High-precision measurements of thermal expansion at cryogenic temperature on stable materials

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1. Introduction

The European Space Agency (ESA) aspires the exploration of the universe using space-borne infrared telescopes with large aperture operating at cryogenic temperature. The Herschel space telescope is currently returning its first scientific observations orbiting from the Lagrange point L2 and the foreseen infrared observatory Spica is currently under study. The large diameter of the primary mirrors and the operation at cryogenic temperature require the use of ultra-stable materials and well-characterised dimensional change of the structure as function of temperature. Dimensional stability and accurate data on the thermal expansion is also relevant for the cryogenic spectographs MIRI and NIRspec on the James Webb Space Telescope JWST, for the high-precision laser interferometer LISA and for spacecraft sent to a hot environment such as BepiColombo and Solar Orbiter.

Although European industry has proven effective at designing and building thermally stable structures, problems have been encountered in measuring the absolute coefficient of thermal expansion (CTE) of relevant stable materials. In one recent mission, the design CTE had to be changed 6 times over a 2 year period and when the telescope was tested in cryogenic conditions an error was found in the back focal plane of over a centimetre [RD1,2].

To assess the current state-of-art in CTE measurements and to further improve European capability, ESA defined a number of activities. A first study was finalised comprising a round-robin test campaign and a second activity is ongoing for the further improvement of an already existing facility [RD20]. The present paper describes the round-robin that surveys various methods for length detection at various test institutes using various temperature profiles and measuring various sample materials. Similar work [RD3-6] was done under supervision of the Jet Propulsion Laboratory (JPL) in frame of the JWST project.

The objectives of the round-robin are a) thermo-mechanical characterisation of sample materials relevant for space applications, b) assessment of test institutes by reviewing their measurement uncertainties and c) recommendations for the future strategy and development of measurement capability in Europe.

1.1 Selection of materials and institutes

The sample materials included in the test campaign are SiC-100 from Boostec, HB-Cesic from ECM, Invar M93 from Imphy Alloys (ArcelorMittal), Schott-Zerodur and Schott-BK7. The first two materials are composites of reinforced Silicon Carbide with a near-zero CTE at cryogenic temperature. The third material is a nickel-steel alloy that achieves a very low CTE due to a strict thermal annealing regime. Zerodur is an optical material with near-zero expansion around room temperature. BK7 was included in this campaign because it is commonly used as traceable standard in classic dilatometry.

The selection of test institutes is based on predicted uncertainty, coverage of a wide temperature range, the method used for length detection and the used temperature profile. The emphasis is on interferometric methods at cryogenic temperature. The participating laboratories, indicated in table 1, offer mostly interferometry, but also capacitance cell dilatometry and LVDT based push-rod dilatometry. Geopolitical motivations played also a role in the selection, as some notable institutes appeared unavailable for participation.

Laboratory (Country)	Expansion detection method	Temperature range	Predicted CTE uncertainty	Determined CTE uncertainty range
PMIC (US)	Michelson Interferometer Probing on 2 sides of sample	LT: 10 K-300 K HT: 300 K-625 K	10 ppb/K	LT: 10 to 50 ppb/K HT: 10 to 400 ppb/K
ATK (US)	Michelson Interferometer Probing on 1 side of sample	20 K-300 K	20 ppb/K	10 to 30 ppb/K
PTB (DE)	Absolute Michelson Interferometer Probing on 1 side of sample	283 K-313 K	5 ppb/K	< 10 ppb/K
SERCO (GB)	Diverging beam interferometer Probing on flat mirror on sample	LT: 100 K-300 K HT: 300 K-625 K	10 ppb/K	LT: 10 to 130 ppb/K HT: 90 to 180 ppb/K
FZK (DE)	Capacitance cell	5 K-300 K	10 ppb/K	10 to 20 ppb/K
CEA (FR)	Push rod quartz dilatometer, LVDT	4 K-300 K	100 ppb/K	10 to 100 ppb/K
MELCO (JP)	Focus distance measurement Probing on mirrors on 2 sides of sample	10 K-300 K	10 ppb/K	10 to 50 ppb/K

Table 1 Participating laboratories with their predicted and determined uncertainties (LT = low temperature, HT = high temperature)

1.2 Summary observations

The analysis of the round-robin includes processing of the raw strain data by polynomial fitting and deriving the CTE, as well as a comparison of uncertainties and review of the laboratory's understanding and analysis of their measurement uncertainty. The round-robin confirmed that at the time of the comparison no facility was available that achieved CTE measurements in accordance with ESA's requirements. ATK and FZK provided low uncertainty in the cryogenic temperature range. The latter demonstrated a unique detection method with an unsurpassed resolution but its measurement uncertainty is limited because correction with a calibration standard is required. PTB demonstrated a low measurement uncertainty and a thorough understanding of its measurement uncertainty analysis but only operated in a limited temperature range. For this reason the PTB facility has been selected for further development to extend the temperature range [RD20].

2. CTE Measurement principle

2.1 Definitions

According to [RD12, RD17] the differential coefficient of linear thermal expansion $\alpha(T)$ is defined as CTE at temperature T and at constant pressure p, in reciprocal K, given by the following equation:

$$\alpha(T) = \frac{dL(T)}{dT} \cdot \frac{1}{L_0} \qquad \text{[Eq.1]}$$

with L_0 is the reference length at room temperature T_0, in the axis of measurement;
$L(T)$ is the length at temperature T, in the axis of measurement;
$dL(T)$ is the change in length over time interval dt;
dT is the change in temperature over time interval dt.

Note that previous definition is different from the more classical mean coefficient of linear thermal expansion A, which is defined as CTE at constant pressure, in reciprocal K, given by the following equation:

$$A = \frac{\Delta L}{\Delta T} \cdot \frac{1}{L_0} \qquad \text{[Eq.2]}$$

with ΔL is the change in length of the test specimen between two temperatures T_1 and T_2;
ΔT is the change in temperature, equal to T_2 - T_1.

2.2 Practical processing approaches

In a strict sense, one should sample equation [Eq.1] at regular temperatures τ_i and measure the local elongation slope (the derivative). Practically speaking we apply equation [Eq.2] locally such that

$$\frac{dL(T)}{dT} \approx \frac{\Delta L}{\Delta T} \qquad \text{[Eq.3]}$$

with ΔL is the change in length in the temperature interval ΔT;
ΔT is the local change in temperature (at temperature τ_i),
with $\Delta T <$ temperature sampling interval $|\tau_i - \tau_{i+1}|$.

