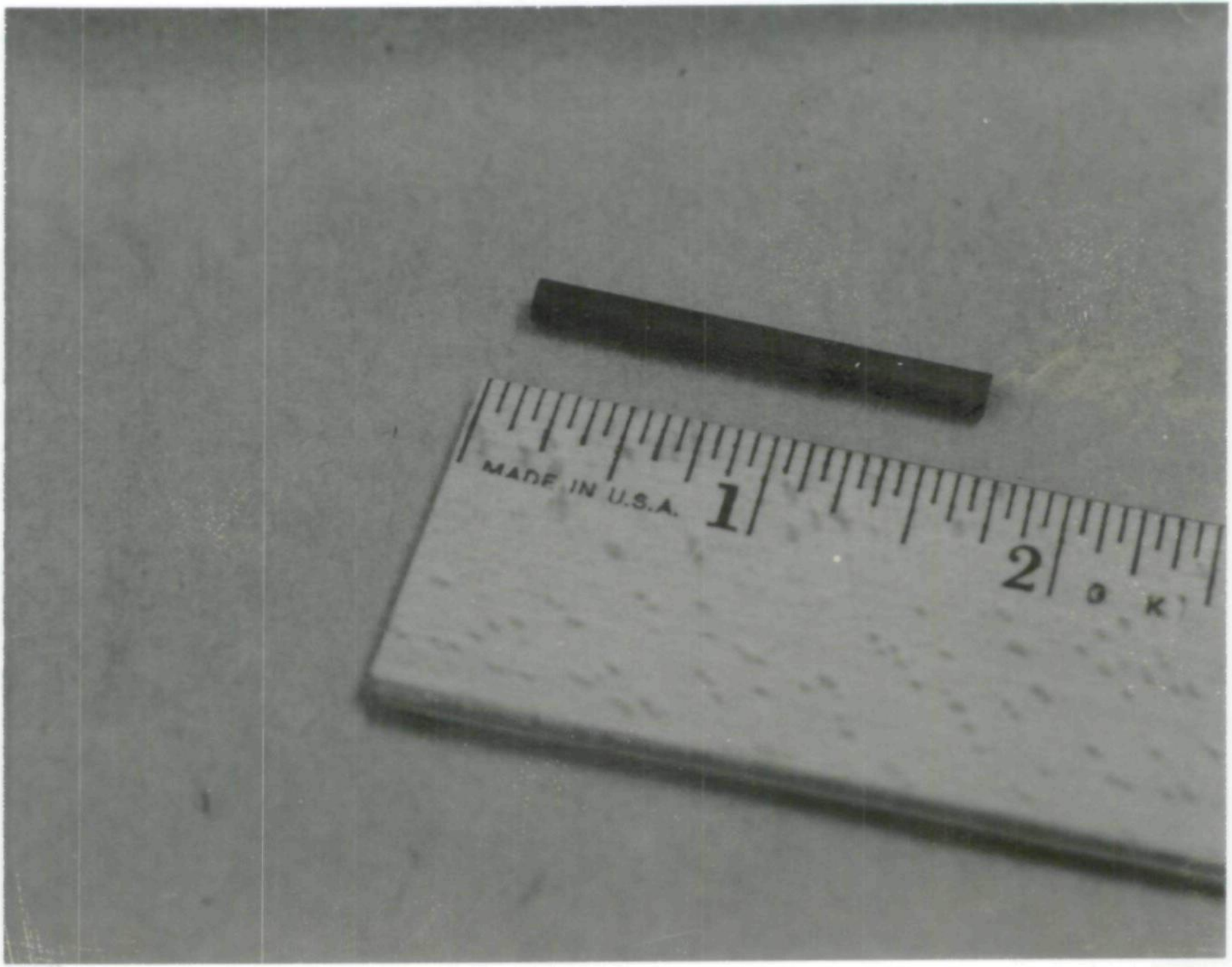
MADE IN U.S.A.
1
2

Fig. 3.1. Torsion Rod Specimen. Scale in Inches.

Fig. 3-1

POLAROID

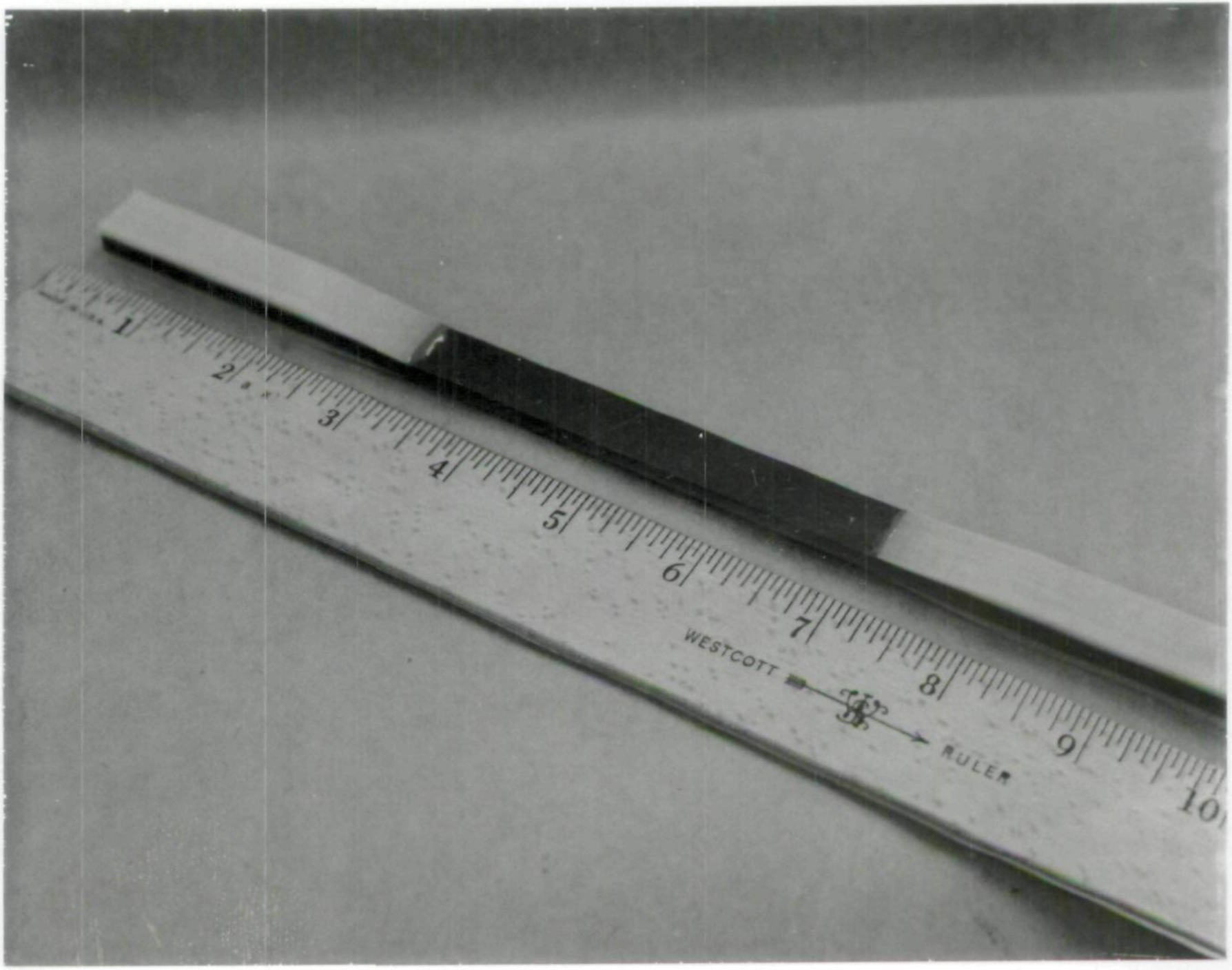
WESTCOTT
RULER

Fig. 3.2(a) Tensile Specimen with Fiberglass Tabs.

NASA Contractor Report 159057

Fig. 3-2(a)

POLAROID L8443208/012A

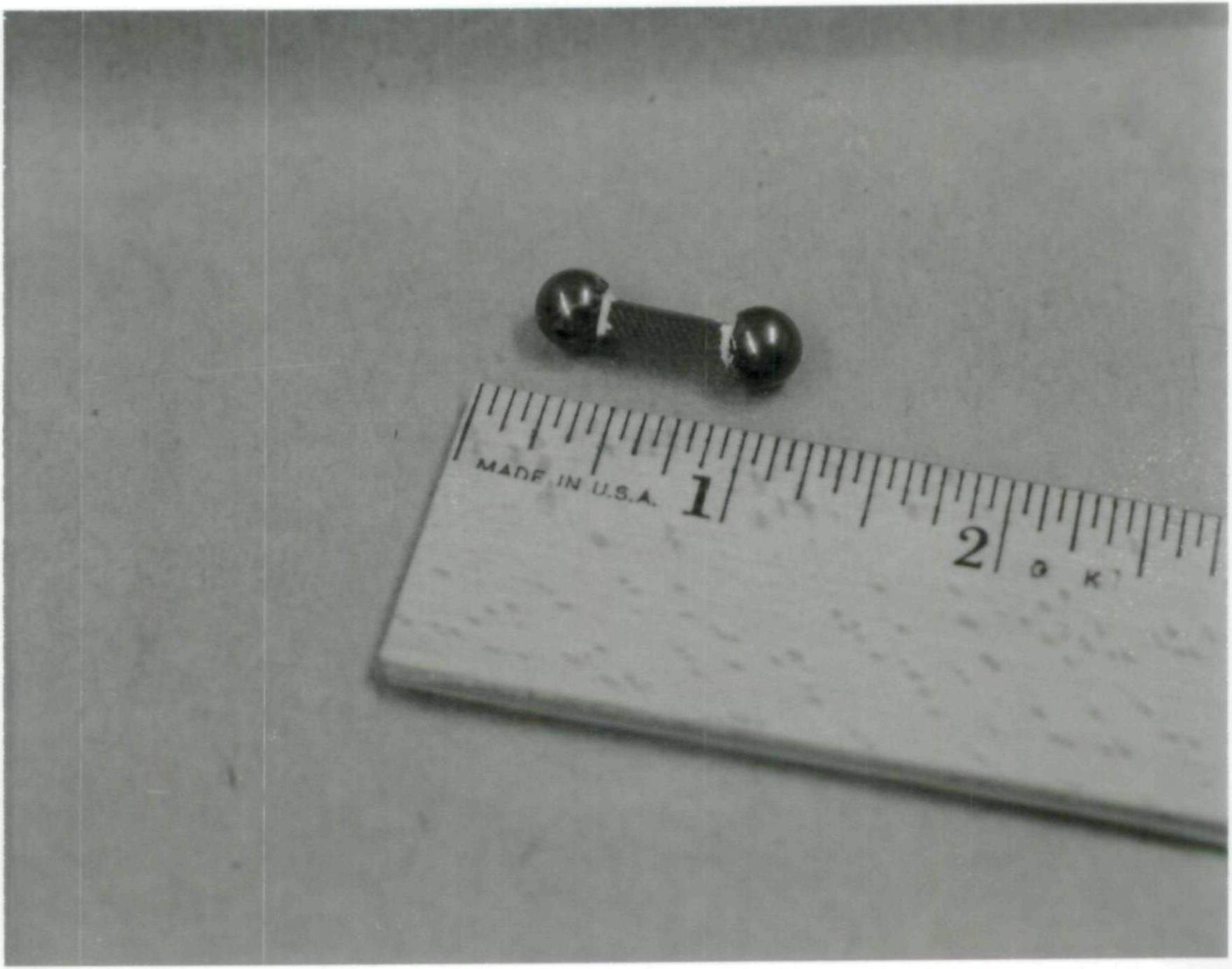
MADE IN U.S.A.
1
2

Fig. 3.2(b) Texaco Compression Sample.

NASA Contractor Report 159057

Fig. 3-2(a)

POLAROID

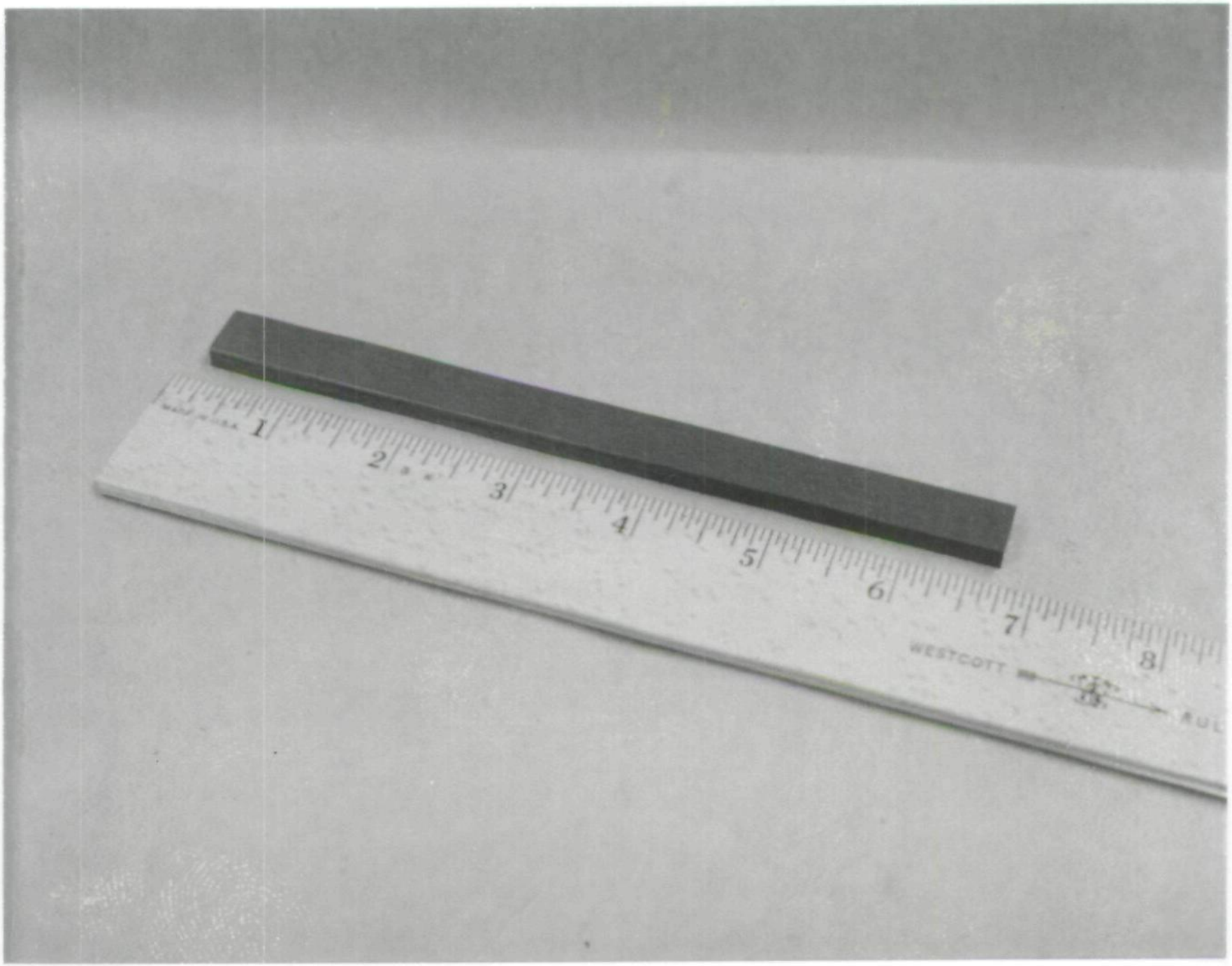
1
2
3
4
5
6
7
8
WESTCOTT

Fig. 3.2(c) Flexural Specimen

NASA Contractor Report 159057

Fig. 3-2 ~~(b)~~ (c)

POLAROID

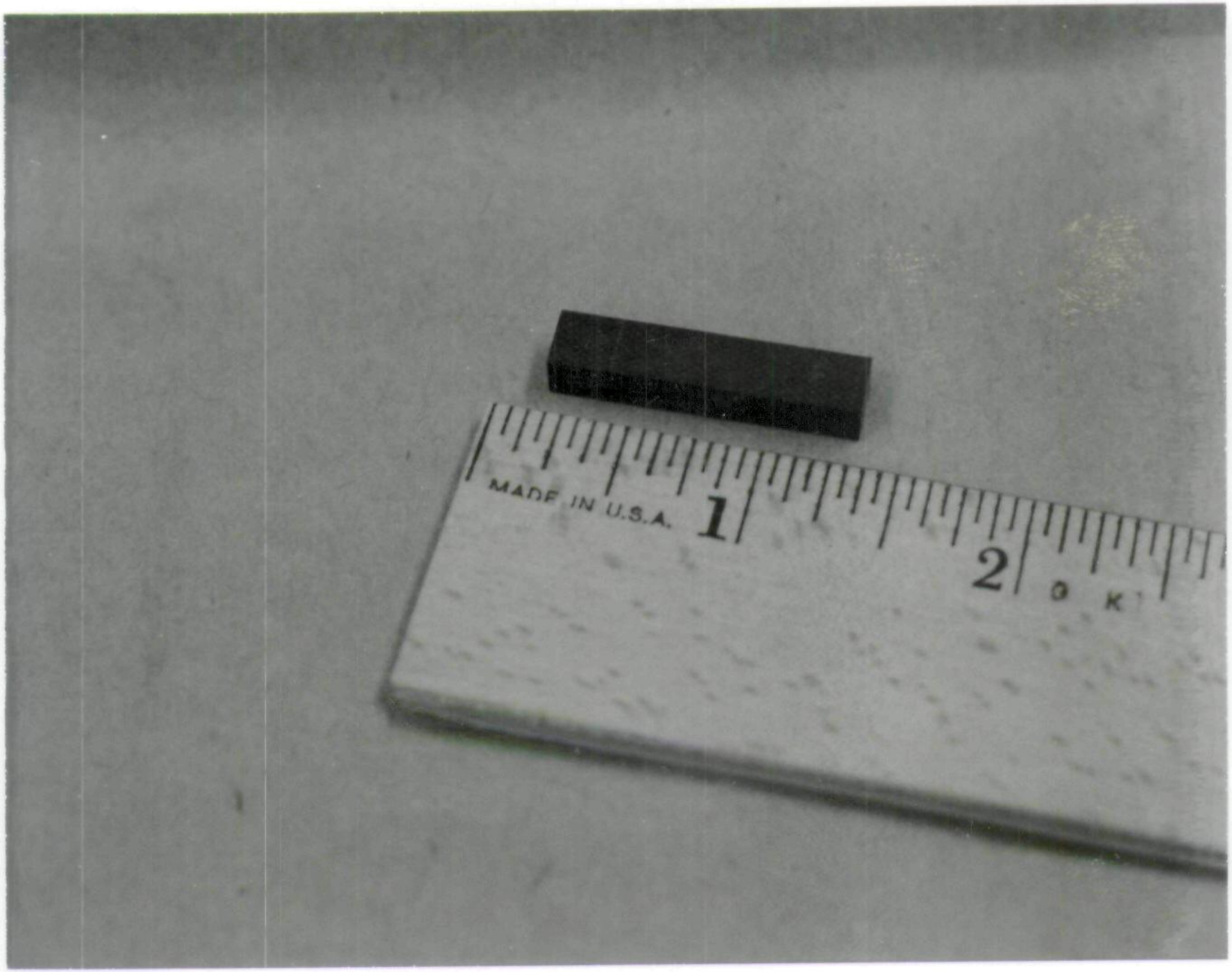
MADE IN U.S.A.
1
2

Fig. 3.2(d) Short Beam Shear Sample

NASA Contractor Report 159057

Fig. 3.2. Physical Property Sample Geometrics. Scale in Inches.

Fig. 3-2(d)

POLAROID

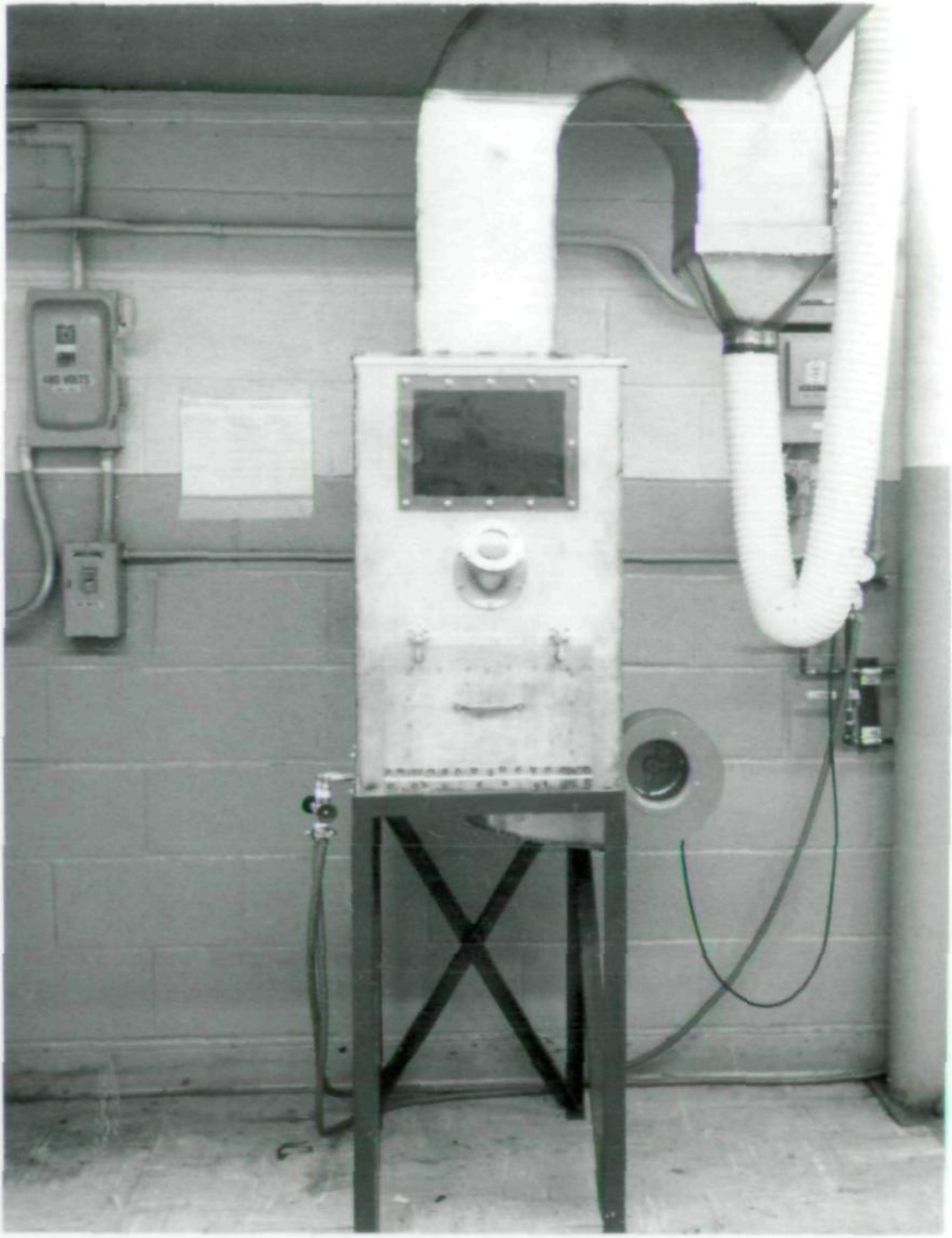
480 VOLTS

Fig. 3.4(a) Chamber showing Collection System.

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Fig. 3.4(a)

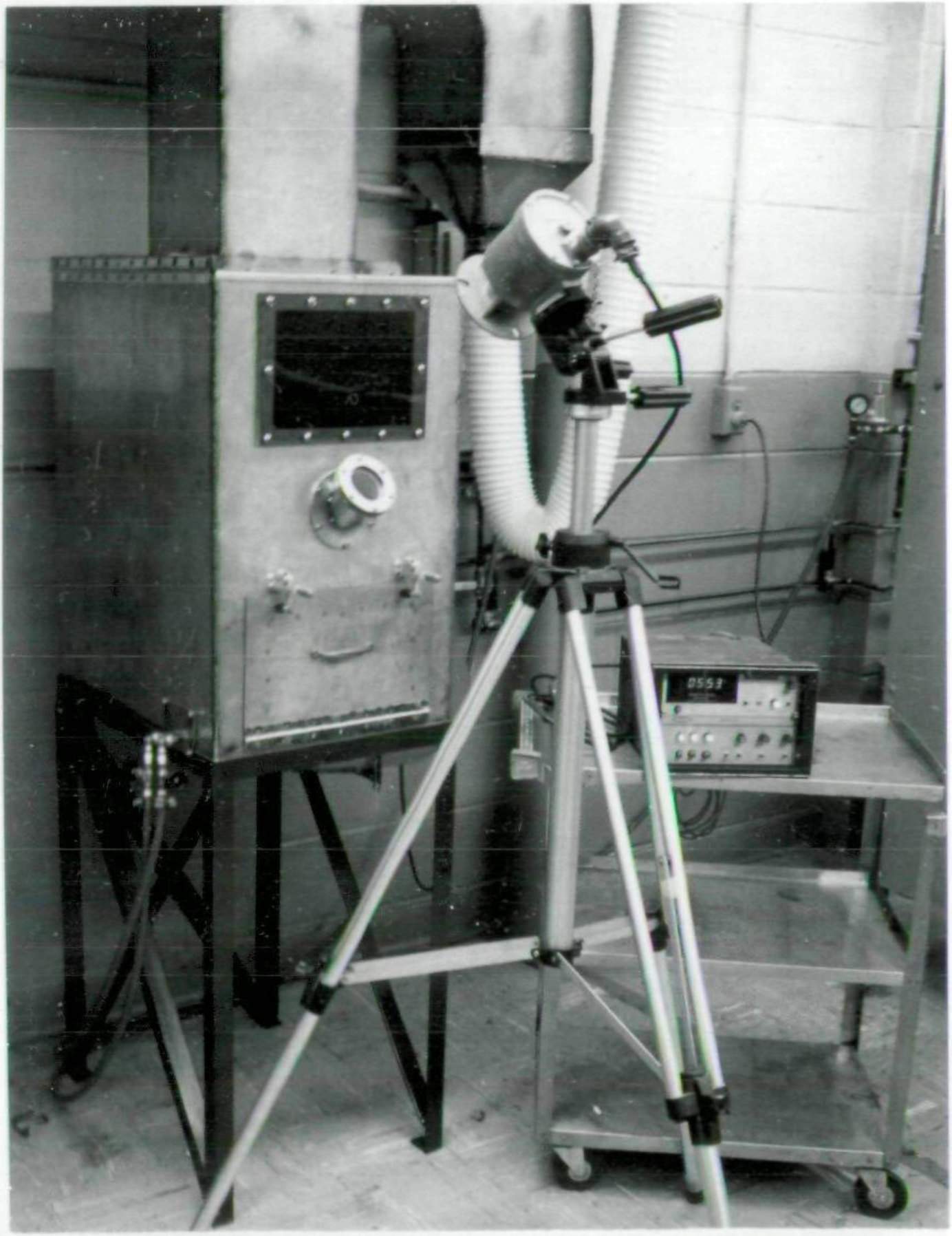

Fig. 3.4(b) Temperature Measurement

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L 80205 1

Fig. 3-4(b)

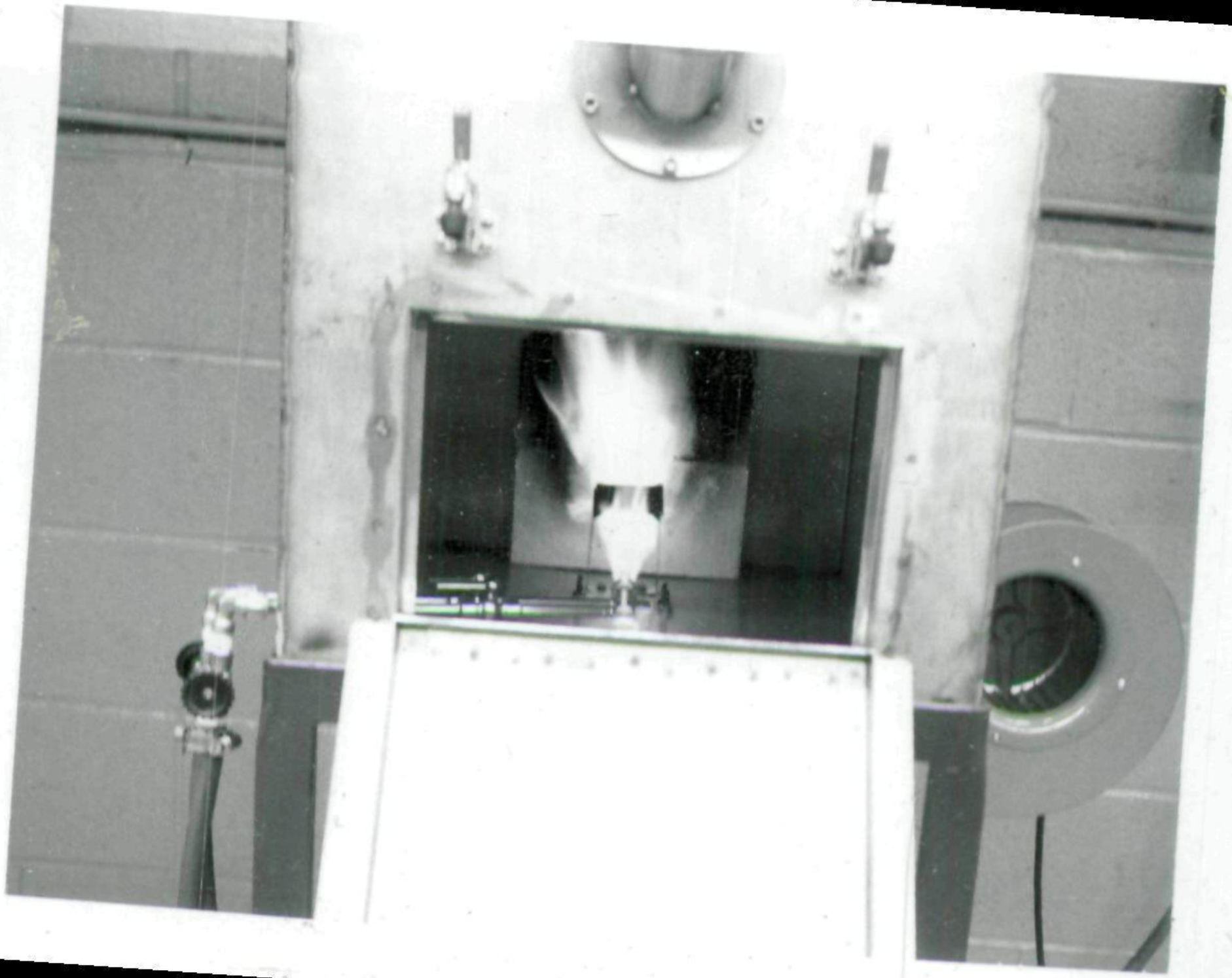

Fig. 3.4(c) Sample Under Test

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Fig. 3.4. Dynamic Burn Test Chamber.

