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DEVELOPMENT OF HIGH STRENGTH, HIGH MODULUS FIBERS

R.N. Fetterolf

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August 1970

Air Force Materials Laboratory
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

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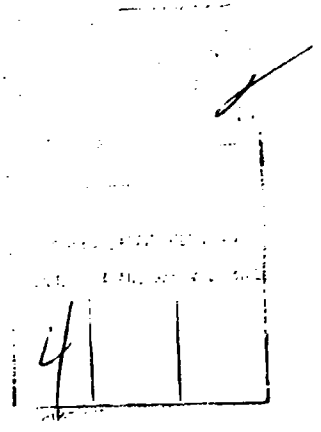

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DEVELOPMENT OF
HIGH STRENGTH, HIGH MODULUS FIBERS,

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FOREWORD

This report was prepared by The Babcock & Wilcox Company, Research Center, Alliance, Ohio, 44601, under United States Air Force Contract No. F 33615-69-C-1235, Project No. 7320. This contract is administered by the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, 45433, with Mr. L.G. Picklesimer, (MANF) as Project Engineer.

The author wishes to express his gratitude to the many people who aided in this research effort: J.A. Kaurich, J.A. Maurer, and R.A. Mordew for the production and testing of fibers and G.K. Dray for performing the X-ray diffraction analyses and aiding in their interpretation. Mr. L.W. Sarver's extensive electron microscopic analyses proved of great aid, and a special thanks is due him.

Finally the author wishes to thank Mr. R.C. Young and Dr. A.V. Illyn for their helpful discussions and suggestions in developing program directions.

The contractor's report number is 7953.

This report has been reviewed and is approved.

Jack H. Ross
Chief, Fibrous Materials Branch
Nonmetallic Materials Division
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ABSTRACT

A low cost process for manufacturing polycrystalline alumina based fibers has been developed. Recently, this process was made capable of producing uniform diameter fibers. The objective of this program was to produce fibers using this process with tensile strengths greater than 400,000 psi and tensile moduli of 60 x 10^6 psi.

This process consists of (1) preparation of extrusible concentrated metal salt solutions, (2) extrusion of the solution to form less than 10 micron diameter "raw" fiber, and (3) drying and thermal conversion of the "raw" fiber to polycrystalline oxide fibers.

The properties of the fibers depend on their crystalline composition and microstructure. Since the process affects microstructure, the influence of both process and composition on the tensile strength and modulus of the fibers was emphasized in this work. The research effort consisted of the following two phases:

1. Evaluation of drying and firing on the properties and microstructure of a mullite composition fiber
2. Evaluation of properties of mullite through pure alumina composition fibers when dried and fired by the "best" method.

In the first phase, the alumina-silica ratios ranged from 3 to 4:1 with additive levels of B_2O_3 up to 10%. Analysis by transmission and scanning electron microscopy, and X-ray diffraction showed that porosity and grain growth were the microstructural factors influencing tensile strength and modulus. The porosity was found to be built into the fibers as a consequence of the process. The B_2O_3 component was found to inhibit grain growth, promote crystallization and minimize the pore size and number distribution change with firing temperature, thereby providing stronger fibers over a wider firing range.

Modulus of elasticity was found to depend primarily on the amount and composition of the crystalline component of the fibers.

The maximum fiber properties were attained in the second phase of this program. Tensile strengths from 225,000 to 250,000 psi and moduli of elasticity from 25 to 28 x 10^6 psi were exhibited routinely in 85% Al_2O_3, 10% SiO_2, 5% B_2O_3 fibers. Pure alumina fibers exhibited 130,000 psi strength and 14 x 10^6 psi moduli values. These fibers were gamma alumina. When heated to produce alpha alumina, they were weak and powdery. Microstructural analysis indicated

that excessive grain growth and porosity probably resulted from the phase transformation, and were the factors limiting the tensile strength of the alpha alumina fibers.

The conclusion reached is that the present process is limited to the production of polycrystalline fibers with tensile strengths of 300,000 psi and moduli of elasticity of 30 x 10^6 psi. Advances beyond this require process changes to control the porosity, grain growth and phase transformations of 95 to 100% alpha alumina fibers. They are required to attain the 60 x 10^6 psi goal for modulus.

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SECTION I
INTRODUCTION

This report summarizes the results of a program aimed at the development of a low cost, strong, high modulus fiber for use as the reinforcement component in composite materials. The "salt decomposition" process used throughout this program and to be described in later sections, has been demonstrated in earlier work to produce low cost polycrystalline oxide fibers[1]. The main task covered in this report has been to use this process to produce fibers with high tensile strength and high Young's modulus of elasticity, "E".

The objective of the program was to develop a low-cost, strong, high-modulus fiber based on aluminum oxide. The desired form is a continuous-length, uniform-diameter fiber. The target properties were:

1. Tensile strength of 400,000 psi.
2. Tensile modulus of 60×10^6 psi.

The fibers studied in this work are based on the alumina-mullite system. The lowest value of true "E" in this system is 30×10^6 psi (for dense mullite) and the highest, 63×10^6 psi (for single crystal alumina).

The technical background and approach used to attain the objective are presented in the following section.

SECTION II
TECHNICAL BACKGROUND AND APPROACH

The Babcock & Wilcox Company has developed a salt decomposition process for making fibers composed of high alumina content crystalline species. The process, which has also been studied by B&W and under several Government contracts, can be divided into the following five operations, each of which has a distinct effect on the final fiber product:

1. Solution preparation.
2. Fiberization.
3. Fiber collection.
4. Fiber drying.
5. Fiber calcination.

A general method of transforming common inorganic salts into highly concentrated inorganic salt solutions has been developed[1] [2]. The physical properties of these viscous salt solutions permit their fiberization. Heat treatment of these fibers produces crystalline ceramic oxide fibers.

The solution preparation techniques have been shown to be applicable to a wide range of compositions[1] [3]. The techniques can be applied to many inorganic salts either alone or in combination. Thus, it is possible to tailor-make solutions of a specific composition to lead to calcined fibers of a desired composition. The fibers studied in this work have been 73 to 100% in alumina content. That is, pure alumina to mullite composition fibers were studied.

Attainment of the contract fiber properties would be meaningless if the fiber were not in a usable form. Integration of the fibers into a composite matrix and translation of the measured fiber properties to that composite must be achieved. This largely depends on the fiber form, which wholly depends on the fiberization technique and collection method employed. A continuous fiber or a bundle of highly oriented fibers is desirable. The fiber should be uniform in diameter along the length of fiber.

A method of fiberization capable of producing uniform-diameter fibers has been developed. This is the extrusion-attenuation method initially detailed under a NASA contract[4] aimed at the production of a continuous ceramic oxide fiber.

Fiber formation based on extrusion of the solutions through small-diameter orifices, has been the technique employed in this program. The solution is pressure extruded through an orifice and collected on a rotary drum. The extruded solution dries in the distance from the orifice to the collector drum and is thus converted from a viscous inorganic salt solution to an inorganic salt fiber. This fiber is picked up by a rotating collection drum, and a pulling or attenuating force is applied to the fiber. That is, the collection rate is faster than the rate of solution extrusion.

The extrusion produces a continuous-length, uniform-diameter fiber and the rotating drum sets the fiber diameter according to its speed and continuously collects the fiber. Collection drums 2 feet and 6 feet in circumference have been used. The drums are driven by variable speed motors, and a drum traversing mechanism has been integrated into the collection system. This extrusion and collection system is capable of producing uniform-diameter fibers which are continuous in the green or unfired state. Current production techniques limit the fired fiber form to highly oriented fiber bundles up to 4 feet in length. Development of a technique for producing continuous-length fired fibers was to be emphasized when fiber properties were high enough to justify the effort.

The salt fibers prepared from the solutions by extrusion and drying are converted to an oxide form by calcining them at temperatures in the range of 1700 to 2500 F. As a result of this calcination step, the salt fibers are converted to crystalline oxides with a tensile modulus higher than that of amorphous oxide filaments such as glass fibers. The "crystallinity" of a specific fiber composition is controlled by the calcination step. Thus, the value of Young's modulus, which depends more on the crystalline makeup of the fiber than on other considerations, is also controlled by the calcination step.

The tensile strength of the fiber, on the other hand, appears to depend more on the overall process. The starting chemistry, solution characteristics, drying conditions, atmosphere, and temperature during calcination all play a role in obtaining the maximum tensile strength possible in a specified system.

The fiberizable solution preparation and unfired fiber formation (extrusion-attenuation) operations had been well studied and brought under control during previous work. The approach taken for research under this contract was to first study and gain control of the fiber drying and firing operations. The measure of successful control of these operations would be the reproducibility

of fiber properties with a given fiber composition. Therefore, the first phase of the program was directed toward the definition of fiber drying and calcination effects on the tensile strength and modulus of elasticity of a constant fiber composition. Solutions were selected for fiberization so that the fired fiber would be 75% alumina, 20% silica, and 5% boric oxide in composition.

While fiber properties in the range of the goals of this contract had never been attained in fibers of this composition, much fiber property data had been generated in past work[4]. Routine attainment of the best properties periodically achieved in the past in fibers of this composition (200,000 psi tensile strength, 20 x 10^6 psi modulus of elasticity) would indicate that control of the fiber drying and firing operations had been achieved.

In addition to the control of the drying and firing operations, an understanding of the effects of drying and firing variables on the physical and chemical properties of the fibers was desired. While fiber strength and stiffness are of primary interest, their measurements cannot indicate why specific fibers have specific strengths. These measurements can only indicate some other physical characteristics built into the fibers during drying and firing. Improvements in fiber strength and stiffness can only result from changes in these physical characteristics. The determination of the physical or chemical characteristics of the fibers affecting fiber strength and stiffness and the dependence of these characteristics on drying and firing have thus been a research goal necessary to the ultimate attainment of increased fiber properties.

Our first research goal was to gain control and an understanding of the fiber drying and firing operations as they affect the properties of a 75% alumina, 20% silica, and 5% boric oxide composition fiber. Once the fiber drying and firing operations were controlled and understood, the search for a fiber composition having a higher modulus of elasticity could proceed with confidence that fiber properties would be reproducible and that knowledge generated in the first research phase would aid in the attainment of the desired fiber properties for the compositions studied.

Therefore the second research goal was to apply the developed fiber drying and firing knowledge to fiber compositions with alumina content increasing from the 73% (remainder boric oxide and/or silica) characteristic of pure mullite to 100% Al_2O_3. The objective was to obtain a crystalline fiber component of inherently higher modulus of elasticity than mullite. Pure α-alumina was the target since its single crystal modulus of elasticity is around 60 x 10^6 psi.

SECTION III
EXPERIMENTAL PROCEDURES AND TESTS

A. FIBERIZABLE SOLUTION PREPARATION

The success of the B&W method of making fibers from a salt solution depends on the physical and chemical properties built into that solution during preparation. The requirements for a successful fiberizable salt solution are:

1. Low surface tension.
2. High viscosity.
3. High equivalent oxide content.
4. Chemical and physical stability.
5. Solvent volatility at room temperatures.
6. Salts decomposable by heat to a solid oxide form.

The fiberizable salt system used by B&W to obtain these desired properties consists of acid-deficient inorganic salts. These salts can be readily made from inexpensive raw materials using standard chemical techniques.

The method used to make the fiberizable salt solution for this work starts with either concentrated inorganic acids or an inorganic acid salt solution or a mixture of both. Aluminum metal is added to either of these acidic solutions. Heating the mix initiates a rapid exothermic reaction in which aluminum is dissolved while hydrogen gas is liberated according to the equation:

$$2Al + 6H_3O^+ \rightarrow 2Al^{3+} + 3H_2 \uparrow + 6H_2O$$

The reaction is allowed to continue until the rate of aluminum dissolution is very slow. The undissolved aluminum is then removed from the solution by filtration. Additives such as colloidal silica, boric acid, and phosphoric acid can be mixed into the aluminum salt solution, as desired, to modify the properties of the finished fiber.

The solution is then further heated to evaporate excess water and to obtain the desired solution viscosity and equivalent solids content. A viscosity in the range of 300 to 3000 poise has been found desirable. At this point the complex salt solution is clear and colorless, and a final addition of acetic acid is made to stabilize the solution with respect to viscosity and time.

B. FIBERIZATION

The fiberizable salt solution has been transformed into monofilaments using the pressure extrusion apparatus shown in Figure 1. In operation, the pressure

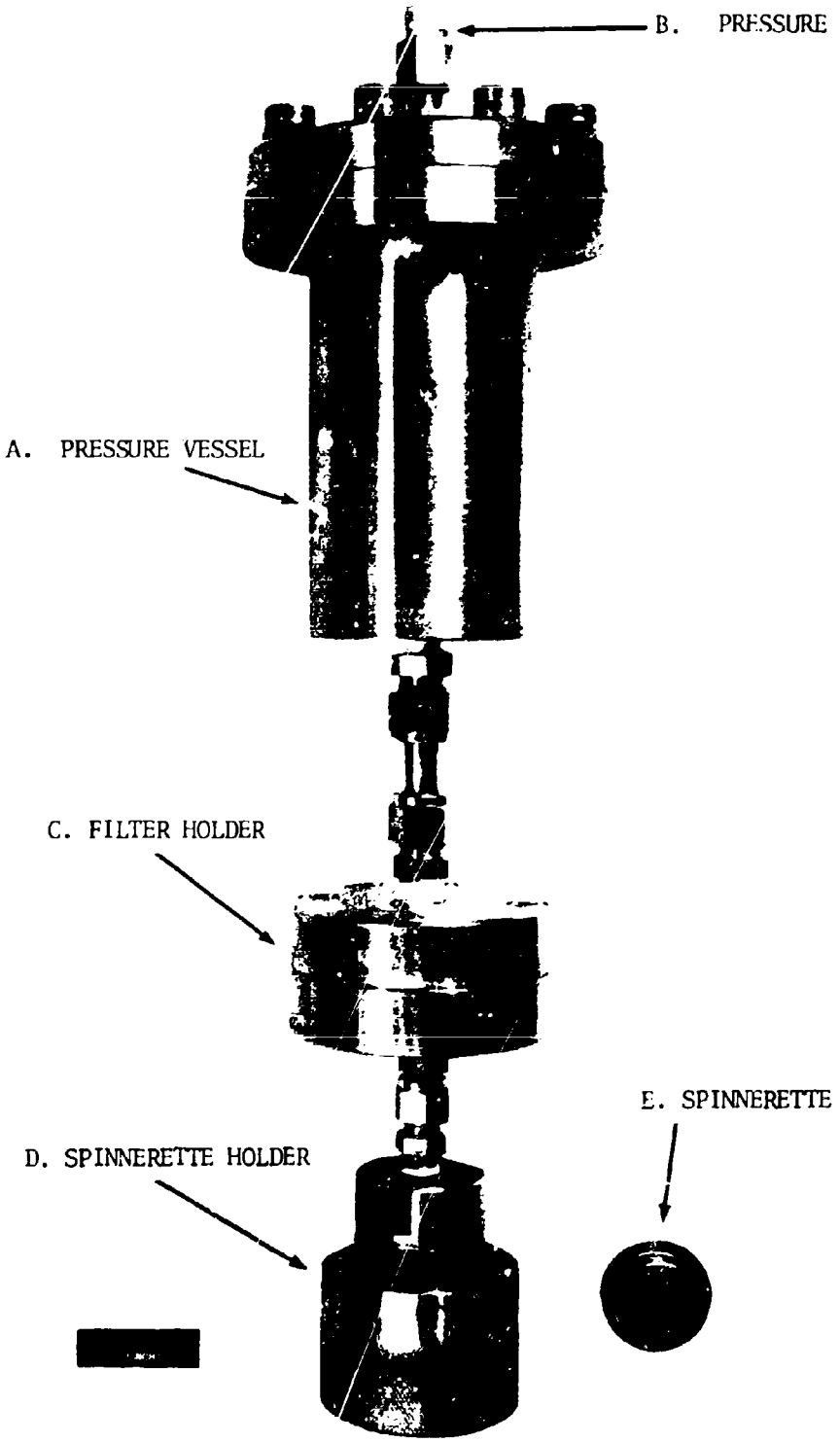


FIGURE 1 STAINLESS STEEL PRESSURE EXTRUSION SYSTEM USED FOR PRODUCING MONOFILAMENT FIBER

vessel (A) is filled with fiberizable solution, and pressure is applied to the system through the fitting (B). The pressure can be varied between 0 and 500 psig using compressed nitrogen gas as the pressure source. The fiberizable solution is pushed through an in-line high-pressure filter holder (C). The high-pressure filter holder is a product of Millipore Corp., and Millipore Mitex (Teflon) filters having pore sizes of 5 and 10 microns have been successfully used in this application. After the filtration operation, the solution proceeds to the spinnerette holder (D), where it is pressure extruded through a spinnerette (E). The spinnerettes were manufactured by Englehard Industries of Carteret, New Jersey. Through use of the in-line filter to remove foreign particles from the fiberizable solutions, orifice sizes of 0.002" to 0.004" in diameter have been successfully used for the production of pressure extruded fibers. All components of this system have been constructed of stainless steel to resist the corrosive nature of the viscous salt solutions.

The "green" or raw fiber produced as described above is allowed to fall by gravity to a collector below the nozzle. The fiber falls 4 feet before being picked up on the drum. A drum 2 or 6 feet in circumference is rotated by a precision speed control motor to further attenuate and collect the extruded fiber. The relatively large drum and vibration-free winding motor have been incorporated into the system to allow faster fiber collection rates at reduced collection drum speed and concurrent reduced air turbulence to avoid mechanical breakage of the fiber.

C. CALCINATION

The collected fibers must then be calcined to the desired oxide form. The collected fibers are removed from the collector drum and either stored under selected conditions of humidity and temperature or immediately placed in an electrical resistance furnace preheated to a desired temperature. The furnace and fibers are then further heated at a selected rate to a maximum temperature of 1750 to 2500 F. The furnace is held at the desired maximum temperature for 15 to 60 minutes. The furnace is gradually cooled to about 1000 F before the calcined fiber is removed from the furnace and stored in plastic boxes to await testing and evaluation. The fibers, placed on a mat or blanket of bulk insulation fiber (Kaowool; e.g.) for the entire calcination, can also be covered with a layer (about 0.5 inch) of bulk fiber to protect them from convection turbulence.

In the second method, the fibers are fired rapidly in a gas fired tunnel kiln. The tunnel kiln, designed by Fostoria Corp. and B&W, is 20 feet long and can calcine the fiber to 1850 to 2100 F in 15 to 140 minutes depending on furnace settings and belt speed. This tunnel kiln has been operated with a normal combustion atmosphere and an introduced steam atmosphere.

D. TESTING PROCEDURES

1. Viscosity Measurements

Two testers are used for the measurement of viscosity. The first apparatus is a hand-held Rion Viscotester, distributed by Fisher Chemical Company. This viscometer is a battery-powered, direct-reading instrument which uses various spindles to cover the range of viscosity desired. The Viscotester is used principally to check hot solutions and mixes whose corrosive vapors may damage the more delicate but more accurate Brookfield viscometer, the second apparatus used to determine viscosity.

The Brookfield, Model HAT, viscometer having eight speeds and seven spindles is used principally to check the viscosity of the fiberizable solutions before fiberizing and to calibrate the Rion Viscotester.

2. Fiber Diameter

Optical methods determine the diameter of the fiber. The apparatus, a Vickers A.E.I. split-image eyepiece mounted on a Leitz light microscope, is used to observe the fiber at 675x magnification. This permits a precision of ±0.1 micron.

3. Tensile Strength

A 10,000-pound capacity Instron Universal Testing Machine, Model TT-C, equipped with tension load cell "A" (0-50 grams) is used to measure tensile strength. Fibers 1 inch long are mounted on specially made metal pins using a thermoplastic glue. The crosshead speed is 0.050"/min with a chart speed of 20"/min. The full-scale deflection across the strip chart recorder is set to either 2 or 10 grams. The breaking load is then read directly from the chart. The diameter of the fiber is determined as previously described at five equidistant positions along the one inch fiber test length. The tensile strength of the fiber is calculated as follows using the average of the five readings as the fiber diameter.

$$TS = \frac{1.811 \times 10^6 \times g}{d^2}$$

where

TS = tensile strength in psi

g = load in grams

d = fiber diameter in microns (avg. of five measurements over the 1-inch gage length)

4. Modulus of Elasticity

The tensile modulus of elasticity (E) is determined on the Instron Model TT-C tester using the stress-strain curve generated in the tensile strength tests. The modulus is calculated from the load-elongation trace as the initial modulus. The indicated elongation is corrected by subtracting the Instron compliance so that the strain over the fiber gage length can be computed. In most tests, the load elongation trace is straight over its length up to fracture. In the cases where a slight curvature is noted, only the straight portion, usually 80% of the length, is used to compute the elongation.

5. Fiber Composition

The composition of the calcined fibers is calculated using the starting materials as the basis for the calculations. The amount of aluminum metal digested is measured. Since the purity of the metal used is 99.7+% Al, 1.88 g of Al_2O_3 is produced for each gram of Al dissolved. The colloidal silica used is a 40% sol. Thus, for each gram of sol used, 0.4 g of SiO_2 is added to the batch. The boric acid is used as the source of B_2O_3, and each gram of acid equals 0.563 gram of B_2O_3. Thus, knowing the total equivalent oxide content of the solution, the percentage of each oxide in the fiber can then be calculated on a weight basis.

6. Electron Microscope Examination of Ceramic Fibers

The general procedure is to examine etched and unetched fractured transverse (or semilongitudinal when available) sections of ceramic fibers. For transmission electron microscopy this is done by replication. It is done by direct examination in the scanning electron microscope.

Bundles of fibers are supported by embedding them in plastic rods. This preparation is essential for the replication required in transmission electron microscopy.

Surfaces to be examined are prepared by simply bending the composite ceramic fiber and plastic rods to induce fracture. In this way hundreds of fibers are set up for ready replication.

Replication consists of making an impression of the fracture surface in a thin sheet of plastic material. For this, the fractography replicating kit marketed by Ladd Research Industries is used. Several replicas are stripped to clean the surface of debris and pulled-out fibers. Replicas are shadowed with palladium (30-45^{o}) followed by carbon deposition at 90^{o}. The heavy plastic is dissolved by acetone washing, which results in finished carbon replicas ready for examination.

Preparation for scanning electron microscopy involves only the metallizing of specimens to provide conducting surfaces. This is done by high-angle palladium shadowing while specimens are rotated in the vacuum evaporator.

Transmission microscopy is done on a JEM-6A instrument, operating with high-contrast parameters - 50 kv and a small condensor aperture. Scanning microscopy is done on a JSM-II instrument.

Specimens are examined in the "as fractured" and etched conditions. Etching consists of immersion in a 20% aqueous solution of 52% HF for 1-1/2 hours and 5 to 6-1/2 hours. Some SEM specimens are etched 24 hours in 10% HF (52%) and H_2O. Natural circumferential surfaces are also examined in the scanning electron microscope.

Fiber porosity is quantified in terms of pore density (pores per square micron) and average size. Density is established by counting the number of pores, shown on photomicrographs, within an area of 4 square microns (2 square microns for fibers with high pore density). Of course, values are scaled by a factor of 30,000x magnification. Values counted are reduced to pores per square micron. Pore size is determined by measurement of selected examples on photomicrographs. Crystallite size is established in a similar fashion.

7. DTA and dTGA

Two different thermal analyzers are used in this work. Both were built by B&W. The first DTA apparatus was modeled after Skinner's apparatus[5] and is capable of reaching temperatures up to 2400 F. The test samples are placed in the container and then heated at a uniform rate of 13.5 or 27 F/min to 2000 F or 2200 F.

The second DTA apparatus uses a Fisher sample holder and a Marshall (Model 2231) furnace capable of temperatures up to 2200 F. The holder is made from fused silica and can hold 100 grams of sample. Heating rates up to 80 F/min can be used, but generally 13.5 or 27 F/min is appropriate.

The thermogravimetric analysis apparatus, also built by B&W, has Leeds and Northrup instrumentation and a platinum wound furnace capable of temperatures to 2700 F. The weight change was obtained using an electronic recording balance. Weight changes of 0.1 mg can be detected in 300 mg samples. Heating rates up to 27 F/min can also be attained with this apparatus.

8. X-ray Analysis

X-ray diffraction patterns are obtained using a General Electric XRD-5 motor driven diffractometer synchronized with a Leeds and Northrup Speedomax Type "G" recorder. The calcined fibers are finely ground in an agate mortar and then packed into a flat aluminum holder. Either cobalt or iron radiation is used, and the sample is scanned at a rate of 2 degrees per minute.

The X-rays are also used to determine crystallite size. Each of the main diffraction lines is scanned at a rate of 0.2 degree/min to measure the breadth of the diffraction line at half peak height. This broadening is compared with the broadening of a standard quartz sample to correct for the broadening due to the instrument.

The average crystallite size is then calculated using the following Klug & Alexander formula[6]:

$$DhKl = \frac{51.57\gamma}{B_1 \cos\theta}$$

where

DhKl = diameter of the crystal

B_1 = broadening due to experimental method employed

51.57 = conversion factor

γ = X-ray wavelength

θ = Bragg angle

SECTION IV

RESULTS AND DISCUSSION

The various unit operations of the salt decomposition fiber making process have been well studied and defined up to the fiber drying step. The conversion of the salt fiber to a polycrystalline ceramic oxide fiber through drying and firing are the following process operations and next in line for study and definition. Research under this contract was thus divided into two phases. First, fiber drying and firing was studied and brought to a degree of control such that reproducible fired fiber properties could be obtained from identical composition unfired fibers. The second research phase was to apply the developed drying and firing process knowledge to various higher alumina content fibers in search of a crystalline composition higher in modulus of elasticity.

The first effort was thus to define the drying and calcination operations and their import on the properties of a constant 75% alumina, 20% silica, and 5% boric oxide composition fiber.

A. STUDY OF DRYING-FIRING OPERATIONS WITH A CONSTANT FIBER COMPOSITION

1. Fiber Firing Rate vs Fiber Properties

Previous work had shown that firing rate and temperature are of prime importance to fiber properties. A broad study of firing rates was made as the first series of experiments.

Fiber extrusion and fiber drying during extrusion and collection were maintained constant. Individual fiber samples were extruded and collected for 1 hour. All reported samples contained about 45% solids when they were stripped from the collection drum. The percentage of solids is defined as the percentage of ash remaining after a 1400 F firing. On this basis the solutions extruded contained the equivalent of 34% solids.

Extruded fibers were stripped from the collection drum and stored at 80 F and 30% relative humidity for 0 to 24 hours prior to firing.

Firing temperatures of 1850 to 2000 F were investigated at firing rates of 25 to 8000 F/hr. The slow and intermediate rates were accomplished in electric kilns, and 30-minute soak times were standard. The faster rates were accomplished in a gas fired tunnel kiln using various belt speeds for fiber transport through the kiln. Soak time in the tunnel kiln depended on belt speed and ranged from about 3.5 minutes (5200 F/hr) to about 12 minutes (2600 F/hr).

Tables I, II, and III show many fiber samples fired to various temperatures at various rates. Fibers with tensile strengths up to 200,000 psi and moduli of elasticity of 20 x 10^6 psi are evident in these tables where relatively short firing times (15 min to 1.5 hours) were employed to take the fibers from room temperature up to temperatures of 1850 to 2000 F. Reproducibility of fiber properties under identical firing conditions is not evident in the tables. However, it can be seen that fiber samples obtained by cutting one large sample into several identical samples, each of which was fired in a different manner, had similar tensile strengths and moduli of elasticity. Samples B-299-2B, 4B, and B-299-4A (Table II) and the B-299-6 to 10, A and A1 samples of Tables I and II illustrate this point well. The implication is that the fiber properties were set before firing was begun; that is, by the drying that occurred in the fiber extrusion and storage operations. The extrusion and storage and consequently drying of the samples in Tables I through III were maintained reasonably constant. However, the data indicate that more precise control is necessary.

Samples B-299-2B, 4B and B-299-2A, 4A (Table II) also indicate that a slow firing can produce fibers comparable in properties to fast fired fibers if the precalcination treatment (fiber drying) is the same. Thus, the only really poor samples appear to be those fired at intermediate rates.

The conclusion drawn from these data was that there is a temperature range in the firing operation where a slow rate is best and a temperature range where a fast rate is best. By firing slowly or rapidly through the entire range, good samples result. But by going neither slowly nor rapidly through any of the firing range, very poor samples result. Obtaining the best fiber properties would seem to depend on the identification of the firing temperature ranges where slow and fast firing rates are best. It was further concluded that fiber drying must be controlled and studied as thoroughly as fiber firing. The next set of experiments was therefore aimed at a more precise definition of fiber firing rates and a study of the effects of fiber drying on fiber properties.

Figures 2 and 3 show a typical dTGA and DTA of unfired, extruded fibers. With the aid of these data and the experiments described above, the fiber drying and calcination operations were broken down into four steps as follows:

1. Fiber room temperature drying (extrusion and storage).
2. Room temperature to 600 F.
3. 600 F to 1200 F.
4. 1200 F to firing temperature.

TABLE I

TENSILE STRENGTH AND MODULUS OF ELASTICITY OF 75% ALUMINA, 20% SILICA, 5% BORIC OXIDE COMPOSITION FIBERS FIRED AT VARIOUS RATES TO 1850 F

Sample No.	Firing Rate (F/hr)	Fiber Diam.* (avg. microns)	Tens. Str.* (avg. psi)	Modulus (E)* (avg. psi x 10^6)
B-305-8A	300-500 F/hr	All samples powdery, no tests could be made.		
B-305-9A	300-500 F/hr			
B-299-1A	∿2500 F/hr	5.9	137,000	20.0
B-299-3A	∿2500 F/hr	5.5	118,000	20.5
B-299-6A1	∿2500 F/hr	5.3	175,000	20.2
B-299-7A	∿2500 F/hr	5.4	147,000	20.9
B-299-8A	∿2500 F/hr	5.4	152,000	21.1
B-299-9A	∿2500 F/hr	5.0	152,000	20.3
B-299-10A1	∿2500 F/hr	5.9	169,000	19.7
B-299-11A	∿2500 F/hr	5.2	206,000	21.0
B-299-11C	∿2500 F/hr	5.5	202,000	20.6
B-299-12A	∿2500 F/hr	5.4	181,000	20.3
B-299-12C	∿2500 F/hr	5.7	199,000	19.6
B-299-12D	∿5000 F/hr	5.1	167,000	20.7
B-299-12B	∿5000 F/hr	5.5	167,000	20.6
B-299-11D	∿5000 F/hr	5.6	162,000	21.1
B-299-11B	∿5000 F/hr	5.5	174,000	20.0

* Individual fiber properties are shown in Table A.1.

TABLE II

TENSILE STRENGTH AND MODULUS OF ELASTICITY OF 75% ALUMINA, 20% SILICA, 5% BORIC OXIDE COMPOSITION FIBERS FIRED AT VARIOUS RATES TO 1950 F

Sample No.	Firing Rate (F/hr)	Fiber Diam.* (avg. microns)	Tens. Str.* (avg. psi)	Modulus (E)* (avg. psi x 10^6)
B-299-2B	25 F/hr	5.7	143,000	20.4
B-299-4B	25 F/hr	5.7	150,000	18.0
B-299-1B	300-500 F/hr	5.9	53,000	13.8
B-305-8B	300-500 F/hr	6.0	127,000	17.3
B-305-9B	300-500 F/hr	5.7	165,000	20.5
B-299-2A	∿2600 F/hr	5.8	145,000	20.5
B-299-4A	∿2600 F/hr	5.6	154,000	19.4
B-299-5A	∿2600 F/hr	5.6	163,000	20.2
B-299-6A	∿2600 F/hr	5.3	187,000	20.0
B-299-7A1	∿2600 F/hr	5.5	152,000	19.7
B-299-8A1	∿2600 F/hr	5.4	149,000	18.6
B-299-9A1	∿2600 F/hr	5.2	184,000	19.1
B-299-10A	∿2600 F/hr	6.4	161,000	20.0
B-305-1A	∿5200 F/hr	6.0	190,000	20.8
B-305-1B	∿5200 F/hr	5.6	144,000	21.6
B-305-3	∿5200 F/hr	5.9	188,000	20.6
B-305-4	∿7800 F/hr	5.8	202,000	20.8
B-305-2B	∿7800 F/hr	5.6	191,000	20.4
B-305-2A	∿7800 F/hr	5.5	162,000	21.0

* Individual fiber properties are shown in Table A.2.

