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(54) Title: CRYSTALLINE HEXAGONAL BORON NITRIDE WITH NITROGEN-15 ISOTOPE ENRICHMENT

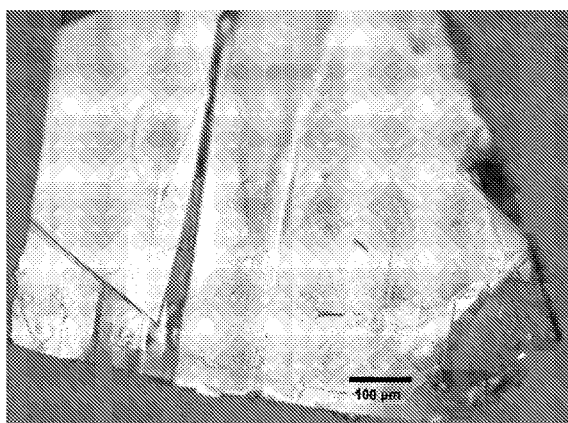


FIG. 1

PRIOR ART

(57) Abstract: A composition comprising crystalline hexagonal boron nitride (hBN) enriched with the nitrogen-15 isotope and a process for preparing the same are provided. The hBN enriched in L5N can be used in the fabrication of electronic devices including quantum sensors of strain and magnetic fields, and infrared chemical sensors.



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CRYSTALLINE HEXAGONAL BORON NITRIDE
WITH NITROGEN-15 ISOTOPE ENRICHMENT

STATEMENT REGARDING FEDERALLY SPONSORED
RESEARCH AND DEVELOPMENT

[0001] This application claims the benefit of U.S. Provisional Patent Application No. 63/509,108, filed on June 20, 2023, which is incorporated by reference herein in its entirety. This invention was made with government support under Contract Nos. N00014-20-1-2474 and N00014-22-1-2582 awarded by the Office of Naval Research. The government has certain rights in the invention.

BACKGROUND OF THE INVENTION

Field of the Invention

[0002] Embodiments according to the present invention are directed toward crystalline hexagonal boron nitride (hBN) enriched with the nitrogen-15 isotope and methods of making the same.

Description of the Prior Art

[0003] Hexagonal boron nitride (hBN) is the second most important two-dimensional material after graphite and its monolayer derivative, graphene. Although hBN and graphite have similar crystal structures and lattice parameters, their properties are quite different. Graphite is electrically conductive; hBN is an electrical insulator. Graphite is opaque; hBN is optically transparent from deep ultraviolet through the infrared wavelengths. Graphite reacts with oxygen and air at low temperatures, while hBN is chemically inert. Thus, in many ways, the properties of hBN and graphite are complimentary.

[0004] The properties of crystalline hBN are advantageous for a wide range of nanophotonic, electronic, and quantum devices. Because its optical properties are

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hyperbolic, it can compress infrared light to extremely small volumes, through the use of hyperbolic phonon polaritons. This is useful in nanophotonics as it increases light-matter interactions, and thereby chemical and biological sensing. It also enables much higher image resolutions, hyperlensing, than is possible using the source free-space light. hBN analogue switches with high power handling and switching speeds have shown promise for high frequency devices, while hBN-based memristors integrated into CMOS devices for computation had superior endurance compared to more mature technologies. hBN is appealing for quantum devices due to its ability to host bright, stable, single photon emitters, some of which are magnetically addressable.

[0005] Hexagonal boron nitride (hBN) has been a commercial product for over 60 years as amorphous fine powders and very fine grain (<20 micron) solids. In contrast, new interest in crystalline (ideally single crystals) hBN for potential electronic, optoelectronic, nanophotonic and quantum devices has accelerated over the past twenty years. This is due in part to crystalline hBN's unique optical properties. It has the potential to be an excellent sensor of specific organic molecules since its infrared frequency range matches those of the vibrational properties of specific organic ligands. hBN is also appealing for quantum devices based on defects that emit at visible light frequencies that are sensitive to magnetic fields.

