US006254677B1

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

(10) **Patent No.: US 6,254,677 B1**
(45) **Date of Patent: Jul. 3, 2001**

(54) **SEMICONDUCTOR CRYSTAL, AND METHOD AND APPARATUS OF PRODUCTION THEREOF**

(75) Inventors: **Katsushi Hashio; Shin-ichi Sawada; Masami Tatsumi**, all of Itami (JP)

(73) Assignee: **Sumitomo Electric Industries, Ltd.**, Osaka (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 : **09/217,349**

(22) Filed: **Dec. 21, 1998**

(30) **Foreign Application Priority Data**

Dec 26, 1997 (JP) 9-360090
Mar 23, 1998 (JP) 10-072969
Dec 11, 1998 (JP) 10-352557

(51) **Int. Cl.**[7] **C30B 29/42**; C30B 35/00

(52) **U.S. Cl.** **117/206**; 117/224; 117/900; 117/954

(58) **Field of Search** 117/206, 224, 117/954, 900, 953, 955, 956, 81–83

(56) **References Cited**

U S PATENT DOCUMENTS

3,615,878	*	10/1971	Chang	117/13
4,404,172	*	9/1983	Gault	117/223
4,704,257	*	11/1987	Tomizawa et al	117/203
5,132,145		7/1992	Valentian	
5,685,907	*	11/1997	Fujikawa et al	117/205

FOREIGN PATENT DOCUMENTS

2142388	6/1972	(DE)
0290322	11/1988	(EP)
0559921	9/1993	(EP)
2130192	5/1984	(GB)
2535312	5/1984	(FR)
2205087	11/1988	(GB)
55-140792	4/1980	(JP)
2-018375	1/1990	(JP)
2-120292	5/1990	(JP)
2-233578	9/1990	(JP)
7-221038	8/1995	(JP)

OTHER PUBLICATIONS

"Growth and Characterization of Single Crystals of PbTe–SnTe", by John W Wagner Becker et al., Transactions of the Metallurgical Society of AIME, vol 242, (1968), pp 366–371

"Effect of Ultrasonic Vibrations on InSb Pulled Crystals", by Yasuhiro Hayakawa et al., Japanese Journal of Applied Physics, vol 21, No 9 (1982), pp 1273–1277

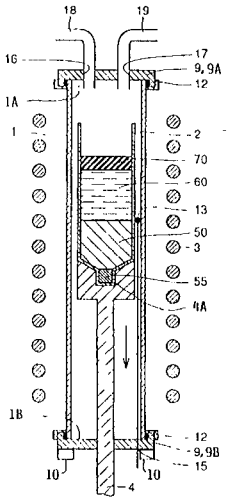
(List continued on next page)

Primary Examiner—Benjamin L Utech
Assistant Examiner—DuyVu Deo
(74) *Attorney, Agent, or Firm*—W. F. Fasse; W. G. Fasse

(57) **ABSTRACT**

An apparatus for and method of producing a large semiconductor crystal at a low cost are provided. The apparatus for producing a semiconductor crystal includes a reactor (**1**) having an open end at both ends thereof, that is formed of any material selected from the group consisting of silicon carbide, silicon nitride, aluminum nitride, and aluminum oxide, or of a composite material including a base 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 including an oxidation-proof or airtight film formed on the surface of the base The apparatus further includes a resistance 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 a material melt (**60**) The material melt is solidified to grow a semiconductor crystal (**50**)

5 Claims, 7 Drawing Sheets



US006110279A

United States Patent [19]

Kito et al.

[11] Patent Number: 6,110,279

[45] Date of Patent: Aug. 29, 2000

[54] **METHOD OF PRODUCING SINGLE-CRYSTAL SILICON CARBIDE**

[75] Inventors: **Yasuo Kito**; **Youichi Kotanshi**, both of Okazaki; **Shoichi Onda**, Toyokawa; **Tatuyuki Hanazawa**, Okazaki; **Eiji Kitaoka**, Anjo, all of Japan

[73] Assignee: **Denso Corporation**, Kariya, Japan

[21] Appl No : **09/049,979**

[22] Filed: **Mar. 30, 1998**

Related U.S. Application Data

[63] Continuation-in-part of application No 08/826,147, Mar 27, 1997, abandoned.

[30] **Foreign Application Priority Data**

Mar 29, 1996 [JP] Japan 8-75775
May 20, 1997 [JP] Japan 9-129875
Jun 19, 1997 [JP] Japan 9-163087

[51] **Int. Cl.**[7] **C30B 17/00**

[52] **U.S. Cl.** **117/105**; 117/109; 117/915

[58] **Field of Search** 117/951, 105, 117/109, 915

[56] **References Cited**

U S PATENT DOCUMENTS

4,147,572 4/1979 Vodakov et al
4,556,436 12/1985 Addamiano .
4,866,005 9/1989 Davis et al .
5,037,502 8/1991 Suzuki et al. .
5,441,011 8/1995 Takahaski et al
5,471,946 12/1995 Scholz et al
5,501,173 3/1996 Burk, Jr et al

FOREIGN PATENT DOCUMENTS

61-243000 10/1966 Japan
53-147700 12/1978 Japan
55-144499 11/1980 Japan
60-14000 4/1985 Japan
61-222992 10/1986 Japan .
63-57400 11/1988 Japan .
1-38080 8/1989 Japan
5-208897 8/1993 Japan .
6-24900 2/1994 Japan .
6-48898 2/1994 Japan .

OTHER PUBLICATIONS

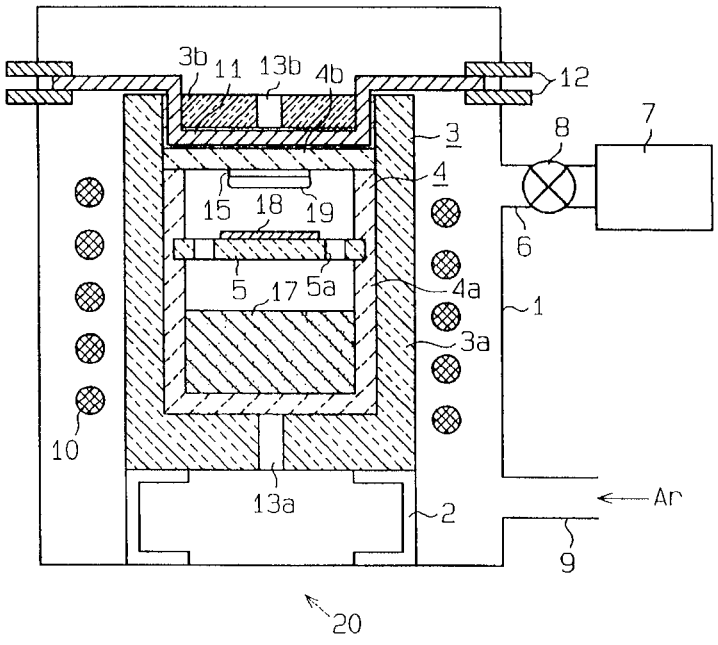
Single Crystal Growth of Hexagonal Sic on Cubic Sic by Intentional Polytype Control, Woo Sik Yoo et al (Also see Appln p. 5), pp 278–283.

Primary Examiner—Felisa Hiteshew
Attorney, Agent, or Firm—Pillsbury, Madison & Sutro, LLP

[57] **ABSTRACT**

A (111) cubic silicon carbide single-crystal layer is formed on a (111) silicon wafer, and then the silicon wafer is removed Thus prepared (111) cubic silicon carbide single-crystal layer is disposed in a graphite crucible to function as a seed crystal Silicon carbide source material powder is also held in the graphite crucible and sublimated in an atmosphere including inert gas, while controlling a temperature of the (111) cubic silicon carbide single-crystal layer to be lower than a temperature of the silicon carbide source material powder. As a result, a (0001) α-type silicon carbide single-crystal layer can be formed on the (111) cubic silicon carbide single-crystal layer with a large diameter and high quality at low cost

48 Claims, 11 Drawing Sheets



US006025289A

United States Patent [19]

Carter et al.

[11] Patent Number: 6,025,289

[45] Date of Patent: Feb. 15, 2000

[54] COLORLESS SILICON CARBIDE CRYSTALS

[75] Inventors: **Calvin H. Carter**, Cary; **Valeri F. Tsvetkov**, Durham; **Robert C. Glass**, Chapel Hill, all of N C

[73] Assignee: **Cree Research, Inc.**, Durham, N C

[21] Appl No : **08/984,938**

[22] Filed: **Dec. 4, 1997**

Related U.S. Application Data

[62] Division of application No. 08/596,526, Feb 5, 1996, Pat. No. 5,718,760.

