Microscopic surface structure of C/SiC composite mirrors for space cryogenic telescopes

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We report on the microscopic surface structure of carbon-fiber reinforced silicon carbide (C/SiC) composite mirrors that have been improved for Space Infrared telescope for Cosmology and Astrophysics (SPICA) and other cooled telescopes. The C/SiC composite consists of carbon-fiber, silicon carbide and residual silicon. Specific microscopic structures are found on the surface of the bare C/SiC mirrors after polishing. These structures are considered to be caused by the different hardness of those materials. The roughness obtained for the bare mirrors is 20 nm rms for flat surfaces and 100 nm rms for curved surfaces. It was confirmed that a SiSiC slurry coating is effective in reducing the roughness down to 2 nm rms. The scattering properties of the mirrors were measured at room temperature and also at 95 K. No significant change was found in the scattering properties through cooling, which suggests that the microscopic surface structure is stable with changes in temperature down to cryogenic values. The C/SiC mirror with the SiSiC slurry coating is a promising candidate for the SPICA telescope.

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1. Introduction

The development of light-weight mirrors is one of key technologies required to realize the next-generation astronomical infrared space telescopes, for which mirrors much larger than the present ones are needed. Silicon carbide (SiC) and its variants are promising candidates of the material for light-weight mirrors. The major characteristics of SiC are its high strength, thermal conductivity, and especially the high ratio of Young’s modulus to density. In particular, because of its high stiffness, mirrors using these materials can be made with thin ribs and skin structures, so that the weight can be much reduced.

SiC mirrors have been adopted for the AKARI mission and the Herschel Space Observatory (ASTRO-F is a Japanese infrared astronomical satellite which was launched in February in 2006 and is now called AKARI). The telescope on AKARI has a 710-mm-diameter primary mirror consisting of porous and chemical vapor deposited SiC. The entire AKARI telescope system is cooled by vapor from liquid helium to 6 K. The Herschel Space Observatory is an infrared astronomical mission from the European Space Agency with a telescope equipped a 3.5 m diameter sintered SiC mirror. This satellite is to be launched in 2008 and operated with a passive cooling system. NASA has urged the development of the James Webb Space Telescope (JWST), which will have deployable light-weight mirrors of beryllium for a 6.5 m aperture telescope in space.

Space Infrared telescope for Cosmology and Astrophysics (SPICA) is a next generation mission led by Japan for infrared astronomy planned to be launched into a halo orbit around one of the Sun-Earth libration points (L2) in the 2010s. The SPICA telescope is designed to have a 3.5 m diameter aperture, and will be cooled to 4.5 K by radiative cooling and mechanical cryo-coolers for observations at infrared wavelengths. It will be optimized for observations in the 5-200 µm wavelength, and have image quality of diffraction limit at 5 µm. The requirement for the surface roughness of the mirrors for the SPICA telescope is 20 nm rms or better at 4.5 K. Table 1 summarizes the optical specifications of the SPICA telescope.

Carbon-fiber reinforced silicon carbide (C/SiC) composite is one of the promising materials
for the SPICA telescope\textsuperscript{10}, while the other is sintered SiC. C/SiC composite has characteristics similar to SiC and can provide a higher fracture toughness than SiC\textsuperscript{10}. Furthermore, the C/SiC composite has another advantage in which it can be machined easily during the C/C stage (see below), and therefore, a lightweight rib structure and complex shaped design for the support mechanism can be realized.

A sufficiently small surface roughness is one of the most fundamental properties required for mirror applications\textsuperscript{11}. However, it is not guaranteed that a surface with sufficiently small roughness can be realized with the C/SiC mirror because of the complicated nature of composite materials. SiSiC slurry coating has often been applied to improve roughness, although it requires additional processes in the fabrication\textsuperscript{10}. Furthermore, the cryogenic performance of C/SiC and SiSiC slurry coatings has not yet been well investigated. The coefficients of thermal expansion (CTE) of the SiC\textsuperscript{12}, Si\textsuperscript{13}, and carbon-fiber\textsuperscript{14} are $2.6 \times 10^{-6} \text{ K}^{-1}$, $3.3 \times 10^{-6} \text{ K}^{-1}$, and $0.8-1.1 \times 10^{-6} \text{ K}^{-1}$ at room temperature, respectively. The difference in the CTEs of these materials causes thermal stress in the C/SiC composite when the mirrors are cooled to cryogenic temperatures, and consequently the microscopic structure might change when cooled.

