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Remarks:

Amended claims 1-5 in accordance with Rule 137(2)
EPC.

(54) **Atmospheres for metal atomization including silane or borane**

(57) The invention relates to a method for producing a metal powder by atomizing a molten metal by means of an atomization gas wherein said atomization gas,

which is based e.g. on nitrogen, comprises a gaseous hydride, e.g. silane or borane.

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Description

[0001] The invention relates to a method for producing a metal powder by atomizing a molten metal by means of an atomization gas.

[0002] Metal atomization is a process for the production of fine metal powders. The raw material is melted and then the liquid metal is broken into fine particles. This is achieved by atomizing a flow of the liquid metal by an atomization agent such as water or a gas. The atomization agent strikes the liquid metal at a high velocity whereby the molten metal is disintegrated into fine droplets which solidify thereafter.

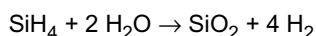
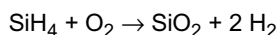
[0003] During the production process the fine metal particles absorb oxygen from the atomization agent which might react with alloying elements and might cause problems in the production of special alloyed metal powder. Thus, instead of using water or compressed air as atomization agent it is already known to atomize the molten metal with an inert gas, for example nitrogen or argon.

[0004] US 4,124,377 discloses a method for producing metal powder wherein the liquid metal is subjected to a reducing atomization agent such as hydrocarbon. In order to protect the powder against oxidation, the actual pulverization process is performed in a closed chamber which is partially filled with a reducing liquid and is under pressure from a gaseous reducing agent.

[0005] It is an object of the present invention to provide a method for atomization of metal wherein oxidation of the produced metal powder is avoided or at least significantly reduced.

[0006] This object is achieved by a method for producing a metal powder by atomizing a molten metal by means of an atomization gas which is characterized in that said atomization gas comprises a gaseous hydride.

[0007] Gaseous hydrides such as silane are known to have a very high reactivity with oxygen containing substances or compounds. For example, at room temperature silane or mono silane undergoes a spontaneous reaction with oxygen as well as with air:



[0008] By the inventive addition of a gaseous hydride to the atomization gas any oxygen remnants or any water vapour present reacts with the gaseous hydride. Thus, the oxygen content in the atomization gas is extremely reduced and an atomization gas with an extreme low dew point and low partial pressure of oxygen is achieved.

[0009] The invention relates to the atomization of molten metal. The term "atomization" in general means the disintegration of the molten metal by addition of a jet stream of an atomization agent. Since the invention is related to the use of a gaseous atomization agent in the following the term "atomization" shall mean the disintegration of molten metal by means of a high speed gas stream.

[0010] Preferably silanes or boranes are added to the atomization gas. Silanes are chemical compounds of silicon and hydrogen. The lowest silanes, mono silane with the chemical formula SiH_4 and di silane with the chemical formula Si_2H_6 , are gaseous and thus are particularly suitable for the invention.

[0011] Boranes are chemical compounds of boron and hydrogen. The two smallest members of the borane group are mono borane or simply borane BH_3 and di borane B_2H_6 . All these compounds are known to be very reactive with oxygen and air. Thus, these boranes are also preferably used for reducing the oxygen content of the atomization gas.

[0012] Prior to atomization the metal is held in the molten state. During that stage there is also a risk that the molten metal is oxidized by contact with the surrounding atmosphere. Thus the molten metal is preferably subjected to a particular process atmosphere with a low oxygen content.

[0013] The molten metal is then mixed with a high velocity stream of the atomization gas and sprayed in order to get fine metal droplets. It has also been found advantageous to spray the resulting metal droplets into a cooling atmosphere with reduced oxygen content.

[0014] Thus, according to a preferred embodiment of the invention the gaseous hydride is not only used to reduce the oxygen content of the atomization gas but also utilized to reduce the oxygen content of the process atmosphere and/or of the cooling atmosphere. Especially it is preferred to add a gaseous hydride to the atomization gas and to the process atmosphere and to the cooling atmosphere.

[0015] The amount of the gaseous and reactive hydride which is added to the atomization gas, the process atmosphere and/or the cooling atmosphere is preferably between 0.00001 % and 2 %, most preferably between 0.001% to 0,05%. The amount of hydride is determined based on one or more of the following factors:

- quality (especially oxygen content) of the atomization gas prior to the addition of the hydride,
- quality (especially oxygen content) of the process atmosphere prior to the addition of the hydride,
- quality (especially oxygen content) of the cooling atmosphere prior to the addition of the hydride,

- required level of oxygen and water in the atomization gas,
- required level of oxygen and water in the process atmosphere,
- required level of oxygen and water in the cooling atmosphere,
- type of metal and alloying elements which shall be atomized ,
- grain size of the metal powder product,
- desired quality of the metal powder product.

[0016] According to the invention it is possible to extremely decrease the oxygen and moisture level of the atomization gas, in the process atmosphere and/or in the cooling atmosphere. Thus, it is possible to atomize metals which could not have been treated in this way before. Further, the invention allows to produce very fine-grained metal powder even if the metal comprises easily oxidizing alloying elements.

