CTE homogeneity, isotropy and reproducibility in large parts made of Sintered SiC

Michel Bougoin
Boostec (a company of Mersen group)
Bazet, France

michel.bougoin@mersen.com

Didier Castel & Franck Levallois
Astrium
Toulouse, France
didier.castel@astrium.eads.net
franck.levallois@astrium.eads.net

Abstract— **For Herschel SiC primary mirror purpose, a new approach of comparative CTE measurement has been developed; it is based on the well known bimetallic effect ("biceramic" in this case) and also optical measurements. This method offers a good CTE comparison capability in the range of 170-420K (extensible to 5-420K) depending of the thermal test facilities performance, with a resolution of only 0.001 µm/m.K. The Herschel primary mirror is made of 12 SiC segments which are brazed together. The CTE of each segment has been compared with the one of a witness sample and no visible change, higher than the measurement accuracy, has been observed. Furthermore, a lot of samples have been cut out from a spare segment, from different places and also from all X, Y and Z direction of the reference frame. No deviation was seen in all of these tests, thus demonstrating the very good homogeneity, reproducibility and isotropy of the Boostec® SiC material. Some recent literature about SiC material measurements at cryogenic temperature shows a better behaviour of Boostec® SiC material in comparison with other kind of SiC which are also candidate for space optics, in particular for isotropy purpose. After a review of the available literature, this paper describes the comparative CTE measurement method and details the results obtained during the measurement campaigns related to Herschel project.**

Index Terms— **Ceramic material, SiC, cryogenic, low CTE, homogeneity, isotropy, reproducibility**

I. INTRODUCTION

The ideal material for space telescopes is a material which should …

i) be easy to polish and allow thin metal coating, for mirrors application,
ii) be able to withstand high loads (high mechanical strength and toughness), high stiffness and low density for the structural parts,.
iii) have a Coefficient of Thermal Expansion (CTE) as low as possible, stable in time, isotropic and ultra-homogeneous,
iv) provide high thermal conductivity thus making thermal gradient effects negligible,
v) withstand cryogenic temperature (close to 0 K) and also moderately high temperature (~ 350 K, for decontamination purpose), without any degradation or physical evolution (e.g. no material structure phase change),
vi) provide a good lightweighing capability compatible with the mass requirements,
vii) show no out-gassing, no moisture absorption nor release,
viii) show no degradation by space radiation.

Among all industrially available materials, the silicon carbide and in particular the Boostec® SiC material appeared as a best choice. During the past decade, Astrium / Boostec team has successfully carried out several projects of large highly stable optical instruments using Boostec® SiC material. They include Herschel [1] [11] [12] [13], Gaia [2] and Nirspec [3] which are operating at cryogenic temperature. For such application, the CTE knowledge of SiC down to 5K is fundamental because it has direct impact on the instrument performance. On technological point of view, the optical instruments require for all SiC parts (even very large ones) i) a perfect homogeneity of the CTE inside a part, ii) a perfect reproducibility of the CTE from a part to another one and iii) a perfect isotropy of the CTE inside a same part.

II. BOOSTEC® SIC MATERIAL

Previously named SiC-100, BOOSTEC® SiC material, is a **sintered silicon carbide**; its basic manufacturing process has been kept the same since the nineties. Its key properties are a high specific stiffness (420GPa / 3.15g.cm^{-3}) combined with a high thermal stability (180W.m^{-1}.K^{-1}/ 2.2 . 10^{-6} K^{-1}). In comparison with the even most recently developed reaction bonded SiC including short chopped carbon fibers [4], it features 24% higher thermal conductivity, 25% higher bending strength, 20% higher stiffness and similar toughness. It shows a better stability in time and a better resistance to the space radiations than the glass-ceramics which have been commonly used in the past for the space mirrors.

