Journal of Astronomical Telescopes, Instruments, and Systems

AstronomicalTelescopes.SPIEDigitalLibrary.org

Thermal stress test of the depth-graded platinum/carbon reflectors

Yoshitomo Maeda
Kou Ichihara
Hiroaki Kan
Yu Shionome
Takuro Sato
Toshiki Sato
Takayuki Hayashi
Manabu Ishida
Yoshiharu Namba
Hideaki Takahashi
Takuya Miyazawa
Kazunori Ishibashi
Michito Sakai
Satoshi Sugita
Yoshito Haba
Hironori Matsumoto
Hideyuki Mori

Thermal stress test of the depth-graded platinum/carbon reflectors

Yoshitomo Maeda,^{a,b,*} Kou Ichihara,^{a,c} Hiroaki Kan,^d Yu Shionome,^{a,c} Takuro Sato,^{a,c} Toshiki Sato,^{a,c} Takayuki Hayashi,^e Manabu Ishida,^{a,b,c} Yoshiharu Namba,^f Hideaki Takahashi,^f Takuya Miyazawa,^e Kazunori Ishibashi,^g Michito Sakai,^g Satoshi Sugita,^h Yoshito Haba,ⁱ Hironori Matsumoto,^e and Hideyuki Mori^{e,j}

^aJapan Aerospace Exploration Agency (JAXA), Institute of Space and Astronautical Science (ISAS), 3-1-1 Yoshinodai, Chuo-Ku, Sagami-hara, Kanagawa 252-5210, Japan

^bSOKENDAI (The Graduate University for Advanced Studies), Department of Space and Astronautical Science, School of Physical Sciences, 3-1-1 Yoshinodai, Chuo-Ku, Sagami-hara, Kanagawa 252-5210, Japan

^cTokyo Metropolitan University, Department of Physics, 1-1 Minami-Osawa, Hachioji, Tokyo 192-0397, Japan

^dOsaka University, Department of Earth and Space Science, Graduate School of Science, 1-1 Machikaneyama, Toyonaka, Osaka 560-0043, Japan

^eNagoya University, Department of Physics, Furocho, Chikusa, Nagoya 464-8602, Japan

^fChubu University, Department of Mechanical Engineering, 1200 Matsumotocho, Kasugai, Aichi 487-8501, Japan

^gJapan Aerospace Exploration Agency (JAXA), Research and Development Directorate, 2-1-1, Sengen, Tsukuba, Ibaraki 305-8505, Japan

^hEhime University, Bunkyo-cho, Matsuyama, Ehime 790-8577, Japan

ⁱAichi University of Education, Igaya-cho, Kariya, Aichi 448-8542, Japan

^jNASA, Goddard Space Flight Center, Code 662, Greenbelt, Maryland 20771, United States

Abstract. The ASTRO-H hard x-ray telescope (HXT) is designed to reflect hard x-rays with energies up to 80 keV. It will make use of thin-foil, multinested conical optics with depth-graded platinum/carbon (Pt/C) multilayers. We report on thermal stress tests of the HXT reflectors. The reflectors were fabricated on a heat-formed aluminum substrate of thickness gauged at 200 μm of the alloy 5052. This was followed by an epoxy replication on Pt/C-sputtered smooth Pyrex cylindrical mandrels to acquire the x-ray reflective surface. For the thermal tests, the reflectors were maintained at three different temperatures: -5 , 50 , and 60°C , respectively, for a week. We found that the surface of the reflectors were significantly changed at temperatures of 60°C or higher. The change appears as wrinkles with a typical scale length of a few tens of microns. No changes on the surface were observed from the -5 and 50°C samples. There was also no change in the x-ray reflectivity for these two temperatures. © The Authors. Published by SPIE under a Creative Commons Attribution 3.0 Unported License. Distribution or reproduction of this work in whole or in part requires full attribution of the original publication, including its DOI. [DOI: [10.1117/1.JATIS.1.3.034001](https://doi.org/10.1117/1.JATIS.1.3.034001)]

Keywords: ASTRO-H; hard x-ray telescope; x-ray optics.

Paper 15008P received Feb. 25, 2015; accepted for publication May 28, 2015; published online Jun. 29, 2015.

1 Introduction

The ASTRO-H¹ hard x-ray telescope (HXT) has conical-foil mirrors with depth-graded multilayer reflecting surfaces that provide reflectivity over a 5 to 80 keV energy range.² The effective area of the HXT is maximized for a long focal length of 12 m giving an effective area of $\sim 350\text{ cm}^2$ at 30 keV for the two HXTs. A depth-graded multilayer mirror reflects x rays not only by total external reflection, but also by Bragg reflection. In order to obtain high reflectivity up to 80 keV, the HXTs have a stack of multilayer reflectors with different sets of periodic length and number of layer pairs with a platinum/carbon (Pt/C) coating.

