NASA CR-54414

PREPARATION and EVALUATION of FIBER METAL NICKEL BATTERY PLAQUES

THIRD QUARTERLY PROGRESS REPORT

February 1, 1965 to April 30, 1965

by

J. L. Bidler and J. I. Fisher

prepared for

NATIONAL AERONAUTICS and SPACE ADMINISTRATION

CONTRACT NAS 3-6006

HUYCK METALS

A DEPARTMENT OF HUYCK CORPORATION

NASA CR-54414

Huyck 805 (F3-25)

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I. SUMMARY

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An experiment to more clearly define the effect of fiber size upon internal surface area and pore size of nickel fiber metal plaques has been completed by separating the finest fibers from AX1 nickel fiber. Two techniques were used for separation. The first, a laboratory technique, separates according to fiber length. The fine, short fibers obtained by this method resulted in a plaque with an increased internal surface area and smaller pores at the expense of porosity. The second method, a production technique, separates according to fiber diameter. Plaques made from this material have increased internal surface area with no significant change in pore size or porosity.

Sintering parameters of 1900±15°F for 20 minutes in dry hydrogen have been established for nickel fiber metal battery plaques. This processing results in maximum internal surface area and tensile strength and minimum electrical resistivity without significantly increasing the density.

Air permeability determinations of internal surface area have been supplemented by a modified B.E.T. Krypton technique. A measurement of AX1 nickel fiber sintered at 1900±15°F for 20 minutes indicated an internal surface area of 590 cm^2/gm by the B.E.T. method as compared with 370 cm^2/gm for the air permeability calculation.

Plaques of AX1 and AX2 nickel fiber metal have been produced at a thickness of 0.028±.001 and sintered for 20 minutes in dry hydrogen. Plaque classification tests have been initiated and preliminary results compare favorably with the results obtained from the sintering study.

Author ↑

II. INTRODUCTION

This program, for the preparation and evaluation of fiber metal nickel battery plaques is intended to define the raw material characteristics of nickel fiber metal and the processing parameters required to produce battery plaques. The resulting plaques are to be classified as to internal surface area, tensile strength, electrical resistivity, porosity and flexibility.

The program outline defines four major work areas, Tasks A-D, which are summarized below along with the current status of each Task and the work performed during this reporting period.

Task A - Raw Material Characterization

Each raw material is to be characterized as to particle size, shape, and particle size distribution. The primary raw materials, two grades of nickel fiber differing in apparent diameter, have been characterized in previous reports. This report includes the characterization of that portion of one of the fiber grades that contains the smallest diameter fibers.

Task B - Sintering Study

This Task is directed toward determining the highest sintering temperature that will result in an acceptable amount of shrinkage when the sintering time is held constant at 20 minutes. The optimum sintering temperature has been established for the raw materials employed by measuring pore size, pore size distribution, internal surface area, electrical resistivity, and tensile strength as a function of sintering temperature. This report contains the results obtained from the small diameter fibers described above, as well as the results obtained previously for the two primary raw materials.

Task C - Plaque Classification

Plaques of the raw material described above are to be processed as the results of T ask B dictate and the plaque classification test performed. The plaques produced to date and the classification results obtained are included in this report.

Task D - Plaque Samples

It is desired that samples of the plaques classified to Task C be furnished to the NASA Project Manager when they are available. No samples have been furnished to date.

III. EXPERIMENTAL APPARATUS and PROCEDURES

Task A - Raw Material Classification

The techniques and apparatus used to classify the raw materials employed in this program have been described in the First and Second Quarterly Progress Reports. These techniques have been used to determine the length and apparent diameter of two modifications of AX1 nickel fiber.

Task B - Sintering Study

The apparatus used to conduct the sintering study was described in the First Quarterly Progress Report. The sintering time has been held constant at 20 minutes for all tests made and the atmosphere has been dry hydrogen.

Plaques of two modifications of AX1 nickel fiber have been sintered at 1600°F, 1800°F, 2000°F, and 2150°F to determine the effect of sintering temperature upon density and to provide samples for pore size, pore size distribution, electrical resistivity, and internal surface area measurements.

Task C - Plaque Classification

Preliminary plaque classification tests have been made in conjunction with the sintering study, Task B, to determine the effect of sintering temperature upon plaque characteristics. The techniques for determining pore size, pore size distribution, density, internal surface area, tensile strength and electrical resistivity were described in the First and Second Quarterly Progress Reports.

