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D E C I S I O N
of 13 July 1999

Case Number: T 0921/95 - 3.2.2

Application Number: 90307683.4

Publication Number: 0408383

IPC: C22B 34/12

Language of the proceedings: EN

Title of invention:
Highly purified titanium material, method for preparation of it and sputtering target using it

Applicant:
Kabushiki Kaisha Toshiba

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-

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Case Number: T 0921/95 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 13 July 1999

Appellant: Kabushiki Kaisha Toshiba
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 13 June 1995 refusing European patent application No. 90 307 683.4 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: W. D. Weiß
Members: R. Ries
J. C. M. De Preter

Summary of Facts and Submissions

I. The appellant (applicant) lodged an appeal against the decision of the Examining Division dated 13 June 1995 to refuse the grant of a patent under application No. 90 307 683.4.

The Examining Division had held that the application did not meet the requirements of Article 54 EPC (lack of novelty) having regard to document

D1: EP-A-0 248 338.

II. In accordance with Article 115 EPC, a third party presented observations concerning the patentability of claims 1 to 4, 11, 13 to 17, and 18 which were challenged with respect to documents

D2: Toshiba Review No. 161, Autumn 1987, pages 38 to 41 and

D3: R. E. Smallman, Modern Phys. Metallurgy, Butterworths, 1962, pages 78 and 79.

III. In a communication dated 26 February 1999 the Board considered document D2 as being the closest prior art. It was pointed out that the thermal conductivity of the newly developed high purity titanium TSB 4N-Ti which is not disclosed in D2 could be calculated by the Wiedemann-Franz law given in

D4: Bird, Stewart, Lightfoot, "Transport Phenomena", J. Wiley, 1960, page 262.

IV. In the appellant's view submitted with the Statement of Grounds of 13 October 1995 and with its letter of 11 June 1999, the impugned decision is essentially based on the erroneous assumption that the materials disclosed in document D1 would exhibit the claimed reduction of area and thermal conductivity because of the low impurity levels of the Ti material. The appellant points to the fact, that the high purity Ti material disclosed in D1 - as well as in D2 - is prepared by a method different to the method used in the present application which would necessarily result in products different from those defined in claim 1.

In support of its argumentation, the appellant refers to results of comparative tests showing that - for essentially the same impurity level of a high purity Ti material - a value of > 16 W/K m for the thermal conductivity is not automatically achieved. Consequently, the impurity levels of oxygen, Fe, Cr, Na, K etc per se are not the only feature to determine the level of thermal conductivity. A thermal conductivity of > 16 W/K m is, however, successfully obtained if the sputtering target Ti material is prepared by the method claimed in the application using a sieving step and/or an acid treatment step of the crude Ti particles before electron beam melting.

The appellant further refers to document

D5: "Handbook of Materials and Processes for Electronics", edited by C. A. Harper, McGraw-Hill, 1970, pp. 9-62 and 9-68

showing a decrease of thermal conductivity with increasing purity of titanium, thus demonstrating that the Wiedemann-Franz-Lorenz law is not applicable in the

present case. Moreover, the theoretical values of the thermal conductivity calculated by the Wiedemann-Franz-Lorenz law given in D3 and D4 deviate significantly from data experimentally determined by the appellant. Therefore, the thermal conductivity of at least 16 W/K m or more is not implicitly disclosed by the alloy composition TSB 4N Ti given in document D2 or by the composition given in document D1.

V. At the oral proceedings held on 13 July 1999 the appellant requested that the decision under appeal be set aside and that the patent be granted on the basis of claims 1 to 17 submitted at the oral proceedings on 13 July 1999, Figures 1 to 3 as originally filed and a description to be adapted.

