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(30) Priority 15.07.1981 US 283696

(71) Applicant DRESSER INDUSTRIES,INC.

(72) Inventor Phillips, James Davis

(54) **Silicon carbide furnace**

(57) A furnace for manufacturing silicon carbide comprises a broken ring (8) of raw materials in which is embedded a graphite electrical heating element (12) The ring (8) is free-standing and defines a central working space (32) in which is located a support (36) The furnace is enclosed by an annular wall (44) spaced from the ring (8) to define an annular working space A track (38) is mounted on top of the wall (44) and a rotatable materials handling device (6) is mounted on the central support (36) and the track (38) Materials are discharged from an outlet (48) to form the ring (8) as the device (6) rotates A geodesic dome roof prevents escape of pollutants to atmosphere

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(30) Priority **29.03.1982 US 362701**

(71) Applicant **NORTON COMPANY**

(72) Inventor **Kuriakose, Areekattuthazhayil Kuruvillai**

(54) **Process & furnace for making silicon carbide**

(57) There is provided a method of making a larger percentage of coarsely crystalline silicon carbide in an Acheson furnace comprising packing the silica and coke ingredients forming a reaction mix around a centrally disposed heat source (14), confining the mix between gates (22, 24) which hold the mix in an insulated zone (20) surrounding said source (14), supplying energy to said source (14) for raising the temperature within the mass to at least about 1800 °C for reacting all of the ingredients in the mix between the heat source (14) and the gates (22, 24) to form finely crystalline silicon carbide, and then increasing the crystal size of the silicon carbide in the reacted mass by increasing the temperature of the reacted mass between the heat source (14) and the gates (22, 24) to a temperature between 2000 °C to 2500 °C

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FIRST 83/002 FST83002
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WO 83/02108

(84) Designated Contracting States.
FR

(30) Priority **16.12.1981 US 331331**

(71) Applicant **ATLANTIC RICHFIELD COMPANY**

(72) Inventors
- **BEATTY, Ronald L.**
- **WYMAN, Floyd H.**

(54) **CONTINUOUS SILICON CARBIDE WHISKER PRODUCTION**

(57) Methods and apparatuses for obtaining silicon carbide whiskers on a continuous basis Coked rice hulls or other carbon - and silicon bearing feed materials are continuously fed to a heating zone (18) in an unagitated state in order to promote whisker growth The heating zone is continuously purged with an inert gas (22a), such as nitrogen or argon The inert gas carries away gaseous impurities through vents (25) which are located directly in the heating zone, and prevents oxidation of the whiskers The feed materials are dried in a dehydrating furnace (14) and are fed in a dry state therefrom to the heating zone in order to prevent erosion of the furnace walls

(57) Procédés et dispositifs permettant d'obtenir des whiskers de carbure de silicium en continu Des cosses de riz cokéifiées ou d'autres substances carbonées et des matériaux d'alimentation contenant du silicium sont amenés en continu vers une zone de chauffage (18) dans un état de non-agitation afin de stimuler la croissance des whiskers La zone de chauffage est purgée en continu avec un gaz inerte (22a) , tel que de l'azote ou de l'argon Le gaz inerte éloigne les impuretés gazeuses au travers d'évents (25) disposés directement dans la zone de chauffage, et empêche l'oxydation des whiskers Les matériaux d'alimentation sont séchés dans un four de déshydratation (14) et d'ici sont envoyés dans un état sec vers la zone de chauffage afin d'éviter l'érosion des parois du four

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(74) Representative: White, Martin David et al
MARKS & CLERK 57/60 Lincoln's Inn Fields
London WC2A 3LS (GB)

(54) A heteroepitaxial growth method.

(57) A heteroepitaxial growth method comprising growing a semiconductor single-crystal film (8) on a semiconductor single-crystal substrate (A) with a lattice constant (a1) different from that (b1) of the semiconductor single-crystal film (B) by chemical vapor deposition, the epitaxial orientation of the semiconductor single-crystal film (B) being inclined at a certain angle (1°-30°) with respect to the semiconductor single-crystal substrate (A)

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(19) Europäisches Patentamt
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WO 89/04055

(84) Designated Contracting States
DE FR GB IT NL SE

(30) Priority 26.10.1987 US 113565

(71) Applicant NORTH CAROLINA STATE UNIVERSITY

(72) Inventors
- DAVIS, Robert, F.
- CARTER, Calvin, H. Jr.
- HUNTER, Charles, Eric

(54) **SUBLIMATION GROWTH OF SILICON CARBIDE SINGLE CRYSTALS**

(57) The present invention is a method of forming large device quality single crystals of silicon carbide (33) The sublimation process is enhanced by maintaining a constant polytype composition in the source materials (40), selected size distribution in the source materials (40), by specific preparation of the growth surface of seed crystals (32), and by controlling the thermal gradient between the source materials (40) and the seed crystal (32)

(57) La présente invention se rapporte à un procédé servant à former de grands monocristaux de carbure de silicium (33) de qualité appropriée pour la production de dispositifs électriques Le procédé de sublimation décrit est amélioré grâce au maintien d'une composition polytype constante dans les matériaux sources (40), grâce à une distribution granulométrique sélectionnée dans les matériaux sources (40), grâce à une préparation spécifique de la surface de croissance des germes cristallins (32) et grâce à une régulation du gradient thermique entre les matériaux sources (40) et le germe cristallin (32)

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(19) Europäisches Patentamt
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ESPACE 90/060 ESP90060
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(84) Designated Contracting States
DE FR GB

(30) Priority **20.06.1989 DE 3920134**

(71) Applicant **Siemens Aktiengesellschaft**

(72) Inventor **Stein, Rene, Dr.**

(54) **Process for producing single crystal silicon carbide**

(57) Single crystals of silicon carbide SiC can be produced by sublimation and partial decomposition of crystalline SiC powder as starting material and growth on a nucleus According to the invention, for crystal growth an excess of silicon is established in the SiC powder used as starting material This process gives pure single crystals

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AT BE DE FR GB IT LU NL SE

(30) Priority 11.01.1989 US 8900114

(71) Applicant THE DOW CHEMICAL COMPANY

(72) Inventors
- WEIMER, Alan, W.
- MOORE, William, G.
- RAFANIELLO, William
- ROACH, Raymond P.

(54) **PROCESS FOR PREPARING SILICON CARBIDE**

(57) A process for preparing silicon carbide by carbothermal reduction involves rapidly heating a particulate reactive mixture of a silica source and a carbon source to form a product which shows improved uniformity of crystal size The product of this process can be used to form a densified part The process comprises passing a particulate reactive mixture (24) of a silica source and a carbon source into a reactor having (a) a reactant transport member (6), the reactant transport member having a wall defining a hollow conduit, the wall having a cooling means and being further characterized as having a concentric inner wall defining an inner annular space (20), the inner annular space having an inlet and being open at the bottom such that a gas can be flowed therethrough, (b) a reactor chamber (16), the reactor chamber having a wall (26), defining a reaction zone (28), the chamber being in fluid connection with the reactant transport member, (c) a heating means (40), the heating means being suitable for heating the particulate reactive mixture in the reaction zone, and (d) a cooling chamber (42), the cooling chamber having a wall (48) defining a cooling zone (44), the wall having a cooling means, the cooling chamber being in fluid connection with the reactor chamber, the temperatures of the reactant transport member, reactor chamber, and cooling chamber being independently controllable, such that the particulate reactive mixture can be fed continuously through the reactant transport member into the reactor zone and then into the cooling zone

