US006562130B2

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

(10) **Patent No.: US 6,562,130 B2**
(45) **Date of Patent: *May 13, 2003**

(54) **LOW DEFECT AXIALLY GROWN SINGLE CRYSTAL SILICON CARBIDE**

(75) Inventors: **Yury Alexandrovich Vodakov**, St. Petersburg (RU); **Evgeny Nikolaevich Mokhov**, St Petersburg (RU); **Mark Grigorievich Ramm**, Rego Park, NY (US); **Alexandr Dmitrievich Roenkov**, St. Petersburg (RU); **Yury Nikolaevich Makarov**, Richmond, VA (US); **Sergei Yurievich Karpov**, St. Petersburg (RU); **Mark Spiridonovich Ramm**, St Petersburg (RU); **Leonid Iosifovich Temkin**, St. Petersburg (RU)

(73) Assignee: **The Fox Group, Inc.**, Livermore, CA (US)

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

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **09/849,615**

(22) Filed: **May 4, 2001**

(65) **Prior Publication Data**

US 2002/0059901 A1 May 23, 2002

Related U.S. Application Data

(63) Continuation-in-part of application No. 09/355,561, filed as application No. PCT/RU97/00005 on Jan. 22, 1997, now Pat. No. 6,261,363.

(51) **Int. Cl.**[7] **C30B 23/06**

(52) **U.S. Cl.** **117/109**; 117/107; 117/200; 117/201; 117/900; 423/345

(58) **Field of Search** 117/109, 107, 117/200; 423/345

(56) **References Cited**

U.S. PATENT DOCUMENTS

2,854,364 A 9/1958 Lely

4,125,756 A 11/1978 Hierholzer, Jr. et al. 219/121

(List continued on next page.)

FOREIGN PATENT DOCUMENTS

DE	3230727	2/1984
JP	59-35099	2/1984

(List continued on next page.)

OTHER PUBLICATIONS

Hofmann et al., "Use of Ta container material for quality improvement of SiC", Institute of Physics Confernece Series vol. 142, pp. 29–32, 1996 abstract only.*

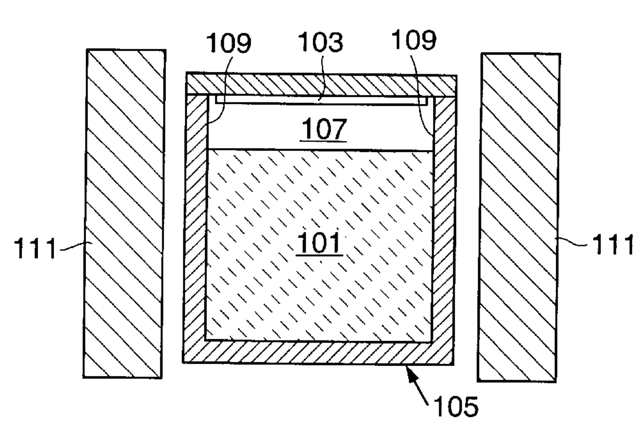
(List continued on next page.)

Primary Examiner—Robert Kunemund
(74) *Attorney, Agent, or Firm*—Bingham, McCutchen, LLP; David G. Beck

(57) **ABSTRACT**

A method and apparatus for axially growing single crystal silicon carbide is provided. Utilizing the system, silicon carbide can be grown with a dislocation density of less than 10^4 per square centimeter, a micropipe density of less than 10 per square centimeter, and a secondary phase inclusion density of less than 10 per cubic centimeter. As disclosed, a SiC source and a SiC seed crystal of the desired polytype are co-located within a crucible, the growth zone being defined by the substantially parallel surfaces of the source and the seed in combination with the sidewalls of the crucible. Prior to reaching the growth temperature, the crucible is evacuated and sealed, either directly or through the use of a secondary container housing the crucible. The crucible is comprised of tantalum or niobium that has been specially treated. As a result of the treatment, the inner surfaces of the crucible exhibit a depth variable composition of Ta—Si—C or Nb—Si—C that is no longer capable of absorbing SiC vapors, thus allowing the vapor-phase composition within the crucible to be close to the SiC—Si system with the partial pressure of Si-vapor slightly higher than that in the SiC—Si system.

