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WADC TECHNICAL REPORT 55-327

AN INVESTIGATION OF THE FEASIBILITY OF PRODUCING METAL BONDED CARBIDE BODIES BY THE EXTRUSION PROCESS

WILLIAM W. WELLBORN

FIRTH STERLING INC.

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MATERIALS LABORATORY
CONTRACT No. AF 33(616)-229
PROJECT No. 7350
TASK No. 73500

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
300 - April 1956

FOREWORD

This report was prepared by Firth Sterling Inc., under USAF Contract Number AF 33(616)-229. This contract was initiated under Project No. 7350, "Ceramic and Cermet Materials", Task No. 73500, "Ceramic and Cermet Materials Development", formerly RDO No. 615-17, "Ceramic Materials", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. L. D. Richardson acting as project engineer.

This report covers the period of work from July 1952 to May 1955.

ABSTRACT

The main purpose of this investigation was to determine if useful metal-bonded carbide objects could be produced by the extrusion process. A further aim of this investigation was the comparison of the properties of such objects as might be produced by extrusion, with the properties of objects of the same composition produced by pressing, shaping, and sintering. The principal equipment used was a 1050-ton extrusion press, which had been built to extrude carbide charges.

The investigation revealed that it was possible to extrude these bodies into some shapes. However, shapes with irregular cross sections, or thin sharp edges, were extremely difficult to extrude and it was impossible to hold size. The physical properties of extruded material were found to be measurably lower than materials compacted by conventional means. The incidence of flaws in extruded material was found to be greater.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. Whitmore
Technical Director
Materials Laboratory
Directorate of Research

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I. INTRODUCTION

It has been common practice in the ceramic and hard metal industries, to produce certain shapes by the powder extrusion process. The process used involves the suspension of the metal or ceramic powders involved in some viscous fluid carrier to enhance their ability to flow. This mixture is placed in a chamber as shown in Figure 1. Pressure is applied by means of the ram, and the material is compressed to the desired shape when it is caused to flow through the correctly shaped nozzle.

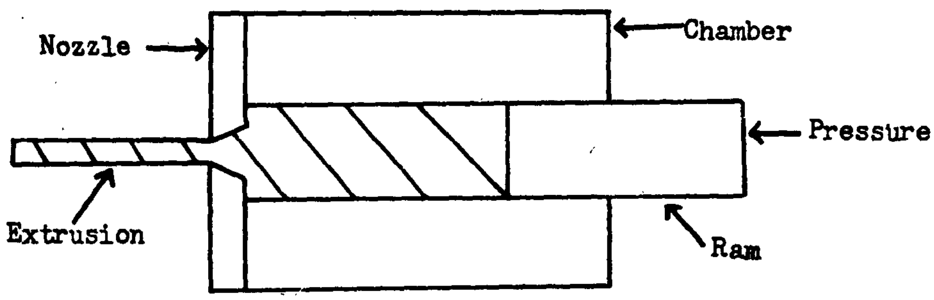


Figure 1. Simplified Illustration of the Powder Extrusion Process

The application of this process to the production of objects useful in aircraft engines, for instance nozzle diaphragm blades, presents difficulties not encountered in either the hard-metal or ceramic industries. Extrusion of ceramics is devoted primarily to the production of various types of ceramic tubing, such as thermocouple protective tubing. These tubes require little more strength than is necessary to support their own weight. High density is not required, since the tubes can be made gas or liquid tight by subsequent glazing. The cross-sections of such material are regular and symmetrical. In the hard-metal industry, powder extrusion is used to produce rods, bars, and tubes of a relatively small diameter and also with regular cross-sections. It is a recognized fact that extruded, cemented carbide compositions have only a percentage of the strength of a conventional cold pressed and sintered composition. They are generally more porous, which to some extent affects the hardness. It is possible to make up for these deficiences however, by adding other carbides or using a different higher strength composition. The case of the metal bonded carbides (mainly titanium carbide bonded with nickel) intended for use at high temperatures in aircraft engines is somewhat different. These bodies which require high resistance to oxidation, high temperature strength, and high impact strength, are generally at their operative limit as conventionally fabricated. There is not room for a decrease in properties, which is attributable to the fabrication process.

It was hoped that methods of powder extrusion could be developed that would either eliminate the drop in physical properties or actually yield improved properties.

The principal points to be investigated were:

(1) Types of plasticizers or extrusion carriers
(2) The quantity and method of adding these plasticizers
(3) Method of elimination or removal of these plasticizers prior to or during sintering
(4) Extrusion die or nozzle design
(5) The effect of pressure and rate of extrusion on the final physical properties.

Evaluation of the effect of these process variables was to be made on 15 stress rupture specimens to be furnished to Wright Air Development Center. If, in addition, the investigation yielded methods that could successfully produce them, 25 nozzle diaphragm blades were to be supplied to Wright Air Development Center for further evaluation tests. The ultimate goal of the investigation then was to develop a method which would produce by powder extrusion, nozzle diaphragm blades which had identical or improved physical properties with respect to those produced by cold pressing and sintering techniques.

II. EXPERIMENTAL PROCEDURES AND RESULTS

A. General Description of Powder Extrusion Process

In Figure 2 is shown a general flow diagram of the powder extrusion process. The equipment used in each numbered step is described in the following paragraphs.

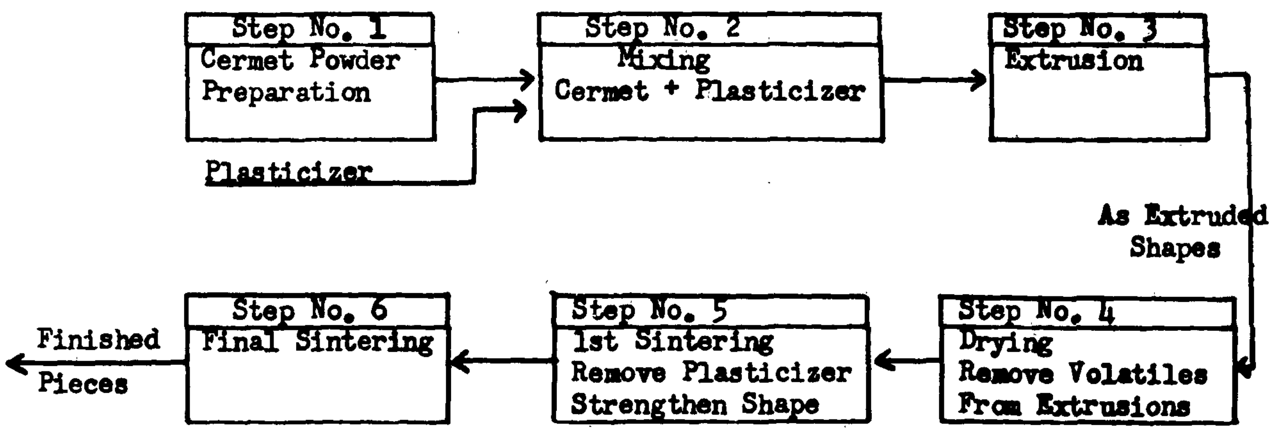


Figure 2. General Flow Sheet for Powder Extrusion Process

Step No. 1 - Cermet Powder Preparation

It was decided that it would be advantageous to use a standard cermet composition. This decision was made because of the obvious advantages of working with compositions whose physical properties as produced were well known. Accordingly, Firth Sterling's grade FS-26 was chosen as one having properties most nearly suited for a diaphragm blade. This grade is composed of titanium carbide (TiC) 54.3%, chromium carbide (Cr_3C_2) 5.7%, nickel (Ni) 40%. The properties of this material are listed in Table I.

Table I. Properties of Firth Sterling Grade FS-26

Density, grams per cc.	6.24
Hardness, Rockwell A	85
Transverse Rupture Strength, psi.	165,000
Oxidation Resistance at 1800°F, Weight gain in mg. per cm^2 after	
50 hours	21.2
100 hours	31.5
200 hours	47.6
Tensile Strength, psi., and Elongation, %	
75°F	32,900-Nil
800°F	41,000-Nil
1200°F	48,200-Nil
1500°F	51,400-Nil
1800°F	41,000-0.8
Impact Resistance, Ft.-lb., on standard size, unnotched Charpy bars	
75°F	4.5
800°F	4.5
1200°F	4.9
1500°F	4.8
1800°F	4.4
Stress, psi., and Elongation, % for 100-hour rupture	
1350°F	42,000-0.8
1500°F	30,000-1.1
1600°F	20,500-1.2
1800°F	11,100-5.6

Step No. 2 - Mixing

The equipment used in the mixing of cermet powder plus plasticizer consisted of a small Lancaster sand muller used in the experimental evaluation of various plasticizers, a large dough mixer, similar to the type used in bakeries, a large Simpson sand muller, and finally a day ribbon mixer which was determined to be the best piece of equipment for adding the plasticizer in the final large batches. In the very early experimental stages, mixing was accomplished by hand using spatulas.

Step No. 3 - Extrusion

Two extrusion presses were used during this project. The small 20-ton capacity extrusion press shown in Figure 3 was used in the experimental evaluation of various plasticizers and in making determinations for extrusion nozzle design for the large 1050-ton extrusion press shown in Figure 4.

Figure 3. Twenty-ton Extrusion Press

The 1050-ton extrusion press in Figure 4 was loaned by the Air Force to Firth Sterling for the accomplishment of this project. The press was originally designed and built for the extrusion of cordite charges. It delivers 1050 tons total load on a 15-inch diameter extrusion ram. It is equipped so that the extrusion chamber may be evacuated. The die section may be either heated to temperatures obtainable with 100 pounds of steam or water cooled. The hydraulic system of this press consists of a high volume pump for rapid traverse, and a high pressure pump to

Figure 4. Ten-hundred fifty Ton Extrusion Press

supplement in order to obtain maximum loading. These pumps are driven by a variable speed drive which enables the operator to obtain some variance in the rate of extrusion. The hydraulic system is coupled directly to the pressure piston of the press, without benefit of an air or water buffer. Accordingly, at some speeds its operation is somewhat erratic. Two dies to produce two different shapes, a solid round, one inch in diameter and an air-foil section, were designed and constructed for this press. These dies are described later in this report.

In addition to the presses, a number of trays were constructed to receive material as it was extruded from the two presses. In general, these trays were made of graphite and each had the shape of the piece being extruded pressed upon it. Additional equipment was used in the extrusion step, such as weighing equipment for weighing out charges, a cart which was constructed to carry the cermet-plus-plasticizer mixture from the mixer to the large extrusion press, and a special hydraulic die puller for removing the die inserts from the large extrusion press.

Step No. 4 - Drying

Two Stokes vacuum drying ovens were available for drying. One of these ovens was equipped so that hot drying air could be circulated through it. The other was conventionally set up for vacuum drying. Some drying was done through the use of infrared bulbs in atmospheric air. In addition, the furnace constructed for preliminary sintering in Step No. 5 was equipped so that the heat could be controlled in five zones. Final drying was accomplished in the first two zones of the preliminary sintering furnace in some cases.

Step No. 5 - Preliminary Sintering

Preliminary sintering was accomplished in the furnace shown in Figure 5. This furnace is equipped with three zones of gas burners, a charge zone at the entrance end, and a cooling zone at the exit end. The Inconel muffle through the furnace has a rectangular cross-section 5 inches by 7 inches. It is equipped in the charging end with a variable rate stoking device. Hydrogen can be put into the muffle from either end or the center of the furnace. The total of seven gas burners can be adjusted separately to obtain three zones of different temperatures; and by controlling the point of entrance and the flow of the hydrogen gas, the temperature of the charging zone and cooling chambers may also be controlled. Some preliminary sintering was accomplished in a conventional vacuum sintering furnace. All preliminary sintering was done by charging the material as it rested on the shaped graphite trays on which it was caught during extrusion. Temperatures were measured at various zones in the hydrogen preliminary sintering furnace and in the vacuum sintering furnace by chromel-alumel thermocouples. A Leeds and Northrup potentiometer was used as the measuring instrument.