(57) Un procédé de préparation de carbure de silicium par réduction carbothermique consiste à chauffer rapidement un mélange particulaire réactif d'une source de silice et d'une source de carbone afin de former un produit présentant une uniformité améliorée de la taille des cristaux On peut utiliser le produit de ce procédé pour former une pièce densifiée Ledit procédé consiste à faire passer un mélange particulaire réactif (24) d'une source de silice et d'une source de carbone dans un réacteur comportant (a) un élément (6) de transport de réactif, ledit élément de transport de réactif présentant une paroi définissant un conduit creux, la paroi étant dotée d'un moyen de refroidissement et étant en outre caractérisée en ce qu'elle comporte une paroi intérieure concentrique définissant un espace annulaire intérieur (20), ce dernier comprenant une admission et étant ouvert au niveau de sa partie inférieure de sorte que du gaz peut s'y écouler, (b) une chambre de réacteur (16), présentant une paroi (26) et définissant une zone de réaction (28), ladite chambre étant en liaison fluidique avec ledit élément de transport de réactif, (c) un moyen de chauffage (40) adapté pour chauffer le mélange particulaire réactif dans ladite zone de réaction, et (d) une chambre de refroidissement (42) présentant une paroi (48) définissant une zone de refroidissement (44), ladite paroi étant dotée d'un moyen de refroidissement, et ladite chambre de refroidissement étant en liaison fluidique avec la chambre du réacteur On peut régler indépendamment les températures de l'élément de transport de réactif, de la chambre du réacteur et de la chambre de refroidissement, de sorte que l'on peut acheminer en continu le mélange particulaire réactif par l'intermédiaire dudit élément de transport de réactif jusque dans la zone du réacteur puis dans ladite zone de refroidissement

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(19) Europäisches Patentamt
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(84) Designated Contracting States
BE DE FR GB SE

(30) Priority 24.07.1990 US 566908

(71) Applicant. HERCULES INCORPORATED

(72) Inventors
- Jensen, James Allen
- Rosenthal, Allen Bruce

(54) **Preparation of silicon carbide whiskers**

(57) Silicon carbide single crystals are prepared by (1) reacting silica gel, silicic acid or silicon dioxide with an inorganic base and a multifunctional alcohol or a multifunctional phenol to produce a carbon-containing chemically activated silicon compound, (2) mixing the activated silicon compound with carbon black or graphite and (3) heating the mixture to 1300° to 1700°C under a non-oxidizing atmosphere

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(84) Designated Contracting States
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(30) Priority 13.10.1989 US 421375

(71) Applicant CREE RESEARCH, INC.

(72) Inventors
- PALMOUR, John, W.
- KONG, Hua-Shuang
- EDMOND, John, A.

(54) **METHOD OF PREPARING SILICON CARBIDE SURFACES FOR CRYSTAL GROWTH**

(57) The invention is a method of forming a substantially planar surface on a monocrystalline silicon carbide crystal by exposing the substantially planar surface to an etching plasma until any surface or subsurface damage caused by any mechanical preparation of the surface is substantially removed The etch is limited, however, to a time period less than that over which the plasma etch will develop new defects in the surface or aggravate existing ones, and while using a plasma gas and electrode system that do not themselves aggravate or cause substantial defects in the surface

(57) Procédé de formation d'une surface plane sur un cristal au carbure de silicium monocristallin, par exposition de la surface plane à un plasma de gravure jusqu'à élimination d'éventuelles détériorations de surface ou de sous-surface provoquées par n'importe quelle préparation mécanique Toutefois, la gravure est limitée à une durée inférieure à celle pendant laquelle la gravure au plasma développe de nouveaux défauts dans la surface ou aggrave les défauts existants, et tandis que l'on utilise un système d'électrode et de gaz au plasma n'aggravant ou ne provoquant pas eux-mêmes les défauts de la surface

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DE FR GB

(30) Priority. **28.01.1992 JP 3720892**

(71) Applicant **NISSHIN STEEL CO., LTD.**

(72) Inventors
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- **Taniguchi, Seiichi, c/o Semiconductor Res. Center**
- **Fukuda, Momoya, c/o Semiconductor Res. Center**

(54) **SiC single crystal growth**

(57) A chamber (10) is divided into a reaction zone (20) and a sublimation zone (30) A gaseous mixture (41) is supplied through a conduit (21) into the reaction zone (20) and heated by a heater (27) The components in the gaseous mixture (41) are reacted with each other to synthesize solid-phase SiC (42) The solid-phase SiC (42) is heated and evaporated by a heater (35), and condensed as a single crystal (43) on a seed crystal attached to a mount base (37) The mount base (37) is rotated and lowered in response to the growth of the SiC single crystal (43) by a rotary shaft (38) Since the SiC single crystal (43) grows from SiC synthesized by the vapor-phase reaction, the obtained product is of very high purity without the substantial inclusion of impurities In addition, a single crystal having a large diameter or length can be obtained without restrictions imposed by the use of a crucible

<IMAGE>

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(54) **Procédé de production de trichites ou whiskers fibreux, longs de carbure de silicium.**

(57) La production de trichites ou whiskers de SiC et de mats de ceux-ci sur un substrat par traitement à 1250 - 1500°C, d'un mélange gazeux comprenant de l'hydrogène et des sources d'atomes de Si et C qui sont sous forme d'au moins un composé dépourvu d'oxygène, en présence d'un catalyseur de type métal, par un procédé semi-continu ou périodique, est caractérisée en ce que durant la période de croissance, un catalyseur Al-Fe est introduit dans la phase gazeuse dans la zone de réaction, au moyen d'une reduction par le carbone de céramiques de type aluminosilicate qui comprennent au moins 73 % en poids de Al_2O_3 et de 0,3 à 3,0 % en poids d'oxydes de fer et le substrat est un tissu de carbone à base de fibre de rayonne carbonisée qui a été prétraité, avant la carbonisation, par une solution de borax et une solution de phosphate de diammonium jusqu'à ce que les quantités de bore et de phosphore dans le tissu ne dépassent pas 4 % et 2 % en poids, respectivement.

Application à la fabrication de trichites ou whiskers longs de SiC.

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Process for the production of long fibrous silicon carbide whiskers

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Inventor(s)	GRIBKOV VLADIMIR N (RU), POLAKOV ALEXANDRE (RU), POKROVCKY DANIEL D (RU), SILAEV VLADMIR A (RU); GOLEROV YURII A (RU), LYACOTA PIOTR P (RU)
Applicant(s)	AEROSPATIALE (FR), VIAM ALL (RU)
Requested Patent	US5614162
Application Number.	US19950390505 19950217
Priority Number(s):	FR19940001826 19940217
IPC Classification	C01B31/36
EC Classification:	C04B35/622F, C30B25/00F
Equivalents	CA2142693, DE69508827D, DE69508827T, EP0668376, B1, ES2132548T, FR2716208, JP8091953

Abstract

The production of SiC whiskers and of mats thereof on a substrate by treatment, at 1250 DEG to 1500 DEG C., of a gaseous mixture including hydrogen and sources of Si and C atoms which are in the form of at least one oxygen-free compound, in the presence of a metal type catalyst, by a semi-continuous or periodic process, is characterized in that, during the growth period, an Al-Fe catalyst is introduced into the gas phase in the reaction zone, by means of carbon reduction of aluminosilicate ceramics, which comprise at least 73 weight % of Al_2O_3 and 0.3 to 3 0 weight % of iron oxides and the substrate is a carbonized rayon fiber based carbon fabric which has been pre-treated, prior to carbonization, with a solution of borax and a solution of diammonium phosphate until the quantities of boron and phosphorus in the fabric do not amount to more than 4 and 2 weight %, respectively.