[0006] Single crystals of hBN have been grown by dissolving boron or boron nitride in molten metals at high temperatures, and then slowly cooling the solution to reduce the solubility of the boron nitride. As a result, boron nitride crystals are precipitated. An example of a crystalline flake produced by this method (approximately 10 microns thick) is shown in Fig. 1.

[0007] There has also been work in growth of hBN crystalline flakes that are enriched in either the boron-10 ($h^{10}\text{BN}$) or boron-11 ($h^{11}\text{BN}$) isotope. Most hBN has the natural distribution of boron isotopes, which is 20% ^{10}B and 80% ^{11}B . However, by using isotopically pure boron-10 or boron-11 in the synthesis of hBN, crystalline hBN flakes can be produced that have superior infrared optical properties, which render the flake preferred for use in nanophotonic devices.

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SUMMARY OF THE INVENTION

[0008] According to one embodiment of the present invention, there is provided a hexagonal boron nitride composition enriched in nitrogen-15 isotope comprising at least 50%, by weight of $h^{10}B^{15}N$, $h^{11}B^{15}N$, or a mixture thereof.

[0009] According to yet another embodiment of the present invention there is provided a method of producing a hexagonal boron nitride composition enriched in nitrogen-15 isotope. The method comprises providing a source of boron and heating the source of boron to a temperature of 1550°C or greater under an N_2/H_2 atmosphere comprising $^{15}N_2$ gas and H_2 gas to create a composition comprising boron nitride. The composition comprising boron nitride is cooled and single crystals of the hexagonal boron nitride are precipitated. The single crystals of the hexagonal boron nitride are recovered from the composition.

[00010] According to still another embodiment of the present invention, electronic devices comprising the hexagonal boron nitride composition as described herein are provided. In certain embodiments, the devices may include an optoelectronic device, a nanophotonic device, or a quantum device.

BRIEF DESCRIPTION OF THE DRAWINGS

[00011] Figure 1 is an optical micrograph of a crystalline hBN flake produced by precipitation from a molten metal solution.

[00012] Fig. 2 is a temperature profile of a two-part process used to produce isotopically-controlled hBN crystals.

[00013] Fig. 3 is an image of the surface of an Ni-Cr-B ingot after precipitation of $hB^{15}N$ crystals on the surface of the ingot.

[00014] Fig. 4 is a micrograph of an $h^{10}B^{15}N$ single crystal mechanically exfoliated from a solidified ingot.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[00015] The properties of hBN can be varied by controlling its concentrations of boron isotopes. There are two stable boron isotopes, which are naturally distributed as

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approximately 20% ^{10}B and 80% ^{11}B . These isotopes have distinctly different properties. Their nuclear spins are 3 and 3/2 for ^{10}B and ^{11}B respectively. ^{10}B has one of the largest neutron capture cross section of any isotope of any element, approximately 3855 barns for neutrons with velocities of 2200 m/s. For the majority of element isotopes, the neutron capture cross section is on the order of one or less.

[00016] The boron isotope enrichment of hBN leads to certain characteristic distinctions. For instance, the energy band gap of hBN is slightly reduced (increased) when enriched in ^{10}B (^{11}B). Also, the $\text{E}_{2\text{g}}$ peak position in the Raman spectra is shifted up from $1,366\text{ cm}^{-1}$ for hBN with the natural distribution of boron isotopes to $1,383\text{ cm}^{-1}$ for h^{10}BN and down to $1,357\text{ cm}^{-1}$ for h^{11}BN . The FWHM of the $\text{E}_{2\text{g}}$ peak at room temperature of the boron monoisotopic hBN is reduced to 3.0 cm^{-1} from 7.5 cm^{-1} for natural hBN. This reduced Raman peak width is due to less isotopic disorder. The maximum peak width is predicted to occur with isotope concentrations of 65% ^{11}B and 35% ^{10}B . The phonon lifetime for monoisotopic hBN is increased by a factor of 2.7 from $\text{h}^{\text{nat}}\text{BN}$. Using scattering type near field optical microscopy (S-NOM) the phonon-polariton propagation length is significantly increased in monoisotopic hBN. The maximum hyperbolic phonon polariton lifetimes measured is 4.2 ps with propagation lengths of $25\text{ }\mu\text{m}$, and an eight-fold increased figure of phonon propagating merit in comparison to $\text{h}^{\text{nat}}\text{BN}$. The longer phonon lifetimes achieved by eliminating isotopic disorder also increases the thermal conductivity of hBN.

