(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 072 525**
A1

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: 82107252.7

(22) Date of filing: 10.08.82

(51) Int. Cl.³: C 21 D 1/46
H 05 B 3/60

(30) Priority: 10.08.81 JP 124950/81

(43) Date of publication of application:
23.02.83 Bulletin 83/8

(84) Designated Contracting States:
CH DE FR GB IT LI SE

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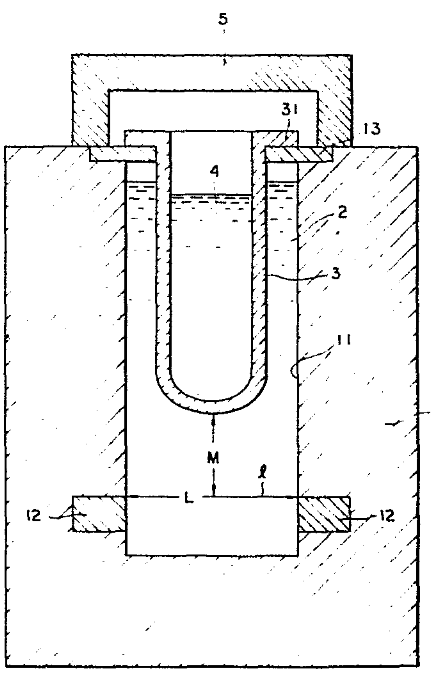
(54) **Indirect heating furnace for the surface treatment of a metal or the like employing a salt bath.**

(57) An indirect heating furnace for the surface treatment of a metal or the like, employing a salt bath, comprises a furnace body (1), provided with a concave portion (11) containing a thermal medium (2) mainly composed of a chloride, and a heat-resistant vessel (3) placed in the concave portion, which contains a treatment material (4) mainly composed of a borate, and at least one pair of electrodes (12) embedded in the side wall of the concave portion (11) adjacent to the bottom portion thereof. The electrodes heat the thermal medium which in its turn heats the surface treatment material. The temperature of the surface treatment material is easily raised within a short period of time so as to extend the actual useful life of the treatment material even at a temperature as high as about 1,200°C.



EP 0 072 525 A1

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INDIRECT HEATING FURNACE FOR THE SURFACE TREATMENT OF A METAL OR THE LIKE EMPLOYING A SALT BATH

The present invention relates to a heating furnace for surface treatment of a metal or the like, employing a salt bath a main ingredient whereof comprises a borate such as borax or the like. In particular, the present invention is intended to provide a heating furnace suitable for maintaining the salt bath at a high temperature in the range of or above 1,100°C.

Conventionally, direct heating furnaces with inside or outside heating, for instance, have been used for heat treatment employing a salt bath, such as quenching and tempering of steel, etc. The outside-heated furnace is provided with a heating source which is supported, for instance, on brick, on the outer side of a vessel containing the salt bath. In the inside-heated furnace, a plurality of electrodes is placed into the salt bath whereupon electric current is applied across the electrodes to heat the bath by electrical resistance heating.

On the other hand, a salt bath which employs a borate such as borax or the like, as its main ingredient is composed of the borate and a specified additive like ferro-boron or

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ferro-vanadium, for example. A metal to be treated is immersed into this bath and maintained there while the electrolytic treatment may be carried out on the metal. As a result, boron, vanadium or the like diffuses and permeates into the surface of the treated metal, thereby forming a surface layer of boride or carbide.

Usually, for baths employing a borate as its main constituent a furnace with outside-heating is used. Use of an inside-heated furnace with a borate bath shortens the life of the furnace because the borate has an extremely corrosive effect on the refractory material and/or the electrodes.

