

WADD TECHNICAL REPORT 60-244

PART II

CONTINUOUS FILAMENT CERAMIC FIBERS

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Research and Development Division*

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**WRIGHT AIR DEVELOPMENT DIVISION
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UNITED STATES AIR FORCE
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FOREWORD

This report was prepared by The Carborundum Company under USAF Contract No. AF 33(616)-6246. This contract was initiated under Project No. 7320, "Fibrous Materials for Decelerators and Structures", Task No. 73201, "Organic and Inorganic Fibers." The work was administered under the direction of the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Mr. J. H. Ross acting as project engineer.

This report covers the work conducted from March 15, 1960 through September 15, 1960.

This research work was conducted by The Carborundum Company under the general administrative supervision of Dr. W. A. Lambertson. The project consultant was Dr. B. W. King of Columbus, Ohio.

WADD TR 60-244, Pt. II.

ABSTRACT

A number of refractory materials were examined to determine their possible usefulness as materials for crucibles for melting, fining, and drawing high-silica glass compositions. Niobium diboride gave the most promising results of the materials examined.

Time and temperature relationships as well as variations in batch composition were investigated in an effort to improve premelt characteristics. The addition of vanadium pentoxide was particularly effective. It had been conceived that a well-fined premelt should be a better starting material for fiber drawing than an unreacted batch composition. Fiber drawing studies, however, showed no improvement in either the drawing characteristics or in the physical characteristics of fiber when well-fined premelt was used.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



C. A. WILLIS, Chief
Fibrous Materials Branch
Nonmetallic Materials Laboratory
Materials Central

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I. INTRODUCTION AND SUMMARY

The purpose of this report is to present a summary of the research carried out under Contract No. AF 33(616)-6246 for the period March 15, 1960 through September 15, 1960.

A small rate of effort (about one man-year) was applied to the project during the six-month extension to the original contract. During the original work, reported in WADD Technical Report 60-244, the objectives of the project were to:

1. Develop a continuous filament ceramic or glass fiber suitable for textile applications and capable of withstanding temperature degradation up to at least 1500°F. (815°C.).
2. Determine the following physical characteristics of the fibers developed:
 - (a) Tensile strength
 - (b) Tenacity
 - (c) Ultimate elongation
 - (d) Modulus of elasticity
 - (e) Loop strength
 - (f) Energy absorption

The physical characteristics were to be determined at standard conditions, at 815°C. (1500°F.), at sufficient intermediate temperature to provide smooth curves showing the effect of temperature on these characteristics, and at room temperature after exposure to these temperatures.

Determinations of tenacity, ultimate elongation, modulus of elasticity, loop strength, and energy absorption were not made as it was felt that the filaments produced thus far were not developed to such a stage as to make knowledge of these properties useful.

The approach used for refractory filament formation on this project was the melting and fining of high silica compositions in refractory crucibles and the drawing of these compositions through orifices into filaments. Because of the instability of the refractory materials used, the melting and drawing of the filaments was done

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in an inert atmosphere chamber, which also provided moisture level control.

Particular emphasis was placed on forming well-fined, homogeneous premelts from which fiber could be drawn. Such a material was formed. Filaments were drawn from it and their tensile strengths were determined.

Several refractory glasses in the baria-alumina-silica, calcia-alumina-silica, and magnesia-alumina-silica systems were successfully drawn into continuous filaments. Tensile strengths of filaments drawn were determined at room and elevated temperatures. Values as high as 138,000 pounds per square inch at room temperature and 132,000 pounds per square inch at 1500°F. (815°C.) were obtained.

Because of the small rate of effort provided for over the six-month period it was decided that the research should be directed to solving some of the major problems which had developed during the first year of work, rather than to continue fiber drawing experiments. The effort was, therefore, directed to the following:

1. Finding a crucible material which would be superior to boron nitride, the material previously used.
2. Investigation of fining conditions and materials which could be fined without excessive loss through vaporization.
3. Investigation of the effect of premelting glass batches as an aid to obtaining uniform, bubble-free melts during fiber drawing.

II. EXPERIMENTAL PROCEDURES AND RESULTS

A. Facilities

All of the facilities used for experimental work during the period covered by this report had been previously obtained or made available and were thoroughly discussed in previous reports. However, minor improvements were made where it was deemed advisable. A brief description of the equipment used is given in the following sections.

1. Furnaces

The Pereco, Model CT-312, horizontal graphite tube furnace was used for melting and fining and for compatibility experiments.

Its temperature is regulated by a proportional controller actuated by a radiation pyrometer. Crucibles, one-inch outside diameter by two inches high, were used for the experiments in this furnace.

The 40 kilowatt Ajax-Northrup induction furnace was used for melting and fining studies and in making large amounts of premelted glass from which fiber was to be drawn. Crucibles as large as seven inches in outside diameter by eight inches high were used.

The melting point furnace, which is a small resistance heated furnace with its heating zone enclosed in a water-cooled shell, was used solely for crucible material studies. Small crucibles 3/8-inch outside diameter by one-inch high were used in this furnace.

