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NTSIC(NEW TECHNOLOGY SIC): THE PROGRESS OF RECENT TWO YEARS

Katsuhiko Tsuno⁽¹⁾, Hiroshi Irikado⁽¹⁾, Kazuhiko Oono⁽¹⁾, Shoko Suyama⁽²⁾, Yoshiyasu, Itoh⁽²⁾

(1) NEC TOSHIBA Space Systems, Ltd., 10, Nisshin-cho 1-chome, Fuchu, Tokyo 183-8551, JAPAN,
Email: tsuno.katsuhiko@ntspace.jp, ono.katsuhiko@ntspace.jp, irikado.hiroshi@ntspace.jp

(2) Metal and Ceramics Technology R&D Department, Power and Industrial Systems Research and Development Center,
Toshiba Corporation, 2-4, Suehiro-cho, Tsurumi-ku, Yokohama, 230-0045, JAPAN,
E-mail: shoko.suyama@toshiba.co.jp, yoshiyasu.ito@toshiba.co.jp

ABSTRACT

New Technology Silicon Carbide (NTSIC®) is a reaction sintered silicon carbide with very high bending strength. Two times higher bending strength than other SiC materials is important characteristics in an optical mirror for space application. The space optics is to endure the launch environment such as mechanical vibration and shock as well as lightweight and good thermal stability of their figure. NTSIC has no open pore. It provides good surface roughness for infrared and visible application, when its surface is polished without additional coatings. Additional advantages are in the fabrication process. The sintering temperature is significantly lower than that of a sintered silicon carbide ceramics and its sintering shrinkage is less than one percent. These advantages will provide rapid progress to fabricate large structures and will enable that one meter mirror will put practical use. The fabrication capability has developed from 250mm to about one meter in these two years, after previous report of NTSIC. It is concluded that NTSIC has potential to provide large lightweight optical mirror.

1. INTRODUCTION

SiC is widely used in semiconductor production equipment parts as well as mechanical parts, since it is very stable in high temperature and it is contamination free material for silicon semiconductor process. SiC is ceramics with remarkable properties, high stiffness, high thermal conductivity, very hard and high melting point, SiC has been proposed as space optical applications in recent years. In space application, thermal stability of the optical system is very important, since the thermal control of the spacecraft and space borne equipment is one of the major issues. Usually glasses with low coefficient of thermal expansion (CTE), such as ULE and Zerodure, have been adopted to avoid thermal deformation of the space optics. Such glasses are heavy and fragile. On the other hand, beryllium is also widely used as lightweight optical mirror. Beryllium is used for less accurate application than low CTE glass due to its large CTE, though it is very light metal without fragility.

Material properties of SiC as well as ULE, Zerodure and Beryllium are summarized in Table 1. Even though the CTE of SiC is about two orders larger than low CTE glasses, it has better thermal stability than the low CTE glasses and beryllium in following parametrical manner. The steady state distortion (κ/α in Table 1) expresses the magnitude of distortion under The transient distortion (α/D) same heat flow. expresses the magnitude of relaxation time scale after transient heat input. High specific stiffness (E/p) and above thermal stability will make SiC best candidates to replace low thermal expansion glass and beryllium in the optical mirror application. Though several different SiC ceramics manufacturing process are developed, for example pressureless sintering (S-SiC), gas pressure sintering, hot pressing, hot isostatic pressing, reaction sintering (RS-SiC), chemical vapor deposition (CVD-SiC) and carbon fiber-reinforced SiC (C/SiC), a few methods have been applied for large scale optical mirror fabrication. Typical light weighted SiC optical mirror fabrication process are thought to be S-SiC^[2], RS-SiC CVD-SiC^{[3][4]} and C/SiC^[5]. Though CVD-SiC has very good surface for polishing, CVD is very slow and expensive process. Other processes may generally include complicated fabrication steps to obtain good optical surface, for example sintering, machining, CVD and polish. New attractive fabrication process, high-strength reaction-sintering process^[6], has developed to obtain high-performance SiC optical substrate. Three and half years study in TOSHIBA and NEC TOSHIBA Space Systems revealed that highstrength RS-SiC for space application, we call New Technology SiC (NTSIC) has potential to replace low thermal expansion glasses and beryllium in large light weighted optical mirror.