[00017] Monoisotopic hBN has also proven advantageous in several applications. The longer hyperbolic phonon polariton propagation distance in isotopically enriched h^{10}BN enables the production of an anomalous, concave wave front on a h^{10}BN metasurface. Demonstrating hyperlensing, objects as small as 44 nm separated by distances of 25 nm can be resolved via N-SOM with light with a free-space wavelength of $6.76\text{ }\mu\text{m}$ using h^{11}BN . Vibrational strong coupling between phonon polaritons in hBN and organic molecules has also been shown. This has the potential to reduce the size and enhance the sensitivity of on-chip spectroscopes. The optical properties of h^{11}BN can be changed by placing it in contact with VO_2 which was changed from a dielectric to a metal by changing its temperature.

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[00018] Further control over the properties of hBN can be achieved by changing the concentration of the nitrogen isotope in crystalline hBN. Natural nitrogen is predominantly ^{14}N (99.6%) with a small amount of ^{15}N (0.4%). As an example, changing the nitrogen isotopes as well as the boron isotopes offers the possibility of shifting its Reststrahlen band, to better match the infrared absorption of specific organic molecules, thus achieving sensors that are better (more sensitive at detecting specific organic compounds). Further, ^{14}N and ^{15}N have different nuclear spins, 1 and $\frac{1}{2}$ respectively, which may prove important in quantum applications. Unlike ^{14}N , ^{15}N reacts with high energy protons to form carbon and an alpha particle.

[00019] In one or more embodiments, hBN crystals can be prepared that are enriched in the nitrogen-15 isotope, both $\text{h}^{10}\text{B}^{15}\text{N}$ and $\text{h}^{11}\text{B}^{15}\text{N}$. In particular embodiments, a hBN composition is provided in which $\text{h}^{10}\text{B}^{15}\text{N}$ and/or $\text{h}^{11}\text{B}^{15}\text{N}$ comprise the predominant hBN species. Preferably, the hBN composition comprises at least 50%, at least 60%, at least 70%, at least 80%, at least 90%, at least 95%, or at least 98% by weight of $\text{h}^{10}\text{B}^{15}\text{N}$, $\text{h}^{11}\text{B}^{15}\text{N}$, or a mixture thereof. Alternatively, the hBN composition is depleted in $\text{h}^{10}\text{B}^{14}\text{N}$ and/or $\text{h}^{11}\text{B}^{14}\text{N}$. In such embodiments, while some $\text{h}^{10}\text{B}^{14}\text{N}$, $\text{h}^{11}\text{B}^{14}\text{N}$, or a mixture thereof is present in the composition, such nitrogen-14 isotope species comprise a minority component, preferably less than 50%, less than 40%, less than 30%, less than 20%, less than 10%, less than 5%, or less than 2% by weight of the composition. In one or more embodiments, the hBN composition comprises hBN flakes of single-crystal hBN.

[00020] There are at least two potential advantages to this ^{15}N enriched material, hB^{15}N . First, the infrared frequency range in $\text{h}^{11}\text{B}^{15}\text{N}$ is shifted to a new region not previously accessible with common hBN. This can greatly improve its sensitivity to specific organic molecules. Second, because the nuclear spin of ^{15}N ($\frac{1}{2}$) is lower than ^{14}N (1), the quantum defects in hB^{15}N have a longer spin coherence lifetime than that of regular hBN. This results in better quantum properties, and thus quantum devices with improved sensitivities.

[00021] Another application in which hB^{15}N can be useful is micro nuclear magnetic resonance (NMR) spectroscopy. With hB^{15}N , NMR with unprecedented high

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resolution can be possible. In certain embodiments, hB¹⁵N is an alternative to the current leading contender for micro-NMR, which is diamond with the nitrogen-vacancy (NV) center.

