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(54) **ATOMISATION OF METALLIC MELTS USING CARBONATED WATER**

(57) A method of producing a metal powder for additive manufacturing process, the method comprising providing a stream of molten metal; atomising the metal

by directing jet of fluid towards the stream of molten metal, wherein the fluid comprises water and carbon dioxide.

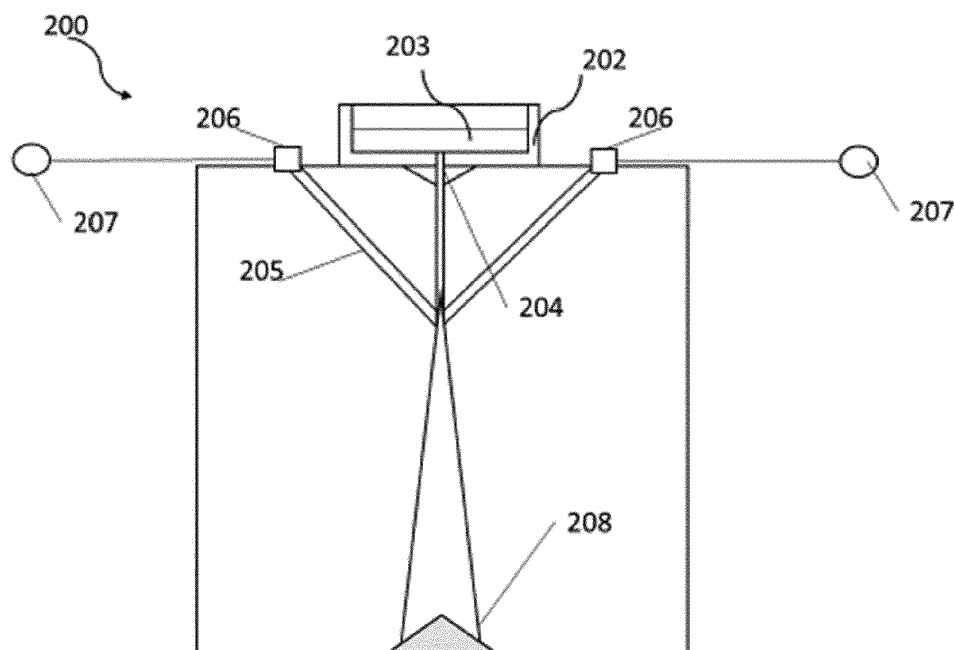


Figure 2

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Description

TECHNICAL FIELD

[0001] The present invention relates generally to a method for use in producing metal powder. In particular, but not exclusively, the present invention relates to a method of producing a metal powder for use in additive manufacturing process.

INTRODUCTION

[0002] In general, additive manufacturing (AM) is a manufacturing technology that includes a methodology whereby a heat source melts a feedstock of material which is deposited onto a substrate. Computer control of the movement of the heat source, and the source of the feedstock, makes it possible to build complex components.

[0003] As mentioned above, in additive manufacturing the material is deposited layer by layer. Some AM methods, for example, melt or soften the material to produce the layers, such as selective laser melting (SLM) or direct metal laser sintering (DMLS), selective laser sintering (SLS), fused deposition modelling (FDM), or fused filament fabrication (FFF). These additive manufacturing processes are summarized by the term laser powder bed fusion (L-PBF).

[0004] For these processes a metal powder is normally used, and for the repeatability of the process it is important to have the powder material with reproducible morphological characteristics, such as shape and size. To provide optimal performance in additive manufacturing operations, it is often desirable that the particles in the powder are as close as possible to spherical, and that they have a narrow size distribution. This facilitates good packaging of the powder and allows the powder to flow well.

[0005] Water atomisation, whereby the molten metal is treated with a stream of water at high pressure reaching 1000 bar, is a widely used technique to produce powder for additive manufacturing. Another technique to produce powder is gas atomisation, whereby an argon gas or a mixture of argon and nitrogen are used to atomise molten metal. For both of these atomisation techniques, the resultant metal particles are then sieved into fractions having predetermined size distributions.