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Table 1 Material	properties	of various	optical	substrate

Material Property	Units	SiC(Typical)	Beryllium	Zerodure	TIN	Pyrex (Bolosillicate)
Density(p)	kg/m ³	3100	1840	2520	2200	2230
Young's Modulus(E)	GPa	420	303	92.9	67	64
Poisson's Ratio(v)		0.25	0.12	0.24	0.17	0.2
Ultimate Tensile Strength	M Pa	400		57		
CTE(a)	10 ⁻⁶ K ⁻¹	2.2	11.5	0.05	0.03	3.25
Specific Heat Capacity(Cp)	J/kg/K	680	1925	821	778	726
Thermal Conductivity(K)	W/m/K	140	216	1.64	1.3	1.13
Specific Stiffness(E/p)	10 ⁹ Nm/kg	0.135	0.165	0.037	0.030	0.029
Thermal Diffusivity(D=κ/Cp/ρ)	10 ⁻⁶ m ² /s	66.4	61.0	0.8	0.8	0.7
Steady State Distortion(α/κ)	10 ⁻⁶ m/W	0.016	0.053	0.030	0.023	2.876
Transient Distortion(α/D)	s/K/m ²	0.033	0.189	0.063	0.039	4.656

2. NTSIC CHARACTERISTICS

NTSIC has following characteristics and advantages which are come from its fabrication process and material properties.

- 1) Near-net shape fabrication
- 2) Visible grade polished surface without additional coating
- 3) High bending strength

2.1 NEAR-NET-SHAPE FABRICATION

NTSIC has small sintering shrinkage less than $\pm 1\%$. This small sintering shrinkage is come from reaction sintering fabrication process. The Fabrication process NTSIC is shown in Fig. 1 and manufacturing facilities for every step are shown in Fig. 2.

- 1) Carbon, SiC powder and some dispersand are mixed and spray-dried.
- 2) The green body is formed by cold pressing
- 3) The green body is machined to form product shape.
- The machined green body is reaction-sintered at about 1700K in vacuum with the contact of molten silicon.
- 5) Some part of the infiltrated silicon turns SiC by reaction with the carbon in the green body and unreacted silicon remains as residuals.
- 6) The reaction sintered SiC is mechanically finished to be final shape.

The process is very similar to conventional RS-SiC (hereafter, we simply call RS-SiC), S-SiC and C/SiC. The NTSIC process has very small sintering shrinkage, since expansion of Si+C reaction has balanced to sintering shrink. Therefore the shape obtained at above green body machining provides very close shape to final product, i.e. near-net-shape fabrication, whereas S-SiC has shrinkage from fifteen

to twenty percent. The near-net-fabrication has many advantages;

- NTSIC product is expected to be as large as full size of machining and furnace capacity, though S-SiC is expected to be about eighty percent of facility limit.
- Small deformation during sintering is expected to be good production yield especially in case of large scale products.
- 3) Rapid production due to fast machining of the soft green body than glass material is suitable characteristics to shorten manufacturing period of large aperture optics.

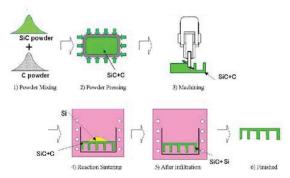


Fig. 1 Fabrication Process of NTSIC



Fig. 2 Manufacturing Facilities of NTSIC

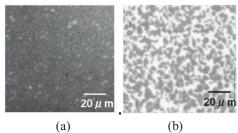


Fig. 3 Optical microstructure of polished surface.
(a) NTSIC, (b) RS-SiC

2.2 High strength

The material parameters of NTSIC are shown in Table 2. NTSIC has remarkable property on its bending strength, a few times higher value than RSSiC and S-SiC. The details are described in separated paper9. The bending strength of NTSIC has been controlled from 400MPa to 1200MPa^[6] by the raw material and other process parameters. The 800 MPa class NTSIC are studied for light weighted mirror according to the trade off between the performance and process control complexity.

The higher bending strength is very important for the space optics, because they should tolerate launch vibration and shock as well as change of thermal condition in the orbit. The mirror mount shall be so soft as to absorb the thermal expansion mismatch between mirror and optical bench. On the other hand, it shall be so hard as to endure launch environment, because the soft mount will decreases the stiffness of the optics and the mechanical load will be increased by the resonance. The higher bending strength enables to be more lightweight design with thin ribbed structure. The mass of the mirror become smaller, the mount is softer and smaller. So the simple kinematic mount will be employed and the optical system will be simple and lightweight. Fig. 4 shows the application point of current NTSIC technologies listed below.

- 1) Thinned skin structure down to 3mm thickness to form mirror surface.
- 2) Ribbed structure up to 100 mm height with 3 mm thickness.
- 4) Visible grade polished surface better than a few nano meters roughness without CVD coating.
- Kinematic mirror mount compatible to a few tens G launch vibration and joining method to NTSIC

Table 3 shows the sample design to demonstrate NTSIC capabilities. The mirror is designed to tolerate vibration with 20G using simple bipod kinematic mount. The first resonant frequency is 189Hz under condition that the mounting surface of the base of the kinematic mounts is rigidly held.

