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"IMPROVEMENTS IN OR RELATING TO METHOD OF SINTERING NICKEL POWDER TO PRODUCE THIN SINTERED PLAQUES FOR ALKALINE BATTERY PLATES"

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT(ACT XXI OF 1860).

The following specification describes the nature of this invention :—

This is an invention by HANDADY VENKATAKRISHNA UDUPA, PENNAGARAM VYSA RAO VASUDEVA RAO, RAMASAMY SABAPATHY, Director, Scientist, and Senior Scientific Assistant respectively of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, all Indian citizens.

This invention relates to the method of sintering nickel powder to produce thin sintered plaques for alkaline battery plates.

Hitherto it has been proposed to produce sintered plaques by loose powder technique under conditions not well defined to obtain thin sintered plaques.

This is open to the objection that thin sintered plaques with high porosity cannot be obtained by following the methods described.

The object of this invention is to obviate these disadvantages by standardising the process of sintering to obtain thin sintered plaques, for alkaline battery plates.

To these ends, the invention broadly consists in the steps described below.

1. *Annealing of nickel plated mild steel grid :*
This process is carried out to obtain very flat grid and to avoid the non-uniformity in thickness of the plaque due to warping of the plated grid. The grids are heat treated between 800—900°C in hydrogen or dissociated ammonia or any suitable inert atmosphere and cooling to the room temperature in steps so that the time taken for cooling is about 3 hours. The grids produced are flat and also devoid of any wrinkles.

2. *Production of sintered plaques :*

Nickel powder (Mond B Grade of Apparent density 0·88 gm/cc or Nickel powder of any similar suitable grade) is spread evenly on a graphite block having a small groove which determines the ultimate thickness of the sintered plaques. After spreading the nickel powder, annealed nickel plated grid is placed over it. Over this grid another layer of powder is sifted and then a glass rod is rolled to produce a uniform layer of Nickel powder upto the height of the groove. Similarly, a number of graphite blocks are filled and placed one over the other and top plate is covered with a plain graphite block. The whole assembly is placed in a stainless steel frame of suitable size with two bolts and nuts at the two ends. Another frame is slid from the top and the nuts are tightened slowly to give a uniform pressure of about 2 tons/sq. inch. The complete assembly is then placed in a stainless steel box and the charge is heated in a furnace between 750°C to 1000°C in hydrogen or dissociated ammonia atmosphere for a duration of 10-60 minutes, with automatic control.

The resultant porosity of the plaques ranged from 75%—85% and predominant pore diameter is approximately 6/μ. The sintered plaques were tested by bending in various dia. rods for the machanical strength. It was found that no peeling off sintered material has been observed upto 1/4" dia rod.

In order to increase porosity and pore diameter volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol and polymethoxy acrylate, and which reduce to nickel powder such as nickel formate, in varying proportions from 10—25% by wt. of nickel powder were used. The resultant plaques have a porosity of 85—90% and predominant pore diameter is approximately 10 microns.

The following are the typical examples illustrating the invention :

EXAMPLE I

16 nickel plated m.s. grids size 7·5 cm×6 cm. were annealed at 800°C for 30 minutes in hydrogen atmosphere. The resultant grids were flat.

Nickel powder was placed in a graphite block of size 16 cm×15 cm with a groove thickness of 0·8 mm and the grid was placed over the powder and then nickel powder was sifted over the grid. The extra powder was then rolled out with the help of a glass rod. One graphite block was then placed over to cover the top and 8 blocks were kept having 16 such grids covered with powder. The entire set up was placed in heating zone of a muffle in an atmosphere of hydrogen or dissociated ammonia and heated continuously at 900°C for 30 minutes. After cooling the porosity of the plaques was determined and the same was 80—82% on average. The predominant pore size was found to be 6 microns.

EXAMPLE II

Following the procedure employed in Example I' except that nickel powder was mixed with 15% by weight of ammonium carbonate and sintering was carried out. The porosity of the sintered plaques ranged from 85—95% and the predominant pore size was 10 microns.