[51] **Int. Cl.**[7] . . . **C04B 35/565**; C30B 29/36

[52] **U.S. Cl.** .... **501/86**; 252/62 3 C; 117/951; 501/88

[58] **Field of Search** . .. . 252/62 3 C; 117/951; 501/86, 88

[56] **References Cited**

U.S. PATENT DOCUMENTS

Re. 34,861	2/1995	Davis et al	
3,956,032	5/1976	Powell et al .. .	. 252/62 3 C
4,866,005	9/1989	Davis et al	437/100
4,966,860	10/1990	Suzuki et al.	
5,030,580	7/1991	Furukawa et al. .	
5,363,798	11/1994	Yodei	117/89
5,433,167	7/1995	Furukawa et al	
5,723,391	3/1998	Hunter et al.	501/86

OTHER PUBLICATIONS

Introduction to Ceramics, W D. Kingery et al , Second Edition, John Wiley & Sons, pp 676–679 No Date

Optical and Electronic Properties of SiC, W H Choyke, *The Physics and Chemistry of Carbides, Nitrides and Borides*, Manchester, England, Sep 1989, pp 1–25 35 al

Woo Sik Yee, "Bulk Crystal Growth of 6–H–SiC on Polytype Controlled Substrates through Vapor Phase and Characterization,", Journal of Crystal Growth; Dec 2, 1991; vol 15, No 1/04; Amsterdam.

Primary Examiner—Karl Group

Attorney, Agent, or Firm—Philip Summa, Patent Attorney

[57] **ABSTRACT**

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

10 Claims, No Drawings

US005989340A

United States Patent [19]

Stephani et al.

[11] Patent Number: 5,989,340

[45] Date of Patent: Nov. 23, 1999

[54] **PROCESS AND DEVICE FOR SUBLIMATION GROWING OF SILICON CARBIDE MONOCRYSTALS**

[75] Inventors: **Dietrich Stephani**, Bubenreuth; **Johannes Völkl**, Erlangen, both of Germany

[73] Assignee: **Siemens Aktiengesellschaft**, Munich, Germany

[21] Appl No.: **08/913,278**

[22] PCT Filed: **Nov. 14, 1995**

[86] PCT No : **PCT/DE95/01576**

§ 371 Date: **Aug. 27, 1997**

§ 102(e) Date: **Aug. 27, 1997**

[87] PCT Pub. No.: **WO96/17113**

PCT Pub. Date: **Jun. 6, 1997**

[51] **Int. Cl.**[6] **C35B 35/00**

[52] **U.S. Cl.** ... **117/204**; 117/84; 117/106; 117/900

[58] **Field of Search** 117/84, 89, 106, 117/200, 204, 900

[56] **References Cited**

U S PATENT DOCUMENTS

2,854,364	9/1958	Lely	117/84
4,147,572	4/1979	Vodakov et al	148/175
5,288,326	2/1994	Maeda et al	118/719
5,667,587	9/1997	Glass et al	117/200
5,704,985	1/1998	Kordina et al	118/725
5,707,446	1/1998	Völkl et al	117/200
5,792,257	8/1998	Kordina et al	117/90

FOREIGN PATENT DOCUMENTS

0 554 047 A1	8/1993	European Pat Off
24 09 005	9/1975	Germany
42 65294	9/1992	Japan .

OTHER PUBLICATIONS

Lilov, S., "Investigation of the Role of Crystal Growth Zone during Silicon Carbide Growth by the Sublimation Method," Cryst Res Technol 29 (1994) 1, pp. 69–75

Vodakov, Y, Epitaxial Growth of Silicon Carbide Layers by Sublimation "Sandwich Method" (I), Kristall und Technik, Bd 14, Nr 6, 1979, Berlin, DE, pp 729–740

Ivanov, P et al., "Recent developments in SiC single-crystal electronics," Semiconductor Science and Technology (1992), No 7 Bristol, GB, pp 863–880.

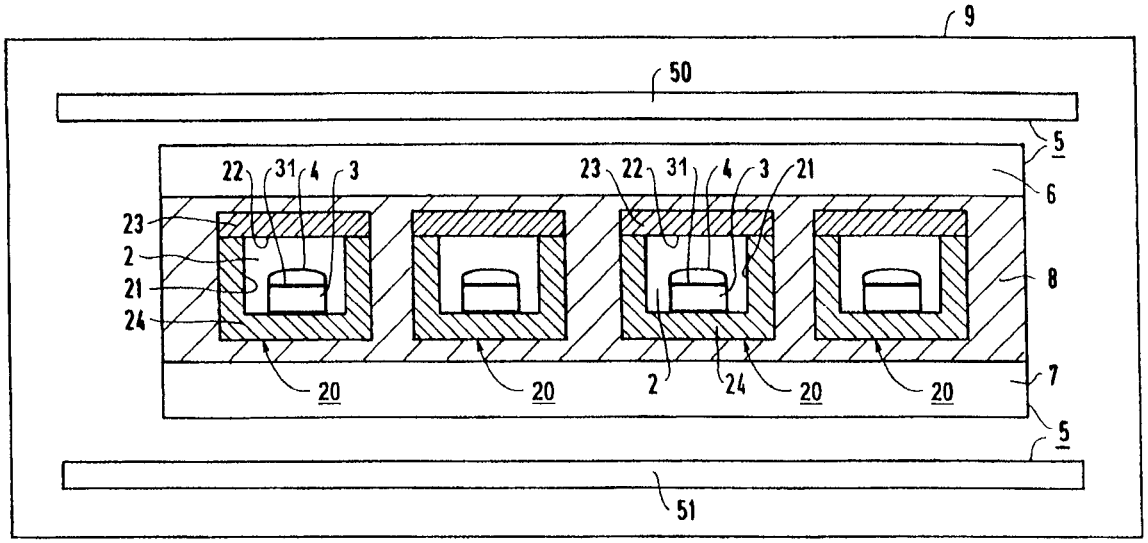
Primary Examiner—Felisa Hiteshew

Attorney, Agent, or Firm—Kenyon & Kenyon

[57] **ABSTRACT**

A reaction chamber (**2**) is enclosed by a gas-tight wall (**20**), made of silicon carbide obtained by a CVD process at least on the inside (**21**) facing the reaction chamber (**2**) At least part of the silicon carbide of the wall (**20**) is sublimated and grown on a seed crystal (**3**) as a silicon carbide monocrystal (**4**)

11 Claims, 6 Drawing Sheets



US005968261A

United States Patent [19]

Barrett et al.

[11] Patent Number: 5,968,261

[45] Date of Patent: Oct. 19, 1999

[54] **METHOD FOR GROWING LARGE SILICON CARBIDE SINGLE CRYSTALS**

[75] Inventors: **Donovan L. Barrett**, Penn Hills Township; **Raymond G. Seidensticker**, deceased, late of Forest Hills, by Joan Seidensticker, legal representative; **Richard H. Hopkins**, Murraysville, all of Pa.

[73] Assignee: **Northrop Grumman Corporation**, Los Angeles, Calif

[21] Appl No : **08/845,119**

[22] Filed: **Apr. 21, 1997**

Related U.S. Application Data

[63] Continuation of application No. 08/523,303, Sep 5, 1995, Pat No. 5,683,507.

