THE JPL CRYOGENIC DILATOMETER: MEASURING THE THERMAL EXPANSION COEFFICIENT OF AEROSPACE MATERIALS.

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ABSTRACT

Opto-mechanical systems such as the James Webb Space Telescope and the Herschel Space Telescope must maintain accurate dimensions over a wide range of temperatures. One piece of information essential to a successful instrument is the thermal expansion coefficient (CTE) of the component materials measured over the expected range of temperatures. Other important data are stability (creep), and for actuators, the stroke as a function of temperature. From room temperature to below 30 K, the JPL dilatometer has measured CTE and creep for a variety of materials including ULE, Zerodur, fused silica, single-crystal silicon, silicon carbide, copper, Invar and PMN actuators. It has also tested the CTE and stroke of piezo-electric actuators.

This paper updates the status of the JPL cryogenic dilatometer, presents improved error estimation, and recent measurements of silicon carbide and Invar.

INTRODUCTION

The JPL cryo-dilatometer[1,2,3,4] was built to support the engineering of space telescopes, such as the James Web Space Telescope (JWST), Terrestrial Planet Finder, the Space Interferometry Mission. The dilatometer measures the thermal strain and CTE of typical optical and opto-mechanical materials from ~20 K to 324 K.

The CTE accuracy can be as good as ~10 ppb (units are 1/K) in CTE, however, actual accuracy is highly sample dependent. Materials with high CTE, rapidly changing CTE as a function of temperature, low thermal conductivity, or inconvenient mechanical properties are problematic. Some of these problems were experienced in the work reported here: the measurement of the thermal strains of SiC and Invar at a 70 K target temperature.

The measurements were challenging because:

- Invar has low thermal conductivity, which enhances thermal gradients in the sample, increasing the temperature uncertainty.
- Invar's strain as a function of temperature is feature-rich, making the CTE uncertainty and to some extent the strain uncertainty more sensitive to temperature errors.
- The multi-crystalline SiC samples could not be polished to a smoothness level that allowed robust optical contacting, hence the samples were extremely fragile. This caused a temperature sensor attachment problem that ultimately increased the temperature uncertainty.

These problems will be discussed further, but first a review of the facility.

THE JPL CRYO-DILATOMETER FACILITY

Figures 1 and 2 indicate the major features of the dilatometer. Four downward beams of laser light form part of a Michelson interferometer that measures the relative distances to the top of a nominally 25 mm tall sample "pillar" and to a sample "base" that the pillar rests on. As the sample temperature varies, the interferometer detects a change in the reflected laser beams' optical phases. By subtracting the temperature dependent phase of the beam reflected from the pillar from the phase of the beams reflected from the base, we obtain the relative pillar height, in units of laser wavelength. This, and other aspects of the dilatometer are explained in greater detail in the references[1,2,3].

