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(54) **Removal of alkali metals and alkaline earth metals from molten aluminium.**

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Description

This invention relates to the removal of contaminant quantities of alkali metals and alkaline earth metals from molten aluminium by reaction with aluminium fluoride.

U.S. 3,620,716 discloses a method of removing magnesium from molten aluminium by fluxing with a cover flux containing AlF_3. According to this document, the flux is stirred into the melt and the melt is then left for at least a few hours to allow reaction between the flux and the contaminant.

A method of treating molten aluminium with a particulate aluminium fluoride-yielding material has been described in EP—A—0065854. This publication is comprised in the state of the art as defined by Article 54(3) and (4) EPC. In that method a charge of contaminated metal is placed in a vessel, in which it is stirred to establish a vortex and flow currents having both downward and lateral components at the bottom of the vortex and upwardly spiralling currents at the periphery of the vessel. The particulate aluminium fluoride-yielding material is supplied to enter the vortex and become entrained in the molten aluminium and the stirring of the molten aluminium is continued until the alkali metal and alkaline earth metal content is reduced to a desired low level.

The vortex was preferably generated by means of a multi-blade impeller having blades inclined to its axis of rotation and in the already described process this was preferably arranged somewhat eccentrically in relation to the axis of a cylindrical vessel having a vertically arranged axis. It was found that optimum results were achieved by careful dimensioning of the impeller in relation to the dimension of the treatment vessel and also to the depth of molten metal to be contained in the vessel, generally a vessel for transferring a body of molten metal from a reduction cell to a casting station.

The preferred relationships were established as follows:

Relationship or dimension	Outside range	Preferred range
d/D	0.1—0.6	0.15—0.40
h/H	0.1—0.7	0.2—0.40
y	0.25H—0.75H	0.4H—0.6H
θ	0°—45°	30°—40°

In the above θ designates the pitch angle of the impeller blades, *d* is the overall diameter of the bladed portion of the impeller, *h* is the height of the impeller blades, *y* is the vertical distance from the bottom of the crucible interior to the midpoint of the impeller blades, *H* is the vertical distance from the bottom of the crucible interior to the quiescent level of molten metal in the crucible, *D* is the internal diameter of the crucible. The impeller was preferably eccentrically located at a distance of 0.1—0.25D and more preferably at a distance of 0.25—0.7 d in relation to the axis of the vessel.

In the method, as described in our said European Patent Application No. 82,302448.4 (EP—A—0065854) the vortex is generated by a stirrer in a mass of molten metal, which is essentially cylindrical in the unstirred condition. It has now been found that the process can be effectively performed on bodies of molten metal which are initially non-cylindrical (in the non-stirred condition). Solid particulate material can be entrained in and reacted with alkali metal contaminants in molten aluminium by generation of a vortex in such non-cylindrical masses with similar efficiency to that achieved when the metal is contained in an upright cylindrical crucible, thus permitting the process to be employed in all forms of transfer crucible, particularly crucibles having an essentially continuous, rounded sidewall surface free from recesses or abrupt angles, which would result in locally stagnant zones within the upwardly spiralling molten metal flow in the peripheral region of the vessel.

Referring now to the accompanying drawings.

Figure 1 is a diagrammatic plan view of the surface of a body of molten metal contained in a cylindrical crucible tilted with respect to the vertical.

Figure 2 is a similar view of a crucible having parallel sides and semi-circular ends.

Figure 3 is a view of a crucible having a downwardly tapering interior arranged at a substantial tilt angle in relation to the vertical, with a stirrer rotating about a vertical axis.

Figure 4 is a similar view to Figure 3, but with the stirrer rotating about an axis parallel to the crucible axis.

In performing the process of the present invention in the crucibles illustrated in Figures 1—4 the stirrer is constructed and arranged to operate in the same way as in our co-pending European Patent Application No. 82.302448.4. The stirrer is supported by a lid (not shown) and a duct is arranged on the lid for the supply of a particulate aluminium fluoride-yielding material (which expression embraces compounds such as $KAlF_4$ on to the surface of the molten metal in the vessel).