26 Claims, 1 Drawing Sheet



U.S. PATENT DOCUMENTS

4,147,572	A		4/1979	Vodakov et al.	148/175
4,302,508	A		11/1981	Hierholzer, Jr. et al.	428/367
4,556,436	A		12/1985	Addamiano	148/175
4,866,005	A		9/1989	Davis et al.	437/100
5,433,167	A		7/1995	Furukawa et al.	117/84
5,964,944	A	*	10/1999	Sugiyama et al.	117/102
6,261,363	B1	*	7/2001	Vodakov et al.	117/104

FOREIGN PATENT DOCUMENTS

JP	11-116398	*	4/1999
JP	11-116399	*	4/1999
RU	403275		7/1973
RU	882247		7/1980
WO	2001-004389	*	10/2001

OTHER PUBLICATIONS

Hoffmann, et al., "Use of Ta Container Material for Quality Improvement of SiC Crystals Grown by Sublimation Technique", Int'l Conf. on Silicon Carbide and Related Materials (1995) pp. 1–4.

E.N. Mokhov, et al., "Crowing of Epitaxial Layers of Silicon Carbide", Vysokochistie veshchesta, No. 3, 1992, pp. 98–105.

S. Yu, et al., "Excess Phase Formation During Sublimation Growth of Silicon Carbide", Int'l Conf. on Silicon Carbide and Related Materials (1995), pp. 1–4.

Yu V. Koritsky, et al., "Electrochemical Materials Manual", (1988), p. 449.

E.N. Mokhov, et al. "Growth of Silicon Carbide Bulk Crystals By the Sublimation Sandwich Method", Materials Science and Engineering B46 (Jun. 4, 1997), pp. 317–323.

E.N. Mokhov, et al., "SiC Growth in Tantalum Containers by Sublimation Sandwich Method", Journal of Crystal Growth, 181 (Apr., 1997) pp. 254–258.

S. Yu Karpov, et al., "Analysis of Sublimation Growth of Bulk SiC Crystals in Tantalum Container", Journal of Crystal Growth, 211 (2000) pp. 347–351.