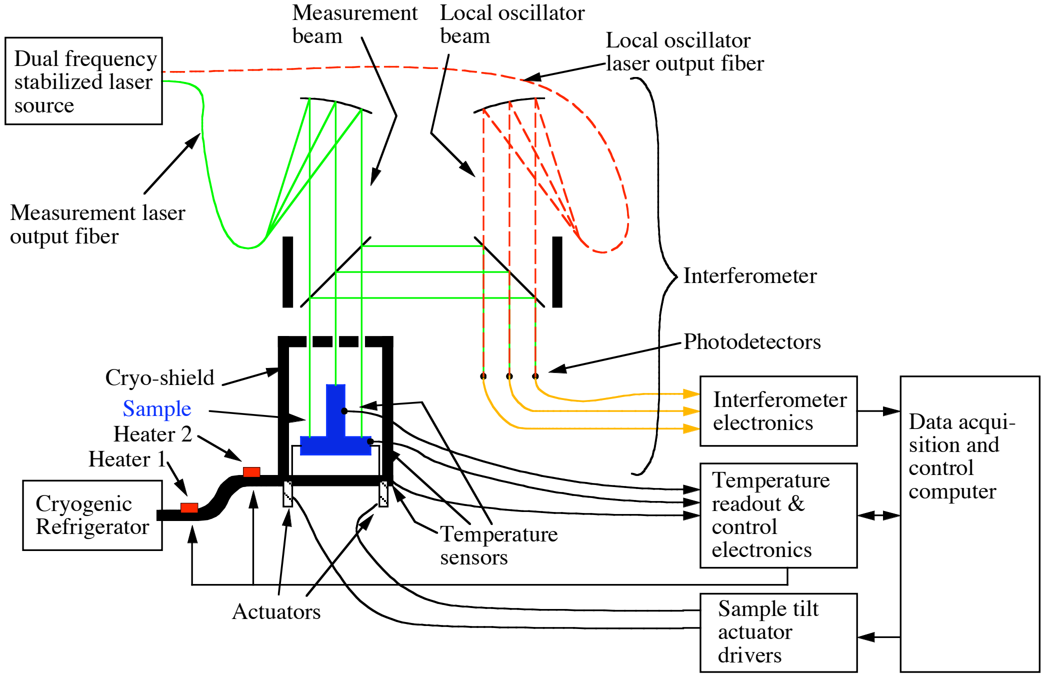

Figure 1. high-level diagram of the JPL cryo-dilatometer.

Dilatometer upgrade: iodine stabilized laser

A recent improvement to the dilatometer was the replacement of the laser. The previous laser, while reasonably stable thanks to a thermo-electric controller, nevertheless had a frequency drift of roughly 50 MHz over several hours. A 50 MHz laser drift would be indistinguishable from a 0.1 ppm strain, for a 25 mm sample pillar.

By locking the laser to an iodine gas cell, the drift is reduced to ~300 Hz, or $5x10^{-13}$ in strain. Some of the details of the new laser are:

- Vendor: Innolight[5]
- Model: “Prometheus” 1 W @532 nm, doubled Nd:YaG
- Iodine stabilization: stable to $5x10^{-13}$ (10,000 seconds)
- Iodine line frequency, wavelength: $5.63260223471x10^{14}$ Hz, 532.613501716 nm

Overall, the effect of the upgrade was to remove laser instability as a significant source of error.

SAMPLES TESTED

Xinetics Silicon Carbide

One SiC sample was provided by Xinetics[6]. Some of its properties[7,8,9] include:

- Multi-crystalline mixture: mostly SiC with some fraction of silicon.
- Complex shapes possible by casting the “green” precursor.
- Can be polished to mirror finish, but crystal boundaries scatter (this could be remedied with silicon or CVD SiC cladding).

The sample pillar and base, shown in figure 3 (left), although polished to a fraction of a wave, could not be optically contacted because of the crystalline micro-texture. As a work-around we chose to "glue" the pillar to the base using thin layer of vacuum grease. The thickness of the grease, ~1 micron, was small enough that its presence is a minor contributor to the strain and CTE measurement error. More problematically, the weak base-pillar bond prevented us from attaching the temperature sensors to the pillar so the temperature readings were restricted to the SiC base, increasing the pillar temperature uncertainty.

