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3,287,248

ELECTROCHEMICAL CELL INCLUDING A TUBULAR FORAMINOUS PARTITION

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FIG. 1

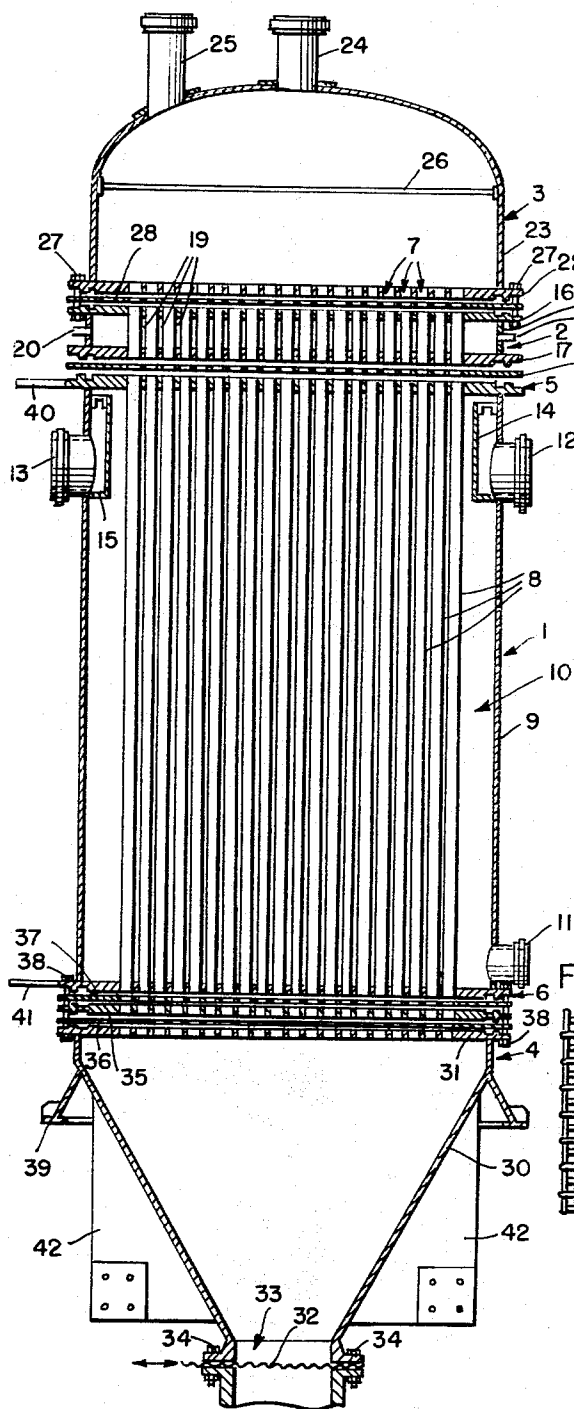


FIG. 2

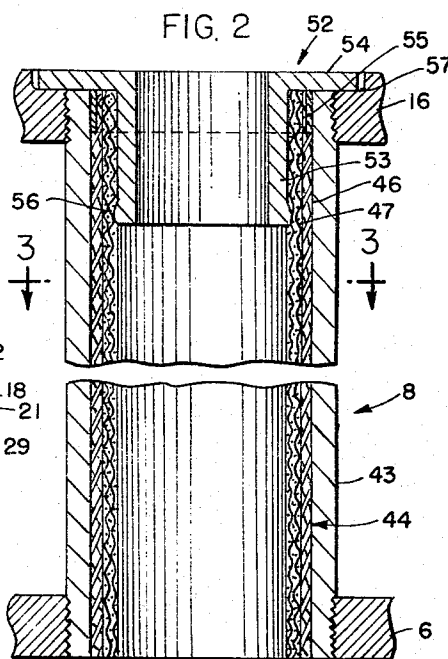


FIG. 3

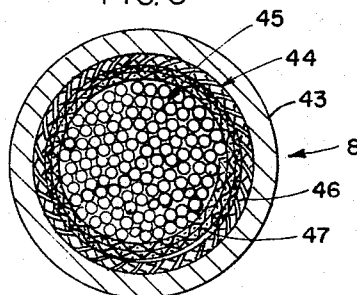


FIG. 4

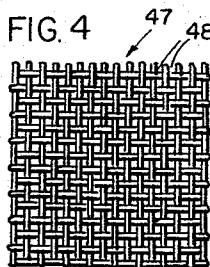


FIG. 5

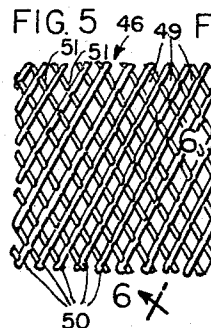


FIG. 6



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**ELECTROCHEMICAL CELL INCLUDING A
TUBULAR FORAMINOUS PARTITION**

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This invention relates to a liner or partition for an electrolytic cell and more particularly to a foraminous partition for location within the annular electrolyzing zone of a cell for electrolyzing a sacrificial anode in a liquid electrolyte. The invention is especially concerned with a new and improved tubular electrically non-conducting liner or partition which is employed in an electrochemical cell for the manufacture of tetraalkyl lead compounds, including, for example, tetraethyl lead, tetramethyl lead, triethylmonomethyl lead, diethyldimethyl lead, monoethyltrimethyl lead, and mixtures thereof, and which is useful in the manufacture of other organic metallic compounds by an electrolytic process involving the use of a sacrificial anode.

The term "sacrificial anode" refers to an anode which is eroded or dissolved during the electrolytic process.

In one type of sacrificial anode process employed for making tetraalkyl lead compounds, the lead anode is in particulate form, that is, particles, spheres or pellets. The electrolyte is an organic magnesium compound, such as a Grignard reagent, dissolved in an anhydrous solvent and this is circulated through the lead particles. The cathode consists of an electrically conducting metal, such as steel and a foraminous partition, liner or diaphragm is placed between the metal cathode and the lead particles in such a way that it is in contact with both. In this way, the cathode and the lead particles are very close to each other and a narrow space between them is provided by the partition. The electrolyte circulates through the anode particles and through the narrow space between these particles and the cathode. It is essential that the foraminous partition be electrically non-conducting and that the lead particles should be held out of direct contact with the cathode by the partition. At the same time, it is desirable that the lead particles and the cathode be as close together as possible and that there be a maximum circulation of the electrolyte in the narrow space between them.

The lead particles being heavy exert considerable drag on the partition with which they are in contact and it is necessary to provide a very durable partition which will not sag and in which the openings will not become enlarged so as to permit the lead particles to contact the cathode directly and short circuit the cell.

One of the objects of the present invention is to provide a new and improved type of foraminous partition which is so constructed as to facilitate passage of a liquid electrolyte between the anode particles and the cathode.