The graphite "hairpin" resistance furnace was located inside a glove box and was used for all filament drawing. This furnace is constructed such that a crucible fits loosely in a graphite "hairpin" heater which is surrounded by concentric rings of silicon carbide foam, boron nitride powder, a Fiberfrax® T30 tube, and finally by copper tubing used for water cooling. The top of the furnace consists of water-cooled copper plates which clamp onto the heater and carry current to it. The bottom is a hot-pressed boron nitride radiation shield and a transite base plate, which is connected to the top by springs. The bottom has a hole through it such that fiber can be drawn down from an orifice in the bottom of the crucible. The crucibles normally used are eight inches long by 1-1/2 inch diameter.

The temperatures at different depths in the furnace were found to vary greatly. In one case a temperature of about 1400°C. (2550°F.) was measured at a point about an inch above the bottom of the graphite heater, and at a point an inch higher the temperature was only 900°C. (1650°F.). Several new heater designs have been suggested to produce a larger, uniform heating area, among which is a double spiral graphite heater. This heater was ordered, but delivery was not made soon enough to permit testing under this contract.

2. Inert Atmosphere Chamber

All fiber drawing studies were conducted in an inert atmosphere chamber because of the instability of the refractory materials in air.

This chamber was made of an extruded aluminum frame, aluminum plate, and Plexiglas® panels. Neoprene rubber gloves

installed in the Plexiglas® panels allow work in the chamber from outside without affecting its atmosphere. Because of these gloves the chamber is often referred to as a "glove box."

A recirculating gas purification system is connected to the chamber to remove particles of dust, vapors, water, oxygen, and nitrogen. The inert gas normally used in this system is argon; however, helium is used occasionally.

3. Tensile Strength Measuring Apparatus

The apparatus used to determine the tensile strengths of the fibers produced can measure the tensile strengths of four fibers simultaneously and consists of a furnace (for measuring the strengths at elevated temperatures), drive motor, and four transducers which signal the movement of cantilevered beams to which one end of each filament is attached. The signals from the transducers represent the deflection of the beams caused by a given amount of force on the filaments.

From the force required to break a given fiber in tension and its diameter as determined with an optical microscope, the tensile strength can be calculated.

B. Crucible Materials Studies

Crucibles for melting, fining, and drawing the fiber were needed which would have the following characteristics at temperatures of 1800°C. (3270°F.) or higher:

1. They must be capable of withstanding sustained use in an inert atmosphere at the given temperatures.
2. They must not react either with the materials contained or with any portion of the furnace being used to heat it, to produce appreciably detrimental effects on any of the reactants.
3. They should not inhibit in any way the drawing of fiber from an orifice in its base.

Boron nitride was previously found to meet the first requirement; however, it was limited by its reaction with carbon at 2200°C. (3990°F.) and by reactions with titania-containing glasses and with

molybdenum heaters at temperatures between 1500°C. (2730°F.) and 2000°C. (3630°F.). Fiber has been drawn from boron nitride crucibles on many occasions, but it was felt that there might be materials from which fibers could be more easily drawn.

Two types of experiments were used in testing the crucible materials. For those materials that could not be readily obtained in the form of crucibles, the materials to be tested were placed in separate boron nitride crucibles with pieces of fused quartz and were heated in graphite heating elements to temperatures above 1800°C. (3270°F.) in argon. The results are given below:

1. Niobium diboride (NbB_2) - No indication of reaction at temperatures to 2200°C. (3990°F.).
2. Titanium diboride (TiB_2) - An indication of a reaction with silica at 1930°C. (3506°F.).
3. The composition 90 percent zirconium diboride (ZrB_2), 10 percent molybdenum disilicide (MoSi_2) - A definite reaction between the boride-silicide and the crucible, or the fused silica, or both, at about 1720°C. (3130°F.).
4. The composition 80 percent zirconium nitride (ZrN), 20 percent zirconia (ZrO_2) - No apparent reaction at temperatures to 2090°C. (3794°F.).
5. Tungsten boride (WB) - No indication of reaction at temperatures to 2150°C. (3902°F.).
6. Hafnium diboride (HfB_2) - Indication of a reaction below 2150°C. (3902°F.). X-ray diffraction of heated material showed major hafnium diboride (HfB_2) and weak hafnium oxide (HfO_2).
7. Tantalum diboride (TaB_2) - Showed slight signs of reaction below 2150°C. (3902°F.). X-ray diffraction of heated material showed major tantalum diboride (TaB_2), moderate to strong tantalum boride (TaB), weak tridymite, and a weak to moderate undefinable phase.

8. Hafnium nitride (HfN) - Indication of a reaction below 2150°C. (3902°F.). X-ray diffraction of heated material showed major hafnium nitride (HfN), moderate to strong calcium hafnate (CaHfO₃) (possibly from impurities in the crucible), weak hafnium diboride (HfB₂), and weak hafnium oxide (HfO₂).