L 6 0 2 0 5 1

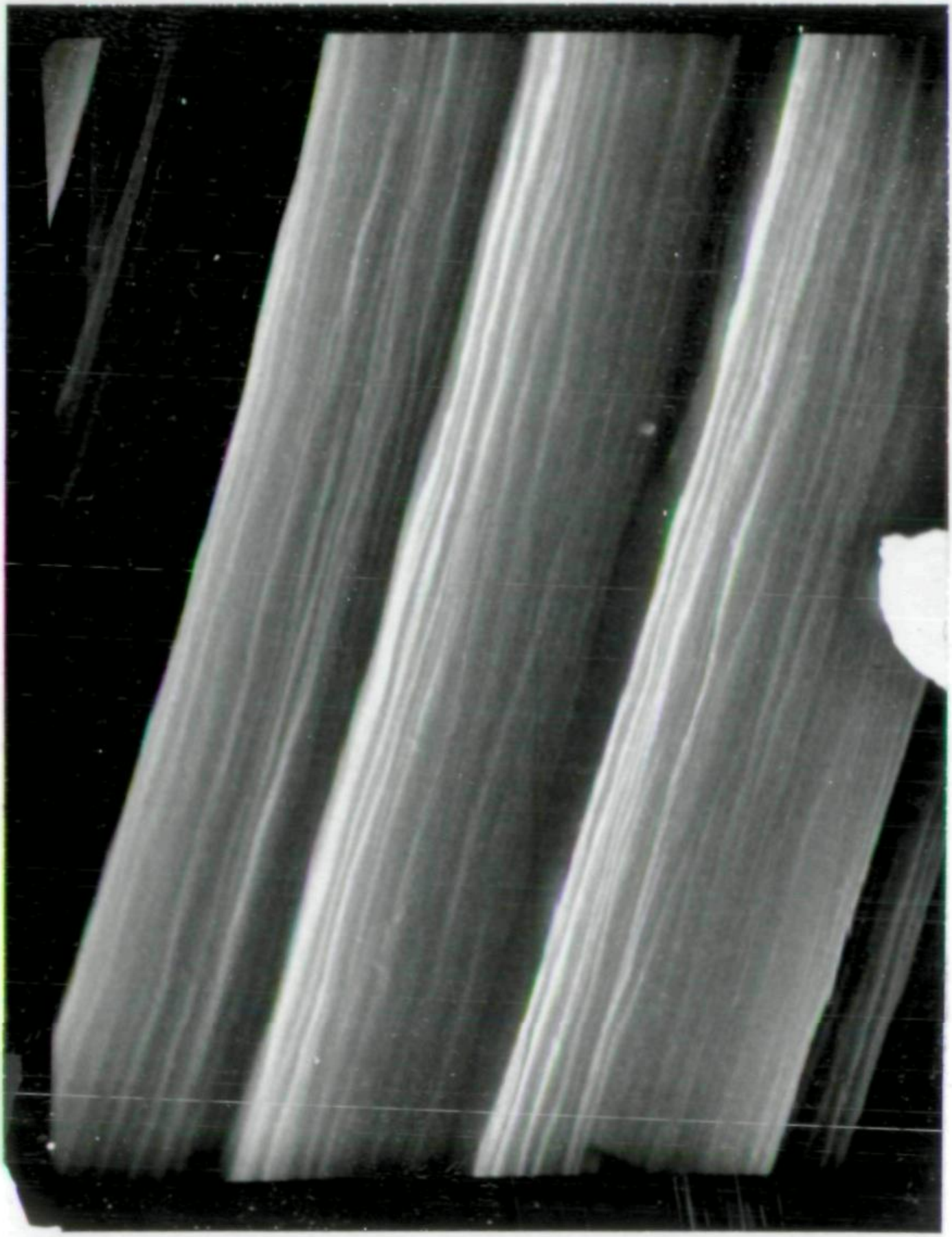

Fig. 4.1(a) UCC L-45 Silicone Fluid, 8 wt.%, Applied from Kerosene Solution. 2000X

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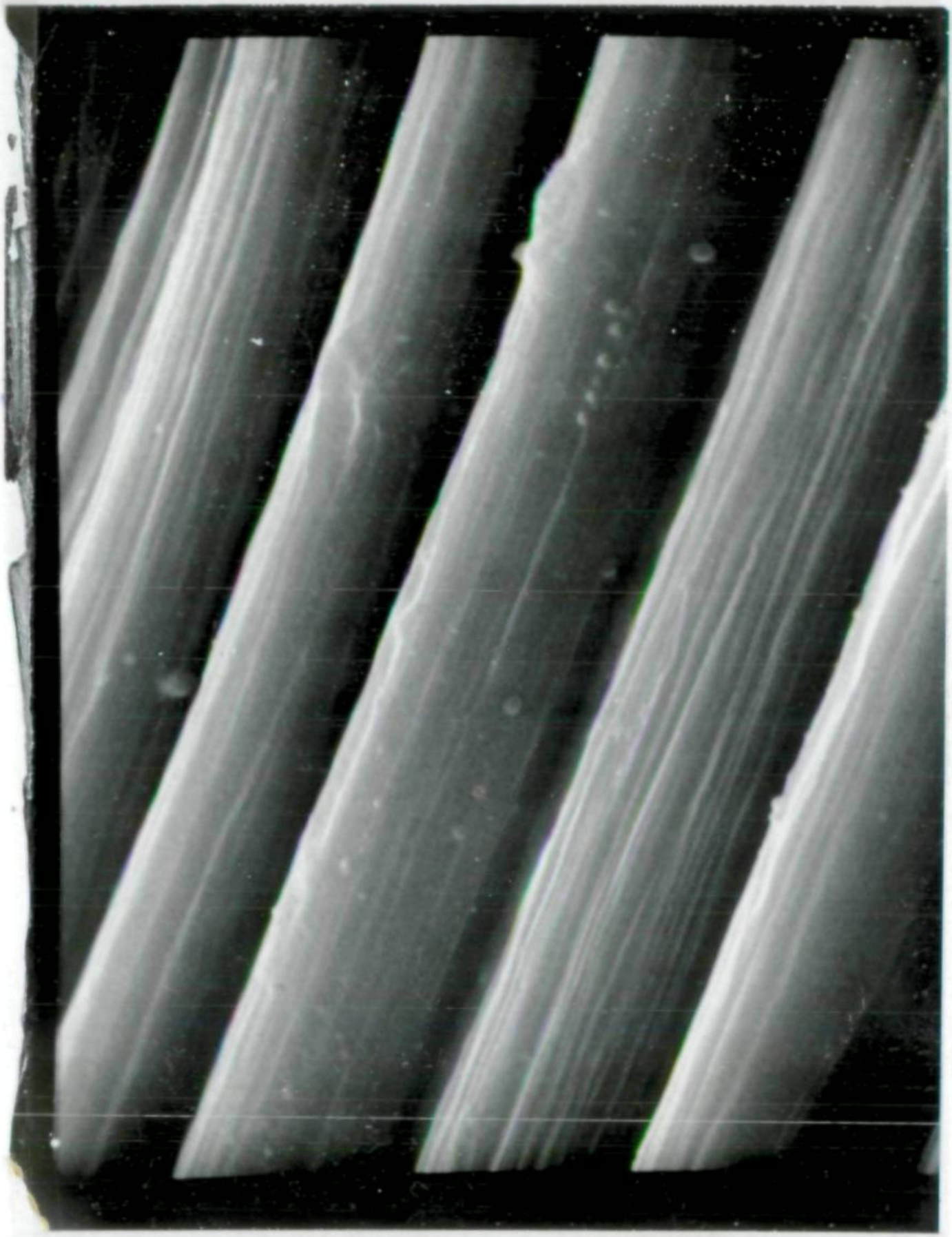

Fig. 4.1(b) UCC L-31 Silicone Fluid, 5 wt. %, Applied from Isopropanol Solution. 2000 x

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Fig. 4-1. SEM Micrographs of Carbon Fibers Coated with Silicone Fluids

(9) 1-4

METRIC 1

Fig. 4-2(a) UCC L-45 Silicone Fluid, 8.4 wt.% Applied from Kerosene Solution.

POLAROID

METRIC 1

Fig. 4-2(b) UCC L-31 Silicone Fluid, 4.6 wt.%, Applied from Isopropanol Solution.

NASA Contractor Report 159057

Fig. 4-2. Silicone Coatings, Showing Bundle Integrity, Scale is Centimeters.

POLAROID L84622G070240

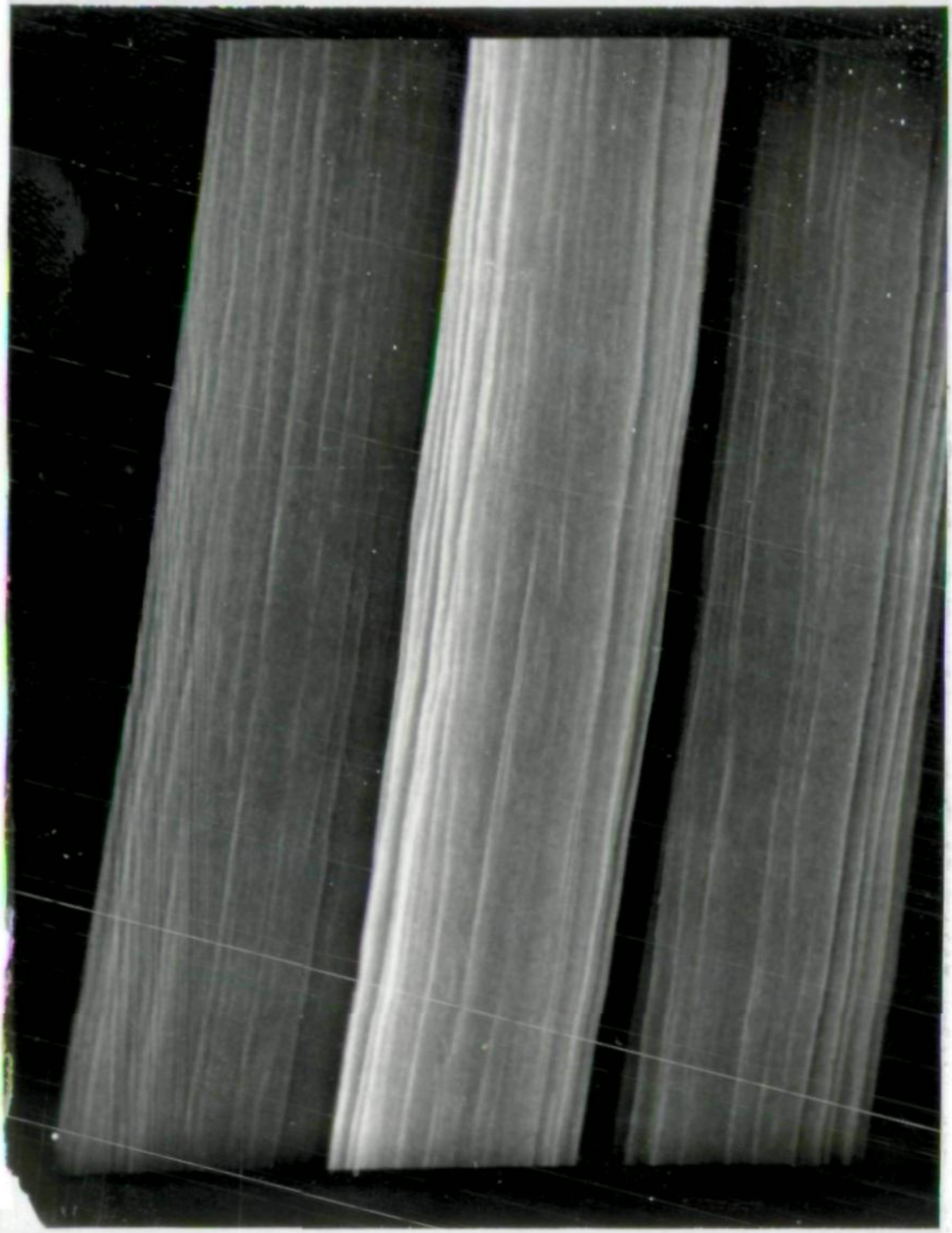

Fig. 4.3. SEM Micrograph of Uncoated Water-Sized "Thornel" 300. 2000 X

NASA Contractor Report 159057

631-1-36

Fig. 4-3

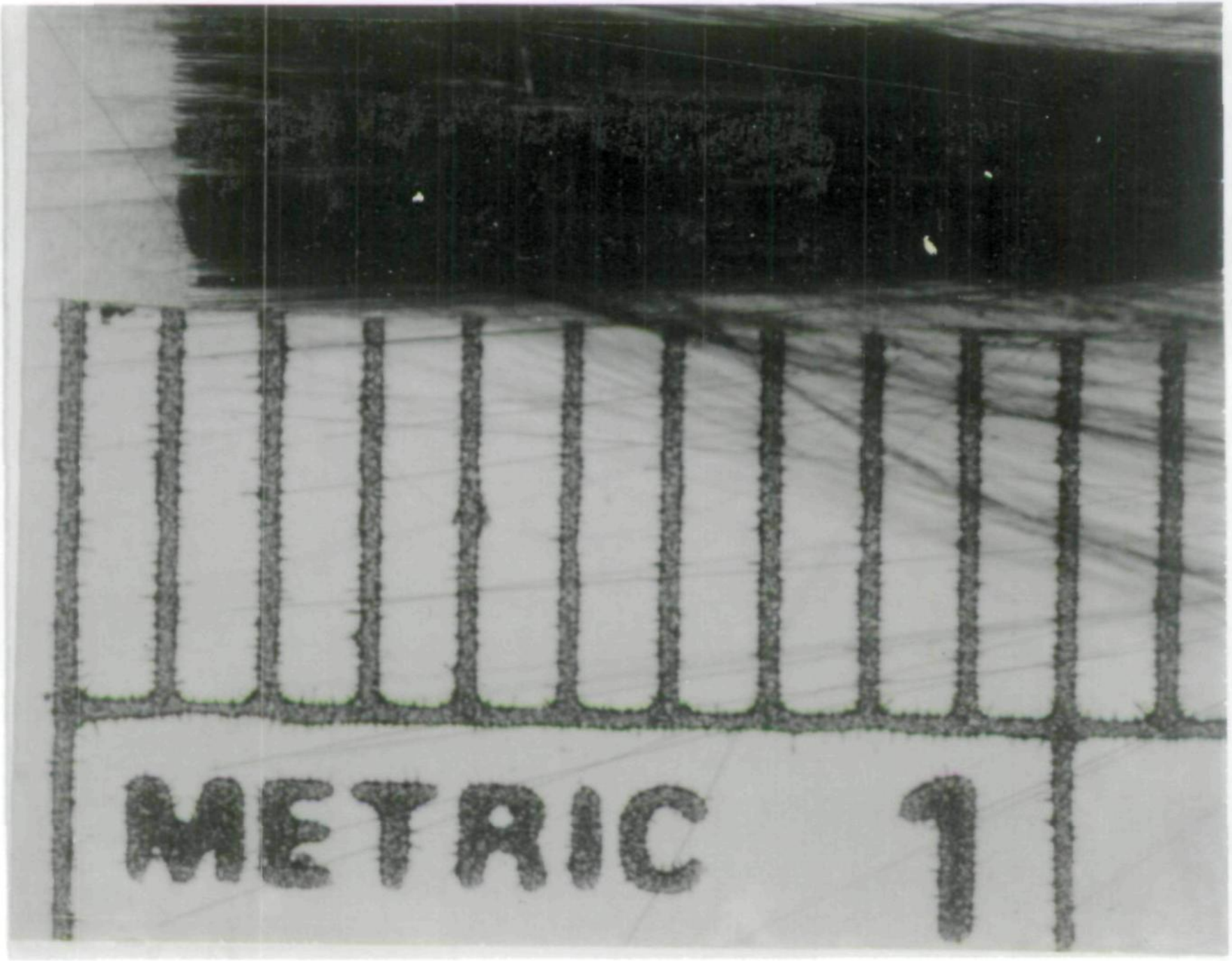
METRIC 1

Fig. 4-4. Uncoated Water-Sized "Thornel" 300 Showing Bundle Integrity, Scale is Centimeters.

NASA Contractor Report 159057

61-8-159

Fig. 4-4

POLAROID

L84622G870240

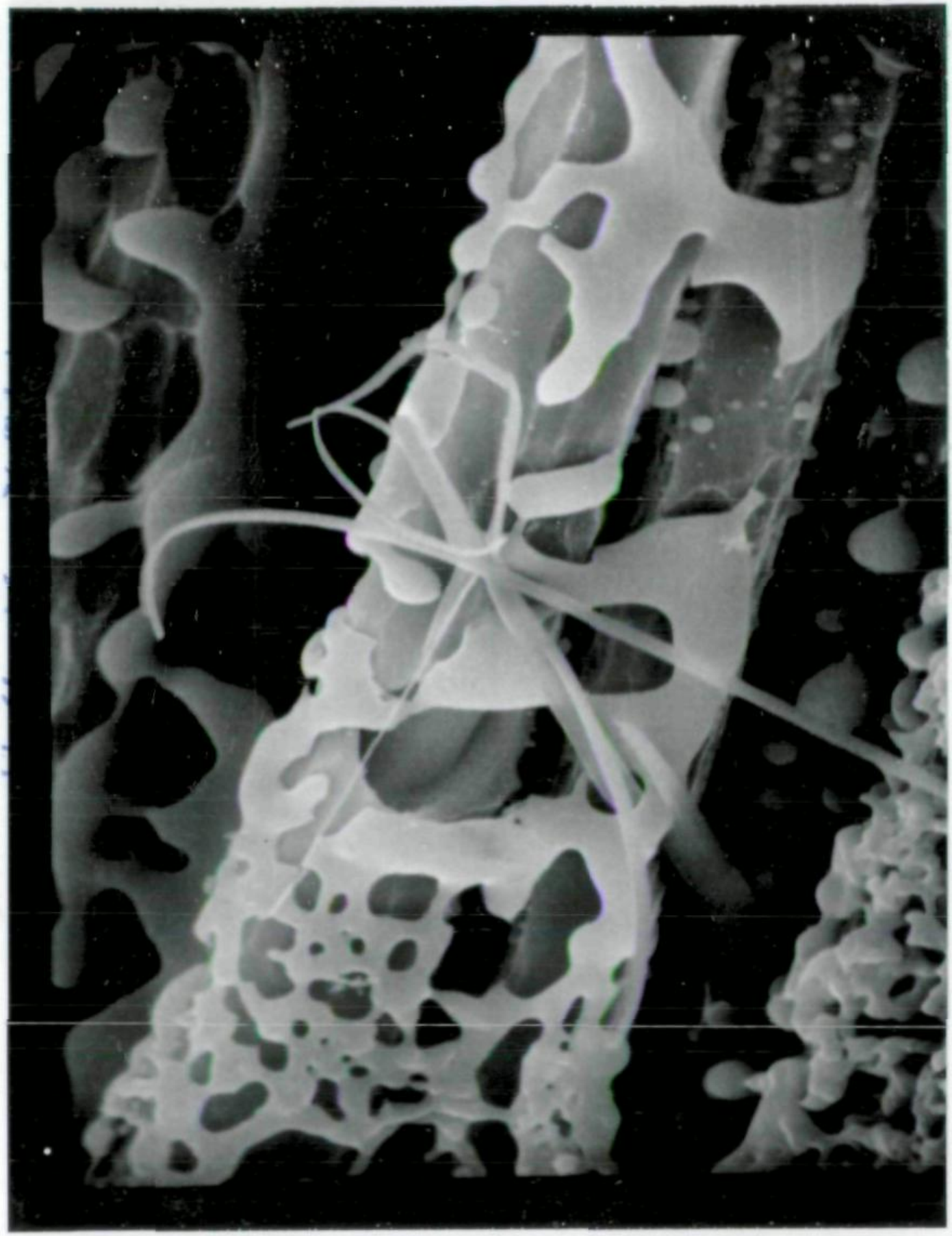

Fig. 4-5(a) UCC L-45 Silicone Fluid Coating, 14 wt.%.2000X

139

Fig. 4-5(a)

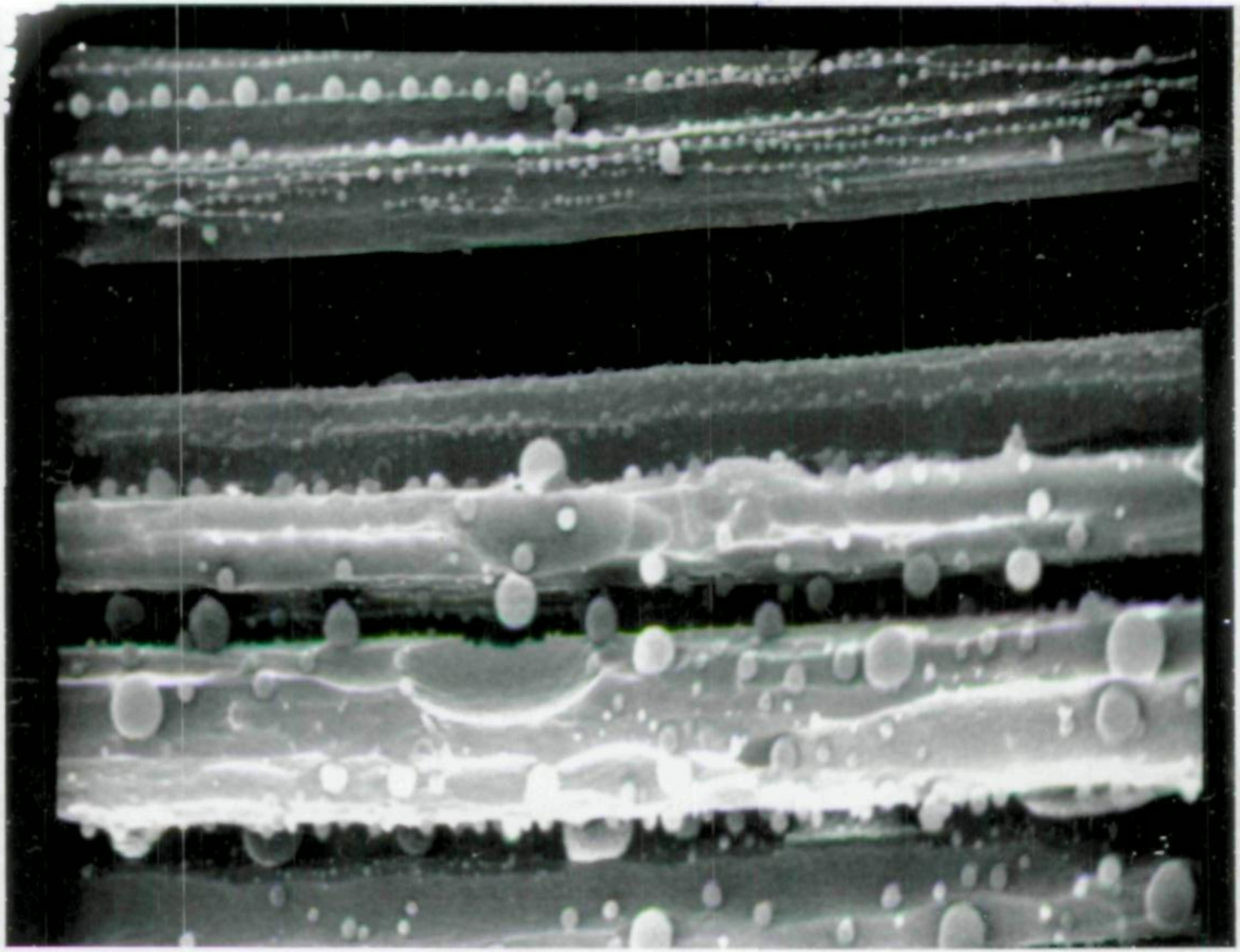

Fig. 4-5(b). UCC L-31 Silicone Fluid Coating, 13 wt.%. 2000X

Fig. 4-5. Silicone Fluid Coated Carbon Fibers After 1000°C Static Burn for 30 Seconds.

NASA Contractor Report 159057

(9)-5-h Fig.