[00022] Conventional hBN flakes have been manufactured by a process in which natural nitrogen (primarily ¹⁴N₂ gas) continuously flows through the furnace during the chemical synthesis and crystal growth. Consequently, many cubic feet of nitrogen are used over the multi-day process to grow the crystals. Due to the low cost of natural nitrogen, the cost is minimal. In contrast, due to the high cost of ¹⁵N enriched nitrogen (more than 100,000 times higher than industrial grade natural nitrogen), the continuous flowing of nitrogen during the process is not economically feasible. Thus, in one embodiment, the present invention pertains to a static process for synthesis of hB¹⁵N, in which the furnace can be filled once with the ¹⁵N enriched gas, and not changed over course of the synthesis process.

[00023] According to one or more embodiments, hB¹⁵N crystalline flakes are produced by precipitation from molten metal solutions. A source of boron is provided and heated to a temperature sufficient to cause the boron to dissolve within one or more metal solvents. In particular embodiments, the molten metal solution is prepared by heating the source of boron to at temperature of 1550°C or greater under a nitrogen/hydrogen atmosphere comprising ¹⁵N₂ gas and H₂ gas to create a composition comprising boron nitride. Preferably, the N₂/H₂ atmosphere comprises isotopically pure ¹⁵N₂ gas (i.e., at least 95%, at least 98%, or at least 99% pure ¹⁵N₂). Preferably, the hydrogen concentration of the N₂/H₂ atmosphere is no greater than 10% so as to avoid surface etching of the hBN crystals. In one or more embodiments, the N₂/H₂ atmosphere a hydrogen to nitrogen molar ratio of from 0.7 to 1.2, or from 0.8 to 1.1, or from 0.9 to 1. In certain embodiments, the N₂/H₂ atmosphere is charged to a furnace containing the source of boron and remains static (i.e., there is no continuous flow of N₂) throughout the heating step.

[00024] The source of boron may be substantially monoisotopic. That is, the source of boron is enriched to nearly 100% ¹⁰B or 100% ¹¹B (i.e., greater than 98%, or greater than 99%); however, this need not always be the case and a mixture of boron isotopes

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can be provided.

[00025] In one or more embodiments, the source of boron can be formed by mixing a boron powder comprising isotopically pure ^{10}B or ^{11}B , or a mixture of ^{10}B and ^{11}B with nickel and chromium powders to form a boron-containing mixture. In an alternate embodiment, iron can be substituted for the nickel in forming the molten metal solution. The mass ratio of nickel or iron to boron, and chromium to boron is from 10:1 to 50:1, from 15:1 to 40:1, 20:1 to 30:1, or about 24:1. The mass ratio of nickel or iron to chromium is from 0.1:1 to 10:1, from 0.25:1 to 4:1, from 0.5:1 to 2:1, or about 1:1. Accordingly, a preferred mass ratio of nickel or iron to chromium to boron in the boron-containing mixture is about 12:12:1.

[00026] The boron-containing mixture is heated under an argon/hydrogen atmosphere to 1550°C or greater to form the metal solution. In a preferred embodiment, the ratio of argon to hydrogen in the argon/hydrogen atmosphere is about 93 to 7. The pressure of the atmosphere within the furnace containing the boron-containing mixture is 600 to 1200 torr, or 700 to 1100 torr, 800 to 1000 torr, or about 850 torr. In certain embodiments, the heating step can be carried out for a period of time of from 2 hr. to 4 days, to 6 hr. to 3 days, from 12 hr. to 2 days, or about 24 hr.

[00027] The boron-containing mixture is then cooled to form a homogeneous boron-metal ingot. The boron-metal ingot comprises the source of boron from which the hB^{15}N is synthesized.

[00028] Returning to the process for producing the hB^{15}N , once formed, the composition comprised of boron nitride and metals is cooled thereby precipitating single crystals of the hB^{15}N . The cooling step comprises slowly cooling the composition comprising boron nitride at a rate of $1\text{--}4^{\circ}\text{C/hr.}$ to a temperature of 1500°C to precipitate the crystals of hB^{15}N .

[00029] The precipitated crystals of hB^{15}N are recovered from the composition. In one or more embodiments, the recovery step comprises recovering an ingot comprising the source of boron having a layer of the single crystals of the hexagonal boron nitride contained thereon. The recovery step can further comprise mechanically exfoliating free-standing hexagonal boron nitride flakes from the ingot comprising the

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source of boron.

