



Figure 1. Aluminum nitride powder and sintered components manufactured by Surmet (Burlington, Mass.).

# Manufacturing of aluminum nitride powder for advanced applications

By Mohan Ramisetty, Suri Sastri, and Uday Kashalikar

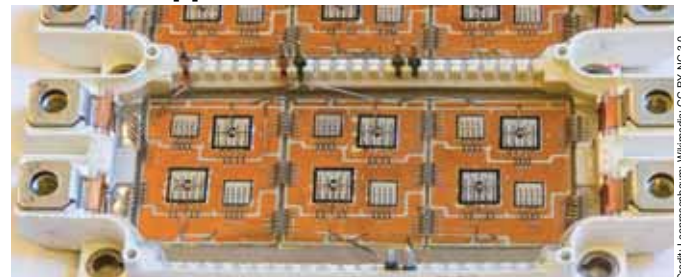
Production of aluminum nitride powder by direct nitridation or carbothermal reduction and nitridation requires balancing trade-offs between cost, carbon footprint, and properties.

**A**luminum nitride (AlN, Figure 1) is a synthetic ceramic with a unique combination of useful thermal and electrical properties. Chief among them are high thermal conductivity, excellent dielectric properties, low

coefficient of thermal expansion (close to silicon), nontoxicity, and chemical- and high-temperature resistance. Also, AlN is a high bandgap semiconductor, so there is growing interest developing around AlN powders for single crystal growth.

Although AlN has been known for more than a century, interest has renewed in the recent decade or so. AlN is more expensive than alumina, which is used for many substrate and dielectric applications. Alumina, however, cannot meet the requirements for many high-brightness LED and power

## Common applications for AlN



**Aluminum nitride is the preferred substrate for insulated-gate bipolar transistors, such as this one.**

- Sintered substrates and heat sinks: Circuitry carriers, sensor carriers, heat sinks for HB-LEDs, insulated-gate bipolar transistor (IGBT) modules, microchanneled coolers, and AlN/direct-bonded copper (DBC) manifold microchannels in power electronics packaging.
- Thermally conductive adhesives: AlN powder/granule fillers increase adhesive thermal conductivity for electronics, power electronics, and semiconductor packaging.
- Semiconductor chambers: Electrostatic chucks, evaporation boats, crucibles, plasma-resistant components, susceptors, and heaters for CVD and dry etching in semiconductor equipment.
- Dielectric and radio-frequency systems: Defense radio systems, radars, and output windows.
- Single crystal AlN for growing GaN devices and AlN for ultraviolet LED devices.

**Table 1. Important engineering properties of sintered aluminum nitride**

Property	Value
Typical thermal conductivity <sup>†</sup> (room temperature) (W/(m·K))	120–270
Electrical resistivity (room temperature) ( $\Omega\cdot\text{cm}$ )	$>10^{13}$
Coefficient of thermal expansion (/K)	$5.6 \times 10^{-6}$
Flexural strength (MPa)	200–370
Knoop hardness (at 200-g load) (kg/mm <sup>2</sup> )	1,300–1,500
Temperature stability in inert atmosphere ( $^{\circ}\text{C}$ )	$>1,900$
Chemical resistance to molten metals	High

<sup>†</sup>Depending on processing conditions, impurities, density, etc.



**Figure 2. Example applications that use aluminum nitride.**

electronics applications. The dramatic growth of these new technologies also drives growing demand for polycrystalline sintered AlN products. Moreover, the relentless push for faster speed, smaller footprint, and high power density of microprocessor chips in the optoelectronic and power electronic industries fuels demand for advanced heat-dissipation solutions.

AlN is an ideal material for applications needing electrical insulation and thermal conductivity (Figure 2). Alumina remains the material of choice for many high-volume–low-cost applications. However, constantly growing demand for miniaturization, long life, and high performance in the electronics and semiconductor industries for packaging, power electronics, inverters for transportation, telecommunications, cooling systems, high-brightness LEDs, and more

make AlN a very attractive solution.