This imposes a stringent uncertainty requirement on the instrument when high sampling rate are applied, because this leads to small values of ΔT and ΔL. Indeed, for small ΔT, there is subsequently a small ΔL. The length measurement instrument needs to be accurate. Low measurement uncertainty, high resolution, and high repeatability would be the more appropriate wording in this case. Indeed, we measure small relative length differences. If α(T) is small and consequently ΔL is below the instrument resolution or sensitivity, ΔL=0 is measured locally.

This problem can be overcome by a step-and-hold thermal profile of the measurement. In such a sequence, $dL(T)/dT$ is derived from two thermal states of the sample at temperatures τ_i and τ_{i+1}. The sample is considered in static thermal equilibrium with its environment and has reached a homogeneous temperature. ΔT needs to be sufficiently large in order to associate an accurate measurement of ΔL (large with respect to the resolution). The CTE α(T) is derived from an interpolated value between these temperatures. The method typically takes a long time because it requires temperature uniformity of the sample.

Many authors [RD3, RD4] derive [Eq.1] with a different method. Length L(t) and temperature T(t) are measured continuously as function of time t. A polynomial is fitted through the L(t) points in function of time. The polynomial expression L(t) or, equivalently, the thermal strain $L(t)/L_0$ is then analytically derived versus temperature T(t). This approach means that our length measurement instrument would be capable to follow the full elongation/contraction of the sample over the

temperature range. Since the length measurements are made over a larger temperature interval, we are generally sure that we exceed the instrument resolution and relative length errors are reduced significantly with increasing integration interval. On the other hand, some concerns can appear on the temperature uniformity in the sample. This approach is proposed by most laboratories.

2.3 Major measurement uncertainty contributions

According to the definition of the CTE, three measurements are combined. From [Eq.1] we derive:

$$\frac{\Delta CTE(T)}{CTE(T)} = \frac{\Delta dL(T)}{dL(T)} + \frac{\Delta dT}{dT} + \frac{\Delta L_0}{L_0} \qquad \text{[Eq.4]}$$

with $\Delta CTE(T)/CTE(T)$ is the relative measurement uncertainty of CTE;
$\Delta dL(T)/dL(T)$ is the relative measurement uncertainty contribution of the elongation;
$\Delta dT/dT$ is the relative measurement uncertainty contribution of the temperature interval;
$\Delta L_0/dL_0$ is the relative measurement uncertainty contribution of absolute sample length.

These three instrumental contributors are statistically independent and can therefore be combined as a root squared sum (RSS):

$$\frac{\Delta CTE(T)}{CTE(T)} = \sqrt{\left(\frac{\Delta dL(T)}{dL(T)}\right)^2 + \left(\frac{\Delta dT}{dT}\right)^2 + \left(\frac{\Delta L_0}{L_0}\right)^2} \qquad \text{[Eq.5]}$$

The relative measurement uncertainty contribution of the sample elongation contains system instabilities like laser wavelength stability (interferometry), length readout, deformation of the optics and supporting hardware. In the relative measurement uncertainty contribution of the temperature interval, we have contribution of the difference between sample temperature and sensor, temperature inhomogeneity, gradients in the sample and calibration of the sensors. These contributors have been estimated by the laboratories and is used as input for this study.

The following contributors, among others, are missing in in the measurement uncertainty analysis:

- The effect of accumulation of contaminants on the sample [RD3] or due material inhomogeneity (non-uniform stress release in metals, non-uniformity in glasses, sample instabilities, creep, and hysteresis). It is therefore recommended to perform measurements on a number of samples.
- Temperature gradients in the sample are probably the most uncontrolled contributors, which explain that a measurement can differ for one measurement set-up to another.

In order to include these effects, the CTE was calculated as the derivative from a polynomial fit of the raw strain data. We fit the data to polynomial order (close to a physical model) and retrieve the fit–residual, i.e. a root mean square RMS deviation $\Delta dL/L_0$ of the data with respect to the model. $(\Delta\, dL/dL)_{RMS\_FIT}$ is finally added to the combined measurement uncertainty of the CTE:

$$\frac{\Delta CTE(T)}{CTE(T)} = \sqrt{\left(\frac{\Delta dL}{dL}\right)_{RMS\_FIT}^{\ 2} + \left(\frac{\Delta dL}{dL}\right)_{Instr}^{\ 2} + \left(\frac{\Delta dT}{dT}\right)^2 + \left(\frac{\Delta L_0}{L_0}\right)^2}$$

And equivalently:

$$\Delta CTE(T) = \sqrt{\left(\frac{1}{dT}\right)^2 \cdot \left(\frac{\Delta dL}{L_0}\right)_{RMS\_FIT}^{\ 2} + \left(\frac{1}{dT \cdot L_0}\right)^2 \cdot \Delta dL_{Instr}^{\ 2} + \left(CTE(T) \cdot \frac{\Delta dT}{dT}\right)^2 + \left(CTE(T) \cdot \frac{\Delta L_0}{L_0}\right)^2}$$

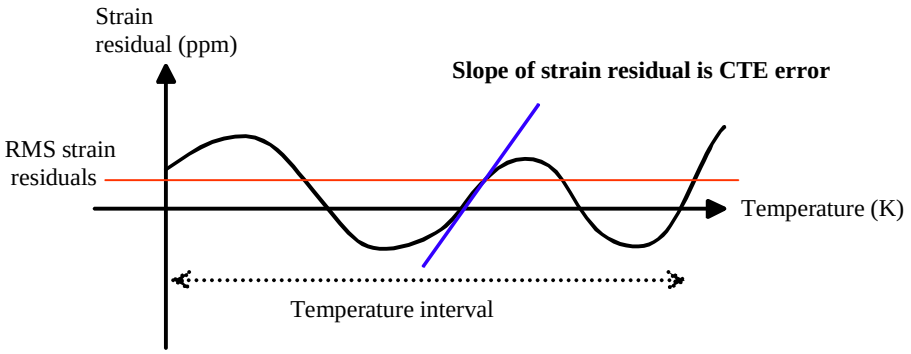
Note that ΔCTE(T) is proportional to the CTE(T) scaled by the sensitivity $\Delta dT/dT$ and $\Delta L_0/L_0$ respectively.