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(22) Date of filing **21.03.1994**

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(30) Priority 01.04.1993 DE 4310744

(71) Applicant **SIEMENS AKTIENGESELLSCHAFT**

(72) Inventors
- **VÖLKL, Johannes**
- **LANIG, Peter**

(54) **DEVICE AND PROCESS FOR PRODUCING SiC SINGLE CRYSTALS**

(57) In a process and a device for producing SiC single crystals (20), a reaction chamber (2), in which there is a seed crystal (21) for the separation of a SiC single crystal (20) from the gas phase, is connected to a storage chamber (4) which is at least partly filled with a supply of SiC (40) by a gas channel (3) with a predetermined cross-section for conveying the SiC in the gas phase The supply of SiC (40) is sublimated in a heating device (6) and a temperature gradient is created in the reaction chamber (2) It is thus possible to produce SiC single crystals of high crystalline quality and single-crystal yield, and having any cross-sectional area because the conveyance rate of the gas molecules can be precisely adjusted

(57) L'invention concerne un procédé et un dispositif permettant de réaliser des monocristaux (20) de carbure de silicium. Une chambre de réaction (2) dans laquelle se trouve un germe cristallin (21) destiné à séparer un monocristal de carbure de silicium (20) de la phase gazeuse, est reliée par l'intermédiaire d'une arrivée de gaz destinée à assurer le transport du SiC dans la phase gazeuse et ayant une section prédéfinie, à un réservoir (4) rempli au moins partiellement d'une réserve de SiC (40) Un dispositif de chauffage (6) permet de sublimer la réserve de SiC (40) et d'instaurer un gradient de température dans la chambre de réaction (2) Ce procédé permet d'obtenir des monocristaux de SiC (20) ayant une section de n'importe quelle superficie, et assure une qualité de cristal élevée et un rendement en monocristaux élevé, du fait que les volumes de molécules gazeuses transportées peuvent être ajustés avec précision

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(30) Priority **26.10.1987 US 113565**

(71) Applicant **NORTH CAROLINA STATE UNIVERSITY**

(72) Inventors
- **Davis, Robert F.**
- **Hunter, Charles Eric**
- **Carter, Calvin H., Jr.**

(54) **Sublimation growth of silicon carbide single crystals**

(57) The present invention relates to a method of forming large device quality single crystals of silicon carbide (33) The sublimation process is enhanced by maintaining a constant polytype composition in the source materials (40), selected size distribution in the source materials (40), by specific preparation of the growth surface of seed crystals (32), and by controlling the thermal gradient between the source materials (40) and the seed crystal (32)

<IMAGE>

EP 0 712 150 A1

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(84) Designated Contracting States
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(30) Priority 02.10.1995 US 538064

(71) Applicant MOTOROLA, INC.

(72) Inventors
- Thero, Christine
- Norton, Patricia A.

(54) **Method of etching silicon carbide**

(57) A mask (12) is applied to a silicon carbide substrate (11) in order to etch the substrate (11) The material used for the mask (12) has a Mohs hardness factor greater than 4 in order to prevent sputtering material from the mask (12) onto the substrate (11) An oxygen and sulfur hexafluoride plasma is utilized to perform the etch **<IMAGE>**

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(30) Priority 30.11.1994 US 346618

(71) Applicant CREE RESEARCH, INC.

(72) Inventors
- DMITRIEV, Vladimir, A.
- RENDAKOVA, Svetlana V.
- IVANTSOV, Vladimir A.
- CARTER, Calvin, H., Jr.

(54) EPITAXIAL GROWTH OF SILICON CARBIDE AND RESULTING SILICON CARBIDE STRUCTURES

(57) A method is disclosed for producing epitaxial layers of silicon carbide that are substantially free of micropipe defects The method comprises growing an epitaxial layer of silicon carbide on a silicon carbide substrate by liquid phase epitaxy from a melt of silicon carbide in silicon and an element that enhances the solubility of silicon carbide in the melt The atomic percentage of that element predominates over the atomic percentage of silicon in the melt Micropipe defects propagated by the substrate into the epitaxial layer are closed by continuing to grow the epitaxial layer under the proper conditions until the epitaxial layer has a thickness at which micropipe defects present in the substrate are substantially no longer reproduced in the epitaxial layer, and the number of micropipe defects in the epitaxial layer is substantially reduced

(57) L'invention présente un procédé visant à produire des couches épitaxiales de carbure de silicium, pratiquement exemptes d'imperfections du type microconduit Ce procédé consiste à faire croître une couche épitaxiale de carbure de silicium sur un substrat de carbure de silicium par épitaxie en phase liquide à partir d'un bain de fusion de carbure de silicium dans du silicium et d'un élément qui augmente la solubilité du carbure de silicium dans le bain de fusion Le pourcentage atomique de cet élément est plus important que celui du silicium dans le bain La poursuite de la croissance de la couche épitaxiale, dans des conditions appropriées, permet d'obturer les microconduits que le substrat y a propagé jusqu'à ce que la couche épitaxiale soit d'une épaisseur telle que les imperfections du type microconduit présentes dans le substrat cessent pratiquement de s'y multiplier et que leur nombre dans cette même couche ait diminué de façon notable

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(19) Europäisches Patentamt
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CH DE FR GB IT LI NL SE

(30) Priority 01.12.1994 DE 4442819

(71) Applicant SIEMENS AKTIENGESELLSCHAFT

(72) Inventors
- STEPHANI, Dietrich
- VÖLKL, Johannes

(54) PROCESS AND DEVICE FOR SUBLIMATION GROWING SILICON CARBIDE MONOCRYSTALS

(57) A reaction chamber (2) is surrounded by a gas-tight wall (20) of which at least the inner side (21) that faces the reaction chamber (2) is made of silicon carbide produced by a CVD process At least part of the silicon carbide that constitutes the wall (20) is sublimated and grown as a silicon carbide monocrystal (4) on a crystal seed (3)

(57) Une chambre de réaction (2) est entourée d'une paroi (20) étanche aux gaz constituée au moins du côté intérieur (21), vis-à-vis de la chambre de réaction (2), de carbure de silicium obtenu par un procédé de dépôt en phase vapeur Au moins une partie du carbure de silicium dont est constituée la paroi (20) est sublimée et forme un monocristal de carbure de silicium (4) sur un germe de cristallisation (3)

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(87) International publication number
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(84) Designated Contracting States
CH DE FR GB LI SE

(30) Priority 27.12.1994 DE 4446866
13.02.1995 DE 19504669

(71) Applicant SIEMENS AKTIENGESELLSCHAFT

(72) Inventors
- STEIN, René
- RUPP, Roland

(54) **METHOD OF PRODUCING BORON-DOPED MONOCRYSTALLINE SILICON CARBIDE**

(57) In a CVD process or a sublimation process an organic boron compound is used for doping a SiC-monocrystal, the molecules of said boron compound comprising at least one boron atom chemically bonded to at least one carbon atom The preferred boron compounds are boron trialkyls

(57) Dans le cadre d'un processus de dépôt chimique en phase vapeur ou d'un processus de sublimation, pour doper un monocristal de SiC, on utilise un composé de bore inorganique, dont les molécules contiennent au moins un atome de bore lié chimiquement à au moins un atome de carbone Les composés de bore préférés sont les trialkyles de bore

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(19) Europäisches Patentamt
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(84) Designated Contracting States
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(30) Priority 10.04.1996 FR 9604450

(71) Applicant COMMISSARIAT A L'ENERGIE ATOMIQUE

(72) Inventors
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- Madard, Roland
- Anikin, Mikhail
- Garcon, Isabelle

(54) Process and apparatus for forming single crystal silicon carbide (SiC) on a seed

(57) Single crystal sublimation growth of silicon carbide An apparatus for SiC growth on a seed includes a crucible chamber (100) delimited by one or more walls (102, 110, 112) and accommodating a SiC seed (122), a SiC powder source (118) and a heating system (120) for producing a thermal gradient between the source and the seed The wall (102, 110, 112) is covered with a SiC layer (116), preferably a powder layer covering the cylindrical side wall (102) of the chamber (100) Also claimed is a process for forming a SiC ingot using the above apparatus, the process involving (a) positioning a SiC seed (112) in the chamber (100), (b) evacuating the chamber, (c) cleaning the seed, and (d) growing SiC on the seed

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(19) Europäisches Patentamt
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(74) Mandataire Signore, Robert
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(54) Dispositif et procédé pour la formation de carbure de silicium (SiC) monocristallin sur un germe