Silicon nitride ( $\text{Si}_3\text{N}_4$ ) is another strong contender for applications that require better thermo-mechanical durability. Although the thermal conductivity of  $\text{Si}_3\text{N}_4$  is much lower ( $\sim 90 \text{ W/m}\cdot\text{K}$ ) than AlN, its high fracture toughness—because of its acicular microstructure, hardness, and strength—makes up for this deficiency. Availability and cost continue as barriers.

AlN has a hexagonal lattice (Wurtzite type) and predominantly covalent bonding between aluminum and nitrogen. Table 1 summarizes key properties that make AlN a useful engineering material.

Low atomic mass, simple crystal structure, strong interatomic forces, and low anharmonicity make AlN intrinsically thermally conductive, unlike typical ceramics.<sup>1</sup> However, thermal conductivity of polycrystalline

sintered AlN is influenced by microstructure; secondary phases, including porosity; impurities, such as oxygen and other cationic impurities; and processing-related factors.

Achieving good thermal conductivity and dielectric properties is directly related to processing. Similar to any other ceramic material, good control of process depends directly on properties of the raw materials. This particularly is true of AlN because impurity content and microstructure—which influence thermal conductivity—directly depend on quality of the starting powders that will be reacted to synthesize AlN.

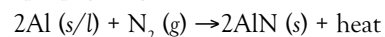
## Two approaches to manufacturing AlN powder

Figure 3 shows the interdependence of steps in ceramic powder processing and ultimate impact of processing on the final properties of sintered ceramic. AlN ceramic powder processing begins with powder synthesis. Properties of the starting powder, including purity, particle-size distribution and morphology, surface area, and bulk or tap densities dictate processing approaches, such as green forming and densification.

AlN powder can be synthesized via various approaches. However, direct nitridation (DN) and carbothermal reduction and nitridation (CRN) are the two most widely used routes for producing AlN powder in tonnage-scale quantities. Each offers advantages and disadvantages. Figure 4 illustrates steps involved in DN and CRN processes.

### Direct nitridation

AlN is a reaction product of aluminum metal and nitrogen formed via the self-propagating exothermic reaction.



The DN process typically involves ignition of aluminum powders in a nitrogen-containing atmosphere. The reaction is self-sustaining. Reacted product requires further processing via milling or classification to produce final powders.

### The advantages of DN synthesis are

- *Energy efficiency*—The exothermic reaction generates  $\sim 328 \text{ kJ}/(\text{mol AlN})$  at 1,800 K. Aluminum melts at 933 K

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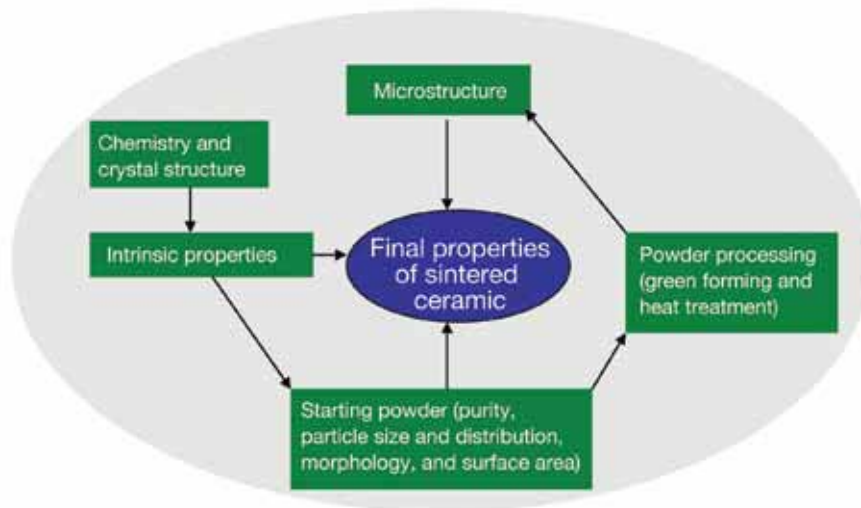


Figure 3. Property-processing diagram for ceramics.

and the reaction with nitrogen begins at  $\sim 1,073$  K, making it a self-sustaining process. Other than the initial ignition, which does not require much energy, no additional external heat is needed to sustain the reaction, making the process extremely energy efficient.