The reflector has a bilayer structure with a $\sim 0.2\text{-}\mu\text{m}$ -thick depth-graded multilayer, a $\sim 20\text{-}\mu\text{m}$ -thick epoxy, and a $200\text{-}\mu\text{m}$ -thick aluminum substrate. Wrinkle formation in such bilayers resulting from compressive stresses due to a heat load is a well-known problem (e.g., Ref. 3). The wrinkles also degrade the performance of the x-ray reflection so that it is critical to know the upper and lower temperature boundaries for which

the wrinkles do not form. We, therefore, performed thermal stress tests of the depth-graded Pt/C reflectors used by the ASTRO-H HXT. Such replicated thin-foil reflectors are planned for many future missions.^{4–9} A record of the storage temperature is, therefore, useful in order to quickly examine the validity of a given thermal environment of such satellite systems.

We prepared samples by randomly choosing two reflectors for flight use. The depth-graded Pt/C multilayer was sputtered onto the glass mandrel, which was then sprayed with epoxy (EPOTEK 301-2) and was transferred onto the aluminum substrate (A5052). The group ID of the sets of periodic length and number of layer pairs is six.² Details of the reflector production can be found in Ref. 2.

2 Experiments

For the stress tests, the prepared samples were placed into a constant-temperature oven and annealed for a period of time. The samples were then removed one at a time to examine for temporal changes of the surface structure using four different methods. The methods we used are summarized in Table 1. All measurements were made under room temperature environment.

*Address all correspondence to: Yoshitomo Maeda, E-mail: ymaeda@astro.isas.jaxa.jp

Table 1 Summary of the methods used.

Method	Machine name	Sample	Sensitive wavelength
X-ray reflectivity	ISAS 4 m x-ray beamline	Full foil	≤ 0.1 nm
Atomic force microscope	Nano-search microscope	20 mm $\times$ 20 mm	~ 20 nm to ~ 5 μ m
Scanning white light Interferometry	NewView200	20 mm $\times$ 20 mm	~ 1 to 250 μ m
Microscope	Nano-search microscope	20 mm $\times$ 20 mm	A few micrometers to ~ 1 mm

Table 2 Root mean square (r.m.s.) of the NewView200 measurements.

	r.m.s. (nm)
No heat load	0.46 ± 0.10
After 50°C load	0.43 ± 0.12
After -5°C load	0.51 ± 0.14

The reflector originally had a height of 20 cm and a radius of 150 mm. Because this is too large to be measured with the atomic force microscope (AFM), a microscope, and an interferometer, one reflector was cut into squares measuring 20 mm by 20 mm. From these, six pieces were chosen randomly and affixed to a brass sheet (Fig. 1). A 20 mm by 20 mm square sample was used for three measurements: AFM, scanning white light interferometry, and microscope.

A different, un-cut reflector was used for the evaluation of the x-ray reflectivity.

3 Measurements of the Surface “Waviness”

3.1 Temperatures of 60°C or Higher

Surface waviness with a scale length of > 1 μ m is measured with a scanning white light interferometer and microscope. The scanning white light interferometer used is the NewView200 (Zygo Co.), while the microscope is an option mode of the nano-search microscope (Olympus Co.). Both machines are located at Chubu University.

Fig. 1 Picture of the 20 mm $\times$ 20 mm sample of the platinum/carbon supermirror reflector.

Fig. 2 Picture taken with the microscope after the exposure at 60°C.

We found that the samples exposed to temperatures of 60°C or higher showed wrinkles. An example taken with the microscope is shown in Fig. 2. The scale length of the wrinkles is ~ 20 μ m, to which both the microscope and the scanning white light interferometer are sensitive.

The ~ 20 - μ m scale wavelength corresponds to the thickness of the epoxy layer (20 μ m), so that the wrinkles can be interpreted as being caused by compressive stress in the Pt/C multilayer during a heat load. The scale length of the wrinkles may be limited by the thickness of the epoxy layer.

It is also notable that the cure temperature of the epoxy used in the reflector is 50°C.² The glass transition temperature of the epoxy is 65°C or higher (EPOTEK 301-2 Technical Data Sheet). The 60°C temperature at which we detected the wrinkles is higher than the cure temperature and lower than the glass transition temperature. The appearance of the wrinkles at 60°C may also depend on the properties of the epoxy.

3.2 Temperatures Between -5 and 50°C

In order to identify the temperature range where there is no damage by a heat load, we made the assumption that the lower and upper temperature boundaries where the performance is

Fig. 3 History of the head load of -5 and 50°C for the reflector samples.

Fig. 4 An example of the three-dimensional surface profile taken with the interferometer NewView200 after the 50°C load.

unaffected are -5 and 50°C . We then tested to see whether or not the assumption is valid.

