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NOTICE OF ENTITLEMENT

675122

We KEELE UNIVERSITY

of Staffordshire ST5 5BG, UNITED KINGDOM

being the Applicant and Nominated Person, in respect of Application No. 62169/94, entitled SOLID OXIDE FUEL CELL STRUCTURES state the following:

Kevin Kendall is the actual inventor of the invention.

The inventor made the invention for and on behalf of Keele University whilst on secondment at Keele Univerisity. By virtue of an agreement between Keele University and the inventor's then employer, it was agreed that all inventions, including the present invention, made by the inventor during his time of secondment at Keele University would be the property of Keele Univerisity. Further, a confirmatory assignment between the inventor's then employer and Keele University has been executed to confirm the transfer of rights in the invention to Keele University.

Convention priority is claimed from the following basic applications:

Basic Applicant	Application Number	Application Date	Country	Country Code
KEELE UNIVERSITY	9305804.8	20 March 1993	United Kingdom	GB
KEELE UNIVERSITY	9305823.8	20 March 1993	United Kindgom	GB

The basic applications were the first applications made in a Convention country in respect of the invention the subject of this request.

DATED this 21st day of November 1996

KEELE UNIVERSITY
By their Patent Attorney

GRIFFITH HACK

OUR REF: P24022-A/RPW:SLC

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(56) Prior Art Documents
US 5106706
GB 1216024
US 3377203

Abstract

(57) Claim

1. A tubular SOFC structure comprising a self-supporting extruded tube a longitudinally extending portion of which is formed from an electrolyte, an electrically conductive inner electrode making electrical contact with the inner wall of the electrolyte, an electrically conductive outer electrode making electrical contact with the outer wall of the electrolyte, a thermally insulating enclosure defining a wall through which the tube extends such that a first portion of the tube extends within the enclosure and a second portion of the tube extends outside the enclosure, a first gas supply conduit connected to the end of the second portion of the tube by a gas-tight seal located outside the enclosure, a second gas supply conduit which opens within the enclosure, one of the first and second gases being a fuel gas and the other containing oxygen, and means for heating the

.../2

interior of the enclosure to a temperature at which the structure operates as a solid oxide fuel cell.

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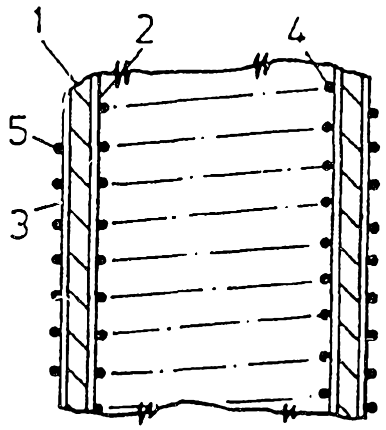

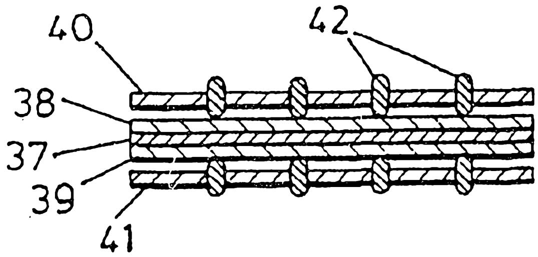

(57) Abstract

Solid oxide fuel cell structures which are capable of relatively rapid temperature changes without cracking and which are simple to seal. In one arrangement a tubular SOFC structure comprises a self-supporting extruded tube of zirconium oxide with inner and outer electrodes. The tube may have an outside diameter of from, for example 1 to 5mm and a wall thickness of from, for example, 50 to 200 microns. In an alternative arrangement, a simple gas type planar interconnect for a planar SOFC is provided in the form of a sheet of ceramic material having electrically conducting bodies of ceramic material embedded in it so as to provide an electrical path through the sheet. To avoid edge sealing problems, fuel gas and air may be delivered to a stacked SOFC structure through tubes extending between adjacent cell sub-assemblies such that the gas is delivered to a central portion of each anode of the cell stack and flows outwards towards the edges of the stack.

