GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

Specification No. 107347. Application No. 107347, dated 5th October, 1966. Complete Specification left on 2nd August, 1967. (Application accepted 29th June, 1968.)

Index at acceptance—14D2 [LVIII(1)].

PROVISIONAL SPECIFICATION.

IMPROVEMENTS IN AND RELATING TO THE PREPARATION OF POROUS CARBON ELECTRODES FROM ACTIVE CARBONS FOR USE IN AIR DEPOLARISED CELLS.

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

The following specification describes the nature of this invention.

This is an invention by Abdul Khader Abdul Waheed (Indian citizen), Scientist, Chinnasamy Chakkaravarthy (Indian citizen), Senior Scientific Assistant, Michael Angelo Vincent Devanathan (citizen of Ceylon), Scientist, and Kadarundalige Sitarama Gururaja Doss (Indian citizen), Director, all of the Central Electrochemical Research Institute, Karaikudi 3, Madras State.

This invention relates to a process for the manufacture of porous carbon electrodes and in particular to the manufacture of porous carbon electrodes by the admixture of high conductivity carbon black and activated vegetable carbon using a high polymer as binder. Such carbon electrodes can be used in Air Depolarised cells.

More particularly it relates to certain improvements which give added advantages to the process described in an earlier specification No. 90957.

In the specification of pending application No. 90957 dated 23-11-63 is described a process for the manufacture of porous carbon electrodes by the admixture of acetylene black and activated vegetable carbon using a high polymer as a binder.

In order to briefly restate the principle of the Air Depolarised cell it may be stated that this is a primary cell which utilises the oxygen of the air as the cathode component and consists of a porous carbon element as the cathode (through which oxygen (air) circulates and takes part in the reaction), zinc as the anode and sodium hydroxide as the electrolyte. Such an Air Depolarised cell has an open circuit voltage of 1.40-1.45 volts, operates at 1.10-1.20 volts and has a constant voltage discharge curve for the entire running period at normal discharge rates; it is designed to be assembled in the field.

The Air Depolarised cell has several application, for instance in railway and marine signalling and switch lamp lighting, telephone and other general instrument operations and emergency lighting.

According to the said specification the invention consisted of a process of making porous carbon electrode elements wherein a mix of activated vegetable carbon, acetylene black and a high polymer binder is pressed in the form of the electrode element using a suitable mould. After pressing the electrode was allowed to weather dry "*in situ*" in the atmosphere before removal from the mould.

The high polymer binder is dissolved in a suitable solvent (such as benzene or trichlorethylene) and acts also as a water proofing agent.

It was stated that an advantage of a porous carbon electrode element constructed in accordance with the invention was that most of the element could be made from indigenously available vegetable carbon, the only other materials necessary being present in minor amounts. For example, acetylene black may be present in a proportion of about 10 per cent. and the high polymer binder may be present in a proportion of about 3 per cent. by weight of the element.

Moreover, the binder used in the manufacture of the porous carbon electrode acts also as a water-proofing agent. This result in reducing the number of steps involved in the manufacture of carbon electrode elements from four, *viz.* those of binding, pressing, baking and water-proofing, as performed in the usual method of making porous carbon electrode element, to two, merely blending and pressing.

Further, the fact that the porous carbon electrode element consists of a blend of vegetable carbon, acetylene black and a high polymer binder and can be made by a simple procedure involving no costly equipment (a mixer and a hydraulic press being all that is required) makes production economical.

Thus in the earlier specification it was emphasised that the invention had for its main object a process whereby a carbon electrode for air depolarised cells could be made as far as possible from indigenously available materials and by a simpler process resulting in economy.

Following up with that objective, it was very desirable that acetylene black be replaced with a material more readily available indigenously, at the same time without detriment to any of the advantages of the said process.

The object of the present invention is to effect specifically two improvements. The first of these relates to materials, namely, the replacement of acetylene black by other forms of highly conducting carbon blacks so as to extend the scope of the earlier process of making the porous carbon electrode. The high conductivity carbon black used in the present invention is more specifically carbon black obtained as a by-product during the combustion of liquid naphtha in the fertiliser plants. A few other forms of high conducting carbons such as lamp black can also be used.

The replacement of the acetylene black by the carbon black results in a porous carbon electrode of equal physical and electrical characteristics.