The sensitivity coefficient of $(\Delta\, dL/L_0)_{RMS\_FIT}$ was derived as described as follows. It is assumed that all the fitting residuals of the strain can be fitted by a polynomial of an order that is larger than the order N of the strain polynomial. We introduce the characteristic temperature step T_P of the polynomial fit residuals (Figure 1). T_P is the temperature interval ($\tau_{min} - \tau_{max}$) of the measurement divided by the strain polynomial order N plus one: N + 1. Then we apply following assumptions and hypotheses:

a) The CTE uncertainty is maximum at the zero transition of a polynomial fit through the strain residuals. This is a polynomial fit of order N+1.
b) According to Descartes rules, all zeros of the polynomial are real. There are N+1 zero crossings. It was also experimentally noticed that the polynomial coefficients alternate in sign.
c) We make the assumption that zeros are equally spaced, and so are the hills and valleys of the polynomial.
d) The effect onto the CTE is approximated by the RMS strain error divided by T_P.
e) This approach averages out spikes, discontinuities in the strain residual fit.

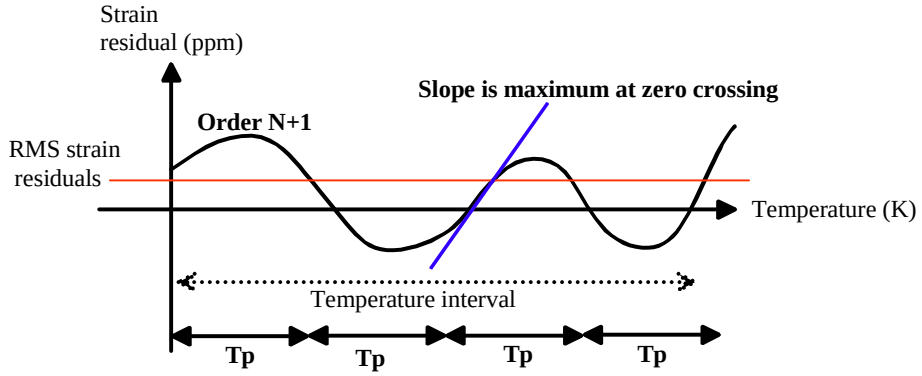
Finally we obtain the following formula that includes the instrumental measurement uncertainties and the sensitivity coefficient:

$$\Delta CTE(T) = \sqrt{\left(\frac{1}{T_P}\right)^2 \cdot \left(\frac{\Delta dL}{L_0}\right)_{RMS\_FIT}^{\ 2} + \left(\frac{1}{dT \cdot L_0}\right)^2 \cdot \Delta dL_{Instr}^{\ 2} + \left(CTE(T) \cdot \frac{\Delta dT}{dT}\right)^2 + \left(CTE(T) \cdot \frac{\Delta L_0}{L_0}\right)^2} \qquad \text{[Eq.6]}$$



Strain Residual (T) = Strain(T) - Polynomial fit order of order N (T)
Strain Residual (T) => Polynomial fit of ordcer > N

Strain Slope is CTE



Tp=Temperature interval/(N+1)

Figure 1 Estimation of the sensitivity coefficient of the statistical uncertainty contribution CTE caused by the polynomial fit

2.4 Test samples selection

A test matrix is proposed in Table 2. The test programme needed to emphasize the stable space materials and the Statement of Work (SoW) required a selection of one or more materials used as calibration standard (BK7). The BK7 standards from NIST are accepted standards around room temperature down to 150 K.

Interferometric test-benches require a high grade optical finish of the surface (e.g. PTB requires RMS Surface Figure Error = 20 nm). In some cases, a high parallelism of the surfaces, about 1 arcsec, is required. In some cases, an Aluminium coating (vapour deposited) is requested by the laboratory. Such characteristics affected the selection of sample materials for each institute.

SiC-100 and HB-Cesic have a CTE which is nearly zero for temperatures below 100 K, so we selected those materials for facilities capable of measuring at temperatures <100 K. One part of the round-robin activity emphasized the investigation of low measurement uncertainty. At PTB, which operated only close to room temperature, BK7 was selected as sample material.

Material	Size (mm)	Amount	LABORATORY	Temperature
SiC-100	50 x 6 x 6	2	ATK - Michelson Interfero	300 K – 25 K
HB-Cesic		2		
INVAR M93		2		
BK7	50 x 6 x 6	2	PTB – Abs Michelson Interfero	313 K – 283 K
HB-Cesic	12 x Ø12	1	SERCO-HT - Diverging beam Interfero	625 K – 300 K
SiC-100		1		
HB-Cesic	12 x Ø12	1	SERCO-LT - Diverging beam Interfero	300 K – 100 K
SiC-100		1		
SiC-100	500 x 30 x 10	2	MELCO - focus distance	300 K – 10 K
SIC 100	9 x 1 x 1	2	FZK - Capacitance Bridge	300 K – 5 K
INVAR M93		2		
HB-Cesic		2		
BK-7		2		
INVAR M93	101 x 6 x 6	3	PMIC-HT - Michelson Interfero	625 K –300 K
SiC-100		3		
BK-7		3		
INVAR M93	101 x 6 x 6	3	PMIC-LT - Michelson Interfero	300 K – 15 K
HB-Cesic		3		
SiC-100		3		
BK-7		3		
INVAR M93	50 x 6 x 6	3	CEA - LVDT Step & Hold	300 K – 4 K
HB-Cesic		3		
SiC-100		3		
BK-7		3		

Table 2 Measurement programme.

3. Results

3.1 Data analysis and reliability assessment

Most of the literature reports the strain ($\Delta L/L_0$) as a change in length versus temperature from some original length, generally measured at 293 K. All participating laboratories have delivered their data as strain versus temperature. While this is a practical way of measuring thermal expansion, the more fundamental property is the coefficient of thermal expansion (the derivative). A good "noise-filtering" basis is achieved by fitting a polynomial through the strain data in function of temperature, and to take the derivative of this polynomial resulting in a the CTE polynomial.

Note that this polynomial fit is only of practical use in the measured temperature interval. It represents a polynomial interpolation between data points based on a least square estimation. This polynomial represents nothing in terms of physics. Indeed, this polynomial fit cannot be extrapolated. In some cases, the material has some phase transition in the measurement range. Consequently, another physical model shall be used in that range.

Furthermore, we noticed that various laboratories proposed various polynomials for interpolation. No physical justification was made for their choice although there may exist a physical model that would give a reliable CTE prediction in the range.

In our approach, we tried to stay close to the physics of the expansivity. From an atomic perspective, thermal expansion is caused by an increase in the average distance between the atoms. Thermal expansion in crystalline materials is a relatively well-understood physical process. The relative rate, at which a material expands with increasing temperature usually falls within the range $1 \cdot 10^{-6}\ K^{-1} < \alpha < 20 \cdot 10^{-6}\ K^{-1}$ [RD7-9].