During the treatment of molten aluminium with AlF_3 powder, alkali and alkaline earth metals react preferentially with AlF_3 (compared to aluminium) to form mixed alkali cryolithionite compounds, e.g. $Na_5Al_3F_{14}$, Na_2LiAlF_6, and $Li_3Na_3Al_2F_{12}$.

These compounds, having a relatively low melting point (compared with pure cryolite), can easily be agglomerated or stick to the crucible walls or float to the melt surface where they react with metal oxide or particles of electrolyte always present after the siphoning of electrolytic cells. During subsequent metal transfer from the crucible by siphoning, most of these compounds will remain inside the crucible.

As previously stated, according to the present invention, it is possible effectively to treat contaminated molten Al metal with solid particulate AlF_3 and maintained in a non-cylindrical mass by a crucible or metal-confining vessel which has a configuration other than cylindrical and/or has an orientation other than vertical. For example, the vessel may be shaped as shown in Figures 2 and 3, and may be axially tilted with even an axially tilted stirrer (Fig. 4), although a vertical axis of stirrer rotation is preferred because of the effect of gravity on vortex generation.

We have found that the preferred dimensional and positional relationships already stated for an upright cylindrical vessel can be more broadly defined with reference to the geometric axis of the vessel, the axis of impeller rotation, and the plane of impeller rotation (viz. the plane, containing the midpoint of the impeller blades, perpendicular to the axis of impeller rotation). Thus, the diameter D is the minimum internal diametrical dimension (i.e. measured through the geometric axis of the vessel) of the metal-containing vessel in the plane of impeller rotation. Blade diameter d and eccentricity x are also measured in the plane of impeller rotation, while blade height h, distance y, and distance H are all measured along the axis of impeller rotation, and pitch angle θ is measured with reference to the axis of impeller rotation.

With the foregoing definitions, all the relationships and ranges of values (i.e. d/D, h/H, etc.) already given, hold true, except for the outside range of eccentricity x. In general, the eccentricity is limited only by the requirement that the minimum distance C from the axis of impeller rotation to the internal wall of the crucible vessel (again measured in the plane of impeller rotation) is at least equal to D/4.

Figure 1 represents the surface, having perimeter 10′, of a body of molten metal contained in a cylindrical crucible tilted with respect to the impeller axis. In Figure 1 G is the geometric axis of the elliptical surface of the molten metal D is the short diameter of the ellipse, and L is the long diameter of the ellipse. The axis of impeller rotation can intersect the plane of impeller rotation (viz. the plane of the drawing) anywhere within the area enclosed by broken-line ellipse E which is spaced inwardly from the crucible wall by a constant distance C_o=D/4. In the illustrated eccentric position of the impeller shaft 18, the eccentricity x is clearly greater than D/4, but the axis of impeller rotation is spaced from the crucible wall by a distance C greater than D/4.

In Figure 2, the cross-sectional configuration of the crucible 10″ in the plane of impeller rotation is bathtub-shaped, having semicircular ends and straight parallel sides spaced apart by a distance equal to the diameter of the semi-circular ends. The minimum diameter D through the geometric axis G in this plane is the distance between the parallel sides (viz. the diameter of the semi-circular ends), while the long diameter L is equal to A+D, where A is the spacing between the centres of the semi-circles. The broken line E, defining the outer limit of eccentricity of the axis of impeller rotation, is again spaced inwardly from the crucible wall by a constance distance C_o=D/4. The impeller shaft 18 shown in an illustrative eccentric position within this outer limit, having an eccentricity x.

