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

(11) EP 0 732 415 A1

(12) # EUROPEAN PATENT APPLICATION

(43) Date of publication:
18.09.1996 Bulletin 1996/38

(51) Int Cl.6: **C22C 1/04**

(21) Application number: **96301717.3**

(22) Date of filing: **13.03.1996**

(84) Designated Contracting States:
AT BE CH DE DK ES FR GB IT LI NL SE

(30) Priority: **14.03.1995 GB 9505094**
21.12.1995 GB 9526166
23.01.1996 GB 9601331

(71) Applicant: **Deritend Advanced Technology Limited**
Oldbury, West Midlands B69 2DH (GB)

(72) Inventors:
- **Zhang, Zhu**
Kings Heath, Birmingham B14 6AN (GB)
- **Emmett, Robert Edward**
Raymonds Hill, Axminster,
Devon EX13 5TD (GB)

(74) Representative: **Leach, John Nigel**
FORRESTER & BOEHMERT
Franz-Joseph-Strasse 38
80801 München (DE)

(54) **Method of making an intermetallic compound**

(57) A method of making a product comprising an intermetallic compound including the steps of permeating in a mould cavity a powder metal component, having a first melting point, with a liquid metal component, having a second melting point which is lower than the first melting point, to produce a product comprising an intermetallic compound of said powder and liquid metal components.

EP 0 732 415 A1

Description

Description of Invention

This invention relates to a method of making a product comprising an intermetallic compound.

Intermetallic compounds, for example titanium aluminides, have good high temperature mechanical properties, a relatively low density and good oxidation resistance. This renders them potential replacements for super alloys and other high temperature materials in, for example, aerospace, industrial gas turbines, electronic and automotive applications.

Hitherto, it has been proposed to make products comprising an intermetallic compound by one of the following methods.

Intermetallic compound ingots may be produced by electroslag or vacuum induction melting for further processing and shaping using hot forging techniques. However, problems with shape deformation and resulting microstructure and mechanical properties have restricted the use of this method from widespread use in manufacturing industry.

Alternatively, it has been proposed to use a lost wax investment casting method to provide a near net shape product by melting intermetallic compound ingots using processes based on conventional titanium alloy melting and pouring processes. These processes require relatively high energy input and have the additional disadvantages of the molten metal reacting with mould surfaces and with a crucible in which the melting operation is performed, together with problems arising from grain growth in the casting.

Another alternative method hitherto proposed is to carry out a powder metallurgical process to produce a initially porous structure which is then hot isostatically pressed to allow the reaction required to produce an intermetallic compound to take place and to fully densify the structure. Complexity and limitations on the size and accuracy of products have restricted the application of this method.

Objects of the present invention are to provide a method of and apparatus for making a product comprising an intermetallic compound whereby the above mentioned disadvantages are overcome or are reduced.

According to the present invention we provide a method of making a product comprising an intermetallic compound including the steps of permeating a powder metal component, having a first melting point, with a liquid metal component, having a second melting point which is lower than the first melting point, to produce a product comprising an intermetallic compound of said powder and liquid metal components.

The method may be performed in a mould cavity. The mould cavity may define the net shape of the product or near net shape of the product.

In a first more specific aspect of the invention:

Said step of permeating a powder metal component with a liquid metal component may be performed to produce an intermediate form and the method may include the step of heat treating the intermediate form to produce said product.

Preferably in this aspect, the powder metal component comprises titanium or may comprise iron and the liquid metal component aluminium.

Alternatively to said first more specific aspect, in a second more specific aspect of the invention:

Said step of permeating a powder metal component with a liquid metal component may be performed under specific conditions to produce said product without a discrete step of heat treating an intermediate form.

Preferably in this aspect the powder metal component comprises nickel or may comprise iron and the liquid metal component aluminium.

The method may include the step of subjecting the liquid metal component to a pressure which is greater than a pressure to which the powder metal component is subjected. The subjection of the components to such a pressure difference may cause or assist said permeating of the powder metal component with the liquid metal component.

The powder metal component and the liquid metal component may be subjected to pressure to cause or assist permeation of the powder metal component with the liquid metal component.

The components may be subject to pressure by the application of fluid pressure such as gas, or by the application of mechanical pressure, such as by a mechanical ram.

The powder metal component may comprise a rigid preform.

Alternatively, the powder metal component may be in flowable form.

The powder metal component may be introduced into the mould cavity and then the liquid metal component may be introduced into the mould cavity.

The powder metal component may be degassed. That is, the powder metal component may be treated to remove gaseous and/or volatile material, for example by heating and evacuation.

The powder metal component may be degassed after being introduced into the mould cavity and before introduction of the liquid metal component.

After the step of permeating the powder metal component the liquid metal component may be allowed to solidify and the solidified intermediate form may then be subjected to said heat treatment to effect a solid state phase transformation to provide said product comprising an intermetallic compound.

A predetermined atomic fraction of said powder metal component and said liquid metal component may be introduced into said mould cavity.

The mould cavity may have a wall which is impermeable as hereindefined.

The liquid metal component may be introduced into the mould cavity via an ingate from a reservoir.

In one more specific aspect of the invention.

The liquid metal component may be introduced into the mould cavity from the reservoir through a passage which communicates with the ingate.

The liquid metal component may be introduced into the mould cavity via an ingate from a reservoir.

The or each ingate may comprise an aperture or a plurality of apertures or a porous material such as a porous ceramic material which may also function as a filter.

The reservoir may be disposed above the mould cavity and the liquid metal component being fed downwardly into the mould cavity through the passage.

The liquid metal component may be fed downwardly into the mould cavity from the reservoir by virtue of flow under gravity.

A valve means may be provided to control the flow of molten metal from the reservoir to the mould cavity.

The valve means may comprise a stopper rod or other valve member moveable relative to a valve seat.

The mould and the reservoir may be mounted for movement relative to a horizontal axis so that in a first position the mould may be disposed above the reservoir and in a second position the mould may be disposed below the reservoir so that metal can flow from the reservoir into the mould cavity under gravity.

The reservoir and mould may be mounted for rotation about said axis or may be mounted for orbital or any other desired movement in which said change of orientation may take place.

The reservoir may be disposed below the mould cavity and the liquid metal may be fed upwardly into the mould cavity through the passage.

Alternatively to said one more specific aspect in another more specific aspect of the invention.

The liquid metal component may be introduced into the mould cavity via an ingate by immersing the ingate in a bath of said liquid metal component held in a reservoir.

Preferably the whole of the mould is immersed in the bath.

The ingate may be at the top of the mould cavity or may be at any other desired position.

Where the ingate is not at the top of the mould cavity only a part of the mould may be immersed in the bath of liquid metal component and the mould cavity being filled by applying pressure to the liquid metal bath to force it into the mould cavity.

The introduction operation may be performed so that the temperature of the metal in the bath is above the melting point of the liquid metal component but below the melting point of the intermetallic compound so that the intermetallic compound will solidify within the mould cavity whilst the mould cavity is immersed or partly immersed in the bath, and the mould containing the solidified intermetallic compound can be removed from the bath.

The mould cavity may be provided with a plurality of ingates.

The or each ingate may be positioned at any desired location in a wall of the mould.

Where the wall of the mould defines a tubular mould cavity, at least one ingate may be provided in the part of the wall of the mould which defines the internal surface of the tubular mould cavity.

In each more specific aspect:

The liquid metal component in the reservoir may be subject to pressure.

The pressure may be mechanical pressure, for example, by advancing a piston in said reservoir to apply pressure to a surface of the liquid metal.

The pressure may be applied by subjecting a surface of the metal in the reservoir to fluid pressure, for example, gaseous pressure.

Said application of pressure may feed or aid the feed of metal from the reservoir into the mould cavity and/or subject the metal in the mould cavity to pressure.

The mould cavity may be disposed in an enclosure, the interior of which may be placed in communication with the reservoir, for example through the ingate and the interior of the enclosure may be subject to said fluid pressure.

The mould cavity may be connected in communication with a degassing means through the ingate or, alternatively, through a conduit separate from the ingate.

Where the mould and the reservoir are mounted for said movement relative to said axis, conduit means, which may comprise a passage, are provided continuously to connect the interior of the enclosure to the source of fluid pressure. Flow of fluid pressure through the passage may be controlled by suitable valve means.

The conduit means may comprise a passage extending lengthwise of a pivot member by which the mould and reservoir are mounted for rotation about said axis.

The pressure may be maintained for a predetermined time, for example, until the liquid metal in the mould cavity has solidified, or, alternatively, for a time which is predetermined so as to ensure complete or substantially complete permeation of the liquid metal component into the powder metal component.

The powder metal component may comprise titanium, iron or nickel.

The liquid metal component may comprise aluminium.

The intermetallic compound may comprise titanium aluminide, which may be $TiAl_3$, TiAl or Ti_3Al, nickel aluminide which may be $NiAl_3$, Ni_2Al_3,NiAl, Ni_3Al or iron aluminide which may be Fe $Al_3Fe_2Al_5$, FeAl or Fe_3Al.

The intermetallic compound may comprise (expressed in Atomic %):

	Powder	Al	Others	When present
	TI + usual impurities			Optionally
			)	
$TiAl_3$	Balance	75	)	1 - 4
			) 1 - 25	
TiAl	Balance	47 - 56	)	1 - 4
			)	
Ti_3Al	Balance	20 - 38	)	7 - 25
	Ni + usual impurities			Optionally
$NiAl_3$	Balance	75	)	1 - 2
			)	
Ni_2Al_3	Balance	58 - 63	)	1 - 2
			) 1 - 15	
NiAl	Balance	45 - 59	)	1 - 2
			)	
Ni_3Al	Balance	25 - 28	)	7 - 14
	Fe + usual impurities			Optionally
$FeAl_3$	Balance	75	)	1 - 4
			)	
Fe_2Al_5	Balance	71 - 73	)	1 - 4
			) 1 - 15	
FeAl	Balance	36 - 55	)	1 - 4
			)	
Fe_3Al	Balance	22 - 36	)	5 - 6

The "other" components, if and when present, may comprise chromium, manganese or niobium and may be present in the range specified above.