In this paper, we report on measurements and the improvement in the microscopic surface structures of both bare and slurry coated mirrors made of the newly developed C/SiC composite. We also carried out measurements of the scattering properties of these mirrors at $\sim 300 \text{ K}$ and $95 \text{ K}$ to examine possible changes in the surface roughness caused by cooling.

2. Surface of the C/SiC composite mirror and its measurement

2.A. Manufacturing process of the mirror

The fabrication process of the improved C/SiC composite is summarized briefly below; the details are described in Ozaki et al. 2004\textsuperscript{10}.

1. Preparation of the carbon-fiber preform : for the new C/SiC composite, the preform is composed of pitch-based milled fibers and binder.
2. The CF preform is impregnated with coal tar pitch. They are carbonized and graphitized in an inert atmosphere to produce the carbon-fiber carbon matrix (C/C).

3. The C/C material is machined into the end-product geometry. The C/C material is easily milled into light-weight structures with thin ribs and skins.

4. If necessary, C/C segments can be joined by adhesives to build larger or more complicated structures.

5. The C/C substrate is infiltrated with liquid silicon to react with the carbon matrix and form SiC. During this process, the joints between the segments become SiC.

6. If necessary, a SiSiC slurry coating is applied.

As a result of improvements in the manufacturing process, particularly owing to the increase in the volume fraction of pitch-based carbon-fiber to more than 30% and to the adequate control of the reactivity, several material properties have been significantly improved. For example, the bending strength, Young’s modulus, the toughness, and the thermal conductivity become 200 MPa, 320 GPa, 3.9 MPa m$^{1/2}$, and 160 W m$^{-1}$K$^{-1}$ at room temperature respectively, while the conventional values are 160 MPa, 260 GPa, 2.4 MPa m$^{1/2}$, and 125 W m$^{-1}$K$^{-1}$.

2.B. Measurement of the surface properties

We prepared small test mirrors to investigate the surface properties and provide quick feedback to the fabrication process. A photograph of the test mirrors is shown in figure 1. They are flat mirrors produced by employing different fabrication processes for the C/SiC composite. The right square mirror in figure 1 is made using the improved C/SiC. Half of the mirror surface is coated with aluminium for the light scattering measurements described in section 4.

Microscope interferometers are used to observe the surface structure of the test mirrors. The interferometers provide high-precision three-dimensional measurements, in which the
resolution is sub-micron parallel to the mirror surface, and sub-nm in the vertical direction. Results of the three dimensional measurements are highly useful not only for estimating the roughness, but also for understanding the origin of the roughness and for improving the surface quality. For each measurement, we used one of the available interferometers, either the ZYGO Maxim-NT, the WYKO NT1100 in National Astronomical Observatory Japan, or the ZYGO New-View in RIKEN. It is difficult to apply microscope interferometers for mirrors at cryogenic temperatures, so measurements of the scattering properties were made as described in section 4.

The top-left panel of figure 2 shows a typical result of the measurement by the microscope interferometer ZYGO Maxim-NT for the surface of a conventional C/SiC composite mirror shown in the left of figure 1. The top-right of figure 2 is a photographic image obtained through the viewer of the interferometer with the fringe pattern removed. The light gray region in the photograph corresponds to residual silicon. The gray region indicates SiC and slender carbon-fiber. The SiC regions tend to be ~100 nm higher than the silicon regions. This tendency is consistent with the difference between the hardness of the SiC and Si; the Mohs hardness of the SiC is 9 and that of Si is 7, respectively\textsuperscript{15}. The carbon-fiber regions tend to be trenches with ~100 nm depth. The average roughness of the whole surface is 40 nm rms.