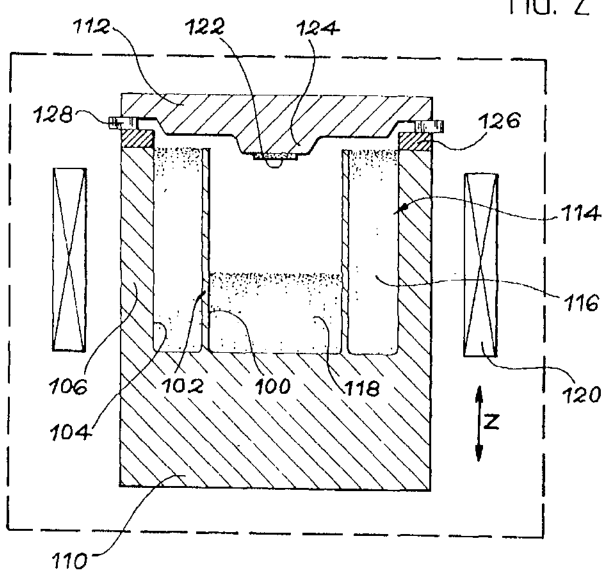
(57) L'invention concerne un dispositif pour la formation de SiC sur un germe

Le dispositif comporte

- une première enceinte (100) délimitée par au moins une paroi (102, 110, 112) et pouvant recevoir un germe de SiC (122),
- un réservoir (118) de poudre de SiC,
- des moyens (120) de chauffage de l'enceinte, et, conformément à l'invention, la paroi (102, 110, 112) est pour l'essentiel recouverte d'au moins une couche (116) de SiC

Application à la fabrication de lingots de SiC

FIG. 2



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Apparatus and process for the formation of monocrystalline silicon carbide (SiC) on a nucleus

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Inventor(s)	MADARD ROLAND (FR); ANIKIN MIKHAIL (FR), GARCON ISABELLE (FR), JAUSSAUD CLAUDE (FR)
Applicant(s)	COMMISSARIAT ENERGIE ATOMIQUE (FR)
Requested Patent.	EP0801155
Application Number.	US19970824093 19970325
Priority Number(s)	FR19960004450 19960410
IPC Classification	C30B23/06
EC Classification	C30B23/00
Equivalents:	FR2747401, JP10036195

Abstract

The invention relates to an apparatus for forming SiC on a nucleus. The apparatus comprises a first enclosure (100) defined by at least one wall (102, 110, 112) and able to receive a SiC nucleus (122), a SiC powder reservoir (118) and means (120) for heating the enclosure and, according to the invention, the wall (102, 110, 112) is essentially covered by at least one SiC layer (116)

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(71) Applicant UBE INDUSTRIES, LTD.

(72) Inventors
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- Inoue, Yoshiyuki c/o Ube Lab. of Ube Ind., Ltd.
- Yamaoka, Hiroyuki c/o Ube Lab. of Ube Ind., Ltd.

(54) Silicon carbide fiber having excellent alkali durability and process for the production thereof

(57) A crystalline silicon carbide fiber excellent not only in mechanical properties but also in alkali durability at high temperatures, which has a density of at least 2 7 g/cm3, contains 55 to 70 % by weight of Si, 30 to 45 % by weight of C, 0.06 to 3 8 % by weight of Al and 0 06 to 0 5 % by weight of B, the total of these elements being 100 % by weight, and has a sintered structure of SiC

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(19) Europäisches Patentamt
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(87) International publication number
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(84) Designated Contracting States
DE FR GB IT SE

(30) Priority 26.06.1995 SE 9502288

(71) Applicants
- ABB RESEARCH LTD.
- OKMETIC OY

(72) Inventors
- KORDINA, Olle
- HALLIN, Christer
- JANZEN, Erik

(54) A DEVICE AND A METHOD FOR EPITAXIALLY GROWING OBJECTS BY CVD

(57) A device for epitaxially growing objects of SiC, a group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate (13) comprises a susceptor (7) having circumferential walls (8) surrounding a room (18) for receiving a substrate and means (11) for heating said circumferential susceptor walls and by that the substrate and a gas mixture fed to the substrate for the growth by feeding means (5) The heating means (11) is arranged to heat the susceptor (7) and by that the substrate (13, 13') above a temperature level from which sublimation of the material grown starts to increase considerably, and the feeding means is arranged to feed said gas mixture with such a composition and at such a rate into the susceptor that a positive growth takes place

(57) Ce dispositif destiné à la croissance épitaxiale d'articles à base de SiC, de nitrure du groupe III ou d'alliages de ceux-ci, par dépôt chimique en phase vapeur sur un substrat (13), comprend un suscepteur (7) présentant des parois (8) circulaires entourant une chambre (18) destinée à recevoir un substrat, des moyens (11) de chauffage desdites parois, ainsi que des moyens (5) d'alimentation servant à apporter un mélange de gaz au substrat afin de permettre ladite croissance Les moyens (11) de chauffage sont conçus pour chauffer le suscepteur (7) et par là même le substrat (13, 13'), afin de porter ce dernier à un niveau de température à partir duquel la sublimation du matériel tiré commence à augmenter considérablement, lesdits moyens d'alimentation étant eux-mêmes conçus pour apporter, dans le suscepteur, le mélange de gaz selon une telle composition et à un tel débit qu'il se produise une croissance positive

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(19) Europäisches Patentamt
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(12) EUROPEAN PATENT APPLICATION

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WO 97/05303

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(30) Priority 27.07.1995 DE 19527536

(71) Applicant SIEMENS AKTIENGESELLSCHAFT

(72) Inventors
- STEIN, René
- RUPP, Roland
- VÖLKL, Johannes

(54) **PROCESS FOR PRODUCING SILICON CARBIDE MONOCRYSTALS**

(57) A new process is disclosed for producing SiC three-dimensional monocrystals SiC powder or another starting material is dissolved under high overpressures in a solvent and grown on a seed

(57) L'invention concerne un nouveau procédé de production de monocristaux tridimensionnels de SiC La poudre de SiC ou un autre matériau de départ est dissout(e) à une surpression élevée dans un solvant et cultivé(e) sur un germe cristallin

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(19) Europäisches Patentamt
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(30) Priority **23.01.1997 JP 1045797**

(71) Applicant **Oji Paper Company Limited**

(72) Inventors
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- **Kato, Hitoshi**
- **Okada, Kaoru**
- **Kubo, Ryoji**

(54) **Process for producing silicon carbide fibers**

(57) Silicon carbide fibers, which may be in the form of a shaped article, for example, a sheet, can be produced with a high efficiency by mixing a silicon-supply source powder containing silicon and/or silicon oxides with activated carbon fibers having a fiber thickness of 1 to 20 μm and a specific surface area of 300 to 2000 m2/g determined by the BET nitrogen absorption method, heating the mixture at a temperature of 1200 to 1500°C in an atmosphere substantially free from substances reactive with carbon, silicon, silicon oxides and silicon carbide, for example, in a flow of an inert gas or under a reduced pressure of 103 Pa of less

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(84) Designated Contracting States
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(71) Applicants
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(72) Inventors
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- HALLIN, Christer
- JANZEN, Erik
- VEHANEN, Asko
- YAKIMOVA, Rositza
- TUOMINEN, Marko

(54) A METHOD FOR EPITAXIALLY GROWING OBJECTS AND A DEVICE FOR SUCH A GROWTH

(57) In a method for epitaxially growing objects of SiC, a Group III-nitride or alloys thereof on a substrate (13) received in a susceptor (7) having circumferential walls (8) these walls and by that the substrate and a source material (24) for the growth are heated above a temperature level from which sublimation of the material grown starts to increase considerably The carrier gas flow is fed into the susceptor towards the substrate for carrying said source material to the substrate for said growth. At least a part of said source material for said growth is added to the carrier gas flow upstream the susceptor (7) and carried by the carrier gas flow to the susceptor in one of a) a solid state and b) a liquid state for being brought to a vapour state in a container comprising said susceptor by said heating and carried in a vapour state to said substrate for said growth

(57) Cette invention concerne un procédé permettant de réaliser la croissance épitaxiale d'objets faits de SiC, d'un nitrure du Groupe III, ou d'un alliage de ces derniers, sur un substrat (13) se trouvant dans un suscepteur (7) à parois circulaires (8) Ces parois ainsi que le substrat et une matière de base (24) pour la croissance, sont chauffés à une température supérieure à celle à partir de laquelle la sublimation de la matière qui s'est développée commence à s'accroître fortement Le flux de gaz porteur est introduit dans le suscepteur et dirigé vers le substrat afin de transporter vers ce dernier la matière de base servant à la croissance Une partie au moins de la matière de base servant à la croissance est introduite dans le flux de gaz porteur en amont du suscepteur (7), puis transportée par ledit flux dans le suscepteur soit (a) en phase solide, soit (b) en phase liquide Cette partie de la matière de base est ensuite portée en phase vapeur par chauffage dans un conteneur renfermant ledit suscepteur, puis envoyée en phase vapeur vers le substrat afin de réaliser la croissance

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(30) Priority 05.02.1996 US 596526

(71) Applicant CREE RESEARCH, INC.