SOLID OXIDE FUEL CELL STRUCTURES

The present invention relates to solid oxide fuel cell (hereinafer referred to as SOFC) structures.

SOFCs of three basic designs are currently being developed. These three basic designs are generally referred to by the term tubular, planar and monolithic. All of these fuel cells are based upon a stabilised zirconia electrolyte which is capable of conducting oxygen ions at elevated temperatures. A typical operating temperature for a SOFC is 1000°C. The known cells provide high electrical efficiencies and can be operated on a variety of fuels including hydrogen, carbon monoxide, coal-derived gases and natural gas. SOFCs also offer high quality exhaust heat for co-generation applications. Of potentially the greatest significance, however, is the fact that SOFCs produce very low emissions as compared with, for example, diesel generators and therefore can be located wherever an electrical generator is required. For example, it would be possible to replace a relatively dirty and noisy diesel generator providing power to a hospital by a SOFC.

The paper "Solid Oxide Fuel Cells - The Next Stage", author Brian Riley, pages 223-238 of "Journal of Power Sources", 29(1990) briefly describes the various known SOFC structures and reference should be made to that document for details of the structure of the known planar, monolithic and tubular geometries. It will be appreciated, however, that all of the known structures incorporate a solid electrolyte one side of which supports an anode to which fuel gas is delivered and the other side of which supports a cathode to which air or oxygen is delivered. When an external load is connected to the anode and cathode, oxygen at the cathode reacts with incoming electrons from the external circuit to form oxygen ions which migrate to the anode through the oxygen-ion conducting electrolyte. At the anode, the fuel is oxidised with these oxygen ions, resulting in the liberation of electrons to the external circuit. Thus the overall reaction is simply the oxidisation of fuel. Typically 50 to 90% of the fuel is utilised in the electrochemical cell reaction, the partially depleated fuel being combusted outside the cell. The exhaust gas from the cell can be used in a co-generation system for producing process steam or in a steam turbine for an all-electric system. As each cell has a theoretical open voltage of about 1 volt it is necessary to interconnect a number of cells to provide an appropriate output voltage.

If SOFCs are to be widely useable, they must be very reliable over long term use. The known cells are prone to two problems which compromise long term reliability, the first problem being the fact that fuel cell structures are very easily damaged if subjected to thermal shocks, and the second problem being related to the difficulty experienced in sealing the fuel cell structures so that fuel and oxygen are reliably delivered to opposite sides of a relatively thin electrolyte and do not come into contact with each other until the fuel has been at least significantly depleated. It has proved difficult to deal with these problems given the high operating temperatures and the fact that it is fundamental to the operation of fuel cells that thin ceramic structures form the interface between the anode and cathode. Such structures crack easily when exposed to varying temperatures. The conventional approach to reducing the significance of these problems is to very slowly heat up the fuel cell structure to the normal operating temperature of 1000°C and to maintain that temperature continuously. Unfortunately in the real world continuous operation of a system cannot be guaranteed. Until such time as manufacturers can assure potential users that, for example, a power failure resulting in rapid cooling of a SOFC would not cause any structural damage to such a cell it is going to be very difficult to convince potential customers that SOFCs are a viable alternative to conventional electrical generation systems.

In the case of conventional tubular electrode structures, the basic cell is in the form of a porous support tube onto which a cathode or air electrode is deposited as a layer by slurry-dipping. The electrolyte is then deposited on the cathode by electrochemical vapour deposition or plasma spraying. The anode or fuel electrode is then formed on the electrolyte by slurry dipping. Electrical connections are made to the anode by a simple nickle felt pad applied to its outer surface. Electrical connections are made to the cathode by forming an elongate strip of electrical conductor along the length of the tube, the electrolyte not covering the electrical conductor.

Tubes can thus be interconnected by appropriately positioning them with the cathode and anode interconnects of adjacent tubes in contact. The exterior surface of the tube is exposed to fuel gas, and air is pumped into the interior through an air tube extending along most of the length of the tube from one end of the tube. The other end of the tube is closed so that injected air flows back through the annular space defined between the air tube and the support tube.