[0006] It has hitherto been the case that gas atomisation produced particles having more spherical morphology than water atomisation, but it has generally been possible to atomise greater amounts of metal in a water atomisation apparatus than in a gas atomisation apparatus. However, it is known that the shape and size of the powder particles are also affected by the composition of the powder. Thus, for example, if iron alloy is used, the carbon content of the alloy affects the morphology of the particles during atomisation -the higher the carbon content, the more spherical powder particles are ob-

tained. It is believed that the evolution of CO₂ during the water atomisation of alloys having a high carbon content helps to produce this spherical morphology, although under certain circumstances it can also lead to the formation of hollow particles and other undesired shapes.

[0007] It is an object of this invention to at least mitigate one or more problems associated with the prior art and to provide a method for producing a metal powder for additive manufacturing process.

SUMMARY OF THE INVENTION

[0008] According to an aspect of the invention for which protection is sought, there is provided a method of producing a metal powder for additive manufacturing process, the method comprising:

providing a stream of molten metal;
atomising the metal by directing a jet of fluid towards the stream of molten metal,
wherein the fluid comprises water and carbon dioxide.

[0009] Advantageously, the high concentration of CO₂ around the metal during the atomisation process is believed to promote the formation of spherical particles. Furthermore, the presence of CO₂ in the atomising medium helps to reduce or even prevent evolution of CO₂ by reactions between the water and the carbon in the stream of molten metal.

[0010] In an embodiment, the carbon dioxide is dissolved in water. Advantageously, when the gas is dissolved in water, for instance by way of pre-mixing with water prior to contact with the molten metal, it allows for better interaction of metal particles with the carbon dioxide in the atomising medium, thus leading to a more efficient metal particle formation.

[0011] In another embodiment, the fluid further comprises carbon monoxide. The presence of carbon monoxide may serve the purpose of further preventing the depletion of carbon from the molten metal and/or reacting with iron to reduce oxides present in the metal.

[0012] In another embodiment, the ratio of carbon dioxide and water is at least 1 wt.%. Advantageously, at this ratio the amount of carbon dioxide is sufficient to interact with the molten metal and to suppress carbon depletion from its surface.

By control of CO₂ content, it is possible to tailor carbon content of produced melt, thus reducing or possible eliminating need of graphite addition to final powder mix. This would result in less hardness variations after sintering/heat-treatment related to component base carbon content.

[0013] In yet another embodiment, the jet of fluid is supplied through a nozzle. In an embodiment, the nozzle is a convergent-divergent nozzle. Advantageously, supplying both molten metal and the pressurised carbon dioxide through the nozzle having both a divergent and

convergent sections allows for better mixing at the point of exit of the two components due to a change in the speed of the components at the narrowing point, and thus preventing blockage of the nozzle opening.

[0014] In another embodiment, the fluid is supplied at between 100 and 1000 bar pressure. In yet another embodiment, the fluid is supplied at between 600 to 850 L/min. Advantageously, by pressurising the fluid and maintaining the flow between 600 and 850 L/min it is possible to achieve high fluid velocities before colliding the fluid with a molten stream of metal. This results in more efficient break down of the molten metal into droplets before the powder solidification occurs.

[0015] In an embodiment, the metal comprises iron or iron alloy, preferably a steel such as alloyed steel. Advantageously, the use of iron or alloyed steel allows for producing a wide variety of components for automotive, industrial, food processing and medical applications.

[0016] In another embodiment, the metal powder comprises substantially spherical solid particles having an aspect ratio between 1 and 1.2. Advantageously, the presence of the spherical or near-spherical particles allows for an enhanced powder flowability and better bulk density which, in turn, will lead to a better quality of the finished product. Smooth round particles possess better packing properties and therefore spread more evenly on the powder bed and form a uniform layer.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] Embodiments of the invention will now be described by way of example only, with reference to the accompanying figures, in which:

Figure 1 shows a system for producing metal powder using a water atomisation process;

Figure 2 shows a rough schematic view of carbonated water atomiser according to the present invention; and

Figure 3 shows a flow chart illustrating a method of producing a metal powder in an embodiment of the present invention.