Step No. 6 - Final Sintering

Final sintering was accomplished in the vacuum sintering furnaces shown in Figure 6. Ultimate sintering was carried out by the Firth Sterling cermet division in equipment and by techniques which were conventional for Firth Sterling grade FS-26.

Finished Pieces

In the case of the 15 stress rupture specimens prepared for submission to WADC Materials Laboratory, these specimens were prepared by centerless grinding 3/4 inch extruded bars to a proper diameter and grinding the reduced section in a cylindrical grinder.

B. Method for Determining Best Plasticizer for Titanium-Carbide-Base Cermets

The effectiveness of various plasticizers was determined by extruding a mixture of FS-26 cermet and the plasticizer in question, through a 1/4 inch nozzle in the 20-ton extrusion press shown in Figure 3. These pieces were then dried, first sintered in hydrogen atmosphere furnaces, and finally sintered in vacuum. Evaluations of the performance of the plasticizer were made at each stage of the process. Factors evaluated at each stage are listed on page 8.

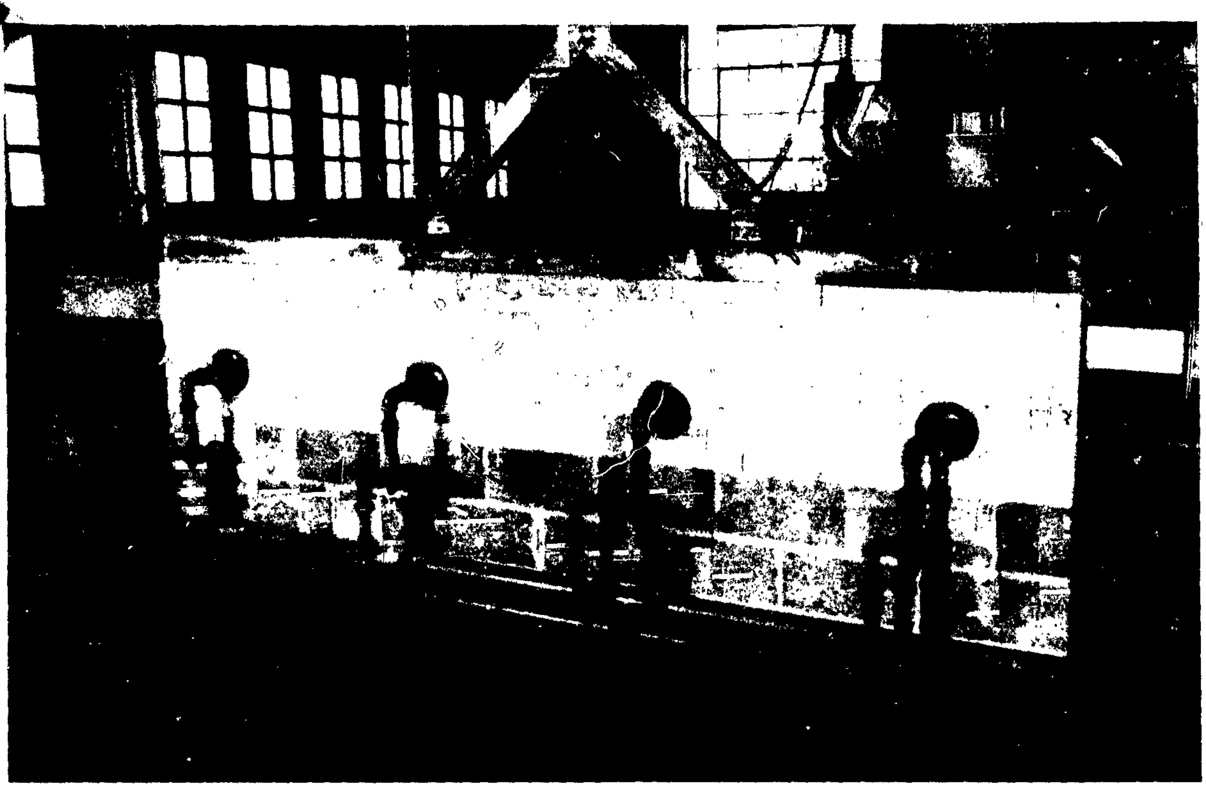


Figure 5. Preliminary Sintering Furnace

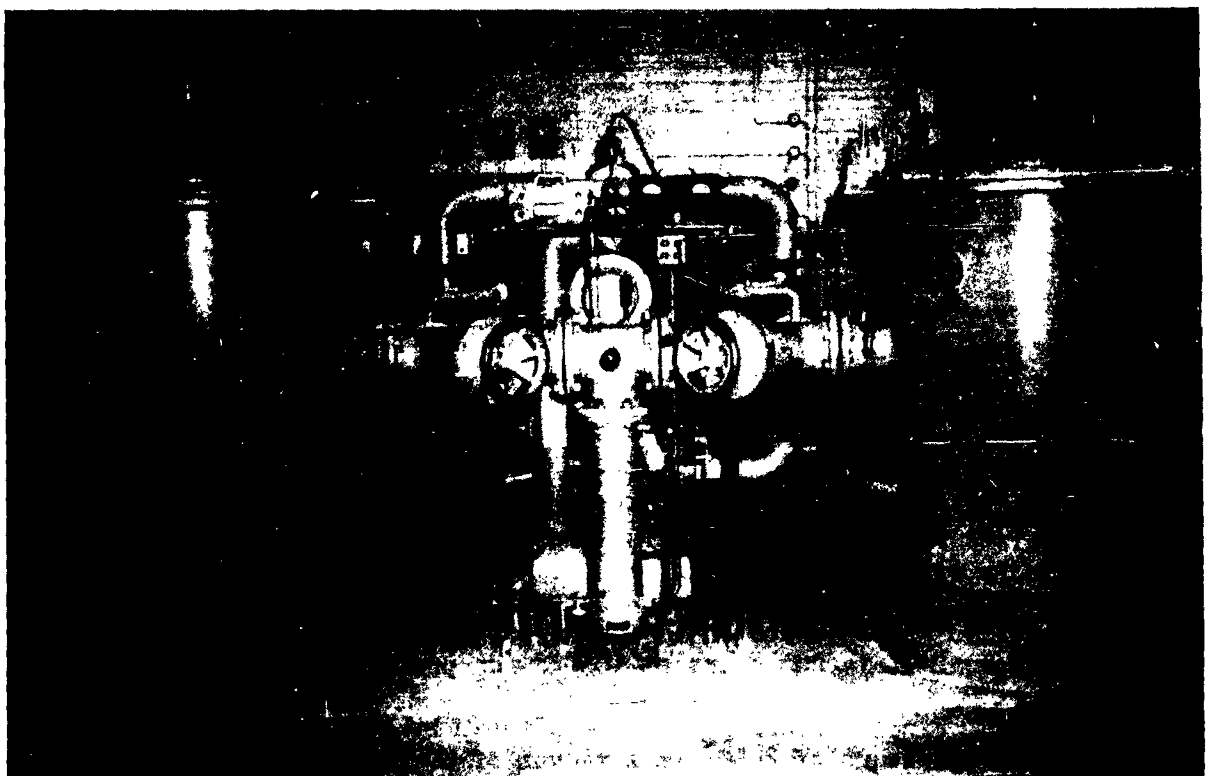


Figure 6. Vacuum Sintering Furnaces

1. Ease of extrusion
2. Ability of the material to hold size and shape
3. Strength and ability to be handled
4. General porosity
5. Amount of internal and external cracking
6. Amount of warpage

All extrusions to evaluate plasticizers were carried out according to the following procedures:

1. Two-hundred grams of FS-26 powder were thoroughly mixed by hand with the plasticizer.

2. This material was placed into a one-inch cylinder in the 20-ton extrusion press of Figure 3.

3. A solid plug was put in place of the die and the material compacted until pressure just began to show on the gage.

4. The solid plug was removed and a .200-inch round die nozzle with a hemispherical entranco angle was put in its place. This gave a reduction of area of approximately 25 times.

5. The extrusion ram was caused to move forward at the rate of 2-1/2 inches per minute, which produced approximately 60 inches per minute of extruded rod.

6. Pressures necessary to maintain this rate were observed and recorded.

7. Extrusions were caught in half-cylindrical graphite boats as illustrated in Figure 3.

8. The extruded material was dried in air at room temperature for 24 hours.

9. Further drying was accomplished by placing the room-temperature dried material in an oven at 150°F. The oven was heated by circulating heated air through it from an external heater. Temperature in the heater was controlled at 350°F.

10. The dried extrusions were first sintered in Inconel boats, still resting on the graphite slabs on which they were extruded. The pieces and slabs were covered with minus 100 plus 120 mesh graphite powder to further protect them from oxidation. The pieces were then first sintered by stoking them through a 30-inch uniform heat zone furnace, regulated at 1550°F, at a rate of 14 inches per hour in a hydrogen atmosphere. This gives approximately two hours at 1550°F for preliminary sintering.

11. Final sintering was accomplished by sintering for 30 minutes at a temperature of 2450° in a vacuum of 50 microns or less.

The following types of plasticizers were investigated:

1. Petroleum jelly
2. Camphor and oil
3. Methyl cellulose - H_2O solutions

Where initial trials of these materials so warranted it, the proportions were varied accordingly. The results of these trials are recorded in the following paragraphs under heading of the appropriate type of plasticizer.

Petroleum Jelly

Mixtures of 6, 10 and 15% by weight Cities Service Petroleum Jelly, the balance FS-26 cermet powder, were prepared in the previously described method. The mixtures containing 6% and 10% petroleum jelly both failed to extrude through the nozzle at a total load of 20 tons. This is equal to a pressure of 25 tons per square inch on the one-inch diameter extrusion cylinder. The maximum pressure obtainable on the large 1050-ton press, to be used later, is approximately 6 tons per square inch. The 6% and 10% mixtures were not considered usable for this reason. The 15% mixture extruded easily and smoothly. It was soft and bent quite easily. After drying for 24 hours in air at room temperature, it was somewhat firm and not as easily bent; however, it still could not be handled without distortion. During drying at 150°F the rods sagged and completely lost their shape. No satisfactory drying procedure was found to prevent this sagging and loss of shape. Petroleum jelly was not considered to be a satisfactory plasticizer.

Camphor and Oil

Two different combinations of camphor and oil were tried and their performance is listed below.

1. Spirits of camphor, 2 parts - Camphorated oil, 1 part

   Forty-five cc. of the above mixture was added to 200 grams of FS-26 powder. This mixture extruded well, the surface was smooth with no apparent surface checks. It would support its own weight in 7-inch lengths, which indicated fair strength. For optimum strength, 12 to 14-inch lengths should be capable of supporting their own weight when held by one end. These pieces were dried, and first sintered without developing any observable flaws. After each of the above steps, the strength was somewhat lower than desired. After final sintering there was neither external nor internal cracks, no appreciable warpage, no loss of shape. The extrusions were porous, however; the density was low and the transverse strength was only 75% of the expected value. It would then appear that the viscosity of this mix was insufficient to obtain the necessary compaction. The performance of this plasticizer was sufficiently good to warrant additional trials of the material in other proportions.

2. Spirits of camphor, 2 parts - Camphorated oil, 1 part

Thirty-five cc. of the above mixture was added to 200 grams of FS-26. This material extruded well, as did the first camphor mixture, with no reading on the pressure gage. There was no evidence of flaws after extrusion, but the strength was inadequate since only a 6-inch length would support its own weight. No further flaws developed in drying. The material broke into small sections during first sintering. It was concluded that the viscosity of this material was insufficient to serve as a proper plasticizer.

