

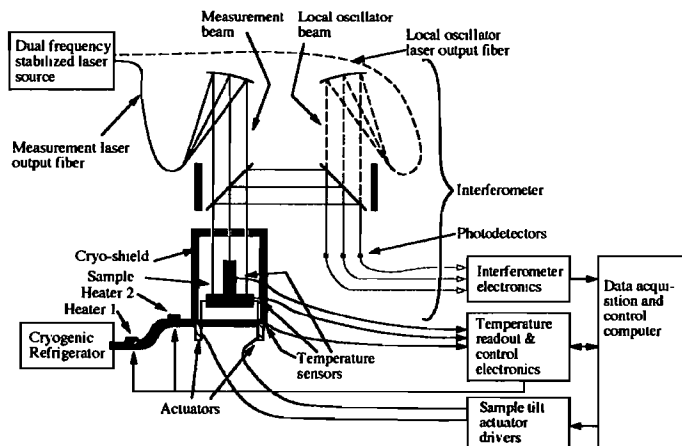
The JPL cryogenic dilatometer: measuring the thermal expansion coefficient of aerospace materials.

Peter G. Halverson, Matthew J. Dudick, Paul Karlmann, Kerry J. Klein, Marie Levine, Martin Marcin, Tyler J. Parker, Robert D. Peters, Stuart Shaklan, David Van Buren
ITCC29/ITES17 conference
June 24-27, 2007
Birmingham, Alabama

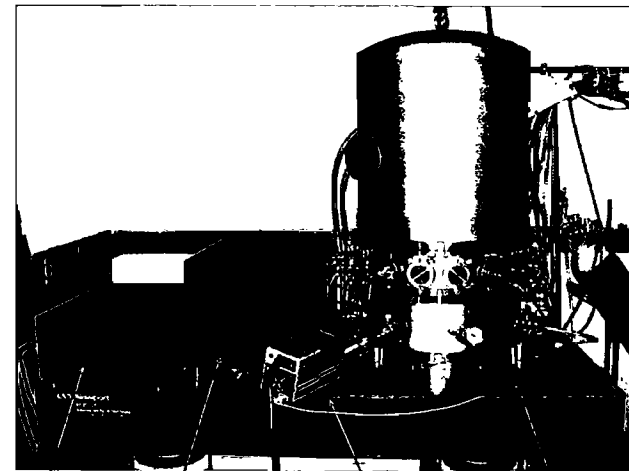
Overview

- Measure thermal strain from ~20 K to 324 K
- Strain accuracy
 - better than +/- 0.5 nm, 2E-8 strain above 180 K
 - better than 50 nm, 2E-6 strain below 180 K
- Temperature accuracy
 - better than +/- 0.25 K near room temperature
 - sample dependent error increases to roughly +/- 1 K around 100 K, to +/- 5 K around 25 K

System diagram



Dilatometer chamber and laser source



Behind chamber:
Liquid He
chiller

Iodine stabilized laser
Heterodyne shifters and coupling to fibers-optics
RGA (temporary)
Chamber



