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PREPARATION and EVALUATION of FIBER METAL NICKEL BATTERY PLAQUES

FINAL REPORT

August 1, 1964 to July 31, 1965

by

J. L. Bidler and J. I. Fisher

prepared for

NATIONAL AERONAUTICS and SPACE ADMINISTRATION

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Space Power Systems Division
William A. Robertson MS 500-201

HUYCK METALS COMPANY
of HUYCK CORPORATION
P. O. Box 30
Milford, Connecticut

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ABSTRACT

Flexible fiber metal plaques can be produced with internal surface areas greater than 500 cm^2/gm, tensile strengths greater than 300 psi, porosities of 90% or greater and electrical resistivities of 400 microhm-cm or less. Although not all of the values indicated above can be achieved in the same plaque, sufficient data are presented to permit an evaluation of the affect of processing parameters upon the plaque characteristics and to indicate which fiber characteristics should be varied to optimize specific plaque properties. A new fiber type has been developed to maximize the internal surface area and porosity. It is anticipated that this material can be processed into plaques with greater than 90% porosity and with an internal surface area in excess of 800 cm^2/gm.

Author

TABLE OF CONTENTS

TABLE OF CONTENTS
Continued

-o0o-

LIST OF FIGURES

LIST OF FIGURES
(Continued)

LIST OF FIGURES

LIST OF FIGURES
(Continued)

LIST OF TABLES

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(Continued)

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1.0 SUMMARY

This report concludes an investigation of the use of three grades of nickel fiber metal for the manufacture of battery plaques. In general, the objective of the program was to produce battery plaques with maximum internal surface area, porosity, and tensile strength and minimum electrical resistivity. This report includes a description of the fibers used, the results of a sintering investigation and the characteristics of sintered nickel fiber metal plaques.

The data presented show that flexible fiber metal plaques can be produced with internal surface areas greater than 500 cm^2/gm, tensile strengths greater than 300 psi, porosities of 90% or greater and electrical resistivities of 400 microhm-cm or less. Although not all of the values indicated above can be achieved in the same plaque, sufficient data are presented to permit an evaluation of the affect of processing parameters upon the plaque characteristics and to indicate which fiber characteristics should be varied to optimize specific plaque properties. Toward this end, a new fiber type has been developed, a description of which is included, to maximize the internal surface area and porosity. It is anticipated that this material can be processed into plaques with greater than 90% porosity and with an internal surface area in excess of 800 cm^2/gm.

In general, this investigation has shown that fiber metal battery plaques have properties equivalent to or better than battery plaques manufactured from currently available materials. In particular, the high porosities attainable, the flexibility, and the wide range of pore sizes available are properties in which fiber metal plaques excel.

2.0 INTRODUCTION

This program, for the preparation and evaluation of fiber metal nickel battery plaques, is intended to characterize the raw materials, define the sintering parameters, and measure the sintered plaque properties of battery plaques made from three grades of nickel fiber. In general, the objective of the program is the development of nickel battery plaques having minimum apparent density and electrical resistivity and maximum tensile strength, internal surface area, and flexibility.

The program is based on the technology called Fiber Metallurgy. The primary purpose of the manufacture of fiber metal is to produce sintered metals having controlled porosity, pore size, surface area, and permeability. The advantages of using metal fibers over other forms of raw material are:

Porosity Range

A wider porosity range can be achieved using fibers than with any other particle form.

Control of Pore Size

Fiber diameter and porosity interact to define pore size. Using the fibers selected for this program, pore sizes ranging from 10 to 80 microns can be obtained at high porosity levels.

Control of Pore Size Distribution

The normal procedure for manufacturing fiber metal is to felt and sinter to a high porosity, then compact to the desired porosity. This affords precise control of the pore size range, which decreases as the porosity decreases.

Maximum Interconnected Porosity

Fiber metal structures with porosities as low as 50% have more than 95% interconnected pores.

Large Surface Area

Fiber size and shape interact to define surface area. The fibers employed in this study have specific surface areas in excess of 450 cm^2/gm.

Strength

Fiber metal bodies have the highest strength of any material at high porosities.

Formability

The compressibility of fiber metal materials permits considerable latitude in forming operations.

The work of the program consisted of three phases. The first involved the classification of the raw materials as to size, size tolerance, and shape. The second phase was concerned with determining the sintering parameters that would produce the optimum combination of density, surface area, tensile strength, pore size distribution and electrical resistivity. The third phase consisted of classifying plaques of each material, sintered under the conditions dictated in the second phase, as to the parameters noted above.

Initially two grades of nickel fiber were selected for evaluation. These grades were designated as AX1 and AX2. As the program progressed, it became apparent that it would be benefical to separate the smaller diameter fibers from the AX1 material and to include this material, designated AX1 Modified, in the program. In addition, a small quantity of a new fiber type, designated DX, became available late in the program. Because DX fiber was believed to be ideally suited to the application, the raw material classification portion of the program was completed for this fiber type.

As indicated above the program outline defined major tasks, A-D, which are summarized below:

2.1 Task A - Raw Material Classification

Each raw material used in the program was characterized as to particle shape, particle size and particle size distribution. Microscopic measurements of fiber lengths and apparent diameter were supplemented by photomicrographs of as-sintered surfaces,

cross sectional areas, and shadographs of typical fibers to present both a statistical and visual description.

2.2 Task B - Sintering Study

It was desired to establish the highest sintering temperature that would produce an acceptable amount of shrinkage when the sintering time is held constant at 20 minutes. The sintered plaques resulting from this portion of the program were evaluated to determine the median pore size, pore size distribution, density, tensile strength, internal surface area, and electrical resistivity. These data were used to define the sintering temperature required to produce plaques with the desired characteristics.

2.3 Task C - Plaque Classification

Plaques processed under the conditions defined in Task B were produced and electrical resistivity, internal surface area, density, median pore size, pore size distribution, tensile strength, and flexibility determined.

2.4 Task D - Plaque Samples

A sample of each plaque upon which the classification tests were performed was provided to the NASA Technical Director.

3.0 EXPERIMENTAL METHODS, APPARATUS AND PROCEDURES

3.1 Task A - Raw Material Classification

3.1.1 Microscopic Determination of Fiber Length and Apparent Diameter

3.1.1.1 Apparatus

Leitz Metallux microscope with micrometer stage and graduated eyepiece.

3.1.1.2 Procedure

Microscopic slides of AX1, AX2, AX1 Modified, and DX nickel fibers were prepared by suspending a typical sample of fibers in a 1% solution of Carbopol in water. A sample of this solution, containing hundreds of fibers, was placed between two glass slides. The slide was mounted on the micrometer stage, indexed, and viewed through the graduated eyepiece. A magnification of 100X was used for length measurements and 200X for apparent diameter measurements.

To facilitate measurements, length class intervals were defined as 25 microns and diameter class intervals as 2.5 microns. The length and apparent diameter of each fiber measured was estimated to the nearest class interval. Duplicate samples were prepared and measured until the reproducibility of the technique was established. Frequency tabulations were prepared for each sample measured.

3.1.2 Photomicrographs

3.1.2.1 Apparatus

Leitz Metallux microscope with Polaroid camera attachments.

3.1.2.2 Procedure

Low magnification (15X) photographs were taken of as-sintered surfaces of AX1, AX2, AX1 Modified and DX nickel fibers sintered at 1900°F.

Metallographic mounts were made of samples sintered at various temperatures by vacuum impregnating them with a catalyzed epoxy resin. Photomicrographs were taken parallel to the felting plane at a magnification of 210X.

Shadographs of AX1, AX2, AX1 Modified and DX nickel fibers were obtained at a magnification of 57X to permit a visual comparison of the primary raw materials.

3.2 Task B - Sintering Study

3.2.1 Sintering

3.2.1.1 Apparatus

Lindberg retort furnace with a 12 x 26 x 8 inch hot zone capable of 2150°F with a dry hydrogen atmosphere.

Burrell laboratory furnace with a 2 inch diameter by 15 inch long hot zone capable of 2150°F with a dry hydrogen atmosphere.

3.2.1.2 Procedure

All sintering was done by bringing the furnace to the appropriate temperature, loading the samples into the retort and charging the retort into the furnace. When the work reached the proper temperature the 20 minute time cycle was begun. At the end of the cycle, the retort was pulled and air cooled to 250°F maximum.

3.2.2 Porosimetry Measurements

3.2.2.1 Apparatus

Aminco Winslow mercury intrusion porosimeter with a 0.2 cm^3 penetrometer.

3.2.2.2 Procedure

Appropriate size samples were obtained for each material tested. The volume of mercury intruded was measured at a variety of absolute pressures and the data were plotted to show pore size versus volume intruded.

3.2.3 Internal Surface Area Measurements

3.2.3.1 Apparatus

An air permeability apparatus is employed for surface area determinations. This equipment is designed to measure accurately the pressure drop across a permeable sample when the sample is exposed to a calibrated flow of air.

3.2.3.2 Procedure

The method used to determine the internal surface area of nickel fiber metal plaques is that described by Orr and Dallavalle[1] wherein the pressure drop of a calibrated flow of air through a bed of fine, fibrous, packed material can be related to the specific surface area of the material by means of the Kozeny-Carman equation:

$$S_v^2 = \frac{g_c}{K\mu V} \quad \frac{(\Delta P)}{L} \quad \frac{\epsilon^3}{(1-\epsilon)^2}$$

where S_v = Specific surface area of solids, surface area/unit volume of solids present

g_c = gravitational constant

μ = viscosity of flowing fluid

V = velocity of flowing fluid

ΔP = pressure drop through packed bed

L = length of packed bed

ϵ = porosity; void volume/total packed bed volume

K = Kozeny constant = 4.5 for spheres, 3.0 for cylinders arranged parallel to flow, 6.0 for cylinders arranged perpendicular to flow.

[1] Superscripts refer to similarly numbered entries in the bibliography.

This method is reliable if the permeability data are taken in the streamline region where the flow rate varies linearly with the pressure drop.

Using air under the conditions of streamline flow and samples of constant porosity the Kozeny-Carman equation can be reduced to

$$S_v^2 = K_2 \frac{\Delta P}{VL}$$

The constant K_2 can be evaluated by obtaining permeability coefficients $(\frac{\Delta P}{VL})$ for samples of known surface area.

For this investigation, an average value of K_2 was calculated by obtaining permeability coefficients for sample plaques made from wire of 0.003, 0.004 and 0.006 inch diameter.

The samples of AX1, AX2 and AX1 Modified nickel fiber plaques sintered at various temperatures were tested at densities of 20% and 30% of theoretical density. At higher porosities the flow rate required to produce accurately measurable pressure drops across thin samples is in excess of 3000 SCFH/ft^2, (standard cubic feet per hour per ft^2), which has been shown to be the upper limit for streamline flow. Since the method used to increase the density is a simple mechanical compaction, the specific surface area is not significantly changed.

Samples of AX1, AX2 and AX1 Modified were placed in a sample holder designed to eliminated edge effects. A stream of filtered air from a controlled supply pressure of 30 psi was passed through each sample at a rate that would produce a pressure drop of 0.1 or 1.0 inches of H_2O. The flow rate was controlled by one of six calibrated orifices, depending upon the flow rate required, and measured by means of a mercury manometer, which determines the pressure drop across the selected orifice.

The pressure drop across the sample was read from an inclined water gage manometer, calibrated in hundredths of inches.

Permeability coefficients were obtained at 20% and 30% of theoretical density for AX1, AX2 and AX1 Modified nickel fiber metal plaques sintered at 1600°F, 1800°F, 2000°F and 2150°F for 20 minutes.

3.2.4 Tensile Strength Measurements

3.2.4.1 Apparatus

Hounsfield Tensometer with 62.5 pound beam. TensilkuT milling machine with ASTM standard E8-54T tensile specimen fixture

3.2.4.2 Procedure

Procedure is in accordance with ASTM standard E8-54T.

Plaques of AX1, AX2 and AX1 Modified nickel fiber sintered at 1600°F, 1800°F, 2000°F and 2150°F were impregnated with a low melting point (275°F) salt to prevent damage to the macrostructure during machining. The salt impregnated plaques were sawed into 2 inch by 6-1/2 inch strips and tensile specimens were milled using a TensilkuT mill and fixture. The tensile specimens had a 2 inch gauge length, were 1/2 inch wide and approximately 0.060 inches thick.

The salt was leached from the specimens using warm water; the specimens were dried and pulled using a Hounsfield Tensometer. The load required to break each specimen was recorded and the original cross sectional area was used in calculating the tensile strength. Duplicate specimens were tested for each material at each sintering temperature.

3.2.5 Electrical Resistivity Measurements

3.2.5.1 Apparatus

Leeds and Northrup Kelvin Bridge Model number 4288.

3.2.5.2 Procedure

Procedure and data reporting are in accordance with ASTM standard A344-64.

Samples of AX1, AX2 and AX1 Modified nickel fiber plaques, 1/2 inch wide by 12 inches long, were prepared as described in 3.2.4.2 for the preparation of tensile specimens. The cut surfaces were surface ground to eliminate spurious edge effects due to compaction or smearing, and to obtain precise width.

The strips were clamped securely in a sample holder designed to compact the fiber metal between two 1/2" radii at each end of the strip. The compacting assured contact and constant contact resistance. The length - which was measured between the line contacts of the radii - resistance, and cross sectional area of each strip was measured and recorded. Duplicate samples of each material at each sintering temperature were tested.

3.2.6 Density Measurements

3.2.6.1 Apparatus

Micrometer, Trip balance, Scale.

3.2.6.2 Procedure

The apparent density of fiber metal samples is calculated by measuring the linear dimensions, calculating the volume and dividing the volume into the weight of the sample; this is then divided by the true density of the metal. Density determinations were made for each sample as sintered and for each specimen prepared for tensile strength, electrical resistivity, porosimetry, and internal surface area testing.

3.3 Task C - Plaque Classification

3.3.1 Tensile Strength, Internal Surface Area, Electrical Resistivity, Porosimetry and Density Measurements

3.3.1.1 Apparatus and Procedure

The apparatus and procedure used to determine these parameters were the same as those used in Task B for the same measurements.

3.3.2 Flexibility Measurements

3.3.2.1 Apparatus

Steel mandrels 1/4, 1/8, and 1/16 inches in diameter.

Bausch and Lomb binocular microscope.

3.3.2.2 Procedure

Strips of each final configuration plaque 3-1/2 inches x 1/2 inch were prepared by salt filling, machining and leaching. Each strip was bent 90° over progressively smaller mandrels and the area of maximum bending was observed for damage under the microscope. Duplicate samples of each material were tested.

3.3.3 Electrical Resistivity Measurements Perpendicular to the Felting Plane

3.3.3.1 Apparatus

Leeds and Northrup Kelvin Bridge model number 4288.

Five ton hydraulic press.

3.3.3.2 Procedure

A sample of AX2 fiber metal, 0.75 x 0.75 x 0.75 inches, was filled with a catalyzed epoxy resin and opposite faces ground parallel. The sample was placed between two copper blocks 1" x 1" x 1/2" thick.

This assembly was placed between the platens of a hydraulic press and resistance versus applied load measurements were made both parallel and perpendicular to the felting plane.

4.0 EXPERIMENTAL RESULTS AND DISCUSSION

4.1 Task A - Raw Material Classification

4.1.1 Microscopic Determination of Fiber Length and Apparent Diameter

The mean, median, and range for the length and apparent diameter of AX1, AX2, AX1 Modified, and DX nickel fiber were calculated[2] and are summarized in Table I. The frequency tabulations from which these data were obtained are included as Tables XVII through XXVIII in the Appendix. In addition, a frequency tabulation was made for the thickness of type DX fiber, since the regular shape and greater length of this type fiber make it possible to mount samples with the ends of the fibers normal to the viewing plane.