Methyl Cellulose - H_2O Solutions

The methyl cellulose used was that marketed by the Dow Chemical Company, under the trade name Methocel. The grade was the 4000 C.P.S. grade. A basic solution consisted of 2% by weight Methocel and 98% by weight distilled water. This basic solution was added to FS-26 powder in various proportions as indicated in the following paragraphs.

1. 23% by weight - 2% Methocel solution, Balance FS-26 powder

This material was far too soft; it squirted through the nozzle easily with no showing of pressure on the gage. It would not hold form at all on extrusion. It was decided to reduce the amount of Methocel solution to obtain greater viscosity.

2. 20% by weight - 2% Methocel solution, Balance FS-26 powder

Material extruded under a total load of 2 tons held form, but rippled and cracked at the top of each ripple. The strength and rigidity were unsatisfactory.

3. 16% by weight - 2% Methocel solution, Balance FS-26 powder

This material extruded smoothly and uniformly under a total load of 5 tons. A 20-inch length successfully supported its own weight. There was no evidence of flaws. After drying, the material showed no evidence of flaws. It was extremely rigid and quite hard. A 12-inch length of the extrusion could be held horizontally by one end without breaking. Density of this material was determined to be in excess of that produced in cold pressed and sintered materials in the as-pressed condition. First sintering produced no evidence of flaws. Final sintering produced excellent results. There was no evidence of cracks or flaws. Warpage was very minor. The shrinkage was 16% during sintering as compared to 19% for cold pressed and sintered material. All measurable properties (density, hardness, transverse strength) were normal for FS-26. Size and shape were uniform. It was concluded that 16% by weight of a 2% methyl cellulose solution was the best plasticizer available for the extrusion of FS-26 powders.

C. Method for Determining the Correct Extrusion Die Nozzle Entrance Angle

In the powder extrusion process, material flowing from the extrusion chamber and out through the extrusion nozzle flows at a definite angle. This angle for purposes of discussion in this report, will be referred to as the nozzle entrance angle. The angle of the flow into the nozzle is affected by a number of variables. It is affected principally by the viscosity of the mix and the friction between the two surfaces along the nozzle entrance angle. Extrusion pressure and rate also influence the nozzle entrance angle, but to a much lesser degree. If no entrance angle is used, in other words if the nozzle is simply a straight hole whose sides are parallel to the extrusion produced, then the material will establish its own entrance angle as illustrated in Figure 7. This will be the maximum allowable entrance angle into the die for minimum pressure and maximum material viscosity. Upon determining this maximum entrance angle, the same angle can be built into the steel nozzle. This will materially reduce the friction and thereby reduce the pressure necessary to obtain the same degree of compaction of materials of like viscosity.

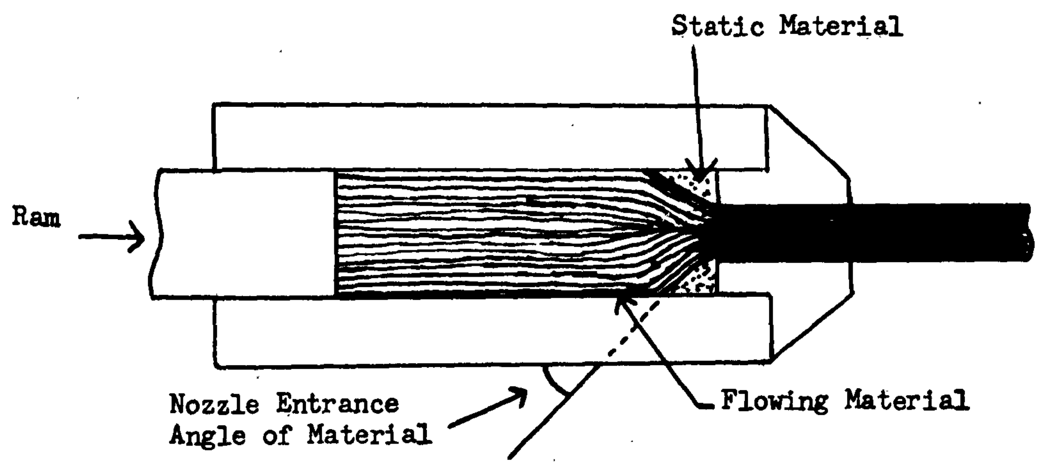


Figure 7. Method of Determining Nozzle Entrance Angle

The maximum nozzle entrance angle possible gives the maximum reduction and maximum flow rate. If this angle is increased above maximum, the material will compensate and retain the maximum angle. If this angle is decreased below maximum reduction, flow rate and compaction are decreased for each given viscosity. The maximum nozzle entrance angle was determined for the FS-26, 16% Methyl cellulose solution mixture, by placing two slightly compacted slugs of the mixture in the chamber of the 20-ton extrusion press, and pressing them through a steel nozzle with a 90° entrance angle. The slugs were placed one on the other, and blue coloring had been added to the lower one and black coloring to the upper one. The material was extruded until a solid round of the black material appeared. The nozzle was then removed and the slug behind it pressed out. The cylindrical slug was then sectioned along its center section and the angle of the black with the blue material was measured. The maximum nozzle entrance angle for the above material was found to be 47°.

By further experimentation it was found that this angle could be increased to as much as 52° by building it into the nozzle. This was made possible by the reduction of friction between the cermet and the metal nozzle surfaces, as compared with the friction of the material sliding over itself. It was decided to standardize on a 45° nozzle entrance angle for this material.

Round Die Design

Accordingly, the extrusion nozzle shown in Figure 8 was then designed for the purpose of extruding solid rounds for test specimens on the 1050-ton extrusion press. The nozzle entrance angle was set as 45°. The bearing was made so as to extrude a one-inch round. The extremely long bearing was not desirable (approximately 14 inches), but was unavoidable because of the distance through the die holding block of the press. A short relief was allowed at the exit end of the nozzle, but any further relief would have resulted in too much expansion and distortion of the extrusion. The cap and stop rod were for the purpose of sealing the press during de-airing of the mix, and to allow for the production of a solid slug in back of the nozzle. The material of construction was mild steel, which had been forged to minimize voids. The best machine finish obtainable was asked for in the nozzle entrance angle and bearing. The bearing was produced by drilling and reaming.

D. Mixing FS-26 Powder and Plasticizer

In the early stages of plasticizer evaluation, mixing was accomplished by using spatulas. It was determined that two types of mixing were desirable.

When the mix was first made it was composed largely of extremely liquid material plus some drier material and a stirring action was necessary. After the liquid carrier for the plasticizer became more evenly dispersed, a kneading action was desirable. Accordingly, a search was made for mixers which incorporated both of these principles.

The first trial was on a sand muller which uses rotating scrapers set at an angle for a stirring action and these scrapers are followed by a roller which may be set at various heights above the bottom of the mixer to obtain different degrees of kneading action. On this type of mixer, a great deal of sticking, particularly sticking to the roller, was encountered. Considerable heat from friction was involved which caused the plasticizer to set and a granulated rather than a kneaded mass was obtained. It was concluded that the sand muller was not satisfactory for mixing.

The second type of mixer tried was a rotating eccentric mixer of the type ordinarily used to stir and knead bread dough in bakeries. This mixer provided the necessary mixing action on small batches, but we were unable to find one capable of handling the necessary 125-pound minimum charge for the 1050-ton extrusion press. In addition, all the available mixers of this type had stainless steel mixing bowls and paddles, which very rapidly wore out in stirring and kneading the very abrasive carbide mix.

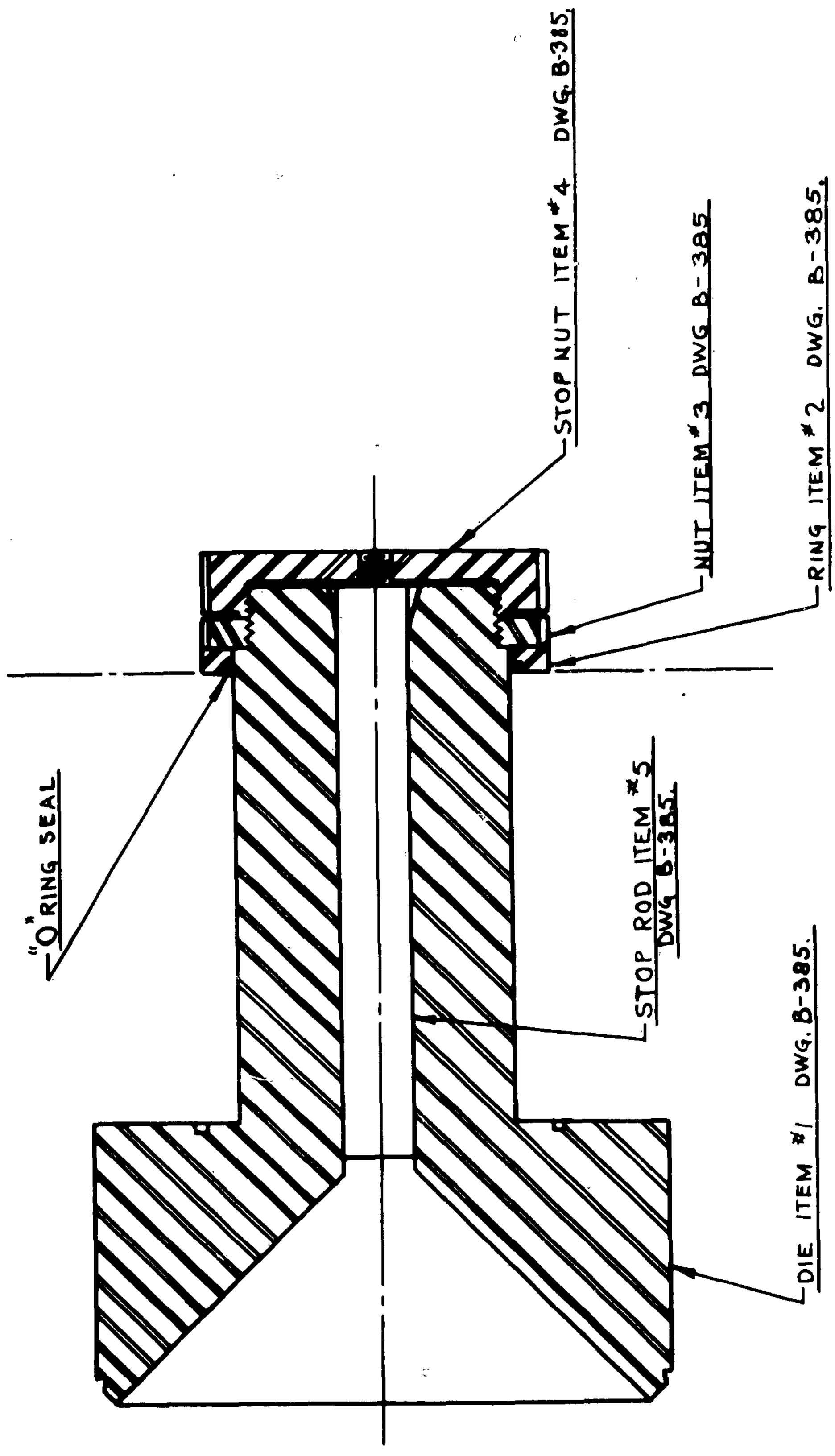


Figure 8. One-Inch Solid Round Extrusion Die

A ribbon type mixer with two entwining helical ribbons and equipped with a jacket which could be used to either heat or cool the charge was tried. This mixer did an excellent job of stirring and kneading. There was a fairly large amount of heat involved, but this was easily removed by putting cold water into the jacket. For this reason the plasticizer and subsequent granulation which was encountered in the sand muller was avoided. It was decided to use the ribbon type mixer for the mixing operation for the 1050-ton extrusion press. The ribbon type mixer is shown in Figure 9.