(72) Inventors
- CARTER, Calvin, H.
- TSVETKOV, Valeri, F.
- GLASS, Robert, C.

(54) GROWTH OF COLORLESS SILICON CARBIDE CRYSTALS

(57) Large single crystals of silicon carbide are grown in a furnace sublimation system The crystals are grown with compensating levels of p-type and n-type dopants (i e , roughly equal levels of the two dopants) in order to produce a crystal that is essentially colorless The crystal may be cut and fashioned into synthetic gemstones having extraordinary toughness and hardness, and a brilliance meeting or exceeding that of diamond

(57) L'invention concerne de grands cristaux uniques de carbure de silicium qui sont tirés dans le four d'un système de sublimation Les cristaux sont tirés avec des niveaux de compensation de dopants de type p et de type n (c -à-d des niveaux à peu près égaux de deux dopants) afin de produire un cristal essentiellement incolore Le cristal peut être découpé et façonné sous forme de pierres précieuses synthétiques présentant une résistance et une dureté extraordinaires, et une brillance équivalente ou supérieure à celle du diamant

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(30) Priority 01.09.1997 JP 251394/97

(71) Applicant **Tokai Carbon Company, Ltd.**

(72) Inventors
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- **Yasunaka, Tomiya**
- **Ushijima, Yuji**
- **Kanai, Kenichi**

(54) **Silicon carbide fabrication**

(57) An SiC fabrication comprising a CVD-SiC fabrication excellent in strength and thermal characteristics The SiC fabrication is prepared with a CVD process (i e CVD-SiC fabrication) which has a thermal conductivity along the direction of the SiC crystal growth between 100 and 300 W/m K, and an average grain diameter of the internal structure between 4 to 12 um It is preferred that the ratio of the thermal conductivity along the direction of the SiC crystal growth to the thermal conductivity in the perpendicular direction is in a range of 1 10 to 1 40

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(71) Applicant NIPPON PILLAR PACKING CO. LTD.

(72) Inventors
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- Hiramoto, Masanobu c/o Nippon Pillar Pack.Co.,Ltd.

(54) Single crystal SiC and a method of producing the same

(57) The single crystal SiC according to the invention is produced in the following manner Two complexes M in each of which a polycrystalline film 2 of β-SiC (or α-SiC) is grown on the surface of a single crystal α-SiC substrate 1 by thermochemical deposition, and the surface 2a of the polycrystalline film 2 is ground so that the smoothness has surface roughness of 200 angstroms RMS or smaller, preferably 100 to 50 angstroms RMS are subjected to a heat treatment under a state where the complexes are closely fixed to each other via their ground surfaces 2a', at a high temperature of 2,000°C or higher and in an atmosphere of a saturated SiC vapor pressure, whereby the polycrystalline films 2 of the complexes M are recrystallized to grow a single crystal which is integrated with the single crystal α-SiC substrates 1 Large-size single crystal SiC in which impurities, micropipe defects, and the like do not remain, and which has high quality can be produced with high productivity

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(71) Applicant NIPPON PILLAR PACKING CO. LTD.

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(54) **Single crystal SiC and a method of producing the same**

(57) The single crystal SiC according to the invention is produced in the following manner The surface 1a of a single crystal α-SiC substrate 1 is adjusted so as to have a surface roughness equal to or lower than 2,000 angstroms RMS, and preferably equal to or lower than 1,000 angstroms RMS On the surface 1a of the single crystal α-SiC substrate 1, a polycrystalline α-SiC film 2 is grown by thermal CVD Thereafter, the complex M is placed a porous carbon container 3, and the outer side of the carbon container 3 is covered with α-SiC powder 4 The complex M is subjected to a heat treatment at a high temperature equal to or higher than a film growing temperature, i e , in the range of 1,900 to 2,400°C in an argon gas flow, whereby single crystal α-SiC is integrally grown on the single crystal α-SiC substrate 1 by crystal growth and recrystallization of the polycrystalline α-SiC film 2 It is possible to stably and efficiently produce single crystal SiC of a large size which has a high quality and in which any crystal nucleus is not generated

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(19) Europäisches Patentamt
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(30) Priority **23.05.1997 JP 17090297**

(71) Applicant **NIPPON PILLAR PACKING CO., LTD.**

(72) Inventor **TANINO, Kichiya, Nippon Pillar Packing Co., Ltd**

(54) **SINGLE CRYSTAL SILICON CARBIDE AND PROCESS FOR PREPARING THE SAME**

(57) According to the invention, a complex (M) which is formed by growing a polycrystalline β-SiC plate (2) on the surface of a single crystal α-SiC base material (1) by the thermal CVD method is heat-treated at a high temperature of 1,900 to 2,400°C, whereby polycrystals of the polycrystalline cubic β-SiC plate (2) are transformed into a single crystal, so that the single crystal is oriented in the same direction as the crystal axis of the single crystal α-SiC base material (1) and integrated with the single crystal of the single crystal α-SiC base material (1) to be largely grown As a result, single crystal SiC of high quality which has a very reduced number of lattice defects and micropipe defects can be efficiently produced while ensuring a sufficient size in the term of area

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(19) Europäisches Patentamt
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(71) Applicant NIPPON PILLAR PACKING CO., LTD.

(72) Inventor TANINO, Kichiya, Nippon Pillar Packing Co., Ltd.

(54) SINGLE CRYSTAL SiC AND PROCESS FOR PREPARING THE SAME

(57) According to the invention, a complex (M) which is formed by stacking a polycrystalline β-SiC plate (2) on the surface of a single crystal α-SiC base material (1) in a close contact state via a polished face or grown in a layer-like manner by the thermal CVD method is heat-treated in a temperature range of 1,850 to 2,400°C, whereby polycrystals of the polycrystalline cubic β-SiC plate (2) are transformed into a single crystal, and the single crystal oriented in the same direction as the crystal axis of the single crystal α-SiC base material (1) is grown As a result, large single crystal SiC of high quality which is free from micropipe defects, lattice defects, generation of grain boundaries due to intrusion of impurities, and the like can be produced easily and efficiently

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(19) Europäisches Patentamt
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(12) EUROPEAN PATENT APPLICATION

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(84) Designated Contracting States **DE FR GB**

(30) Priority **25.06.1997 JP 20703997**

(71) Applicant **NIPPON PILLAR PACKING CO., LTD.**

(72) Inventor **TANINO, Kichiya, Nippon Pillar Packing Co., Ltd.**

(54) **SINGLE CRYSTAL SiC AND PROCESS FOR PREPARING THE SAME**

(57) According to the invention, a complex (M) which is formed by growing a polycrystalline β-SiC plate (2) having a thickness of 10 um or more on the surface of a single crystal α-SiC base material (1) by the PVD method or the thermal CVD method is heat-treated at a temperature of the range of 1,650 to 2,400°C, whereby polycrystals of the polycrystalline cubic β-SiC plate (2) are transformed into a single crystal, and the single crystal oriented in the same direction as the crystal axis of the single crystal α-SiC base material (1) is grown As a result, single crystal SiC of high quality which is substantially free from micropipe defects and defects affected by the micropipe defects can be produced easily and efficiently

