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3,689,320

**METHOD FOR MAKING A BATTERY PLATE
USING CELLULOSIC MATERIAL**

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Int. Cl. H01m 35/18

U.S. Cl. 136—75

7 Claims

ABSTRACT OF THE DISCLOSURE

Porous metal articles having good strength and conductivity are produced by impregnating a natural cellulosic material such as cotton cloth with a metal salt solution, drying the impregnated cloth, and heating the dried cloth in a reducing atmosphere to an elevated temperature to destroy the cellulosic material, convert the metal salt to metal and sinter the metal to a coherent body.

This application is a continuation of 751,726 filed Aug. 12, 1968, now abandoned.

The production of metal bodies in the form of knitted, woven or felted bodies such as mesh is desirable for many purposes including filters, catalyst supports, fuel cell electrodes, battery plates, etc. Porous sintered bodies can be produced from metal powders and mesh can be produced by weaving fine wire but such processes have been attended by commercial disadvantages, including high cost, insufficient strength and conductivity, limitations as to size and shape and other difficulties.

The present invention is based upon the discovery of a relatively quick and inexpensive procedure adaptable to high production of porous metal bodies having a knitted, woven or felted configuration.

It is an object of the present invention to provide a process for producing porous metal bodies having a knitted, woven or felted configuration which involves only a limited number of operations.

It is a further object of the present invention to provide a porous metal battery plaque having improved strength and conductivity.

Other objects and advantages of the invention will become apparent from the following description.

The invention comprises impregnating a natural cellulosic fibrous material or thread with a solution of one or more thermally decomposable metal salts, drying the impregnated material or thread, and by a single heating in a reducing atmosphere destroying the cellulosic material or thread, decomposing the salt or salts to metal and sintering the resultant metal to a coherent body.

The invention is applicable to the production of porous sheets or threads of many different metals, but particularly to nickel, cobalt, iron and copper and alloys of these metals. Other suitable metals are tungsten, molybdenum, gold, silver, and metals of the platinum group.

The metal salt must decompose completely and must not form any stable oxide, that is to say, one which is not reduced in the sintering atmosphere used. Suitable salts of nickel, iron, cobalt and copper are halides, for example, nickel chloride. Nitrates or complex salts of these metals may also be used. To produce an alloy product, salts of different metals are mixed together in solution. Generally, aqueous impregnating solutions are employed, so the im-

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pregnating salts should have substantial solubility in water.

The fibrous material or thread must have adequate absorptive power for the impregnating salt solution, and it is essential for this reason that it must be a natural cellulosic material. Equivalent results are not obtained with synthetic cellulosic or polymeric fibers. Cotton and paper are particularly suitable, but jute, linen, ramie and sisal may also be used.

The invention is particularly applicable to the production of very thin sheets of open-mesh metal, for which purpose the starting material may be any natural cellulosic fibrous material that is in the form of sheet, for example, a fabric, which may be woven, knitted or non-woven, or blotting paper; or again the natural cellulosic fibrous material may be cotton wool or paper pulp compressed into a sheet. We prefer to use a woven or knitted fabric, and then the diameter of the yarn and the aperture size of the fabric determine the mesh size of the final metal sheet.

The invention is also useful in the production of metal thread. In addition it may be used in the production of other shaped porous bodies, for example, a catalyst support, the cellulosic material then advantageously being cotton, wool or paper pulp compressed into the shape of the body. It will be understood, of course, that the fibrous body must be of such thickness and shape that the cellulosic material is completely removed by the heating in the reducing atmosphere.

It is an essential feature of the invention that the heating to decompose the salt and sinter the metal is effected in a reducing atmosphere and it is surprising that the cellulosic material is completely removed during this heating. It is important that the carbon be removed, otherwise one does not achieve adequate strength in the porous metal sheet or thread, nor does one obtain the optimum conductivity. Heating may be conducted at a rapid rate, e.g., 800° C. per hour, with heating rates of 5° C. to about 35° C. per minute being satisfactory. The permissible rapid heating rate in a reducing atmosphere makes the process simple and adaptable to high production rates of porous metal in knitted, woven or felted configuration. Heating in an oxidising atmosphere to destroy the cellulosic material followed by heating in a reducing atmosphere to reduce the metal oxide formed leads to inferior products in terms of strength, conductivity, etc. Moreover, the overall time is much less when the metal is directly formed by decomposition of the salt in a reducing atmosphere. In the invention, a sintering temperature of at least 900° C. is desirable, but the period during which the material is exposed to a temperature of 900° C. or more need not, and preferably does not, exceed 60 minutes. The product may, if desired, be resintered in order to increase its strength. During the sintering, sinter bonds are formed at the points where the metal fibers intersect and are in contact.