A sample of AX1 nickel fiber metal was submitted to Nuclear Materials and Equipment Corporation, Apollo, Pennsylvania, for a surface area determination using a modified B.E.T. technique developed by A. C. Nyce, J. E. Scott and B. L. Vondra, Jr.[1]

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

IV. EXPERIMENTAL RESULTS and DISCUSSION

Task A - Raw Material Classification

An evaluation of the effect of fiber size upon the internal surface area, pore size, and pore size uniformity of nickel fiber metal battery plaques was initiated by separating AX1 nickel fiber into size ranges.

Two methods are available for separating fiber metal into size ranges. The first, a laboratory technique, separates according to length. This method was used to obtain a rapid determination of the degree of improvement that could be expected from using small diameter fibers. The finest fraction of AX1 nickel fiber separated by this method was obtained and designated AX13. The second method for fiber separation separates according to fiber diameter. The finest fraction of AX1 nickel fiber obtained by this method was designated AX1 Modified.

The fraction of fibers obtained by the first method, AX13, contains the smallest diameter and the shortest fibers of AX1 nickel fiber. The fraction of fibers obtained by the second method, AX1 Modified, contains the smallest diameter fibers of AX1 nickel fiber regardless of length.

A summary of the frequency tabulation data of the length and apparent diameter of AX1, AX2, AX13, and AX1 Modified is shown in Table I.

TABLE I

Summary of Frequency Tabulation Data of Length and Apparent Diameter of AX1, AX2, AX13, and AX1 Modified Nickel Fiber

Material	Apparent Diameter - Microns				Length - Microns			
	Mean	Median	Standard Deviation	Range	Mean	Median	Standard Deviation	Range
AX1	11.2	5-7.5	10.3	1-50	139	88-112	163	1-1500
AX2	14.9	10-12.5	11.5	1-50	159	88-112	178	1-1500
AX13	14	9-11	9.3	1-40	121	63-88	111	1-900
AX1 Modified	13.7	8.75-11.25	10	1-45	215	125-175	188	1-1300

The mean and median apparent diameter of AX13 and AX1 Modified are shown to be larger than the mean and median apparent diameter of AX1 nickel 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 all three materials, AX1, AX13, and AX1 Modified, should be expected to be similar.

The significance of the difference in fiber length between AX13 and AX1 Modified is discussed under Task B.

These data of the description of AX13 and AX1 Modified nickel fiber complete Task A of the contract.

Task B - Sintering Study

1. Porosity vs Sintering Temperature

Plaques of AX13 and AX1 Modified nickel fiber metal were sintered at 1600°F, 1800°F, 2000°F, and 2150°F for 20 minutes in dry hydrogen. The effect of sintering temperature upon density is shown in Table II.

TABLE II

Density versus Sintering Temperature for AX13 and AX1 Modified Nickel Fiber Metal Plaques

Material	Sintering Temperature °F	Density % of Theoretical
AX13	1600	15.4
	1800	16.8
	2000	18.9
	2150	21.0
AX1 Modified	1600	9.0
	1800	9.3
	2000	9.8
	2150	10.5

It is apparent that the retention of long fibers in the AX1 Modified material significantly decreases the density. For the same weight of nickel, a battery plaque that is 10% dense can theoretically hold twice as much active material as a battery plaque 18% dense. This factor would be important in applications where weight must be minimized.

2. Pore Size versus Sintering Temperature

The median pore size and the per cent of the porosity due to pores 10 to 50 microns in diameter are shown in Table III for AX13 and AX1 Modified nickel fiber metal plaques, along with the data previously obtained for AX1 and AX2 nickel fiber metal battery plaques.

TABLE III

Median Pore Size and Volume of Porosity Due to Pores 10 to 50 Microns in Diameter for AX1, AX2, AX13, and AX1 Modified Nickel Fiber Metal Plaques as a Function of Sintering Temperature

Material	Sintering Temperature	Median Pore Size, Microns	Volume of Pores 10-50 Microns, %
AX1	1600	38.9	49.5
	1800	37.2	
	2000	47.3	
	2150	47.3	39.3
AX2	1600	47.9	36.9
	1800	51.5	
	2000	43.8	
	2150	52.2	41.6
AX13	1600	20	84
	1800	20	88
	2000	21	88
	2150	21	85
AX1 Modified	1600	54	42
	1800	54	41
	2000	54	42
	2150	57	41

These pore size measurements for AX13 and AX1 Modified nickel plaques were made at the densities shown in Table II. The short fibers, AX13, result in a plaque with a smaller pore size. It is apparent that, by controlling the fiber size and the density, nickel fiber metal battery plaques can be produced with a broad range of median pore sizes.

