Synthesis of nitrides and silicon carbide using sodium

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Studies on the synthesis of nitrides, nitride-related compounds and silicon carbide by using Na as a flux and reaction enhanced medium are reviewed. Using a Na melt, ternary and quaternary nitrides and nitride-related compounds containing Ba and Sr were prepared at around 1000 K and N2 pressure below about 10 MPa. Crystal growth of a binary nitride GaN by heating Ga and N2 with Na was also found during the studies of the multinary compounds. AlN powder was prepared above the Al melting temperature of 932 K. Single crystals of CrN and Mn2N were obtained by heating Cr and Mn with a Na–Ga flux and a Na–In flux, respectively. β-type SiC powder and porous bulks were prepared by reaction of Si and fullerene with Na or Si and amorphous carbon with Na at 1000 K. The role of Na in the formation of the nitrides and silicon carbide is herein discussed.©2009 The Ceramic Society of Japan

Key-words: Nitrides, Zintl anions, Silicon carbide, Na flux, Single crystal, Crystal structure

Table 1. Multinary Nitrides and Nitride-related Compounds Prepared by Using a Na Flux

<table>
<thead>
<tr>
<th>nitridometallate anions</th>
<th>Zintl-polyanion</th>
<th>ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sr2ZnN2</td>
<td>[Zn(N2)]4–</td>
<td>15)</td>
</tr>
<tr>
<td>Ba/Zn(N2)</td>
<td>[Zn(N2)]4–</td>
<td>18)</td>
</tr>
<tr>
<td>Ba/Ga(N2)</td>
<td>[Ga(N2)]4–</td>
<td>16)</td>
</tr>
<tr>
<td>Ba/Si(N2)</td>
<td>[Si(N2)]4–</td>
<td>17)</td>
</tr>
<tr>
<td>SrCuN</td>
<td>[Cu(N2)3]2–</td>
<td>19)</td>
</tr>
<tr>
<td>Sr/Si/Cu(N2)</td>
<td>[Cu(N2)3]2–</td>
<td>21)</td>
</tr>
<tr>
<td>Ba/Cu(N2)</td>
<td>[Cu(N2)3]2–</td>
<td>20)</td>
</tr>
<tr>
<td>Ba/Sr/Cu(InN3)</td>
<td>[Cu(N3)]4– / [In(N3)]4–</td>
<td>21,22)</td>
</tr>
<tr>
<td>Ba/Cu(InN3)</td>
<td>[Cu(N3)]4– / [In(N3)]4–</td>
<td>23)</td>
</tr>
<tr>
<td>Ba/In(N3)</td>
<td>[In(N3)]4– / [In(N3)]4–</td>
<td>24)</td>
</tr>
<tr>
<td>Ba/In(N3)2</td>
<td>(In defect 3D network)</td>
<td>25)</td>
</tr>
</tbody>
</table>

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into Na and N₂ gas at around 570 K. The N₂ pressure in the sealed Nb tube was estimated to be 4–11 MPa at 1023 K from the inner volume of the sealed tube and the amount of NaN₃.

The samples obtained after heating were a mixture of Na and crystals of unknown compounds, and sometimes contained intermetallic compounds of Na and other source metals. Since all the compounds listed in Table 1 are unstable in air and against water, the products were washed in liquid NH₃. Na can also be removed from the sample by evaporation in a furnace under a temperature gradient condition or reduced pressure if the obtained compounds are stable at the conditions. In the case of nitrides stable in air and alkaline water, Na metal remaining in the products can be removed by reaction with 2-propanol and ethanol, or water vapor.

Figure 1 shows a photograph of a platelet transparent yellow single crystal of Ba₃Ga₂N₄ sealed in a glass capillary with Ar gas. The size was about 0.5 to 1.0 mm and large enough for single crystal X-ray diffraction. Figure 2 shows the crystal structure of Ba₃Ga₂N₄ drawn with the program VESTA. In this structure, GaN₄ nitrogen tetrahedra share their edges and form one-dimensional chains.

The starting metal materials used for the synthesis of Ba₈Cu₃In₄N₅ and Ba₁₄Cu₂In₄N₇ were Ba, Cu, In and Na. These metals were charged in a BN crucible, which was placed in a stainless steel container. A gas feed line was connected to the container as shown in Fig. 3(a) and N₂ gas was supplied up to a total pressure of 7 MPa. The container was heated at 1023 K and cooled to 823 K at a rate of 2 K/h.