Figure 9. Ribbon Type Mixer

A study was begun to determine the best method of adding the various ingredients to the mixer in order to accomplish the best mixing. Addition of a 2% solution of methyl cellulose to FS-26 powder proved to be practical but slow and it was difficult to control the consistency of the mixture because of the heat involved by friction. In addition, a considerable amount of evaporation of water from the mix occurred, making it necessary to add water during the process in order to obtain the desired consistency of the mix. It was then proposed to try to blend in the mixer FS-26 powder and powdered methyl cellulose. The water was then added in small increments and thoroughly blended in while cold water was being circulated through the mixer jacket. The process proved very successful. Later, ice water was added which greatly improved the consistency of the mix, and finally, the necessary water was added as crushed ice and was allowed to melt during blending. The standard charge for the mix consisted of 125 pounds of FS-26 powder, 2-1/2 pounds of Methocel powder, and 22-1/2 pounds of crushed ice. This mixture was blended for two hours with cold water circulating through the mixer jacket.

E. Extrusion of One-Inch Solid Rounds

Figure 10 is a photograph of the 1050-ton extrusion press with the various parts labeled on the photograph. Figure 11 is a photograph of the front end of this press, with the nozzle swung out for charging. By referring to these two labeled photographs and the following description the reader may obtain a general knowledge of the extrusion process.

In order to carry out a powder extrusion, the nozzle assembly is swung out as in Figure 11. The powder plasticizer mixture is charged into the extrusion chamber, and the nozzle assembly is closed and locked as shown in Figure 10. The pumps are started and oil is pumped into

Figure 10. Ten-hundred fifty Ton Extrusion Press in Operation

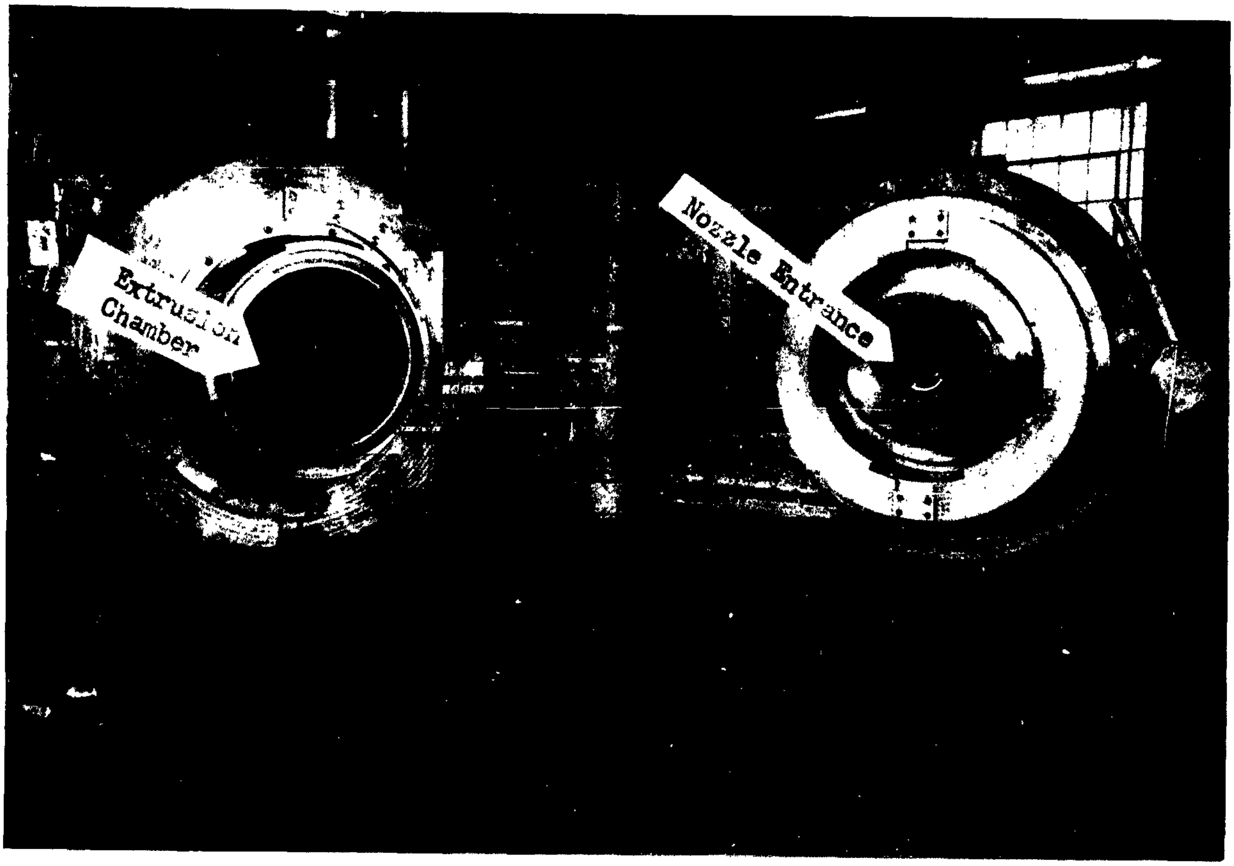


Figure 11. Ten-hundred fifty Ton Press With Nozzle Assembly Swung Out for Charging

the hydraulic pressure cylinder. This causes the extrusion pressure ram to move into the extrusion chamber. When the extrusion pressure ram closes the extrusion pressure chamber, the nozzle plug and cap, as shown in Figure 8, are inserted and tightened. This completely seals the extrusion chamber. The extrusion chamber is evacuated through a series of small holes, just behind the face of the extrusion pressure ram. This removes the air from the extrusion mix. When the pressure can be pumped down to the desired degree (100 mm. of Hg or less), the ram is again caused to advance. The material then forms into a solid slug in the chamber and the extrusion entrance nozzle. This slug is essentially void of air space. The nozzle plug and cap are removed. The ram is caused to advance at a predetermined rate. This forces material out through the nozzle. These extrusions are caught on shaped graphite boats, and cut to the desired length. Extrusion rate is controlled by presetting a bypass valve in the hydraulic lines to one of six available positions. This bypasses oil back to the reserve and causes the extrusion ram to move slower if more oil is bypassed and faster if less oil is bypassed. The rates of extrusion for a one-inch diameter cross section according to rate control valve settings are as follows:

Position No.	1	-	80	feet	per	minute
"	2	-	120	"	"	"
"	3	-	148	"	"	"
"	4	-	170	"	"	"
"	5	-	210	"	"	"
"	6	-	240	"	"	"

Table II is a summary of the results obtained in extruding one-inch diameter solid rounds under the various possible operating conditions.

Table II. Summary of Extrusion Conditions

Rate of Extrusion Feet Per Minute	Total Pressure Exerted, Tons	Maximum Uniform Length Obtainable	General Comments
170	800	4 inches	Extrusion buckled, extruded jerkily, cracked, Rate too fast
120	760	18 inches	Same as 170 foot rate. Still too fast but an improvement
80	600	40 inches	Extruded smoothly. No evidence of cracks nor buckling

The rods as extruded at the 80 feet per minute rate were chosen as standard. It was desirable to cut the rate even further, but the press would not operate at lower speeds. This material was approximately 84% of theoretical density as extruded, and contained approximately 14.5% moisture. It became rigid enough to handle with care after setting for 45 minutes at room temperature. There was no evidence of either internal or external cracks in the as-extruded material. Charge weight for the extrusion was 125 pounds and the unextrudable portion remaining in the press after operation comprised 62 pounds. This unextrudable material comprises the fill in the nozzle and nozzle entrance.

F. Drying of One-Inch Solid Rounds

A number of methods of drying the various extruded shapes were investigated. No single method proved satisfactory. A combination of methods proved to be the most advantageous procedure.

After extrusion, the rods set up in approximately 45 minutes. This was the result of the loss of a small amount of water from the material during extrusion, and subsequent further evaporation to the air shortly after extrusion. The extrusion process itself produces some heat in the powder during extrusion. The combination of heat and loss of moisture content causes an increase in the viscosity of the Methocel and the material sets up. Once this setting up occurs the material may be handled with care and the drying can proceed.

The initial extrusions were divided into 4 groups. Each group was to test a drying procedure.

Group 1 was charged into a Stokes vacuum dryer. The pressure was pumped down to 35 mm. of Hg and held for 24 hours. The temperature was then raised to 150°F at a rate of 25° per hour, and this temperature was maintained for 48 hours. Starting moisture content of the pieces was 14.5% by weight. Final moisture content was 0.35% after drying. The pieces were badly warped and severely cracked; this method of drying was unsatisfactory.

Group 2 was placed in a circulating air oven after the rods had set up. The temperature of the oven was raised to 150°F at a rate of 25°F per hour. This temperature was maintained for 72 hours. The initial moisture content of the specimens was 14.5%, final moisture content was 0.39%. The pieces did not warp but showed cracks in the bottom surfaces which rested on the drying slab. There was some evidence of internal cracking.

Group 3 was allowed to dry in air at room temperature for 196 hours. The temperature of the room was controlled at 72°F; the average relative humidity over the 196-hour period was 42%. The starting moisture content of this group was 14.5%; final moisture content was 5.8%. The moisture content was measured three times at 24-hour intervals during the last 72 hours. It was constant at 5.8%. It was decided that insufficient drying could be obtained by this method. No cracking or warping was evident in this material.

Group 4 was dried for 24 hours at room temperature (72°F) and a relative humidity of 42%. After 24 hours the moisture content had decreased from 14.5% to 6.2%. Group 4 was then dried for 72 hours at 150°F in circulating air. Final moisture content of this material was 0.301%. There was no evidence of either internal or external cracks and no evidence of warpage. The dried extrusions were extremely strong and quite capable of very rough handling. This drying procedure was considered adequate.

G. Preliminary Sintering of One-Inch Solid Rounds

Two different techniques were developed for first sintering FS-26 extrusions after drying was completed. Preliminary sintering was done in vacuum and in hydrogen. Both techniques were satisfactory and both produced satisfactory physical properties.

Pre-sintered material was produced in the hydrogen furnace by creating a uniform heat zone 24 inches long in the rear. The preliminary sintering furnace is shown in Figure 5. The last two burners were adjusted to maintain this 24-inch uniform zone at a temperature of 1600°F. The two preceding burners were adjusted to maintain temperature zones of 1200° and 600°F above each burner. Hydrogen gas was introduced at the cooling zone, or exit end, of the furnace and allowed to flow through the length of the tube before exiting. This produced a temperature in the entrance zone of approximately 230°F. When the material was introduced and mechanically stoked through these various zones, it was brought from room temperature to 1600°F in approximately two hours and maintained at 1600°F for 30 minutes. This produced a pre-sintered rod with excellent strength and capable of being shaped by either grinding or turning. Several rods were sliced down the middle and showed no evidence of internal cracks. In a few isolated cases, very shallow surface cracks developed on the bottom of some of the rods during preliminary sintering. These cracks could be removed by grinding and were of average depth of .005 inch. They developed as a result of incomplete removal of moisture from the bottom surface. During preliminary sintering in hydrogen a one-inch length of the extruded material expanded approximately .006 inch. This expansion is for a temperature of 1600°F. Temperatures below 1600°F produced weak pieces and greater expansions. Temperatures above 1600°F produced pieces which were too hard for subsequent shaping. No shrinkage was encountered during preliminary sintering until a temperature of 1900°F was reached.

Vacuum preliminary sintering was accomplished by packing pieces to be sintered in carbon (-20 + 80 mesh graphite powder) and sintering at 1800°F for 30 minutes in a vacuum which was maintained at less than 100 microns. There was neither an expansion nor contraction in this material as sintered in vacuum. Pieces sintered below 1800°F were too soft and too weak for shaping. Pieces sintered at 1900°F or above were too hard for shaping. The graphite powder was necessary in order to prevent surface oxidation in the early stages of the vacuum sintering.