The data in Table I indicate that there is little difference in the apparent diameter of AX1, AX2 and AX1 Modified nickel fiber. The primary differences between these three grades are the greater length of AX1 Modified, which results in increased porosity, and in the thickness which decreases in the order AX2, AX1, AX1 Modified. This decrease in thickness, which was not measured due to the difficulty of orienting irregular fibers to obtain an end view, is evident in the surface area measurements which are reported in subsequent sections of this report.

The AX1 Modified material, which was separated from AX1 fiber by a technique that separates according to diameter regardless of length, is shown to have a larger apparent diameter than AX1 fiber. This anomaly can be explained, in part, by the observation that many of the small, symetrically shaped, particles were eliminated and that the number of small fibers is much greater than the number of large fibers. Consequently, a statistical description is more heavily weighted by the small fibers and the mean and median apparent diameter of both materials should be expected to be similar.

TABLE I

SUMMARY OF FREQUENCY TABULATION DATA FOR THE LENGTH AND APPARENT DIAMETER OF AX1, AX2, AX1 MODIFIED, AND DX NICKEL FIBER

Material	Diameter - Microns		
	Mean	Median	Range
AX1	11.2	5 - 7.5	1- 50
AX2	14.9	10 -12.5	1- 50
AX1 Mod.	13.7	8.7-11.2	1- 48
DX	61.8	63.7-66.2	1-121

Material	Length - Microns		
	Mean	Median	Range
AX1	139	88-112	13-1500
AX2	159	88-112	13-1500
AX1 Mod.	215	125-175	25-1375
DX	540	575	25-1325

Material	Thickness - Microns		
	Mean	Median	Range
DX	2.3	1.2-1.7	0.2-11.7

A test for significant difference between two standard deviations, the F test[3], was made for several duplicate determinations of fiber length and apparent diameter. The results show no significant difference at the 5% level of significance, indicating that the measuring technique is reproducible and that the resulting frequency distributions are typical of the entire population.

The data in Table I for type DX fiber show that this material has a greater length and apparent diameter than the other three materials. The apparent diameter measurement is clearly a width measurement for this type fiber as shown by photomicrographs. The greater length permits processing to a higher porosity while the smaller thickness results in greater internal surface area.

It is evident that a precise geometrical description of the fiber employed in this program is not possible. To supplement the statistical data, shadographs of typical fibers are shown in Figures 1 - 4. In addition photomicrographs of cross sectional areas are shown in Figures 5 - 8. These visual presentations, in conjunction with the microscopic measurements, provide an overall description of the fibers and of the differences between the grades of fiber.

Low magnifications photographs of as sintered surfaces of each of the materials are shown in Figures 9 - 12.

4.2 Task B - Sintering Study

4.2.1 Density versus Sintering Temperature Measurements

The effect of sintering temperature, at a constant time of 20 minutes, upon the density of AX1, AX2, and AX1 Modified nickel fiber metal plaques is shown in Table II. These data are plotted in Figure 13 and show a gradual increase in density with increasing sintering temperature. The effect of fiber length upon the maximum porosity obtainable is indicated by the lower initial density of AX1 Modified plaques,

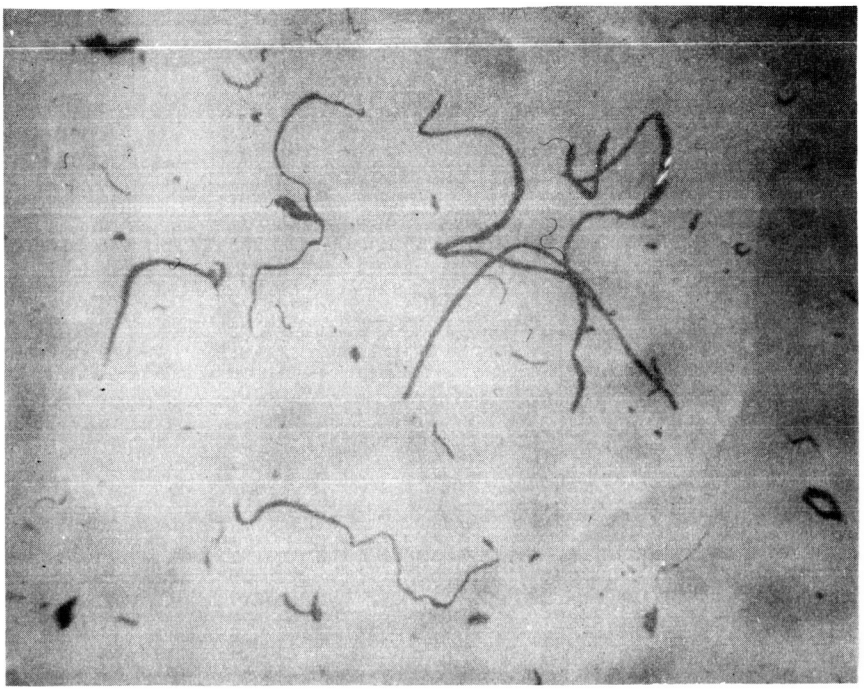


Fig. 1 Shadograph of AX1 Nickel Fiber X57

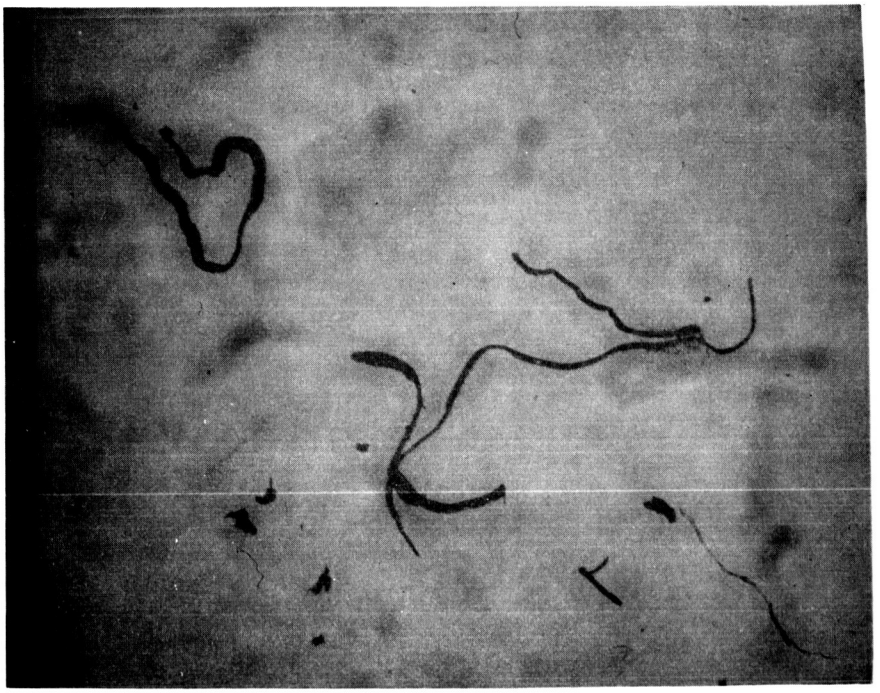


Fig. 2 Shadograph of AX2 Nickel Fiber X57

Fig. 3 Shadograph of AX1 Modified Nickel Fiber X57

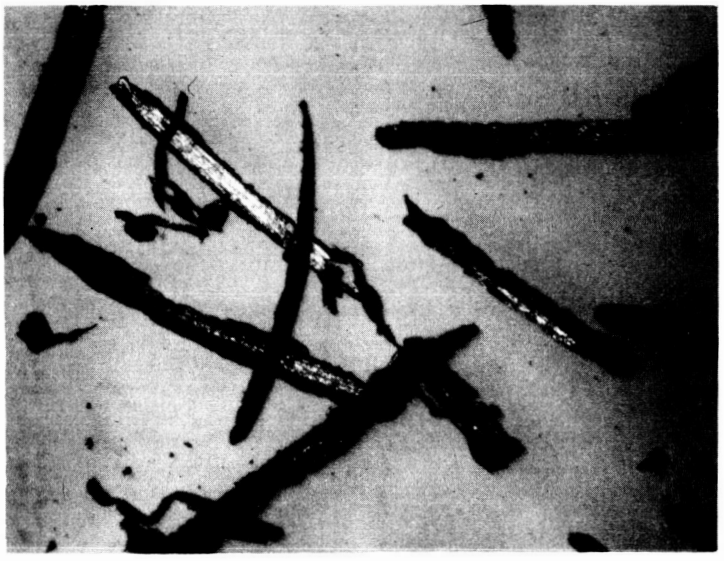


Fig. 4 Shadograph of DX Nickel Fiber X57

Fig. 5 Photomicrograph of AX1 Nickel fiber sintered plaque 11% dense. Section is perpendicular to felting plane. X210

Fig. 6 Photomicrograph of AX2 Nickel fiber sintered plaque 12% dense. Section is perpendicular to felting plane. X210

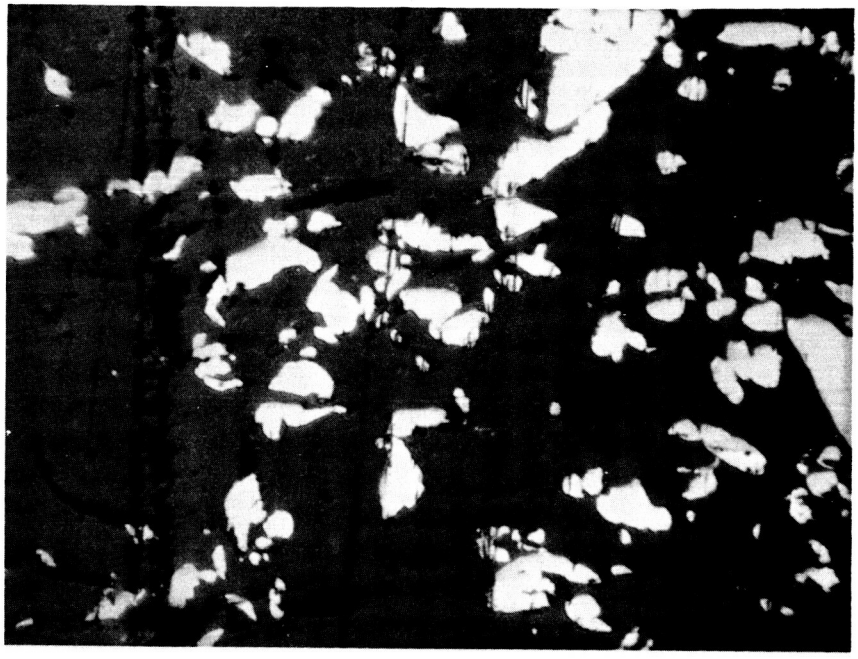


Fig. 7 Photomicrograph of AX1 Modified Nickel fiber sintered plaque 10% dense. Section is perpendicular to the felting plane. X210

Fig. 8 Photomicrograph of DX Nickel fiber sintered plaque 10% dense. Section is perpendicular to the felting plane. X210

