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Datasheet for the decision of 23 May 2019

Case Number: T 1501/16 - 3.3.05

Application Number: 07842416.5

Publication Number: 2059946

IPC: C30B23/00, C30B29/36, H01L21/20, H01L21/02

Language of the proceedings: EN

Title of invention:
MICROPIPE-FREE SILICON CARBIDE AND RELATED METHOD OF MANUFACTURE

Applicant:
Cree, Inc.

Headword:
Micropipe-free SiC single crystal/CREE

Relevant legal provisions:
EPC Art. 54(1), 54(2), 56, 84, 123(2)

Keyword:
Amendments - allowable (yes)
Claims - clarity (yes)
Novelty - (yes)
Inventive step - (yes)

Decisions cited:

Catchword:

Beschwerdekammern
Boards of Appeal
Chambres de recours

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Case Number: T 1501/16 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 23 May 2019

Appellant:
(Applicant)
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Representative:
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted on 12 February 2016 refusing European patent application No. 07842416.5 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman	A. Haderlein
Members:	T. Burkhardt
	R. Winkelhofer

Summary of Facts and Submissions

I. The appeal lies from the decision of the examining division to refuse European patent application EP 07 842 416.

II. The following documents were considered during the examination proceedings:

D1 EP 1 659 198 A1
D2 US 2006/0075958 A1

III. According to the decision under appeal, the subject-matter of claim 11 of the then main request was obvious within the meaning of Article 56 EPC in view of document D2.

It was held that the subject-matter of claim 11 differed from D2 in that the silicon carbide wafer had a micropipe density of zero (item II.2.2). It would have been evident for the skilled person to seek a silicon carbide single crystal having a defect density as low as possible in order to assure the functioning of semiconductor devices.

Claim 10 of the then first auxiliary request was identical to claim 11 of the then main request, and therefore its subject-matter was also not inventive within the meaning of Article 56 EPC.

For several additional reasons, it was concluded that the requirements of Article 84 EPC were not met:
- the requirement in claim 11 of the then main request and in claim 10 of the then first auxiliary request

that the wafer had no micropipe defects was a "result to be achieved";
- since the "SiC sublimating flux" in claim 1 of the then main request would primarily depend on the temperature of the "source material", it was unclear how the sublimating flux could be "controlled" by means of a "ramped increase in the growth temperature",
- it was unclear how the "growth nucleation temperature" could cause the "closing-off of micropipes" (via a modification of the Si/C ratio) as stipulated in claim 1 of the then main request. According to the description, the closing-off would rather be caused by a low start temperature. Hence, the low start temperature was an essential feature missing in claim 1 of the main request.

IV. With its grounds of appeal, the applicant (appellant) filed a main (sole) request.

V. After the issuance of a communication under Article 15(1) RPBA and two telephone conversations, the appellant filed a new main request with submission of 15 May 2019 and submitted replacement pages of the description on 15 and 20 May 2019.

In the communication under Article 15(1) RPBA, the following document had been introduced:

D3 Nakamura *et al.*, "Ultrahigh quality silicon carbide single crystals", Nature, 430, 2004, 1009-1012.

VI. The independent claims of the main (sole) request read:

"1. A method of growing a single-crystal (26) of silicon carbide, SiC, in the nominal c-axis growth

direction using a physical vapor transport, PVT, process in a sublimation system (12), wherein the crystal is completely free of micropipe defects, the method comprising:

attaching a seed material (22) to a seed holder (24) and forming a uniform thermal contact between the seed material and seed holder, wherein the seed material comprises a SiC seed wafer having a micropipe density of less than 2 cm^{-2}*;*

placing a source material (20) and the seed material attached to the seed holder in a reaction crucible (14), wherein constituent components of the sublimation system including at least the source material, the seed holder, and the reaction crucible are substantially free from metallic impurities, wherein the source material has a total concentration of less than 5 parts per million by weight of metallic impurities; and

controlling growth temperature, growth pressure, SiC sublimation flux and composition, and a temperature gradient between the source material and the seed material or the SiC crystal growing on the seed material during the PVT process,

comprising controlling the growth pressure to be in a range of from 40,000 Pa to 13 Pa (300 to 0.1 torr);

controlling the growth temperature to have a start temperature in a growth nucleation temperature range of 2000° to 2200° C during an initial growth nucleation phase of bulk growth such that a modified Si/C ratio is induced which promotes the closing-off of micropipes that may exist in the seed material during the initial stages of bulk growth;

controlling the temperature gradient to be in a range of from 50 to 150 deg C/cm; and *controlling the growth temperature to have a ramped increase in the range of 0.3 to 10 deg C/hr and to be in the range of 2000° to 2500° C;*

thereby eliminating micropipe-inducing process instabilities and growing the micropipe free SiC crystal on the seed material."

"8. A semiconductor wafer comprising:

a silicon carbide, SiC, substrate sliced from a crystal having a minimum diameter of 100 mm, the SiC substrate having opposing first and second surfaces: and

an epitaxial layer on at least the first surface of the SiC substrate and having a concentration of n-type or p-type dopant atoms defining a conductivity type for the epitaxial layer;

characterized in that the SiC substrate is a micropipe-free substrate having a micropipe density of zero."

