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**Datasheet for the decision  
of 19 November 2018**

**Case Number:** T 1047/16 - 3.3.05

**Application Number:** 10773822.1

**Publication Number:** 2493813

**IPC:** C01B35/04

**Language of the proceedings:** EN

**Title of invention:**

METHODS OF MAKING TITANIUM DIBORIDE POWDERS

**Applicant:**

Alcoa USA Corp.

**Headword:**

Titanium diboride powders/ALCOA

**Relevant legal provisions:**

EPC Art. 83, 84, 111(1), 123(2)

**Keyword:**

Amendments - allowable (yes)

Claims - clarity (yes)

Sufficiency of disclosure - (yes)

Remittal to the department of first instance - (yes)

**Decisions cited:**

**Catchword:**



**Beschwerdekammern**  
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Case Number: T 1047/16 - 3.3.05

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.05**  
**of 19 November 2018**

**Appellant:** Alcoa USA Corp.  
(Applicant) 201 Isabella Street  
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**Representative:** Trinks, Ole  
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**Decision under appeal:** **Decision of the Examining Division of the  
European Patent Office posted on 3 December 2015  
refusing European patent application No.  
10773822.1 pursuant to Article 97(2) EPC.**

**Composition of the Board:**

**Chairman** G. Glod  
**Members:** A. Haderlein  
S. Fernández de Córdoba

## Summary of Facts and Submissions

- I. The applicant (appellant) lodged an appeal against the decision of the examining division refusing European patent application No. 10 773 822. The application concerns methods for making titanium diboride powders.
- II. The examining division held *inter alia* that the main request and auxiliary requests 1 to 3 underlying the impugned decision complied neither with Article 83 EPC nor with the clarity requirement of Article 84 EPC.
- III. The reasons given in the decision under appeal, where relevant to the present decision, may be summarised as follows:

*Main request and auxiliary requests 1 and 2 underlying the impugned decision*

- Claim 1 did not contain any indications of what the target size might be and how the amount of sulfur should vary to attain the desired effect;
- the expression "a small amount of sulfur corresponds to a finer average particle size" rendered the claim unclear;
- the feature "an amount of sulfur" encompassed any quantity of sulfur and it was highly implausible that a reaction among the components of the precursors would afford titanium diboride with any amount of sulfur;
- the feature "reacting the precursor mixture in a reactor" was unclear because it lacked any indication on the type of process which could afford the desired product;
- the expression "process variables" in claim 1 did not contain any indication of how they should be selected

and what their effect on the final particle size might be;

- the skilled person wishing to reproduce the process of claim 1 to obtain titanium diboride particles having a desired particle size would be subject to an undue burden of experimentation in order to find the values of the amount of sulfur and the other processing variables that would afford the desired product.

*Auxiliary request 3 underlying the impugned decision*

- It was not clear whether the weight percentage of sulfur referred to the carbon source, as in example 3, or to the entire reaction mixture; if the weight percentage referred to the reaction mixture, example 4 in table 3 could be considered to fall within the claimed range for the amount of sulfur; the 2 wt% of example 4 in table 3 was based on the amount of carbon and corresponded to 0.56 wt% based on the entire precursor mixture; this example however did not afford the target average particle size of not greater than 7 microns, but it was actually 9.56 microns;

- the requirement of sufficiency was not met because claim 1 was limited to a target average particle size of not greater than 7 microns and to an amount of sulfur not exceeding 1 wt%, but in table 3 of the application at an amount of 1 wt% of sulfur, a D50 value of 7.99 microns was obtained.

IV. With its grounds of appeal, the appellant filed a main and seven auxiliary requests.

V. In a communication under Article 15(1) RPBA, the board informed the appellant that none of the then pending requests seemed to be allowable. In particular, it raised an objection under Article 84 EPC with respect

to the expression "average particle size".

VI. At the oral proceedings, the appellant filed a new main request and withdrew all pending requests.

VII. Claim 1 of the main request reads as follows:

*"1. A method of producing a titanium diboride product having a target average D50 particle size, comprising the following steps:*

- (a) selecting the target average D50 particle size of the titanium diboride product to be produced;*
  - (b) providing a precursor mixture comprising a titanium source, a boron source, a carbon source and an amount of sulfur, wherein the amount of sulfur is selected on the selected target average D50 particle size;*
  - (c) reacting the precursor mixture in a reactor;*
  - (d) deagglomerating the actual titanium diboride product to remove a plurality of agglomerations in the titanium diboride product,*
- wherein reacting the precursor mixture further comprises selecting at least one processing variable from a group, comprising the soak time, the reaction temperature and the inert gas flow rate through the reactor, and selecting a condition of the at least one processing variable, such that the actual titanium diboride product having an actual average D50 particle size is produced, wherein the soak time refers to the amount of time that the precursors in the precursor mixture are held at a specific temperature or within a temperature range and interact;*
- wherein, due to the amount of sulfur, the actual average D50 particle size corresponds to the target average D50 particle size, wherein the amount of sulfur is not greater than 1.0 wt.% of the weight of the carbon source in the precursor mixture and the actual*

*average titanium diboride D50 particle size is not greater than 7 microns;  
wherein reacting the precursor mixture includes carbothermically reacting the precursor mixture;  
wherein the precursor mixture further comprises iron oxide as a catalyst;  
wherein increasing the amount of sulfur in the precursor mixture leads to an increased average D50 particle size of the titanium diboride product actually obtained;  
wherein increasing the soak time, increasing the reaction temperature or lowering the inert gas flow leads to an increased average D50 particle size of the titanium diboride product actually obtained;  
wherein SEM analysis is used to confirm deagglomeration along with laser diffraction particle size analysis."*

Claims 2 to 4 relate to particular embodiments of the method according to claim 1.

