Overview of the production of sintered SiC optics and optical sub-assemblies, S. Williams, CoorsTek, Inc.; P. Deny, BOOSTEC Industries (France) [5868-04]
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Abstract:

The following is an overview on sintered silicon carbide (SSiC) material properties and processing requirements for the manufacturing of components for advanced technology optical systems. The overview will compare SSiC material properties to typical materials used for optics and optical structures. In addition, it will review manufacturing processes required to produce optical components in detail by process step. The process overview will illustrate current manufacturing process and concepts to expand the process size capability. The overview will include information on the substantial capital equipment employed in the manufacturing of SSIC.

This paper will also review common in-process inspection methodology and design rules. The design rules are used to improve production yield, minimize cost, and maximize the inherent benefits of SSiC for optical systems. Optimizing optical system designs for a SSiC manufacturing process will allow systems designers to utilize SSiC as a low risk, cost competitive, and fast cycle time technology for next generation optical systems.

Keywords:
SiC, silicon carbide, space optics

Background

Sintered SiC has been a useful material for several industrial applications for over forty years because of it's high strength, hardness, stiffness, polishability and thermal stability. In the last 10 years, new processing techniques have been developed so that this high performance material can be applied to highly complex and large optical systems. Previously, the high temperature processing requirements and unrefined forming techniques limited SSiC to small simple shaped components. The two primary applications for SSiC were seal rings, generally for automotive water pumps and armor where SSiC is a highly regarded material for its mass relative to ballistic resistance.

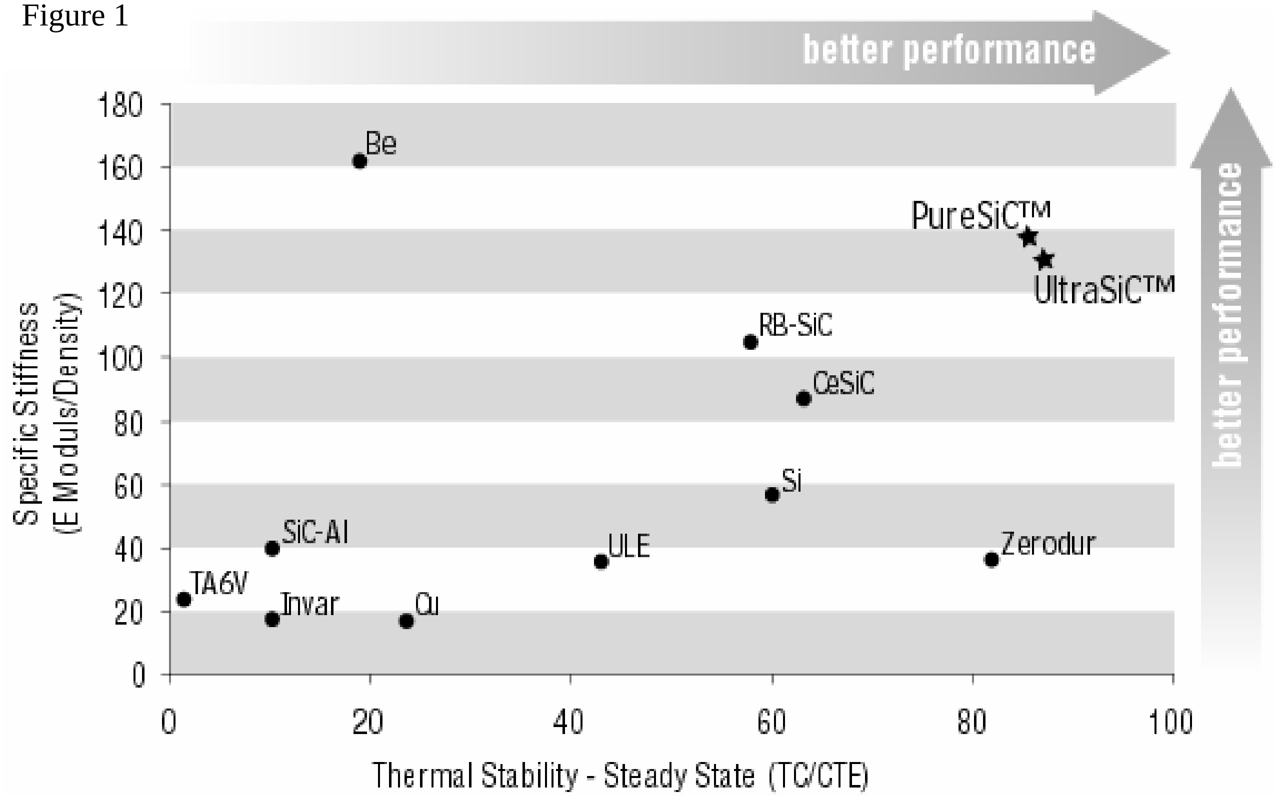
SSiC is one of many materials commonly referred to as silicon carbide. There are two general families of silicon carbides, single phase and multiple-phase materials. Single-phase SiC materials are chemical vapor deposition (CVD) SiC, hot pressed SiC, and sintered SiC. These materials are 99% to 99.9999% pure SiC. The multiple-phase materials include reaction bonded SiC, converted SiC, and CeSiC. These materials have SiC phases intermixed with phases of silicon and sometimes carbon.