TABLE III

TENSILE STRENGTH AND MODULUS OF ELASTICITY OF 75% ALUMINA 20% SILICA, 5% BORIC OXIDE COMPOSITION FIBERS FIRED AT VARIOUS RATES TO 2000 F

Sample No.	Firing Rate (F/hr)	Fiber Diam.* (avg. microns)	Tens. Str.* (avg. psi)	Modulus (E)* (avg. psi x 10^6)
B-305-16	~2700 F/hr	5.4	162,000	19.1
B-305-20	~5400 F/hr	5.3	162,000	21.2
B-305-19	~8100 F/hr	5.2	175,000	19.2
B-305-11	~8100 F/hr	5.5	173,000	21.3
B-305-7	~8100 F/hr	5.6	221,000	23.3

* Individual fiber properties are shown in Table A.3.

FIGURE 2. DIFFERENTIAL THERMOGRAVIMETRIC ANALYSIS OF UNFIRED EXTRUDED FIBERS

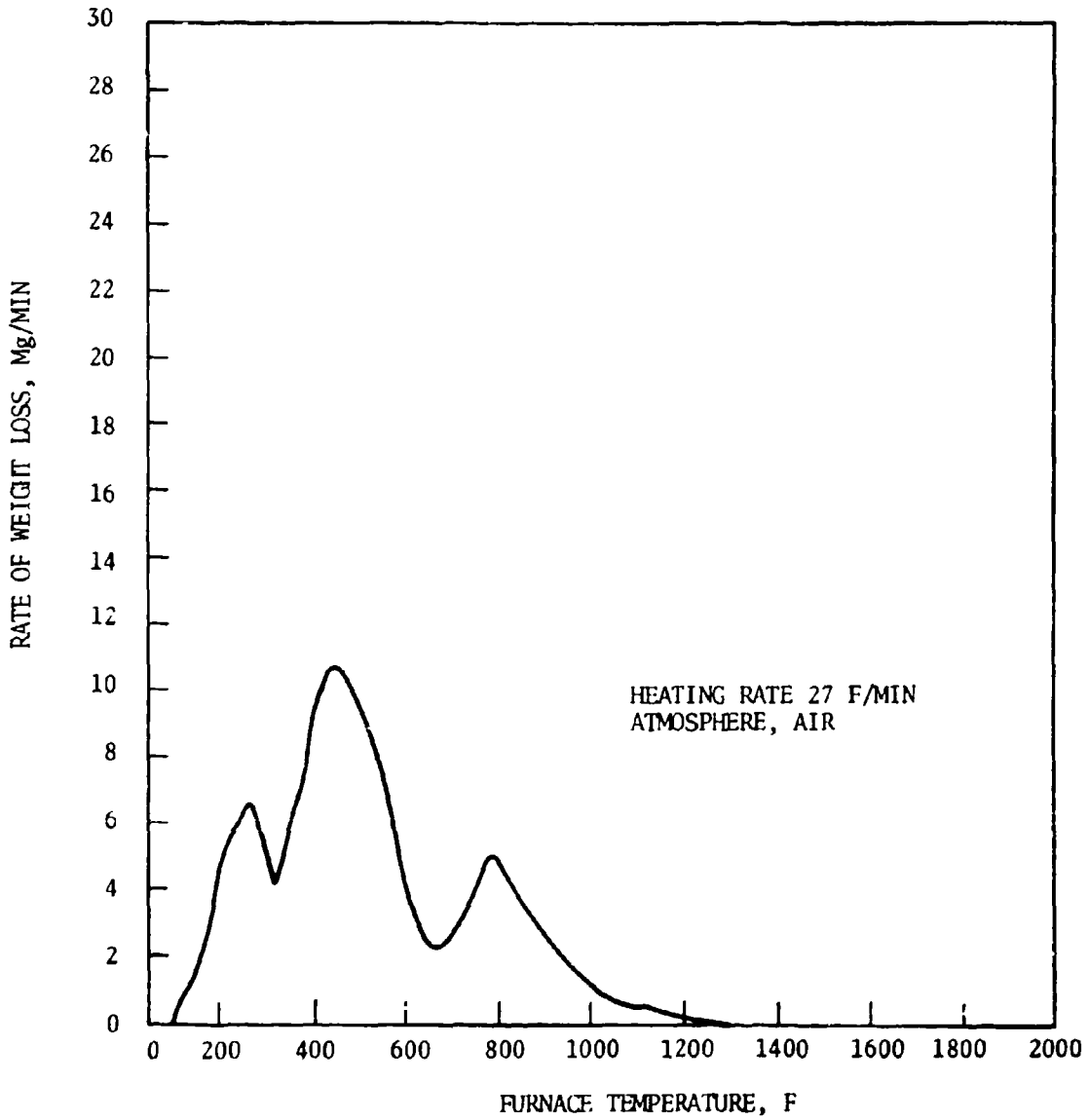
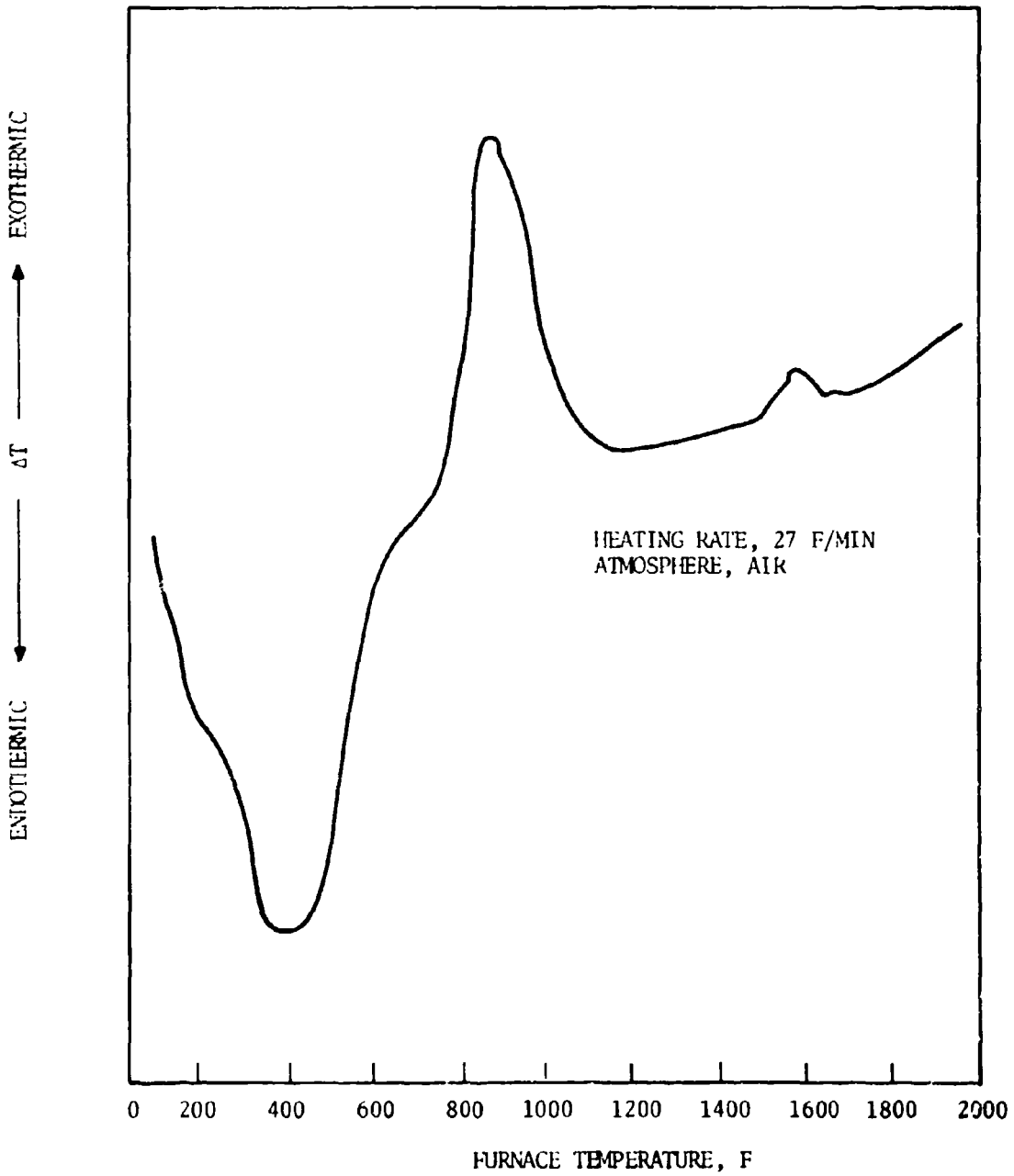


FIGURE 3. DIFFERENTIAL THERMAL ANALYSIS
OF UNFIRED EXTRUDED FIBERS



The important variables in fiber drying seem to be relative humidity and temperature for both extrusion and storage in addition to storage time. The relative humidity and temperature during fiber extrusion are part of the fiber formation operation and are set for optimum unfired fiber production. These values were thus left constant and were not studied with respect to fiber drying. However, fiber drying during storage could be studied by measuring unfired fiber weight change after storage for a given time at various relative humidities. Fiber firing from ambient temperatures to 600 F involves the driving off of all volatiles and the decomposition of the complex inorganic salts in the unfired fiber. The dTGA indicates two discrete reactions, but the DTA shows them blended into one large endothermic reaction.

The temperature range from 600 to 1200 F involves a weight loss according to the dTGA. However, this weight loss is not as simple as the driving off of volatiles. The dehydration of a hydrated alumina compound and the resultant transformation of an amorphous alumina hydrate to γ-alumina show up as an exothermic reaction on the DTA plot. Above 1200 F, no further weight loss is evident, and only a small exothermic reaction at 1670 F is indicated. Over this temperature range a sintering or densification probably occurs in addition to the transformation from γ-alumina to mullite.

The most important variables in the three firing ranges are firing rate, atmosphere, soak time, and ultimate firing temperature. The assignment of ranges to each of the variables in each of the drying-firing stages results in an experimental matrix of large proportions. The complete study of this matrix would be a long-term program. A review of the firing rate studies above and the dTGA and DTA results coupled with some short experiments brought the fiber drying-firing experimental matrix into manageable proportions.

Tables I through III show that fiber properties of 200,000 psi tensile strength and 20 x 10^6 psi modulus of elasticity were obtained using an air or combustion gas atmosphere throughout the firing. Therefore, study of the calcination atmosphere was deferred, and a constant air atmosphere was selected for all fiber drying-firing experiments.

Firing rates for the three firing ranges were selected with the aid of some experiments and judgment.

The tunnel kiln entrance is known to be at about 600 F. This is the first higher temperature the fiber encounters, and the increase from room temperature to 600 F is very rapid in a tunnel kiln firing. The dTGA-DTA work showed that

95% of the volatiles were removed from the fiber at about 1000 F. From about 600 to 1100 F, however, a phase transformation was also suspected. These two temperatures were thus convenient for a first attempt at determination of firing range vs firing rate. Several samples were placed in periodic kilns which had been preheated to 600 or 1000 F. The samples were then taken to 1950 F at 500 F/hr. The tabulation below summarizes these data:

Sample No.	Kiln Preheat Temp. (F)	Fiber Diam.* (avg. microns)	Tens. Str.* (avg. psi)	Modulus (E) (avg. psi x 10^6)
B-299-13	600	5.4	175,000	20.2
B-305-5	1000	5.8	89,000	16.1

* Individual fiber properties are shown in Table A.4.

A respectable sample was obtained from B-299-13 but that from B-303-5 was very poor. A fast firing rate thus appears best for the firing range from room temperature to 600 F, but a slower rate seems necessary for the phase-changing temperature range of 600 to 1200 F. Bearing in mind that a relatively fast overall firing rate was desired, best guess values were then assigned to most of the variables involved in fiber calcination. The following two methods were derived for firing the fiber from room temperature to 1950 F:

	Firing Technique 1		Firing Technique 2	
Firing Stage	Firing Time*	Soak Time at Upper Temp.	Firing Time*	Soak Time at Upper Temp.
Room temp. to 600 F	0**	1 hr	0**	1 hr
600 to 1200 F	0.5 hr	0	3 hr	0
1200 to 1950 F	50 min	0.5 hr	50 min	0.5 hr

* Uniform firing rate.

** Fibers are placed in periodic kiln maintained at 600 F.

The firing is carried out in periodic kilns in an air atmosphere. The fibers are protected from temperature gradients and convection currents by placing them between thin blankets of insulation fiber.

The work was thus a general study of unfired fiber drying during storage, as previously described, with respect to fiber properties after firing by either of the above two methods. Following this general study, the better drying technique could then be combined with the better firing technique to yield reproducible fired fibers with high tensile strengths and moduli of elasticity.

2. Fiber Drying vs Fired Fiber Properties

a. Extent of Fiber Drying

Fiber drying during extrusion and collection was kept constant by maintaining temperature and relative humidity constant. Fiber samples were collected for a uniform 1-hour period. Fiber forming and collection conditions caused the 35% solids content in the solution to change to 45% solids content in the fiber sample.

Varying degrees of fiber drying beyond the collection step were accomplished by storing the fibers under various relative humidity conditions. Fiber samples were weighed immediately upon removal from the collection drum and periodically during storage. The weight changes reported are based on the initial fiber weight as stripped from the collector. Positive weight changes signify a weight gain, and negative weight changes signify lost weight.

Table IV shows the properties of fibers dried to various degrees at 80 F before being fired in either of the two selected ways. A correlation between storage relative humidity and fiber weight loss is apparent. Fiber storage over magnesium perchlorate in a desiccator has resulted in a routine weight loss of 12 to 16% at the apparent 0% relative humidity. This weight loss is similar to that observed with fiber storage at 20% relative humidity, indicating that this is the maximum amount of fiber drying that can be achieved at 80 F and ambient atmospheric pressure.

A slower firing schedule through the 600 to 1200 F range appears to be better for fiber properties than the faster firing schedule regardless of the amount of fiber drying. However, good fiber properties were obtained with both firing methods depending on the amount of fiber drying. Based on this information, the following two fiber drying techniques were chosen for further work:

1. Storage over magnesium perchlorate at 80 F to an equilibrium weight loss.
2. Storage at 600 F.

The optimum combination of one of the drying methods with one of the selected firing techniques was sought.

b. Tensile Strength vs. Storage Time over Magnesium Perchlorate

Table V shows the average tensile strength and modulus of elasticity of fibers stored for various times over magnesium perchlorate and then fired in either of two ways. Samples fired using Technique 2 (3 hr at 600 to 1200 F) are almost universally identical in properties. This is not surprising since it has

TABLE IV

AVERAGE FIBER PROPERTIES FOR FIBERS DRIED AT 80 F AND VARIOUS RELATIVE HUMIDITIES AND FIRED IN VARIOUS MANNERS

Sample No.	Storage Time (hr)	Storage Rel. Hum. (%)	Wt. Change (%)	Firing Technique 1		Firing Technique 2	
				Tens. Str.* (psi)	Mod.(E)* (psix10^6)	Tens. Str.** (psi)	Mod.(E)** (psix10^6)
B-313-9	0	--	0	200,000	20.2		
B-3056-1-2	87	40	+2.6			180,000	21.1
B-3056-2-7	87	40	+4.1	136,000	20.5		
B-3056-1-3	87	40	+4.8			200,000	22.6
B-3056-2-8	87	40	+5.3	178,000	22.7		
B-3056-1-5	87	30	-7.9			202,000	22.1
B-3056-2-10	87	30	-9.3	83,000	17.3		
B-308-2R	95	20	-11.7			221,000	21.8
B-308-1R	95	20	-15	Powdery - could not test.			
B-313-10	118	0***	-13			164,000	21.8
B-314-2	67	0***	-14	Powdery - could not test.			

* Individual fiber properties are shown in Table A.5.

** Individual fiber properties are shown in Table A.6.

*** Magnesium perchlorate desiccator.

TABLE V

AVERAGE FIBER PROPERTIES VS. UNFIRED
FIBER STORAGE TIME OVER MAGNESIUM PERCHLORATE

		Firing Technique 2*		Firing Technique 1	
Sample No.	Storage Time (hr)	Tens. Str. (avg. psi)	Mod. (E) (avg. psix10^6)	Tens. Str. (avg. psi)	Mod. (E) (avg. psix10^6)
B-313-15	19	190,000	22.6	All samples were brittle and powdery; could not test. (40 to 200 hr storage times)	
B-313-13	22	236,000	22.2		
B-313-10	118	164,000	21.8		
B-313-7	141	195,000	22.2		
B-313-3	165	216,000	21.6		
B-313-4	285	218,000	20.1		

* Individual fiber properties are shown in Table A.7.

been found that about 16 hours over magnesium perchlorate produces a green fiber dried to an equilibrium weight loss of 12 to 16%. According to DTA-dTGA studies, this weight loss occurs at temperatures up to 212 F.

These data confirm the statements above. Firing Technique 2 generally results in fibers of 200,000 psi tensile strength and 20 to 22 x 10^6 modulus of elasticity. Firing Technique 1 can yield fibers of this quality irregularly. Sample B-308-1R (Table IV) was identical in drying amount (15% loss) to these samples, and Firing Technique 1 yielded brittle and powdery fibers in that case also.

c. Tensile Strength vs. Soak Time at 600 F

Table VI shows fiber samples fired to 1950 F in the two selected manners after soaking for various times at 600 F. All samples were placed in a periodic kiln at 600 F immediately after being stripped from the collection drum.

Regardless of soak time at 600 F, most of the fiber samples exhibit almost identical properties. However, a soak time from 16 to 24 hours most consistently yields fired fibers of ~200,000 psi tensile strength.

Thus, the two firing techniques can yield routine 200,000 psi tensile strength and 20 x 10^6 psi modulus of elasticity for fibers of 75% alumina, 20% silica, and 5% boric oxide composition. The fibers can be stripped immediately from the collection wheel and fired in either of the two methods described above or they can be prepared for the firing in the following two manners:

1. Fibers can be stored to an equilibrium weight loss (12 to 16% in 24 hr) over magnesium perchlorate and then must be fired using Technique 2 to obtain reproducible 200,000 psi tensile strength and 20 to 22 x 10^6 psi modulus of elasticity.
2. Fibers can be immediately placed in a kiln at 600 F and soaked for 1 to 24 hours at 600 F before being fired by either of the two methods.

3. Microstructural Analysis

The study of the drying-firing steps of the process produced fiber samples having wide ranges of tensile strength and modulus. Understanding this could serve as a guide to developing fibers with reproducible as well as higher strengths and elasticity values. Consequently, numerous batches of fibers were examined using electron microscope and X-ray diffraction techniques in an attempt to correlate the characteristics of these fibers with their tensile and modulus of

TABLE VI

AVERAGE FIBER PROPERTIES FOR FIBERS SOAKED VARIOUS TIMES AT 600 F AND FIRED TO 1950 F IN TWO MANNERS

		Firing Technique 1*		Firing Technique 2**	
Sample No.	Soak Time (hr at 600 F)	Tens. Str. (psi)	Mod. (E) (psix10^6)	Tens. Str. (psi)	Mod. (E) (psix10^6)
B-314-11	1	200,000	22.8		
B-313-30	1			180,000	20.4
B-313-20	17			198,000	20.3
B-313-29	19			194,000	20.1
B-313-27	21			233,000	22.9
B-314-7	22	227,000	21.3		
B-314-6	24	193,000	22.0		
B-314-5	40	202,000	21.0		
B-313-23	44			174,000	20.1
B-313-18	88			197,000	21.2

* Individual fiber properties are shown in Table A.8.

** Individual fiber properties are shown in Table A.9.

elasticity properties. The fiber batches selected for this evaluation, and the range of properties exhibited by these typical samples are shown below.

Sample No.*	Fiber Diam. (avg. microns)	Tens. Str. (avg. psi)	Mod. (E) (avg. psix10^6)	Etch Time (hr)	Figure No.
B-305-7	5.6	221,000	23.3	3.5	4
B-305-1A	6.0	190,000	20.8	2.5	7
B-299-3A	5.6	118,000	20.5	8	6
B-299-11B	5.5	174,000	20.0	8	7,8
B-305-8B	6.0	127,000	17.3	2.5	6
B-299-1B	5.9	53,000	13.8	3.5	5

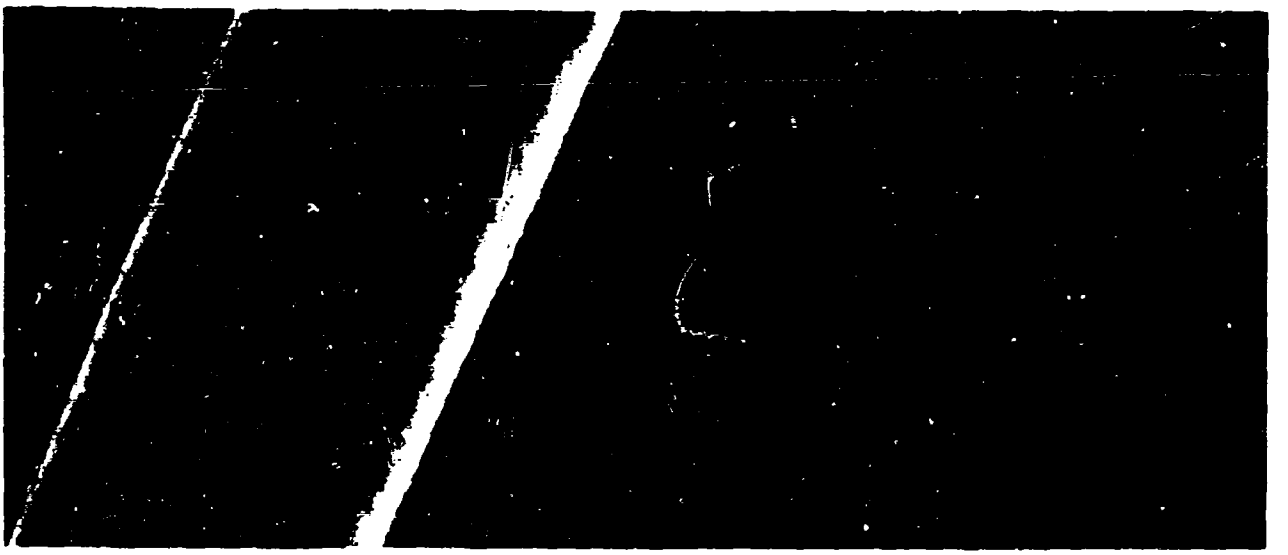
* Individual fiber data shown in Tables A.1, A.2, and A.3.

Figure 4 shows a high-strength, high-modulus fiber as opposed to a low-strength, low-modulus fiber (Figure 5). The natural surfaces of the two fibers are similar. However, when both samples had been exposed to a chemical etch (10% aqueous HF), a dramatic difference became apparent. The low-strength, low-modulus sample, B-299-1B, was very badly eroded. Figure 6 shows etched fibers which were comparable in tensile strength but differed in modulus. The low-modulus sample, B-305-8B, is badly eroded, but the low-strength, high-modulus sample, B-299-3A, is only slightly eroded, similar to the high-strength, high modulus sample, B-305-7. Figure 7 shows two more good fibers which were not eroded badly by the chemical etching. Figure 8 shows end views of etched sample B-299-11B. The end views show a very homogeneous cross section of rather fine grain size.

In summary, the fibers that were easily etched were the ones that exhibited the lower modulus. No relationship seemed to exist between tensile strength and the degree of chemical attack by the etchant as evidenced in the scanning electron photomicrographs. The possible explanations for these effects consider differences in the porosity and/or crystallinity of these samples.

It would be reasonable to assume that the fibers which exhibited the low-modulus had higher porosity, and therefore would be attacked more readily by the etchant. The differences in crystallinity; i.e., the size of the grains and their degree of order could also reflect difference in the rate of chemical attack.

A further attempt to establish the microstructural reasons for the higher rate of chemical attack on fibers with low-modulus of elasticity was made using transmission electron microscopy.



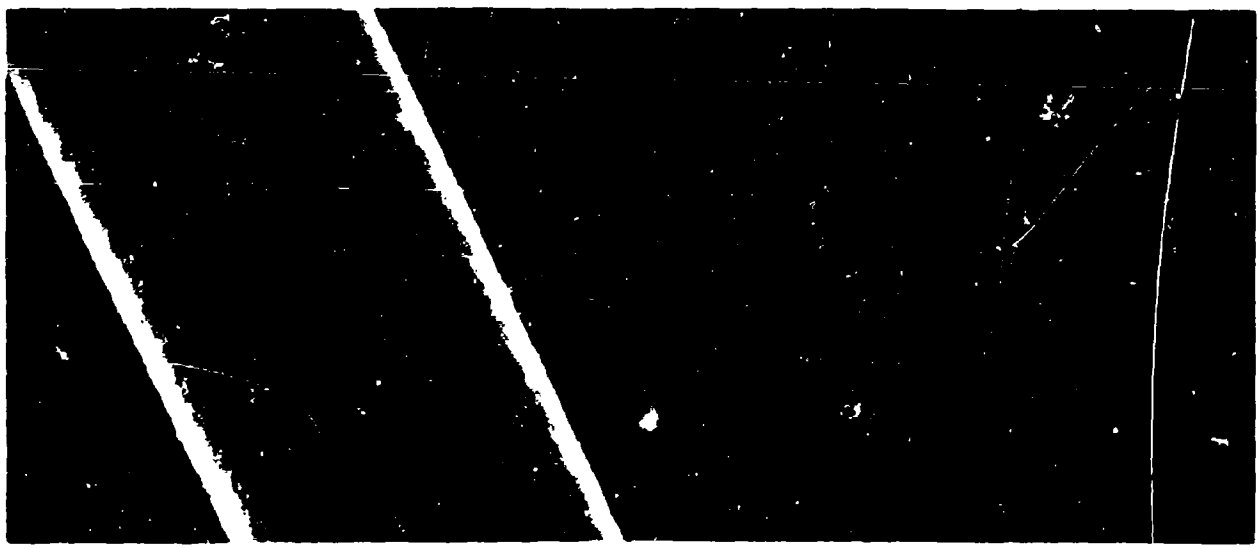
x10,000 NATURAL

x30,000 NATURAL

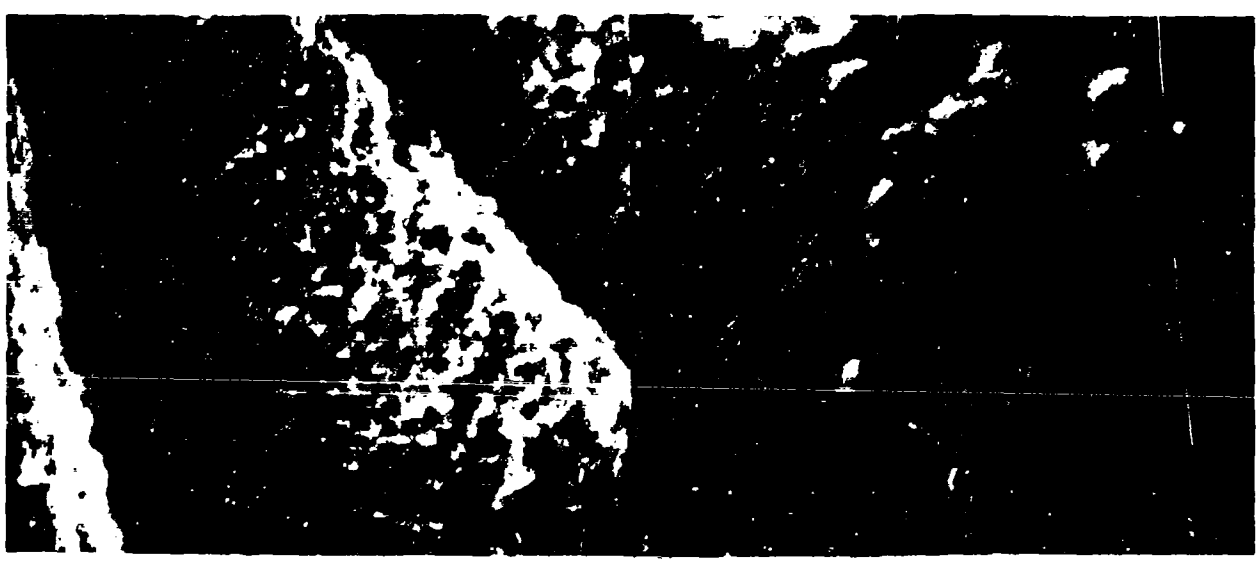
x10,000 ETCH

x30,000 ETCH

FIGURE 4. SCANNING ELECTRON MICROGRAPHS OF SAMPLE B-305-7 WITH NATURAL SURFACE AND AFTER 3.5 HOUR ETCH IN 10% HF



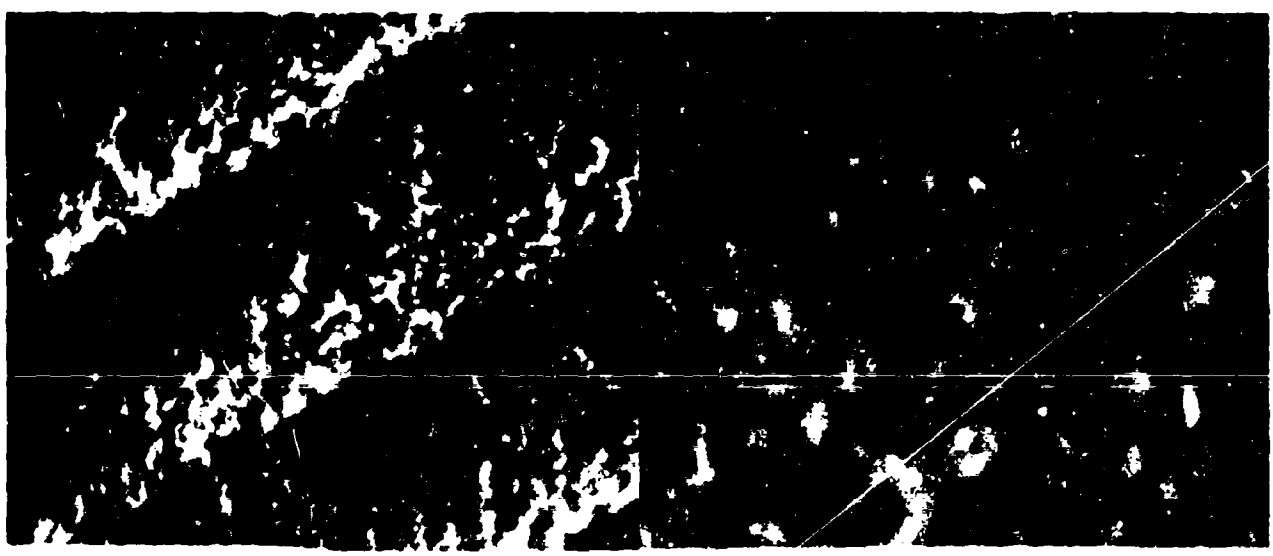
x10,000 NATURAL x30,000 NATURAL



x10,000 ETCH x30,000 ETCH

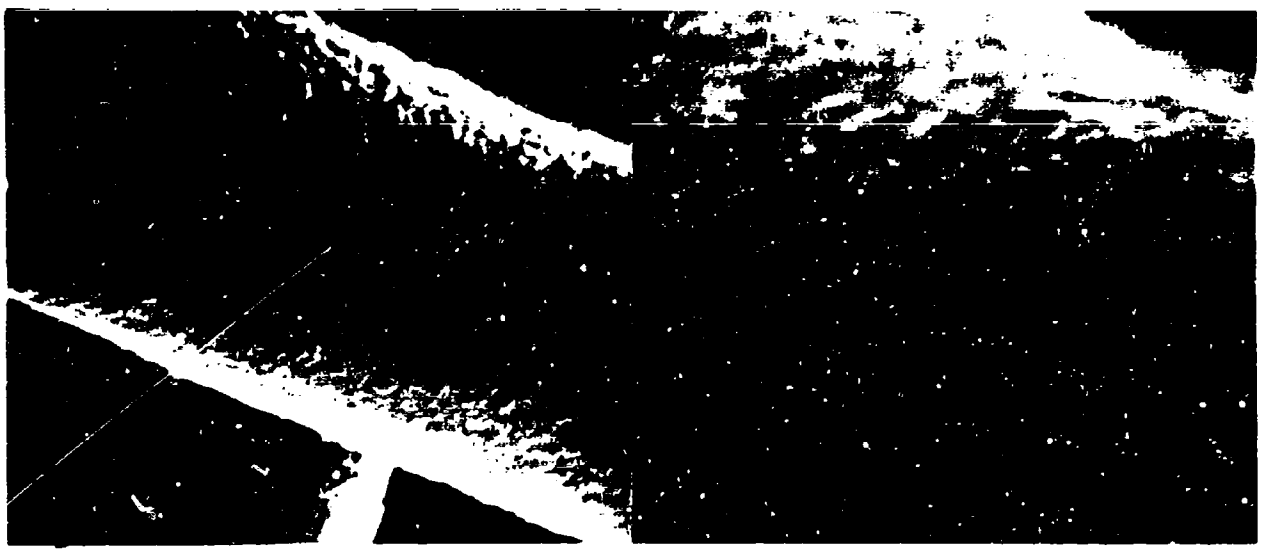
FIGURE 5. SCANNING ELECTRON MICROGRAPHS OF SAMPLE B-299-1B WITH NATURAL SURFACE AND AFTER 3.5-HOUR ETCH IN 10% HF