VII. The appellant's arguments, as far as relevant for the present decision, are summarised as follows:

The claims of the main request and the adapted description fulfil the requirements of the EPC.

VIII. The appellant requests that the decision be set aside and that a patent be granted on the basis of:
- claims 1-12 of the main request as filed with submission of 15 May 2019;
- description pages 1, 13 and 16 as submitted on 15 May 2019, pages 3-5, 11 and 14 as submitted on 20 May 2019, and pages 2, 7-10, 12 and 15 as originally filed, page 6 being deleted; and
- drawing sheets 1/5-5/5 as originally filed.

Reasons for the Decision

1. Article 123(2) EPC

Independent claim 1 is based on claim 1 as originally filed (reference is made to the A-publication). The properties of the starting materials are disclosed in paragraphs [46, 53] as originally filed, and the parameters of the crystal growth process are disclosed in paragraphs [53, 57, 59]. The replacement of the "unintentional impurities" by "metallic impurities" is supported by paragraphs [46, 47] as originally filed.

Independent claim 8 is based on claim 40 as originally filed. The nature of the dopant atoms is based on paragraph [36] as originally filed.

The dependent method claims 2-7 are based on original claims 2, 10-12, 20, 22 and 23 and on original paragraph [58].

The dependent device claims 9-12 are based on original claims 32-34, 41 and 43.

Hence, the requirements of Article 123(2) EPC are fulfilled.

2. Clarity

For the following reasons, the present main request fulfills the requirements of Article 84 EPC:

2.1 In the decision under appeal, it was held that the mere definition of the semiconductor wafer in claim 1 of the then main request amounted to defining the invention by a "result to be achieved".

In contrast, the claims of the present main request do not merely define the underlying problem. Indeed, as will be explained below (see especially point 3.1), method claim 1 comprises a specific feature combination that is sufficient to yield silicon carbide single crystals with a micropipe density of zero, namely a relatively defect-free and pure seed material and a precise set of operating parameters - in particular numerical ranges for the growth pressure, the growth temperature, the thermal gradient between the source material and the seed material, and the ramped increase of the growth temperature.

2.2 The examining division had also held that it was unclear how the "SiC sublimating flux" could be "controlled" by means of a "ramped increase in the growth temperature". The flux would rather depend on the temperature of the "source material".

Since claim 1 now makes clear that the SiC sublimation flux is controlled not only by the ramped increase of the growth temperature but also by further parameters, such as the growth temperature and the thermal gradient between the source material and the seed material, this objection is now moot.

2.3 Likewise, since in claim 1 the "start temperature" is now specified to be in the range of 2 000°C to 2 200°C, the objection in the contested decision that this was a missing essential feature is also moot.

3. Novelty

Novelty was already acknowledged in the decision under appeal, since D2 does not disclose SiC single crystals with a micropipe density of zero. For the following reasons, the board shares the view that the requirements of novelty are met:

3.1 D1 discloses a method of growing a silicon carbide single crystal without defects, such as micropipes, and the possible use of this crystal in a semiconductor wafer (see paragraphs [4, 16, 136] and claim 1).

The method of D1 also involves the sublimation of a seed material and its recrystallisation around a seed material (paragraphs [94, 95]), the use of a high purity raw material (paragraph [61]), specific temperature and pressure ranges for the sublimation-recrystallisation (paragraphs [110, 144], Table 1) and the possible use of dopants (paragraph [62]).

Moreover, the absence of micropipes in the silicon carbide single crystal is mentioned *in principle* in D1: For example, in paragraph [136] it is stated that "[t]he silicon carbide single crystal of the present invention contains no crystal defects such as ... micropipes". Likewise, it is indicated in paragraph [163] that "[a]ccording to the present invention, a high quality silicon carbide single crystal ... showing no defects such as ... micropipes ... can be provided."

However, the board notes firstly that D1 is silent on various numerical values for parameters in method claim 1, such as the start temperature, the temperature gradient between the seed material and the source material, and the ramped increase of the growth temperature, let alone on a ramped increase with a specific slope.

More importantly, in D1 the *actually* observed pipe defect density in the silicon carbide single crystals is different from zero: it is indicated in the experiments that "[w]hen the resulted silicon carbide single crystal 60 was evaluated, ... crystal defect of micropipes was as scarce as 4 cm^{-2}" (paragraph [148]). In other words, the disclosure of micropipe-free crystals in D1 is not enabling the obtention of single crystals with a micropipe density of zero.

Yet, it is established case law that subject-matter described in a document can only be regarded as having been made available to the public if the information given therein is sufficient to enable the skilled person, at the relevant date, to practise the technical teaching, taking also into account the general knowledge at that time (Case Law of the Boards of Appeal, 8th edition, I.C.4.11, first paragraph).

The specific feature combination of claim 1 involves a relatively defect-free and pure seed material and a precise set of operating parameters - namely ranges for the growth pressure, the growth temperature, the thermal gradient between the source material and the seed material, and the ramped increase of the growth temperature. As explained in paragraphs [45, 48] of the application, these parameters allow the different sources for micropipe generation to be eliminated.