[51] **Int. Cl.**[6] **C30B 15/00**

[52] **U.S. Cl.** **117/13**; 117/902; 117/937

[58] **Field of Search** 117/13, 14, 15, 117/902, 937

[56] **References Cited**

U S PATENT DOCUMENTS

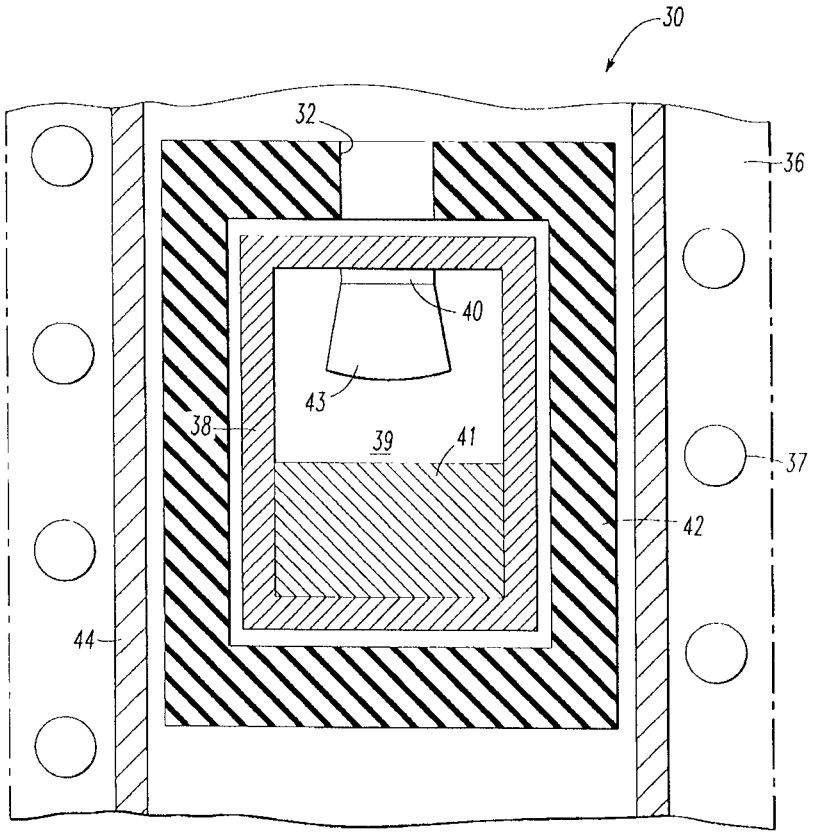
2,854,364	9/1958	Levy	117/84
4,866,005	9/1989	Davis et al	117/98
5,211,801	5/1993	Stein	117/84

Primary Examiner—Felisa Garrett

[57] **ABSTRACT**

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 with walls that border and define a crucible cavity. A silicon carbide source material provided at a first location of the crucible cavity, and a monocrystalline silicon carbide seed is provided at a second location of the crucible cavity A heat path 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, in which a side of the stepped surface sidewall opposite to the furnace heat path is bordered by the thermal insulating member

6 Claims, 5 Drawing Sheets



US005964944A

United States Patent [19]

Sugiyama et al.

[11] Patent Number: 5,964,944

[45] Date of Patent: Oct. 12, 1999

[54] METHOD OF PRODUCING SILICON CARBIDE SINGLE CRYSTAL

[75] Inventors: **Naohiro Sugiyama; Atsuto Okamoto; Toshihiko Tani**, all of Nagoya; **Nobuo Kamiya**, Nisshin, all of Japan

[73] Assignee: **Kabushiki Kaisha Toyota Chuo Kenkyusho**, Aichi-ken, Japan

[21] Appl No : **08/820,888**

[22] Filed: **Mar. 21, 1997**

[30] **Foreign Application Priority Data**

Mar 29, 1996 [JP] Japan 8-103719

[51] **Int. Cl.**[6] **C30B 29/36**

[52] **U.S. Cl.** **117/107**; 117/2; 117/902; 117/935; 117/951; 438/931

[58] **Field of Search** 117/2, 107, 902, 117/935, 951; 438/931

[56] **References Cited**

U S PATENT DOCUMENTS

3,074,887	1/1963	Carroll	117/2
3,275,415	9/1966	Chang	117/951
3,520,740	7/1970	Addamiano	117/951
3,755,541	8/1973	Strepkoff	117/951
4,512,825	4/1985	Addamiano	148/175
4,855,254	8/1989	Eshita	117/902
4,866,005	9/1989	Davis	117/107

FOREIGN PATENT DOCUMENTS

2-293398	12/1990	Japan
6-1698	1/1994	Japan
6-56596	3/1994	Japan .
6-128094	5/1994	Japan
6-298600	10/1994	Japan

OTHER PUBLICATIONS

Kobayashi et al. "Evaluation of structural quality of a silicon carbide (6H–SiC) ingle crystal grown by a vapor transport method by Rutherford backscattering spectroscopy", Journal of Applied Physics, vol 65, No. 4, pp. 1790–1792, Feb. 15, 1989.

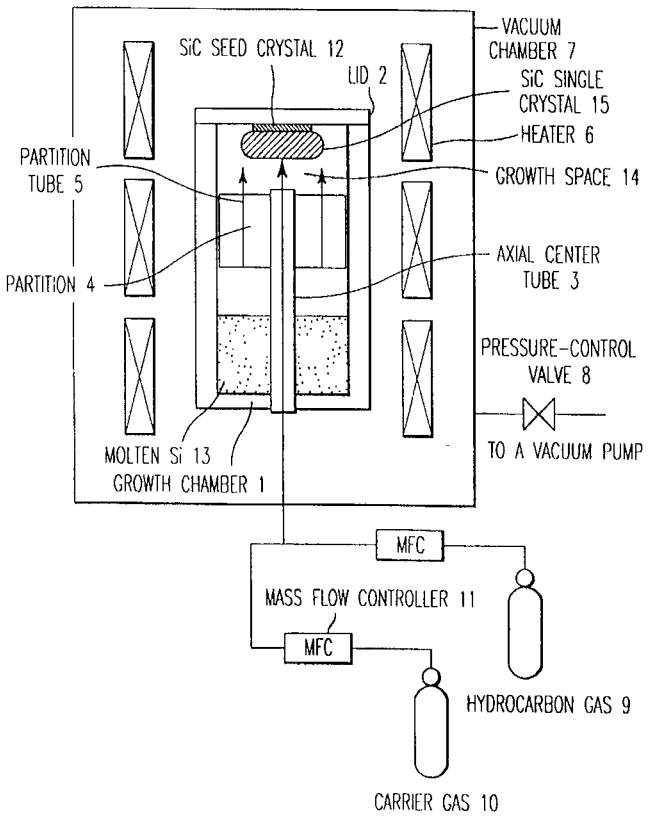
Primary Examiner—Robert Kunemund

Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt, P.C.

[57] **ABSTRACT**

An easy and low-cost method of producing a large-size and high-purity silicon carbide (SiC) single crystal includes reacting silicon vapor directly with a carbon-containing compound gas under a heated atmosphere (growth space **14**) to grow a silicon carbide single crystal (**15**) on a silicon carbide seed crystal (**12**), in which the silicon vapor generated from molten silicon (**13**) is used as a silicon vapor source, and a hydrocarbon gas (**9**) (e.g , propane gas) is used as the carbon-containing compound gas

9 Claims, 1 Drawing Sheet



US005958132A

United States Patent [19]

Takahashi et al.

[11] Patent Number: 5,958,132

[45] Date of Patent: Sep. 28, 1999

[54] **SIC SINGLE CRYSTAL AND METHOD FOR GROWTH THEREOF**

[75] Inventors: **Jun Takahashi; Masatoshi Kanaya; Yuichiro Fujiwara; Noboru Ohtani,** all of Kanagawa-ken, Japan

[73] Assignee: **Nippon Steel Corporation,** Japan

[21] Appl No.: **08/856,248**

[22] Filed: **May 14, 1997**

Related U.S. Application Data

[63] Continuation of application No. 08/410,731, Mar. 27, 1995, abandoned, which is a continuation-in-part of application No 08/264,744, Jun 22, 1994, abandoned, which is a continuation of application No 07/870,639, Apr 20, 1992, abandoned

[30] **Foreign Application Priority Data**

Apr 18, 1991	[JP]	Japan	3-087020
Jan 20, 1992	[JP]	Japan	4-007684
Mar. 25, 1994	[JP]	Japan	6-056036

[51] **Int. Cl.**[6] **C30B 23/06**

[52] **U.S. Cl.** **117/84**; 117/87; 117/109; 117/951; 148/DIG 148

[58] **Field of Search** 117/109, 84, 87, 117/951; 148/DIG 148

[56] **References Cited**

U S PATENT DOCUMENTS

2,854,364	9/1958	Lely	117/951
3,634,149	1/1972	Knippenberg	117/951
4,556,436	12/1985	Addamiano	117/951
4,866,005	9/1989	Davis et al	
5,011,549	4/1991	Kong et al	148/DIG 148
5,200,022	4/1993	Kong et al	148/DIG 148

FOREIGN PATENT DOCUMENTS

63-57400	11/1988	Japan	
2-48495	2/1990	Japan .	
02290084	11/1990	Japan	117/951
2290084	11/1990	Japan .	