The crucible 10‴ shown in elevational cross-section in Figures 3 and 4 has a geometric axis G tilted with respect to the vertical. In Figure 3 the axis of rotation of the impeller 14 is vertical; hence y is a vertical distance (from the midpoint of the impeller blades to the point on the crucible floor vertically beneath that midpoint) and eccentricity x is measured in the horizontal plane P of impeller rotation, which is at an oblique angle to G. In Figure 4, the axis of rotation of the impeller 14 is tilted to the vertical so as to be parallel to G; y is again measured along the impeller axis, now at an oblique angle to the vertical, and x is measured in rotational plane P which is perpendicular to G but at an oblique angle to the horizontal. The pitch angle θ, as indicated, is in each case measured with reference to the axis of impeller rotation. In both Figures 3 and 4 the crucible 10‴ tapers so that its cross-sectional diametrical dimensions decrease in a downward direction; the value of diameter D which determines the various aforementioned dimensional relationships is, in each instance, measured in the plane P.

These different arrangements permitted the removal of Li contamination from Al metal, withdrawn from electrolytic reduction cells, to be achieved within reasonable time, but not in all cases with the same process time as could be achieved with a stirrer eccentrically related to the axis of an upright vertical vessel.

It will be appreciated that a mass of molten metal, held in a tilted cylindrical crucible, assumes an essentially non-cylindrical shape, having an elliptical surface. The top surface of the body of metal in the tilted, tapering crucible of Figures 3 and 4 is also ellipsoidal in shape.

We have found that a vortex can be generated satisfactorily where the impeller is tilted by an angle of up to 15° to the vertical, but with decreasing efficiency as the tilt angle is increased. It is however preferred that the tilt angle of the impeller should not exceed about 10° to the vertical. The tilting of the axis of the crucible is of less importance. However, increase of the tilt angle of the crucible will decrease its molten metal-holding capacity. In all cases however improved results are obtained when the crucible (of whatever shape) is maintained in an upright condition and confines the molten metal to a non-cylindrical mass.

It should be noted that the treatment material may comprise aluminium fluoride, possibly containing up to 50% inert material, such as aluminium oxide. Alternatively aluminium fluoride may be added in chemically-bound form, such as a fluoaluminate of sodium or potassium. Conveniently it may be added as NaF/AlF_3 having a low NaF/AlF_3 ratio by weight, for example 0.6—0.7/1.

Aluminium fluoride may also be added admixed with other alkali metal fluorides or chlorides or alkaline earth metal fluorides or chlorides. As an alternative to aluminium fluoride, any active fluorine-containing compound could be added, i.e. any such compound which on addition to molten aluminium will liberate a fluoride which is reactive towards alkali or alkaline earth metal contaminants and does not introduce other undesirable contaminants into the molten aluminium. KBF_4 and K_2TiF_6 are examples of such compounds.

All such materials are for convenience in the appended claims considered as being embraced by the terms "aluminium fluoride" or "aluminium fluoride-yielding material".

Claims

1. A method of removing contaminant alkali metals and alkaline earth metals from molten aluminium comprising:—

(1) stirring a mass of molten contaminated aluminium in a vessel by means of an impeller also contained in the vessel, the axis of which impeller being located eccentrically in relation to the axis of the vessel and the blades of which impeller being pitched downwardly in relation to the axis of rotation, under conditions to establish a vortex therein and flow currents in said molten aluminium having both downward and lateral components at the bottom of said vortex and upwardly spiralling currents in the region of the periphery of the said vessel

(2) supplying particulate aluminium fluoride-containing material to the surface of said molten aluminium for entry into said vortex

(3) continuing the stirring of the molten aluminium until the alkali metal and alkaline earth metal content is reduced to a desired low level, and

(4) separating the molten aluminium from the alkali and alkaline earth metal fluoaluminate reaction products, wherein the vessel is non-cylindrical and/or has its axis inclined to the vertical such that it confines the molten metal to a non-cylindrical mass.

2. A method according to claim 1, wherein the molten metal is treated with powdered AlF_3 or $NaF \cdot AlF_3$ having a weight ratio of $NaF:AlF_3$ in the range of from 0.6—0.7:1.