This specific feature combination yields silicon carbide single crystals that are "*completely* free of micropipe defects" (claim 1) and wafers with a micropipe-free substrate having a micropipe density of zero (claim 8), as proven in the photographs of Figures 4A-C and 5 (see also paragraphs [51, 52]). In contrast, a method of growing where "differences in growth rate" exist promotes the formation of polytype inclusions, which in turn result in micropipes as shown in Figure 3 (see also paragraphs [42, 43]).

Hence, the present application discloses an enabling way to produce SiC single crystals that are completely free of micropipes or that have a micropipe density of zero.

Consequently, the subject-matter of claims 1 and 8 is novel over D1 (Article 54(1) and (2) EPC).

3.2 The disclosure of D2 is similar to that of D1, except that D2 does not mention entirely micropipe-free crystals and that the information concerning how the growth process is carried out is less detailed:

According to D2, "producing larger high quality bulk single crystals of silicon carbide with low basal plane defect levels in crystals formed in the seeded sublimation system remains a constant technical commercial goal" (paragraph [14]). According to paragraph [11], micropipes are a special form of dislocations.

According to the method as claimed in D2, the density of residual dislocations is up to 50 cm^{-2} in the best (lowest) case (claim 8). In the example, a defect density of 44 cm^{-2} is consistently obtained (paragraph [33]).

While D2 mentions a growth temperature in the claimed range (paragraph [50]), the features ramped temperature increase with a specific slope and specific temperature gradient are not disclosed. It is merely stated that "the temperature gradients and other conditions (pressure, carrier gases, etc.) are properly maintained" (paragraph [51])

Contrarily, as explained above for D1, the present application discloses an enabling way to produce micropipe-free silicon carbide single crystals.

Consequently, the subject-matter of claims 1 and 8 is novel also vis-à-vis D2 (Article 54(1) and (2) EPC).

3.3 For the sake of completeness, the fact that residual micropipes remain when the growth methods of the prior art are used is also confirmed by the serial publication D3, which is referred to in both the present application (paragraph [8]) and in D2 (paragraph [7]).

In the abstract of D3, it is stated that "some macroscopic defects (about 1-10 cm^{-2}) ... remain within the crystal" when silicon carbide single crystals are grown.

The board notes that even with the alternative growth method suggested in D3, i.e. not a growth along the c-axis as in claim 1 of the present application but along the a-axis ("repeated a-face growth process" in Figure 1 of D3), it is merely "considered" that the "[perfect elimination of] dislocations" and the possibility "to enlarge the diameter to several inches" would be possible "in the near future" (page 1011, column 2, last paragraph).

4. Inventive step

4.1 The invention

The application relates to a method of growing micropipe-free single crystals of silicon carbide and to a semiconductor wafer comprising a micropipe-free silicon carbide substrate with a micropipe density of zero.

4.2 Closest prior art

Since D1 also relates to a method of growing a silicon carbide single crystal without defects, such as micropipes, and to the use of this crystal in a semiconductor wafer (see paragraphs [4, 16, 136] or claim 1), D1 is considered as the closest prior art of the subject-matter of claims 1 and 8.

4.3 Problem to be solved

The problem to be solved by the present application is to improve the fabricability, operability and reliability of semiconductor devices which contain SiC substrates (see paragraphs [7, 163]).

4.4 Proposed solution

The solution proposed is a SiC single crystal growth method involving the specific combination of a relatively pure raw material and operating conditions during the crystal growth process stipulated in claim 1, and resulting in SiC single crystals that are completely micropipe-free.

In claim 8, the integration of such a crystal into a semiconductor wafer is proposed.

4.5 Success of the solution

The results of Figures 4A-C and 5 and paragraphs [51, 52] show that the obtained SiC crystals are micropipe-free and that the posed problem is successfully solved.

4.6 Obviousness

In the absence of an *enabling* teaching in the prior art (see point 3. above), it was not obvious at the filing date to arrive at a method of growing a SiC single crystal that is completely free of micropipe defects or at a semiconductor wafer with a micropipe-free SiC substrate.

In the decision under appeal, it was stated that it was evident for the skilled person to seek a wafer with as few defects as possible, and that the mere "definition" of a micropipe-free wafer could not confer an inventive step. However, the board does not share this view, since the present application does not only define such a wafer but also discloses an enabling way to achieve it.

Consequently, an inventive step within the meaning of Article 56 EPC is acknowledged.

5. For the same reasons, the subject-matter of the dependent claims is also novel and inventive.

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 with the order to grant a patent on the basis of:
   - claims 1-12 of the main request as filed with submission of 15 May 2019;
   - description pages 1, 13 and 16 as submitted on 15 May 2019, pages 3-5, 11 and 14 as submitted on 20 May 2019, and pages 2, 7-10, 12 and 15 as originally filed, page 6 being deleted; and
   - drawing sheets 1/5-5/5 as originally filed.

The Registrar: The Chairman:

K. Götz-Wein A. Haderlein

Decision electronically authenticated