Examples of unconventional thermal expansion behaviour are well known, and often these have highlighted some interesting and important physical processes in the respective materials. For example, the balance between lattice thermal expansion and magneto-restriction in INVAR FeNi alloys gives rise to their well-known and widely exploited near-zero thermal expansion around room temperature.

One must be careful to the physical meaning when selecting the power in temperature terms of the thermal expansion models [RD9]. At low temperature, it is even better to use a Debye function. The Debye model is a solid-state equivalent of Planck's law of black body radiation, where one treats electromagnetic radiation as a gas of photons in a box.

According to models reported in literature, we relied on a third order polynomial fit model (as the Debye model) for the strain , except for INVAR. In most practical cases, we experienced the need to fit with a higher order model.

The following chapters show a summary of the most relevant CTE curves. For the complete data set see [RD19].

3.2 BK7

There is agreement within 0.4 ppm/K between the models obtained with LVDT (CEA), capacitive measurements (FZK) and interferometry (PMIC-LT).

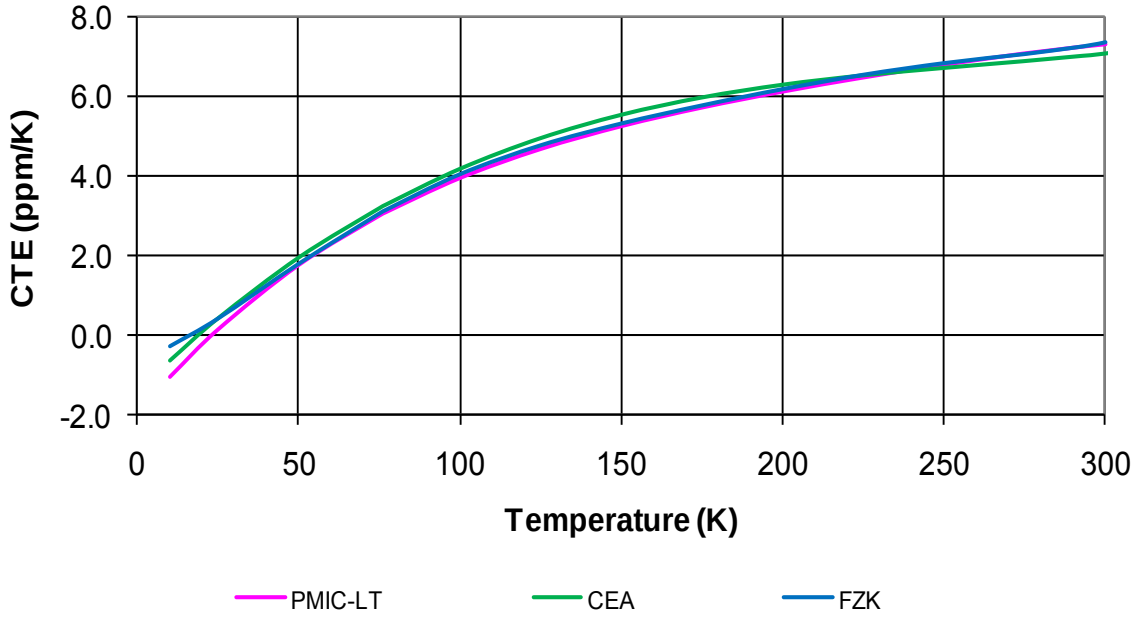


Figure 1. BK7 CTE models derived from data of various laboratories between 10 K and 300 K.

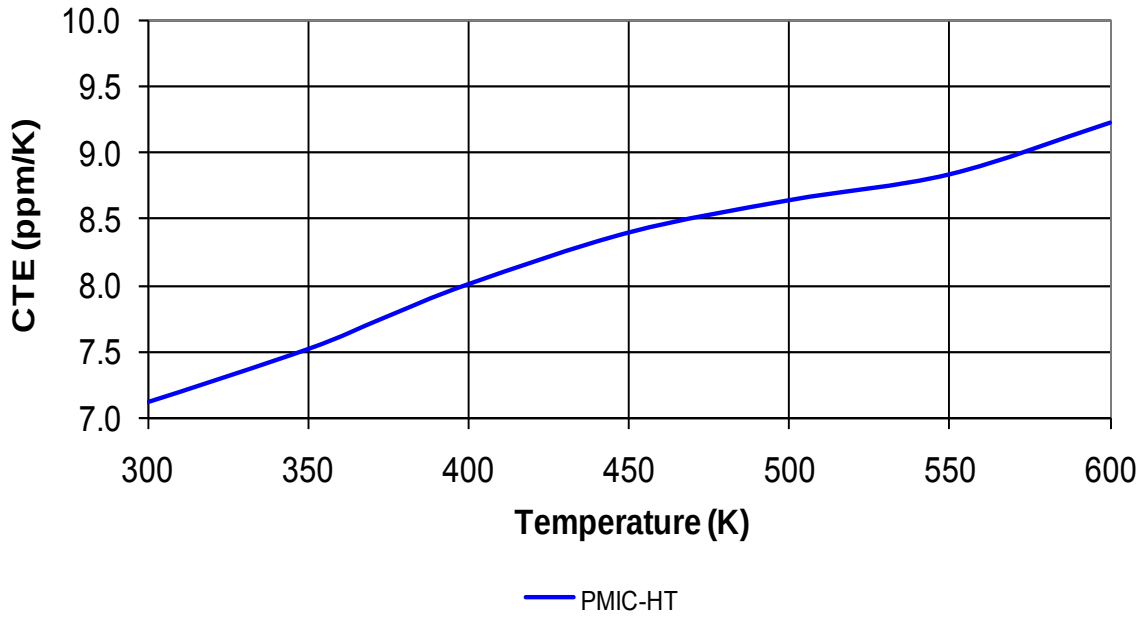


Figure 2. BK7 CTE model derived from strain data between 300 K and 600 K.

3.3 SiC-100

Fig 4 shows good agreement between 4 different measurement methods performed at MELCO, ATK, FZK and PMIC-LT. The models derived from ATK, FZK and MELCO data agree within 0.2 ppm/K in this temperature range. The SiC-100 CTE values agree within 0.1 ppm/K with respect to the data provided by JPL [RD3, RD4].

The models in fig 5 agree within 0.2 ppm/K over the temperature range from 300K to 600K.