The known tubular arrangement is effective but cannot be heated or cooled rapidly without cracking. The incorporation of the axially extending air tube simplifies the problem of sealing, as a seal can be made relatively easily to the relatively cool air tube, and in addition enables some preheating of the air delivered to the interior of the support tube, thereby reducing thermal shocks. Unfortunately, providing the air tube increases costs substantially both because of increased material costs and because the structure is relatively difficult to manufacture. Furthermore, the deposition of the electrolyte and interconnect on the cathode is a very costly process step.

The manufacturers of the known tubular structures have recognised that the incorporation of the support tube accounts for some 70% of the total weight of the cell which results in a relatively low energy density for the design. With a view to reducing the overall weight, it has been proposed to replace the calcia-stabilised support tube by a self-supporting cathode to improve the energy density. This approach may improve the energy density but does not address the problems of cracking or cost outlined above.

It is an object of the present invention to obviate or mitigate the problems outlined above.

According to the present invention there is provided a tubular SOFC structure comprising a self-supporting extruded tube a longitudinally extending portion of which is formed from an electrolyte, an electrically conductive inner electrode making electrical contact with the inner wall of the electrolyte, an electrically conductive outer electrode making electrical contact with the outer wall of the electrolyte, a thermally insulating enclosure defining a wall through which the tube extends such that a first portion of the tube extends within the enclosure and a second portion of the tube extends outside the enclosure, a first gas supply conduit connected to the end of the second portion of the tube by a gas-tight seal located outside the enclosure, a second gas supply conduit which opens within the enclosure, one of the first and second gases being a fuel gas and the other containing oxygen, and means for heating the interior of the enclosure to a temperature at which the structure operates as a solid oxide fuel cell.

By relying upon the electrolyte structure itself to be self-supporting the tube may be made with a sufficiently small diameter and small wall thickness to make it highly resistant to cracking. For example a tube of this structure can be heated at any point along its length with a very localised flame to the normal operating temperatures of an SOFC (typically 1000^{0}C) without incurring any damage. Thus an SOFC based on such tubular components can be rapidly heated and cooled without damage, dramatically improving the reliability of such systems and making them useable in many more applications, for example as emergency power supplies.

Further preferred features of the invention are referred to in attached claims 2 to 19.

Embodiments of the present invention will now be described, by way of example, with reference to the accompanying drawings, in which :

Figure 1 is a section through a tubular SOFC structure which may be used in accordance with the present invention;

Figure 2 is a section through the structure of Figure 1 after the structure of Figure 1 has had internal and external spiral wires attached to it;

Figure 3 is a schematic illustration of an SOFC system in accordance with the present invention which has been used to provide the utility of the tubular structure illustrated in Figures 1 and 2;

Figure 4 illustrates a fuel cell system which may be fabricated using a plurality of the tubular structures illustrated in Figures 1 and 2;

Figure 5 illustrates the structure of a further embodiment of the invention with a co-extruded internal electrode;

Figure 6 illustrates an alternative arrangement to that of Figure 5 with a co-extruded electrical interconnect provided in the wall of the tube to enable the interconnection of a series of tubular structures;

Referring to Figures 1 and 2, the illustrated arrangement comprises an extruded electrolyte tube 1 supporting an inner electrode 2 in the form of a nickel/zirconia cermet ink defining an anode. A strontium doped lanthanum manganite layer 3 is formed on the outside of the tube to define a cathode or air electrode. As shown in Figure 2, electrical contact is made with the internal electrode 2 by winding a spiral wire 4 inside the tube. Electrical contact to the outer electrode 3 is similarly achieved by winding a spiral wire 5 onto the layer 3. If such a tube is placed in an oxygen-containing enclosure maintained at about 1000^{0}C and fuel gas is supplied to the interior of the tube a solid fuel cell (SOFC) is formed as a result current will flow to any external circuit connected to wire 4 and 5.