H. Final-Sintering of One-Inch Solid Rounds

Final-sintering was carried out according to standard procedures developed for the final-sintering of Firth Sterling's grade FS-26. The material was placed on a "V" shaped graphite slab separated from the graphite by carburized paper and sintered at 2500°F for 30 minutes. The pressure was maintained at 100 microns. There was no evidence of internal nor external flaws after final-sintering. Warpage was not excessive. The removal of ten thousandths stock completely cleaned up a 3/4-inch rod, 12 inches long. The shrinkage during final sintering was 16%.

I. Evaluation of Physical Properties of Extruded FS-26

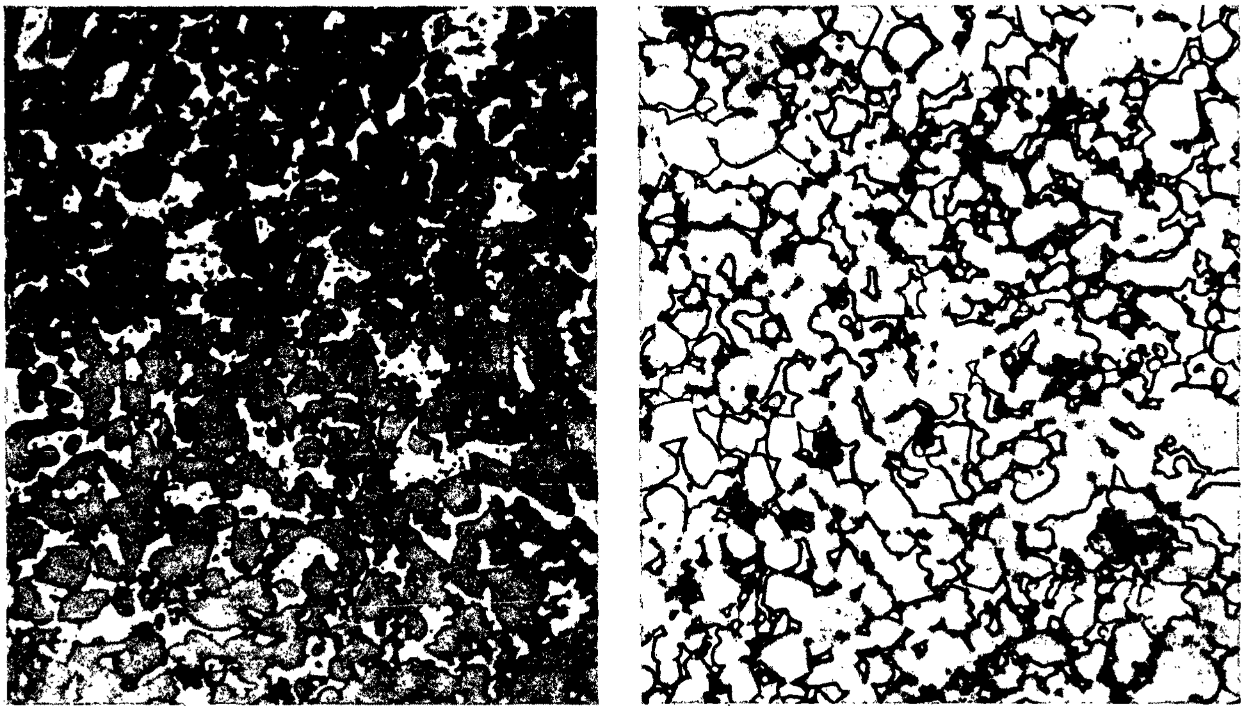
Various necessary test specimens were produced from extruded FS-26 bars by shaping the pre-sintered piece and final sintering or by grinding the required shape from the final-sintered material. Table III is a comparison of the properties of extruded FS-26 and FS-26 produced normally by cold pressing and sintering.

Table III. Comparative Properties of Cold Pressed and Sintered and Extruded FS-26

Property	Cold Pressed FS-26	Extruded FS-26
Density, grams/cc	6.24	6.24
Hardness, Rockwell "A"	85	84.5
Transverse Rupture Strength, psi	165,000	145,000
Oxidation Resistance		
Weight gain mg/sq cm		
after 100 hours	31.5	35.3
Impact Resistance Ft-lbs		
on Standard Unnotched Charpy Bars		
at 75°F	4.5	2.9
at 1800°F	4.4	5.4
Tensile Strength, psi		
and % Elongation		
at 75°F	32,900 - Nil	29,000 - Nil
at 1800°F	41,000 - 0.8	40,000 - 1.0
Stress, psi and Elongation %		
for 100-hour rupture at 1800°F	11,100 - 5.6	10,300 - 6.0

It is significant to note that one of the conclusions that might be drawn from a study of this comparison is the lack of room temperature impact strength as shown by the extruded FS-26 while the impact strength at 1800°F showed a considerable improvement. There is also a lack of room temperature strength indicated by both the tensile strength and transverse-rupture strength. In the case of stress-rupture there seems to have been no improvement in strength at either low or high temperatures, although elongation may have increased slightly. High temperature tensile strengths showed no significant improvement either. The increase in impact strength at elevated temperatures and elongation at elevated temperatures may be attributed to possible orientation, during sintering, of the TiC grains. The microstructure of the extruded material is shown in Figure 12. As compared with the microstructure of normally prepared FS-26, there appears to be a more regular distribution of the TiC ordinarily equiaxed parts. There was no definite evidence of elongation in the direction of extrusion attained, however. It is not felt that any of the properties were significantly improved by extrusion.

Figure 13 is an X-ray print of stress-rupture bars of extruded FS-26. X-ray scattering has obliterated the reduced section but careful observation of the major diameter sections shows many porous areas and internal flaws. This appears to have been a more or less typical condition of extruded material. No way has ever been found to eliminate random voids of up to .016 inch in diameter, generally near the center of the material. These voids and porous areas tend to make the extruded material somewhat unreliable in long lengths.



Extruded FS-26, 1500X

Cold Pressed & Sintered FS-26, 1500X

Figure 12. Extruded and Cold Pressed and Sintered FS-26

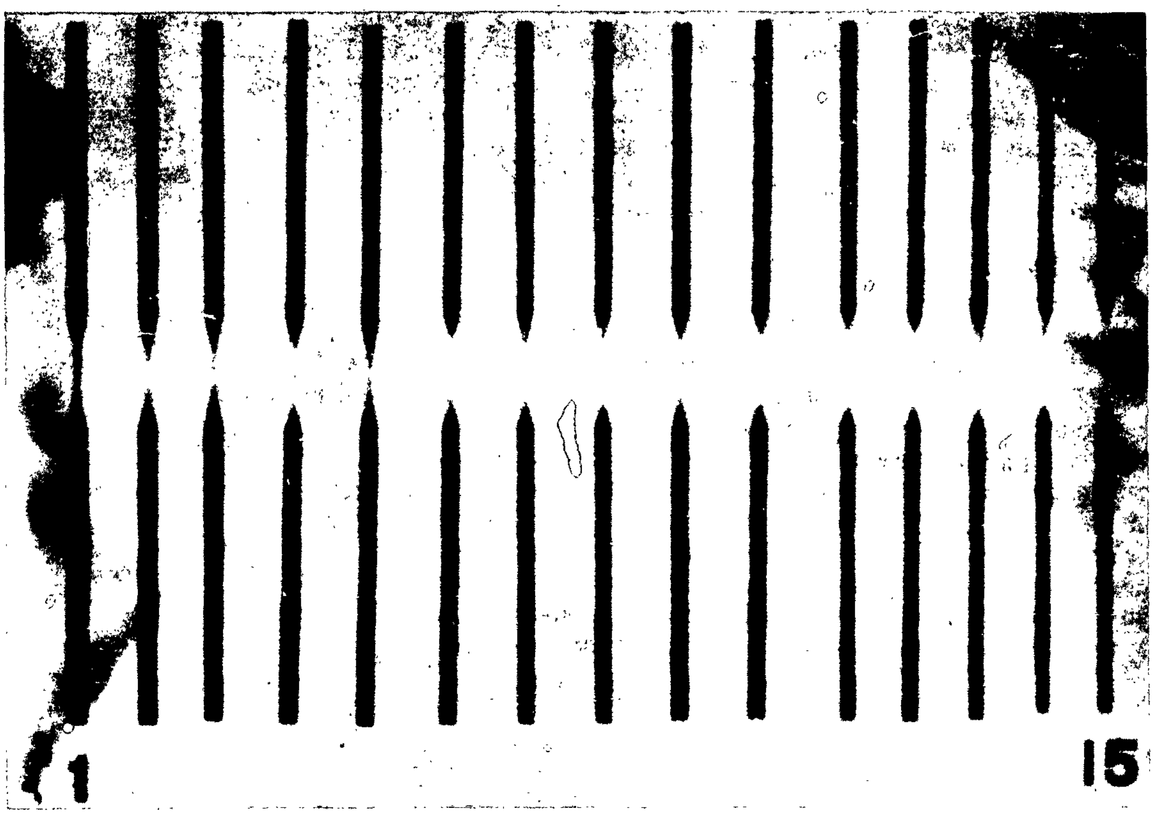


Figure 13. Radiograph of Extruded FS-26 Bars

J. Extrusion of Airfoil Sections

Design of Airfoil Section Die Nozzles

All information essential for the design of the airfoil section nozzle had been determined by measurement of the experimental extrusions performed on the one-inch round nozzle. The essential points were the correct nozzle entrance angle, the amount of shrinkage obtained when the extrusions were sintered, which would indicate the amount of oversize necessary to make the nozzle, and the amount of relief which could be put into the exit end of the nozzle without causing undue expansion. The nozzle entrance angle was definitely established as being an optimum 45°; the shrinkage of the extrusions was established to be 16%; and it was established that expansion of the extrusion occurred even with a 1/8 x 45° relief on the round nozzle. It was decided accordingly that relief at the exit end of the die would be held to a minimum. The distance through the die block press established the length of the die bearing to be nearly 10 inches. From the beginning this was considered extremely detrimental to the progress of the experimental extrusions, but there was no way of maintaining a 45° entrance angle from the bearing to the 15-inch diameter of the extrusion barrel without a bearing of this length. An extrusion press which was specifically designed for this purpose would have circumvented this difficulty. In addition, since it was impossible to relieve at the exit end of the nozzle, the other method of shortening this bearing was made impractical. Accordingly, the nozzle die incorporating all of the foregoing principles was designed and constructed according to the drawing in Figure 14. The die was designed to produce a section which, after sintering, would be an airfoil section the same dimension as that required in WADC Drawing No. S52D9145. In designing the die, a constant 45° entrance angle into the airfoil section was maintained. This meant that material entered the constant dimension airfoil bearing at different points along its length, as shown in Figure 15. The entrance end is at the left and an end view of this entrance is shown in Figure 16. A circular cross section to airfoil cross section nozzle entrance is inserted as a separate part as illustrated in Figure 17.

Construction of this die proved extremely difficult. The irregular curves proved nearly impossible to produce accurately by any known extrusion method. Out of 8 die making concerns contacted only one would attempt construction of the die. This company delivered the die nearly 11 months beyond the predicted delivery date. In general, the die was produced by casting the large nozzle entrance angle of meehanite and making up special laps for finishing to the desired finish. The long bearing proved nearly impossible to produce with accuracy of better than plus or minus .005 inch. Construction of such dies to produce shapes in closely controlled and specified dimensions proved to be virtually impossible. The desired degree of dimensional accuracy of the part is beyond the accuracy obtainable in the construction of the nozzle itself. The full die assembly after construction is shown in Figure 18.