Another object is to provide a new and improved tubular electrically non-conducting foraminous partition for location within an annular electrolyzing zone of a cell for electrolyzing a sacrificial anode in a liquid electrolyte which is so constructed that the partition will maintain its shape while at the same time directing the flow of the electrolyte.

A further object of the invention is to provide a foraminous partition of the type described comprising a plurality of electrically non-conducting materials.

A more specific object of the invention is to provide a foraminous partition of the type described comprising a first foraminous tubular electrically non-conducting material having filaments therein overlapping one another and running obliquely with respect to the annular electrolyzing zone and a second foraminous tubular electrically non-conducting material concentrically arranged with respect to said first material.

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Other objects and advantages of the invention will appear from the following description in conjunction with the accompanying drawing in which

FIGURE 1 is an elevational sectional view of an electrolyzing cell of the type with which the invention is particularly concerned;

FIGURE 2 is an enlarged sectional view of a part of one of the electrodes of the cell of FIGURE 1;

FIGURE 3 is a plan sectional view taken along the line 3-3 of FIGURE 1;

FIGURE 4 is a fragmentary view of one type of material employed in constructing a foraminous partition as shown in FIGURES 2 and 3;

FIGURE 5 is a fragmentary view of another type of material employed in constructing a foraminous partition as shown in FIGURES 2 and 3; and

FIGURE 6 is a view taken along the lines 6-6 of FIGURE 5.

In accordance with the invention a foraminous partition is provided which is adapted to be used within an annular electrolyzing zone of a cell for electrolyzing a sacrificial anode in a liquid electrolyte and which comprises a tubular electrically non-conducting material having filaments therein overlapping one another and running obliquely with respect to the longitudinal axis of the electrolyzing zone. The overlapping filaments are secured together at the points where they overlap. The filaments employed preferably overlap at angles of 60° and about 120°. They are preferably extruded filaments. In the preferred structure, there are outer and inner filaments. All of the outer filaments run in the same direction parallel to each other and all of the inner filaments run parallel to each other and in a different direction from the outer filaments so that the spaces between them form oblong parallelograms. Where the filaments intersect or overlap, they are secured together so as to provide a structure which is flexible transversely but relatively rigid or substantially inflexible longitudinally. In other words, the tubular structure can be deflected from the vertical plane but does not yield when pulled longitudinally.