TABLE I. BASIC PROPERTIES OF BOOSTEC® SiC

Properties	Typical Values @ 293 K
Density	3.15 g.cm^{-3}
Young's modulus	420 GPa
Bending strength / Weibull modulus (coaxial double ring bending test)	400 MPa / 11
Poisson's ratio	0.17
Toughness (K_{1C})	3.5 MPa.m$^{1/2}$
Coefficient of Thermal Expansion (CTE)	2.2 . 10^{-6} K^{-1}
Thermal Conductivity	180 W.m^{-1}.K^{-1}
Electrical conductivity	10^{5} Ω.m

The thermal expansion and the CTE (the strain derivative) of the Boostec® SiC material are displayed respectively in Fig.1 and Fig.2. It must be noticed that the thermal expansion is very low at cryogenic temperatures and that the CTE decreases down to zero when the temperature approaches the absolute zero. Thanks to its isotropic microstructure, the physical properties of this alpha type SiC are isotropic and reproducible inside a same large part or from batch to batch (as demonstrated here after for CTE). The sintered SiC of BOOSTEC shows no mechanical fatigue, no outgassing and no moisture absorption nor release. It has been fully qualified for space application at cryogenic temperature such as NIRSpec instrument which will be operated at only 30K [3].

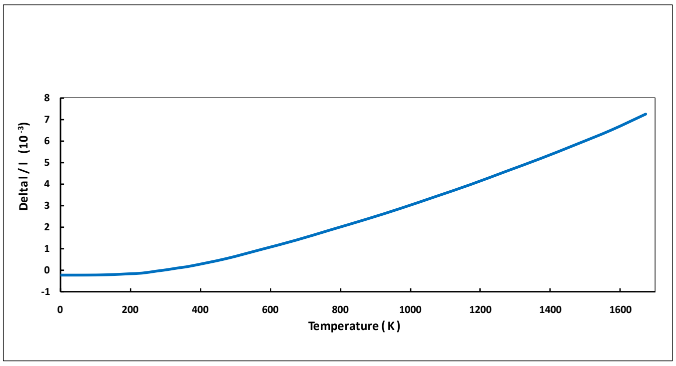


Fig.1. Thermal Expansion of Boostec® SiC versus Temperature

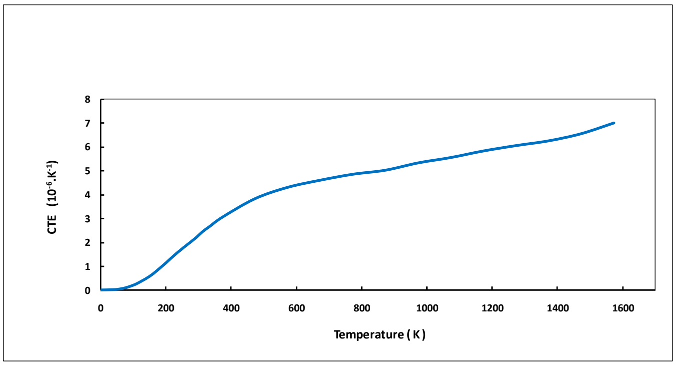


Fig.2. Coefficient of Thermal Expansion of Boostec® SiC

III. ABSOLUTE CTE MEASUREMENT

A. State of the art through the literature

All absolute CTE measurements are made with use of interferometric heads giving quite good accuracy but several laboratories claim some difficulties [5], [6], [7], [8], [9], [10].

For its Zerodur® material, Schott claims improved dilatometric measurements with an accuracy of +/- 0.0062 . 10^{-6} K^{-1}and a reproducibility of +/- 0.0012 . 10^{-6} K^{-1}. These glass ceramics show CTEs which are statistically distributed around zero, from batch to batch and also inside a batch. Schott distinguishes 3 classes of Zerodur® [5]:

- class 0 for batch ≤ 18 tons, CTE absolute value # +/- 0.02 . 10^{-6} K^{-1} and batch ΔCTE # +/- 0.03 . 10^{-6} K^{-1},
- ii) class 1 for batch ≤ 6 tons, CTE absolute value # +/- 0.05 . 10^{-6} K^{-1} and batch ΔCTE # +/- 0.02 . 10^{-6} K^{-1},
- iii) class 2 for batch ≤ 0.3 tons, CTE absolute value # +/- 0.1 . 10^{-6} K^{-1} and batch ΔCTE # +/- 0.01 . 10^{-6} K^{-1}.

Furthermore, due to structural relaxation, the Zerodur® thermal expansion shows a small hysteresis effect which is dependent on the rate of temperature change, when thermally cycling around room temperature [5].

