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(54) **METHOD AND APPARATUS FOR SINGLE CRYSTAL GALLIUM NITRIDE (GAN) BULK SYNTHESIS**

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Related U.S. Application Data

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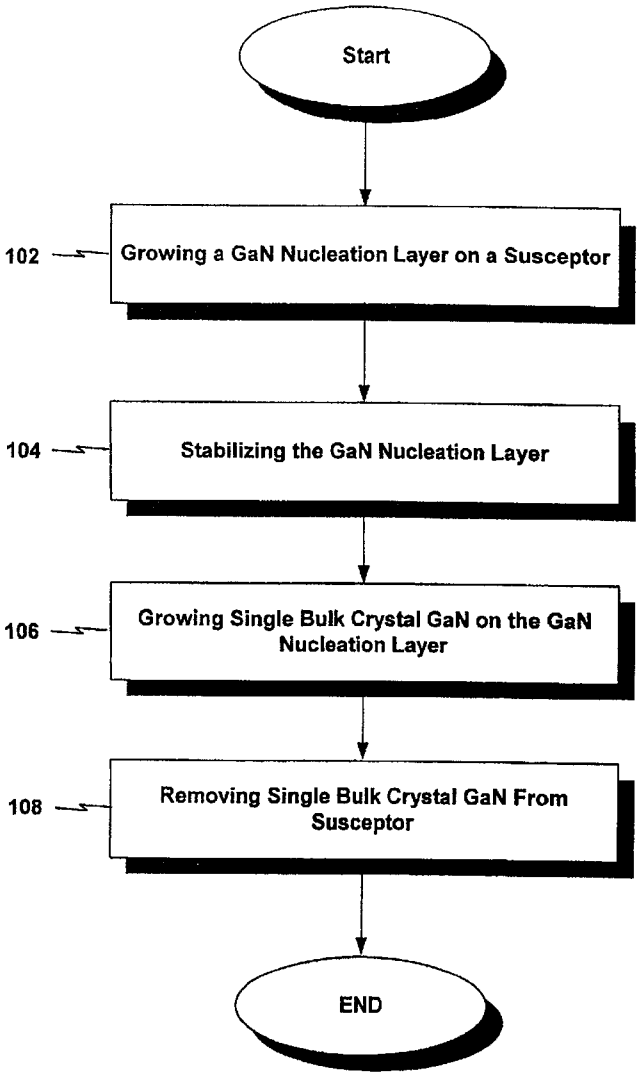
(52) **U.S. Cl.** **117/2**

(57) **ABSTRACT**

A method and apparatus for homoepitaxial growth of free-standing, single bulk crystal Gallium Nitride (GaN) are provided, wherein a step of nucleating GaN in a reactor results in a GaN nucleation layer having a thickness of a few monolayers. The nucleation layer is stabilized, and a single bulk crystal GaN is grown from gas phase reactants on the GaN nucleation layer. The reactor is formed from ultra low oxygen stainless steel



Group 2

CRYSTALLINE GALLIUM NITRIDE AND METHOD FOR FORMING CRYSTALLINE GALLIUM NITRIDE

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Applicant(s):: GEN ELECTRIC (US)

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Equivalents:

Abstract

A gallium nitride growth process forms crystalline gallium nitride. The process comprises the steps of providing a source gallium nitride (15); providing mineralizer (17); providing solvent (17); providing a capsule (10); disposing the source gallium nitride, mineralizer and solvent in the capsule; sealing the capsule; disposing the capsule in a pressure cell (1); and subjecting the pressure cell to high pressure and high tempeature (HPHT) conditions for a length of time sufficient to dissolve the source gallium nitride and re-precipitate the source gallium nitride into at least one gallium nitride crystal. The invention also provides for gallium nitride crystals formed by the processes of the invention.

(12) МЕЖДУНАРОДНАЯ ЗАЯВКА, ОПУБЛИКОВАННАЯ В СООТВЕТСТВИИ С ДОГОВОРОМ О ПАТЕНТНОЙ КООПЕРАЦИИ (PCT)

(19) ВСЕМИРНАЯ ОРГАНИЗАЦИЯ ИНТЕЛЛЕКТУАЛЬНОЙ СОБСТВЕННОСТИ
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(84) **Указанные государства** *(регионально):* ARIPO патент (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), евразийский патент (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), европейский патент (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), патент OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

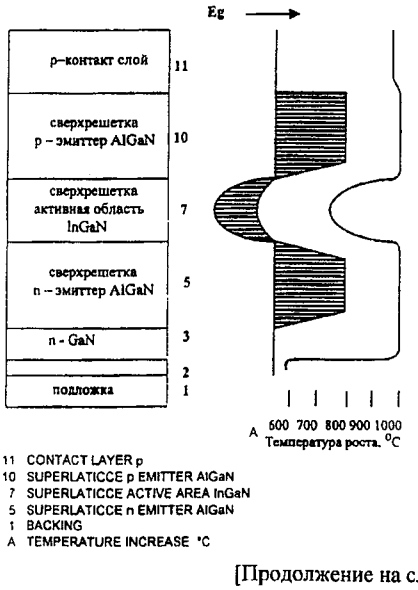
Опубликована
С отчётом о международном поиске.

В отношении двухбуквенных кодов, кодов языков и других сокращений см. «Пояснения к кодам и сокращениям», публикуемые в начале каждого очередного выпуска Бюллетеня PCT.