It is usually preferable for the purpose of the invention to have the outer set of filaments somewhat larger in diameter than the inner set of filaments. Thus, the outer set of filaments can be, for example, 0.06 inch in diameter and the inner set of filaments can be, for example, 0.03 inch in diameter. A fusible or thermoplastic material is preferably used in forming the filaments and when such a material is used, the filaments are fused together at the points where they intersect or overlap. A particularly suitable material from which the filaments can be formed for the purpose of the invention is polypropylene. Polypropylene can readily be extruded to produce filaments of the desired size which are then helically wound to produce a structure of the type previously described.

In practicing the invention it is also desirable in order to prolong the life of the foraminous partition to coat the tubular electrically non-conducting material with a resin which is substantially inert under the conditions of cell operation. A suitable resin, for example, is an epoxy resin. A specific example of a suitable resin is the epoxy resin obtained by impregnating the tubular liner or partition with an epichlorhydrin-bis-phenol type polymer, such as, for example, a mixture of epichlorhydrin epoxidized dihydroxydiphenyldimethylmethane having an epoxide equivalent of 175 to 210 (Epon 828) cured with 15% by weight monohydroxypropyldiethylenetriamine at 250° F.

It is possible to employ a tubular electrically non-conducting material of the type previously described as the sole foraminous partition in contact with the cathode and the anode of the electrolyzing cell. In this case, however, the openings should be sufficiently large to permit passage of a liquid electrolyte but small enough to permit

passage of the sacrificial anode material where the latter is in particulate form, such as, lead particles, spheres or pellets. From a practical standpoint it is better to employ a first tubular electrically non-conducting material of the type previously described which is in direct contact with the cathode and a second tubular electrically non-conducting material having openings therein which is in contact with the anode particles, said second material being concentrically arranged within said first material. The second material can then be readily more flexible and have smaller openings which are preferably rectangular and this material is preferably wound so as to form at least two layers within the first tubular material. The second tubular material is preferably composed of woven filaments, such as glass fibers, or linear polyamide filaments, such as nylon. The preferred structure is one in which the first tubular electrically non-conducting material is composed of polypropylene filaments and the second tubular electrically non-conducting material concentrically disposed within the first tubular material is composed of a woven nylon cloth. Other electrically insulating materials can be employed in both the first and second materials, including, for example, polyethylene and a polymer of tetrafluoroethylene. Both the first and the second tubular materials can be impregnated or coated with a resin, preferably an epoxy resin of the type previously described. All of these insulating materials are inherently chemically inert.

The cell structure shown in FIGURE 1 does not in itself constitute a part of the invention but is given in order to illustrate one type of structure in which the invention can be employed. In the cell structure shown there is a hollow main shell 1, a second shell 2, a top end closure member 3 and a bottom end closure member 4. The main shell 1 is provided with an upper end plate 5 and a lower end plate 6. Each of the end plates 5 and 6 is substantially circular in cross section and is provided with aligned openings or apertures. Electrically conducting tubes 8 which are preferably constructed of steel are welded or otherwise secured in the apertures of the end plates 5 and 6. The number of these tubes will vary depending upon the size of the main shell 1 and the desired capacity of the unit. The cylindrical shell 9 is welded or otherwise secured in liquid-tight relationship to the end plates 5 and 6. Likewise, the metal tubes 8 are welded or otherwise secured in liquid-tight relationship to the same end plates. The space around the metal tubes 8 forms a chamber 10 into which a heat exchange liquid is introduced through an inlet opening 11 and removed or recirculated through outlets 12 and 13 which are provided with baffles 14 and 15, respectively.

The second shell 2 consists of two end plates 16 and 17 having apertures therein corresponding to the apertures in the end plates 5 and 6 of the main shell 1. A circular sheet metal housing 18 is welded or otherwise secured to the end plates 16 and 17 of the second shell 2 to form a liquid-tight enclosure. Short tubes 19, preferably made of steel and corresponding in diameter to the tubes 8, are welded or otherwise secured in liquid-tight engagement in the apertures of the plates 16 and 17 of the second shell 2. Openings 20 and 21 are provided as inlet and outlet openings to introduce and remove heat exchange fluid, if desired, or for the purpose of draining condensate from the interior of the second shell 2.

The top end closure 3 consists of a base plate 22 having apertures 7 therein. These apertures correspond in size and are aligned with the apertures in plates 5, 6, 16 and 17. A hollow metal shell 23 is welded or otherwise secured in liquid-tight engagement to the base plate 22. At the top of this shell is an inlet 24 where anode material, such as lead particles, spheres, or pellets, can be charged and also an opening 25 where the electrolyte can be introduced or withdrawn. A metal reinforcing band 26 is welded to the inside the shell 23.