Increasing sintering temperature in general, tends to slightly increase the median pore size, but has little effect upon the per cent of the porosity due to pores 10 to 50 microns in diameter.

3. Surface Area and Electrical Resistivity versus Sintering Temperature

Internal surface area data, obtained from air permeability measurements, and the electrical resistivity of AX1, AX2, AX13, and AX1 Modified nickel fiber metal battery plaques are shown in Table IV as a function of sintering temperature.

TABLE IV

Internal Surface Area and Electrical Resistivity of AX1, AX2, AX13, and AX1 Modified Nickel Fiber Metal Battery Plaques as a Function of Sintering Temperature

Material	Sintering Temperature °F	Surface Area cm^2/gm	Electrical Resistivity Microhm-cm at 15% Dense
AX1	1600	449	1090
	1800	419	443
	2000	329	355
	2150	302	324
AX2	1600	369	778
	1800	343	433
	2000	315	354
	2150	296	315
AX13	1600	755	425
	1800	647	292
	2000	550	226
	2150	464	207
AX1 Modified	1600	645	622
	1800	546	453
	2000	492	388
	2150	448	321

The electrical resistivity data have been normalized to 15% density because the varying sintering temperatures result in varying densities. It has been shown[2] 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 1, a plot of electrical resistivity versus density for AX2 nickel fiber metal, indicate that at low densities the resistivity is not proportional to density. Normalizing the data to 15% density in Table IV does not result in accurate data, but does give an indication of the difference in electrical resistivity between the different fiber grades. The actual resistivities and the densities at which they were obtained are included in the appendix.

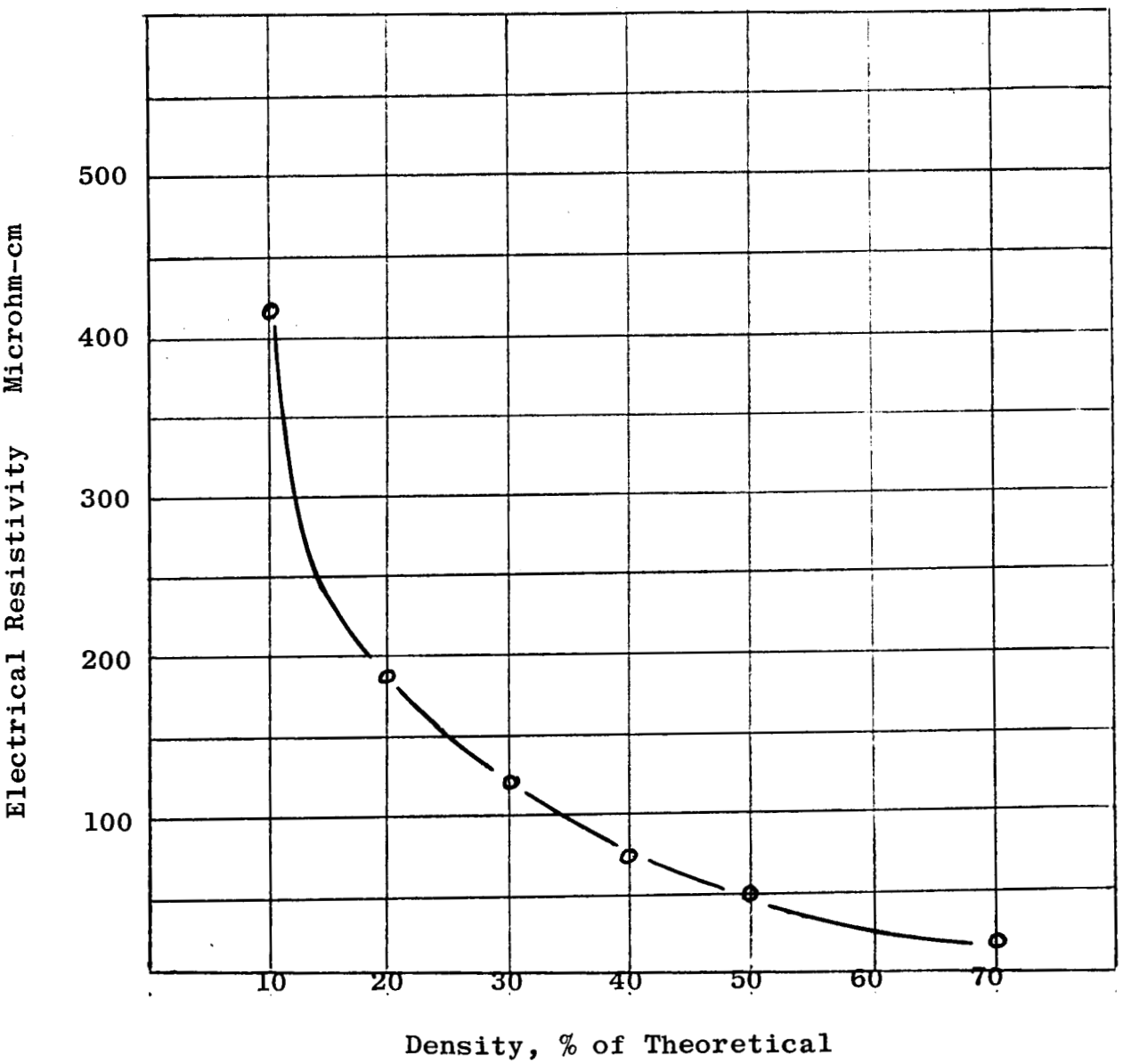
The internal surface area of AX13 and AX1 Modified nickel fiber metal plaques is greater than that of AX1 and AX2 nickel fiber metal plaques over the sintering range studied. The electrical resistivity of AX13 nickel fiber metal is lower than the other three materials at all sintering temperatures studied. The increased surface area and decreased electrical resistivity at the expense of porosity for AX13 nickel fiber metal is similar to that experienced with some grades of nickel powder plaques[3].

The electrical resistivity of AX1 Modified nickel fiber metal is the same as that of AX1 nickel fiber metal at sintering temperatures of 1800°F and greater. The combination of high porosity and increased surface area in conjunction with process capabilities indicate that the AX1 Modified nickel fiber metal should be included in the program and plaque classification test be made at a sintering temperature of 1900°F.

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. 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.

Figure 1

Electrical Resistivity versus Density
of AX2 Nickel Fiber Metal



The data accumulated to date have been used to establish the optimum sintering parameters of 1900±15°F for 20 minutes in dry hydrogen for AX1, AX2 and AX1 Modified nickel fiber metal battery plaques. This determination completes Task B of the contract.

Task C - Plaque Classification

Plaques of AX1 and AX2 nickel fiber metal have been made at a thickness of 0.028±.001 inches and sintered at 1900°F for 20 minutes.

The classification tests performed to date are density determinations and electrical resistivity measurements. These data are shown in Table V.

TABLE V

Density and Electrical Resistivity of AX1 and AX2 Nickel Fiber Metal Battery Plaques Sintered at 1900±15°F for 20 Minutes

Material	Density % of Theoretical	Electrical Resistivity Microhm-cm
AX1	12.5	499 468 Ave. 484
AX2	12.0	562 544 Ave. 553

Task D - Plaque Samples

Samples of plaques classified in Task C will be furnished to the NASA Project Director.

V. FUTURE WORK

Work during the next reporting period will be directed toward:

1. Obtaining plaques of AX1 Modified nickel fiber metal for classification testing.

2. Continued classification testing of AX1 and AX2 nickel fiber metal plaques.

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VII. APPENDIX

UNNORMALIZED RESISTIVITIES and DENSITIES of DATA PRESENTED in TABLE IV.

Material	Sintering Temperature °F	Density % of Theoretical	Resistivity Microhm-cm
AX1	1600	11.3	1450
AX1	1800	12.3	541
AX1	2000	12.4	423
AX1	2150	17.5	324
AX2	1600	11.4	1022
AX2	1800	11.2	578
AX2	2000	13.6	392
AX2	2150	15.7	302
AX13	1600	15.4	414
AX13	1800	16.8	292
AX13	2000	18.9	226
AX13	2150	21.0	207
AX1 Modified	1600	8.7	1070
AX1 Modified	1800	9.1	746
AX1 Modified	2000	9.5	611
AX1 Modified	2150	11.0	438

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