In the second series of crucible material experiments, crucibles of given materials or coated with given materials containing an 80 percent silica, 15 percent alumina, 5 percent calcia composition were heated to greater than 1800°C. (3270°F.) by graphite heaters in argon. The results are given below:

1. Stabilized zirconia (ZrO₂) - Strongly eroded and dissolved by the melt at temperatures of 1900°C. (3452°F.) and above.
2. Graphite (C) crucibles coated with a monoclinic zirconia (ZrO₂) wash - The wash did not protect the crucible at 1900°C. (3452°F.) and there appeared to be signs of a reaction between the graphite and the melt and/or the zirconia (ZrO₂) and the melt.
3. Graphite crucibles coated with a boron nitride (BN) wash - Appeared to protect the crucible from the melt at temperatures up to 1950°C. (3542°F.).
4. Tungsten (W) - Reacted with the graphite (C) heater below 2000°C. (3632°F.) forming ditungsten carbide (W₂C).
5. Niobium diboride (NbB₂) - No indication of any reaction with either the graphite heater or the melt up to 2450°C. (4442°F.); however, all of the melt quickly evaporated at this temperature.
6. Zircon (ZrSiO₄) - A definite reaction between the crucible and melt at 1950°C. (3542°F.).
7. Graphite (C) - Was somewhat eroded by the composition at 2000°C. (3632°F.).
8. Silicon carbide (SiC) - Slight signs of reaction at 1900°C. (3452°F.).

Contrails

The zirconia (ZrO_2), zircon ($ZrSiO_4$), graphite (C), and silicon carbide (SiC) crucibles were obtained commercially. The boron nitride and zirconia washes were formed by mixing fine powders of each material with alcohol and then painting them onto the sides of the graphite crucibles and allowing them to dry. The tungsten crucible was formed by plasma arc spraying, and the niobium diboride crucible by hot pressing.

Several of the materials which showed signs of slight reaction may be useful as crucible material, but niobium diboride (NbB_2) appears to be by far the most promising of all the materials tested. It has been previously reported on this project that tungsten (W) should be good where no carbon is present.

C. Melting and Fining Studies

The purpose of these studies was to try to obtain a well-fined, high-melting temperature glass which could be premelted and used as starting material for fiber drawing. The procedures used and results obtained in these experiments are given in Table No. I. The fining characteristics of many of the melted batches are shown pictorially in Figures 1 through 27.

Batch numbers 1, 21, 22, 23, 24, 32, 33, 34, 36, 37, 38, 39, 40, 41 and 42 were chosen somewhat at random on the basis of their refractoriness and the possibility that they might form glasses if cooled rapidly. None appeared to be very promising.

Batch numbers 2 through 20 were experiments used to determine the effect of adding certain of the oxides as different compounds (Nos. 4 through 7) and minor additions of various materials (Nos. 8 through 20) on a highly viscous, refractory glass melt. (The added weight of the various materials are over and above the original composition weight which totalled 100). For the purpose of this study, Batch Nos. 2 and 3 are used as standards of comparison since both are of the base composition, disregarding the difference of $150^{\circ}C$ ($270^{\circ}F$) in melt temperature. Barium oxide is not usually added to a mix as the single oxide because it rapidly picks up water. In the previous work barium peroxide was used in place of barium oxide. It was felt, however, that the oxide might possibly be added with better results in some other form. Barium carbonate (No. 5), barium fluoride (No. 6) and barium sulfate (No. 4) were tried but none proved to be any better than the barium peroxide. Aluminum phosphate (No. 7) was substituted directly for the alumina in one case but was found to produce poorer fining.

The effects of carbon (No. 8), boron nitride (No. 9), and ferric oxide (No. 10), were suggested as being harmful to the glass compositions. For this reason minor amounts of these materials were added to the given batch and their effects were observed. Carbon definitely caused much poorer fining; boron nitride had no observable effect; and ferric oxide produced a lighter colored and somewhat better fining, possibly by lowering the viscosity and melting point.

Ammonium nitride (No. 11), ammonium sulfate (Nos. 12 and 13), and tungsten trioxide (No. 14) were suggested as agents to improve fining but were found instead to produce adverse effects.

The effects of vanadium pentoxide (Nos. 15, 16 and 17) and zirconia (Nos. 18, 19 and 20) on the fining characteristics of the melt were expected to be quite different from one another. This proved to be the case. The vanadium pentoxide-containing compositions produced bubble-free and apparently low viscosity melts in contrast to the apparently high viscosity many-bubbled zirconia (ZrO_2) containing melts.

In Nos. 25 through 29 the fining characteristics of a given melt were studied and a well-fined premelt was produced for fiber drawing experiments.

Nos. 30 and 31 were conducted to determine the effects of vanadium pentoxide (V_2O_5) and zirconia (ZrO_2), respectively, on the melt composition of Nos. 25 through 29. The melt with vanadium pentoxide was well-fined while the one with zirconia was completely vaporized.