Single-phase materials generally provide better and more predictable performance than multiple phase materials for optical applications. The material uniformity and purity of single-phase materials inherently exhibits higher repeatability and uniformity in mechanical properties and thermal properties. SSiC in particular been demonstrated to be completely isotropic.

Alternatively, multiple-phase materials inherently introduce internal stress during cooling after sintering due to the differing coefficients of thermal expansions of the different phases of materials. Thus, multiple-phase materials are subject non-uniform and less repeatable mechanical and thermal performance.

<u>Benefits of SSiC as a candidate material for optical applications</u>

Figure 1



The two primary reasons for using SSiC are its specific stiffness and thermal stability (Figure 1).

Other benefits of using SSiC in optical applications are as follows:

- Generic tooling - New designs can be produced rapidly with little to no tooling expense as the tooling required is generic in nature.
- Highly industrialized and predictable manufacturing process
- Material can be used for both optical and structural components thus making the complete optical loop athermal
- Polishing – SiC optics can be polished to comparable figure accuracy and surface roughness to any other optical materials.
- All typical optical coating processes designed for glass are compatible with SiC optics.
- Flight heritage for SSiC has been established.

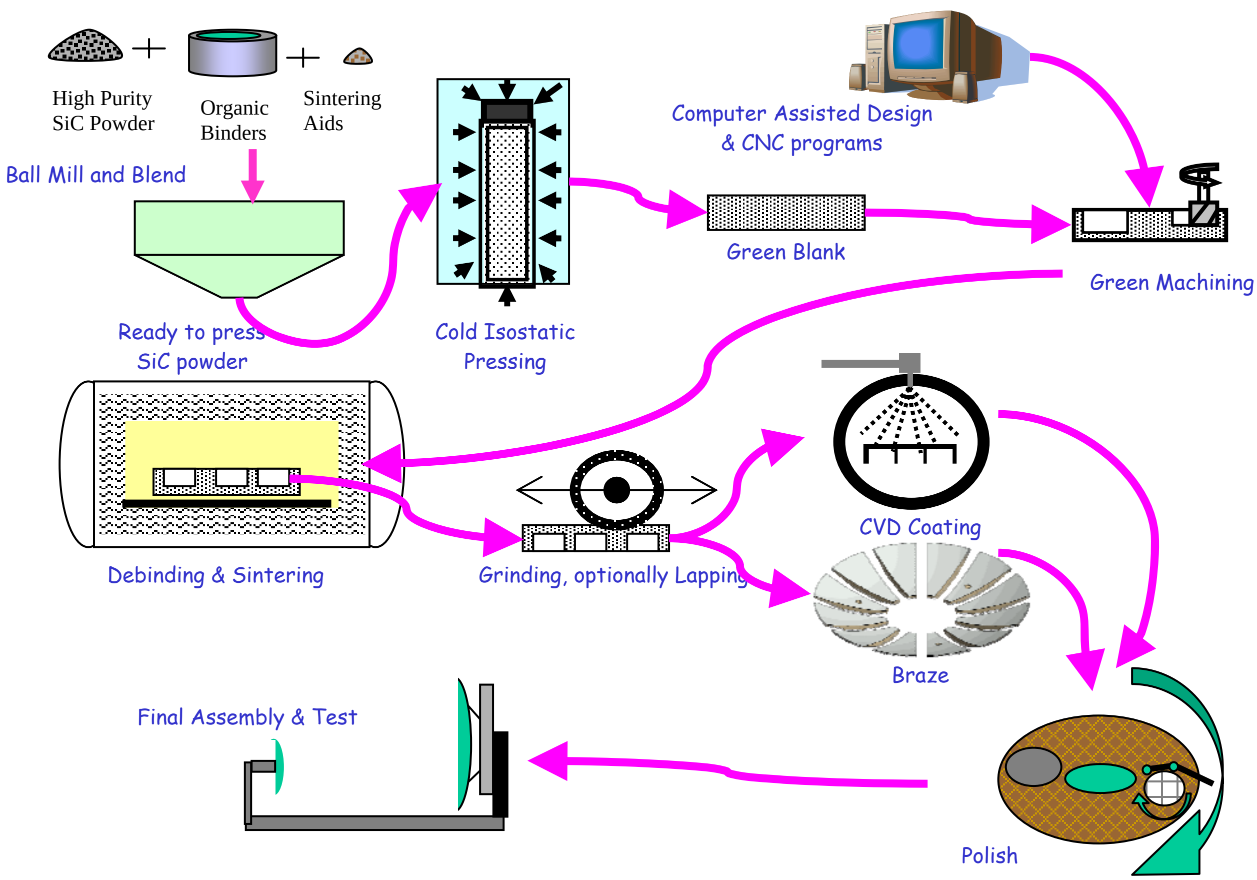


Figure 2 - General Manufacturing Process

<u>Powder production</u>
Sintered silicon carbide (SiC) raw material is produced by mixing high purity SiC with sintering aids and binder systems to aid in producing large-scale products from this fine-grained material. Initially, the powder is ball milled to a precise sub-micron size distribution. When the target particle size has been reached, sintering aids, typically boron carbide and phenolic resin, are introduced to the milling operation. The sintering aids enable the SiC to reach full density during sintering. These sintering aids comprise less than 1% by weight of the sintered material.