OTHER PUBLICATIONS

Tairov et al "General Principles of Growing Large-Size Single Crystals of Various Silicon Carbide Polytypes", Jour of Crystal Growth vol 52 (1981) pp 146–150

Koga et al , Single Crystal Growth of 6H-SiC by a Vacuum Sublimation Method, Ext. ABS. of the 17th Conf. on Solid State Materials 1985 pp. 249–252

Data EFM–88–24, p. 24, published by Electric Science Society (Japan), Electronic Material Study Group, on Sep. 5, 1988.

Tairov et al , *Journal of Crystal Growth,* 36, 147–151 (1976)

Tairov et al , *Journal of Crystal Growth,* 43, 209–212 (1978)

Tairov et al., *Journal of Crystal Growth,* 52, 146–150 (1981)

Koga et al., *Vacuum,* 30(11), 886–892.

Koga et al., Extended Abstracts of the 17th Conference on Solid State Devices and Materials, Tokyo, pp 249–252 (1985)

Primary Examiner—Robert Kunemund

Attorney, Agent, or Firm—Wenderoth, Lind & Ponack, L.L P

[57] **ABSTRACT**

A method for the growth of a SiC single crystal comprising

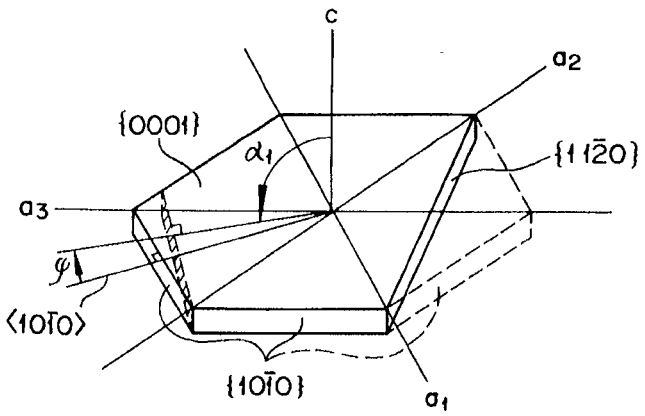
introducing a seed crystal of SiC single crystal having an exposed face deviating from the {0001} plane by an angle α_1 of about 60° to about 120°, typically about 90° and SiC powder as a raw material into a graphite crucible,

elevating the temperature of the SiC powder in an atmosphere of inert gas to a level sufficient for sublimation, meanwhile

elevating the temperature of the exposed face of the seed crystal to a level slightly lower than the temperature of the SiC powder, and

keeping the SiC powder and the seed crystal at the specific temperatures for a period enough for a SiC single crystal of the same polytype as the seed crystal to grow to a desired height on the exposed face of the seed crystal.

15 Claims, 5 Drawing Sheets



US005653798A

United States Patent [19]

Parsons et al.

[11] Patent Number: 5,653,798

[45] Date of Patent: Aug. 5, 1997

[54] **METHOD OF MAKING SUBSTRATES FOR THE GROWTH OF 3C-SILICON CARBIDE**

[75] Inventors: **James D. Parsons**, Beaverton; **Ajay Kumar Chaddha**; **Her Song Chen**, both of Portland; **Jin Wu**, Beaverton, all of Oreg.

[73] Assignee: **Oregon Graduate Institute of Science and Technology**, Beaverton, Oreg.

[21] Appl. No.: **485,982**

[22] Filed: **Jun. 7, 1995**

Related U.S. Application Data

[63] Continuation of Ser. No 83,903, Jun 23, 1993, Pat. No. 5,492,752, which is a continuation-in-part of Ser No. 986, 999, Dec. 7, 1992, abandoned.

[51] **Int. Cl.**[6] **C30B 25/02**

[52] **U.S. Cl.** **117/2**; 117/104; 427/248.1

[58] **Field of Search** 117/2, 84, 88, 117/104, 937; 427/248.1; 437/235

[56] **References Cited**

U.S. PATENT DOCUMENTS

4,639,028	1/1987	Olson	294/34
4,738,937	4/1988	Parsons	437/180
4,767,666	8/1988	Bunshah et al	428/334
4,912,063	3/1990	Davis et al	117/97
4,923,716	5/1990	Brown et al	427/249
4,946,547	8/1990	Palmour et al	156/643
5,043,773	8/1991	Precht et al	357/16
5,184,199	2/1993	Fujii et al.	257/77
5,326,992	7/1994	Yoder	257/77
5,492,752	2/1996	Parsons et al	428/212

FOREIGN PATENT DOCUMENTS

3613012	11/1986	Germany
2010772	1/1990	Japan
2199098	8/1990	Japan .

OTHER PUBLICATIONS

Otani, et al., "Effect of W Doping on the Growth of TiC Crystal by the Floating Zone Method," *Journal of Crystal Growth*, 92 (1988) pp. 359–363.

Zhao, et al., "Defect Structure In Single Crystal Titanium Carbide," *J. Mater. Res.*, vol. 9, No. 8, Aug. 1994, pp. 2096–2101.

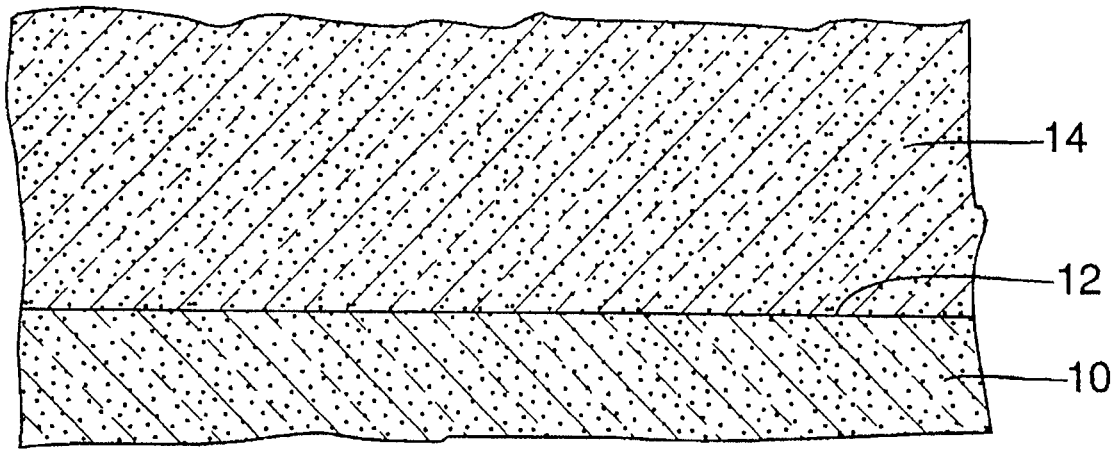
Primary Examiner—Felisa Garrett

Attorney, Agent, or Firm—Marger, Johnson, McCollom & Stolowitz P.C.

[57] **ABSTRACT**

A substrate for the growth of monocrystalline β-SiC is formed by providing a body of monocrystalline hexagonal material having a planar surface with a lattice parameter that is within ±5% of the lattice parameter of 6Hα-SiC in the basal plane and growing a body of monocrystalline cubic material on the surface to provide a planar cubic material surface that is without grain boundaries, subgrain boundaries, double positioning boundaries, and pits. The cubic material, for example TiC, ZrC, HfC, or TiN, has a rock salt structure and a lattice parameter within ±5% of the lattice parameter of β-SiC. Monocrystalline β-SiC can be nucleated and grown on the surface of the cubic material.

33 Claims, 1 Drawing Sheet



US005441011A

United States Patent [19]

Takahaski et al.