The intermetallic compound alternatively may comprise molybdenum silicide or niobium beryllides or chromides.

At least one additional component may be introduced into the mould cavity prior to permeating the powder metal component with the liquid metal component. The non-metallic reinforcement may be added to the intermetallic compound to create a metal matrix composite (MMC) having modified thermophysical properties. The additional component may comprise ceramic particles such as silicon carbide or alumina particles, fibres such as silicon carbide or alumina fibres, whiskers such as silicon carbide whiskers or monofilament, multifilament or random fibres.

In a further more specific aspect of the invention:

Said step of permeating a powder metal component with a liquid metal component may be performed to provide a product having a first portion which has a melting point which is higher than the melting point of a second portion of the product.

Said step of permeating a powder metal component with a liquid metal component may be performed with different atomic proportions by atomic weight of powder metal component and liquid metal component in at least one predetermined part of the mould.

The method may comprise a step of introducing said powder metal component into a first part of the mould cavity and permeating the powder metal component in said first part with said liquid metal component and introducing another component, selected from a powder metal component and a liquid metal component, in a second part of the mould cavity not occupied by the powder metal component.

The other component may be a liquid metal component.

Alternatively, the other component may be at least one powder metal component the or each of which provides a different atomic proportion of powder metal component to liquid metal component to the proportion provided in the first part of the cavity and the method including a further step of permeating the or each other powder metal component with a liquid metal component.

In the latter case, the mould cavity may comprise a third part unoccupied by a powder metal component and into which a liquid metal component is introduced.

The compositions and proportions by atomic weight in each part may be in accordance with the values tabulated in Table 1 hereof.

In consequence a product, in accordance with the further more specific aspect of the invention, may be provided with different physical properties such as melting points, in different regions of the product. For example, in the case of an automotive piston or an aerospace heat shield a part of the product adjacent a surface which, in use, is subjected to high temperature such as the crown of an automotive piston may be provided with a higher melting point than the remainder of the product.

In the regions of the boundaries between said parts there may be an abrupt change in composition or a gradual change of composition. It is believed that providing the powder metal components in a flowable form facilitates an extended transition zone between the compositions of the parts.

For example, where the powder metal component comprises a surface part having an atomic proportion to provide a surface part of NiAl, there may be transition regions of a Ni_2Al_3 and then $NiAl_3$ as an integral part of the product, as the composition approaches that of aluminium or an aluminium alloy the remainder of which comprises aluminium or an aluminium alloy. In between these parts, for example between NiAl and Ni_2Al_3, there may be a boundary layer consisting of a mixture of precipitates of NiAl and Ni_2Al_3.

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

FIGURES 1 - 8 illustrate, diagrammatically, stages in a method embodying the invention;
FIGURE 9 is a diagrammatic illustration of one form of apparatus which may be used in the method illustrated with reference to Figures 1 - 8;
FIGURE 10 is a diagrammatic illustration of an alternative form of apparatus for use in the method of Figures 1 - 8;
FIGURE 11 is a diagrammatic illustration of a further alternative form of apparatus for use in the method of Figures 1 - 8;
FIGURE 12 is a diagrammatic illustration of a further alternative form of apparatus for use in the method of Figures 1 - 8;
FIGURE 13 is a diagrammatic illustration of a further alternative form of apparatus for use in the method of Figures 1 to 8;
FIGURE 14 is a diagrammatic illustration of a yet further alternative form of apparatus for use in the method of Figures 1 to 8, and
FIGURE 15 is a diagrammatic illustration of a yet further embodiment of the invention.

Referring to Figure 1 of the drawings, a mould cavity 10 of a desired net shape, that is to say, a shape which is the same as, or similar to, a desired final shape of a product, and having an ingate I is provided in a mould or die 11 by suitable means such a shaped mould can be distinguished from, for example, an ingot mould by virtue of the product having a desired three dimensional shape and generally being not formed to a final shape by further forming . In the present example the cavity is made using a lost wax disposable pattern in a refractory material which in the present example is silica, but which may be zirconia and/or alumina.

The refractory material of the mould in the present example comprises silica bonded with silicate formulated so as to make the mould impervious, or substantially impervious, to liquid or gas.

The mould is then fired in conventional manner to a temperature sufficient to remove volatile material and to stabilise the mould.

If desired, the cavity may be made in any other suitable manner and need not be made to net shape, although such is preferred. For example, the mould may be made in one of said refractory materials, or in plaster or any suitable moulding material using removable patterns. For example, if, alternatively, the mould is made of alumina it may be made, for example, of alumina silicate which renders the mould, in itself, impermeable. Further alternatively, the mould may be made by machining a suitable mould material and where, for example, the mould material is metal, then the cavity is generally referred to as being made in a die.

In all cases described herein the mould is made in such a way as to be impermeable. By "impermeable" we mean herein that the mould is at least sufficiently impermeable a) for liquid metal to be cast into the mould so as to prevent penetration of the mould surface by the liquid metal surface, and b) to prevent passage of gas from the interior of the wall of the mould to prevent ingress of gas into the interstices between the powder particles, of the powder metal component, from within the interior of the mould wall prior to being permeated with the liquid metal component.

By "interior of the mould wall" we mean gas which has either permeated into the mould wall and been held there within the mould wall or gas which has passed through the interior of the mould wall from the exterior of the mould wall into the mould cavity. The degree of "prevention" can vary with the desired application and/or manufacturing technique including the mould material of the product concerned. For example, for some applications a relatively rough surface is desirable.

The mould may be made of material which is itself impermeable as well as a mould which is coated or otherwise treated so as to be impermeable. The porosity, for example the pore size of the mould, may vary with temperature within the above-mentioned limits. An important requirement is that the mould is, in practice, sufficiently impermeable to liquid metal to be cast into the mould as well as to passage of gas from the interior of the wall of the mould to produce satisfactory results.

Referring to Figure 2.

The mould cavity then has a powder metal component introduced thereinto, through the ingate I as shown at 12 in Figure 2.

The morphology and the packing density of the material is adjusted to provide the required volume fraction. In the present example the powder is titanium metal powder, but may comprise other suitable material for producing intermetallic compounds, such as nickel or iron.

If desired, the powder metal component, instead of comprising metal powder in flowable form, may comprise a rigid preform made by conventional powder metallurgical techniques to produce a preform with interstices between the powder metal particles.

In the present example the titanium powder had an average particle size of 70 µm and a purity of about 99.75%. The particle size may lie in the range, for example 10 - 150 µm or, for example, 30 - 250 µm. The volume fraction introduced in the present example was about 50% but the volume fraction may lie in the range 35% to 75%.

Where the powder metallurgical component comprises a preform the preform may be pressed as necessary to achieve the desired volume fraction and the mould cavity is configured so as to provide a predetermined clearance with the preform. The clearance may, for example, be the minimum necessary to allow introduction of the preform into the cavity.

Referring now to Figure 3,

The mould 11 is disposed in the chamber 13. The chamber 13, in the present example, comprises a lower part 14 in which the mould 11 is disposed and an upper part 15 in which a crucible 16 for receiving liquid metal component is received.

The mould 11 may be introduced into a lower part 14 of the chamber either before or after filling the mould 11 with the powder metal component.

The crucible 16 is filled with the desired amount of liquid metal 17 which in the present example comprises aluminium, but which, if desired, may comprise other suitable material for any of the alternative powder metal components.

Suitable means are provided to maintain the metal in the crucible 16. Such means may comprise a magnetic field, a mechanical plug or other means as described hereinafter with reference to Figures 9 - 15.

The chamber 13 is then sealed from atmosphere and is evacuated to remove air from the interior of the chamber and consequently from the interstices between the powder particles 12 in the mould 11.

If desired, the inside of the chamber 13 and or the mould 11 may be heated to facilitate the above described degassing process and the removal of volatile materials from the mould and the chamber.

Referring now to Figure 4.

When degassing has been completed the liquid metal component 17 is poured from the bottom of the crucible 16 through a tap hole 18 into a feeder system 19 of the mould 11 and, hence, via the ingate I, into the mould 11. Initially the molten metal 17 rests temporarily in the feeder system 19 on top of the powder component 12 so as to seal the evacuated interstices inside the impermeable mould 11.

Referring now to Figure 5.

The powder and liquid components within the mould 11 are and then subjected to pressure to ensure that the powder metal component is permeated with the liquid metal component so that all, or substantially all, of the interstices between the particles of the powder metal components are filled with liquid metal. The application of this pressure overcomes flow resistance and surface tension effects and ensures complete permeation.

The pressure may be applied using any one of the casting apparatus described hereinafter and illustrated with reference to Figures 9 - 15.

When the liquid metal component 17 has solidified, the pressure in the chamber 13 is reduced to atmospheric pressure and the upper and lower parts 15, 14 are separated and the mould 11 is removed, as shown at 21 in Figure 6.

Mould material and excess metal 20 in the feeder system 19 are removed to leave a near net shape intermediate product, shown at 22 in Figure 7. The intermediate product 22 contains the correct ratio of finely distributed components necessary to produce the intermetallic compound.

The thus produced intermediate product is then subject to a solid state phase transformation heat treatment to create an intermetallic compound.

In the present example, where the liquid metal and powder metal components comprise approximately 50% volume fraction titanium and aluminium, the heat treatment to produce a titanium aluminide intermetallic compound of desired microstructure and mechanical properties comprises a two stage solid state phase transformation in the range 520 - 600°C, typically 570°C and in the range 1200°C up to the limit of stability, typically 1350°C.

If desired, the powder metal component may comprise titanium, iron or nickel.

The liquid metal component may comprise aluminium.

