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Datasheet for the decision of 29 July 2008

Case Number: T 1473/06 - 3.2.03

Application Number: 04250309.4

Publication Number: 1440752

IPC: B22F 9/18, B23K 35/00, C23C 20/00

Language of the proceedings: EN

Title of invention:
Fabrication and utilization of metallic powder prepared without melting

Applicant:
GENERAL ELECTRIC COMPANY

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56

Relevant legal provisions (EPC 1973):
-

Keyword:
"Primary request - novel (yes)"
"Inventive step (no)"
"Auxiliary request - remittal to Examining Division"

Decisions cited:
-

Catchword:
-

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Case Number: T 1473/06 - 3.2.03

D E C I S I O N
of the Technical Board of Appeal 3.2.03
of 29 July 2008

Appellant: GENERAL ELECTRIC COMPANY
1 River Road
Schenectady
NY 12345 (US)

Representative: Goode, Ian Roy
London Patent Operation
General Electric International, Inc.
15 John Adam Street
London WC2N 6LU (GB)

Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 13 April 2006 refusing European application No. 04250309.4 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: U. Krause
Members: G. Ashley
K. Garnett

Summary of Facts and Submissions

I. European patent application EP-A-1 440 752 concerns a method of making a metallic powder by chemical reduction and then applying it to a substrate. The Examining Division was of the view that the methods defined in claims 1 of both the main and auxiliary requests lacked novelty (Article 54 EPC); it therefore decided to refuse the application.

II. In reaching its decision, the Examining Division took the following documents into account:

D3: JP-A-01 294810
D4: JP-A-11 291087
D5: US-A-2002/073804
D6: GB-A-883429

The patent application itself refers, amongst other documents, to:

D7: US-A-5 958 106

III. The decision was posted on 13 April 2006; the Appellant (applicant) filed notice of appeal on 8 June 2006, paying the appeal fee at the same time; a statement containing the grounds of appeal was filed on 22 August 2006.

IV. In accordance with Article 15(1) of the Rules of Procedure of the Boards of Appeal, the Board issued a preliminary opinion together with a summons to attend oral proceedings, setting out its view on the issue of

novelty. The oral proceedings were duly held on 29 July 2008.

V. During the oral proceedings, the Appellant submitted two sets of claims as its primary and auxiliary requests, and requested that the decision of the Examining Division be set aside and a patent be granted on the basis of the claims submitted as the primary or auxiliary request.

VI. Claim 1 of the primary request reads as follows:

"1. A method for fabricating and utilizing a metallic alloy made of metallic constituent elements, comprising the steps of:

furnishing a mixture of nonmetallic precursor compounds of the metallic constituents;

chemically reducing the mixture of nonmetallic precursor compounds to produce a metallic alloy as a metallic alloy powder comprising a plurality of particles, each particle comprising the metallic alloy material, without melting the metallic alloy powder and without melting the nonmetallic alloy precursor compounds (20); and thereafter

applying the metallic alloy powder to a surface of a substrate article (30)."

Dependent claims 2 to 11 define preferred embodiments of the method of claim 1.

Claim 1 of the auxiliary request is as follows:

"1. A method for fabricating and utilizing a metallic alloy made of metallic constituent elements, comprising the steps of:

furnishing a mixture of nonmetallic precursor compounds of the metallic constituents;

chemically reducing the mixture of nonmetallic precursor compounds to produce a metallic alloy as a metallic alloy powder comprising a plurality of particles, each particle comprising the metallic alloy material, without melting the metallic alloy powder and without melting the nonmetallic alloy precursor compounds (20), further comprising controlling the oxygen content of the metallic alloy powder by not carrying the chemical reduction process to completion in a fused salt solid phase reduction process or by adding oxygen to react with the metallic alloy material in a rapid plasma quench reduction process; and thereafter

applying the metallic alloy powder to a surface of a substrate article (30)."

Dependent claims 2 to 8 are as those of the primary request.

VII. Submissions of the Appellant

The Appellant's submissions concerning inventive step starting from D7 are relevant for this decision.

The Appellant argued essentially that there is no incentive in D7 to apply the powder to a substrate. The inventors have identified particular advantages in

powders produced in the absence of melting that lead to improved materials for application to substrates. In particular, there is a reduction in defects, such as inclusions, segregation and trapped gas, in the deposited material; it is also possible to make alloys that are normally difficult to achieve, for example those involving immiscible components.