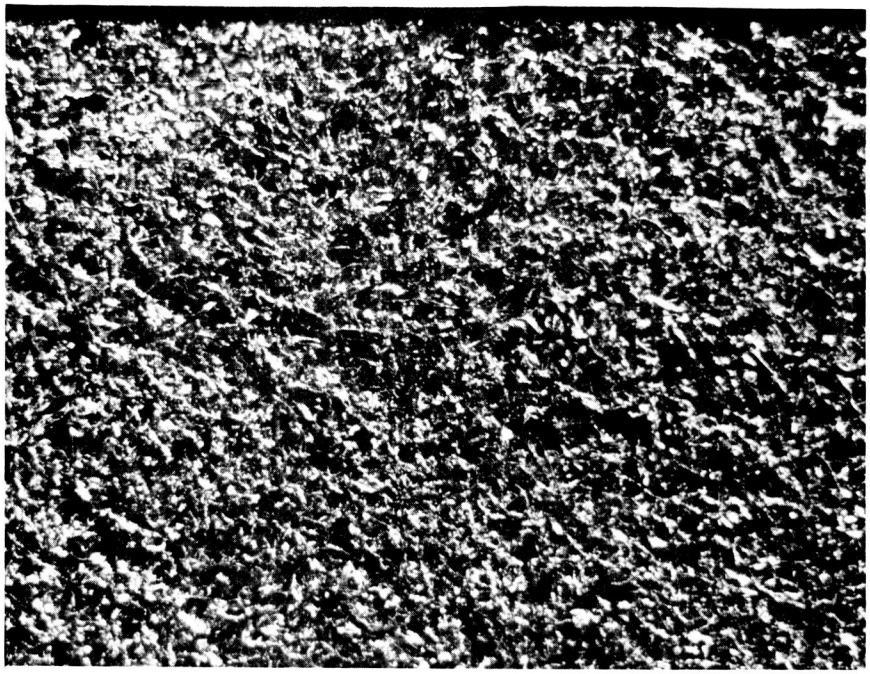


Fig. 9 As sintered surface of AX1 Nickel fiber plaque X15

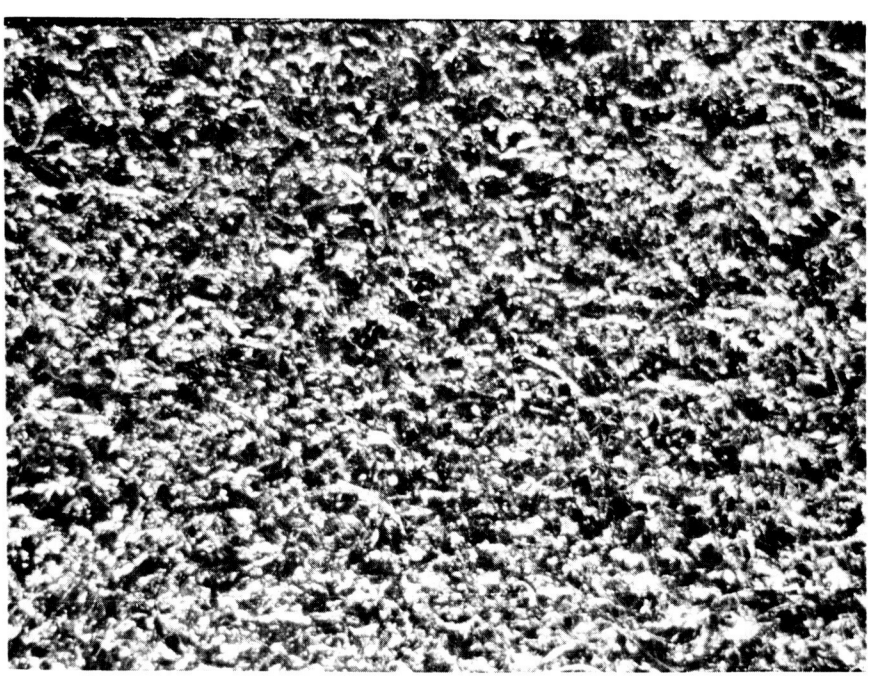


Fig. 10 As sintered surface of AX2 Nickel fiber plaque X15

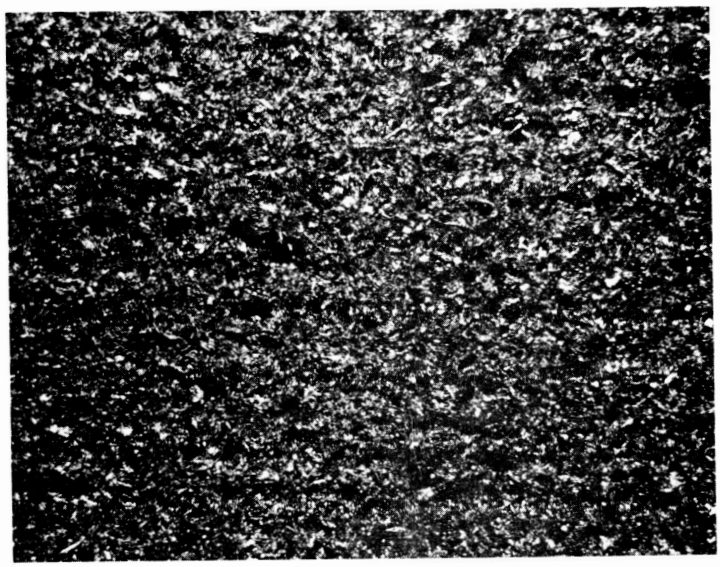


Fig. 11 As sintered surface of AX1 Modified Nickel fiber plaque X15

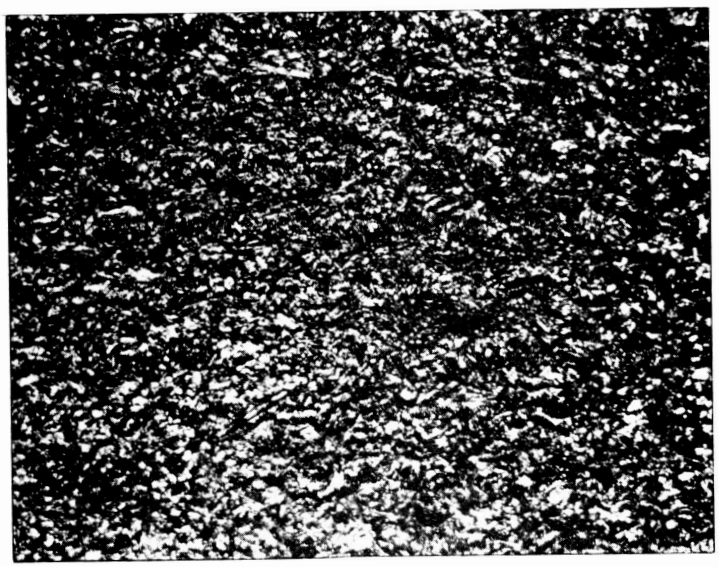


Fig. 12 As sintered surface of DX Nickel Fiber plaque X15

TABLE II

DENSITY VS SINTERING TEMPERATURE
of
NICKEL FIBER METAL PLAQUES

Material	Sintering Temperature °F	Density % of Theoretical
AX1	1600	11.6
	1800	11.5
	2000	11.7
	2150	12.9
AX2	1600	10.6
	1800	10.9
	2000	11.8
	2150	12.4
AX1 Modified	1600	9.0
	1800	9.3
	2000	9.8
	2150	10.5

which have the longest fibers, and the higher initial density of AX1 plaques, which have the shortest fibers. For the same weight of nickel fiber, a battery plaque that is 10% dense can theoretically hold twice as much active material as a battery plaque 18% dense. To maximize the porosity of nickel fiber metal battery plaques the sintering temperature should be as low as the other plaque characteristics dictate.

4.2.2 Porosity versus Sintering Temperature Measurements

Pore size distribution versus sintering temperature data are shown graphically in Figures 14, 15, and

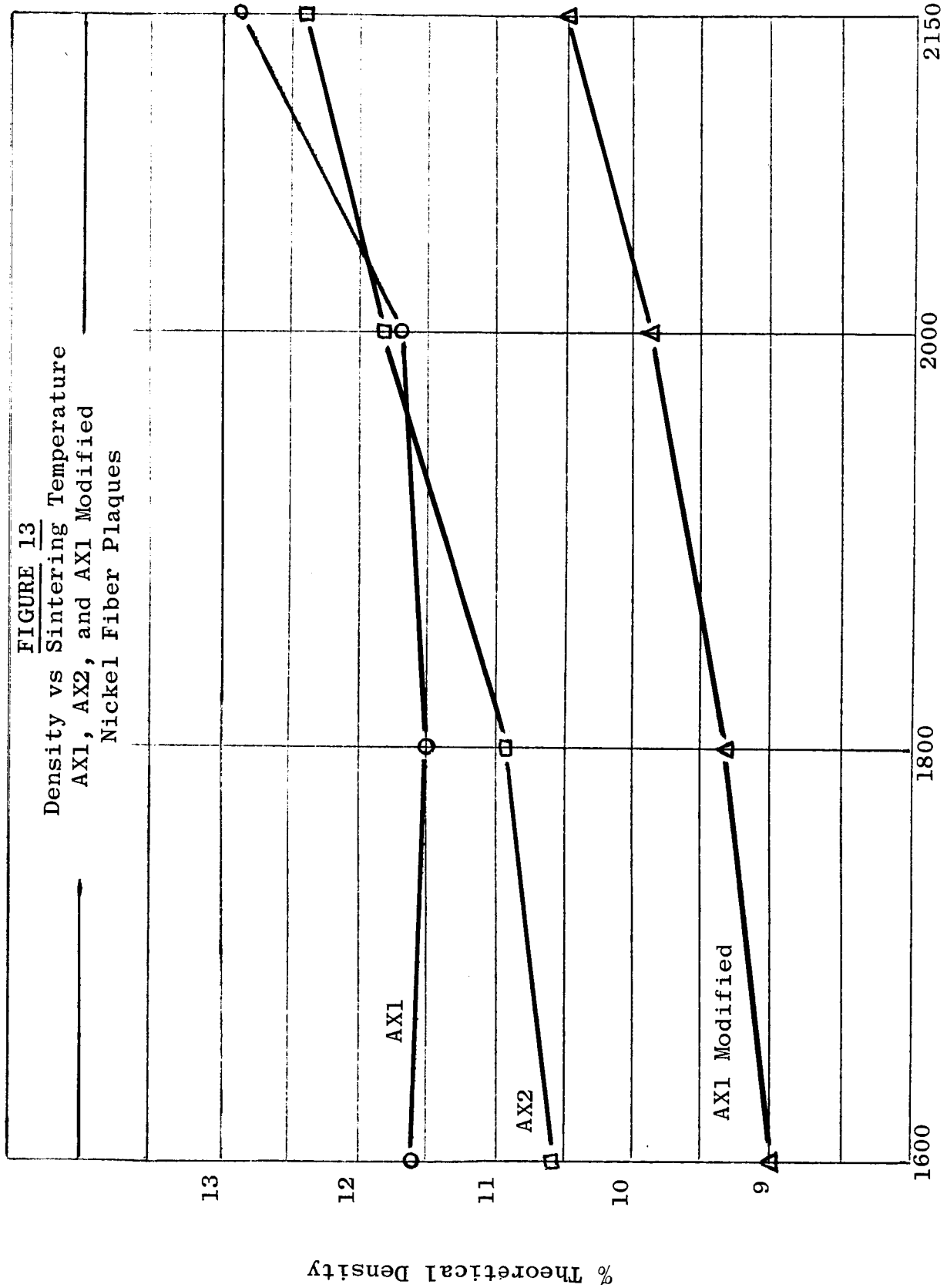
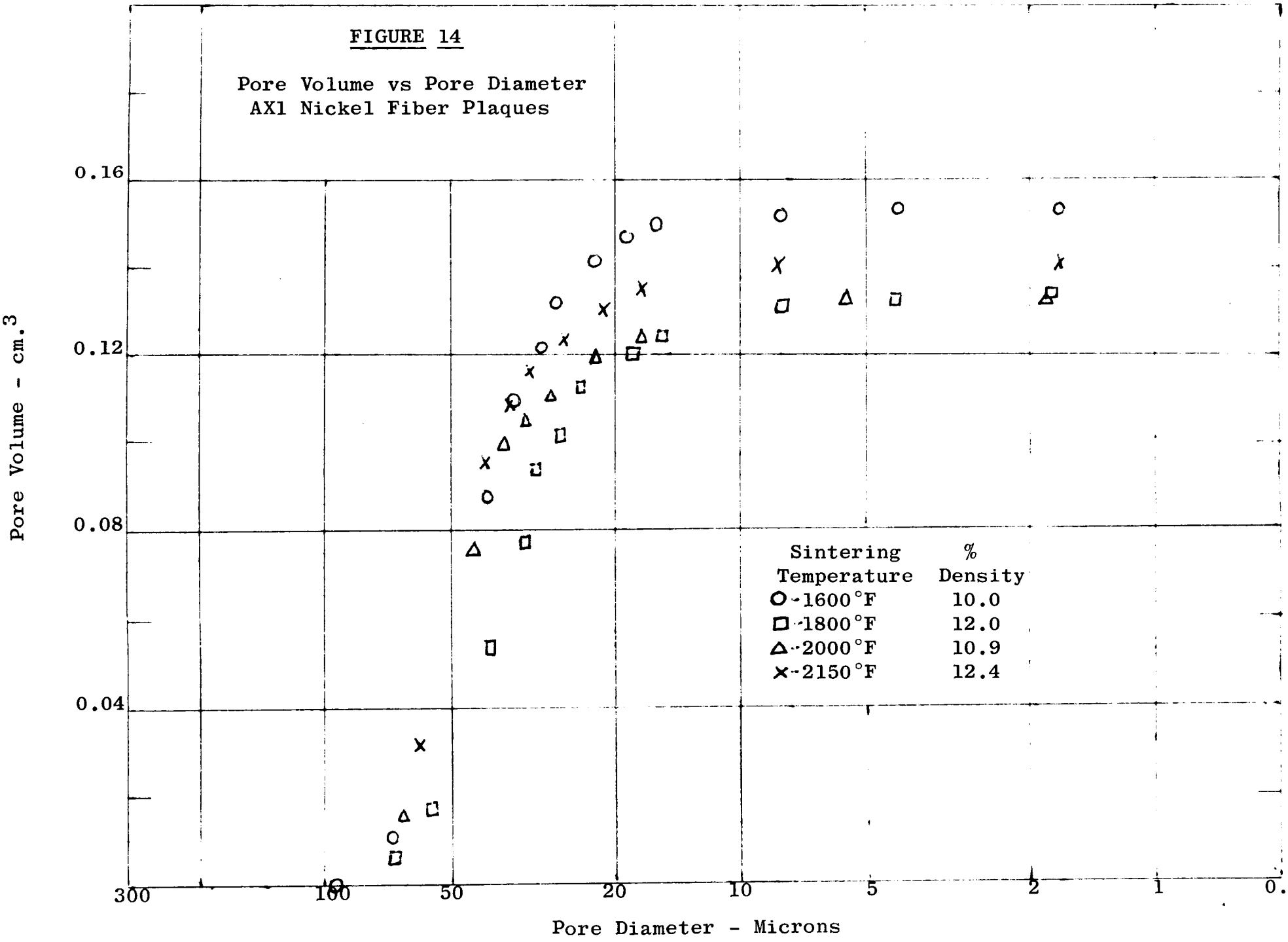


FIGURE 13
Density vs Sintering Temperature
AX1, AX2, and AX1 Modified
Nickel Fiber Plaques