Laser Source

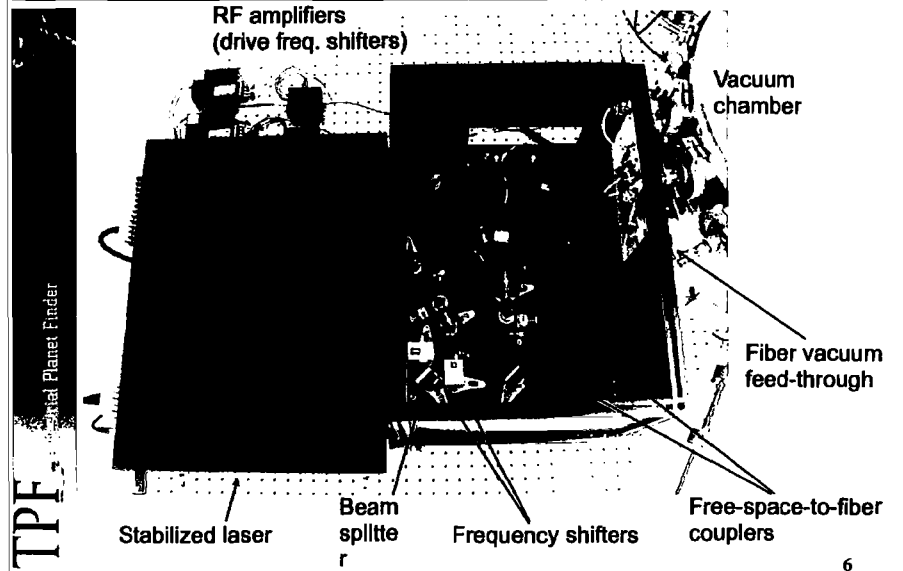
- **Laser:**
 - Innolight “Prometheus” 1 W @532 nm, doubled Nd:YAG
 - Iodine stabilization: stable to 5E-13 (10,000 seconds)
 - Iodine line frequency: 5.63260223471E+14 Hz, 532.613501716 nm
- **Laser frequency shifters**
 - Brimrose acousto-optic frequency shifters, one at 80 MHz, the other at 879.984 MHz provide 16 kHz frequency difference for heterodyne interferometry
- **Coupled to two fibers using two OFR “Snap-on” fiber collimators.**
- **Two laser signals brought into the test chamber on polarization-maintaining fibers with vacuum feed-through by Oz Optics.**

