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Datasheet for the decision of 10 May 2016

Case Number: T 1213/12 - 3.4.03

Application Number: 02021450.8

Publication Number: 1298709

IPC: H01L21/20, C30B29/38

Language of the proceedings: EN

Title of invention:
III-nitride epitaxial substrate, epitaxial substrate for III-nitride element and III-nitride element

Applicant:
NGK Insulators, Ltd.

Headword:

Relevant legal provisions:
EPC Art. 52(1), 123(2)
EPC 1973 Art. 56

Keyword:
Inventive step - (yes)

Decisions cited:

This datasheet is not part of the Decision.
It can be changed at any time and without notice.

Catchword:

This datasheet is not part of the Decision.
It can be changed at any time and without notice.

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Case Number: T 1213/12 - 3.4.03

D E C I S I O N
of Technical Board of Appeal 3.4.03
of 10 May 2016

Appellant: (Applicant)
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted on 29 December 2011 refusing European patent application No. 02021450.8 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman G. Eliasson
Members: S. Ward
T. Bokor

Summary of Facts and Submissions

I. The appeal is against the decision of the Examining Division refusing European patent application No. 02 021 450 on the ground that the claimed subject-matter did not involve an inventive step within the meaning of Articles 52(1) and 56 EPC.

II. At the end of the oral proceedings held before the Board the appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the following documents:

Description: pages 1, 2, 2a, 3-12 as filed during the oral proceedings before the Board, originally filed page 12a being deleted;

Claims 1-11 of the main request as filed during the oral proceedings before the Board at 10.50 hours;

Drawings: Figure 1 as originally filed.

III. The following documents are referred to:

D1: US 6 252 255 B1;

D2: EP 1 065 705 A2;

D9: Structural defects and their relationship to nucleation of GaN thin films; Weida Qian et al; Materials Research Society Symposium Proceedings; Volume 423, Pages 475-486, 1996; XP009189415.

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

"A method for producing a III nitride element comprising a III nitride epitaxial substrate, said method comprising the steps of:
employing a c-faced sapphire single crystal base material of which a crystal orientation of a main surface is inclined from the <0001> orientation, that is, the c-axis orientation by a range within 0.02-0.3 degrees;
nitriding the main surface of the base material to form a surface nitride layer thereon;
forming an III nitride underfilm including at least Al element directly on said main surface of said sapphire single crystal base material via said surface nitride layer by means of a MOCVD method in a state where the base material is heated at a temperature in the range of 1100°C or over and 1250°C or below; and
forming a III nitride film on the nitride underfilm by a MOCVD method,
wherein a full width at half maximum of the underfilm at (10-12) reflection in X-ray rocking curve is 2000 seconds or below and of which a surface roughness Ra within 5 µm area is 3,5 Å or less."

V. The findings of the Examining Division, insofar as they are relevant to the present decision, may be summarised as follows:

Starting from document D2, the subject matter of claim 1 differed only in that the crystal orientation of a main surface was inclined from the <0001> orientation by a range within 0.02 - 0.3 degrees, and that the rocking curve measurement was performed at the (10-12) reflection.

The objective technical problem was the improvement of the quality of epitaxial III-nitride layers on sapphire.

The minimization of FWHM of the rocking curve at the (10-12) orientation lacked an inventive step since it represented a generally desirable property for a semiconductor device.

D1 taught that using a sapphire substrate with a miscut in the range of 0.05°to 0.2° from the <0001> orientation led to a very small roughness (less than 1 nm) and a low dislocation density (< 3 x 10^{10} cm2). Since the skilled person would know that dislocation density and roughness were key quality metrics for epitaxial III-nitride layers, he would employ a sapphire substrate with a miscut in the range of 0.05° to 0.2°from the <0001> orientation in a process according to D2.