FIGURE 14

Pore Volume vs Pore Diameter
AX1 Nickel Fiber Plaques



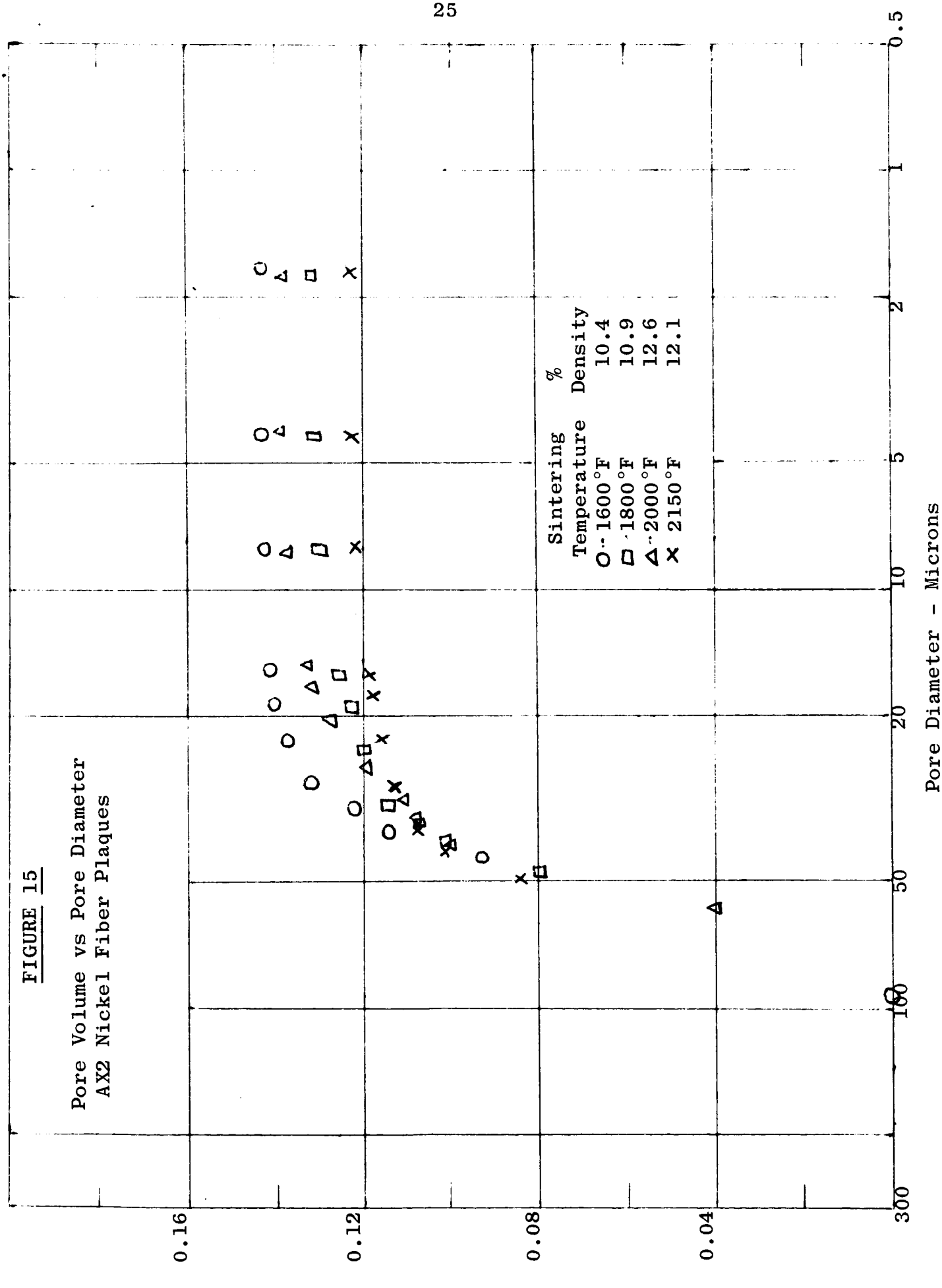


FIGURE 15

Pore Volume vs Pore Diameter
AX2 Nickel Fiber Plaques

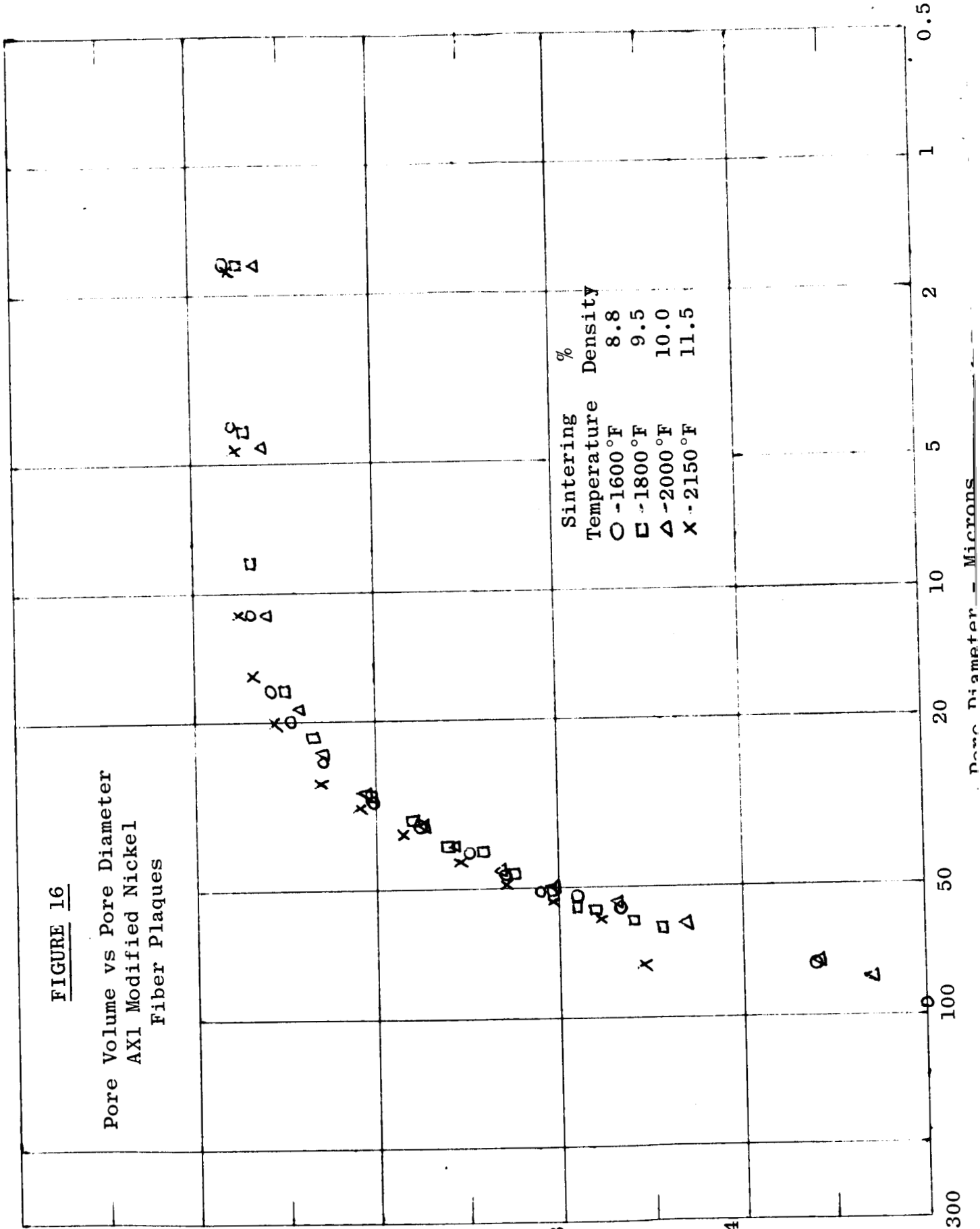


FIGURE 16

Pore Volume vs Pore Diameter
AX1 Modified Nickel
Fiber Plaques

16 for AX1, AX2, and AX1 Modified nickel fiber metal plaques respectively. The effect of sintering temperature is shown by plotting the results obtained at different temperatures on the same graph. It is evident that sintering temperature has little effect upon the pore size distribution and that all the materials studied have an appreciable number of pores in the desired range. Since the sintering temperature does not affect the pore size distribution significantly, this parameter was not considered significant in determining the optimum sintering temperature.

TABLE III

SUMMARY OF POROSIMETRY DATA FOR AX1, AX2, and AX1 MODIFIED NICKEL FIBER METAL PLAQUES

Material	Sintering Temperature °F	Density % of Theoretical	Median Pore Size, Microns	Volume of Pores 10-50 Microns, %
AX1	1600	10.0	43	67.5
	1800	12.0	38	78.5
	2000	10.9	48	53.9
	2150	12.4	48	55.0
AX2	1600	10.4	48	72.8
	1800	10.9	52	42.6
	2000	12.6	44	56.2
	2150	12.1	53	36.9
AX1 Modified	1600	8.8	54	42
	1800	9.5	54	41
	2000	10.0	54	42
	2150	11.5	57	41

It should be noted, however, that the pore size distribution is determined by the interaction between fiber diameter and density and that the mean pore

diameter can be varied over a wide range at the expense of porosity and/or fiber diameter.

Table III shows the median pore size and volume of pores 10 to 50 microns in diameter. The remaining porosity is predominately in the 50 to 100 micron range and over 99% of all the pores are interconnected.

4.2.3 Tensile Strength versus Sintering Temperature Measurements

The tensile strengths determined for AX1, AX2, and AX1 Modified nickel fiber, as a function of sintering temperature, are shown in Table IV.

TABLE IV

SINTERING TEMPERATURE AND TENSILE STRENGTH DATA FOR AX1, AX2, AND AX1 MODIFIED NICKEL FIBER METAL PLAQUES

Material	Sintering Temperature °F	Density % of Theoretical	Tensile Strength lbs per in^2
AX1	1600	10.0	41.2
	1800	12.1	144
	2000	13.7	363
	2150	15.3	451
AX2	1600	10.5	53.3
	1800	9.7	72.2
	2000	12.5	227
	2150	12.7	265
AX1 Modified	1600	8.95	19.6
	1800	9.31	58.6
	2000	9.75	69.6
	2150	10.5	161

The density of the samples shown varies as a result of the increasing sintering temperature. It is known that the tensile strength increases in direct proportion to the density in the range from 10% to 20% density; consequently the strengths have been normalized linearly to 10% density and plotted in Figure 17. The deviation from linearity of the strength - density relationship is considered to be insignificant in the range of the tests.

The difficulty of tensile testing fragile specimens of a material that is composed mostly of pores and notches is evident in some of the erratic data obtained. The values reported should be regarded as indicative and not absolute. The strength of all three fiber materials is sufficient to permit handling and processing. The manufacturing process for fiber metal plaques is such that a reinforcing screen could readily be incorporated into the structure if additional strength was required.

The tensile strength - sintering temperature interaction can be related to the size, surface area, and shape of the fibers involved. Smaller diameter fibers result in more contact points per volume of material at a given density. Material with high surface areas tend to sinter faster, at a given temperature, and small particles promote the sintering of larger particles.[4]

It would be expected that as time increased at any temperature the strength of the three materials would tend to converge. To maximize the tensile strength of any of the materials, the sintering temperature should be as high as the other plaque characteristics permit.

4.2.4 Surface Area versus Sintering Temperature Measurements

The permeability coefficients of plaques made from 0.003, 0.004 and 0.006 inch diameter wire are shown in Table V with the average value of the constant K_2.

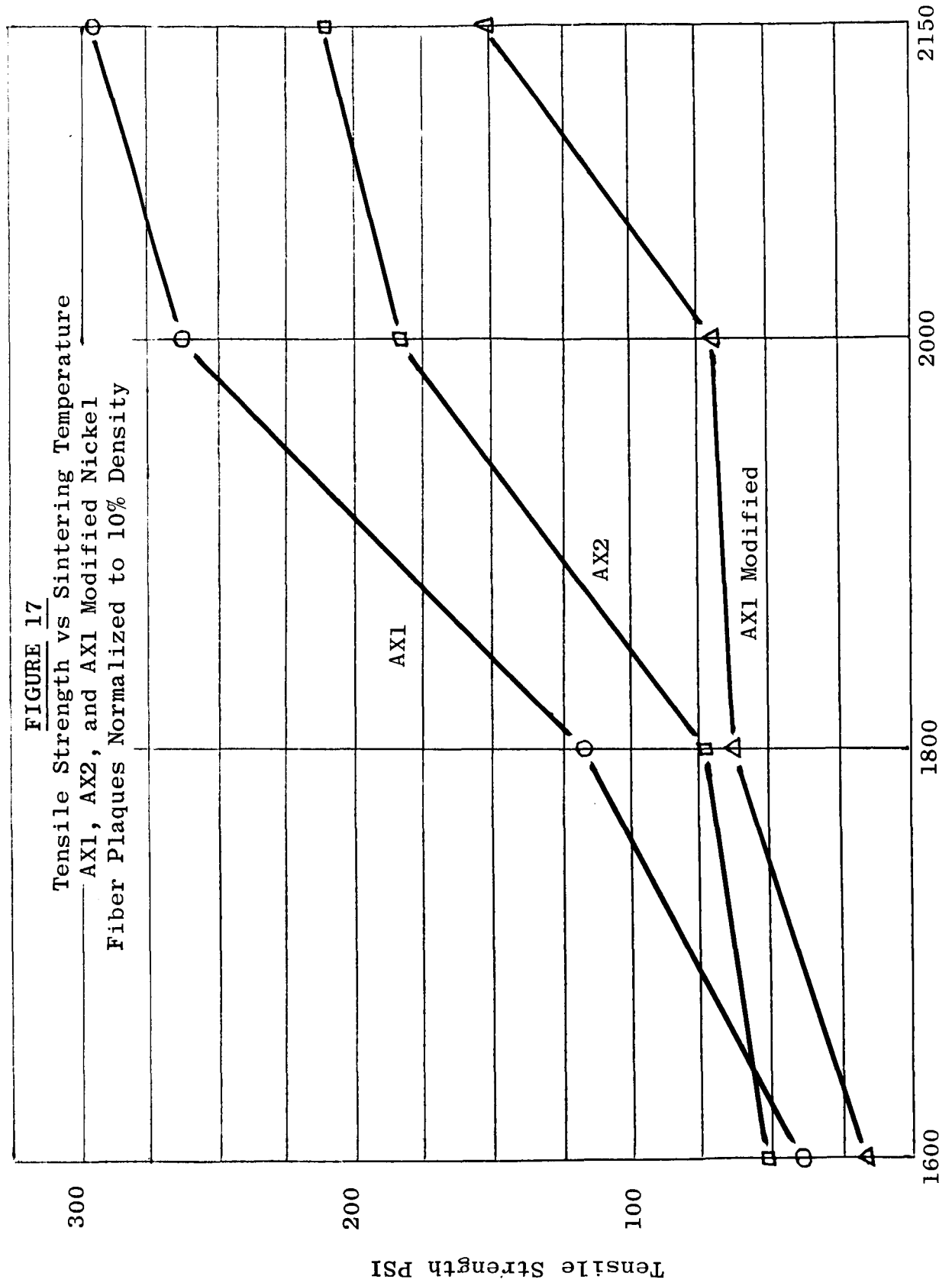


FIGURE 17
Tensile Strength vs Sintering Temperature
AX1, AX2, and AX1 Modified Nickel
Fiber Plaques Normalized to 10% Density

TABLE V

PERMEABILITY COEFFICIENTS, SURFACE AREA AND ORIENTATION FACTOR FOR PLAQUES MADE FROM WIRE

	Permeability Coefficient $\frac{VL}{\Delta P}$ SCFH/ft^2/in. H_2O/in. thickness	Known Surface Area cm^2/gm	K_2
30% dense 0.003" wire	2150	525	6.0×10^8
30% dense 0.004" wire	2000	394	7.8×10^8
30% dense 0.006" wire	8300	262	5.6×10^8
Average			6.5×10^8

The permeability coefficients (VL and ΔP) obtained for AX1, AX2, and AX1 Modified nickel fiber metal plaques at two densities are shown in Table VI. Figures 18, 19 and 20 are semi-logarithmic plots of density versus flow rate at a constant pressure drop. To keep the various parameters in compatible units and to determine the surface area at the same density that the K_2 constant was derived at, it is necessary to calculate the flow rate at a density of 30% of theoretical and a thickness of one inch. The data presented in Table VII were calculated using a density of 30% and a pressure drop of 0.1 inches of H_2O.

The specific surface area in cm^2/gm is obtained by dividing the specific surface area in cm^2/cm^3 by the density of nickel which is 8.9 gm/cm^3.

A plot of internal surface area versus sintering temperature, Figure 21, shows that the surface area increases in the order AX2, AX1, AX1 Modified. The increase in surface area is not apparent from the statistical description of the fibers presented in Task A. It is evident that the "thickness", or

TABLE VI

PERMEABILITY COEFFICIENTS FOR AX1, AX2 AND AX1 MODIFIED NICKEL FIBER PLAQUES SINTERED AT VARIOUS TEMPERATURES

Material	Sintering Temperature °F	Density % of Theoretical	Flow Rate $SCFH/Ft^2$ $\Delta P = 0.1$ inch H_2O
AX1	1600	18.1	420
	1600	27.8	178
	1800	19.3	417
	1800	28.2	172
	2000	19.2	500
	2000	31.6	209
	2150	19.4	570
	2150	28.6	308
AX2	1600	18.1	580
	1600	28.3	238
	1800	18.6	520
	1800	28.8	234
	2000	19.0	625
	2000	29.8	266
	2150	19.3	705
	2150	28.8	334
AX1 Modified	1600	18.3	374
	1600	26.9	111
	1800	18.9	423
	1800	26.3	153
	2000	19.5	451
	2000	28.3	147
	2150	19.4	550
	2150	28.4	176

FIGURE 18

FLOW RATE VS DENSITY AND SINTERING TEMPERATURE FOR AX1 NICKEL FIBER PLAQUES AT A PRESSURE DROP OF 0.1 INCH OF H_2O AND A THICKNESS OF 0.030 INCH

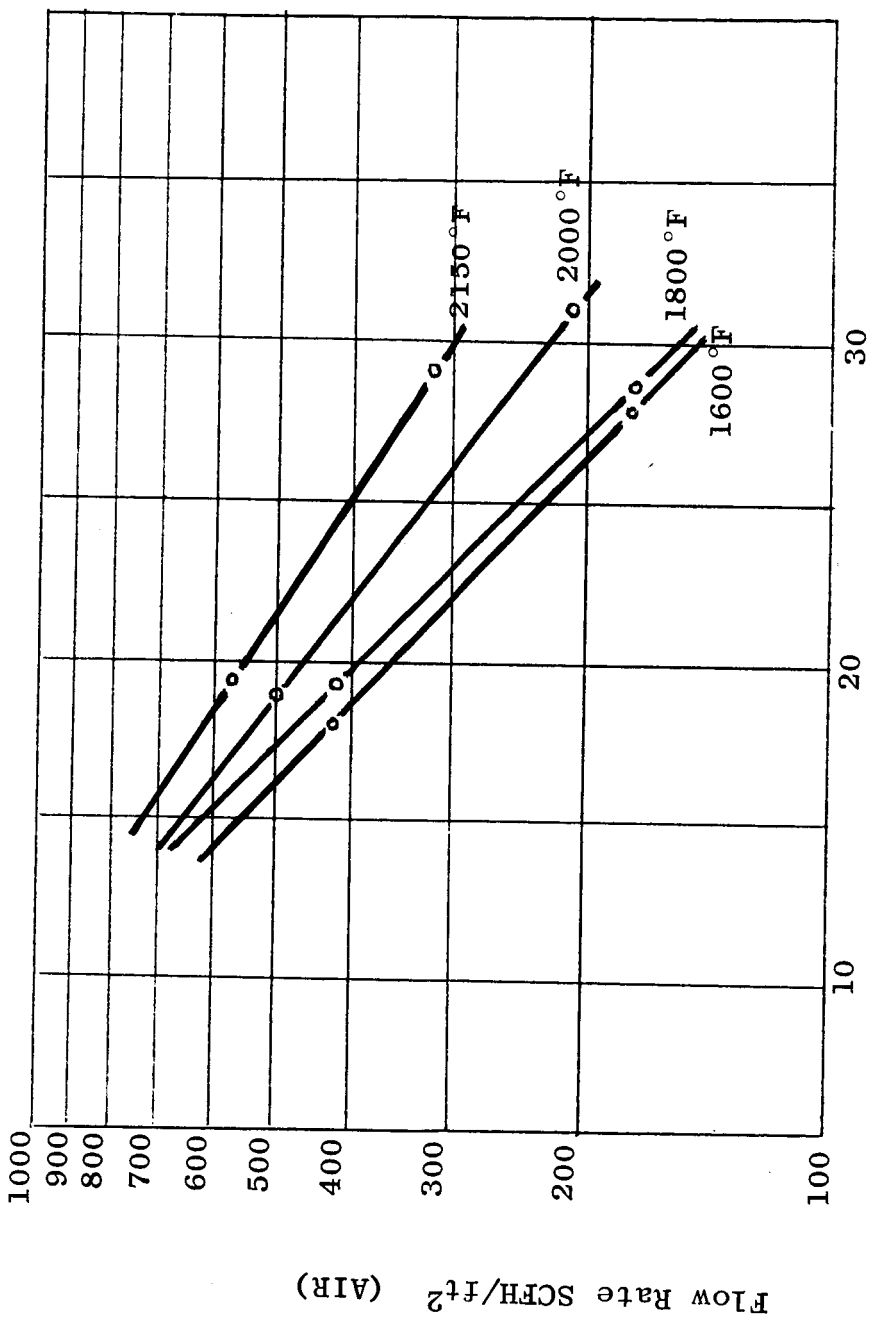


FIGURE 19

FLOW RATE VS DENSITY AND SINTERING TEMPERATURE
FOR AX2 NICKEL FIBER PLAQUES AT A PRESSURE DROP
OF 0.1 INCH OF H_2O AND A THICKNESS OF 0.030 INCH