3. A method according to claim 1, further characterised in that the containing vessel is non-cylindrical and has a minimum internal diameter D, and is filled with the molten body of metal to a height H, and the impeller has a diameter d and a blade height h, such that the ratio d/D is between 0.1 and 0.6 and the ratio h/H is between 0.1 and 0.7, D and d being measured in the plane of impeller rotation and H and h being measured along the axis of impeller rotation.

4. A method according to claim 3, further characterised in that the minimum spacing between the axis of impeller rotation and the vessel wall is D/4, measured in the plane of impeller rotation.

5. A method according to claim 3, further characterised in that the midpoint of said blades is spaced above the bottom of said vessel by a distance y, measured along the axis of impeller rotation, said distance y being 0.25H and 0.75H.

6. A method according to claim 3, further characterised in that the axis of impeller rotation is eccentric in relation to the vessel axis by a distance, x, having a value of 0.25d—0.7d in the plane of impeller rotation.

7. Apparatus for mixing particulate aluminium fluoride-yielding material with molten aluminium to remove dissolved contaminant alkali metals and alkaline earth metals from the molten aluminium said apparatus comprising

(a) a non-cylindrical vessel, having a vertical geometric axis and minimum internal diameter D, for containing a body of molten aluminium to a height H above the floor of the vessel; said vessel being free from internal baffles and having a generally rounded interior surface;

(b) a cover for said vessel supporting a multi-bladed impeller and means for driving said impeller about a vertical axis and means for rotating the impeller, said impeller having a diameter, d, and its blades having a height, h, the midpoint of said blades being spaced above the floor of the vessel by a distance, y, the axis of impeller rotation being spaced from said geometric axis by a distance x, and said blades having major surfaces pitched downwardly at an angle θ to the vertical;

(c) the values of d, D, h, H, x and θ being such that d/D is between 0.1 and 0.6, h/H is between 0.1 and 0.7, x is between 0.1 D—0.25 D, y is between 0.25H and 0.75H, and θ is greater than 0° but not greater than 45°;

(d) the minimum spacing between the axis of rotation of the impeller and the vessel, measured in the plane of impeller rotation, is D/4, the values of D, d and x being measured in the plane of impeller rotation, the values of H and h being measured along the impeller axis of rotation.

8. Apparatus according to claim 7, further characterised in that d/D is between 0.15 and 0.40, h/H is between 0.2 and 0.40, x is 0.25d—0.7 d, y is between 0.4H and 0.6H, and θ is between 30° and 40°.

Patentansprüche

1. Verfahren zur Entfernung von verunreinigenden Alkali- und Erdalkalimetallen aus geschmolzenem Aluminium umfassend:

(1) Rühren einer Masse aus geschmolzenem verunreinigtem Aluminium in einem Gefäß mittels eines Schnellrührers, wobei die Achse des Schnellrührers exzentrisch in Beziehung zur Achse des Gefäßes ist und die Flügel des Rührers schräg nach unten in Beziehung zur Drehachse eingestellt sind, unter Bedingungen, unter denen ein Wirbel und Fließströme in dem geschmolzenen Aluminium mit nach unten und zur Seite ausgebildeten Komponenten am Boden des Wirbels und nach oben spiralförmig aufsteigende Ströme in der Region des Umfanges des Gefäßes ausgebildet werden;

(2) Zugabe von feinteiligem Aluminiumfluorid enthaltenden Material zu der Oberfläche des geschmolzenen Aluminiums zum Eintritt in den Wirbel;

(3) weiteres Rühren des geschmolzenen Aluminiums bis der Alkali- und Erdalkalimetallgehalt auf das gewünschte Niveau vermindert ist, und

(4) Abtrennen des geschmolzenen Aluminiums von den Alkali- und Erdalkalimetall-fluoaluminat-Reaktionsprodukten, wobei das Gefäß nicht-zylindrisch ist und/oder dessen Achse zur Senkrechten derart geneigt ist, daß es das geschmolzene Metall als eine nichtzylindrische Masse umschließt.