The results of these measurements were taken into account in the material production process improvement trials\textsuperscript{10} and were very convenient because of their immediacy. The middle panel of figure 2 shows the data of the improved C/SiC composite mirror obtained in the same manner as the first one. The height difference between the SiC and the residual silicon regions has become smaller as a result of the reduction in the amount of residual silicon in the improved material and polishing improved for the new composite. Carbon-fiber is still the dominant factor in the surface roughness. Surface roughness to be 20 nm rms has been achieved for flat mirrors made using the improved C/SiC composite.
3. Microscopic surface quality of the spherical mirrors

3.A. Surface without SiSiC slurry coating

To test the surface quality of a more realistic mirror than the test pieces, we fabricated a 160 mm diameter spherical test mirror with a flat outer region near the edge using the improved C/SiC composite material, as shown in figure 3. The radius of curvature of the spherical part is 900 mm. Both the spherical and flat regions were polished. Three segments were joined at three of the six ribs to examine the effects of the joints.

An example of the microscopic surface structure of the flat region is shown in the top left and right of figure 4. The roughness of the outer flat region is similar to that of the improved flat test mirrors made from the improved C/SiC composite (∼20 nm rms), which satisfies the requirement of the SPICA telescope.

However, the structure of the spherical part was different from the outer flat region. The middle-left and right of figure 4 show the results of measurements on the spherical part. The roughness of the spherical part is typically 100 nm rms, which is significantly worse than that of the flat region. Mirrors of this quality can be useful for mid- to far-infrared observations, however, SPICA requires a better quality. The major sources of the roughness are the trenches due to the carbon-fiber and pits due to the residual silicon. As a result, we conclude that the current bare mirrors made of C/SiC composite do not meet the requirements for the SPICA telescope.

3.B. Surface of the joint of segments

The joint is one of the key techniques needed to realize large monolithic mirrors from small segments. The joint material for joining C/SiC composite parts together is made of SiC. The CTE, Young’s modulus, and the other properties of the joint are similar to those of the mirror.

The bottom of figure 4 shows the microscopic surface structure of the joint of the 160 mm spherical mirror. The width of the joint in this mirror is about 0.5 mm. Most of the area of
the joint consists of SiC, but there is also residual silicon here. We find that the height of the residual silicon region tends to be a few tens of nanometers lower than the SiC region, similar to the difference in height between the SiC and silicon regions in the flat test mirrors. Based on this result, we adjusted the viscosity of the adhesive to optimize it for the improved C/SiC composite. Finally, the thickness of the joint now becomes 0.2 mm and the residual silicon is much reduced.

3.C. Surface with SiSiC slurry coating

Since the presence of carbon-fiber is a major factor in making the polishing process difficult and increasing the surface roughness, we applied a SiSiC slurry coating\textsuperscript{10,11} to the C/SiC to make a surface without carbon-fiber. The typical thickness of the slurry coating is 200 µm.

The polished surface of the improved C/SiC composite mirror with the SiSiC slurry coating appears quite different from those of bare polished C/SiC composite mirrors. The bottom of figure 2 shows the microscopic surface structures of a slurry coated and polished flat mirror measured with the ZYGO New-View. The typical roughness is \(~2 \text{ nm rms}\) or less. The improvement in the roughness is highly significant and the obtained surface may be applied even for visible light observations. In the photograph (bottom-right of the figure 2), the dark gray spot-like regions correspond to SiC, and the light gray region indicates silicon. We examined the correlation between the 3-D data and the optical appearance, which indicates that the SiC region has become bumpy. This trend is similar to that seen in the bare polished surface of the improved C/SiC composite. However, both the scale and height of the SiC regions are much smaller in the slurry coated surface than in the bare ones.

We prepared a 160 mm diameter spherical test mirror with a SiSiC slurry coating similar to that in the previous section. The microscopic surface structure of this mirror was measured with an interferometer. The roughness and appearance of the surface are similar to those of the flat test mirror, not only in the outer flat region but also in the central spherical part. The surface of the joint was also examined and no significant differences were found between
the joint and other regions.

Therefore, we conclude that the C/SiC composite mirror with the SiSiC slurry coating is a promising candidate for the SPICA telescope.

Table 2 summarizes the microscopic surface properties of the mirrors developed in this work.

4. Light scattering measurements

4.A. Experimental method

Space-borne infrared telescopes must be cooled to achieve high sensitivity and therefore the mirrors for the telescope must satisfy their specifications at cryogenic temperatures. The C/SiC composite consists of materials with different CTEs as described in section , and consequently the surface of the mirrors can be deformed or damaged on the microscopic scale by thermal stress at low temperatures. Direct observations with a microscope interferometer are difficult to perform at cryogenic temperatures. In this section, we examine the surface roughness of C/SiC composite mirrors using the light scattering method.