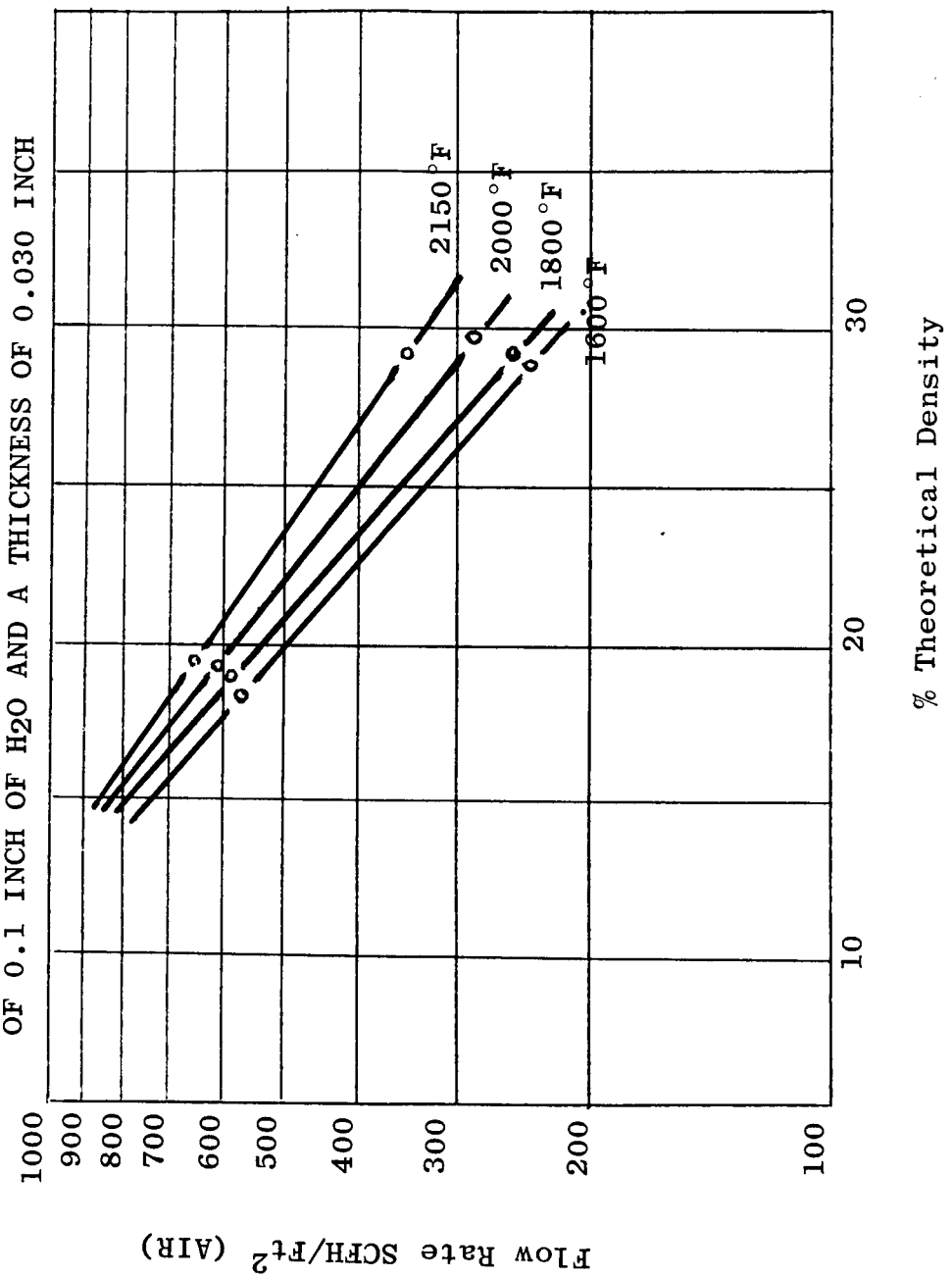
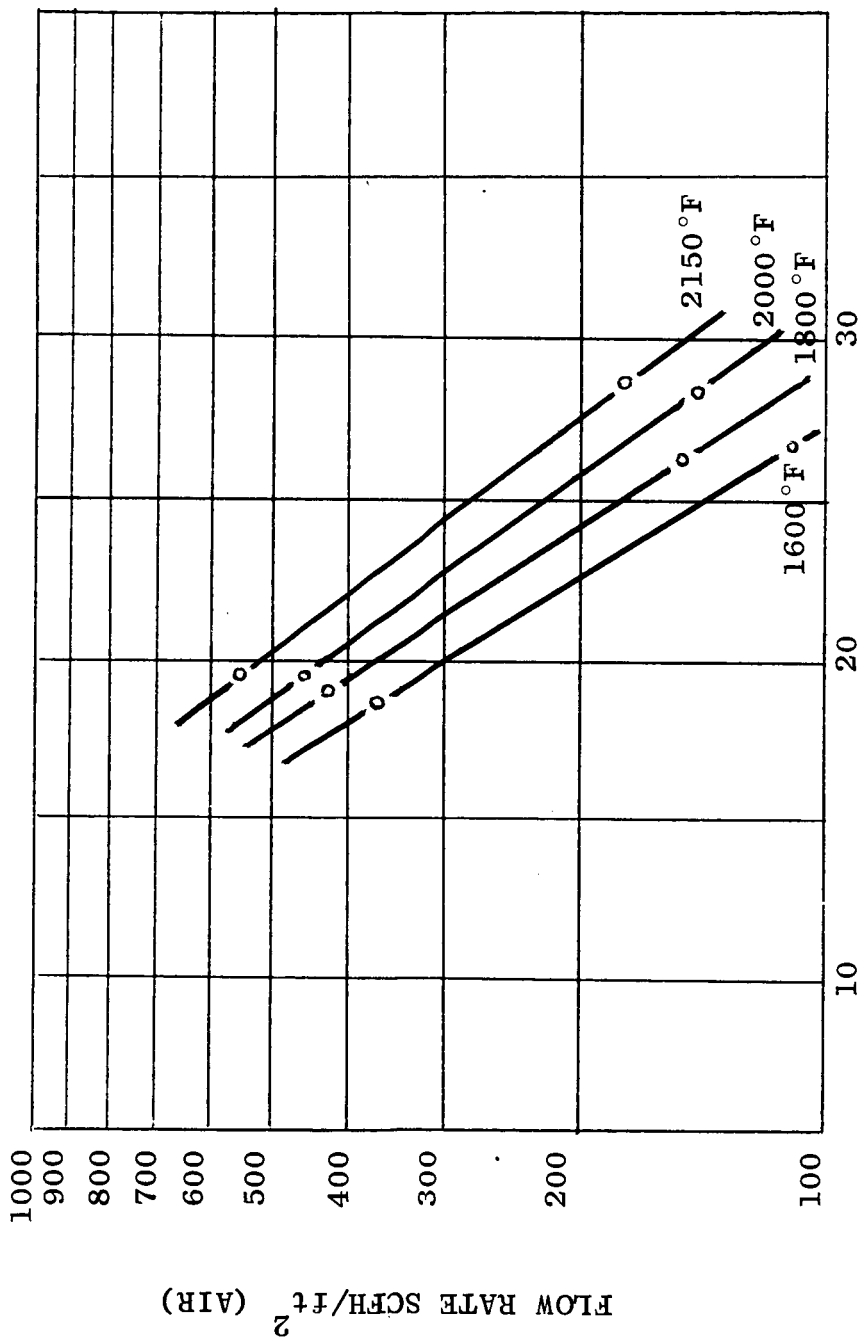


FIGURE 20

FLOW RATE VS DENSITY AND SINTERING TEMPERATURE FOR AX1 MODIFIED NICKEL FIBER PLAQUES AT A PRESSURE DROP OF 0.1 INCH OF H_2O AND A THICKNESS OF 0.030 INCH



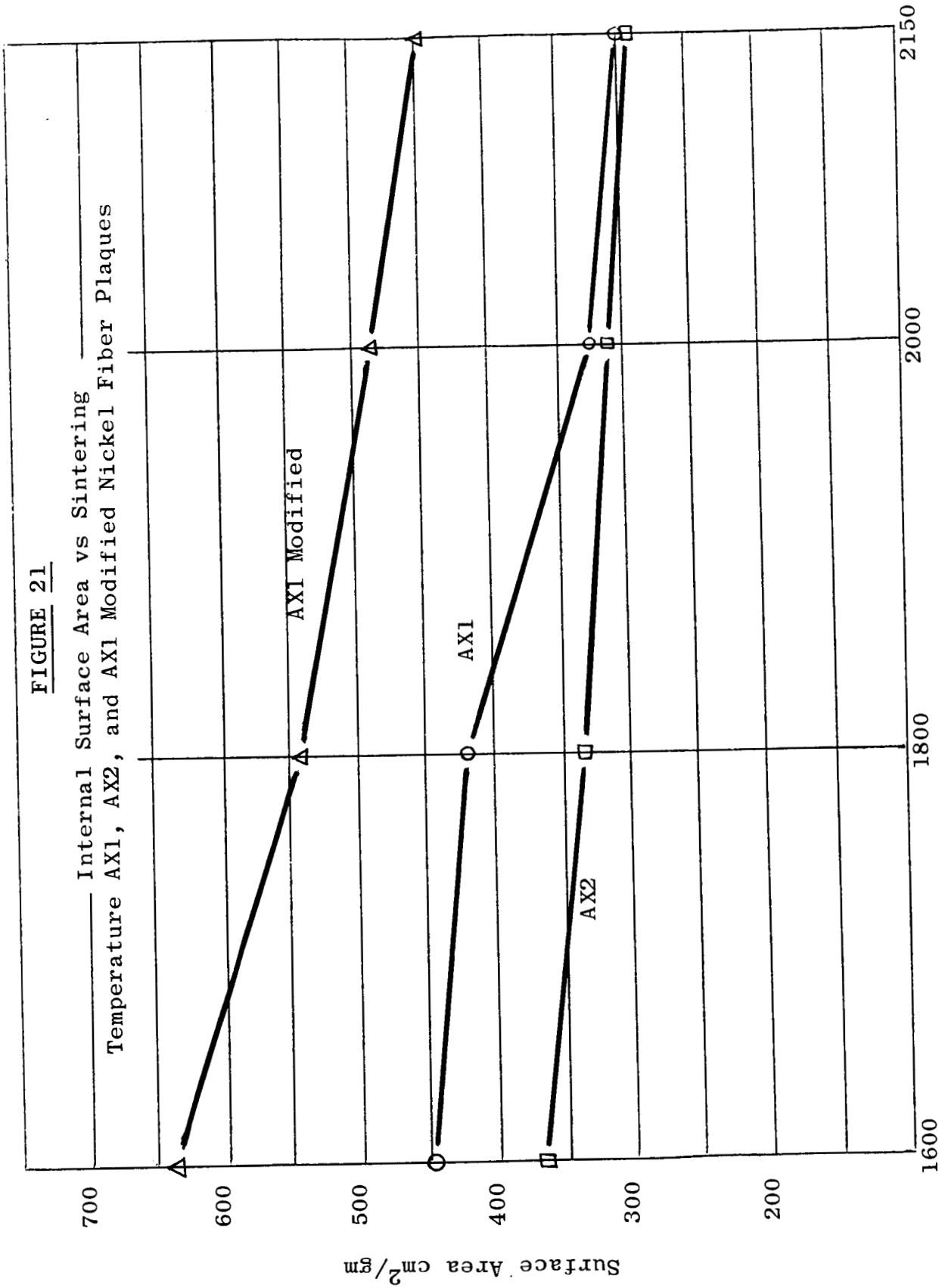


FIGURE 21
Internal Surface Area vs Sintering Temperature AX1, AX2, and AX1 Modified Nickel Fiber Plaques
AX1 Modified
AX1
AX2
Surface Area cm2/gm
700
600
500
400
300
200
1600
1800
2000
2150
Sintering Temperature - °F

TABLE VII

SPECIFIC SURFACE AREA OF AX1, AX2, AND AX1 MODIFIED NICKEL FIBER METAL PLAQUES SINTERED AT VARIOUS TEMPERATURES

Material	Sintering Temperature °F	$\frac{VL}{\Delta P}$ SCFH/ft^2/ in. H_2O/in. Thickness	S_v^2	S_v cm^2/cm^3	S_v cm^2/gm
AX1	1600	42	15.4×10^6	3920	440
	1800	46.5	13.9×10^6	3730	419
	2000	75	8.6×10^6	2930	329
	2150	90	7.2×10^6	2690	302
AX2	1600	60	10.8×10^6	3280	369
	1800	69.5	9.3×10^6	3050	343
	2000	82.5	7.8×10^6	2800	315
	2150	93	6.9×10^6	2640	296
AX1 Mod.	1600	19.8	32.8×10^6	5740	645
	1800	27.6	23.6×10^6	4860	546
	2000	33.9	19.2×10^6	4380	492
	2150	42.8	15.2×10^6	3900	438

the dimension of the fiber that lies in the viewing plane when observed through a microscope, must decrease in the order AX1 Modified, AX1, AX2.