The impregnation step may also be short, immersion for a few minutes, e.g., 5 or 10 minutes up to one or two hours, in a solution of the salt being enough, especially when the metal salt employed is soluble to the extent of about 50 grams (gm.) or more in 100 cc. of water.

Some cellulosic materials available as the starting materials are slightly greasy and, advantageously, these are first washed in water containing a surfactant.

When a nickel sheet or other nickel product is being made, it is preferred to use nickel chloride as the salt. Nickel sulfate, for example, does not yield so strong a product. The strength of the impregnating solution is not critical but we prefer a solution containing 120 gm. of NiCl₂·6H₂O per 100 cc. of water, which will be referred to as the 120 gm. nickel chloride solution.

The invention leads to products of high porosity, remarkable strength for such porous products and low electrical resistivity. The strengths are reported herein-

after as the force in grams required to break a strand or a strip 1 centimeter (cm.) wide when subjected to a straight tensile test. The porosities of the products are reported hereinafter as a percentage derived from a determination of the density of the product in a mercury balance, the balance being sensitive to all pores below 300 microns.

Plaques made according to the invention have good bend strength and low electrical resistivity. These factors contribute good handling characteristics to the plaques and enable the production of cells having low internal resistance. Their porosity depends on the nature of the starting material and the processing conditions, but even when it is not so high as that of plaques of sintered powder, their electrical properties when converted into battery plates are better.

Naturally, the electrical resistance of a plaque according to the invention is less than that of one of the sheets from which it is made but, nevertheless, is essentially related to it, so the suitability of sheets for the production of plaques can be determined by ascertaining their resistivity.

In making the plaques, it is preferred to bond the assembly of sheets together with both compacting and sintering. A plaque made by compactant alone, or by sintering an uncompacted assembly of sheets, may tend to delaminate in use, and battery plates made from such plaques may need to be specially supported to counteract this tendency.

A preferred aspect of the invention is the production of plaques for alkaline batteries wherein porous metal sheets of nickel, iron, copper or cobalt produced in accordance with the invention are placed together in face to face relation and bonded together by compacting or sintering or both. It is found that such plaques, after suitable impregnation with active material, have surprisingly advantageous properties. Plaques of nickel are particularly useful in the production of plates for nickel-cadmium and nickel-iron batteries.

In making sheets for the production of battery plaques, the thermal treatment may comprise heating under oxidizing conditions to destroy the organic material, followed by a heating under reducing conditions, but better products are obtained when the thermal treatment comprises a single heating of metal salt impregnated natural cellulosic material in a reducing atmosphere to decompose the salt directly to metal, sintering the metal, and destroying the organic material at the same time.

When the metal salt is directly decomposed to metal, the organic material is preferably cotton or other natural cellulosic fibrous material, but when the metal is first oxidized and then reduced in the thermal treatment, the organic material is preferably a synthetic cellulosic material such as rayon, although much longer soaking times in the impregnating salt solution are then required.

In the production of battery plates for an alkaline accumulator, active mass may be introduced into nickel plaques by impregnating them with solutions of nickel salts and subsequently converting these to hydroxide by immersion with or without electrolysis in a solution of caustic alkali. Alternatively, the plaques may be filled initially with nickel hydroxide active mass, either by pressing the material into the plaques or treating the plaques with a slurry of nickel hydroxide which is taken up by the pores of the plaques. It will be understood that cadmium hydroxide may be put into the plaque to form a negative plate.

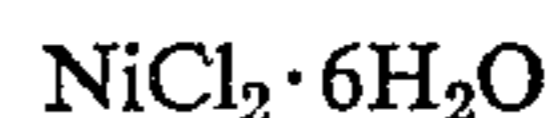
It is often desired to produce plaques with varying porosity throughout their thickness, and this desired result can readily be obtained by sintering together sheets of different porosity produced as aforescribed.