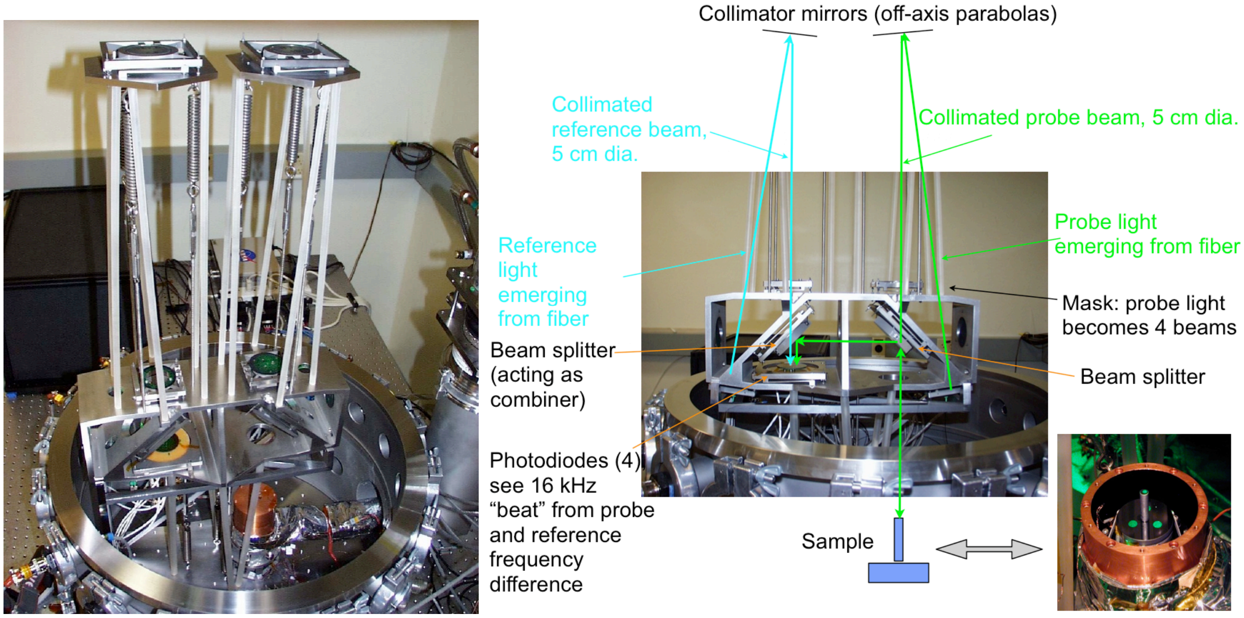

Figure 2. Inside the dilatometer vacuum chamber: the interferometer assembly as seen from above (left) and from the side (right) with the reference and probe laser beams indicated. Inset shows a silicon carbide sample (from Xinetics) illuminated by the four measurement beams.

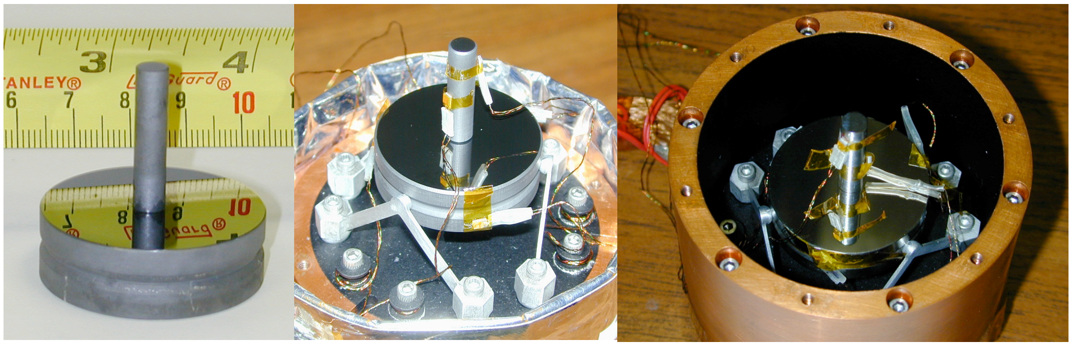

Figure 3. Xinetics silicon carbide sample (left), Boostec silicon carbide sample with temperature sensors attached (middle), and Imphy Invar M93 sample (right). A very thin (~1 micron) layer of vacuum grease tacked the Xinetics pillar to its base while the Boostec SiC pillar was successfully optically contacted. For the Invar, a small magnet under the base immobilized the pillar.

Boostec Silicon Carbide

Another SiC sample, provided by ESA, was fabricated by Boostec[10]. Some of the properties of this material include[11]:

- Multi-crystalline, nearly 100% SiC.
- Complex shapes possible by machining the "green" precursor.

- Can be polished to mirror finish, but there is a surface porosity that scatters (this could be remedied with CVD SiC cladding).

The sample pillar, figure 3 (middle), was optically contacted to the sample base. However because the bond was very weak, we had difficulty attaching temperature sensors to the pillar and eventually the lower sensor detached. These problems cause an increased sample temperature uncertainty.