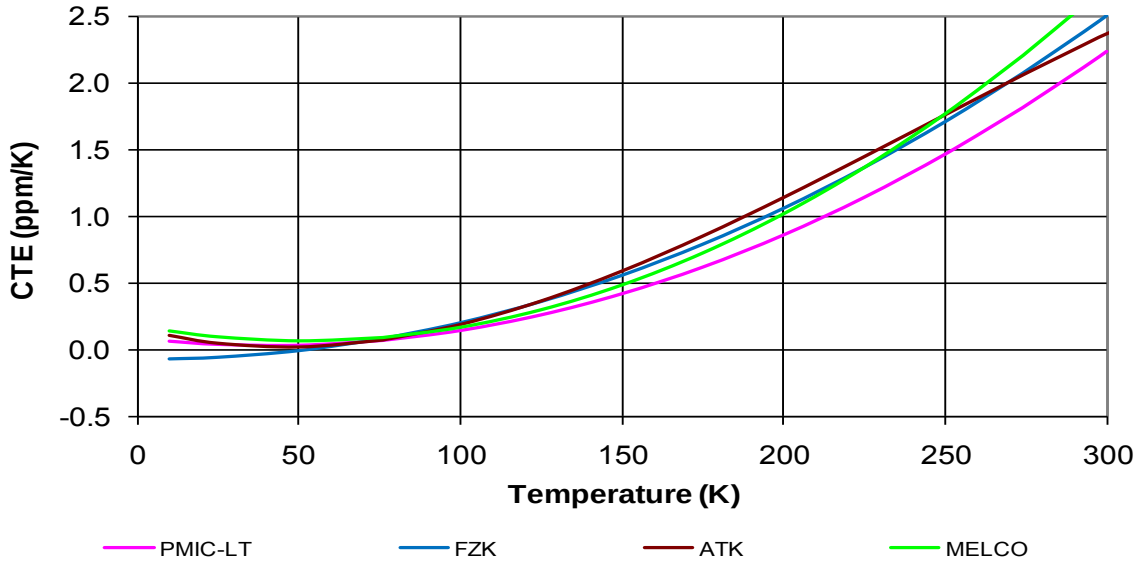


Figure 3. SiC-100 CTE models derived from data of various laboratories between 10 K and 300 K.

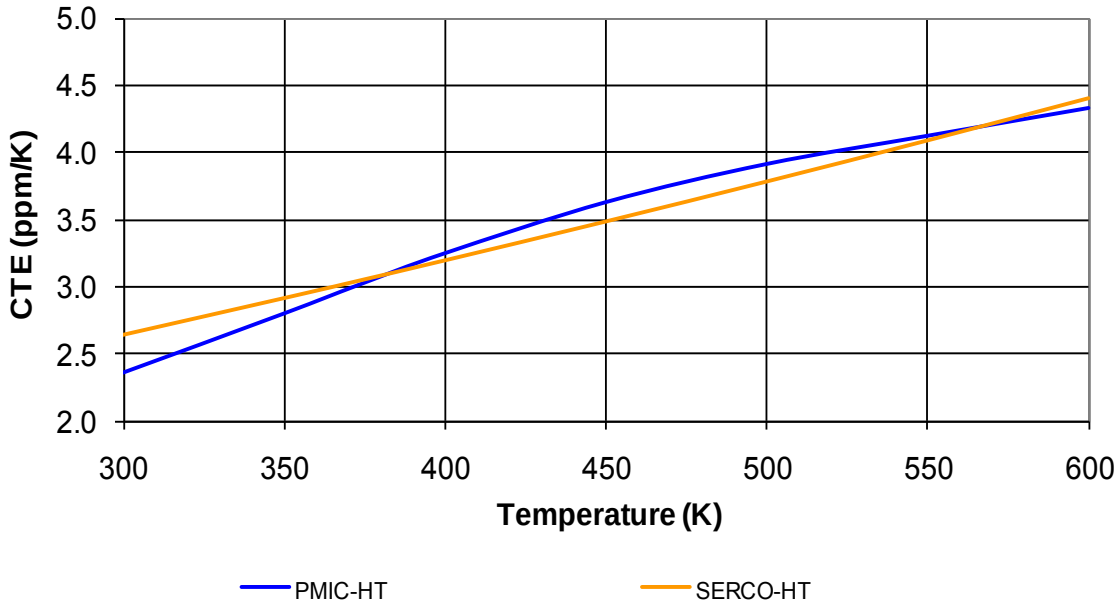


Figure 4. SiC-100 CTE models derived from data of various laboratories between 300 K and 600 K.

3.4 HB-Cesic

Fig 6 shows that the CTE models derived from data of FZK (based on capacitive sensor) and ATK, SERCO (based on interferometry) agree within 0.2 ppm/K above 100 K. The CTE values obtained are close to the ones measured by ECM (Figure 7). Some deviation appears at low temperatures (<25 K) where the CTE predictions of ATK and FZK differ from PMIC-LT as well as previous measurements from ECM, where a near zero CTE was measured. This could be due to a too high polynomial order of our fitting. The CTE differences with respect to tabulated data are within 0.2 ppm/K.

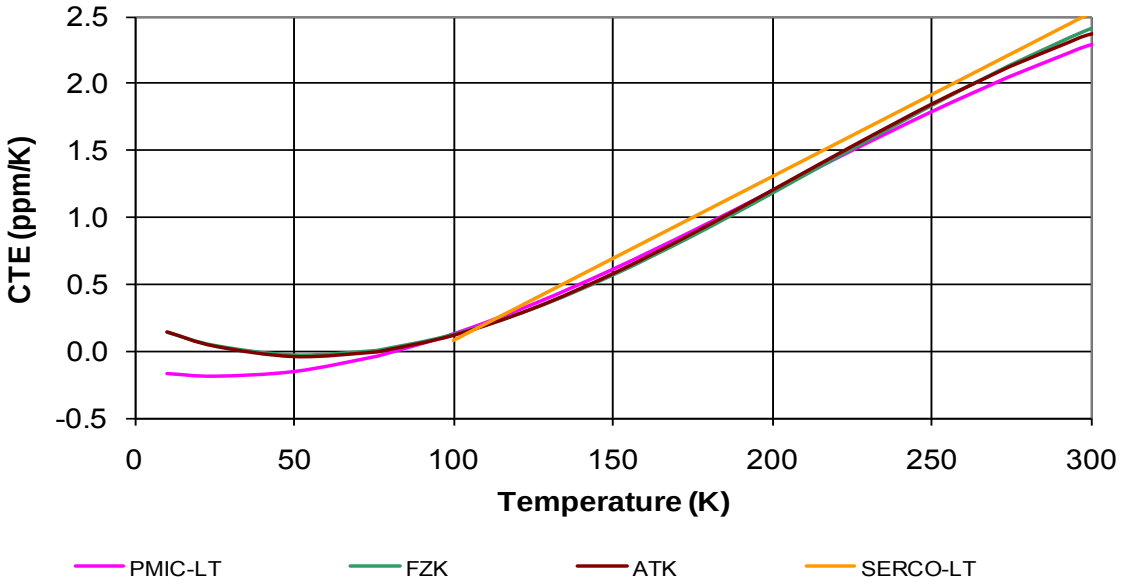


Figure 5. *HB-Cesic CTE models derived from data of various laboratories between 10 K and 300 K.*