No. 35 is a high potash feldspar. It was hoped that the high viscosity of this material at its melting temperature would produce strong fibers; however, from the fining experiment, bubbles appear to be a difficulty.

Nos. 43 through 46 were conducted to investigate the effects of the conditions encountered with previous melting and fining experiments on a common window glass composition. Of particular interest was the lack of vaporization from this comparatively low melting glass.

Pieces of melts No. 3 and No. 29 were examined under an optical microscope. No signs of non-homogeneous material were observed in No. 3; however, No. 29 was found to contain many small (approximately one micron in diameter) spherical particles of unknown origin. These particles were dark in color and spherical in shape. No crystalline phase could be detected by x-ray diffraction.

D. Filament Drawing Studies

Fiber was drawn on several occasions from approximately 1/2-inch diameter chunks of 80 percent silica, 15 percent alumina, 5 percent calcia premelt listed in Table No. I as Nos. 28 and 29. It was hoped that by starting with this relatively well-fined material a strong, homogeneous, bubble-free filament would be produced.

The room temperature tensile strength of the filaments produced varied from 31,900 pounds per square inch to 116,000 pounds per square inch with the fiber diameter varying from 39 to 18.5 microns. Tested at 815°C. (1500°F.), the filaments did not break but simply stretched out.

The filaments were examined under an optical microscope and were found to have small crystallites on their surfaces and to contain striations and bubbles.

III. CONCLUSIONS

1. Niobium diboride appears to be a promising crucible material for melting high silica glass compositions.
2. Vanadium pentoxide aids in the melting and fining of high silica glass compositions.
3. No advantages were gained by using premelt as a starting material for filament drawing.



Plate No. 7936

Figure 1 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂ - Heated 15 Minutes
at 2050°C. (3720°F.)

Contrails

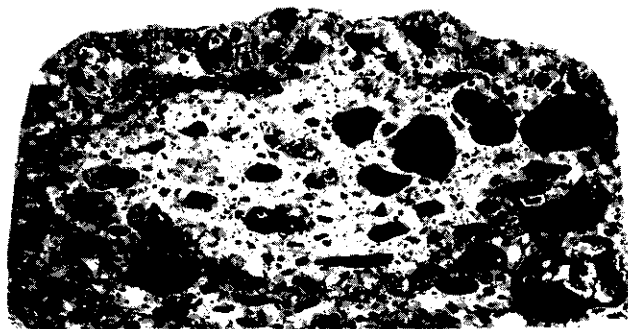


Plate No. 7938

Figure 2 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂, 5 C - Heated 15
Minutes at 2050°C. (3720°F.)

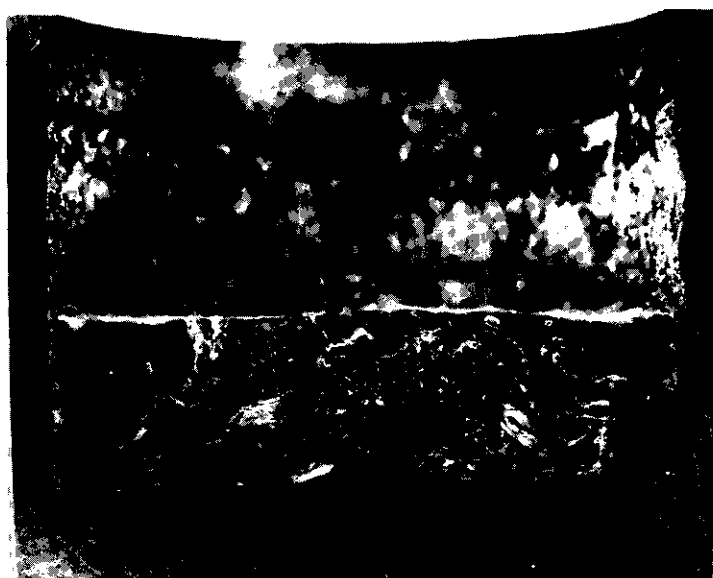


Plate No. 7939

Figure 3 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂, 5 BN - Heated 15
Minutes at 2050°C. (3720°F.)