Imphy Invar M93

A second sample provided by ESA was made of Invar, a low CTE iron-nickel steel fabricated by Imphy Alloys[12]. Some of its properties are

- M93 alloy, mostly Fe, 35 to 36.5% Ni, 0.2 to 0.4% Mn
- Used in liquified natural gas transport applications (pipelines, tankers), 110 Kelvin typical operating temperature
- Ferromagnetic.

The sample pillar provided, figure 3 (right), could not be optically contacted to the sample base, probably because the surfaces were not sufficiently polished. As a work-around, a small magnet placed under the base created a magnetic field just strong enough the to hold the pillar onto the base's surface. A check of the residual magnetic field confirmed that the temperature sensors would not be affected[13]. (This is often a concern when measuring cryo temperatures near superconducting magnets.)

Single-crystal silicon

JPL maintains a stock of single-crystal silicon sample pillars, optically contacted to silicon bases. High purity silicon is easily obtained from microelectronics industry and its CTE is independent of crystal orientation, fabrication method and source, thus it is a convenient reference material for comparison with other labs. JPL CTE measurements[4] agree with the literature[14] to ~10 ppb (35 to 300K), and JPL strain measurements agree to better than 1.3 ppm down to 30 K.

RESULTS

The measured strain and CTE values are presented in figures 4 and 5, together with results from the literature for single crystal silicon[14] and for single-crystal silicon carbide[15,16]. For readers who wish to reproduce the curves, the polynomials in tables 1 and 2 can be used to reconstruct the strains and CTEs as a function of temperature.

Considerations For Communicating Thermal Expansion Measurement Accuracies

Because of the highly disparate thermal/mechanical properties of materials measured, and because of the diversity of uses the measurements will be used for, it is important that we present enough information for future users of the data to establish how accurate the data is *for their application*. This means that it is not enough to just say "we can measure CTE (or strain) to accuracy *E*".

At the risk of sounding pedantic, we can state that for thermal strain and CTE measurements to be useful, we must answer the following questions over the temperature range under consideration:

1. How well do we know the sample temperature?

2. How well do we know the change in sample length?

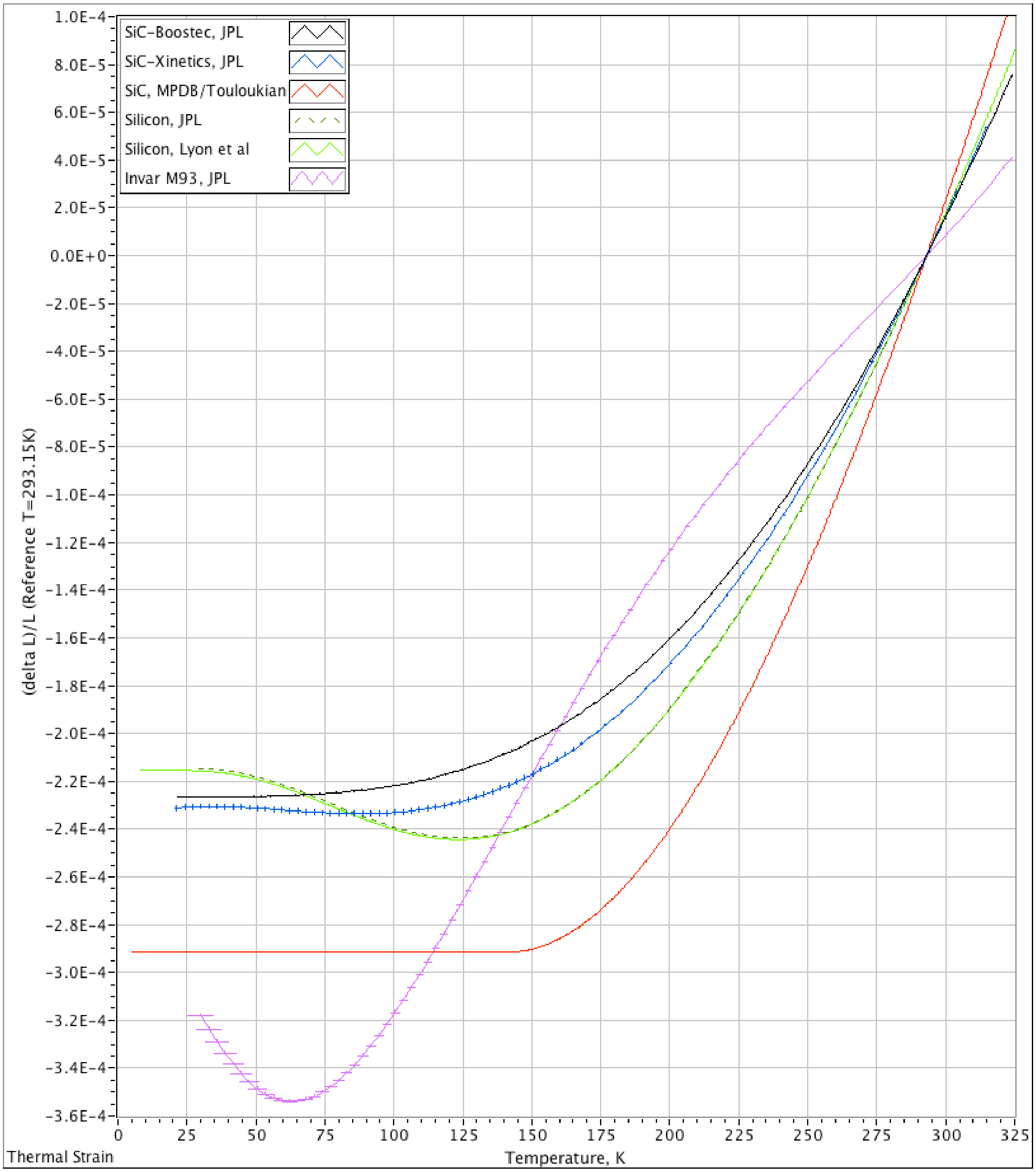

Figure 4. Measured strains, referred to 293.15 K, for Boostec SiC, Xinetics SiC, Imphy M93 Invar and single crystal silicon. For comparison, measurements from the literature have been added for single-crystal silicon[14] and SiC[15,16]. All of the JPL measurements, except for the silicon, have horizontal error bars for the temperature uncertainty, and vertical error bars (almost imperceptible at this scale) for the length change uncertainty.