The greater surface area of the smaller apparent diameter fibers renders them more susceptible to sintering than the coarser fibers because the rate of sintering is sensitive to surface free energy.[5] Consequently, the decrease in surface area is most rapid for AX1 and AX1 Modified at the lower sintering temperatures. Figures 22 and 23 are photomicrographs of AX1 nickel fiber metal plaques sintered at the indicated temperatures. As the sintering temperature increases, the reduction in surface energy is accelerated; the sharp

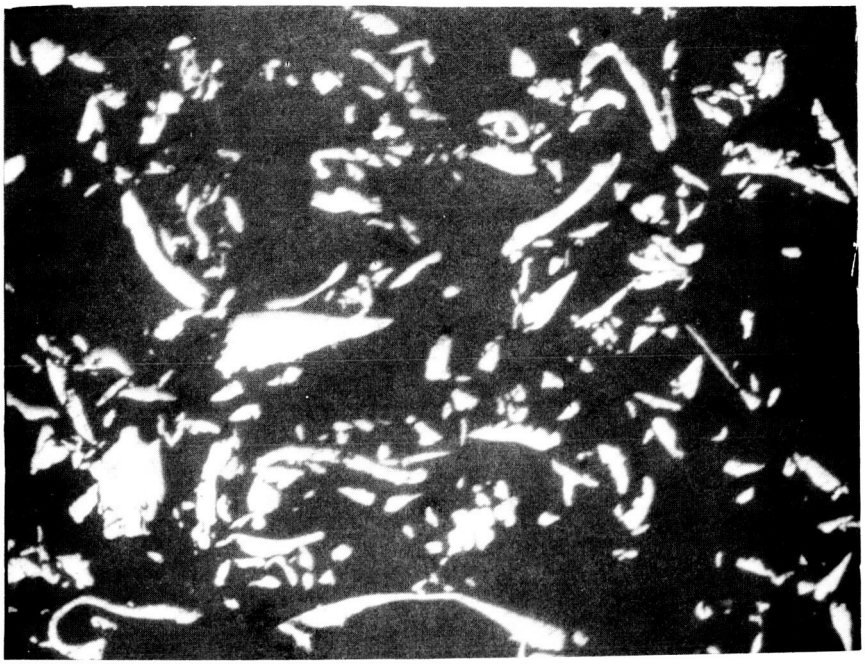


Fig. 22 Photomicrograph of AX1 nickel fiber plaque sintered at 1600°F. Section is perpendicular to felting plane. X 210

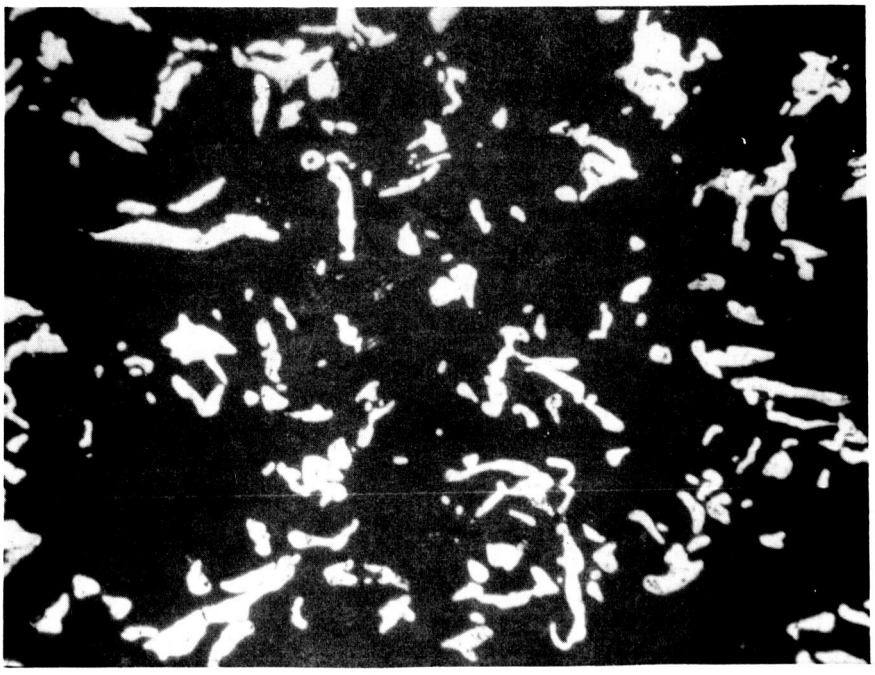


Fig. 23 Photomicrograph of AX1 nickel fiber plaque sintered at 2000°F. Section is perpendicular to felting plane. X 210

edges of the fibers become more rounded and their cross section takes on a more symmetrical shape. The surface area is, therefore, decreased.

Internal surface area measurements using air permeability techniques have been supplemented by a B.E.T. Krypton determination. A sample of AX1 nickel fiber metal sintered at 1900±15°F for 20 minutes was submitted to Nuclear Materials and Equipment Corporation for a gas adsorption surface area determination.[6] Air permeability calculations indicated an area of 370 cm^2/gm. Krypton adsorption indicated an area of 590 cm^2/gm.

These results show the difficulty experienced in accurately determining the surface area of materials in the 100-1000 cm^2/gm range and indicate that the most satisfactory test for nickel fiber metal battery plaques would be to evaluate them as electrodes in a battery.

The results obtained in this study indicate that the sintering temperature for nickel fiber metal plaques should be as low as practical to maximize the surface area.

4.2.5 Electrical Resistivity versus Sintering Temperature Measurements

Electrical resistivity data as a function of sintering temperature for AX1, AX2, and AX1 Modified nickel fiber metal plaques are shown in Table VIII. The density increases as the sintering temperature increases, as noted previously. It has been shown[7] that the electrical resistivity of fiber metal composites is dependent upon the density of the composite and not upon the contact section. The data in the above referenced work were obtained at relatively high densities. The data shown in Figure 24, a plot of electrical resistivity versus density for AX2 nickel fiber metal, indicate that at low densities the resistivity is not proportional to density. In fiber structures, as opposed to powder structures, the

resistivity is determined by the cross section of the fiber once sintering has progressed to the point where the cross sectional area of the sinter bond contacts is equal to or greater than the cross sectional area of the fiber. At very low densities there are few contact points per fiber and the resistivity may be determined by the size of the contact area as in powder structures. As the number of contact points increase (density increases) the resistivity is determined by the fiber cross section. With the materials investigated, it appears that the resistivity becomes proportional to density between 10% and 15% density.

TABLE VIII

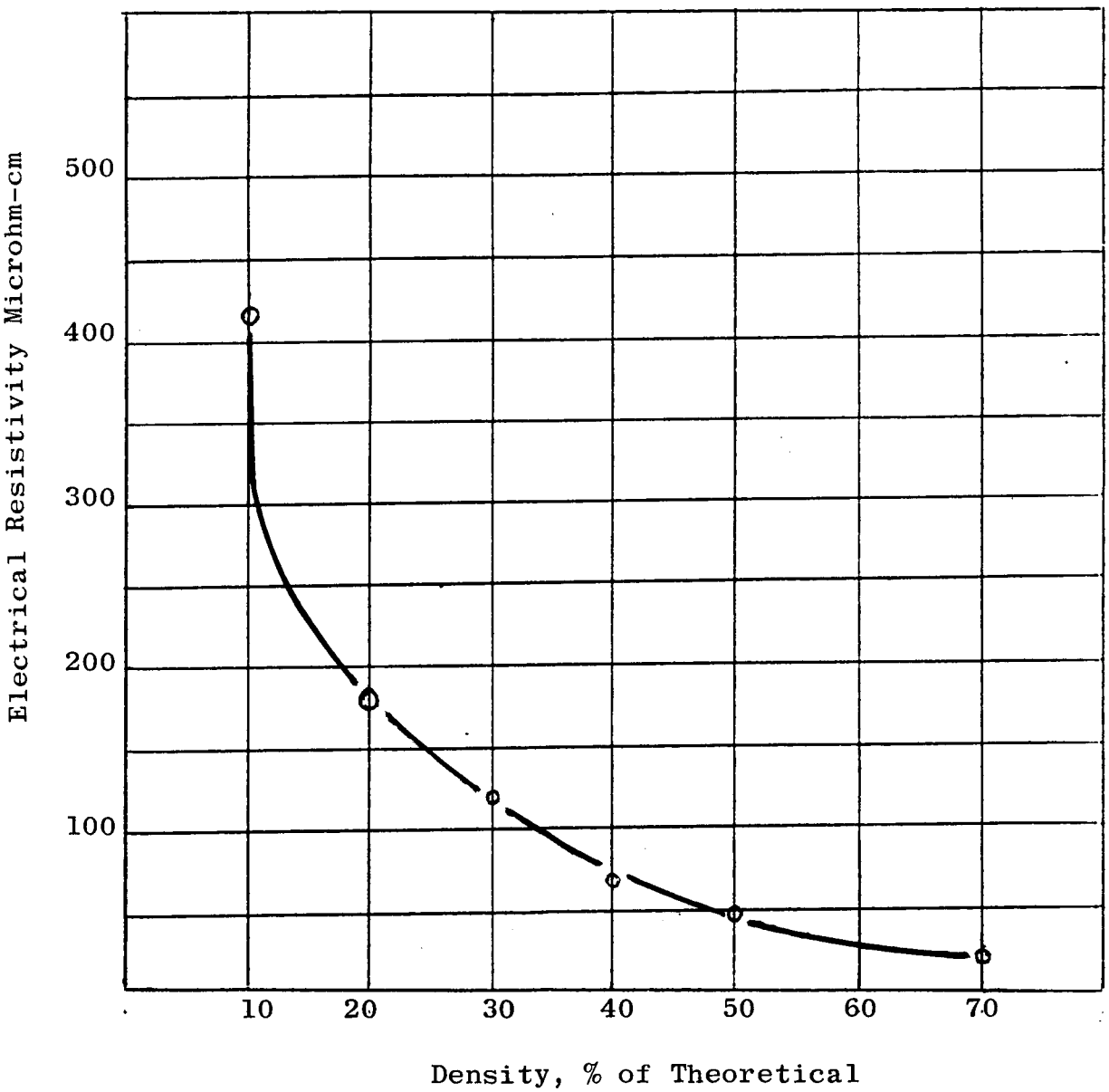
ELECTRICAL RESISTIVITY AND DENSITY FOR AX1, AX2, AND AX1 MODIFIED NICKEL FIBER PLAQUES SINTERED AT VARIOUS TEMPERATURES

Material	Sintering Temperature °F	Density % of Theoretical	Resistivity Microhm-cm
AX1	1600	11.3	1450
	1800	12.3	541
	2000	12.4	423
	2150	17.5	324
AX2	1600	11.4	1022
	1800	11.2	578
	2000	13.6	392
	2150	15.7	302
AX1 Mod.	1600	8.7	1070
	1800	9.1	746
	2000	9.5	611
	2150	11.0	438

In Figure 25, a plot of sintering temperature versus electrical resistivity, the resistivities have been normalized to 15% density; consequently, while

FIGURE 24

ELECTRICAL RESISTIVITY VS DENSITY OF
AX2 NICKEL FIBER METAL



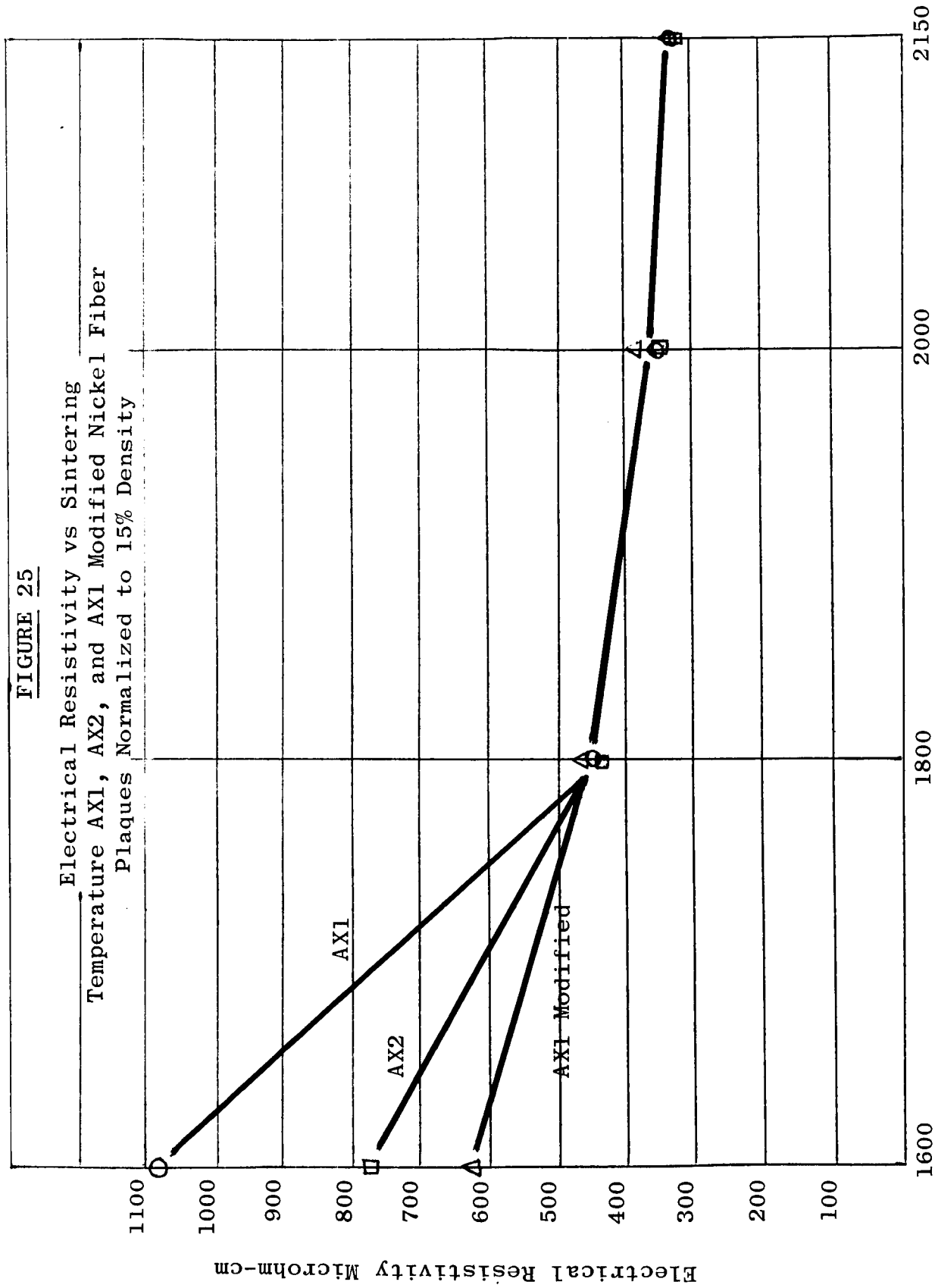


FIGURE 25

Electrical Resistivity vs Sintering Temperature AX1, AX2, and AX1 Modified Nickel Fiber Plaques Normalized to 15% Density

the data may not be absolute it does give an indication of the difference in electrical resistivity between the fiber grades.

The values obtained are similar to those obtained for some grades of nickel powder plaques.[8] The resistivity of fiber metal plaques can be decreased, of course, by incorporating a screen into the structure or by embossing a grid pattern into the plaque to obtain an interconnected path of high density material.

The results of this study show that the electrical resistivity decreases with increasing density and increasing sintering temperature. To minimize the resistivity the sintering temperature should be as high as possible without producing an excessive amount of shrinkage.

4.3 Task C - Plaque Classification

4.3.1 Sintering

The data presented in Task B are sufficient to permit an evaluation of the variation of plaque characteristics with sintering temperature. It is evident that any single characteristic can be optimized at the expense of other characteristics. A sintering temperature of 1900±15°F for 20 minutes was selected to compromise the decreasing surface area and porosity and the increasing tensile strength and electrical conductivity. Final configuration plaques of AX1, AX2 and AX1 Modified nickel fiber were produced at a thickness of 0.028" ± 0.001" and sintered as indicated above. Portions of these plaques were used to determine pore size distribution, surface area, tensile strength, electrical resistivity, and flexibility.

4.3.2 Density Measurements

The densities of all three materials are shown in Table IX.

TABLE IX

DENSITY OF NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Density % of Theoretical
AX1	11.7
AX2	11.2
AX1 Modified	12.2

In addition to the densities of the complete plaques, measurements were made for the samples used in subsequent testing. As will be noted, the density varies somewhat due to the machining operations required to shape the samples. Since many of the plaque characteristics are sensitive to density, measurements were made of each test sample before testing.

4.3.3 Pore Size Distribution

Duplicate samples of each material were tested to determine the pore size distribution. The resulting data were averaged for each material and plotted as shown in Figure 26.

The average median pore size and average volume of pores in the 10 to 50 micron range are shown in Table X. The remaining porosity is predominately in the 50 to 100 micron range and over 99% of all porosity is interconnected.