[0017] In general, the invention is useful for the atomization of all kind of metals. Examples for preferred metals and metal powders to be used with the inventive method are iron, low alloyed steel, austenitic stainless steel, ferritic stainless steel and duplex stainless steel, copper and copper alloys, nickel and nickel alloys.

[0018] According to a preferred embodiment of the invention a little amount of silane is added to the atomization gas, to the process atmosphere and/or to the cooling atmosphere. The atmosphere will preferably contain pure nitrogen with addition of silane.

[0019] In general, the inventive atomization gas is either plain nitrogen, plain hydrogen, plain argon or a mixture of these gases blended with a gaseous hydride. Preferred gases or gas mixtures are

- plain nitrogen,
- a mixture of an inert gas and hydrogen,
- a mixture of nitrogen and hydrogen,
- a mixture of argon and hydrogen,
- a mixture of nitrogen, argon and hydrogen,

wherein to each of the above atmospheres a gaseous hydride, especially silane, is added.

[0020] The same applies to the process atmosphere and to the cooling atmosphere. It is also preferred to use an inert gas blended with a gaseous hydride, especially with silane. Preferred mixtures are

- plain nitrogen plus a gaseous hydride,
- a mixture of an inert gas and hydrogen plus a gaseous hydride,
- a mixture of nitrogen and hydrogen plus a gaseous hydride,
- a mixture of argon and hydrogen plus a gaseous hydride,
- a mixture of nitrogen, argon and hydrogen plus a gaseous hydride.

[0021] The mixture of nitrogen and hydrogen or the mixture of argon and hydrogen are preferably produced by blending pure nitrogen with hydrogen or pure argon with hydrogen, respectively.

[0022] The inventive method is not only useful to decrease the oxygen and water vapour content of the atomization gas, of the process atmosphere and/or of the cooling atmosphere. It can also be used as a reducing agent to react with other impurities present in the atomization gas, of the process atmosphere and/or of the cooling atmosphere, for example organic compounds.

Claims

1. Method for producing a metal powder by atomizing a molten metal by means of an atomization gas, **characterized in that** said atomization gas comprises a gaseous hydride.
2. Method according to claim 1 **characterized in that** a process atmosphere is provided above said molten metal and wherein said process atmosphere comprises a gaseous hydride.
3. Method according to any of claims 1 or 2 **characterized in that** after atomizing said molten metal the resulting metal powder is subjected to a cooling atmosphere comprising a gaseous hydride.
4. Method according to any of claims 1 to 3 **characterized in that** said atomization gas, said process atmosphere and/or said cooling atmosphere comprise silane or borane.

5. Method according to any of claims 1 to 4 **characterized in that** the concentration of said gaseous hydride in said atomization gas, in said process atmosphere and/or in said cooling atmosphere is between 0.00001 % and 2 %, preferably between 0.001% to 0,1%.

6. Method according to any of claims 1 to 5 **characterized in that** said atomization gas, said process atmosphere and/or said cooling atmosphere consist of

- nitrogen and said gaseous hydride or of
- argon, hydrogen and said gaseous hydride or of
- an inert gas and hydrogen and said gaseous hydride.

Amended claims in accordance with Rule 137(2) EPC.

1. Method for producing a metal powder by atomizing a molten metal by means of an atomization gas, **characterized in that** said atomization gas comprises a gaseous hydride and that the concentration of said gaseous hydride in said atomization gas, in said process atmosphere and/or in said cooling atmosphere is between 0.00001 % and 2 %, preferably between 0.001 % to 0,1%.

2. Method according to claim 1 **characterized in that** a process atmosphere is provided above said molten metal and wherein said process atmosphere comprises a gaseous hydride.

3. Method according to any of claims 1 or 2 **characterized in that** after atomizing said molten metal the resulting metal powder is subjected to a cooling atmosphere comprising a gaseous hydride.

4. Method according to any of claims 1 to 3 **characterized in that** said atomization gas, said process atmosphere and/or said cooling atmosphere comprise silane or borane.

5. Method according to any of claims 1 to 4 **characterized in that** said atomization gas, said process atmosphere and/or said cooling atmosphere consist of

- nitrogen and said gaseous hydride or of
- argon, hydrogen and said gaseous hydride or of
- an inert gas and hydrogen and said gaseous hydride.



European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 07 02 0458

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	DE 44 12 768 A1 (UNIV IOWA RES FOUND [US]) 20 October 1994 (1994-10-20) * the whole document *	1-6	INV. B22F9/08
X	US 5 147 448 A (ROBERTS PETER R [US] ET AL) 15 September 1992 (1992-09-15) * the whole document *	1,4	
X	US 2004/065171 A1 (HEARLEY ANDREW K [GB] ET AL) 8 April 2004 (2004-04-08) * paragraphs [0041], [0042] *	1,4	
			TECHNICAL FIELDS SEARCHED (IPC)
			B22F
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 3 June 2008	Examiner von Zitzewitz, A
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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 07 02 0458

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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03-06-2008

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 4412768 A1	20-10-1994	JP 7048609 A US 5368657 A	21-02-1995 29-11-1994
US 5147448 A	15-09-1992	NONE	
US 2004065171 A1	08-04-2004	AU 2003279056 A1 WO 2004031642 A2 US 2004213998 A1	23-04-2004 15-04-2004 28-10-2004

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 4124377 A [0004]