2. Verfahren gemäß Anspruch 1, bei dem geschmolzene Metall mit pulverisiertem AlF_3 oder $NaF \cdot AlF_3$ mit einem Gewichtsverhältnis von $NaF:AlF_3$ im Bereich von 0,6 bis 0,7:1 behandelt wird.

3. Verfahren gemäß Anspruch 1, weiter dadurch gekennzeichnet, daß das aufnehmende Gefäß nicht-zylindrisch ist und einen kleinsten Innendurchmesser D hat, und mit dem geschmolzenen Metallkörper bis zu einer Höhe H gefüllt ist, und der Schnellrührer einen Durchmesser d und eine Flügelhöhe h hat, und daß das Verhältnis d/D zwischen 0,1 und 0,6 und das Verhältnis h/H zwischen 0,1 und 07 beträgt, und d in der Ebene der Rührerdrehung gemessen ist und H und h längs der Achse der Rührerdrehung gemessen sind.

4. Verfahren gemäß Anspruch 3, weiter dadurch gekennzeichnet, daß der minimale Abstand zwischen der Achse der Rührerdrehung und Gefäßwand D/4, gemessen in der Ebene der Rührerdrehung ist.

5. Verfahren gemäß Anspruch 3, weiter dadurch gekennzeichnet, daß sich der Mittelpunkt der Flügel in einem Abstand y oberhalb des Bodens des Gefäßes, gemessen längs der Achse der Rührerdrehung befindet, wobei dieser Abstand y 0,25H und 0,75H beträgt.

6. Verfahren gemäß Anspruch 3, weiter dadurch gekennzeichnet, daß die Achse der Rührerdrehung exzentrisch in Beziehung zu der Gefäßachse in einer Entfernung x, die einen Wert von 0,25d bis 0,7d in der Ebene der Rührerdrehung hat.

7. Vorrichtung zum Mischen von feinteiligen Aluminiumfluorid ergebenden Material mit geschmolzenem Aluminium zur Entfernung von gelösten verunreinigenden Alkali- und Erdalkalimetallen aus dem geschmolzenen Aluminium, wobei die Vorrichtung umfaßt

(a) ein nicht-zylindrisches Gefäß mit einer senkrechten geometrischen Achse und einem kleinsten Innendurchmesser D für die Aufnahme eines Körpers aus geschmolzenem Aluminium bis zu einer Höhe H oberhalb des Bodens des Gefäßes, wobei das Gefäß keine inneren Prallbleche aufweist und einem im allgemeinen abgerundete Innenoberfläche hat;

(b) eine Abdeckung für das Gefäß, welche einen mehrflügeligen Schnellrührer und Einrichtungen zum Antreiben des Schnellrührers längs einer senkrechten Achse und Einrichtungen zum Drehen des Schnellrührers trägt, wobei der Schnellrührer einen Durchmesser d und dessen Flügel eine Höhe h haben, der Mittelpunkt der Flügel in einem Abstand y vom Boden des Gefäßes entfernt ist, die Achse der Rührerdrehung sich in einem Abstand x von der geometrischen Achse befindet und die Hauptoberflächen der Flügel in einem Winkel θ zur Senkrechten nach unten geneigt sind;

(c) die Werte von d, D, h, H, x und θ derart sind, daß d/D zwischen 0,1 und 0,6 ist, h/H zwischen 0,1 und 0,7 ist, x zwischen 0,1 D bis 0,25 D ist, y zwischen 0,25H und 0,75H ist, und θ größer als 0° aber nicht größer als 45° ist;

(d) der kleinste Abstand zwischen der Drehachse des Rührers und des Gefäßes gemessen in der Ebene der Rührerdrehung D/4 ist, die Werte von D, d und x in der Ebene der Rührerdrehung gemessen sind und die Werte von H und h längs der Drehachse des Rührers gemessen sind.