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(19) Europäisches Patentamt
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(71) Applicant **SUMITOMO ELECTRIC INDUSTRIES, LTD.**

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(54) **Semiconductor crystal, and method and apparatus of production**

(57) An apparatus and method of providing a large semiconductor crystal at a low cost are provided The apparatus of producing a semiconductor crystal includes a reactor (1) having an open end at both end sides, formed of any one material selected from the group consisting of silicon carbide, silicon nitride, aluminum nitride, and aluminum oxide, or of a composite material with any one material selected from the group consisting of silicon carbide, silicon nitride, aluminum nitride, boron nitride, aluminum oxide, magnesium oxide, mullite, and carbon as a base, and having an oxidation-proof or airtight film formed on the surface of the base, a kanthal heater (3) arranged around the reactor (1) in the atmosphere, a flange (9) attached at the open end to seal the reactor (1), and a crucible (2) mounted in the reactor (1) to store material of a semiconductor crystal The material stored in the crucible (2) is heated and melted to form material melt (60) The material melt is solidified to grow a semiconductor crystal (50)

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(19) Europäisches Patentamt
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(71) Applicants
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(54) A DEVICE FOR EPITAXIALLY GROWING OBJECTS AND METHOD FOR SUCH A GROWTH

(57) A device for epitaxially growing objects of for instance SiC by Chemical Vapour Deposition on a substrate has a first conduit (24) arranged to conduct substantially only a carrier gas to a room (18) receiving the substrate and a second conduit (25) received in the first conduit, having a smaller cross section than the first conduit and extending in the longitudinal direction of the first conduit with a circumferential space separating it from inner walls of the first conduit The second conduit is adapted to conduct substantially the entire flow of reactive gases and it ends as seen in the direction of said flows, and emerges into said first conduit at a distance from said room

(57) Un dispositif de formation par épitaxie d'objets constitués, par exemple, de SiC, par dépôt chimique en phase vapeur sur un substrat, présente un premier conduit (24) conçu pour le transport de sensiblement seulement un gaz vecteur dans un espace (18) dans lequel est placé le substrat, et un second conduit (25) situé dans le premier conduit, présentant une section inférieure au premier conduit et s'étendant dans l'axe longitudinal du premier conduit, un espace circonférentiel le séparant des parois intérieures du premier conduit Le second conduit qui est conçu pour le transport de sensiblement la totalité du flux de gaz réactifs, se termine, comme l'indique le sens d'écoulement des flux, dans ledit premier conduit, à une certaine distance dudit espace

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(19) Europäisches Patentamt
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(12) EUROPEAN PATENT APPLICATION

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26.01.1998 JP 1264698

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(54) **Method of making SiC single crystal and apparatus for making SiC single crystal**

(57) An apparatus comprises an Si-disposing section in which solid Si is disposed, a seed-crystal-disposing section in which a seed crystal of SiC is disposed, a synthesis vessel adapted to accommodate the Si-disposing section, the seed-crystal-disposing section, and carbon, heating means adapted to heat the Si-disposing section and the seed-crystal-disposing section, and a control section for transmitting to the heating means a command for heating the Si to an evaporation temperature of Si or higher and heating the seed crystal to a temperature higher than that of Si, wherein the Si evaporated by the heating means is adapted to reach the seed-crystal-disposing section

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(19) Europäisches Patentamt
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(30) Priority 18.12.1996 US 769090

(71) Applicant Northrop Grumman Corporation

(72) Inventors
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(54) **APPARATUS FOR GROWING SILICON CARBIDE CRYSTALS**

(57) A silicon carbide growth container for placement into a crystal growing furnace The growth container has a liner of pyrolytic graphite which seals the inside of the container and allows for easy removal of the grown silicon carbide crystal

(57) L'invention concerne un contenant pour la croissance de carbure de silicium, destiné à être placé dans un four de production de cristaux Ce récipient présente un revêtement de graphite pyrolitique qui permet d'étanchéifier l'intérieur dudit récipient et de retirer facilement le cristal de carbure de silicium ayant été produit

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(71) Applicants
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- **Ramm, Mark Spiridonovich**
- **Temkin, Leonid Iosifovich**

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- **Mokhov, Evgeny Nikolaevich**
- **Ramm, Mark Grigorievich**
- **Ramm, Mark Spiridonovich**
- **Roenkov, Alexandr Dmitrievich**
- **Temkin, Leonid Iosifovich**
- **Vodakov, Yury Alexandrovich**

(54) SILICON CARBIDE MONOCRYSTAL GROWTH

(57) A sublimation technique of growing silicon carbide single crystals, comprising a parallel arrangement, opposite each other, of the evaporating surface of a silicon carbide source (1) and the growing surface of at least one seed crystal (2) of a specified politype, to define a growth zone (4), and generation of a reduced pressure and an operating temperature field with an axial gradient in the direction from the seed crystal (2) towards the source (1), providing evaporation of silicon carbide of the source (1) and vapour-phase crystallization of silicon carbide on the growing surface of the seed crystal (2) The growth zone (4) is here sealed before the operating temperatures are reached therein, and the process is run with a solid solution of tantalum and silicon carbides in tantalum and their chemical compounds present in the growth zone (4) The material of the source (1) employed for implementing the sublimation technique of growing silicon carbide crystals is silison carbide ceramics

(57) Cette invention concerne un technique de sublimation destinée à la croissance de monocristaux de carbure de silicium, laquelle technique fait appel à un système parallèle comprenant une surface d'évaporation d'une source de carbure de silicium (1) qui fait face à une surface de croissance d'un ou plusieurs cristaux germes (2) d'un politype prédéterminé, les deux surfaces définissant une zone de croissance (4) Cette technique consiste à créer une chute de pression et un champ de températures fonctionnelles, tout en observant un gradient axial selon une direction menant du cristal germe (2) à la source (1) On procède ensuite à l'évaporation du carbure de silicium de la source (1), puis à la cristallisation en phase vapeur du carbure de silicium sur la surface de croissance du cristal germe (2) La zone de croissance (4) est scellée avant que l'on y atteigne les températures fonctionnelles, et le processus se déroule dans ladite zone de croissance (4) en présence d'une solution solide de tantale contenant des carbures de silicium, et en présence de leurs composés chimiques Le matériau utilisé pour la source (1) et permettant de mettre en oeuvre cette technique de sublimation, afin d'obtenir la croissance de cristaux de carbure de silicium, se compose de céramiques de carbure de silicium

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(19) Europäisches Patentamt
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(71) Applicant NORTHROP GRUMMAN CORPORATION

(72) Inventors
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- SEIDENSTICKER, Raymond, G.

(54) **APPARATUS FOR GROWING LARGE SILICON CARBIDE SINGLE CRYSTALS**

(57) An apparatus for growing single-polytype, single crystals of silicon carbide utilizing physical vapor transport as the crystal growth technique The apparatus has a furnace which has a carbon crucible (60) with walls that border and define a crucible cavity A silicon carbide source material (64) provided at a first location of the crucible cavity, and a monocrystalline silicon carbide seed (62) is provided at a second location of the crucible cavity A heat path (72) is also provided in the furnace above the crucible cavity The crucible has a stepped surface that extends into the crucible cavity The stepped surface has a mounting portion upon which the seed crystal is mounted The mounting portion of the stepped surface is bordered at one side by the crucible cavity and is bordered at an opposite side by the furnace heat path The stepped surface also has a sidewall that is bordered at one side by and surrounds the furnace heat path The apparatus may also have a thermal insulating member (59), in which a side of the stepped surface sidewall opposite to the furnace heat path is bordered by the thermal insulating member