We carried out measurements of the scattered light from test mirrors at $\sim 300$ K and 95 K. The configuration used for the measurement is shown in figure 5. As described above, the diameter of the carbon fiber is about 10 $\mu$m and its length and the size of the residual Si region are larger than 10 $\mu$m. Light scattered by these structure can spread into a few degrees due to the diffraction of light at 0.63 $\mu$m, so we set up an experiment to measure the scattered light in the range of a few degrees. A flat test mirror was installed in a dewar with a BK7 flat window. The test mirror was thermally connected to the cold work surface of the dewar, which was cooled by liquid nitrogen. A He-Ne laser of 0.63 $\mu$m wavelength and 1 mm diameter was irradiated onto the aluminum coated part of the mirror.

The reflected light was measured by a Si photo-diode with an electrical amplifier. No AC chopping was used. The profile of the scattered light was measured by scanning the detector in a linear direction. The background signals were measured without the light irradiation
and were subtracted from the measurements. The incident angle of the irradiated beam was 5 degrees. Each test mirror was measured both at $\sim 300\,\text{K}$ and $95\,\text{K}$. The temperature was measured with a platinum resistance temperature sensor placed directly on the surface of the mirror. As a reference, a BK7 flat mirror with an aluminum coating was also measured.

4.B. Results

Obtained profiles of the scattered light from the mirrors are shown in figure 6. In the plot, the data are transformed into the intensity in a solid angle. Profiles in the top figure indicate the results for the improved C/SiC composite mirror without a SiSiC slurry coating shown on the right of figure 1. Compared with the results for the BK7 mirror, significant scattered light is clearly detected. No appreciable difference is found between the results at $300\,\text{K}$ and $95\,\text{K}$. The results for the SiSiC coated mirror in the bottom of figure 2 are presented in the middle of figure 6. No significant difference is seen between the SiSiC coated mirror and the BK7 mirror surface. No appreciable difference is detected between the results at $\sim 300\,\text{K}$ and $95\,\text{K}$ also for this test mirror. The limit to the sensitivity of the scattered light measurements is $10^{-5.5} \sim 10^{-6}$ for the scale of figure 6. The measured positions on the mirror surface may change with cooling because of the shrinkage of the cold work surface. However, the stability of the scattered light between $\sim 300\,\text{K}$ and $95\,\text{K}$ indicates that this effect is not significant.

The sensitivity of the present measurement is limited by the detector dark current and the laser light scattered at the window and in the air. The former was estimated to be about $5 \times 10^{-6}$ in units of figure 6 when measured with the laser light off. This dark current has been subtracted from the plots of figure 6. The latter cannot be measured directly in the present setup, but the comparison of the C/SiC with and without slurry coat (and BK7) measurements indicates that the present measurements have an total uncertainty of $\sim \pm 1 \times 10^{-6}$, which is limited by the uncertainty in the dark current.

The scale of the surface structures that produce the peak profile in the central $\pm 0.2$ degree is estimated to be $\sim 180\,\mu\text{m}$ if diffraction dominates the scattering. The length of the trenches
in the carbon-fiber regions are of a similar scale, though it is difficult to distinguish the scattered light from the reflected light itself in the vicinity of the specularly reflected beam. On the other hand, the width of the trenches is much smaller and the length of them is not uniform. The width of the trenches and the length of the shortest trenches are $\sim 10\mu m$ which corresponds to $\pm 3.6$ degree. The scattering angle from smaller structures or structures of other geometry is expected to be larger. Thus the results of the measurements suggest that the roughness caused by the carbon-fiber does not increase during cooling.

From the surface roughness of the C/SiC mirror obtained from the interferometer measurements and the results of the light scattering measurements, we can estimate the total integrated scatter of the mirror at cryogenic temperatures at optical and infrared wavelengths. Here we use the following estimate for the total integrated scattering\textsuperscript{16}:

$$TIS = \frac{E_s}{E_a} = \left(\frac{4\pi\sigma \cos \theta_i}{\lambda}\right)^2,$$

where $TIS$ is the total integrated scattering, $E_s$ is the scattered energy and $E_a$ is the total energy of the incident beam, $\sigma$ is rms roughness, $\theta_i$ is the incident angle, and $\lambda$ is the wavelength. The values of $TIS$ derived from the roughness of the test mirrors shown in table 2 are 0.17 and $1.7 \times 10^{-3}$ for the bare C/SiC flat mirror and the mirror with the SiSiC slurry coating, respectively, with $\theta_i = 5$ degree and $\lambda = 0.63\mu m$. At $\lambda = 5\mu m$, $TIS$ derived from equation (1) is $2.5 \times 10^{-3}$, $2.5 \times 10^{-5}$, 0.06 for the bare C/SiC flat mirror, the mirror with the SiSiC slurry coating, and the bare spherical mirror, respectively. Therefore, the fraction of the energy lost by scattering at the microscopic surface structure is estimated to be insignificant in the mid-infrared region for slurry coated mirrors. To estimate the $TIS$ from the scattering experiment directly, measurement of light with a larger scattering angle is needed.

The difference in the profiles between the bare mirror and the others shown in figure 6 is significant at which the relative intensity is smaller than $\sim 10^{-4.5}$. With this value, scaled by equation (1), for the sensitivity of detecting scattered light $\sigma = 20\, \text{nm}$ for the bare mirror,
the limit to the sensitivity for measuring roughness is $3.6 \sim 6.5 \text{ nm rms}$. This limit is larger than the roughness of the BK7 mirror measured by the interferometer, and therefore it is reasonable that no significant scattered light was detected with the BK7 mirror.

The present experiment was carried out with the available He-Ne laser. The measurement sensitivity for roughness can be improved if we use a laser with shorter wavelengths. The measurement with infrared wavelengths is also useful to confirm directly the scattering property for the SPICA application. Examinations of the scattering properties are complementary to measurements with microscope interferometers. Measurements of the scattered light profiles provide the optical performance of mirrors directly. Furthermore, this method can be applied at cryogenic temperatures. On the other hand, measurements by interferometer give us useful information about the origin of the roughness. In this work, the results of the two method are consistent with each other

5. Summary

The microscopic surface structure of mirrors made of improved C/SiC composite was investigated. The improved C/SiC is a candidate material for the SPICA mirrors because of its superior properties: high toughness, high stiffness, machinability, and the feasibility of making large single dish mirrors.

We examined flat test mirrors and 160mm diameter spherical mirrors with and without a SiSiC coating. The surface of the bare C/SiC composite consists of carbon-fiber, silicon carbide and silicon. Specific structures were found at the surface of the bare C/SiC surface after polishing, which is consistent with the difference in hardness of those materials.

The achieved surface roughness was 20 nm rms or less for bare C/SiC composite flat mirrors, which just about satisfies the requirement of the SPICA telescope. For the curved surface of the bare C/SiC mirror, the nominal roughness was about 100 nm, which is larger than that obtained for flat mirrors. At present, the roughness of the curved bare surface is not small enough for the SPICA telescope, though such mirrors still can be useful for mid
to mid- and far-infrared observations. The joint between the segments was examined and an improvement was made in the adhesive process. The amount of residual silicon was reduced and the width of the joint was narrowed down to 0.2 mm from 0.5 mm. We can confirm that the SiSiC slurry coating is effective for reducing the roughness of both flat and curved surfaces down to 2 nm rms. For polished slurry coated mirrors with a spherical surface, no significant difference was detected between the joint and the other parts.

The change in the scattering properties of the C/SiC composite at cryogenic temperatures was examined with a 0.63 µm laser. Light scattered from the microscopic surface structures was detected for bare C/SiC composite mirrors, while SiSiC slurry coated mirrors showed scattering properties similar to glass mirrors. No significant change was found between the scattering properties at 95 K and ∼300 K, suggesting that the microscopic structures of the surface are stable against cooling. The TIS of the examined mirrors at room temperature and 95 K is discussed.

We conclude that the mirrors made with the improved C/SiC with a SiSiC slurry coating satisfy the specifications for the SPICA telescope, and therefore they are one of quite promising candidates for the SPICA mirrors. Other candidate materials (e.g. sintered SiC) are also being examined for the SPICA telescope using the same methods. On the other hand, the surface quality of the bare mirrors is suitable for use at mid- and far- infrared wavelengths where cooling of the optics is quite important.