While a furnace with outside-heating does not have the disadvantages explained in relation to the inside-heated furnace, it requires a long time to raise the temperature. Moreover, a salt bath vessel used in this furnace must offer particularly good resistance to oxidation and high-temperature corrosion because the outer wall of the furnace is heated in air or in a combustion-gas atmosphere for a long time. Simultaneously, high-temperature strength is also required to withstand the weight of the vessel itself or of the salt bath. In particular, when the salt bath is at a high temperature, e.g. on the order of 1,100°C or more, the aforesaid characteristics for the salt bath vessel are definitely indispensable. The number of materials for the vessel, which satisfy these demands on an industrial scale, is very limited and the materials extremely expensive. Moreover, another important problem of the outside-heated furnace resides in the fact that the temperature of the furnace can be increased at a low rate only, and this disadvantage of the furnace is aggravated when the required temperature is high and there is only a small difference between the temperature of a heat source and the heating temperature of the furnace. Therefore, in this case, the time required to raise the salt bath temperature becomes so long that, compared to the life of the salt bath, it is no longer a negligible factor on an industrial scale. In general, the life of a salt bath employing a borate as its main constituent rapidly decreases as the temperature of the used bath increases. This phenomenon varies remarkably with the composition of the bath. For example, when a vanadium-carbide coating bath, which is prepared with addition of a ferro-vanadium powder to borax, is used at a temperature of 1,200°C, the life of the bath is less than 10 to 20 hours; therefore, a heating time of several hours to reach the operating temperature is no longer negligible in industry.

The invention as claimed is intended to remedy these drawbacks. It solves the problem of how to design a heating furnace employing a borate bath for surface treatment of metal or the like which allows a high temperature of the borate bath, which allows this temperature to be reached in a short period of time and which has a long life time.

Further objects of the invention are, that due to the fact that the temperature of a surface treatment material is easily raised within a short period of time so that time for which the treating material is actually usable, is prolonged; the furnace is low in cost as a low-grade material can be used, and consumption of the thermal medium can be reduced; an improved operating environment may be created by the use of a cap or lid on the furnace; and a compact heating furnace is provided having electrodes disposed at predetermined positions within the inner wall of the furnace body.

The indirectly heated furnace according to the present invention, which employs a salt bath and is useful for surface treatment of a metal or the like, comprises: a) a furnace body made of a non-conductive refractory material and having a concave portion with an open top end, for containing a thermal medium mainly composed of a chloride; b) a heat-resistant vessel having an upper opening, positioned in said concave portion and supported at the upper portion of said furnace body, in a manner to be replaceable, said vessel containing a treatment material mainly composed of a borate for surface treatment of a metal or the like; and c) at least one pair of electrodes provided in said furnace body and arranged at a specified location in the inner wall of said furnace body, which defines said concave portion, said at least one or more pairs of electrodes being so located that a line connecting the upper ends of the electrodes of each pair is located below said heat-resistant vessel, and that the smallest distance between said line connecting the upper ends of the electrodes of that pair, which is located nearest to said heat resistant vessel, and said heat-resistant vessel is not less than 1/4 of the distance between said pair of electrodes.

The figure illustrates a sectional view showing the heating furnace according to the present invention.

The indirect-heating furnace according to the present invention comprises: a furnace body 1 as the heating portion, which is made of a non-conductive refractory material and provided with a concave portion 11 opening at the upper end of the body thereof, which contains a thermal medium 2 mainly composed of a chloride; a heat-resistant vessel 3 supported at the upper portion of the concave portion 11 in a manner to be replaceable, and containing a treatment material 4 for the surface treatment of a metal or the like. The surface treatment material 4 comprises a borate as its main component. Within the wall portion of the furnace body 1, which defines the concave portion 11, at least one pair of heating electrodes 12 is embedded. The electrodes are arranged such that the line ℓ connecting the upper ends of each electrode of a pair of electrodes is located below the heat-resistant vessel 3; the smallest distance M between the aforesaid line ℓ of that pair located nearest to said vessel 3 and the heat-resistant vessel 3 is not less than 1/4 of the distance L between the aforesaid two electrodes 12.