The second relates to an improvement in the process of making the porous carbon electrode. The improvement lies in the fact that the electrode is ejected from the mould and dried at controlled temperature, instead of being kept in the mould and allowed to dry *in situ*, as described in the earlier specification No. 90957.

For comparison, the physical properties of carbon black and acetylene black used in the invention are given in Table 1.

TABLE I

	Carbon black	*Acetylene black*
1. Apparent density	0.18 gm/c.c.	0.08 gm/c.c.
2. Resistivity	0.44 ohm.cm.	0.36 ohm.cm.

The performance characteristics of the air depolarised cell incorporating the carbon electrode made either with the inclusion of carbon black or acetylene black are shown in Table II.

TABLE II

Composition of the electrode	Open circuit voltage	Short circuit current	Closed circuit voltage at 1 ohm load
10% acetylene black 90% activated coconut shell charcoal and 3% binder	1.40 volts	10.5 amps.	1.10 volts
20% carbon black 80% activated coconut shell charcoal and 3% binder	1.40 volts	11.5 amps.	1.10 volts.

Price : TWO RUPEES.

In order that the present invention, which effects the aforesaid improvements, may be clearly understood and practised, the following procedure for making porous carbon electrode element for the special railway type air depolarised cell of capacity 500 watt hours (the special railway type air depolarised cell is more fully described in B.S. 1335 : 1946) is described in detail in the following example.

Example

A blend consisting of activated carbon (such as activated coconut shell charcoal) in powdered form, preperably between —100 mesh and +140 mesh size, and a small percentage of carbon black (ranging from 10 to 30 per cent. but preferably 20 per cent. wt/wt) to which a high polymer binder cum waterproofing agent such as perspex (ranging from 1.5 to 3 per cent. preferably 3 per cent. of the mass of the electrode) dissolved in a suitable solvent (such as benzene or trichlorethylene) had been added, was used for making the electrodes. The addition of high polymer in ratio of less than 1.5 per cent. tends to affect the mechanical strength adversely whereas a ratio of greater than 3 per cent. tends to increase the resistance.

Approximately 560 gms. of activated coconut shell charcoal and approximately 140 gms. of carbon black were mixed using a kneading machine with the addition of about 20 gms. of a high polymer (such as perspex) dissolved in about 500 cc. of a suitable solvent (such as benzene or trichlorethylene). The carbon mix was then pressed in the form of an electrode at a pressure of about half ton per sq. inch in the hydraulic press, using a mould such as one made of wood. After pressing, the electrode was ejected out from the mould using a suitable plunger in the hydraulic press. The electrode was then dried at controlled temperature (around 100°C.) for nearly twelve to eighteen hours.

(For electrical contact, a central mild steel rod ½ dia. (nickel plated) with a smaller mild steel disc (nickel plated) on top and a bigger one at the bottom of the electrode was used. The discs were kept in contact with the electrode using suitable nuts.)

A seamless plastic or rubber ring 2″ wide and 1/16″ thick was then inserted around the top portion of the electrode. Wax coating was given at the lower end of the plastic or rubber ring to avoid seepage of the electrolyte to the top surface of the carbon electrode. The electrode made in the above manner could then be assembled into an AIR DEPOLARISED CELL.

The air depolarised cell assembled from the porous carbon electrode element made according to the invention described hereinbefore gives an open circuit voltage of 1.4 volts and short circuit current of about ten amperes (as measured with a 20 ampere range ammeter)

For the shape and dimensions of the porous carbon electrode, reference may be made to Figures 1 and 2 of the accompanying drawings showing the top view and the sectional elevational view respectively of the porous carbon electrode.

The example describes in detail the method of making porous carbon electrode element, in particular, that of the special railway type air depolarised cell of 500 watt hour capacity. However, it will be appreciated that the invention may be utilised in the manufacture of porous carbon electrode elements for other types of air depolarised cells, as also porous carbon electrodes for any other use

ADVANTAGES

By virtue of the advantage of being able to use an alternative material such as carbon black in the place of acetylene black, the alternative material at the same time being indigenously available, as also by using the ejection method, which makes the process faster and also by using more controlled conditions of subsequent drying of the electrodes, the complete process becomes versatile.

R. BHASKAR PAI,

Patents Officer,

Council of Scientific & Industrial Research.

