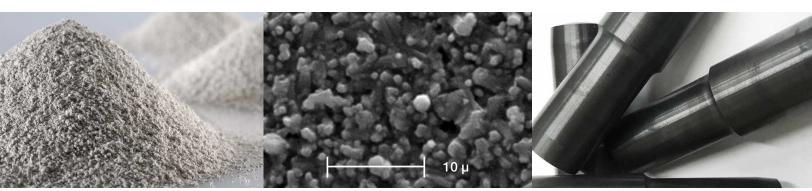


DIRECT PRESSURE SINTERED SILICON NITRIDE (Si3N4): BALANCING HIGH PERFORMANCE WITH COST EFFECTIVENESS



Microscopic photography showing the surface of Si₃N₄

OVERVIEW

Superior Technical Ceramics has introduced a new Direct Pressure Sintered Silicon Nitride powder-to-part component manufacturing process that provides a high-performance, cost-effective material solution as an alternative to both Reaction Bonded Silicon Nitride and Hot Pressed Silicon Nitride.

Silicon nitride (Si₂N₂) is a strong, lightweight, and commercially important non-oxide ceramic material. It was first fabricated in the mid-1800s, however it did not see widespread industrial use until approximately a century later, when advanced processing techniques became available and allowed the material to be utilized in a wide variety of industries and applications.

Raw silicon nitride powder has a gray color and is typically fabricated by exposing pure metallic silicon powder to high temperature nitrogen gas under pressure, although naturally occurring deposits have also been found as small inclusions in certain meteorites. Fully sintered (dense) silicon nitride has a dark gray to black coloration and component surfaces can be ground to a smooth polish. It is often utilized in demanding applications in which strength, wear resistance, fracture toughness, and dimensional stability are all required at high temperatures and/or in corrosive environments.

STRUCTURE, PROPERTIES, PERFORMANCE

Mechanical

One particular advantage of silicon nitride over other ceramic materials is its high strength-to-weight ratio, which compares favorably even with metallic nickel-based "superalloys". This strength is the result of the material's high-energy, predominantly covalent, bonding structure, which maintains its strength even at high temperatures (up to ~1000°C). The high fracture toughness of silicon nitride results from the presence of strong, acicular (needle-shaped) B-Si_aN, grains in the sintered microstructure, whose high tensile strength allows them to bridge cracks and slow crack propagation. The balance of the microstructure is comprised of a-Si_aN_a grains, which confers hardness and wear resistance to the material. Silicon nitride exhibits excellent tribological performance and is often utilized in wear applications.

STRUCTURE, PROPERTIES, PERFORMANCE (cont.)

Thermal

Silicon nitride also exhibits an unusually low coefficient of thermal expansion, again due to its high-energy covalent bonding. This is a potentially useful property for designers working with high temperature applications. The thermal conductivity of silicon nitride is comparable to high purity alumina and it possesses a higher thermal shock resistance compared to most ceramics due to its strength, toughness, and low thermal expansion properties. The maximum use temperature, dependent on strength requirements, stretches into the range of 1000 - 1400°C in an oxidizing atmosphere and beyond 1500°C in inert atmospheres. The material will dissociate into silicon and nitrogen at approx. 1850°C.

Electrical

As with most other ceramic materials, silicon nitride is an excellent electrical insulator and exhibits a higher dielectric strength compared to most high purity aluminas and zirconias.

COMPONENT FABRICATION TECHNIQUES

The strong covalent bonding of silicon and nitrogen atoms results in low diffusion coefficients on the surface of silicon nitride powder particles during the sintering process. As described above, the material also dissociates into silicon and nitrogen at 1850°C, which, combined with slow atomic diffusion at lower temperatures, inhibits complete densification of pure silicon nitride components during pressureless sintering processes. Therefore, fully dense sintered silicon nitride is typically obtained by applying nitrogen gas (or purely mechanical) pressure during firing and/or adding refractory glass-phase additives such as yttria (Y2O3), alumina (Al2O3), magnesia (MgO), or other rare earths combined with silica (SiO2) to the powder body in order to facilitate liquid-phase sintering.

While an exhaustive list of Si₂N₄ fabrication processes reaches beyond of the scope of this paper, the primary categories of industrial manufacturing approaches and the resulting material characteristics are summarized and compared here.

Reaction Bonded Silicon Nitride (RBSN)

» Process

Pure Si powder is isostatically pressed into a porous net-shape preform and then exposed to N2 gas at high temperatures, converting the Si to Si₂N₄ with minimal dimensional change. The nitridation reaction is exothermic and must be carefully controlled to avoid melting the silicon powder. This process may take up to several days.

» Result

Finished RBSN is a porous material, typically contains residual Si at the center of the powder particles, and exhibits poor mechanical properties compared to other finished materials in the Si, N, processing family. The material is easily machined into complex geometries after firing.

» Application

Due to the low cost of the raw materials (Si powder and N2 gas only) and the simplicity of the processing, this is an attractive option for applications with light performance requirements and/or for form-and-fit prototyping.

Sintered Reaction Bonded Silicon Nitride (SRBSN)

» Process

Quite similar to RBSN, except for the addition of sintering aids to the powder body and an additional sintering step (which may or may not include applied N2 gas pressure).

» Result

While the material and processing costs are increased, the resulting properties are superior to those of standard RBSN. However, the resulting material still typically contains residual Si and the material properties are still inferior to other Si₃N₄ fabrication options.

Typically used for applications with light to moderate performance requirements and/or form-fit-andfunction prototyping.