[00030] As indicated above, the hBN crystals made according to any embodiment of the present invention can be used in fabricating various electronic devices. In one embodiment, the electronic device comprises an optoelectronic device, a nanophotonic device, or a quantum device. The electronic device may comprise an analogue switch or a memristor. In addition, the device may comprise a sensor for detecting one or more organic molecules.

EXAMPLE

[00031] In this Example, hBN single crystals were prepared that are enriched in nitrogen-15. Note, this Example is provided by way of illustration and should not be taken as limiting upon the scope of the present invention.

[00032] A two-step process was used. In the first step, isotopically pure (>99%) ^{10}B or ^{11}B powder was mixed with Ni and Cr powder in an alumina crucible in the mass ratio 12/12/1 Ni/Cr/B, respectively. Note, crucibles formed from other suitable materials, such as zirconia (ZrO_2) and magnesia (MgO), may also be used. This was then placed in a horizontal tube furnace, then evacuated of air and purged with a mixture of 93/7 argon/hydrogen continuously flowing through it at 270 sccm and 850 torr. The furnace was heated to 1550°C, held there for 24 hours, and cooled back down as illustrated by the dashed line in Fig. 2. The purpose of this step is to create a homogenous ingot and remove some of the impurities brought into the system by the source materials. Thus, no nitrogen is used because the formation of hBN interferes with thorough mixing of the components. Hydrogen, diluted with argon for safety, is included in this step to help remove oxygen impurities by reacting with residual oxygen to form water vapor and carry it out of the system.

[00033] In the second step, the ingot produced in the first step is placed in the same furnace purged of air and backfilled with a mixture isotopically pure $^{15}\text{N}_2$ gas and hydrogen. The gas mixture comprised between 8%-9% hydrogen and 91%-92% nitrogen. Due to the high cost of $^{15}\text{N}_2$ gas, it was not continuously flowed through the system and was instead sealed off once the pressure rose to atmospheric pressure. Then

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the furnace was heated to 1550°C, held there for 24 hours, and slowly cooled at 1°C/hr. to 1500°C to carefully precipitate hBN single crystals as illustrated by the solid line in Fig. 2. Since isotopically pure boron (^{10}B or ^{11}B) and isotopically pure ^{15}N are the only boron and nitrogen sources present in the system, any hBN that forms will have these isotopes. This process also has the flexibility to produce hBN with precise mixtures of isotopes by controlling how much ^{10}B and ^{11}B powder is added in the first step and the ratio of $^{14}\text{N}_2$ and $^{15}\text{N}_2$ gas introduced to the system.

[00034] Once the system has cooled, the alumina boat contains a solidified metal ingot with a layer of hBN crystals covering its top surface. Fig. 3 is an image of the surface of the ingot surface comprising hB ^{15}N crystals. Mechanical exfoliation of the hBN from the ingot produces free-standing hBN flakes, which are shown in Fig. 4.

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WE CLAIM:

1. A hexagonal boron nitride composition enriched in nitrogen-15 isotope comprising at least 50% by weight of $h^{10}B^{15}N$, $h^{11}B^{15}N$, or a mixture thereof.
2. The hexagonal boron nitride composition of claim 1, wherein the composition comprises at least 90% by weight of $h^{10}B^{15}N$, $h^{11}B^{15}N$, or a mixture thereof.
3. The hexagonal boron nitride composition of claim 1, wherein the composition comprises at least 95% by weight of $h^{10}B^{15}N$, $h^{11}B^{15}N$, or a mixture thereof.
4. The hexagonal boron nitride composition of claim 1, further comprising $h^{10}B^{14}N$, $h^{11}B^{14}N$, or a mixture thereof, wherein the $h^{10}B^{14}N$, $h^{11}B^{14}N$, or a mixture thereof is present in an amount of less than 50% by weight.
5. The hexagonal boron nitride composition of claim 4, wherein the $h^{10}B^{14}N$, $h^{11}B^{14}N$, or a mixture thereof is present in an amount of less than 10% by weight.
6. The hexagonal boron nitride composition of claim 4, wherein the $h^{10}B^{14}N$, $h^{11}B^{14}N$, or a mixture thereof is present in an amount of less than 5% by weight.
7. The hexagonal boron nitride composition of any of claims 1 to 6, wherein the composition comprises crystalline boron nitride.
8. A method of producing a hexagonal boron nitride enriched in nitrogen-15 isotope comprising:
 - providing a source of boron and heating the source of boron to a temperature of 1550°C or greater under an N_2/H_2 atmosphere comprising $^{15}N_2$ gas and H_2 gas to create a composition comprising boron nitride;
 - cooling the composition comprising boron nitride and precipitating single crystals of the hexagonal boron nitride; and