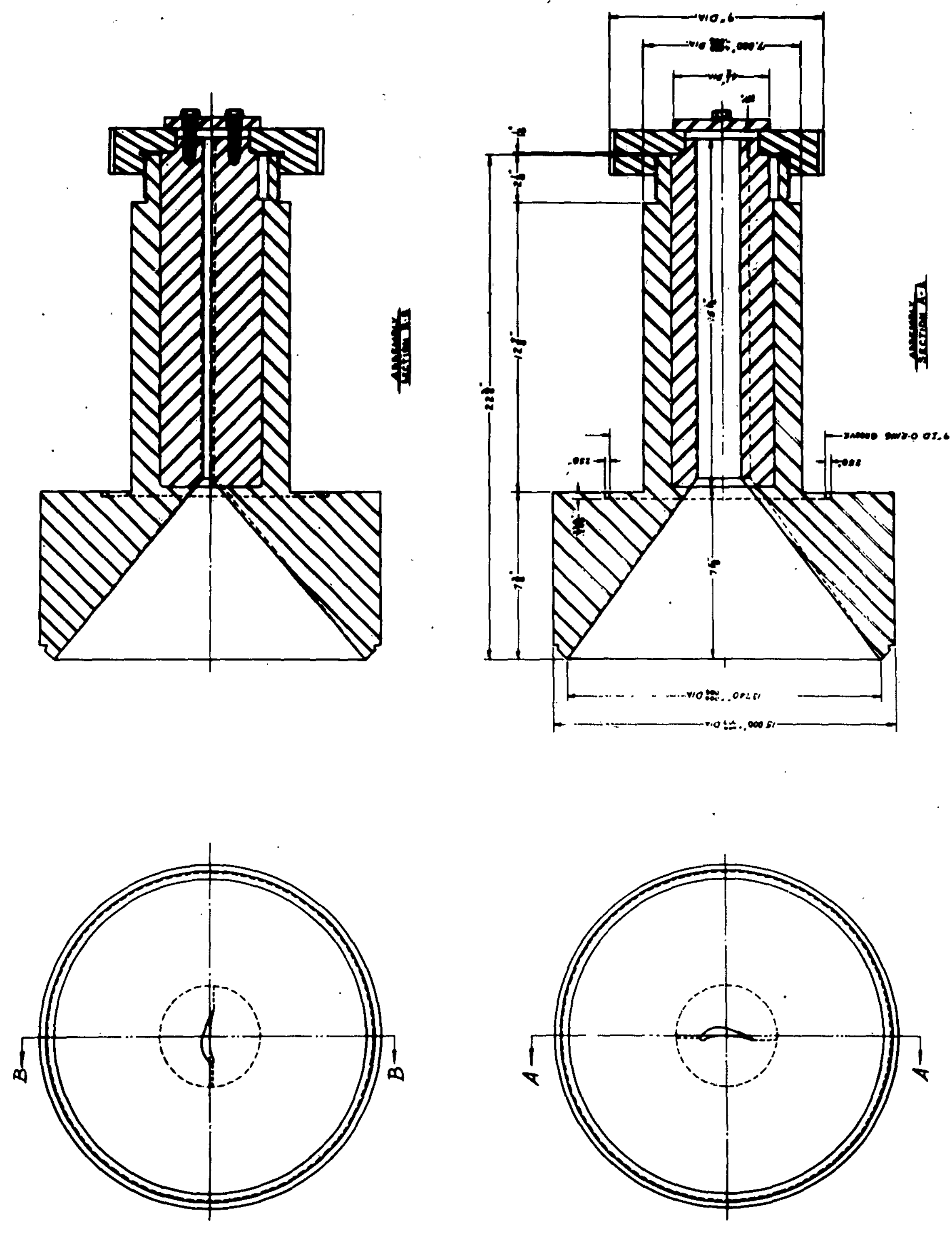


Figure 14. Drawing of Airfoil Section Die

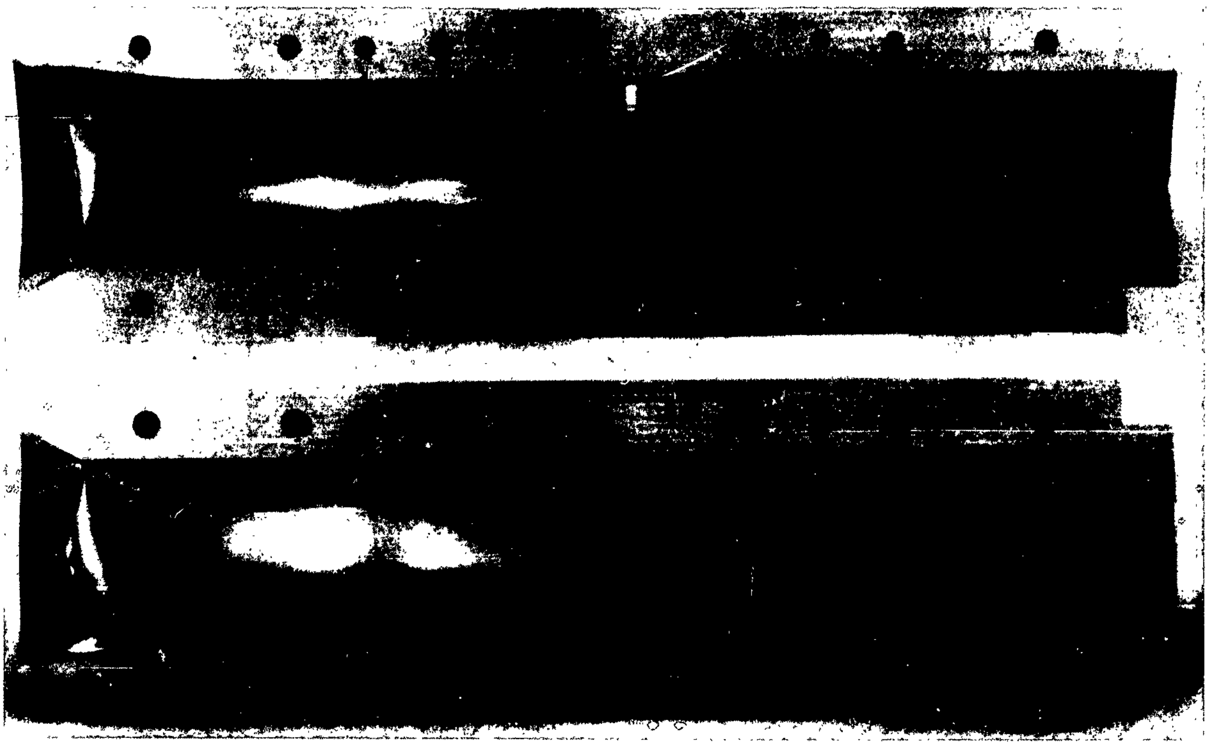


Figure 15. Nozzle Bearing and Entrance Angle

Figure 16. End View Nozzle Entrance

Figure 17. Separate Nozzle Entrance

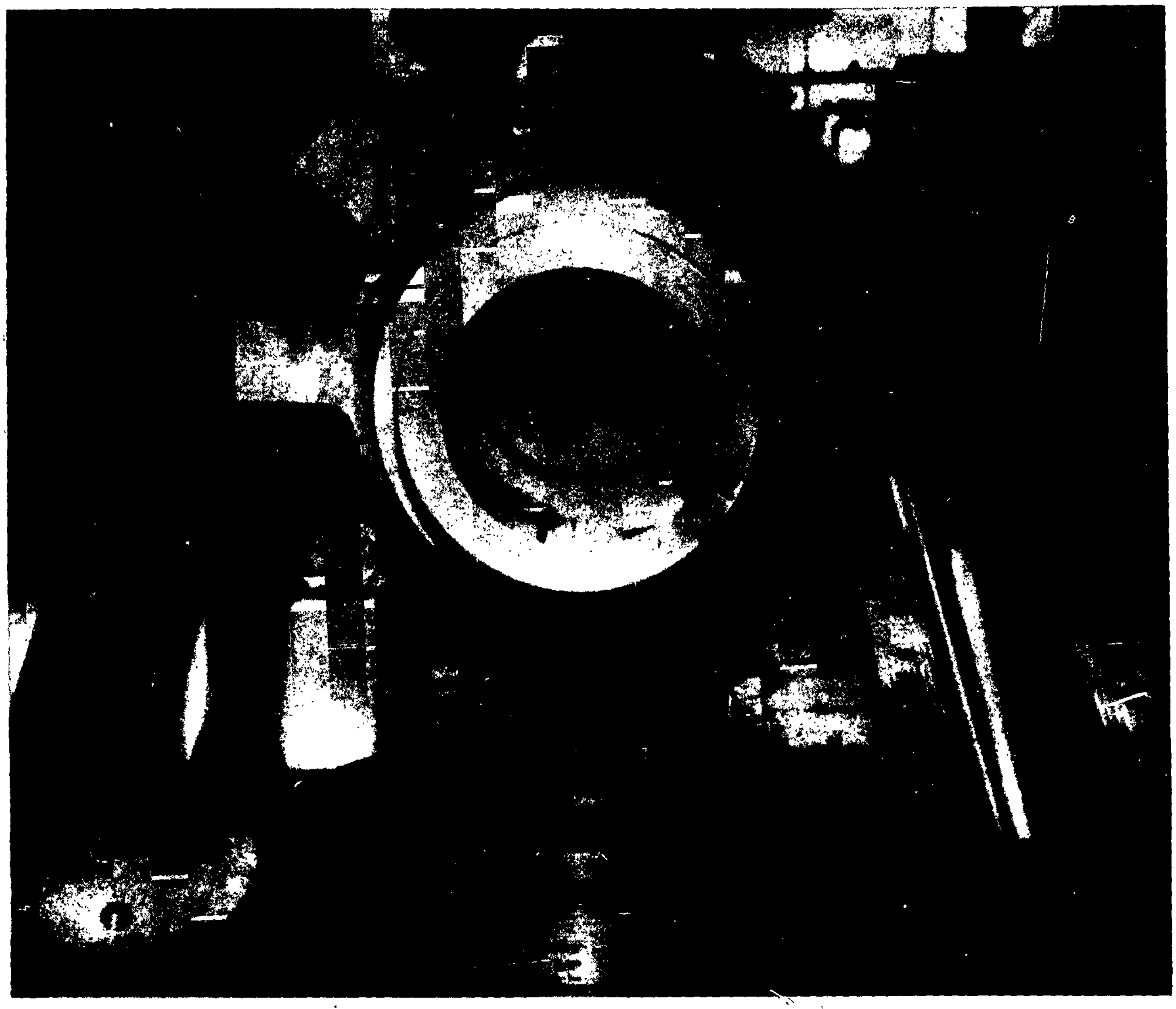


Figure 18. Die Assembly

Extrusion of Airfoil Cross Sections

The first attempt at extrusion of an airfoil cross section was made according to the conditions determined during the extrusion of one-inch solid rounds. A comparison of the cross sectional area of the airfoil section to the cross section of the one-inch solid round indicated that the setting for the pumping rate should be the No. 3 position in order to achieve the 80 feet per minute extrusion rate found best for the one-inch solid nozzles. This was due to a somewhat greater cross sectional area in the airfoil section. At this setting a required pressure to produce an extrusion proved to be 800 tons. The material extruded from the nozzle is shown in Figure 19.