TYPICAL PROPERTIES* (SI | IMPERIAL)

• Hot (Isostatic) Pressed Silicon Nitride (H(I)PSN)

» Process

Pre-nitrided Si powder (pure Si_3N_4) is mixed with small amounts of sintering aids and pressed at extremely high pressure and temperature, either uniaxially or isostatically, to create a fully dense blank. Uniaxial pressing limits the component geometry to simple shapes but is typically faster (and thus cheaper) than isostatic pressing.

» Result

The resulting Si_3N_4 material generally has the highest densities and the most impressive properties of any current fabrication technique available, but the costs of the raw material, processing, and tooling are similarly high in comparison with the other options. Additionally, non-simple geometries will necessitate expensive post-fire diamond grinding.

» Application

Typically utilized for applications with unforgiving requirements and/or critical applications where cost is not a primary concern.

Sintered Silicon Nitride (SSN)

» Process

Pure Si_3N_4 powder is mixed with moderate amounts of sintering aids and pressed into a preform at lower pressure, either uniaxially or isostatically, but without applied heat (cold pressing), resulting in a "green" preform which can possess complex geometry. Additional geometric features can easily and quickly be machined into this green preform. The preform is then sintered (with or without N2 gas pressure) to achieve a fully dense component. Subsequent diamond grinding is also possible if extremely tight tolerances or fine surface finishes are required.

» Result

This fabrication method occupies an advantageous middle ground, in that the processing costs are decreased compared with the H(I)PSN method while the geometric flexibility of SRBSN processing is preserved. While the raw materials (pre-nitrided, ready-to-press $\mathrm{Si_3N_4}$ powder) are more expensive than those utilized with RBSN or SRBSN techniques, SSN processing eliminates the need for a nitridation step, drastically reducing overall processing time. Material produced with this technique contains no residual Si and the resulting densities and mechanical properties are only slightly inferior to H(I)PSN-produced $\mathrm{Si_3N_4}$.

» Application

The material of choice for applications with demanding performance requirements coupled with moderate cost constraints.

Direct Pressure Sintered Silicon Nitride (DPSSN)

Superior Technical Ceramics has developed a direct sintering process, which is a proprietary method based on the SSN technique described above. We utilize a brand new nitrogen gas pressure kiln and custom kiln furniture capable of sintering silicon nitride components up to approx. 8" in diameter. We believe that our material presents the optimal solution for applications with both stringent material performance requirements and reasonable cost constraints.

Our finely tuned sintering cycles provide consistent, state-of-the-art material performance and our top-tier facilities, equipment, and staff consistently deliver high quality, tightly toleranced component geometries ranging from simple to complex at both prototype and production quantities.

TYPICAL PROPERTIES* (SI | IMPERIAL)

	Property	ASTM Method	Units	Silicon Nitride (Si ₃ N ₄)
General	Crystal Size (Average)	Thin Section	Microns	4
	Color			Black
	Gas Permeability		atms-cc/sec	gas tight <10 ⁻¹⁰
	Water Absorption	C 20-97	%	0
Mechanical	Density	C 20-97	g/cc	3.25
	Hardness	Vickers 500 gm	GPa (kg/mm²)	15 (1529)
	Hardness		R45N	83
	Fracture Toughness	Notched Beam	MPam ^{1/2}	6
	Flexural Strength (MOR) (3 point) @ RT	F417-87	MPa (psi x 10³)	900 (130)
	Tensile Strength @ RT		MPa (psi x 10 ³)	537 (78)
	Compressive Strength @ RT		MPa (psi x 10 ³)	2500 (362)
	Elastic Modulus	C848	GPa (psi x 10°)	300 (44)
	Poisson's Ratio	C848		0.28
Thermal	C.T.E. 25 - 600° C	C 372-96	x 10 ⁻⁶ /C	2.9
	Thermal Conductivity @ RT	C 408	W/m K	29
	Max Use Temp (non-loading) (at high strength)		Fahrenheit (°F)	2552
			Celsius (°C)	1400
Electrical	Dielectric Strength (.125" Thick)	D 149-97A	V/mil	330
	Dielectric Constant @ 1 MHz	D 150-98		9.2
	Volume Resistivity, 25°C	D 257	ohms-cm	> 1 x 10 ¹⁴

Note: The information in this data sheet is for design guidance only. STC does not warrant this data as absolute values. Forming methods and specific geometry could affect properties. Slight adjustments can be made to some of the properties to accommodate specific customer requirements. Most of the dense materials in the table are resistant to mechanical erosion and chemical attack. STC has performed ASTM testing qualification for certain compositions, in accordance with ASTM D2442. Please consult our technical staff for appropriate material and specific test results. In addition to the above compositions, STC offers a wide range of alternative materials. Please contact one of our applications engineers for material requirements that may not be shown above.

PROCESS COMPARISON TABLE

Ranking (1=Best)	Overall Cost	Processing Time	Green Forming/ Machining Ability	Mechanical Properties	Applications
RBSN	1	4*	2	4	Form/Fit Prototypes Mild Requirements
SRBSN	2	3*	1	3	Functional Prototypes Moderate Requirements
H(I)PSN	4	2**	3	1	Critical Applications Extreme Requirements
SSN/DPSSN	3	1	1	2	Versatile Usage Demanding Requirements

^{*}Due to the slow nitridation process. **Excessive diamond grinding compared to DPSSN may be required.

Call or submit an inquiry on our website to speak with our helpful, friendly, and knowledgeable customer service representatives and application engineers. We will work collaboratively with you to solve your problems and discover ceramic material solutions that will fulfill your specific project requirements.