The intermetallic compound may comprise titanium aluminide, which may be $TiAl_3$, TiAl or Ti_3Al, nickel aluminide which may be $NiAl_3$, Ni_2Al_3, NiAl, Ni_3Al or iron aluminide which may be $FeAl_3$ Fe_2Al_5, FeAl or Fe_3Al.

The liquid metal and powder metal components may comprise compositions selected from the ranges set out below (expressed in Atomic %):

TABLE 1

	Powder	Al	Others	When present
	Ti + usual impurities			Optionally
			)	
$TiAl_3$	Balance	75	)	1 - 4
			) 1 - 25	
TiAl	Balance	47 - 56	)	1 - 4
			)	
Ti_3Al	Balance	20 - 38	)	7 - 25
	Ni + usual impurities			Optionally
$NiAl_3$	Balance	75	)	1 - 2
			)	
Ni_2Al_3	Balance	58 - 63	)	1 - 2
			) 1 - 15	
NiAl	Balance	45 - 59	)	1 - 2
			)	
Ni_3Al	Balance	25 - 28	)	7 - 14
	Fe + usual impurities			Optionally
$FeAl_3$	Balance	75	)	1 - 4
			)	
Fe_2Al_5	Balance	71 - 73	)	1 - 4
			) 1 - 15	
FeAl	Balance	36 - 55	)	1 - 4
			)	
Fe_3Al	Balance	22 - 36	)	5 - 6

For example, the resultant intermetallic components may be as set out in Table 2.

TABLE 2

Intermetallic	Density, g/cm^3	Aluminium, wt%	Melting point, °C
Ni_3Al	7.293	13.28	1395
NiAl	5.91	31.49	1639
Ni_2Al_3	4.787	40.81	1133
$NiAl_3$	3.957	57.96	854
Fe_3Al	6.648	13.87	1502
FeAl	5.585	32.57	1215
Fe_2Al_5	3.963	54.70	1171
Ti_3Al	4.216	15.81	1600
TiAl	3.63	36.03	1460
$TiAl_3$	3.371	62.82	1340

Where the powder metal component comprises Nickel, due to the heat energy during the exothermic transformation from separate Ni and Al into NiAl intermetallic, the respective intermetallic component may be formed during the permeation operation without the need for a subsequent heat treatment to cause a solid state transformation to the respective intermetallic component. When the powder metal component comprises iron or titanium a combination of intermetallic formation during permeation and during a subsequent solid state heat treatment occurs. This condition is possible but it is not necessarily always like this. In fact, Ni, Ti and Fe have an essentially similar nature, that is, during infiltration of an aluminium melt a more or less partial reaction may occur depending upon the powder volume fraction, the melt temperature, the applied pressure, the particle morphology and hence, generally, the velocity of infiltration.

The "other" component if and when present may comprise chromium, manganese or niobium, or any desired combination of two or more of the above.

The intermetallic compound alternatively may comprise molybdenum silicide or niobium beryllides or chromides depending on the powder and liquid metal components.

At least one additional component may be introduced into the mould cavity prior to permeating the powder metal component with the liquid metal component. The non-metallic reinforcement may be added to the intermetallic compound to create a metal matrix composite (MMC) having modified thermophysical properties. The additional component may comprise ceramic particles such as silicon carbide or alumina particles, fibres such as silicon carbide or alumina fibres, or whiskers such as silicon carbide whiskers.

Other examples are relatively large diameter monofilaments, typically 120-140μm diameter or relatively small diameter multifilaments, typically 3-20μm diameter. Random fibres are usually about 3μm and over in diameter and have a typical fibre length of about 50μm. Whiskers are single crystals, so rarely grow larger than 0.5μm in diameter. Said additional component does not form an intermetallic compound with the liquid metal component.

The resultant net shape product comprising titanium aluminide intermetallic compound is illustrated at 23 in Figure 8 and may comprise a single product or may comprise a plurality of products depending on the configuration of the mould cavity 10.

One form of apparatus for applying pressure to the components is illustrated in Figure 9.

In this embodiment the mould cavity 10 is defined in a die 25, having an ingate I, and which in the present example is made by machining in a suitable die steel. The wall of the mould cavity is impermeable since the steel is impermeable itself and split lines in the die are provided with suitable seals. Above the die 25 is provided a melting chamber 26 in which a crucible 27 is positioned, surrounded by a heating coil 28 such as an induction or resistance heating coil. The crucible 27 is made of a suitable material to withstand the temperature and mechanical stresses and has a generally cylindrical interior 30.

A generally cylindrical ram 29 is positioned above the crucible 27. The ram 29 is adapted to be advanced into the interior 30 of the crucible 27 by a suitable mechanical means such as a hydraulic piston, illustrated diagrammatically at 31. The ram 29 in the present case is of generally cylindrical configuration and closely conforms to an internal surface 32 of the interior 30 of the crucible 27.

The cavity 10 is connected by a conduit 33 and a valve 34 to a vacuum system 35. A clamp 38 is provided to hold the die 25 in engagement with the melting chamber 26.

In use, the cavity 10 is filled with powder metal component as described hereinbefore, and the crucible 27 is filled with a desired amount of liquid metal component.

In this example the liquid metal is, substantially, retained in the crucible 30 simply by virtue of the liquid metal flowing through the opening 18 until it rests on the upper surface of the powder metal component in the cavity 10. If desired, however, liquid metal may be retained wholly within the crucible 27 by any other suitable means such as those described hereinbefore.

A degassing operation is carried out as described previously using the vacuum system 35 and then liquid metal is permitted to leave the crucible through the tap hole 18. The hydraulic piston 31 is operated to advance the ram 29 into the interior 30 of the crucible 27 and thus apply mechanical pressure to the molten metal in the crucible 27 and maintain this pressure in the range 10 - 30 bar as the molten metal is caused to permeate the powder metal component. The pressure may be maintained until the liquid metal component has solidified, or may be removed or reduced prior to complete solidification of the liquid metal component. The die 25 is then separated from the melting chamber 26 and is opened and the method continued as described hereinbefore.

Referring now to Figure 10, in this example the apparatus comprises a mould chamber 50 in which is housed an impermeable disposable ceramic mould 11 having an ingate I and made from plaster or refractory material such as silica, zirconia and/or alumina as described hereinbefore and defining a mould cavity 10. The mould cavity 10 in this embodiment has a feeder portion 19 similar to that described previously and illustrated in Figure 3. The mould 11 is rendered impervious by virtue of a silicate coating, as described hereinbefore. The interior of the mould chamber 50 is connected by a manifold 52 and valves 53, 54 to a vacuum system 55 or a pressure source 56 respectively.

Removably sealed to the mould chamber 50 is a melting chamber 57 which houses a crucible 58 surrounded by a heating coil 51. The crucible 58 has a hollow interior 59 into which molten metal is fed via a gate valve 61. A stopper rod 62 is provided to close a pouring hole 63 of the crucible 58.

A metal loading port 60 above the crucible 58 is connectable by a manifold 64 and valves 65, 66 to a pressure source 56 or a vacuum system 55 respectively.

The method is performed as described hereinbefore with reference to Figures 1 - 8, feed of liquid metal into the feed portion 19 being controlled by the stopper rod 62. Prior to feeding the molten metal into the feeder portion 19, the above described degassing operation is performed using the vacuum system 55.

When the metal pouring operation is completed the valves 53, 66 are closed and the valves 54, 65 opened so as to apply gaseous pressure to the top of the metal in the feeder portion 19 and thus pressuring the interior of the cavity 10.

In other respects the method is performed as described previously.

Referring now to Figure 11, a mould chamber 70 is releasably mounted on and sealed to a melting chamber 71 which is mounted for rotation about a horizontal axis 72, by virtue of a stub axle 73 received in a bearing 74 and a hollow stub axle 75 being received in a bearing 76. The bearings 74, 76 are supported by bearing frames 77. The hollow interior of the stub axle 75 is connected at one end by an annular aperture 78 to the interior of the mould chamber 70 and by virtue of a transverse aperture 79 at the other end and a valve 80 to a pressure source 81 and by a second, diametrically opposite transverse aperture 82 and a valve 83 to a vacuum system 84.

A crucible 85 is disposed within the melting chamber 71 and surrounded by a heating coil 86. An impermeable disposable ceramic mould 11, made as described hereinbefore, with at least one passage 89 thereof, is provided within the mould chamber 70 and is held in place by a clamp means 87.

Initially the liquid metal component 17 is introduced into the crucible 85 and then the mould chamber 70 is sealingly mounted on the melting chamber 71. A degassing operation is then performed using the vacuum system 84 with the valve 83 open and the valve 80 closed.

When the degassing operation has been completed and the melt temperature measured by a pyrometer 88, the assembly of melting chamber and mould chamber 71, 70 is rotated through 180° about the axis 72 so that liquid metal from the crucible 85 is poured into the feeder portion 19 and through the ingate I into the mould 11 and then the interior of the assembly 70, 71 is pressurised from the source 81 through the valve 80, the valve 83 being closed, and so gaseous pressure is applied to the surface of metal in the feeder portion 19 via the passage 89. The remainder of the process is performed as described hereinbefore.

In this version the powder material is either in the form of a rigid or semi-rigid preform, or is introduced into the mould cavity in flowable form held in place by a filter or is subsequently compacted to sufficient extent so as to retain its shape and position in the mould 11 whilst in an inverted position. Referring now to Figure 12.

In this embodiment the mould cavity 10 having an ingate I is defined in an impermeable permanent die 90 which in the present example is made by machining in a suitable die steel. Below the die 90 is provided a melting chamber 91 in which a crucible 92 is positioned, surrounded by a heating coil 93.

The lower end of a generally cylindrical feed tube 94 is immersed in the liquid metal component contained in the crucible 92. The upper end of the feed tube 94 is removably sealed to the inlet to the die 90.

The cavity 10 is connected by a conduit 95 and a valve 96 to a vacuum system 97. A valve 99 similarly connects the melting chamber 91 to the vacuum system 97.