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Table 1. The optical specifications for the SPICA telescope

| Parameter                          | Specification                      |
|-----------------------------------|------------------------------------|
| Telescope optics type             | On axis Ritchey-Chretien           |
|                                   | Consists of monolithic mirrors     |
| Image quality                     | 5 $\mu$m diffraction limit         |
| Diameter of the primary mirror    | 3.5 m                              |
| Optimal wavelength                | 5 - 200 $\mu$m                     |
| Working temperature               | 4.5 K                              |
| Weight                            | $< 700$ kg                         |
| Focal length                      | $\sim 18$m                         |
| Field-of-view diameter            | $\sim 30$ arcmin                   |
| Total wave-front error            | $< 350$ nm (rms)                   |
| Surface roughness of the mirrors  | $< 20$ nm (rms)                    |
Table 2. Microscopic surface quality of C/SiC composite mirrors

| Sample                  | Coating      | Roughness (rms) | Origin of roughness                  |
|-------------------------|--------------|-----------------|--------------------------------------|
| Flat test mirror:       | Conventional| 40 nm           | Si dimple, carbon-fiber trench       |
|                         | Improved     | 20 nm           | Carbon-fiber trench                  |
| φ160mm mirror:          | Flat part    | 20 nm           | Carbon-fiber trench                  |
|                         | Spherical part| 100 nm         | Carbon-fiber trench                  |
| Flat test mirror:       | SiSiC slurry | 2 nm            | SiC bump                             |
| φ160mm mirror:          | Flat part    | SiSiC slurry    | SiC bump                             |
|                         | Spherical part| SiSiC slurry   | SiC bump                             |
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Figure 1. Examples of flat test mirrors for examining the manufacturing process of the C/SiC composite material, the polishing technique and the microscopic surface structures. The left and the right mirror are sample before and after the trial in which the carbon fiber was more dispersed and the residual Si was reduced. The middle and the right mirror are test piece in the trial of the improvement. The size of the right mirror is 50 mm × 50 mm × 10 mm. Half of the polished surface is coated with aluminum for measurements of the scattered light described in section 4.

Figure 2. Top: The left figure shows the microscopic surface structures obtained from the interferometer (ZYGO Maxim-NT) for a flat test mirror of the conventional C/SiC composite. The right figure is a photographic image of the same part of the surface with visible light. The relatively light gray area indicates residual Si. The dark gray part corresponds to SiC and carbon-fiber. The average surface roughness of this sample is 40 nm rms. Middle: Microscopic surface structure of a flat test mirror of the improved C/SiC composite. The average value of surface roughness is 20 nm rms. Bottom: Microscopic surface structure of a SiSiC slurry coated flat test mirror using the ZYGO New-View. The relatively bright and dark gray areas in the photograph correspond to silicon and SiC, respectively. SiC regions are higher than silicon regions. The morphology of the slurry coated surface is quite different to that of the bare surface. The average roughness of the coated surface is 2 nm rms. Note that the vertical scale is not the same for each figure.

Figure 3. The left and the right show front and back view of the 160 mm diameter test mirror of the improved C/SiC composite. The radius of curvature of the spherical part is 900 mm, while the periphery is flat. Three segments which were fan shaped with 120 degree central angle were joined at the ribs.
Figure 4. Top: Microscopic surface structure of the 160 mm spherical mirror of the bare C/SiC composite. Left and right figures are the results of interferometer measurements and photographic images of the flat part obtained using the WYKO NT1100, respectively. The average roughness is 20 nm rms. The observed structures are similar to those of the flat test mirrors shown in figure 2. Middle: Left and right show the spherical part of the mirror by the same method. The average roughness of this part is 100 nm rms. Bottom: Joint in the 160 mm spherical mirror of the bare C/SiC composite. Left and right figures are the results of interferometer measurements and photographic images. The average width of the join is 0.5 mm, where no carbon-fiber is found. The bright region in the right figure corresponds to residual silicon. The silicon region is 20-50 nm lower than the SiC regions.

Figure 5. Configuration used for the scattered light measurements.

Figure 6. Light scattered from flat test mirrors at room temperature and cryogenic temperatures. The top and middle figures show the results of the improved C/SiC bare mirror and the SiSiC coated mirror. The bottom figure shows the data for a BK7 glass mirror as a reference.
Fig. 1. Enya et al.
Fig. 3. Enya et al.
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