* cited by examiner

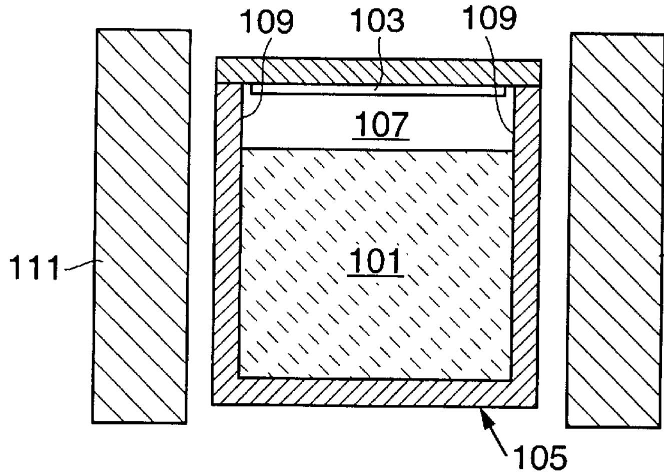


FIG. 1

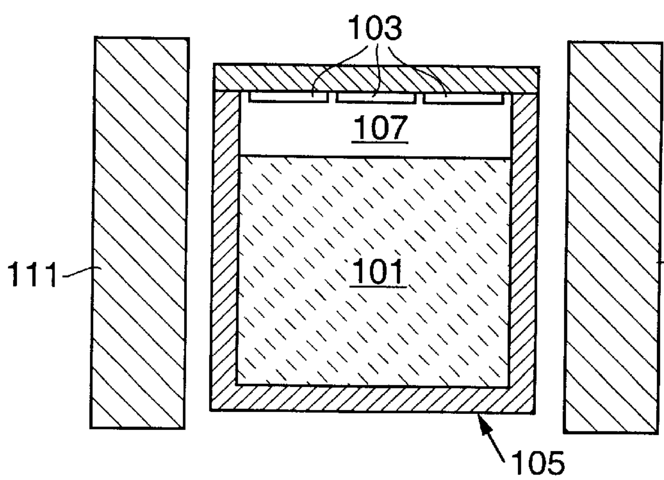


FIG. 2

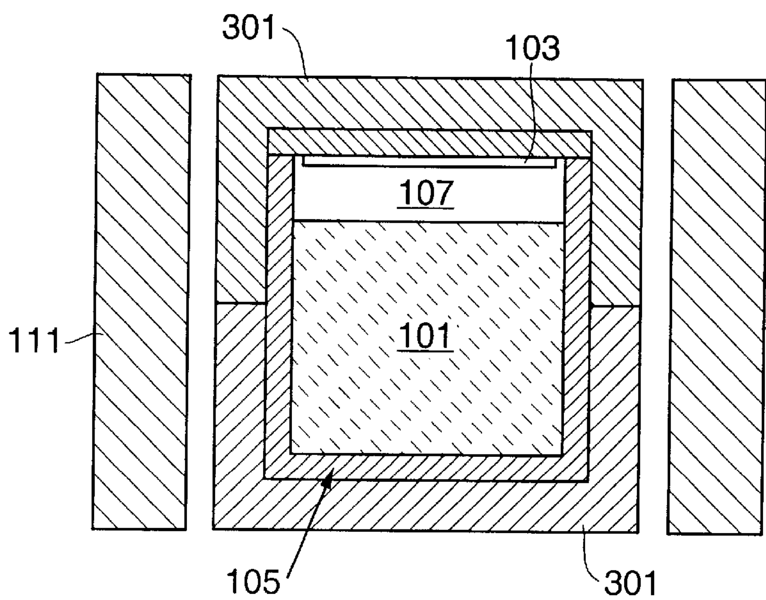


FIG. 3

LOW DEFECT AXIALLY GROWN SINGLE CRYSTAL SILICON CARBIDE

CROSS-REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of U.S. patent application Ser. No. 09/355,561, filed Jul. 20, 1999 now U.S. Pat. No. 6,261,363, which is a 371, and claims priority from PCT Application Serial No. PCT/RU97/00005, filed Jan. 22, 1997.

FIELD OF THE INVENTION

The present invention relates generally to the generation of monocrystalline materials and, more particularly, to a method and apparatus for growing monocrystalline silicon carbide.

BACKGROUND OF THE INVENTION

Silicon carbide (SiC) is wide-band-gap semiconductor material that has a number of characteristics that make it an ideal candidate for a variety of semiconductor applications including, but not limited to, light sources, power diodes, field-effect transistors, and photodiodes. The ability to realize the benefits offered by SiC is largely controlled by the purity of the material as well as its structural characteristics.

The methods most commonly used in producing SiC single crystals are sublimation techniques based on the Lely method, this method utilizing vapor-phase crystallization of evaporated solid silicon carbide. (See, for example, U.S. Pat. Ser. Nos. 2,854,364 and 4,866,005). As shown by Karpov et al. in an article entitled Excess Phase Formation During Sublimation Growth of Silicon Carbide, 6th Int. Conf. on Silicon Carbide, Kyoto, Japan, 74–75 (September 1995), in order to achieve SiC monocrystalline growth from vapor without forming secondary-phase inclusions, the external silicon (Si) flux on the growing surface must exceed the carbon (C) flux. The ability to achieve the desired excess silicon flux depends on the temperature of the growing surface and, in the case of sublimation techniques, the composition of the vapor adjacent to the growth surface.

As silicon molecules have the maximum concentration in the gaseous phase, any drift of the substance from the growth zone will result in the vapor phase within the growth zone being depleted of silicon and enriched with carbon. Excessive carbon in the growth zone leads to source graphitization, crystal quality degradation, and eventually the discontinuation of the growth process. On the other hand, excessive silicon in the growth zone may result both in the formation of defects on the growing surface of the SiC crystal, primarily due to the precipitation of excess silicon drops, and in the generation of polytypes differing from the seed polytype. Accordingly, it has been established that the best characteristics for the as-grown SiC single crystal are achieved when the vapor composition in the growth zone is shifted the towards the vapor phase corresponding to the SiC—Si system.

U.S. Pat. Ser. No. 2,854,364 discloses locating SiC powder with a mass of more than three times the mass of the single crystal to be grown in the growth zone in order to maintain a relatively constant vapor phase composition, the powder serving as a source of silicon carbide vapors. As disclosed, the drift of SiC vapors into the space outside the growth zone is balanced by the generation of SiC vapors from the SiC powder. The duration of the stable growth process is limited by the quantity of SiC powder located in the growth zone. Once the source of SiC vapors becomes depleted, the vapor composition shifts to the non-advantageous SiC—C system.