Some examples will now be given. In all of them, the heating step in the production of the sheets was effected in a tube furnace ten feet long with an internal diameter of rectangular section 6.5" x 3" through which the im-

pregnated and dried material was carried on a belt. In the middle of the furnace, there was a hot zone 12 inches long, the temperature in which was thermostatically controlled. An atmosphere of cracked ammonia (approximately 75% hydrogen, balance nitrogen) flowing at the rate of 100 cubic feet per hour was maintained in the furnace. The flow rate was sufficient to provide good sweeping of decomposition products from the furnace and to prevent internal explosions.

EXAMPLE 1

Knitted cotton fabric (stockinette) was soaked in a solution of nickel chloride consisting of 60 gm.



in 100 cc. water. The impregnated fabric was then dried and passed continuously at a speed of one inch per minute through a sintering furnace in which the hot zone was held at a temperature of 1000° C., the residence time in this zone being 12 minutes. An atmosphere of cracked ammonia was maintained in the furnace. The product was a strong and flexible porous sheet of substantially pure nickel. The diameter of the cotton thread was from 0.01 to 0.02 inch and the aperture size of the original knitted fabric from 0.06 to 0.09 inch. In the final product, which resembled the original fabric in configuration, the corresponding figures were 0.002 to 0.005 inch and 0.03 to 0.05 inch, respectively. The product had a porosity of 95.1%, a breaking strength of 170 gm. and a resistivity of 449 microhm-cm.

Seven pieces of the sintered material were packed face to face between two ceramic plates and resintered under the same conditions to give a strong plaque approximately 3" x 2.25" x 0.059" with a porosity of 95.4% and a resistivity of 1,630 microhm-cm.

EXAMPLE 2

A woven cotton cloth was impregnated in the same way as in Example 1 and sintered at 1.25 inches per minute at 1050° C., residence time 9.7 minutes. The diameter of the cotton thread was from 0.008 to 0.010 inch and the aperture size of the original cotton fabric was from 0.007 to 0.010 inch. In the product, the corresponding figures were each from 0.002 to 0.005 inch. The product porosity was 89.3%, the breaking strength 260 gm. and the resistivity 197 microhm-cm.

Thirteen sheets of this material were assembled face to face and resintered under the same conditions to give a plaque 0.059 inch thick with a porosity of 89.9%, a resistivity of 398 microhm-cm. and a bend strength of 47 kilograms per square centimeter (kg./cm.²).

A further five sheets were pressed together under a pressure of 5 tons per square inch to give a compact 0.005 inch thick with a porosity of 62.2% (pressure bonded).

EXAMPLE 3

Sheets of blotting paper approximately 12" x 6" x 0.019" were impregnated by immersion in nickel chloride solution (120 gm. NiCl₂·6H₂O in 100 cc. water) for 5 minutes, removed from the solution, allowed to drain for 10 minutes to remove excess solution and then dried at 70° C. for 30 minutes. The dried sheets were sintered in the furnace at one inch per minute at 10.50° C. to give nickel sheets 0.0065 inch thick with a porosity of 81.8% and a resistivity of 159 microhm-cm. Seven of these sheets were assembled face to face and sintered for one hour at 1150° C. in hydrogen under a load of 0.10 gm./cm.² to give a plaque 0.0354 inch thick with a porosity of 72.8% and a bend strength of 258 kg./cm.².

EXAMPLE 4

A sheet of cotton wool approximately 12" x 6" x 1.25" was immersed in the 120 gm. nickel chloride solution for 5 minutes, removed and pressed between two plates under a weight of 14 pounds (0.194 pound per square inch) for

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5 minutes to remove excess solution. The pressed sheet was dried at 70° C. for 22 hours to give a final thickness of 0.181 inch. The dried sheet was then sintered at one inch per minute at 1050° C. in the furnace to give a porous nickel plaque 0.074 inch thick with a porosity of 88%, bend strength over two supports 0.75 inch apart 33 kg./cm.², resistivity 320 microhm-cm. After resintering at one inch per minutes at 1150° C. to increase the strength, the porosity fell to 86%, the bend strength was 44.7 kg./cm.² and the resistivity 193 microhm-cm.