Tubes of the type described have been found to be easy to manufacture, to resist cracking even if heated or cooled rapidly, and to be easy to seal. The tubes can be readily extruded using a mix of, for example, zirconia powder and polymer formulation. After firing to full density, these tubes can be heated to 1000^{0}C rapidly and cooled equally rapidly without damage. Furthermore, the tubes can be passed through an insulating layer defining the wall of a chamber, the interior of which is maintained at 1000°C even though one end of the tube is at room temperature and the other end is at the temperature of the enclosure. Cold gas can then be passed down the tube without causing it to crack. Sealing the tube on the cold end is simple, for example using simple plastics connections. Thus the problems associated with known tubular SOFC structures are overcome.

The thin walled zirconia tube may be made from stabilised zirconia, for example using yttria stabiliser. Between 3 and 12 mol% may be used although preferably from 6 to 10 mol% is used and the preferred amount of stabiliser is 8 mol%. It will be appreciated that stabilisers other than yttria may be used, for example magnesia, calcia, ceria, alumina and others known in the art. The outside diameter of the tube may be from 1 to 5mm, although other sizes are possible. The tube need not necessarily be round, as fluted or "wavy" cross sections can prove to be advantageous and are readily extrudable. The zirconia wall thickness is preferably between 50 and 200 microns (10^{-6} metres) to allow for the ready passage of oxygen ions during fuel cell operation. If the zirconia is extruded with the anode, the zirconia could be relatively thin, for example from 5 to 10 microns. The inner and outer electrodes may be deposited in the form of inks containing active powders. The inks are sintered and then electrical connections are made to them to enable current to be drawn from the cell. Metal or ceramic components may be used to make the electrical connections, for example the illustrated wires shown in Fig. 2. Each tube may form a single cell, although several cells could be positioned on each tube. In such a case, interconnects would have to be positioned along the length of the tube. Alternatively, a line of interconnect can be formed in the wall of the tube and this can be achieved by extrusion in a single shot process.

Many tubes must be interconnected to make an SOFC assembly of reasonable output power. The necessary interconnections may be achieved in a number of ways. For example, each tube may be a single cell which is connected externally to neighboring cells, or each tube may form multiple cells with internal interconnections along the length of the tube, each tube then being connected externally to other tubes.

As a further alternative each tube may be a single cell which is connected internally to surrounding tubes by means of interconnect strips extending along the lengths of the tubes. Tubes may be connected in series or parallel or in combinations of series and parallel connections. The tubes will normally be straight and parallel, but could also be curved or shaped so as to channel gas in desirable directions. Fuel gas may be delivered either to the inside or the outside of the tubes and residual fuel gas may be recycled to the fuel gas inlet.

A tube of the type illustrated in Figs. 1 and 2 was fabricated using 8 mol% yttria stabiliser zirconia powder which was mixed with polyvinyl butyral and cyclohexanone in proportions by weight of 100/8/9. The composition was mixed intensively in a Dow mixer to break down any agglomerates. The plastics mix was pressed into a sheet under 5 MPa pressure and then extruded in a tube die to give a 5mm diameter tube of 0.2mm wall thickness. After drying, the tube was supported inside an alumina tube of slightly larger diameter and fired in a furnace. The polymer binder was first removed at 1°C per minute until a temperature of 500°C was achieved, and then the tube was sintered to 1500°C at 5°C per minute and held at that temperature for one hour. Nickel/zirconia cermet ink was coated on the inside of the tube to form the anode and strontium doped lanthanum manganite was coated on the outside to form the air electrode. These electrodes were fired at 1200°C with platinum wires attached as current leads.

The resultant tube was then placed in a furnace of the type schematically illustrated in Fig. 3. The tube 6 was passed through a bore drilled in a thermally insulating plug 7 inserted in the top of a tubular body 8 of insulating material. An exhaust outlet tube 9 was also positioned in the plug 7. The tubular body 8 was supported on a base 10. An air inlet tube 11 extended into the lower portion of the enclosure defined within the insulating body.

A rubber tube 12 was connected to the upper end of the SOFC tubular structure 6. Fuel gas was then passed through the tube 12 and the tube 6 into the enclosure and burnt with air delivered through tube 11. When the temperature within the enclosure had reached 1000°C the tube was seen to act as an SOFC. Turning off the supply of fuel gas resulted in a rapid fall in temperature within the enclosure but this did not damage the tube 6, nor did the tube suffer any damage as a result of the large temperature differentials between its two ends.