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recovering the single crystals of the hexagonal boron nitride from the composition.

9. The method of claim 8, wherein the source of boron is formed by:
mixing a boron powder comprising isotopically pure ^{10}B or ^{11}B , or a mixture of ^{10}B and ^{11}B with nickel and chromium powders to form a boron-containing mixture;
heating the boron-containing mixture under an argon/hydrogen atmosphere to 1550°C ;
cooling the boron-containing mixture to form a homogeneous boron-metal ingot, the boron-metal ingot comprising the source of boron.
10. The method of claim 9, wherein the ratio of argon to hydrogen in the argon/hydrogen atmosphere is about 93 to 7.
11. The method of claim 9, wherein the argon/hydrogen atmosphere is flowed through a furnace containing the boron-containing mixture at a pressure of 600 to 1200 torr.
12. The method of claim 9, wherein the mass ratio of nickel to chromium to boron in the boron-containing mixture is about 12 to 12 to 1.
13. The method of claim 8, wherein the N_2/H_2 atmosphere comprises isotopically pure $^{15}\text{N}_2$ gas.
14. The method of claim 8, wherein the N_2/H_2 atmosphere is charged to a furnace containing the source of boron and remains static throughout the heating step.
15. The method of claim 8, wherein the cooling step comprises cooling the composition comprising boron nitride at a rate of $1\text{-}4^{\circ}\text{C/hr.}$ to a temperature of 1500°C .

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16. The method of claim 8, wherein the recovery step comprises recovering an ingot comprising the source of boron having a layer of the single crystals of the hexagonal boron nitride contained thereon.

17. The method of claim 16, wherein the recovery step further comprises mechanically exfoliating free-standing hexagonal boron nitride flakes from the ingot comprising the source of boron.

18. An electronic device comprising the hexagonal boron nitride composition of claim 1.

19. The electronic device of claim 18, wherein the device comprises an optoelectronic device, a nanophotonic device, or a quantum device.

20. The electronic device of claim 18, wherein the device comprises an analogue switch or a memristor.

21. The electronic device of claim 18, wherein the device comprises a sensor for detecting one or more organic molecules.