VIII. The appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the claims of the sole request filed during oral proceedings before the Board of appeal.

### **Reasons for the Decision**

1. Main request - amendments

1.1 Claim 1 is based on claim 1 as originally filed and includes additional features. Below, the additional features are listed with their respective basis in brackets:

- average particle size being the D50 particle size

(examples, figures, in particular paragraphs [0073], [0074], [0111], Figures 13 and 14);

- selection of the amount of sulfur; average particle size corresponding to target average particle size; precursor mixture; reacting in a reactor (paragraph [0015]);
- deagglomerating (paragraph [0013]);
- selecting at least one variable out of the soak time, the reaction temperature and the inert gas flow rate through the reactor (paragraphs [0004], [0015], [0021], [0022]; for the definition of "soak time" see paragraph [0038], last six lines);
- amount of sulfur not greater than 1.0 wt.% of the weight of carbon source and D50 not greater than 7 microns (paragraph [0006], claim 2, examples, in particular paragraph [0103]);
- carbothermically reacting (paragraphs [0037] and [0082] et seq.);
- iron oxide as a catalyst (paragraph [0048], penultimate line, paragraphs [0088], [0094], [0103] and [0124])
- dependencies of the particle size actually obtained on the process variables (in particular paragraph [0057]).

1.2 As to the features in the last two lines of claim 1, the board observes that these are disclosed in paragraph [0108] in the context of a specific example and also in combination with "confirming... the presence of fractured particles" and with a milling step, these features being absent from claim 1.

For the skilled person reading the application as originally filed it is however clear that SEM analysis and laser diffraction are the methods that are applied in the examples and can be applied in general in the



methods disclosed in the application (see in particular paragraph [0094], cf. also paragraphs [0026] and [0027]). Moreover, it is also clear to the skilled person that by the laser diffraction method the particle size distribution and thus the D50 value can be determined independently of whether the particles are "fractured" or not. Also, the aforementioned analysis methods do not depend on how the deagglomeration step is carried out, i.e. using milling or other means of deagglomerating (cf. paragraph [0051], last four lines). These are methods that the skilled person knows to be applicable in general to particles having a particle size as claimed. The feature "confirming the presence of fractured particles" and the milling step disclosed in paragraph [0108] are therefore not inextricably linked with the features in the last two lines of present claim 1. By omitting these features from claim 1 its subject-matter does not extend beyond the content of the application as originally filed.

1.3 Dependent claims 2 to 4 are based on the following passages of the application as filed:

- claim 2: paragraph [0046], claims 14 and 15;
- 1.4 - claim 3: paragraph [0051];
- claim 4: paragraphs [0004], last two lines, [0016], claim 18.

1.5 The amendments thus comply with the requirements of Article 123(2) EPC.

2. Main request - clarity of the claims
  - 2.1 The claims also comply with the clarity requirement of Article 84 EPC.
  - 2.2 The objections raised by the examining division in the impugned decision with respect to clarity (see III above) have been overcome by the amendments carried out. In particular, it is now clear that the wt.% of the sulfur is based on the weight of the carbon source. It is also clear how the particle size depends on the process variables and which reaction is employed in the claimed method.
  - 2.3 Also, the expression "average particle size" is now clearly defined so as to refer to the D50 value (cf. V above). The last two lines of claim 1 now clearly specify that the average D50 particle size is measured by laser diffraction particle size analysis.
3. Main request - sufficiency of disclosure
  - 3.1 The application contains sufficient information to obtain titanium diboride particles having a D50 of not greater than 7 microns by using sulfur amounts of not greater than 1 wt.% per amount of carbon (see in particular example 3, Table 3). The tests, where a D50 value exceeding 7 microns at sulfur concentrations of not greater than 1 wt.% is obtained, relate to the titanium product obtained before deagglomeration (Table 3, column "As-reacted PSD"; Fig. 12, y-axis labelled "As Reacted Particle Size"), contrary to claim 1, which requires deagglomerating in step (d). Likewise, sample 2 in Table 2 relating to graphite having a sulfur content of as low as 0.008% (see Table 1) leads to a higher D50 value than samples 1

and 3 having sulfur contents of as high as 1.2 or 1.3 % (see Table 1), but sample 2 relates to a state before deagglomeration (see paragraph [0095]).

3.2 There are also no longer serious doubts substantiated by verifiable facts with respect to the sufficiency of disclosure (see the Case Law of the Boards of Appeal of the EPO, 8th edition 2016, II.C.6.1.4, second paragraph).

3.3 Thus, the requirement of sufficiency of disclosure set forth in Article 83 EPC is met.

4. Remittal

The decision under appeal deals only with the provisions of Articles 123(2), 84 and 83 EPC and in particular does not deal with the requirements of Articles 54 and 56 EPC. The board thus exercises its discretion (Article 111(1) EPC) and remits the case to the department of first instance 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 department of first instance for further prosecution.

The Registrar:

The Chairman:



C. Vodz

G. Glod

Decision electronically authenticated