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Laser Source



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Interferometer

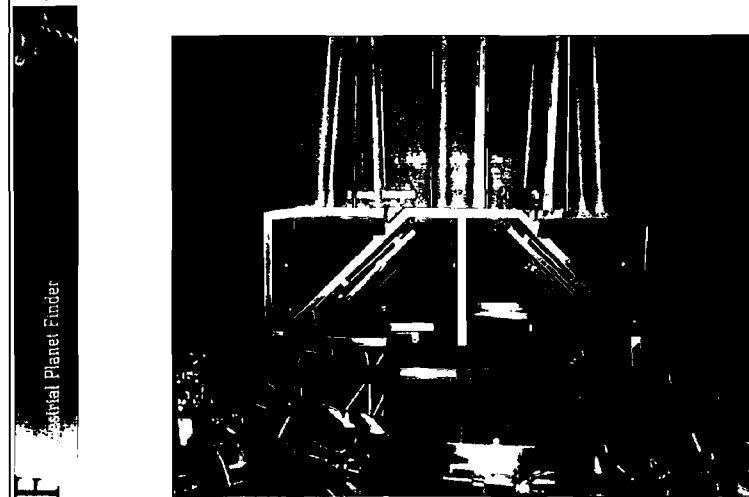


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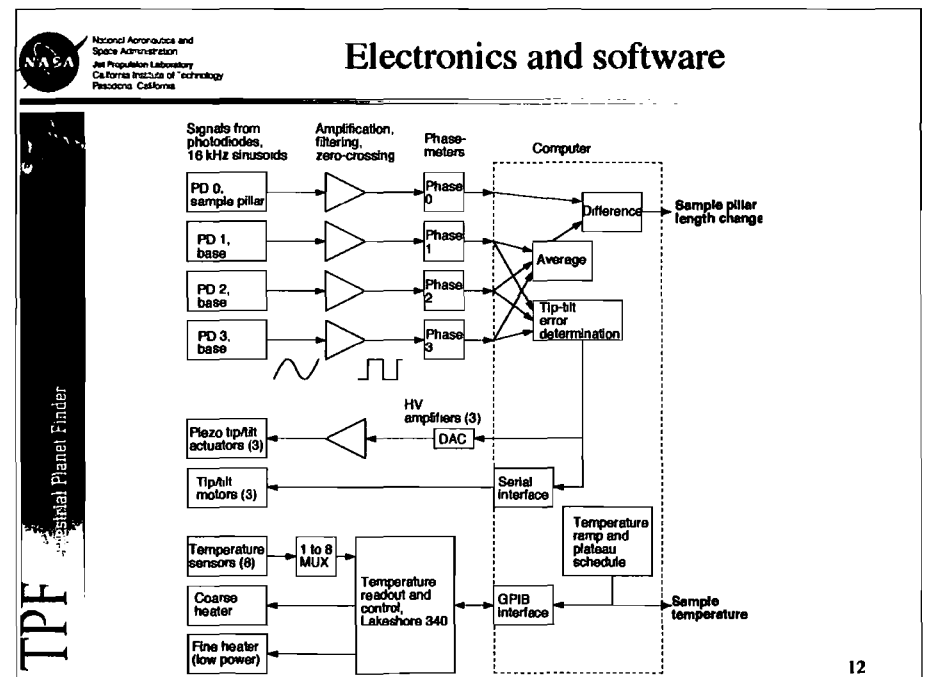
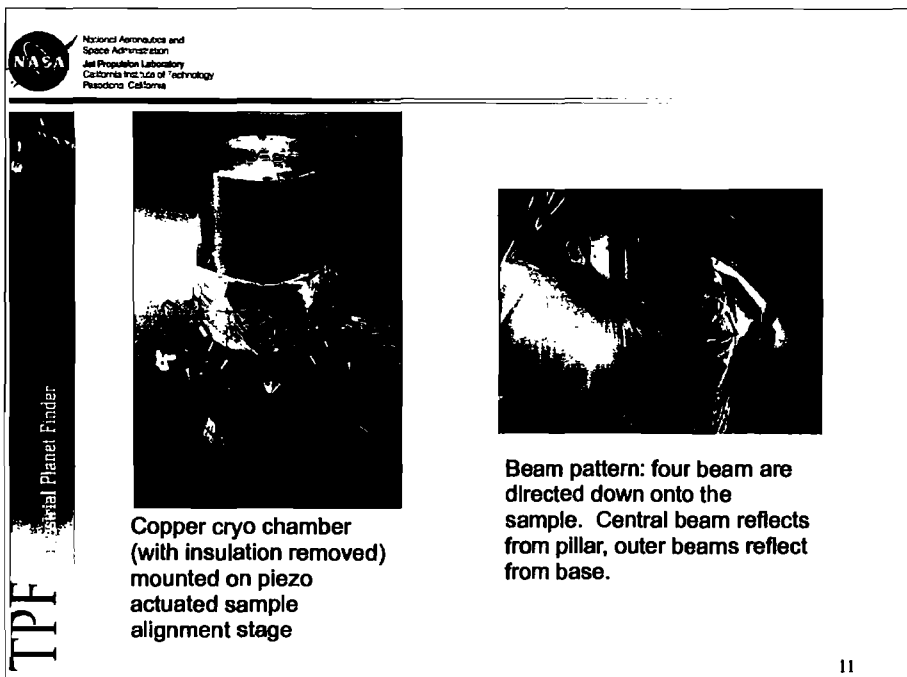
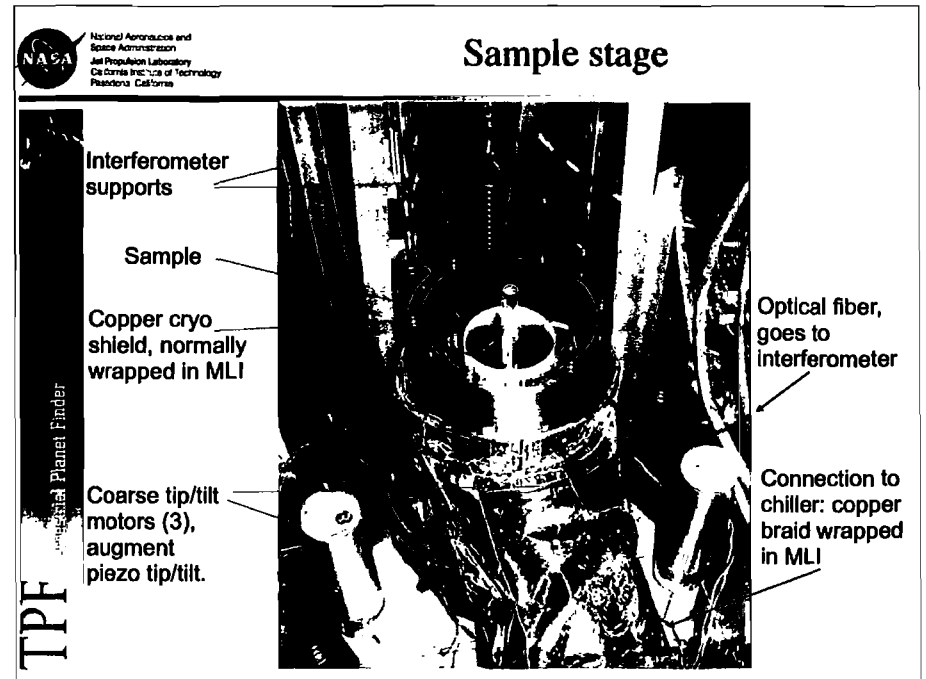
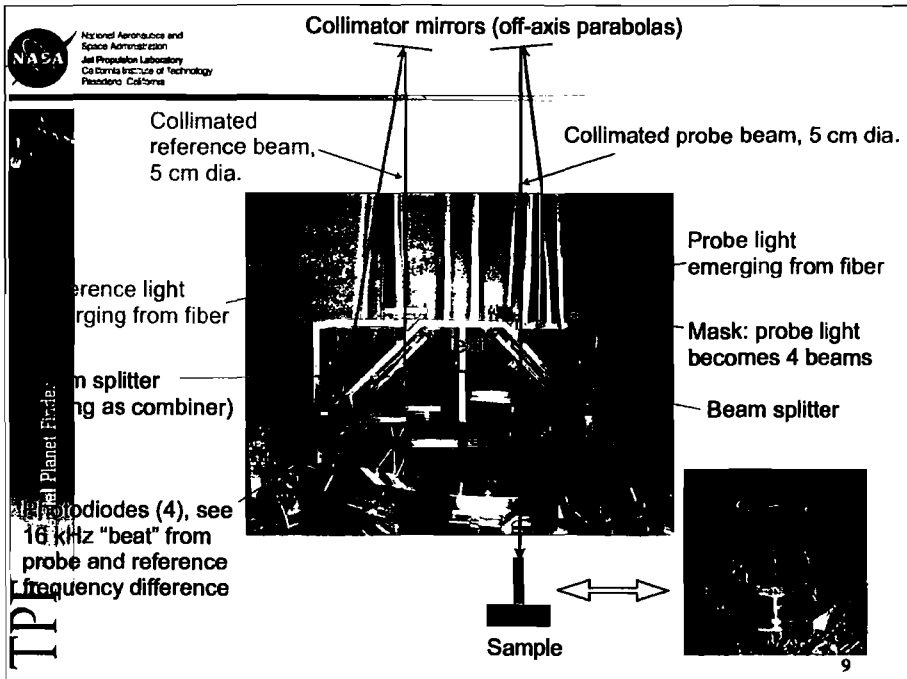