In U.S. Pat. Ser. No. 4,866,005 a technique is disclosed that continuously feeds small portions of SiC powder into a temperature zone of the growth chamber. Although this technique does allow a SiC—Si system to be maintained indefinitely, it is an inefficient process due to the SiC material consumed in addition to the SiC source as well as the growth zone geometry. As disclosed, the evaporating surface of the SiC vapor source is approximately 10 centimeters from the growing surface of the seed crystal, a distance that far exceeds the Si, Si_2C, SiC_2 molecular track length at the working pressure in the growth zone.

U.S. Pat. Ser. No. 4,147,572 discloses a growth technique in which the evaporating surface of the SiC source and the growth surface of the SiC seed crystal are arranged in parallel at a distance that is less than 20 percent of the maximum linear dimension of the source. The single crystals are grown in a graphite crucible in an inert gas atmosphere at temperatures of 1600 to 2000° C. with an axial thermal gradient of 5 to 200° C. per centimeter. This technique is limited to relatively small crystals, typically less than 1 millimeter thick, due to a sharp drop in the growth rate as the crystallization time increases. The change in growth rate is due to the silicon at the edge of the growth zone being volatilized, thereby causing excessive carbon to be released from the evaporating surface of the SiC source and the growing surface of the grown crystal. Single crystals obtained by this technique show defects such as secondary-phase inclusions (predominantly, graphite), micropipes with a density of more than 100 per square centimeter, and dislocations of at least 10^4 per square centimeter. These crystals also have relatively high concentrations of residual impurities such as boron, oxygen, etc.

In an article by D. Hofmann et al. entitled The Use of Tantalum Container Material for Quality Improvement of SiC Crystals Grown by the Sublimation Technique, 6th Int. Conf. on Silicon Carbide, Kyoto, Japan, 15 (September 1995), it was shown that the inclusion of tantalum (Ta) during the sublimation growth of monocrystalline SiC resulted in the vapor medium produced in the growth zone being close to the SiC—Si system. The favorable aspects were found to occur both in an inert gas atmosphere and in vacuum. Unfortunately it was also found that during the early stages of growth, secondary-phase inclusions of tantalum or its compounds were formed. An increased concentration of dissolved tantalum in the monocrystalline SiC was also noted. Lastly, due to the carbon enrichment of the vapor phase that results from silicon drifting outside of the growth zone, carbon dust was embedded into the growing crystal, further reducing the quality of the growing crystal while simultaneously decreasing the transferal efficiency of source material to the growing crystal.

Another problem associated with the use of a Ta container as disclosed in the previously cited article arises during the initial stage of the growth process when the silicon vapors formed by the evaporating SiC source interact with the material of the tantalum container. As a result of this interaction, a low-melting-point tantalum silicon alloy is formed which can lead to the destruction of the container at the normal growth temperature.

In known sublimation techniques for growing SiC single crystals, the vapor source may be either a pre-synthesized SiC powder of the specified dispersity or a polycrystalline or monocrystalline SiC wafer produced, for example, by the

Lely method. Although the use of SiC powder is more economical than the use of wafers, providing a continuous supply of powder into the growth zone, as required to grow large single crystals, is quite complicated. Additionally, SiC powder often includes impurities such as graphite or other dust that are transported to the growth surface along with the SiC molecules. These impurities lead to a high density of micropipes and dislocations in the growing crystal, thus substantially impacting the crystal quality.

Accordingly, what is needed in the art is a method and system that allows high quality SiC single crystals to be grown. The present invention provides such a method and system.

SUMMARY OF THE INVENTION

The present invention provides a method and apparatus for growing low dislocation density single crystal silicon carbide. Utilizing the system of the invention, silicon carbide can be grown with a dislocation density of less than 10^4 per square centimeter, preferably less than 10^3 per square centimeter, and more preferably less than 10^2 per square centimeter. The density of micropipes in the as-grown material is less than 10 per square centimeter. The density of secondary phase inclusions is less than 10 per cubic centimeter and preferably less than 1 per cubic centimeter. Depending upon the construction of the crucible, the concentration of tantalum or niobium impurities is less than 10^{17} per cubic centimeter.

In accordance with the invention, a SiC source and a SiC seed crystal of the desired polytype are co-located within a crucible with the distance separating the source evaporating surface from the growing surface being comparable to the track length of a SiC molecule. The growth zone is defined by the substantially parallel surfaces of the source and the seed in combination with the sidewalls of the crucible. Prior to reaching the growth temperature, the crucible is evacuated and sealed, either directly or through the use of a secondary container housing the crucible.