In Pasadena Ca, the J.P.L. has measured the absolute CTE of Silicon Crystal, Invar M93 from Imphy, SiC from Xinetics and Boostec® SiC in the range of 320 to 20 K [6]. From RT to 70K (the Herschel telescope temperature of use), they obtained a strain of -232.7 +/-1 ppm for Xinetics SiC and -225.5 +/- 0.15 ppm for Boostec® SiC.

Old publication relates some CTE measurements made by Mitsubishi in Japan, on 15 CeSiC® samples, between 300K and 20K [7]. They obtained a strain of -238.59 +/- 5.29 ppm and also concluded that the material showed no anisotropy.

In Japan again, with a JAXA support, the absolute CTE of HBCeSiC® material has been measured in the range of 293K to 10K, with an accuracy estimated better than 0.01 . 10^{-6} K^{-1} and a reproducibility better than 0.001 . 10^{-6} K^{-1} [8]. They obtained an average CTE of 0.805 +/- 0.003 . 10^{-6} K^{-1} in the XY plane and 0.837 +/- 0.001 . 10^{-6} K^{-1} in the Z perpendicular direction. This significant anisotropy is due to the included carbon fibers (even short); it is in contradiction with the former CeSiC® measurements. Such anisotropy has been stated unacceptable for future large space optics operating at cryogenic temperature like SPICA.

The same authors also measured the CTE of SiC-100 (now named Boostec® SiC) in the same range of 293K to 10K; they report a CTE of 0.816 +/- 0.005 . 10^{-6} K^{-1}, similar to the HBCeSiC® ones but no anisotropy was detected [9]. Furthermore, no significant CTE difference was seen between 3 samples manufactured from 3 different batches. This +/- 0.005 . 10^{-6} K^{-1} dispersion was stated to be smaller than the measurement uncertainty and also compatible with the requirements of the future SPICA telescope.

More Recently in Germany, in the frame of an ESA project, the Physikalisch-Technische Bundesanstalt (PTB) has developed an improved and impressive Ultra Precision Interferometer. Absolute lengths measurements have been performed in the overall temperature range from about 7 K to 323 K for samples made of Boostec® SiC and HB-Cesic® [10].

Uncertainty of the resulting CTE was mostly below 0.003×10^{-6} K^{-1} which was the ESA target. The CTE of both types of samples revealed considerable reduction towards low temperatures, i.e. it was less than $0.01\text{x}10^{-6}$ K^{-1} at temperatures lower than 30K (Fig.3). Beside the continuous reduction of the CTE towards cryogenic temperature observed for Boostec® SiC material, HB-Cesic® exhibited small negative CTE values at temperatures < 80 K and a minimum around 50 K before it returns to be nearly zero around 40 K [10]. With this project, absolute length measurements down to cryogenic temperature could be demonstrated for the first time ever.

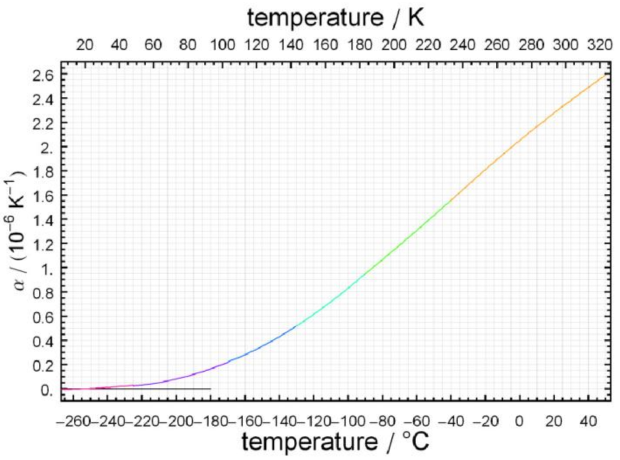


Fig.3. Accurate CTE measurement of Boostec® SiC from [10]

B. Absolute CTE measurement in Astrium

In the eighties, Astrium has developed its own CTE measurement facility in Toulouse, also based on interferometric measurements; it was mainly dedicated to the characterization of CFRP materials. It allows low CTE measurements in the 243K – 293K range. It has also been used in the nineties for the development of the SiC technology.