Interferometer

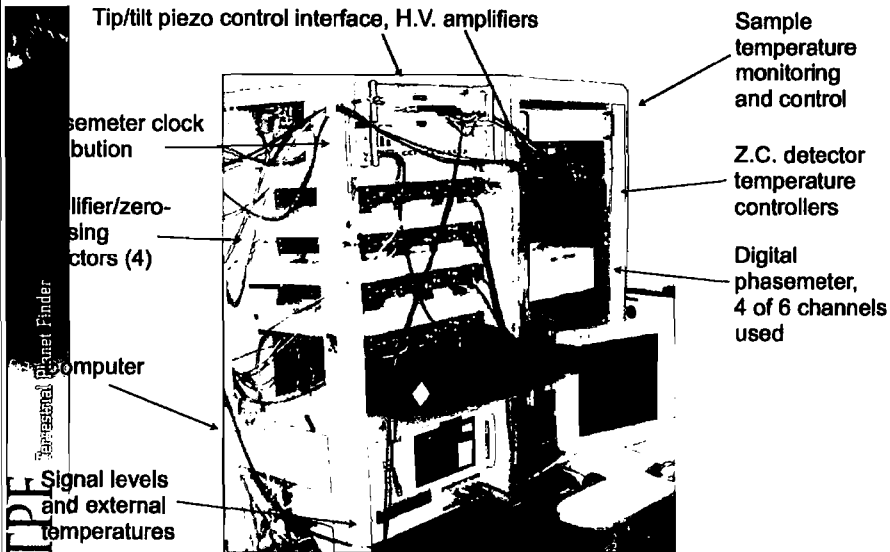


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Electronics & Software



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Accuracy/Error Budget (briefly)

