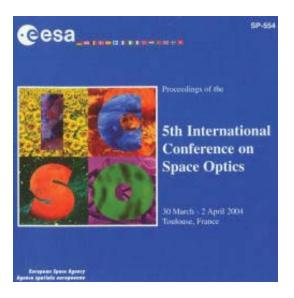
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### **REACTION-SINTERED SILICON CARBIDE: NEWLY DEVELOPED MATERIAL FOR** LIGHTWEIGHT MIRRORS

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#### ABSTRACT

Newly developed high-strength reaction-sintered silicon carbide is an attractive material for lightweight optical mirror with two times higher bending strength than other SiC materials. The polished surface has no pore and is suited to visible region as well as infrared without CVD SiC coating. The fabrication process, with low temperature and small shrinkage, is also suited to develop large scale objects.

#### 1. INTRODUCTION

Silicon Carbide (SiC) has been proposed for space optical applications in recent years as well as semiconductor production equipment parts and fusion reactor structural application. Its unique material properties, high thermal stability ( $\lambda/\alpha$ ; thermal conductivity to CTE ratio) and high stiffness ( $E/\rho$ ; Young's modulus to density ratio), will make SiC best candidates to replace low thermal expansion glass in the optical mirror application. The material properties of the typical optical substrate are listed in Table 1 and SiC is obviously thought to be one of the best materials. Though several different SiC ceramics manufacturing process are developed, for example pressureless sintering, gas pressure sintering, hot pressing, hot isostatic pressing, reaction sintering, chemical vapor deposition (CVD), 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 pressureless sintering (PLS-SiC)[1], reaction sintering (RS-SiC), CVD-SiC[2][3] and C/SiC[4]. These 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[5], has developed to obtain high-performance SiC optical substrate. Two years study in TOSHIBA and NEC TOSHIBA Space Systems revealed that high-strength RS SiC, we call New Technology SiC (NT-SiC) has potential to replace low thermal expansion glass and beryllium in large light weighted optical mirror.

Table 1 Pro	perties of Miri	ror Substi	rate Mate	rial		
Material Property	Units	SiC(Typical)	Beryllium	Zerodure	ULE	Pyrex (Bolosillicate)
Density(p)	kg/m <sup>3</sup>	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	MPa	400		57		
$CTE(\alpha)$		2.2	11.5	0.05	0.03	3.25
Specific Heat Capacity(Cp)	J/kg/K	680	1925	821	778	726
Thermal Conductivity( $\kappa$ )	W/m/K	180	216	1.64	1.3	1.13
Specific Stiffness(p/E)	10 <sup>-9</sup> kg/N/m	7.4	6.1	27.1	32.8	34.8
Thermal Diffusivity( $D = \kappa / Cp / \rho$ )	$10^{-6} \text{m}^2/\text{s}$	85.4	61.0	0.8	0.8	0.7
Steady State Distortion( $\alpha/\kappa$ )	10 <sup>-6</sup> m/W	0.012	0.053	0.030	0.023	2.876
Transient Distortion( $\alpha$ /D)	$s/K/m^2$	0.026	0.189	0.063	0.039	4.656

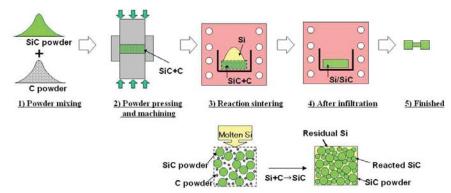


Fig.1 Flowchart of fabrication process for NT-SiC

#### 2. New Technology REACTION-SINTERED SiC

NT-SiC fabrication process is shown Fig. 1.

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

Apparent difference of NT-SiC form commercial RS-SiC is the fraction of residual silicon. Fig. 2 shows the polished surfaces of NT-SiC in comparison with that of commercial SiC. Though the commercial RS-SiC surface can be observed that SiC particles are embedded in silicon matrix, NT-SiC surface can be observed that small silicon particles are distributed in SiC matrix. The NT-SiC has a few times higher bending strength than commercial RS-SiC as shown in Fig.3. The bending strength of NT-SiC has been characterized by the particle size of residual silicon as well as the fraction of silicon. These parameters are controlled by the size of the SiC and carbon powders and the carbon to SiC ratio, and the bending strength can be controlled from 400MPa to 1200MPa [5]. The 800 MPa class NT-SiC are studied for light weighted mirror. The NT-SiC has many advantages in comparison with other type of SiC, for example;

- The NT-SiC is composed of SiC and Si and it has no micro pores on its surface. Its optical surface roughness of a few nm may be achieved by polishing without CVD coating.
- The sintering temperature is significantly lower than the pressureless sintered SiC of sintering temperature 2300K.