The new compounds prepared by the Na flux method contain alkaline earth elements of Ba and Sr. The electrons from Ba and Sr are used to form nitridometallate anion groups and Zintl (poly-)anions of Ge and In. Edge-sharing Sr-centered nitrogen tetrahedra were first discovered in Ba₃Si₃N₄. The crystal structures of Ba₃GeGe₄N₉, Ba₃Cu₄In₅N₇ and Ba₃Cu₃In₃N₇ are new combinations of nitridometallate anions and Zintl (poly-)anions. Figures 4 and 5 show a photograph of a single crystal of Ba₃Cu₄In₅N₇ taken under Ar atmosphere and the crystal structure, respectively. It contains Zintl anions [In₅]⁻ and nitridometallate anions [CuN₂]⁻ in which one Cu atom is almost linearly coordinated by two N atoms. The nitridometallate (poly-)anions have the same valence electrons as the compounds and molecules of main-group elements. For example, [CuN₂]⁻ is iso-electric with [ZnN₂]⁺ in Sr₂ZnN₂, Ba₂ZnN₂, and Ba₂ZnN₂O and has the same 16 valence electrons as [BN₂]⁻ in Li₃BN₂ and as a CO₂ molecule.

3. Single crystal growth of GaN

The first experiment in which the author found the growth of GaN single crystals by using a Na melt was carried out at 1023 K with Ga, Na and NaN₃ which were directly sealed in a Nb tube. The initial N₂ pressure in the tube was estimated to be about 7 MPa. By the sealed method, N₂ pressure decreased with increasing the GaN single crystal formation. The stainless steel container schematically illustrated in Fig. 3(a) was designed so as to synthesize GaN at a constant N₂ pressure. Because GaN is stable in air and aqua regia, the product was first reacted...
with alcohol to remove Na and to decompose the Na–Ga intermetallic compound into Ga, and then Ga was dissolved with aqua regia. GaN crystals left in the crucible were washed with distilled water.

Figure 6 shows the growth temperature and pressure conditions of GaN single crystals by the Na flux method. The equilibrium N₂ pressure for the formation of GaN reported by Karpinski et al. and the melting point of GaN reported by Utsumi et al. are also plotted in this figure. The melting point of GaN is 2493 K under a high N₂ pressure of 6 GPa. Utsumi et al. grew GaN single crystals with a size over 10 mm were grown in a Ga melt at around 1770 K and 2 GPa of N₂ pressure by Porowski et al. This is regarded to be a self-flux method where nitrogen is dissolved in the Ga melt and GaN single crystals grow from the Ga solution. The temperature and N₂ gas pressure conditions of the Na flux method are around 970 to 1120 K and 3 to 10 MPa, respectively. These conditions are much more moderate than those of the other growth methods in which Ga and N₂ react.

The morphologies and yields of GaN crystals depend on the growth conditions. Formation of pyramidal black GaN crystals 0.1 mm in size was achieved by heating at 973 and 1023 K at a pressure of 1 MPa for 200 h with a premixed Na–Ga melt of Na/(Na + Ga) = 0.67. The yields under this condition were 6 and 13% against the initial Ga content. No GaN crystal was obtained at 1073 K and 1 MPa but sub-millimeter sized prismatic crystals were formed at 1073 K and 2–4 MPa. The yields increased from 68% to almost 100% with increasing the nitrogen pressure. Platelet crystals approximately 1 mm in length and 0.1–0.2 mm in thickness were obtained at 1073 K and 5 MPa of N₂.

As shown in Fig. 7, platelet crystals with a size of over 3 × 3 mm were obtained at 1023 K and 5 MPa of N₂ by reducing the area of contact between the Na–Ga melt and the surface of a sintered BN crucible with a cone-shaped cavity. Furthermore, by using Na purified by distillation and a pyrolytic BN crucible with a smooth surface, platelet crystals having a size of 10 mm in the longest direction were grown at 1048 K and 5 MPa. The thickness of the platelet crystals obtained was 0.1–0.3 mm and they included lustrous thin layers of Na.

The crystal morphology of GaN changed from platelet to prismatic or needle-like by the addition of the 3d-transition metals Mn, Fe, Co and Ni in a Na–Ga melt. Colorless transparent prismatic crystals 1.5 mm in size were obtained at 1073 K and 5 MPa by the addition of Ni to the melt. GaN single crystals were doped with 0.10–0.35 at% of Mn. The Mn-doped GaN single crystals were transparent reddish brown and exhibited Curie-like paramagnetism. The other transition metals were not detected in the obtained GaN crystals by inductively coupled plasma atomic emission spectroscopy.