Dated this 29th day of September 1966.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN AND RELATING TO THE PREPARATION OF POROUS CARBON ELECTRODES FROM ACTIVE CARBONS FOR USE IN AIR DEPOLARISED CELLS.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH RAFI MARG, NEW DELHI-1, INDIA, A INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXII 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 : Abdul Khader Abdul Waheed (Indian citizen), Scientist, Chinnasamy Chakkaravarthy (Indian citizen), Senior Scientific Assistant, Michael Angelo Vincent Devanathan (Citizen of Ceylon), Scientist, and Kadarundalige Sitarama Gururaja Doss, (Indian citizen), Director, all of the Central Electrochemical Research Institute, Karaikudi-3, Madras State.

This invention relates to a process for the manufacture of porous carbon electrodes and in particular to the manufacture of porous carbon electrodes by the admixture of high conductivity carbon black and activated vegetable carbon using a high polymer as binder. Such carbon electrodes can be used in Air Depolarised cells.

More particularly it relates to certain improvements which give added advantages to the process described in an earlier specification No. 90957. In the specification of 90957, dated 23.11.1963 (sealed), is described a process for the manufacture of porous carbon electrodes by the admixture of acetylene black and activated vegetable carbon using a high polymer as binder.

The well known air depolarised cell is a primary cell which utilises the oxygen of the air as the cathode component and consists of a porous carbon element as the cathode (through which oxygen (air) circulates and takes part in the reaction) zinc as the anode and sodium hydroxide as the electrolyte. Such an air depolarised cell has an open circuit voltage of 1.40-1.45 volts, operates at 1-10-1.20 volts and has a constant voltage discharge curve for the entire running period at normal discharge rates ; it is designed to be assembled in the field.

The cell reactions are as follows :

Over all anodic reaction :

$$Zn + 4\overline{OH} \rightarrow Zn\overline{O}_4 + 2H_2O + 2e$$

Overall cathodic reaction : $2e + O + H_2O \rightarrow 2\,OH$

Overall cell reaction :

$$Zn + 2\overline{OH} + O \rightarrow Zn\overline{O}_2 + \overline{H}_2O$$

The air depolarised cell has several civil and military applications ; however, it finds its special use on a large scale in railway signalling.

According to the said specification, the invention consisted of a process of making porous carbon electrode elements wherein a mix of activated vegetable carbon, acetylene black and a high polymer binder is pressed in the form of the electrode element using a suitable mould. After pressing, the electrode was allowed to weather dry '*in situ*' in the atmosphere before removal from the mould. The high polymer binder is dissolved in a suitable solvent (such as benzene or trichlorethylene) and acts also as a water proofing agent.

The advantages of the invention lay in the fact of the main constituent, namely the activated vegetable carbon being available indigenously, the only other materials necessary being in small amounts, for instance acetylene black (5-15 per cent.) and high polymer binder (1.5-3 per cent.), as also in the fact of the binder being at the same time a water-proofing agent; the latter aspect eliminates the steps of baking and separate water-proofing; in the conventional method, thus resulting in simplification and economy.

Following up with the objectives of indigenous availability and economy, it was very desirable that acetylene black be replaced with a material more readily available indigenously at the same time without detriment either to any of the advantages of the said process or to the physical and electrical characteristics of the resulting electrode.

The object of the present invention is to effect specifically two improvements.

According to the present invention, the process for making porous carbon electrode element for use in air depolarised cells by mixing activated vegetable charcoal, more particularly activated cocoanut shell charcoal with carbon black and a high polymer, dissolved in a solvent pressure moulding and drying the carbon element is characterised in that the carbon element is ejected from the mould in the wet condition prior to drying.

The drying is done at a controlled temperature of about 70°C. The drying is complete within six hours.

A highly conducting carbon black obtained as a by-product in the combustion of liquid naphtha in the fertilizer industry is used. A highly conducting carbon such as lamp black may also be used.

Carbon black ranging from 10-30 per cent. by weight of the electrode is used.

The invention includes within its scope a porous carbon electrode element for use in air depolarised cells prepared according to the invented process and also the porous carbon electrode element when used in an air depolarised cell.

The process thus replaces acetylene black by other forms of highly conducting carbon blacks so as to extend the scope of the earlier process of making the porous carbon electrode. The high conductivity carbon black used in the present invention is more specifically a carbon black obtained as a by-product during the combustion of liquid naphtha in fertilizer plants. It is usually obtained in the form of carbon pellets containing 65-70 per cent by weight of fuel oil. In order that the carbon black is in a form suitable for use in the making of the porous carbon electrode, the oil from the pellets is removed, for instance by subjecting the pellets to ignition. A few other forms of high conducting carbons such as lamp black can also be used.