In further accordance with the invention, the crucible is comprised of tantalum or niobium that has been specially treated. As a result of the treatment, the inner surfaces of the crucible exhibit a depth variable composition of Ta—Si—C or Nb—Si—C that is no longer capable of absorbing SiC vapors as the monocrystalline silicon carbide is grown. Accordingly, during crystal growth the vapor-phase composition within the crucible is close to the SiC—Si system with the partial pressure of Si-vapor slightly higher than that in the SiC—Si system. Additionally, the resultant Ta—Si—C or Nb—Si—C material is refractory, thus allowing it to withstand the operating temperatures required to grow the silicon carbide.

The crucible is initially fabricated from tantalum or niobium that is preferably at least 99.9 percent pure. Once the crucible is shaped, it undergoes a series of processing steps to clean the surfaces and remove surface contaminants. A thin, near-surface layer of Ta—C or Nb—C is then formed and annealed, resulting in a surface that will not interact with carbon particles. Lastly the crucible is annealed in silicon containing vapor that is diluted by an inert gas, preferably argon, resulting in the formation of a depth variable composition of Ta—Si—C or Nb—Si—C on the crucible surfaces.

A further understanding of the nature and advantages of the present invention may be realized by reference to the remaining portions of the specification and the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a cross-sectional view of a crucible according to the invention;

FIG. **2** is a cross-sectional view of a crucible similar to that shown in FIG. **1** with multiple silicon carbide seeds contained therein; and

FIG. **3** is a cross-sectional view of a crucible similar to that shown in FIG. **1** with a separate, sealable container.

DESCRIPTION OF THE SPECIFIC EMBODIMENTS

Growth Process

According to the invention and as illustrated in FIG. **1**, a silicon carbide (SiC) source **101** and a SiC seed crystal **103** of the desired polytype (e.g., 4H, 6H, 3C, etc.) are co-located within a crucible **105**. An axial growth zone **107** is defined by the substantially parallel surfaces of source **101** and seed **103** in combination with sidewalls **109** of crucible **105**. If multiple seed crystals **103** are used as illustrated in FIG. **2**, their growth surfaces are located within the same plane and parallel to the evaporating surface of source **101**.

The distance between the evaporating surface of source **101** and the growing surface of seed crystal **103** is preferably not much in excess of the track length of a SiC molecule. This configuration enhances the crystal growth rate as the precipitation of source vapors outside of the seed crystal growth surface is minimized.

In the preferred embodiment, source **101** is comprised of SiC ceramics that are produced from SiC powder that has been sintered at a temperature that permits partial over-sublimation of the SiC grains. The sintering process is preferably carried out in an inert gas environment (e.g., argon) within a temperature range of 1500 to 2300° C. In addition to achieving partial binding of the powder, during the sintering process basic background impurities and dust are removed from the powder, thus preventing the dust composition from being transferred from the evaporating surface of source **101** in the vapor phase. The SiC ceramics used for source **101** can also be fabricated by compressing SiC powder.

Additionally, during the fabrication of the SiC ceramics, a doping agent can be deliberately introduced. By using SiC ceramics in which the dopant has been uniformly distributed throughout the entire volume, a uniformly doped single crystal can be grown.

In another embodiment of the invention, a SiC polycrystal or mono-crystal source is used.

As shown in FIG. **1**, in the preferred embodiment SiC source **101** is located on the bottom of crucible **105** and seed crystal **103** is mounted to the top surface of the crucible. Alternately, source **101** can be mounted on the top surface of the crucible with seed crystal **103** mounted to the crucible's bottom surface. In order to prevent the loss of SiC source material due to precipitation of SiC vapors outside the growth surface, preferably the inner dimensions of crucible **105** in general, and crucible sidewalls **109** in particular, do not exceed the dimensions of growth zone **107**. Accordingly, in this embodiment of the invention, sidewalls **109** define the periphery of growth zone **107**. If the inner dimensions of sidewalls **109** do exceed the dimensions of growth zone **107**, preferably it is by a minor amount.