x10,000 x25,000

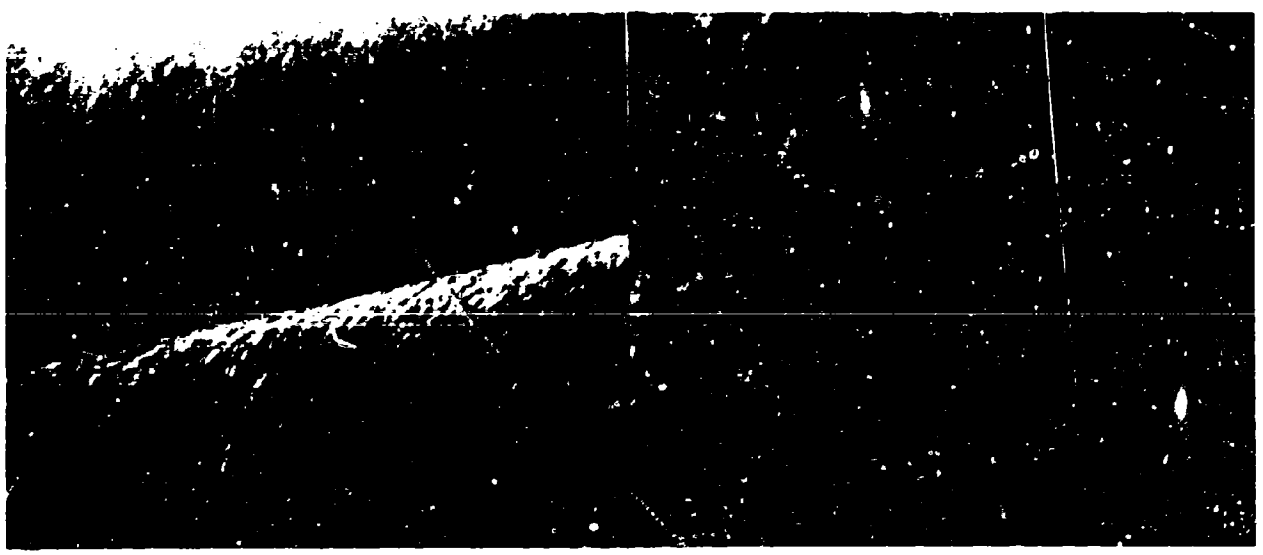


x10,000 x30,000

FIGURE 6. SCANNING ELECTRON MICROGRAPHS OF SAMPLE B-299-3A (top) AFTER 8 HOUR ETCH IN 10% HF AND SAMPLE B-305-8B (bottom) AFTER 2.5 HOUR ETCH IN 10% HF



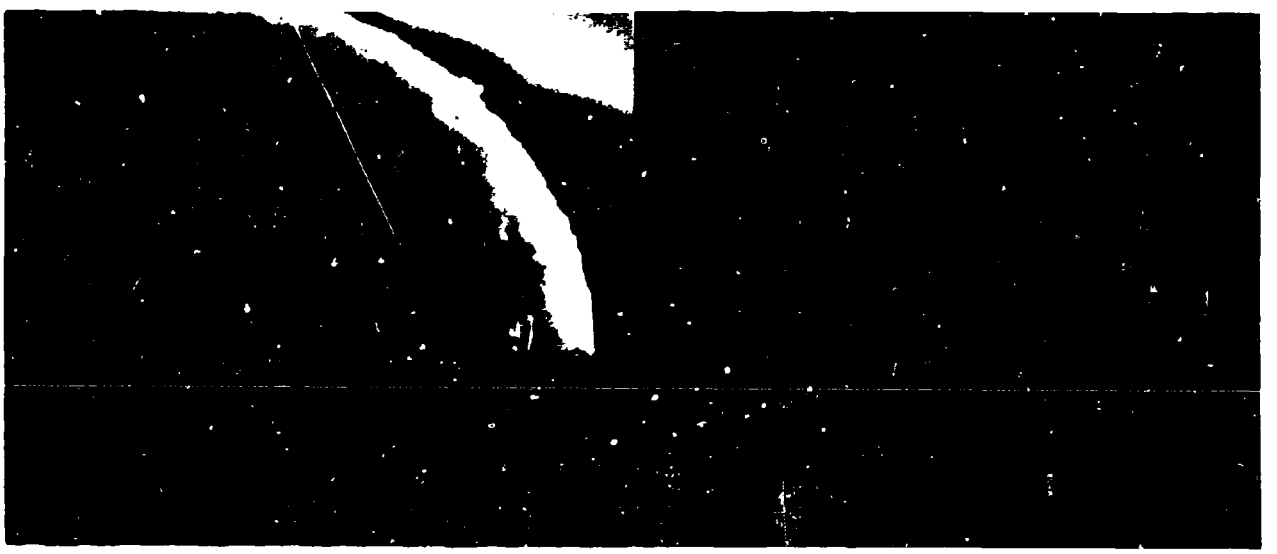
x10,000 x30,000



x10,000 x30,000

FIGURE 7. SCANNING ELECTRON MICROGRAPHS OF SAMPLE B-299-11B (top) AFTER 8 HOUR ETCH IN 10% HF AND SAMPLE B-305-1A (bottom) AFTER 2.5 HOUR ETCH IN 10% HF

x5,000 x10,000



x25,000 x50,000

FIGURE 8. SCANNING ELECTRON MICROGRAPH END VIEWS OF SAMPLE B-299-11B AFTER 4 HOUR ETCH IN 10% HF

The pertinent data for these samples are summarized below:

Sample No.	Tens. Str. (avg. psi)	Mod. (E) (avg. psix10^6)	Crystalline Const., X-ray Major	Minor	Cryst. Size
B-305-7	221,000	23.3	Mullite	γ-Al_2O_3	350 Å
B-3056-9A	242,000	20.6	"	"	<300 Å
B-299-3A	118,000	20.5	"	"	<300 Å
B-305-8B	127,000	17.3	"	-------	450 Å

Qualitative Porosity Level:

B-305-7	very low
B-3056-9A	low
B-299-3A	inhomogeneous (low)
B-305-8B	high

High-tensile strength, high-modulus of elasticity, and a low rate of chemical erosion are characteristic of low-porosity specimens (B-305-7, Figure 9 and B-3056-9A, Figure 10). Specimen B-299-3A (Figure 11) shows a dual cross sectional structure; that is, somewhat more porosity than that of B-3056-9A is apparent in some areas, and a structure comparable to that of B-305-7 is apparent in other areas in a given cross section. Observations suggest that the dual microstructure in B-299-3A is partially responsible for its lower tensile strength.

Specimen B-305-8B (Figure 12) contains a larger volume of pores than any of the other three specimens and also was most rapidly attacked by aqueous hydrofluoric acid. X-ray data indicate that this specimen also differs with respect to crystallinity and size of the major crystalline phase. B-305-8B contains only mullite, whereas the others contain a major amount of mullite and minor amount of γ-alumina. The mullite in B-305-8B has a crystallite size about 30% larger than mullite in the other specimens. Mullite crystal growth and high void content probably contribute to the low measured properties of sample B-305-8B. The high void content is also responsible for the higher rate of chemical attack noted above.

In brief the results suggest that porosity has an inverse effect on mechanical properties. Observations of specimen B-299-3A (Figure 11) indicate that porosity distribution is also critical in controlling mechanical properties. Fibers which exhibit substantially different mechanical properties do so because of variations in microstructure. Unquestionably the lowest mechanical properties occur in the most porous specimens. Since fired fiber properties have been brought to a stage of reproducibility at reasonable values, the above information indicates that the microstructure is also reproducible. That is, the drying-firing operations are building the same porosity and crystallinity into all fiber samples.

FIGURE 9. SAMPLE NO. B-305-7 X10,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite - Minor, γ-Alumina
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 221,000 psi - MODULUS: 23.3×10^6 psi

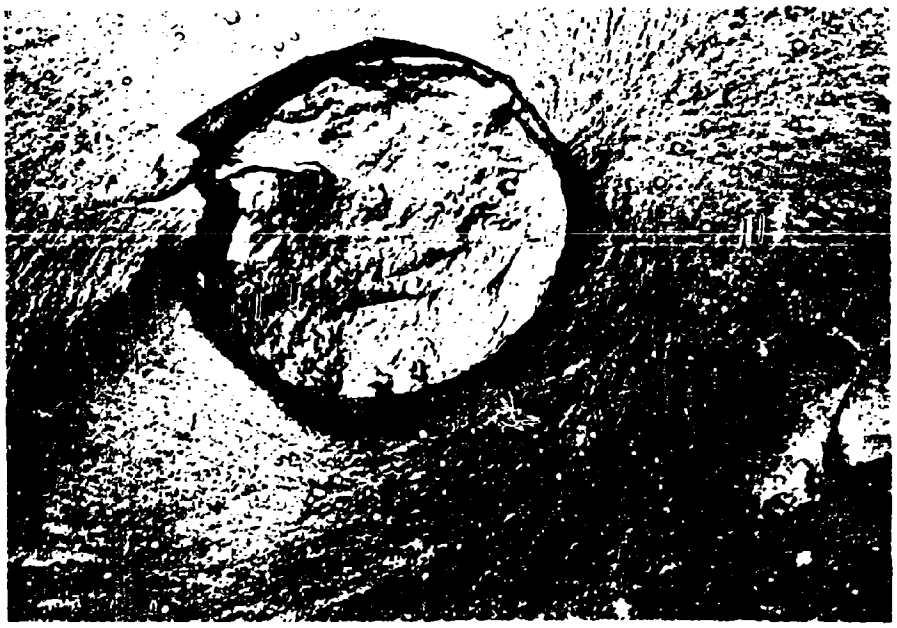


FIGURE 10. SAMPLE NO. B-3056-9A X10,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite - Minor, γ-Alumina
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 242,000 psi - MODULUS: 20.6×10^6 psi

FIGURE 11. SAMPLE NO. B-299-3A X10,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite - Minor, γ-Alumina
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 118,000 psi - MODULUS: 20.5×10^6 psi

FIGURE 12. SAMPLE NO. B-305-8B X10,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 127,000 psi - MODULUS: 17.3×10^6 psi

Further property improvements in a given fiber composition now depend on refinement of the drying-firing operations to reduce the porosity level or create a more perfect crystalline fiber structure.

4. Fiber Properties vs. Fiber Diameter

Fiber diameter has been reported to influence fiber properties[1,2]. Smaller diameter fibers have sometimes exhibited significantly higher measured tensile strength and modulus of elasticity than identical fibers of larger diameter. Since reproducibility of fiber properties had been attained through control of the fiber drying and firing operations, a check of fiber diameter vs. properties was run on the 75% alumina, 20% silica, and 5% boric oxide fibers.

Several fiber samples of different diameters were prepared and fired to 1950 F using Firing Technique 1 with zero storage time and 1-hour soak at 600 F. The tabulation below describes the average properties of these samples:

Sample No.	Diameter* (avg. microns)	Tens. Str.* (avg. psi)	Modulus (E)* (avg. psix10^6)
B-313-4	4.7	247,000	23.5
B-313-9	6.0	200,000	20.2
B-313-1	7.3	220,000	23.3

* Individual fiber properties are shown in Table A.10.

While the sample with the smallest diameter does exhibit the best properties, there does not appear to be a large difference in fiber properties depending on fiber diameter, at least within the range observed. Perusal of the individual fiber data in Table A.10 leads to essentially the same conclusion. Proportionately more small-diameter fibers have high strength than larger fibers, but some larger fibers show strengths equivalent to those of the fine-diameter fibers. The diameter range and the amount of data observed are rather small, and it is still possible that fibers with diameters greater than 10 microns will exhibit decreased properties and that fibers of less than four microns in diameter will exhibit increased properties. However, fibers in the diameter range being investigated do not appear to significantly vary in properties as the diameter changes.

B. STUDY OF FIBER COMPOSITION WITH CONSTANT DRYING-FIRING OPERATIONS

Routine and reproducible 200,000 psi tensile strength and 20 to 22 x 10^6 psi modulus of elasticity were accomplished in fibers of 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition. These properties were not assumed to be the maximum

attainable from fibers of the given composition. However, the properties were reproducible, implying that the process was under control if not fully optimized. Further optimization of the process could probably raise the fiber properties somewhat, but the increase would be small relative to the contract goals. Therefore, experimentation with fiber composition in search of higher properties was begun as the second research phase.

Fiber compositions from pure mullite (73% Al_2O_3 and 27% SiO_2) to pure alumina (100% Al_2O_3) were chosen for study. Fiber properties through a range of firing temperatures were monitored for each fiber composition. The drying-firing techniques developed during the first research phase were also applied to fibers varying in boric oxide and phosphorous pentoxide content from 0 to 10%. The object of the research was to find a composition which would yield a fiber containing a crystalline species of inherently high modulus of elasticity.

1. Influence of Boric Oxide and Phosphorous Pentoxide Additives on Fiber Properties

a. Tensile Strength and Modulus of Elasticity vs. Additive Content

Mullite crystals have always been found in a variety of fiber compositions as long as the Al_2O_3/SiO_2 ratio has been close to that of true mullite ($3Al_2O_3 \cdot 2SiO_2$). The additives B_2O_3 and/or P_2O_5 in about 5 weight percent quantity had been found beneficial to fiber properties for an unknown reason. The study of pure mullite fibers (73% Al_2O_3 and 27% SiO_2) was begun in hopes of attaining fibers with values of "E" modulus close to the "true" value for mullite and determining, if possible, the effect of the additives on the fired fiber.

Fibers of 73% Al_2O_3 and 27% SiO_2 composition were prepared, fired to a variety of temperatures, and tested. The following tabulation describes the results of mechanical testing and X-ray diffraction analysis:

				X-ray Diffraction Crystallinity		
Sample No.	Firing Temp (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^6*)	Major	Minor	Cryst. Size
M-100-2	1950	115,000	12.3	γ-Al_2O_3	------	------
M-100-4	2100	151,000	17.3	"	Mullite	------
M-103-4	230	112,000	20.7	Mullite	------	1600 Å
M-102-1B	2500	41,000	26.6	"	------	"

* Individual fiber data are shown in Table A.11.

These data are shown in graphical form in Figure 15. As the firing temperature increases, the degree of crystallinity, crystallite size, and modulus of elasticity increases while the tensile strength increases and then decreases at temperatures greater than 2100 F. For comparison, Figure 14 and the tabulation below show the same relationships for the 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition fibers studied in the drying-firing investigations reported above.

Sample No.	Firing Temp. (F)	Tens. Str.* (avg. psi)	Modulus (E) (avg. psix10^6*)	X-ray Diffraction Crystallinity Major	Minor	Cryst. Size
B-314-12	1700	211,000	21.8	γ-Al_2O_3	Mullite	300 Å
B-314-4	1950	223,000	20.5	Mullite	γ-Al_2O_3	350 Å
B-313-25	2400	140,000	24.4	"	-------	600 Å

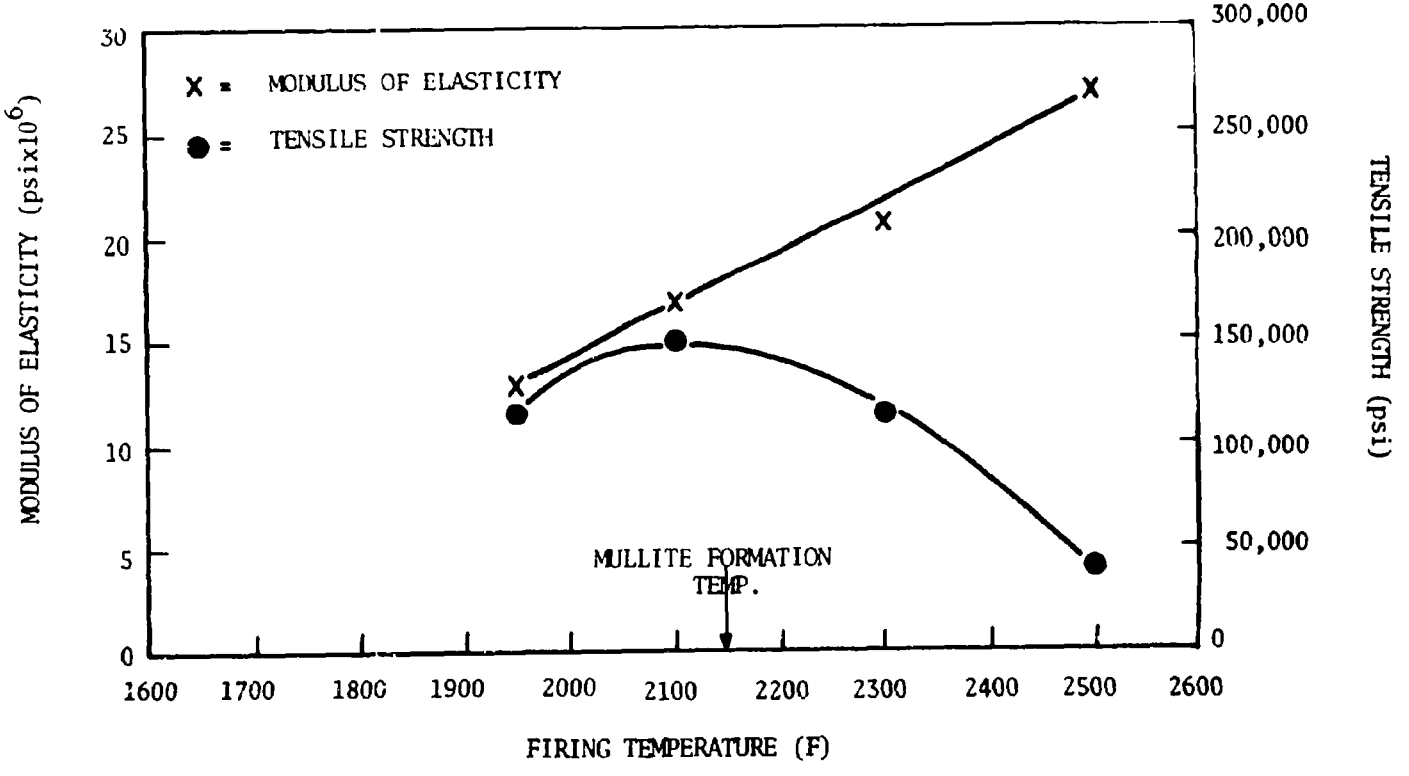
* Individual fiber data are shown in Table A.12.

Mullite formation occurs between 1800 and 1950 F for the 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition fibers, whereas it occurs around 2150 F for pure mullite fibers. Above these temperatures the tensile strength and firing temperature lines for the two compositions are approximately parallel. However, the fiber containing 5% B_2O_3 is about 50,000 psi stronger in tensile strength over the indicated temperature range. The B_2O_3 promotes mullite formation at a lower firing temperature. This is probably the reason for the higher modulus values at lower temperatures exhibited by the fibers containing boric oxide. At firing temperatures greater than 2400 F, the two modulus curves would appear to coincide while the 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition maintains its 50,000 psi tensile strength superiority over the pure mullite fiber. Addition of B_2O_3 to a 75% Al_2O_3 and 25% SiO_2 fiber base composition thus results in increased tensile strength at any firing temperature. The effect of a boric oxide addition on fiber modulus merely lowers the temperature where the fiber is converted to the higher modulus crystalline form.

The crystallite size differences in the above two tabulations could account at least partially for the 50,000 psi tensile strength difference. The B_2O_3 apparently not only lowers the mullite formation temperature but also is a crystallite growth inhibitor. This crystalline growth limitation should be beneficial to the production of higher strength fibers.

To further check the effect of B_2O_3 and P_2O_5 additives, fibers were prepared from a solution so that the composition of the fiber would be 77.6% Al_2O_3, 19.5% SiO_2, 1.6% B_2O_3 and 1.4% P_2O_5 after firing. These fibers were fired to various temperatures and tested as tabulated below and shown in Figure 15.

FIGURE 13. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 73% Al_2O_3 AND 27% SiO_2 FIBERS



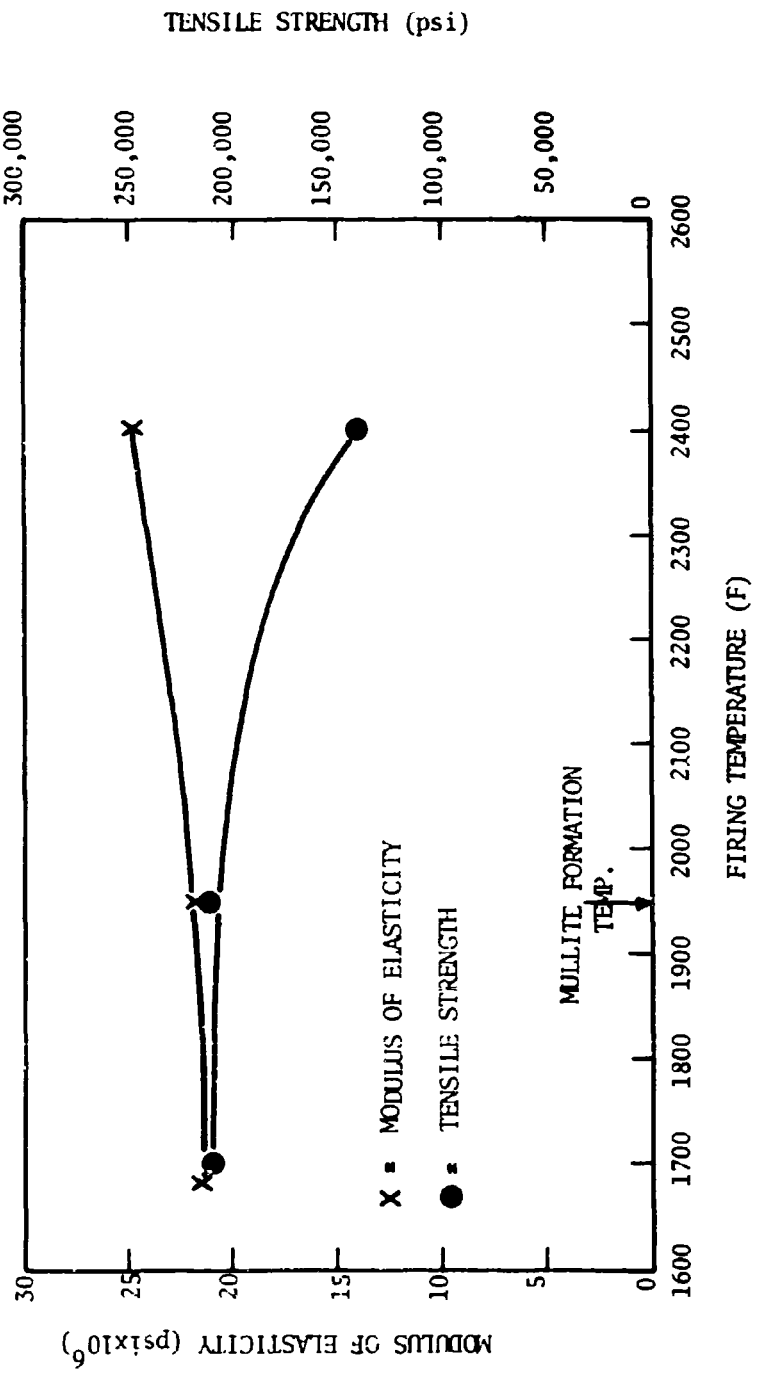
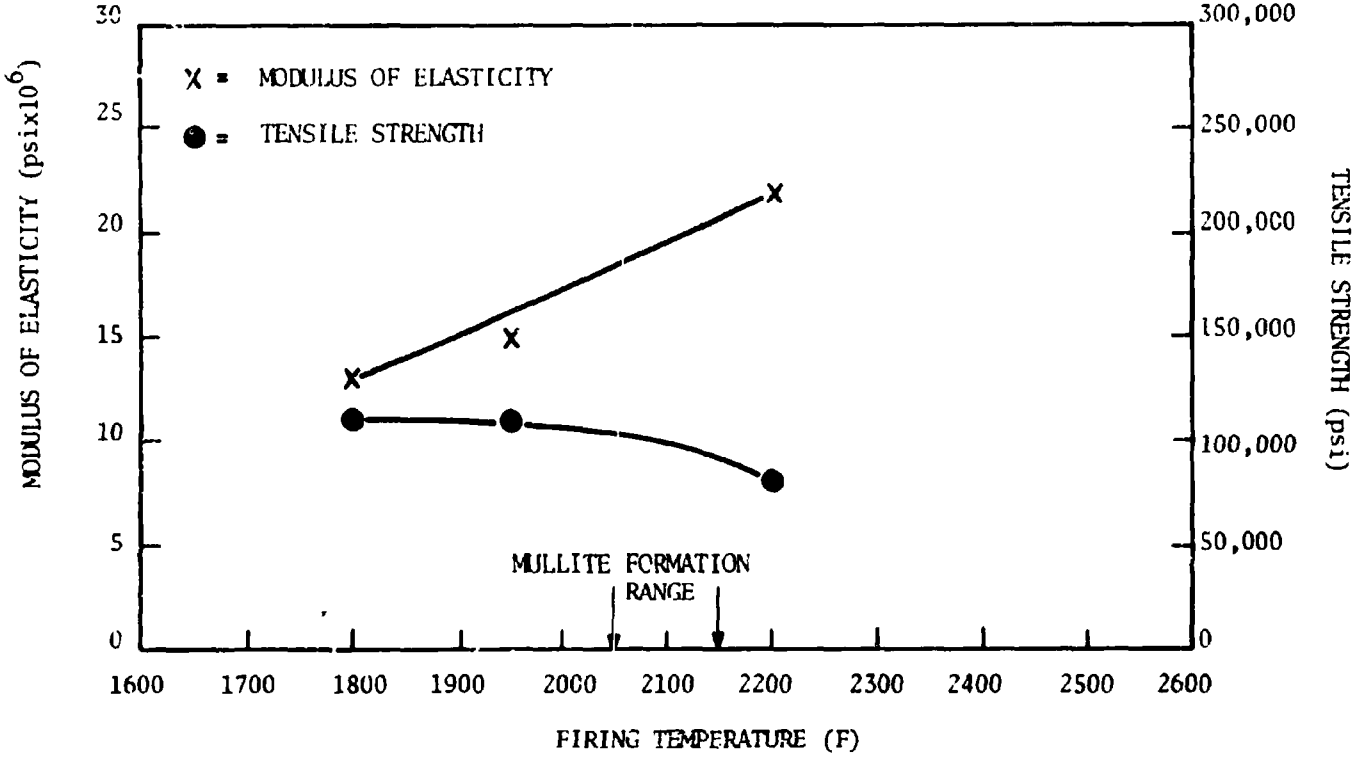


FIGURE 14. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 75% Al_2O_3, 20% SiO_2, AND 5% B_2O_3 FIBERS

FIGURE 15. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 77.6% Al_2O_3, 19.5% SiO_2, 1.6% B_2O_3 AND 1.4% P_2O_5 FIBERS



Sample No.	Firing Temp. (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^{6}*)	X-ray Diffraction Crystallinity Major	Minor	Cryst. Size
P-100-6	1800	110,000	12.6	γ-Al_2O_3	----	----
P-100-4	1950	101,000	14.6	"	----	----
P-100-5	2200	80,000	21.9	Mullite	----	1450 Å

* Individual fiber properties are shown in Table A.13.

The X-ray diffraction analyses indicate that the mullite formation temperature is about 2050 to 2150 F. This is higher than that found for the fibers containing 5% B_2O_3 but lower than that for mullite composition fibers with no additives. Again, the tensile strength line appears to parallel the lines in Figures 13 and 14, but at a lower overall tensile strength value. The line for modulus of elasticity appears to converge with those of Figures 13 and 14 at the higher temperatures.

Another series of firing temperatures was run on fibers of 74.5% Al_2O_3, 19.7% SiO_2, 4.7% B_2O_3, and 1.2% P_2O_5 composition as tabulated below and shown in Figure 16. X-ray diffraction data indicates that the mullite formation temperature of 1800 F is somewhat lower for these samples than for the 5% B_2O_3 containing fibers.

Sample No.	Firing Temp. (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^{6}*)	X-ray Diffraction Crystallinity Major	Minor	Trace
P-103-7	1800	200,000	21.0	γ-Al_2O_3	Mullite	----
P-103-8A	1950	203,000	21.9	Mullite	----	γ-Al_2O_3
P-103-8B	2200	194,000	23.5	"	----	----

* Individual fiber properties are shown in Table A.14.

With the slightly higher B_2O_3 and P_2O_5 contents, the modulus of elasticity and tensile strength curves follow the curves for 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 fiber rather closely at all positions.

Figure 17 shows the modulus of elasticity and tensile strength of several fiber compositions fired to 1950 F. These values are plotted as a function of the total contained percentage of B_2O_3 and P_2O_5. Both modulus of elasticity and tensile strength increase as the percentage of additives increases from 0 to 5 or 6%. Above 5 or 6% the values decrease and appear to level off. The modulus increase for additives increased from 0 to 5% is not unexpected since it has been

FIGURE 16. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 74.5% Al_2O_3, 19.7% SiO_2, 4.7% B_2O_3, AND 1.2% P_2O_5 COMPOSITION FIBERS.