Like any other conventional furnace, the furnace body 1, which constitutes the heating furnace according to the present invention, is made of a refractory material, such as alumina or the like. The concave portion 11 in the furnace body 1 is axially arranged at the center to contain the thermal medium 2. In a heating furnace as that shown in the figure, the concave portion 11 has a hollow round or square shape so as to have a deep bottom. Furthermore, a pair of electrodes 12 is embedded in the side wall of the concave portion 11 adjacent the bottom portion thereof. These electrodes 12 are made, for instance, of heat-resistant steel, and are connected to an external electric power source (not shown). A reinforcing ring 13, made, for instance, of heat-resistant steel, is mounted at the upper end portion of the concave portion 11, too.

The concave portion 11 of the furnace body 1 contains the thermal medium 2. Chlorides, such as sodium chloride (NaCl), barium chloride ($BaCl_2$) and the like, are suited for use as the thermal medium 2. These thermal media are in a sufficiently liquid state at temperatures in excess of 800°C. On the other hand, when the thermal medium 2 is a solid its conductivity is inadequate for an electric-resistance type exothermic element. Therefore, in such cases, an exothermic element, such as a nichrome wire or the like, is provided at the bottom of the concave portion 11 to heat and melt the solid thermal medium. When the thermal medium 2 has been melted a voltage is applied across the electrodes 12 to heat the thermal medium 2 by the heat generated by the electric resistance of the thermal medium 2 itself. In the course of the process, the exothermic element is removed from the concave portion 11 when the thermal medium becomes melted. If the thermal medium 2 cools down the exothermic element should be re-introduced into the concave portion 11 before the thermal medium solidifies.

The heat-resistant vessel 3 is made of heat-resistant steel, for example, and is introduced into the melted thermal medium 2 contained in the concave portion 11 of the furnace body 1. In the heating furnace shown in the Figure, the heat-resistant vessel 3 is provided with a flange portion 31 at its upper end, and has a cylindrical shape. The vessel 3 is inserted into the reinforcing ring 13 of the concave portion 11, and is supported by that ring 13 at its flange portion 31. The position of the heat-resistant vessel 3 relative to the electrodes 12 is defined as follows: The lowermost part of the heat-resistant vessel 3 is positioned above the line ℓ which connects the upper ends of the two electrodes 12. The smallest distance M between the aforesaid line ℓ and the heat-resistant vessel 3 is not less than 1/4 of the distance L between the electrodes. Furthermore, the distance between the inner wall of the furnace body 1, which defines the concave portion 11, and the outer wall of the heat-resistant vessel 3 is not less than 50 mm. The opening of the concave portion 11, which is defined by the inner wall of the furnace body 1 and the outer side wall of the heat-resistant vessel 3, is sealed by the reinforcing ring 13 and the flange portion 31 of the heat-resistant vessel 3. Moreover, a pipe for introduction of an inert gas may be provided at any portion desired of the upper part of the concave portion 11.

The surface treatment material 4 is contained in the heat-resistant vessel 3. A borate is the main constituent of the surface treatment material. Borax has proved to be the most practical compound for this purpose. The surface treatment material 4 is prepared by adding to the borate boron carbide, ferro-boron, ferro-aluminum or the like, or a IVa, Va or VIa group element of the Periodic Table, such as niobium, chromium, vanadium, titanium or the like.

In the surface treatment material, one or more of the following compounds may be admixed to the borate: a halide, such as sodium chloride (NaCl), potassium chloride (KCl) and sodium fluoride (NaF); an oxide, such as phosphorus oxide (P_2O_5); a hydroxide, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH); a sulfate; a carbonate; and a nitrate; so as to vary the viscosity of the surface treatment material. The surface treatment material 4 is heated to melt the borate, such as borax or the like. The boron source or the metal element source is dissolved in the molten boride.

The borate may fuse at a relatively low temperature, and acts as a kind of flux capable of keeping the surface of the article to be treated clean while preventing formation of an oxide film thereon. A substantial amount of a boride-forming or a carbide-forming element may be fused in a borate bath melt with one or more of such elements being added in its element or alloy form, e.g.

a ferro-alloy. In practice, however, the carbide-forming element is added, for instance, to the fused treatment material in a proportion of about 1 to 50 % by weight based on the total weight of the treatment material. The additive for forming a boride is admixed to the fused treatment material in a percentage of about 5 to 40 % by weight based on the total weight of the treatment material. With less than 1 % by weight of the carbide-forming element, or 5 % by weight of the boride-forming additive the rate of formation of the carbide or boride layer is too low for practical purposes. With more than 50 % by weight of the carbide-forming element, or 40 % by weight of the boride-forming additive the viscosity of the fused treatment material is increased to a value so high that it may become practically impossible to immerse the article to be treated into the treatment bath.