Figure 3 shows a history of the reflector samples. We measured the surface at room temperature and placed the samples at 50°C in a constant-temperature oven in the atmosphere and annealed for five days. We then made the measurements and placed them at -5°C in vacuum for five days. We then repeated the measurements in the atmosphere.

Figure 4 shows an example of a three-dimensional image taken with the interferometer NewView200. We measured six pieces of the $20\text{ mm} \times 20\text{ mm}$ sample and three local spots per sample. The root mean square (r.m.s.) of the image height of the 18 datasets was obtained. The average of the r.m.s. is also

listed in Table 2. The results showed that the surface is not significantly changed after heat loads of 50 and -5°C .

4 Roughness Measurements

Small-scale angstrom-level waviness (roughness) with a scale length of $\sim 0.1\text{ }\mu\text{m}$ was measured using two methods for the samples taken at 50 and -5°C . One method was x-ray reflectivity measurements: The x-ray reflectivity depends on the roughness of the reflector's surface, where the sensitivity of the x-ray reflectivity to surface roughness is typically a few angstroms. We thus measured the x-ray reflectivity of the reflectors at the ISAS 4 m x-ray beam facility, using a collimated x-ray beam as narrow as 0.05 mm and at a distance of $\sim 4\text{ m}$ from the x-ray generator (RIGAKU RU-200). The incident energy is fixed at 8.05 keV ($\text{Cu-K}\alpha$). A proportional counter was used as the detector. The results of the reflectivity are shown in Fig. 5. The x-ray reflectivity of the three samples at the different temperature loads are consistent with each other.

The second roughness measurement was made with the AFM in the dynamic force microscopy mode. We used the nano-search microscope provided by Olympus Co. The roughness was $3\text{ }\text{\AA}$ (r.m.s.) from the surface of the samples that had been maintained at 50 and -5°C . This confirmed that there was no significant change in the roughness for any sample.

5 Summary

A thermal stress test was made for the reflector of the ASTRO-H HXT. The reflector has a bilayer structure with a $\sim 0.2\text{-}\mu\text{m}$ -thick Pt/C depth-graded multilayer, a $\sim 20\text{-}\mu\text{m}$ -thick epoxy, and a $200\text{-}\mu\text{m}$ -thick aluminum substrate. The epoxy is EPOTEK 301-2 and is cured at 50°C .

Wrinkles with a typical scale length of a few tens of microns were observed at the surface after it had been exposed to a 60°C temperature environment. On the other hand, no significant changes were seen for the reflectors exposed to temperatures of -5 and 50°C .

Acknowledgments

The authors are grateful to all full-time engineers and part-time workers in Nagoya University for support in the mass production of the hard x-ray telescope mirror reflectors. We thank C. Baluta for English correction.

Fig. 5 X-ray reflectivity at 8.0 keV . Black, red, and blue correspond to the data taken before the thermal stress (circle), after the 50°C experience (triangle), and after -5°C (square).

References

1. T. Takahashi et al., “The ASTRO-H x-ray astronomy satellite,” *Proc. SPIE* **9144**, 914425 (2014).
2. H. Awaki et al., “Hard x-ray telescopes to be onboard ASTRO-H,” *Appl. Opt.* **53**(32), 7664–7676 (2014).
3. P. J. Yoo and H. H. Lee, “Evolution of a stress-driven pattern in thin bilayer films: spinodal wrinkling,” *Phys. Rev. Lett.* **91**, 154502 (2003).
4. M. Beilicke et al., “Design and tests of the hard x-ray polarimeter X-Calibur,” *Nucl. Instrum. Methods Phys. Res. A* **692**, 283–284 (2012).
5. K. Hayashida et al., “X-ray gamma-ray polarimetry small satellite PolariS,” *Proc. SPIE* **9144**, 91440K (2014).
6. Y. Ogasaka et al., “Characterization of Pt/C multilayer at 200 keV soft gamma-ray,” *Proc. SPIE* **6688**, 66880S (2007).
7. S. Tachibana et al., “Development of multi-layer-supermirror design for future x-ray telescopes with a large effective area for the Fe K lines,” in *Suzaku-MAXI 2014: Expanding the Frontiers of the X-ray Universe*, p. 232 (2014).
8. Y. Tawara et al., “Development of the four-stage x-ray telescope (FXT) for the DIOS mission,” *Proc. SPIE* **9144**, 914461 (2014).
9. H. Tsunemi et al., “Formation Flight Astronomical Survey Telescope (FFAST) mission in hard x-ray,” *Proc. SPIE* **9144**, 91442R (2014).

Yoshitomo Maeda is an assistant professor at the Institute of Space and Astronautical Science (ISAS), Japan Aerospace Exploration Agency (JAXA). He is a member of the ASTRO-H collaborations.

Biographies for the other authors are not available.