In use, the cavity 10 is filled with powder metal component as described hereinbefore, and the crucible 92 is filled with a desired amount of liquid metal component.

A degassing operation is carried out as described previously using the vacuum system 97. Simultaneously vacuum valves 96 and 99 are closed and pressure valve 101 is opened applying pressure to the molten metal in the crucible. The molten metal is caused to rise up feed tube 94 and permeate the powder metal component. The pressure may be maintained until the liquid metal component has solidified whereupon the die 90 separated from the melting chamber 91 is opened and the method continued as described hereinbefore.

Referring now to Figure 13.

In this embodiment the mould cavity 10 is defined in an impermeable disposable ceramic mould 11 made from plaster or refractory material and which is rendered impervious by virtue of a silicate coating as described hereinbefore. The mould cavity is provided with an ingate I at the entrance to which is provided a filter 103 and the filter 103 also retains the powder metal component within the mould cavity. If desired the mould cavity may be provided with a plurality of ingates at desired positions to ensure proper filling of the mould cavity depending upon the mould cavity configuration. A filter or a plurality of filters 103 being provided for each ingate. The or each filter may comprise a porous ceramic material.

A crucible 106 provides a reservoir which is charged with liquid metal component to a level L and the temperature of the liquid metal component is controlled by an electrical heating coil 107. The mould 11 containing the powder metal component is mounted on a rod 104 which can be moved vertically through an opening 104<u>a</u> in a lid 105 of an enclosure or melting chamber 102. The lid 105 is removably sealed to the chamber 102 so as to provide a fluid tight enclosure.

The chamber 102 is provided with a degassing system 108 connectable with the interior of the chamber 102 by a valve 109 and with a gaseous pressure system 110 connectable to the interior of the chamber 102 by a valve 111.

A degassing operation is then performed as described previously using the vacuum 108 with the 109 open and the valve 111 closed.

When the degassing operation has been completed the mould 11 is immersed in the liquid metal in the crucible 106 by operation of the hydraulic piston 112 to lower the rod 104.

The valve 109 is closed and the valve 111 is open so as to apply gaseous pressure from the pressure system 110 to the liquid metal contained in the crucible 106. The liquid metal is thus caused to enter the mould cavity via the or each ingate and associated filter or filters 103 and thus permeates the powder metal component.

Fluid pressure is maintained until the two components have intimately combined or the reaction process is complete to form the intermetallic compound. Thereafter the mould 11 is withdrawn from the liquid metal by reversing the movement of the hydraulic piston 112. The valve 111 is closed and pressure in the chamber 102 reduced to atmospheric pressure and the lid 105 is then unsealed from the chamber 102 and the mould 11 removed. The remainder of the process is performed as described hereinbefore.

If desired, a plurality of moulds may be immersed in the crucible 106 by a suitable mounting on the rod 104 or by providing a plurality of rods 104 moveable simultaneously or independently by appropriate hydraulic pistons similar to the piston 112.

The temperature of the metal in the crucible 106 is maintained above its melting point but below the melting point of the intermetallic compound. For example, when the intermetallic compound comprises y titanium aluminide, melting point 1450°C, and the liquid metal component is aluminium, melting point 660°C, the aluminium may be maintained at a temperature lying in the range 700°C to 900°C.

A similar temperature differential, lying in the range 400°C to 1000°C, may be provided between the melting point of the liquid metal component and of the intermetallic compound where these differ from the particular example described hereinbefore.

Referring now to Figure 14, there is shown apparatus for use in a method similar to that described with reference to Figure 13 but Figure 14 shows an impermeable disposable ceramic mould 11 which defines a tubular or part tubular product from which it will be seen that a plurality of ingates I are provided disposed at desired positions over the whole of the wall of the mould 11 which defines the internal surface of the tubular product, or tubular part of the product, to be made. As previously mentioned, each ingate could comprise a single aperture or comprise a porous material or indeed a single ingate covering the whole internal surface could be provided. In this case, the ingate may have a plurality of discrete apertures or comprise a porous ceramic body.

Such a disposition of ingates on the internal surface facilitates the casting of products incorporating a tubular configuration.

Another example of the invention will now be described with reference to Figure 15 which is a view of the apparatus shown in Figure 9 and the same reference numerals have been used to refer to corresponding parts. The cavity 10 is again defined in an impermeable permanent die and, in the present example, is configured to produce an automotive engine piston. In this example, instead of the cavity 10 being filled with powder metal component as was the case of the embodiment previously described with reference to Figure 9, the cavity has the powder metal component introduced into a first part P1 of the mould cavity adjacent the bottom end thereof. The powder metal component may comprise, for example, a layer of nickel powder in flowable form, or alternately, if desired, nickel powder in the form of a rigid preform. In either case the powder metal component may be, for example, approximately 1-2mm thick and may be of shallow cup shape as shown in Figure 15 or of any other desired configuration to provide a desired part of the final product with desired properties.

Liquid metal component such as aluminium or a suitable aluminium alloy is then introduced into the mould cavity as described in connection with the previous embodiment described with reference to Figure 9 so that the powder metal component disposed in the first part P1 of the mould cavity is permeated with the liquid metal component and a second part P2 of the mould cavity, which does not contain a powder metal component is occupied with the desired amount of liquid metal component.

If desired, the first part of the mould cavity may have a powder metal component of a volume fraction introduced thereinto and a second part of the mould cavity adjacent the first part may have another powder metal component introduced thereinto of different volume fraction to the powder metal component in the first part. Liquid metal component is introduced into the mould cavity to permeate the powder metal components disposed in the first and second parts of the mould cavity and to occupy to a desired extent a third part of the mould cavity, not occupied by the powder metal components, with liquid metal component.

Where, for example, the powder metal component in the first part comprises nickel and the liquid metal component in the second part comprises aluminium or an aluminium alloy, the resultant product may have a region such as a surface part which comprises Ni Al, then a region comprising Ni_2 Al_3 and then a region comprising Ni Al_3 all as integral regions of the product, as the composition approaches that of aluminium or an aluminium alloy, the remainder of which comprises aluminium or aluminium alloy. The intermetallic compound, for example NiAl, Ni_2Al_3, or $NiAl_3$ may be of constant composition throughout the respective region. Alternatively, the composition of the intermetallic may vary in accordance with the phase diagram, according to the range of volume fraction provided in the region and/or according to the range of volume fraction which results from introduction of the liquid metal component into the region.

There may be transition regions between these parts where the composition comprises a mixture of the intermetallic components of the adjacent regions. For example, the transition region between the NiAl region and the Ni_2 Al_3 region may comprise a mixture of the two intermetallic compounds which varies from 100% NiAl progressively to 100% Ni_2 Al_3. Similarly for other transition regions.

If desired, a powder metal component or components may be disposed in any desired part or parts of the mould cavity and powder metal components of different volume fraction may be provided at different parts of the mould cavity.

If desired, the above mentioned partial occupation of the mould cavity by at least one powder metal component described hereinbefore with reference to Figure 15 may be applied in any of the other examples described hereinbefore and the parameters described hereinbefore in connection therewith are applicable thereto.

In all cases the volume fraction can be varied by powder metallurgical techniques for example by virtue of using different particle morphology, and/or size and/or by putting a different amount of powder in the or a desired region.

In all cases the reaction of the powder metal component and the liquid metal component to produce the intermetallic compound may occur either as the liquid metal part advances through the powder metal component or generally within the product, for example, after a short delay. When the reaction occurs in an intermediate body the reaction may again occur as described above. The composition of the intermetallic compound may vary without falling outside the scope of this patent application for example by a few percent although those stated are preferred compositions.

In all the embodiments described hereinbefore the pressure applied to the liquid component lies in the range 10 - 30 bar and is typically 20 bar.

However, higher pressures may be used, for example, up to 100-500 bar or even higher pressures eg 1000 bar may be used where, for example, conventional squeeze casting is used to apply a mechanical pressure directly to the liquid metal.

If desired a pressure below atmospheric pressure may be applied to the powder component and atmospheric pressure applied to the liquid metal component to cause or assist the permeation. Indeed, any suitable means of providing a pressure difference between the liquid and powder components in which the pressure acting on the liquid component is relatively greater than that acting on the powder component may be provided.

In this specification by powder metal we mean discrete particles of metal with a maximum dimension of less than 1mm (1,000 μm). Generally, the particles of the powder metal component are dry prior to introduction of the liquid metal component.

The features disclosed in the foregoing description, or the accompanying drawings, expressed in their specific forms or in terms of a means for performing the disclosed function, or a method or process for attaining the disclosed result, or a class or group of substances or compositions, as appropriate, may, separately or in any combination of such features, be utilised for realising the invention in diverse forms thereof.

Claims

1. A method of making a product comprising an intermetallic compound including the steps of permeating in a mould cavity a powder metal component, having a first melting point, with a liquid metal component, having a second

melting point which is lower than the first melting point, to produce a product comprising an intermetallic compound of said powder and liquid metal components.

2. A method as claimed in Claim 1 wherein said step of permeating a powder metal component with a liquid metal component is performed to produce an intermediate form and the method includes the step of heat treating the intermediate form to produce said product.

3. A method as claimed in Claims 1 or 2 wherein said step of permeating a powder metal component with a liquid metal component is performed under conditions to produce said product without a discrete step of heat treating an intermediate form.

4. A method as claimed in any one of the preceding claims wherein the powder metal component and the liquid metal component are subjected to pressure to cause or assist permeation of the powder metal component with the liquid metal component.

5. A method as claimed in any one of the preceding claims wherein the powder metal component comprises a rigid preform.

6. A method as claimed in any one of Claims 1 to 4 wherein the powder metal component is in flowable form.

7. A method as claimed in Claim 3 or any one of Claims 4 to 6 when dependent directly or indirectly on Claim 3 wherein after the step of permeating the powder metal component the liquid metal component is allowed to solidify and the solidified intermediate form is then be subjected to said heat treatment to effect a solid state phase transformation to provide said product comprising an intermetallic compound.