8. Vorrichtung gemäß Anspruch 7, weiter dadurch gekennzeichnet, daß d/D zwischen 0,15 und 0,40 ist, h/H zwischen 0,2 und 0,40 ist, x 0,25d bis 0,7 d ist, y zwischen 0,4H und 0,6H ist, und θ zwischen 30° und 40° ist.

Revendications

1. Procédé d'élimination des métaux alcalins et alcalino-terreux contaminants de l'aluminium fondu, dans lequel:

(1) on agite une masse d'aluminium contaminé fondu dans un récipient au moyen d'une roue à aubes également contenue dans le récipient, l'axe de ladite roue étant situé de manière excentrée par rapport à l'axe du récipient et les aubes de ladite roue à aubes étant inclinées vers le bas par rapport à l'axe de rotation, dans des conditions permettant d'y établir un tourbillon et des courants de circulation dans ledit

aluminium fondu ayant des composantes descendantes et latérales au fond dudit tourbillon et des courants montant en spirale dans la région de la périphérie dudit récipient

(2) on fournit une matière en particules contenant du fluorure d'aluminium à la surface dudit aluminium fondu pour pénétrer dans ledit tourbillon

(3) on continue d'agiter l'aluminium fondu jusqu'à ce que la teneur en métal alcalin et en métal alcalino-terreux soit réduite au niveau désiré, et

(4) on sépare l'aluminium fondu des produits de réaction fluoaluminate de métal alcalin et de métal alcalino-terreux, dans lequel le récipient est non cylindrique et/ou a son axe incliné par rapport à la verticale de manière qu'il confine le métal fondu en une masse non cylindrique.

2. Procédé selon la revendication 1, dans lequel le métal fondu est traité avec AlF_3 ou $NaF \cdot AlF_3$ ayant un rapport pondéral de $NaF{:}AlF_3$ dans un intervalle de 0,6—0,7:1.

3. Procédé selon la revendication 1, caractérisé en outre en ce que le récipient contenant est non cylindrique et a un diamètre interne minimal D, et est rempli de la masse fondue de métal à une hauteur H, et la roue à aubes a un diamètre d et une hauteur d'aubes h, tels que le rapport d/D est compris entre 0,1 et 0,6 et le rapport h/H est compris entre 0,1 et 0,7, D et d étant mesurés dans le plan de rotation de la roue à aubes et H et h étant mesurés le long de l'axe de rotation de la roue à aubes.

4. Procédé selon la revendication 3, caractérisé en outre en ce que l'espacement minimum entre l'axe de rotation de la roue à aubes et la paroi du récipient est de D/4, mesuré dans le plan de rotation de la roue à aubes.

5. Procédé selon la revendication 3, caractérisé en outre en ce que le milieu desdites aubes est espacé au-dessus du fond dudit récipient d'une distance y, mesurée le long de l'axe de rotation de la roue à aubes, ladite distance y étant entre 0,25H et 0,75H.

6. Procédé selon la revendication 3, caractérisé en outre en ce que l'axe de rotation de la roue à aubes est excentré par rapport au récipient d'une distance x, ayant une valeur de 0,25d—0,7d dans le plan de rotation de la roue à aubes.

7. Appareil pour mélanger une matière donnant des particules de fluorure d'aluminium avec de l'aluminium fondu pour enlever les métaux alcalins et métaux alcalino-terreux contaminants de l'aluminium fondu, ledit appareil comprenant

(a) un récipient non cylindrique, ayant un axe géométrique vertical et un diamètre interne minimum D, pour contenir une masse d'aluminium fondu à une hauteur H au-dessus du plancher du récipient; ledit récipient étant dépourvu de chicanes internes et ayant une surface intérieure généralement arrondie;