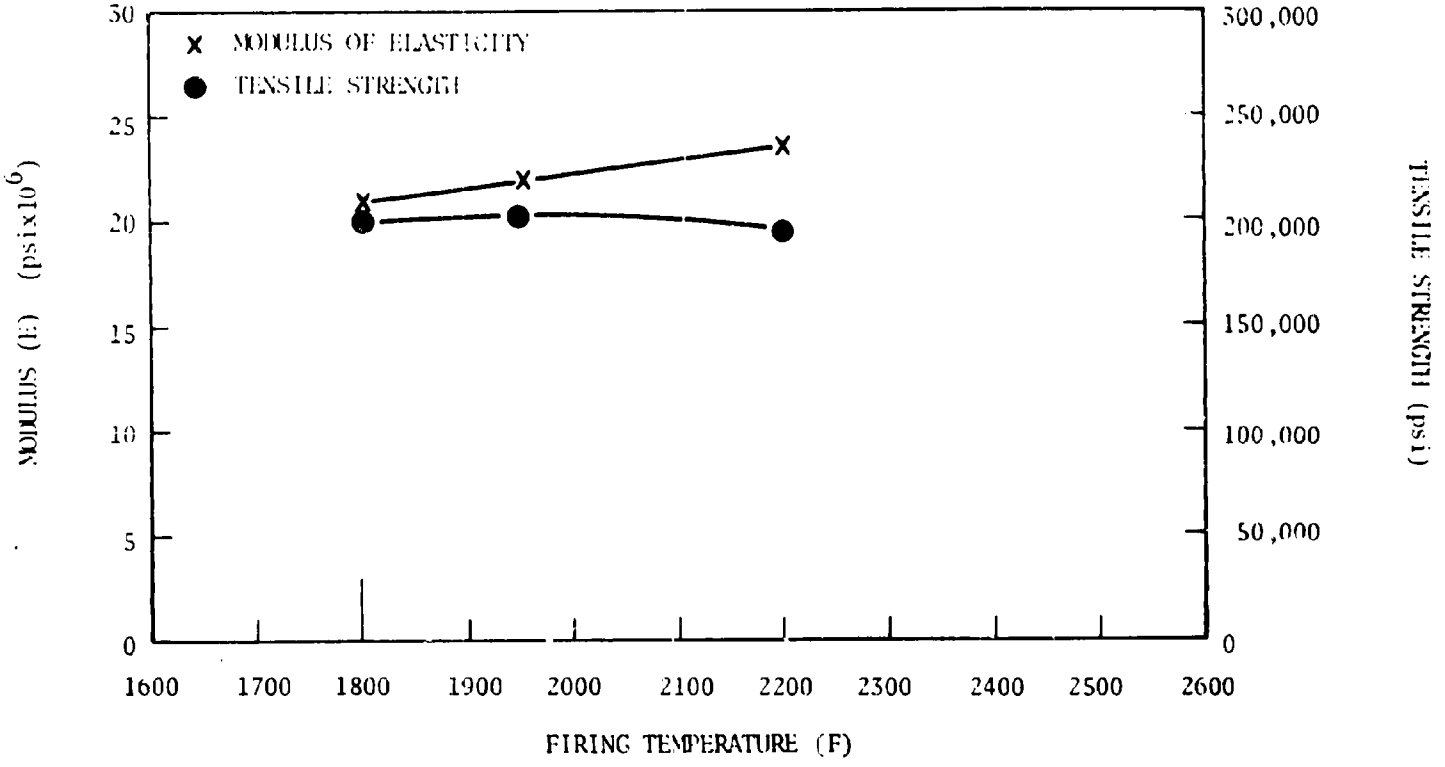
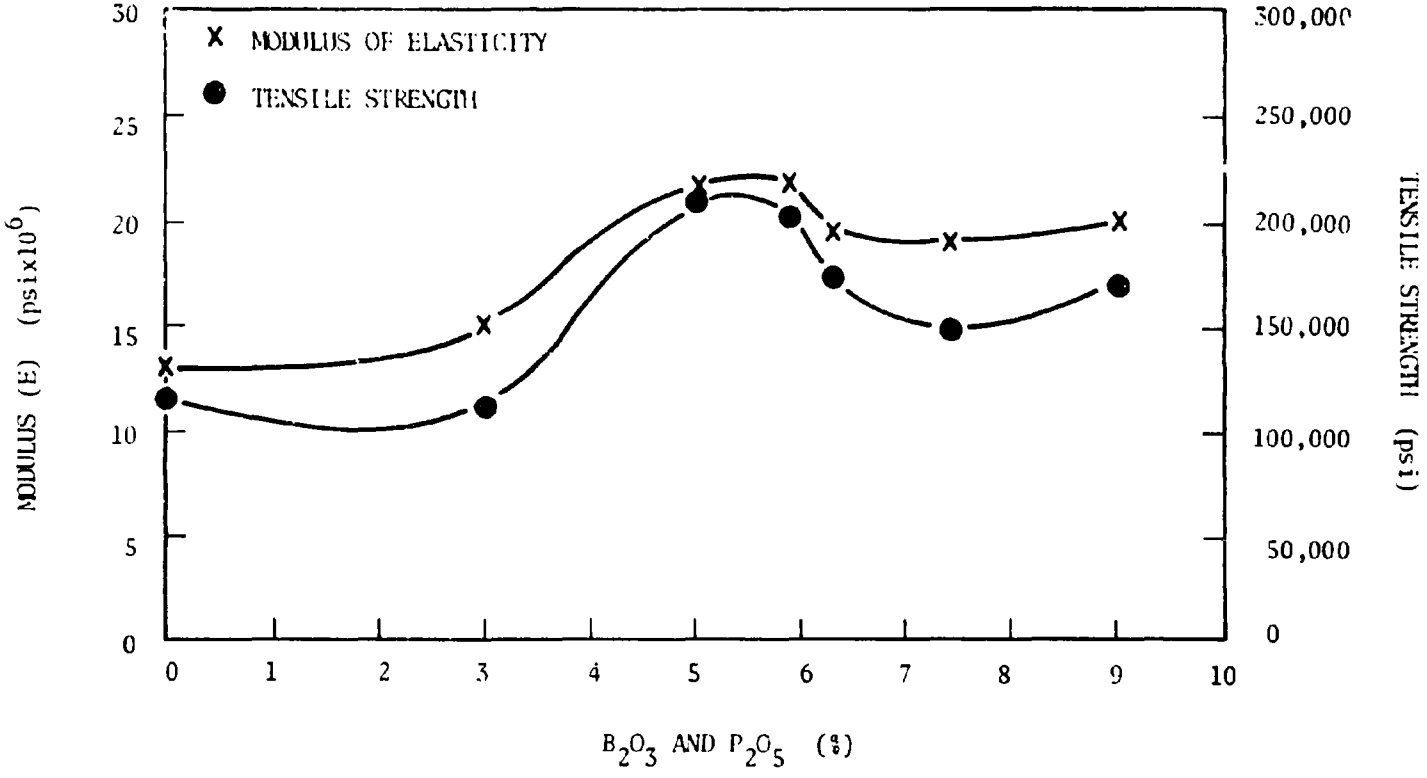


FIGURE 17. TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. B_2O_3 AND P_2O_5 CONTENT OF 1950 F FIRED FIBERS WITH ALUMINA-SILICA RATIO BETWEEN 3/1 AND 4/1.



shown in this range that the degree of crystallinity at any lower temperature is directly related to the amount of additives present. The degree of crystallinity is intimately related to the modulus of elasticity of the fiber.

Since the percentage of additives has been shown to influence the reaction of a fiber to a given firing temperature, the percentage of additives was plotted against fiber properties at the firing temperature where mullite formation occurs. This relationship is shown in Figure 18 for the samples containing 0 to 6% additives. The tensile strength drops appreciably for the fibers containing 3% total additives and increases rather significantly when the additives are increased to 5%. The modulus of elasticity climbs somewhat from about 18 x 10^6 psi to about 21 x 10^6 psi over the additive range shown. This climb is perhaps insignificant. Modulus values are routinely obtained in this entire range for the fibers containing 5 to 6% boric oxide. Thus, the modulus of elasticity attained with a given fiber composition is apparently independent of the additive content and is characteristic of the Al_2O_3/SiO_2 ratio and crystalline species derived therefrom. The additive content however, does determine the firing temperature necessary to convert the amorphous fiber to a higher modulus crystalline material and influences strongly the tensile strength of that crystalline material.

b. Microstructural Analysis

Transmission electron micrographs of the pure mullite fiber series, the fiber series containing 5% boric oxide and the fiber series containing 6% boric oxide and 1.4% phosphorous pentoxide were obtained for the purpose of clarifying through microstructural analysis the tensile strength trends noted in the preceding figures and to help explain the role of B_2O_3 and P_2O_5 additives.

Table VII summarizes conditions studied, variables considered, and results obtained in mechanical tests, X-ray diffraction analyses, and microstructural analyses.

Electron microscopic investigation of mullite compositions revealed that rounded features were prominent within the structures of all specimens. The size and number of these features was different for different firing temperatures. Representative results may be studied in Figures 19 through 28.

In general, higher firing temperatures reduced the number but increased the size of these microstructural features. In all series a size range and certain oversize features were noted, but only the average size was estimated. The

FIGURE 18. MODULUS OF ELASTICITY AND TENSILE STRENGTH VS. B_2O_3 + P_2O_5 CONTENT OF FIBERS FIRED TO MULLITE FORMATION TEMPERATURE.

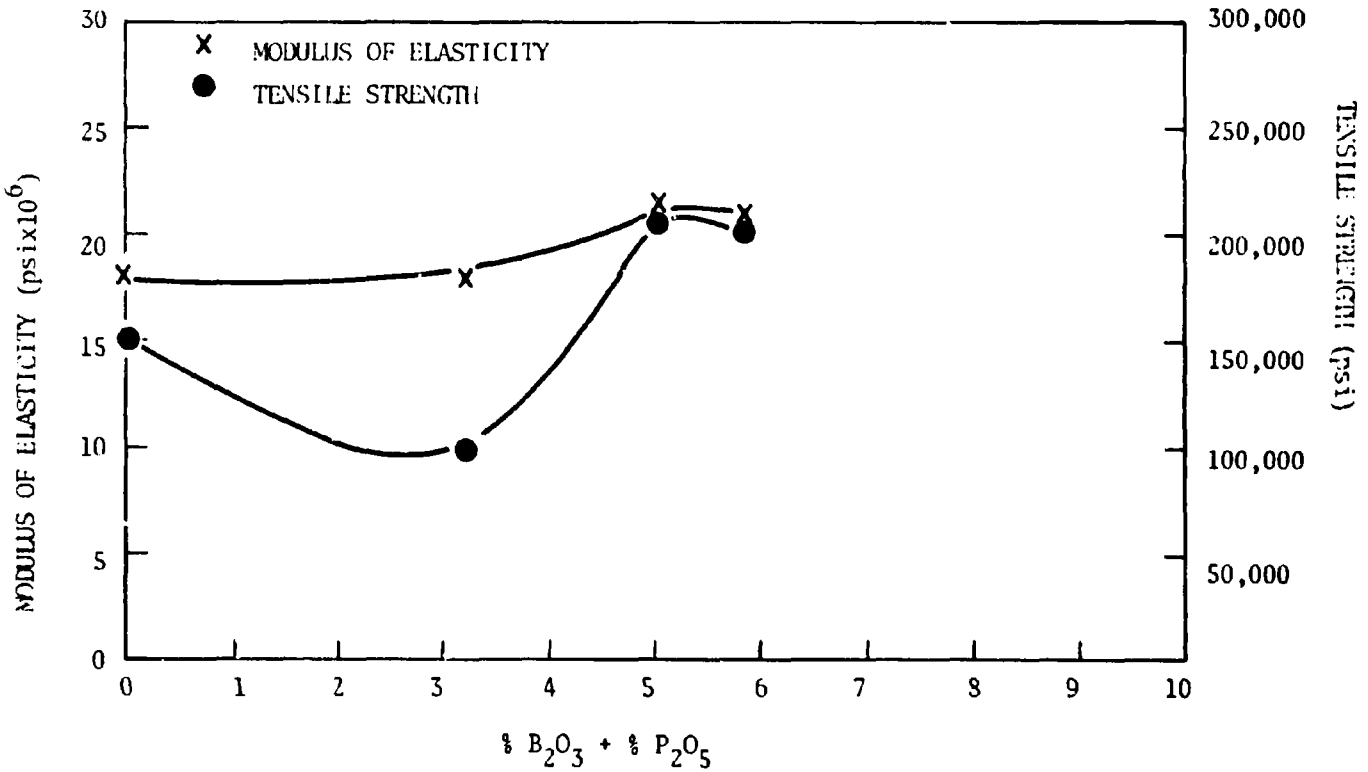


TABLE VII

DESCRIPTION OF FIBER SAMPLES ANALYZED
BY TRANSMISSION ELECTRON MICROSCOPY

Fiber Comp.	Sample No.	Firing Temp. (F)	Mechanical Tests: Tens. Str. (avg. psi)	Mechanical Tests: Modulus (E) (avg. psix10^6)	X-ray Diffraction Crystallinity: Major	X-ray Diffraction Crystallinity: Minor	X-ray Diffraction Crystallinity: Cryst. Size (Å)	Pore Dens./Sq. Micron	Avg. Pore Size (Å)	Calc. Pore Vol. (%)
70% Al_2O_3	B-314-12	1700	211,000	21.8	γ-Al_2O_3	Mullite	300	120	400	15
20% SiO_2	B-314-4	1950	223,000	20.5	Mullite	γ-Al_2O_3	350	186	300	13
5% B_2O_3	B-313-25	2400	140,000	24.4	Mullite	-------	600	81	900	52*
77.6% Al_2O_3	P-100-6	1800	110,000	12.6	γ-Al_2O_3	-------	---	365	200	11
19.5% SiO_2	P-100-4	1950	101,000	14.6	γ-Al_2O_3	-------	---	187	400	24
1.6% B_2O_3	P-100-5	2200	80,000	21.9	Mullite	-------	1450	87	700	34*
1.4% P_2O_5										
73% Al_2O_3	M-100-2	1950	115,000	12.3	γ-Al_2O_3	-------	---	482	200	15
27% SiO_2	M-100-4	2100	151,000	17.3	γ-Al_2O_3	Mullite	---	120	300	9
	M-103-4	2300	112,000	20.7	Mullite	-------	1600	49	700	19
	M-102-1B	2500	41,000	26.6	Mullite	-------	1600	94	500	18

* Reevaluation suggests subjective error; real value ~10%.

** Probable subjective error; real value ~18%.

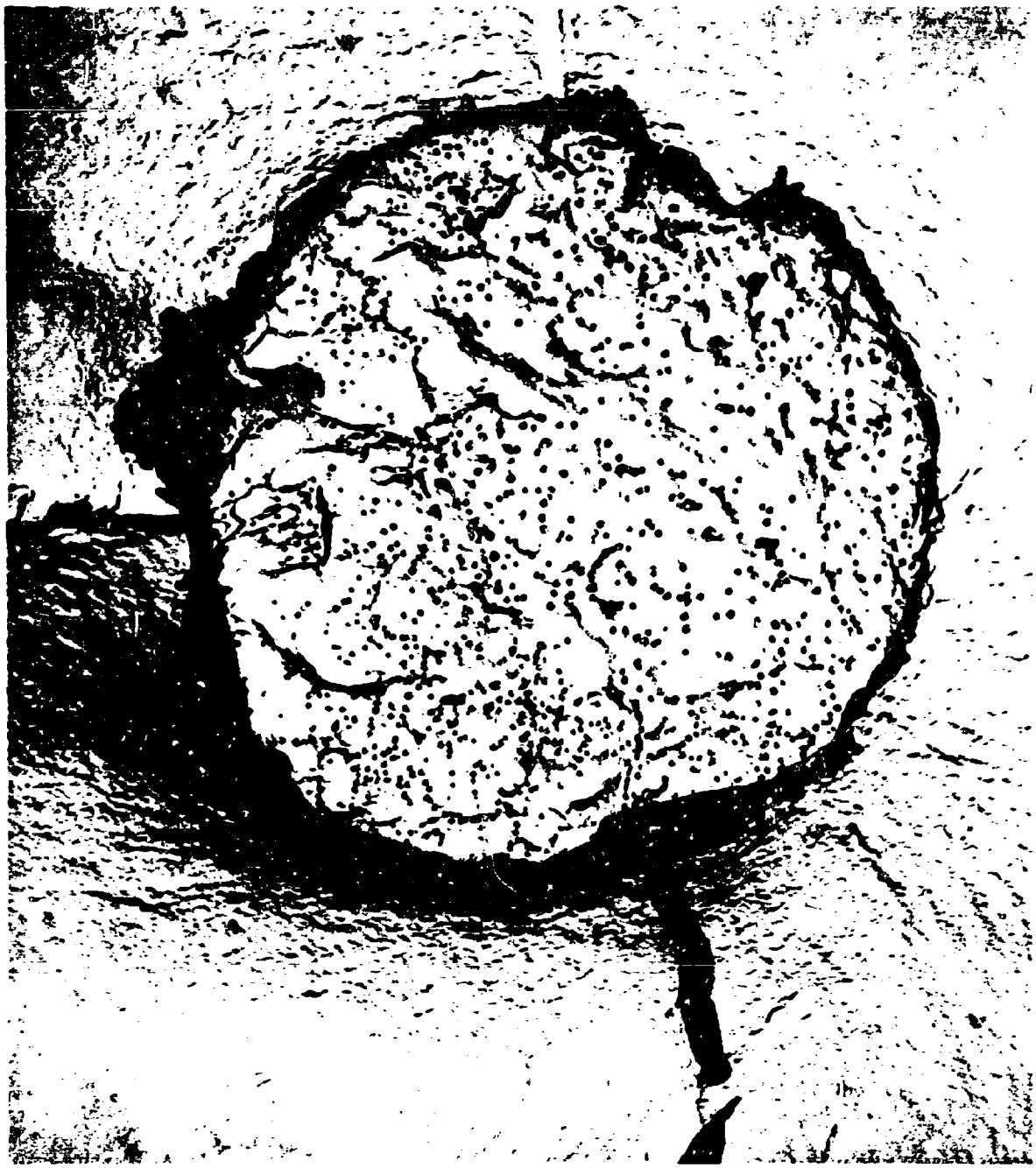


FIGURE 19. SAMPLE NO. B-314-12 X30,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, γ-Alumina - Minor, Mullite
FIRING TEMPERATURE: 1700 F
TENSILE STRENGTH: 211,000 psi - MODULUS: 21.8×10^6 psi

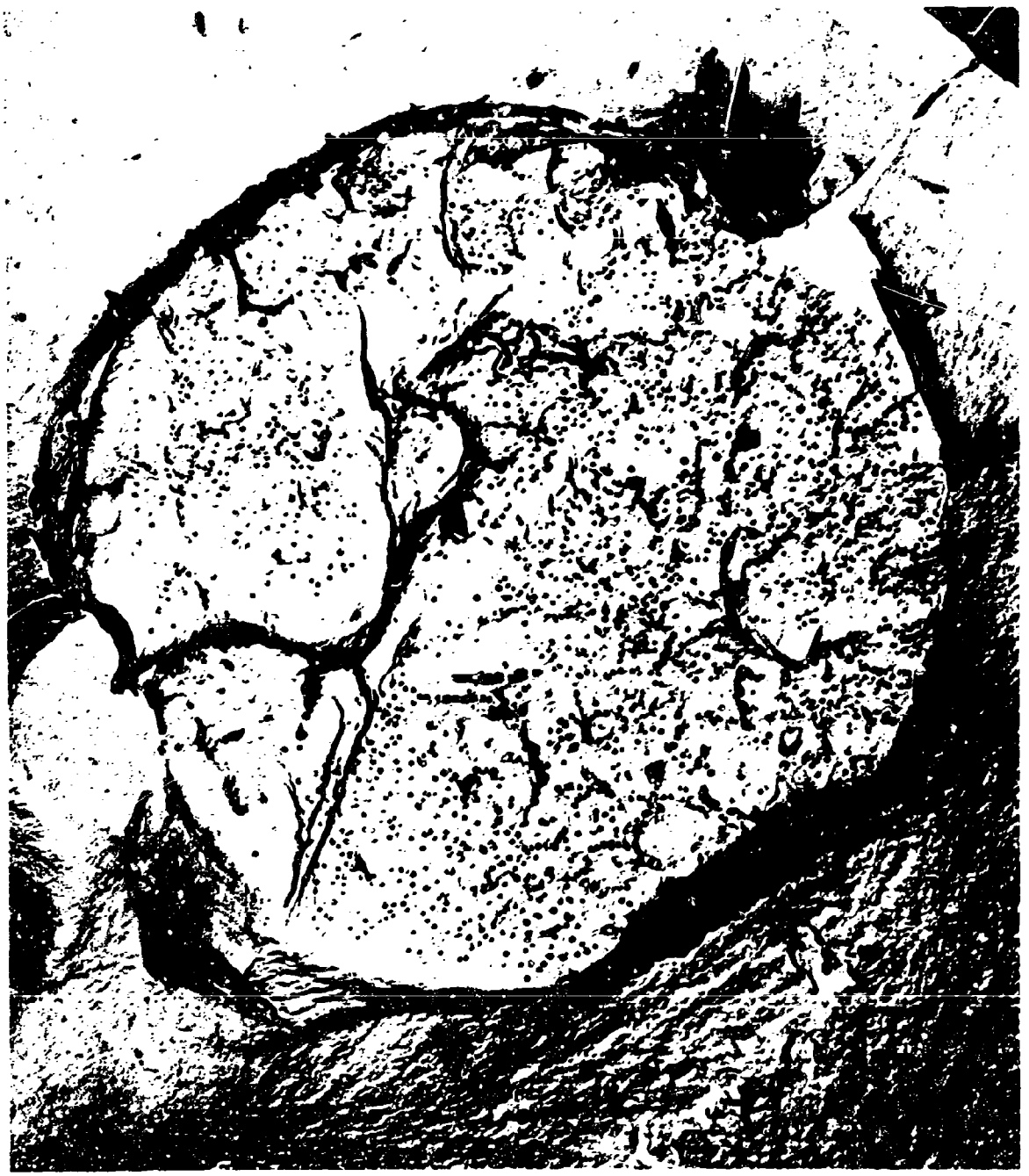


FIGURE 20 SAMPLE NO. B-314-4 X30,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 223,000 psi - MODULUS: 20.5×10^6 psi

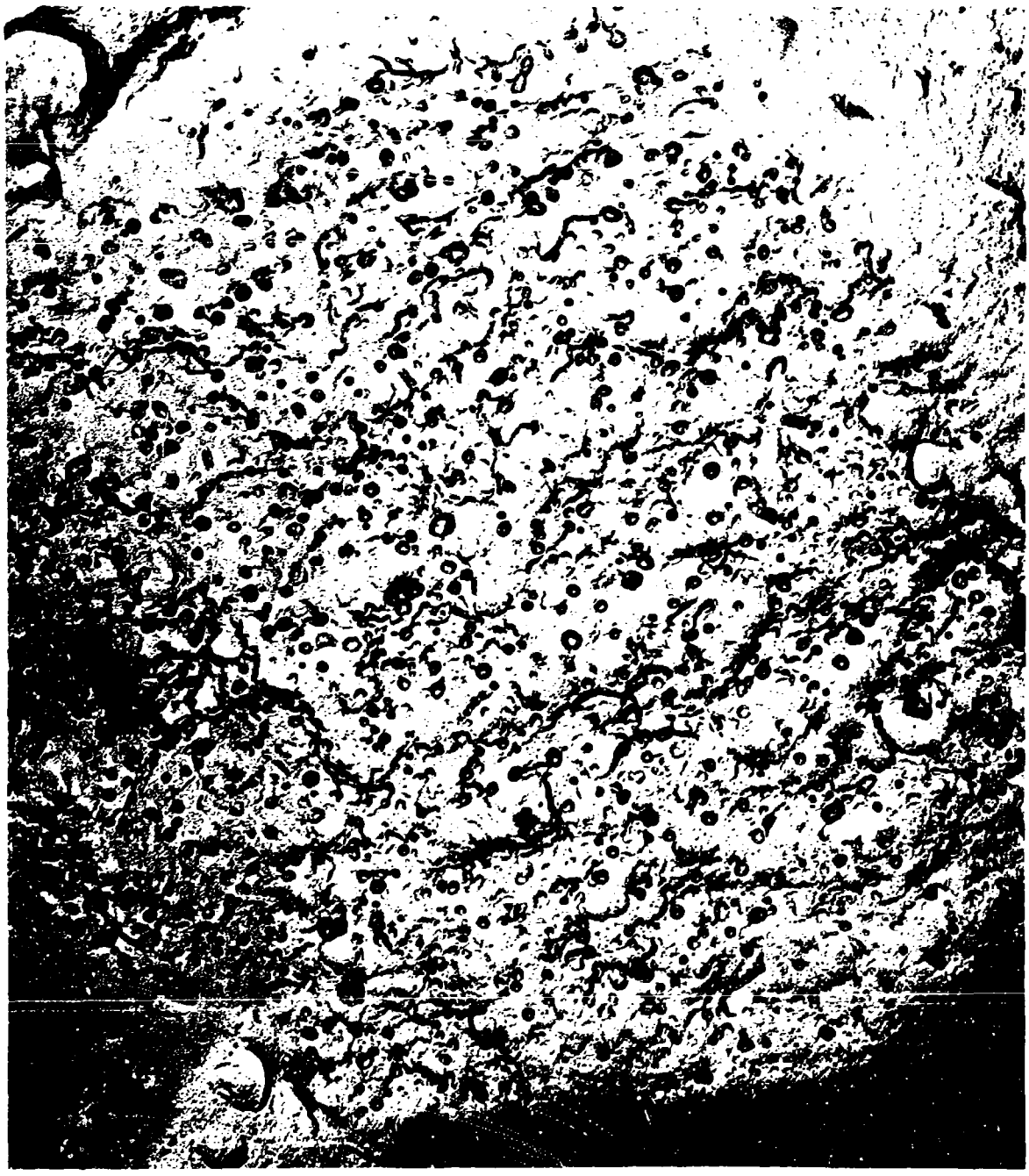


FIGURE 21. SAMPLE NO. B-313-25 X30,000
CHEMISTRY: 75% Al_2O_3, 20% SiO_2, 5% B_2O_3
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 2400 F
TENSILE STRENGTH: 140,000 psi - MODULUS: 24.4×10^6 psi

FIGURE 22. SAMPLE NO. P-100-6 X30,000
CHEMISTRY: 73% Al_2O_3, 24% SiO_2, 1.5% B_2O_3, 1.5 P_2O_5
CRYSTAL: Major, γ-Alumina
FIRING TEMPERATURE: 1800 F
TENSILE STRENGTH: 110,000 psi - MODULUS: 12.6×10^6 psi

FIGURE 23. SAMPLE NO. P-100-4 X30,000
CHEMISTRY: 73% Al_2O_3, 24% SiO_2, 15% B_2O_3, 1.5% P_2O_5
CRYSTAL: Major, γ-Alumina
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 101,000 psi - MODULUS: 14.6×10^6 psi

FIGURE 24. SAMPLE NO. P-100-5 X30,000
CHEMISTRY: 73% Al_2O_3, 24% SiO_2, 1.5% B_2O_3, 1.5% P_2O_5
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 2200 F
TENSILE STRENGTH: 80,000 psi - MODULUS: 21.9×10^6 psi

FIGURE 25. SAMPLE NO. M-100-2 X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, γ-Alumina
FIRING TEMPERATURE: 1950 F
TENSILE STRENGTH: 115,000 psi - MODULUS: 12.3×10^6 psi

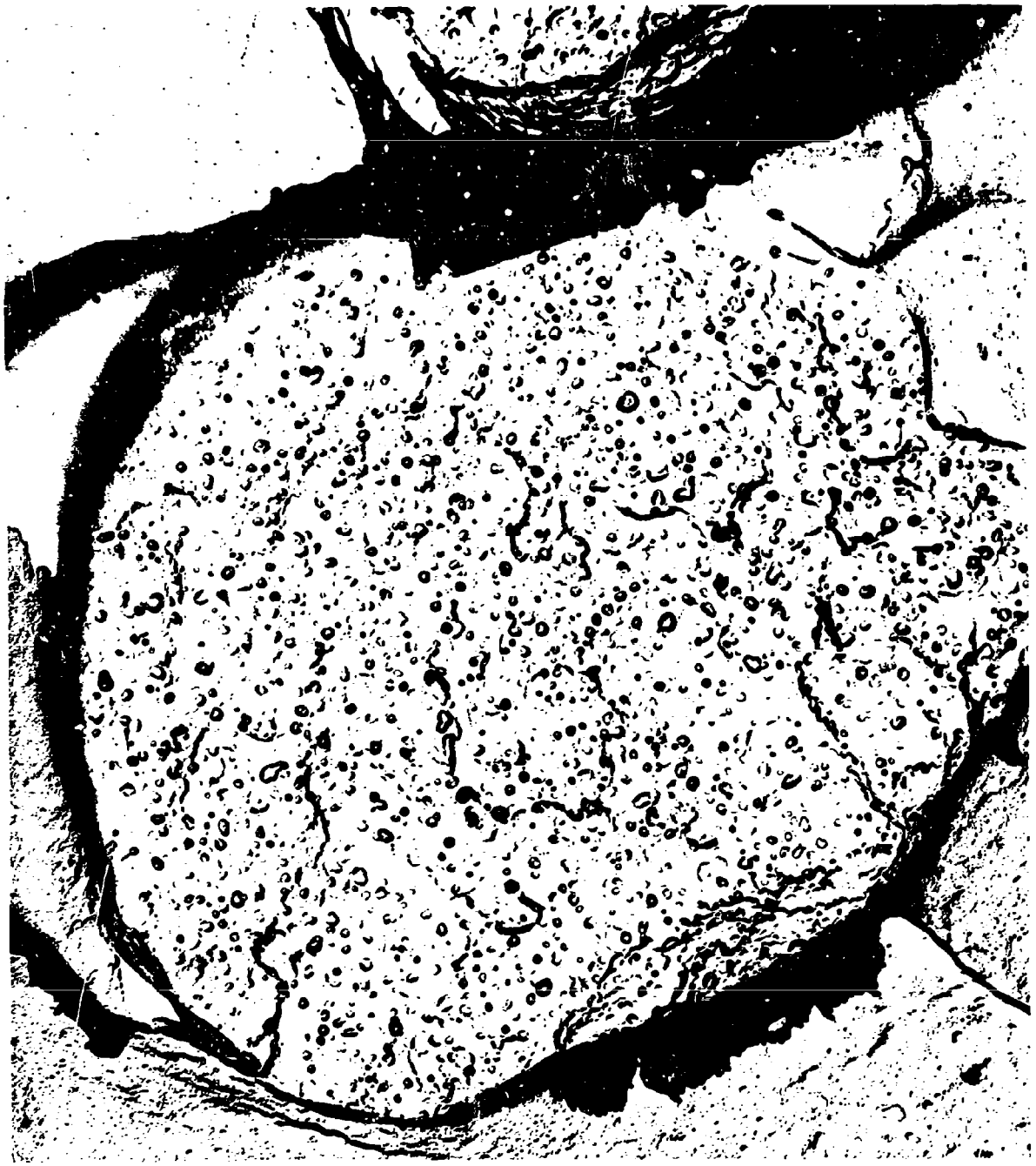


FIGURE 26. SAMPLE NO. M-100-4 X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, γ-Alumina - Minor, Mullite
FIRING TEMPERATURE: 2100 F
TENSILE STRENGTH: 151,000 psi - MODULUS: 17.3×10^6 psi

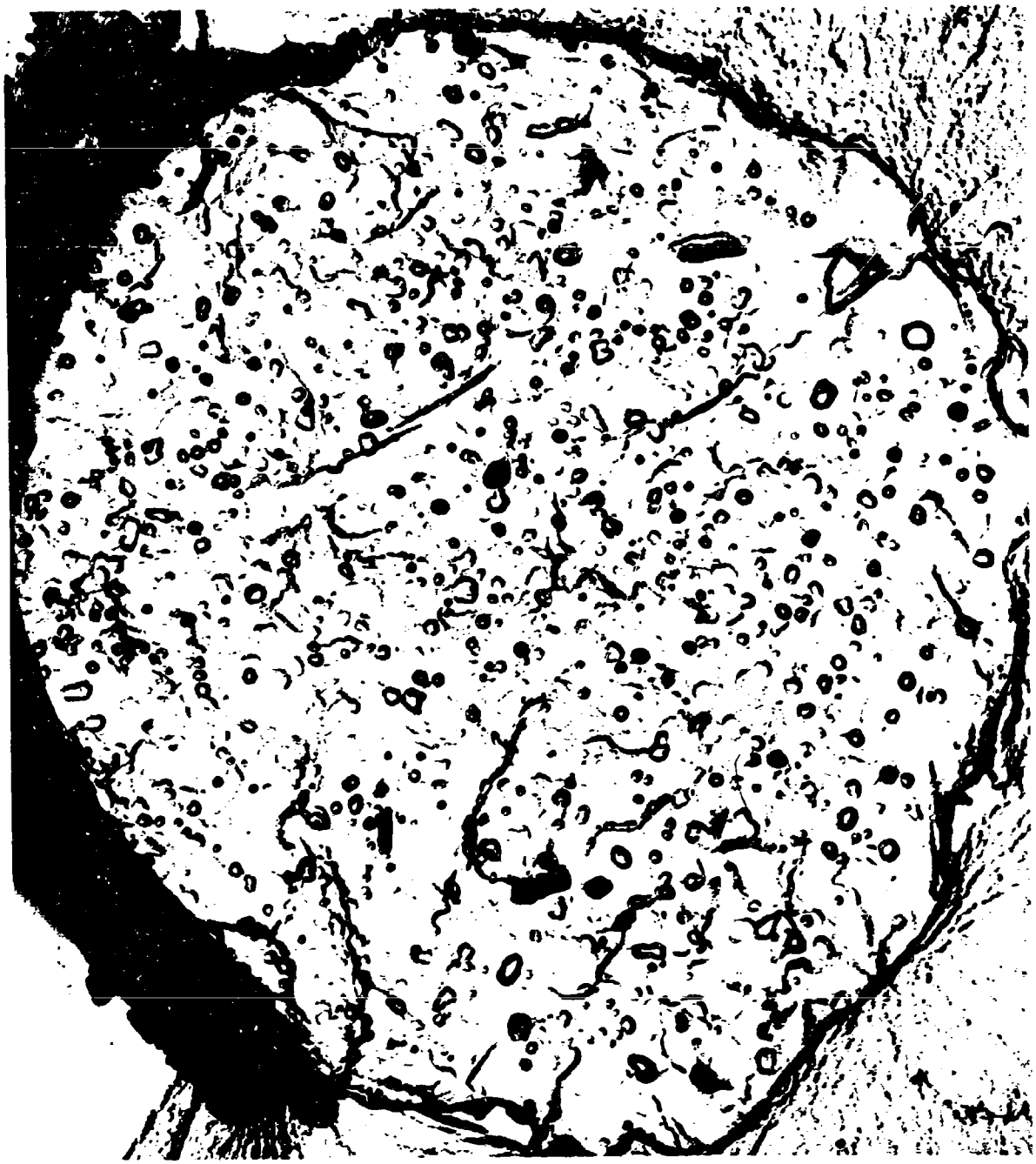


FIGURE 27. SAMPLE NO. M-103-4 X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 2300 F
TENSILE STRENGTH: 112,000 psi - MODULUS: 20.7×10^6 psi

FIGURE 28. SAMPLE NO. M-102-1B X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, Mullite
FIRING TEMPERATURE: 2500 F
TENSILE STRENGTH: 41,000 psi - MODULUS: 26.6×10^6 psi

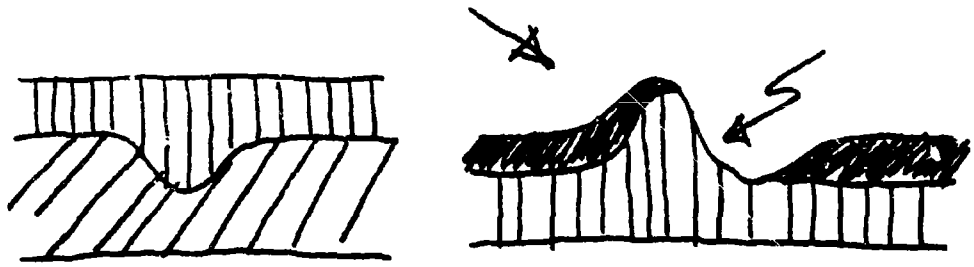
density is a count of the number of features per unit area and is undoubtedly imperfect due to subjective error and the fact that measurements were made from projected surfaces.