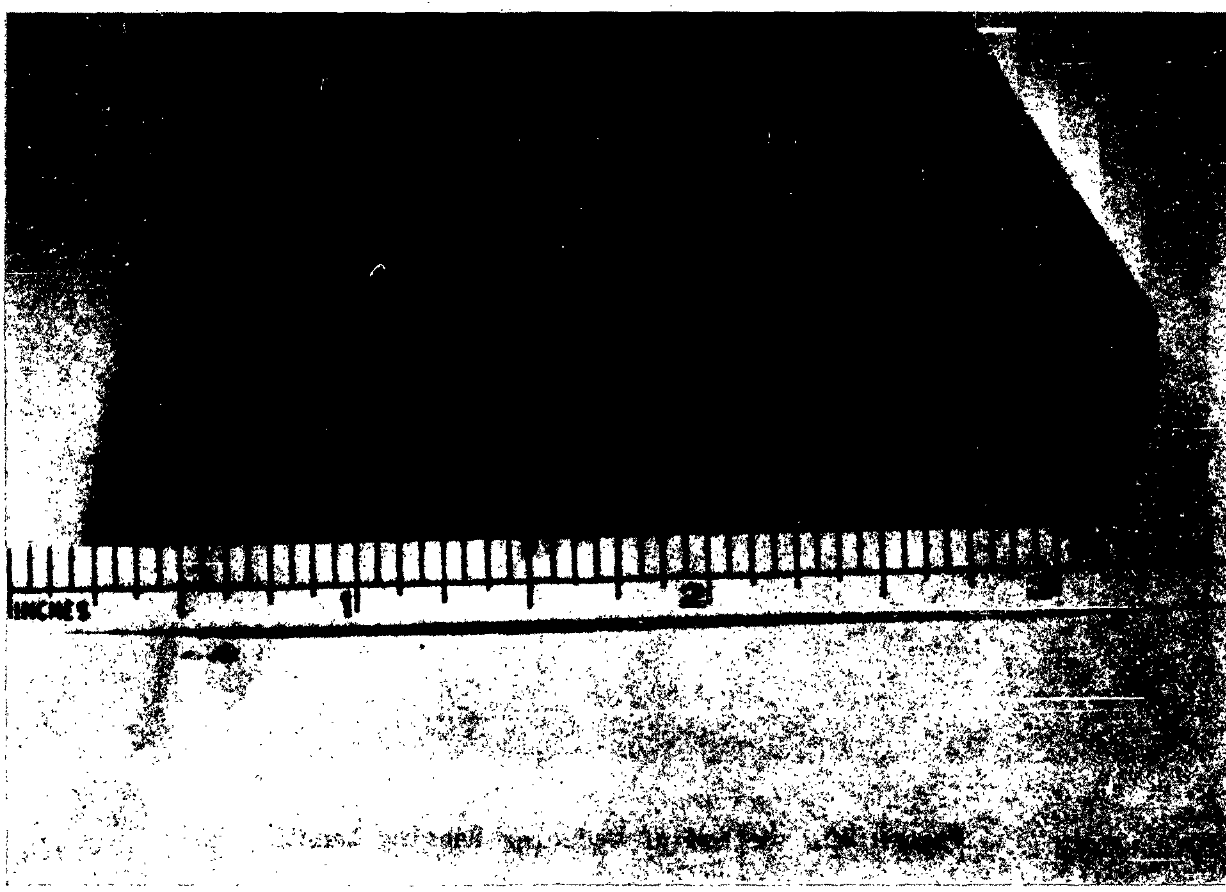


Figure 19. Material Extruded from Nozzle

It held form well but the leading and trailing edges did not extrude completely. Approximately 1/8 inch of the leading edge did not extrude and 3/4 inch of the trailing edge failed to extrude with the original bearing die design. No amount of varying extrusion rates or the viscosity of the mix would produce a satisfactory extrusion. Addition of more plasticizer to reduce the viscosity produced pieces which would not hold shape. Reduction of the amount of plasticizer increased the amount of cross section of the airfoil section which did not extrude. Increase and decrease in extrusion rate served only to increase and decrease extrusion pressure. It was concluded that a reduction in the amount of die friction would produce a better extrusion. Accordingly it was decided to materially reduce the bearing length by relieving the exit end by a very sharp (90°) angle. Accordingly, the average bearing length was reduced to approximately 3-1/2 inches as shown in Figure 20.

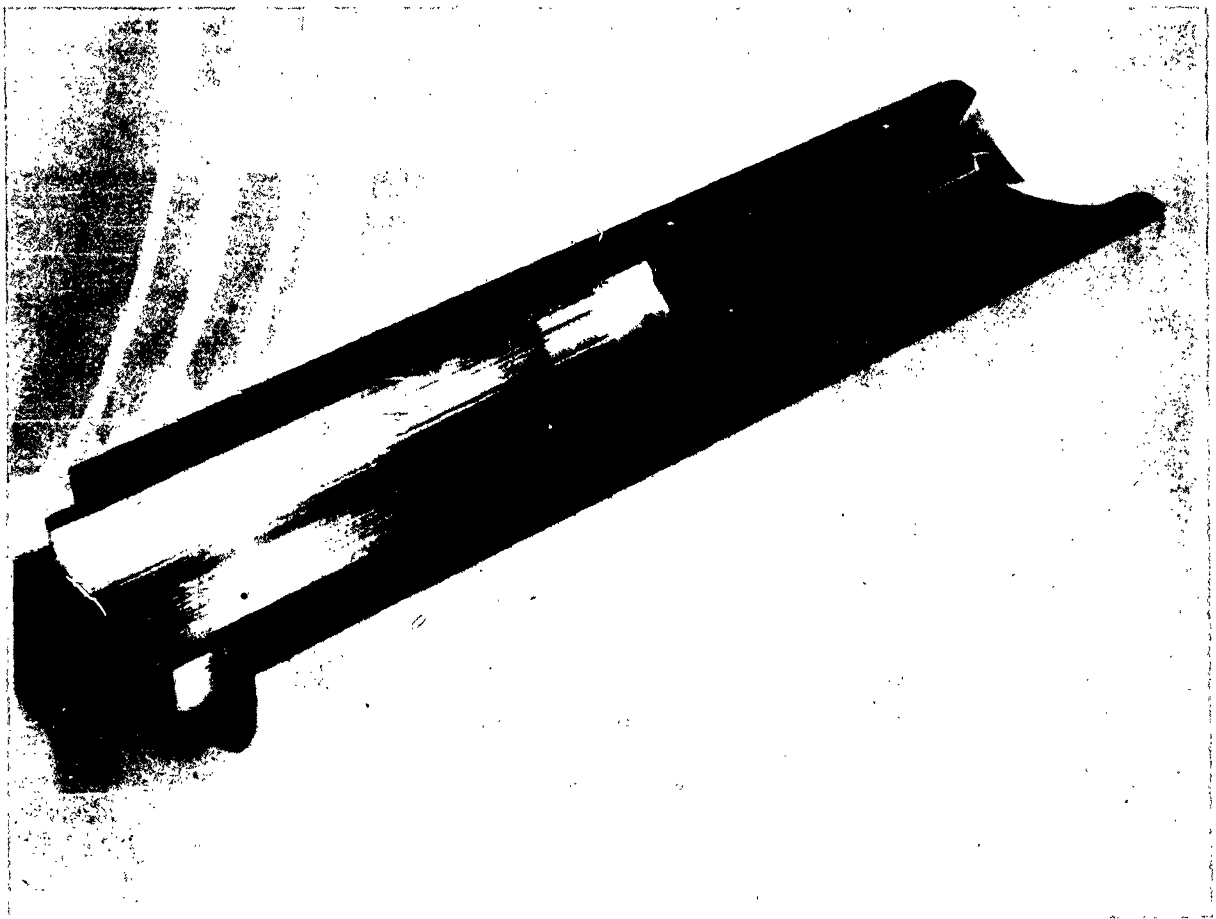


Figure 20. Method of Reducing Bearing Length

This was a definite improvement since the leading edge remained essentially intact and only 1/2 inch of the trailing failed to extrude. In exiting into the relief, however, the extrusion apparently dragged against the trailing edge and expanded into the relief section. This produced a perfectly flat underside of the airfoil section as illustrated in Figure 21. It was then decided to further reduce the bearing length on the side of the trailing edge and to taper the relieved section so as to prevent the flattening of the underside of the extrusion. Accordingly, the die was tapered as shown in Figure 22. The taper on the side of the trailing

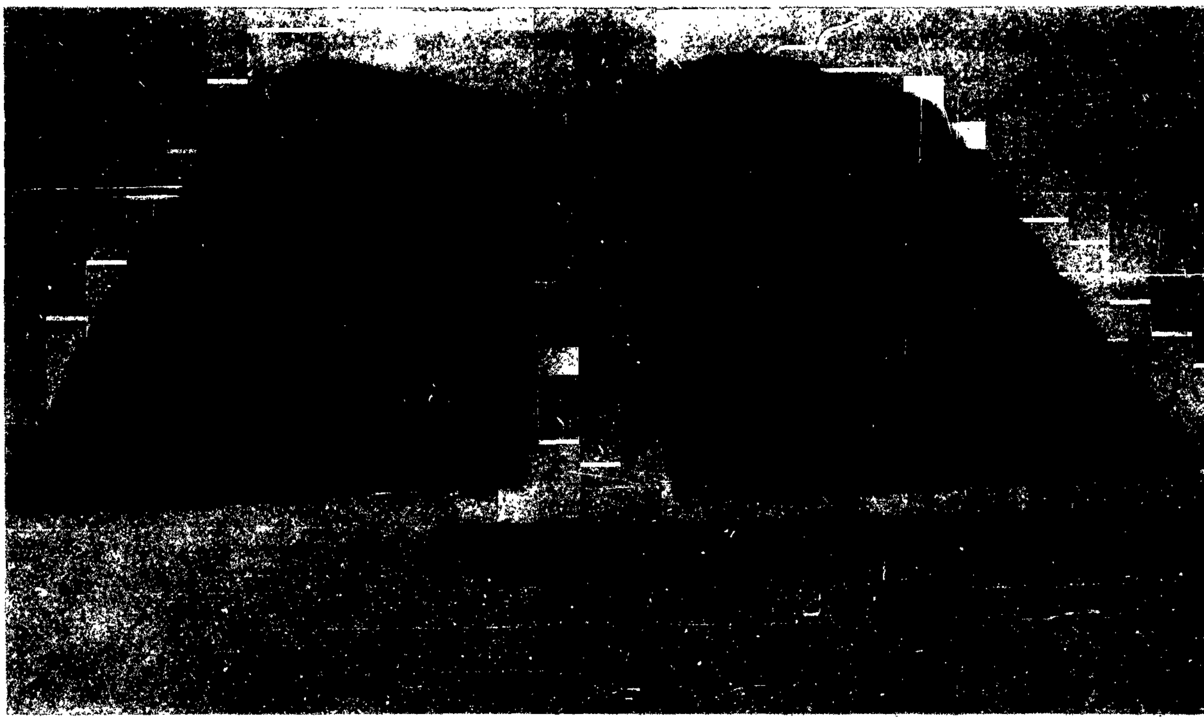


Figure 21. Extrusion Produced by Relieving Bearing Section

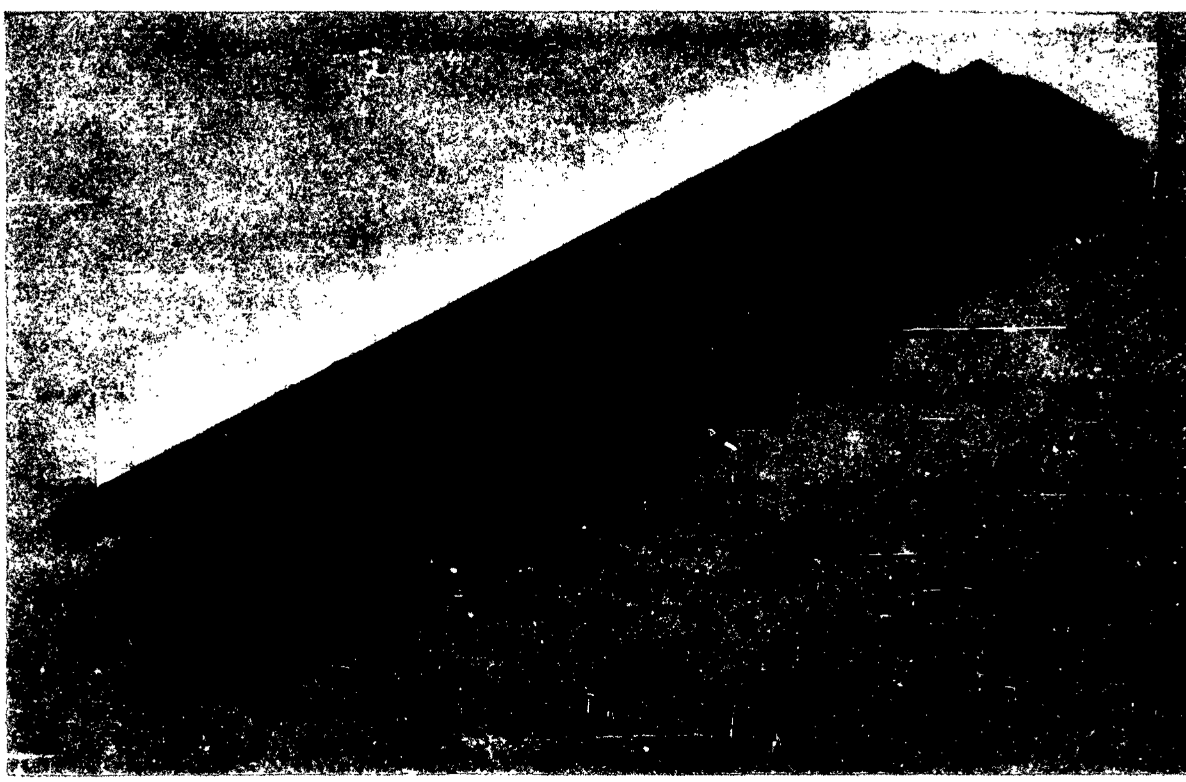


Figure 22. Reduced Bearing Section With Tapered Relief

edge was put in from the entrance side in order to assure a greater flow through that side. The 90° relief was polished out. This resulted in the production of the extrusion shown in Figure 23 in which the cross section is virtually perfect, although some minor abrasion of the leading and trailing edges was evident.