Fig. 4-6. SEM Micrograph, Water-Sized "Thornel" 300 After 1000°C Static Burn for 30 Seconds. 2000X

NASA Contractor Report 159057

63

Fig. 4-6

Fig. 4-7. SEM Micrograph of Boron Nitride, from Ammonium Borate, Coating on Carbon Fibers. 5000X

Contractor Report 159057

POLAROID L84432-87912A

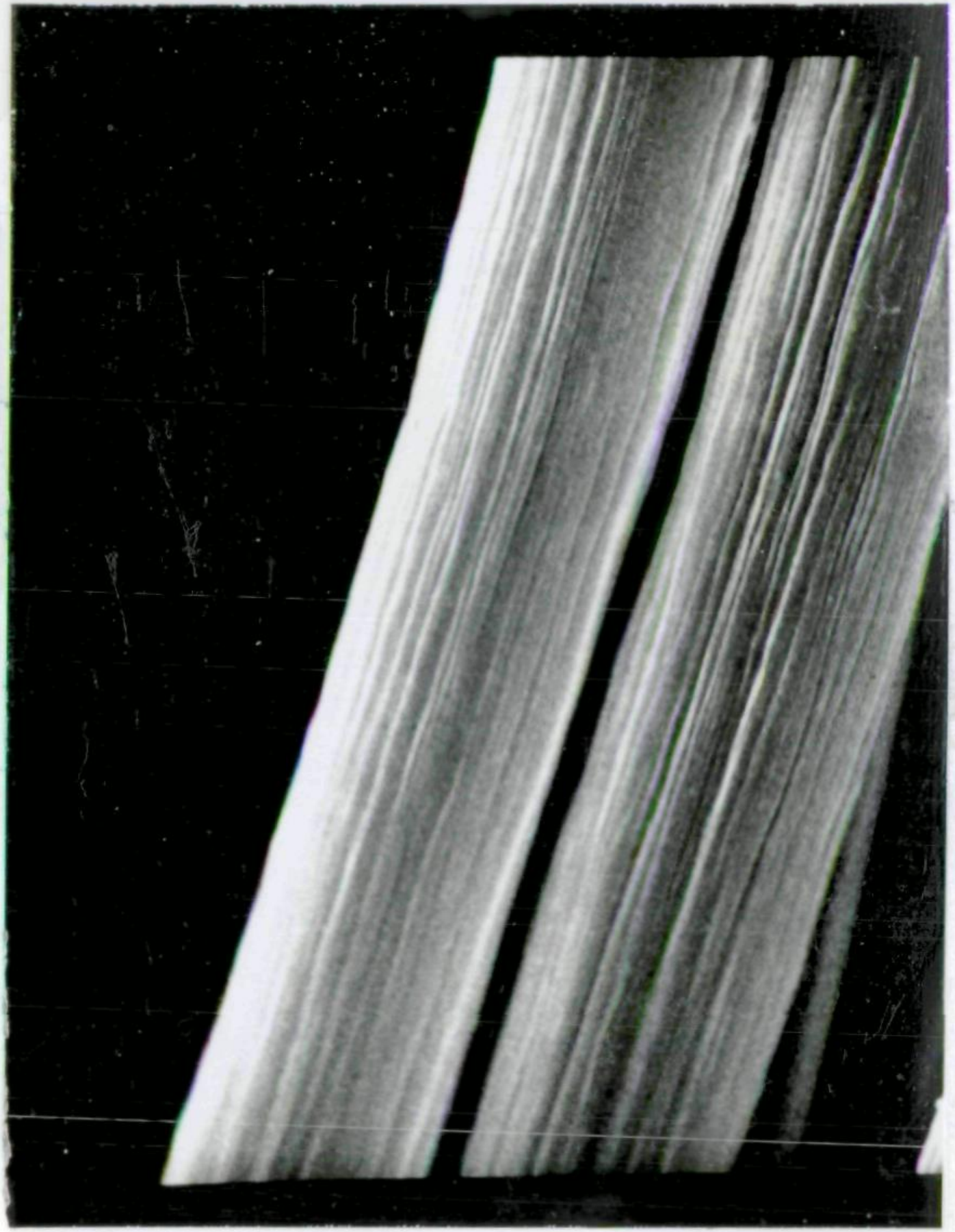

Fig. 4-8 (a) Boric Acid Coating, 5 wt.%.
2000X

NASA Contractor Report 159057

J 71 6 2 2 2 A

63

Fig. 4-8(a)

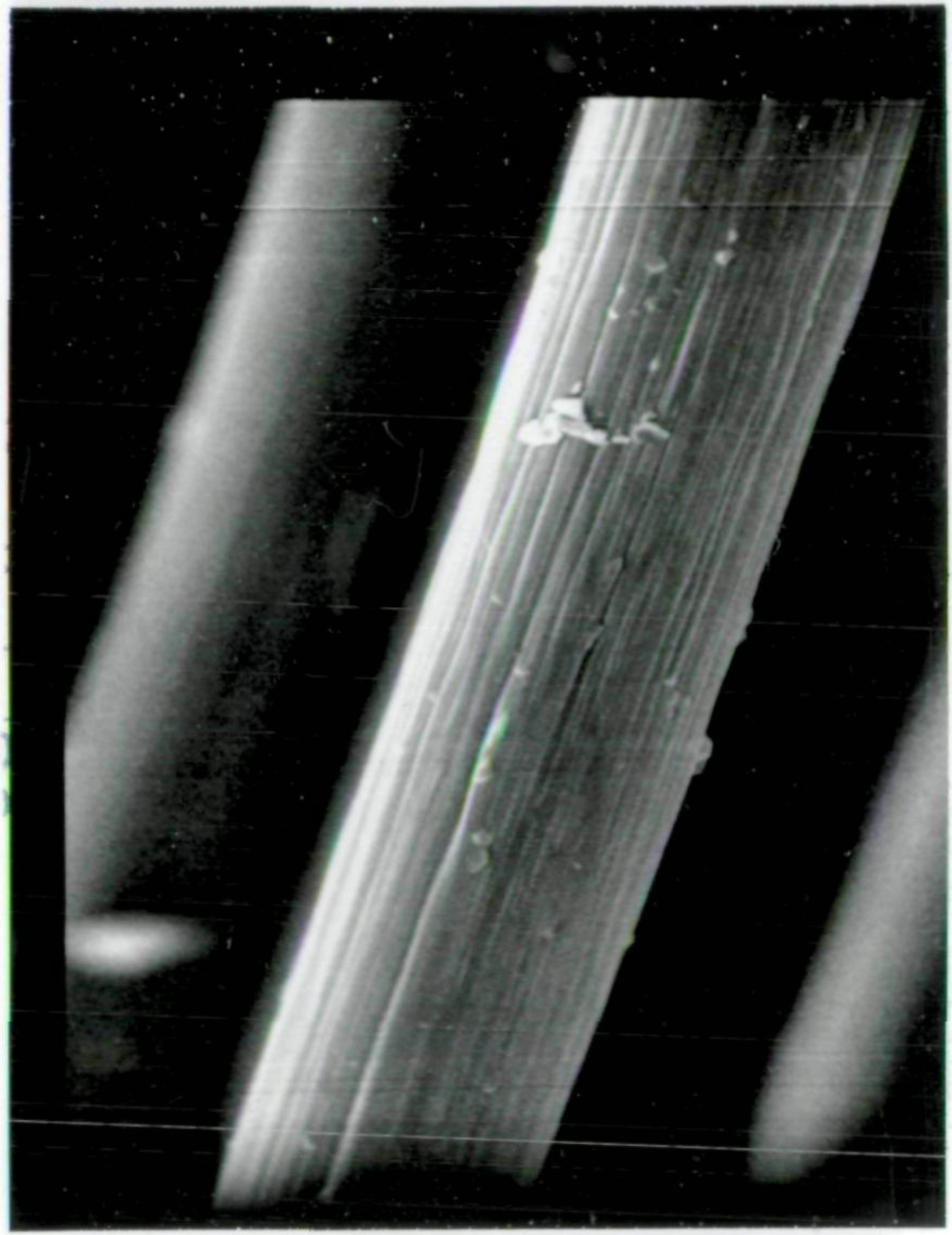

Fig. 4-8(b) Ammonium Borate Coating, 10 wt.%. 2000X

Fig. 4-8. SEM Micrographs of Coated Carbon Fibers

NASA Contractor Report 159057

631

Fig. 4-8(b)

Fig. 4-9. SEM Micrograph of Boron Nitride, from Ammonium Borate, Coated Carbon Fibers After 30 Seconds Static Burn Test at 1000°C. 400X

POLAROID

L84622c070240

Fig. 4-9

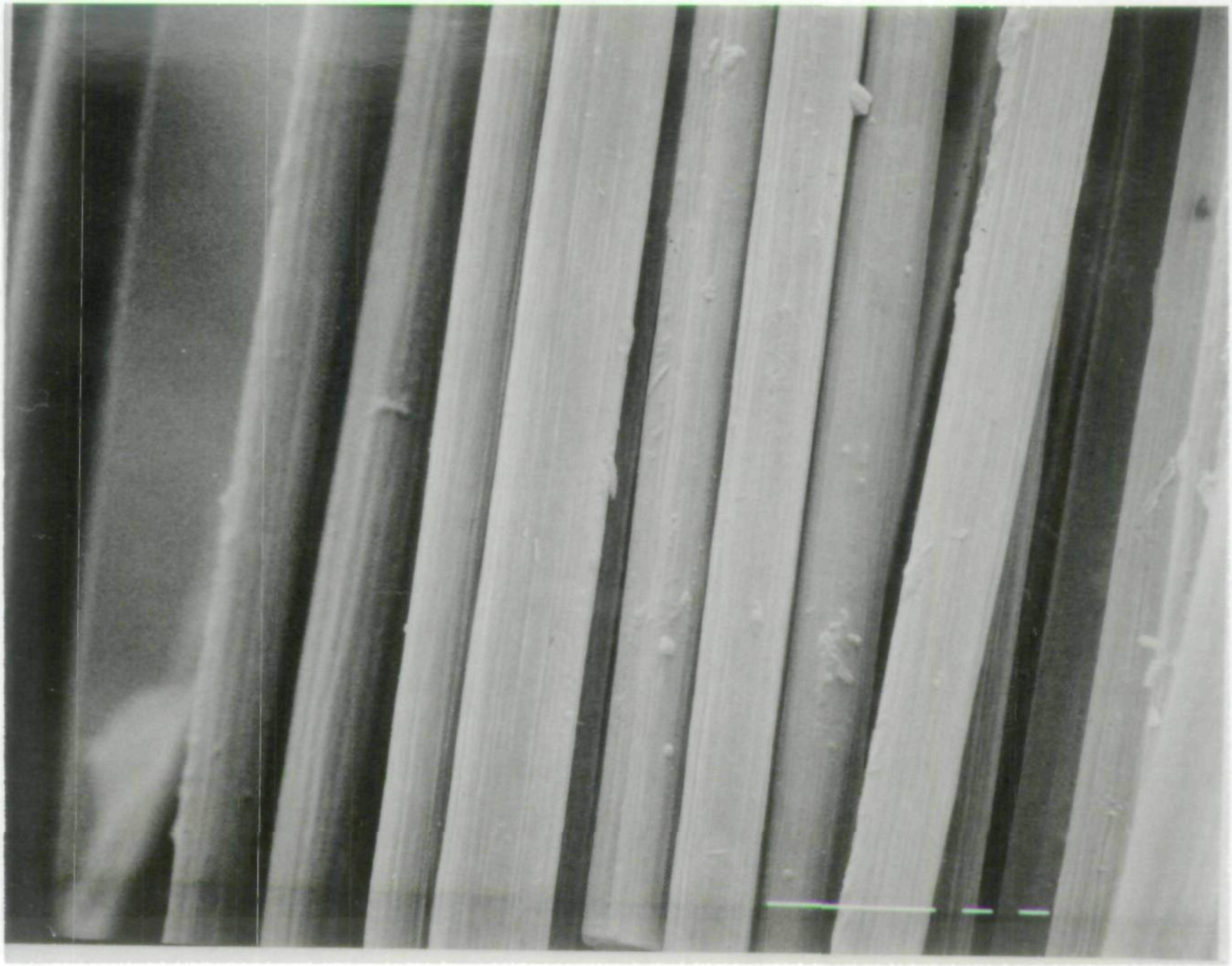

Fig. 4-10. SEM Micrograph of Boron Nitride Coated Fiber Composite After 1000°C Static Burn Test for 30 Seconds. 1350X

POLAROID

L84322C37923C

Fig. 4-11. SEM Micrograph of Water-Sized "Thornel" 300 Fiber Composite After 1000°C Static Burn Test for 30 Seconds. 1200 X

Contractor Report 159057

POLAROID

METRIC 1 2 3 4 5 6 7 8 9

Fig. 4-12(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.

Fig. 4-12(b) SEM Micrograph from Cooler Corner of Composite During Burn Test. Epoxy Char is Holding Fibers Together. 180X

Fig. 4-12(c) SEM Micrograph from Hottest Area of Composite Where Fiber Burning was Taking Place. 180X

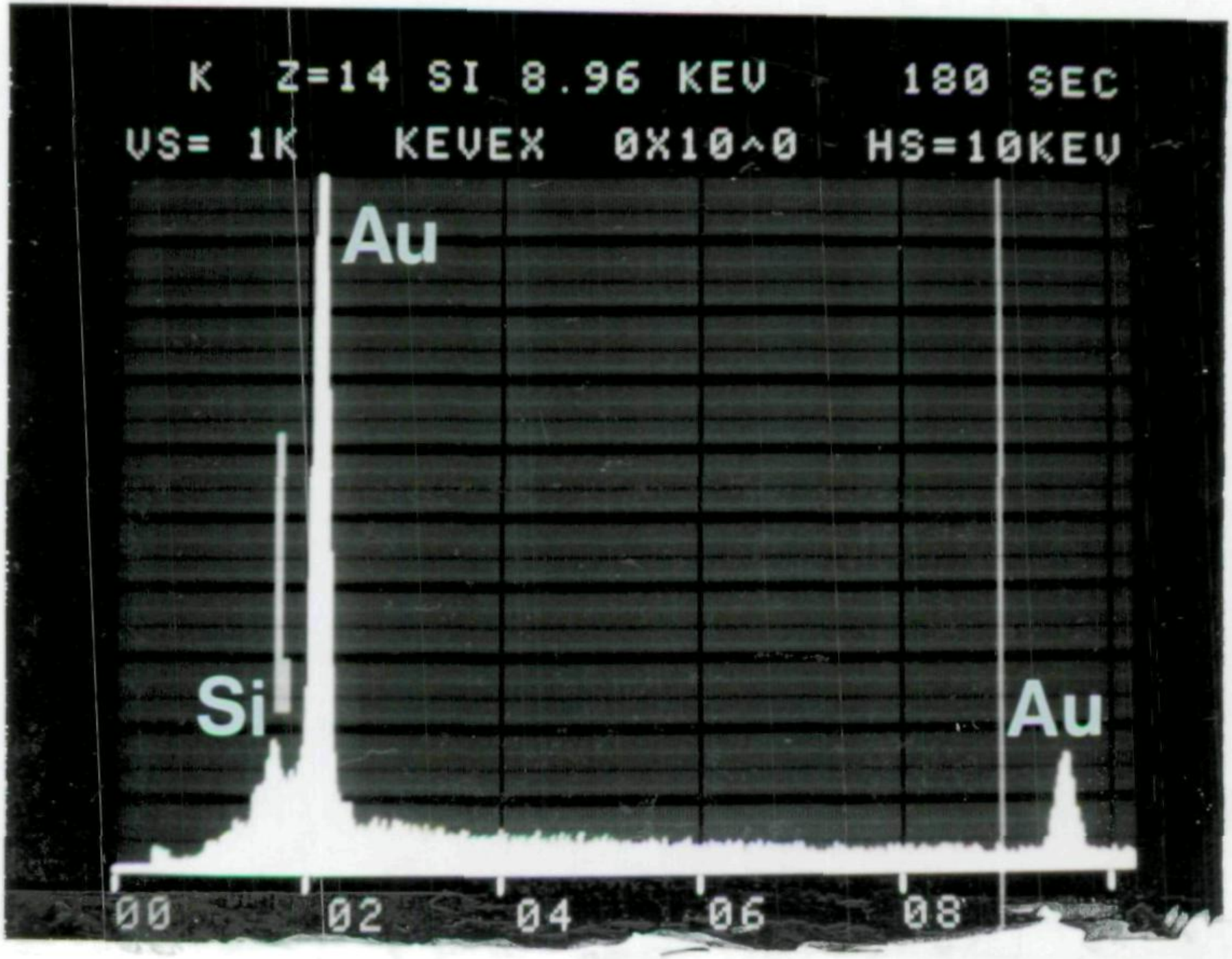
K Z=14 SI 8.96 KEV 180 SEC
VS= 1K KEVEX 0X10^0 HS=10KEV
Au
Si
Au
00
02
04
06
08

Fig. 4-12 (d) Kevex Trace of Burned Composite Showing Gold Coating and Small Silicon Background.

Fig. 4-12. Boron Nitride, from Ammonium Borate, Coated Fiber Composite After Dynamic Burn Test.

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Fig. 4-12 (d)

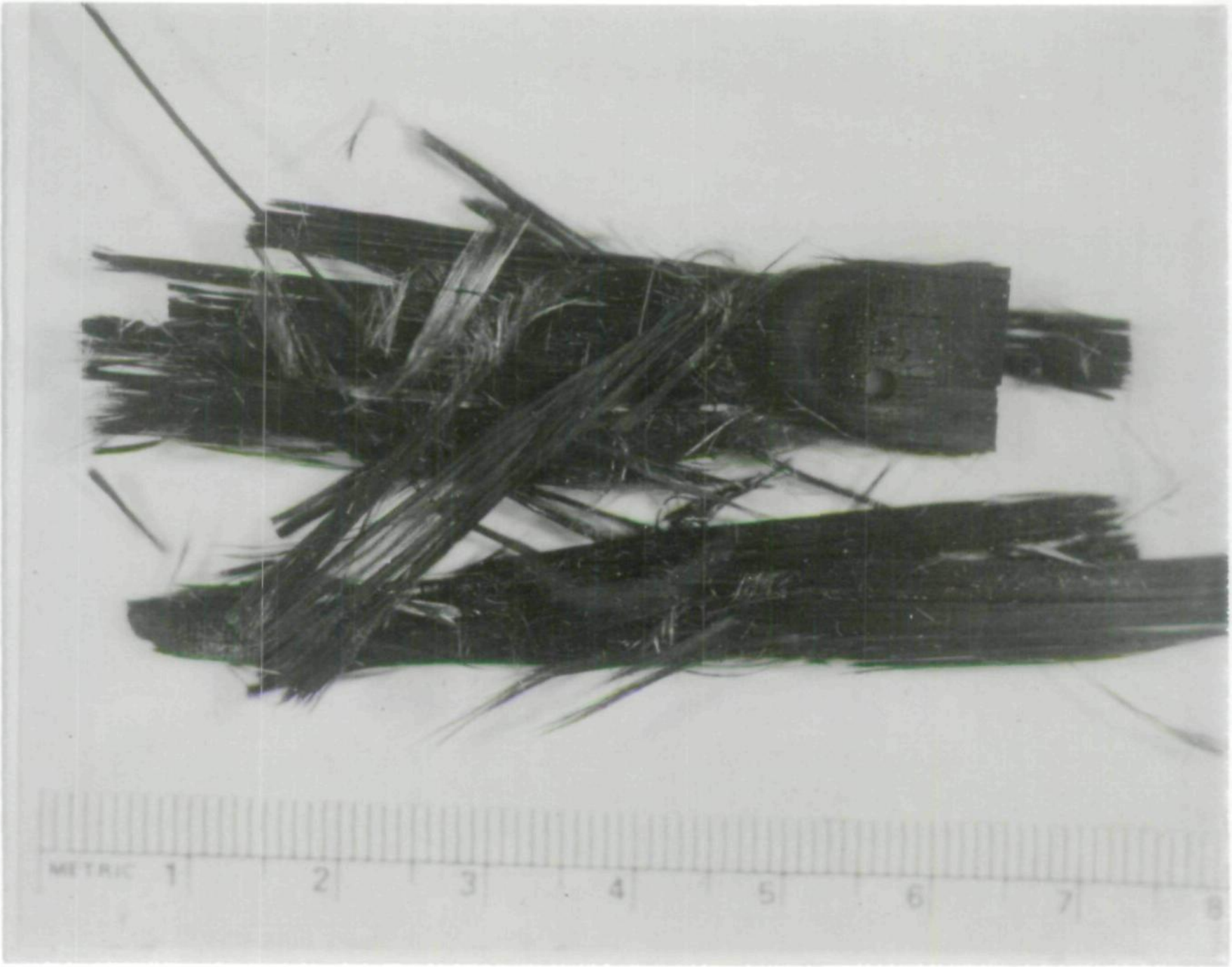
METRIC 1 2 3 4 5 6 7 8

Fig. 4-13(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 11 M/sec. Air Velocity. Approximately 60% of Fibers Burned Away.

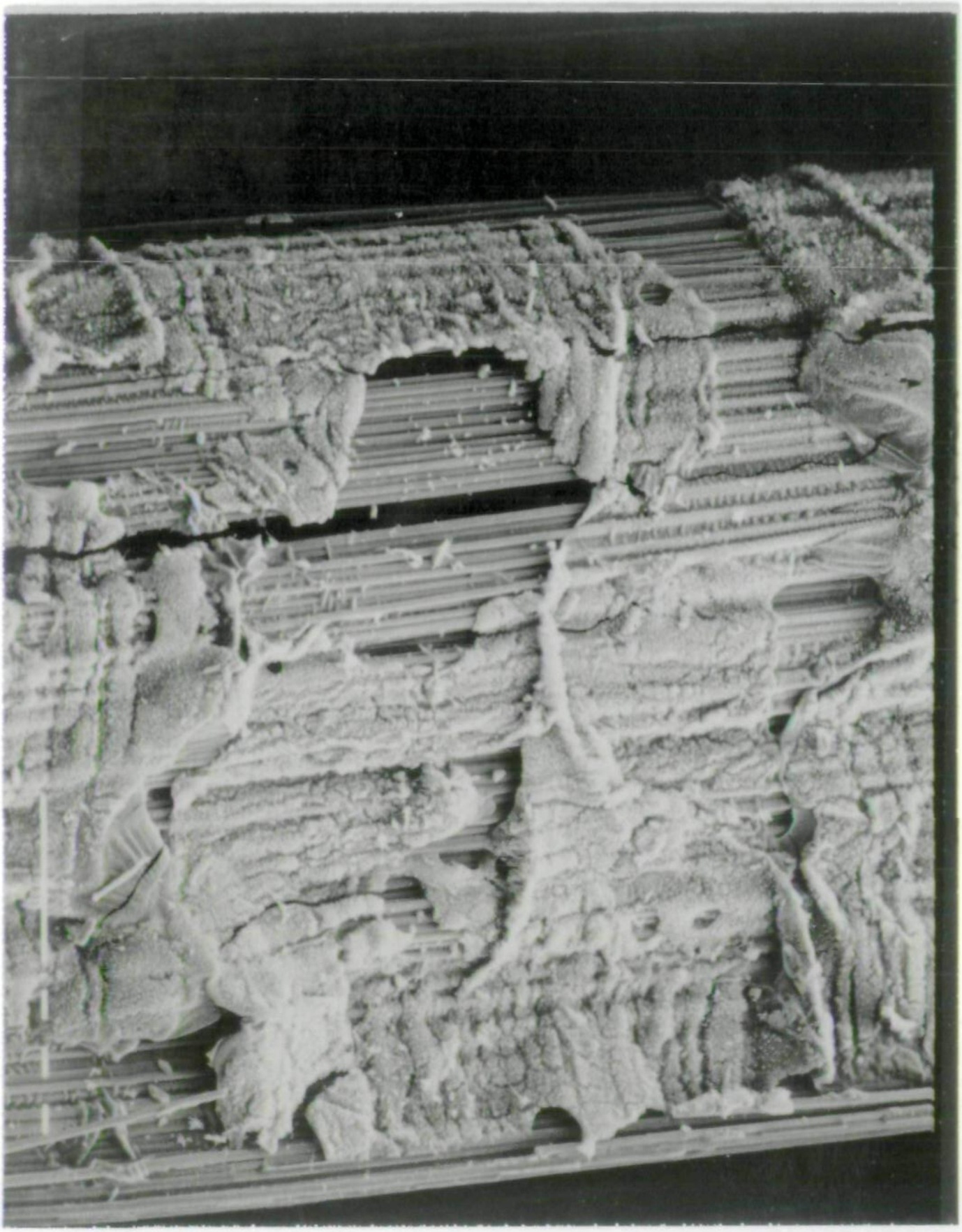

Fig. 4-13(b) SEM Micrograph from Cooler Area of Composite During Burn Test. Epoxy Char Holds Fibers Together. 120X

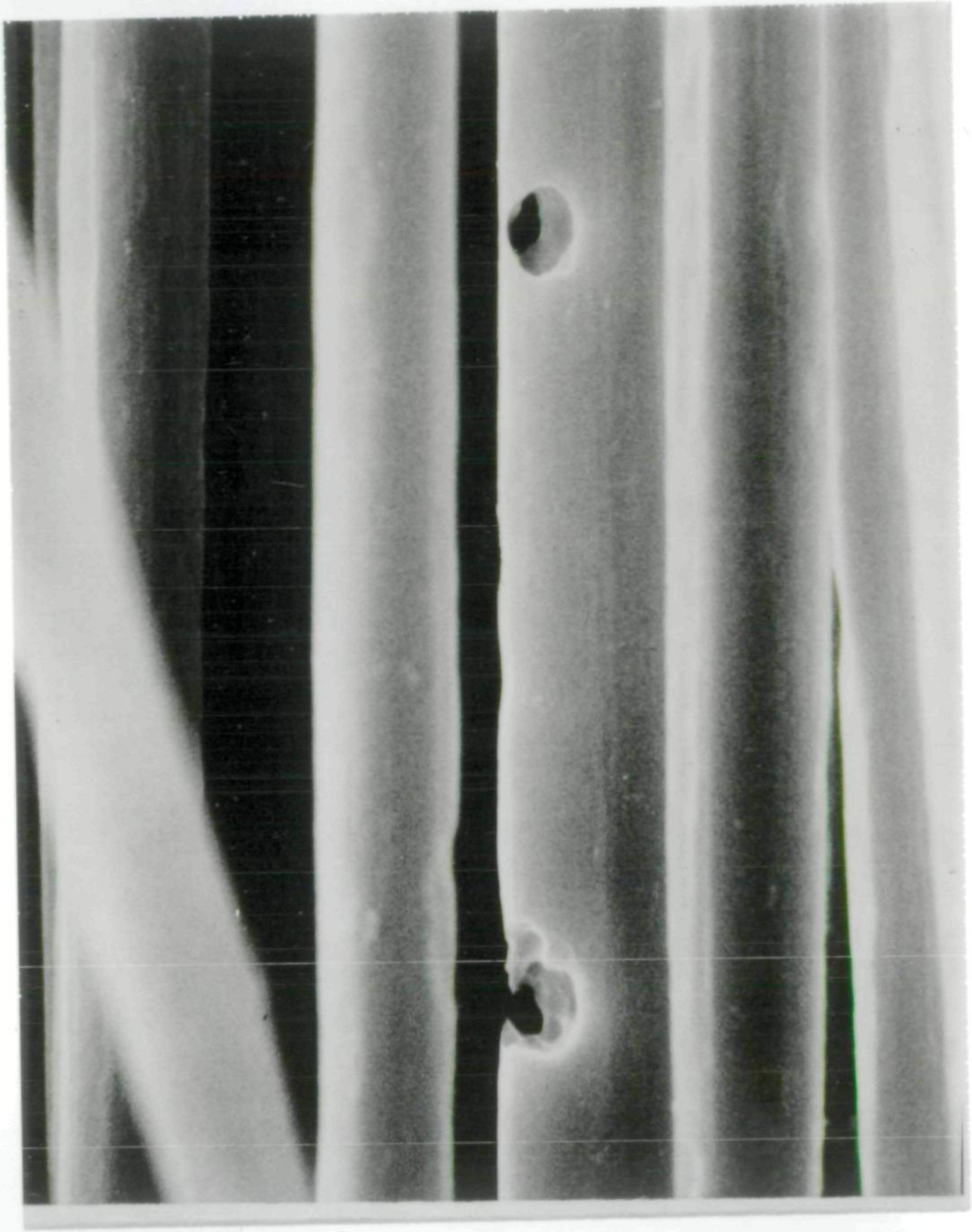

Fig. 4-13(c) SEM Micrograph of Fibers from Hotter Area of Sample Where Fiber Burning was Taking Place. 2700X

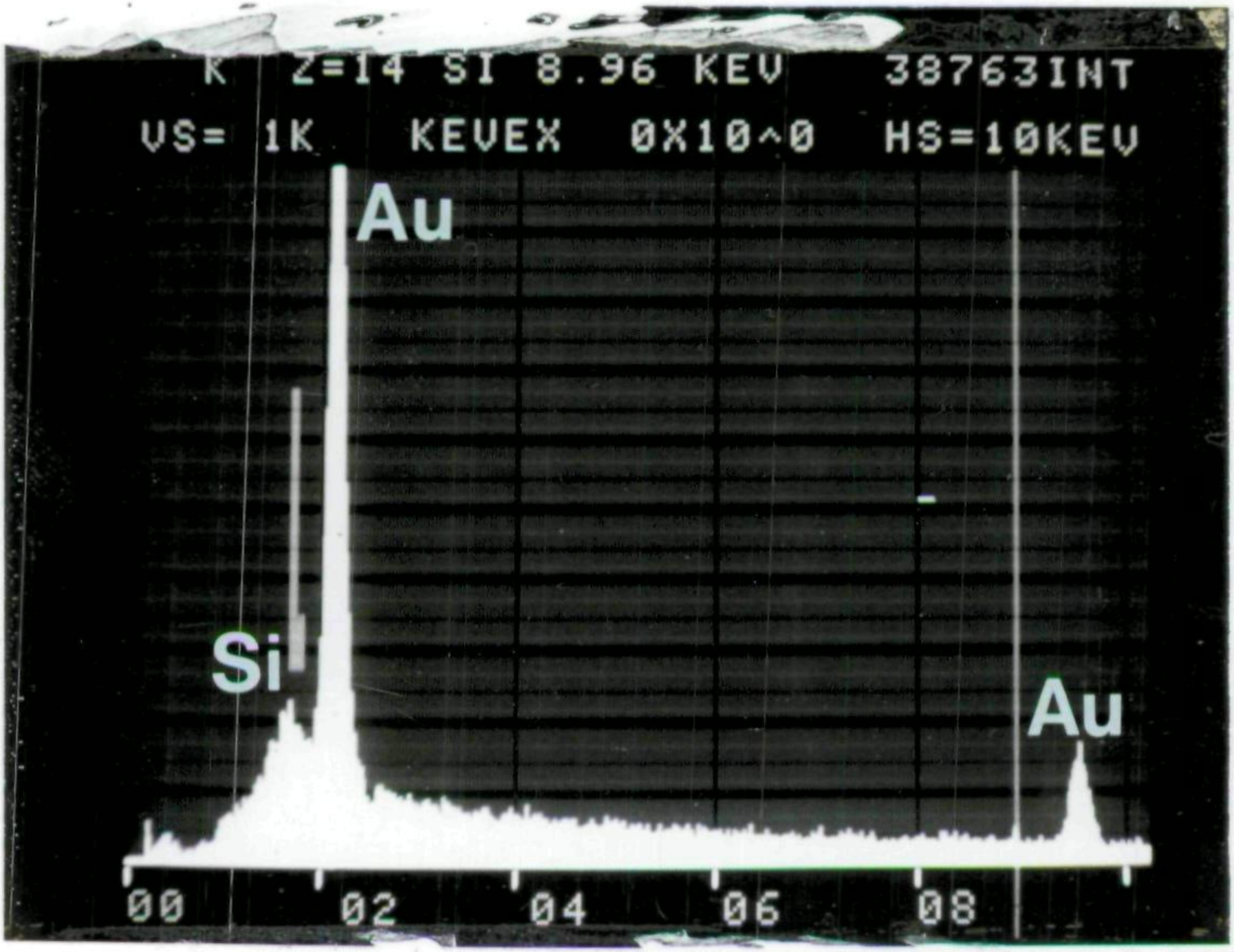
K Z=14 SI 8.96 KEV 38763INT
VS= 1K KEVEX 0X10^0 HS=10KEV
Au
Si
Au
00
02
04
06
08

Fig. 4-13(d) Kevex Trace of Burned Composite Showing Gold Coating and Small Silicon Background.