[11] Patent Number: 5,441,011

[45] Date of Patent: Aug. 15, 1995

[54] SUBLIMATION GROWTH OF SINGLE CRYSTAL SIC

[75] Inventors: Jun Takahaski; Masatoshi Kanaya, both of Sagamihara, Japan

[73] Assignee: Nippon Steel Corporation, Tokyo, Japan

[21] Appl. No.: 213,055

[22] Filed: Mar. 15, 1994

[30] Foreign Application Priority Data

Mar. 16, 1993 [JP] Japan 5-055687

[51] Int. Cl.[6] C30B 23/00; H01L 21/20

[52] U.S. Cl. 117/84; 117/105; 117/951; 437/100; 148/DIG. 148

[58] Field of Search 117/84, 88, 89, 951, 117/105; 437/100; 148/DIG. 148

[56] References Cited

U.S. PATENT DOCUMENTS

2,854,364	9/1958	Lely .	
3,147,159	9/1964	Lowe	437/100
3,236,780	2/1966	Ozarow	117/951
3,343,920	9/1967	Lowe	117/951
3,615,930	10/1971	Knippenber et al	17/951
4,147,572	4/1979	Vodakov et al.	117/951
4,866,005	9/1989	Davis et al. .	
5,211,801	5/1993	Stein	117/951

FOREIGN PATENT DOCUMENTS

63-57400	11/1988	Japan .	
WO89/04055	5/1989	WIPO	437/100

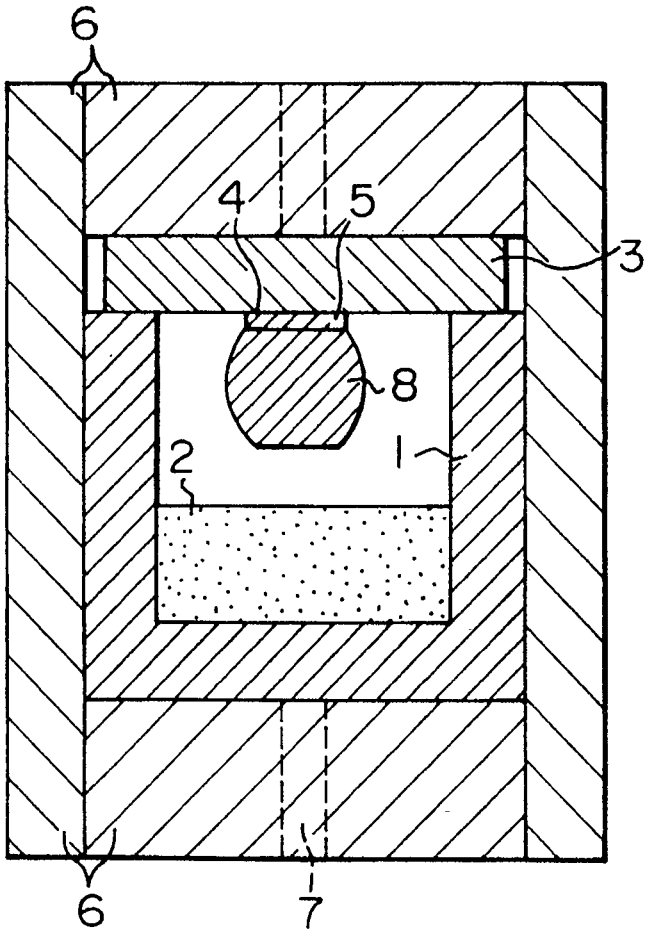
Primary Examiner—Mary Wilczewski

Attorney, Agent, or Firm—Pollock, Vande Sande & Priddy

[57] ABSTRACT

A method of growing a first SiC single crystal on a seed crystal including a second SiC single crystal, comprises the steps of setting a SiC source material at an initial temperature, growing the first SiC single crystal on the seed crystal including the second SiC single crystal at a temperature lower than the initial temperature of the source material and gradually decreasing the source material temperature from the initial temperature during at least a predetermined period during the growing step.

7 Claims, 2 Drawing Sheets



US005363800A

United States Patent [19]

Larkin et al.

[11] Patent Number: 5,363,800

[45] Date of Patent: Nov. 15, 1994

[54] **PROCESS FOR THE CONTROLLED GROWTH OF SINGLE-CRYSTAL FILMS OF SILICON CARBIDE POLYTYPES ON SILICON CARBIDE WAFERS**

[75] Inventors: **David J. Larkin**, Fairview Park; **Powell, J. Anthony**, North Olmsted, both of Ohio

[73] Assignee: **The United States of America as represented by the United States National Aeronautics and Space Administration**, Washington, D.C.

[21] Appl. No.: **973,505**

[22] Filed: **Nov. 9, 1992**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 718,315, Jun. 12, 1991, abandoned.

[51] **Int. Cl.**[5] **H01L 21/306**; H01L 21/20

[52] **U.S. Cl.** **117/95**; 117/97; 117/101; 117/106; 117/913; 117/923; 117/951; 437/100; 148/DIG. 168; 148/DIG. 148

[58] **Field of Search** 156/612; 148/DIG. 168, 148/DIG. 148; 437/100; 117/95, 97, 106, 913, 923, 101, 951

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,956,032	5/1976	Powell et al.	148/175
4,333,792	6/1992	Smith	156/612
4,612,072	9/1986	Morrison et al.	156/612
4,855,254	8/1989	Eshita et al	473/100
4,865,659	9/1989	Shigeta et al.	148/33
4,866,005	9/1989	Davis et al.	437/100
4,912,063	3/1990	Davis et al.	437/100
4,912,064	3/1990	Kong et al.	437/100
4,946,547	8/1990	Palmour et al.	156/643
5,190,613	3/1993	Yamagata	156/612
5,200,022	4/1993	Kong	156/612
5,230,768	7/1993	Furukawa et al.	156/612
5,248,385	9/1993	Powell	156/612

FOREIGN PATENT DOCUMENTS

270398	11/1988	Japan	156/612

OTHER PUBLICATIONS

V. J. Jennings et al, The Epitaxial Growth of Silicon Carbide; Journal of the Electrochemical Society, Jul. 1966, p. 730.

J. A. Powell et al, Growth of Improved Quality 3C-SiC Films on 6H-SiC Substrates Appl. Phys. Lett. 56(14), 2 Apr. 1990.

J. A. Powell et al, Growth of High Quality 6H-SiC Epitaxial Films on Vicinial (0001) 6H-SiC Wafers; Appl. Phys. Lett 56(15), 9 Apr. 1990.

J. A. Powell et al, Silicon Carbide, A Semiconductor for Space Power Electronics NASA Technical Memorandum 103655, Jan. 6-10, 1991.

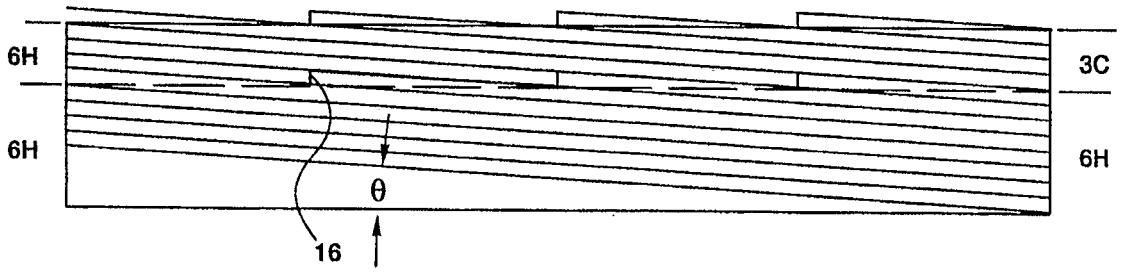
J. A. Powell et al, Controlled Growth of 3C-SiC and 6H-SiC Films on Low-Tilt-Angle Vicinal (0001) 6H-SiC Wafers, Appl. Phys. Lett. 59(3), 15 Jul. 1991.

Primary Examiner—R. Bruce Breneman
Assistant Examiner—Linda J. Fleck
Attorney, Agent, or Firm—Gene E. Shook; Guy M. Miller; James A. Mackin

[57] **ABSTRACT**

This invention is a method for the controlled growth of single-crystal semiconductor-device-quality films of SiC polytypes on vicinal (0001) SiC wafers with low tilt angles. Both homoepitaxial and heteroepitaxial SiC films can be produced on the same wafer. In particular, 3C-SiC and 6H-SiC films can be produced within selected areas of the same 6H-SiC wafer.

36 Claims, 4 Drawing Sheets



US005248385A

United States Patent [19]

Powell

[11] Patent Number: 5,248,385

[45] Date of Patent: Sep. 28, 1993

[54] **PROCESS FOR THE HOMOEPITAXIAL GROWTH OF SINGLE-CRYSTAL SILICON CARBIDE FILMS ON SILICON CARBIDE WAFERS**

[75] Inventor: **J. Anthony Powell**, North Olmsted, Ohio

[73] Assignee: **The United States of America, as represented by the Administrator, National Aeronautics and Space Administration**, Washington, D.C.