While all of the embodiments of D2 referred to a-plane sapphire, paragraph [0025] taught that the use of the a-plane orientation, while preferred, is only optional. Stated differently, D2 contained no teaching against the use of the c-plane orientation. Thus the skilled person would not hesitate to apply the teachings of D1 (regarding the use of miscut c-plane oriented sapphire) to D2, especially because in the field of epitaxy, the use of a crystal miscut was a routine measure to improve epitaxial quality, and c-plane was a very well-known alternative from a limited list of possible sapphire orientations (r-plane, a-plane and c-plane).

The main teaching of Dl was clearly the use of a miscut of 0.05° to 0.2° from <0001 >, rather than the use of a low temperature buffer layer.

VI. The appellant's arguments, insofar as they are relevant to the present decision, may be summarised as follows:

Document D1 (using miscut c-plane orientated sapphire) was the closest prior art.

It was very unlikely that one skilled in the art would ignore the critical difference in terms of crystal structure, i.e. c-plane vs. a-plane, between Dl and D2. D2 was strongly based on an application to the a-plane of sapphire single crystal. The Examples of D2 unanimously used the a-plane of sapphire single crystal (paragraphs [0071], [0074], [0086] and [0091]) with no mention of a c-plane alternative.

The objective problem was to provide a method for producing an improved epitaxial substrate with a high crystallinity nitride film, particularly an Al-including nitride film, including fewer dislocations.

Documents D2 and Dl were irrelevant to each other because they simply represented different method concepts for producing a III nitride epitaxial substrate. While a skilled person might be motivated to switch from the method of D1 to that disclosed in document D2 (including the use of the a-plane), he would not incorporate isolated features from D2 into D1.

Furthermore, it should be noted that the method of Dl formed the buffer layer (underfilm) at relatively low temperature around 600°C just like the prior art mentioned in paragraph [0032] of the EP specification. In contrast, D2 formed the corresponding buffer layer at a temperature "in the range from 1000 to 1200°C". D2

and Dl strongly contradicted each other in this regard, rendering an unconsidered combination of features belonging to different method concepts even more unlikely.

Further, document Dl was completely silent about the claimed feature relating to the FWHM of the underfilm at the (10-12) reflection.

Reasons for the Decision

1. The appeal is admissible.

2. *Main Request: Article 123(2) EPC*

2.1 Claim 1 is directed to a method of producing a III nitride element comprising a III nitride epitaxial substrate, and can be seen as being based chiefly on features from claim 1 as originally filed adapted to correspond to method steps (the original claims did not comprise any method claim).

2.2 Further aspects of claim 1 are disclosed in the description as originally filed as follows: formation of a "nitride element" - paragraph [0001]; nitriding the main surface - paragraphs [0018],[0028]; depositing the underfilm by MOCVD where the base material is heated to between 1100°C and 1250°C - paragraph [0030]; "directly" - examples 1-5; forming a III nitride film on the underfilm by MOCVD - paragraphs [0036]- [0038]; 3.5 Å "or less" - paragraph [0019].

2.3 The dependent claims 2-11 are method claim adaptations of the originally filed dependent claims 2-11.

2.4 The Board is therefore satisfied that the requirements of Article 123(2) EPC are met.

3. *Closest prior art*

3.1 The invention concerns a method of producing a III nitride element comprising a III nitride epitaxial substrate, and involves *inter alia* forming layers on a main surface of a sapphire substrate slightly inclined to the c-plane (within a range of 0.02-0.3 degrees from the c-axis orientation).

3.2 In the prior art, various crystal planes of sapphire, including the c-plane and the a-plane, have been used for the deposition of III-nitride films.

3.3 Document D1 discloses a method of producing a III nitride element on a main surface of a sapphire substrate which is slightly inclined (0.05-0.2 degrees) to the c-plane (see e.g. example 1, claim 1), essentially in accordance with the teaching of present claim 1.

3.4 In document D2, which was taken as the closest prior art in the contested decision, possible substrates including sapphire are listed in paragraphs [0024] and [0058], and the a-plane of a sapphire substrate is said to be preferred (paragraphs [0025],[0060], claim 9).