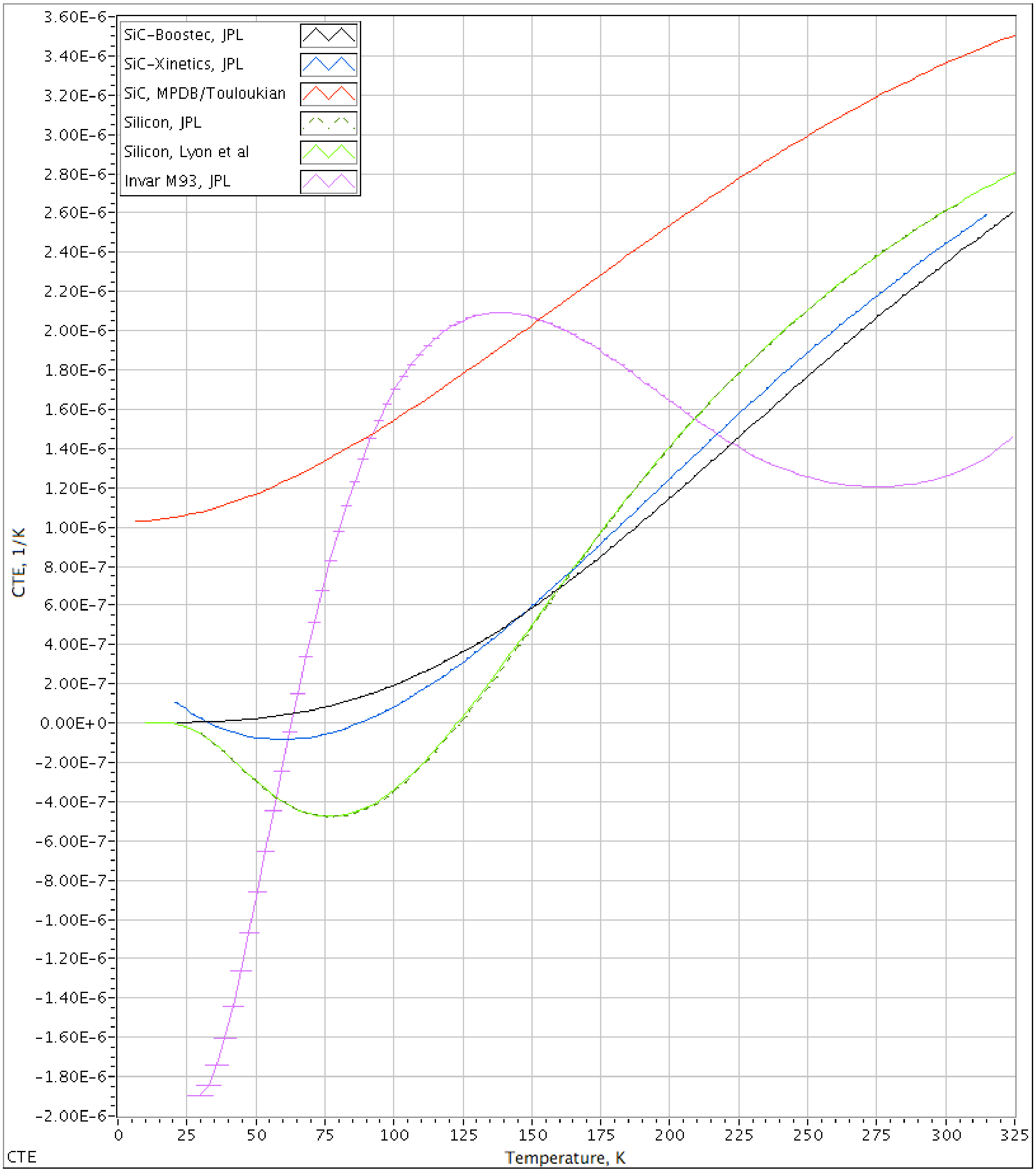

Figure 5. Measured CTEs (1st derivatives of strains) for Boostec SiC, Xinetics SiC, Imphy M93 Invar and single crystal silicon. For comparison, measurements from the literature have been added for single-crystal silicon[14] and SiC[15,16]. All of the JPL measurements, except for the silicon have horizontal error bars for the temperature uncertainty. (Vertical error bars have not been included.)

As an example, the horizontal error bars in figure 4 indicate the uncertainty in the sample temperature which is large for Invar because of its low thermal conductivity. The vertical error bars for the strain uncertainties are small enough that they are difficult to be seen on this figure. What this tells us is that although our answer to question 2 is “very accurately indeed” (to a few nm), in the case of the Invar measurements at least, that accuracy is somewhat undermined by the large temperature uncertainty.

Accuracy/Error Budget

The work reported here gives special attention to the strain at 70 K, as this was the most likely operating temperature of the equipment the samples represented.

The errors in measuring the strain can be categorized and estimated as follows with the dominant errors (at 70 K) shown in bold:

- System instabilities, inherent. This includes laser wavelength, length readout, temperature readout, deformation of the optics and supporting hardware. (+/- 300 pm or +/- 1.2x10^{-8})
- System instabilities, temperature dependent. (+/- 250 pm or +/- 1x10^{-8})
- Error due to the accumulation of contaminants on the sample. This has emerged as a significant issue but it only affects data taken below 170 K. (+/- 2 nm or **+/- 8x10^{-8}** for Boostec SiC, +/- 25 nm or **+/-1x10^{-6}** for Xinetics SiC, -19 nm or **-7.6x10^{-7}** for Invar) The error for Invar was asymmetric.
- Interferometer nonlinearity. (+/- 115 pm or +/- 4.6x10^{-9})
- Sample instabilities, creep and hysteresis. (Zero; undetectable for these samples.)
- Sample length at room temperature measurement error. Strain error scales with strain. (+/- 1 micron or +/- 9x10^{-9} for SiC at 70K, +/- 1.4x10^{-8} for Invar at 70 K)
- Temperature measurement errors: calibration[13]. Strain error scales with CTE. (+/- 0.25 K or +/- 1.6x10^{-8} for SiC, +/- 1.1x10^{-7} for Invar at 70 K)
- Temperature measurement errors: sensor contact with sample, sample temperature gradients and thermal lag. Strain error scales with CTE. (+/- 0.1 K or +/- 6.6x10^{-9} for SiC at 70 K. +/- 2.5 K or **+/- 1.1x10^{-6}** for Invar at 70 K.)