As described in further detail below, crucible **105** is comprised of tantalum (Ta) or niobium (Nb) that has been specially treated. It has been established by the inventors that as a result of such treatment, the crucible exhibits a depth-variable composition of Ta—Si—C or Nb—Si—C that is no longer capable of absorbing SiC vapors as the monocrystalline silicon carbide is grown. Consequently, during crystal growth the vapor-phase composition within crucible **105** is close to the SiC—Si system with the partial pressure of Si-vapor slightly higher than that in the SiC—Si system. Additionally, the resultant variable composition Ta—Si—C or Nb—Si—C is a refractory material that can withstand the operating temperatures required to grow the SiC single crystal.

Preferably crucible **105** is capable of being evacuated and sealed, either directly or indirectly through the use of an external container **301** as illustrated in FIG. **3**. In one embodiment of the invention, after crucible **105** is loaded with source **101** and seed **103**, it is placed within a high temperature oven **111**. Oven **111** provides an axial temperature gradient from seed **103** to source **101**, resulting in the evaporation of the SiC of source **101** and vapor phase crystallization of SiC on the growing surface of seed **103**. In this embodiment crucible **105** (or container **301**) is sealed before the final operating temperature is reached, sealing being accomplished using any of a variety of sealing systems (e.g., vacuum welding, graphite or other based sealants, etc.). In an alternate embodiment, crucible **105** (or container **301**) is evacuated and hermetically sealed prior to placement within high temperature furnace **111**.

If the crystal growing process is run for an extended time period, for example as required to grow an exceptionally large crystal, the gradually increasing thickness of the grown crystal is accompanied by a corresponding decrease in the thickness of source **101**. Accordingly, in order to maintain the growth process, a large source must be used, for example, a source rod of SiC that can be continuously fed into growth zone **107**.

In the illustrated embodiment, furnace **111** provides the required thermal gradient, either through the use of multiple temperature zones (e.g., one zone for source **101** and one zone for seed crystal **103**) or other means. During crystal growth, a stable temperature profile must be maintained throughout the entire growth period. Preferably this is achieved by altering the relative positions of crucible **105** and furnace **111**, for example by moving crucible **105** within furnace **111** at a rate that is substantially equivalent to the growth rate.

As previously disclosed, preferably growth zone **107** is evacuated and sealed prior to initiation of the sublimation process. As a result, material losses from source **101** are substantially reduced. Additionally, sealing crucible **105**, either directly or through the use of separate container **301**, prevents foreign impurities from the environment from entering into growth zone **107**.

By isolating the growth zone from the environment and using a crucible exhibiting a depth-variable composition of Ta—Si—C or Nb—Si—C, the vapor phase in the growth zone shifts from the SiC—C system to the SiC—Si system. Furthermore, as the depth-variable composition of Ta—Si—C or Nb—Si—C remains relatively unchanged for an extended period of time, a stable composition of the vapor phase within growth zone **107** can be achieved, thereby resulting in improvements in both crystal quality and size. For example, the present invention allows SiC single crystals to be grown with a dislocation density of less than 10^4 per square centimeter, preferably less than 10^3 per square centimeter, and more preferably less than 10^2 per square centimeter. The density of micropipes in the as-grown material is less than 10^2 per square centimeter. The density of secondary phase inclusions is less than 10 per cubic centimeter and preferably less than 1 per cubic centimeter. Depending upon the construction of the crucible, the concentration of tantalum or niobium impurities is less than 10^{17} per cubic centimeter and typically in the range of 10^{16} to 10^{17} per cubic centimeter.

In order to grow a SiC crystal of the 4H polytype, a 4H polytype single crystal is used as seed **103** and the growth process is carried out in the presence of tin vapors that have been introduced into the crucible prior to its sealing.

Crucible Preparation

As previously disclosed, crucible **105** is preferably fabricated from tantalum or niobium, thereby allowing the vapor composition during growth to remain close to equilibrium thus reducing crystal graphitization. The tantalum or niobium must be pre-treated, however, to prevent the metals from interacting with the Si—C vapor and forming low-temperature eutectics. The pre-treatment consists of two stages. First, tantalum or niobium carbide layers are formed on the surface. During SiC crystal growth, binding of carbon vapor leads to deeper diffusion of carbon atoms into the pure metal. Second, tantalum or niobium silicide surface layers are formed, the existence of which increases silicon containing species partial pressure.