The powder processing operation is a well-refined, high volume material process. Companies, like CoorsTek, have material processing systems designed to process in excess of 100 tons of SSiC powder per year. High volume processing has been needed for applications including seals for automotive water pumps and armor tiles for defense applications. These applications require very high repeatability in material properties and processing attributes to maintain performance requirements and low cost. The process has been refined to produce consistent material properties over several years. The remarkable consistency in mechanical and thermal properties enables a highly useful and predictable material for optical applications.

The first manufacturing step for most SSiC optical products is isostatic pressing. This process involves filling a sealable container, typically a rubber bag, with the SSiC powder. The rubber bag is usually supported with a skeletal structure, typically a steel box, which has several perforations to allow the iso-static force media, generally water, to exposure to the full surface area of the bag. This toolset containing the bag of powder is placed into pressure vessel where it is subjected to hydraulic pressure as high as 30 thousand pounds per square inch (KPSI).

In some cases it is beneficial to have hollow shapes pressed into the press billet, such as an internal circular cavity to form a cylinder. For these types of features, a forming mandrel is used. The bag presses the material against the mandrel. After pressing, the forming mandrel is extracted, leaving a precise negative shape of the mandrel in the as-pressed billet. This is a very helpful technique for removing excess material that would only be machined away later.

Green Forming

Green forming, also known as green machining is the process of using manual or CNC machine tools for near net shaping the unsintered SiC to its final design. Near net shaping at the green state is generally applied when possible because the SiC machines 20 to 30 times faster in the green state when compared to the fired state. Although, diamond tooling is typically used in green machining, the feed rates and milling operations are quite similar to common techniques used in milling more common materials such as aluminum. Features are specified for machining in the green state if the feature tolerance allows a linear shrinkage of +/- 0.2% to 1%. In addition, most features are net shaped in the green state and require relatively quick post-sinter machining operation, to finish the feature to specification.