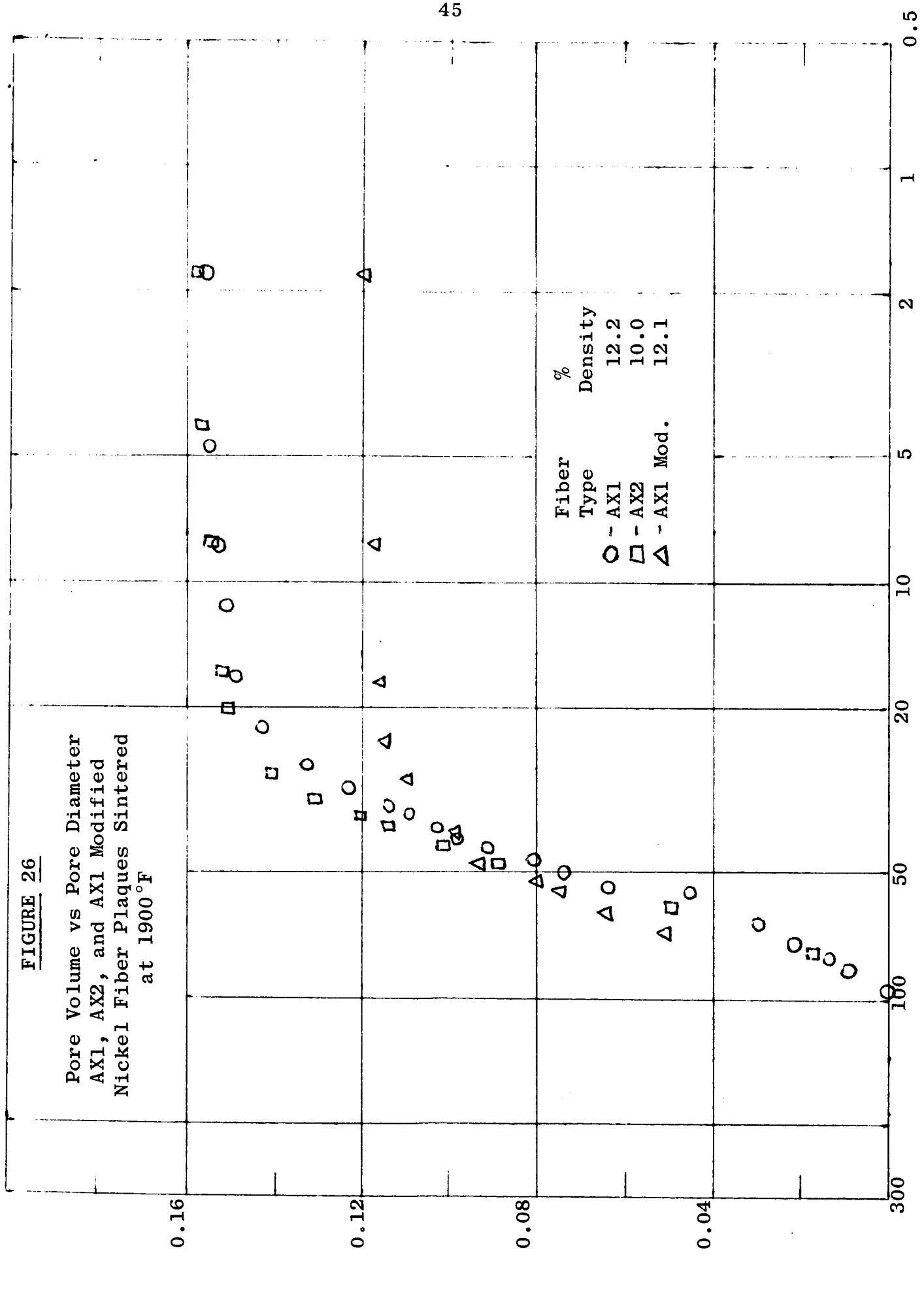


FIGURE 26

Pore Volume vs Pore Diameter AX1, AX2, and AX1 Modified Nickel Fiber Plaques Sintered at 1900°F

TABLE X

DENSITY, MEDIAN PORE SIZE AND VOLUME OF POROSITY DUE TO PORES 10 to 50 MICRONS IN DIAMETER OF NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Density % of Theoretical	Median Pore Size Microns	Volume of Pores 10-50 Microns %
AX1	12.3	50.0	47.4
	13.1	47.5	54.9
		Ave. 48.8	Ave. 51.2
AX2	10.2	61.0	29.8
	10.0	65.0	27.5
		Ave. 63.0	Ave. 28.7
AX1 Modified	12.0	49.0	50.0
	12.1	49.0	51.0
		Ave. 49.0	Ave. 50.5

4.3.4 Tensile Strength

The average tensile strength of each material at the indicated density is shown in Table XI. The data presented are significantly different from those reported in Task B. These erratic data can be expected when attempting to tensile test fragile specimens of a material composed largely of notches and pores.

TABLE XI

DENSITY AND TENSILE STRENGTHS OF NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Density % of Theoretical	Tensile Strength lbs. per in.^{2}
AX1	11.5	96.9
	11.4	97.6
AX2	11.1	53.8
	11.3	61.6
AX1 Modified	12.5	167.0
	12.6	183.5

4.3.5 Surface Area

Flow rate versus pressure drop data are shown in Table XII for AX1, AX2, and AX1 Modified nickel fiber metal plaques.

TABLE XII

DENSITY AND FLOW RATE DATA FOR NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Density % of Theoretical	Flow Rate SCFH/ft^2 $\Delta P = 0.1''$ H_2O
AX1	20.3	342
	20.2	496
	28.5	128
	29.4	156
AX2	19.0	562
	19.5	688
	28.4	164
	30.7	127

TABLE XII
Continued

Material	Density % of Theoretical	Flow Rate SCFH/ft^2 ΔP = 0.1" H_2O
AX1 Modified	19.8	415
	19.7	390
	30.2	104
	30.1	105

These data were used to plot the density versus flow rate curves shown in Figure 27. Surface area calculations were made as described in Task B and the specific surface area obtained are shown in Table XIII.

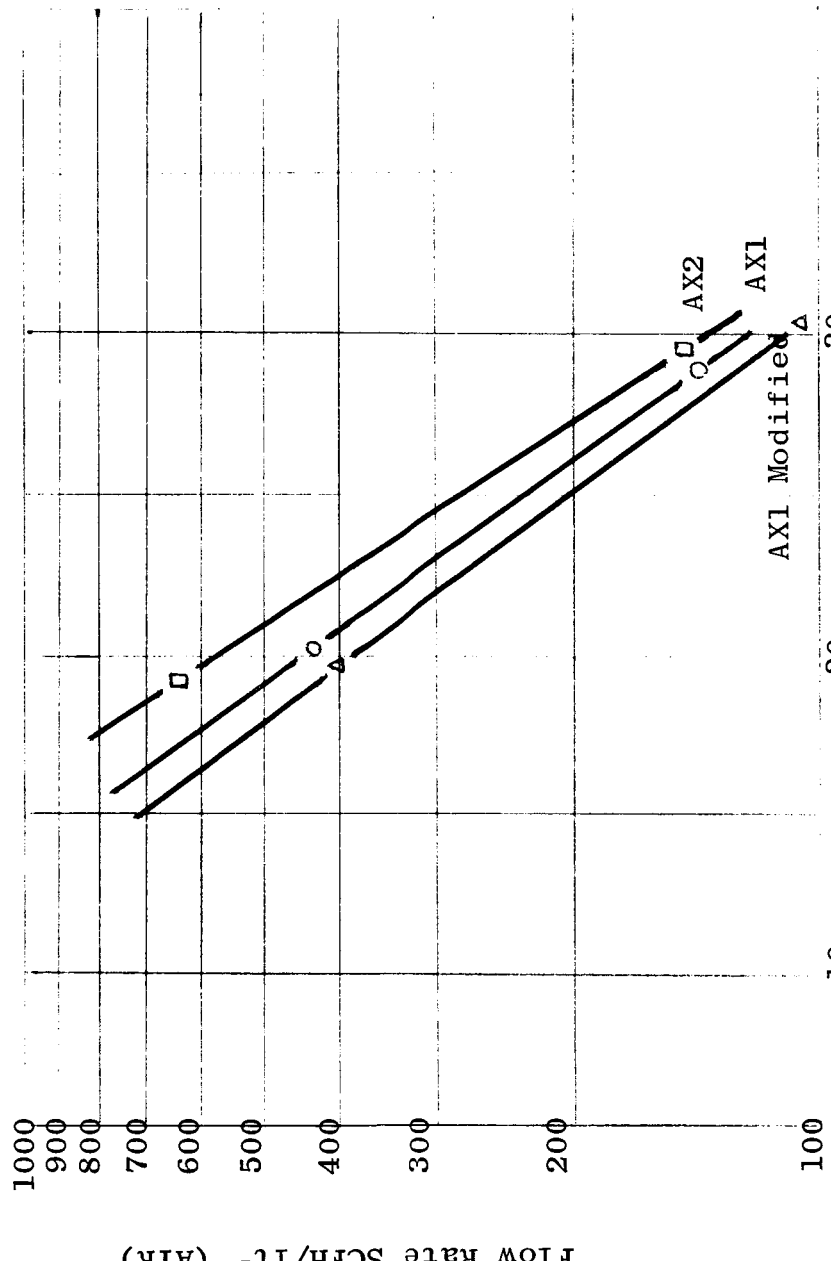
TABLE XIII

SPECIFIC SURFACE AREAS FOR NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	$\frac{VL}{\Delta P}$ SCFH/ft^2/ in. H_2O/in. Thickness	S_v^2	S_v cm2/cm3	S_v cm^2/gm
AX1	35.4	18.4 x 10^6	4290	482
	38.1	17.1 x 10^6	4140	464
AX2	39.9	16.3 x 10^6	4040	454
	42.3	15.4 x 10^6	3920	441
AX1 Modified	32.1	20.2 x 10^6	4500	506
	32.1	20.2 x 10^6	4500	506

FIGURE 27

FLOW RATE VS DENSITY AT A PRESSURE DROP OF 0.1 INCH OF H_2O AND A THICKNESS OF 0.030 INCH FOR AX1, AX2, AND AX1 MODIFIED NICKEL FIBER PLAQUES SINTERED AT 1900°F



4.3.6 Electrical Resistivity

The electrical resistivities obtained at the indicated densities are shown in Table XIV.

TABLE XIV

DENSITY AND ELECTRICAL RESISTIVITY OF NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Density % of Theoretical	Electrical Resistivity Microhm - cm
AX1	12.3	499
	12.8	468
AX2	12.1	562
	12.2	544
AX1 Modified	12.6	504
	12.5	522

The experiment to determine the electrical resistivity perpendicular to the felting plane was moderately successful. The compressibility of low density fiber metal structures makes it difficult to contact the surfaces with enough pressure to overcome the contact resistance without densifying the plaque. Two methods were available for determining this resistivity. The first would be to produce a very thick felt and cut resistivity strips perpendicular to the felting plane. Resistivity measurements could then be made in the same manner that they were parallel to the felting plane. Since producing a plaque this thick is not practical, a 0.75 inch plaque was made using AX2 fiber. A cube of this material 0.75 inches on a side was filled with a catalyzed epoxy resin and opposite surfaces were ground parallel. This cube was placed in a hydraulic press between the contacts of a Kelvin Bridge and pressure was applied

until a constant resistance reading was obtained. The readings recorded, at 500 psi, were 400 microhm-cm parallel to the felting plane and 895 microhm-cm perpendicular to the felting plane. The increase in resistivity perpendicular to the felting plane is expected because the fibers are oriented parallel to the felting plane. Consequently, the resistive path is longer when the resistance is measured perpendicular to the felting plane.

4.3.7 Normalized Data

For convenience the surface area, density, tensile strength normalized to 10% density and electrical resistivity normalized to 15% density are shown in Table XV.

TABLE XV

SUMMARY OF DATA FOR DUPLICATE SAMPLES OF THREE GRADES OF NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	% Density	Surface Area cm^2/gm $\Delta P = 0.1"$	Electrical Resistivity Microhm-cm. At 15% Density	Tensile Strength lbs. per $in.^2$ At 10% Density
AX1	11.7	482	411	84.2
		464	400	85.6
		Ave. 473	Ave. 406	Ave. 84.9
AX2	11.2	454	454	48.5
		441	442	54.5
		Ave. 448	Ave. 448	Ave. 51.5
AX1 Modified	12.2	506	423	134
		506	435	146
		Ave. 506	Ave. 429	Ave. 140

4.3.8 Flexibility

Samples of each of the materials, 0.028 inches thick, were bent 90° over progressively smaller mandrels. As the data in Table XVI show, no damage was evident in any of the samples.

TABLE XVI

FLEXIBILITY DATA FOR NICKEL FIBER METAL PLAQUES SINTERED AT 1900±15°F

Material	Diameter of Mandrel Used	Result
AX1	1/4 "	A
	7/32"	A
	3/16"	A
	5/32"	A
	1/8 "	A
	3/32"	A
	1/16"	A
AX2	1/4 "	A
	7/32"	A
	3/16"	A
	5/32"	A
	1/8 "	A
	3/32"	A
	1/16"	A
AX1 Modified	1/4 "	A
	7/32"	A
	3/16"	A
	5/32"	A
	1/8 "	A
	3/32"	A
	1/16"	A

A* No damage to the specimen was evident upon inspection under a 15 power binocular microscope

4.4 Task D - Plaque Samples

Samples of each of the plaques evaluated in Task C were sent to the NASA Technical Director.

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6.0 APPENDIX

TABLE XVII

FREQUENCY TABULATION OF FIBER LENGTH OF AX1 NICKEL FIBER

Sample 1

Length Class Interval Microns	Class Midpoint X Microns	Frequency F	FX	FX % of Total	X^2	FX^2
13-37	25	228	5700	2.9	625	142500
38-62	50	193	9650	4.9	2500	482500
63-87	75	111	8325	4.2	5625	624375
88-112	100	135	13500	6.9	10000	1350000
113-137	125	143	17875	9.1	15625	2234375
138-162	150	74	11100	5.6	22500	1665000
163-187	175	62	12950	6.6	30625	1898750
188-212	200	74	14800	7.5	40000	2960000
213-237	225	35	7875	4.0	50625	1771875
238-262	250	28	7000	3.6	62500	1750000
263-287	275	18	4950	2.5	75625	1361250
288-312	300	35	10500	5.3	90000	3150000
313-337	325	12	3900	2.0	105625	1267500
338-362	350	15	5250	2.7	122500	1837500
363-387	375	12	4500	2.3	140625	1687500
388-412	400	16	6400	3.3	160000	2560000
413-437	425	8	3400	1.7	180625	1445000
438-462	450	9	4050	2.1	202500	1822500
463-487	475	10	4750	2.4	225625	2256250
488-512	500	11	5500	2.8	250000	2750000
513-537	525	4	2100	1.1	275625	1102500
538-562	550	5	2750	1.4	302500	1512500
563-587	575	3	1725	0.9	330625	991875
588-612	600	3	1800	0.9	360000	1800000
613-637	625	4	2500	1.3	390625	1562500
638-662	650	5	3250	1.7	422500	2112500
663-687	675	5	3375	1.7	455625	2278125
688-712	700	11	7700	3.9	490000	5390000
788-812	800	2	1600	0.8	640000	1280000
888-912	900	2	1800	0.9	810000	1620000
988-1012	1000	2	2000	1.0	1000000	2000000
1188-1212	1200	1	1200	0.6	1440000	1440000
1288-1312	1300	1	1300	0.7	1690000	1690000
1488-1512	1500	1	1500	0.8	2250000	2250000