An article, such as a metal or the like, to be treated is immersed into the molten surface treatment material 4 and is maintained therein. On the other hand, an electrolytic treatment may be carried out, employing the article to be treated as the cathode while the article is immersed into and held in the treatment material 4. Iron, iron steel, alloy steel, graphite or the like can be used as the materials to be treated. If the material to be treated contains carbon and the surface treatment material 4 also contains a carbide-forming element such as a IVa, Va or VIa group element of the Periodic Table, a carbide coating (composed of the metal element) will be formed on the surface of the treated article. On the other hand, when boron is dissolved in the surface treatment material 4 and an electrolytic process is carried out in the bath, a boride coating is usually formed on the treated article.

The treatment temperature may theoretically range between the fusion point of the surface treatment material and the melting point of the article to be treated. In practice, however, it ranges preferably between 800°C and 1,300°C, in consideration of the rate of formation of the carbide or the boride layer as well as embrittlement caused by grain growth and the like.

The treatment time depends upon the thickness of the boride or the carbide layer to be formed. Treatment shorter than one hour will practically not furnish an acceptable formation of a boride or a carbide layer although the final determination of the treatment period depends upon the treatment temperature. An increase in the time of treatment will correspondingly increase the thickness of the boride or the carbide layer.

This process is carried out either in ambient air or in an inert gas atmosphere.

While the surface treatment is carried out and the thermal medium 2 is heated, the concave portion 11 of the furnace body 1 is covered with the cap 5. This cap 5 prevents emission of gases from the thermal medium 2 or from the treatment material 4 into the environment to a certain extent while it suppresses radiation of generated heat. The cap 5 is made of a refractory material or iron steel or the like.

In a heating furnace according to the present invention, the heat-resistant vessel 3 is introduced, for instance, into the thermal medium 2 after the thermal medium 2 has been heated to a sufficient temperature. The treatment material 4 in the heat-resistant vessel is thus rapidly heated by the thermal medium 2. When this surface treatment process has been finished the heat-resistant vessel may be removed from the thermal medium 2 and separated from the furnace body 1 so that it may cool. In this way, the time required for heating and cooling the surface treatment material 4 may be remarkably reduced. As a result of this reduction in time, the useful life of the molten surface treatment material in the heat-resistant vessel 3 is considerably prolonged.

Furthermore, in the heating furnace according to the present invention, the relative position of the heating electrodes 12 and the heat-resistant vessel 3 is defined in such a manner that the line ℓ connecting the upper ends of the two electrodes 12 is located below the heat-resistant vessel 3, and that the smallest distance M between the line ℓ and the heat-resistant vessel 3 is not less than 1/4 of the distance L between the electrodes of the pair. In other words, the pair of electrodes 12 is arranged at a sufficient distance below the heat-resistant vessel 3 and therefore there is no danger of current flowing to the heat-resistant vessel 3 itself, when a voltage is applied across the electrodes 12. As a result, the heat-resistant vessel 3 itself is not partially overheated and thus there is no deterioration of the heat-resistant vessel 3 due to such a partial overheating of the vessel. Furthermore, since the heating is carried out at a specified location below the heat-resistant vessel 3 and as also the distance between the inner wall surface of the heating portion 1, which defines the concave portion 11, and the outer side wall of the heat-resistant vessel 3 is not less than 50 mm, the thermal medium 2 surrounding the heat-resistant vessel 3 is maintained at a comparatively uniform temperature by normal convection.