There are a few limitations in green forming sintered SiC. The material in the pressed state has mechanical strength of chalk. It is quite brittle and low strength in the green state. Thus, care must be taken when handling large thin sections. Maintaining machine tools is critical for machining the ever-increasing aspect ratios of today’s light weighting designs. Aspect ratios approaching 80 to 1 height to thickness have been achieved for light-weight ribs. Recently advanced machining techniques utilizing high speed (up to 20,000 rpm) 3 and 5 axis milling heads with harmonically optimized tools have reduced machine times as much as 70% when compared to machining capabilities used 5 years ago.

<u>Sintering</u>

Sintering techniques have been dramatically improved in the last 30 years. Newer furnace technology with computer controlled zonal elements, optimized gas flow, and vacuum environments allow much larger and more complex products to be sintered today than previously thought possible. The challenge is producing a uniform temperature during the sintering process that peaks in excess of 2100 C while maintaining an environment to allow binder removal and prevent oxygen exposure to the SiC.

SiC powder is highly vulnerable to oxidation at temperatures over 1000 C. Thus, it must be kept under vacuum or an inert atmosphere during the entire sintering process. The sintering process typically takes 20 to 120 hours depending on size and complexity of the load. During this process, there are 3 basic stages; binder burnout up until 500 C; densification (shrinkage of ~ 20%) up to 2100 C; then cool-down. This process must be precisely controlled. Not reaching full sintering temperature for the required time will produce a part that has not reached full density, and the material properties will be compromised. Over-sintering can cause enlarged grain growth throughout the part, which can be detrimental to material properties.

<u>Grinding</u>

Grinding sintered SiC is similar to grinding other types of ceramic materials. Fixed abrasive diamond wheels are effective at removing excess material. Keeping the wheel sharp and true will produce good results for surface finish and profile accuracy. Typically, grinding is done with a large flow of water-based coolant to prevent excess heat built up.

For optical applications, grinding should be avoided to maintain the ultimate strength of the material found in the as-fired surface. As with most ceramics, grinding leaves behind sub-surface damage that can lower the strength by 50% or more when compared to an as-fired or highly polished surface. However, lapping and polishing operations can remove the damage done by grinding and recover the strength. Care must be taken when grinding thin sections such as mirror face sheets. However, grinding sparsely supported mirror face sheets down to as little as 0.7 mm has been demonstrated.

Milling

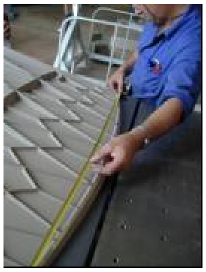
Features such as interface attachment holes and recessed mounting surfaces must be milled to precision geometric and dimensional requirements. CNC milling machines utilizing up to 5 axes typically perform these operations. Machining times during these operations are relatively slow operations. Only diamond tooling with substantial coolant is effective for milling operations.

Polishing

Most optical polishing techniques can be applied to SSiC optics. Conventional loose abrasive polishing with pads and pitch have been demonstrated as well as advanced techniques such as computer controlled polishing, ion beam figuring[i], and magneto-rheological finishing[ii]. Diamond is the only abrasive that is effective in polishing. Removal rates can be optimized for each process to be comparable with removal rates of more traditional optical substrate materials.

Inspection
There are several in-process and final inspection steps that are applied to SSiC optical projects. Most products have the following step, although many products have design specific testing and qualification requirements.

- Green body qualification – The raw material is qualified using test coupons for density, pore distribution, and strength



- As-pressed blank – The blank is visually inspected for cracks and measured for dimensional requirements

- As-sintered part – The sintered part undergoes a dye penetrant inspection for cracks and porosity

- Ground /machined parts – Dye penetrant inspection for cracks. CMM inspection for dimensional and geometric requirements.

- Proof testing – Final part may undergo proof testing and/or vibration testing prior to integration into the optical system.

Manufacturing Size Constraints.
Physical size constraints are typically restricted by the functional capacity of the capital equipment employed by the manufacturers of SSiC. The following table lists the theoretical size constraints at the time of publication in terms finished part size (after machining, shrinkage). Design features and dimensions determine actual size limitations.