In the above-mentioned crystal growth of GaN by the Na flux method, Na and Ga were premixed and melted in a BN crucible before GaN formation. The crystals which precipitated on the crucible surface were always black, but became transparent...
during the crystal growth. However, when a Ga melt in a BN crucible was heated at 1073 K and 5 MPa of N₂ in Na vapor, colorless transparent prismatic crystals of GaN grew on the crucible surface, as shown in Fig. 8(a). Figure 3(b) is a schematic drawing of the apparatus used for single crystal growth of GaN by using Na vapor. The boiling point of Na is 1156 K and Na has a relatively high vapor pressure at 973–1073 K. The Na vapor was probably taken into the Ga melt with N and formed a N-containing Na–Ga melt before nucleation of GaN on the surface of the BN crucible. As shown in Fig. 8(b), some prismatic single crystals included the Na melt and formed negative crystals. The shape of the negative crystals may suggest the equilibrium form of GaN single crystals at the growth temperature.

4. AlN powder synthesis

AlN and InN, like GaN, are also group 13 nitrides having the wurtzite-type structure. AlN has been used for the ceramic substrates of electronic devices due to its high thermal conductivity. Many methods of AlN synthesis have been reported, but AlN powder had not been prepared by the reaction of Al and N₂ at 973 K. However, almost 100% of the Al reacted with N₂ gas and formed AlN hexagonal platelet nano-powder (grain size: 20–50 nm) at 973 K from a mixture of Al, Na and NaN₃ which was sealed in a stainless steel tube.9 Below the melting point of Al (932 K), Al metal in a Na melt did not react with N₂.

The preparation of InN was also attempted, but no crystal could be obtained at 773–1073 K and N₂ pressure of 0.1–5 MPa. InN is unstable at these temperature and pressure conditions, as indicated in Fig. 6.40 On the other hand, the condition for AlN powder formation is in the AlN stable condition and far from the condition of the equilibrium N₂ pressure at the temperature. This may be related to the formation of AlN powder.

5. Single crystal growth of CrN, Mn₄₋ₓGaₓN and MnₓN

CrN, which crystallizes in the cubic NaCl-type structure, has been used for hard coating materials with excellent corrosion, oxidation and wear resistance. CrN single crystals cannot be grown from the melt because of decomposition into Cr₂N and N₂ at 1375 K at 0.1 MPa of N₂. During the studies of the effect of 3d element addition on the growth of GaN, Cr did not result in any change in the crystal morphology and size of GaN, but Aoki et al. found the formation of CrN with GaN.7,39 Regular octahedral crystals of CrN 75 μm in size were obtained at 1123 K and 3 MPa of N₂. Platelet CrN crystals with a size of about 200 μm were also prepared at 1073 K and 5 MPa of N₂.

Figure 9 shows temperature and N₂ pressure conditions for the formation of CrN. GaN crystals do not form, but CrN crystallizes under conditions of lower N₂ pressure and higher temperature. This corresponds to the absolute value of CrN formation free energy, which is greater than that of GaN. CrN was not prepared from Na and Cr in N₂ without Ga. According to the Cr–Ga binary phase diagram,40 the melting point of Cr is 2136 K and about 10 at% of Cr was dissolved in a Ga melt at 1073 K. This means that Ga acts as a flux to dissolve Cr into the melt.

The lattice constant of CrN refined with the powder X-ray diffraction pattern was \(a = 0.1467(8)\) nm at 298 K. The crystal structure was confirmed by single crystal X-ray diffraction with an \(R\) factor of 1.8%. A very sharp phase transition from paramagnetism to antiferromagnetism with a temperature range less than 3 K was observed at 290 K.

In the Mn–Ga–N system with Na, besides the formation of Mn-doped GaN, cubic single crystals of Mnₓ₋₄GaₓN (anti-perovskite-type, \(Pm-3m, a = 0.38886(9)\) nm) having a maximum size of 500 μm were obtained at 1023 K and 5 MPa of N₂ pressure.43 The crystals showed magnetic phase transitions from a ferromagnetic phase with a spin-glass-like disorder to an antiferromagnetic phase at 107 K and then to a paramagnetic phase at 270 K. The composition \(x\) was estimated to be 0.93 (Mnₓ₋₄Gaₓ₋₄N) based on previously reported information regarding the transition temperatures and compositions.44

Platelet single crystals of MnₓN 100 μm in size (\(P6₃/mmc, a = 0.28185(6), c = 0.45371(12)\) nm) were obtained at 973 K and 5 MPa of N₂ pressure from starting materials of Mn and In with Na.9 The In–Mn binary phase diagram exhibits the formation of a Mn-containing In melt at this temperature45 and In did not form InN under these conditions, as mentioned in the above section.

These results suggest that single crystals of other nitrides containing high-melting temperature metals could also be prepared by using a Na melt and a metal flux such as Ga and In.