Fig. 4 illustrates a proposed system incorporating tubes of the type illustrated in Figs. 1 to 3 in an assembly capable of providing reasonable power output levels. An array of tubes 13 is supported within a thermally insulating container 14 from which combustion products can escape through exhaust passageway 15. Fuel gas is supplied to the container 14 through a valve 16, a manifold 17 and the tubes 13, the bottom ends of which simply open into the interior of the container. Air is supplied to the container from a blower fan 18 via an inlet pipe 19 that extends through the exhaust outlet 15. The air is pre-heated as it passes through the tube 19. A water heat exchanger 20 is also provided in the exhaust outlet 15 to extract heat energy from the exhaust gas stream.

The internal electrodes (not visible in Fig. 4) within the tubes 13 are connected to a negative terminal 21 of a battery 22 whereas the outer electrodes of the tubes 13 are connected to the positive terminal 23 of the battery 22. The output of the battery is converted from DC to AC by a converter 24 to deliver the required AC output as represented by arrow 25. The system is controlled by a controller 26 that monitors the condition of the battery, the temperature within the container 14 as indicated by a thermocouple connected to terminal 27, and the operation of the DC to AC converter 24. In addition the controller 26 energises the valve 16 and an igniter positioned within the container 14 and connected to terminal 28.

The illustrated system was designed to provide both electric power and heat in the 0.2 to 20 kWe scale. The system is well integrated to give the smallest number of parts, and those parts are designed to enable the system to be switched on and off as required without damage. Such a system is ideally suited to replace conventional power generators such as diesel or turbine engines.

The tubes 13 are fed with fuel from the manifold 17. The rate of supply of gas is controlled by the valve 16 in response to control signals generated by the controller 26. The controller also controls the blower 18, the speed of which is modulated in response to variations in the temperature within the container 14 as monitored by the thermocouple. As the air and fuel are brought together inside the container, the gas is ignited by the igniter. Gas flows could be typically in the range of 10 to 1000 ml/s, while air flows are typically in the range of 100 to 10,000 ml/s.

The output of the blower 18 is connected by an air bleed line 29 to the gas supply line. Mixing air with the supplied fuel gas prevents the formation of carbon within the tubes 13. This is important as given that the tubes have small internal diameters they could easily be blocked by carbon deposits. The rate of supply of air through the bleed line 29 will typically be the same as the rate of supply of fuel gas.

The detailed geometry of the assembly of tubes 13 may vary depending on the size and purpose of the device. For example, a small device may contain 20 sub-units each containing 30 short tubes 13. A larger device may contain 40 sub-units each containing 100 long tubes. The overall layout of the system will, however, remain the same in both cases.

It will be appreciated that in larger installations several air feed tubes may be appropriate, and that the water heater may be omitted in certain applications, for example where there is a need for hot gas to drive a heater or chiller.

In the arrangement of Fig. 4, partially depleated fuel gas is simply burnt within the container 14, the combustion products leaving the container through the exhaust 15. It would be possible, however, to re-cycle partially depleated fuel, for example simply by passing the tubes 13 through the bottom wall of the container 14 and recycling the gas within them to the manifold 17. This approach would enable steam reforming to take place, again with a view to preventing carbon build up in the system. Of course, no problems will arise with carbon if the fuel gas is, for example, hydrogen or methanol.

As briefly mentioned above, alternatives are available to the tubular structure illustrated in Figs. 1 and 2. In particular, advantages will arise from reducing the number of process steps and the number of components required to make any particular tube. As illustrated in Fig. 5, the process steps necessary to form the inner electrode shown in Figs. 1 and 2 can be avoided by the simple expedient of co-extruding the zirconia electrode 30 with an anode 31.

The anode could be formed from zirconia and nickel and would enable current to be carried along the length of the tube. Such an arrangement would avoid the need for depositing an ink inside the ceramic tube shown in Figure 2 and for providing a contact wire within the tube.