Process	Size Limitation
Isostatic Pressing	Cylindrical: 1.0 meter Rectangular: 1.0 x 1.7 meters Rectangular: 0.6 x 2.0 meters
Green machining	Cylindrical: 1.5 meters Rectangular: 1.0 x 3.0 meters
Sintering	Rectangular: 1.4 x 1.8 meters
Grinding	Cylindrical 3.5 meters Rectangular: 1.0 x 5.0 meters
CVD SiC	Cylindrical 1.5 meters
Brazing	Cylindrical 3.5 meters

Workarounds on size limitations have been explored due the high cost of obtaining larger process equipment.

- Green joining techniques – Several approaches are being explored to produce larger green blanks than currently available isopresses are capable of producing. Such approaches involve bonding smaller components together either before green machining or post green machining. Boostec Industries has demonstrated one of these techniques in the manufacture of a 1 meter light-weight mirror blank[iii]. CoorsTek has used this approach for joining backplates on pocketed mirrors to produce closed back mirrors.

- Brazing – An alternative approach to producing large multi-meter class optics and/or optical structures has been demonstrated through brazing. The silicon based braze is used to produce a high strength thermally matched SiC to SiC joint.[iv, v]

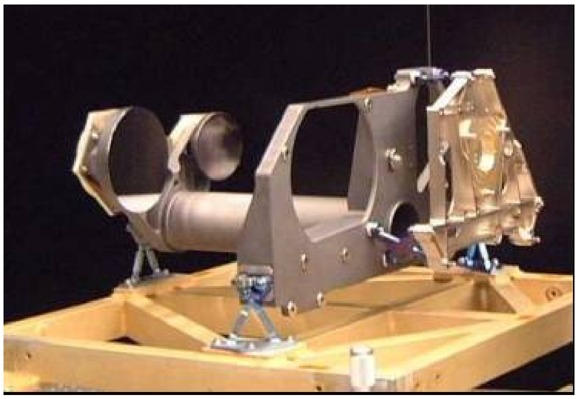
Mechanical Design Rules

The following are general design guidelines for manufacturing SSiC components. Actual product design parameters have many factors that determine prudent design rules, and should be discussed on case-by-case basis to assure an optimal design.

- Thickness > 0.5, < 20 mm
- Keep thickness transitions to less than 8 to 1 ratio
- Web thickness minimum 0.5 mm aspect ratio (height to thickness) 80 to 1
- Exterior chamfers 0.5 to 2 mm
- Fillets > 1.5 mm for bottom of pockets
- Fillets > 3 mm for vertical
- As-fired (no post sinter machining) +/- 1% linear tolerance for pockets and non-critical features

Examples of Space Qualified Sintered SiC Optical Products

ROSETTA
•Three mirror anastigmat (TMA) telescope
•Focal length 700 mm
•Weight less than 5 kg
•Optical quality (WFE) 30 nm



ROCSAT2 : Earth observation
•Cassegrain telescope diameter 600 mm
•Focal length 2896 mm
•Weight 55 kg
•Optical quality (WFE) 40 nm



Conclusion
Sintered SiC optical products are production ready have demonstrated many attributes that compare favorably to traditional optical materials in most applications. It is expected, in the next few years, SSiC will be space qualified in several new applications. The obvious benefits and design flexibility will allow new systems to reach previously unattainable system performance while keeping schedule and cost competitive.

[i] M. Fruit, A. Schindler, T. Hansel, "Ion Beam Figuring of SiC Mirrors Provides Ultimate WFE Performances for Any Type of Telescope," 2000.

[ii] A. Shorey, "Magneto-Rheological Finishing (MRF) of Large and Lightweight Optics," Mirror Tech Days, Marshall Space Flight Center, Huntsville, Al, August, 2004

[iii] D. Castel, D. Denaux, K. Mercier, E. Riviere, M. Bougoin, "New Technology for Manufacturing Very Large SiC Mirrors," CNES, Toulouse, 2002.

[iv] P. Deny, "A New Generation of Large Silicon Carbide Telescopes for Space Applications," SPIE Optical Science and Technology, August 2004, Denver, CO

[v] P. Antoine, M. Fruit, "SiC Telescope Demonstrator (Mirrors& Structure) Opto-mechanical Perfomances", Toulouse, France, 2000