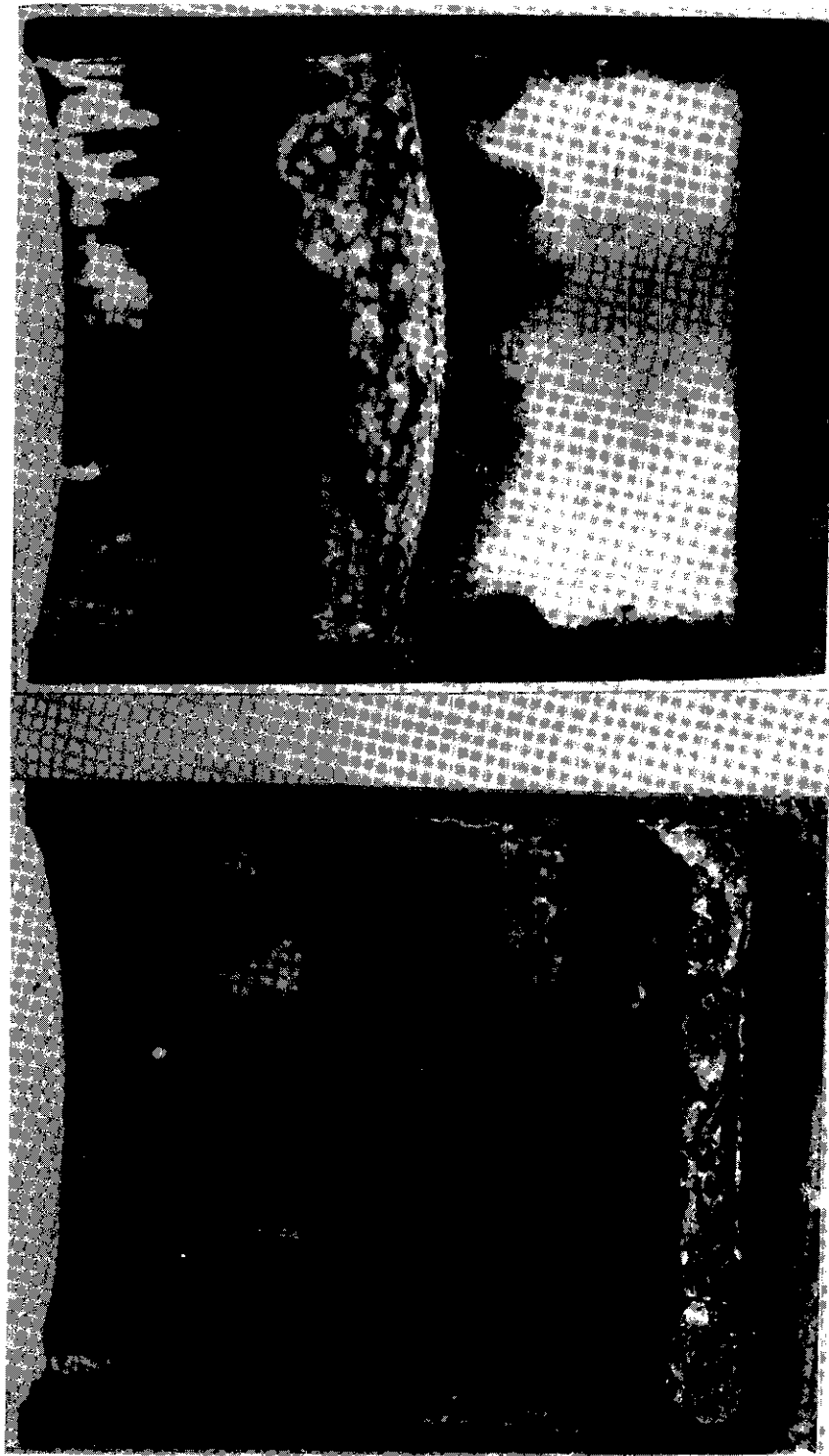


Plate No. 7949

Figure 4 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
2.5 Fe₂O₃ - Heated 15
Minutes at 2050°C. (3720°F.)

Plate No. 7940

Figure 5 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
1 NH₄NO₃ - Heated 15 Minutes
at 2050°C. (3720°F.)



Plate No. 7954

Figure 7 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
5 (NH₄)₂SO₄ - Heated 15 Minutes
at 2050°C. (3720°F.)

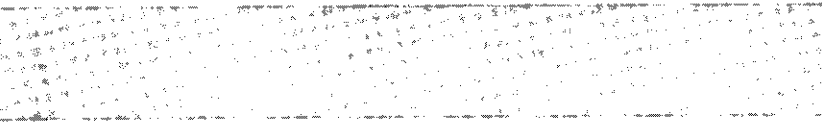


Plate No. 7953

Figure 6 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂
2.5 (NH₄)₂SO₄ - Heated 15
Minutes at 2050°C. (3720°F.)



Plate No. 7943

Figure 9 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
2.5 V₂O₅ - Heated 15 Minutes
at 2050°C. (3720°F.)



Plate No. 7941

Figure 8 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
1 WO₃ - Heated 15 Minutes
at 2050°C. (3720°F.)

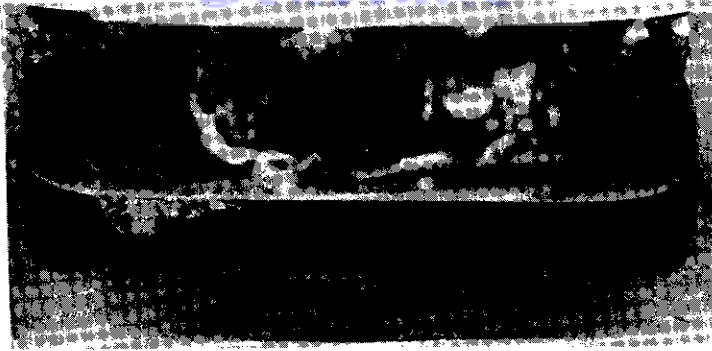


Plate No. 7942

Figure 10 - 90 SiO_2 , $6 \text{ Al}_2\text{O}_3$, 4.4 BaO_2 , $5 \text{ V}_2\text{O}_5$ - Heated 15 Minutes at 2050°C . (3720°F .)