- *Environmental friendliness*—The reaction produces no known harmful byproducts or greenhouse gases.

The disadvantages are

- *Milling required*—Uncontrollable reaction sequence and exothermic temperature induce considerable neck-

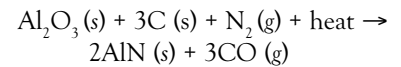
ing between AlN particles, resulting in agglomeration that is difficult to break up during milling. Consequently, multiple particle size reduction steps are necessary to achieve required particle sizes.

- *Impurities*—Milling steps can introduce potential impurities.

## Carbothermal reduction and nitridation

Typical precursors are aluminum oxide ( $\text{Al}_2\text{O}_3$ ) powders mixed with a source of carbon as a reducing agent. The precursors are combined and heat-

treated at  $1,400^\circ\text{C}$ – $1,800^\circ\text{C}$  in the presence of nitrogen or nitrogen-containing gas. The overall reaction is:



Theoretically, the reaction needs three moles of carbon for every mole of  $\text{Al}_2\text{O}_3$ . However, practical issues, such as surface area and mixing limitations, require a substantial amount of excess carbon ( $\sim 15\%$ – $30\%$  additional carbon) to promote full conversion to AlN.

The advantages of CRN synthesis are

- *Quality*—For the most part, precursor particle size and synthesis temperature determine final particle size and purity levels of CRN powders.
- *Minimal or less milling required*—Finer particle size is possible without excessive milling because of availability of fine precursor powders.

The disadvantages are

- *High cost*—Expensive precursors and energy-intensive multiple heat-treatment steps makes this powder very expensive compared with DN powders. Powder cost may not be a big factor in some advanced applications, such as growing single crystals for UV-LEDs, phosphors, and advanced microelectronic packaging. However, for applications such as HB-LEDs, CRN powders are simply unaffordable.

- *Energy intensive*—Production of high-purity  $\text{Al}_2\text{O}_3$  powders is itself an energy-intensive process requiring initial conversion of ore to metal and then purified metal into oxide. Added to that are the reduction and nitridation heat-treatment steps. Typically, synthesis occurs at temperatures well above  $1,400^\circ\text{C}$  and approaching  $1,800^\circ\text{C}$ , usually in graphite furnaces. Calcination is necessary to burn off excess carbon after synthesis.

- *Carbon footprint and environmental impact*—In addition to the carbon emissions that result from energy required for several heat-treatment steps, the reaction itself produces a significant amount of carbon (in the form of  $\text{CO}$  and  $\text{CO}_2$ ) as a byproduct. For every 100 g of AlN, the reaction produces  $\sim 160$  g of  $\text{CO}_2$ —not counting the excess carbon needed from practical considerations for complete conversion of  $\text{Al}_2\text{O}_3$  to AlN.