(57) L'invention concerne un appareil de croissance de polytypes uniques, de monocristaux de carbure de silicium par utilisation de transport de vapeur physique comme technique de croissance cristalline L'appareil comporte un four possédant un creuset de carbone (60) doté de parois délimitant et définissant une cavité de creuset Une substance de base (64) de carbure de silicium est disposée à un premier emplacement de la cavité de creuset, et un germe (62) de carbure de silicium monocristallin est disposé à un deuxième emplacement de la cavité de creuset Une trajectoire de chaleur (72) est également créée dans le four au-dessus de la cavité de creuset Le creuset présente une surface à gradins s'étendant dans la cavité de creuset La surface à gradins comporte une partie de montage sur laquelle est monté le cristal germe La partie de montage de la surface à gradins est délimitée d'un côté par la cavité de creuset, et du côté opposé par la trajectoire de chaleur du four La surface à gradins comporte également une paroi latérale délimitée d'un côté par la trajectoire de chaleur qu'elle entoure L'appareil peut également comprendre un élément d'isolation thermique (59), un côté de la paroi latérale de la surface à gradins opposée à la trajectoire de chaleur du four étant délimité par l'élément d'isolation thermique

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(19) Europäisches Patentamt
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(30) Priority **26.02.1996 SE 9600705**

(71) Applicant **ABB RESEARCH LTD.**

(72) Inventors
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- **KORDINA, Olle**
- **NILSSON, Roger**

(54) **A SUSCEPTOR FOR A DEVICE FOR EPITAXIALLY GROWING OBJECTS AND SUCH A DEVICE**

(57) A susceptor for a device for epitaxially growing objects of one of a) SiC, b) a Group 3B-nitride, and c) alloys thereof on a substrate to be received in the susceptor has a channel (1) adapted to receive said substrate and through which a source material for the growth is intended to be fed The walls (11-14) of the susceptor surrounding said channel are made of a material which may be heated by induction created by heating means intended to surround the susceptor The susceptor is made of at least two separate susceptor wall pieces (11 14), and it comprises means (15) for securing said wall pieces of the susceptor to each other for forming the susceptor

(57) Suscepteur pour dispositif de croissance épitaxiale d'objets constitués par a) SiC, b) un nitrure du groupe 3B et, c) des alliages de ces matières, sur un substrat destiné à être placé sur le suscepteur Ce dernier comporte un canal conçu pour accueillir ledit substrat et par lequel la matière source de croissance doit être apportée Les parois (11-14) du suscepteur entourant ledit canal sont réalisées dans un matériau qui peut être chauffé par induction par un dispositif de chauffage conçu pour entourer le suscepteur Le suscepteur est constitué d'au moins deux parois distinctes (11-14) et comporte un dispositif (15) d'immobilisation desdites parois l'une par rapport à l'autre pour constituer ce suscepteur

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(30) Priority 13.04.1998 JP 10149298

(71) Applicant NIPPON PILLAR PACKING CO. LTD.

(72) Inventors:
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(54) **Single crystal SiC and a method of producing the same**

(57) In single crystal SiC according to the present invention, a single crystal α-SiC substrate 1 and a polycrystalline β -SiC plate 2 are laminated each other for fixation, the single crystal α-SiC substrate 1 and the polycrystalline β-SiC plate 2 are subjected to heat treatment under an inert gas atmosphere and a saturated SiC vapor atmosphere, whereby the single crystallization owing to solid-phase transformation of the polycrystalline β-SiC plate 2 and a progress of the single crystallization to a surface direction wherein a contact point is regarded as a starting point make a whole surface of layer of the polycrystalline β-SiC plate 2 grow efficiently into a single crystal integrated with the single crystal α -SiC substrate 1, whereby it is possible to produce single crystal SiC having high quality with high productivity, which is substantially free from lattice defects and micropipe defects

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(71) Applicant NIPPON PILLAR PACKING CO., LTD.

(72) Inventor TANINO, Kichiya, Nippon Pillar Packing Co., Ltd

(54) SINGLE CRYSTAL SiC AND PROCESS FOR PREPARING THE SAME

(57) According to the invention, a complex (M) which is formed by growing a polycrystalline β-SiC plate (2) by the thermal CVD method on crystal orientation faces (2a) which are unified in one direction of plural plate-like single crystal α-SiC pieces (2) that are stacked and closely contacted is subjected to a heat treatment at a temperature in the range of 1,850 to 2,400°C, whereby a single crystal which is oriented in the same direction as the crystal axes of the single crystal α-SiC pieces (2) is grown from the crystal orientation faces (2a) of the single crystal α-SiC pieces (2) toward the polycrystalline β-SiC plate (2) As a result, single crystal SiC of a high quality in which crystalline nuclei, impurities, micropipe defects, and the like are not substantially generated in an interface can be produced easily and efficiently

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21.07.1998 JP 22109998
17.07.1998 JP 20369698

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- **Fukushima, Yoshiaki, c/o Toyota Chuo Kenkyusho**
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- **Onda, Shoichi, c/o Denso Corporation**
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- **Kuriyama, Haruyoshi, c/o Denso Corporation**
- **Hasegawa, Takeshi, c/o Denso Corporation**

(54) **Method for manufacturing single crystal of silicon carbide**

(57) Micropipe defects existing in a silicon carbide single crystal are closed within the single crystal At least a portion of the micropipe defects opened on the surface of the silicon carbide single crystal (SiC substrate) is sealed up with a coating material Then heat treatment is performed so as to saturate the inside of the micropipe defects with silicon carbide vapors By this, the micropipe defects existing in the SiC substrate can be closed within the SiC substrate, not in a newly grown layer Further, the micropipe defects can be efficiently closed by filling the micropipe defects with a silicon carbide material by preliminarily using super critical fluid and the like

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(30) Priority 26.08.1998 US 140824

(71) Applicant DOW CORNING CORPORATION

(72) Inventors
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- Lipowitz, Jonathan
- Nguyen, Kimmai Thi

(54) **Process for making silicon carbide fibers using a controlled concentration of boron oxide vapor**

(57) A process for producing polycrystalline silicon carbide includes heating an amorphous ceramic fiber that contains silicon and carbon in an environment containing boron oxide vapor The boron oxide vapor is produced in situ by reaction of a boron containing material such as boron carbide and an oxidizing agent such as carbon dioxide and the amount of boron oxide vapor is controlled by varying both the amount and rate of addition of the oxidizing agent

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(19) Europäisches Patentamt
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(30) Priority 12.09.1997 JP 24891697
22.05.1998 US 86605 P

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(54) **METHOD AND APPARATUS FOR PRODUCING SILICON CARBIDE SINGLE CRYSTAL**

(57) A silicon carbide single crystal is produced by allowing a vapor evaporated from a silicon raw material to pass through a heated carbon member and then reach a seed crystal substrate on which a silicon carbide single crystal grows For this production, an apparatus is used, which has a reaction tube, a heating device and a graphite crucible, wherein the lower part of the crucible constitutes a silicon raw material-charging part, a seed crystal substrate is situated at the top of the crucible, and a carbon member, through which the vapor evaporated from a silicon raw material is capable of passing, is disposed intermediately between the silicon raw material-charging part and the seed crystal As the carbon member, a porous carbon structure, a carbon plate having a plurality of through holes and a carbon particle-packed layer can be mentioned

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(19) Europäisches Patentamt
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(11) **EP 1 038 055 A1**

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(71) Applicant **Northrop Grumman Corporation**

(72) Inventors
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- **BALAKRISHNA, Vijay**
- **HOBGOOD, H., McDonald**
- **HOPKINS, Richard, H.**
- **THOMAS, R., Noel**

(54) **METHOD AND APPARATUS FOR GROWING HIGH PURITY SINGLE CRYSTAL SILICON CARBIDE**

(57) Method and apparatus for growing semiconductor grade silicon carbide epitaxial layers or boules Pure silicon feedstock is melted and vaporized The vaporized silicon is reacted with a high purity carbon-containing gas, such as propane, and the gaseous species resulting from the reaction are deposited on a silicon carbide seed crystal, or substrate, resulting in the growth of monocrystalline silicon carbide

(57) Ce procédé et cet appareil servent à faire croître des lingots monocristallins ou des couches épitaxiales de carbure de silicium de qualité semi-conductrice A cet effet, on fait fondre la matière brute de silicium pur et on la vaporise Le silicium ainsi vaporisé est alors amené à réagir avec un gaz contenant du carbone de grande pureté, tel que le propane, et les espèces gazeuses produites par cette réaction sont déposées sur un cristal germe de carbure de silicium ou sur un substrat portant un tel cristal, ce qui entraîne la croissance de carbure de silicium monocristallin

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(19) Europäisches Patentamt
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AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE

(30) Priority 17.12.1997 US 992157

(71) Applicant CREE, INC.