DETAILED DESCRIPTION

[0018] Figure 1 shows a water atomisation system 100 for producing metal particles for additive manufacturing. The metal is supplied into a ladle or a melter 101 where it is heated up to the desired temperature, and then transferred into a tundish 102 to avoid splashing and give a smoother flow. A stream of the molten metal is then supplied into an atomising chamber 103 where it is mixed with water with carbon dioxide ("carbonated water") dissolved in it, said mixture is supplied through the nozzles 104a, 104b. The molten metal undergoes atomisation process under contact with carbonated water to form micron sized particles. It is understood that the carbonated water is supplied into the chamber 103 under pressure

of between 200 and to 1000 bar and a flow rate between 600 to 850 L/min. The water and metal particles form a slurry at the bottom of the chamber 103, said slurry comprising about 90% water and 10% metal particles. The slurry then is transferred either in a leaching tank 105 or into a dewatering system 106. The recovered water from either process is then transferred into a system 107 for cooling down, filtering and further recycling. A pressure or a high pressure pump 108 pumps the recycled water back into the atomising system. It is understood that an apparatus for conventional water atomisation can be also used for carbonated water atomisation, with minimal changes to the set-up required. In some embodiments, the nozzles 104a, b may be modified to supply a mixture of water and CO₂, and/or an additional unit may be provided for enriching water with carbon dioxide prior to contact with the molten metal. Alternatively, the CO₂ and water may be premixed and supplied as a ready to use solution, in which case a water atomisation system as shown in figure 1 may be used without structural modification.

[0019] Figure 2 shows an atomiser 200 to be used for producing metal powder according to an embodiment of the present invention. It comprises the chamber 201 where the atomising of the molten metal by the carbonated water occurs, and a furnace 202 connected to the chamber. A metal 203 is supplied to the furnace 202 and is subsequently heated to reach the melting temperature. It is understood that the metal 201 can be iron or an iron alloy. The iron alloy may include, besides other elements typically used in alloys, carbon from 0.03% to greater than 1% to increase hardness and strength of iron. The temperature in the furnace 202 depends on the type of metal or metal alloy and must be sufficient to maintain the metal in a molten state.

[0020] The molten metal 203 is then supplied into the chamber 201 through an opening 204. The molten metal 203 flows downwards under gravity and then it is subsequently treated with a one or more jets 205 of fluid. The fluid may be supplied into the chamber under pressure, then a pressure system 207 may be used. The fluid is supplied into the chamber 201 through a nozzle 206. It is understood that different kind of nozzles can be used, such as Venturi nozzle or de Laval nozzle, with the latter being the preferred choice of nozzle. Advantageously, by using the de Laval, or divergent-convergent, nozzle it is possible to achieve higher acceleration rates of the passing fluid, thus resulting in a better mixing of the fluid with the molten metal.

[0021] The fluid 205 can be water with pre-dissolved carbon dioxide. Optionally, the fluid can further comprise carbon monoxide, to achieve a more efficient suppression of the gas evolution from the molten metal 203.

[0022] It is known that during conventional gas and/or water atomisation process of metal particles manufacturing, a migration of carbon may occur for carbon-containing alloys. This is disadvantageous as the final composition of the alloy will change, thus in turn changing its

physico-chemical properties. In the present invention this problem is alleviated by using carbonated water as atomising medium, thus preventing the depletion of carbon from the final composition. Furthermore, it has been discovered that by using carbonated water it is possible to atomise a large amount of molten metal whilst retaining better control over the shape and size of the resultant metal particles than could be obtained using non-carbonated water.

[0023] Once the jet of fluid leaves the nozzle 206, it meets the stream of molten metal 204. Due to the rapid expansion of water and dissolved CO₂, the solid spherical or near-spherical uniform particles of metal powder 208 are formed and further deposited at the bottom of chamber 201. By the "spherical or near-spherical particles" it is understood that a majority of the particles have an aspect ratio of between 1 and 2, preferably between 1 and 1.5, more preferably between 1 and 1.2. It is hypothesised that the presence of a high concentration of CO₂ in the water around the particles as they are atomised assists with the formation of spherical particles.

[0024] Advantageously, the formation of spherical or near-spherical particles leads to a better spreadability of the resultant powder when it is subsequently used for an additive manufacturing process.

[0025] A method of producing a powder using carbonated water will be described in more detail below, with respect to Figure 3.