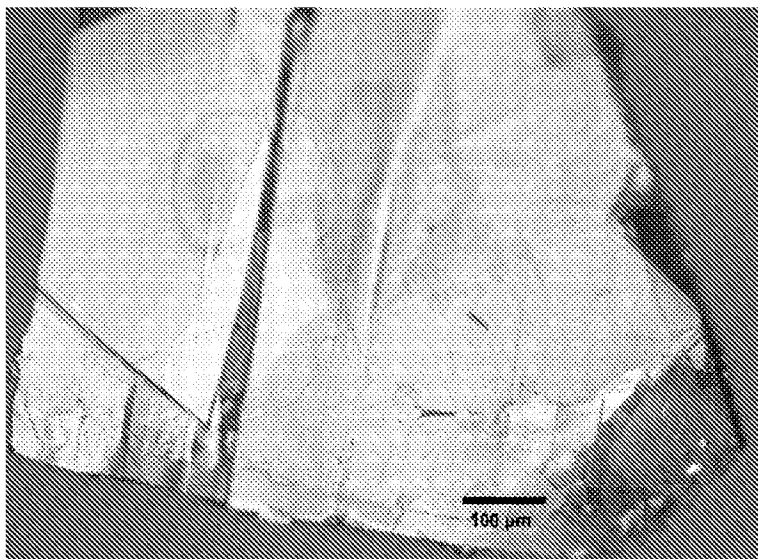


FIG. 1

PRIOR ART

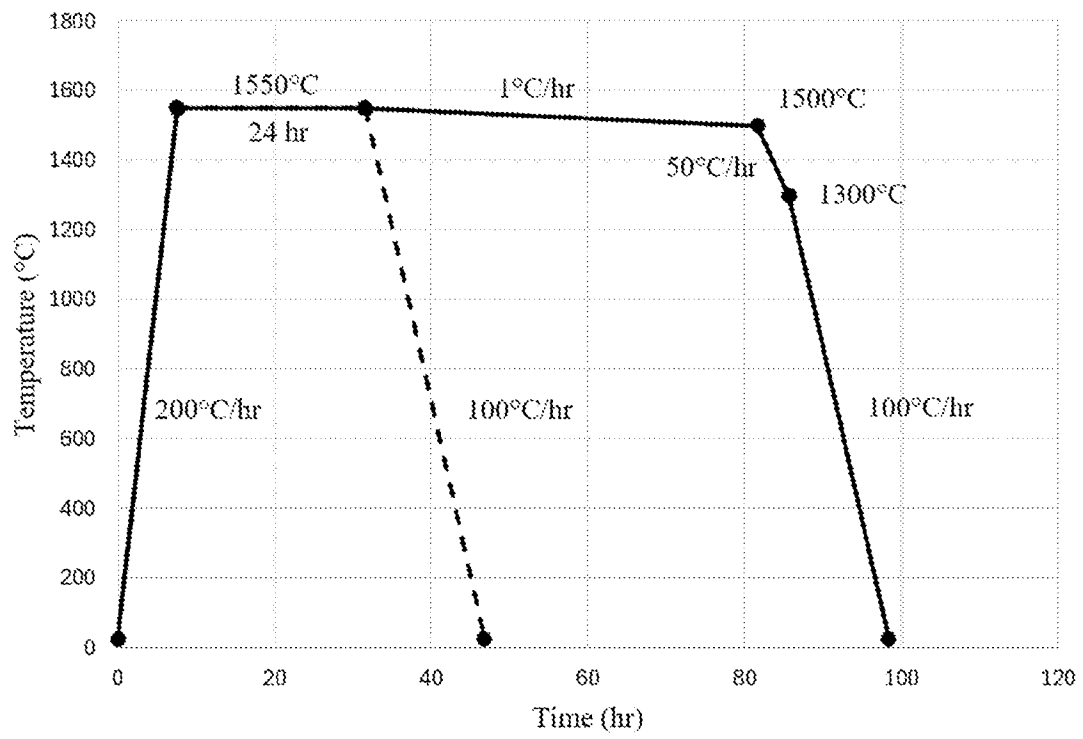