[21] Appl. No.: **718,314**

[22] Filed: **Jun. 12, 1991**

[51] **Int. Cl.**[5] **H01L 21/306**; H01L 21/20

[52] **U.S. Cl.** **156/645**; 148/33; 148/DIG. 148; 156/646; 156/662; 156/612; 156/DIG. 64; 252/79.1; 423/346; 427/249; 427/309; 437/100

[58] **Field of Search** ..... 156/636, 645, 646, 657, 156/662, 610-614, DIG. 64; 252/79.1, 79.2; 437/100, 105; 427/38, 309, 249; 148/33, 33.1, DIG. 148; 428/627, 446; 423/345-347

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,275,415	9/1966	Chang et al.	23/208
3,520,740	7/1970	Addamiano	148/175
4,664,944	5/1987	Hsu et al.	427/87
4,866,005	9/1989	Davis et al.	437/100
4,912,063	3/1990	Davis et al	437/100
4,912,064	3/1990	Kong et al.	437/100
4,946,547	8/1990	Palmour et al	156/643

OTHER PUBLICATIONS

Jennings, V. J. et al., "The Expitaxial Growth of Silicon Carbide," Journal of the Electrochemical Society, vol. 113, No. 7, Jul., 1966, pp. 728-731.

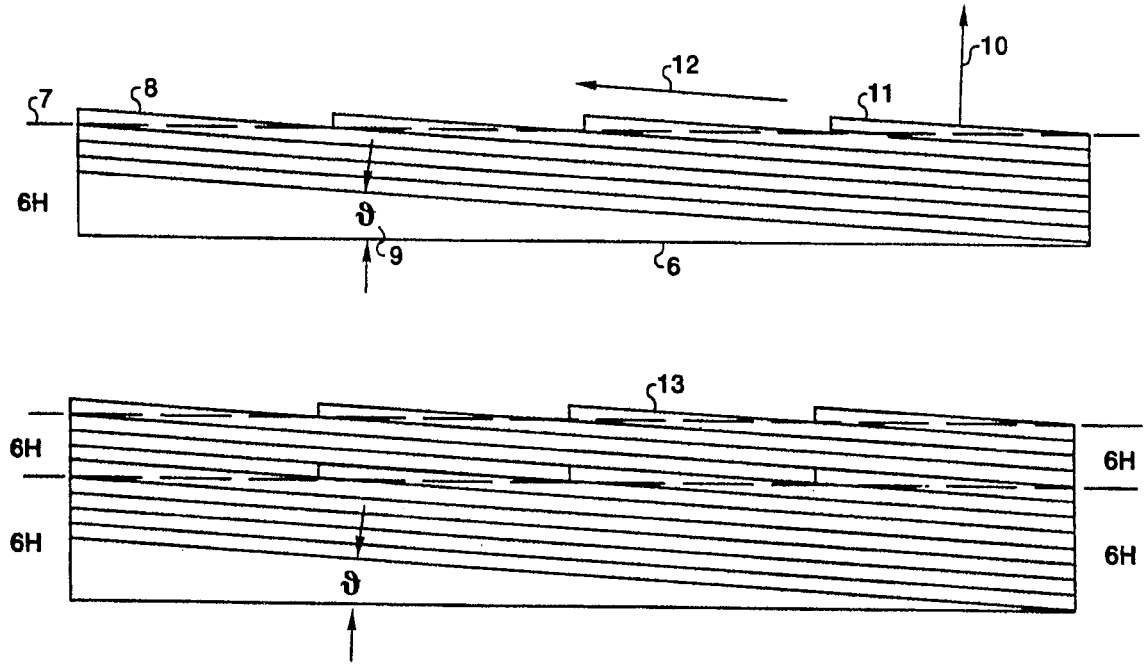
Primary Examiner—William A. Powell

Attorney, Agent, or Firm—James A. Mackin; Gene E Shook; Guy M Miller

[57] **ABSTRACT**

The invention is a method for growing homoepitaxial films of SiC on low-tilt-angle vicinal (**0001**) SiC wafers. The invention proposes and teaches a new theoretical model for the homoepitaxial growth of SiC films on (**0001**) SiC substrates. The inventive method consists of (1) preparing the growth surface of SiC wafers slightly off-axis (from less than 0.1° to 6°) from the (**0001**) plane, (2) subjecting the growth surface to a suitable etch, and then (3) growing the homoepitaxial film using conventional SiC growth techniques.

18 Claims, 2 Drawing Sheets



US005230768A

United States Patent [19]

Furukawa et al.

[11] **Patent Number: 5,230,768**

[45] **Date of Patent: Jul. 27, 1993**

[54] **METHOD FOR THE PRODUCTION OF SIC SINGLE CRYSTALS BY USING A SPECIFIC SUBSTRATE CRYSTAL ORIENTATION**

[75] Inventors: **Katsuki Furukawa**, Sakai; **Akira Suzuki**; **Yoshihisa Fujii**, both of Nara, all of Japan

[73] Assignee: **Sharp Kabushiki Kaisha**, Osaka, Japan

[21] Appl. No.: **845,500**

[22] Filed: **Feb. 28, 1992**

Related U.S. Application Data

[63] Continuation of Ser. No. 675,351, Mar. 25, 1991, abandoned.

[30] **Foreign Application Priority Data**

Mar. 26, 1990 [JP] Japan 2-77765
Mar. 30, 1990 [JP] Japan 2-87067

[51] **Int. Cl.**[5] **H61B 21/205**

[52] **U.S. Cl.** **156/612**; 148/DIG. 148; 148/33.1; 423/346; 427/249; 427/255.1; 437/93; 437/100

[58] **Field of Search** 423/345, 346; 156/612; 427/249, 255, 255.1; 437/160, 93; 148/DIG. 148, 33, 33.1

[56] **References Cited**

U.S. PATENT DOCUMENTS

T951,008	10/1976	Regin .	
3,698,944	10/1972	Dyer .	
3,821,783	6/1974	Sugita et al. .	
3,920,492	11/1975	Sugita et al .	
4,050,964	9/1977	Rode .	
4,512,825	4/1985	Addamiano et al.	156/610
4,614,672	9/1986	Addamiano	427/249
4,623,425	11/1986	Suzuki et al	156/613
4,855,254	8/1989	Eshita et al.	437/100
4,865,659	9/1989	Shigeta et al. .	
4,912,064	3/1990	Kong et al.	
5,011,549	4/1991	Kong et al. .	

OTHER PUBLICATIONS

E. A. Fitzgerald, et al., Appl. Phys Lett., vol. 52, No. 18, May 2, 1989, pp. 1496-1498.

M. Shigeta, et al., Appl. Phys. Lett., vol. 55, No. 15, Oct. 9, 1989, pp. 1522-1524.

D. Dijkkamp, et al., Appl. Phys. Lett., vol. 56, No. 1, Jan. 1, 1990, pp. 39-41.

D. E. Aspnes and J. Ihm, J. Vac. Sci. Technol. B, vol. 5, No. 4, Jul./Aug. 1987, pp. 939-944.

Appl. Physc. Lett. 42(5), 1 Mar. 1983—Nishino et al. pp. 460-462.

Appl. Phys. Lett. 50(26), 29 Jun. 1987—Shibahara et al. pp. 1888-1890.

J. Mater. Res. 3(3), May/Jun. 1988—Kong et al. pp. 521-530.

J. Appl. Phys. 66(9), 1 Nov. 1989—Oshita et al. pp. 4535-3537.

Powell et al., Appl. Phys. Lett. 51(11), 14, Sep. 1987, pp. 823-825.

Zheng et al., J. Vac. Sci. Technol. A 6(3) May/Jun. 1988, pp. 696-698.

Palmour et al., J. Appl. Phys. 64(4) 15 Aug. 1988, pp. 2168-2177.

Bean et al., J. Electrochem. Soc. 114(11) Nov. 1967, pp. 1158-1161.

Liaw et al., J. Electrochem. Soc. 132(3) Mar. 1985 pp. 642-648.

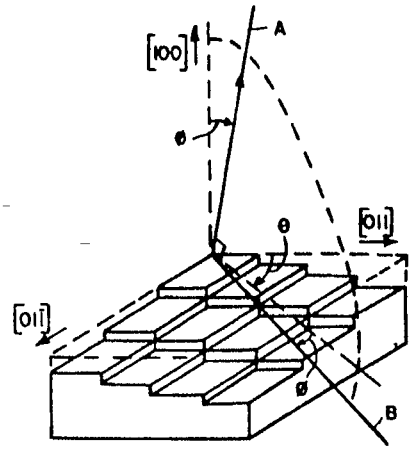
Furumura et al., J. Electrochem Soc. 135(5) May 1988 pp. 1255-1260.