Plate No. 7944

Figure 11 - 90 SiO_2 , $6 \text{ Al}_2\text{O}_3$, 4.4 BaO_2 , $10 \text{ V}_2\text{O}_5$ - Heated 15 Minutes at 2050°C . (3720°F .)



Plate No. 7945

Figure 12 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
2.5 ZrO₂ - Heated 15
Minutes at 2050°C. (3720°F.)



Plate No. 7946

Figure 13 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
5 ZrO₂ - Heated 15 Minutes
at 2050°C. (3720°F.)

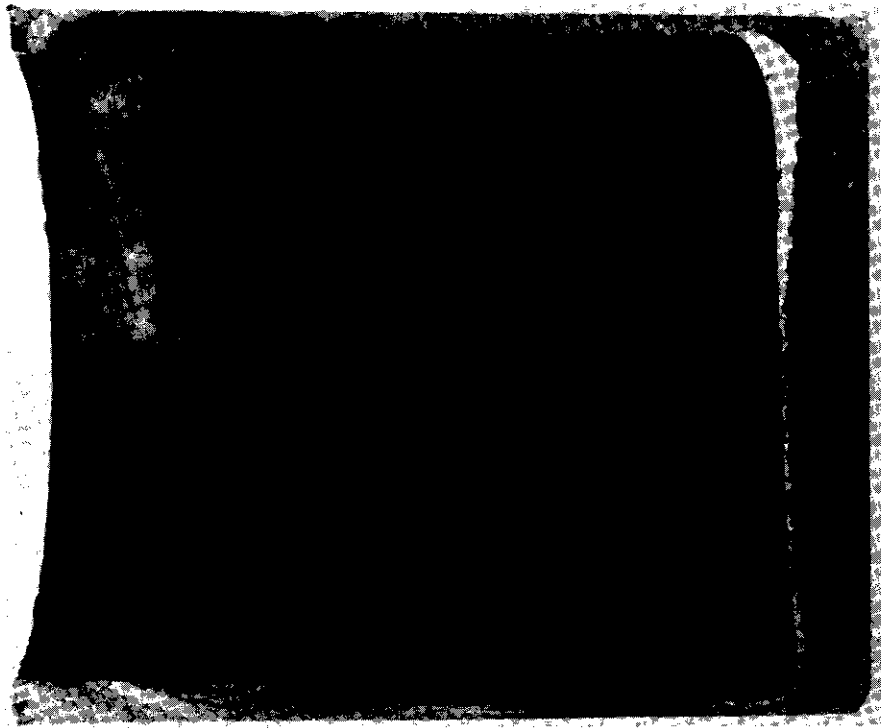


Plate No. 7947

Figure 14 - 90 SiO₂, 6 Al₂O₃, 4.4 BaO₂,
10 ZrO₂ - Heated 15 Minutes
at 2050°C. (3720°F.)



Plate No. 7933

Figure 15 - 90 SiO₂, 8 Al₂O₃, 1 K₂O, 1 MgO
- Heated 15 Minutes at 2050°C.
(3720°F.)



Plate No. 7771

Figure 17 - 80 SiO₂, 15 Al₂O₃, 5 CaO -
Heated 5 Minutes at 1850°C.
(3360°F.)



Plate No. 7934

Figure 16 - 84 SiO₂, 13.5 Al₂O₃, 5 Na₂O,
2 K₂O - Heated 15 Minutes
at 1950°C. (3540°F.)

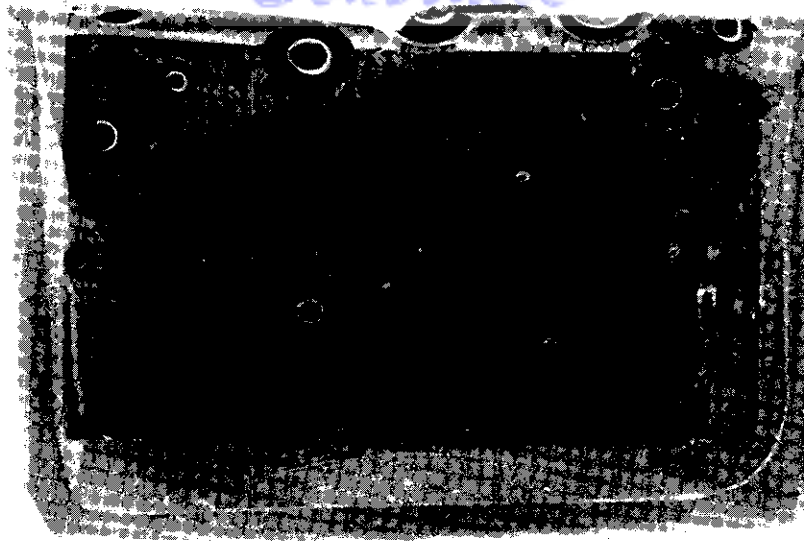


Plate No. 7830

Figure 18 - 80 SiO₂, 15 Al₂O₃, 5 CaO - Heated 5 Minutes at 1900°C. (3450°F.)