(b) un couvercle pour ledit récipient portant une roue à plusieurs aubes (ou ailettes) et un moyen pour diriger ladite roue à aubes autour d'un axe vertical et un moyen pour faire tourner la roue à aubes, ladite roue à aubes ayant un diamètre, d, et ses aubes ayant une hauteur, h, le milieu desdites aubes étant espacé au-dessus du plancher du récipient d'une distance, y, l'axe de rotation de la roue à aubes étant espacé dudit axe géométrique d'une distance x, et lesdites aubes ayant leur surface majeure inclinée vers le bas à un angle θ par rapport à la verticale;

(c) les valeurs de d, D, h, H, x et θ étant telles que d/D est compris entre 0,1 et 0,6, h/H est compris entre 0,1 et 0,7, x est entre 0,1 D et 0,25 D, y est entre 0,25 H et 0,75 H, et θ est supérieur à 0° mais non supérieur à 45°;

(d) l'espacement minimum entre l'axe de rotation de la roue à aubes et le récipient, mesuré dans le plan de rotation de la roue à aubes, est D/4, les valeurs de D, d et x étant mesurées dans le plan de rotation de la roue à aubes, les valeurs de H et h étant mesurées le long de l'axe de rotation de la roue à aubes.

8. Appareil selon la revendication 7, caractérisé en outre en ce que d/D est compris entre 0,15 et 0,40, h/H est entre 0,2 et 0,40, x est entre 0,25 d et 0,7 d, y est entre 0,4 H et 0,6 H, et θ est entre 30° et 40°.