With the arrangements of Figures 1 and 5 current is taken from the internal electrode to one end of the tube. Alternative arrangements are, however, possible and one such arrangement is illustrated in Figure 6. In this arrangement three components are extruded in a single step, that is a zirconia oxide electrolyte 32, an electrically conducting interconnect 33, and an internal electrode 34. An outer nickel-containing electrode 35 is then formed on the tube in a position so that it does not contact the interconnect 33. Adjacent tubes of an identical structure can then be interconnected in series as shown in Figure 6 by sandwiching a nickel felt pad 36 between adjacent tubes.

CLAIMS

1. A tubular SOFC structure comprising a self-supporting extruded tube a longitudinally extending portion of which is formed from an electrolyte, an electrically conductive inner electrode making electrical contact with the inner wall of the electrolyte, an electrically conductive outer electrode making electrical contact with the outer wall of the electrolyte, a thermally insulating enclosure defining a wall through which the tube extends such that a first portion of the tube extends within the enclosure and a second portion of the tube extends outside the enclosure, a first gas supply conduit connected to the er of the second portion of the tube by a gas-tight seal located outside the enclosure, a second gas supply conduit which opens within the enclosure, one of the first and second gases being a fuel gas and the other containing oxygen, and means for heating the interior of the enclosure to a temperature at which the structure operates as a solid oxide fuel cell.

2. A tubular SOFC structure according to claim 1, wherein the first gas is fuel gas and the second gas is air, the tube terminates in an open end

located within the enclosure, and an exhaust conduit is provided for conveying combustion products from the enclosure.

3. A tubular SOFC structure according to claim 1 or 2, wherein the self-supporting tube is made from stabilised zirconia.

4. A tubular SOFC structure according to claim 3, wherein the stabiliser is yttria.

5. A tubular SOFC structure according to any preceding claim, wherein the inner electrode extends the length of the extruded tube.

6. A tubular SOFC structure according to claim 5, wherein the inner electrode is a spiral wire in contact with a porous conductive layer formed by depositing a conductive ink inside the tube.

7. A tubular SOFC structure according to any preceding claim, wherein the outer electrode comprises a porous layer of doped lanthanum manganite.

8. A tubular SOFC structure according to claim 7, wherein the outer electrode comprises a spiral wire in contact with the lanthanum manganite.

9. A tubular SOFC structure according to any one of claims 1 to 5, wherein the inner electrode is extruded as part of the self-supporting tube.

10. A tubular SOFC structure according to claim 9, wherein the inner electrode is formed from a mixture of nickel and zirconia oxide.

11. A tubular SOFC structure according to any preceding claim, wherein the self supporting tube is made from electrolyte and a longitudinally extending strip of electrically conductive material that extends radially through the tube wall and makes contact with the inner electrode.

12. A tubular SOFC structure according to any preceding claim, wherein the extruded tube has an outside diameter of from 1 to 5 mm.

13. A tubular SOFC structure according to any preceding claim, comprising a pre-heater heated by combustion products in the gas supply conduit through which the gas containing oxygen is supplied.

14. A tubular SOFC structure according to any preceding claim, comprising means for mixing air with the fuel gas before it is delivered to the gas supply conduit through which the fuel gas is supplied.

15. A tubular SOFC structure according to any preceding claim, comprising a water heat exchanger to extract heat from combustion products.