FIG. 2

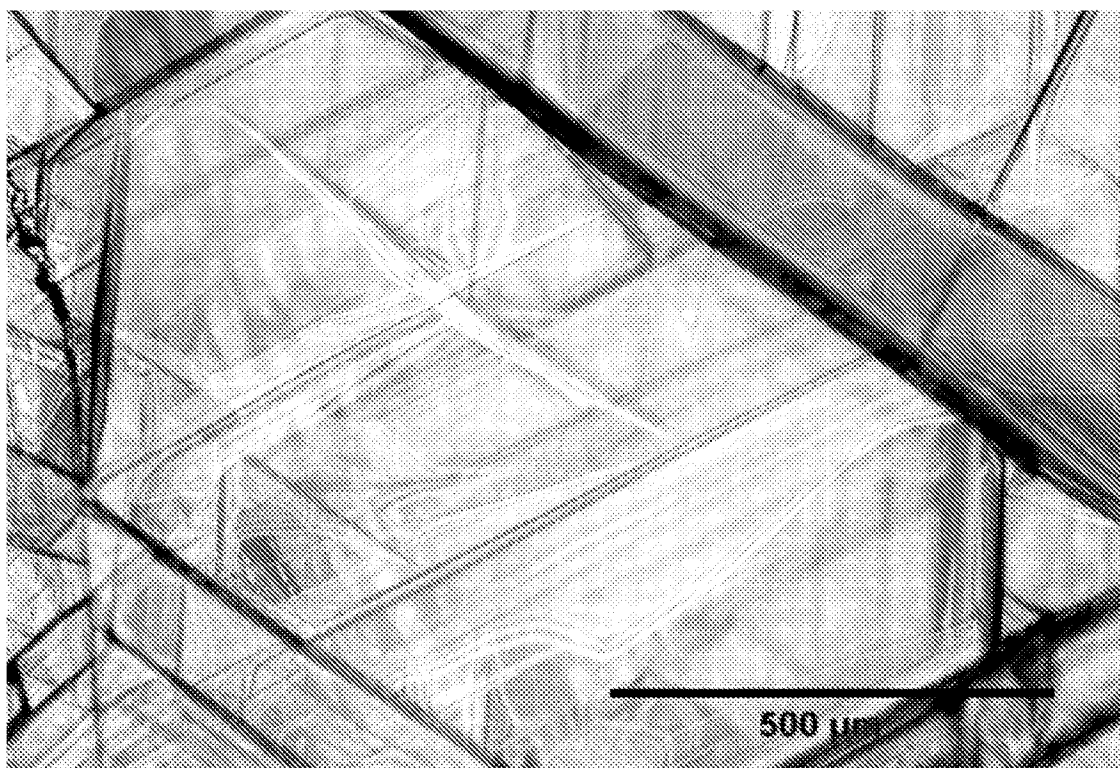


FIG. 3

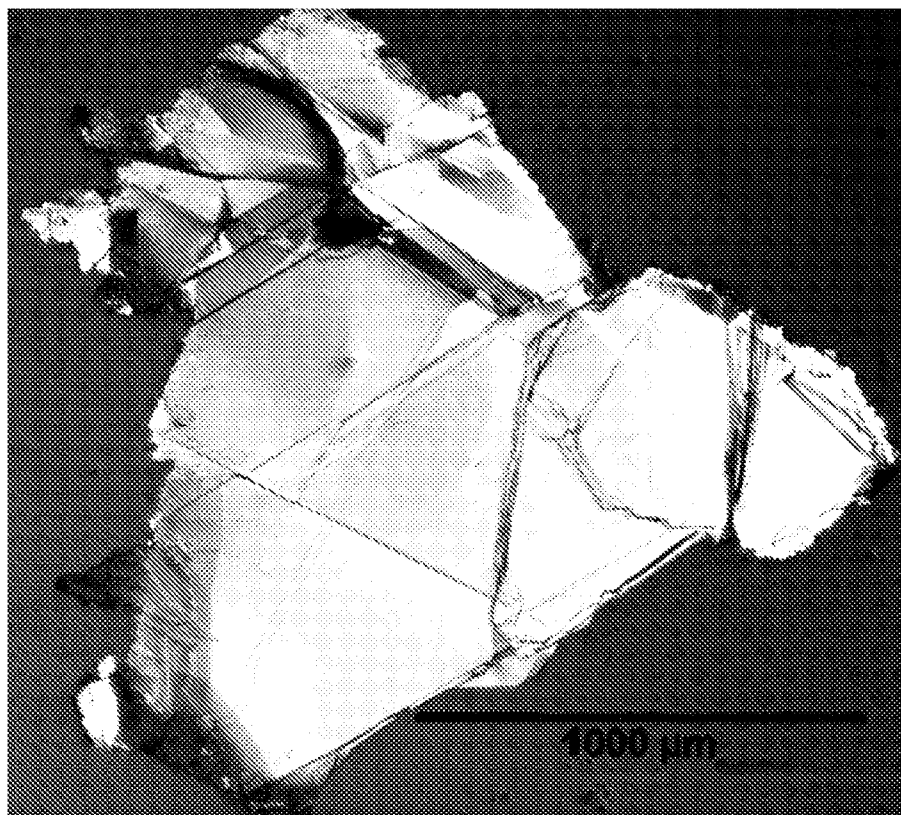


FIG. 4