As the pair of electrodes 12 is arranged at a position below the heat-resistant vessel 3, the furnace space requirement can be reduced. In the heating furnace as illustrated in the Figure, the open end of the concave portion 11 is moreover sealed with the flange portion 31 of the heat-resistant vessel 3 so that inconvenient effects such as seeping of thermal medium 2, penetration of the thermal medium 2 into the treatment material 4, etc., are avoided. On the other hand, the space defined between the vessel 3 and the inner wall of the furnace body may be filled with a protective gas, such as argon gas or the like, as this space is sealed by the flange portion 31 of the heat-resistant vessel 3.

As has already been described, with the heating furnace according to the present invention, it is possible to improve the life time of the heat-resistant vessel 3 or to use a low-grade material for the vessel 3 even when the furnace is employed at temperatures in excess of 1,100°C. Furthermore, the consumption of the thermal medium 2 can be reduced and the working environment can be also improved.

EXAMPLE:

An indirectly heated furnace according to the present invention was built as shown in the Figure. The furnace body 1 is made of a refractory alumina material. The concave portion 11 in the furnace body 1, which contains the thermal medium 2, has a cylindrical shape and a diameter of 347 mm and a depth of 635 mm. A pair of graphite electrodes 12 is provided in the inner wall of the concave portion 11 of the furnace body 1. The concave portion 11 contains the thermal medium 2 which is mainly composed of barium chloride. The heat-resistant vessel 3 is introduced into the melted thermal medium 2 in the concave portion 11. The vessel 3 has a cylindrical shape, is made of a heat-resistant steel, has an inner diameter of 150 mm and a depth of 430 mm. One hundred and fifty grams of powder mixture containing 80 % of borax and 20 % of ferro-vanadium (-100 mesh) were heated and melted for use as the surface treatment material. The surface treatment material thus obtained was contained in the heat-resistant vessel 3. The heat-resistant vessel 3 was provided with a flange portion 31 at its upper end, which was supported by the reinforcing ring 13 around the concave portion 11. The opening of the concave portion 11 of the furnace body 1 was thus sealed with the reinforcing ring 13 and the flange portion 31 of the heat-resistant vessel 3. The lowermost part of the heat-resistant vessel 3 was located above of the line ℓ connecting the upper ends of the electrodes 12. Furthermore, the smallest distance M between the aforesaid line ℓ and the heat-resistant vessel 3 was 100 mm. Moreover, the distance between the inner wall of the furnace body 1, which defined the concave portion 11, and the outer wall of the heat-resistant vessel 3, was 85 mm.

The indirectly heated furnace was heated as follows.

First, a small amount of barium chloride, used as the thermal medium, was introduced into the concave portion 11 of the furnace body 1. The barium chloride filling the gap between a pair of electrodes 12 in the concave portion 11 of the furnace body 1, was heated and melted by employing a heating equipment for local dissolution, employing a nichrome wire as the exothermic element. After the barium chloride was melted, a voltage of 25 V was applied across the electrodes 12 and additional barium chloride was filled into the concave portion 11 of the furnace body 1, preparing a barium chloride bath. The heat-resistant vessel 3 containing the surface treatment material was introduced into the barium chloride bath after the chloride had been heated to a sufficient temperature. Then the treatment material 4 in the heat-resistant vessel 3 was rapidly heated and melted to 1,200°C for half an hour.

The surface treatment process forming a vanadium carbide layer was continued, employing the thus prepared surface treatment material. High speed steel (JIS SKH 9) bars having a diameter of 7 mm and a length of 400 mm were immersed into the aforesaid surface treatment material for 10 minutes.

With time progressing oxidation of the treatment material occurred at the upper part of the steel bars. Accordingly, the carbide-forming ability of the treatment material in the vessel 3 decreased in the course of time until it became impossible to form a carbide layer on the steel surface. When the melted treatment material was continuously employed the actual useful life of the treatment material melt was 7.5 hours before the extent by which no carbide layer was formed on the article reached one half of the article.

The heating furnace of the present invention is frequently used both intermittently and continuously. When surface treatment is not carried out the heat-resistant vessel may be detached from the furnace body to prevent deterioration of the treatment material.

Under practical conditions, the melted surface treatment material can be used for several days.

For reference purposes the same surface treatment material was heated to 1,200°C, employing a conventional outside heating furnace. It took as long as five hours to raise the temperature of the surface treatment material, and the resulting useful life of the melted treatment material for forming a vanadium carbide layer was only 3 hours.

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WHAT IS CLAIMED IS:

1. An indirect heating furnace for surface treatment of a metal or the like, employing a salt bath, comprising:

a furnace body (1) made of a non-conductive refractory material and having a concave portion (11) with an open top end, for containing a thermal medium (2) mainly composed of a chloride;

a heat-resistant vessel (3) having an upper opening, positioned in said concave portion and supported at the upper portion of said furnace body, in a manner to be replaceable, said vessel containing a treatment material mainly composed of a borate for surface treatment of a metal or the like; and

at least one pair of electrodes (12) provided in said furnace body (1) and arranged at a specified location in the inner wall of said furnace body, which defines said concave portion (11), said at least one or more pairs of electrodes (12) being so located that a line (ℓ) connecting the upper ends of the electrodes (12) of each a pair is located below said heat-resistant vessel (3), and that the smallest distance (M) between said line connecting the upper ends of the electrodes of that pair which is located nearest to said heat resistant vessel and said heat-resistant vessel (3) is not less than 1/4 of the distance (L) between said pair of electrodes.

2. An indirect heating furnace according to claim 1, wherein the distance between the inner wall of said furnace body (1) and the outer side wall of said heat-resistant vessel (3) is not less than 50 mm.

3. An indirect heating furnace according to claim 1, wherein said heat-resistant vessel (3) is made of heat-resistant steel or graphite.

4. An indirect heating furnace according to claim 1, wherein said electrodes (12) are made of heat-resistant steel or graphite.

5. An indirect heating furnace according to claim 1, wherein said heat-resistant vessel (3) is provided with a flange portion (31) at its upper end for sealing the open top end of said concave portion.

6. An indirect heating furnace according to claim 1, furthermore comprising a cap (5) for covering the open top end of said concave portion (11) and the upper opening of said heat-resistant vessel (3).

7. An indirect heating furnace according to claim 1, wherein said chloride is selected from the group including sodium chloride (NaCl) and barium chloride ($BaCl_2$).

8. An indirect heating furnace according to claim 1, wherein said surface treatment material contains borate and a boride-forming or a carbide-forming additive.

9. An indirect heating furnace according to claim 8, wherein said boride-forming additive is a member selected from the group consisting of boron carbide, ferro-boron and ferro-aluminum.

10. An indirect heating furnace according to claim 8, wherein said carbide-forming additive is at least one member selected from the group consisting of IVa, Va and VIa group elements of the Periodic Table.

11. An indirect heating furnace according to claim 10, wherein said carbide-forming additive is in the form of an alloy.

12. An indirect heating furnace according to claim 8, wherein the percentage of said boride-forming additive ranges from 5 % to 40 % by weight based on the total weight of said treatment material.

13. An indirect heating furnace according to claim 8, wherein the percentage of said carbide-forming additive ranges from 1 % to 50 % by weight based on the total weight of said treatment material.