The replacement of the acetylene black by the carbon black results in a porous carbon electrode of equal physical and electrical characteristics.

The second improvement relates to the process of making the porous carbon electrode. The improvement lies in the fact that the electrode is ejected from the mould and dried at controlled temperature at about 70°C., instead of being kept in the mould and allowed to dry '*in situ*', as described in the earlier specification No. 90957.

For comparison, the physical properties of acetylene black and carbon black used in the invention are given in Table I.

TABLE I

	Acetylene black	*Carbon black*
1. Apparent density	0.08 gm/c.c.	0.18 gm/c.c.
2. Resistivity	0.36 ohm.cm.	0.44 ohm.cm.

The performance characteristics of the air depolarised cell incorporating the carbon electrode made either with the inclusion of acetylene black or carbon black are shown in Table II.

TABLE II

Composition of the electrode	Open circuit voltage	Short circuit current.	Closed circuit vol. at one ohm. load
10% acetylene black 90% activated coconut shell charcoal and 3% binder	1.40 volts	10.5 amps	1.10 volts
20% carbon black 80% activated coconut shell charcoal and 3% binder	1.40 volts	11.5 amps	1.10 volts

In order that the present invention, which affects the aforesaid improvements, may be clearly understood and practised, the following procedure for making porous carbon electrode element for the special railway type air depolarised cell of capacity 500 watt hours (the special railway type air depolarised cell is more described in B.S. 1335 : 1946) is described in detail in the following example.

EXAMPLE

A blend consisting of activated carbon (such as activated coconut shell charcoal) in powdered form, preferably between −100 and +140 mesh size, and a small percentage of carbon black (ranging from 10 to 30 per cent. but preferably 20 per cent wt/wt) to which a high polymer binder-cum-waterproofing agent such as perspex (ranging from 1.5 to 3 per cent., preferably 3 per cent. of the mass of the electrode) dissolved in a suitable solvent (such as benzene or trichlorethylene) had been added, was used for making the electrodes. The particle size of the activated coconut shell charcoal has a bearing on the mechanical strength and porosity of the finished electrode. Similarly regarding the quantities of constituents, it may be stated that carbon black if present in amounts less than 10 per cent. does not impart sufficient electrical conductivity whereas if present in amounts more than 30 per cent. adversely affects the mechanical strength of the resulting electrode. Similarly if the high polymer binder (such as perspex) were added only in an amount of upto 1.5 per cent. by weight, the mechanical strength of the completed electrode element would be adversely affected whereas if more than 3 per cent. by weight were added, the resistance of the electrodes would tend to increase.

Approximately, 560 gms of activated coconut shell charcoal and approximately 140 gms of carbon black were mixed using a Kneading machine with the addition of about 20 gm of a high polymer (such as perspex) dissolved in about 500 c.c. of a suitable solvent (such as benzene or trichlorethylene). The carbon mix then pressed in the form of an electrode at a pressure of about half ton per square inch in the hydraulic press using a mould such as one made of wood. Placing of a seamless plastic or rubber ring 2″ wide and 1/16″ thick around the top portion of the electrode is also done at this stage. After pressing, the electrode was ejected out from the mould using a suitable plunger in the hydraulic press. The electrode was then dried at controlled temperature (around 70°C.) for nearly 12 to 18 hours.

For electrical contact, a central mild steel rod ½″ diameter (nickel plated) with a smaller mild steel disc (nickel plated) on top and a bigger one at the bottom of the electrode was used. The discs were kept in contact with the electrode using suitable nuts.

The air depolarised cell assembled from the porous carbon electrode element made according to the invention described hereinbefore gives an open circuit voltage of 1.40 volts and short circuit current of about 10 amps. (as measured with a 20 amp. range ammeter).

The example describes in detail the method of making porous carbon electrode element, in particular that of the special railway type air depolarised cell of

500 watt hour capacity. However, it will be appreciated that the invention may be utilised in the manufacture of porous carbon electrode elements for other types of air depolarised cells, as also porous carbon electrodes for any other use.