For specific case of strain at 70 K, these errors sum to give an uncertainty of +/- 1x10^{-6} for Xinetics SiC, +/-1.4x10^{-7} for Boostec SiC and +1.1x10^{-6}/-1.9x10^{-6} for Invar. (Using RSS sums gives somewhat smaller errors.) In the case of Invar, the error is asymmetric. If one thinks of *error bars* as indicating where the *truth* lies, then the “answers” at 70 K are:

•Strain, Xinetics SiC at 70 K = -232.7 +/- 1 ppm
•Strain, Boostec SiC at 70 K = -225.5 +/- 0.14 ppm
•Strain, Imphy Invar M93 at 70 K = -352.4 +1.9/-1.1 ppm
•Silicon, for comparison:
JPL: -225.9 ppm
Lyon et al: -226.9
relative to 293.15 K.

The procedure for estimating strain uncertainty at temperatures other than 70 K is as follows:

1. The temperature uncertainty can be estimated with the data in figure 6. These plots show the differences in temperature from one location on the sample to another, i.e. apparent temperature gradients, which are indicative of the temperature errors.
2. The length measurement errors listed above that applied to 70 K can still be used with the exception of the contamination error.

3. The effect of the contaminants is a step function: above 170 K it is zero, below 170 it is the value in the list above.

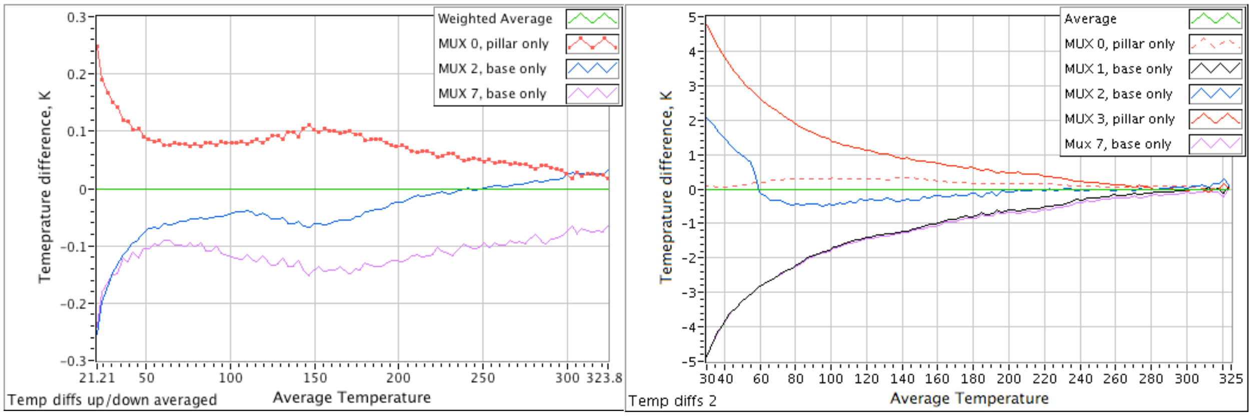

Figure 6. Disagreement between temperature sensors placed on the Boostec SiC (left) and Imphy Invar (right). The temperature differences are mainly due to uneven cooling of the samples. Invar has a low thermal conductivity which leads to larger temperature gradients.