- **System instabilities, inherent.** This includes laser wavelength, length readout, temperature readout, deformation of the optics and supporting hardware.
- **System instabilities, temperature dependent.**
- **Error due to the accumulation of contaminants on the sample.** This has emerged as a significant issue but it only affects data taken below 180 K.
- **Interferometer nonlinearity.**
- **Sample instabilities.** This includes sample creep and hysteresis.
- **Sample length at room temperature measurement error.**
- **Temperature measurement errors: calibration.**
- **Temperature measurement errors:** sensor contact with sample, sample temperature gradients and thermal lag.
- **(Error is dominated by the two underlined sources: contaminants and sensor contact and gradients.)**

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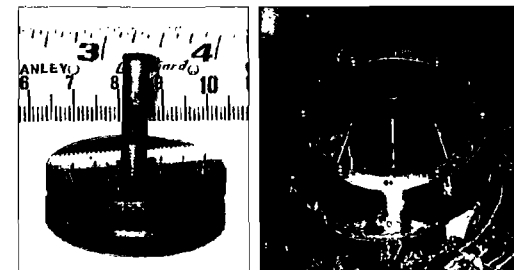
Materials

- **This work:**
 - Silicon Carbide, Boostek flavor
 - Silicon Carbide, Xinetics flavor
 - Invar M93, Imphy
 - Silicon, single crystal reference material for accuracy check
- **Past materials:**
 - ULE, Corning
 - Zerodur, Schott
 - PMN & custom PZT actuators, Xinetics

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Xinetics Silicon Carbide

- **Mixture: mostly SiC with some fraction of Silicon.**
- **Complex shapes possible by machining the “green” precursor.**
- **Can be polished to mirror finish, but crystal boundaries scatter.**
- **Could not optically contact pillar to base.**
 - Had to “glue” pillar to base using thin layer of vacuum grease
 - Yes, this means CTE of grease will cause a small error
 - More importantly, this weak the base-pillar bond prevented us from attaching the temperature sensors to the pillar.

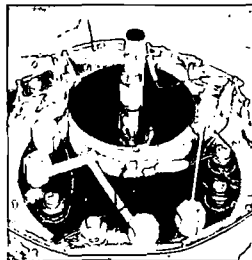


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Boostek Silicon Carbide

- **Mixture: mostly SiC.**
- **Complex shapes possible by machining the "green" precursor.**
- **Can be polished to mirror finish, but crystal boundaries scatter.**
- **ESA was able to optically contact pillar to base.**



Silicon carbide sample with temperature sensors attached

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Imphy Invar M93

- **Iron-Nickel 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 temperature**
- **Could not optically contact sample pillar to base; used magnet under base to hold pillar.**



Invar sample with temperature sensors attached

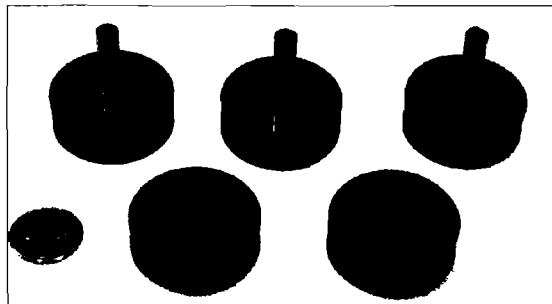
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Single-crystal silicon

- **High purity material, easily obtained from microelectronics industry.**
- **Reference material used major standards labs (NIST, AIST, PTB...)**
- **CTE is not sensitive to crystal orientation, preparation method.**
 - **Lab-to-lab CTE inter-comparison is consistent to a few ppb.**
- **High thermal conductivity, easily polished, easy to optically contact.**