Primary Examiner—Olik Chaudhuri
Assistant Examiner—Ken Horton
Attorney, Agent, or Firm—David G. Conlin; Peter F. Corless

[57] **ABSTRACT**

There is provided a method for the production of a silicon carbide single crystal, which includes the steps of: providing a silicon single-crystal substrate having a growth plane with a crystal orientation inclined from the [100] direction toward an off-direction, wherein the crystal orientation is defined by a deviation angle θ of 5 to 40 degrees, as measured from the [011] direction toward the [01$\bar{1}$] direction, and a tilt angle ϕ of 1 to 7 degrees, as measured from the [100] direction toward the off-direction; and growing a silicon carbide single crystal on the substrate.

5 Claims, 4 Drawing Sheets



United States Patent [19]

Kong et al.

[11] **Patent Number: 5,011,549**

[45] **Date of Patent: Apr. 30, 1991**

[54] **HOMOEPITAXIAL GROWTH OF ALPHA-SIC THIN FILMS AND SEMICONDUCTOR DEVICES FABRICATED THEREON**

[75] Inventors: **Hua-Shuang Kong**, Raleigh; **Jeffrey T. Glass**, Apex; **Robert F. Davis**, Raleigh, all of N.C.

[73] Assignee: **North Carolina State University**, Raleigh, N.C.

[21] Appl. No.: **422,032**

[22] Filed: **Oct. 16, 1989**

Related U.S. Application Data

[62] Division of Ser. No. 113,573, Oct. 26, 1987, Pat No. 4,912,064.

[51] **Int. Cl.**[5] **H01L 21/20**; H01L 21/203

[52] **U.S. Cl.** **148/33.1**; 148/33; 148/DIG. 148; 156/612; 156/DIG. 64; 437/105; 437/106; 427/429

[58] **Field of Search** ..... 437/100, 105, 106, 103; 148/DIG. 148, 33, 33.4, 33.1; 156/600, 610, 612, DIG. 64; 427/248.1, 249

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,520,740	7/1970	Addamiano	437/100
3,956,032	5/1976	Powell et al.	156/612
3,960,619	6/1976	Seiter	156/612
4,147,572	4/1979	Vodakov et al.	156/DIG. 64

FOREIGN PATENT DOCUMENTS

0043200	4/1979	Japan	156/DIG. 64
0035090	2/1984	Japan	156/DIG. 64
0155512	7/1987	Japan .	

OTHER PUBLICATIONS

Matsunami et al., "Epitaxial Growth of α-SiC Layers by Chemical Vapor Deposition Technique", J. Crystal Growth, 31, (1975), pp. 72–75.

Brander et al., "Solution Grown SiC p–n Junctions", Brit. J. Appl. Phys., (J. Phys. D), 1969, Ser. 2, vol. 2, pp. 309–318.

Harris et al., "Growth Characteristics of Alpha-Silicon Carbide I. Chemical Vapor Deposition", J. Electrochem. Soc , vol. 118, No. 2, Feb. 1971, pp. 335–337.

Harris et al., "Growth Characteristics of Alpha-Silicon Carbide II. Equilibrium Considerations", J. Electrochem. Soc., vol. 118, No. 2, Feb. 1971, pp. 338–340.

Wessels et al., "Epitaxial Growth of Silicon Carbide by Chemical Vapor Deposition", Int'l. Conf. on Silicon Carbide, Columbia, S.C., Univ. of S.C., 1973 (abstract).

Powell, "Silicon Carbide: Progress in Crystal Growth", Novel Refractory Semiconductors Symposium, ed. Emin, D., Aselage, T. L. and Wood, C.; Mat'l. Res. Soc., 1987, pp. 159–170.

Carter et al., "Kinetics and Mechanisms of High-Temperature Creep in Silicon Carbide: II, Chemically Vapor Deposited" J. Am. Cer. Soc., vol. 67, No. 11, Nov. 1984, pp. 732–740.

Kong et al., "Chemical Vapor Deposition and Characterization of 6H-Silicon Carbide Thin Films on Off-Axis 6H-Silicon Carbide Substrates," J. Appl. Phys., vol. 64, No. 5, 1988, pp. 2672–2679.

Wessels, B., Gotos, H. C., and Witt, A. F. "Epitaxial Growth of Silicon Carbide by Chemical Deposition," *Silicon Caride* p. 25 (1974).

Nishino, S., Matsunami, H. and Tanaka, T., "Growth and Morphology of 6H-Sic Epitaxial Layers by CVD" *J. Crystal Growth,* 144 (1978).

Muench, W. V. and I. Pfaffeneder, "Epitaxial Deposition of Silicon Carbide from Silicon Tetrachloride and Hexane," *Thin Solid Films,* 31, 39 (1976).

Powell, J. A., and Will, H. A., "Epitaxial Growth of 6H SiC in the Temperature Range 1320–1390 C", *J. Appl. Phys.*, 44, 177 (1973).

(List continued on next page.)

Primary Examiner—Olik Chaudhuri
Assistant Examiner—M. Wilczewski
Attorney, Agent, or Firm—Bell, Seltzer, Park & Gibson

[57] **ABSTRACT**

Device quality monocrystalline Alpha-SiC thin films are epitaxially grown by chemical vapor deposition on Alpha-SiC [0001] substrates prepared off axis.

9 Claims, 8 Drawing Sheets

United States Patent [19]

Kong et al.

[11] **Patent Number: 4,912,064**

[45] **Date of Patent: Mar. 27, 1990**

[54] **HOMOEPITAXIAL GROWTH OF ALPHA-SIC THIN FILMS AND SEMICONDUCTOR DEVICES FABRICATED THEREON**

[75] Inventors: **Hua-Shuang Kong**, Raleigh; **Jeffrey T. Glass**, Apex; **Robert F. Davis**, Raleigh, all of N.C.

[73] Assignee: **North Carolina State University**, Raleigh, N.C.

[21] Appl. No.: **113,573**

[22] Filed: **Oct. 26, 1987**

[51] **Int. Cl.**[4] **H01L 21/20**; H01L 21/203

[52] **U.S. Cl.** **437/100**; 156/612; 156/DIG. 64; 148/DIG. 148; 437/105; 437/106; 427/429

[58] **Field of Search** 437/100, 105, 106, 103; 148/DIG. 148; 156/DIG. 64, 610, 612, 600; 427/248.1, 249

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,520,740	7/1970	Addamiano	437/100
3,956,032	5/1976	Powell et al.	156/612
3,960,619	6/1976	Seiter	156/612
4,147,572	4/1979	Vodakov et al	156/DIG. 64

FOREIGN PATENT DOCUMENTS

0043200	4/1979	Japan	156/DIG. 64
0035099	2/1984	Japan	156/DIG. 64
0155512	7/1987	Japan .	

OTHER PUBLICATIONS

Matsunami et al., "Epitaxial Growth of α-SiC Layers by Chemical Vapor Deposition Technique", J. of Crystal Growth 31 (1975), pp. 72–75.

Brander et al., "Solution Grown SiC p–n Junctions", Brit. J. Appl. Phys. (J. Phys. D), 1969, Ser. 2, vol. 2, pp. 309–318.

Carter et al., "Kinetics and Mechanisms of High-Temperature Creep in Silicon Carbide: II, Chemically Vapor Deposited", J. Am. Cer. Soc., vol. 67, No. 11, Nov. 1984, pp. 732–740

Kong et al., "Chemical Vapor Deposition and Characterization of 6H–Silicon Carbide Thin Film on Off Axis 6H–Silicon Carbide Substrates", J. Appl. Phys., vol. 64, No. 5, 1988, pp. 2672–2679.

Harris et al., "Growth Characteristics of Alpha–Silicon Carbide I, Chemical Vapor Deposition", J. Electrochem. Soc., vol. 118, No. 2, Feb. 1971, pp. 335–337.

Harris et al., "Growth Characteristics of Alpha–Silicon Carbide II, Equilibrium Considerations", J. Electrochem. Soc , vol 118, No. 2, Feb. 1971, pp. 338–340.

Wessels et al., "Epitaxial Growth of Silicon Carbide by Chemical Vapor Deposition", Int'l. Conf. on Silicon Carbide, Columbia, S.C., Univ. of S.C., 1973 (Abstract).

Powell, "Silicon Carbide: Progress in Crystal Growth", Novel Refractory Semiconductors Symposium, Ed. Emin., D ; Aselage, T. L.; Wood, C., Mat'l. Res. Soc., 1987, pp. 159–170.

Wessels, B., Gatos, H. C., and Witt, A. F. "Epitaxial Growth of Silicon Carbide by Chemical Deposition," *Silicon Carbide*, p. 25 (1974).

Nishino, S., Matsunami, H. and Tanaka, T., "Growth and Morphology of 6H–SiC Epitaxial Layers by CVD" J. *Crystal Growth*, 144 (1978).