Fig. 4-13. Water-Sized "Thornel" 300 Fiber Composite After Dynamic Burn Test.

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METRIC 1

Fig. 4-14(a) Sodium Silicate Coated Fiber, 5 wt.%.

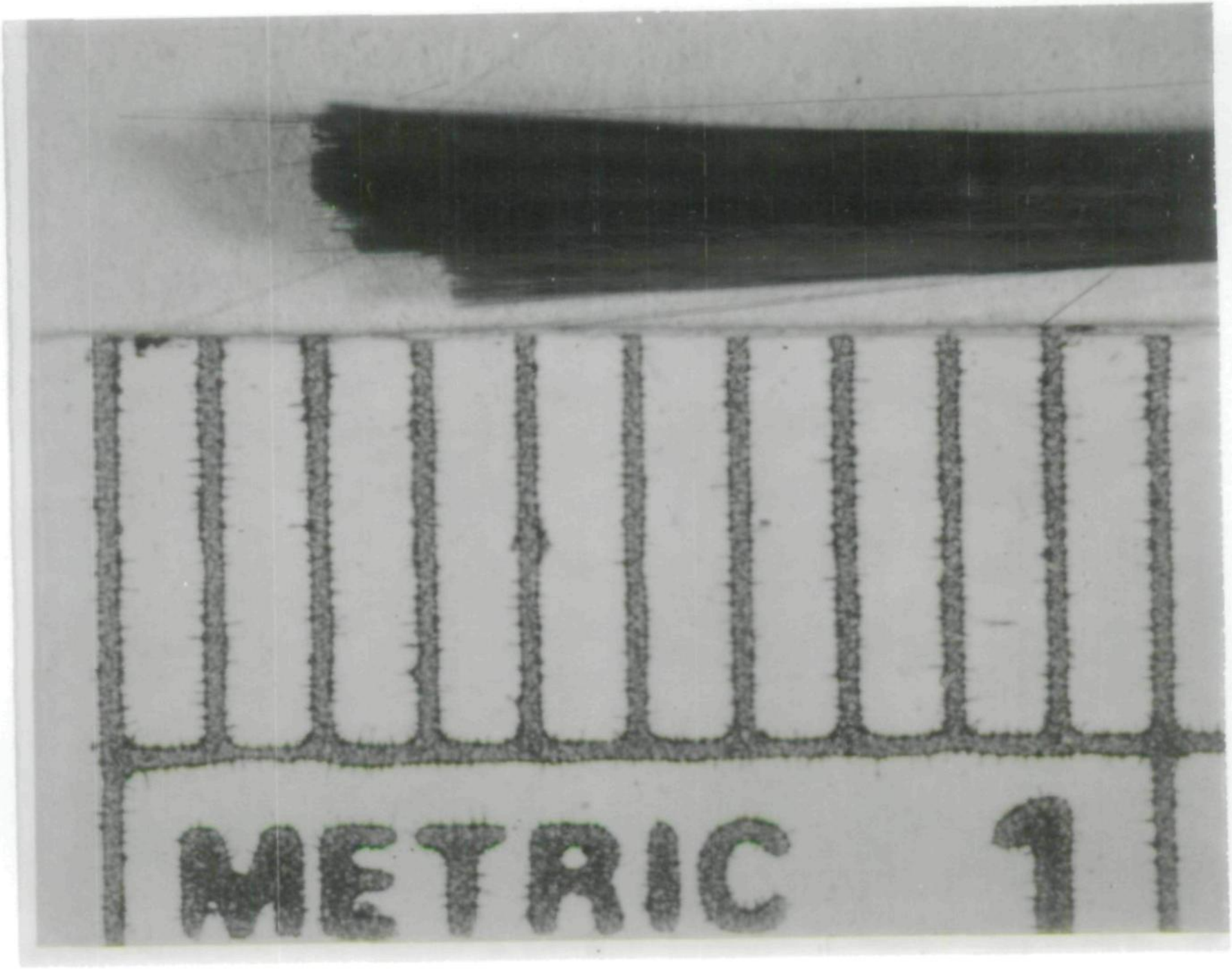
METRIC 1

Fig. 4-14(b) Silica Gel Coated Fibers, 6 wt.%.

NASA Contractor Report 159057

Fig. 4-14 b

L84622c079240

POLAROID

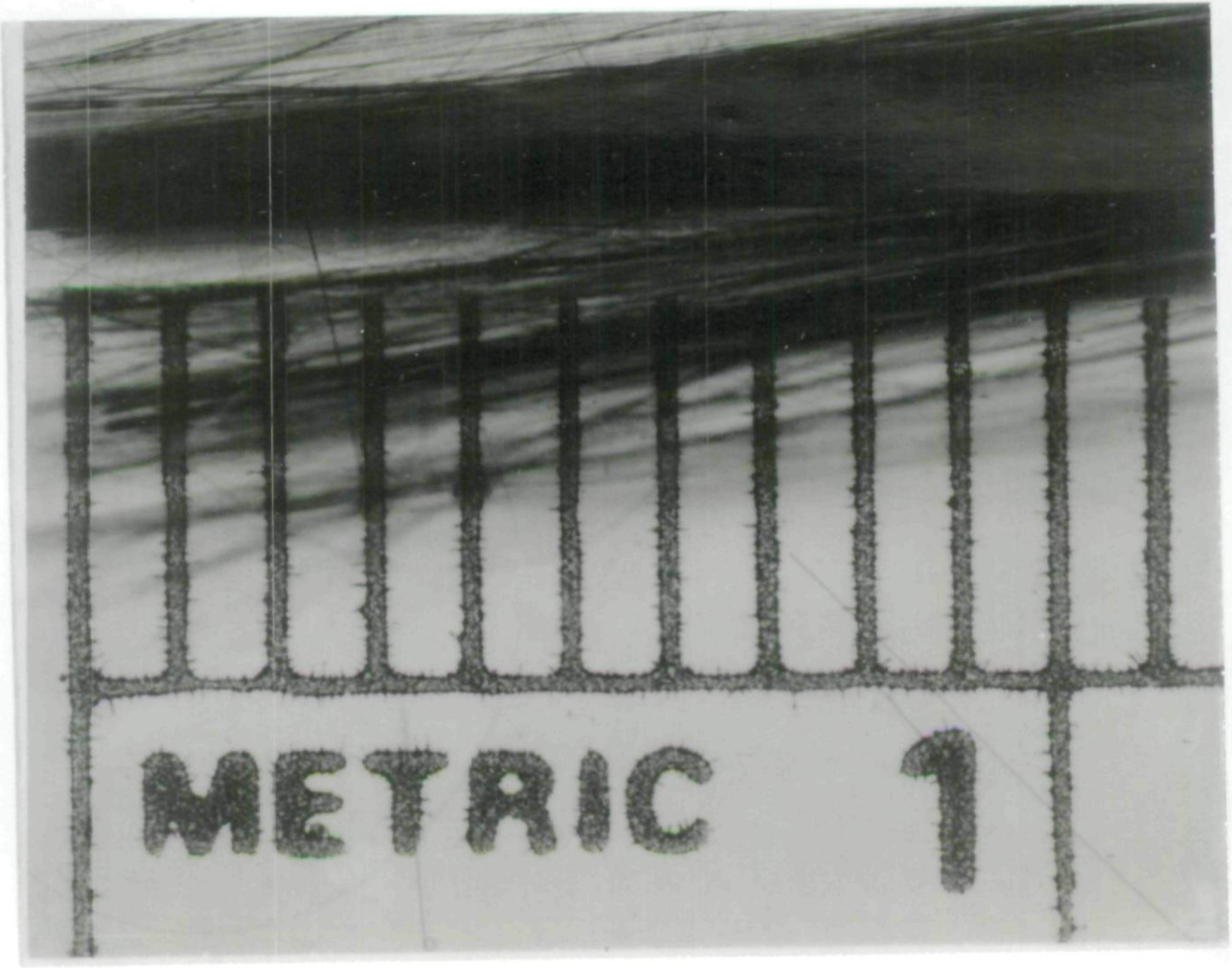
METRIC 1

Fig. 4-14(c) Ethyl Silicate Coated Fibers, 3.5 wt.%.

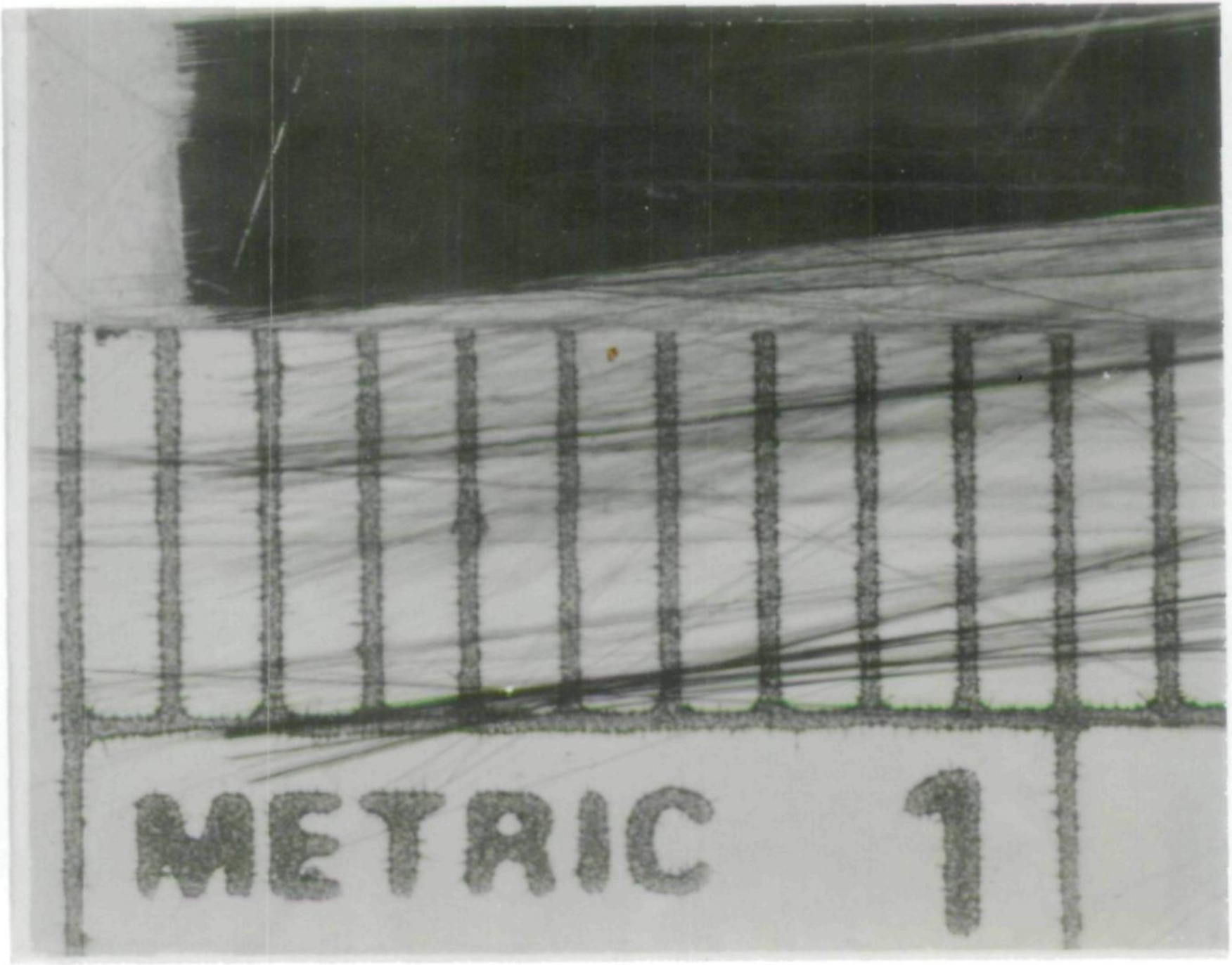
METRIC 1

Fig. 4-14(d) Boric Acid Coated Fibers, 5 wt.%.

NASA Contractor Report 159057

631-2-11

Fig. 4-14d

POLAROID

L84622037924C

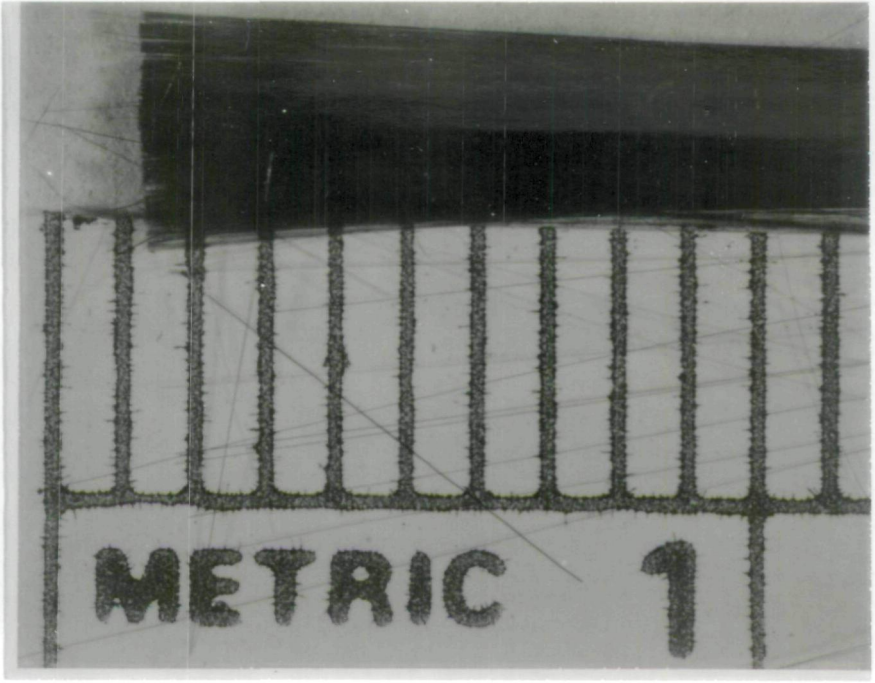
METRIC 1

Fig. 4-14 (e) Ammonium Borate Coated Fibers, 10 wt.%

NASA Contractor Report 159057

Fig. 4-14. Task III Fiber Coatings Showing Bundle Integrity. Scale is Centimeters.

POLAROID

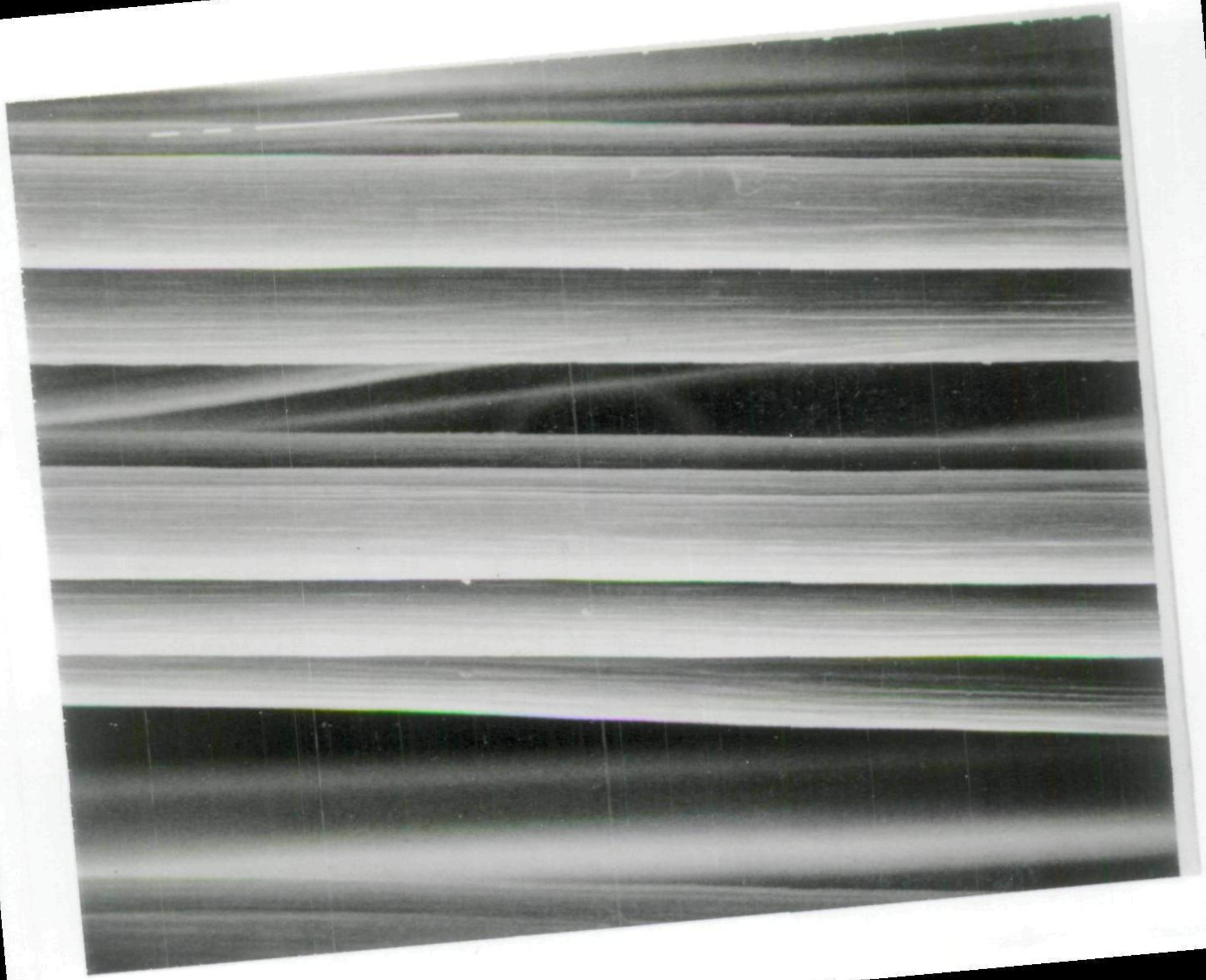

Fig. 4-15. SEM Micrograph of Ethyl Silicate, from an Isopropanol Solution, Coated Carbon Fibers.

POLAROID L84622G373240

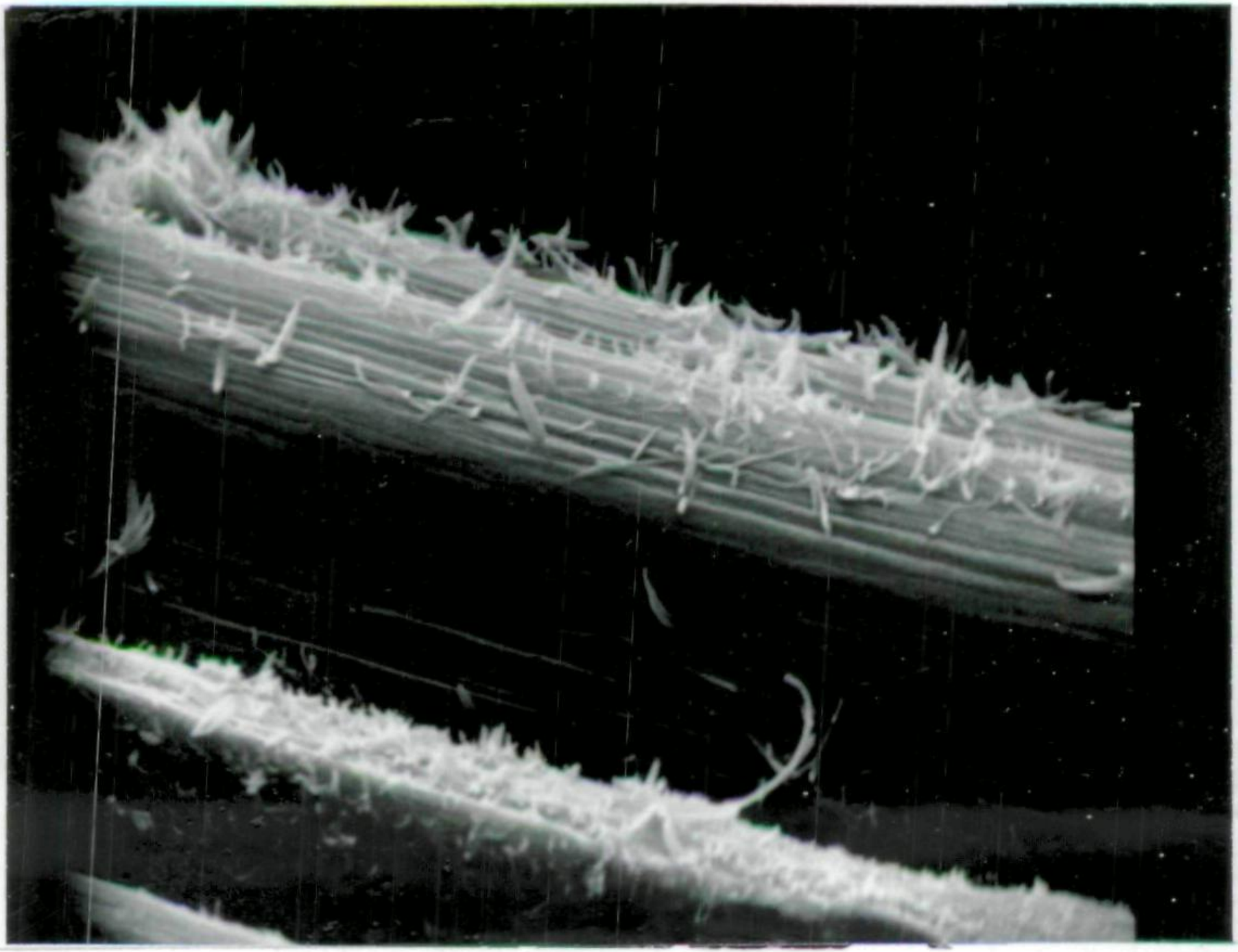

Fig. 4-16(a) Sodium Silicate,
10 wt.%, Coated Carbon
Fiber. 2000X

NASA Contractor Report 159057

Fig. 4-16(a)

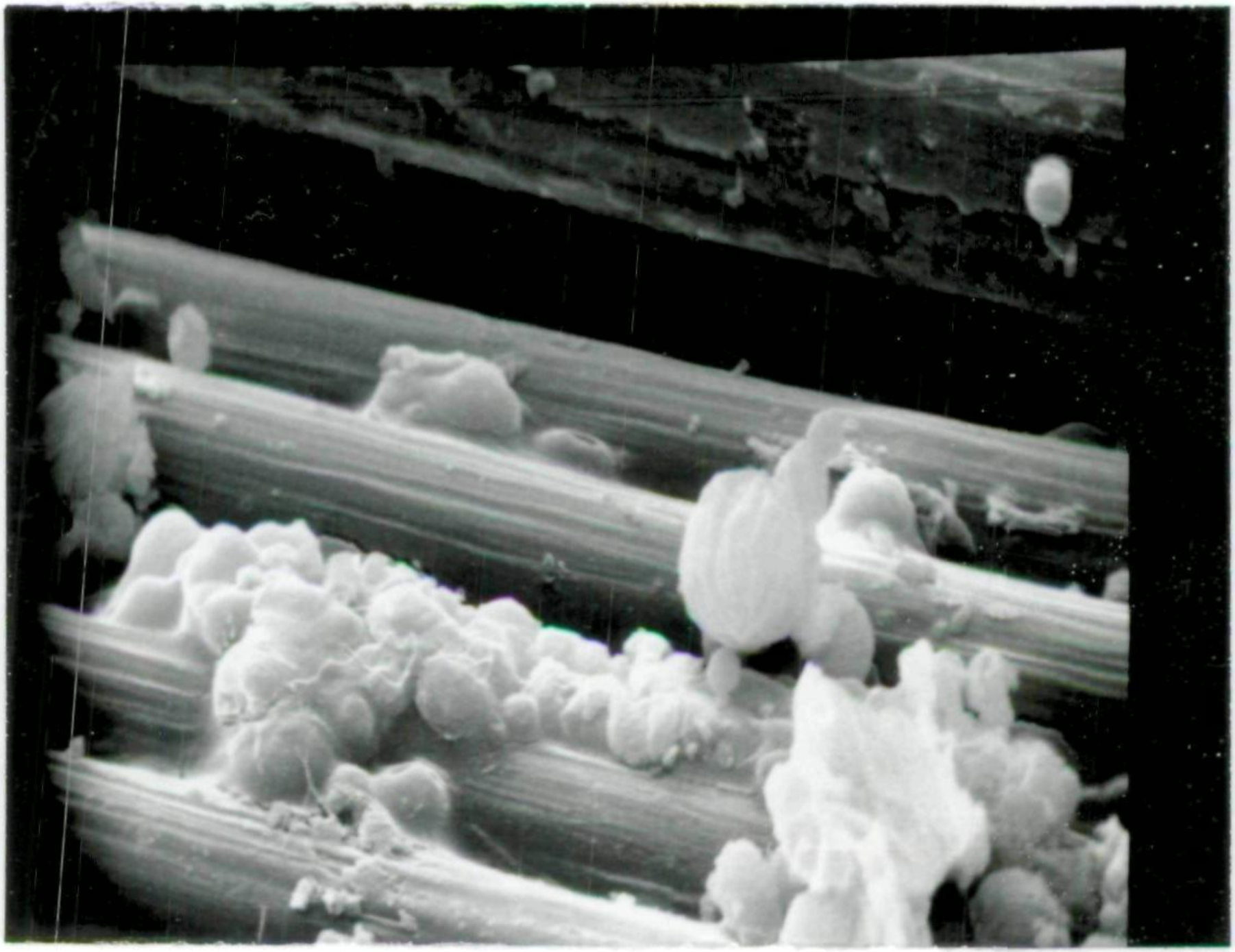

Fig. 4-16(b) Silica Gel, 6 wt.%, Coated Carbon Fiber. 2000X

NASA Contractor Report 159057

Fig. 4-16. SEM Micrographs of Sodium Silicate and Silica Gel Coated Carbon Fibers.