Material	Strain Polynomial, referred to 293.15 K	Range
SiC, Boostec	-2.2657759E-4 -5.8446496E-9T +3.1697174E-10T^2 -7.9364135E-12T^3 +1.3477868E-13T^4 -3.6873692E-16T^5 +3.2131869E-19T^6	22 to 324 K
SiC, Xinetics	-2.3629734E-4 +4.1102516E-7T -9.2041575E-9T^2 +6.6373348E-11T^3 -1.3110218E-13T^4 +9.3714352E-17T^5	22 to 315 K
Silicon crystal	-2.09686420742702E-04 -6.79449065133125E-07T+3.73588146712838E-08T^2 -9.54332538414880E-10T^3 +1.14551460901481OE-11T^4 -8.00348056849206E-14T^5 +3.67876889144067OE-16T^6 -1.14556044207323E-18T^7 +2.33790823596450OE-21T^8 -2.81507343297412E-24T^9 +1.50799224891955OE-27T^10	30 to 324 K
Invar M93, Imphy	-3.0831959E-4 +3.9075588E-6T -2.7049666E-7T^2 +5.9917892E-9T^3 -6.9024193E-11T^4 +4.9563418E-13T^5 -2.3406273E-15T^6 +7.2741764E-18T^7 -1.4311513E-20T^8 +1.6146381E-23T^9 -7.9527239E-27T^10	35 to 305 K

Table 1. Measured strains, converted to polynomial fits. The large number of digits are needed for accurate polynomial evaluations and do not imply accuracy beyond what is claimed elsewhere in this paper. Extrapolation beyond the specified temperature ranges is not recommended.

Material	CTE, 1/K	Range
SiC, Boostec	-5.8446496E-9 +6.3394348E-10T -2.38092405E-11T^2 +5.3911472E-13T^3 -1.8436846E-15T^4 +1.9279121E-18T^5	22 to 324 K
SiC, Xinetics	+4.1102516E-7 -1.8408315E-8T +1.99120044E-10T^2 -5.2440872E-13T^3 +4.6857176E-16T^4	22 to 315 K
Silicon crystal	-6.794490651331250E-7 +7.471762934256760E-8T -2.862997615244640E-9T^2 +4.582058436059240E-11T^3 -4.001740284246030E-13T^4 +2.207261334864402E-15T^5 -8.018923094512611E-18T^6 +1.870326588771600E-20T^7 -2.533566089676708E-23T^8 +1.507992248919550E-26T^9	30 to 324 K
Invar M93, Imphy	+3.90755880E-6 -5.40993320E-7T +1.79753676E-8T^2 -2.76096772E-10T^3 +2.47817090E-12T^4 -1.40437638E-14T^5 +5.09192348E-17T^6 -1.14492104E-19T^7 +1.45317429E-22T^8 -7.95272390E-26T^9	35 to 305 K

Table 2. JPL measured CTE, first derivatives of the strain polynomials. The large number of digits are needed for accurate polynomial evaluations and do not imply accuracy beyond what is claimed elsewhere in this paper. Extrapolation beyond the specified temperature ranges is not recommended.

DISCUSSION/CONCLUSIONS

The strains at 70 K (relative to 293.15 K) for three popular low-expansion materials have been presented, with polynomial fits to enable extracting strains at other temperatures. CTEs have been obtained by taking the 1st derivatives of the strains. Estimated temperature and length uncertainties have been provided as well.

In the case of the Invar M93, an unresolved question is whether the mechanical action of fabricating and polishing the samples could have significantly affected the CTE. Moreover, there are many "flavors" of Invar, and each has its own CTE profile, which can be further altered by the material's heat-treatment and history. The reader should consider this before relying on the Invar data presented here. (Nevertheless, these results agree with the M93 CTE at -180 C published by Imphy Alloys[17].)

Preventable problems encountered in these measurements were (a) contamination at low temperatures and (b) temperature sensor attachment failures. Problem (a) could be solved by removing MLI insulation from the cryo-chamber and adding a "getter" to the coldest part of the system, the cryo-finger. Problem (b) could be solved by using larger, tubular samples instead of thin pillars. This would also make the samples easier (less expensive) to fabricate since polishing small end-faces to a fraction of a wave is technically demanding.

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