The top closure member 3 is assembled with the cell by means of bolts 27 extending through bolt holes in the flanges 16 and 22. An insulating sheet 28 is placed between the end plates 16 and 22 thereby electrically insulating the end closure 3 from the second shell 2. The second shell 2 is similarly bolted to the main shell 1 by means of bolts passing through holes, not shown, in the outer ends of plates 5 and 17. An insulating sheet 29 is placed between the plates 5 and 17 thereby electrically insulating the second shell 2 from the main shell 1.

At the bottom of the cell the end closure 4 consists of a generally conically shaped housing 30 provided at the top with an end plate 31 which is welded or otherwise secured in liquid-tight engagement to the housing 30 and has apertures therein corresponding to the apertures in the plates 5, 6, 16, 17 and 22. A metal grate or screen 32 is placed over the opening 33 in the bottom of the member 4 to support the anode material and to permit the passage of liquid electrolyte without permitting the passage of the anode material. This screen or grate is held in place by means of bolts 34 or any other suitable manner. The arrangement is such that by removing the bolts 34 the screen or grate 32 can be removed to permit cleaning of the cell and removal of all of the anode material, when necessary.

A circular plate 35 with apertures therein is placed between the end plates 6 and 31 and is insulated with electrically non-conducting sheets 36 and 37. The bottom closure member 4 is assembled in liquid-tight engagement to the main shell 1 by means of bolts 38 passing through holes in the outer edges of the plates 6, 31 and 35. The cell is supported from a supporting structure 39 which is suitably mounted to provide a firm base.

The insulating members 28, 29, 36 and 37 all contain apertures corresponding in size and alignment to the openings 7 and are made of a suitable electrically non-conducting material, such as, polyethylene, polypropylene or a polymer of tetrafluoroethylene (Teflon). Extensions 40 and 41 are welded or otherwise secured to the end plates 5 and 6, respectively, and are connected to a negative source of potential, that is, a source of negative direct current suitable for operation of the cell. It is desirable to employ a number of these extensions secured to the end plates 5 and 6 at equally spaced distances. For example, in a cell of the type described in the drawings, eight such extensions are preferred.

The end closure 4 contains downwardly extending triangularly shaped members 42 which are welded or otherwise connected to the shell 30 and serve as connections to a positive source of electrical potential. Again, it is preferable to employ a plurality of these anode connections equally spaced from one another and in a cell of the type described, eight such extensions would be used.

As shown in FIGURES 2 and 3 each of the tubular electrodes 8 comprises an outer metal tube 43, a foraminous partition generally indicated at 44 and an anode material, for example, spherical lead particles, generally indicated at 45. In FIGURE 2 the tubular electrodes are shown without the anode material.

The foraminous partition 44 consists of a tubular electrically non-conducting material 46, the outer surface of which is in direct contact with the inner surface of the metal tube 43 and a second tubular electrically non-conducting material 47 which is disposed concentrically within the tubular material 46, the inner surface of which is in contact with anode material 45.

The inner tubular material 47 is preferably a woven filamentary material composed of glass filaments, polyamide (nylon) filaments or polypropylene filaments and the structure is such that the openings 48 are small enough to prevent the passage of the anode material but large enough to permit the flow of a liquid electrolyte. In a preferred embodiment as shown in the drawings, a double wrap of nylon cloth is used having a 92 x 92 thread

count and a 1/1 plain weave. The threads in this case are made from nylon monofilaments and the double wrap is so arranged that the ends wrapped together overlap each other by about one-half inch.

The foraminous tubular member 46 has openings 49 therein which can be described as oblong parallelograms. The acute angles of the parallelograms extend in a generally vertical direction. In the structure shown in FIGURE 6, there is an outer set of filaments 50 extending at an angle to the vertical and an inner set of filaments 51 which also extend at an angle to the vertical but in the opposite direction. Thus, the outer set of filaments overlaps the inner set of filaments and they are secured together at their points of intersection. It is preferable to form both sets of filaments of a thermoplastic material, for example, polypropylene, and to fuse them together at the points where they overlap or intersect. This produces an exceptionally strong structure which is flexible transversely but substantially inflexible in a vertical direction.