Table 2 Properties of NTSIC

Density	3030 kg/m^3
Yung's Modulus	360GPa
Poisson Ratio	0.18
Fracture Toughness	3.0MPam ^{1/2}
Bending Strength	850MPa
CTE	2.5×10 ⁻⁶ K ⁻¹ (@300K)
Specific Heat Capacity	680J/kg/K
Thermal Conductivity	130(W/m/K)
Hardness	2000 Hv
Solar Absorption (α)	0.74
Infrared Emittance (ε)	0.68

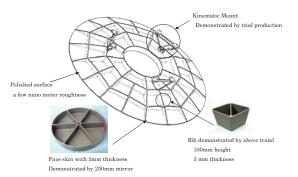


Fig. 4 Sample Design of Light Wight NTSIC Mirror

Table 3 Parameters of the Sample Design of Light Wight NTSIC mirror

Diameter	710mm	
Focal Length	796mm	
Apperture Ratio	1.12	
Maximum Rib Hight	30mm	
Weight	5.1kg	
Areal Density	14.0 kg/m ²	
Maximum Load	20G	
Lowest Natural Frequency w/o mirror mount	525Hz	
Lowest Natural Frequency w/ mirror mount	189Hz	

2.2 Surface Property

S-SiC has open pore on its surface and its polished surface has significant scattering in the visible region du to the open pore. So CVD coating is usually applied for the visible application. C/SiC and RS-SiC as well as NTSIC may have no open pore. C/SiC has residual carbon fiber, which does not react with silicon. Its material inhomogeneity may prevent to obtain good polished surface. Therefore it may be coated by silicon and silicon carbide slurry before polishing.

NTSIC is a kind of RS-SiC and they are the same composition, i.e. silicon and silicon carbide. Apparent and important difference of NTSIC form RS-SiC is the fraction of residual silicon.

Fig. 5 shows the microscopic photos of polished surfaces of NTSIC in comparison with that of RS-SiC.

Though the RS-SiC surface can be observed that SiC particles are embedded in silicon matrix, NTSIC surface can be observed that very small silicon particles are distributed in SiC matrix. The polishing the material with silicon and SiC mixture is somewhat difficult than S-SiC material, since silicon and SiC have different rubbing off speed. But NTSIC seems to be more suitable material for polishing than RS-SiC, since it seems be to more uniform than RS-SiC in scale of micro meter. Some optical fabrication companies tried to conventional optical polishing on NTSIC with diamond slurry and have good result as shown in Fig. 6. The surface roughness better than one nano meter is achieved without any difficulty and found small silicon inclusions are dented in a few tens nano meters. Therefore we can conclude conventional polishing is effective method to make optical surface with visible application.

Fig. 7 shows the result of trial polishing of lightweight mirror of 250mm diameter with 3 mm skin and 6 radial ribs. About 60nm rms surface accuracy after removing power are obtained using conventional Oscar type polishing machine. The result shows that the deformation due to the polishing pressure is very small comparatively large triangular cell size of about 125 mm formed by ribs. Maximum displacement of the thin surface supported by ribs with separation of is expressed by relation $W \max \infty Pa^4 / Eh^3$, where P, E and h denote pressure, Young's modulus and thickness of the surface material respectively. This accurate surface mainly thanks to high Young's modulus of SiC. The structure and size of the ribs are designed to be desired result.

Numerical controlled polishing will also produce much more accurate surface. Further study for accurate processing with ion beam figuring (IBF) was evaluated and found significant surface roughness degradation due to removing speed differences between silicon and SiC. IBF may useful when very small surface modifications.

Magnetorheological Finishing (MRF) is now tried to identify suitable condition.

The other candidate is Electolytic In-Process Dressing (ELID) Grinding Technique^{[7][8]} which is unique method to finish the visible grade surface without polishing and is expected to be rapid and low cost operations.

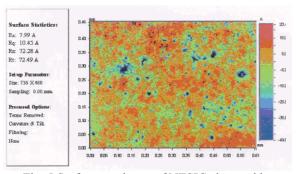


Fig. 5 Surface roughness of NTSIC observed by microscopic interferometer. The roughness less than 1 nm is smooth enough for visible application.

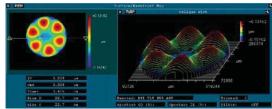


Fig. 6 Polished results of lightweight mirror demonstrator "Pathfinder 250mm plane mirror"

3. CURRENT DEVELOPMENT STATUS AND FURTHER WORK

The first goal of the NTSIC development is that we can fabricate the NTSIC mirror of about one meter aperture. First two years of this work we have developed the above element of the lightweight technique, thin surface and thin ribs with 100mm height and demonstrated the technology by 250mm light weight mirror^[1]. Following two years, we have great progress in size from 250mm to 650mm shown in Fig. 8 and Fig. 9. We will have large fabrication facility compatible to over one meter size mirrors in a few years. Continuous work based on above technologies will lead us to our first goal in a few years.