ADVANTAGES

By virtue of the advantage of being able to use an alternative material such as carbon black in the place of acetylene black, the alternative material at the same time being indigenously available, as also by using the ejection method, which makes the process faster and by using more controlled conditions of subsequent drying of the electrode, the complete process becomes versatile.

We claim :

1. A process for making porous carbon electrode element for use in air depolarised cells by mixing activated vegetable charcoal, more particularly activated cocoanut shell charcoal with carbon black and a high polymer, dissolved in a solvent, pressure moulding and drying the carbon element which is characterised in that the carbon element is ejected from the mould in the wet condition prior to drying.

2. A process as claimed in Claim 1 wherein the drying is done at a controlled temperature of about 70°C.

3. A process as claimed in Claim 1 or 2 wherein the drying is complete within six hours.

4. A process as claimed in any of the preceding claims wherein is used a highly conducting carbon black obtained as a by-product in the combustion of liquid naphtha in the fertilizer industry.

5. A process as claimed in any of the preceding claims wherein a highly conducting carbon such as lamp black is used.

6. A process as claimed in any of the preceding claims wherein is used carbon black ranging from 10-30 per cent. by weight of the electrode.

7. Porous carbon electrode element for use in air depolarised cells, made by the process substantially described herein.

R. BHASKAR PAI

Patents Officer,

Council fo Scientific & Industrial Research.

Dated this 29th day of July 1967.

MGIPC—S20—9 Patent/71—30-6-73—200.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

PROVISIONAL SPECIFICATION

No. OF SHEETS :- 1.
SHEET No. :- 1.