Initially crucible **105** is fabricated from metallic tantalum or niobium, the metal being at least 99.9 percent pure and of any suitable shape (e.g., rod, rolled, etc.). It is understood that the shape of crucible **105** is not limited to the shape shown in FIGS. **1–3**. Once the crucible has been shaped, for example using standard machining processes, it is initially cleaned with standard organic solvents. The crucible is then boiled for 30 minutes in a pre-heated acid solution comprised of a 3:1 mixture of HCl and HNO_3, the acid solution removing metallic remnants left on the surface after crucible shaping. The crucible is then etched in a room temperature 1:1 mixture of HNO_3 and HF for approximately 20 to 30 seconds. This etching step must be short to insure that the etchant does not damage the surface finish quality of the crucible. After etching, the crucible is washed in distilled or deionized boiling water for 10 minutes, the water being changed three times during the process. Lastly, the crucible is dried.

After the crucible has been fabricated and the surfaces cleaned, preferably following the above-described process, the crucible is processed in carbon containing vapor in order to form a thin, near-surface layer of Ta—C or Nb—C. After further temperature processing, this layer protects the crucible's surfaces from interaction with carbon particles.

During carbon vapor processing, the crucible is placed within a furnace and annealed at a pressure of at least 10^{-3} Torr in graphite that is at least 99.99 percent pure. Alternately, the crucible is annealed at a pressure of at least 10^{-1} Torr in vapor that contains carbon species. Preferably a step-wise annealing process is used, such that the crucible is annealed at 800° ±50° C. for one hour, followed by a 1500° ±50° C. anneal for one hour, and ending with a 2000° ±50° C. anneal for two hours. As a result of this annealing process, a thin near-surface layer comprised of Ta—C or Nb—C carbides is formed on the entire surface of the crucible. The depth of carbon penetration for crucibles in which the material is prepared by vacuum melting or metal rolling is approximately 5 to 30 microns while the penetration depth for crucibles formed of materials prepared by powder metallurgy is approximately 100 to 500 microns, the greater penetration depth due to accelerated diffusion along grain boundaries. Quality of the layer as well as the boundary between the metal and the carbide is governed by the technique used to fabricate the metal (e.g., powder metallurgy, rolled metal, vacuum melting, etc.).

After formation of the carbide layer, the crucible is subjected to further temperature processing, thus assuring that the layer protects the surface of the crucible from interaction with carbon particles. During this processing step the crucible is placed in graphite powder in an argon atmosphere, the graphite powder being at least 99.99 percent pure with a grain size of less than 100 microns and the argon being at least 99.999 percent pure. If the crucible is formed of tantalum it is annealed at a temperature of between 2200 and 2500° C. If the crucible is formed of niobium it is annealed at a temperature of between 2000 and 2300° C. The annealing time for either material is at least 2 hours, the final annealing time being governed by the thickness of the crucible.

Lastly the crucible is annealed for at least 2 hours in silicon containing vapor (i.e., SiC or Si vapor) that is diluted by purified argon. The annealing temperature for a crucible formed of tantalum is between 2200 and 2500° C. while the annealing temperature for a crucible formed of niobium is between 2000 and 2300° C. During annealing, the source of Si-vapor should not come into contact with the crucible. Thus, for example, SiC powder is located in a higher temperature zone than the crucible with a temperature difference of between 10 and 20° C.

After completion of each of the annealing steps outlined above, the crucible is cooled to room temperature at a cooling rate less than 20° C. per minute. Additionally, during each annealing step the temperature variation across the surface of the crucible should be less than 20° C. After all stages of annealing are complete, the amount of carbon that has penetrated into the tantalum or niobium crucible surfaces should be more than 0.02 grams per square centimeter while the amount of silicon that has penetrated into the tantalum or niobium crucible surfaces should be more than 0.0005 grams per square centimeter.

EXAMPLES

SiC crystals have been grown using the disclosed processes, examples of which are provided below. A pretreated tantalum crucible was used. During growth, the evaporating surface of source **101** was separated from the growing surface of seed **103** by approximately 3 to 18 millimeters. An operating temperature of between 1800–2500° C. was used with an axial temperature gradient of between 20 and 25 degrees per centimeter, yielding a growth rate of between 0.7 and 1.2 millimeters per hour.

Seed crystal polytypes included 6H SiC growing in direction [0001] Si; 4H SiC growing in direction [0001] C.; and 6H SiC growing in a direction lying at an angle of 5 degrees to direction [0001].