Grain structure was revealed by etching M series specimens. Other series were not as thoroughly studied. In a given specimen, grains were found to be about four times the size of the round features.

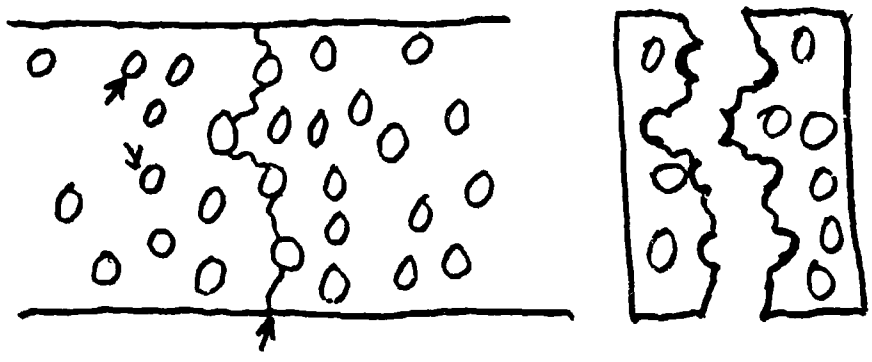
A comparison of etched M-100-2 and M-100-4 specimens, which roughly bracket the γ-alumina to mullite transformation (Figures 29 and 30), shows that the development of granularity corresponds with mullite development. Mullite crystal size (by X-ray measurement) and round microstructural feature size (by micrographs) have been similar. It would be most curious, however, if only 10 to 20% of the high-temperature fired fiber were mullite, as this observation might suggest.

The following observations led to the firm conclusion that round features observed in the microstructures are voids or porosity.

1. Close inspection of critically shadowed portions of replicas showed that all features cast shadows outside themselves. Thus, it is indicated that the features are holes in the original specimen surface (see schematic).



2. Only a porous condition could produce this effect (see schematic).



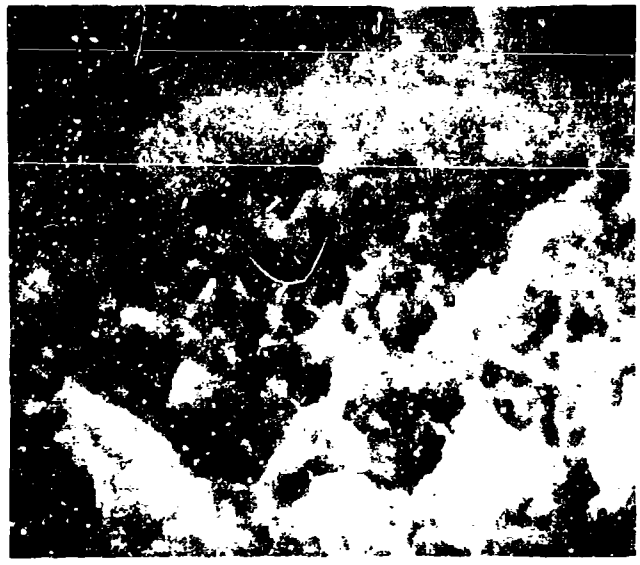


FIGURE 29. SAMPLE NO. M-100-2 X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, γ-Alumina
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS

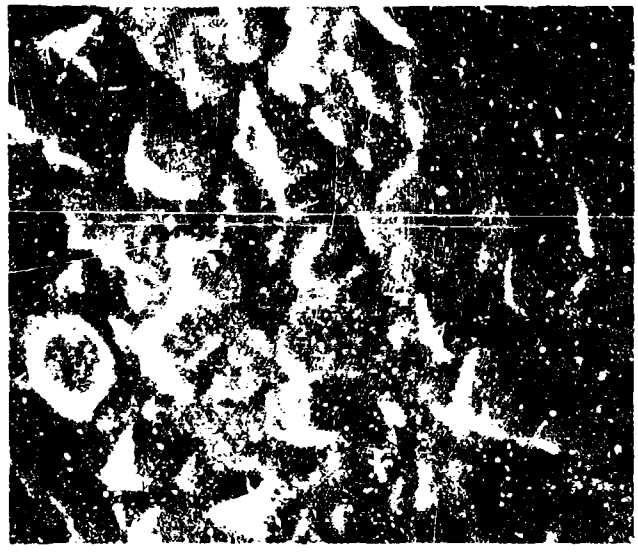
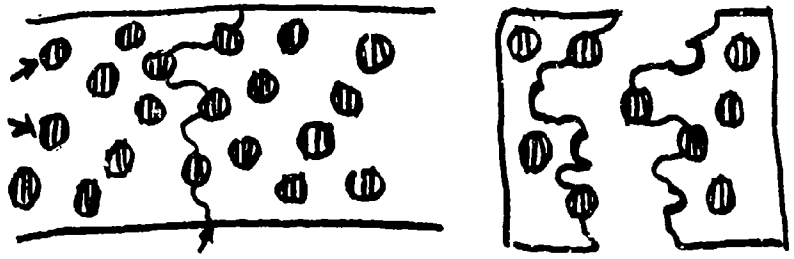


FIGURE 30. SAMPLE NO. M-100-4 X30,000
CHEMISTRY: 73% Al_2O_3, 27% SiO_2
CRYSTAL: Major, γ-Alumina - Minor, Mullite
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS

3. If the features were due to interfacially cracked secondary phases on the replicas, one would expect about half to cast outside shadows and half to remain inside shadows according to the following:



Round microstructural features defined as "pores" were found in all specimens, differing only with respect to size and distribution, but roughly constant in volume within a series. As specimens were fired at progressively higher temperatures, pores coalesced and developed into fewer pores of larger sizes.

X-ray diffraction analyses showed that firing at progressively higher temperatures caused fibers to transform from γ-alumina to mullite crystal structure. It was noted that the average pore size within a particular series was about the same as the mullite crystal size determined by X-ray diffraction line-broadening measurements. Because of this correspondence of sizes it was necessary to investigate the possibility that pores could be mullite crystals.

A hypothesis that pores are a representation of mullite crystals is subject to criticism immediately on the basis that pores also exist in totally γ-alumina structure. If pores represent mullite crystals, they constitute at most about 10 to 20% of the fiber volume. Fibers having complete mullite crystal structure and 10 to 20% pores contain a large volume of mass which is difficult to account for if mullite crystals exist only in the pore space.

The question is clearly resolved by studying etched mullite fibers. Figures 29 and 30 show the effect of increasing the firing temperature, through the γ-alumina to mullite transformation region, on the microstructure of M series fibers. It is seen that relatively homogeneous large grained structure was formed at the higher firing temperature; X-ray diffraction results showed that a major amount of mullite was formed concurrently. Thus the large grains are identified as mullite. Further, a hypothesis that pores are mullite crystals can be dispensed with.

There is a conflict with the grain size measurement of mullite determined by X-ray diffraction line broadening. Micrographic evidence suggests a discrepancy of a factor of about 3. Line-broadening methods, though valuable for comparative study, may not be precise in these cases.

Consider the following comparisons, some shown in Figure 31.

Specimen	XRD	Micrograph	Crystal
B-313-25	600 Å	1500 Å	Mullite
B-314-4	350 Å	800-1000 Å	Mullite
P-100-5	1450 Å	3000 Å	Mullite
M-100-4	----	4000 Å	Mullite
M-103-4 (TEM)	1600 Å	4000 Å	Mullite

X-ray measurements of mullite crystal size are consistently about one third of micrograph measurements. This is probably due to faulting within the mullite crystals, which in effect produces subgrains, each of which is counted as a perfect grain.

It is noted that the tensile strength decreases in all fiber series as the firing temperature is increased. It has been concluded from the microstructures that mullite crystals form in a small grained γ-alumina matrix and that large voids develop from the coalescence of small voids. In series B, high tensile strength was achieved in fibers containing γ-alumina plus mullite crystalline structure. Therefore, it would seem that the void coalescence is important in the control of tensile strength within a series. In series M, P, and B larger pores correlated with lower tensile strength. Reduced tensile strength also correlated with increased crystallite size.

The samples exhibiting the highest modulus properties (B-313-25 and M-102-1B) were fired at temperatures farthest above their respective mullite-transformation temperatures (+450 F and +350 F, respectively). High-temperature firing is apparently necessary for high modulus, but it may be detrimental to tensile properties unless porosity can be eliminated or minimized.

From the above analysis of the electron micrographs, several conclusions appear warranted concerning microstructure of the fibers with respect to their measured properties.

1. The round features present in all fiber series are voids (porosity).
2. Void density (voids per unit area) varies within all series, decreasing with increasing firing temperature.

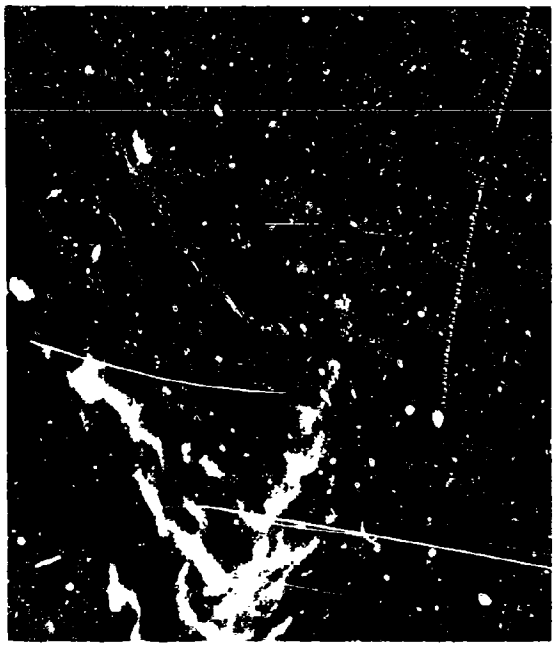


Figure 31 a No. B-314-4
Magnification X10,000
Mullite Crystal Size

X-Ray Diffraction	Micrograph
350 Å	900 Å

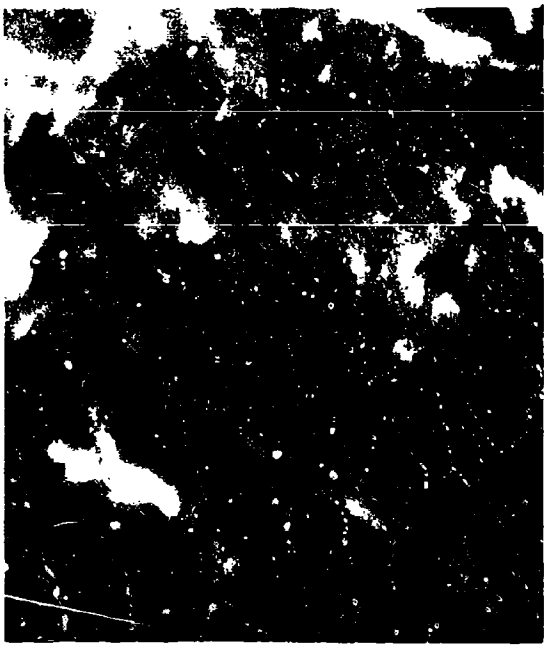


Figure 31 b No. B-313-25
Magnification X30,000
Mullite Crystal Size

X-Ray Diffraction	Micrograph
600 Å	1500 Å

Figure 31 c No. P-100-5
Magnification X20,000
Mullite Crystal Size

X-Ray Diffraction	Micrograph
1450 Å	3000 Å

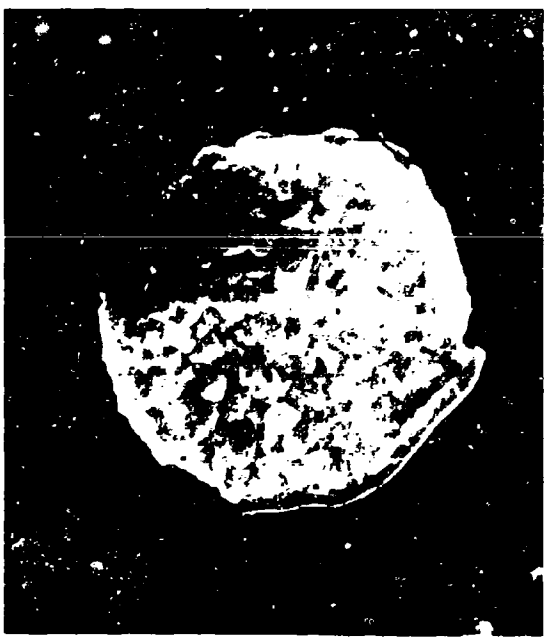


Figure 31 d No. M-100-4
Magnification X10,000
Mullite Crystal Size

X-Ray Diffraction	Micrograph
--	4000 Å

3. Void content (volume percentage) is roughly constant within a series.
4. Small voids coalesce as fibers are fired at increasingly higher temperatures and eventually form large voids in specimens fired at the highest temperature.
5. The void coalescence process is similar in M- and P-series fibers, both of which exhibit roughly the same void density. B-series fibers show less void coalescence.
6. Void coalescence seems to be related to degradation of tensile strength in all series.
7. High modulus was attained in mullite fibers fired at the highest temperature; considerable void coalescence also occurred.

It appears that the attainable modulus of elasticity of the fibers depends only on the crystallites being formed. In all the above cases, the crystallite is a mullite formed from about the same Al_2O_3/SiO_2 ratio, and the modulus of elasticity attained is always in the same 22 to 25 x 10^6 psi range. Tensile strength variations appear to be due to porosity variations both in pore size and pore number. Within a given fiber series the coalescence of many small pores to fewer large pores coincides with a decrease in fiber tensile strength. However, crystallite size also increases with tensile strength decrease and could be more significant than the change in pore size. The B_2O_3 and P_2O_5 additives have been shown to limit mullite crystallite growth and yield fibers with less porosity variation with firing temperature. However, this is not universally true for any arbitrary weight percentage of these additives. An optimum additive level for lessening fiber porosity must be maintained. Deviation from this optimum level results in fibers of increased porosity.

2. Properties of 84 to 97% Al_2O_3 Fibers

From the studies discussed above, the pursuit of a fiber composition having higher modulus of elasticity logically should be directed toward higher Al_2O_3/SiO_2 ratios. In addition, some amount of B_2O_3 or P_2O_5 additive is necessary for porosity and crystallite size control and maintenance of fiber tensile strength.

Tensile strength and modulus of elasticity data vs. firing temperature have been gathered for several fiber compositions of various alumina-to-silica ratios between mullite and pure alumina. These compositions are as follows:

1. 84.2% Al_2O_3, 10% SiO_2, 5.6% B_2O_3
   (40-60 mμ colloidal silica component)

2. 84.8% Al_2O_3, 9.9% SiO_2, 5.8% B_2O_3
   (7 mμ colloidal silica component)
3. 89.4% Al_2O_3, 5.2% SiO_2, 5.4% B_2O_3
4. 92.5% Al_2O_3, 5% SiO_2, 2.5% B_2O_3
5. 95% Al_2O_3, 2.4% SiO_2, 2.5% B_2O_3
6. 97.5% Al_2O_3, 2.5% B_2O_3

Figure 32 shows graphically the fiber property data for the 84.2% Al_2O_3, 10% SiO_2, and 5.8% B_2O_3 composition fibers described in the tabulation below.

Sample No.	Firing Temp. (F)	Tens. Str. (psi*)	Modulus (E) ($psi \times 10^6$*)
B-334-7	1700	207,000	20.0
B-334-4	1800	212,000	22.8
B-334-6	1950	220,000	22.6
B-324-11	2100	219,000	25.5
B-324-10	2150	224,000	25.2
B-324-7	2250	152,000	25.0
B-324-6	2350	67,000	23.5

* Individual fiber properties are shown in Table A.15.

The data show that tensile strengths around 225,000 psi are obtained at 2100 to 2200 F firing temperatures while the modulus of elasticity is around 25 to 26 x 10^6 psi.

The colloidal silica used as a source of silica in all fiber compositions was 40 to 60 mμ in particle size. A smaller colloidal silica size could produce a more homogeneous solution and fiber. This would be more likely at a smaller percentage of silica. A solution was thus prepared having the above composition using a 7 mμ colloidal silica size. Fibers were prepared, fired to various temperatures, and tested for tensile strength and modulus of elasticity. The results are shown in Figure 33 and tabulated below.

FIGURE 32. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 84.2% Al_2O_3, 10% SiO_2, 5.6% B_2O_3 COMPOSITION FIBER.

(40-60 mμ COLLOIDAL SiO_2 SIZE)

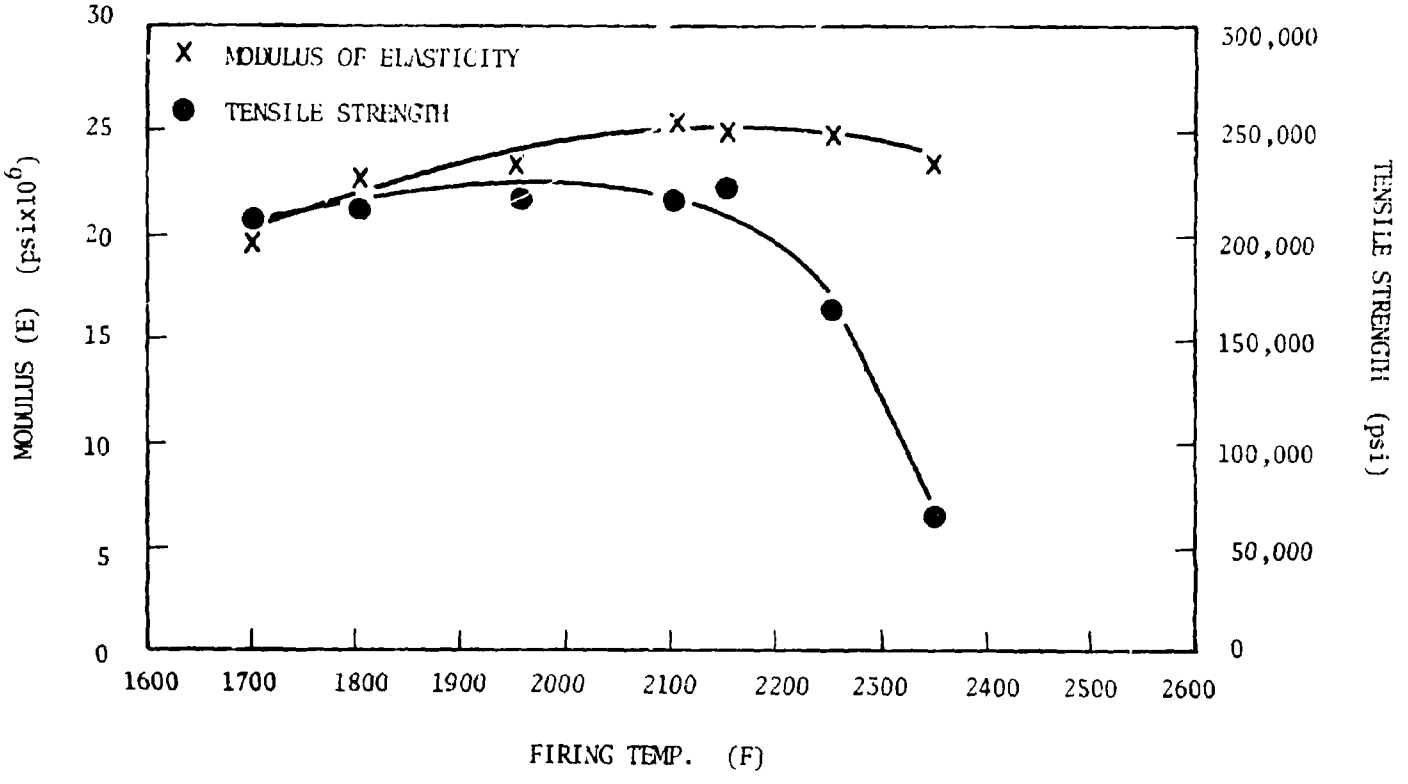
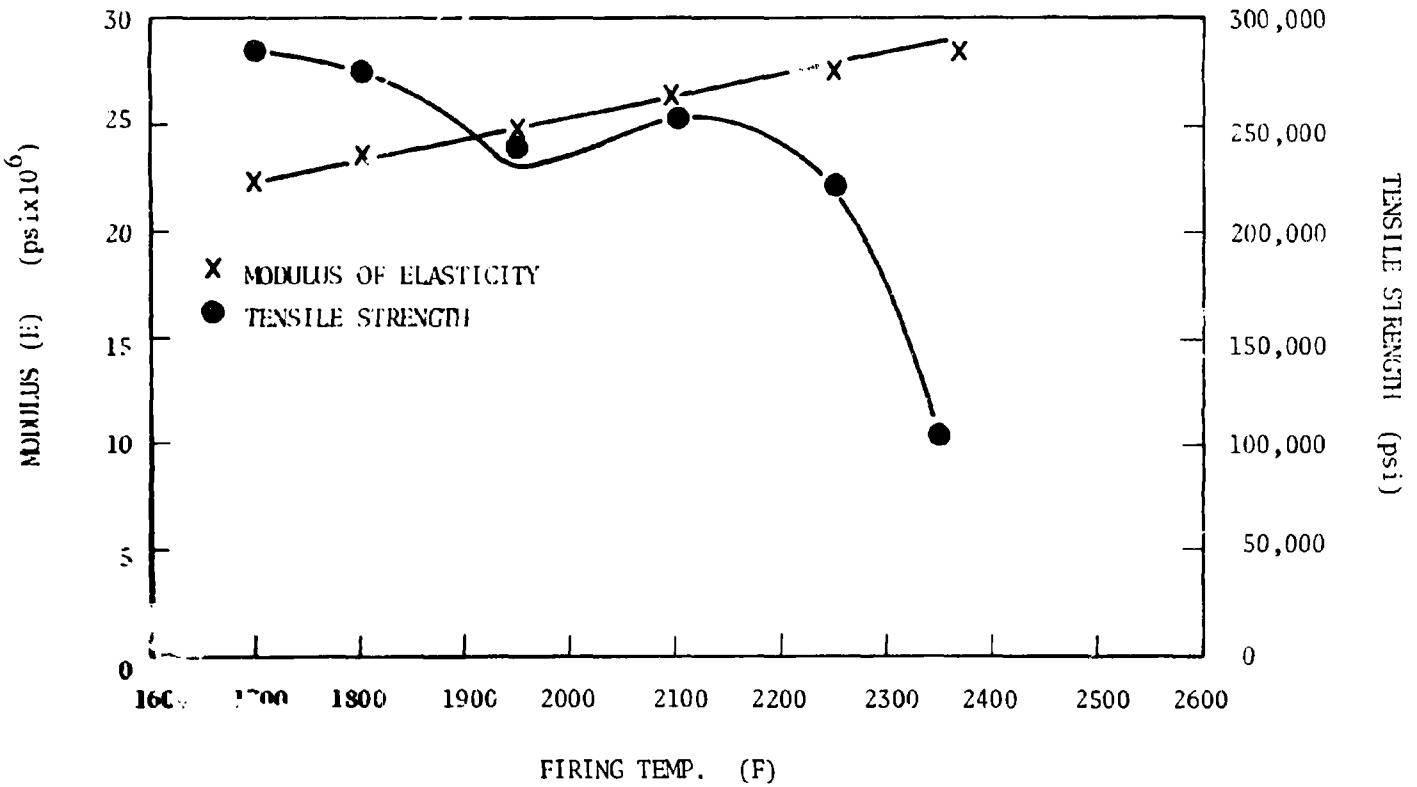


FIGURE 33. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 84.8% Al_2O_3, 9.9% SiO_2, 5.8% B_2O_3 COMPOSITION FIBERS.
(7 mu COLLOIDAL SiO_2 SIZE)



Sample No.	Firing Temp. (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^6*)	X-ray Diffraction Crystallinity		
				Major	Minor	Trace
B-327-3	1700	283,000	22.9	NA**	NA	NA
B-327-6	1800	275,000	23.7	Mullite	-----	-Al_2O_3
B-327-20	1950	240,000	24.5	"	-----	"
B-327-10	2100	256,000	27.6	"	-----	"
B-327-7	2250	221,000	27.5	"	-----	"
B-327-4	2350	103,000	28.5	"	-----	$9Al_2O_3 \cdot 2B_2O_3$

* Individual fiber properties are shown in Table A.16.

** Not available.

A high tensile strength average of 283,000 psi was obtained with an average modulus of elasticity of 22.9 x 10^6 psi at a low firing temperature of 1700 F. The modulus of elasticity approached 28 x 10^6 psi at firing temperatures of 2100 to 2300 F while the tensile strength was maintained around 225,000 psi. Thus, increasing the alumina content of the fibers by 10% at the expense of the silica and going to a source of silica with a smaller particle size brought about a 25 to 30% increase in fiber modulus of elasticity and tensile strength.

Increasing the alumina content of the fibers still further did not result in continuation of this increasing trend in fiber properties. Figures 34 and 35 show the plots of tensile strength and modulus of elasticity vs. firing temperature for an 89.4% alumina fiber and a 92.5% alumina fiber. The average properties of these fibers are tabulated below.

Alumina Content (%)	Sample No.	Firing Temp. (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^6*)
89.4	B-326-1**	1700	132,000	19.1
89.4	B-326-5**	1950	170,000	24.5
89.4	B-326-4**	2100	161,000	23.4
92.5	B-325-10***	1800	177,000	22.9
92.5	B-325-1***	1950	101,000	16.2
92.5	B-325-3***	2100	107,000	16.4

* Individual fiber properties are shown in Table A.17.

** 89.4% Al_2O_3, 5.2% SiO_2 (40-60 mμ size), 5.4% B_2O_3.

*** 92.5% Al_2O_3, 5.0% SiO_2 (40-60 mμ size), 2.5% B_2O_3.

Fiber properties at any firing temperature are relatively poor. In addition, the properties of fibers of these compositions appear to be much more sensitive to firing temperature changes than the preceding compositions.

FIGURE 34. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 89.4% Al_2O_3, 5.2% SiO_2, AND 5.4% B_2O_3 COMPOSITION FIBERS.