[0026] Figure 3 shows a method 300 which is implemented by a controller. In step 301 the controller is arranged to supply a molten metal 203 through an opening 204 of the furnace into the chamber 201. In step 302 the supply of carbonated water occurs through the at least one nozzle 206. It is understood that the controller also controls the carbonated water supply parameters, such as pressure and/or temperature. In step 303 the atomised powder 208 is produced by mixing the stream of molten metal 204 with the jet 205 of atomised water used as atomising medium. If it is determined that substantially all the metal has been transformed into powder, the method proceeds to step 305 at which the removal of water occurs. It will be understood that the removal of carbonated water can be performed using conventional equipment for dewatering of metal powders that have been water atomised.

[0027] If it is determined that the molten metal is still present in the furnace 202, then the process returns to step 302 of supplying more carbonated water into the nozzle. The carbonated water is supplied through the nozzles 104a, 104b as the jets which hit the stream of molten metal 203 at high velocity. The molten metal 203 is then atomised and deposited at the bottom of the chamber 103. Once all the previous steps 301-305 are completed, the process ends 306.

[0028] It will be appreciated that embodiments of the present invention can be realised in the form of hardware, software or a combination of hardware and software. Any such software may be stored in the form of volatile or

non-volatile storage such as, for example, a storage device like a ROM, whether erasable or rewritable or not, or in the form of memory such as, for example, RAM, memory chips, device or integrated circuits or on an optically or magnetically readable medium such as, for example, a CD, DVD, magnetic disk or magnetic tape. It will be appreciated that the storage devices and storage media are embodiments of machine-readable storage that are suitable for storing a program or programs that, when executed, implement embodiments of the present invention. Accordingly, embodiments provide a program comprising code for implementing a system or method as claimed in any preceding claim and a machine readable storage storing such a program. Still further, embodiments of the present invention may be conveyed electronically via any medium such as a communication signal carried over a wired or wireless connection and embodiments suitably encompass the same.

[0029] All of the features disclosed in this specification (including any accompanying claims, abstract and drawings), and/or all of the steps of any method or process so disclosed, may be combined in any combination, except combinations where at least some of such features and/or steps are mutually exclusive.

[0030] Each feature disclosed in this specification (including any accompanying claims, abstract and drawings), may be replaced by alternative features serving the same, equivalent or similar purpose, unless expressly stated otherwise. Thus, unless expressly stated otherwise, each feature disclosed is one example only of a generic series of equivalent or similar features.

[0031] The invention is not restricted to the details of any foregoing embodiments. The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any method or process so disclosed. The claims should not be construed to cover merely the foregoing embodiments, but also any embodiments which fall within the scope of the claims.

Reference signs

[0032]

100	- water atomisation system
101	- ladle/melter
102	- tundish
103	- chamber
104a, 104b	- nozzles
105	- leaching tank

106	- dewatering system		
107	- cooling/filtering/recycling system		
108	- (High Pressure) Pump	5	
200	- atomiser		
201	- chamber		
202	- furnace	10	
203	- molten metal		
204	- opening	15	
205	- jet		
206	- nozzle		
207	- pressure system	20	
208	- metal powder		

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Claims

1. Method of producing a metal powder (208) for additive manufacturing process, the method comprising:
 - providing a stream of molten metal (203);
 - atomising the metal by directing jet of fluid (205) towards the stream of molten metal, wherein the fluid comprises water and carbon dioxide.
2. The method of claim 1, wherein the carbon dioxide is dissolved in the water.
3. The method of claim 1 or 2, wherein the fluid (205) further comprises carbon monoxide.
4. The method of claim 1 or 2, wherein the ratio of carbon dioxide to water is at least 1 wt%.
5. The method of any preceding claim, wherein the jet of fluid (205) is supplied through a nozzle (206).
6. The method of claim 5, wherein the nozzle (206) is a convergent-divergent nozzle.
7. The method of claim 1, wherein the fluid (205) is supplied at between 100 and 1000 bar pressure.
8. The method of claim 1, wherein the fluid (205) is supplied at a flow rate between 600 to 850 L/min.
9. The method of any preceding claim, wherein the metal (203) comprises iron or iron alloy.