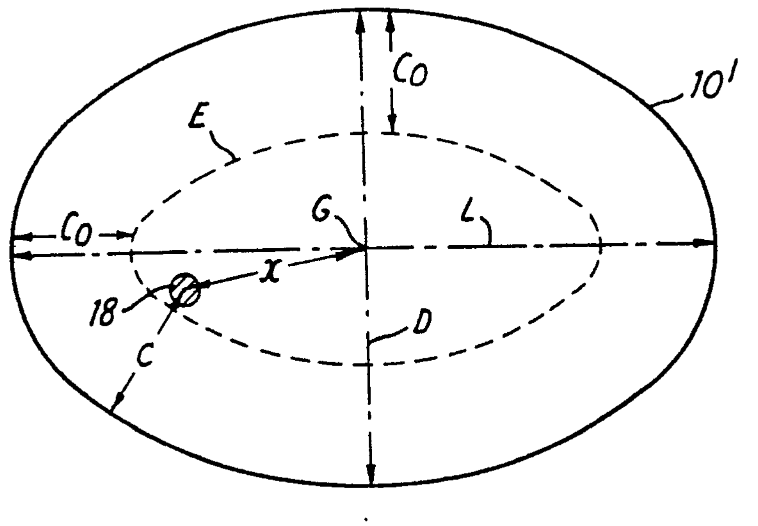

FIG.1

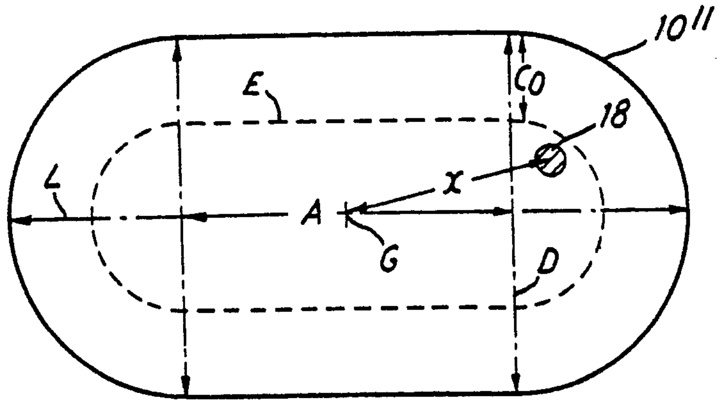

FIG.2

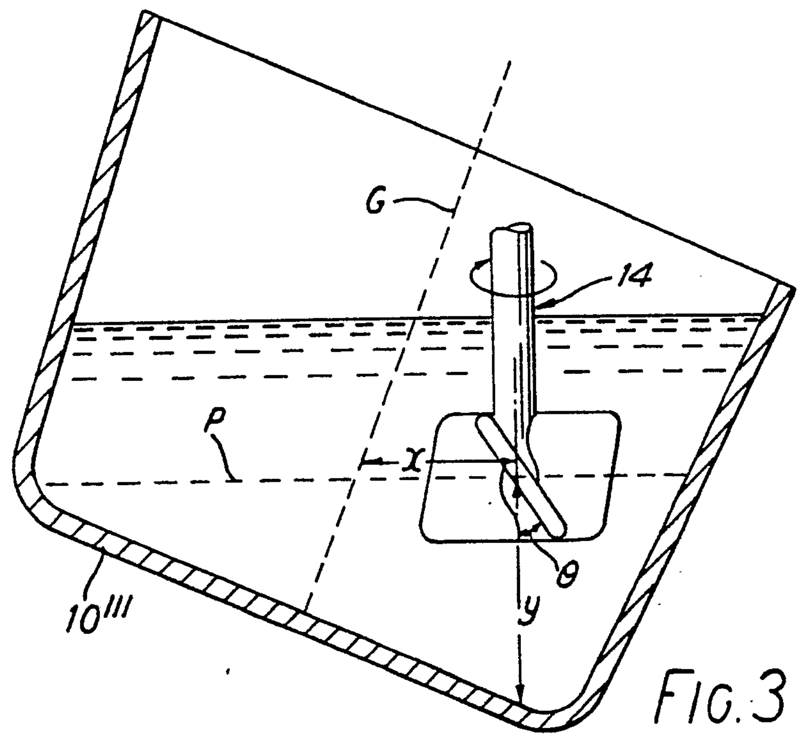

FIG.3

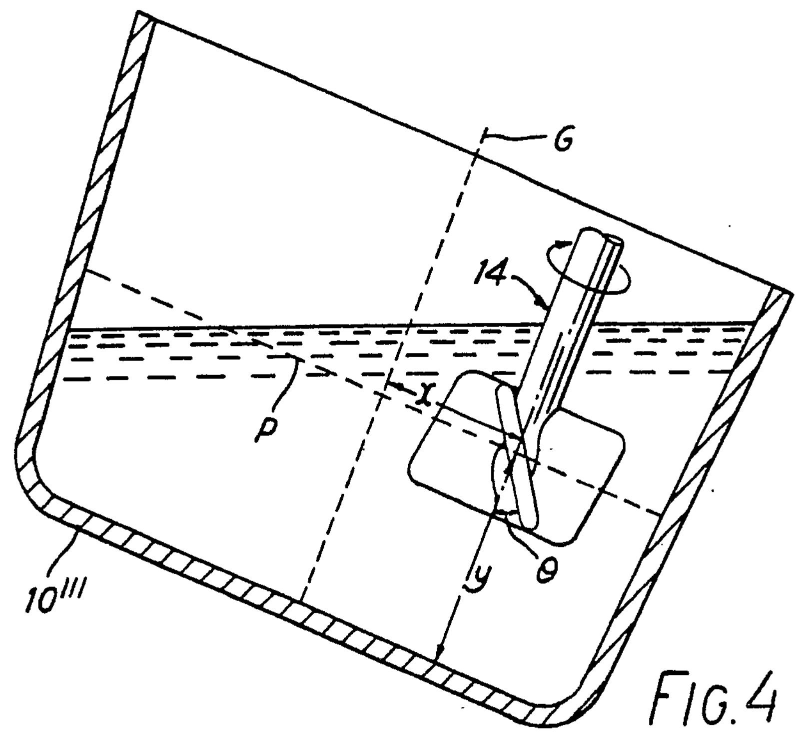

FIG.4