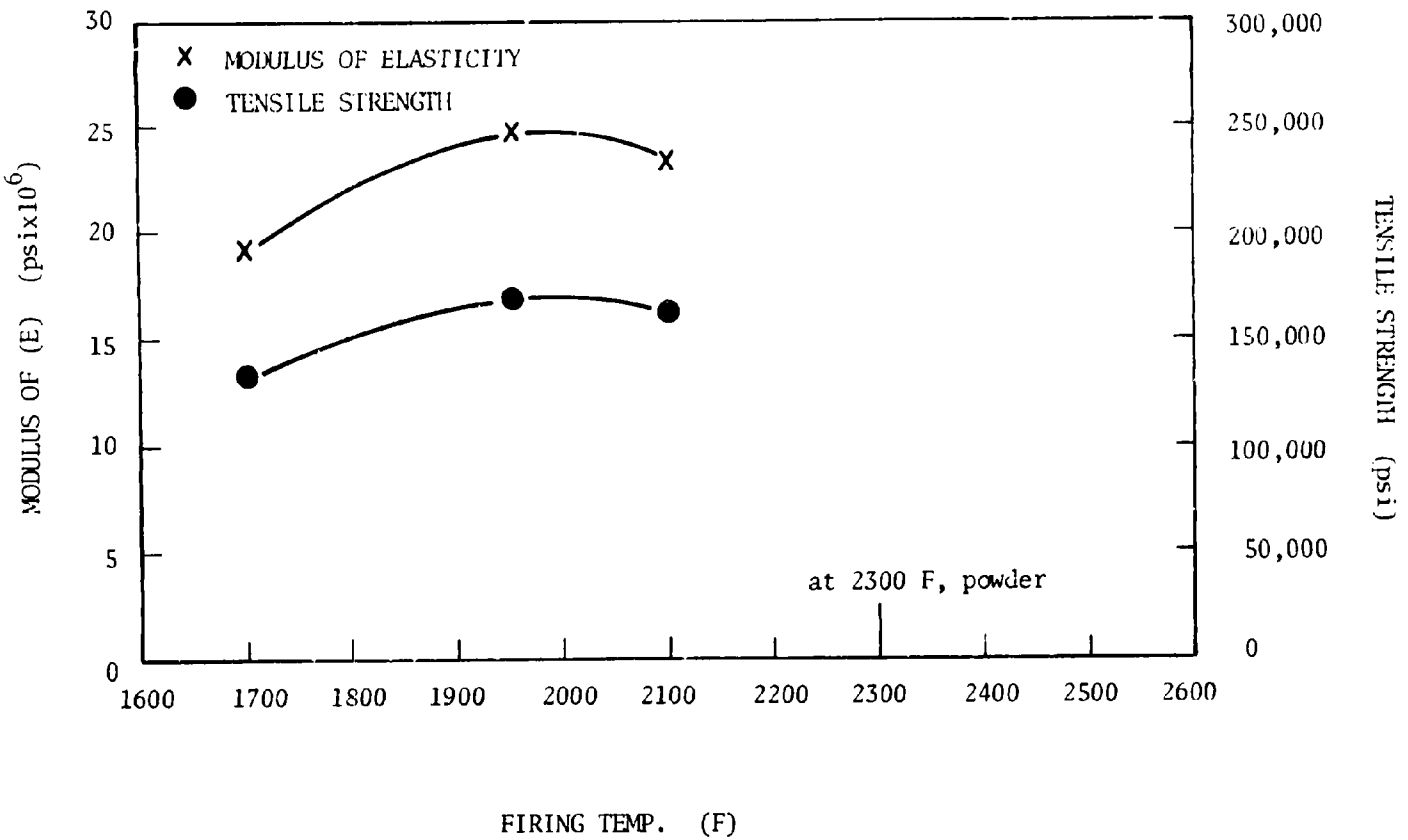
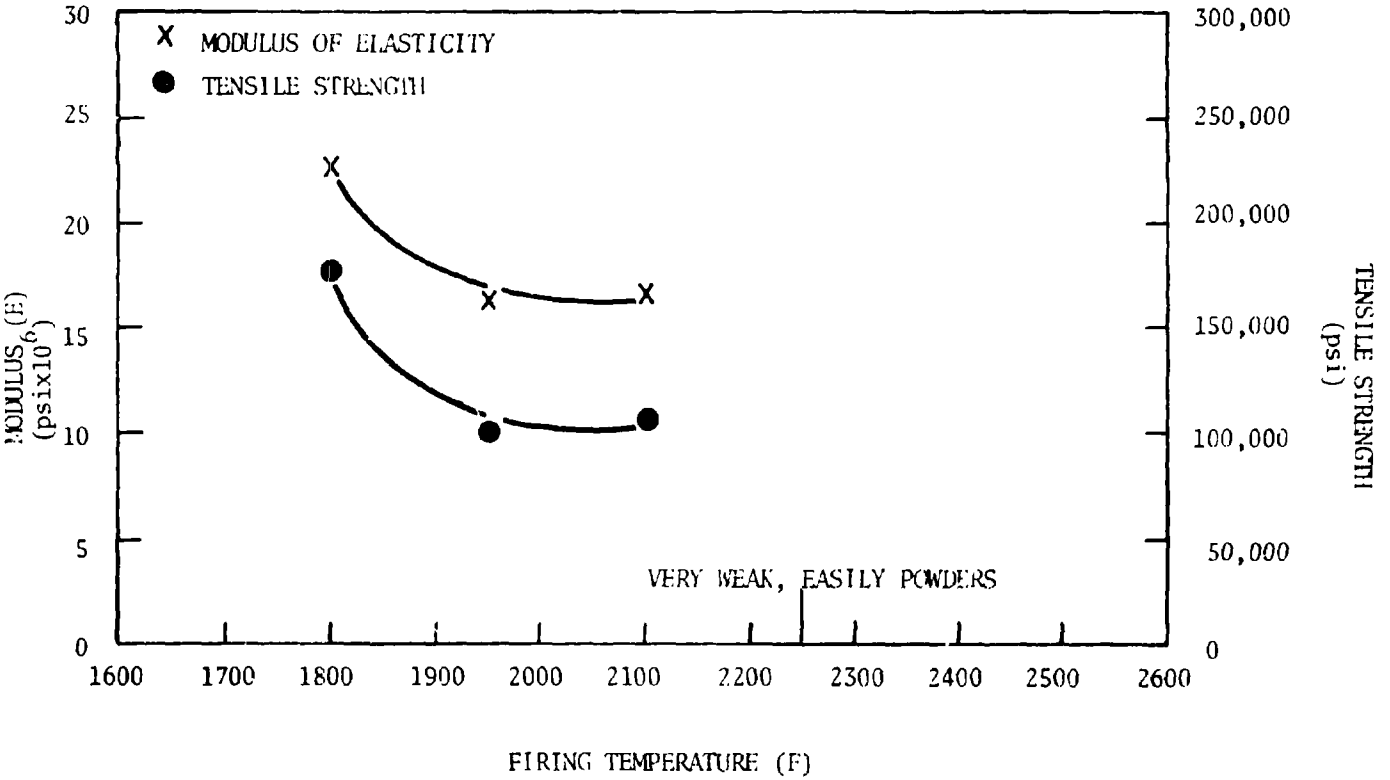


FIGURE 35. AVERAGE TENSILE STRENGTH AND MODULUS OF ELASTICITY VS. FIRING TEMPERATURE OF 92.5% Al_2O_3, 5% SiO_2, 2.5% B_2O_3 COMPOSITION FIBERS.



Fibers of 95% Al_2O_3, 2.4% SiO_2, and 2.5% B_2O_3 composition and 97.5% Al_2O_3 and 2.5% B_2O_3 composition were prepared and fired to temperatures from 1800 to 2100 F with only weak, powdery samples resulting. X-ray diffraction analyses indicated the presence of major or minor amounts of mullite in all these high-alumina samples depending on firing temperature. In addition, the X-ray diffraction analyses picked up some α-Al_2O_3 in the 97.5% alumina composition fiber samples fired to 1950 or 2100 F. An alumina-boria compound could resemble mullite in an X-ray diffraction pattern. Iota-alumina is also possible and is similar to mullite in its X-ray pattern.

Since the fibers became more sensitive to firing temperature changes and became weaker and more powdery with increasing alumina content, increased fiber porosity and crystallite size may be the reasons behind the low properties. These aspects of fiber microstructure were shown in other compositions to have large influences on fiber properties and to depend on the drying and firing conditions.

3. 100% Alumina Fibers

a. Fiber Properties

The drying and firing techniques described in previous sections were applied to several samples of 100% alumina fiber. Periodic kilns were used for firing these fibers either inside crucibles, between thin blankets of insulation fiber, or completely uncovered to temperatures from 1550 to 2100 F.

The resultant fired fibers were universally weak, powdery, and dull in appearance. Photographs of these fibers to 320x magnification showed them to be mostly of a "milky" texture. Occasional glassy, transparent fibers were seen, but even these showed surface defects, which appeared to be air bubbles or notches in the fiber surface.

It was found that carbon (from acetic acid) does not burn out of the 100% alumina fibers until temperatures above about 1200 F are reached. The previously studied fibers (75% Al_2O_3, 20% SiO_2, 5% B_2O_3) lost carbon at about 600 F. Placing the 75% Al_2O_3 unfired fibers directly into a 600 F kiln resulted in the best fired fiber properties. Consequently, some 100% alumina fibers were placed directly into a tube furnace at 1200 F and then fired as tabulated below. These fibers were glassy in appearance and exhibited properties as described below.

Sample No.***	Entrance Temp. (F)	Soak Time at Entrance (min)	Time at 1200 F (min)	Firing Temp. (F)**	Avg. Properties Diam. (μ)	T.S. x10^3	Mod. x10^6
AL-100-7-1-1	1250	20	10	1550	7.6	55	8.3
AL-100-7-2-1	1250	45	10	1575	6.8	99	11.6
AL-100-7-3-1	1250	10	10	1625	6.7	61	12.3
AL-100-7-4-1	1250	10	12	1750	6.8	129	13.5
AL-100-7-5-1	1250	10	20	2100	brittle, powdery		
AL-100-7-6-1	600*	15	12	1550	6.8	91	13.7

* 600 to 1200 F firing time of 15 min with a 10 min soak at 1200 F.

** 10 min soak at firing temperature.

*** Individual fiber properties are shown in Table A.18.

Since the samples fired in the tube furnace were so much better than the periodic kiln samples, one fiber bundle (AL-100-7-6-1) was placed in the tube furnace at 600 F. As the tabulation shows, it too exhibited fair properties.

The firing schedules shown above were applied to more 100% alumina fibers in a periodic kiln. Powdery and brittle samples again resulted at any firing temperature. The maximum properties thus attained for 100% Al_2O_3 fibers were 129,000 psi tensile strength and 13.5 x 10^6 psi modulus of elasticity. These properties were attained on fibers fired to only 1750 F and consequently were γ-Al_2O_3 in structure. All efforts to produce an α-Al_2O_3 structured fiber resulted in weak, powdery fibers which could not be tested. These fibers undoubtedly had a high modulus of elasticity, but the tensile strength was so low that they could not be tested. Typical samples were analyzed by X-ray diffraction and transmission and scanning electron microscopy.

b. Microstructural Analysis

Several of the 100% alumina fiber samples were analyzed to determine the characteristics limiting fiber strength. The samples investigated were:

Sample No.	Firing Temp. (F)	Tens. Str. (avg. psi*)	Modulus (E) (avg. psix10^6*)	Crystalline Const., X-ray Major	Minor	Cryst. Size
AL-100-7-B	1550	too weak to test		γ-Al_2O_3	----	--------
AL-100-7-6-1	1550	91,000	13.7	"	----	--------
AL-100-7-4-1	1750	129,000	13.5	"	----	--------
AL-100-7-5-1	2100	too weak to test		α-Al_2O_3	----	1300 Å

* Individual fiber properties are shown in Table A.18.

AL-100-7-6-1 (fired at 1550 F) contained a high density of small voids (Figure 36). Higher temperature firing did not significantly alter the void distribution within the γ-alumina crystal (AL-100-7-4-1 at 1750 F) although some slight pore coalescence was indicated (Figure 37). Careful inspection of the fiber fracture surface is necessary to pick up the porosity; it is masked to some extent by granular (γ-alumina) boundary markings on the surface.

Granular structure in the γ-alumina shows most clearly in scanning electron micrographs of etched AL-100-7-6-1 (Figure 38) and AL-100-7-4-1 (Figure 39). These micrographs show that higher firing temperature promotes γ-alumina grain growth. The higher tensile strength in AL-100-7-4-1 (Figure 37) seems significant, but the apparent void coalescence and grain growth noted in this specimen fired to 1750 F would seem to preclude a tensile strength higher than that for specimens fired at 1550 F (AL-100-7-6-1, Figure 36). Microstructural differences, as evidenced by relief differences between the micrographs, suggest that other factors are involved.

Specimen AL-100-7-5-1 was fired at 2100 F and thus experienced the γ-α alumina transformation. Electron microscopy shows that it is virtually riddled with porosity (Figure 40). The γ-α alumina transformation involves considerable volume change in the crystallites. AL-100-7-5-1 was so brittle that it could not be mechanically tested. This could be attributed to its porosity and to stresses resulting from the γ-α Al_2O_3 phase change.

Scanning microscopy showed that a fine α-alumina grain size (about four times smaller than that of AL-100-7-4-1) was generated in the AL-100-7-5-1 treatment (Figure 41). X-ray diffraction indicated α-alumina grain size of 1305 Å. From micrographs it was estimated as 1000 Å.

Alumina specimen AL-100-7-B was examined because it exhibited seemingly anomalous properties. It was fired at 1550 F to form γ-alumina (verified by X-ray diffraction) which was so brittle that it could not be tensile tested.

Micrographic examination showed that these fibers (AL-100-7-B) had duplex structure. Fiber core areas were apparently fine grained but certainly extremely porous (Figure 42). Large grains comprised a rim of varying dimensions surrounding the core. Scanning micrographs showed that the core area was extremely susceptible to chemical attack by hydrofluoric acid etchant. Figure 43 shows that the core area varies from about 20 to 80% of the fiber volume and that the γ-alumina grains in the rim are massive. The structure does indeed suggest that AL-100-7-B fibers could exhibit a range of mechanical properties, probably with extreme brittleness. These lower temperature fibers exhibiting microstructural inhomogeneity may have resulted

X30,000

FIGURE 36. SAMPLE NO. AL-100-7-6-1
CHEMISTRY: 100% Al_2O_3
CRYSTAL: γ-Alumina
FIRING TEMPERATURE: 1550 F
TENSILE STRENGTH: 91,000 psi - MODULUS: 13.7×10^6 psi

FIGURE 37. SAMPLE NO. AL-100-7-4-1 X30,000
CHEMISTRY: 100% Al_2O_3
CRYSTAL: γ-Alumina
FIRING TEMPERATURE: 1750 F
TENSILE STRENGTH: 1[illegible]000 psi - MODULUS: 13.5×10^6 psi

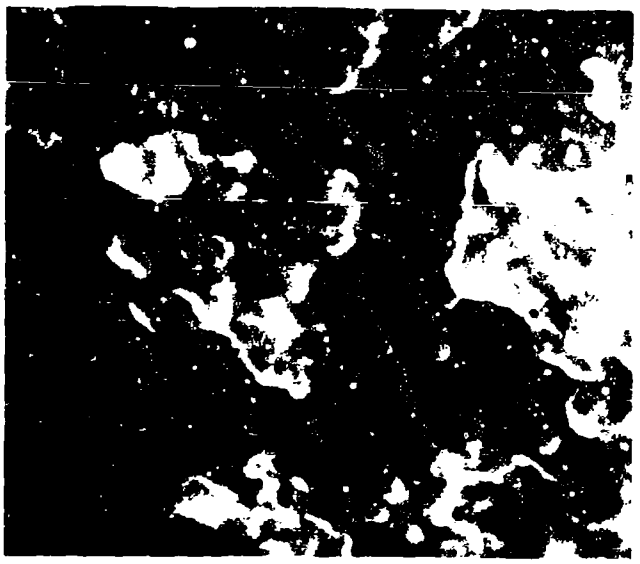


FIGURE 38. SAMPLE NO. AL-100-7-6-1 X30,000
CHEMISTRY: 100% Al_2O_3
CRYSTAL: γ-Alumina
FIRING TEMPERATURE: 1550 F
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS

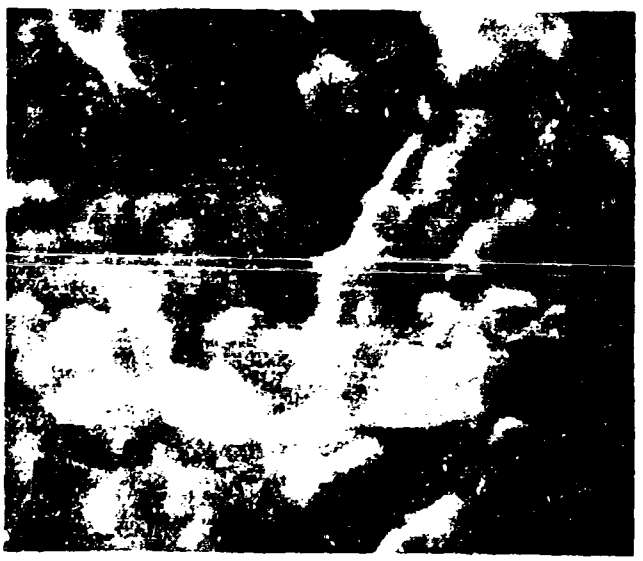


FIGURE 39. SAMPLE NO. AL-100-7-4-1 X30,000
CHEMISTRY: 100% Al_2O_3
CRYSTAL: γ-Alumina
FIRING TEMPERATURE: 1750 F
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS

from small differences in the processing steps.

From all the above information, it is concluded that the γ-α Al_2O_3 phase transformation stresses the fiber excessively and causes an extremely weak fiber.

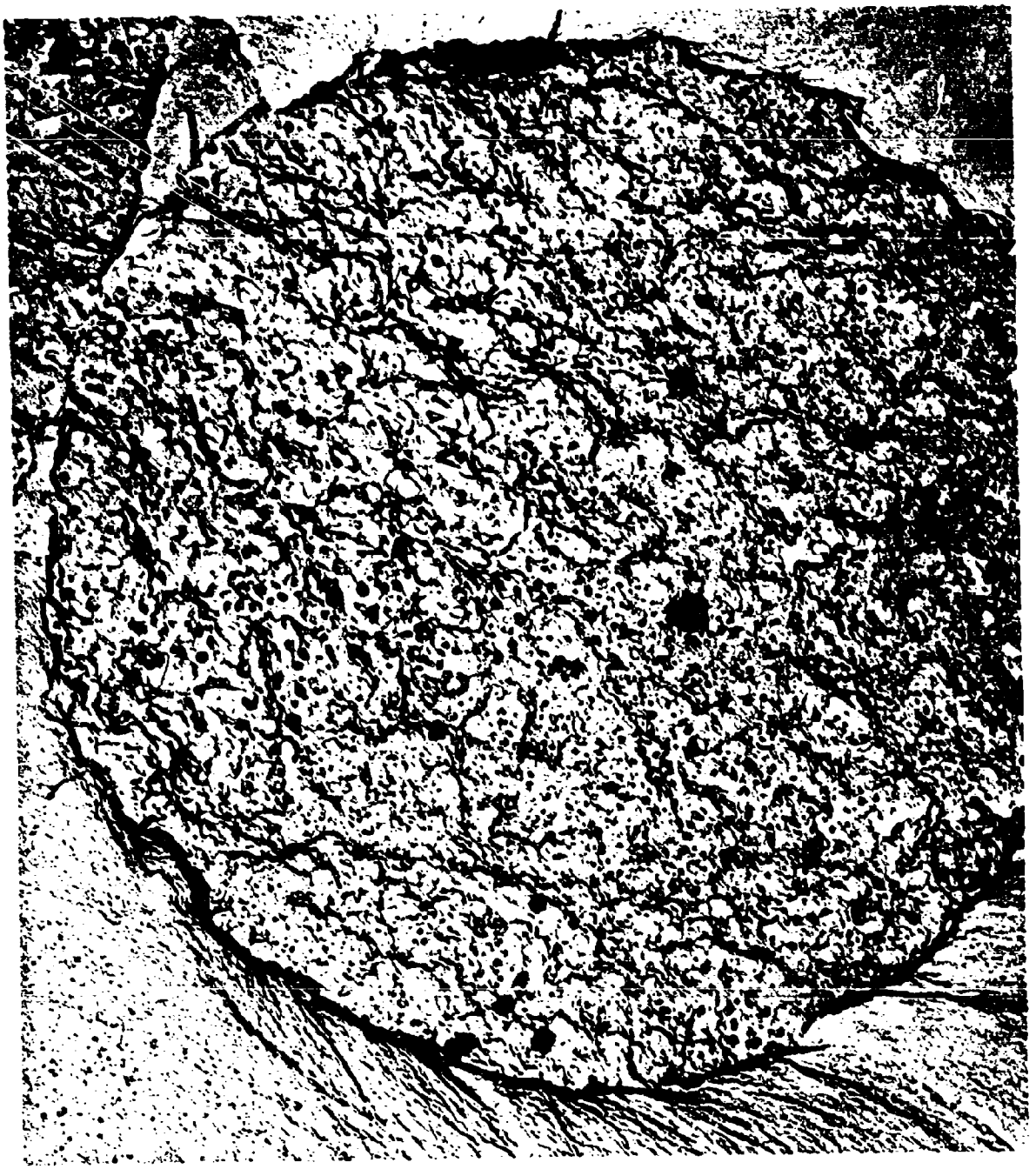


FIGURE 40 Sample No. Al-100-7-5-1 X30,000
Chemistry: 100% Al_2O_3
Crystal: α-Al_2O_3
Firing Temperature: 2100 F
Not Possible to Run Mechanical Tests Due to Brittleness of Samples.

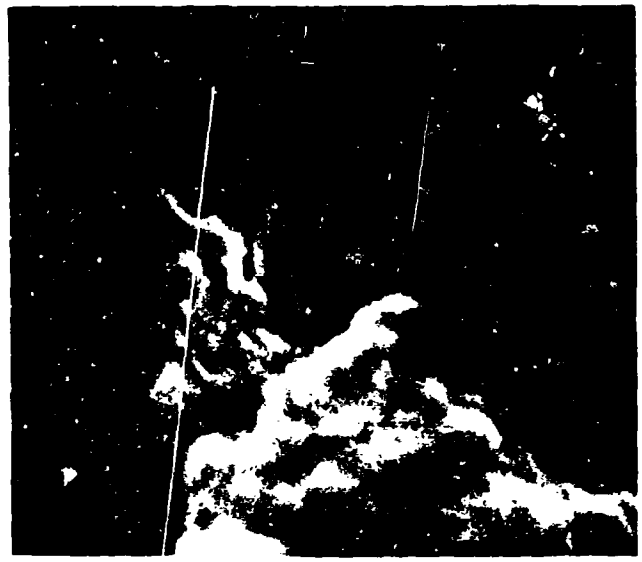


FIGURE 41. SAMPLE NO. AL-100-7-5-1 X30,000
CHEMISTRY: 100% Al_2O_3
CRYSTAL: α-Al_2O_3
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS

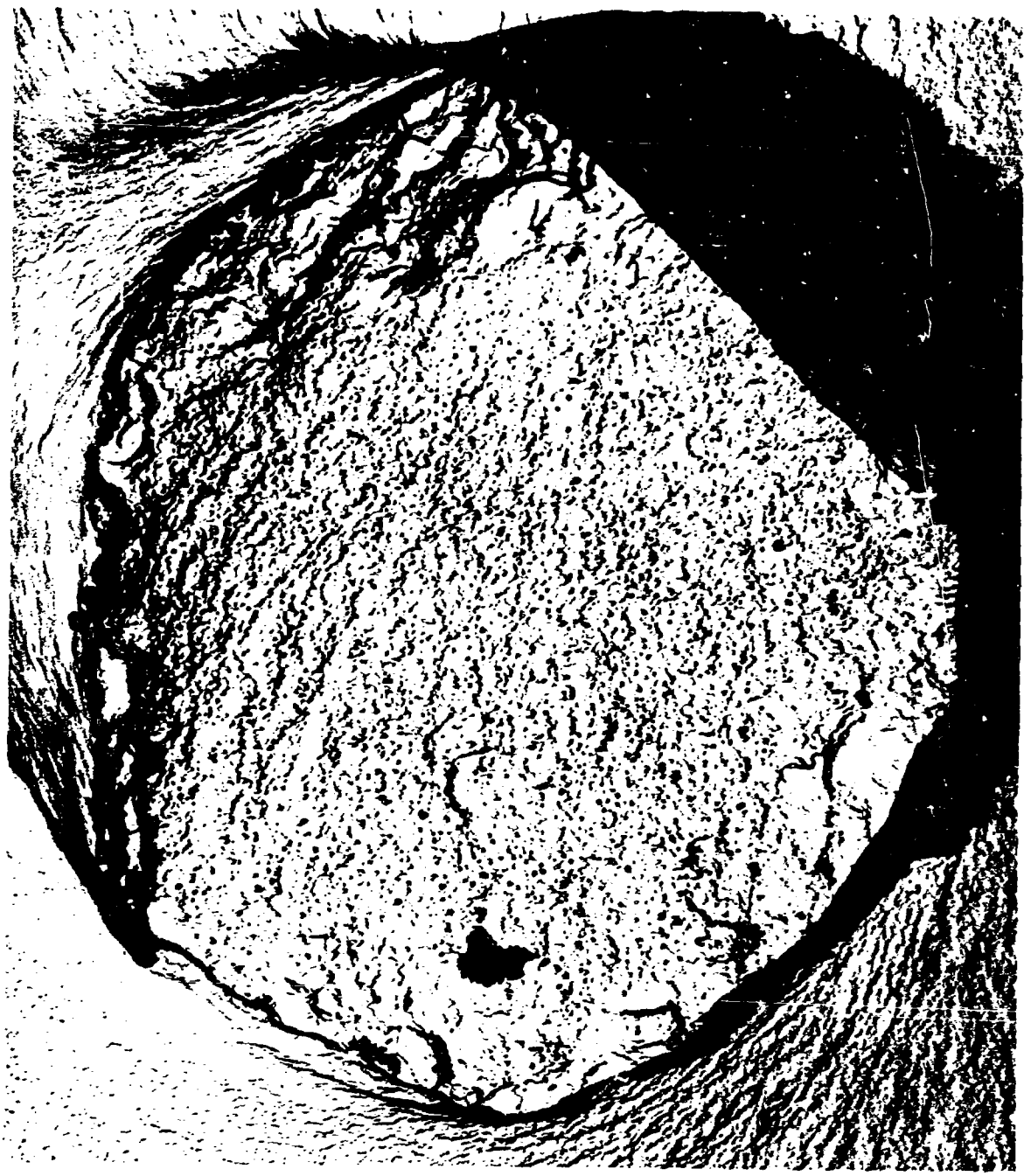
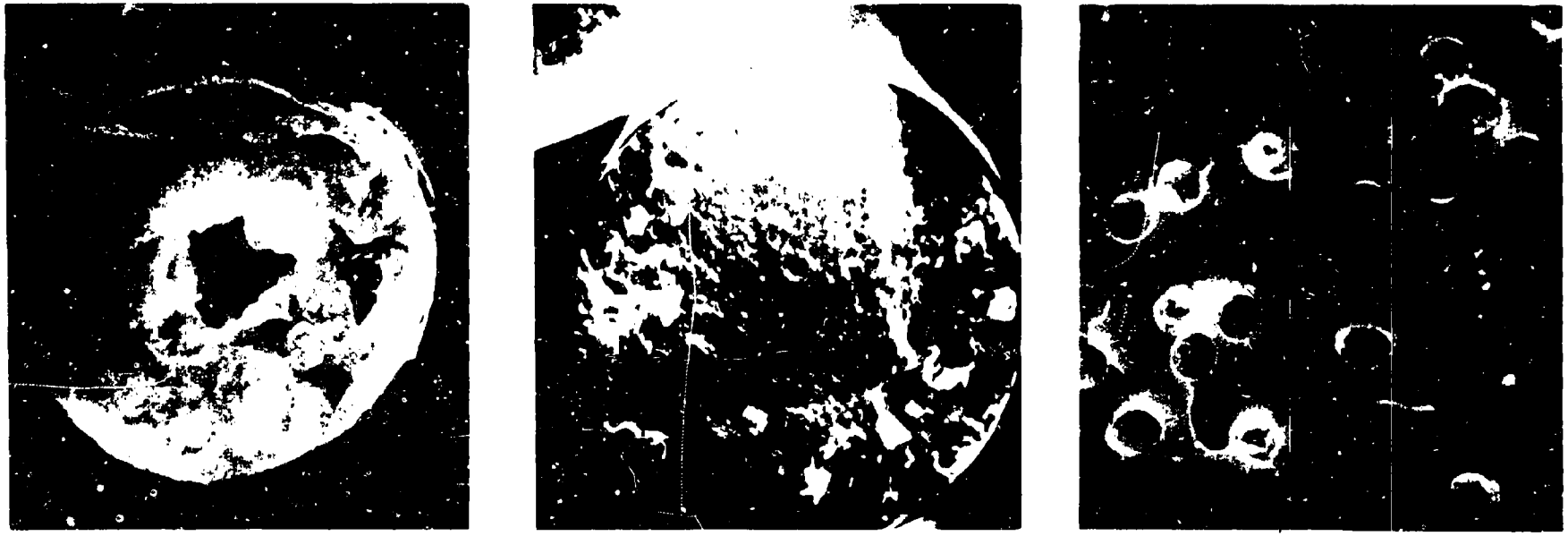


FIGURE 42. SAMPLE NO. AL-103-7-B X30,000
CHEMISTRY: 100% Al_2O_3
CRYSTAL: γ-Alumina
FIRING TEMPERATURE: 1550 F

NOT POSSIBLE TO RUN MECHANICAL TESTS DUE TO BRITTLENESS OF SAMPLES.



a. b. c.

FIGURE 43. SAMPLE NO. AL-100-7-B
ETCHED IN 10% HF (52%) AND H_2O FOR 24 HOURS
a. X10,000 CORE ETCHED OUT
b. X10,000 UNETCHED
c. X1000 IRREGULAR ETCHING BEHAVIOR

SECTION V

SUMMARY OF RESULTS

The first phase of the research for this contract was a process study of the fiber drying and firing operations. The degree of control and understanding of fiber drying and firing led to 200,000 psi tensile strength and 21 x 10^6 psi modulus of elasticity routinely and reproducibly in constant 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition fibers. Two firing techniques were developed as follows:

	Firing Technique 1		Firing Technique 2	
Firing Stage	Firing Time*	Soak Time at Upper Temp.	Firing Time*	Soak Time at Upper Temp.
Room Temp. to 600 F	0**	1 hr	0**	1 hr
600 to 1200 F	0.5 hr	0	3 hr	0
1200 to 1950 F	50 min	0.5 hr	50 min	0.5 hr

* Uniform firing rate.

** Fibers are placed in periodic kiln maintained at 600 F.

Fibers are extruded from solutions which contain 34% solids (percentage of ash after 1400 F firing). The extrusion conditions cause drying of the collected fibers to 45% solids content. These fibers can be stripped immediately from the collection wheel and fired by either of the two methods described above or they can be prepared for firing in either of the following two ways:

1. Fibers can be stored to an equilibrium weight loss (~16% in 24 hours) over magnesium perchlorate and then must be fired according to Technique 2 to obtain reproducible 200,000 psi tensile strength and 20 to 22 x 10^6 psi modulus of elasticity.
2. Fibers can be immediately placed in a kiln at 600 F and soaked for 1 to 24 hours at 600 F before being fired by either of the two methods.

Variations in storage time either at 600 F or over magnesium perchlorate were not found to influence fiber properties.

Electron microscope analyses showed that porosity in the fibers was dependent on the fiber drying and firing steps. In addition, fiber tensile strength was qualitatively shown to be inversely proportional to percent porosity. The modulus of elasticity appears to be independent of percent porosity. No significant correlation of fiber properties with fiber diameter in the range of 4.5 to 7 microns was found. It was further determined that a firing temperature increase to 2400 F over the "standard" 1950 F caused a fiber (75% Al_2O_3, 20% SiO_2, and 5% B_2O_3

composition) modulus increase to about 25 x 10^6 psi. However, the tensile strength correspondingly dropped to about 150,000 psi.

A modulus of elasticity of 20 to 25 x 10^6 psi was felt to be the practical limit of attainment for the 75% Al_2O_3, 20% SiO_2, and 5% B_2O_3 composition fiber. Thus, when drying and firing were sufficiently defined to enable routine attainment of modulus values in this range, other fiber compositions were subsequently studied as the second research phase of this contract.

Tensile strength and modulus of elasticity were obtained for a variety of mullite fiber compositions (Al_2O_3/SiO_2 weight ratios of about 3:1). The principal variables studied were B_2O_3 and P_2O_5 additive level (0 to 10%) and firing temperature (1700 to 2500 F). Although this composition study resulted in fibers of varying tensile strength and modulus of elasticity, in no case did these equal or exceed the values previously attained, 200,000 psi tensile strength and 20 to 25 x 10^6 psi modulus of elasticity. Electron microscopy and X-ray diffraction analyses were applied to these fibers to determine and correlate additive level and firing temperature with fiber characteristics responsible for observed tensile strength and modulus of elasticity variations.