6. Synthesis of \(\beta\)-SiC and a novel ternary carbide Ba₆Si₄C₂

Silicon carbide is a representative non-oxide material because of its excellent properties of high strength, hardness, corrosion resistance and stability at high temperature and in acid solutions.
Powder, dense polycrystalline bulk, porous bulk and single crystals of SiC are used as abrasives, structural materials, filters, and semiconductor devices. Many synthesis methods have also been reported for SiC powders, and the processing temperature of crystalline SiC formation is usually at or above 1273 K. However, it was found that $\beta$-type SiC could be prepared at 900 to 1000 K by reaction of Si and carbon with Na.\(^{10,11}\)

For the synthesis of $\beta$-type SiC powder,\(^{10}\) silicon and amorphous carbon (carbon black) or fullerene and Na were charged in a BN crucible in a Ar-filled glove box and the crucible was sealed in a stainless steel tube under Ar atmosphere as shown in Fig. 10(a). The tube was heated at 900–1000 K for 24 h and cooled in the furnace. The Na metal in the product was reacted with 2-propanol and ethanol. The resulting SiC powder was suspended in the alcohol and separated with a centrifuge. The powder was then rinsed with distilled water and the resuspended powder in the water was again centrifuged. Agglomerates of SiC particles 20–40 nm in size were observed by transmission electron microscopy. The $\beta$-type 3C cubic structure ($a = 0.438$ nm) was confirmed by X-ray powder diffraction and electron diffraction. SiC was not obtained by using graphite as a carbon source at 1000 K. NaSi intermetallic compound was crystallized in the crucible. No SiC formation resulted from heating Si and amorphous carbon or fullerene at 1000 K without Na.

SiC porous bulk was prepared by heating a shaped mixture of Si and carbon powders at 1000 K for 24 h in Na vapor (Fig. 10(b)), the original shape of the mixture being maintained.\(^{11}\) Na was removed by reaction with alcohol or evaporation in a temperature gradient furnace. The porosity of the SiC porous bulk prepared from a starting mixture with a Si:C molar ratio 1:1 was 70%. A photograph of the porous bulk and a scanning electron micrograph of the fracture surface are shown in Fig. 11. A surface area of 11–17 m$^2$/g and mesopore size distribution in the range of 2–10 nm were shown by a nitrogen adsorption technique. Porous bulk samples having a porosity of 75–80% were prepared by using carbon-rich starting mixtures. After formation of SiC with Na vapor and Na evaporation from the sample, residual carbon was removed as CO$_2$ by heating the product in air.

Figure 12 shows a photograph and a scanning electron micrograph of the $\beta$-SiC bulk prepared by heating carbonized balsa wood with Si and Na at 973 K for 24 h.\(^{46}\) The as-prepared sample was covered with Na which was removed by reaction with alcohol. The structure and texture of the original carbonized wood were well maintained. It was also possible to change activated granular charcoal into bulk $\beta$-SiC by heating in a mixture of Si and Na at 973 K.\(^{47}\) After the heating, Na was removed by evaporation. The density of the obtained SiC granular bulk sample was 69% of the theoretical density of $\beta$-SiC. The fracture stress of the bulk filed into a circular cylinder with the flat surfaces (diameter 6.5 mm) was evaluated to be 47 MPa by a compressive test at room temperature. The Vicker’s hardness of the bulk surface was about 1300 HV.

In previous studies, SiC wood ceramics were prepared by immersing carbonized wood in a Si melt (melting point of Si: 1230 K). However, it was found that $\beta$-type SiC could be prepared at 900 to 1000 K by reaction of Si and carbon with Na.\(^{10,11}\)

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1687 K). The obtained SiC wood ceramics often contain Si in the cavities. The low-temperature formation of SiC bulk from porous carbon bodies with a mixture of Si and Na suggested the existence of a Si-containing Na melt at 973 K.

The original motivation of our study was to prepare novel ternary carbides by using Na metal. By using Ba, carbon black or fullerene and Si with Na, binary compounds such as BaC2 and BaSi2 were prepared. Suzuki et al. prepared single crystals of a new ternary silicide carbide Ba3Si4C2 by using Na and β-SiC powder prepared by the Na flux method.88 Single crystal grains of Ba3Si4C2 about 10–40 μm in size were obtained by slow cooling from 1123 K at a rate of ~5.5 K/h. The crystal structure was determined by single crystal X-ray diffraction. The structure is isotypic with Ba3Si4C2 (perovskite CaTiO3 type structure) and crystallizes in a tetragonal cell (a = 0.87693(4) and c = 1.23885(6) nm, space group 4I4/mcm (No. 140)). Zintl anions of [Si4]4– and carbide anions of [C2]2– are contained at the sites with 6-fold and 12-fold Ba atom coordination. The [C2]2– dumbbell anions are disordered at the octahedral sites.