Independent claims 1, 6, 12 and 17 read as follows:

"1. Highly purified Ti material having an oxygen content of not more than 350 ppm, Fe, Ni and Cr contents of not more than 15 ppm each, and Na and K contents of not more than 0.5 ppm each, a reduction area as a material characteristic of not less than 70%, and a thermal conductivity of not less than 16 W/m K, obtainable by a method (1) consisting of:

a step of sieving crude Ti particles prepared by molten salt electrolysis, Kroll method or Hunter method coarsely prepared to classify them depending on each particle diameter according to contents of impurities, a step of selecting one type or a mixture of at least two types of the crude Ti particles having a desired particle diameter from the above classified crude Ti particles and a step of melting the selected crude Ti particles by an electron beam,

or a method (2) consisting of:

a step of acid treating the crude Ti particles prepared by molten salt electrolysis, Kroll method or Hunter method or Jodide method to remove a contaminated layer existing on the surface of the crude Ti particles, and a step of melting the acid-treated crude Ti particles by an electron beam.

6. A method for preparation of highly purified Ti material according to any of claims 1 to 5, the method consisting of:

a step of sieving crude Ti particles prepared by molten salt electrolysis, Kroll method or Hunter method and coarsely prepared to classify them depending on each particle diameter according to contents of impurities,

a step of selecting one type or a mixture of at least two types of the crude Ti particles having a desired particle diameter from the above classified crude Ti particles, and

a step of melting the crude Ti particles by an electron beam.

12. An integrated circuit comprising a Ti wiring network on a semiconductor device surface, wherein said wiring network comprises highly purified Ti material according to any of claims 1 to 5.

17. A wiring network consisting of the highly purified Ti material according to any of claims 1 to 5."

Dependent claims 2 to 5, 7 to 11 and 13 to 16 concern particular embodiments of the Ti material according to claim 1 or the method for producing such a material according to claim 6 or the integrated circuit according to claim 12, respectively.

Reasons for the Decision

1. Amendments

Claim 1 finds support in originally filed claims 1, 5, 8 and 10 in combination with the description as filed (see EP-A-0 408 383, page 2, lines 32 to 35, page 3, lines 34 to 39, line 50). Claim 2 is based on originally filed claims 5 and 9. Claims 3 to 5, 7 and 8 correspond to originally filed claims 2 to 4, 6 and 8, respectively. Claim 6 is supported by originally filed claim 5 in combination with page 4, lines 11 to 13.

Claim 9 results from a combination of former claims 5 and 8, and claims 10 and 11 correspond to claims 10 and 11 as filed. Claims 12 and 17 are based on the description as filed, page 2, lines 1 to 3, page 5, lines 18 to 21 and claims 13 to 16 find support on page 2, lines 39 to 41 and page 10 lines 4 to 6, respectively.

Hence there are no formal objections under Article 123(2) EPC to claims 1 to 17.

2. *Novelty*

Document D2 which represents the closest prior art discloses in paragraph 2 a Newly Developed High Purity Ti Target Material called TSB 4N-Ti which exhibits a chemical composition of: 80 ppm oxygen, <5 ppm Fe, <5 ppm Ni, <5 ppm Cr, <0.1 ppm Na, <0.1 ppm K, <1 ppb U, <1 ppb Th, and a reduction of area of 82.6% (cf. page 39, paragraph 2.2, Tables 1 and 2).

In comparison with claim 1, document D2, however, fails to mention explicitly the thermal conductivity k of TSB 4N-Ti which is claimed to be not less than 16 $W \cdot K^{-1} \cdot m^{-1}$ according to the present application.

A similar high purity Ti material is disclosed in document D1, comprising 100-150 ppm O, 0.4-0.8 ppm Fe, 0.3-0.5 ppm Cr, 0.1-0.3 ppm Ni, 0.05 ppm Na, 0.05 ppm K, < 0.001 ppm U, <0.001 ppm Th without giving any data for the reduction of area and the thermal conductivity. The remaining documents are more remote.

It may be argued that, by applying the Wiedemann-Franz-Lorenz relationship described in D3 and D4, material TSB 4N-Ti of D2 can be expected to exhibit a thermal conductivity in the range of 17 to 22, i.e. meeting the proviso of $\geq$ 16 $W \cdot K^{-1} \cdot m^{-1}$ required by claim 1.