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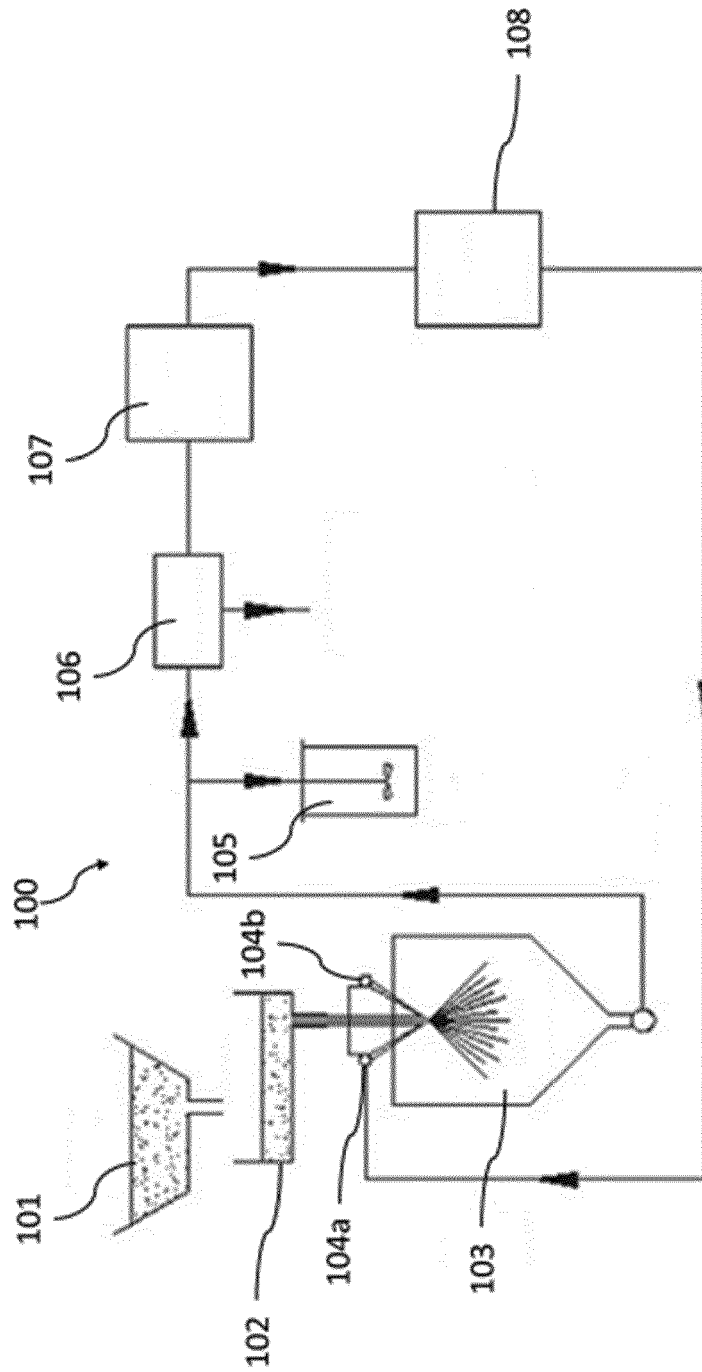


Figure 1

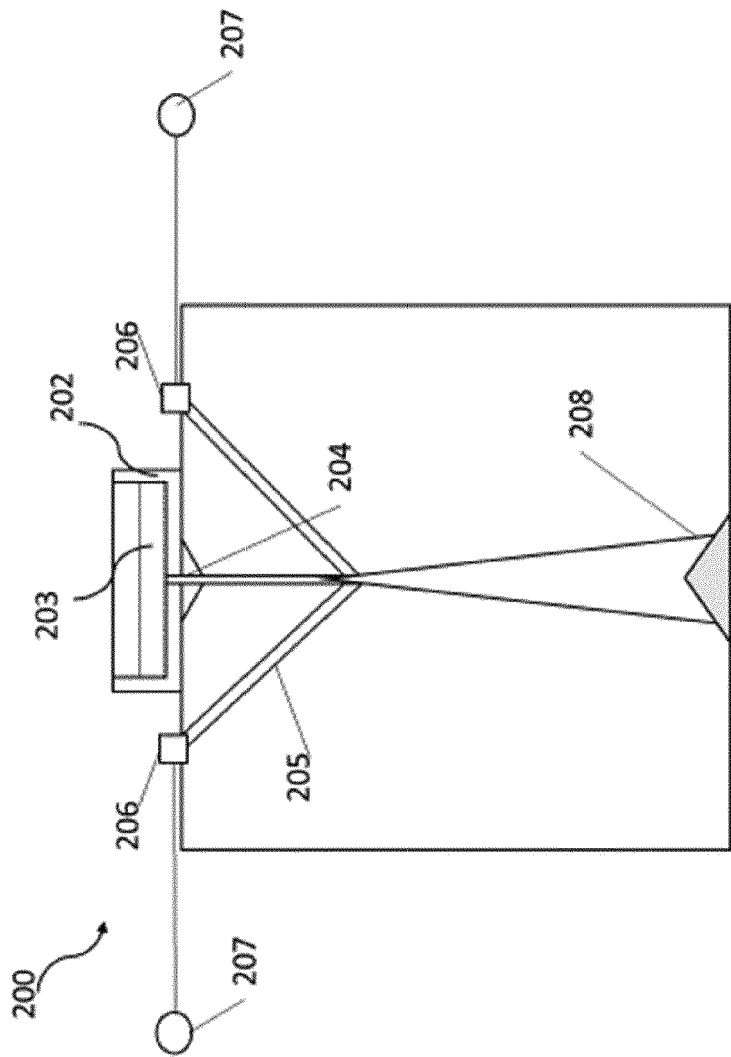


Figure 2

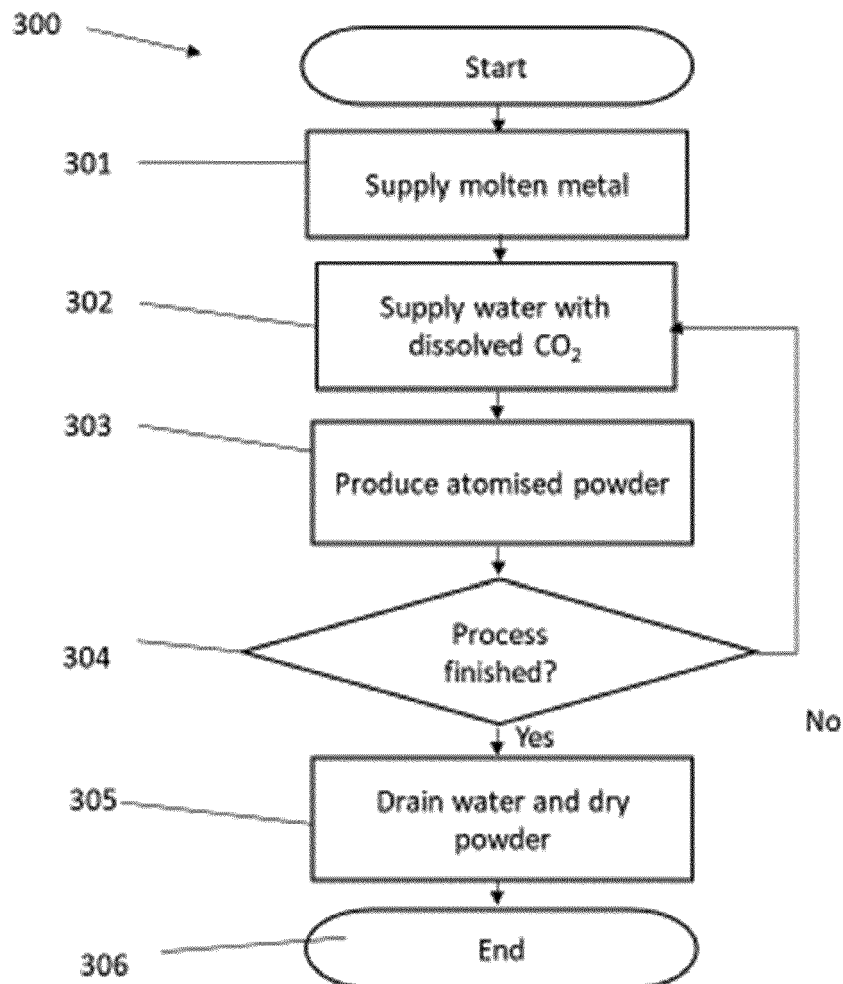


Figure 3



EUROPEAN SEARCH REPORT

 Application Number
 EP 20 02 0640

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			TECHNICAL FIELDS SEARCHED (IPC)
			B22F
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
The Hague		5 March 2021	Forestier, Gilles
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			

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 EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 20 02 0640

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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