7. The role of Na for the nitrides and SiC formation

Na metal melts at 371 K and liquid Na boils at 1154 K under atmospheric pressure. No stable nitride was prepared for Na nor any other alkaline elements except Li3N. In 2002, sodium nitride Na3N was reported as a metastable phase.49 It decomposes at 360 K. Li2C2 is directly synthesized by reaction of Li with C. However, Na2C2 and other alkaline metal carbides cannot be prepared by direct reaction.50 They are synthesized by using H2C2 gas as a carbon source. According to the report by Huberstey, the solubility of nitrogen in liquid Na is extremely low (7.1 × 10–5 mol% at 873 K).51 He also reported a tremendous increase of the solubility by the addition of Ba and Sr to a Na melt. The increase was explained as being due to the presence of soluble species of Ba3N and Sr3N in the Na melt. All the nitrides and nitride-related compounds shown in Table 1 contain Ba and/or Sr. Much more nitrogen is dissolved in the Na melt with Ba than in the melt with Sr.51 For the synthesis of multinary novel nitrides and nitride-related compounds, it seems that the compounds containing Ba were prepared more easily than those containing Sr. The nitrides and nitride-related compounds probably crystallize via the soluble species in the Na melt.

As shown by the growth conditions of GaN single crystals, Na acts as a kind of catalysis which promotes nitridation at low temperatures.52 Na is an electro-positive atom and has a low ionization energy. The author speculated that N2 molecule adsorbed on the Na melt surface may receive an electron from Na.52 The bonding state of the N2 molecule is fully occupied and the lowest unoccupied molecular orbital is anti-bonding. The received electron probably enters the anti-bonding orbital, resulting in a decrease of the bond order of the N2 molecule. This means that the dissociation energy of the N2 molecule decreases. Without Na, large thermal energies at high temperatures are necessary to dissociate N2 molecules.

In this sense, Na may be a nitride formation enhanced medium, other atoms such as Ba, Sr, Ga, Al, Cr and Mn being needed to hold nitrogen in the Na melt for the single crystal growth of nitrides. In order to dissolve Cr and Mn metals into the melt below their melting points, Ga and In were added as fluxes for the single crystal growth of CrN and MnN.

In the case of silicon carbide formation, the role of Na seems to be different. SiC can be synthesized by using reactive carbon sources such as amorphous carbon (carbon black, carbonized wood, charcoal) and fullerene. The results of low temperature formation of SiC powder and bulk suggested the presence of a Si-containing Na melt. Recently, Morito et al. experimentally determined a Na–Si binary phase diagram, showing the presence of Na–Si melt in a wide composition range above 953 K.53 Single crystals of Si were obtained at 1173 K by Na evaporation (solvent evaporation) based on the information of the phase diagram. Yamada et al. have reported the low temperature synthesis of β-FeSi2 by using Fe powder and a Na–Si melt at around 1000 K.54

Elemental metals having low melting temperatures, such as Ga, In, Sn, Pb and Al, were used as fluxes for the crystal growth and low temperature syntheses of non-oxide materials of phosphides, borides, carbides, and intermetallic compounds.55 These conventional metal fluxes have high boiling temperatures. Therefore, the fluxes are dissolved in solutions such as hydrochloric acid or aqueous sodium hydroxide solutions. In the case of Ga and In fluxes, the crystals were separated from the fluxes by the centrifugation techniques. The boiling point of Na is lower than the boiling points of those conventional metal fluxes. This is both an advantage and a disadvantage. Na can be provided to the source materials from the vapor phase and can be removed by evaporation as shown in this review, and high-purity Na can be collected and easily reused. However, a closed or semi-closed system is needed for the preparation due to the high vapor pressure of Na.

8. Summary

The synthesis of nitrides and silicon carbide with a Na melt by the author and his coworkers was reviewed. The studies were initially intended to find novel compounds in the field of ternary nitrides and carbides, and some new compounds were prepared as single crystals. It was found that the synthesis method with Na was also effective to obtain single crystals of binary nitrides GaN, CrN, MnN and powder of AlN at temperatures lower than those of the conventional methods. It was found that SiC powders and SiC porous bulk could also be prepared at around 1000 K with a Na melt and vapor, respectively. The process with Na has a potential for wide application to the low temperature synthesis of silicides and other non-oxide inorganic materials in the future.

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References

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References:


