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DEVELOPMENT OF REACTION-SINTERED SiC MIRROR FOR SPACE-BORNE OPTICS

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ABSTRACT

We are developing high-strength reaction-sintered silicon carbide (RS-SiC) mirror as one of the new promising candidates for large-diameter space-borne optics.

In order to observe earth surface or atmosphere with high spatial resolution from geostationary orbit, larger diameter primary mirrors of 1-2 m are required. One of the difficult problems to be solved to realize such optical system is to obtain as flat mirror surface as possible that ensures imaging performance in infrared - visible - ultraviolet wavelength region. This means that homogeneous nano-order surface flatness/roughness is required for the mirror.

The high-strength RS-SiC developed and manufactured by TOSHIBA is one of the most excellent and feasible candidates for such purpose. Small RS-SiC plane sample mirrors have been manufactured and basic physical parameters and optical performances of them have been measured. We show the current state of the art of the RS-SiC mirror and the feasibility of a large-diameter RS-SiC mirror for space-borne optics.

1. INTRODUCTION

Traditionally, low expansion glass materials have been mainly used for mirrors that are carried on low earth orbit earth observation satellites. As the requirement for earth observation with higher spatial resolution is getting stronger, much lighter, stiffer and larger optical systems that cannot be realized by glass mirrors need to be developed. Also the requirement for geostationary earth observation satellites with multi-band spectroscopic-imagers of high spatial resolution and is increasing these days. In order to realize such observing system, a large diameter (~ 2 m) telescope system is needed due to integration time constrained by attitude stability of a satellite. Hence a new material that will meet such requirements should be investigated and developed.

Silicon carbide has many favorable properties for space

optics, such as high stiffness, high thermal conductivity, and low thermal expansion. Hence the material has been actively developed and recently it has been applied to space borne optical systems.

Astro-F, a Japanese infrared space telescope that will be launched in 2005, employs a porous SiC mirror of which diameter is 70 cm [1]. Porous SiC has an advantage in enabling light weight telescope system. It is, however, technically very difficult to manufacture larger diameter mirrors with this material. One of the reasons is that the manufacturing and sintering process of large diameter substrate has not been well developed and matured yet. Another reason is that a large diameter CVD pot, that is unavailable for the time being, is indispensable to realize homogeneous mirror surface with very small micro roughness.

Sintered SiC has also been applied to space-borne optics and medium and large diameter mirrors are now being developed for astronomical space missions such as HSO (Herschel Space Observatory) [2] and GAIA [3], and also for earth observation satellites. Although the technique of manufacturing 3.5 m diameter mirrors for HSO has matured, there might still be challenges to obtain small surface roughness that meets requirements of near infrared - visible - ultraviolet applications.

High-strength RS-SiC manufactured by TOSHIBA is expected to be one of the most excellent and feasible materials for visible - infrared space-borne optics. We expect that this material has some advantages in developing light weight large telescope system. Also it is the most promising candidate for the telescope system of JASMINE (Japan Astrometry Satellite Mission for INfrared Exploration) [4] that is planned and developed by National Astronomical Observatory of Japan. The whole telescope system, including secondary and tertiary mirrors, trusses, as well as the primary mirror, of its proto model, nano-JASMINE, will be constructed with the high-strength RS-SiC.

In the following sections, we show the characteristics and the current state of the art of this material, and discuss the applicability and feasibility of a large-diameter RS-SiC

mirror for space-borne optics.

2. OUTLINE OF DEVELOPING RS-SiC MIRROR

The high-strength RS-SiC manufactured by TOSHIBA was originally developed for the use as turbine-blades of power plants [5]. The manufacturing method of RS-SiC is different from other SiC manufacturing methods such as conventional sintering method using sintering agent, CVD method, hot press method, etc. The raw material SiC powder and carbon powder are mixed sufficiently to achieve homogeneous and dense substrate so that surface roughness would be suppressed as small as possible and that the strength would be as large as possible. Then a green body is formed by pressing and it is sintered with metal silicon under reduced pressure of inactive atmosphere so that the metal silicon is melt and penetrates into the SiC green body to form SiC by reaction. By this method, residual silicon, that would be a cause of micro crack, are well controlled and distributed, and the size of the residual silicon can be fully dispersed and suppressed to nano-order scale. The details of the manufacturing process of RS-SiC are explained in [5, 6, 7] and in the presentation by Mr. Tsuno from NEC-TOSHIBA Space Systems [8]. The main characteristics of this material are as follows.

- (1) Micro-scale structure is so homogeneous and fine to nano-order level that a few nanometer scale surface roughness can be achieved and hence, CVD coating will not be necessary.
- (2) Porosity is nearly zero, that results in almost no shrinkage (less than 1%) during sintering process.
- (3) Thermal expansion coefficient is smaller than those of other SiCs, therefore, thermal distortion is also smaller than others.
- (4) Bending strength is the world's highest ($> 1000\text{MPa}$), the specific strength is as large as that of FRP (fiber reinforced plastic), and the specific stiffness is as large as that of beryllium.

Therefore we expected that RS-SiC is one of the excellent candidates for large space-borne mirrors and started to develop the material to optimize the manufacturing and processing parameters to employ as a mirror.

We obtained a small sample substrate to study the characteristics of the RS-SiC more in detail to confirm the possibility and applicability of the material. The first step was to investigate (1) condition of inner crystal

grains, (2) absorbance of solar light, (3) infrared emissivity, (4) the correlation between the surface roughness and polishing, and (5) scattering characteristics. The sample mirror was polished three times. The surface wavefront and roughness and scattering characteristics were measured after each polishing. Then the parameters of manufacturing and polishing processes were confirmed and determined. The second step was to investigate the easiness and reliability of metal vapor coating. Three sample mirrors were manufactured and polished adopting the parameters determined in the first step. Then three kinds of metals were vapor coated on the surfaces of the three mirrors. In order to confirm and evaluate the reliability of those metal coatings, rubbing test etc. are to be tried, after measuring optical performances.

The physical properties of the high-strength RS-SiC lot that we are developing are summarized in Table 1.

Tab.1 Physical Properties of the sample RS-SiC

Density (g/cm^3)	3.05
Young's modulus (GPa)	390
Thermal expansion coefficient (ppm/K)	3.9
Bending strength (MPa)	800
Fracture toughness ($\text{MPa}\cdot\text{m}^{1/2}$)	3.3
Thermal conductivity (W/mK)	130
Specific heat (J/kgK)	6.8
Vicker's hardness	1700

The sample substrate is a plane of which dimensions are 50 mm (length), 50 mm (width), and 10 mm (thickness). The diameter of raw material SiC powder was $1\mu\text{m}$ and that of material carbon powder was $0.3\mu\text{m}$. The green body was sintered at 1693 centigrade under reduced pressure. The surface roughness before polishing was around several tens or several hundreds nanometers (rms).

The substrate was polished three times by pitching using diamond slurry as a polishing agent. The polishing time and the diameter of the polishing agent adopted for each polishing are summarized in Tab.2. The polishing speed was about $1\mu\text{m/h}$.

Tab.2 Polishing Parameters

	Time (hours)	Diameter (μm)
1 st	8	2
2 nd	15	2
3 rd	6	1

The surface flatness measured with ZYGO interferometer after the first and the third polishing is shown in Figs.1 and 2, respectively.

After the first polishing, hairline like scratches is clearly identified. It may be due to uneven distribution of residual silicon. Also marble-like feature of black and white was clearly appeared (Fig.3). Hence in order to study the differences of properties and to identify the causes of such inhomogeneity in appearance, polishing parameters were varied as shown above. These three different points (scratched area, white area, and black area) were measured for scattering property and surface roughness. After the third polishing, the marble-like feature disappeared completely (Fig.4), although the hairline like scratches was still confirmed.

Applying the parameters of manufacturing and polishing processes thus determined, three more samples were manufactured and polished to study the applicability of metal vapor coating on the RS-SiC mirror surface. Gold, silver, and aluminum have just been vapor coated on each mirror surface. The results of the coating reliability and optical performances will be published elsewhere.

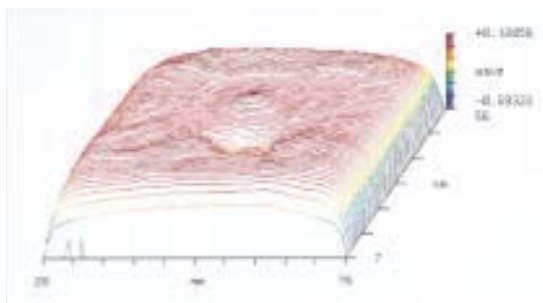


Fig.1 Surface wavefront after the first polishing

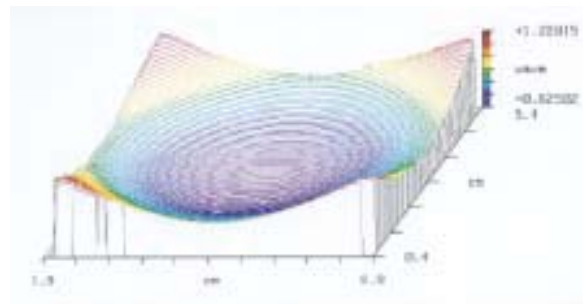


Fig.2 Surface wavefront after the third polishing

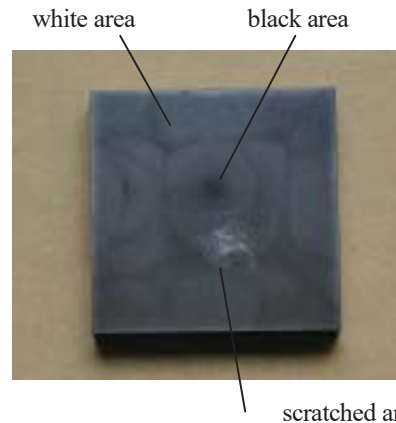


Fig.3 Marble-like feature of the sample mirror



Fig.4 Mirror surface after the third polishing (the direction of the mirror is the same as in Fig.3)

3. MEASUREMENTS

3.1 SEM Observation

The surface conditions of the sample mirror before and after the first polishing were observed with a scanning electron microscope (SEM). The figures are shown in Figs.5 and 6, respectively. The trace of grinding can be seen in Fig.5. There are some $10\mu\text{m}$ scale basins on the surface after the polishing.



Fig.5 RS-SiC surface condition before polishing

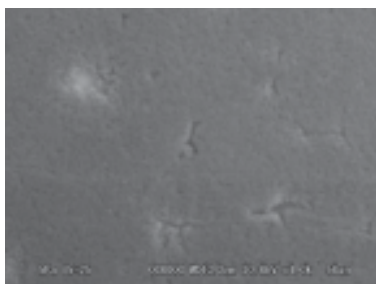


Fig.6 RS-SiC surface condition after polishing

3.2 TEM Observation

In order to observe the inner conditions of the RS-SiC substrate, the sample was observed with a transmission electron microscope. Two thin pieces of samples were sliced from the sintered bulk close to the sample mirror (about 1 mm away from the sample mirror) by ion-milling method. Hitachi H-9000UHR was used. The acceleration voltage was 300kV.

The large and small scale figures of the sample mirror are shown in Figs.7 and 8, respectively. In Fig.7, a few micron scale residual silicon can be identified. Fig.8 shows dense and homogeneous crystal grains of SiC of 500nm - 1 μ m scale and porous structure is not observed.

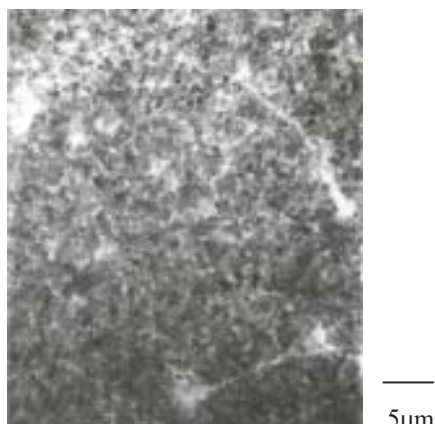


Fig.7 Inner condition of RS-SiC

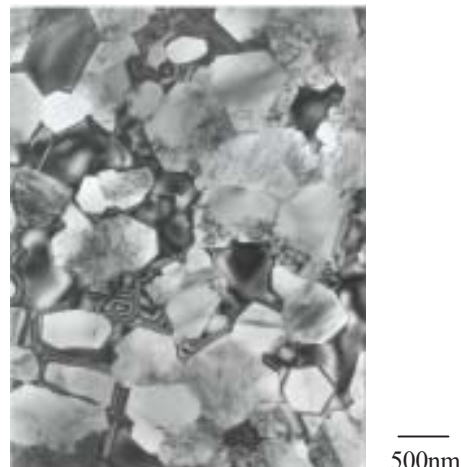


Fig.8 Inner condition of RS-SiC

3.3 Absorbance and Emissivity

The absorbance of solar light and infrared emissivity measured before and after polishing are summarized as below (Tab.3). Both the values after polishing are smaller than those before polishing. The differences may be within measurement error due to the surface conditions.

Tab.3 Absorbance and emissivity

Absorbance	before polishing	0.759
	after polishing	0.720
Emissivity	before polishing	0.689
	after polishing	0.662

3.4 Surface Roughness

In order to study the correlation between polishing and surface roughness to optimize the polishing parameters, the sample mirror was polished three times by changing polishing time and particle size of the agent. Each time after polishing, the surface roughness at several points on the mirror were observed by using laser confocal scanning microscopes, KEYENCE VK8500, OLYMPUS OLS1200, or ZYGO New View 5000.

The surface roughness measured by these instruments are shown in Figs. 9-13. The roughness at the scratched area is obviously larger than those at other areas. The measured values are roughly summarized in Tab. 4.

Generally, there isn't an obvious difference between white and black areas. As silicon is tend to be polished

faster than SiC, the areas that relatively larger residual silicon remains (5-10 μm or larger) are lower than SiC area by 200-400 nm (residual silicon basin). However areas where residual silicon is smaller, the surface roughness is less than 10 nm (rms). Also the roughness of the residual silicon basin area is around 10nm (rms). After the third polishing, the fluctuation of the surface roughness seems to be smaller.

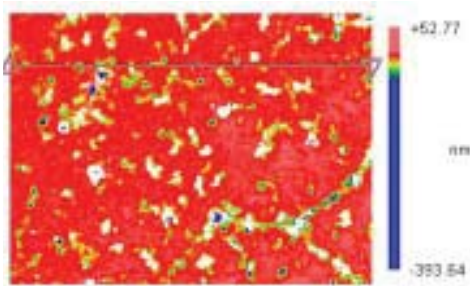


Fig.9 Surface roughness after the first polishing measured with New View 5000

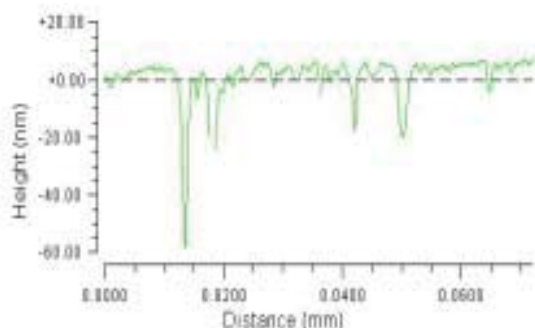


Fig.10 Cross-sectional profile along the line in Fig.9

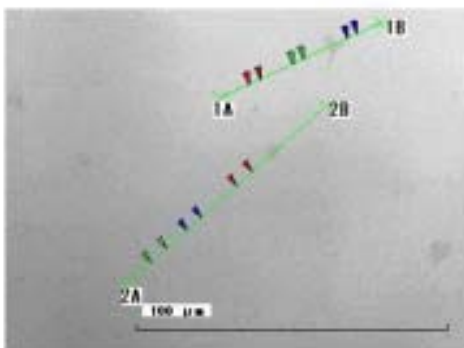


Fig.11 Surface roughness after the third polishing measured with VK8500

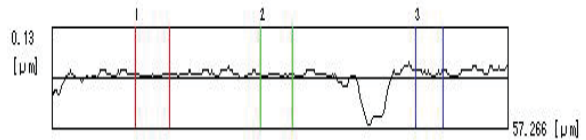


Fig.12 Cross-sectional profile of 1A-1B in Fig.11

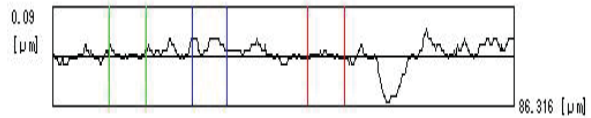


Fig.13 Cross-sectional profile of 2A-2B in Fig.11

Tab.4 Surface roughness

	P-V (nm)	RMS (nm)	Ra (nm)
1 st	4 - 300	2 - 100	0.6 - 65
2 nd	15 - 340	4 - 130	3 - 120
3 rd	10 - 100	10 - 20	10 - 20

3.6 Scattering

The horizontal angular dependency of scattering was measured after each polishing. The wavelengths of the lasers used for the measurements were 404nm, 543.5nm and 633nm. The laser beam was introduced to the sample mirror surface via a chopper. The scattered light of incident laser beam reflected by the sample mirror was detected by a Si-detector. The camera together with the detector was fixed on a turntable that was controlled by a stepping motor of high angular resolution. The azimuthal sampling interval was 0.02-0.03 degree.

The result after the second polishing is shown in Fig.14. The horizontal axis shows the offset angle from the light axis. Vertical axis shows the relative energy level that is normalized by the peak value of each measurement.

As is shown in the figure, scattering profiles of white and black areas are close to that of optical reference mirror. But the scattering profile at the scratched area is larger than those at other areas and the reference mirror.

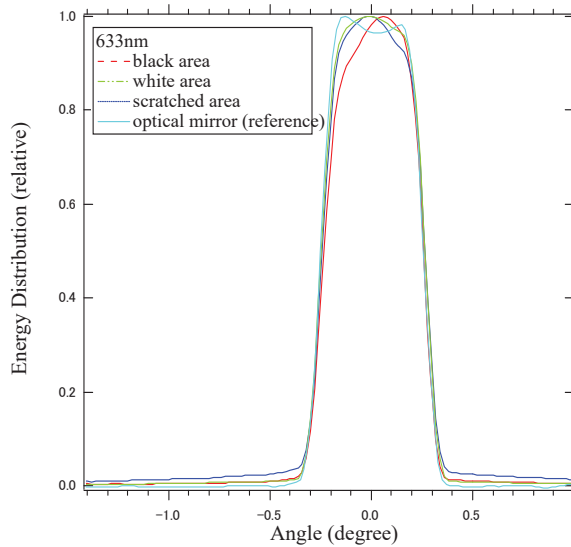


Fig.14 Scattering angular profile after the second polishing

4. DISCUSSION

The observations by SEM and TEM show that the microscopic structure of this high-strength RS-SiC sample mirror is very dense and homogeneous and that the porosity is almost zero. The diameters of the crystal grains are between several hundreds nanometers and 1 μm . There are, however, some large areas (40-50 μm) of residual silicon basin. Such area is supposed to be a cause of hairline scratches that appeared after polishing.

The results of the measurements of surface roughness and scattering after the first and the second polishing show that there was not a remarkable improvement in the surface properties in spite of the longer polishing time. This suggests that the polishing time (polished volume) does not largely effect on the surface roughness, and that 8 hours polishing may be sufficient.

On the other hand, the appearance and the surface roughness were both greatly improved after the third polishing. The marble-like feature disappeared completely and the surface roughness at the scratched area decreased to around 100 nm (PV). Also the fluctuation in surface roughness became smaller, that suggests that the surface obtained by the third polishing is more even. Therefore the diameter of the diamond slurry used as a polishing agent is considered to be an important factor to obtain small surface roughness. From these results, it is concluded that the diameter of the diamond slurry should be smaller than 1 μm .

The hairline scratches themselves did not disappeared completely by the third polishing, although the roughness of the residual silicon basin at the area was improved. In order to improve or eliminate such defect, it is required that the raw material particles of SiC and carbon should be mixed sufficiently and homogeneously so that residual silicon would disperse more evenly to nano scale level.

Comparing with conventional glass mirrors, the obtained surface roughness of 10 nm (rms) is not still preferable for the use in ultraviolet wavelength region. In this sense, polishing parameters and the control of microstructure at nano scale need to be further investigated.

The scattering profiles of white and black areas are close to that of optical reference mirror. But the scattering profile at the scratched area is still broader than those at other areas and the reference pyrex mirror, that also suggests that the surface roughness of the sample mirror is larger than that of the pyrex mirror. It is obvious that the scratches effect largely on the scattering properties of the mirror surface. Hence it is still an important problem to be solved to eliminate the localized residual silicon.

Although there still remains such a technical challenge to improve the surface roughness that is applicable to ultraviolet mirrors, the optical performance of the RS-SiC is expected to be preferable in optical-infrared wavelength region. It is expected that CVD coating is unnecessary for this material, that is one of the most advantageous factors to manufacture a large diameter mirrors.

Though there are many merits and advantage to develop high-strength RS-SiC mirrors, there are still some other important challenges that have to be considered and solved to manufacture large diameter mirrors and to realize large space-borne optics, such as the relationship between the polishing pressure, back-structure of a mirror, and wave front of a mirror surface, etc.

5. SUMMARY

One of the difficult problems to be solved to realize large space-borne optical systems is to obtain as flat mirror surface as possible that ensures imaging performance in infrared-visible-ultraviolet wavelength region. This means that homogeneous nano-order surface flatness/roughness is required for the mirror. The high-strength RS-SiC manufactured by TOSHIBA is one of the most excellent and feasible candidates for such purpose. Small RS-SiC plane sample mirrors have been

manufactured and basic physical parameters and optical performances of the sample mirror have been obtained.

We studied (1) condition of inner crystal grains, (2) absorbance of solar light, (3) infrared emissivity, (4) the correlation between the surface roughness and polishing, and (5) scattering characteristics.

The observations by SEM and TEM show that the microscopic structure of this RS-SiC sample mirror is very dense and homogeneous and that the porosity is almost zero.

From the results of measurements after each polishing, it is concluded that polishing time is not an important factor to obtain small surface roughness. However, the diameter of the diamond slurry used as a polishing agent is considered to be an important factor to realize small surface roughness. From these results, it is concluded that the diameter of the diamond slurry should be smaller than 1 μm .

There are hairline-like scratches appeared after polishing and they did not eliminated completely although it was polished with finer slurry. These scratches are supposed to be appeared due to localized residual silicon. The scattering property at the scratched area is worse than that of an optical reference mirror, although those of other points on the polished surface are as excellent as that of the optical reference mirror. Hence we concluded that it is very important to eliminate such defects of large residual silicon to apply the RS-SiC to large diameter space-borne mirrors, and that technical improvements in manufacturing the RS-SiC are required.

In spite that such a problem still remains, it is expected that CVD coating is unnecessary for this material that is one of the most advantageous factors to manufacture large diameter mirrors.

In order to manufacture large diameter mirrors and to realize large space-borne optics, there are still some other important challenges that have to be considered and solved, such as the relationship between the polishing pressure, back-structure of a mirror, and wave front of a mirror surface. We are planning to continue the study and develop the material to solve such problems to realize a large diameter optical system applicable to geostationary earth observation satellite.

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