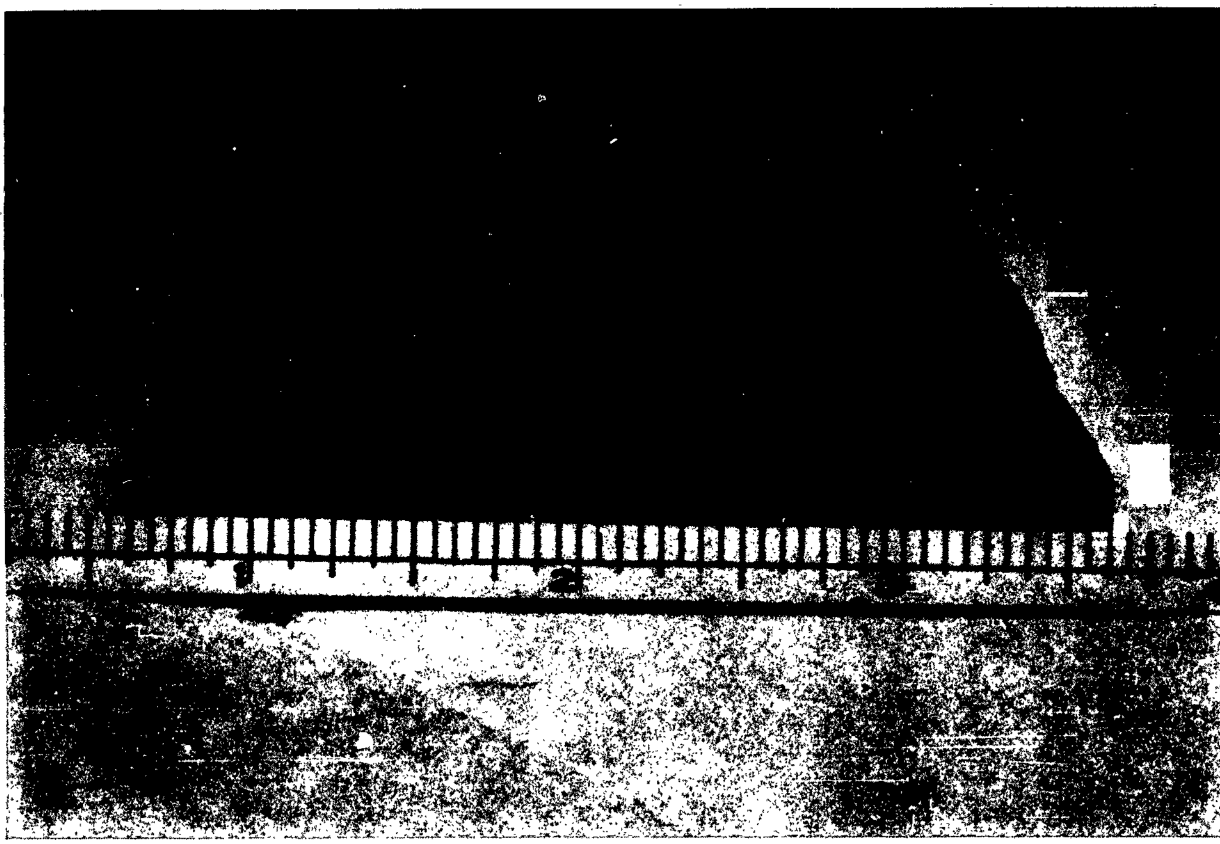


Figure 23. Cross Section of Extrusion Produced by Bearing Section of Figure 22

The pieces would not hold form laterally, however, as illustrated in Figure 24. Drag in the relief caused the pieces to curve with the result that cracks appeared in both the leading and trailing edges. No amount of variance in conditions would relieve this situation.

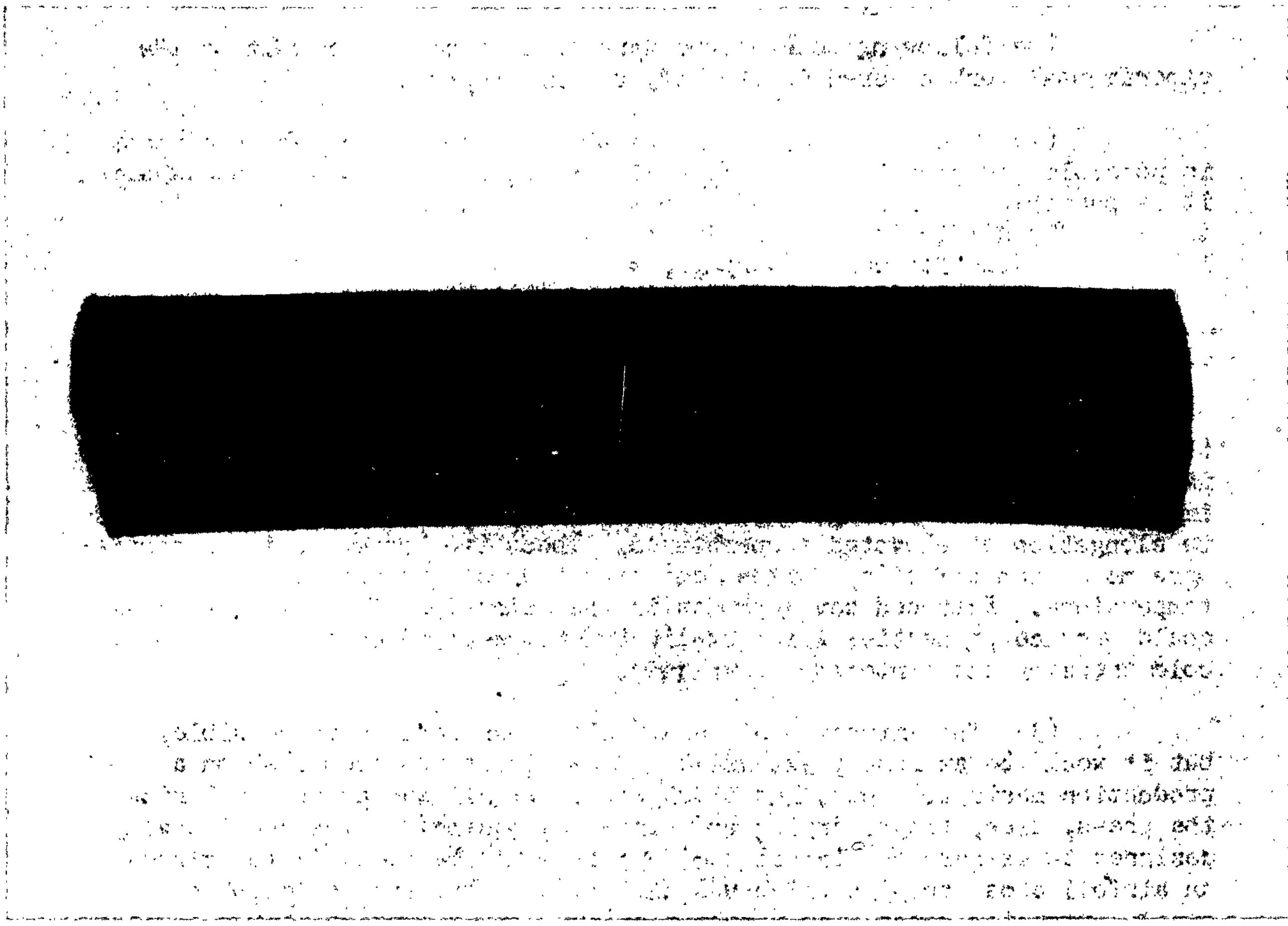


Figure 24. Lateral View of Extrusion Showing Curving and Cracking

III. CONCLUSIONS

The following conclusions were arrived at as a result of the experimental work covered in the body of this report.

(1) The extrusion of useful objects from carbide-base cermets is possible and practical to only a limited extent. By way of explanation it is possible to extrude a definite shape from these materials, but it is extremely difficult to maintain accuracy better than plus or minus 1/16 inch. Such factors as warpage, surface reaction during sintering and porosity pose additional difficulties. Slight variations in the starting raw materials produce correspondingly larger variations in the dimensions of the final sintered extrusion.

(2) The physical properties of cermets are not enhanced by the extrusion process. During this project, only two instances of improvement in physical properties were encountered. There was a slight improvement on the average impact strength at 1800°F and a slight increase in elongation at elevated temperatures. These two increases in properties were more than offset by decided deterioration of properties at room temperature. Extended developments in the extrusion of a specific shape could produce properties which duplicated those produced by conventional cold pressing and sintering techniques.

(3) The extrusion of an airfoil cross section is possible, but it would be extremely expensive and difficult to accomplish on a production basis to controlled dimensions. If all equipment, including the press, dies, trays, drying and sintering equipment, was specifically designed to extrude an airfoil section; it would be possible to extrude an airfoil cross section which was uniform over the entire length of a single extrusion batch. As a result of this investigation it would appear at the present time, that control of the various stages in the extrusion process is insufficiently accurate to allow the extrusion and sintering of airfoil cross sections to final dimensions which would meet any reasonable dimensional tolerance. Variations in chemical compositions of cermets, mixing the cermet-plus-plasticizer and extrusion rate would each cause inaccuracy in final size. The expense of constructing the die to extrude an airfoil cross section is extreme in the case of a single lot of powder. The prospect of having to construct a different size nozzle bearing insert for each variation in powder characteristics is prohibitive.

(4) The following suggestions are offered for future investigations:

a. The use of the extrusion process might be coupled with a continuous mechanical shaping device for producing airfoil cross sections. The production of rectangular or round billets by extrusion is very practical. If a mechanical device such as a grinder or milling cutter should be incorporated at some point in the extrusion process, then allowances could be made for size variation by adjustments in the mechanical device.

b. An injection molding process using cermet powders and the extrusion plasticizers developed in this program appears to be very promising. If plasticizers were used to make a powder mixture which could be caused to flow into a graphite or cast ceramic mold under extremely high pressures, then the expense of making metal molds could be eliminated and size control would be less a problem. Cermet powders plus methyl cellulose plasticizers appear to have the necessary flow characteristics to accomplish this end.

c. Changes in design of the sections to be extruded would accomplish a great improvement in the process. If a uniform symetrical shape could be devised to do the required job, it would undoubtedly be simpler to extrude and to control.

d. The extrusion of more ductile powders would be simpler to accomplish.