One of the useful application of NTSIC is a cryogenic optics, such as Herschel and SPICA projects, since SiC has large thermal conductivity and low coefficient of thermal expansion (CTE), especially nearly zero CTE lower temperature than 80K. First measurement of CTE of NTSIC shows similar behaviour to the other SiC materials. Further study of homogeneity of cryogenic CTE will follows.

In the next step, NTSIC development will be enhanced to many way, for example more light, lager, athermal telescope and so on. When the design and analysis result of present mirror is reviewed, there is large design margin to fracture in almost all areas. In a word, the feature of very high strength of NTSIC cannot be fully utilized in current design. This margin can be turned to lightening. NTSIC is too hard to grind in short time, so it is not efficient to thin large SiC

product after reaction sintering. Therefore effort to make thinner green body before reaction sintering shall be continued. The lighter mirror at same size shall be available by this study. The goal of the areal density is less than $10~\text{kg/m}^2$.

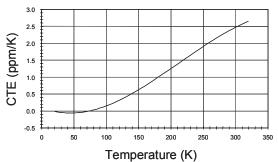


Fig. 7 Temperature dependence of CTE

On the other hand, thinner process is not unnecessary for much lager mirror than one meter, since higher stress is applied in larger and heavy structure. It is not more difficult to prepare large-scale facility such as furnace because the sintering temperature of NTSIC is about 1400 degrees Celsius. However, the serious problem arises that the large fragile green body may be damaged while they are processed. Logistics of the fragile body with size of a few meters and with thickness of a few millimeters is awful.

Alternative way to have large scale mirror is bonding small pieces into larger objects to minimize handling large fragile objects. The machined green bodies are adhered by glue and it is reaction sintered as shown in Fig. 10. In that process, both green bodies and glue turn to reaction sintered SiC (NTSIC), which we call reaction bonding or reaction joining. Therefore the bonded materials expected to be as strong as normal NTSIC. Figure 8 shows the bonded NTSIC which shows very uniform structure. Especially there are no visible boundaries between original green body and adhesive. This reaction bonding demonstration for small test pieces will be the first step toward large scale space optics beyond the a few meters.

We also study athermal optics which is thought to be an ideal optics design. Silicon carbide including NTSIC has finite CTE. Though the deformation of figure will be small enough due to high thermal conduction, significant change of mirror power may be occur due to mirror curvature change. The athermal design, where all telescope mirrors and structures are materials with same CTE, is thermally stable, when temperature change is uniform in whole telescope. The study of the optical telescope design shown in Fig. 12 revealed that following issues are identified as next key technological elements to be studied;

1) The structural elements with complicated shape.

- Mechanical joining of structure with high thermal conductivity.
- Mirror mount with mechanically stability and high thermal conductivity

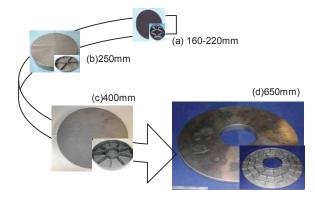


Fig. 8 Large Mirror Development Heritage

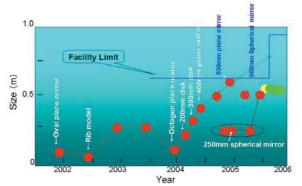


Fig. 9 History of Large Mirror Development

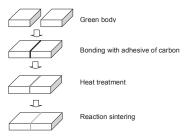


Fig. 10 NTSIC Joining Process

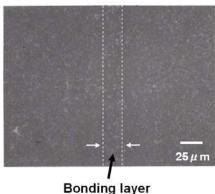


Fig. 11 Joined NTSIC

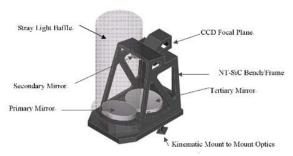


Fig. 12 Study of Athermal Optics

4. CONCLUSION

According to extensive NTSIC study shows that it is one of the best mirror materials for space by following result.

- 1) NTSIC surface without CVD coating is polished to provide good mirror from infrared to visible region.
- 2) Lightweight mirror with 650mm diameter is now available and one meter class will be available in near future.
- 3) Lighter NTSIC mirror with thinner design can be available in near future.
- 4) NTSIC has characteristics suited for larger application which will require bonded mirror, which is enhanced by similar bending strength to normal NTSIC.
- 5) Athermal telescope by NTSIC is worth to study and develop

NTSIC development will be continued to have lighter, larger mirror and optics and will be proposed for Japanese future programs^{[9][10]} and others.

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