Fig. 4-16(b)

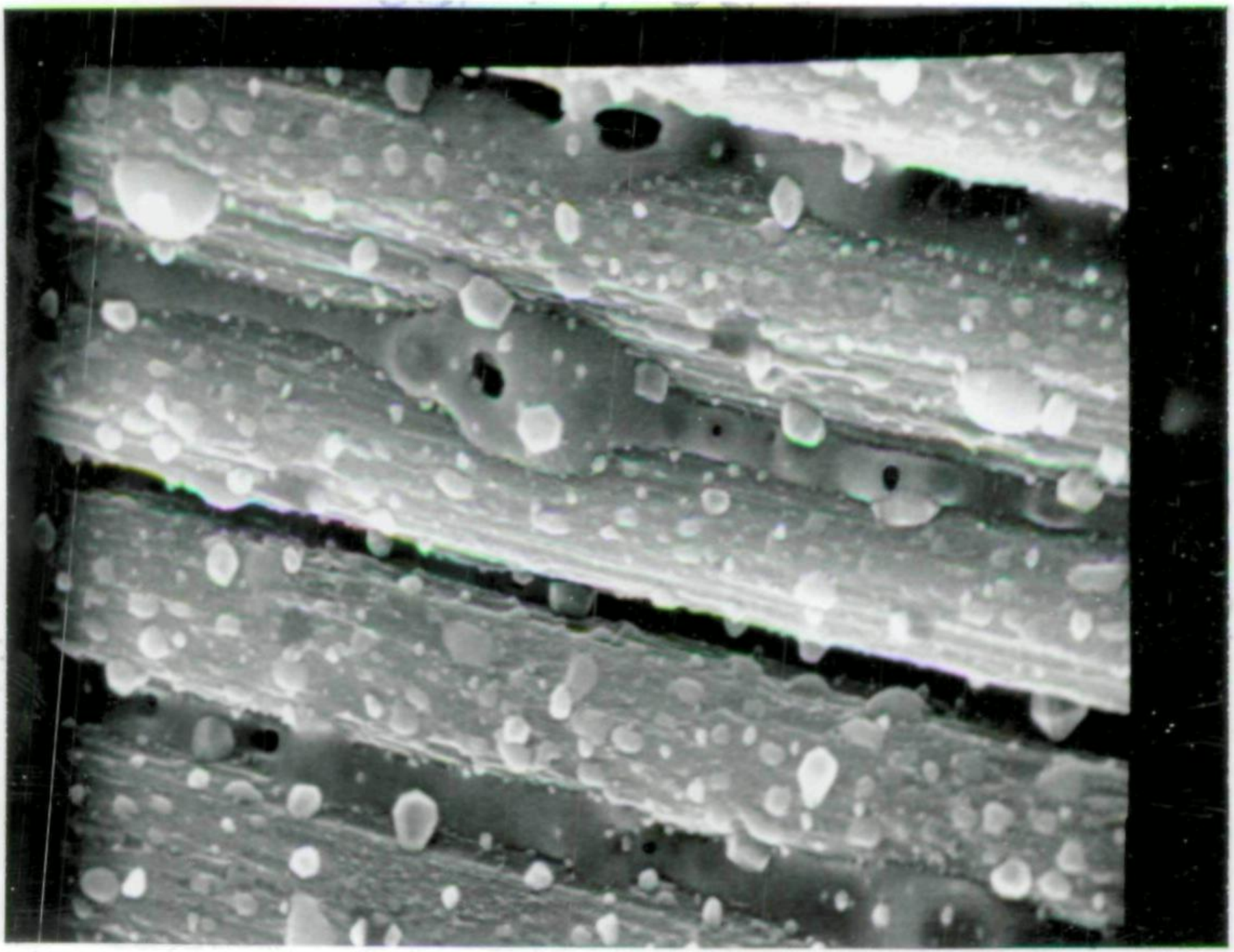

Fig. 4-17(a) Sodium Silicate, 10 wt.%, Coated Fibers. 2000 X

NASA Contractor Report 159057

631-1-79

Fig. 4-17(a)

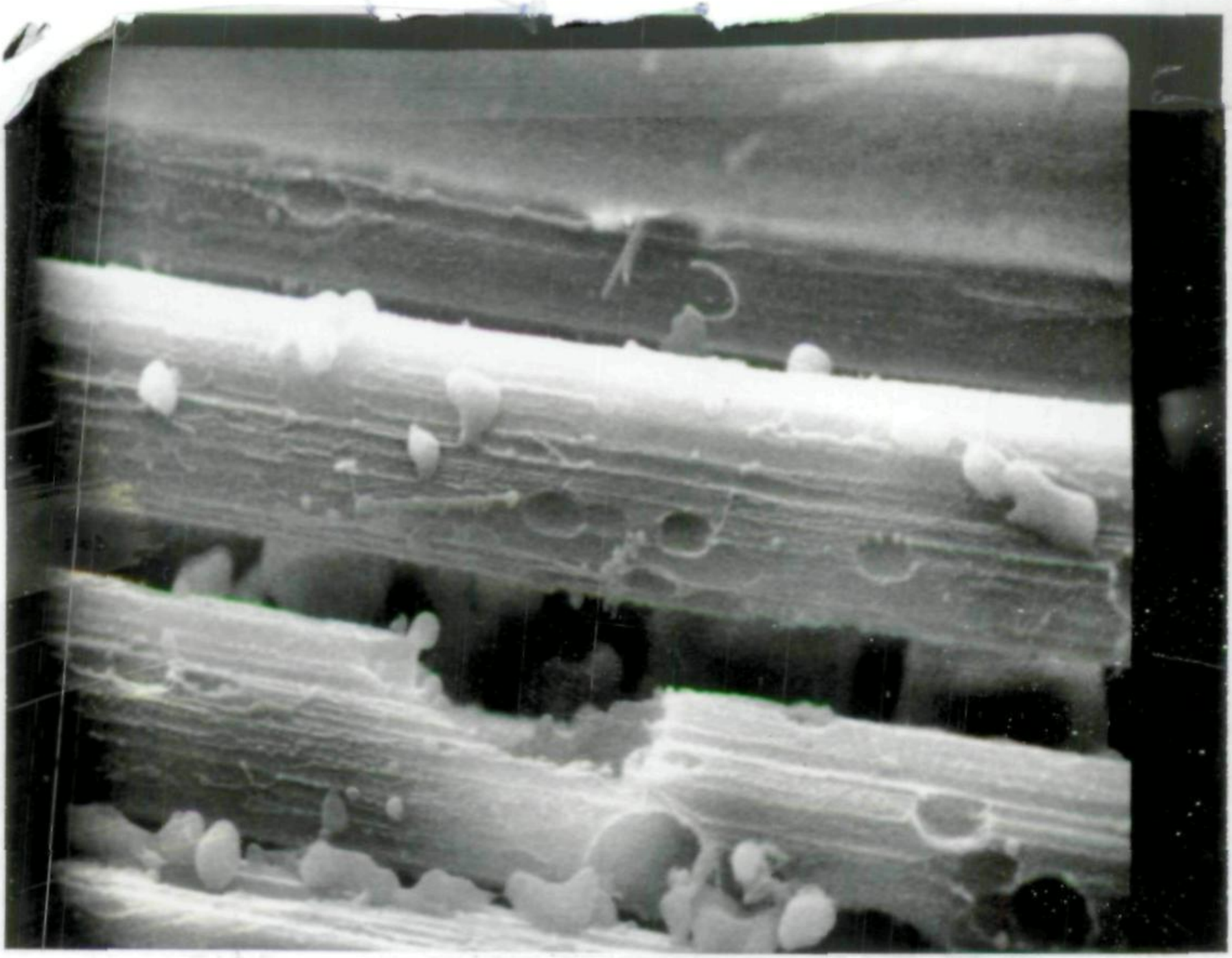

Fig. 4-17(b) Silica Gel, 6 wt.%, Coated Fibers. 2000X

NASA Contractor Report 159057

631-1-22

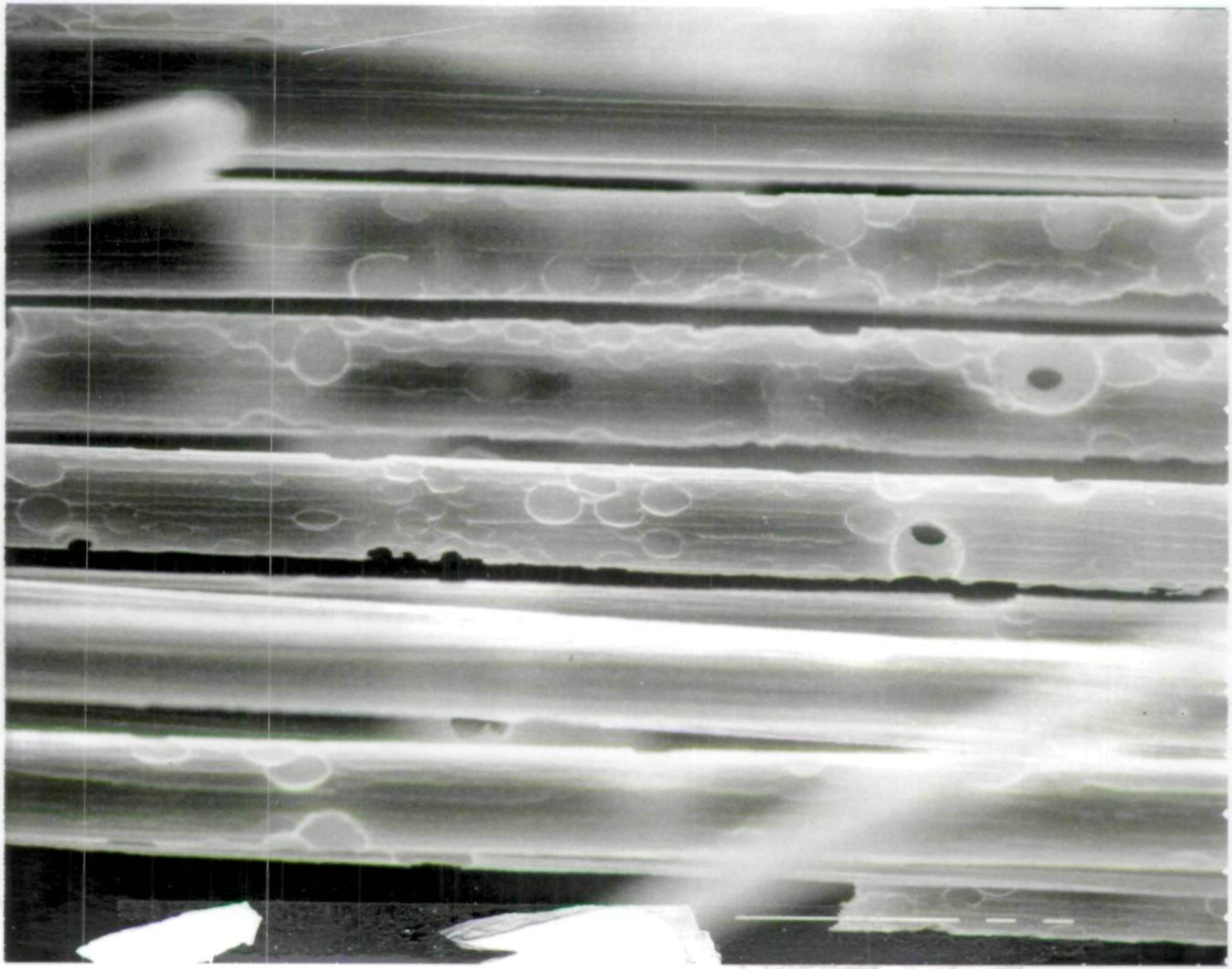

Fig. 4-17(c) Ethyl Silicate, 3.5 wt.%, Coated Fibers. 1600X

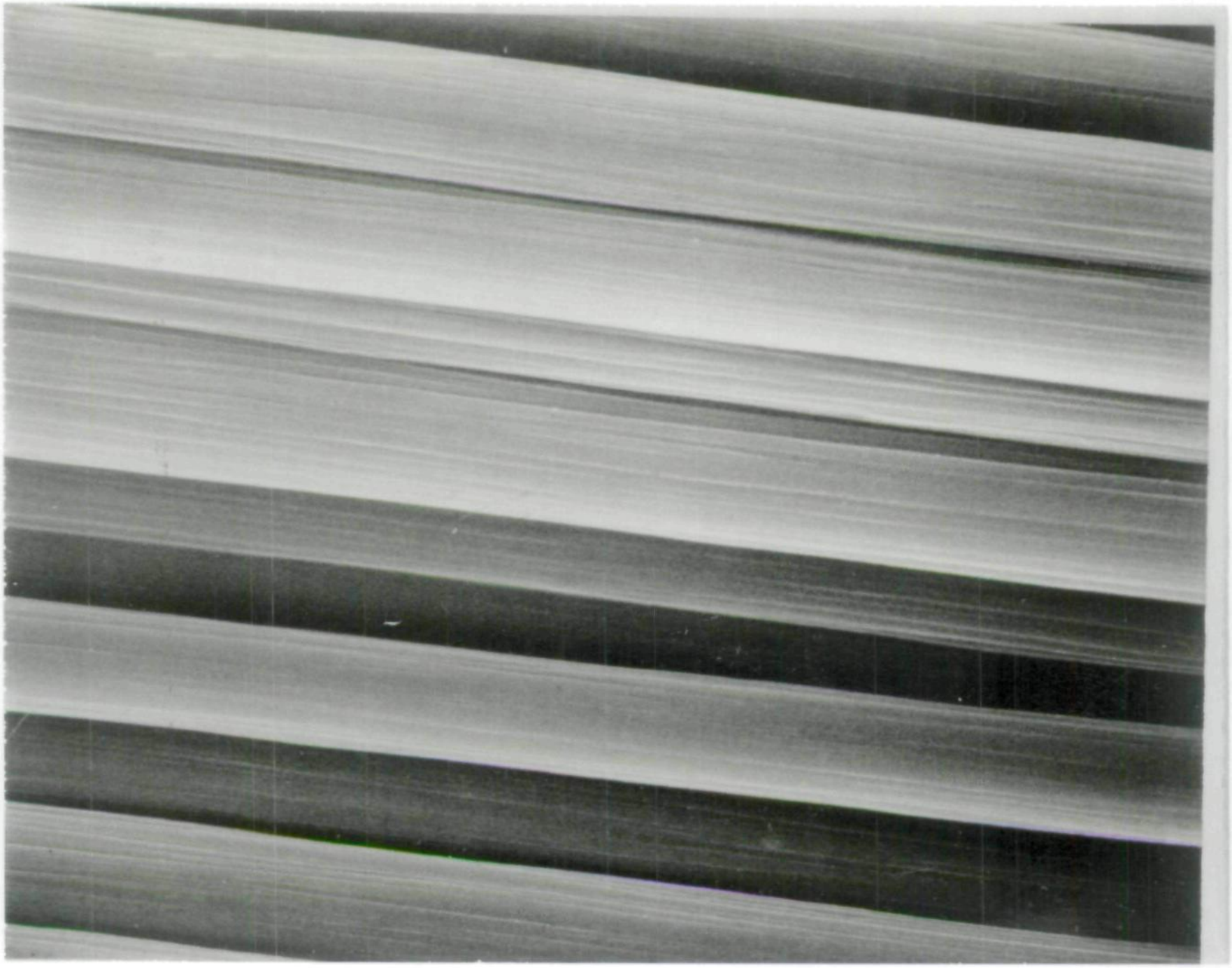

Fig. 4-17(d) Boric Acid, 7 wt.%,
Coated Fibers. 1800X

NASA Contractor Report 195057

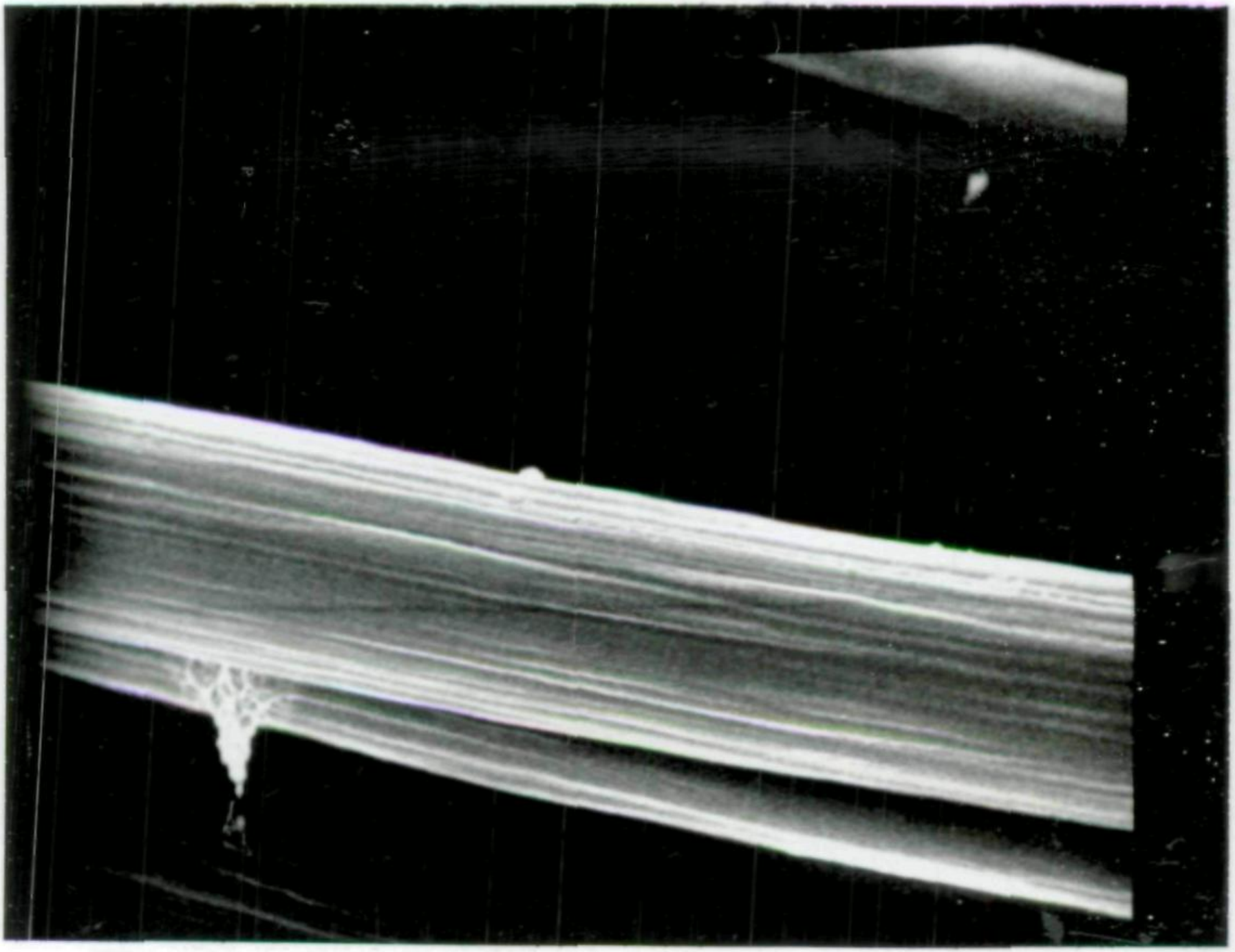

Fig. 4-17(d) Ammonium Borate, 10 wt.%, Coated Fibers. 2000X

Fig. 4-17. SEM Micrograph of Task III Coatings on Carbon Fibers After 1000°C Static Burn Test for 30 Seconds.

NASA Contractor Report 195057

Fig. 4-17(c)

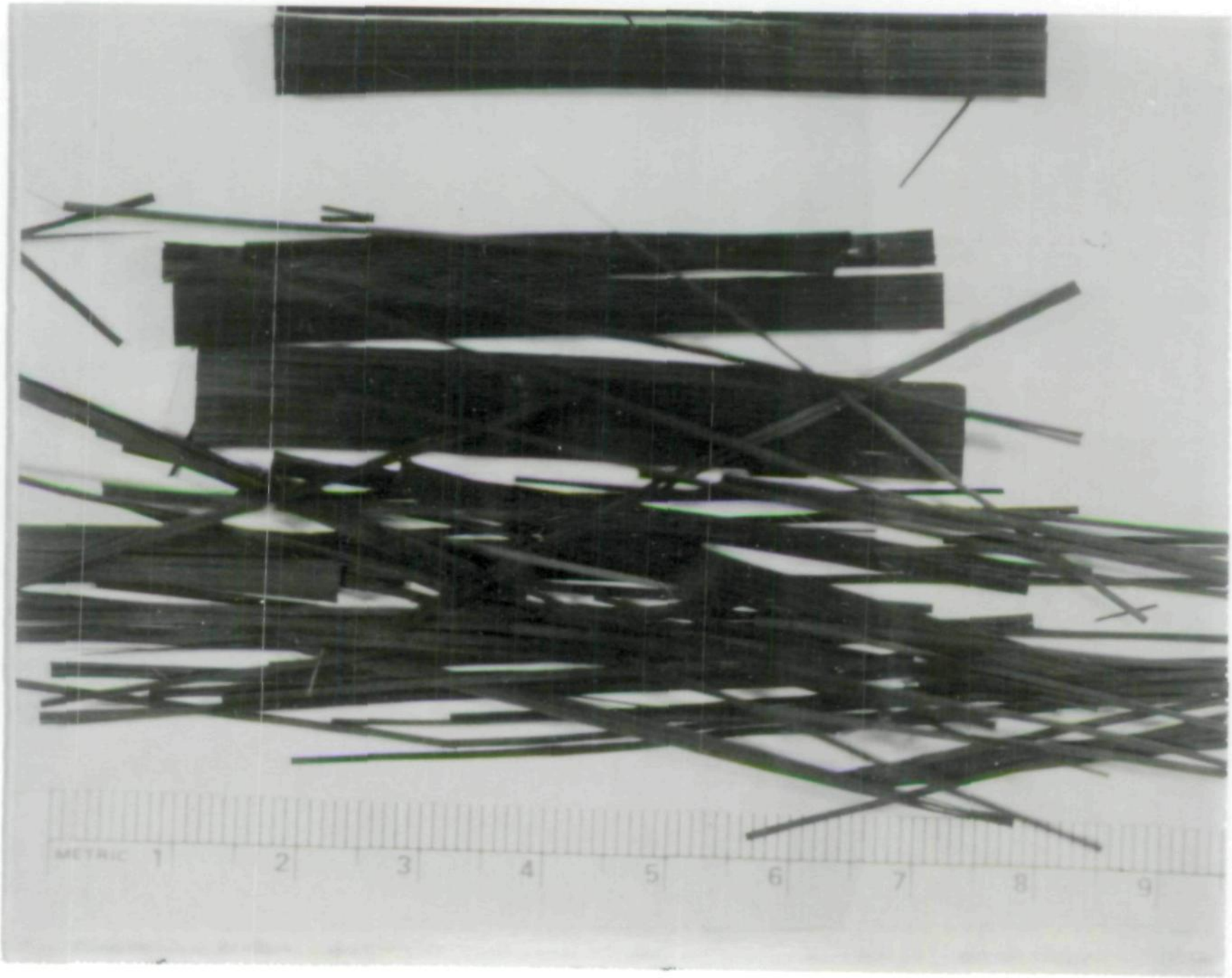
METRIC 1 2 3 4 5 6 7 8 9

Fig. 4-18(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.

NASA Contractor Report 159057

4-18A

Fig.

L84322C37923C

POLAROID

Fig. 4-18(b) SEM Micrograph from One of Larger Pieces of Sample After Burn Testing. 1350X

Fig. 4-18(d) Kevex Trace of Burned Composite Showing Gold Coating, Silicon, and Sodium.

NASA Contractor Report 159057

Fig. 4-18. Sodum Silicate Coated Fiber Composite After Dynamic Burn Test.

Fig. 4-18(d)

Fig. 4-18(c)

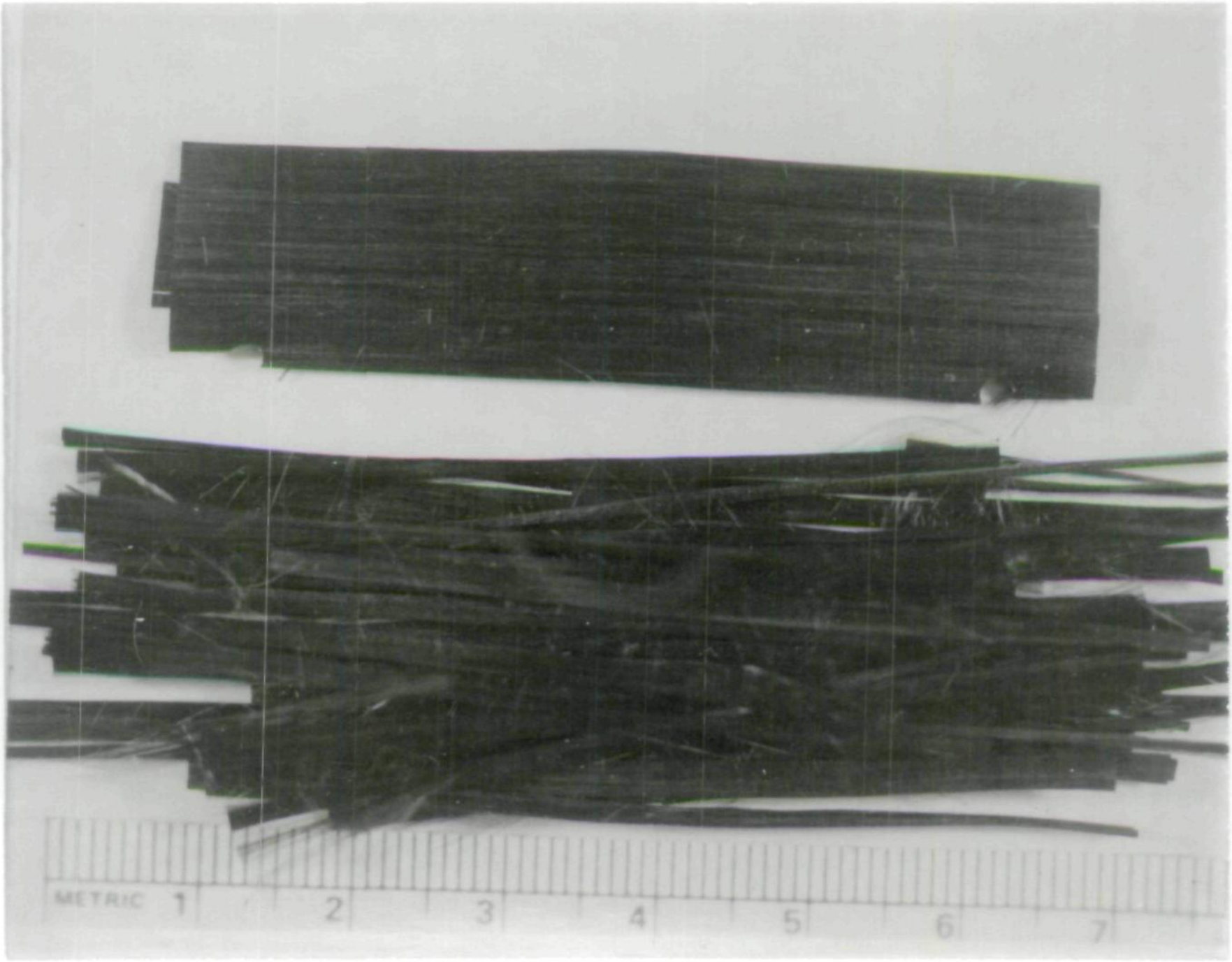
METRIC
1
2
3
4
5
6
7

Fig. 4-19(a) Composite After 10 Minutes Dynamic Burn Best, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 12 M/sec. Air Velocity. Scale is Centimeters.