Despite these general statements, it appears from paragraph [0018] that the inventors' practical investigations were confined solely to sapphire, and in all of the examples, the a-plane of a sapphire

substrate is used exclusively (paragraphs [0039], [0040],[0046],[0056],[0071],[0074],[0086] and [0091]). In short, there is no disclosure in document D2 of deposition on a sapphire c-plane, or indeed on any sapphire face other than the a-plane.

Since the concrete technical teaching to be derived from document D2 concerns a method of producing a III nitride element on the a-plane of a sapphire substrate, document D1 would appear to be *prima facie* the more suitable choice of closest prior art.

3.5 It could perhaps be argued that document D2 might still be a candidate for the closest prior art if it turned out that deposition on the a-plane involved basically the same technical considerations as deposition on the c-plane, the choice of crystal plane being essentially arbitrary. However, this is not the case.

3.6 It is well-known (see e.g. Table I of document D9) that the lattice parameter in the c-plane of sapphire (12.991 Å) is significantly larger than that in the a-plane (4.758 Å), and that the lattice mismatch between III nitride compounds (e.g. GaN, AlN) and sapphire is much greater for deposition on the sapphire c-plane than on the a-plane. The successful deposition of high quality III nitride films on the c-plane would therefore involve different considerations and require different measures compared with deposition on the a-plane.

For the above reasons, the Board considers that document D1 is the suitable choice for the closest prior art.

4. *Difference*

4.1 Document D1 discloses the following features of present claim 1: a method for producing a III nitride element comprising a III nitride epitaxial substrate, said method comprising the steps of:
- employing a c-faced sapphire single crystal base material of which a crystal orientation of a main surface is inclined from the <0001> orientation, that is, the c-axis orientation by a range within 0.02-0.3 degrees (see column 2, lines 44-51; example 1 etc.);
- nitriding the main surface of the base material to form a surface nitride layer thereon (see column 4, lines 22-30);
- forming an III nitride underfilm including at least Al element directly on said main surface of said sapphire single crystal base material via said surface nitride layer by means of a MOCVD method in a state where the base material is heated (see column 4, lines 5-7 and 16-19: the "buffer layer" may be of AlN); and
- forming a III nitride film on the nitride underfilm by a MOCVD method (see column 4, lines 7-9 and 19-22).

4.2 Claim 1 differs in that in forming the III nitride underfilm including Al by means of MOCVD:

"the base material is heated at a temperature in the range of 1100°C or over and 1250°C or below";

Furthermore, document D1 does not disclose the following claimed performance parameters:

"a full width at half maximum of the underfilm at (10-12) reflection in X-ray rocking curve is 2000 seconds or below and of which a surface roughness Ra within 5 µm area is 3,5 Å or less."

5. *Problem and Solution*

5.1 As explained in paragraph [0012] of the description:

"*Since the epitaxial substrate of the present invention can exhibit good crystal quality of low dislocation density, the dislocation density of a III nitride film particularly including Al element to be formed on the epitaxial substrate can be reduced, and thus, the crystal quality can be enhanced.*"

5.2 The problem to be solved is therefore to produce a III nitride device on a c-faced sapphire base material with improved crystal quality and reduced dislocation density.

The solution according to the present invention (paragraphs [0030]-[0032]) resides in the higher temperature ("1100°C or over and 1250°C or below") of the MOCVD process in which the III nitride underfilm is formed (in combination with the other claimed features known from document D1). The remaining distinguishing features (relating to the X-ray rocking curve and surface roughness) appear to define the degree to which the problem is solved.

5.3 For the following reasons, the Board can accept that this represents a plausible solution to the above problem.

5.4 In document D1, the corresponding layer (buffer layer) is formed at "about 600°C" (example 1) or "from about 500°C to about 650°C" (example 2). The present invention starts from the premise that a higher temperature MOCVD step can produce a higher quality crystal underfilm (paragraph [0030]), and that a higher

quality underfilm could, in principle, result in a higher crystallinity III nitride device layer (paragraph [0012]).

Nevertheless, as explained in the present application (paragraph [0031]), the reason that low-crystallinity buffer layers, grown at low temperatures, are preferred in the conventional art is that highly crystalline layers would not be suitable as a buffer between c-plane sapphire and III nitrides (e.g. GaN) due to the large mismatch in lattice constant.