Electron microscope analysis showed fiber volume percent porosity to be 10 to 20% regardless of fiber composition or firing temperature as long as the drying-firing techniques described above were used. However, it was noted that, as firing temperature increased and tensile strength decreased, pore size increased, pore number decreased, and crystallite size increased. The modulus of elasticity increased with firing temperature, as did the amount of crystalline mullite being formed at the expense of γ-Al_2O_3.

An optimum level of B_2O_3 (5 to 6%) was found to have three effects on the physical characteristics of a fired fiber:

1. Mullite crystallite growth is inhibited by the presence of B_2O_3 (500 Å vs. 1600 Å for 5% and 0% B_2O_3 containing fibers respectively).
2. The B_2O_3 causes the chemical reaction yielding crystalline mullite from γ-Al_2O_3 and silica to occur at lower temperatures.
3. The 5 to 6% B_2O_3 appears to stabilize the porosity change vs. firing temperature. That is, the change in pore size and pore numbers with a change in firing temperature is less in B_2O_3 containing fibers than in fibers without B_2O_3 even though the volume percent porosity is nearly identical in all cases.

The fiber composition study was extended to increased alumina content fibers. Compositions with alumina contents from 75 to 95% with the balance being some proportion of silica and boric oxide were studied in addition to the 100% alumina fibers.

Maximum fiber properties of 130,000 psi tensile strength and 14 x 10^6 psi modulus of elasticity were obtained from 100% alumina fibers fired to 1750 F. These 100% alumina fibers contained only γ-Al_2O_3 as the crystalline phase. Fibers with more than 85% but less than 100% alumina content were obtained. However, these fibers had less than 200,000 psi tensile strength and less than 24 x 10^6 modulus of elasticity. All attempts to produce a fiber containing the α-Al_2O_3 phase giving high modulus of elasticity yielded only weak, powdery fibers. Electron micrographs qualitatively showed the weak α-Al_2O_3 fibers to have a higher volume percentage of porosity and larger crystallite size than γ-Al_2O_3 fibers or other composition fibers studied. It was concluded that the γ-Al_2O_3 to α-Al_2O_3 phase change resulted in increased porosity, rapid α-Al_2O_3 grain growth, and microstructural stresses.

The maximum fiber properties attained during this study were exhibited by an 85% alumina, 10% silica, and 5% B_2O_3 composition fiber. Tensile strengths from 225,000 to 250,000 psi were obtained in fibers fired to 2100 to 2250 F. Moduli of elasticity in this firing temperature range are routinely 25 to 28 x 10^6 psi. A maximum tensile strength of 280,000 psi and a modulus of elasticity of 22 x 10^6 psi resulted for fiber fired at about 1700 F.

SECTION VI
CONCLUSIONS

A review of the work carried out during this contract leads to several conclusions. First, the composition of 85% Al_2O_3, 10% SiO_2, and 5% B_2O_3 produces an optimum mix of mullite and γ-Al_2O_3 over a convenient firing temperature range. That is to say that, using specific drying and firing techniques, the best combination of tensile strength and modulus of elasticity is obtained with this composition. It is further recognized that porosity reductions from the current 10 to 20 volume percent would be difficult if not impossible to achieve and would probably provide only about 30×10^6 psi modulus of elasticity and 250,000 to 300,000 psi tensile strength in the 85 to 90% Al_2O_3 fiber compositions.

Further work in firing 90 to 95% Al_2O_3 fibers could increase the modulus of elasticity, but attaining values above 35×10^6 psi would be questionable.

It has become apparent that a 95 to 100% α-Al_2O_3 fiber is necessary to achieve a modulus of elasticity greater than 40×10^6 psi. In every instance where α-Al_2O_3 was present in a fiber, that fiber was weak and powdery. This weakness is attributed to stresses developed within the fiber during the γ-Al_2O_3 to α-Al_2O_3 phase change and to rapid and excessive α-Al_2O_3 grain growth. The weakness of α-Al_2O_3 containing fibers could also be due to porosity, which results from the process as well as being introduced during the phase change.

Tensile strength of salt decomposition fibers is inversely dependent on volume percent porosity contained in the fibers. The porosity is built into the fibers during the drying and firing operations. Tensile strength is also inversely dependent on crystallite size which is directly dependent on firing temperature.

The modulus of elasticity depends on the crystalline phase of the fibers. Both quantity of crystalline phase and composition of that crystalline phase are important to the fiber modulus of elasticity. The maximum quantity of crystalline fiber component and thus the maximum modulus of elasticity for that fiber composition is achieved at higher firing temperatures. However, the higher firing temperatures also result in crystallite growth which lowers fiber tensile strength.

The economics of the process were not reviewed in any more depth than described in the initial work of 1967[1]. The experience gained since then has not altered the analysis. It is anticipated that the fiber would be in the range of $4/lb when made in commercial quantities.

SECTION VII
RECOMMENDATIONS

Polycrystalline oxide fiber with a modulus of elasticity in excess of 40×10^6 psi must consist of 95 to 100% Al_2O_3. Moreover the fiber must be fired high enough to yield alumina in the alpha form. Controlling this conversion to alpha alumina without loss of strength in the fiber is the principal problem. Its solution could result from one or more of the following recommended future approaches:

1. Establish precisely the inversion temperature of the gamma-alpha system. Precisely control the temperature of the fiber near this inversion point and its exposure time to this temperature. This treatment should minimize grain growth as well as relieve stresses within the fiber.
2. Fire the fibers as in (1) above and induce densification through control of furnace atmosphere and sintering aids.
3. Inhibit grain growth of the alpha form through chemical treatment of the gamma form "precursor" fiber.
4. Seed the fiberizable solutions to induce the formation of α-alumina directly from the salts.

Future work should also consider the feasibility of using the polycrystalline alumina fiber as the starting material or "precursor" for conversion to a continuous single crystal α-alumina fiber.

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5. K.G. Skinner, "A Differential Thermal Analysis Apparatus for Temperatures up to 1575 C", Naval Research Laboratory, Report 4942.

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APPENDIX A

INDIVIDUAL FIBER PROPERTIES

TABLE A.1

INDIVIDUAL FIBER DATA FOR FIBERS
FIRED AT VARIOUS RATES TO 1850 F

Test Data: Instron Tester, 1-inch gage length, Cell A corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 75% Al_2O_3, 20% SiO_2, 5% B_2O_3.

Sample No.	Firing Rate (F/hr)		Modulus (E) ($psi \times 10^6$)	Tens. Str. (psi)	Diameter (microns)
B-299-1A	2500		20.1	80,000	6.3
			21.0	160,000	5.5
			21.2	72,000	5.6
			19.4	215,000	5.9
			19.0	127,000	5.9
			19.0	79,000	6.0
			18.9	170,000	6.3
			18.4	184,000	5.6
			19.9	127,000	6.0
			23.0	152,000	5.4
		Avg.	20.0	137,000	5.9
		Std. Dev.	1.4	49,500	0.3
B-299-3A	2500		19.8	192,000	5.5
			19.6	63,000	5.4
			25.5	71,000	5.0
			20.4	89,000	5.7
			23.3	207,000	5.3
			19.6	92,000	5.7
			19.1	110,000	5.6
			18.8	108,000	5.8
			19.3	129,000	5.5
			20.0	94,000	5.6
		Avg.	20.5	118,000	5.5
		Std. Dev.	2.1	52,300	0.2
B-200-6A1	2500		20.7	148,000	4.9
			20.2	69,000	4.9
			20.2	206,000	5.3
			20.6	177,000	5.1
			19.9	225,000	5.5
			19.5	232,000	5.4
			19.5	148,000	5.1
			19.8	132,000	5.5
			21.3	195,000	5.7
			20.4	213,000	5.0
		Avg.	20.2	175,000	5.3
		Std. Dev.	0.54	50,700	0.3

TABLE A.1 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus (E) ($psix10^6$)	Tens. Str. (psi)	Diameter (microns)
B-299-7A	2500	20.7	68,000	5.5
		19.0	116,000	5.5
		21.4	115,000	5.8
		21.9	145,000	6.6
		20.2	107,000	5.1
		21.0	236,000	4.8
		21.1	117,000	5.3
		19.8	113,000	5.0
		23.0	254,000	5.6
		21.3	199,000	5.1
		Avg. 20.9	147,000	5.4
		Std. Dev. 1.1	6,100	0.5
B-299-8A	2500	20.3	143,000	5.7
		20.5	179,000	5.6
		21.6	120,000	5.0
		23.4	236,000	5.1
		21.4	193,000	6.1
		20.0	78,000	5.3
		22.3	91,000	5.1
		19.9	176,000	5.6
		19.7	147,000	5.4
		21.9	155,000	5.5
		Avg. 21.1	152,000	5.4
		Std. Dev. 1.2	47,500	0.3
B-299-9A	2500	18.8	145,000	5.0
		20.5	92,000	4.9
		20.1	200,000	5.9
		20.2	78,000	4.9
		22.3	175,000	5.2
		17.8	141,000	5.2
		20.0	198,000	5.8
		20.3	152,000	4.8
		20.1	171,000	5.1
		22.4	168,000	4.9
		Avg. 20.3	152,000	5.0
		Std. Dev. 1.4	40,500	0.4

TABLE A.1 (Cont'd.)

Sample No.	Firing Rate (F/hr)		Modulus (E) ($psix10^6$)	Tens Str. (psi)	Diameter (microns)
B-299-10A1	2500		19.6	151,000	6.3
			18.1	208,000	6.2
			19.7	117,000	5.4
			20.3	185,000	6.0
			18.7	199,000	5.8
			20.1	187,000	5.9
			18.3	170,000	5.4
			19.3	193,000	5.3
			22.6	183,000	5.6
			20.3	98,000	6.8
		Avg.	19.7	169,000	5.9
		Std. Dev.	1.3	36,300	0.5
B-299-11A	2500		19.4	123,000	5.2
			20.0	232,000	4.8
			20.9	199,000	5.2
			20.6	245,000	5.3
			21.2	249,000	5.5
			19.4	195,000	5.1
			20.3	195,000	5.0
			22.2	174,000	5.1
			20.9	206,000	6.0
			25.1	238,000	5.0
		Avg.	21.0	206,000	5.2
		Std. Dev.	1.7	38,000	0.2
B-299-11B	5000		19.3	179,000	5.5
			21.5	68,000	5.2
			19.8	206,000	5.6
			22.9	224,000	5.2
			19.2	186,000	6.0
			18.9	180,000	5.4
			19.5	213,000	5.5
			19.3	190,000	5.6
			19.5	76,000	5.6
			20.1	218,000	5.2
		Avg.	20.0	174,000	5.5
		Std. Dev.	1.3	56,000	0.2

TABLE A.1 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus$_6$(E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-299-11C	2500	20.3	161,000	5.6
		19.4	185,000	5.6
		21.9	215,000	5.8
		20.6	185,000	6.0
		18.5	198,000	5.6
		20.1	229,000	5.0
		20.0	239,000	5.6
		20.5	188,000	4.8
		20.1	224,000	5.9
		24.7	199,000	5.0
		Avg. 20.6	202,000	5.5
		Std. Dev. 1.7	24,000	0.4
B-299-11D	5000	21.0	206,000	5.2
		20.4	144,000	5.7
		23.3	216,000	4.9
		22.7	147,000	5.4
		22.2	151,000	6.1
		17.2	69,000	5.5
		21.2	131,000	5.6
		20.7	199,000	5.6
		21.5	177,000	5.5
		20.7	177,000	5.4
		Avg. 21.1	162,000	5.6
		Std. Dev. 1.7	43,000	0.4
B-299-12A	5000	21.2	178,000	5.6
		19.9	202,000	6.4
		21.4	184,000	6.0
		20.4	208,000	5.2
		19.4	177,000	4.9
		21.9	157,000	5.3
		18.5	159,000	5.0
		19.7	201,000	5.5
		20.9	154,000	5.0
		19.4	189,000	5.3
		Avg. 20.3	181,000	5.4
		Std. Dev. 1.1	20,000	0.5

TABLE A.1 (Cont'd.)

Sample No.	Firing Rate (F/hr)		Modulus$_6$(E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-299-12B	5000		22.4	110,000	4.6
			18.8	159,000	5.3
			19.3	114,000	5.1
			20.9	157,000	9.9
			22.2	161,000	5.0
			21.6	197,000	5.2
			17.6	114,000	5.4
			22.7	195,000	4.6
			19.9	244,000	5.1
			20.5	220,000	5.3
		Avg.	20.6	167,000	5.5
		Std. Dev.	1.7	46,500	1.6
B-299-12C	2500		19.4	203,000	5.6
			20.7	253,000	5.2
			21.4	197,000	4.7
			21.4	230,000	6.4
			18.3	129,000	9.1
			18.2	228,000	5.2
			19.9	184,000	5.3
			18.7	201,000	5.3
			19.7	232,000	5.2
			18.0	132,000	5.1
		Avg.	19.6	199,000	5.7
		Std. Dev.	1.3	41,000	1.3
B-299-12D	5000		19.8	110,000	5.5
			20.4	195,000	4.9
			19.2	181,000	5.6
			20.8	162,000	5.5
			21.6	116,000	4.5
			23.3	96,000	5.0
			21.2	244,000	5.1
			20.4	157,000	4.8
			20.4	225,000	5.0
			20.3	180,000	4.8
		Avg.	20.7	167,000	5.1
		Std. Dev.	1.1	49,000	0.4

TABLE A.2

INDIVIDUAL FIBER DATA FOR SAMPLES
FIRED AT VARIOUS RATES TO 1950 F

Test Data: Instron Tester, 1-inch gage length, Cell A corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 75% Al_2O_3, 20% SiO_2, 5% B_2O_3.

Sample No.	Firing Rate (F/hr)	Modulus (E) ($psix10^6$)	Tens. Str. (psi)	Diameter (microns)
B-299-2-13	25	20.3	155,000	5.6
		21.7	197,000	5.9
		18.7	128,000	5.7
		20.0	161,000	5.6
		21.3	206,000	5.8
		21.0	129,000	5.5
		20.8	145,000	5.7
		19.9	75,000	5.9
		21.4	50,000	5.6
		18.9	181,000	5.6
	Avg.	20.4	143,000	5.7
	Std. Dev.	1.0	14,300	0.1
B-299-4B	25	18.3	125,000	5.9
		19.3	90,000	5.5
		18.7	171,000	5.5
		18.3	168,000	5.8
		18.7	178,000	5.7
		17.1	116,000	5.8
		18.0	144,000	5.6
		17.1	150,000	5.7
		18.2	201,000	5.6
		16.4	158,000	5.8
	Avg.	18.0	150,000	5.7
	Std. Dev.	0.9	32,000	0.1
B-299-1B	300-500	17.5	64,000	5.8
		----	13,000	6.1
		16.5	73,000	5.8
		19.5	80,000	5.9
		15.3	37,000	6.1
	Avg.	13.8	53,000	5.9
	Std. Dev.	7.9	28,000	0.2

TABLE A.2 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus (E) ($psix10^6$)	Tens. Str. (psi)	Diameter (microns)
B-305-8B	300-500	14.4	91,000	6.1
		20.0	121,000	5.8
		19.0	168,000	6.2
		17.0	79,000	6.3
		19.2	148,000	5.9
		18.3	70,000	5.9
		12.0	138,000	6.4
		17.5	153,000	6.0
		17.1	137,000	6.1
		18.6	181,000	5.7
	Avg.	17.3	127,000	6.0
	Std. Dev.	2.4	37,000	0.2
B-305-9B	300-500	20.2	148,000	5.9
		18.4	126,000	5.6
		20.4	159,000	5.4
		21.9	138,000	5.7
		21.0	133,000	6.0
		20.0	165,000	5.6
		21.5	225,000	5.6
		22.2	162,000	5.6
		19.9	201,000	5.7
		20.7	197,000	5.6
	Avg.	20.5	165,000	5.7
	Std. Dev.	1.0	33,000	6.2
B-299-2A	2500	19.3	110,000	6.1
		26.4	179,000	5.9
		21.1	98,000	5.4
		21.0	172,000	5.7
		20.8	188,000	5.7
		19.1	78,000	5.7
		19.6	182,000	5.8
		19.0	44,000	5.8
		19.3	203,000	5.7
		18.9	200,000	5.9
	Avg.	20.5	145,000	5.8
	Std. Dev.	2.3	57,000	0.2
B-299-4A	2500	17.0	122,000	5.8
		20.8	206,000	5.8
		23.9	117,000	6.2
		18.9	150,000	5.6
		19.3	146,000	5.6
		19.1	188,000	5.4
		22.6	107,000	5.3
		20.0	170,000	5.6
		12.2	183,000	5.5
		19.6	153,000	5.5
	Avg.	19.4	154,000	5.6
	Std. Dev.	3.0	33,000	0.3

TABLE A.2 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus$_6$(E) (psix10^6)	Tens. Str. (psi)	Diameter (microns
B-299-5-4	2500	16.7	153,000	5.9
		18.8	188,000	5.1
		25.3	87,000	6.5
		21.6	163,000	5.7
		16.9	181,000	4.6
		20.1	186,000	4.9
		19.9	156,000	5.4
		22.4	126,000	9.7
		19.1	168,000	7.8
		21.4	219,000	5.0
	Avg.	20.2	163,000	5.6
	Std. Dev.	3.0	36,000	1.0
B-299-6A	2500	19.7	144,000	5.5
		17.9	167,000	5.5
		20.3	189,000	4.6
		20.6	212,000	5.6
		21.0	199,000	5.4
		19.5	187,000	5.2
		21.2	183,000	5.4
		20.2	203,000	5.2
		19.2	213,000	5.3
		20.4	173,000	5.2
	Avg.	20.0	187,000	5.3
	Std. Dev.	1.0	22,000	0.3
B-299-7-A-1	2500	20.0	219,000	5.9
		20.8	93,000	5.7
		20.4	120,000	5.0
		19.3	155,000	5.0
		22.3	169,000	7.0
		19.4	108,000	5.0
		19.9	152,000	5.1
		18.6	210,000	5.4
		20.2	156,000	5.6
		18.2	144,000	5.3
	Avg.	19.7	152,000	5.5
	Std. Dev.	1.2	39,000	0.6

TABLE A.2 (Cont'd.)

Sample No.	Firing Rate (F/hr)		Modulus (E) ($psi \times 10^6$)	Tens. Str. (psi)	Diameter (microns)
B-299-8-A-1	2500		18.5	170,000	5.6
			18.9	104,000	5.8
			16.4	104,000	5.2
			15.0	112,000	5.4
			20.0	145,000	5.2
			16.6	191,000	5.3
			18.9	181,000	5.2
			18.3	155,000	5.4
			18.4	157,000	5.2
			23.7	168,000	5.3
		Avg.	18.6	149,000	5.4
		Std. Dev.	2.0	32,000	0.2
B-299-9-A-1	2500		25.5	227,000	4.6
			20.4	162,000	5.1
			19.9	187,000	5.1
			16.4	168,000	4.9
			19.6	198,000	5.0
			17.3	189,000	5.5
			19.2	208,000	5.4
			17.4	212,000	5.4
			16.4	96,000	5.5
		Avg.	19.1	184,000	5.2
		Std. Dev.	2.7	37,000	0.3
B-299-10A	2500		17.7	207,000	6.0
			17.4	195,000	5.7
			20.9	208,000	5.8
			19.4	115,000	5.7
			20.2	80,000	5.5
			18.9	188,000	6.2
			18.8	108,000	6.5
			20.5	179,000	6.1
			26.5	202,000	9.0
			19.8	131,000	7.5
		Avg.	20.0	161,000	6.4
		Std. Dev.	2.5	48,000	1.1

TABLE A.2 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus$_6$(E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-305-1A	5000	18.9	200,000	6.0
		19.9	155,000	9.2
		20.3	142,000	5.5
		19.9	195,000	5.5
		19.6	207,000	5.7
		20.7	217,000	6.3
		26.9	182,000	5.3
		20.2	192,000	5.5
		20.3	217,000	5.8
		21.3	192,000	5.4
	Avg.	20.8	190,000	6.0
	Std. Dev.	2.2	25,000	1.2
B-305-2-4	7800	20.7	160,000	5.6
		22.2	208,000	5.5
		22.1	158,000	5.6
		21.8	187,000	5.5
		20.9	161,000	5.6
		20.2	144,000	5.5
		19.6	113,000	5.6
		20.4	108,000	5.5
		20.1	175,000	5.5
		21.6	202,000	5.6
	Avg.	21.0	162,000	5.5
	Std. Dev.	0.9	34,000	0.1
B-305-1B	5000	21.2	123,000	5.6
		20.7	82,000	5.8
		21.2	157,000	5.6
		22.6	97,000	5.5
		22.1	198,000	5.9
		19.9	205,000	5.7
		22.3	122,000	5.7
		22.3	124,000	5.4
		21.8	166,000	5.3
		22.1	161,000	5.5
	Avg.	21.6	144,000	5.6
	Std. Dev.	0.8	41,000	0.2

TABLE A.2 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus$_6$ (E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-305-2B	7800	19.3	141,000	5.8
		20.9	231,000	5.6
		19.7	203,000	5.6
		22.2	227,000	5.5
		20.7	119,000	5.7
		20.7	220,000	5.5
		19.4	99,000	5.5
		20.2	228,000	5.5
		20.2	212,000	5.6
		21.3	233,000	5.5
	Avg.	20.4	191,000	5.6
	Std. Dev.	0.9	51,000	0.1
B-305-3	5000	22.0	201,000	6.0
		22.3	144,000	5.4
		21.0	189,000	5.6
		20.0	155,000	6.3
		20.5	157,000	5.5
		21.2	202,000	5.8
		21.0	232,000	5.7
		16.7	225,000	5.8
		20.3	182,000	5.5
		21.2	196,000	6.0
	Avg.	20.6	188,000	5.9
	Std. Dev.	1.6	29,000	0.3
B-305-4	7800	19.2	169,000	5.7
		23.3	207,000	6.6
		20.1	193,000	6.5
		20.5	210,000	5.7
		22.1	199,000	5.1
		21.3	187,000	5.6
		22.1	185,000	5.3
		20.0	222,000	6.1
		19.8	228,000	5.6
		19.9	218,000	5.5
	Avg.	20.8	202,000	5.8
	Std. Dev.	1.3	19,000	0.4

TABLE A.3

INDIVIDUAL FIBER DATA FOR FIBERS
FIRED AT VARIOUS RATES TO 2000 F

Test Data: Instron Tester, 1-inch gage length, Cell A corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 75% Al_2O_3, 20% SiO_2, 5% B_2O_3.

Sample No.	Firing Rate (F/hr)	Modulus$_6$ (E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-305-7	7800	23.9	185,000	6.2
		38.8	316,000	5.1
		21.7	248,000	5.2
		21.2	245,000	5.3
		21.0	220,000	6.8
		22.6	240,000	6.4
		20.9	153,000	5.5
		22.0	205,000	5.5
		20.6	153,000	5.5
		20.8	296,000	4.7
	Avg.	23.3	221,000	5.6
	Std. Dev.	5.5	50,000	0.6
B-305-11	7800	21.0	246,000	5.7
		22.2	191,000	8.2
		21.2	82,000	4.5
		21.0	70,000	5.4
		22.3	210,000	5.5
		22.3	231,000	5.3
		20.2	145,000	4.7
		23.2	211,000	5.4
		21.1	169,000	5.4
		18.9	155,000	5.3
	Avg.	21.3	173,000	5.5
	Std. Dev.	1.2	59,000	1.0
B-305-19	7800	18.3	180,000	4.9
		18.0	167,000	5.0
		18.9	165,000	5.8
		19.3	192,000	5.3
		21.4	98,000	5.2
		18.2	164,000	5.2
		19.9	196,000	5.0
		19.5	204,000	5.0
		18.8	182,000	5.1
		15.8	196,000	5.4
	Avg.	19.2	175,000	5.2
	Std. Dev.	1.6	30,000	0.3

TABLE A.3 (Cont'd.)

Sample No.	Firing Rate (F/hr)	Modulus$_6$ (E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-305-20	5000	20.7	102,000	5.0
		21.0	154,000	5.6
		21.9	181,000	5.0
		21.8	100,000	5.2
		27.6	221,000	4.7
		19.6	164,000	5.1
		20.7	207,000	5.0
		18.8	150,000	7.2
		21.6	159,000	5.0
		18.2	178,000	5.7
	Avg.	21.2	162,000	5.3
	Std. Dev.	2.6	39,000	0.7
B-306-16	2500	19.1	151,000	5.3
		20.3	102,000	5.6
		17.4	137,000	5.6
		21.1	193,000	4.7
		19.7	196,000	5.6
		18.2	183,000	5.2
		21.7	134,000	5.4
		16.9	174,000	5.4
		20.1	197,000	5.4
		17.0	152,000	5.9
	Avg.	19.1	162,000	5.4
	Std. Dev.	1.7	31,900	0.3

TABLE A.4

INDIVIDUAL FIBER DATA FOR FIBERS PLACED IN PREHEATED KILN AND FIRED TO 1950 F AT 500 F/HR

Test Data: Instron Tester, 1-inch gage length, Cell A corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 75% Al_2O_3, 20% SiO_2, 5% B_2O_3.

Sample No.	Kiln Preheat Temp. (F)		Modulus$_6$(E) (psix10^6)	Tens. Str. (psi)	Diameter (microns)
B-305-5	1000		15.7	79,000	5.8
			13.6	129,000	5.9
			17.3	97,000	5.8
			18.4	82,000	5.8
			15.5	65,000	5.6
		Avg.	16.1	89,000	5.8
		Std. Dev.	1.8	25.0	0.1
B-299-15	600		20.5	127,000	6.4
			20.1	238,000	5.3
			19.6	250,000	5.7
			21.5	197,000	5.1
			20.3	238,000	5.7
			19.0	73,000	5.3
			19.2	85,000	5.0
			20.4	228,000	5.3
			19.9	203,000	5.0
			21.8	113,000	5.1
		Avg.	20.2	175,000	5.4
		Std. Dev.	0.9	68.5	0.4

TABLE A.5

INDIVIDUAL FIBER PROPERTIES FOR FIBER SAMPLES
DRIED IN VARIOUS MANNERS AND FIRED ACCORDING TO TECHNIQUE 1.

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" Cell.

Sample No.	Diameter* (microns)	Tens. Str. (psi)	Modulus (E) ($psix10^6$)
B-3056-2-9	5.7	223,000	21.7
	6.1	260,000	22.6
	5.6	199,000	20.4
	6.3	205,000	17.3
	7.0	152,000	19.0
	5.9	153,000	20.2
	5.7	191,000	20.2
	5.1	237,000	20.6
	6.5	181,000	23.7
	5.8	194,000	15.9
Avg.	6.0	200,000	20.2
Std. Dev.	0.5	34,000	2.3
B-3056-2-7	5.6	83,000	20.5
	5.5	104,000	20.2
	6.3	198,000	21.6
	5.6	128,000	16.1
	5.4	186,000	18.1
	5.3	133,000	24.3
	6.2	63,000	22.5
Avg.	5.7	136,000	20.5
Std. Dev.	0.4	55,000	2.7
B-3056-2-8	5.7	194,000	21.8
	5.5	183,000	21.2
	5.6	167,000	21.3
	5.6	231,000	22.8
	5.3	206,000	22.3
	5.5	172,000	20.7
	5.6	186,000	22.0
	5.3	175,000	22.3
	5.9	131,000	28.8
	6.2	139,000	23.3
Avg.	5.5	178,000	22.7
Std. Dev.	0.3	30,000	2.3
B-3056-2-10	5.3	75,000	22.2
	6.8	65,000	21.1
	5.3	141,000	21.0
	5.4	47,000	----
	5.4	87,000	22.1
Avg.	4.9	83,000	21.6
Std. Dev.	0.6	35,000	

* Average of five readings along the 1-inch gage length.

TABLE A.6

INDIVIDUAL FIBER PROPERTIES FOR FIBER SAMPLES
DRIED IN VARIOUS MANNERS AND FIRED ACCORDING TO TECHNIQUE 2

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" ce-1.

Sample No.	Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)
B-3056-1-2	5.4	239,000	21.9
	5.7	178,000	21.2
	5.7	237,000	22.4
	6.0	179,000	21.6
	5.6	193,000	21.8
	6.1	166,000	20.3
	5.6	107,000	17.7
	5.5	179,000	22.0
	5.6	211,000	22.3
	5.3	112,000	19.9
Avg.	5.6	180,000	21.1
Std. Dev.	0.2	45,000	1.5
B-3056-1-3	5.8	211,000	21.3
	5.6	162,000	22.3
	5.6	214,000	21.1
	5.9	272,000	22.7
	5.7	152,000	22.1
	5.7	169,000	23.6
	5.7	241,000	22.7
	5.7	135,000	24.0
	6.1	225,000	22.1
	5.7	223,000	22.8
Avg.	5.7	200,000	22.6
Std. Dev.	0.2	44,000	0.9
B-3056-1-5	5.6	167,000	21.7
	5.5	215,000	22.2
	5.7	268,000	23.5
	7.5	229,000	23.7
	5.6	163,000	21.4
	5.6	133,000	19.5
	5.3	186,000	24.1
	6.2	221,000	21.7
	5.3	197,000	21.9
	5.7	245,000	21.5
Avg.	5.8	202,000	22.1
Std. Dev.	0.6	42,000	1.4

* Average of five readings along the 1-inch gage length.

TABLE A.6 (Cont'd.)

Sample No.	Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)
B-308-2-R	7.7	193,000	22.9
	5.2	278,000	22.8
	5.3	229,000	21.1
	5.3	271,000	22.9
	6.3	190,000	21.7
	5.8	189,000	20.8
	5.9	230,000	21.6
	7.1	179,000	21.6
	6.5	197,000	22.4
	5.5	257,000	20.7
Avg.	6.1	221,000	21.8
Std. Dev.	0.8	38,000	0.9

TABLE A.7

INDIVIDUAL FIBER PROPERTIES FOR SAMPLES STORED VARIOUS TIMES OVER MAGNESIUM PERCHLORATE AND FIRED TO 1950 F

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell

Sample No.	Storage Time (hr)		Diameter* (microns)	Tensile Strength (psi)	Modulus$_6$ (E) (psix10^6)
B-313-5	19		5.8	199,000	23.7
			5.6	213,000	24.4
			5.6	143,000	22.8
			5.9	188,000	22.4
			5.9	209,000	22.6
			5.7	188,000	20.7
			5.8	141,000	22.9
			5.7	179,000	22.4
			5.9	221,000	21.5
			5.7	216,000	22.4
		Avg.	5.8	190,000	22.6
		Std. Dev.	0.1	29,000	1.0
B-313-13	22		6.1	224,000	21.1
			6.0	207,000	22.5
			6.1	253,000	18.7
			5.7	224,000	23.6
			6.0	252,000	21.2
			5.9	271,000	23.2
			5.8	262,000	22.5
			5.8	244,000	21.7
			6.1	186,000	24.1
			5.9	236,000	23.3
		Avg.	5.9	236,000	22.2
		Std. Dev.	0.1	26,000	1.6
B-313-10	118		5.6	185,000	23.2
			5.5	140,000	21.4
			5.6	117,000	21.3
			5.6	181,000	20.9
			5.7	213,000	22.4
			5.4	209,000	22.5
			5.5	169,000	22.7
			5.5	98,000	20.7
			5.5	181,000	20.6
			6.0	142,000	22.2
		Avg.	5.6	164,000	21.8
		Std. Dev.	0.2	38,000	0.9

* Average of five readings along the 1-inch gage length.

TABLE A.7 (Cont'd.)