Plate No. 7928

Figure 19 - 80 SiO₂, 15 Al₂O₃, 5 CaO - Heated 15 Minutes at 1950°C. (3540°F.)

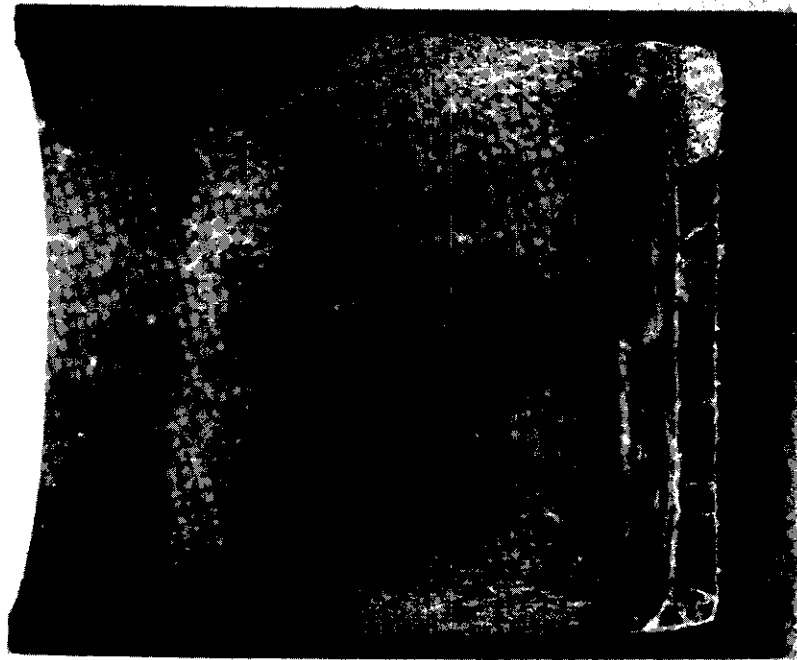


Plate No. 7951

Figure 21 - 80 SiO₂, 15 Al₂O₃, 5 CaO, V₂O₅ -
Heated 15 Minutes at 2050°C. (3770°F.)

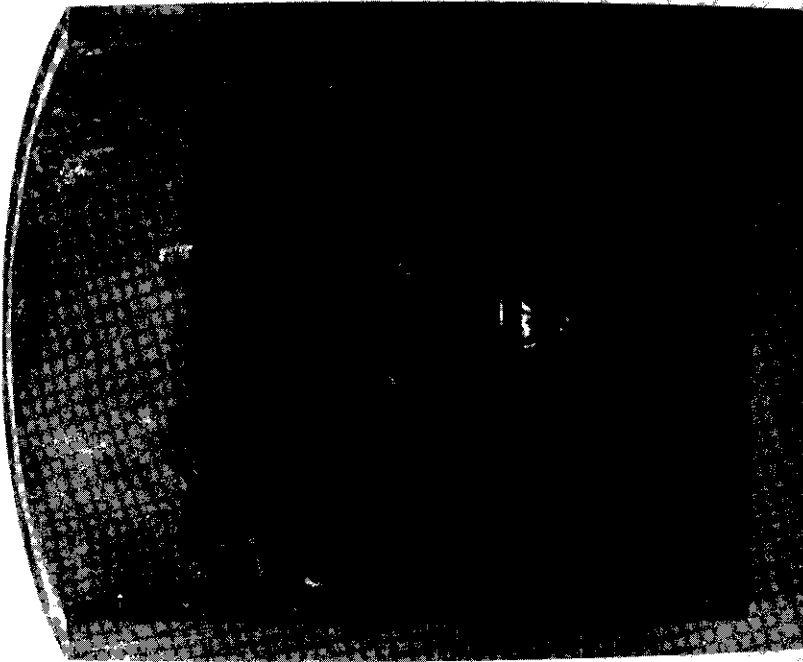


Plate No. 7831-A

Figure 20 - 80 SiO₂, 15 Al₂O₃, 5 CaO -
Heated 20 Minutes at 1950°C.
(3540°F.)