5.5 The claimed features of the epitaxial substrate (an angle of 0.05° to 0.2° between the main surface and the c-plane, nitriding the main surface and providing an underfilm including Al) are also disclosed in document D1, where they are said to provide various advantages, such as reducing dislocations (column 3, lines 53-56).

The insight behind the present invention is that these features also provide an additional advantage, namely that they allow a highly crystalline layer to be used, formed by MOCVD at a higher temperature, without misfit dislocations being created due to the difference in lattice constants (paragraphs [0031],[0032]). The resulting improvements are illustrated in the examples and comparative examples.

6. *Assessment of inventive step*

6.1 It is therefore to be determined whether, starting from document D1, the claimed solution would be obvious to the skilled person on the basis of the prior art.

6.2 The claimed invention would not be obvious from document D1 alone; there is no suggestion in this

document to deviate from the conventional low crystalline (low temperature) buffer layer.

6.3 Document D2 confirms (paragraphs [0003]-[0014]) much of the background to the present invention, in particular that it is conventional to grow a buffer layer at a low temperature of about 400°C to improve the crystallinity of the device layer (paragraph [0005]), and that such a layer is "amorphous or polycrystalline" (paragraphs [0010],[0011]).

Moreover, document D2 (paragraph [0012]) notes a suggestion in the prior art that a higher quality crystal underfilm could be produced by employing a higher temperature MOCVD step, and that this should result in a higher crystallinity III nitride device layer (paragraph [0013]).

6.4 However, document D2 goes on to state that according to an examination carried out by the inventors, using a higher temperature MOCVD step in the manner set out in the cited prior art has been found not to produce sufficient crystallinity in the buffer layer to form a device layer with good crystallinity (paragraph [0014]).

6.5 An object of the invention of document D2 is therefore to find "a preferred condition for formation of a group III nitride compound semiconductor layer on a high temperature buffer layer" (paragraph [0017]). By achieving this object, not only may large process temperature changes be avoided (paragraphs [0007], [0020]), but also "the crystallinity of the device function layer becomes equal to or higher than the crystallinity of a device function layer formed on a

low-temperature growth buffer layer used generally" (paragraph [0023]).

The invention of document D2 therefore represents a solution to the objective problem mentioned above, but one in which - for all embodiments disclosed in a manner which would enable the skilled person to carry them out - deposition takes place on the a-plane of a sapphire substrate (see point 3.4, above).

6.6 Starting from document D1, and attempting to improve crystal quality, if the skilled person decided to persist with growing the device on the c-plane (as disclosed in document D1), the teachings of document D2 about deposition on the a-plane would be seen as irrelevant, given the very different lattice mismatches referred to above (see point 3.6), and the skilled person would not combine documents D1 and D2.

Alternatively, it could be argued that the skilled person might be persuaded by document D2 to abandon the c-plane entirely and switch to deposition on the a-plane, which would lead to subject-matter different from that claimed in the present application.

6.7 What is not plausible to the Board, however, is that the skilled person would persist with deposition on the c-plane (as in document D1), but nevertheless import particular features disclosed only in relation to deposition on the a-plane (as in document D2).

To put it another way, document D2 teaches that depositing the buffer layer at high temperatures can lead to a poor result, and then discloses conditions that, for deposition on a sapphire a-plane, lead to a more successful outcome. Document D2 contains no

information on how this might be achieved when deposition takes place on the c-plane.

6.8 The Board therefore judges that the subject-matter of claim 1 of the main request involves an inventive step within the meaning of Article 52(1) EPC and Article 56 EPC 1973.

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 with the following documents:

   Description, pages 1, 2, 2a, 3-12 as filed during the oral proceedings before the Board;

   Claims 1-11 of the Main Request as filed during the oral proceedings before the Board at 10.50 hours;

   Drawings: Figure 1 as originally filed.

The Registrar: The Chairman:

S. Sánchez Chiquero G. Eliasson

Decision electronically authenticated