16. A tubular SOFC structure substantially as herein described with reference to the accompanying drawings.

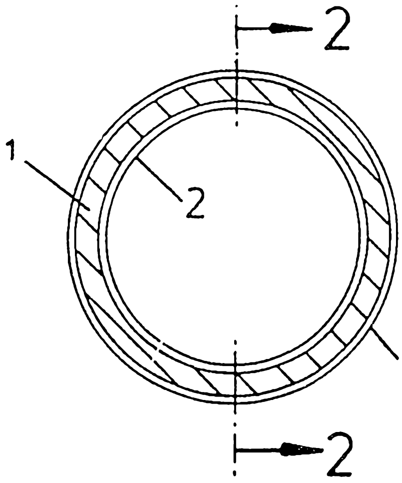

FIG.1

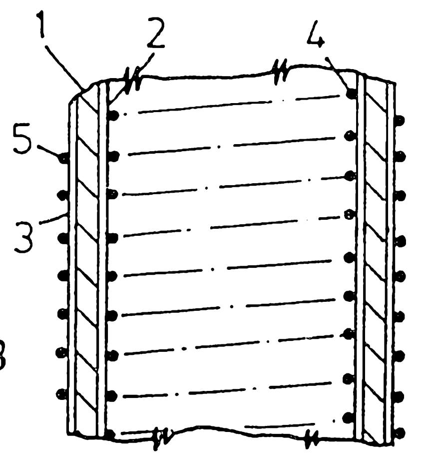

FIG.2