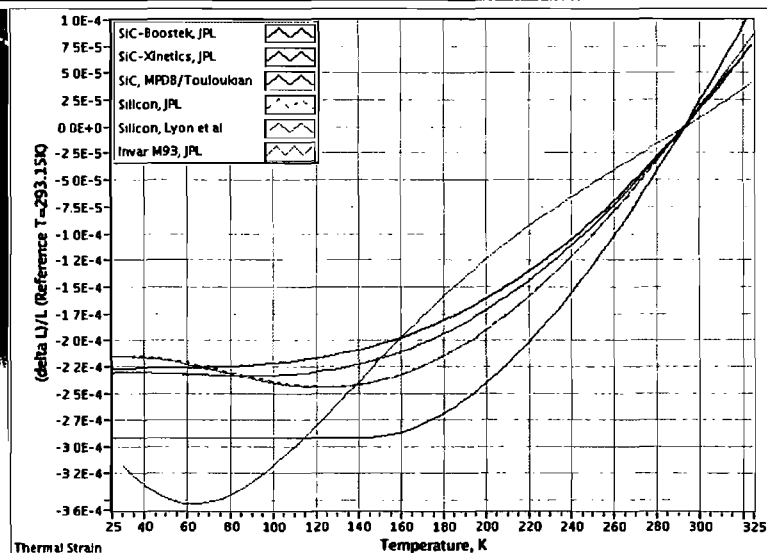


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Results: Strain curves



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References for non-JPL strain curves

- SiC
 - MPDB (Materials Property Database), JAHM Software, <http://www.jahm.com/>
 - MPDB data for SiC is from Thermophysical Properties of Matter, v13, Y.S. Touloukian, R.K. Kirby, R.E. Taylor & T.Y.R. Lee, 1977, IFI/Plenum, NY, NY
- Silicon
 - “Linear thermal expansion measurements on silicon from 6 to 340 K”, K.G. Lyon et al., Journal of Applied Physics, Vol. 48, No. 3, March 1977
- JPL silicon results: “Linear Thermal Expansion Measurements of Single Crystal Silicon for Validation of Interferometer Based Cryogenic Dilatometer”, Karlmann, P.B., et al., ADVANCES IN CRYOGENIC ENGINEERING. AIP Conference Proceedings, Volume 824, pp. 35–42 (2006)

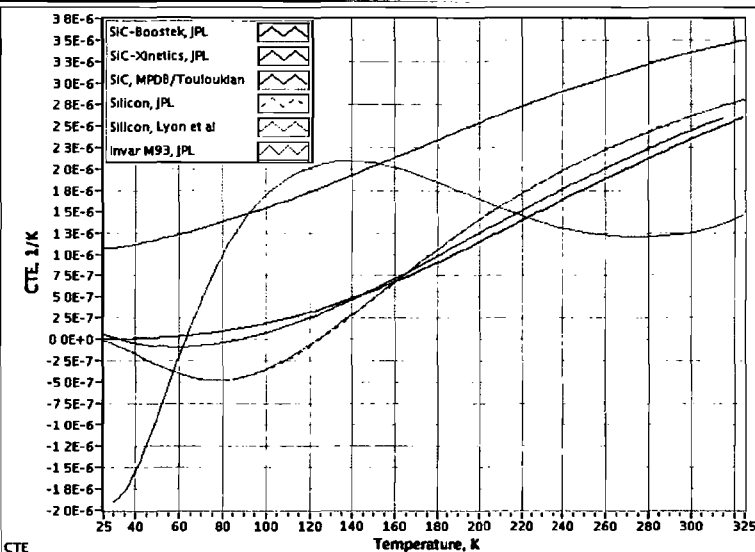
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JPL Strain fits