8. A method as claimed in any one of the preceding claims wherein the mould cavity has a wall which is impermeable as herein defined.

9. A method as claimed in or any one of the preceding claims wherein the liquid metal component is introduced into the mould cavity via an ingate from a reservoir.

10. A method according to Claim 9 wherein the liquid metal component is introduced into the mould cavity from the reservoir by virtue of the expedient comprising introducing the liquid metal through a passage which communicates with the ingate.

11. A method as claimed in Claim 10 wherein in the expedient

a) the or each ingate comprises an aperture or a plurality of apertures or a porous material such as a porous ceramic material,
b) the reservoir is disposed above the mould cavity and the liquid metal component is fed downwardly into the mould cavity through the passage, c) the mould and the reservoir are mounted for movement relative to a horizontal axis so that in a first position the mould is disposed above the reservoir and in a second position the mould is disposed below the reservoir so that metal can flow from the reservoir into the mould cavity under gravity,
d) the reservoir is disposed below the mould cavity and the liquid metal is fed upwardly into the mould cavity through the passage.

12. A method as claimed in Claim 9 wherein the liquid metal component is introduced into the mould cavity via the ingate by the expedient of immersing the ingate in a bath of said liquid metal component held in the reservoir.

13. A method as claimed in Claim 12 wherein in the expedient

a) the whole of the mould is immersed in the bath, and
b) wherein the ingate is not at the top of the mould cavity and only a part of the mould is immersed in the bath of liquid metal component and the mould cavity is filled by applying pressure to the liquid metal bath to force the liquid metal into the mould cavity, and/or
c) the introduction operation is performed so that the temperature of the metal in the bath is above the melting point of the liquid metal component but below the melting point of the intermetallic compound so that the

intermetallic compound will solidify within the mould cavity whilst the mould cavity is immersed or partly immersed in the bath, and the mould containing the solidified intermetallic compound can be removed from the bath, and/or

d) the wall of the mould defines a tubular mould cavity and at least one ingate is provided in the part of the wall of the mould which defines the internal surface of the tubular mould cavity.

14. A method as claimed in any of the preceding claims wherein the intermetallic compound comprises titanium aluminide, nickel aluminide, or iron aluminide.

15. A method as claimed in any one of the preceding claims wherein at least one additional component is introduced into the mould cavity prior to permeating the powder metal component with the liquid metal component.

16. A method as claimed in any one of the preceding claims wherein said step of permeating a powder metal component with a liquid metal component is performed to provide a product having a first portion which has a melting point which is higher than the melting point of a second portion of the product.

17. A method as claimed in Claim 16 wherein said step of permeating a powder metal component with a liquid metal component is performed with different atomic proportions of powder metal component and liquid metal component in at least one pre-determined part of the mould.

18. A method as claimed in Claim 16 or Claim 17 wherein the method comprises a step of introducing said powder metal component into a first part of the mould cavity and permeating the powder metal component in said first part with said liquid metal component and introducing another component, selected from a powder metal component or a liquid metal component, in a second part of the mould cavity not occupied by the powder metal component.

19. A method as claimed in Claim 18 wherein the other component is a liquid metal component.

20. A method as claimed in Claim 19 wherein the other component is at least one powder metal component the or each of which provides a different atomic proportion of powder metal component to liquid metal component to the proportion provided in the first part of the cavity and the method including a further step of permeating the or each other powder metal component with a liquid metal component.

21. A method as claimed in Claim 20 wherein the mould cavity comprises a third part unoccupied by a powder metal component and into which a liquid metal component is introduced.