NASA Contractor Report 159057

POLAROID

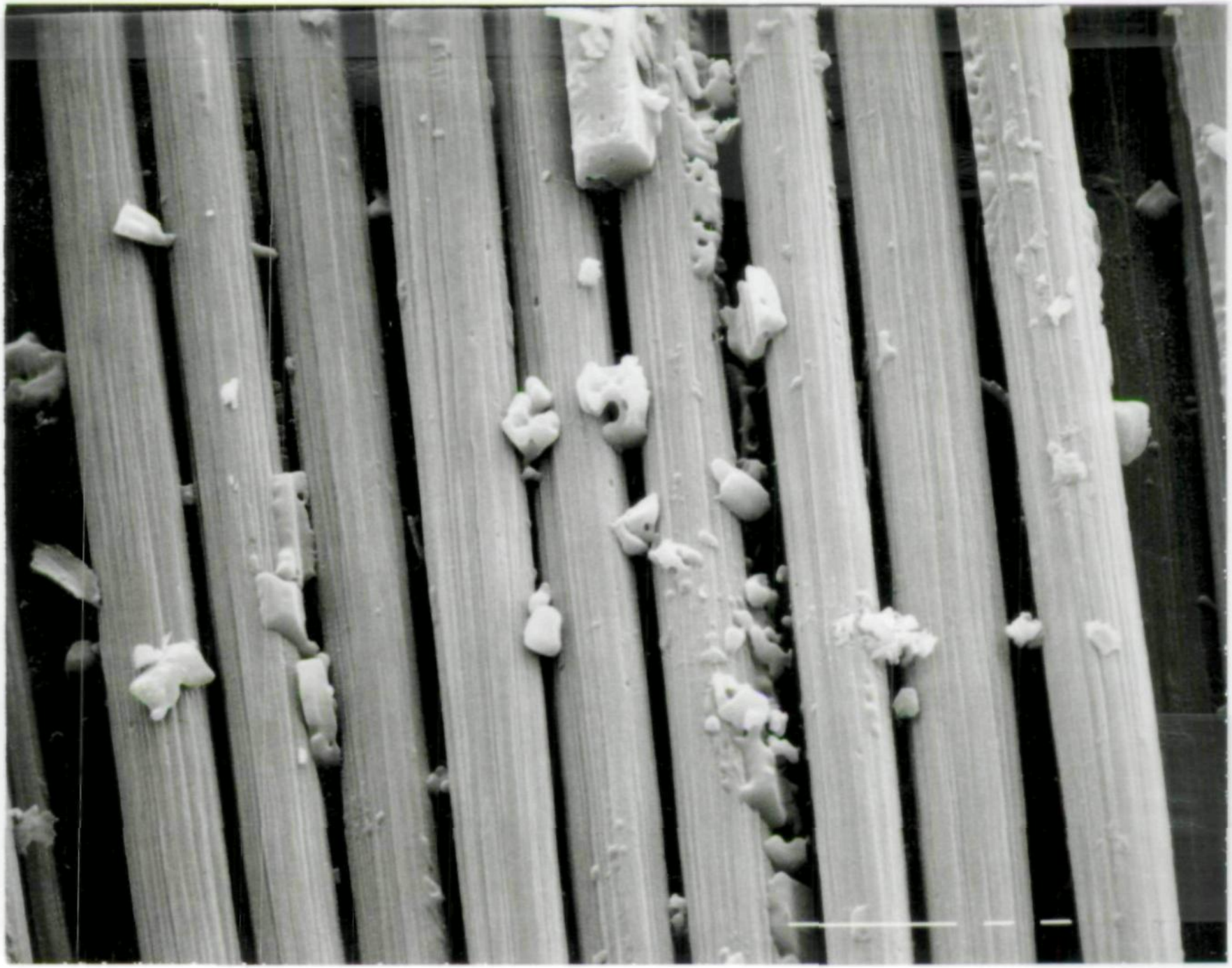

Fig. 4-19(b) SEM Micrograph from One of the Smaller Fiber Bundles After Burn Testing. 1350X

NASA Contractor Report 159057

631-4-8

POLAROID

Fig. 4-19(b)

Fig. 4-19(c) SEM Micrograph from One of the Larger Fiber Bundles After Burn Testing. 1200X

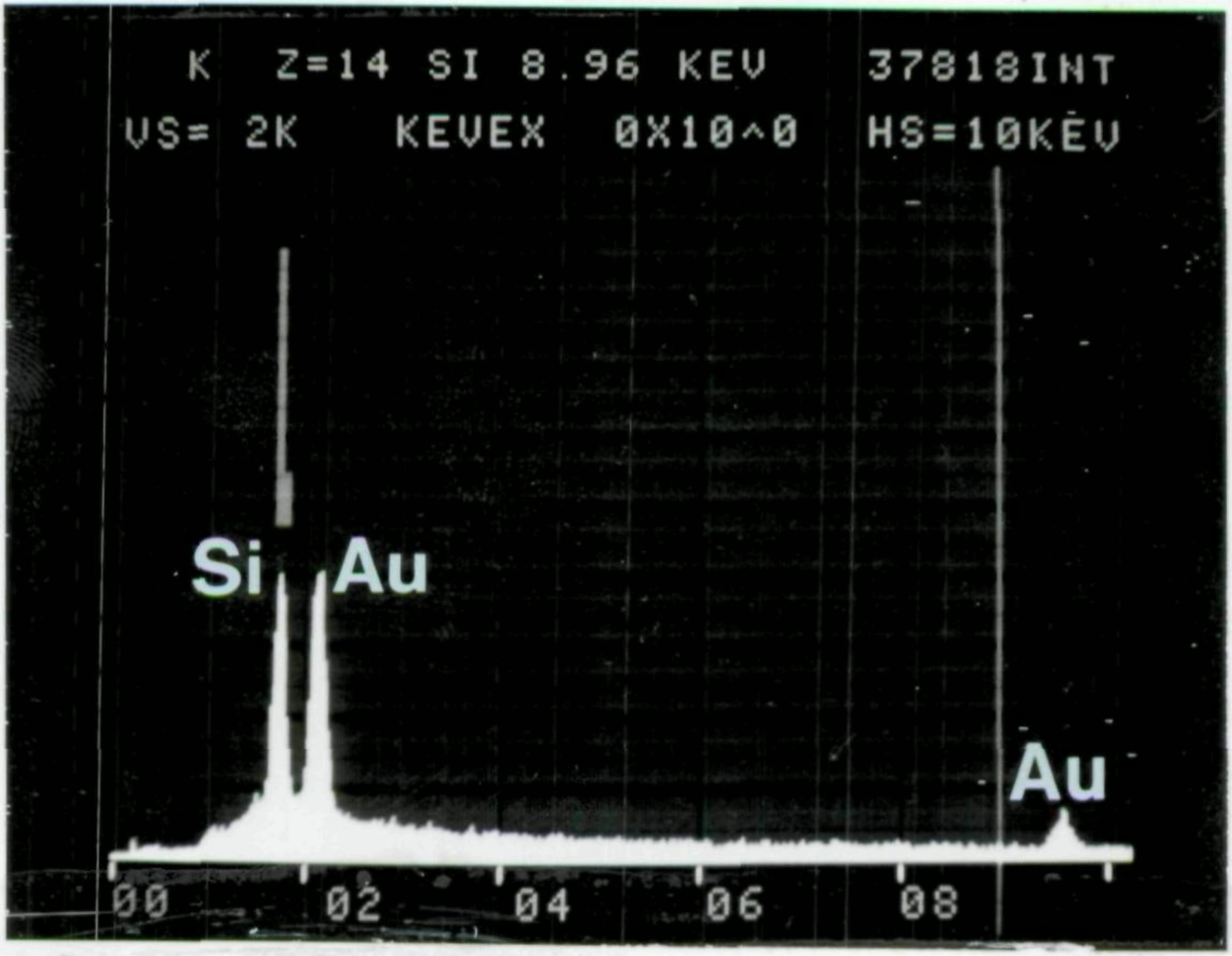
K Z=14 SI 8.96 KEV 37818INT
VS= 2K KEVEX 0X10^0 HS=10KEV
Si
Au
Au
00
02
04
06
08

Fig. 4-19(d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon.

NASA Contractor Report 159057

Fig. 4-19. Silica Gel Coated Fiber Composite After Dynamic Burn Test.

Fig. 4

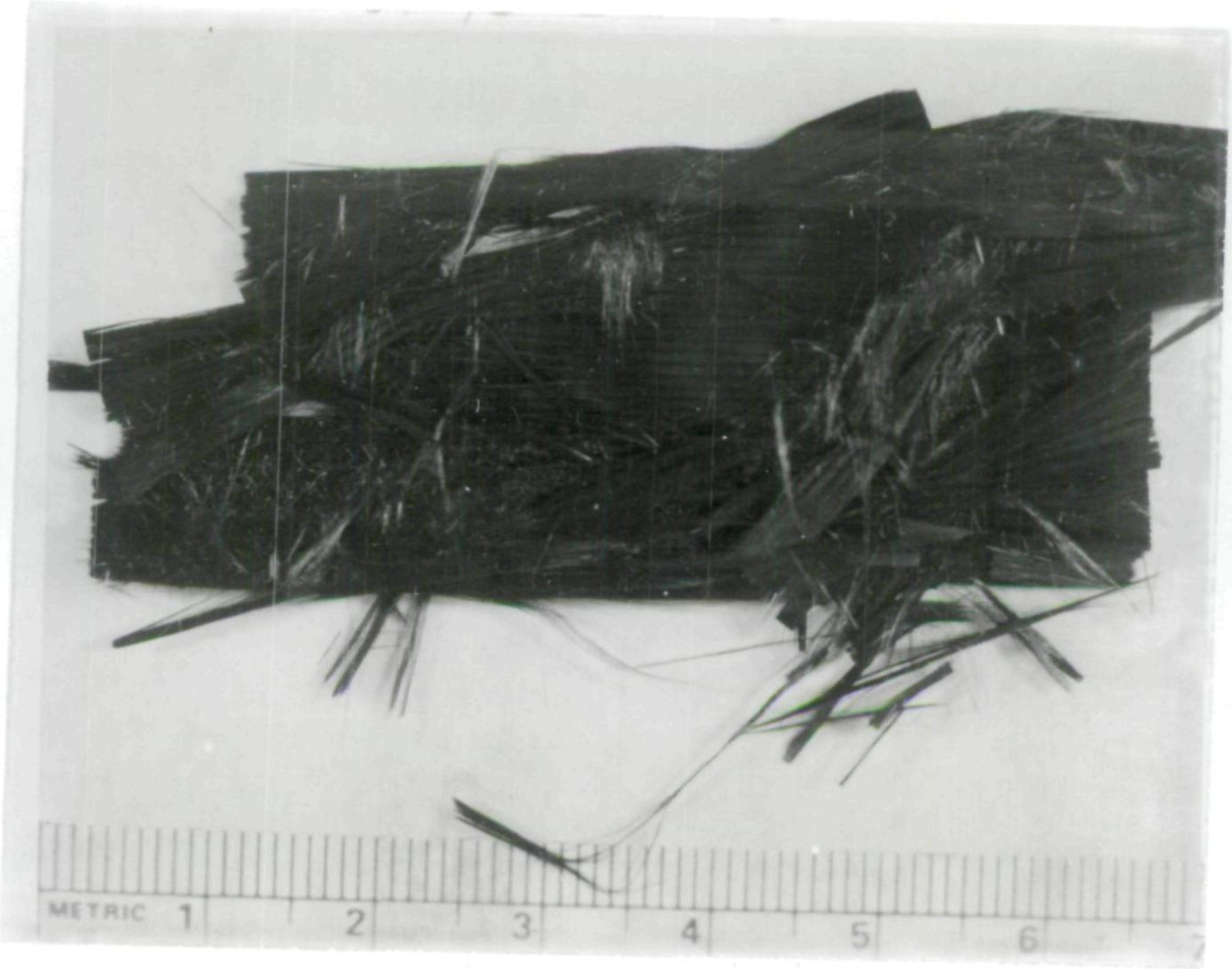
METRIC 1 2 3 4 5 6

Fig. 4-20(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 11 M/sec. Air Velocity. Approximately 50% of Fibers Burned Away. Scale is Centimeters.

Fig. 4-20(b) SEM Micrograph from Cooler Region of the Largest Piece of Sample After Burn Testing.

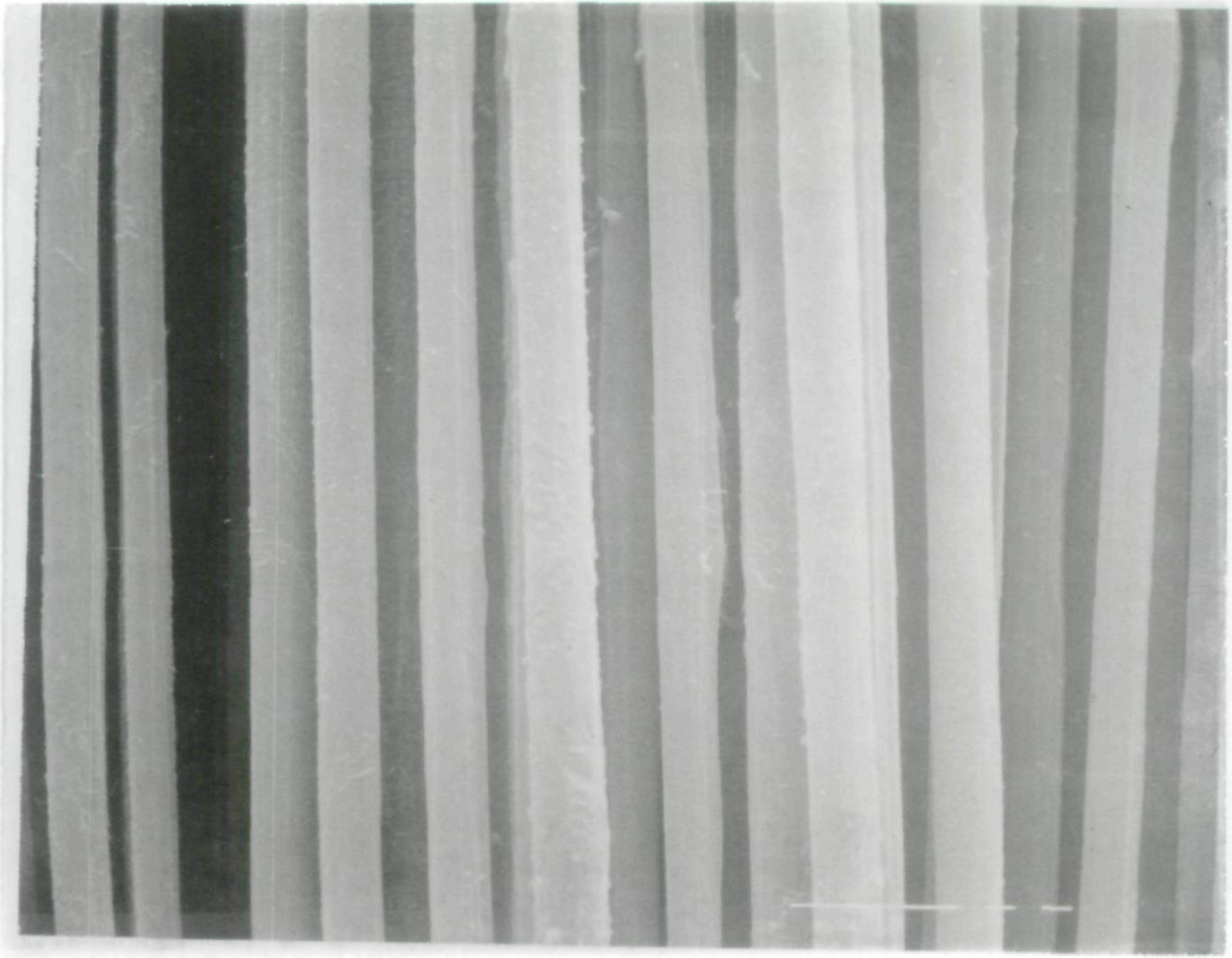

Fig. 4-20(c) SEM Micrograph from Small Fiber Strands at Edge of Sample After Burn Testing.

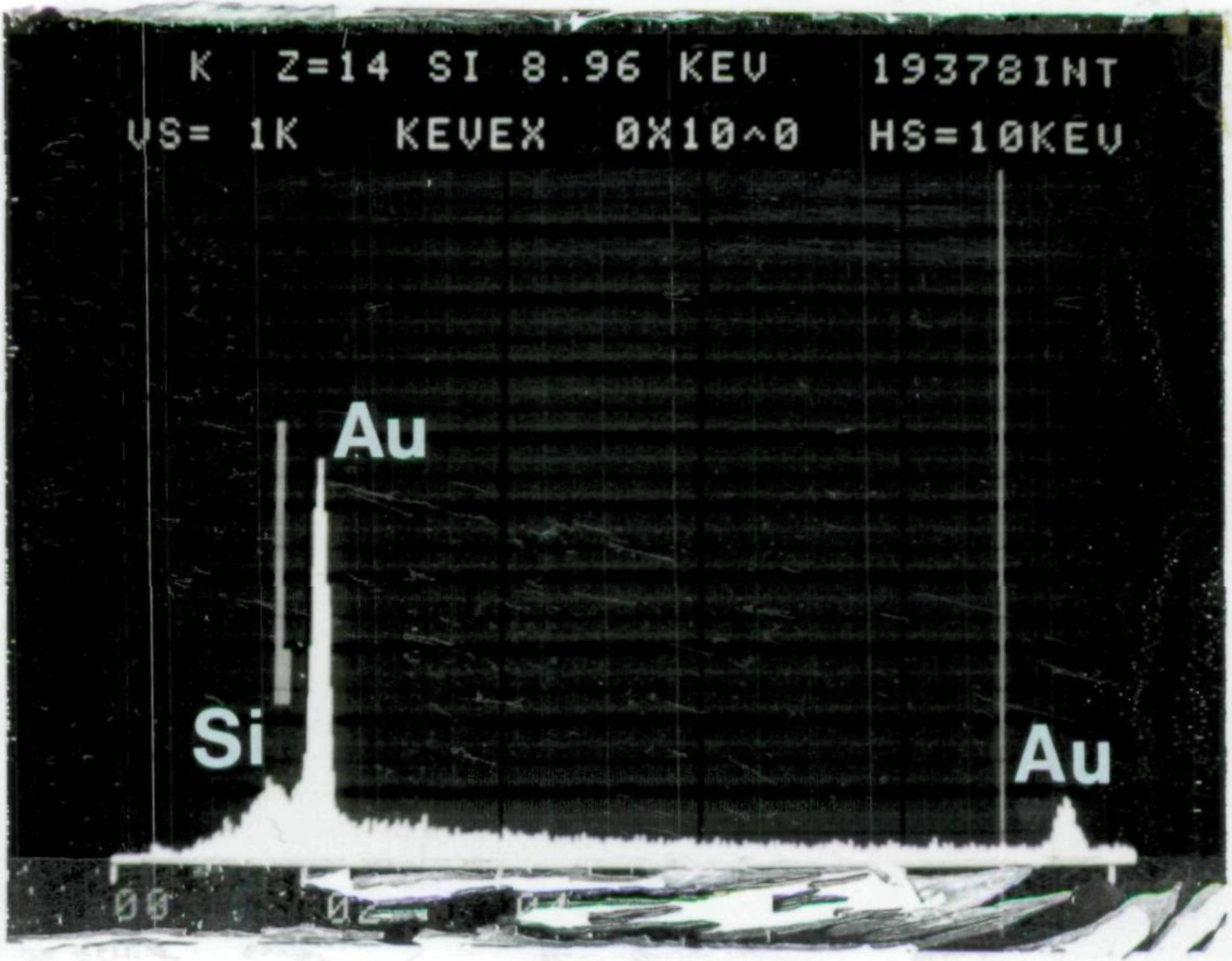
K Z=14 SI 8.96 KEV 19378INT
VS= 1K KEVEX 0X10^0 HS=10KEV
Au
Si
Au
00
02

Fig. 4-20(d) Kevex Trace of Burned Composite Showing Gold Coating and Small Amount of Silicon.

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Fig. 4-20 Ethyl Silicate Coated Fiber Composite After Dynamic Burn Test.

Fig. 4-20 (d)

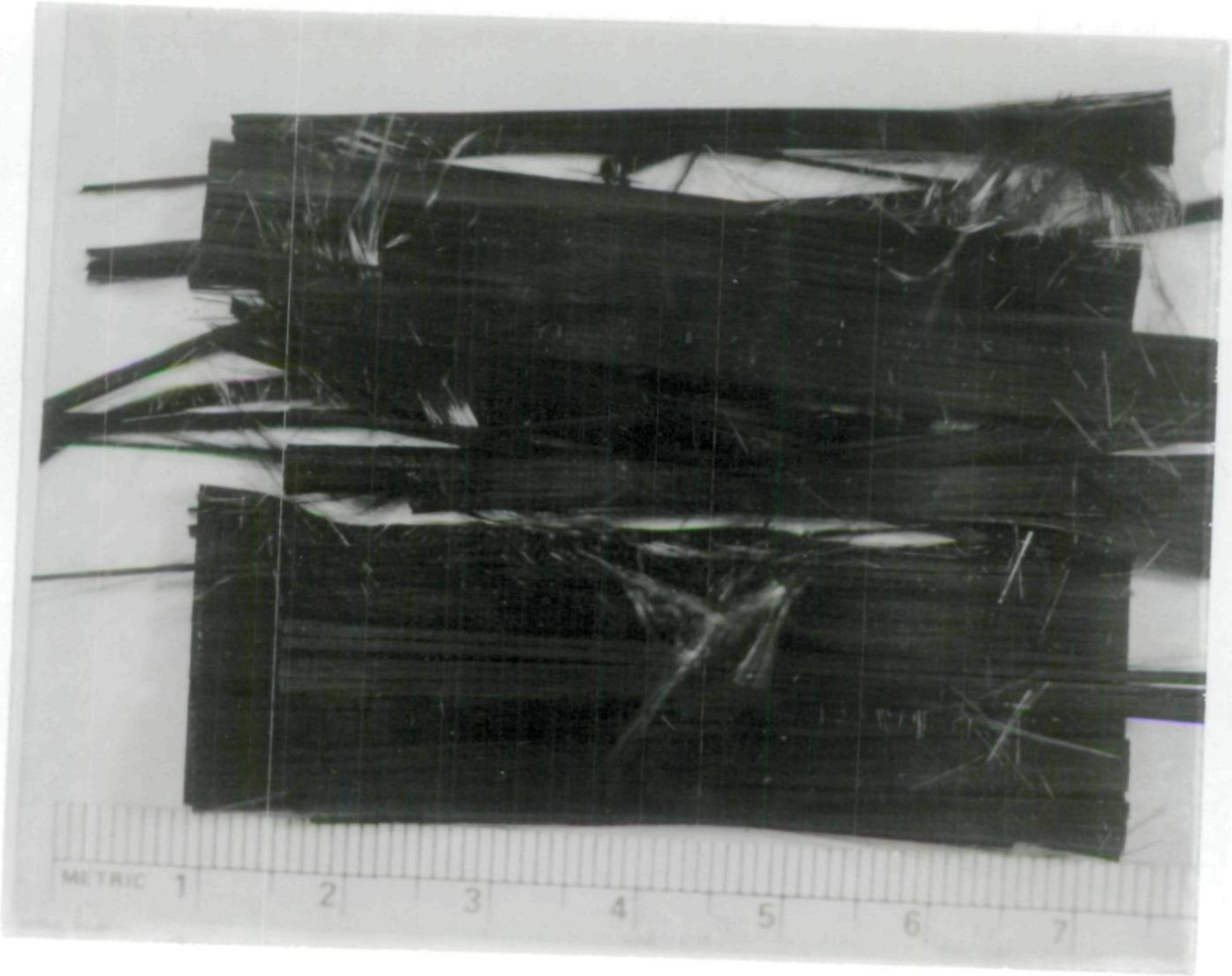
METRIC
1
2
3
4
5
6
7

Fig. 4-21(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 11 M/sec. Air Velocity. Scale is Centimeters.

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4-21A

Fig.

POLAROID L84432087912A

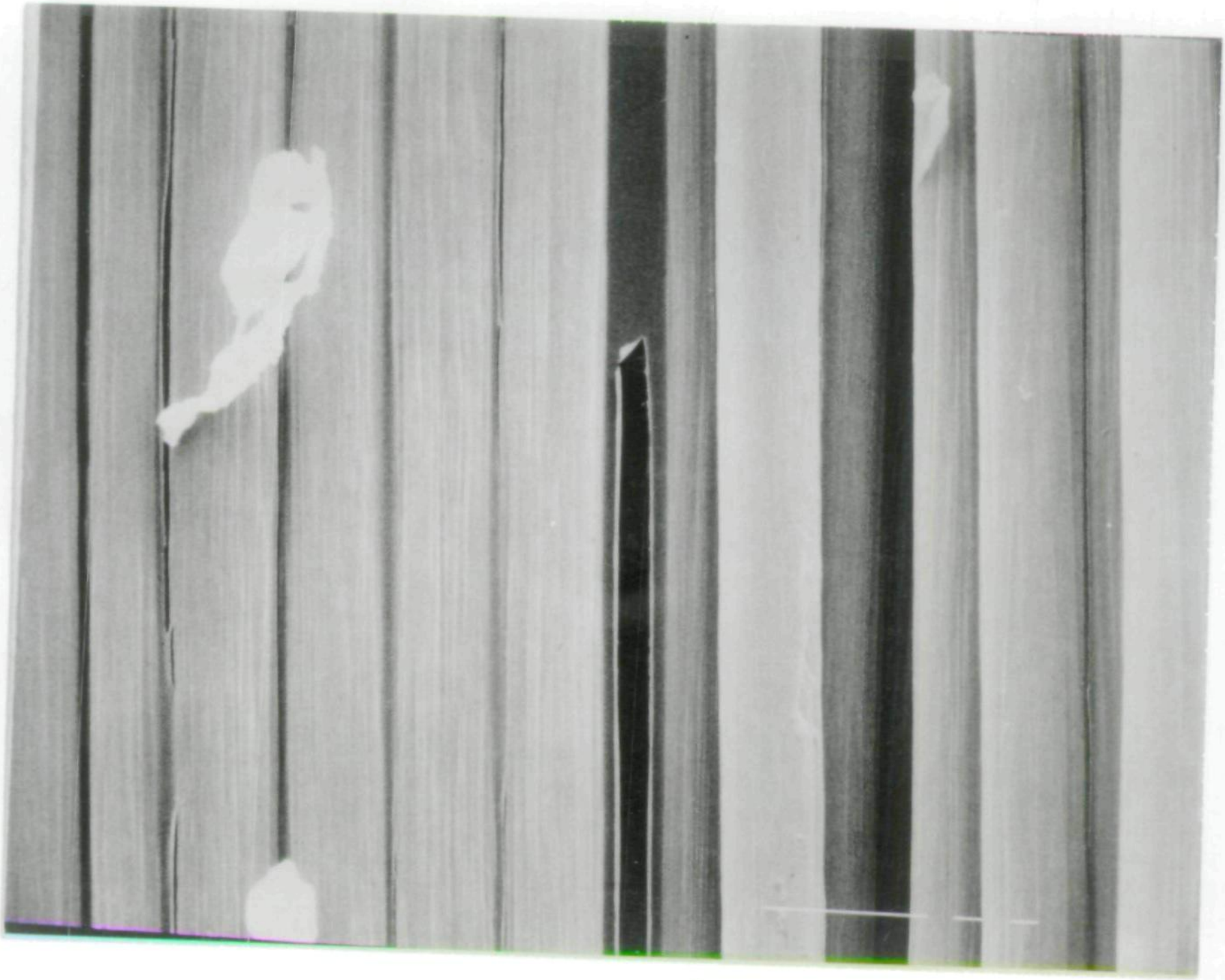

Fig. 4-21(b) SEM Micrograph from Large Piece of Sample Remaining After Burn Testing.