EXAMPLE 5

Impregnated paper pulp was produced by agitating flock used for chemical filtration in the 120 gm. nickel chloride solution. A mat produced from the pulp was pressed between two plates under a load of 0.204 pound per square inch for 5 minutes to remove excess solution. The sheet of pressed mat was dried for 16 hours at 70° C. to give a final thickness of about 0.200 inch and then sintered for 30 minutes at 1050° C. in cracked ammonia to give a nickel plaque 0.056 inch thick with a porosity of 90.6%.

EXAMPLE 6

Cotton string 0.091 inch diameter was impregnated by immersion in the 120 gm. nickel chloride solution for 10 minutes, dried and sintered at 1050° C. (9.7 minutes in the hot zone). The resultant nickel thread had a diameter of 0.053 inch, a porosity of 85.5%, a breaking strength of 232 gm. and a resistivity of 634 microhm-cm.

EXAMPLE 7

Sisal string 0.190 inch diameter treated in the same way as the cotton string in Example 6 had a diameter of 0.085 inch, a porosity of 91.8%, a breaking strength of 671 gm. and a resistivity of 104 microhm-cm.

EXAMPLE 8

Jute sacking impregnated and sintered in the same way as the cotton string in Example 6 gave a nickel sheet 0.016 inch thick with a porosity of 93.7%, a breaking strength of 232 gm. and a resistivity of 634 microhm-cm.

EXAMPLE 9

This shows the use of another nickel salt as the source of the nickel. A woven cotton fabric with a warp count of 72 and a weft count of 60 was immersed for 10 minutes in 100 cc. of water containing 38 grams of nickel sulfate (NiSO₄·6H₂O), dried at 70° C. in air and sintered with a residence time of 9.7 minutes in the hot zone at 1050° C. The product was nickel mesh of 95.8% porosity but of very low strength, resistivity 5,000 microhm-cm. Increasing the impregnation time to 18 hours gave 94.2% porosity, breaking strength 26.4 gm. and resistivity 941 microhm-cm.

EXAMPLE 10

This is an example of the production of an alloy mesh. A piece of the same 72/60 cotton fabric as in Example 9 was impregnated in a solution of 124.4 gm. NiCl₂·6H₂O and 35.6 gm. CuCl₂·2H₂O in 100 cc. of water for 10 minutes, dried at 70° C. in air and sintered with a residence time of 9.7 minutes in the hot zone at 1050° C. The product was mesh of an alloy containing 70% nickel and 30% copper with a porosity of 95.3%. The mesh was resintered at 1050° C. for 9.7 minutes to strengthen it, and the porosity fell to 88.2%, with breaking strength 226 gm. and a resistivity 3,020 microhm-cm. resulting.

EXAMPLE 11

Cotton fabric having 72 warp threads and 60 weft threads to the inch was impregnated for 10 minutes at room temperature in a solution of 80 gm. ferric chloride in 100 cc. of water, dried in air at 70° C. and sintered with a residence time of 8 minutes in the hot zone at

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1050° C. The product was iron mesh with a porosity of 96.9%.

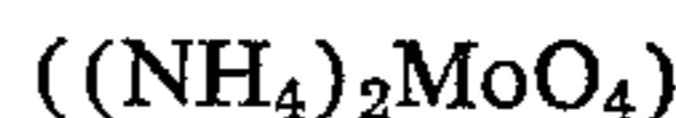
Twelve such sheets can be sintered together face to face at 1100° C. to form a battery plaque.

EXAMPLE 12

Fabric as in Example 11 was impregnated with a solution of 100 gm. copper chloride (CuCl₂·2H₂O) in 100 cc. of water. The product was copper mesh with a porosity of 95.6%. Twelve sheets of this mesh can be sintered together face to face at 1000° C. to form a plaque.

EXAMPLE 13

Fabric as in Example 11 was impregnated in a solution consisting of 40 gm. ammonium molybdate



in 100 cc. water. The product was a molybdenum mesh with a porosity of 96.9%, which fell to 92.2% when the product was sintered for 6 hours at 1300° C. in hydrogen. The breaking strength was 427 gm. and the resistivity 1,214 microhm-cm.

EXAMPLE 14

Fabric as in Example 11 was impregnated in a solution consisting of 10 gm. of platinum as ammonium chloroplatinate in 70 cc. of water. After soaking for 10 minutes at 70° C., the fabric was dried and sintered at 1050° C. in cracked ammonia while passing through the furnace at the rate of one inch per minute, so that its residence time in the hot zone was 12 minutes. The product was platinum mesh having a porosity of 97.3%, a breaking strength of 29 gm. and a resistivity of 1,937 microhm-cm.