The foraminous partition 44 is placed inside of each of the tubular electrodes 8 by first assembling it on a short flanged tube 52 as shown in FIGURE 2. This tube 52 has a tubular portion 53 and a flanged portion 54. The foraminous member 47 is placed around the short tubular portion 53 and the foraminous member 46 is placed around the foraminous member 47 so that they are concentrically arranged and the tops of both the foraminous members 46 and 47 are in contact with the lower surface of the flange 54. They are then clamped in place by means of a clamping ring 55. If desired, the tube 53 can be slightly enlarged or flared at 56 in order to insure that the clamping ring cannot possibly slip down below the lower end of tube 53. The flanged tube 52 with the foraminous partition 44 mounted thereon is then inserted in the tubular electrode 8 so that the flange 54 sits in a recess 57 in the upper part of plate 16. The tubular member 46 is then in contact with the inner surface of the shell 43. More specifically, the outer filaments 50 of the tubular member 46 are in contact with the inner surface of the shell 43. The inner filaments 51 are in contact with the second tubular member 47 which consists of a double wrap of a woven fabric made from nylon or other suitable material. The latter in turn is in contact with the anode material which may consist, for example, of lead pellets, spheres, or particles. In a like manner, other foraminous partitions are suspended or supported in other tubes 8 within the cell. Each of these foraminous partitions extends from the plate 16 to the bottom of the plate 6 and can, if desired, extend into the lower end closure member 4.

Since the foraminous partition is constructed entirely of electrically insulating materials, it is not necessary to take precautions to keep the partition from going beyond the insulating element 37. On the other hand, if the partition were constructed even in part of electrically conducting materials in contact with the cathode any slipping or sagging of the partition might produce a short circuit. The structure of the foraminous partition provided in accordance with the invention is such that it offers the maximum resistance to stress or pressure imparted to it by the anode material. Furthermore, the arrangement of the filaments 50 and 51 in the part of the partition adjacent the cathode provides a series of channels or passageways for electrolyte whereby the electrolyte is circulated in contact with both the anode and the cathode. Thus, the foraminous partition is so constructed that it will maintain its shape while at the same time directing the flow of the electrolyte.

The invention is hereby claimed as follows:

1. In an electrolytic cell having a first electrode in the form of a tube and a second electrode in the form of particles, and a foraminous partition in contact with the inner surface of said first electrode and the outer bound-

daries of said particles, said foraminous partition comprising a tubular, chemically inert, electrically non-conductive material having openings therein formed from filaments overlapping one another, said filaments running obliquely with respect to the longitudinal axis of said first electrode, and secured together at the points where they overlap, and said tubular foraminous partition being flexible transversely but substantially inflexible longitudinally.

2. In an electrolytic cell as claimed in claim 1, a foraminous partition in which said filaments overlap at angles of about 60° and about 120°.

3. In an electrolytic cell as claimed in claim 1, a foraminous partition in which said overlapping filaments are fused together.

4. In an electrolytic cell as claimed in claim 1, a foraminous partition in which there is an outer set of filaments and an inner set of filaments, the filaments in the outer set being of larger diameter than those in the inner set.

5. In an electrolytic cell as claimed in claim 1, a foraminous partition in which said filaments are resin coated.

6. In an electrolytic cell as claimed in claim 1, a foraminous partition in which said filaments are coated with an epoxy resin.

7. In an electrolytic cell as claimed in claim 1, a foraminous partition in which said filaments are formed of polypropylene.

8. In an electrolytic cell having a first electrode in the form of a tube and a second electrode in the form of particles, and a foraminous partition in contact with the inner surface of said first electrode and the outer boundaries of said particles, said foraminous partition comprising a first tubular, chemically inert, electrically non-conducting material having openings therein in the shape of an oblong parallelogram, said openings being formed from filaments intersecting each other, running obliquely with respect to the annular electrolyzing zone, and secured together at their points of intersection, and a second tubular electrically non-conducting material having openings therein, said second material being concentrically arranged with respect to said first material.

9. In an electrolytic cell as claimed in claim 8, a foraminous partition in which said second material comprises rectangular openings of smaller size than the openings in said first material, said openings in said second material being sufficiently large to permit passage of a liquid electrolyte but small enough to prevent passage of sacrificial anode particles.

10. In an electrolytic cell as claimed in claim 8, a foraminous partition in which said second material is wound to form a double thickness of said material.

11. In an electrolytic cell as claimed in claim 8, a foraminous partition in which said second material is a woven fabric made from linear polyamide filaments.

12. In an electrolytic cell as claimed in claim 8, a foraminous partition in which said first material consists of helically wound polypropylene filaments and said second material consists of a fabric woven from linear polyamide filaments.

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