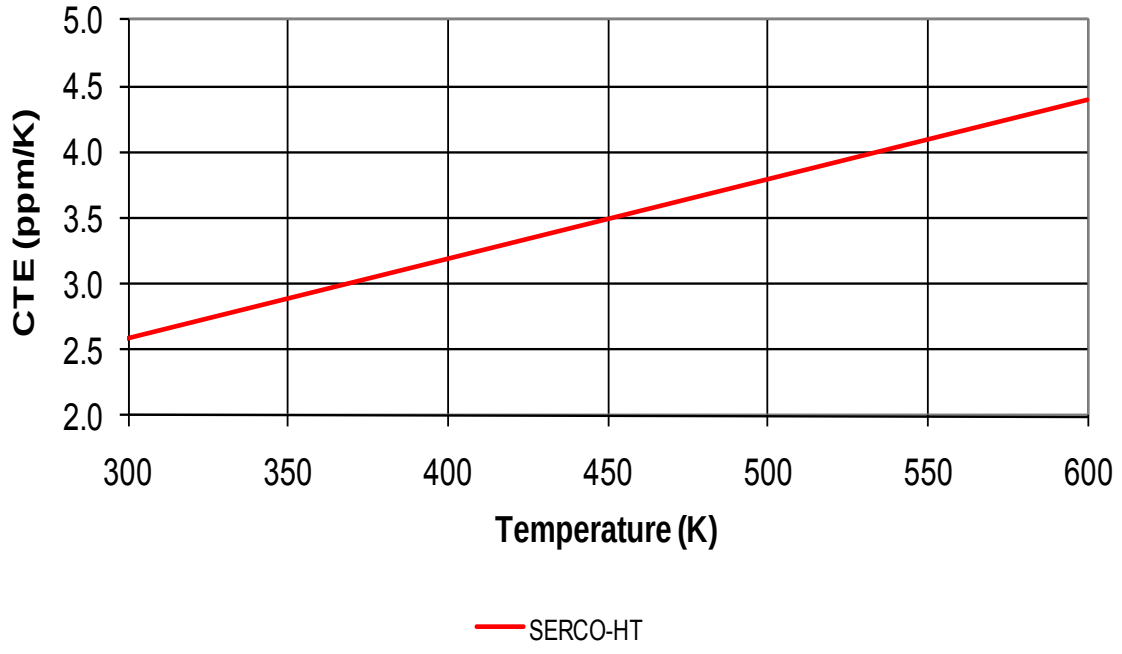


Figure 6. *HB-Cesic CTE model derived from strain data between 300 K and 600 K.*

Cesic® Data-sheet

Type MF – optical grade

Feed material	**Micro-fiber felt**
Fiber orientation	**Isotrop**
Max. temperature at permanent operation	**1673 K**

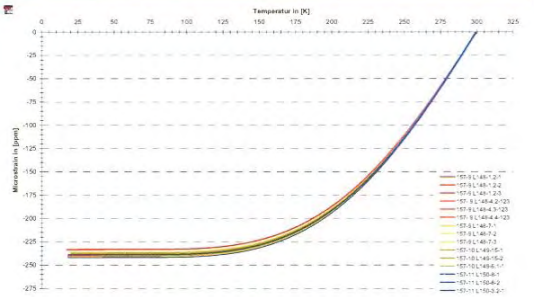
Mechanical Properties

Density	**2.65 – 2.70 g/cm³**		
3-Point bending strength			
293 K	**164 MPa***	Std. Dev. **22**	
90 K	**163 MPa***	Std. Dev. **20**	
4-Point bending strength			
293 K / 150 x 30 x 3 mm	**111 MPa***	Std. Dev. **16**	Weibull **11**
293 K / 80 x 10 x 3 mm	**149 MPa***	Std. Dev. **13**	Weibull **14**
Young's modulus	**249 GPa***	Std. Dev. **20**	
Fracture toughness K_{IC}	**4.62 MPa $m^{1/2}$ ****		
Poisson ratio	**0.17**		

Thermal Properties

CTE

20 K – 85 K	**0.00 10^{-6} / K***
85 K – 120 K	**0.06 10^{-6} / K***
120 K – 180 K	**0.43 10^{-6} / K***
180 K – 220 K	**1.07 10^{-6} / K***
220 K – 300 K	**2.09 10^{-6} / K***
313 K – 393 K	**2.74 10^{-6} / K****



Thermal conductivity (λ)	
293 K	**121 W / m K****
Specific heat capacity	
293 K	**0.8 J / g K****
1473 K	**1.2 J / g K****
Thermal shock parameter R_1	**169 K****

(R_1 equals the max. temperature increase, ΔT, applied suddenly to the surface that Cesic® can tolerate without damage.)

Thermal shock parameter $R_2 = \lambda R_1$ **2.04 10^4 W / m****

Electrical Properties

Electrical conductivity (293 K) **6.1 10^{-3} Ωm****

*Reference to ESA, **ECM measurements

Figure 7. ECM's HB-Cesic data.

3.5 INVAR M93

Fig 9 shows the CTE models for various measurement methods (ATK and PMIC-LT based on interferometry, CEA based on LVDT and FZK based on capacitive cell). The CEA curve is systematically higher than the other curves. PMIC-LT and ATK models correspond within 0.2 ppm/K. The INVAR CTE values differ less than 0.2 ppm/K with respect to data obtained by JPL [RD3]. We noticed discrepancies at low temperatures (<25 K) where all curves show significant disagreement, probably caused by a too high polynomial order.