The as-grown single crystals were approximately 10 millimeters thick with diameters ranging between 20 and 75 millimeters. The density of dislocations was in the range of 10^2 and 10^4 per square centimeter, the density being dependent upon the doping. The density of micropipes was less than 10 per square centimeter while the density of secondary-phase inclusions (i.e., carbon and silicon) was 10 per cubic centimeter. The measured concentration of background impurities was 10^{16} per cubic centimeter for nitrogen; 10^{16} per cubic centimeter for boron; and 10^{16} to 10^{17} per cubic centimeter for tantalum. There was no measurable graphitization of the source or the growing surface during a 10 hour growth procedure which was performed at a temperature of 2000° C. and at a pressure of 10^{-5} Torr. The seed polytype reproducibility was 80 percent for a 6H SiC seed growing in direction [0001] Si; 70 percent for a 4H SiC seed growing in direction [0001] C; and 100 percent for a 6H SiC seed growing in a direction lying at an angle of 5 degrees to direction [0001].

As will be understood by those familiar with the art, the present invention may be embodied in other specific forms without departing from the spirit or essential characteristics thereof. Accordingly, the disclosures and descriptions herein are intended to be illustrative, but not limiting, of the scope of the invention which is set forth in the following claims.

What is claimed is:

1. A silicon carbide material comprising an axial region of re-crystallized single crystal silicon carbide with a density of dislocations of less than 10^4 per square centimeter, a density of micropipes of less than 10 per square centimeter, and a density of secondary phase inclusions of less than 10 per cubic centimeter.

2. The silicon carbide material of claim **1**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of less than 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

3. The silicon carbide material of claim **1**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

4. The silicon carbide material of claim **1**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of less than 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

5. The silicon carbide material of claim **1**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

6. The silicon carbide material of claim **1**, wherein said density of secondary phase inclusions is less than 1 per cubic centimeter.

7. A silicon carbide material comprising an axial region of re-crystallized single crystal silicon carbide with a density of dislocations of less than 10^3 per square centimeter, a density of micropipes of less than 10 per square centimeter, and a density of secondary phase inclusions of less than 10 per cubic centimeter.

8. The silicon carbide material of claim **7**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of less than 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

9. The silicon carbide material of claim **7**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

10. The silicon carbide material of claim **7**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of less than 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

11. The silicon carbide material of claim **7**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

12. The silicon carbide material of claim **7**, wherein said density of secondary phase inclusions is less than 1 per cubic centimeter.

13. A silicon carbide material comprising an axial region of re-crystallized single crystal silicon carbide with a density of dislocations of less than 10^2 per square centimeter, a density of micropipes of less than 10 per square centimeter, and a density of secondary phase inclusions of less than 10 per cubic centimeter.

14. The silicon carbide material of claim **13**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of less than 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

15. The silicon carbide material of claim **13**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

16. The silicon carbide material of claim **13**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of less than 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

17. The silicon carbide material of claim **13**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

18. The silicon carbide material of claim **13**, wherein said density of secondary phase inclusions is less than 1 per cubic centimeter.

19. A silicon carbide material, comprising:

a single crystal silicon carbide seed crystal, said single crystal silicon carbide seed crystal having a growth surface; and

a region of axially re-crystallized silicon carbide, said region of axially re-crystallized silicon carbide initiating at said growth surface of said single crystal silicon carbide seed crystal, said region of axially re-crystallized silicon carbide having a density of dislocations of less than 10^4 per square centimeter, a density of micropipes of less than 10 per square centimeter, and a density of secondary phase inclusions of less than 10 per cubic centimeter.

20. The silicon carbide material of claim **19**, wherein said density of dislocations in said region of axially re-crystallized silicon carbide is less than 10^3 per square centimeter.

21. The silicon carbide material of claim **19**, wherein said density of dislocations in said region of axially re-crystallized silicon carbide is less than 10^2 per square centimeter.

22. The silicon carbide material of claim **19**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of less than 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

23. The silicon carbide material of claim **19**, wherein said axial region of re-crystallized single crystal silicon carbide has a tantalum impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said tantalum impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

24. The silicon carbide material of claim **19**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of less than 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

25. The silicon carbide material of claim **19**, wherein said axial region of re-crystallized single crystal silicon carbide has a niobium impurity concentration of between 10^{16} and 10^{17} per cubic centimeter, said niobium impurity concentration uniformly distributed throughout the re-crystallized single crystal silicon carbide material.

26. The silicon carbide material of claim **19**, wherein said density of secondary phase inclusions is less than 1 per cubic centimeter.

* * * * *