- The sintering shrinkage of NT-SiC is less than 1 %. This characteristic is thought to be great advantage to fabricate precise structures and the production yield.
- The bending strength of 800 MPa is about 2 times higher than the commercial SiC, whereas other parameters are similar to other type. Higher strength material above 1000MPa may be utilized in future.

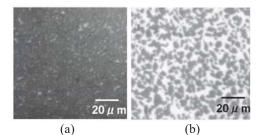


Fig 2.Optical microstructure of polished surface. (a) NT-SiC, (b) commercial RS-SiC

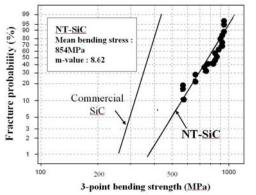


Fig.3 Weibull probability plot for 3-point bending tests

#### 3. LIGHT WEITHTED MIRROR DESIGN

The study of fabrication of the ultra light weighted and low cost mirror has been initiated according to these advantageous characteristics. After basic fabrication trial for thin surface, thin libs and other structures, the thickness less than 3 mm and ribs higher than 100mm are available. Pathfinder, 250mm diameter flat mirrors to demonstrate the thin open back structure, has been fabricated and evaluated. Fig.4 shows the Pathfinder and its material parameters are listed in Table 2. The weight of the Pathfinder of 1050g corresponds to the areal density of 21.4kg/m<sup>2</sup>. Second trial, 700mm diameter spherical mirror shown in Fig. 5 is designed to be lighter than  $15 \text{kg/m}^2$ . The lowest natural frequency of 312Hz is obtained by high Young's modulus of NT-SiC material. It is under fabrication and first sintering is planned in the first quarter in 2004.

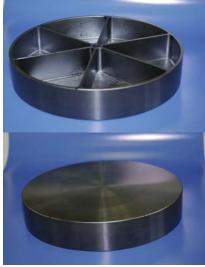


Fig.4 Pathfinder (250mm Flat Mirror)

Table 2: Pathfinder Material Parameters		
SiC Density	3020 kg/m <sup>3</sup>	
Yung's Modulus <sup>*2</sup>	400GPa	
Fracture Toughness <sup>*2</sup>	3.3MPam <sup>1/2</sup>	
Bending Strength	850MPa	
$CTE^{*1}$	$3.9 \times 10^{-6} \text{K}^{-1} (\text{RT-1073K})$	
Specific Heat Capacity <sup>*2</sup>	680J/kg/K	
Thermal Conductivity <sup>*2</sup>	130(W/m/K)	

\*1: CTE below room temperature is under measurement.\*2: Data of other batch, the parameters of the Pathfinder batch is under measurements.

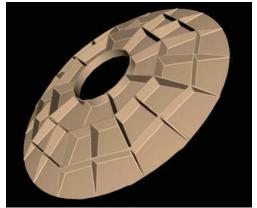


Fig. 5: Second Pathfinder (700mm Spherical) Design

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Surface Shape	Sphare		
Diameter	710mm		
Focal Length	796mm		
Apperture Ratio	1.12		
Maximum Rib Hight	30mm		
Weight	5.1kg		
Areal Density	$14.0 \text{ kg/m}^2$		
Lowest Natural Frequency	321Hz		

Table 3 Second Pathfinder Design Parameter

#### 4. SURFACE QUALITY of NT-SiC

First candidate of surface finish for NT-SiC is polish with diamond slurry. Polish is studied by JAXA and smaller roughness than a few nm rms will be achievable. The study is presented in separate paper.

Other candidate is Electolytic In-Process Dressing (ELID) Grinding Technique [6]. The ELID Grinding is an attractive candidate for a low cost large-scale optical surface finish technique. The detail of the ELID is also presented in separate paper. Detailed study of NT-SiC surface finish by ELID has been performed on the test piece shown in Fig.6.