IV. COMPARATIVE CTE MEASUREMENT

A. Herschel primary mirror case

The Herschel primary mirror is 3.5m in diameter and its aerial density is only 25 kg/m². It is made of 12 sintered silicon carbide segments brazed together in a single run [11]. Each large segment is made from a different batch of “premix” SiC powder. Its 3 mm thick face sheet is stiffened by ribs of up to 100 mm high. The required WFE of this M1 is 3 µm rms for polishing purpose and the 293K-70K cool down allocated contribution is only 2.5 µm rms [12]. It is then of prime importance having a SiC CTE highly homogeneous and isotropic in all 12 segments.

Fig.4. The HERSCHEL M1 is made of 12 SiC segments brazed together

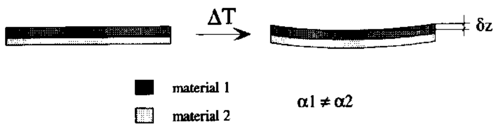
For Herschel primary mirror, Astrium has calculated and allocated a SiC CTE mismatch budget between each segment of $0.015 \cdot 10^{-6}$ K^{-1}.

B. Need of accurate comparative CTE knowledge

As seen here above, measuring the SiC CTE at cryo temperature with an accuracy close to $0.003 \cdot 10^{-6}$ K^{-1} seems now possible [10]. But in the 2000-2005 period, a precision better than $0.010 \cdot 10^{-6}$ K^{-1} was hardly obtained. On the other hand, the problem for Herschel M1 was not having accurate value of SiC samples absolute CTE but rather having an accurate knowledge of possible CTE mismatch between samples. This is the reason why Astrium has developed an accurate method of comparative CTE measurement.

C. Comparative CTE measurement in Astrium

The principle of the comparative CTE measurement is based on a “bi-metallic” effect, according to Fig.5.



With this configuration, the bending deflection (δz) is given by the following relation :

$$\delta z = \frac{3a^2}{8e}.(\alpha_1 - \alpha_2).\Delta T.\left[1 - \frac{h}{2e} - \frac{hk}{4eE} - \frac{1}{16}\left(\frac{k}{E}\right)^2\right] \qquad (1)$$

with : δz = maximal bending deflection due CTE mismatch (m) ;
2a = total length of the sample (m) ;
α1, α2 = CTE of material 1 and 2 (°K⁻¹);
e = thickness of material 1 (m) ;
e + h = thickness of material 2 (m) ;
E = Young's modulus of material 1 (Pa) ;
E + k = Young's modulus of material 2 (Pa) ;
ΔT = difference of temperature (°K) ;

Fig. 5. Principle of the comparative CTE measurement method

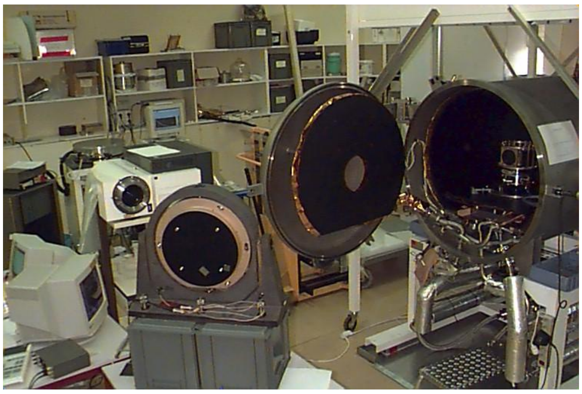


Fig. 6. Astrium comparative CTE measurement facilities

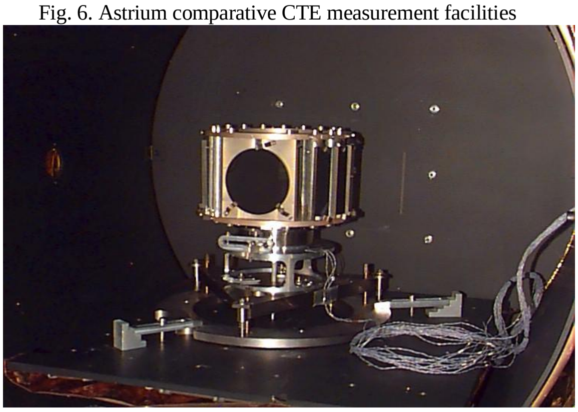


Fig. 7. Circular SiC reference mirror and rectangular samples are mounted on a rotating wheel and placed inside the thermal vacuum chamber