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POLAROID

L84622C37324C

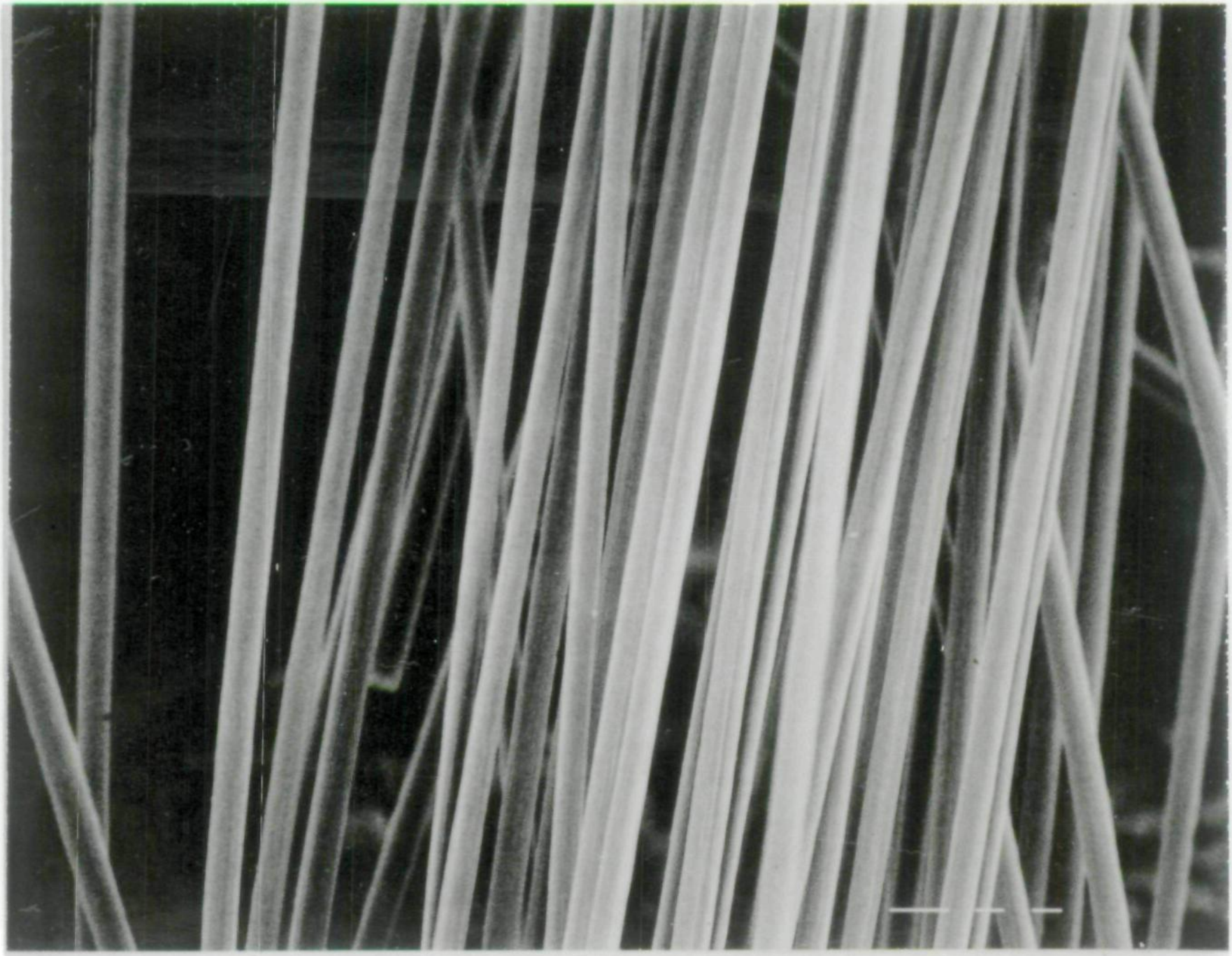

Fig. 4-21(c) SEM Micrograph from Fuzzy Edge of a Loose Strand After Burn Testing.

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Fig. 4-21 c

POLAROID L84622C079240

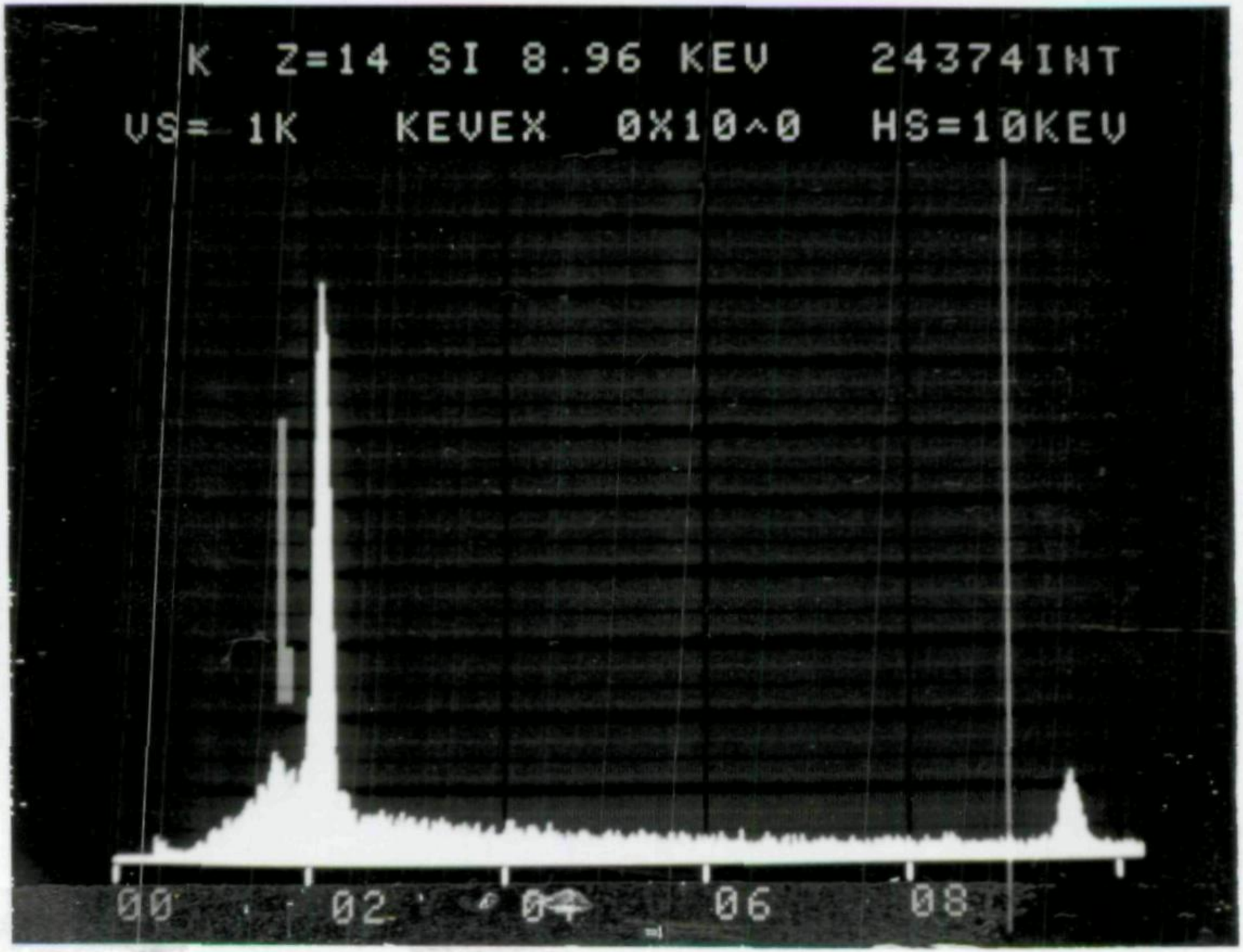
K Z=14 SI 8.96 KEV 24374INT
VS= 1K KEVEX 0X10^0 HS=10KEV
00
02
04
06
08

Fig. 4-21(d) Kevex Trace of Burned Composite Showing Gold Coating and Background Silicon.

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Fig. 4-21 Boric Acid Coated Fiber Composite After Dynamic Burn Test.

Fig. 4-21(d)

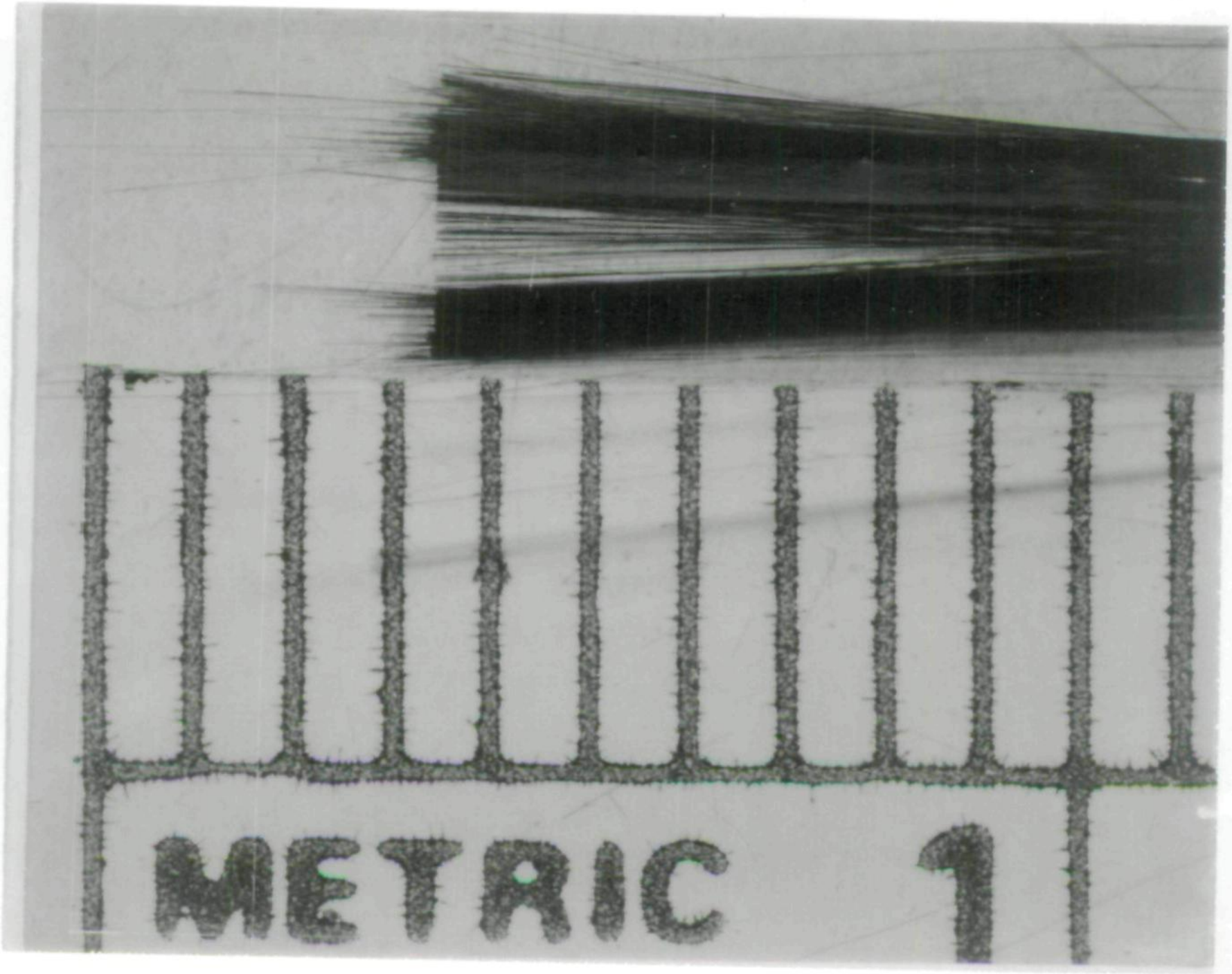
METRIC 1

Fig. 4-22(a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. Scale is Centimeters.

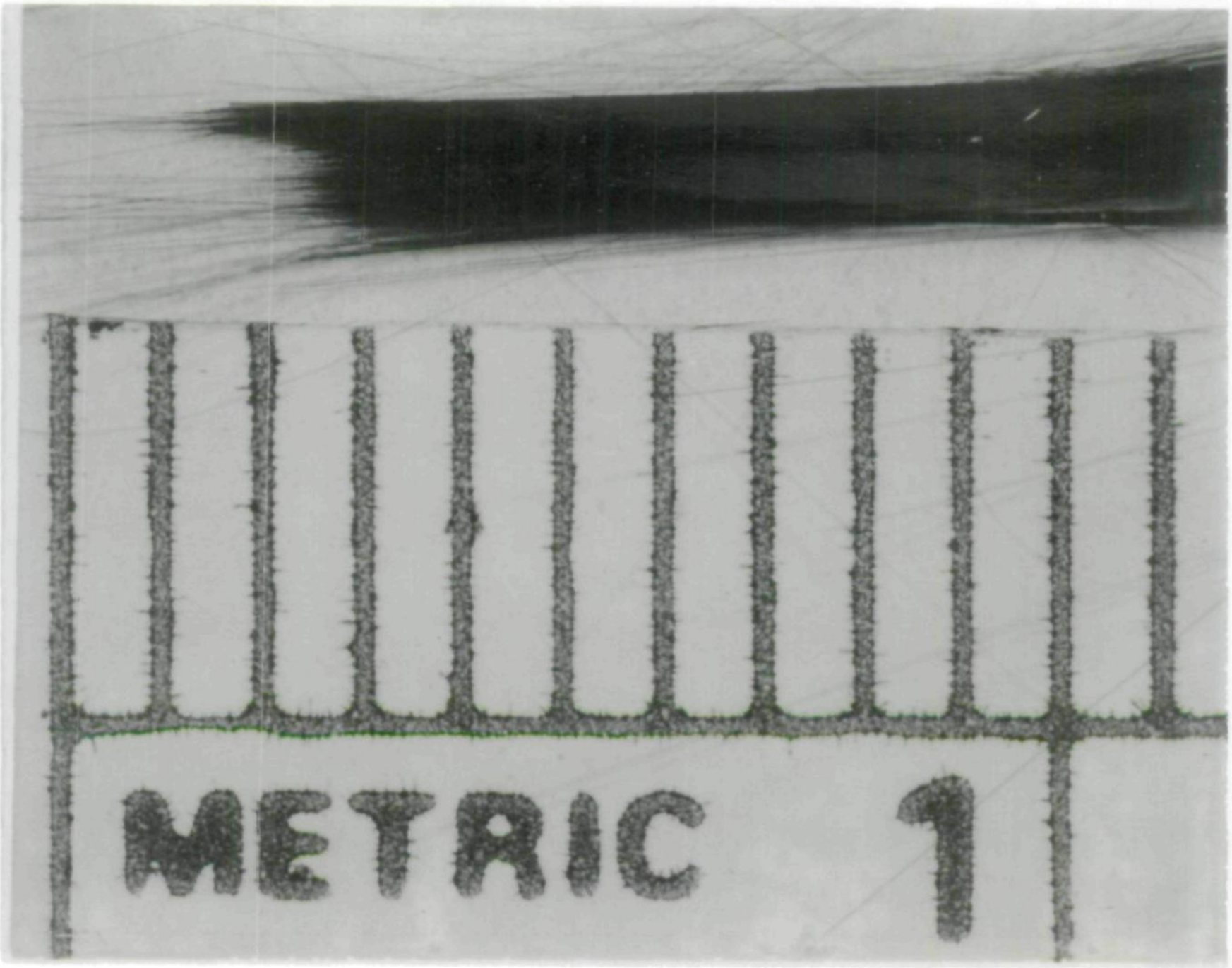
METRIC 1

Fig. 4-22(b) Silicon Carbide Coating from UCC L-31 Silicone Fluid. Scale is Centimeters.

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Fig. 4-22. Silicon Carbide Coated Carbon Fibers Showing Bundle Integrity.

POLAROID

L84622C073240

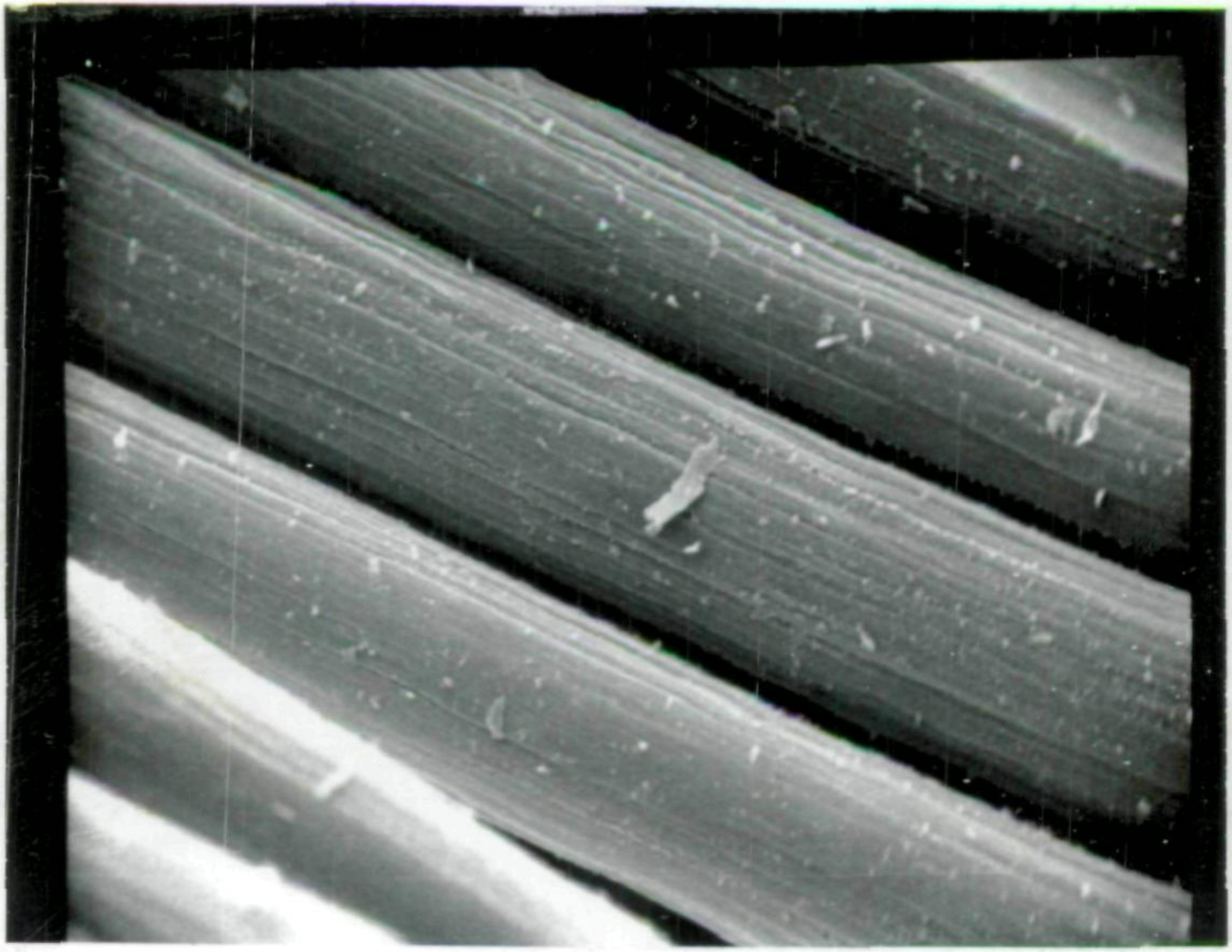

Fig. 4-23(a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. 2000X

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Fig. 4-23(a)

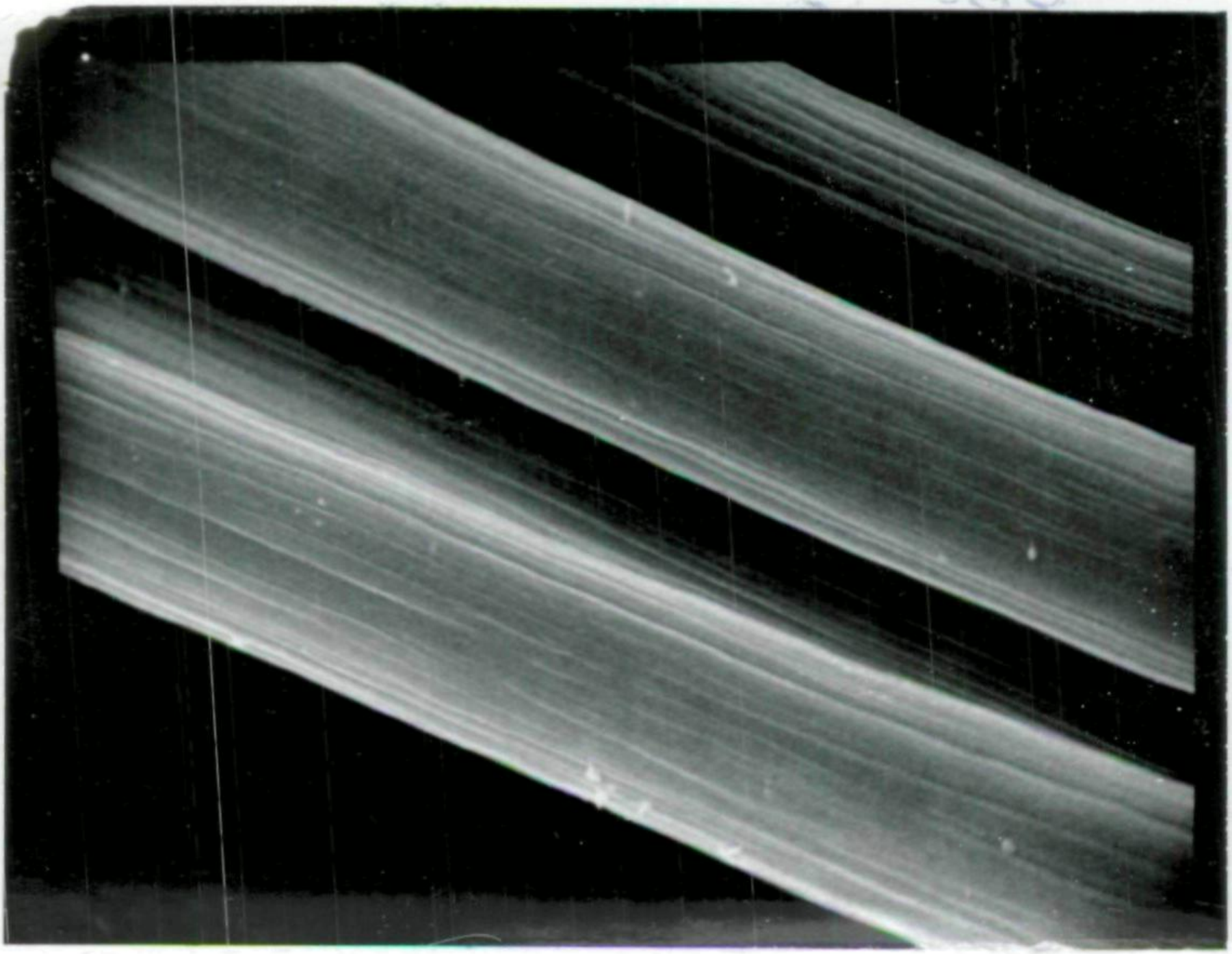

Fig. 4-23(b) Silicon Carbide Coating from UCC L-31 Silicone Fluid. 2000X

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Fig. 4-23. SEM Micrograph of Silicon Carbide Coated "Thornel" 300 Carbon Fibers.