EXAMPLE III

Following the procedure employed in Example I, except that the nickel powder mixed with 15% by wt. of polyvinyl alcohol was used and the sintered plaques obtained were having porosity 85—90% and predominant pore size was 12 microns.

The following is the main advantage of the invention.

1. The sintered plaques obtained have high porosity, with optimum pore diameter and are of a good mechanical strength.

Dated this 29th day of June, 1972.

Sd/-

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Council of Scientific and Industrial Research.

Price: TWO RUPEES

THE PATENTS ACT, 1970

COMPLETE SPECIFICATION

Section 10

"IMPROVEMENTS IN OR RELATING TO METHOD OF SINTERING NICKEL POWDER TO PRODUCE THIN SINTERED PLAQUES FOR ALKALINE BATTERY PLATES"

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed :—

This is an invention by HANDADY VENKATAKRISHNA UDUPA, Director, PENNAGARAM VYSA RAO VASUDEVA RAO, Scientist and RAMASAMY SABAPATHY, Senior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, all Indian citizens.

This invention relates to method of sintering nickel powder to produce thin sintered plaques for alkaline battery plates.

Hitherto it has been proposed to produce sintered plaques by loose powder technique under conditions not well defined.

The sintered plaques with high porosity cannot be obtained by following methods described.

The object of the invention is to obviate these disadvantages by standardising the process of sintering to obtain thin sintered plaques for alkaline battery plates.

Optimum conditions here has been worked out to produce thin sintered plaques for alkaline battery plates.

According to the present invention, there is provided a process for the production of sintered plaques for alkaline battery plates which consists in annealing nickel or nickel plated grids between 800°C to 900°C and sintering nickel powder over the annealed grids between 750°C to 1000°C.

The annealing is carried out preferably at 800°C in hydrogen or dissociated ammonia or any suitable inert atmosphere, for a duration between 10 minutes to 2 hours preferably for 30 minutes.

The sintering is carried out over the annealed grids using nickel powder of suitable grade preferably carbonyl nickel powder.

The sintering process is carried out preferably at 900°C in hydrogen or dissociated ammonia preferably using dissociated ammonia, for a duration of 10 to 60 minutes preferably 30 minutes.

Volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol and polymethoxy acrylate and nickel formate which gets reduced to nickel powder during sintering are added alone or in combination, to the nickel powder used for sintering so that a highly porous sintered plate is produced.

The volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol, polymethoxy acrylate and nickel formate which reduces to nickel powder during sintering are added to nickel powder in varying proportions ranging between 10—25% by weight of nickel powder preferably 15% by weight.

The process consists of the steps described here in below :—

(i) Annealing of nickel or nickel plated mildsteel grid :

The process is carried out to obtain very flat grid and to avoid the non uniformity in thickness of the plate due to warping of the plain on plated grid. The grids are heat treated between 800 to 900°C in hydrogen or dissociated ammonia or any suitable inert atmosphere for a duration between 10 minutes to 2 hours and cooling to the room temperature in steps so that the time taken for cooling is about 3 hours. The grids produced are flat and also devoid of any wrinkles.

(ii) Production of sintered plaques :—

Nickel powder (Mond B grade of apparent density 0·88 gm/cc or nickel powder of any similar suitable grade) is spread evenly on a graphite block having a small groove which determines the ultimate thickness of the sintered plaques. After spreading the nickel powder, annealed nickel or nickel plated grid is placed over it. Over this grid another layer of powder is sifted and then a glass rod is rolled to produce a uniform layer of nickel powder upto the height of the groove. Similarly, a number of graphite blocks are filled and placed one over the other and top plates is covered with a plain graphite block. The whole assembly is placed in a stainless steel frame of suitable size with two bolts and nuts at the two ends. Another frame is slid from the top and the nuts are tightened slowly to give a uniform pressure of about 2 tons/sq. in. The complete assembly is then placed in a stainless steel box and the charge is heated in a furnace between 750°C to 1000°C in hydrogen or dissociated ammonia for a duration of 10—60 minutes with automatic control.

The resultant porosity of the plaques ranged from 75—85% and predominant pore diameter is approximately 6/μ The sintered plaques were tested by bending in various dia. rods for mechanical strength. It was found that no peeling of sintered material has been observed upto $\frac{1}{4}$" dia rods.