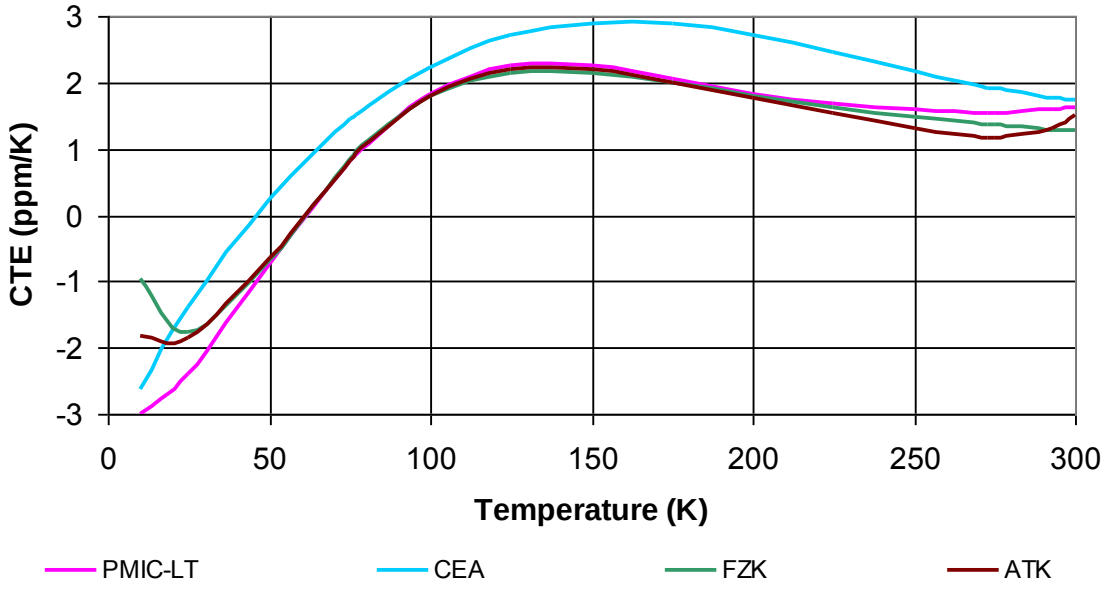


Figure 8. *INVAR CTE models derived from data of various laboratories between 10 K and 300 K.*

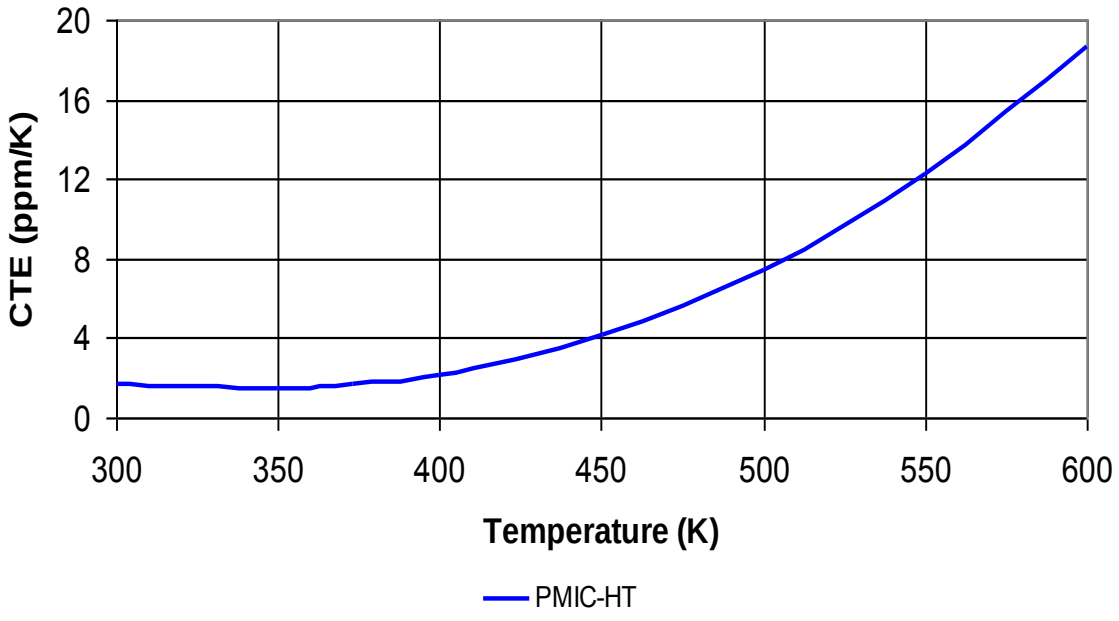


Figure 9. *INVAR CTE model derived from strain data between 300 K and 600 K.*

4. Conclusions

A first general conclusion is that laboratories have mostly achieved their claimed CTE measurement uncertainties, as shown in the last column of table 1.

Our analysis has conducted to the conclusion that FZK and ATK provide high(est) reliability on the CTE models in the cryogenic temperature range. FZK has the set up with the highest (relative) resolution, lowest noise in their data in the largest temperature range with the smallest temperature in-homogeneities. The weak point of this set-up is that it requires an absolute CTE calibration standard, to allow correction for the systematic error caused by the intrinsic expansion of the cell. This drawback is similar for push rod dilatometers such as for CEA where an absolute standard needs to be used to remove the push-rod CTE contribution.

The good agreement between the CTE models constructed based on FZK and ATK data are generally confirmed by PMIC-LT datasets followed by CEA and MELCO. The last two laboratories have a weaker system resolution which is compensated by large temperature intervals (i.e. 50 °C) in the case of CEA and by large sample lengths (i.e. 500 mm) for MELCO. Nevertheless, it is very useful to have data sets with various measurement methods as origin (interferometry, capacitive sensor, LVDT, optical focussing) converge to the same CTE models, like in our case within 0.2 ppm/K. In this case, one can state that the obtained CTE values have an uncertainty of 0.2 ppm/K between 10 K and 300K.

PTB has demonstrated a very low uncertainty at room temperature (283 K-313 K). At the time of this study there was no evidence that the proposed set-up can be transposed “as such” to cryogenic temperature and provide the same (exceptional) uncertainty. The most problematic effect that we expected was the equilibrium temperature of the residual temperature gradients that are present in an experimental set-up combining optics and cryogenics.

The following discusses the facilities and methodologies allowing the most accurate and reliable CTE determination available today, including the test conditions within which they are the best ones. Besides FZK and ATK, the following three calibration laboratories showed potential advantages but did not fulfil all requirements:

- PTB => Participated in the study but did not cover full temperature range
- NMIJ National Metrology Institute of Japan => Not available to participate
- JPL => Not available to participate due to ITAR restrictions

The limitations of the facilities and methodologies and the uncertainties on the CTE value measured:

- ATK’s facility is the most versatile from the point of view of sample size and shape as it measures non-optical and optical materials.
- PTB’s facility is good for optical materials with reflective sides.
- FZK’s facility has specific and critical requirements in terms of samples size and shape.