In its letter dated 11 June 1999, however, the appellant submitted test results for material TSB 4N-Ti disclosed in D2. In particular, the appellant has experimentally determined the thermal conductivity of TSB 4N-Ti to be 15.3 $W \cdot K^{-1} \cdot m^{-1}$ showing that the theoretical relationship underlying the calculation according to the Wiedemann-Franz-Lorenz law is actually not applicable to the high purity Ti material claimed in the present application. This estimation is supported by document D5, Table 34 on page 9-65 which discloses a thermal conductivity for 99.5% Ti of 9 Btu/(hr)(ft)(°F) corresponding to 15.6 $W \cdot K^{-1} \cdot m^{-1}$ and further shows an increase of thermal conductivity with increasing impurity levels rather than the contrary expected by theoretical considerations. In any case, the measured value of 15.3 $W \cdot K^{-1} \cdot m^{-1}$ of material TSB 4N-

Ti, which on the basis of the technical information available to the Board is not subject to any doubt, significantly deviates from the theoretical value of 17 to 21 and is below the lower limit of ≥16 $W \cdot K^{-1} \cdot m^{-1}$ claimed in claim 1 of the application.

Moreover, claim 1 is drafted as a "product-by-process" claim which implies that the claimed material must exhibit the properties and structure of the material brought about by the process. It also implies a lower limit as well as a certain range (in ppm or ppb) for the various impurities and trace elements typically associated with the specific process (Hunter process, salt electrolysis, jodide process). In consequence of these considerations, the scope of claim 1 does not encompass elemental titanium which is (theoretically) free from any impurity and which, according to the general knowledge of a skilled person, indispensably adopts the values for the reduction of area and the thermal conductivity required by claim 1.

In this context, the applicant has submitted further experimental test results for various properties of high purity Ti materials having essentially the same composition but which, however, were produced either using the claimed process or, alternatively, using the process according to the prior art D1. It is clearly apparent from Table 4 that production of the alloys by the process in prior art document D1 does not include a sieving and/or acid treatment step before electron beam melting, and thermal conductivity of ≥16 $W \cdot K^{-1} \cdot m^{-1}$ is not obtained. In consequence thereof, the properties of the sputtered film are not satisfactory. This all goes to show that for the high purity Ti material claimed in the present application the lower limit of ≥16 $W \cdot K^{-1} \cdot m^{-1}$

actually represents a technically relevant property which is not inherent to the composition and which is not automatically brought about by the processes of the prior art D1 or D2. Consequently, the subject matter of claim 1 is novel.

3. *Inventive step*

According to document D2, the impurity levels of oxygen, U, Th, Na, K, Fe, Ni, Cr have a negative impact on various mechanical and electrical properties of Ti sputtered films utilized in semiconductor devices. Hence, the impurity level of the Ti material should be as low as possible. However, document D2 is silent about the thermal conductivity and the surface and inside conditions of the sputtering target material which - according to the application - have to be sufficiently high to uniformly discharge the thermal energy applied to the target during sputtering and which are responsible for changes in the electric resistance and other properties of the sputtered film, (cf. page 2, lines 24 to 30, lines 45 to 49 of the published application).

Starting from document D2 as the closest prior art, the problem underlying the present application, therefore, is seen in providing a sputtering target material which shows an excellent match in thermal conductivity and the surface and inside conditions, sufficient to meet the above mentioned requirements.

The solution to this problem consists in the high purity Ti material which is produced by the process given in claim 1 of the present application. More specifically, the desired thermal conductivity and

surface and inside conditions of the target material are brought about by sieving the crude Ti particles, selecting a particular particle size and/or acid treating the crude Ti particles before electron beam melting.

None of the documents D1 or D2 or any other document discloses the above mentioned steps or gives any inducement to do so to afford a person skilled in the art a better perspective as to the nature of the high purity Ti sputtering target material, as does the present application. Consequently, the subject matter of claim 1 involves an inventive step.

The material according to claim 1 being novel and inventive, the same statement is true for the method of producing this material according to claim 6 and the products produced from this material defined in claims 12 and 17, respectively.

The claimed subject matter, therefore, meets the requirements of the EPC.

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 with the order to grant the patent on the basis of claims 1 to 17 submitted at the oral proceedings, Figures 1 to 3 as originally filed and a description to be adapted.

The Registrar: The Chairman:

S. Fabiani W. D. Weiß

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