- SIC-Boostek, 22 to 324 K
 - Strain= $-2.2657759E-4 - 5.8446496E-9T + 3.1697174E-10T^2 - 7.9364135E-12T^3 + 1.3477868E-13T^4 - 3.6873692E-16T^5 + 3.2131869E-19T^6$
- SIC-Xinetics, 22 to 315 K
 - Strain= $-2.3629734E-4 + 4.1102516E-7T - 9.2041575E-9T^2 + 6.6373348E-11T^3 - 1.3110218E-13T^4 + 9.3714352E-17T^5$
- Silicon crystal, 30 to 324 K
 - Strain= $-2.09605420742702E-04 - 6.79449065133125E-07 + 3.73588146712838E-08T^2 - 9.54332538414880E-10T^3 + 1.14551460901481E-11T^4 - 8.00348056849206E-14T^5 + 3.67876889144067E-16T^6 - 1.14556044207323E-18T^7 + 2.33790823596450E-21T^8 - 2.81507343297412E-24T^9 + 1.50799224891955E-27T^10$
- Invar M93, Imphy, 35 to 305 K
 - Strain= $-3.0831959E-4 + 3.9075588E-6T - 2.7049666E-7T^2 + 5.9917892E-9T^3 - 6.9024193E-11T^4 + 4.9563418E-13T^5 - 2.3406273E-15T^6 + 7.2741764E-18T^7 - 1.4311513E-20T^8 + 1.6146381E-23T^9 - 7.9527239E-27T^10$
- Of course, number of digits does not indicate accuracy. Rather, this many digits are necessary for the fits to accurately reproduce the experimental results.

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Results: CTE



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JPL CTE, 1st derivative of strain fits

- SIC-Boostek, 22 to 324 K
 - Alpha= $-5.8446496E-9 + 6.3394348E-10T - 2.3809240E-11T^2 + 5.3911472E-13T^3 - 1.8436846E-15T^4 + 1.9279121E-18T^5$
- SIC-Xinetics, 22 to 315 K
 - Alpha= $+4.1102516E-7 - 1.8408315E-8T + 1.9912004E-10T^2 - 5.2440872E-13T^3 + 4.6857176E-16T^4$
- Silicon crystal, 30 to 324 K
 - Alpha= $-6.7944907E-7 + 7.4717629E-8T - 2.8629976E-9T^2 + 4.5820584E-11T^3 - 4.0017403E-13T^4 + 2.2072613E-15T^5 - 8.0189231E-18T^6 + 1.8703266E-20T^7 - 2.5335661E-23T^8 + 1.5079922E-26T^9$
- Invar M93, Imphy, 35 to 305 K
 - Alpha= $+3.9075588E-6 - 5.4099332E-7T + 1.7975368E-8T^2 - 2.7609677E-10T^3 + 2.4781709E-12T^4 - 1.4043764E-14T^5 + 5.0919235E-17T^6 - 1.1449210E-19T^7 + 1.4531743E-22T^8 - 7.9527239E-26T^9$
- Number of digits does not indicate accuracy. Rather, this many digits are necessary for the fits to accurately reproduce the experimental results.

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Error budget revisited

	Error source	Magnitude	Strain	Comments
1	System instabilities, inherent laser wavelength, length readout, temperature control, deformation of optics and supporting hardware	+/- 300 pm	+/- 1.2E-8	
2	System instabilities, temperature dependent.	+/- 250 pm	+/- 1E-8	
3	Contamination of sample	25 nm typical?	-8E-7 Invar	Depends on accumulation time, system bakeout. Evaluate for each run. Asymmetric
4	Interferometer non-linearity (cyclic error)	+/- 115 pm	+/- 4.6E-9	Only affects plateaus and near-zero CTE regions
5	Sample instabilities. This includes sample creep and hysteresis	Nearly 0	Nearly 0	Set to zero, redundant with error sources 1 and 2
6	Sample length at room temperature measurement error	+/- 1 micron	+/- 1.4E-8	
7	Temperature measurement errors, calibration	+/- 0.25 K	+/- 1.6E-8 SiC @70K +/- 1.1E-7 Invar @70K	Depends on CTE. Subsumed by next line, error source 8.
8	Temperature measurement errors: sensor contact with sample, sample temperature gradients	+/- 2.5 K Invar @ 70K	+/- 1.1E-6 Invar @70K	Depends on CTE. Varies with temperature and sample conductivity



Acknowledgment

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