Fig. 4-23(b)

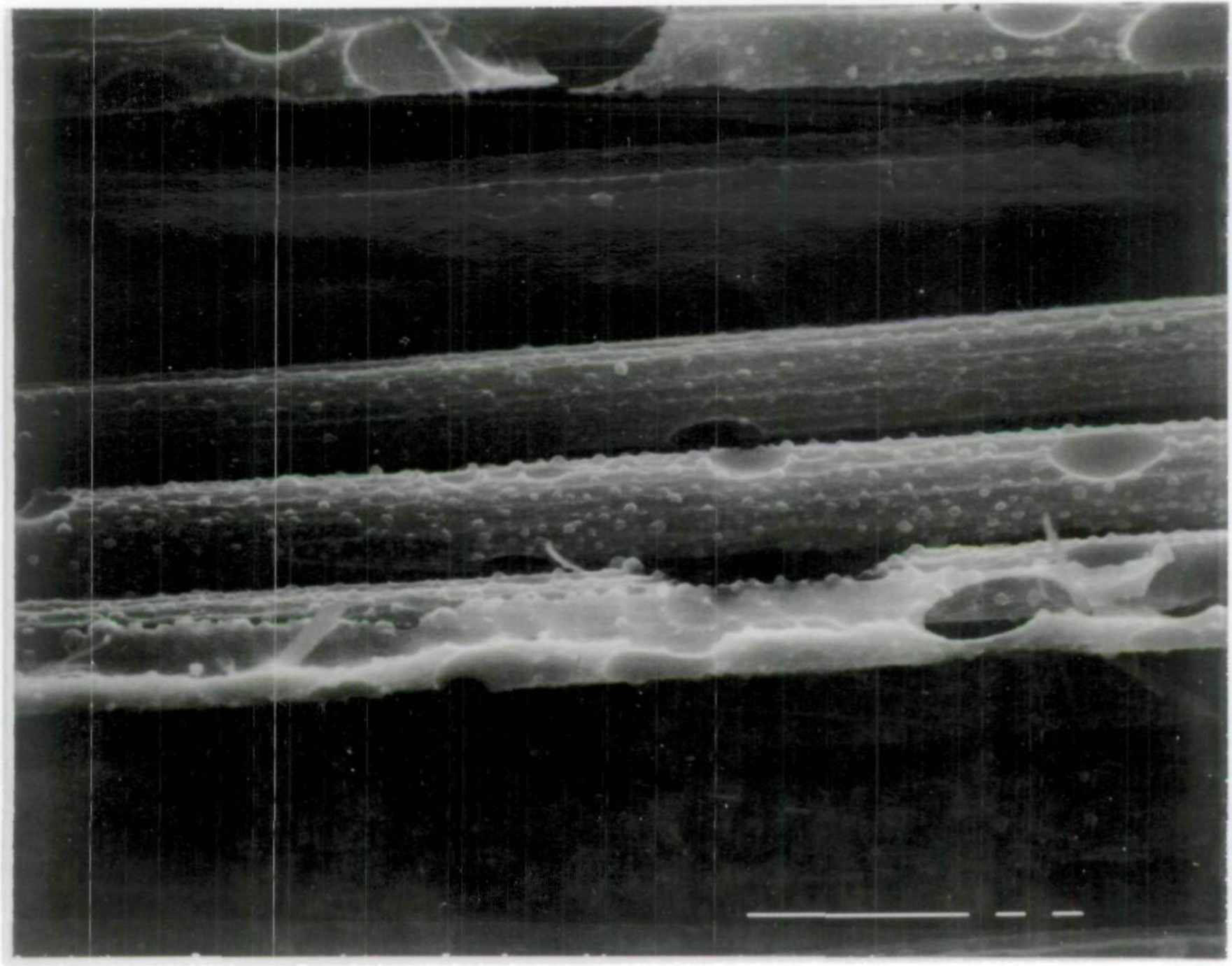

Fig. 4-24(a) Silicon Carbide Coating from UCC L-45 Silicone Fluid. 1600X

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Fig. 4-24 A

POLAROID L8443208/912A

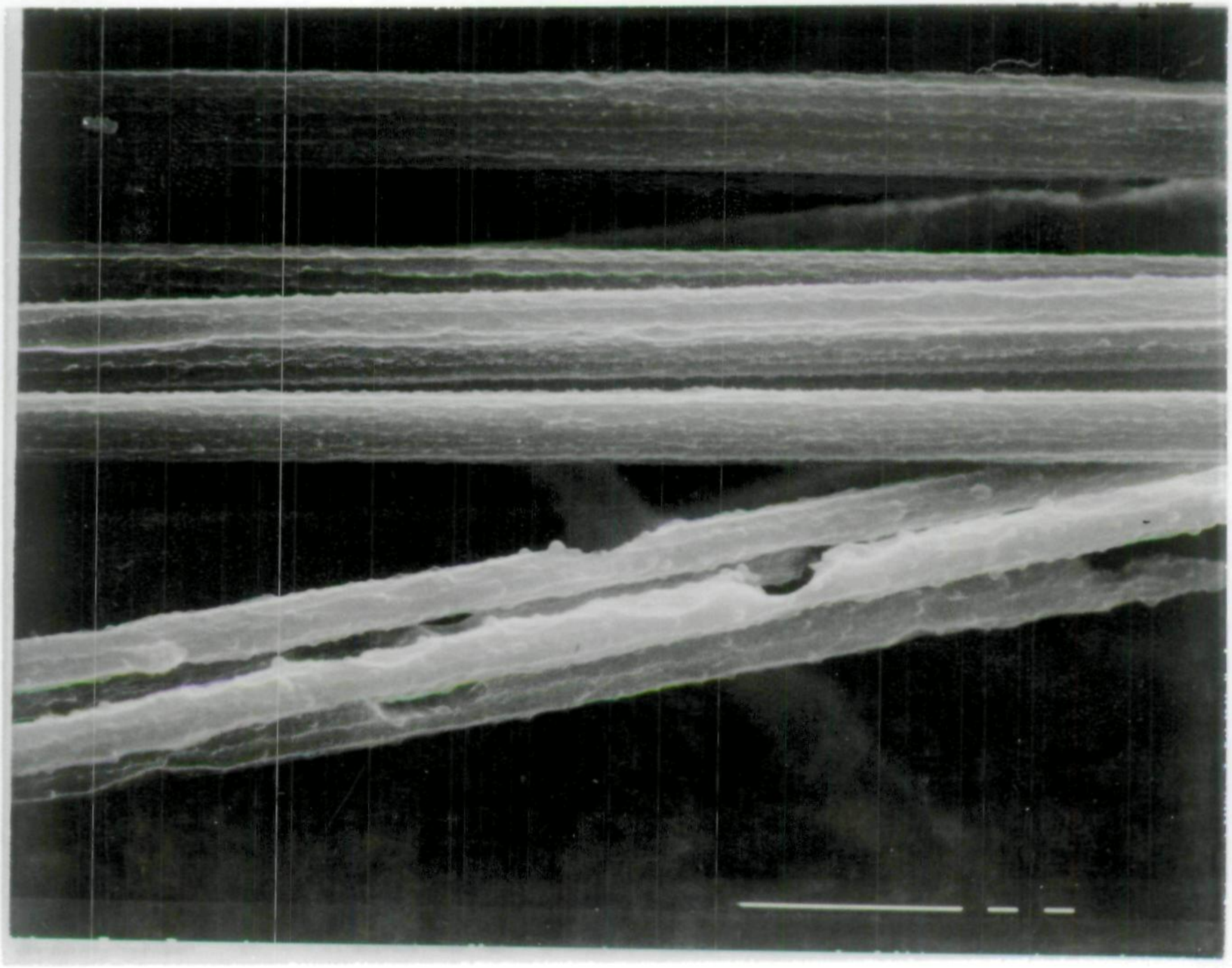

Fig. 4-24(b) Silicon Carbide Coating from UCC L-31 Silicone Fluid 1350X

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Fig. 4-24. SEM Micrograph of Silicon Carbide Coated Carbon Fibers After 30 Seconds Static Burn at 1000°C.

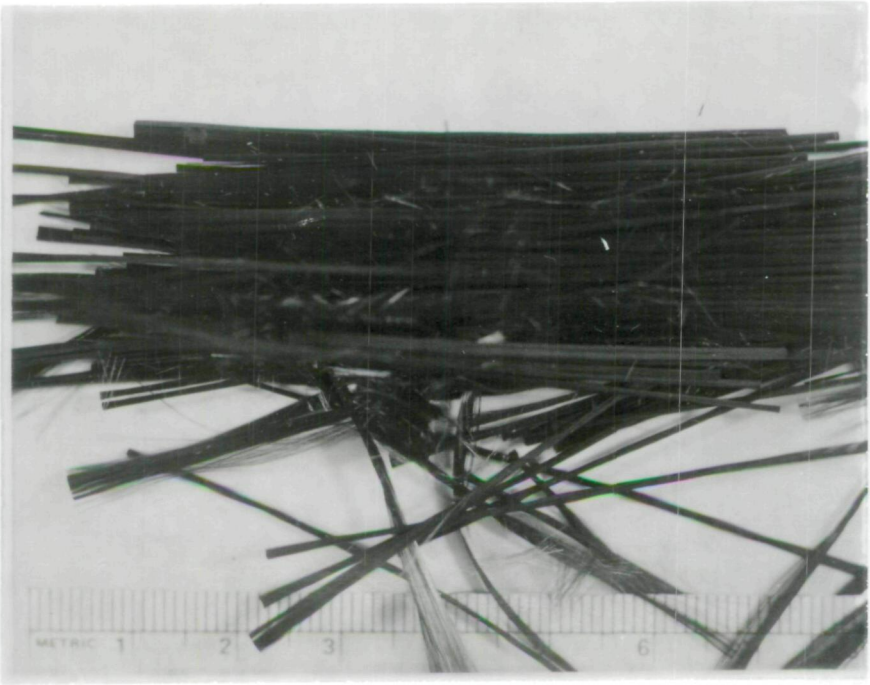
METRIC 1 2 3 6

Fig. 4-25(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 11 M/sec. Air Velocity. Scale is Centimeters.

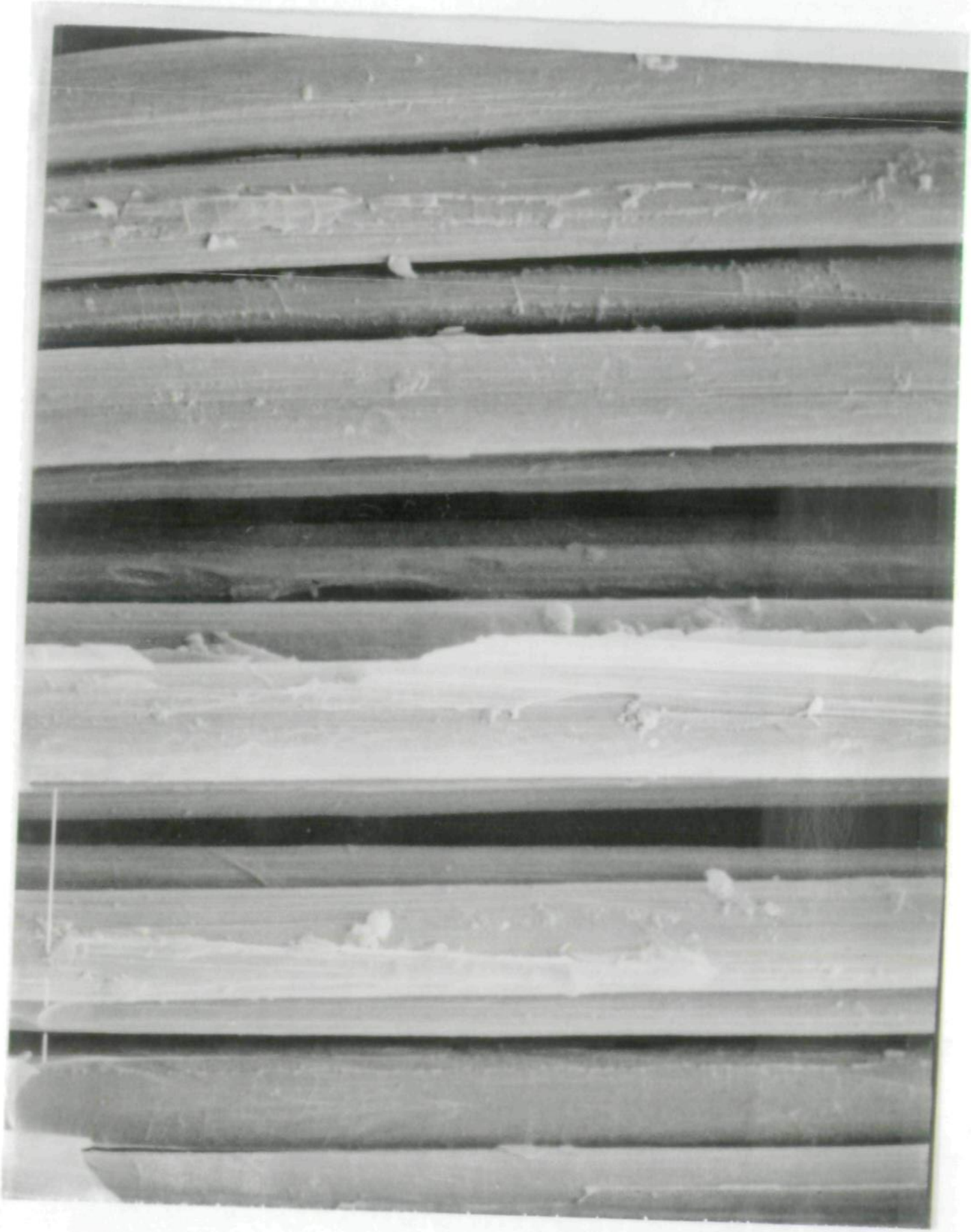

Fig. 4-24(b) SEM Micrograph from One of the Larger Pieces Remaining After Burn Testing. 1350X

POLAROID L8443238791 2A

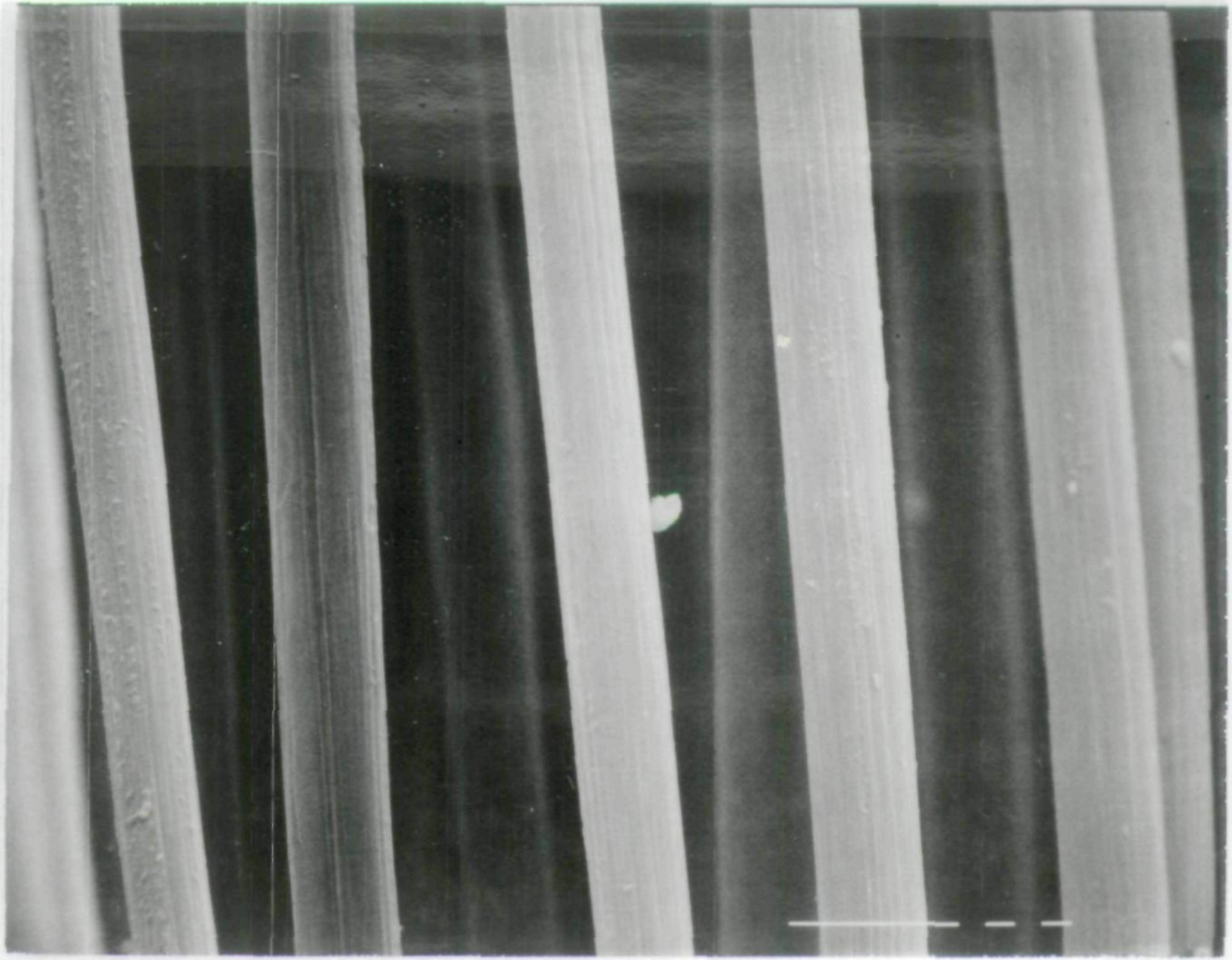

Fig. 4-25(c) SEM Micrograph from Fuzzy Edge of a Smaller Piece Remaining After Burn Testing.

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POLAROID

L8443208/912A

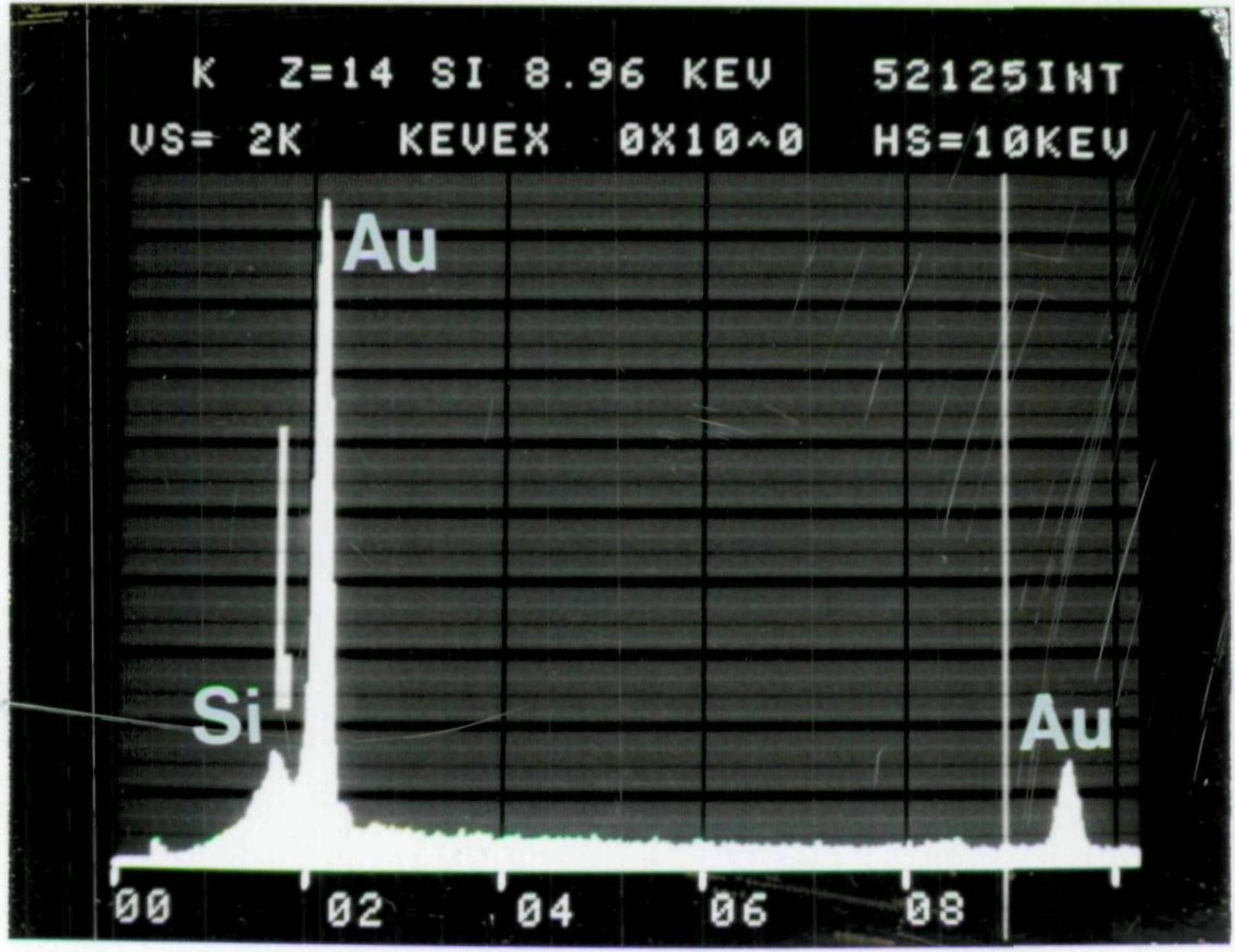
K Z=14 SI 8.96 KEV 52125INT
VS= 2K KEVEX 0X10^0 HS=10KEV
Au
Si
Au
00
02
04
06
08

Fig. 4-25(d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon Levels Similar to Control Samples.

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Fig. 4-25. Silicon Carbide, from UCC L-45 Silicone Fluid, Coated Fiber Composite After Dynamic Burn Test

Fig. 4-25(d)

METRIC 1 2 3 4 5 6 7 8 9

Fig. 4-26(a) Composite After 10 Minutes Dynamic Burn Test, 850°C Max., 1.5×10^6 dyne·cm Impact Energy, 11 M/sec. Air Velocity. Approximately 50% of Fibers Burned Away. Scale is Centimeters.

Fig. 4-26(a)

POLAROID

Fig. 4-26(b) SEM Micrograph from One of the Larger Pieces Remaining After Burn Testing.

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Fig. 4-26(b)

POLAROID

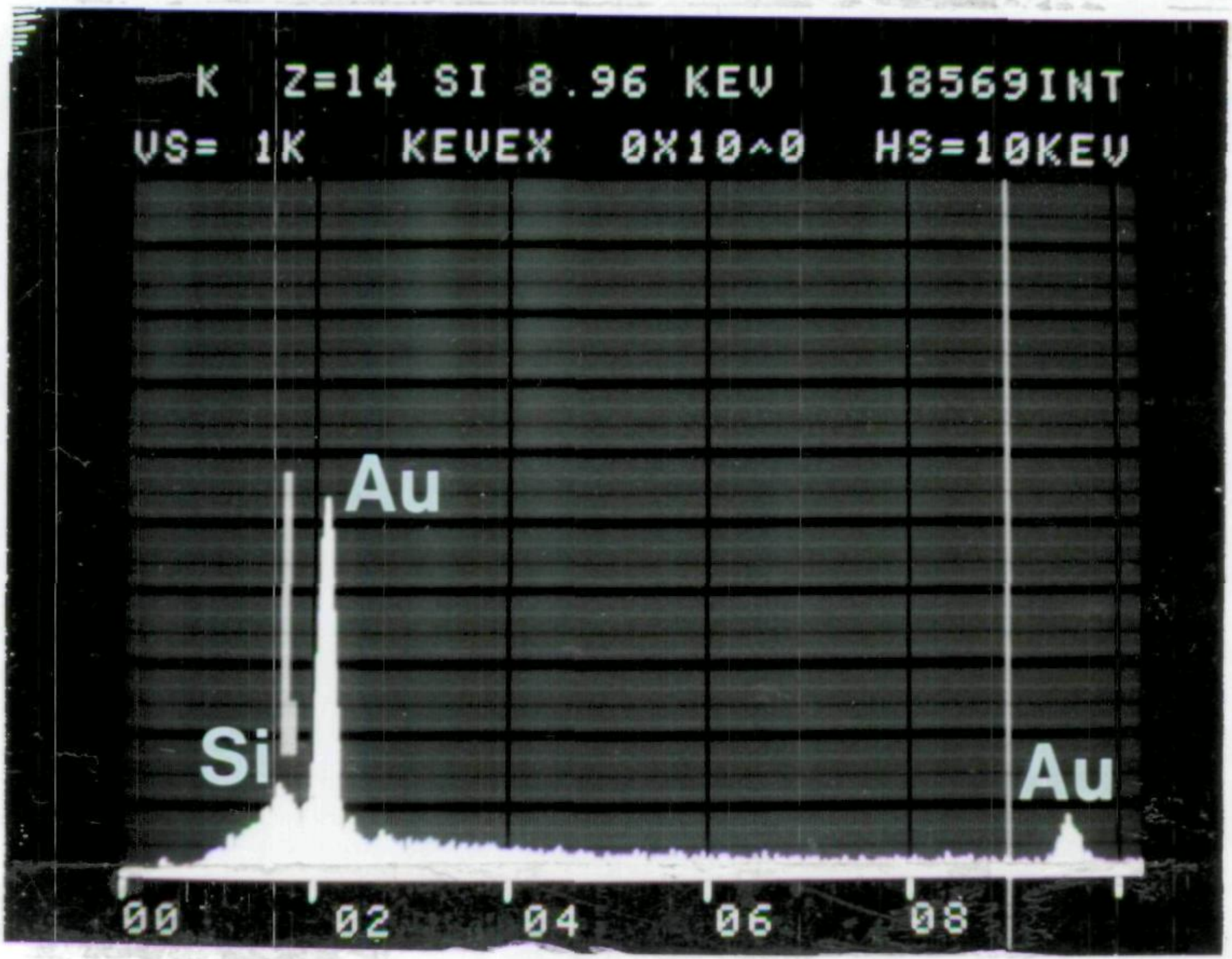
K Z=14 SI 8.96 KEV 18569INT
VS= 1K KEVEX 0X10^0 HS=10KEV
Au
Si
Au
00
02
04
06
08

Fig. 4-26(d) Kevex Trace of Burned Composite Showing Gold Coating and Silicon Levels Similar to Control Samples.

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Fig. 4-26. Silicon Carbide, from L-31 Silicone Fluid, Coated Fiber Composite After Dynamic Burn Test.

Fig. 4-26(d)

Fig. 4-26(c) SEM Micrograph from Edge of a Sample Piece Remaining After Burn Testing. 1350X

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Fig. 4-26(c)

POLAROID

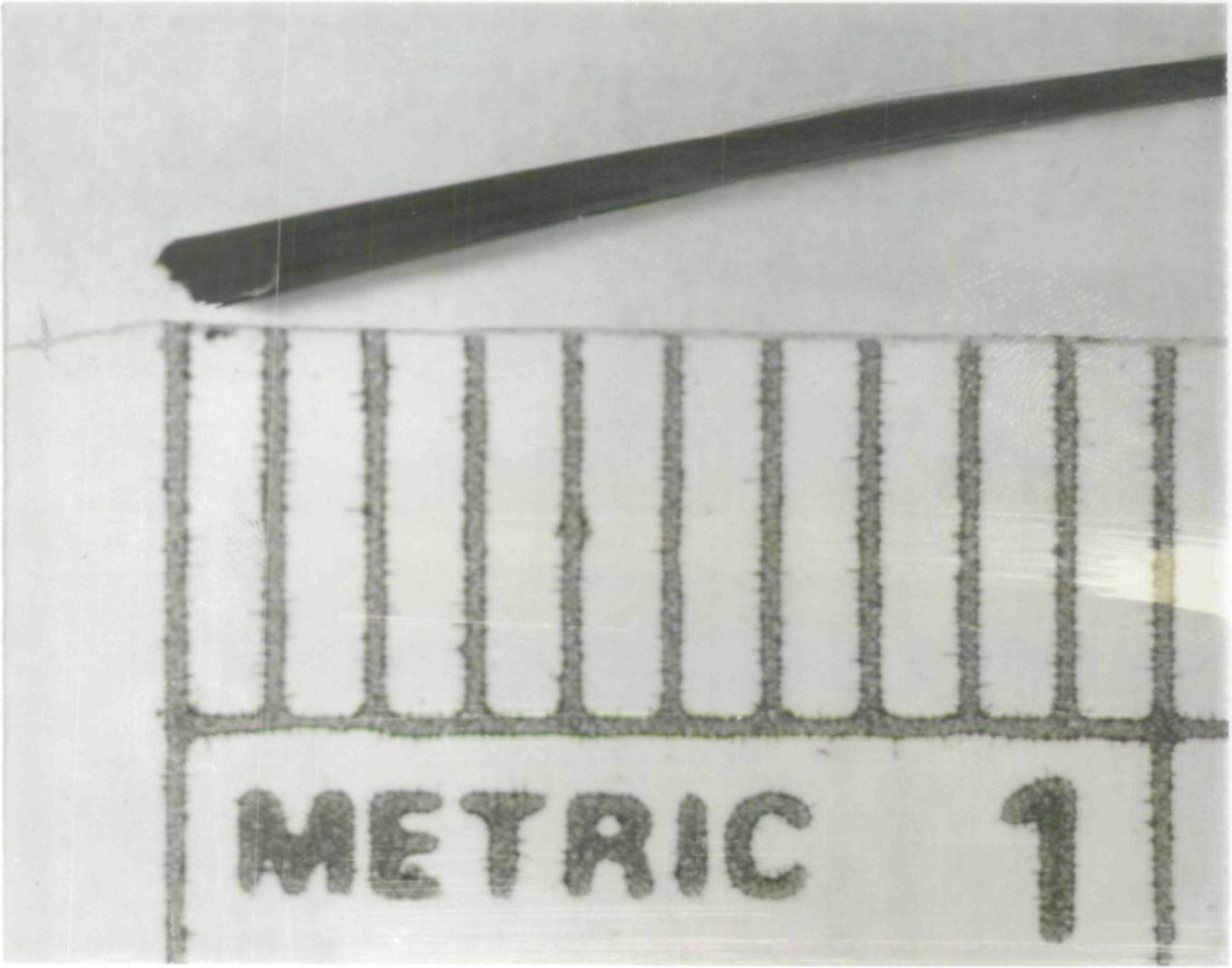
METRIC 1

Fig. 4-27. "Thornel" 300 Carbon Fiber with UC-309 Size Showing Bundle Integrity. Scale is Centimeters.

POLAROID

L84622C87924C

Fig. 4-27