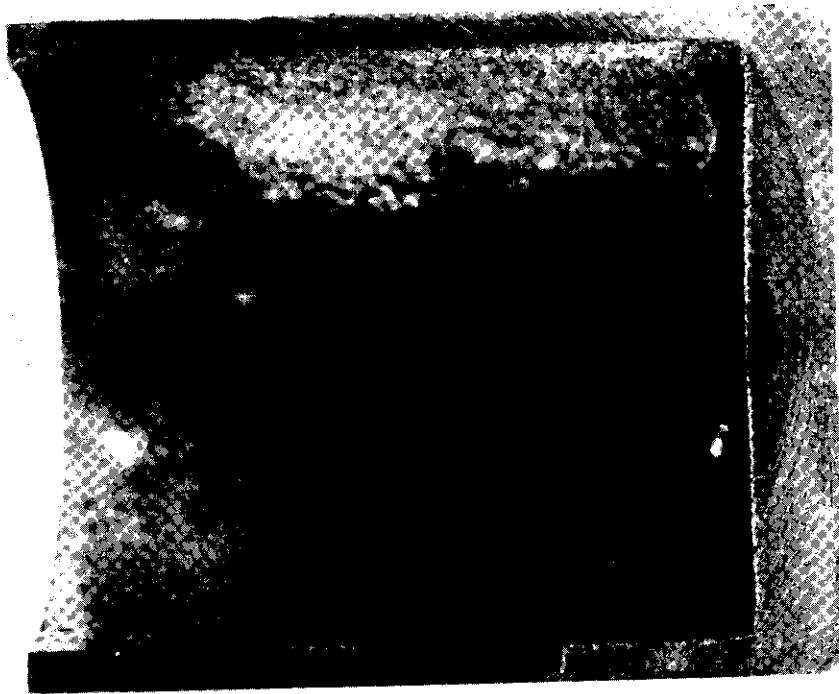


Plate No. 7950

Figure 22 - 80 SiO₂, 15 Al₂O₃, 5 CaO, 5 ZrO₂
- Heated 15 Minutes at 2050°C.
(3720°F.)

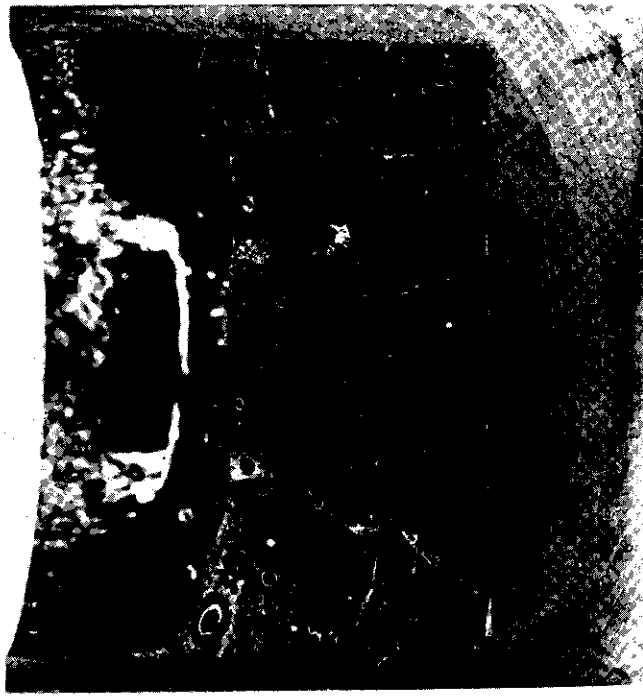


Plate No. 7935

Figure 23 - 68.6 SiO₂, 18.2 Al₂O₃, 3 Na₂O,
10 K₂O, .2 MgO - Heated 15
Minutes at 1750°C. (3180°F.)



Plate No. 7929

Figure 24 - 73.5 SiO₂, 10.5 CaO, 16.0 Na₂O - Heated 15 Minutes
at 1500°C. (2730°F.)

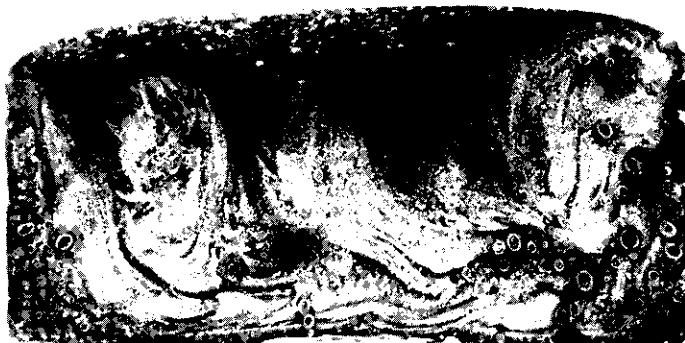


Plate No. 7930

Figure 25 - 73.5 SiO₂, 10.5 CaO, 16.0 Na₂O - Heated 15 Minutes
at 1700°C. (3090°F.)



Plate No. 7932

Figure 27 - 73.5 SiO₂, 10.5 CaO, 16.0 Na₂O -
Heated 15 Minutes at 1950°C.
(3540°F.)



Plate No. 7931

Figure 26 - 73.5 SiO₂, 10.5 CaO, 16.0 Na₂O -
Heated 15 Minutes at 1850°C.
(3360°F.)