Muench, W. V. and I. Pfaffeneder, "Epitaxial Deposition of Silicon Carbide from Silicon Tetrachloride and Hexane," *Thin Solid Films*, 31, 39 (1976).

Powell, J. A , and Will, H. A., "Epitaxial Growth of 6H SiC in the Temperature Range 1320–1390 C", *J. Appl. Phys.*, 44, 177 (1973).

Yoshida, S., E. Sakuma, S. Misawa and S. Gonda, "A New Doping Method Using Metalorganics in Chemical Vapor Deposition of 6H–SiC," *J. Appl. Phys.*, 55 (1), 169 (1984).

Minagwa, S. and H. C. Gatos, "Epitaxial Growth of a–SiC From the Vapor Phase", *Japanese J. of Applied Phys.*, 10 (12), 1680 (1971).

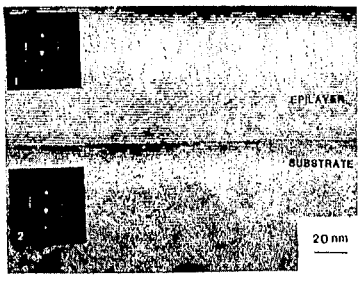
(List continued on next page.)

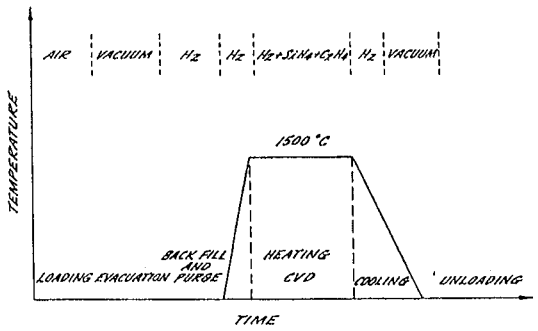
Primary Examiner—Brian E. Hearn
Assistant Examiner—Mary Wilczewski
Attorney, Agent, or Firm—Bell, Seltzer, Park & Gibson

[57] **ABSTRACT**

Device quality monocrystalline Alpha-SiC thin films are epitaxially grown by chemical vapor deposition on Alpha–SiC [0001] substrates prepared off axis.

21 Claims, 8 Drawing Sheets





United States Patent [19]

Davis et al.

[11] **Patent Number: 4,912,063**

[45] **Date of Patent: Mar. 27, 1990**

[54] **GROWTH OF BETA-SIC THIN FILMS AND SEMICONDUCTOR DEVICES FABRICATED THEREON**

[75] Inventors: **Robert F. Davis; Hua-Shuang Kong,** both of Raleigh; **Jeffrey T. Glass,** Apex; **Calvin H. Carter, Jr.,** Raleigh, all of N.C.

[73] Assignee: **North Carolina State University,** Raleigh, N.C.

[21] Appl. No.: **113,921**

[22] Filed: **Oct. 26, 1987**

[51] **Int. Cl.**[4] **H01L 21/20**

[52] **U.S. Cl.** **437/100;** 437/105; 437/106; 148/DIG. 148; 156/610; 156/DIG. 64

[58] **Field of Search** ..... 437/100, 105, 106, 949, 437/970; 148/DIG. 148; 156/610, DIG. 64

[56] **References Cited**

U.S. PATENT DOCUMENTS

2,854,364	9/1958	Lely	437/100

FOREIGN PATENT DOCUMENTS

2159592	6/1973	France	437/100
2030699	2/1987	Japan	437/100
1031783	6/1966	United Kingdom	437/100
1039748	8/1966	United Kingdom	437/100
1054519	1/1967	United Kingdom	437/100

OTHER PUBLICATIONS

Kong et al., "Epitaxial Growth of β-SiC Thin Films on 6H α-SiC Substrates via Chemical Vapor Deposition", Appl. Phys. Lett., vol. 49, No. 17, Oct. 27, 1986, pp. 1074–1076.

Kuroda et al., "Step-Controlled VPE Growth of SiC Single Crystals at Low Temperatures", Extended Abstracts of the 19th Conference on Solid State Devices and Materials", Tokyo, Aug 25–27, 1987, pp. 227–230.

Shibahara et al., "Surface Morphology of Cubic SiC(100) Grown on Si(100) by Chemical Vapor Deposition", J. of Crystal Growth, vol. 78, 1986, pp. 538–544.

Powell et al., "Improved β-SiC Heteroepitaxial Films using Off-Axis Si Substrates", Appl. Phys. Lett., vol. 51, No. 11, Sep. 14, 1987, pp. 823–825.

Saidov et al., "Current-voltage Characteristics of Silicon Carbide Heterojunctions", Sov. Phys. Semicond., vol. 13, No. 9, Sep. 1979, pp. 1054–1056.

Pilyankevich et al., "Change in Real Structure During the B→α SiC Conversion", Izv. Akad. Nauk. SSSR Neorg. Mater., vol. 19, No. 10, Oct. 1983, pp. 1557–1559.

Kong et al., "The Effect of Off-Axis Si(100) Substrates on the Defect Structure and Electrical Properties of β-SiC Thin Films", J. Mater. Res., vol. 3, No. 3, May/-Jun. 1988, pp. 521–530.

Kong et al., "Growth Rate, Surface Morphology and Defect Microstructures of β-SiC Films Chemically Vapor Deposited on 6-H-SiC Substrates", J. Mater. Res., vol. 4, No. 1, Jan./Feb. 1989, pp. 204–214.

Furumura et al., "Characteristics of β-SiC Films Grown from an $SiHCl_3$–C_3H_8–H_2 System", Electronics and Communications in Japan, Part 2, vol. 70, No. 5, 1987, pp. 53–63.

Palmour et al., "High Temperature Depletion-Mode Metal-Oxide-Semiconductor Field-Effect Transistors in Beta-SiC Thin Films", Appl. Phys. Lett., vol. 51, No. 24. Dec. 14, 1987, pp. 2028–2030.

Shibahara et al., "Antiphase-Domain-Free Growth of Cubic SiC on Si(100)", Appl. Phys. Lett., vol. 50, No. 26, Jun. 29, 1987, pp. 1888–1890.

Palmour et al., "Characterization of Device Parameters in High-Temperature Metal-Oxide-Semiconductor Field-Effect Transistors in β-SiC Films", J. Appl Phys., vol. 64, No. 4, Aug. 15, 1988, pp. 2168–2177.

Zheng et al., "Scanning Tunneling Microscopy of Cubic Silicon Carbide Surfaces", J. Vac. Sci. Technol. A, vol. 6, No. 3, May/Jun. 1988, pp. 696–698.

(List continued on next page.)

Primary Examiner—Brian E Hearn
Assistant Examiner—M. Wilczewski
Attorney, Agent, or Firm—Bell, Seltzer, Park & Gibson

[57] **ABSTRACT**

Device quality thin films of Beta-SiC are epitaxially grown on substrates of Alpha-Sic.

14 Claims, 6 Drawing Sheets

United States Patent [19]

Lundberg

[11] **4,349,407**

[45] **Sep. 14, 1982**

[54] **METHOD OF FORMING SINGLE CRYSTALS OF BETA SILICON CARBIDE USING LIQUID LITHIUM AS A SOLVENT**

[75] Inventor: **Lynn B. Lundberg**, Los Alamos, N. Mex

[73] Assignee: **The United States of America as represented by the United States Department of Energy**, Washington, D.C.

[21] Appl. No.: **37,247**

[22] Filed: **May 9, 1979**

[51] **Int. Cl.**[3] **C30B 9/10**

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[56] **References Cited**

U.S. PATENT DOCUMENTS

2,996,456	8/1961	Hergenrother	156/624
3,205,101	9/1965	Mlavsky	252/62.3 C
3,278,274	10/1966	Liebmann et al.	156/624
3,669,763	6/1972	Perusek	252/62.3 C
3,956,032	5/1976	Powell et al.	252/62.3 L
4,032,371	6/1977	Andersen	252/62 3 C

OTHER PUBLICATIONS

Pellegrini et al., Silicon Carbide-1973 Ed. Marshall, pp. 161-167, U.S.C. Press, Columbia S.C. 1974.

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[57] **ABSTRACT**

A method of growing single crystals of beta SiC from solution using molten lithium as a solvent for polycrystalline SiC feed material. Reasonable growth rates are accomplished at temperatures in the range of about 1330° C. to about 1500° C.

4 Claims, No Drawings