Mean 154 Standard Deviation 158

Median 88-112

TABLE XVIII

FREQUENCY TABULATION OF FIBER LENGTH OF AX1 NICKEL FIBER

Sample 2

Length Class Interval Microns	Class Midpoint X Microns	Frequency F	FX	FX % of Total	X^2	FX^2
13-37	25	312	7800	6.0	625	195000
38-62	50	151	7550	5.8	2500	377500
63-87	75	96	7200	5.5	5625	540000
88-112	100	151	15100	11.6	10000	1510000
113-137	125	72	9000	6.9	15625	1125000
138-162	150	41	6150	4.7	22500	922500
163-187	175	22	3850	3.0	30625	673750
188-212	200	38	7600	5.8	40000	1520000
213-237	225	23	5175	4.0	50625	1164375
238-262	250	12	3000	2.3	62500	750000
263-287	275	7	1925	1.5	75625	529375
288-312	300	21	6300	4.8	90000	1890000
313-337	325	6	1950	1.5	105625	633750
338-362	350	12	4200	3.2	122500	1470000
363-387	375	6	2250	1.7	140625	843750
388-412	400	8	3200	2.5	160000	1280000
413-437	425	7	2975	2.3	180625	1264375
438-462	450	4	1800	1.4	202500	810000
463-487	475	4	1900	1.4	225625	902500
487-512	500	12	600	0.5	250000	3000000
513-537	525	0	0	0	275625	0
538-562	550	4	2200	1.7	302500	1210000
563-587	575	1	575	0.4	330625	330625
588-612	600	1	600	0.5	360000	360000
613-637	625	2	1250	1.0	390625	781250
638-662	650	10	6500	5.0	422500	4225000
663-687	675	2	1350	1.0	455625	911250
688-712	700	6	4200	3.2	490000	2940000
788-812	750	2	1500	1.1	562500	1125000
888-912	800	3	2400	1.8	640000	1920000
988-1012	900	2	1800	1.4	810000	1620000
1188-1212	1000	5	5000	3.8	1000000	5000000
1188-1212	1200	3	3600	2.8	1440000	4320000

Mean 125 Standard Deviation 169

Median 88-112

TABLE XIX

FREQUENCY TABULATION OF FIBER LENGTH OF AX2 NICKEL FIBER

Sample 1

Length Class Interval Microns	Class Midpoint X Microns	Frequency F	FX	FX % of Total	X^2	FX^2
13-37	25	119	2975	4.3	625	74375
38-62	50	56	2800	4.0	2500	140000
63-87	75	41	3075	4.4	5625	230625
88-112	100	49	4900	7.0	10000	490000
113-137	125	38	4750	6.8	15625	593750
138-162	150	18	2700	3.9	22500	405000
163-187	175	16	2800	4.0	30625	490000
188-212	200	30	6000	8.6	40000	1200000
213-237	225	8	1800	2.6	50625	405000
238-262	250	12	3000	4.3	62500	750000
263-287	275	1	275	0.4	75625	75625
288-312	300	9	2700	3.9	90000	810000
313-337	325	8	2600	3.7	105625	845000
338-362	350	8	2800	4.0	122500	980000
363-387	375	5	1875	2.7	140625	703125
388-412	400	4	1600	2.3	160000	640000
413-437	425	3	1275	1.8	180625	541875
438-462	450	5	2250	3.2	202500	1012500
463-487	475	2	950	1.3	225625	445250
488-512	500	1	500	0.7	250000	250000
513-537	525	2	1050	1.5	275625	551250
538-562	550	2	1100	1.6	302500	605000
563-587	575	1	575	0.8	330625	330625
588-612	600	1	600	0.9	360000	360000
613-637	625	4	2500	3.6	390625	1562500
638-662	650	1	650	0.9	422500	422500
663-687	675	4	2700	3.9	455625	1822500
688-712	700	4	2800	4.0	490000	1960000
737-762	750	1	750	1.1	562500	562500
788-812	800	1	800	1.1	640000	640000
813-837	825	1	825	1.2	680625	680625
838-862	850	1	850	1.2	722500	722500
913-937	925	1	925	1.3	855625	855625
988-1012	1000	1	1000	1.4	1000000	1000000
1013-1037	1025	1	1025	1.5	1050625	1050625

Mean 152 Standard Deviation 172

Median 88-112

TABLE XX

FREQUENCY TABULATION OF FIBER LENGTH OF AX2 NICKEL FIBER

Sample 2

Length Class Interval Microns	Class Midpoint X Microns	Frequency F	FX	FX % of Total	X^2	FX^2
13-37	25	66	1650	2.0	625	41250
38-62	50	85	4250	5.1	2500	212500
63-87	75	56	4200	5.1	5625	315000
88-112	100	65	6500	7.8	10000	650000
113-137	125	46	5750	6.9	15625	718750
138-162	150	29	4350	5.2	22500	652500
163-187	175	8	1400	1.7	30625	245000
188-212	200	28	5600	6.7	40000	1120000
213-237	225	13	2925	3.5	50625	658125
238-262	250	12	3000	3.6	62500	750000
263-287	275	4	1100	1.3	75625	302500
288-312	300	15	4500	5.4	90000	1350000
313-337	325	4	1300	1.6	105625	422500
338-362	350	5	1750	2.1	122500	612500
363-387	375	5	1875	2.3	140625	703125
388-412	400	8	3200	3.9	160000	1280000
413-437	425	5	2125	2.6	180625	903125
438-462	450	6	2700	3.2	202500	1215000
463-487	475	2	950	1.1	225625	451250
488-512	500	10	5000	6.0	250000	2500000
513-537	525	2	1050	1.3	275625	551250
538-562	550	2	1100	1.3	302500	605000
563-587	575	2	1150	1.4	330625	661250
588-612	600	1	600	0.7	360000	360000
613-637	625	0	0	0	390625	0
638-662	650	1	650	0.8	422500	422500
663-687	675	0	0	0	455625	0
688-712	700	6	4200	5.1	490000	2940000
738-762	750	2	1500	1.8	562500	1125000
788-812	800	2	1600	1.9	640000	1280000
888-912	900	1	900	1.1	810000	810000
988-1012	1000	2	2000	2.4	1000000	2000000
1238-1262	1250	1	1250	1.5	1562500	1562500
1338-1362	1350	1	1350	1.6	1822500	1822500
1488-1512	1500	1	1500	1.8	2250000	2250000

Mean 167 Standard Deviation 183

Median 88-112

TABLE XXI

FREQUENCY TABULATION OF FIBER DIAMETER OF AX2 NICKEL FIBER

Sample 1

Class Midpoint X Microns	Class Midpoint X	Frequency F	FX	FX % of Total	X^2	FX^2
1-3.7	2.5	47	117.5	2.2	6	282
3.8-6.2	5.0	44	220.0	4.1	25	1100
6.3-8.7	7.5	43	322.5	6.0	56	2408
8.8-11.2	10.0	34	340.0	6.4	100	3400
11.3-13.7	12.5	35	437.5	8.2	156	5460
13.8-16.2	15.0	25	375.0	7.0	225	5625
16.3-18.7	17.5	20	350.0	6.5	306	6120
18.8-21.2	20.0	16	320.0	6.0	400	6400
21.3-23.7	22.5	9	202.5	3.8	506	4554
23.8-26.2	25.0	13	325.0	6.1	625	8125
26.3-27.5	27.5	6	165.0	3.1	756	4536
28.8-31.2	30.0	14	420.0	7.8	900	12600
31.3-33.7	32.5	4	130.0	2.4	1056	4224
33.8-36.2	35.0	4	140.0	2.6	1225	4900
36.3-38.7	37.5	8	300.0	5.6	1406	11248
38.8-41.2	40.0	4	160.0	3.0	1600	6400
41.3-43.7	42.5	6	255.0	4.8	1806	10836
43.8-46.2	45.0	4	180.0	3.4	2025	8100
46.3-48.7	47.5	3	142.5	2.7	2256	6768
48.8-50	50.0	9	450.0	8.4	2500	22500

Mean 15.4 Standard Deviation 12.4
Median 10.0-12.5

TABLE XXII

FREQUENCY TABULATION OF FIBER DIAMETER OF AX2 NICKEL FIBER

Sample 2

Class Midpoint X Microns	Class Midpoint X	Frequency F	FX	FX % of Total	X^2	FX^2
1- 3.7	2.5	69	172.5	2.8	6	431
3.8- 6.2	5.0	48	240.0	3.9	25	1200
6.3- 8.7	7.5	54	405.0	6.6	56	3024
8.8-11.2	10.0	48	480.0	7.9	100	4800
11.3-13.7	12.5	29	362.5	5.9	156	4524
13.8-16.2	15.0	31	465.0	7.6	225	6975
16.3-18.7	17.5	30	525.0	8.6	306	9180
18.8-21.2	20.0	23	460.0	7.5	400	9200
21.3-23.7	22.5	16	360.0	5.9	506	8096
23.8-26.2	25.0	28	700.0	11.5	625	17500
26.3-27.5	27.5	9	247.5	4.1	756	6804
28.8-31.2	30.0	15	450.0	7.4	900	13500
31.3-33.7	32.5	3	97.5	1.6	1056	3168
33.8-36.2	35.0	8	280.0	4.6	1225	9800
36.3-38.7	37.5	6	225.0	3.7	1406	8436
38.8-41.2	40.0	3	120.0	2.0	1600	4800
41.3-43.7	42.5	3	127.5	2.1	1806	5418
43.8-46.2	45.0	3	135.0	2.2	2025	6075
46.3-48.7	47.5	1	47.5	0.8	2256	2256
48.8-50	50.0	4	200.0	3.3	2500	10000

Mean 14.1 Standard Deviation 11.4
Median 10.0-12.5

TABLE XXIII

FREQUENCY TABULATION OF FIBER DIAMETER OF AX1 NICKEL FIBER

Diameter Class Interval Microns	Class Midpoint X Microns	Frequency F	FX	FX % of Total
1- 3.7	2.5	164	410.0	5.8
3.8- 6.2	5.0	99	495.0	7.0
6.3- 8.7	7.5	74	555.0	7.8
8.8-11.2	10.0	80	800.0	11.3
11.3-13.7	12.5	51	637.5	9.0
13.8-16.2	15.0	23	345.0	4.9
16.3-18.7	17.5	26	455.0	6.4
18.8-21.2	20.0	17	340.0	4.8
21.3-23.7	22.5	18	405.0	5.7
23.8-26.2	25.0	30	750.0	10.6
26.3-28.7	27.5	3	82.5	1.2
28.8-31.2	30.0	12	360.0	5.1
31.3-33.7	32.5	4	130.0	1.8
33.8-36.2	35.0	7	245.0	3.4
36.3-38.7	37.5	7	262.5	3.7
38.8-41.2	40.0	1	40.0	0.6
41.3-43.7	42.5	1	42.5	0.6
43.8-46.2	45.0	1	45.0	0.6
46.3-48.7	47.5	2	95.0	1.3
48.8-50	50.0	12	600.0	8.4

Mean 11.2
Median 5.0-7.5

TABLE XXIV

FREQUENCY TABULATION OF FIBER LENGTH
OF AX1 MODIFIED NICKEL FIBER

Class Midpoint X Microns	Frequency F	FX	FX % of Total
50	53	2650	3.7
100	78	7800	10.8
150	46	6900	9.6
200	48	9600	13.4
250	26	6500	9.0
300	22	6600	9.2
350	18	6300	8.8
400	7	2800	3.9
450	10	4500	6.3
500	6	3000	4.2
550	3	1650	2.3
600	2	1200	1.7
650	6	3900	5.4
700	1	700	1.0
750	4	3000	4.2
800			
850			
900			
950	1	950	1.3
1000			
1050			
1100			
1150			
1200	1	1200	1.7
1250			
1300	1	1300	1.8
1350	1	1350	1.9
	Total 334	71,900	

Mean 215 microns
Median 125-175 microns

TABLE XXV

FREQUENCY TABULATION OF FIBER DIAMETER
OF AX1 MODIFIED NICKEL FIBER

Class Midpoint X Microns	Frequency F	FX	FX % of Total
2.5	21	52.5	1.4
5.0	45	22.5	6.2
7.5	50	375	10.3
10.0	26	260	7.1
12.5	23	287.5	7.9
15.0	22	330	9.0
17.5	16	280	7.7
20.0	12	240	6.6
22.5	11	247.5	6.8
25.0	5	125	3.4
27.5	6	165	4.5
30.0	9	270	7.4
32.5	7	227.5	6.2
35.0	3	105	2.9
37.5	3	112.5	3.1
40.0	1	40	1.1
42.5	4	170	4.7
45.0	3	135	3.7
	Total 267	3647.5	

Mean 13.7 microns
Median 8.75-11.25 microns

TABLE XXVI

FREQUENCY TABULATION OF FIBER LENGTH OF DX NICKEL FIBER

Class Midpoint X Microns	Frequency F	FX	FX % of Total
50	11	550	0.4
100	15	1500	1.0
150	21	3150	2.1
200	16	3200	2.1
250	12	3000	2.0
300	12	3600	2.3
350	9	3150	2.1
400	11	4400	2.9
450	12	5400	3.5
500	11	5500	3.6
550	12	6600	4.3
600	6	3600	2.3
650	15	9750	6.4
700	7	4900	3.2
750	10	7500	4.9
800	9	7200	4.7
850	8	6800	4.4
900	26	23400	15.3
950	29	27550	17.9
1000	17	17000	11.1
1050	3	3150	2.1
1100	1	1100	0.7
1150			
1200			
1250			
1300	1	1300	0.8
	Total 284	153,300	

Mean 540 microns
Median 575 microns

TABLE XXVII

FREQUENCY TABULATION OF FIBER DIAMETER OF DX NICKEL FIBER

Class Midpoint X Microns	Frequency F	FX	FX % of Total
2.5	1	2.5	0
5	1	5	0
7.5			
10	1	10	0.1
12.5	1	12.5	0.1
15	3	45	0.3
17.5	2	35	0.2
20	1	20	0.1
22.5	1	22.5	0.1
25	4	100	0.6
27.5	2	55	0.3
30	5	150	0.9
32.5	4	130	0.8
35	8	280	1.7
37.5	8	300	1.8
40	5	200	1.2
42.5	9	382.5	2.3
45	8	360	2.2
47.5	7	332.5	2.0
50	14	700	4.2
52.5	15	787.5	4.7
55	6	330	2.0
57.5	9	517.5	3.1
60	6	360	2.2
62.5	13	812.5	4.9
65	16	1040	6.2
67.5	17	1147.5	6.9
70	9	630	3.8
72.5	11	797.5	4.8
75	12	900	5.4
77.5	8	620	3.7
80	15	1200	7.2
82.5	5	412.5	2.5
85	11	935	5.6
87.5	9	787.5	4.7

TABLE XXVII
(Continued)

Class Midpoint X Microns	Frequency F	FX	FX % of Total
90	5	450	2.7
92.5	5	462.5	2.8
95	4	380	2.3
97.5	1	97.5	0.6
100	2	200	1.2
102.5			
105			
107.5	2	215	1.3
110	1	110	0.7
112.5	1	112.5	0.7
115			
117.5			
120	2	240	1.4
Total	270	16,687.5	

Mean 61.8 microns
Median 63.75 - 66.25 microns

TABLE XXVIII

FREQUENCY TABULATION OF FIBER THICKNESS OF DX NICKEL FIBER

Class Midpoint X Microns	Frequency F	FX	FX % of Total
0.5	4	2	1.0
1	21	21	10.2
1.5	22	33	16.0
2	12	24	11.7
2.5	8	20	9.7
3	7	21	10.2
3.5	1	3.5	1.7
4	3	12	5.8
4.5	2	9	4.4
5	1	5	2.4
5.5	2	11	5.3
6			
6.5			
7			
7.5	2	15	7.3
8			
8.5	1	8.5	4.1
9			
9.5	1	9.5	4.6
10			
10.5			
11			
11.5	1	11.5	5.6
	Total 88	206	

Mean 2.34 microns
Median 1.25 - 1.75 microns

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