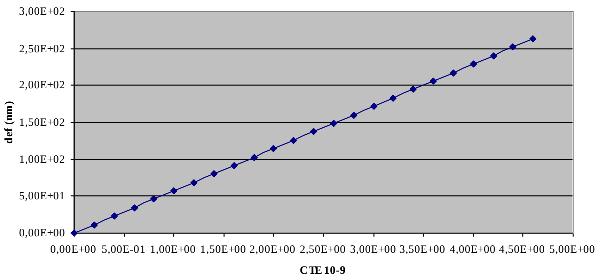


Fig. 8. Bending flexure of SiC/SiC samples versus their CTE mismatch (case of 2x4mm thick samples)

Two sintered SiC samples (110mm x 26mm, 2 to 4mm thick) are ground flat on both large faces and then brazed together according to Boostec process [11], thus obtaining a 110mm x 26mm bi-material. The brazed joint is very thin (< 10µm) and located at mid-thickness; its effect on the thermal deflection is accordingly negligible. The brazed assembly is then polished flat on one 110mm x 26mm face.

The Astrium set-up holds up to 14 bi-material samples and a SiC reference mirror according to Fig. 6 and 7. They are mounted on a wheel which can rotate around a vertical axis. This device is placed inside a thermal vacuum chamber allowing a measurement temperature range of 173K-423K. These all 14 samples can be measured in a same run. The possible samples deflection is obtained from their surface shape measurement (on 100mm length) with help of a Zeiss Direct-100 interferometer. The reference mirror allows making the necessary corrections mainly due to the chamber glass window thermal deformation and also other effects. The temperature is measured directly on the samples with help of thermocouples. Several steady state measurements can be achieved in the 173K-423K temperature range and, furthermore, several up and down cycles can be followed on. The estimated accuracy of +/- 20 nm on the optical measurement and the geometry of the samples give an accuracy about 0.0002 . 10^{-6} K^{-1}. The bending flexure is directly proportional to the CTE mismatch as shown in Fig. 5 and 8.

V. HERSCHEL COMPARATIVE CTE MEASUREMENT CAMPAIGN

Two campaigns dedicated to Herschel M1 are described here after.

A. *12 Segments of Herschel M1 Second Flight Model (FM2)*

Assuming that these M1 segments are from different batches and are named 1 to 12, 11 bi-material samples have been manufactured according to § IV.C. here above, thus obtaining the witness pairs 1/12, 2/12, 3/12, …10/12 and 11/12. They have all been measured in the 169K-413K temperature range. The bending deflections were comprised between -0.056µm and +0.015µm and the resulting CTE mismatch was -0.0004 +/- 0.00062 . 10^{-6} K^{-1}.

It was concluded that the CTE mismatch between each segments could be claimed in the range of +/- 0.001 . 10^{-6} K^{-1} resulting from: i) +/- 0.0006 . 10^{-6} K^{-1} observed dispersion, ii) +/- 0.0002 . 10^{-6} K^{-1} accuracy of the optical measurement device and iii) +/- 0.0002 . 10^{-6} K^{-1} accuracy of the data processing.

B. *Destructive check of an Herschel M1 lost segment*

A spare segment of the Herschel primary mirror has been checked in a destructive way by cutting out 23 samples thus witnessing different locations and orientations (side ribs, internal ribs and optical face sheet) as shown in Fig. 9. The sample N°23 is not visible in Fig.9 but it was cut out from the optical face sheet (similarly as samples N° 21 and 22), but close to the mirror center.

Among these 23 cut out samples, 17 have been selected (representing all locations and orientations), brazed individually on a reference SiC sample and measured in the 163K-273K temperature range. The 17 reference samples were all coming from the same manufacturing batch. The bending deflections were comprised between -0.045µm and +0.020µm and the resulting CTE mismatch was -0.00055 +/- 0.00066 . 10^{-6} K^{-1}.