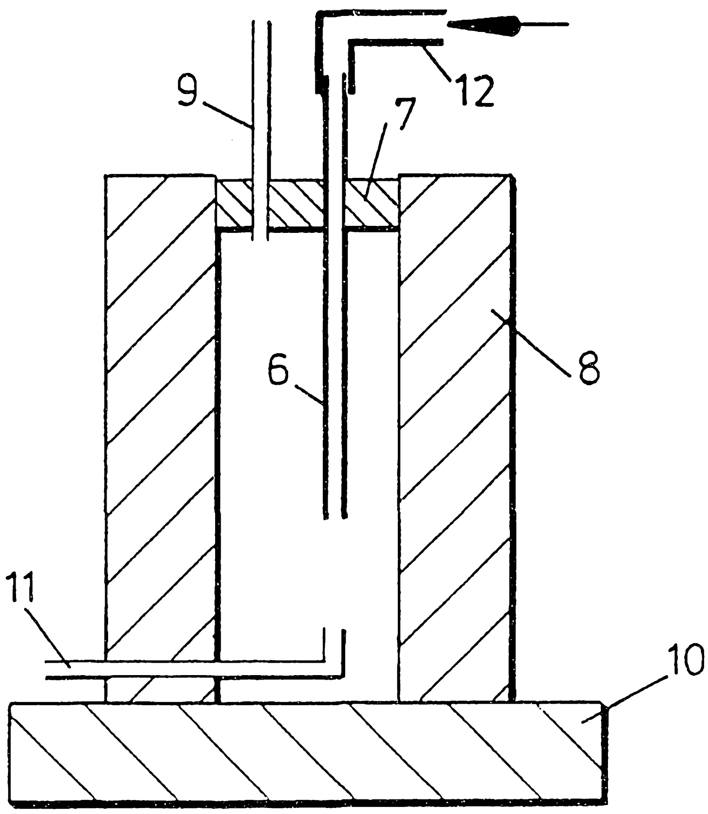

FIG.3

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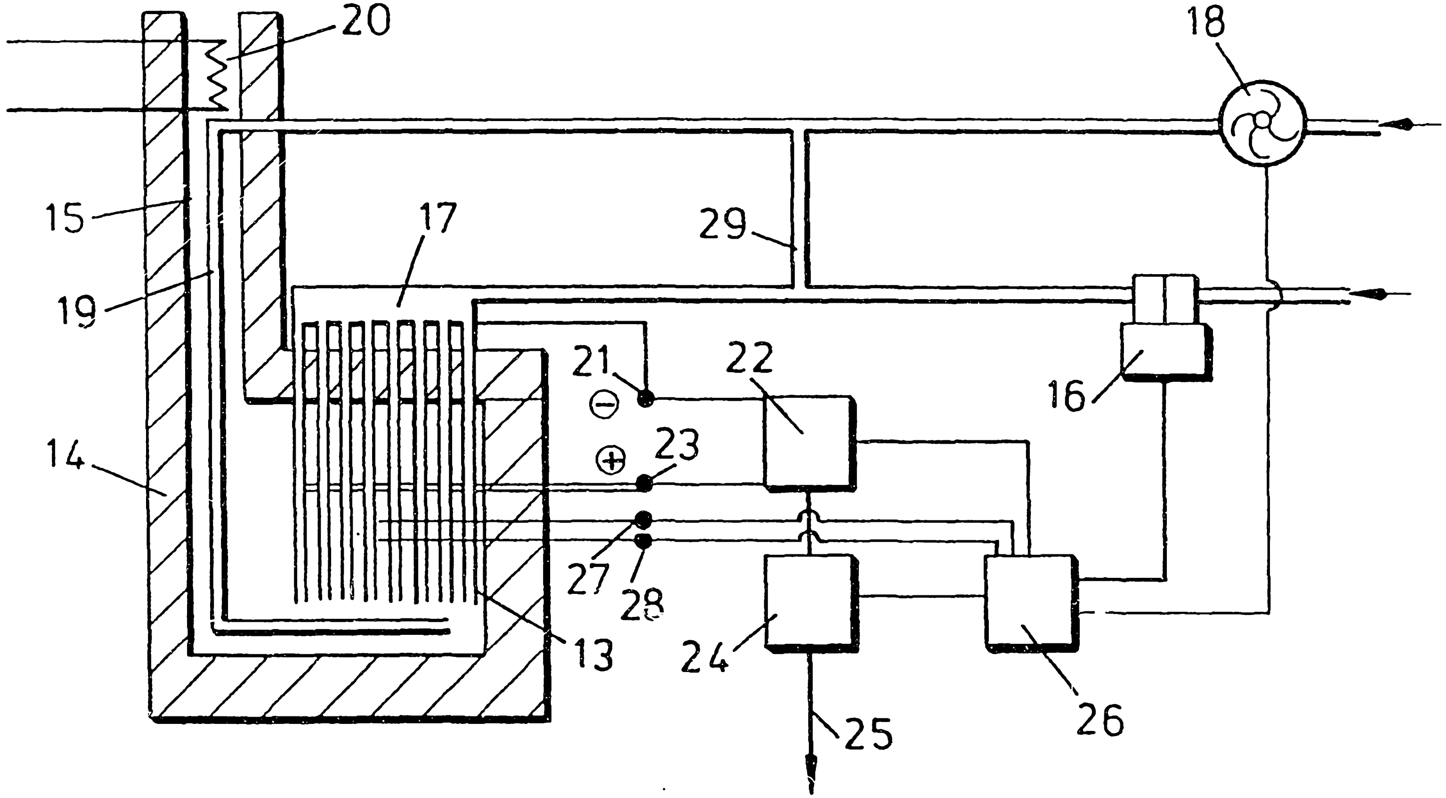

FIG. 4

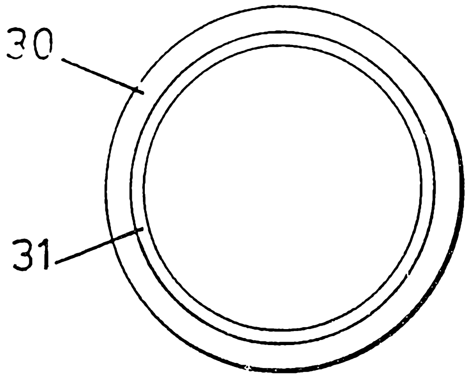

FIG.5

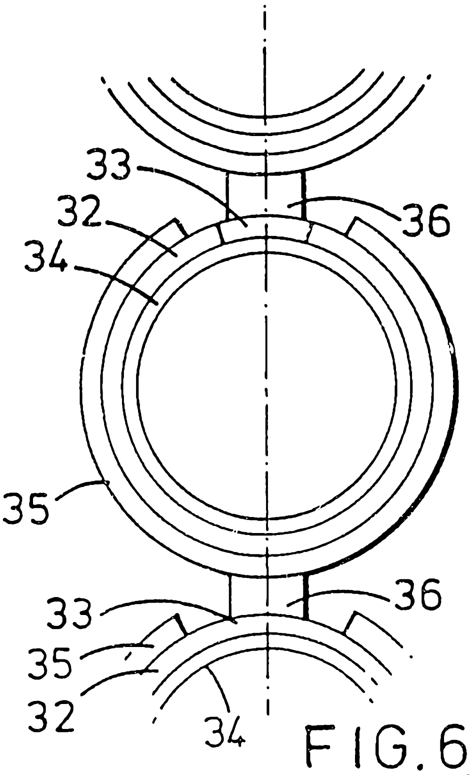

FIG.6