D7 does not unambiguously disclose that the powders are not melted during production; the prevention of melting is a measure deliberately chosen by the inventors. In particular, claim 35 only requires that "substantially all" of the reaction products are below the sintering temperature; the significance of the expression "substantially all" is that some reaction products are melted. In addition one of the metals expressly mentioned in D7 is gallium, which has a melting point of 27.76°C; according to D7 gallium can be reduced by means of an alkali earth metal. Should francium, which has a melting point of 27°C, be selected as the alkali earth metal for the reduction, then it would be practically impossible to avoid melting, especially as the reaction is highly exothermic.

Further, it is not clearly stated in D7 that each of the particles comprise the metallic alloy, so they could merely comprise the elemental components, rather than the alloy, as is required by claim 1.

Reasons for the Decision

1. The appeal is admissible.

Primary Request

2. Claim 1 has been amended to require that each particle comprises the metallic alloy material. The basis for this amendment can be found in paragraph [0025] of the published application, and hence the requirements of Article 123(2) EPC are met.

3. Novelty (Article 54 EPC)

3.1 The Examining Division considered that the claimed method lacked novelty in light of D3, D4, D5 or D6.

3.2 JP-A-01 294810 (D3)

D3 concerns the production of magnetic metal powder for magnetic recording. The starting material in D3 is iron oxide containing the metals nickel, cobalt, zinc or manganese; there is therefore no mixture of non-metallic precursor compounds. The claimed subject-matter is thus novel over D3.

3.3 JP-A-11 291087 (D4)

D4 relates to the manufacture of a tin-bismuth alloy powder. According to the method of D4, a tin salt and a bismuth salt are mixed together with an alkaline earth oxide, carbonate or hydroxide. The mixture is reduced in an atmosphere containing hydrogen at a temperature of 400°C to 800°C, after which the alkaline earth salt is removed from the mixture.

It is inevitable that in the process of D4 the metallic alloy powder undergoes melting, because the temperature

at which reduction is carried out is significantly higher than the melting point of both tin (232°C) and bismuth (271°C). The purpose of adding the alkaline earth salt, which has poor wettability with the molten metal, is allow formation of particles when the melt solidifies.

The subject-matter of claim 1 is thus novel with respect to D4.

3.4 US 2002/073804 (D5)

D5 relates to a method for recycling tungsten which is contaminated by thorium oxide. The starting material for the process of D5 is a powder mixture of tungsten and thorium oxide, which undergoes an oxidation step to convert the tungsten to tungsten oxide. The oxide mixture is then reduced in hydrogen to yield tungsten powder having a controlled content of thorium oxide (paragraph [0025]). A mixture of an oxide powder with an elemental metal powder does not fall within the term "alloy powder" in the sense of the application, and in addition, only one metallic constituent is disclosed, whereas claim 1 requires a plurality. Therefore the feature of claim 1, that a mixture of non-metallic precursor compounds is chemically reduced to produce a metal alloy powder is not disclosed in D5.

3.5 GB-A-883429 (D6)

According to the method of D6, a mixture of reducible compounds of copper, silver and tungsten and/or molybdenum is reduced to form alloy powders. Reduction is carried out at 800°C to 1100°C (see D6, claim 7) and

it seems that some fusion of the particles takes place, as the powders are pulverised after reduction (page 3, lines 23 to 25). The powders are then sintered into compacts, so there is no disclosure of them being applied to a substrate. The claimed method is thus novel over D6.

4. Inventive Step (Article 56 EPC)

4.1 Document D7 describes the making of metals by vapour-phase reduction process, and is cited in the description of the present application as a preferred method of making the metal alloy powder. It therefore presents an appropriate starting point for the assessment of inventive step.

4.2 According to D7, non-metallic precursor compounds in the form of gaseous metal halides are reduced to metallic form by a liquid alkali metal or alkaline earth metal. In the particular example of D7, titanium tetrachloride vapour is contacted with molten sodium to form reaction products of titanium metal and sodium chloride. D7 also teaches that it is possible to make alloys of a predetermined composition by providing mixed metal halides in the required molecular ratio at the beginning of the process (see column 3, lines 32 to 34, and column 7, lines 47 to 51). It is an important feature of D7 that the temperature of the reactants is maintained at a temperature lower than the sintering temperature of the alloy to be produced, and thus below the melting point of the material being produced (see the abstract, column 2, lines 43 to 47, column 4, lines 6 to 17).