(54) Title: METHOD FOR CRYSTALLINE GROWTH IN EPITAXIAL HETEROSTRUCTURES BASED ON GALLIUM NITRIDE

(54) Название изобретения: МЕТОД КРИСТАЛЛИЧЕСКОГО РОСТА ЭПИТАКСИАЛЬНЫХ ГЕТЕРОСТРУКТУР НА ОСНОВЕ НИТРИДА ГАЛЛИЯ

(57) Abstract: The inventive method is a method for an epitaxial gas-phase chemical deposition of a compound solid-state composition in a wurtzite structure based on gallium nitride expressed by general formula $B_xAl_yIn_zGa_{1-x-y-z}$ (wherein, $0 \le x \le 0,2$, $0 \le y \le 1-x-z$, $0 \le 1-x-y$) on a backing, made for instance of sapphire, silicium carbide, gallium or aluminium nitrides in which at least one parameter (x,y, or z) is changed in respect to a layer thickness in accordance with a selected law (linear, parabolic, stages etc) in order to reduce a discrepancy of grid parameters and mechanical stress between both the backing and a heterostructure and between layers or functional parts of a light-emitting heterostructure. . Fig.3 11 Contact layer 10 Superlaticce p Emitter AlGaN 7 Superlaticce Active area 5 Superlaticce n Emitter AlGaN 3 n-GaN Backing



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Group 6

(12) **United States Patent**
Shibata et al.

(10) **Patent No.: US 6,270,569 B1**
(45) **Date of Patent: Aug. 7, 2001**

(54) **METHOD OF FABRICATING NITRIDE CRYSTAL, MIXTURE, LIQUID PHASE GROWTH METHOD, NITRIDE CRYSTAL, NITRIDE CRYSTAL POWDERS, AND VAPOR PHASE GROWTH METHOD**

(75) Inventors: **Masatomo Shibata**; **Takashi Furuya**, both of Tsuchiura (JP)

(73) Assignee: **Hitachi Cable Ltd.**, Tokyo (JP)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U S.C. 154(b) by 0 days.

(21) Appl. No : **09/096,458**

(22) Filed: **Jun. 11, 1998**

Related U.S. Application Data

(63) Continuation-in-part of application No 09/092,020, filed on Jun. 5, 1998.

(30) **Foreign Application Priority Data**

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Aug 18, 1997 (JP) 9-221628
Oct 24, 1997 (JP) 9-292561

(51) **Int. Cl.**[7] **C30B 11/04**; C30B 11/06

(52) **U.S. Cl.** **117/68**; 117/73; 117/74; 117/75; 117/77; 117/78; 117/952

(58) **Field of Search** 117/952, 67, 78, 117/73, 74, 75, 77

(56) **References Cited**

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(List continued on next page.)

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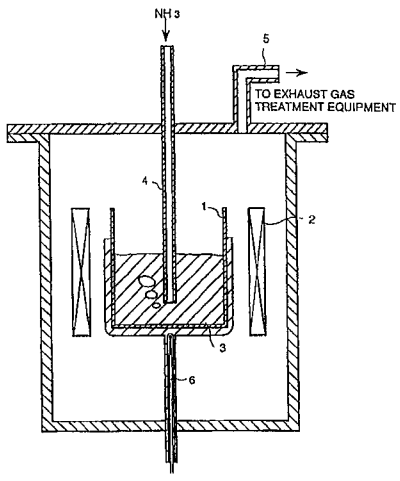
(74) *Attorney, Agent, or Firm*—Antonelli, Terry, Stout & Kraus, LLP

(57) **ABSTRACT**

A Group III metal element is heated so as to melt, a gas NH_3 containing nitrogen atoms is injected into a melt 3 of the Group III metal element at a temperature lower than the melting point of a nitride to be obtained, thereby producing a nitride microcrystal of the Group III element having high wettability with the melt 3 in the melt 3 of the Group III metal element A mixture of the Group III nitride microcrystal obtained as mentioned above and the Group III metal element solution is used as a starting material of a liquid phase growth or Group III nitride powders obtained by removing the Group III metal material from the mixture are used as a starting material of a vapor phase growth. Further, a seed crystal or a substrate crystal is immersed in a melt of a Group III element such as gallium, bubbles of a gas containing nitrogen such as ammonia are intermittently come into contact with the surface of the crystal, and the Group III element and the gas containing nitrogen are allowed to react with each other on the surface of the seed crystal or the substrate crystal, thereby allowing the nitride crystal of the Group III element to be grown on the surface of the seed crystal or substrate crystal.

36 Claims, 12 Drawing Sheets



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(19) **United States**

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TISCHLER et al. (43) **Pub. Date: Jul. 19, 2001**

(54) **BULK SINGLE CRYSTAL GALLIUM NITRIDE AND METHOD OF MAKING SAME**

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(*) Notice: This is a publication of a continued prosecution application (CPA) filed under 37 CFR 1.53(d).

(21) Appl. No.: **08/955,168**

(22) Filed: **Oct. 21, 1997**

Related U.S. Application Data

(63) Continuation-in-part of application No. 08/188,469, filed on Jan. 27, 1994, now Pat No. 5,679,152

Publication Classification

(51) **Int. Cl.**[7] **B32B 1/08**

(52) **U.S. Cl.** **428/34.1**; 428/64.1

(57) **ABSTRACT**

A single crystal M*N article, which may be made by a process including the steps of: providing a substrate of material having a crystalline surface which is epitaxially compatible with M*N; depositing a layer of single crystal M*N over the surface of the substrate; and removing the substrate from the layer of single crystal M*N, e.g., with an etching agent which is applied to the substrate to remove same, to yield the layer of single crystal M*N as said single crystal M*N article. The bulk single crystal M*N article is suitable for use as a substrate for the fabrication of microelectronic structures thereon, to produce microelectronic devices comprising bulk single crystal M*N substrates, or precursor structures thereof.