Fig. 6: ELID Grinded Test Piece

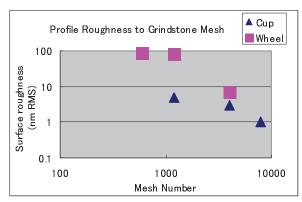


Fig.7: Roughness Derived from Line Profile.

The grinding with cup and disk iron cast grindstone was tried. The cup and disk methods are usually applied for flat and curved surface respectively. Fig.7 shows the dependence of the surface quality, roughness, on the mesh size of the grindstone by respective trials. The figure shows that the finer mesh (larger number) than 10000 will make smooth surface better than 1 nm rms roughness. Observations of the grinded surface with Zygo NewView interferometric microscope, see Fig.8 and 9, obviously show following results.

- 1) Surface quality is good enough for the visible mirror without additional CVD coating.
- 2) Tool mark are visible, even if fine grindstone with larger mesh number than 4000 is applied.
- 3) No open pore is observed on the surface.
- 4) Large silicon inclusions form significantly low basins of depth of a few tens nm. These basins have only negligible effect on the surface quality, since the large inclusions are rare and they are shallow.

Additional trial has been performed on the one of the Pathfinder to demonstrate light weighted thin structured mirror finish. The simple grinding with disk wheel grindstone has tried and successfully finished without any damage to the thin substrate. The precision figure controlled finish will be tried at the next step.

#### 5. CONCLUSION

This study, large scale fabrication and surface finish without CVD coating, demonstrates that NT-SiC has potential to be one of the best light weighted large mirror substrate. Two times higher bending strength than other SiC materials is obtained on large scale thin structure, while other material parameters are similar to others. Since polished bare NT-SiC surface will be utilized for visible as well as infrared application, expensive CVD process is unnecessary. Our goal is the optics with characteristics; 1) high performance, 2) light weight, 3) short lead time and 4) low cost.

Current work is focused on second Pathfinder to

establish the manufacturing process for one meter class lightweight mirror with the areal density less than 10kg/m<sup>2</sup>. On the other hand, the study to fabrication of the complicate figured objects has initiated for peripheral structure of the optics such as optical bench, support truss, spider of the Cassegrain telescope, and so on. All NT-SiC athermal design optics will be available. The first NT-SiC optics will be employed by LASER optical communication instruments for the SmartSat small satellite.

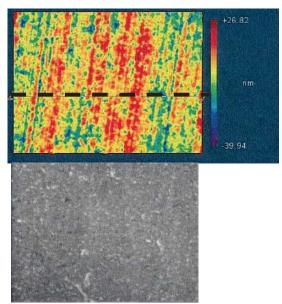


Fig. 8: Height Map (upper panel) and Intensity Map (lower panel). Surface Finished by #4000 Disk Grindstone.

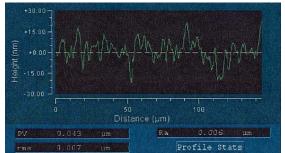


Fig. 9: Line Profile at Dashed Line on the Height Map of Fig.7.

#### References

- [1] Emaniel Sein et al, Space Application of Silicon Carbide, Proceedings of ISTS, (2002), 2002-c-18.
- [2] K.Saito et al, MUSES-C Navigation and Attitude Sensors, SANE98, 31-41, 98,125 (1998), pp59-66 in

Japanese.

- [3] Hidehiko Kaneda et al, Optical performance of the ASTRO-F telescope at cryogenic temperatures, Proc. SPIE Vol.4850 (2003). pp230-240.
- [4] B.Harnish el al, Development of an Ultra-lightweight Scanning Mirror for the Optical Imager of the Second Generation METEOSAT (MSG), Proc. SPIE Vol.2210 (1994), pp305-406.
- [5] S.Suyama, T.Kameda and Y.Itoh, Development of high-strength reaction-sintered silicon carbide, Diamond and Related Materials, 12 (2003), pp.1201-1024
- [6] H. Ohmori, Electrolytic In-Process Dressing(ELID) Grinding Technique for Ultra-Precision Mirror Surface Machining, Int.Journal of JSPE, 26/4(1992), 273