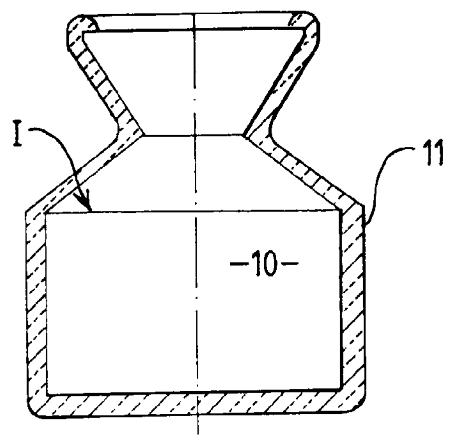


FIG 1

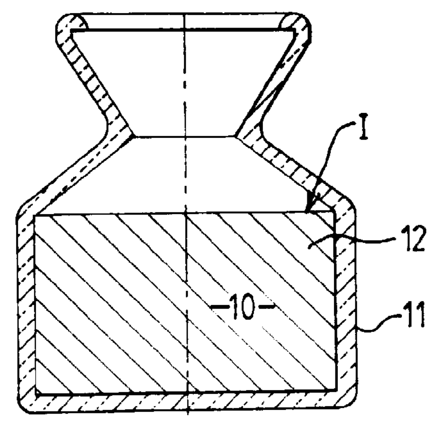


FIG 2

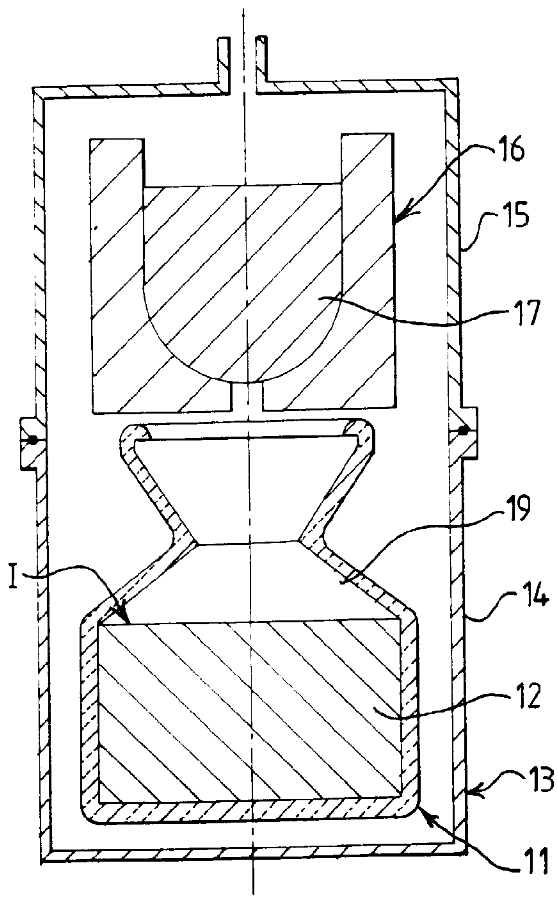


FIG 3

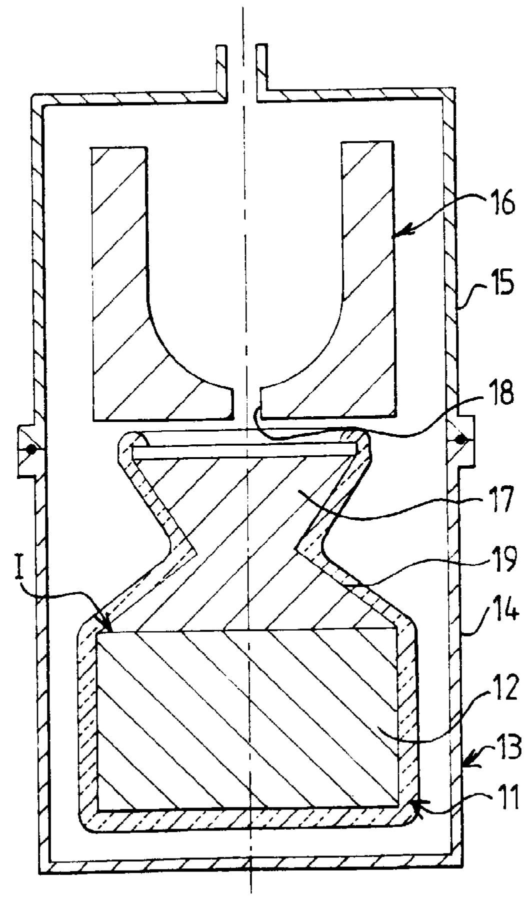


FIG 4

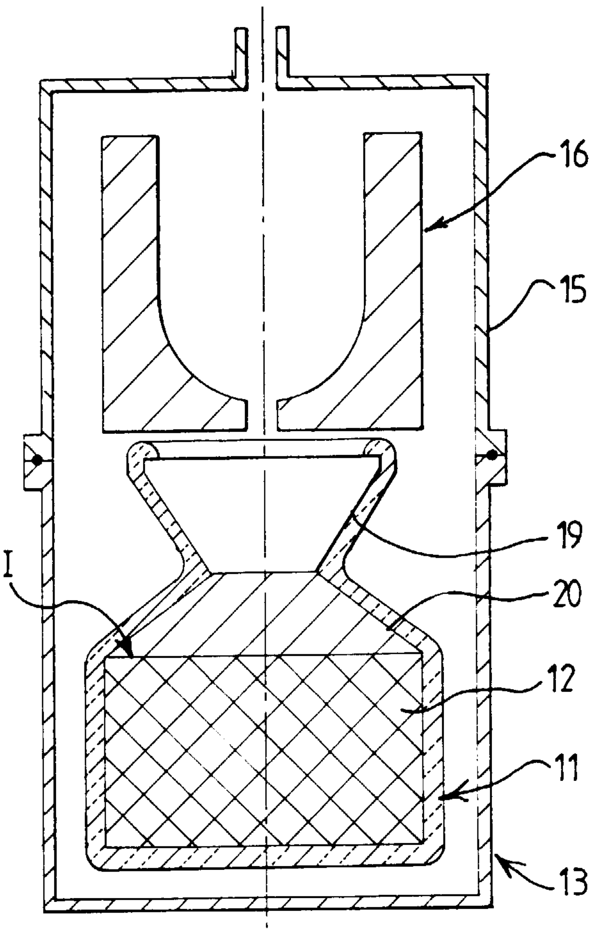


FIG 5

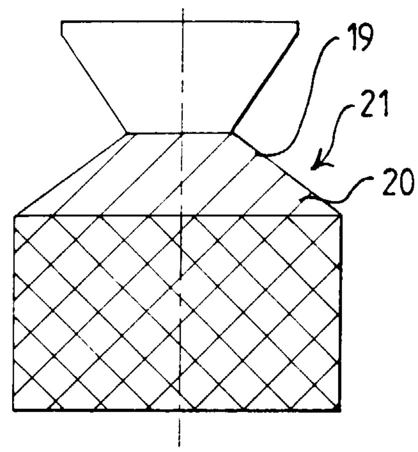


FIG 6

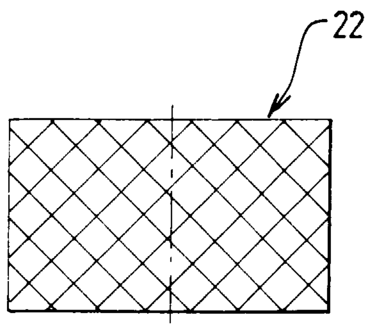


FIG 7

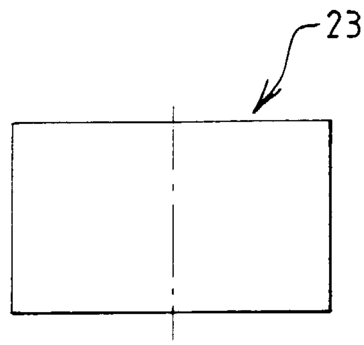


FIG 8

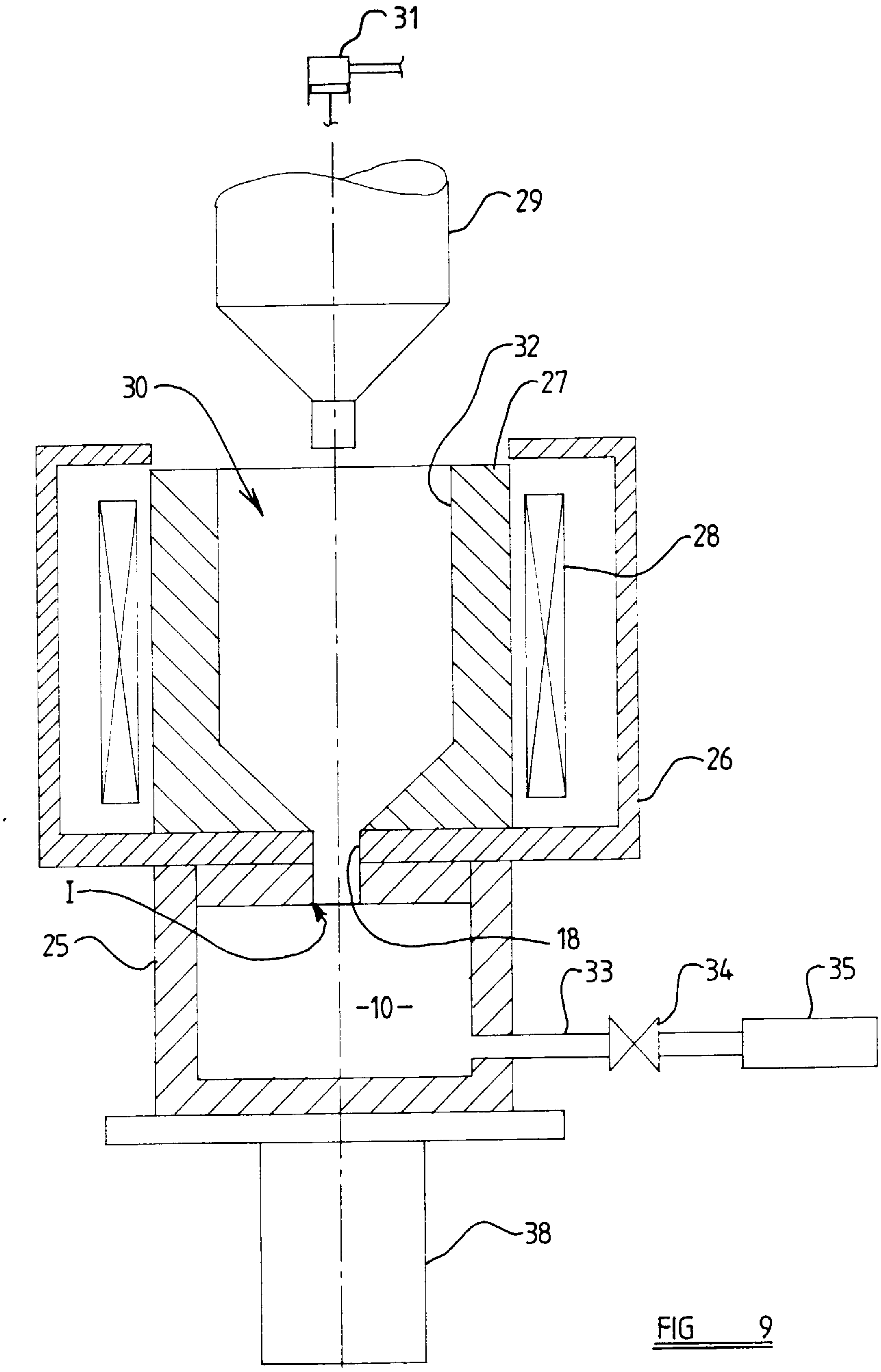


FIG 9

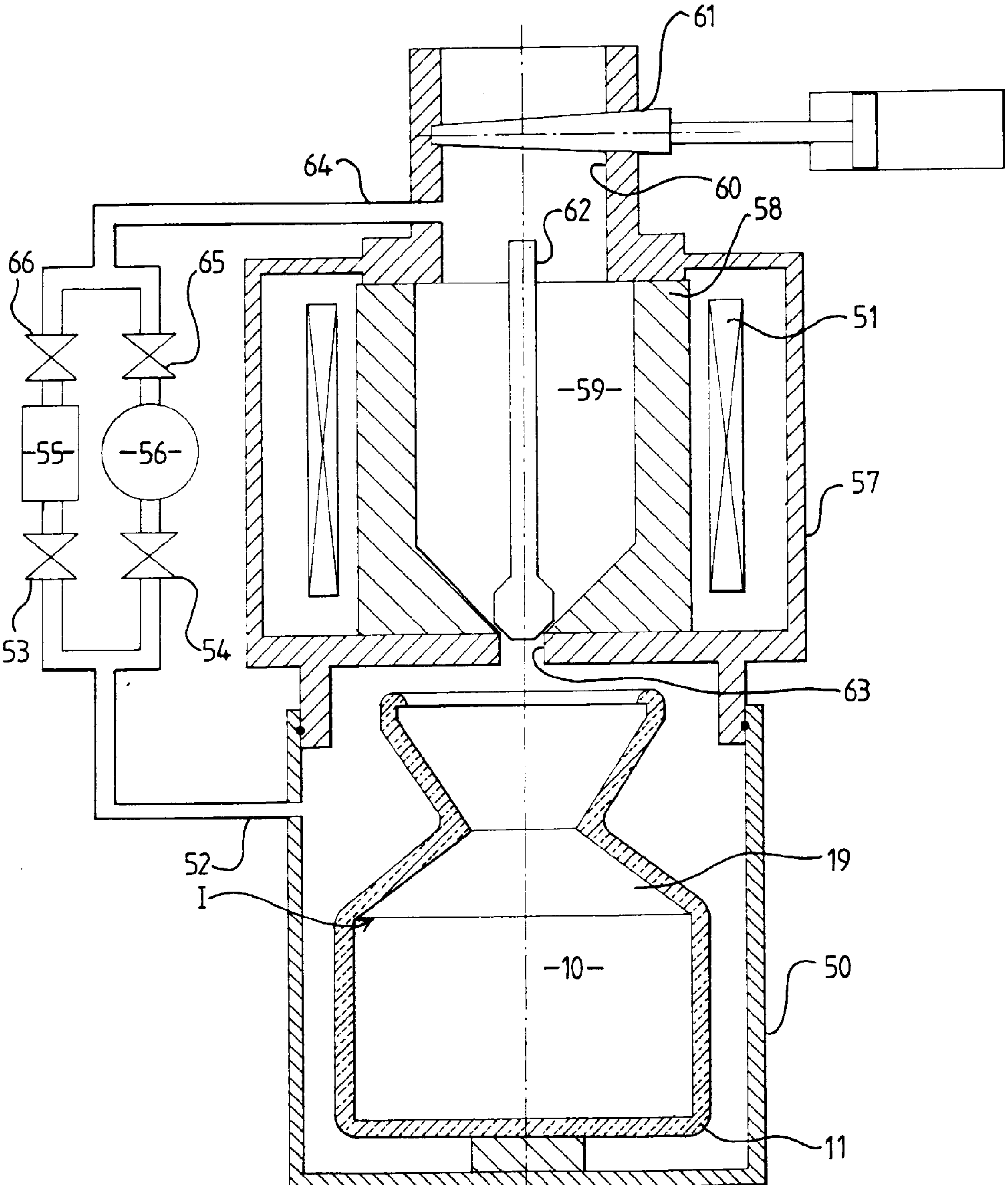


FIG 10

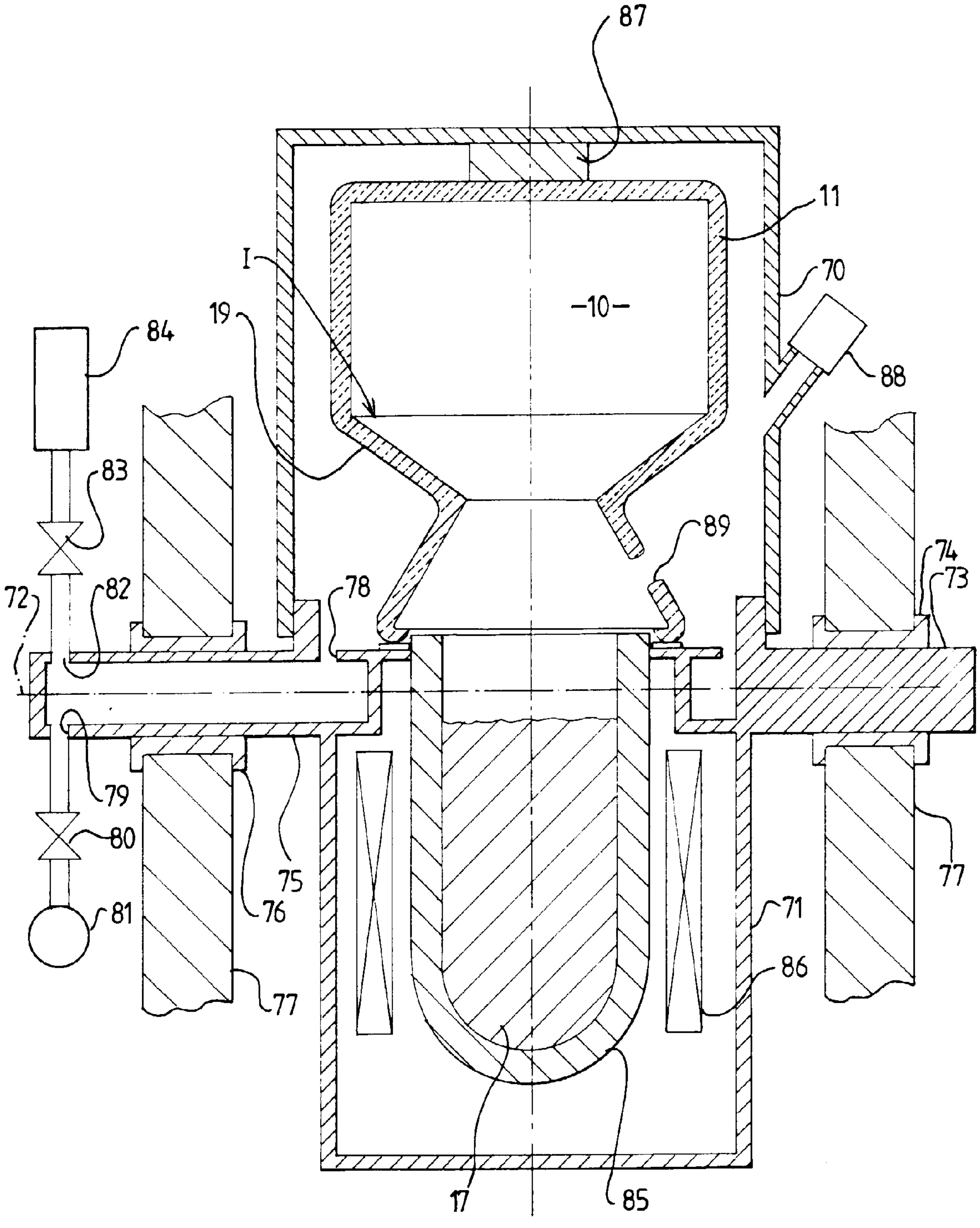