No. 107347

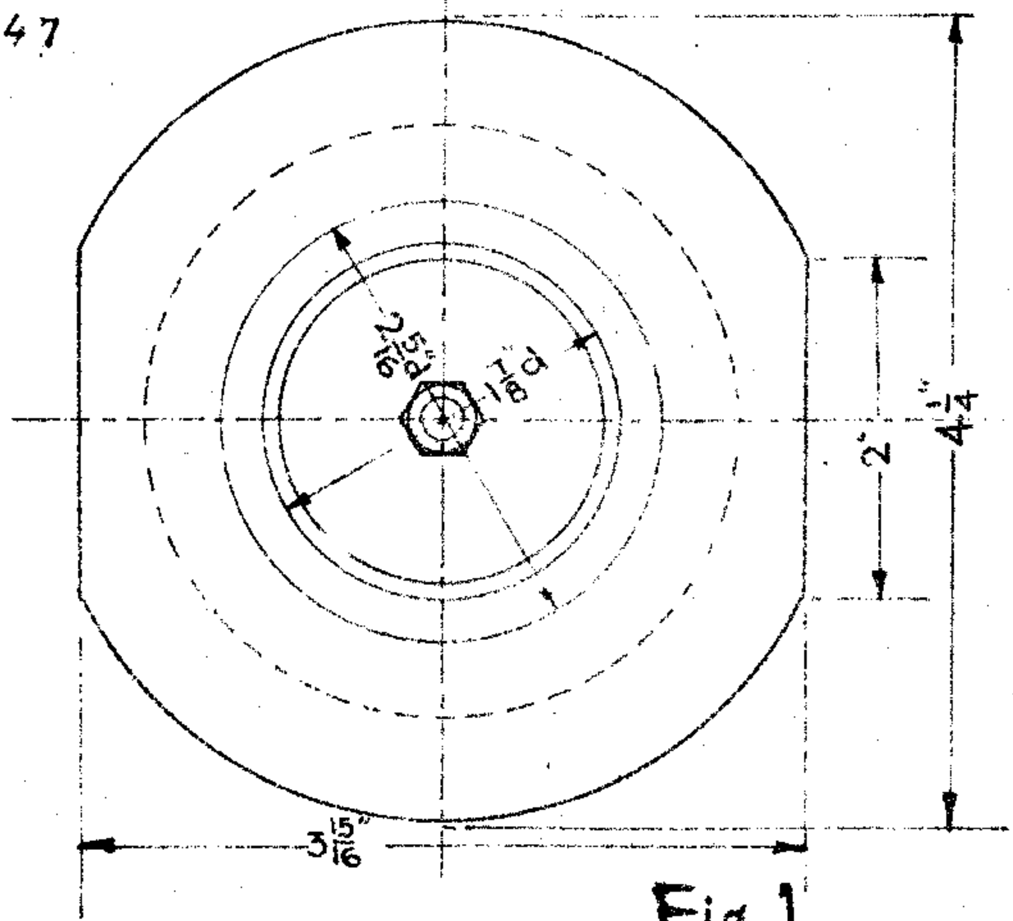

Fig. 1

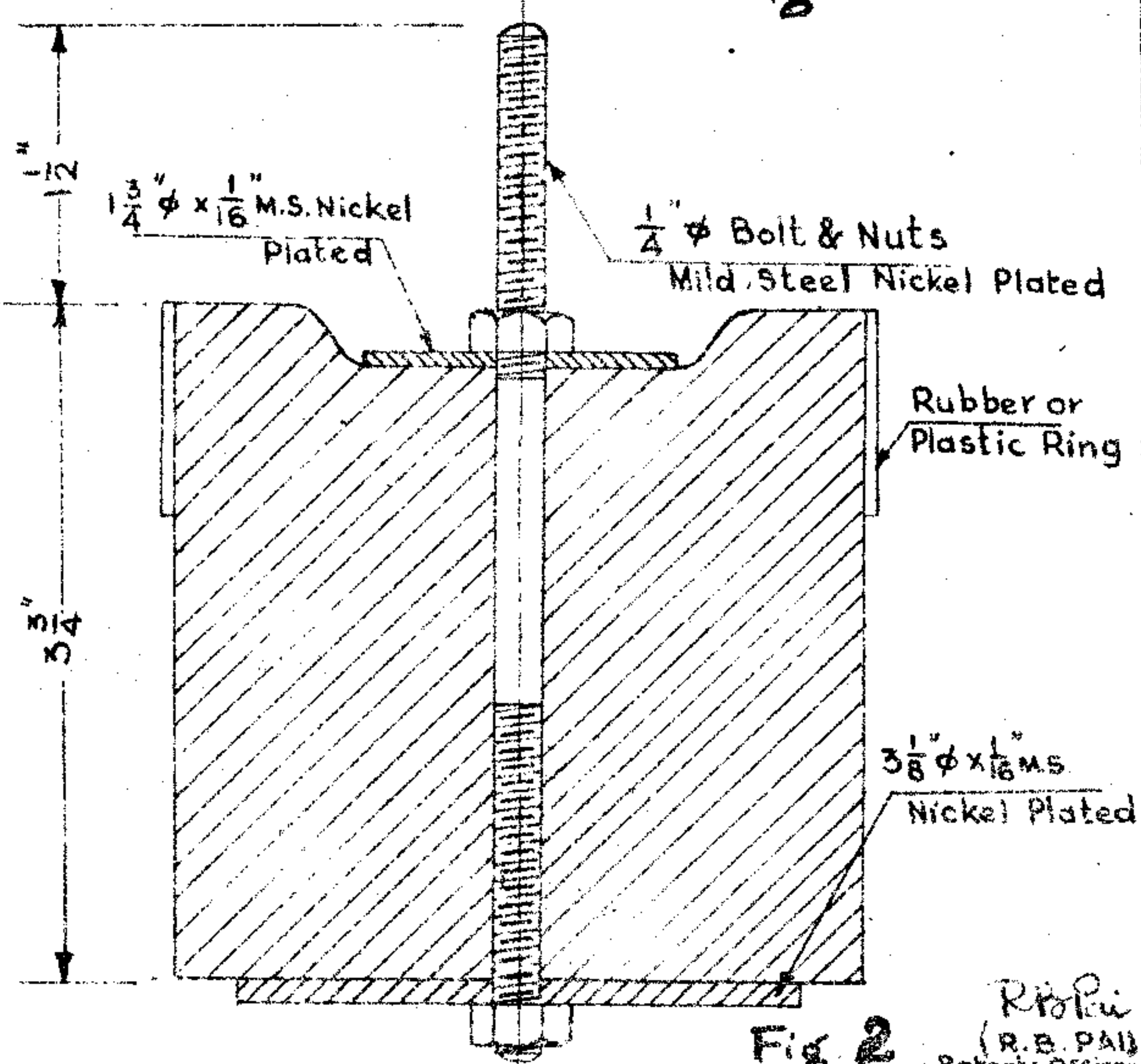

Fig. 2

R. B. Pai
(R. B. PAI)
Patents Officer,
C. S. I. R.

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