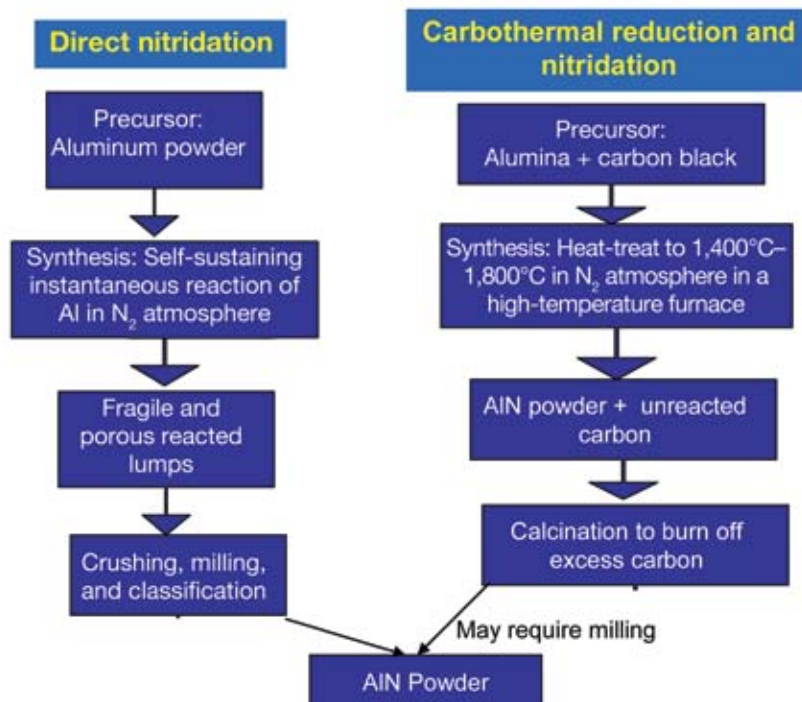


Figure 4. Process steps for synthesizing aluminum nitride powder via direct nitridation or carbothermal reduction and nitridation.



## AlN powder and sintered components manufacturing at Surmet

- **Powder:** Surmet (Burlington, Mass.) has capacity to produce tonnage quantities of AlN powder by direct nitridation and carbothermal reduction. Surmet manufactures and supplies several commercial grades of AlN powders.



- **Sintered components:** Surmet manufactures complex and large AlN sintered components for specialty applications, such as large domes (right), disks, and tiles up to 35-in. wide. Surmet's sintering technology has produced large AlN parts with >200-W/m-K thermal conductivity and good mechanical properties.



The common perception that CRN powders provide better properties for thermal management applications may not be justified. A brief literature review and considerable hands-on experience suggest that powder synthesis method has little impact on the final properties, including thermal conductivity of the sintered product.<sup>2,5</sup> What matters are properties of the AlN powder, such as purity, crystallinity, and particle size and distribution. Carefully engineered DN powders should perform as well or better than CRN powders as long as the powder properties are matched. It is admittedly more challenging to achieve finer particle size and high purity by DN, but, by using proper high-purity, fine-particle-size, large-surface-area aluminum precursor powders and closely controlling synthesis reaction rate and subsequent milling, high-quality DN AlN powder can be made economically in tonnage quantities.

### Sintering powders into products

The strong covalent bonding nature of AlN makes it difficult to achieve full densities via pressureless solid-state sintering. However, liquid-phase sintering is an

effective alternative approach. The liquid phase either solidifies along the grain boundaries or volatilizes at high temperatures without causing significant decreases in thermal conductivity. The most commonly used and suggested sintering aid in the literature is yttrium oxide ( $Y_2O_3$ ). Studies show that  $Y_2O_3$  scavenges the oxygen impurity (typically present as alumina) in the AlN powders. A yttrium aluminate compound forms along the grain boundaries (ideally at triple points) and promotes densification to near theoretical density.<sup>6</sup>

## References:

- <sup>1</sup>G. Partridge, "Ceramic materials possessing high thermal conductivity," *Adv. Mater.*, **4** [1] 51-55 (1992).
- <sup>2</sup>A.V. Virkar, T.B. Jackson, and R.A. Cutler, "Thermodynamic and kinetic effects of oxygen removal on the thermal conductivity of aluminum nitride," *J. Am. Ceram. Soc.*, **72** [11] 2031-42 (1989).
- <sup>3</sup>T.B. Jackson and A.V. Virkar, "High thermal conductivity aluminum nitride ceramics: The effect of thermodynamic, kinetic, and microstructural factors," *J. Am. Ceram. Soc.*, **80** [6] 1421-35 (1997).
- <sup>4</sup>R.-R. Lee, "Development of high thermal conductivity aluminum nitride ceramic," *J. Am. Ceram. Soc.*, **74** [9] 2242-49 (1991).
- <sup>5</sup>A. Franco Jr. and D.J. Shanafield, "Thermal conductivity of polycrystalline aluminum nitride (AlN) ceramics," *Ceramica*, **50**, 247-53 (2004).
- <sup>6</sup>S. Mitra, G. Dutta, and I. Dutta, "Effect of heat treatment on the microstructure and properties of dense AlN sintered with  $Y_2O_3$  additions," *J. Am. Ceram. Soc.*, **78** [9] 2335-44 (1995).

## About the authors

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