EXAMPLE 15

Fabric as in Example 11 was impregnated for 10 minutes in a solution containing 60 gm. of silver nitrate in 100 cc. of water. The dried fabric was sintered 30 minutes at 900° C. in a cracked ammonia atmosphere. The product was silver mesh 0.006 inch thick with a porosity of 94.7%, a resistivity of 28.6 microhm-cm., and a breaking load of 504.6 grams.

EXAMPLE 16

Products were made by immersion of fabrics for 10 minutes in the 120 gm. nickel chloride solution, air-drying at 70° C. and sintering with a residence time of 9.7 minutes in the hot zone at 1050° C. Further products were made by an oxidation-reduction thermal process wherein fabrics were immersed for 18 hours in nickel chloride solution (102.2 gm. NiCl₂·6H₂O in 100 ml. water), air-dried at 70° C., heated in air at 10° C. per hour to 375° C., held at this temperature for 24 hours, heated in hydrogen for ½ hour at 400° C., raised at 25° C. per hour to 800° C., and held at this temperature for 30 minutes.

Three fabrics were used, namely, the 40/40 cotton (A), a cotton fabric with 72 warp and 60 weft threads per inch (B) and rayon fabric with 76 warp and 41 weft threads per inch (C). The products A1, B1 and C1 were made by the direct heating in cracked ammonia atmosphere and the products A2, B2 and C2 by the oxidation-reduction thermal process.

The properties of the products are shown in Table I below:

TABLE I

Product:	Porosity, percent	Resistivity, microhm-cm.	Strength, gm./cm.
A1.....	89.8	252.2	807.2
A2.....	92.8	398.5	483.7
B1.....	89.6	139.4	870.9
B2.....	91.6	353.1	331.6
C1.....	92.0	1,244.4	426.8
C2.....	92.1	272.5	620.8

It will be seen by comparing A1 with A2 and B1 with B2 that although the process according to the invention leads to some slight reduction in the porosity, it gives products of much reduced resistivity and increased strength. The need to use a natural material is clearly apparent from the properties of C1.

EXAMPLE 17

The improved properties obtained in plates according to the invention in comparison with those of plates of sintered nickel powder are shown in Table II below. In Table II, four plates 1 to 4 made according to the invention are compared with a plate 5 made by sintering nickel powder to both sides of woven nickel mesh. Plates 1 and 2 were made by sintering sheets themselves made from further pieces of the cotton fabric A, plates 3 and 4 from cotton fabric B, in the way described in Example 11 to produce sheets, and then sintering the sheets in face to face relation under a load of 10 gm./cm.² for one hour at 1150° C. in hydrogen, plates 1 and 2 being made from plaques comprising 10 sheets and plates 3 and 4 from plaques comprising 12 sheets. Plate 5 was made by sintering nickel powder on each side of a sheet of woven nickel mesh. The sintered plaques and the sintered powder were chemically impregnated with nickel hydroxide by a method involving seven cycles of conventional procedure.

Table II below shows the porosity, the bend strength, the resistivity and the thickness of the five plates. It also shows the electrical properties. Since the thicknesses differed, the table further shows the capacity adjusted to a thickness of 0.040 inch in each case, so that a proper comparison of the capacities may be made.

TABLE II.—COMPARISON OF SINTERED POWDER PLATE WITH SINTERED METAL CLOTH PLATES

Plate Number:	Porosity, percent	Bend strength, kg./cm. ²	Resistivity, microhm-cm.	Thickness, inch	Capacity, a.h./dm. ²		Coefficient of utilization at 7.75 a./dm. ² , percent	Capacity a.h./dm. ² at 7.75 a./dm. ² corrected to 0.040 inch thick
					Discharge current, 1.55 a./dm. ²	Discharge current, 7.75 a./dm. ²		
1.....	80.5	108.0	71.7	0.027	2.68	2.02	54	2.98
2.....	80.5	108.0	71.7	0.025	2.69	2.14	53	3.41
3.....	77.5	146.8	84.5	0.050	4.93	4.08	72	3.25
4.....	77.5	146.8	84.5	0.052	4.81	3.97	56	3.05
5.....	86.7	19.0	202.3	0.060	3.22	3.01	42	2.01

NOTE.—a./dm.²=Amperes per square decimeter; a.h./dm.²=Ampere hours per square decimeter.