In order to increase the porosity and pore diameter volatile addition agents like ammonium chloride ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol and poly methoxy acrylate and which reduce to nickel powder such as nickel formate, in varying proportions from 10—25% by weight of nickel powder were used. The resultant plaques have a porosity of 85—90% and predominant pore diamenter is approximately 10 microns.

In the invented method for sintering nickel powder to produce thin sintered plaques for alkaline battery plates, the new steps are annealing of nickel or nickel plated mild steel grids and sintering nickel powder with volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol and polymethoxy acrylate, which reduce to nickel powder like nickel formate.

The following are the typical examples illustrating the invention :

EXAMPLE I

16 nickel plated m.s. grids of size 7·5 cm×6 cm were annealed at 800°C for 30 minutes in hydrogen atmosphere. The resultant grids were flat.

Nickel powder was placed in a graphite block of size 16 cms×15 cm with a groove thickness of 0·8 mm and the grid was placed over the powder and the nickel powder was sifted over the grid. The extra powder was

then rolled out with the help of a glass rod. One graphite block was then placed over to cover the top and 8 blocks were kept having 16 such grids covered with powder. The active set up was placed in heating zone of a muffle in an atmosphere of hydrogen or dissociated ammonia and heated continuously at 900°C for 30mis. After cooling the porosity of the plaques was determined and the same was 80—82% on average. The predominant pore size was found to be 6 microns.

EXAMPLE II

Following the procedure employed in Example I, except that nickel powder was mixed with 15% by weight of ammonium carbonate and sintering was carried out. The porosity of the sintered plaques ranged from 85—95% and predominant pore size was 10 microns.

EXAMPLE III

Following the procedure employed in Example I, except that nickel powder mixed with 15% by weight of polyvinyl alcohol was used and the sintered plaques obtained were having porosity 85—90% and predominant pore size was 12 microns.

The invention consists in the production of sintered plaques for alkaline battery plates comprising mainly of the nickel powder with volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol and polymethoxy acrylate, sintered on to nickel or nickel plated m.s. grids. The conditions of sintering has been standardised so that the sintered plaques obtained have high porosity, with optimum pore diameter and are of good mechanical strength.

WE CLAIM :

1. A process for the production of sintered plaques for alkaline battery plates which consists in annealing nickel or nickel plated grids between 800°C to 900°C and sintering nickel powder over the annealed grids at 750°C to 1000°C.

2. A process as claimed in claim 1 wherein the annealing is carried out at 800°C.

3. A process as claimed in claims 1 or 2 wherein the annealing is carried out in hydrogen or dissociated ammonia or any suitable inert atmosphere.

4. A process as claimed in any of the preceding claims wherein the annealing is carried out for a duration between 10 minutes to 2 hours preferably for 30 minutes.

5. A process as claimed in any of the preceding claims wherein sintering is carried out over the annealed grids using nickel powder of suitable grade preferably carbonyl nickel powder.

6. A process as claimed in any of the preceding claims wherein the sintering process is carried out preferably at 900°C.

7. A process as claimed in any of the preceding claims wherein the sintering process is carried out in hydrogen or dissociated ammonia preferably using dissociated ammonia.

8. A process as claimed in any of the preceding claims wherein the sintering process is carried out for a duration of 10 to 60 minutes preferably 30 minutes.

9. A process as claimed in any of the preceding claims wherein volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyviny alcohol and polymethoxy acrylate and nickel formate which gets reduced to nickel powder during sintering are added alone or in combination, to the nickel powder used for sintering so that a highly porous sintered plate is produced.

10. A process as claimed in any of the preceding claims wherein the volatile addition agents like ammonium chloride, ammonium carbonate, ammonium bicarbonate, polyvinyl alcohol, polymethoxy acrylate and nickel formate which reduces to nickel powder during sintering are added to nickel powder in varying proportions ranging between 10—25% weight of nickel powder preferably 15% by weight.

11. A process for the production of sintered plaques for alkaline battery plates substantially herein before described.

Dated this 20th day of July, 1973.

Sd/-

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