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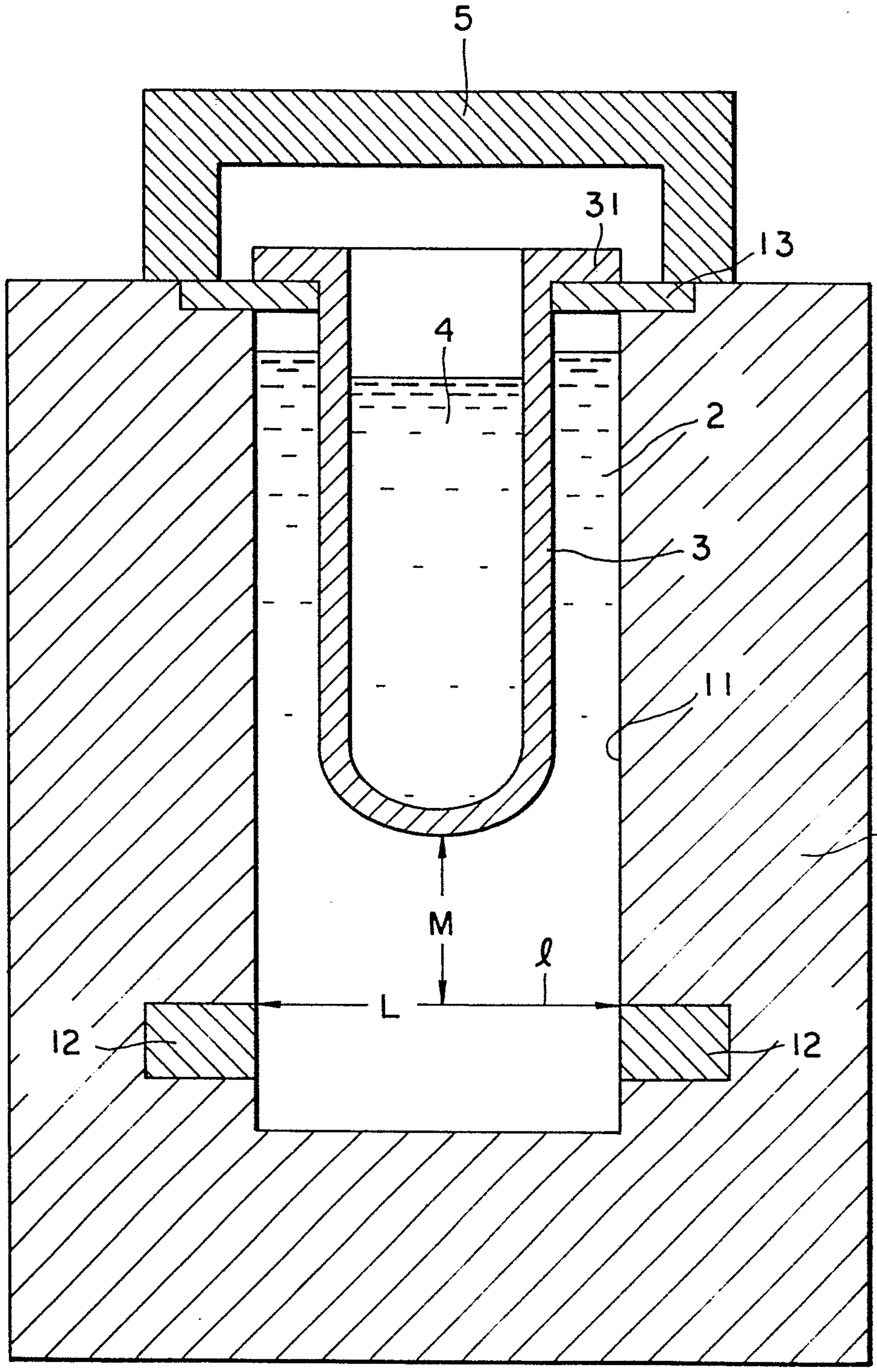

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EUROPEAN SEARCH REPORT

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Application number

EP 82 10 7252

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
Y	FR-A- 701 149 (SIEMENS-SCHUCKERTWERKE A.G) *Abstract; page 1, line 28; figures*	1,3,5, 7	C 21 D 1/46 H 05 B 3/60
Y	US-A-1 776 128 (OTIS) *Claims; figures*	1,2,3, 5,6	
Y	GB-A- 10 884 (RUSSELL) *The whole document*	1,3	
A	US-A-2 231 010 (HOLT) *Line 50; claims*	7	
A	FR-A- 913 062 (IMPERIAL CHEMICAL INDUSTRIES)	8	**TECHNICAL FIELDS SEARCHED (Int. Cl. 3)**
A	US-A-1 736 457 (MERTEN)		C 21 D C 23 C C 22 B H 05 B

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	15-11-1982	COULOMB J.C.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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