87
11
I
70
-10-
19
84
88
83
89
74
73
72
82
78
79
80
75
77
81
76
71
77
86
17
85

FIG 11

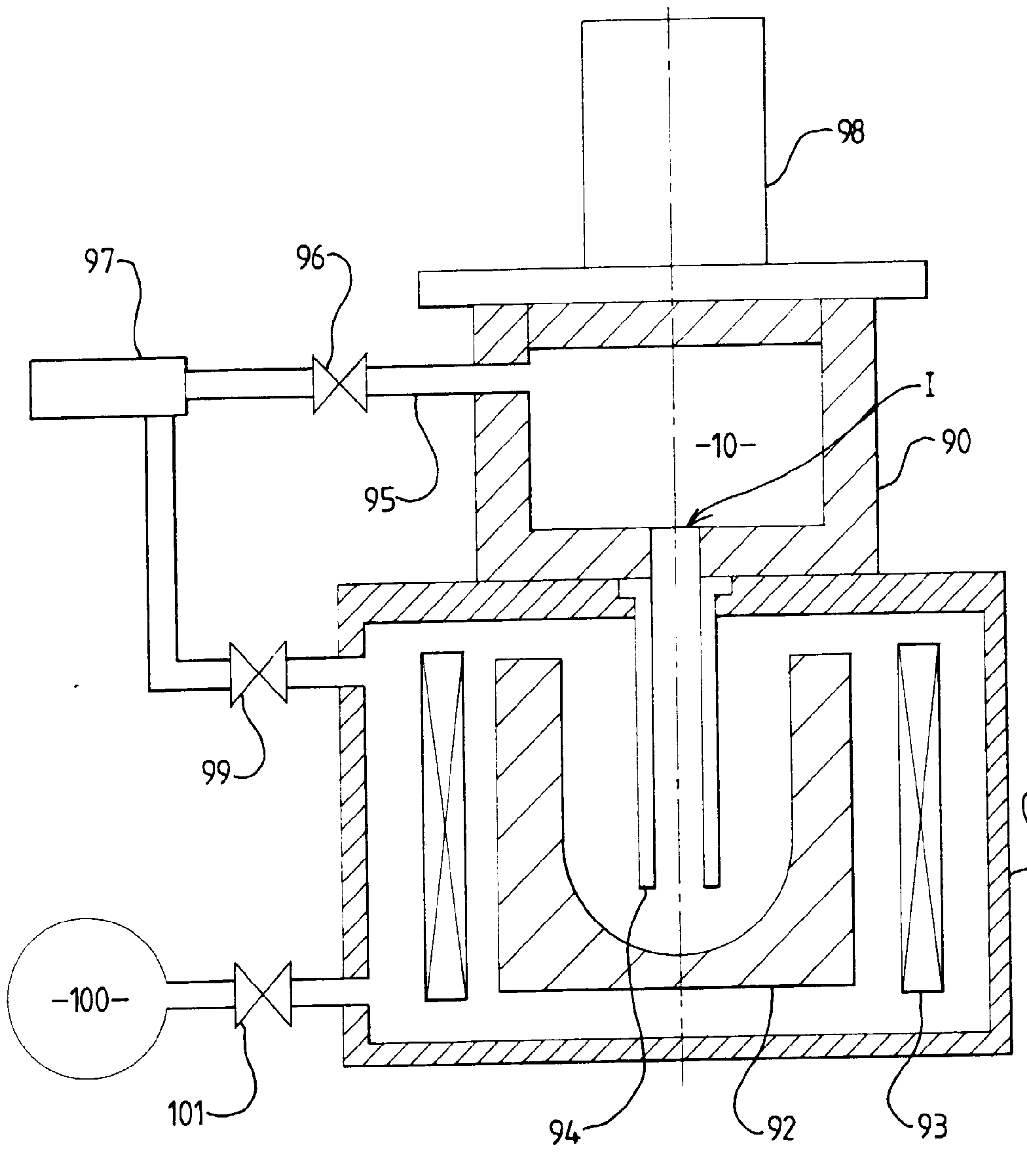


FIG 12

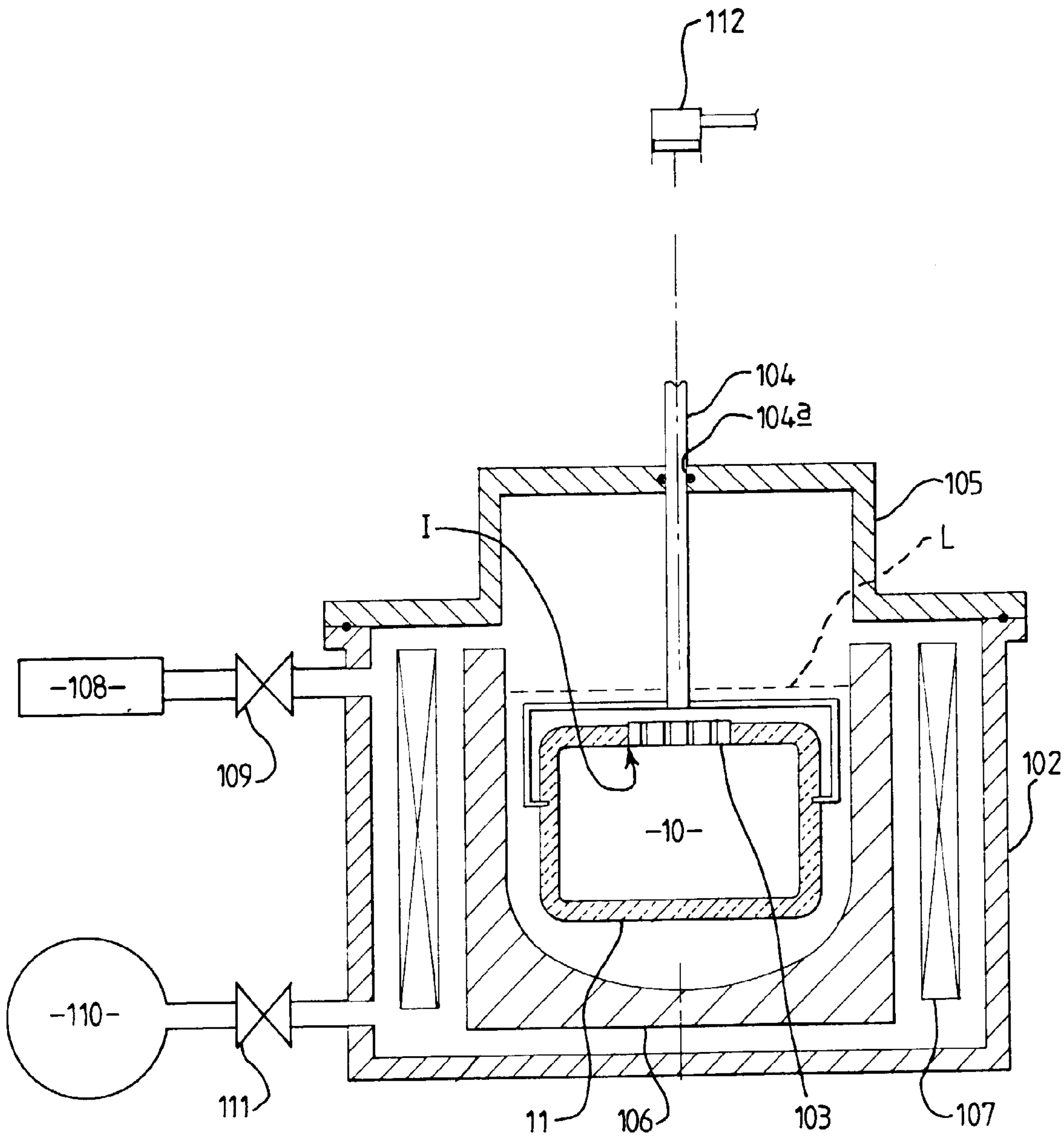
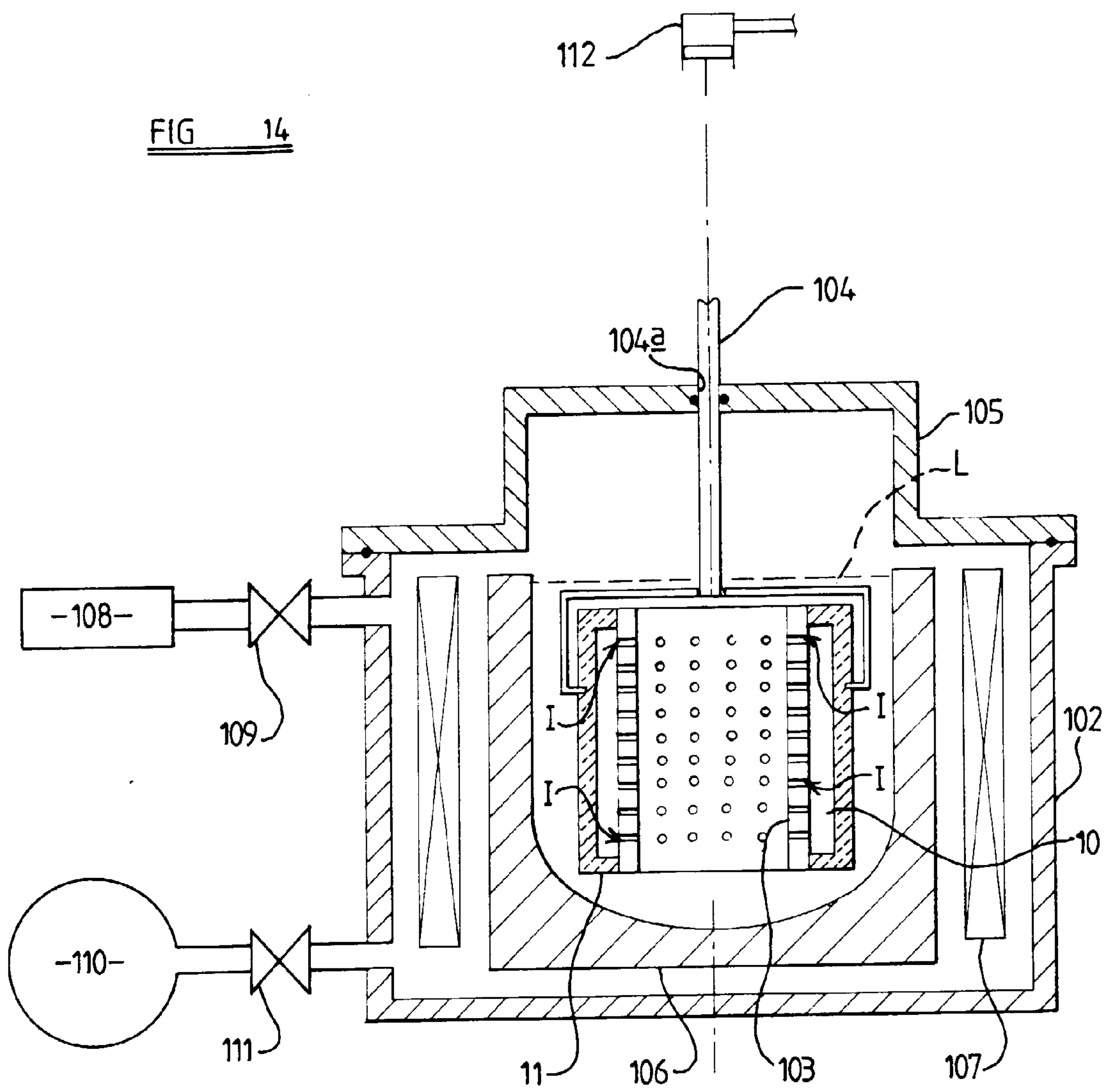


FIG 13

FIG 14



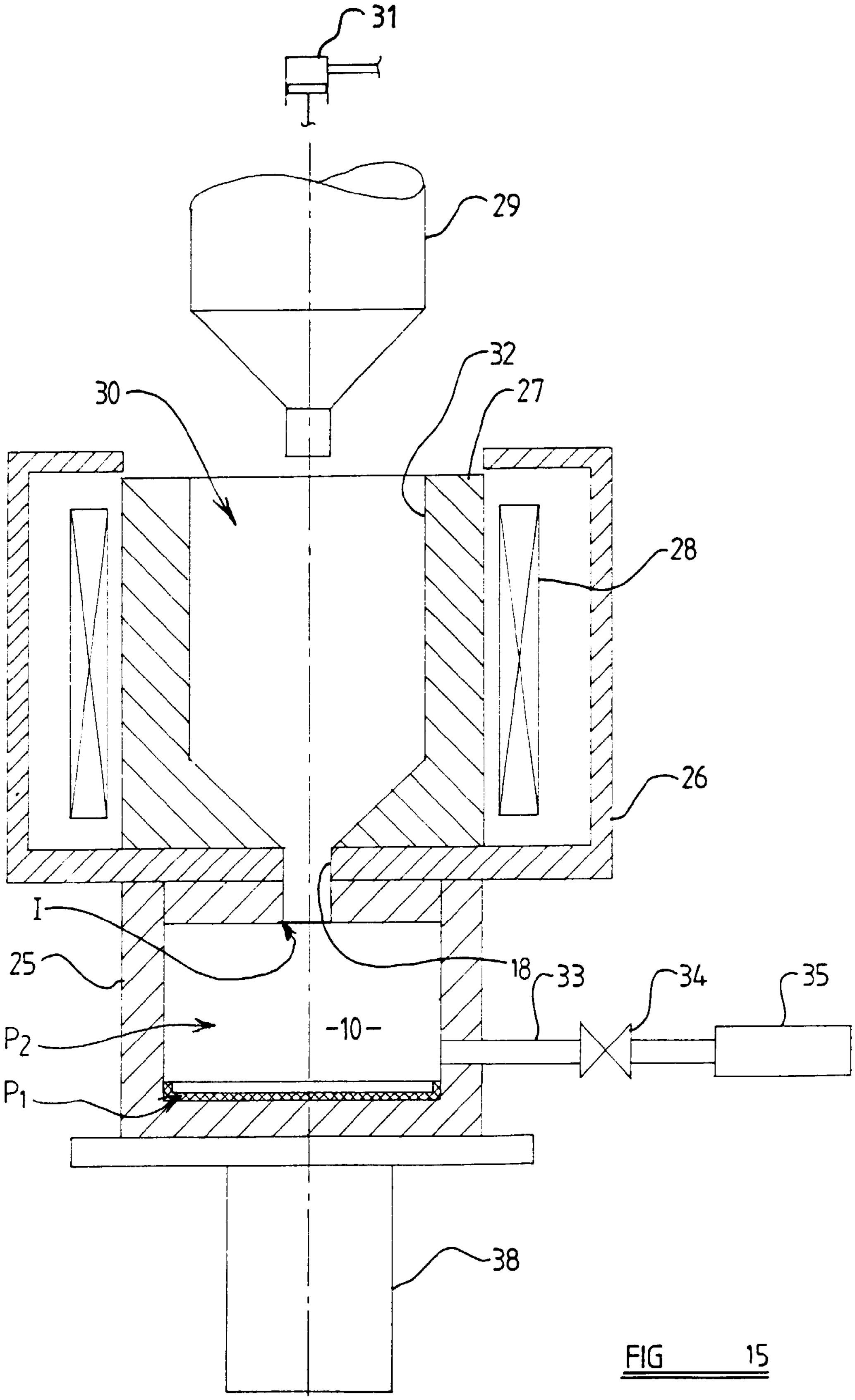


FIG 15

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 96 30 1717

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.6)
X	WO-A-94 21407 (MASSACHUSETTS INST TECHNOLOGY) 29 September 1994	1-6, 14-16, 18,20	C22C1/04
Y	* claims 1-5,9,15-20,24-26 *	8	

Y	US-A-5 015 440 (BOWDEN DAVID M) 14 May 1991 * column 1, line 22 - line 27; claims 1,10; example *	8	

A	DE-A-38 09 550 (VAW VER ALUMINIUM WERKE AG) 19 October 1989 * column 5, line 60 - column 6, line 2; claim 1 *		

A	WO-A-92 17297 (ALUMINUM CO OF AMERICA) 15 October 1992	9-13	

			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C22C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	25 June 1996	Schruers, H

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document