Sample No.	Storage Time (hr)	Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)
B-313-7	141	6.2	239,000	22.7
		6.0	226,000	25.3
		6.0	161,000	22.2
		6.0	174,000	21.8
		6.1	238,000	22.1
		6.1	215,000	21.4
		6.2	122,000	20.4
		6.1	222,000	21.2
		6.3	235,000	23.7
		6.0	121,000	22.9
	Avg.	6.1	195,000	22.2
	Std. Dev.	0.1	47,000	1.0
B-313-3	165	5.9	246,000	24.3
		5.3	271,000	22.1
		5.4	170,000	22.1
		5.1	169,000	23.7
		5.0	215,000	19.5
		5.3	210,000	20.6
		5.4	262,000	20.1
		5.5	235,000	19.8
		5.6	191,000	23.3
		5.4	190,000	20.4
	Avg.	5.4	216,000	21.6
	Std. Dev.	0.3	37,000	1.7
B-310-4	285	6.1	216,000	22.7
		6.3	249,000	20.4
		6.0	192,000	22.6
		5.6	215,000	18.8
		5.0	250,000	20.7
		5.5	167,000	17.2
		5.[illegible]	203,000	18.9
		5.8	257,000	20.7
		5.8	188,000	19.3
		5.7	238,000	20.0
	Avg.	5.7	218,000	20.1
	Std. Dev.	0.4	30,000	1.7

TABLE A.8

INDIVIDUAL FIBER PROPERTIES FOR FIBERS SOAKED VARIOUS TIMES AT 600 F AND FIRED TO 1950 F

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell.

Sample No.	Soak at 600 F (hr)		Diameter* (microns)	Tensile Strength (psi)	Modulus$_6$(E) (psix10^6)
B-314-11	1		6.0	219,000	22.4
			6.3	266,000	29.1
			5.9	88,000	22.0
			5.7	181,000	22.5
			5.5	217,000	23.5
			5.0	290,000	23.2
			5.6	222,000	21.8
			7.8	174,000	20.3
			6.2	151,000	22.1
			5.7	187,000	21.7
		Avg.	6.0	200,000	22.9
		Std. Dev.	0.7	57,000	2.4
B-314-7	22		5.3	191,000	19.8
			5.0	242,000	21.8
			4.9	235,000	21.4
			5.6	233,000	21.4
			5.4	248,000	21.3
			6.0	169,000	22.9
			4.8	239,000	21.1
			5.1	241,000	21.4
			5.7	225,000	21.0
			5.7	249,000	1.2
		Avg.	5.3	227,000	21.3
		Std. Dev.	0.4	26,000	0.77
B-314-5	24		5.1	127,000	23.1
			5.7	225,000	21.1
			6.3	233,000	22.8
			6.0	254,000	22.6
			5.6	203,000	20.0
			5.6	142,000	22.7
			5.0	174,000	20.5
			5.3	184,000	21.3
			5.2	188,000	23.0
			5.6	198,000	22.8
		Avg.	5.5	193,000	22.0
		Std. Dev.	0.4	39,000	1.2

* Average of five readings along the 1-inch gage length.

TABLE A.8 (Cont'd.)

Sample No.	Soak at 600 F (hr)	Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psix10^6$)
B-314-5	40	6.6	225,000	20.3
		6.9	243,000	22.1
		5.6	199,000	20.3
		6.6	204,000	20.2
		6.2	227,000	20.7
		5.8	194,000	21.1
		5.2	208,000	22.2
		5.0	134,000	21.9
		6.7	18[illegible],000	20.7
		5.3	206,000	20.6
	Avg.	6.0	202,000	21.0
	Std. Dev.	0.7	30,000	0.77

TABLE A.9

INDIVIDUAL FIBER PROPERTIES FOR SAMPLES SOAKED VARIOUS TIMES AT 600 F AND FIRED TO 1950 F

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell.

Sample No.	Soak at 600 F (hr)		Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psix10^6$)
B-313-30	1		6.7	214,000	22.2
			6.2	205,000	14.1
			5.0	160,000	19.8
			5.9	204,000	19.0
			5.4	210,000	22.7
			5.6	166,000	19.1
			6.4	241,000	18.6
			6.2	123,000	20.6
			5.1	157,000	21.0
			5.1	217,000	21.4
		Avg.	5.8	180,000	20.4
		Std. Dev.	0.6	45,000	1.5
B-313-20	17		7.2	232,000	21.3
			6.7	207,000	23.8
			7.4	206,000	20.5
			7.5	196,000	22.7
			5.6	217,000	21.9
			7.0	130,000	13.8
			5.7	213,000	20.4
			4.9	212,000	16.3
			7.1	201,000	21.4
			8.1	170,000	21.0
		Avg.	6.7	198,000	20.3
		Std. Dev.	1.0	29,000	3.0
B-313-29	19		5.8	238,000	20.4
			6.3	105,000	22.2
			5.6	222,000	20.7
			5.2	191,000	12.5
			5.6	220,000	21.5
			5.9	185,000	21.5
			7.9	187,000	21.1
			6.3	164,000	18.9
			6.0	194,000	20.5
			5.7	237,000	21.7
		Avg.	6.0	194,000	20.1
		Std. Dev.	0.7	40,000	2.8

* Average of five readings along the 1-inch gage length.

TABLE A.9 (Cont'd.)

Sample No.	Soak at 600 F (hr)		Diameter* (microns)	Tensile Strength (psi)	Modulus$_6$ (E) (psix10^6)
B-313-27	21		6.0	247,000	25.2
			5.3	176,000	21.8
			5.8	228,000	21.9
			6.2	236,000	21.1
			5.8	252,000	24.3
			5.6	214,000	22.6
			5.5	256,000	24.8
			5.5	231,000	22.9
			5.1	228,000	22.5
			5.6	220,000	23.5
		Avg.	5.6	233,000	22.9
		Std. Dev.	0.3	26,000	1.1
B-313-23	44		5.6	180,000	18.7
			6.2	213,000	20.4
			9.6	191,000	21.3
			6.6	121,000	19.8
			6.8	126,000	19.7
			5.2	154,000	20.1
			6.1	190,000	20.1
			5.9	194,000	20.8
			5.4	189,000	20.4
			5.6	179,000	19.6
		Avg.	6.3	174,000	20.1
		Std. Dev.	1.3	30,000	0.7
B-313-18	88		5.6	191,000	20.2
			5.6	205,000	20.1
			5.8	221,000	20.1
			5.7	192,000	20.9
			5.2	238,000	23.0
			5.7	173,000	20.0
			5.8	234,000	22.9
			5.9	177,000	22.9
			6.3	146,000	19.5
		Avg.	5.7	197,000	21.2
		Std. Dev.	0.3	30,000	1.4

TABLE A.10

INDIVIDUAL FIBER PROPERTIES OF VARIOUS DIAMETER FIBER SAMPLES OF 75% Al_2O_3, 25% SiO_2, 5% B_2O_3 COMPOSITION

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell

Sample No.		Diameter* (microns)	Tensile Strength (psi)	Modulus$_6$ (E) (psix10^6)
B-314-4		4.9	251,000	20.8
		5.0	241,000	23.2
		4.5	179,000	27.0
		4.8	260,000	22.9
		4.7	254,000	21.8
		4.6	257,000	22.0
		4.7	251,000	24.4
		4.7	255,000	24.1
		4.7	257,000	26.2
		4.8	267,000	22.2
	Avg.	4.7	247,000	23.5
	Std. Dev.	0.1	25,000	2.0
B-313-9		5.7	223,000	21.7
		6.1	260,000	22.6
		5.6	199,000	20.4
		6.3	205,000	17.3
		7.0	152,000	19.0
		5.9	153,000	20.2
		5.7	191,000	20.2
		5.1	237,000	20.6
		6.5	181,000	23.7
		5.8	194,000	15.9
	Avg.	6.0	200,000	20.2
	Std. Dev.	0.5	34,000	2.3
B-313-1		7.5	229,000	----
		7.5	231,000	23.2
		7.5	155,000	21.8
		7.4	236,000	22.4
		6.9	235,000	21.8
		7.4	239,000	23.0
		7.2	218,000	24.4
		7.1	190,000	22.1
		7.5	238,000	22.4
		6.9	225,000	22.7
	Avg.	7.3	220,000	23.3
	Std. Dev.	0.2	27,000	1.7

* Average of five readings along the 1-inch gage length.

TABLE A.11

INDIVIDUAL FIBER DATA FOR MULLITE
(73% Al_2O_3,27% SiO_2) FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements.

Sample No.	Firing Temp. (F)		Diameter (microns)	Tensile Strength (psi)	Modulus$_6$(E) (psix10^6)
M-100-2	1950		6.4	124,000	12.11
			6.2	127,000	11.59
			6.4	116,000	11.68
			5.8	97,000	12.26
			6.6	133,000	11.48
			6.2	115,000	11.28
			6.1	54,000	10.82
			6.2	130,000	15.11
			7.1	129,000	13.85
		Avg.	6.3	115,000	12.3
		Std. Dev.	0.3	24,000	1.3
M-100-4	2100		7.2	161,000	17.8
			5.6	165,000	18.4
			7.0	144,000	17.7
			6.9	143,000	16.2
			5.4	184,000	19.8
			5.8	129,000	18.1
			5.6	136,000	15.6
			5.6	167,000	16.5
			5.6	163,000	16.1
			5.6	135,000	17.1
		Avg.	6.0	151,000	17.3
		Std. Dev.	0.7	21,000	1.3
M-103-4	2300		7.3	104,000	19.6
			7.5	131,000	21.7
			8.0	102,000	20.0
			7.4	118,000	21.7
			6.7	93,000	19.4
			7.6	110,000	19.9
			7.6	123,000	21.7
			8.2	108,000	21.1
			7.2	110,000	19.3
			5.8	118,000	22.4
		Avg.	7.3	112,000	20.7
		Std. Dev.	0.7	11,000	1.2

TABLE A.11 (Cont'd.)

Sample No.	Firing Temp. (F)	Diameter (microns)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)
M-102-1B	2500	10.6	29,000	30.7
		8.6	45,000	25.4
		7.9	36,000	27.3
		7.8	13,000	23.9
		9.4	77,000	28.0
		11.2	49,000	27.3
		8.2	29,000	25.5
		8.5	63,000	27.8
		6.7	26,000	25.3
		9.6	44,000	25.4
	Avg.	8.8	41,000	26.6
	Std. Dev.	1.4	19,000	2.1

TABLE A.12

INDIVIDUAL FIBER PROPERTIES FOR FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell. Composition 75% Al_2O_3, 20% SiO_2, 5% B_2O_3.

Sample No.	Firing Temp. (F)		Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psix10^6$)
B-314-12	1700		8.0	165,000	22.8
			5.5	251,000	21.4
			5.4	267,000	22.2
			5.8	231,000	21.0
			5.9	131,000	21.1
			5.1	218,000	21.4
			6.6	198,000	21.6
			5.5	251,000	22.0
			5.5	210,000	21.9
			5.6	186,000	22.2
		Avg.	5.9	211,000	21.8
		Std. Dev.	0.8	42,000	0.6
B-314-4	1950		4.0	211,000	20.1
			5.2	259,000	21.5
			6.0	209,000	18.7
			5.5	180,000	20.4
			5.6	262,000	21.9
			5.0	262,000	22.4
			6.0	186,000	19.6
			5.0	209,000	20.7
			5.6	219,000	19.8
			5.2	228,000	20.3
		Avg.	5.3	223,000	20.5
		Std. Dev.	0.6	30,000	1.1
B-313-25	2400		5.5	159,000	24.9
			6.9	100,000	25.5
			8.1	143,000	26.0
			6.4	158,000	25.4
			5.4	129,000	15.5
			6.6	135,000	25.2
			5.1	149,000	26.2
			6.7	157,000	24.8
			7.3	153,000	24.8
			5.7	104,000	26.1
		Avg.	6.4	140,000	24.4
		Std. Dev.	0.9	23,000	3.2

* Average of five readings along the 1-inch gage length.

TABLE A.13

INDIVIDUAL FIBER PROPERTIES FOR FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, Cell "A" corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 77.6% Al_2O_3, 19.5% SiO_2, 1.6% B_2O_3, 1.4% P_2O_5.

Sample No.	Firing Temp. (F)		Modulus (E) ($psix10^6$)	Tensile Strength (psi)	Diameter (microns)
P-100-6	1800		13.5	135,000	6.2
			14.2	116,000	7.3
			13.5	73,000	7.4
			12.8	122,000	7.5
			11.1	116,000	7.6
			12.7	107,000	7.8
			11.2	90,000	7.9
			13.2	107,000	8.2
			11.5	110,000	6.8
			12.5	119,000	8.3
		Avg.	12.6	110,000	7.5
		Std. Dev.	1.1	17,000	0.6
P-100-4	1950		13.9	115,000	6.4
			14.9	122,000	4.4
			14.8	117,000	4.9
			13.7	81,000	5.2
			14.1	96,000	6.0
			15.5	86,000	6.7
			13.6	111,000	6.0
			16.0	99,000	5.2
			14.4	122,000	6.2
			15.5	63,000	5.9
		Avg.	14.6	101,000	5.7
		Std. Dev.	0.9	20,000	0.7
P-100-5	2200		21.3	55,000	7.5
			21.5	120,000	7.9
			23.0	60,000	7.8
			20.2	80,000	7.8
			21.8	92,000	6.4
			22.6	86,000	6.6
			21.8	53,000	7.2
			22.5	73,000	7.0
			22.8	113,000	9.4
			21.6	64,000	7.3
		Avg.	21.9	80,000	7.4
		Std. Dev.	0.9	23,000	0.8

TABLE A.14

INDIVIDUAL FIBER PROPERTIES FOR FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, Cell "A" corrected for extension, 0.02"/min crosshead speed, 20.00"/min chart speed, diameter avg. of five measurements. Composition 74.5% Al_2O_3, 19.7% SiO_2, 4.7% B_2O_3, 1.2% P_2O_5.

Sample No.	Firing Temp. (F)		Modulus (E) (psix10^6)	Tensile Strengt' (psi)	Diameter (microns)
P-103-7	1800		21.9	196,000	7.8
			21.5	204,000	7.3
			22.1	182,000	7.8
			21.0	187,000	7.6
			19.3	224,000	7.6
			19.8	206,000	7.3
			20.0	212,000	7.2
			21.7	217,000	7.2
			20.3	176,000	7.6
			21.9	192,000	7.6
		Avg.	21.0	200,000	7.5
		Std. Dev.	1.01	15,700	0.2
P-103-8A	1950		19.3	191,000	9.1
			21.8	164,000	10.2
			24.2	180,000	10.8
			21.1	216,000	7.5
			21.8	238,000	7.3
			21.6	220,000	8.8
			22.4	204,000	7.6
			22.9	198,000	9.9
			21.6	194,000	7.7
			22.8	220,000	6.9
		Avg.	21.9	203,600	8.6
		Std. Dev.	1.29	21,800	1.4
P-103-8B	220		21.9	187,000	7.1
			24.4	192,000	6.6
			24.3	224,000	7.2
			20.5	142,000	6.4
			22.9	231,000	6.6
			22.6	152,000	7.6
			25.9	227,000	8.2
			22.5	174,000	7.0
			22.3	206,000	6.7
			22.2	202,000	6.8
		Avg.	23.5	194,000	7.0
		Std. Dev.	1.65	30,700	0.5

TABLE A.15

INDIVIDUAL FIBER PROPERTIES FOR FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell, diameter avg. of five measurements Composition 84.2% Al_2O_3,10% SiO_2 (40-60 mμ colloidal SiO_2),5.6% B_2O .

Sample No.	Firing Temp. (°F)		Tensile Strength (psi)	Modulus$_6$(E) (psix10^6)	Diameter (microns)
B-334-7	1700		252,000	21.6	5.4
			228,000	22.0	5.0
			226,000	19.9	5.1
			208,000	20.6	5.2
			181,000	21.7	5.4
			185,000	21.2	5.6
			152,000	18.3	5.3
			215,000	30.5	5.4
			204,000	21.0	5.8
			216,000	20.4	5.2
		Avg.	207,000	22.6	5.3
		Std. Dev.	28,000	6.03	0.2
B-334-4	1800		236,000	22.0	5.4
			248,000	21.9	5.4
			174,000	23.9	5.6
			241,000	22.9	5.2
			201,000	21.8	5.4
			247,000	24.6	5.4
			107,000	22.9	5.4
			223,000	20.8	5.5
			219,000	22.3	5.4
			222,000	25.2	5.5
		Avg.	212,000	22.8	5.4
		Std. Dev.	43,000	1.38	0.1
B-334-6	1950		219,000	22.8	5.1
			219,000	22.8	5.3
			251,000	24.0	5.1
			232,000	23.9	5.0
			210,000	21.8	5.0
			193,000	21.6	5.4
			174,000	22.3	5.4
			242,000	24.0	5.2
			226,000	22.5	5.1
			235,000	21.6	5.0
		Avg.	220,000	22.6	5.2
		Std. Dev.	23,000	0.88	0.2

TABLE A.15 (Cont'd.)

Sample No.	Firing Temp. (F)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)	Diameter (microns)
B-324-10	2150	223,000	23.7	4.6
		231,000	25.1	4.7
		227,000	26.0	4.8
		256,000	24.0	4.7
		155,000	26.0	4.7
		193,000	28.8	4.6
		234,000	24.1	4.7
		242,000	26.1	4.6
		256,000	24.5	4.9
		225,000	23.7	4.7
	Avg.	224,000	25.2	4.7
	Std. Dev.	30,000	1.58	0.1
B-324-7	2250	180,000	23.8	6.0
		71,000	23.5	5.9
		182,000	25.5	5.9
		88,000	25.5	5.9
		173,000	23.7	5.6
		193,000	25.0	5.8
		172,000	24.4	5.8
		157,000	25.5	5.5
		169,000	25.2	5.6
		137,000	27.4	5.6
	Avg.	152,000	25.0	5.8
	Std. Dev.	41,000	1.18	0.2
B-324-6	2300	69,000	22.8	5.5
		65,000	22.7	5.4
		74,000	23.8	5.2
		60,000	24.2	5.2
		73,000	24.9	5.5
		75,000	22.6	5.6
		74,000	24.3	5.5
		67,000	22.5	5.4
		61,000	24.6	5.6
		56,000	22.6	5.5
	Avg.	67,000	23.5	5.4
	Std. Dev.	7,000	0.97	0.1

TABLE A.16

INDIVIDUAL FIBER PROPERTIES FOR FIBERS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell, diameter avg. of five measurements
Composition 84.8% Al_2O_3, 9.9% SiO_2 (7 mμ colloidal SiO_2), 5.8% B_2O_3.

Sample No.	Firing Temp. (F)	Tensile Strength (psi)	Modulus (E) ($psix10^6$)	Diameter (microns)
B-327-3	1700	303,000	23.5	5.1
		292,000	22.2	5.1
		286,000	24.9	5.4
		220,000	21.9	5.2
		299,000	23.9	5.1
		311,000	22.3	4.9
		300,000	22.5	5.3
		265,000	23.6	5.1
		296,000	21.9	5.2
		258,000	22.0	5.2
	Avg.	283,000	22.9	5.2
	Std. Dev.	28,000	1.03	0.1
B-327-6	1800	264,000	23.0	4.5
		320,000	24.3	4.2
		243,000	25.4	4.3
		302,000	23.7	4.5
		310,000	23.2	4.4
		276,000	26.1	4.2
		244,000	24.6	4.4
		236,000	22.2	4.2
		244,000	23.5	4.6
		313,000	21.9	4.5
	Avg.	275,000	23.7	4.4
	Std. Dev.	33,000	1.30	0.1
B-327-20	1950	239,000	26.6	5.7
		246,000	25.6	6.1
		224,000	24.1	5.9
		275,000	24.7	6.4
		219,000	23.5	6.2
		200,000	22.9	6.6
		302,000	24.5	6.2
		233,000	23.1	6.0
		181,000	25.1	6.8
		284,000	24.7	5.6
	Avg.	240,000	24.5	6.1
	Std. Dev.	38,000	1.16	0.4

TABLE A.16 (Cont'd.)

Sample No.	Firing Temp. (F)	Tensile Strength (psi)	Modulus$_6$(E) (psix10^6)	Diameter (microns)
B-327-10	2100	275,000	27.4	4.9
		284,000	27.0	4.8
		262,000	26.2	4.7
		240,000	34.4	4.8
		241,000	25.6	4.8
		259,000	27.6	4.8
		248,000	27.1	4.6
		262,000	26.8	4.7
		258,000	26.7	4.6
		230,000	26.8	4.7
	Avg.	256,000	27.6	4.7
	Std. DEv.	17,000	2.46	0.1
B-327-7	2250	172,000	27.3	4.7
		222,000	27.2	5.0
		272,000	28.0	4.7
		238,000	27.5	4.9
		264,000	26.9	4.9
		155,000	27.6	4.7
		197,000	27.6	4.8
		252,000	27.8	4.8
		223,000	28.0	4.6
		213,000	27.3	4.7
	Avg.	221,000	27.5	4.8
	Std. Dev.	38,000	0.35	0.1
B-327-4	2350	119,000	28.5	4.8
		102,000	26.4	4.8
		106,000	28.5	4.9
		102,000	31.7	4.8
		68,000	28.3	4.8
		124,000	30.3	4.8
		112,000	28.6	4.9
		91,000	26.6	5.3
		110,000	28.4	4.8
		96,000	27.9	5.0
	Avg.	103,000	28.5	4.9
	Std. Dev.	16,000	1.57	0.2

TABLE A.17

INDIVIDUAL FIBER PROPERTIES FOR FIBERS OF TWO HIGH Al_2O_3 COMPOSITIONS FIRED TO VARIOUS TEMPERATURES

Test Data: Instron Tester, 1-inch gage length, Cell "A" corrected for extension, 0.02"/min crosshead seped, 20.00"/min chart speed, diameter avg. of five measurements.

Sample No.	Composition	Firing Temp. (F)	Tens. Str. (psi)	Modulus (E) ($psix10^6$)	Diameter (microns)
B-326-1	89.4% Al_2O_3	1700	152,000	18.3	6.6
	5.2% SiO_2		151,000	20.0	5.6
	5.4% B_2O_3		157,000	19.7	4.6
			154,000	19.3	6.5
			85,000	18.4	6.2
			120,000	19.0	6.3
			127,000	18.8	5.2
			125,000	17.6	6.7
			151,000	19.8	5.9
			96,000	19.9	6.6
		Avg.	132,000	19.1	6.0
		Std. Dev.	26,000	0.8	0.7
B-326-5	89.4% Al_2O_3	1950	140,000	23.0	6.0
	5.2% SiO_2		177,000	24.9	5.5
	5.4% B_2O_3		132,000	26.2	5.1
			174,000	24.5	5.6
			197,000	26.1	4.8
			185,000	23.2	4.8
			171,000	27.6	5.0
			164,000	24.1	5.1
			211,000	24.5	5.6
			156,000	21.1	5.5
		Avg.	170,000	24.5	5.3
		Std. Dev.	26,000	1.86	0.4
B-326-4	89.4% Al_2O_3	2100	193,000	21.4	5.4
	5.2% SiO_2		129,000	19.2	7.4
	5.4% B_2O_3		172,000	24.9	5.9
			166,000	23.9	5.4
			124,000	22.1	5.4
			98,000	23.3	5.4
			185,000	24.9	5.6
			203,000	27.7	6.2
			144,000	25.0	4.4
			196,000	21.3	5.0
		Avg.	161,000	23.4	5.6
		Std. Dev	36,000	2.3	0.8

TABLE A.17 (Cont'd.)

Sample No.	Composition	Firing Temp. (F)	Tens. Str. (psi)	Modulus$_6$(E) (psix10^6)	Diameter (microns)
B-325-10	92.5% Al_2O_3	1800	180,000	21.6	5.6
	5.0% SiO_2		191,000	22.9	5.1
	2.5% B_2O_3		188,000	22.4	5.1
			167,000	21.1	5.0
			197,000	26.9	4.8
			203,000	24.3	4.8
			184,000	22.7	5.2
			181,000	22.9	4.9
			130,000	22.1	5.0
			146,000	22.4	5.1
		Avg.	177,000	22.9	5.1
		Std. Dec.	23,000	1.64	0.2
B-325-11	92.5% Al_2O_3	1950	101,000	15.6	5.4
	5.0% SiO_2		107,000	14.3	5.4
	2.5% B_2O_3		121,000	16.6	5.6
			96,000	15.7	5.2
			105,000	15.5	5.6
			68,000	17.0	5.4
			130,000	15.9	5.4
			55,000	16.1	5.5
			128,000	19.3	5.3
			102,000	15.7	5.6
		Avg.	101,000	16.2	5.4
		Std. Dev.	24,000	1.31	0.1
B-325-3	92.5% Al_2O_3	2100	94,000	15.6	7.0
	5.0% SiO_2		111,000	16.8	6.4
	2.5% B_2O_3		100,000	16.6	6.3
			126,000	15.9	6.0
			110,000	16.3	6.3
			89,000	15.7	7.0
			100,000	16.5	6.2
			78,000	16.4	6.5
			122,000	17.7	6.1
			136,000	17.0	6.1
		Avg.	107,000	16.4	6.4
		Std. Dev.	18,000	0.64	0.4

TABLE A.18

INDIVIDUAL FIBER PROPERTIES FOR ALUMINA FIBERS CALCINED IN VARIOUS MANNERS

Test Data: Instron Tester, 1-inch gage length, 0.02"/min crosshead speed, 20.00"/min chart speed, "A" cell.

Sample No.	Firing Temp. (F)		Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psix10^6$)
Al-100-1-1	1550		6.9	46,000	8.4
			7.9	80,000	8.3
			8.1	58,000	8.1
			7.6	49,000	8.5
			7.3	42,000	8.1
		Avg.	7.6	55,000	8.3
		Std. Dev.	0.5	15,000	0.2
Al-100-7-2-1	1575		6.6	89,000	10.9
			6.9	100,000	12.1
			6.6	85,000	12.5
			7.1	99,000	12.2
			6.8	114,000	11.3
			7.0	95,000	12.2
			7.2	116,000	10.2
			6.6	114,000	11.8
			6.6	89,000	11.3
			6.8	92,000	11.8
		Avg.	6.8	99,000	11.6
		Std. Dev.	0.2	12,000	0.7
Al-100-7-3-1	1625		6.2	40,000	12.7
			6.9	63,000	11.5
			6.8	67,000	13.9
			6.7	51,000	10.8
			6.8	86,000	12.5
		Avg.	6.7	61,000	12.3
		Std. Dev.	0.3	17,000	1.19
Al-100-7-4-1	1750		7.0	122,000	13.9
			6.7	140,000	15.1
			7.6	130,000	14.3
			7.2	133,000	13.1
			6.5	141,000	11.5
			6.3	103,000	13.5
			6.8	129,000	13.0
			6.8	129,000	12.9
			5.8	152,000	14.9
			6.9	111,000	12.8
		Avg.	6.8	129,000	13.5
		Std. Dev.	0.5	14,000	1.07

* Average of five readings along the 1-inch gage length.

TABLE A.18 (Cont'd.)

Sample No.	Firing Temp. (F)	Diameter* (microns)	Tensile Strength (psi)	Modulus (E) ($psi \times 10^6$)
A1-100-7-6-1	1550	6.6	42,000	13.6
		6.4	133,000	13.9
		6.2	92,000	14.3
		7.1	126,000	12.7
		6.5	116,000	14.1
		6.4	93,000	14.0
		9.5	58,000	15.1
		6.5	103,000	13.6
		6.6	89,000	12.7
		6.7	54,000	12.9
	Avg.	6.8	91,000	13.7
	Std. Dev.	1.0	31,000	0.77

APPENDIX B

PRODUCTION OF A QUANTITY OF FIBER

This contract required that a quantity of fiber be submitted to the AFML for evaluation. In compliance with this requirement, 0.5 pound of fiber was prepared and submitted.

The fiber chosen for production and evaluation by the AFML was the 85% Al_2O_3, 10% SiO_2 (7 mμ colloid), 5% B_2O_3 composition fibers fired to 2100 to 2150 F after being dried to equilibrium weight loss over magnesium perchlorate.

Eight to 25 grams of fiber consisting of five to eight individual samples were obtained from each firing. In order to obtain straight and aligned fiber samples, 6-foot-long unfired samples were cut in two and fired to form straight bundles 18 to 24 inches long.

One sample per firing was tested. The results of this fiber production are tabulated below:

Firing No.	No. of Samples	Total Sample Wt. (g)	Modulus (E)* ($psix10^6$)	Tens. Str.* (psi)	Diameter* (microns)
1	3	3.5	26.1	241,000	5.8
2	6	10.8	26.2	208,000	6.1
3	5	9.4	25.5	232,000	5.2
4	4	10.0	26.4	230,000	5.2
5	6	17.4	26.2	238,000	5.4
6	6	13.9	25.8	226,000	5.2
7	5	9.1	27.5	209,000	5.8
8	6	11.2	28.3	229,000	6.4
9	5	9.2	25.6	222,000	4.9
10	5	10.4	27.5	241,000	5.6
11	5	16.3	25.0	245,000	4.8
12	6	20.3	25.7	242,000	5.0
13	6	16.3	27.6	240,000	5.2
14	7	17.3	27.6	215,000	5.2
15	6	8.3	23.2	182,000	3.8
16	4	10.0	23.5	175,000	6.9
17	3	17.4	26.2	215,000	7.0
18	5	23.8	24.0	181,000	7.4
19	6	33.8	Poor, no testing.		
20	6	13.1	Poor, no testing.		
21	5	13.6	23.8	205,000	6.6
22	5	14.3	27.6	182,000	6.3

*Average of individual tests on ten fibers from on sample per firing.

The quality and quantity of fiber produced and delivered to the AFML are summarized as follows:

Fiber Properties		Fiber Weight (g)
Tensile Strength	>215,000 psi	172.6
Modulus of Elasticity	>25 x 10^6 psi	
Tensile Strength	>200,000 psi, <215,000 psi	33.5
Modulus of Elasticity	>25 x 10^6 psi	
Tensile Strength	<200,000 psi	103.3
Modulus of Elasticity	>23 x 10^6 psi, <28 x 10^6 psi	

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11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY
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13 ABSTRACT

The objective of the program was to obtain low cost fibers with tensile strengths greater than 400,000 psi and tensile moduli of 60 x 10^6 psi. A low cost process for converting concentrated aluminum salt solutions into uniform diameter polycrystalline oxide fibers via thermal decomposition had been developed.

The process was used to prepare fibers ranging in composition from 73% alumina, 27% silica to 100% alumina. Fibers in which 0-10% boric oxide was added to the basic alumina-silica composition were also prepared and studied.

The properties and microstructure of these fibers were studied with respect to the fiber composition and various thermal decomposition methods used.

Electron microscope and X-ray diffraction analyses indicated that grain size, pore size, and pore number could be related to fiber tensile strength and these factors were shown to vary with fiber composition.

Modulus of elasticity was found to depend primarily on the amount and composition of the crystalline component of the fibers. Maximum fiber properties of 250,000 psi tensile strength and 28 x 10^6 psi tensile modulus were obtained from 85% alumina, 10% SiO_2, 5% B_2O_3 composition fibers in the mullite crystalline form. Gamma alumina fibers with 130,000 psi tensile strength and 14 x 10^6 psi tensile modulus were obtained. Conversion to alpha alumina resulted in low tensile strength strength fibers in which excessive grain growth and porosity were apparent.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Alpha alumina						
Alumina fiber						
Ceramic fiber						
Composites						
Crystalline fibers						
Gamma alumina						
Grain growth - porosity - strength relationship						
High modulus fibers						
High strength fibers						
Inorganic fibers						
Mullite fiber						
Polycrystalline oxide fiber						
Reinforcement fiber						
Salt decomposition process						

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