It was concluded that the CTE mismatch between each sample from the same large segment could be claimed in the range of +/- 0.0021 . 10^{-6} K^{-1} resulting from: i) +/- 0.0007 . 10^{-6} K^{-1} observed dispersion, ii) +/- 0.0007 . 10^{-6} K^{-1} accuracy of the optical measurement device and iii) +/- 0.0007 . 10^{-6} K^{-1} accuracy of the data processing. It must be noticed that these 2 last uncertainties were significantly increased because the 17 samples were measured in 2 different runs.

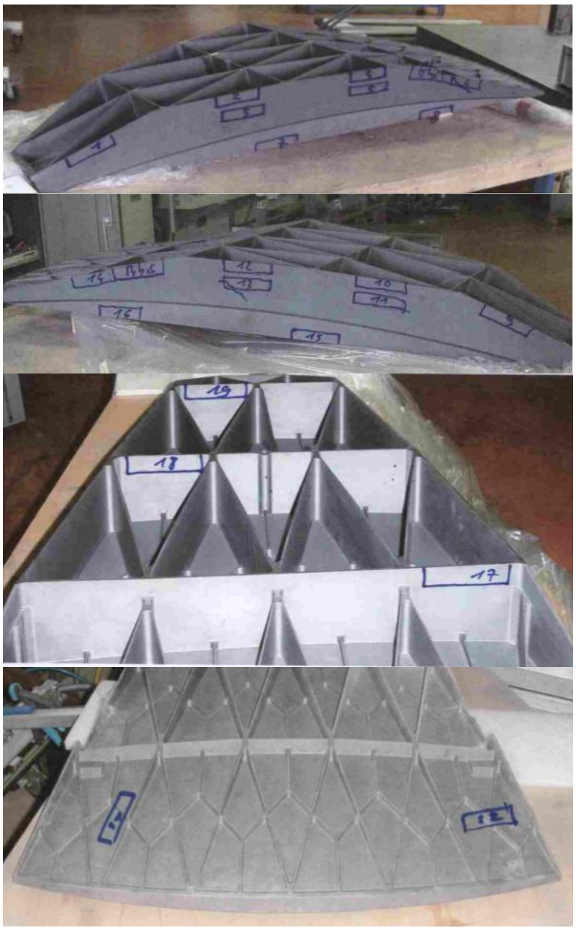


Fig. 9. SiC samples to be cut out from a Herschel M1 spare segment

VI. DISCUSSION

All CTE mismatches measured in § V here above are in the same range as the measuring method uncertainties. It is then demonstrated that no significant CTE mismatch is detectable from Boostec® SiC parts, from batch to batch (§ V.A.) and inside a large SiC part, whatever its location or its orientation in the part (§ V.B.). These very promising results have been confirmed afterwards through other Astrium measurements and also by JAXA ones [9]. The Boostec® SiC CTE mismatches were about ten times better than Herschel M1 requirements; they should also be compliant with the ones of the future scientific and earth observation missions.

The outstanding isotropy of the Boostec® SiC is obtained thanks it is made of small (< 5 µm) equiaxed grains and also because it is nearly free of secondary phase. On the other hand, the very good batch to batch reproducibility is obtained thanks to both i) a process fully under control and ii) a probably quite low sensitivity of the CTE to the process, inside elementary part and also part to part since the beginning of Boostec® SiC history.

VII. CONCLUSION

Astrium has developed a comparative CTE measurement method which allows quantifying the CTE mismatch of pairs of different SiC samples on the 173K-423K temperature range, with an accuracy about 10^{-9} K^{-1}. The Boostec® SiC CTE has been demonstrated highly reproducible, in this range of 10^{-9} K^{-1}, while the glass ceramic reproducibility is only in the 10^{-8} K^{-1} to 10^{-7} K^{-1}range. In other words, the CTE of Boostec® SiC is a generic value which is shared by all manufactured batches.

With same accuracy (~ 10^{-9} K^{-1}), no anisotropy has been detected in the Boostec® SiC. This is not the case of other SiC ceramics candidates for space application.

These outstanding thermal expansion properties greatly simplify the design, the manufacturing, the integration and the control of the large optics. For instance, no cryo-figuring is necessary when polishing the mirrors dedicated to cryo application. This makes Boostec® SiC a material of choice for the future large space scientific and earth observation missions.

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