The potential complementarities and recommendations for equipment and technologies leading to a more accurate determination of CTE and / or extension of the temperature range:

- Interferometric methods (ATK, PTB, JPL, NMIJ) and capacitance cell (FZK) methods are the most accurate methods.
- Interferometric methods require the use of wavelength stabilized lasers (ATK, PTB, JPL) or heterodyning (JPL).
- Use of absolute calibrated cryogenic silicon-diode sensors such as Lakeshore DT470 down to 4 K (thermocouples are not accurate enough) (ATK, PMIC, FZK) is required.
- Recurrent calibration of the facility with a standard material (e.g. single crystal silicon SCS) and cross-checking the calibration with a second calibration standard material (e.g. Copper) is required.

- Thermal design of the cryogenic facilities (ATK) (understanding heat exchange with the sample, temperature gradients in steady state, transient temperature effects, adjustment of heat and cooling rate) or the use of step and hold methods (CEA, PTB) are required.
- Use of a thermally conductive gas (e.g. 100 mbar He) is required to allow temperature homogenisation around the samples.
- Methods which are useable for optical as well as non-optical materials are required, which may drive the choice for non-interferometric (FZK, CEA) or interferometric (PMIC, ATK, SERCO) methods.

The most promising concepts of methodology to be developed to decrease the measurement uncertainty of CTE measurements are:

- Extend the temperature range of the absolute interferometric length measurement method as proposed by PTB to the cryogenic and high temperature range.
- Develop high-precision calibration standards to compare the best laboratories world-wide having absolute measurement capability and to improve the absolute CTE modelling of other laboratories.
- Take advantage of the unprecedented relative resolution of the capacitive technique used by FZK by calibration with high-precision standards to decrease the uncertainty on the absolute CTE values. Another improvement would be to develop a capacitance cell using low-CTE material instead of the Copper cell used by FZK.

5. Lessons learned

5.1 Objectives

The objectives of this measurement program are a) to identify the facilities with 10 ppb/K CTE measurement uncertainties and b) to make a comparison among these centres by measuring the CTE of low CTE materials (1 ppm/K) from typically 600 K to 5 K (emphasis set on 300 K to 5 K). The project has partially succeeded with respect to these two (ambitious) aims. In our quest to these goals, we have faced several practical difficulties. These practical problems have clearly modelled our road map. We highlight them in this chapter.

5.2 <10 ppb/K uncertainty CTE metrology in Europe

At the time of the activity, we identified two metrology facilities claiming in their publications to reach better than 10 ppb/K CTE uncertainties in the range of 300 K to 5 K: JPL (US) and NMIJ (Japan). Both have declined participation to the round-robin activity.

The only laboratory in Europe which has proven to have a <5 ppb/K CTE uncertainty in the 283 K-313 K range is PTB (Germany) in very favourable and precise sample conditions (polished samples with optical quality). At the time of this study there was no proof that this uncertainty can be reproduced under other conditions, but we believe that 10 ppb/K is achievable based on the experiments performed by NMIJ and JPL.

There is one laboratory that has proven 1 ppb/K resolution in the 5K-300K range: the capacitance cell of FZK (Germany). The method is a relative CTE measurement. The CTE of the capacitance cell needs to be subtracted by using an absolute calibration standard. The uncertainty of the absolute measurement is limited to the uncertainty of that standard, whereas the uncertainty on the relative measurement is orders of magnitude lower.

We discovered also that the need of a <10 ppb/K uncertainty in CTE metrology (300K-5K) is mostly driven by the space sector. Currently, there are not many industrial needs (potential customers) for low uncertainty at such low temperatures. Some laboratories, having set-ups for cryogenic temperature are historically associated to the nuclear sector (FZK, SERCO, CEA).

5.3 Need for calibration standards

High-precision calibration standards are needed. They allow calibrating out systematic errors of a set-up and they are suitable for round-robin comparisons. We recommend that calibration standards shall be made of a mono-crystalline material with high purity that can be polished (to allow comparison with optical and non-optical metrology methods). Single crystalline Silicon (SCS) seems in this respect a suitable material; it has a moderate CTE around room temperature (about 3 ppm/K). SCS can be mono-crystalline and very pure, when grown by means of chemical vapour deposition (CVD).

5.4 The sample problem

We faced an immediate problem within the round-robin activity as it was impossible to let circulate a unique sample for measurement comparison. Most laboratories in the USA comply with a sample shape of 6 x 6 x 50 mm, simply, because NIST provides standards with this shape. Laboratories have all used various shapes and sizes (PTB, MELCO, CEA, FZK, SERCO). In some cases a certain shape was a strict requirement for the validity of the calibration (such as for CEA).

We recommend that a measurement laboratory shall also be responsible to manufacture and machine the sample to their required standard. We recommend also that an acceptable shape shall be used, to allow transfer of the calibration standards. In order to allow comparison with non-European countries 6 x 6 x 50 mm is suggested.

The surface preparation of some sample materials cannot be performed to the optical quality required by some facilities to achieve the best possible measurement conditions. The measurement setups need to be capable to cope with optical (polishable) and non-optical materials. Artefacts affecting the uncertainty shall be quoted in advance by the test facilities.

The material history is also important. Materials shall be tested taking into account the thermal history they have undergone. Thermal ageing can also be of importance, especially at this degree of uncertainty. In some cases, this may not allow for retesting as the measurement method itself may affect the expansion behaviour of the material. This typically results in a hysteresis of the measurement when passing a temperature repeatedly in various cycles.

We touch here also a fundamental problem related to the low uncertainty of the CTE measurement and the real thermal expansion undergone by a structure in space. A strict CTE definition is only possible when the material is free from time dependent effects and hysteresis. We know that in space, structures or experiments (especially the one observing the earth) can be submitted to varying thermal loads. It may be necessary to know the thermo-elastic behaviour in representative transition conditions, as well as to know the CTE difference between materials coupled within the structure. The possibility to measure a fast transition representative to space environment and CTE mismatch starts with the capability to measure CTE with low uncertainty.

5.5 Thermal design and measurement time

The operational cost of a measurement cycle is typically proportional to the measurement time. Our observation was that development of most set-ups is driven by metrology capability. Low uncertainty temperature sensors are used (Lake Shore DT-470), which allow quoting of the uncertainties of the set-up. However, most of the time there is no associated thermal design providing analysis of temperature uniformity.

We recommend, when building cryostats for low uncertainty CTE measurements, that the design is supported by thermal analysis of the sample temperature distribution at equilibrium and the evolution of the sample temperature in transient regimes. This will allow recommending a transient temperature (heating) rate for various materials (having a thermal conductivity and heat capacity, which are also temperature dependent).

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