(72) Inventors
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- KORDINA, Olle, Claes, Erik
- PAISLEY, Michael, James

(54) **GROWTH OF VERY UNIFORM SILICON CARBIDE EPITAXIAL LAYERS**

(57) An improved chemical vapor deposition method is disclosed that increases the uniformity of silicon carbide epitaxial layers and that is particularly useful for obtaining thicker epitaxial layers The method comprises heating a reactor to a temperature at which silicon carbide source gases will form an epitaxial layer of silicon carbide on a substrate in the reactor, and then directing a flow of source and carrier gases through the heated reactor to form an epitaxial layer of silicon carbide on the substrate with the carrier gases comprising a blend of hydrogen and a second gas in which the second gas has a thermal conductivity that is less than the thermal conductivity of hydrogen so that the source gases deplete less as they pass through the reactor than they would if hydrogen is used as the sole carrier gas

(57) L'invention concerne une méthode améliorée de dépôt chimique en phase vapeur, qui augmente l'uniformité de couches épitaxiales de carbure de silicium et s'avère particulièrement efficace pour donner des couches épitaxiales plus épaisses La méthode consiste à chauffer un réacteur à une température à laquelle les gaz source de carbure de silicium forment une couche épitaxiale de carbure de silicium sur un substrat disposé dans le réacteur La méthode consiste ensuite à faire circuler un flux de gaz source et de gaz vecteurs à travers le réacteur chauffé pour former sur ledit substrat une couche épitaxiale de carbure de silicium Les gaz vecteurs contiennent un mélange d'hydrogène et un deuxième gaz, lequel deuxième gaz présente une conductivité thermique inférieur à celle de l'hydrogène, ce qui fait que les gaz source s'épuisent moins lorsqu'ils passent à travers le réacteur qu'il n'est le cas lorsque l'hydrogène est utilisé comme unique gaz vecteur

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(19) Europäisches Patentamt
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(11) **EP 1 060 301 A1**

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(84) Designated Contracting States
DE GB

(71) Applicant **Northrop Grumman Corporation**

(72) Inventors
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- **THOMAS, Linard, M.**

(54) **CEILING ARRANGEMENT FOR AN EPITAXIAL GROWTH REACTOR**

(57) A ceiling arrangement for a high temperature epitaxial growth reactor in which silicon carbide epitaxial layers may be grown The ceiling includes an upper layer of carbon foam and a lower layer of graphite bonded thereto A support structure for the ceiling is coupled to a nozzle assembly, holding a gas delivering nozzle The support structure has a lower flange portion which includes an upwardly extending projection defining a knife edge upon which the ceiling rests The arrangement minimizes unwanted heat transfer from the ceiling to the nozzle assembly and nozzle

(57) Cette invention concerne un système de plafond qui est destiné à un réacteur de croissance épitaxiale à haute température dans lequel on peut faire pousser des couches épitaxiales de carbure de silicium Ce plafond comprend une couche supérieure de mousse de carbone ainsi qu'une couche inférieure de graphite collée à la couche supérieure Une structure de support du plafond est couplée à un système d'injecteurs qui comprend un injecteur d'alimentation en gaz Cette structure de support possède une partie flanc inférieur comportant une protubérance qui est orientée vers le haut et qui forme un bord de type lame sur lequel repose le plafond Ce système permet de minimiser tout transfert de chaleur indésirable depuis le plafond vers le système d'injecteurs et l'injecteur

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(19) Europäisches Patentamt
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(12) EUROPEAN PATENT APPLICATION

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(71) Applicants
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- OYANAGI, Naoki, Showa Denko K.K.

(54) **METHOD AND DEVICE FOR CUTTING AND MIRROR FINISHING SINGLE CRYSTAL SILICON CARBIDE**

(57) The present invention comprises a metal bond grind stone having a flat plate portion 10a and a tapered portion 10b, an electrode 13 opposed to the metal bond grind stone with a gap therebetween, voltage applying means 12 for applying a direct-current pulse voltage between the metal bond grind stone and the electrode, conductive liquid supplying means 14 for supplying a conductive liquid 15 between the metal bond grind stone and the electrode, and grind stone moving means 16 for moving the metal bond grind stone in a direction orthogonal to the shaft center thereof, and an ingot 1 of a single crystal SiC is thereby cut at the tapered portion 10b of the metal bond grind stone and the cut surface is then specular-worked at the flat plate portion 10a

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(19) Europäisches Patentamt
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(54) **Silicon carbide and process for its production**

(57) Silicon carbide having a resistivity of from 10^3 to 10^6 Ω cm and a powder X-ray diffraction peak intensity ratio of at least 0.005 as represented by Id1/Id2 where Id1 is the peak intensity in the vicinity of 2θ being 34° and Id2 is the peak intensity in the vicinity of 2θ being 36°

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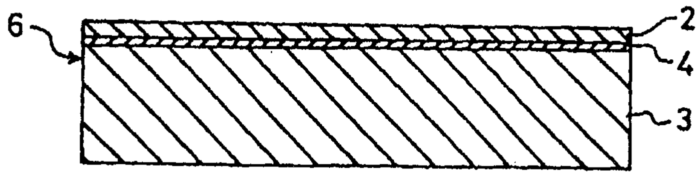
(54) Single crystal SiC composite material for producing a semiconductor device and a method of producing the same

(57) In a single crystal SiC composite material for producing a semiconductor device, and a method of producing the same according to the invention, a single crystal SiC film 2 which is produced on an Si substrate by the heteroepitaxial growth method and obtained by removing the Si substrate is stacked and bonded via a film-like SiO_2 layer 4 onto the surface of a polycrystalline plate 3 consisting of Si and C atoms in a closely contacted manner, and the composite member 6 is then heat-treated, whereby single crystal SiC in which the crystal is transformed in the same orientation as the single crystal of the single crystal SiC film 2 is integrally grown on the polycrystalline plate 3 The thickness and the strength which are requested for producing a semiconductor device can be ensured, and lattice defects and micropipe defects seldom occur, so that an accurate and high-quality semiconductor device can be produced

F I G. 3



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(30) Priority 14.07.1998 DE 19831556

(71) Applicant SIEMENS AKTIENGESELLSCHAFT

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(54) **METHOD AND DEVICE FOR PRODUCING AT LEAST ONE SILICON CARBIDE MONOCRYSTAL**

(57) The invention relates to a device for producing a silicon carbide (SiC) monocrystal (10), comprising a crucible (20) with a storage area (30) for receiving a supply (31) of solid SiC and a crystal area (12) for receiving an SiC crystal seed (11) An insert (51) made of glassy carbon is arranged in the crucible (20) According to the method provided for by the invention solid SiC is sublimated by heating the supply (31) thereof and SiC in its gaseous phase is created which is transported to the SiC crystal seed (11) and there grows into a SiC monocrystal (10) A heat supply (61) is controlled via the glassy coal insert (51)

(57) L'invention concerne un dispositif permettant de produire un monocristal de carbure de silicium (SiC) (10), qui comporte un creuset (20) pourvu d'une zone à réserve (30) destinée à recevoir une réserve (31) de SiC solide et d'une zone à cristal (12) destinée à loger un germe cristallin (11) Dans le creuset (20) est placé un insert (51) en carbone vitreux Selon ledit procédé, du SiC solide est sublimé par chauffage de ladite réserve (31), et du SiC est produit en phase gazeuse et amené au germe cristallin de SiC (11) où il se développe sous forme de monocristal de SiC (10) Un flux de chaleur (61) est régulé par l'intermédiaire de l'insert (51) en carbone vitreux

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