4.3 The vapour-phase reduction process of D7 is mentioned in the present application as being one of the preferred techniques for achieving the chemical reduction required by the claimed invention; an outline of the technique is given in paragraph [0022] of the application, which refers the reader to D7 as providing a more detailed description. It is therefore intended that the method of D7 will result in a metallic alloy powder whose particles comprise alloy material without the melting of either the non-metallic precursor or the resultant alloy powder.

According to the detailed description of the process of D7 (column 4, lines 6 to 16), titanium tetrachloride vapour is mixed with liquid sodium. The temperature is controlled by the quantity of flowing sodium so that it is kept below the sintering temperature; the temperature of the sodium away from the immediate reaction zone is maintained in the range 200 to 400°C, which acts to quench the particles leaving the reaction zone. Any melting that may occur initially, as a result of the exothermic reaction between titanium tetrachloride and sodium, falls within the meaning given in the application for the expression "without melting" (see the present application, column 6, lines 49 to 52), which is said to include very brief melting in the order of 10 seconds or less. The expression "substantially without melting", as used in claim 35, is seen as reflecting such brief melting.

The Appellant argues that D7 discloses an embodiment for which melting during production of the powder would be inevitable. The example quoted is that of gallium, which has a melting point of 27.9°C. Should gallium

tetrachloride (melting point 201°C) be reacted with francium (melting point 27°C) then melting would be inevitable. Gallium is one element in a list of 15 mentioned in column 1, lines 19 to 20 of D7, and francium, although an alkali metal, is the least stable of all naturally occurring elements, having a half-life of 22 minutes. This example seems to be a fairly obscure example and contrary to the tenor of D5, namely that the temperature should remain below the sintering temperature, and hence the melting point, of the alloy to be produced.

As set out above, it is clear from D7 that all attempts should be made during production of the alloy powder to maintain the temperature below the melting point. The skilled person would immediately recognise that the example cited by the Appellant is not realistic and is contrary to the overall teaching of D7.

4.4 The Appellant submits that it is not apparent from D7 that the powder particles comprise the alloy. Although D7 teaches that alloys can be produced, it does not explicitly state that each of the resulting powder particles is itself an alloy. However, the present application itself puts forward the technique of D7 as a preferred means of producing the powder of claim 1. Following the instructions given in the application (paragraph [0022]), the skilled person would expect to achieve the alloy powder of the invention without the need to take any further essential measures.

4.5 In light of the above reasoning, the method of claim 1 differs from that of D7 only in that the powders are applied to the surface of a substrate.

4.6 D7 itself indicates that the powders are suitable for powder metallurgical applications (column 1, lines 46 to 48). Although it is not expressly mentioned in D7 that the powders are applied to a substrate, such a use of powders is common-place, eg as braze material or for providing a coating, to the extent that, given no evidence that the powders of D7 would *prima facie* not be suitable, it is an obvious application.

4.7 Consequently, the method of claim 1 of the primary request lacks an inventive step.

Auxiliary Request

5. Claim 1 of the auxiliary request has the additional feature that the oxygen content of the metallic alloy powder is controlled by not carrying the chemical reduction process to completion in a fused salt solid phase reduction process or by adding oxygen to react with the metallic alloy material in a rapid plasma quench reduction process.

5.1 The basis for this amendment is to be found at column 6, lines 50 to 55 and in paragraph [0023] respectively; the requirements of Article 123(2) EPC are thus met.

5.2 The invention is now directed to the control of the oxygen content.

Claim 1 of the auxiliary request before the Examining Division contained the feature that oxygen content is controlled by not carrying out the reduction to completion. The Examining Division was of the view that

this feature was also disclosed in D5, and hence the claimed process also lacked novelty; however, for the reasons given above, the Board considers the claim to be novel with respect to D5.

The present claim 1 of the auxiliary request is specifically directed to the control of oxygen content in the processes of fused salt solid phase reduction and rapid plasma quench reduction. These topics have neither been searched nor subjected to substantive examination by the department of first instance. The case must therefore be remitted to the Examining Division for further prosecution.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the Examining Division for further examination on the basis of the auxiliary request filed during the oral proceedings.

The Registrar: The Chairman:

A. Counillon U. Krause