It will be appreciated that in a fabric the warp and the weft can be of threads of different sizes, and by using such a starting fabric, a metal fabric can be made in which, for example, the warp is of larger-diameter threads than the weft. In a battery plate made from such a fabric, the sheets can with advantage be so orientated that the larger-diameter metal threads of the warp run in the most advantageous direction of the current flow, since these will have superior conductance to the metal threads of the weft.

It is also possible to start with a woven fabric in which the warp or the weft consists wholly or partially of a number of metal wires, the remainder of the fabric being of organic fibers. After the treatment of the fabric by the process of the invention, the original metal wires will have greater density than, and superior conductance to, those of the metal threads formed on the organic fibers.

Threads or pieces of mesh produced by the invention may be incorporated in the active mass of pocket and tubular electrodes to replace graphite or metallic flake.

Other uses for sheets produced according to the invention are as electrodes for fuel cells, catalyst supports and filters. In the case of fuel cell electrodes, the careful stacking of meshes of varying aperture can provide an electrode with conical pores. The meshes should have the same thread count but the threads will be of different diameters.

Threads produced according to the invention may be woven into suitable products. Either the sheets or the

threads may be used as metallic reinforcement in composite metal, plastic, glass and ceramic structures, or to provide a conductive path through plastics or other non-metallic materials.

The products of the invention may also be used as, or in, mechanical- and sound-damping materials, and magnetic screening materials. Decoration may be produced on metal strip by rolling mesh of the invention into it and developing contrasting colors by chemical treatment.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

We claim:

1. A method of producing a porous metal product which comprises impregnating a natural cellulosic fibrous material with one or more thermally decomposable metal salts of a metal from the group consisting of nickel, cobalt, iron, copper, tungsten, molybdenum, gold, silver and platinum group metals by immersing said material in an aqueous solution of said metal salt, drying the impregnated material and heating the dried material at a rapid rate of at least about 5° C. per minute in a single operation to a temperature of at least about 900° C. in a flowing gaseous atmosphere reducing to said metal to destroy the cellulosic material and to completely remove the carbon, to decompose the salt or salts to metal and to sinter the resultant metal to a coherent body.

2. The method according to claim 1 wherein the cellulosic material is selected from the group consisting of cotton, paper, jute, linen, ramie and sisal.

3. The method according to claim 2 wherein the impregnation is conducted for a time period of a few minutes to about two hours.

4. The method for producing a plaque for an alkaline battery plate which comprises impregnating a porous sheet of natural cellulosic material by immersing said material in a solution of a metal salt of at least one metal from the group consisting of nickel, iron, copper and cobalt, drying the impregnated sheet, decomposing the metal salt to metal by a single thermal treatment in a flowing gaseous atmosphere reducing to said metal, at a rapid heating rate of at least 5° C. per minute, at a temperature of at least about 900° C. to destroy the cellulosic material and to completely remove the carbon, and to sinter the resultant metal to a coherent porous sheet-form body, thereafter assembling in face to face relation a plurality of porous sheet-form bodies so obtained and sintering them together in a reducing atmosphere to form a battery plaque.

5. The method according to claim 4 wherein the cellulosic material is a natural material from the group consisting of cotton, paper, jute, linen, ramie and sisal.

6. The method according to claim 4 wherein the cellulosic material is a knitted or woven cotton fabric and the metal salt is nickel chloride.

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7. The method according to claim 4 wherein the battery plaque is formed by bonding a plurality of porous sheet-form bodies together using both heat and pressure.

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10 WINSTON A. DOUGLAS, Primary Examiner

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U.S. Cl. X.R.

15 136—36, 122

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,689,320

Dated September 5, 1972

Inventor(s) Thomas Brian Ashcroft, Victor Allen Tracey
Walter Betteridge and

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 4, line 59, for "NiCl₂·6H₂O" read --NiCl₂·6H₂O--.

Line 63, for "10.50°" read --1050°--.

Col. 5, line 8, for "minutes" read --minute--.

Line 29, for "232" gm" read --467 gm-- and
for "634 microhm-cm" read
--157 microhm-cm--.

Signed and sealed this 24th day of April 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents