Linear Thermal Expansion Measurements of Single Crystal Silicon for Validation of Interferometer Based Cryogenic Dilatometer

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LINEAR THERMAL EXPANSION MEASUREMENTS OF SINGLE CRYSTAL SILICON FOR VALIDATION OF INTERFEROMETER BASED CRYOGENIC DILATOMETER

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ABSTRACT

Linear thermal expansion measurements were performed for high-purity P-type single crystal silicon over a temperature range of 30K to 310K to validate the accuracy of JPL's interferometer-based Cryogenic Dilatometer Facility. This system was developed to better characterize thermophysical properties of precision engineering materials at cryogenic temperatures for space-based optical systems. An accurate measurement of these properties is critical for the success of missions such as the James Webb Space Telescope and the Terrestrial Planet Finder Coronagraph where picometer-level instabilities and thermal deformations impact performance. Results from these single crystal silicon measurements show a mean system repeatability of 4 ppb/K in the coefficient of thermal expansion (CTE) from 35K to 305K. Comparison with NIST/CODATA recommended values shows agreement of better than 2 ppb/K from 30K to 80K, better than 11 ppb/K from 80K to 165K, and better than 2 ppb/K from 165K to 305K.

KEYWORDS: single crystal silicon, thermal expansion, thermal strain, dilatometer
PACS: 65.40.De, 42.62.Eh

INTRODUCTION

The JPL Cryogenic Dilatometer Facility is designed to provide high precision measurements of thermal strain, dimensional stability and material hysteresis to support NASA's next generation of astronomical missions. Primary support for the development and operation of the facility is provided by the James Webb Space Telescope Project. Additional support is provided by the Terrestrial Planet Finder Coronagraph as well as the Space Interferometry Mission.

In 2004, a series of thermal expansion measurements were performed on single crystal silicon to help validate the performance of the JPL Cryo-Dilatometer and to provide a calibration reference standard against which other test facilities could be compared. Silicon is an excellent thermal expansion reference standard because of (a) its single crystal structure, (b) availability in high-purity forms, (c) insensitivity to machining stresses, (d) it doesn't require special heat-treating, (e) negligible variation from supplier to supplier, and (f) it has a high thermal conductivity and therefore low thermal gradients. Consequently, any two test samples of single crystal silicon, of different geometries, prepared years apart by different suppliers will likely have nearly identical thermal expansion behavior.

Following completion of this series of tests, the JWST Project requested that one of the silicon test samples be shipped to Southern Research Institute (SRI) in Alabama and another to ATK Space Systems in San Diego for validation of those measurement facilities. The third silicon test sample remains at JPL as a system reference standard.

APPARATUS

The basic design of the JPL Cryogenic Dilatometer has been previously described by Dudik et al. [1] with subsequent improvements and modifications to the system detailed by Karlmann et al. [2]. In general, the system consists of a high-precision heterodyne interferometer and laser source combined with a cryocooler-based thermal control system and custom built electronics to provide picometer-level measurements with milli-Kelvin-level thermal control. The test sample consists of a small cylindrical sample pillar optically contacted to a reference base. The interferometrically measured expansion and contraction of the sample pillar is obtained by subtracting the measurement of the central beam reflecting off the sample pillar from the average measurement of the three outer beams reflecting off the reference base. Temperature of the sample is controlled by a thermal shroud actively cooled by a vibration-isolated, closed-cycle 4K helium cryocooler. The interferometer is a custom design based on technology development work for the metrology system of NASA's Space Interferometry Mission. Photos of the test hardware and test samples are shown in Figure 1.

TEST SAMPLE PREPARATION

The raw material used for the test samples is high purity single crystal p-type silicon grown in the [100] orientation via the Czochralski or modified Czochralski method. Although thermal expansivity is independent of crystal orientation, each test sample was fabricated with its thermal expansion measurement axis parallel to the [100] crystal direction. All critical surfaces were polished to a flatness of 32 nm , and the parallelism between the top and bottom surfaces of each sample pillar is held to less than 4 arcsec. A total of three sample pillars and five sample bases were fabricated by Insync Optics in Albuquerque, New Mexico. Prior to delivery, each sample pillar was optically contacted to a sample base as shown in Figure 1.

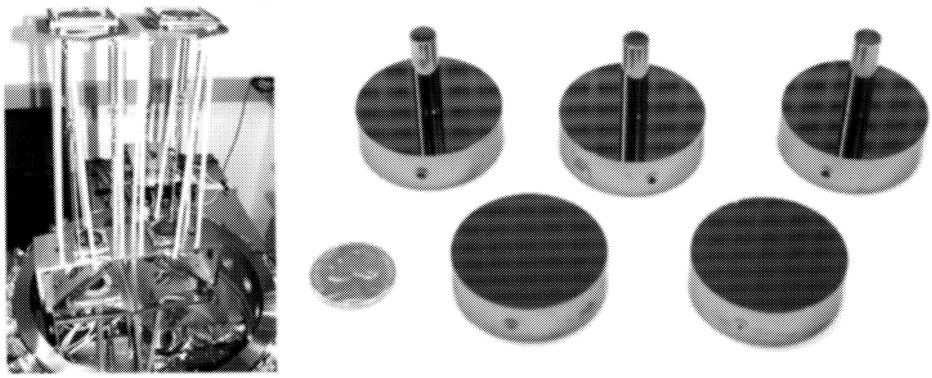

FIGURE 1. (Left) Custom heterodyne interferometer installed inside its vacuum belljar. (Right) Three single crystal silicon test samples and two reference bases as manufactured by Insync Optics in Albuquerque, New Mexico.

TEST METHODOLOGY

A total of 10 measurements were performed for single crystal silicon Samples #1(A) and #2(B) as described below. The two testing methodologies utilized during the course of the testing are described as a transient and a steady state temperature approach. The transient testing approach had a nominal thermal profile of 310 K to 30 K to 310 K with nominal temperature transitions of 15 K/hr. The steady state temperature approach has the same nominal thermal profile and temperature transitions; however, the temperature was incremented in 10 K intervals and held for approximately four hours once the sample had reached a steady state temperature stability of +/- 5 mK. Due to the quality and repeatability of the data, it was determined that the derivation of a temperature-dependent CTE using the transient approach could be utilized while still minimizing the associated errors.

The temperature-dependent CTE is established by determining the derivative of an nth order least squares polynomial curve fit of the raw strain curve. Due to end effects associated with curve fitting techniques that can result in large uncertainties, the lowest order curve fit that can characterize the system should be chosen. However, proper characterization of the system requires a fit that will minimize both nonlinearities and deviations of the residual associated with the curve fit. Since the curve fitting process is highly unsystematic and largely up to the discretion of the data analyst, it is necessary to follow the aforementioned guidelines in order to minimize analysis induced errors.

RESULTS & DISCUSSION

Shown in Figure 2 is a plot of the thermal strain as a function of temperature for 10 separate measurements of samples #1(A) and #2(B) between 30K and 310K. Raw data from both the transient and the steady state approach was used. Each curve is a 10^{th} order least squares polynomial curve fit of the raw measurement data. For all of these plots, a single thermal cycle includes data obtained during both cooling and heating. Also, for all of the raw strain data, zero strain has been defined at 293.15K. Also shown are the reported values for strain as a function of temperature given by Lyon et al. [4] and eventually adopted as the recommended values by CODATA [3]. The quality of the curve fitting process can be evaluated in Figure 3 which shows the residuals of the curve fit results minus the raw strain data.

By taking the derivative of the curve fits shown in Figure 2, the CTE as function of temperature can be obtained. Shown in Figures 4 and 5 are the derived CTE as a function of temperature curves for each thermal cycle, as well as, the standard deviation of the CTE as a function of temperature. Also shown in Figure 4 are the recommended values of CTE given by Lyon et al. [4] and by CODATA [3]. An estimate of the system repeatability can be obtained by noting that for 10 separate measurements between two separate test samples, the standard deviation in the data varies from about 1 to 8 ppb/K.

Comparison with Literature Data

For the comparison of these results with literature data, there are few available references. The most useful reference is by Lyon et al. [4] who performed capacitance-based expansion measurements in 1976 on single crystal silicon from 6K to 340K. Later, the CODATA Task Group on Thermophysical Properties published their recommended values for thermal expansivity of silicon from 0K to 1000K [3]. Not surprisingly, the CODATA values below 300K were merely taken from the results of Lyon et al. Supporting the results of Lyon et al., Yamada and Okaji [5] were able to obtain data that agreed to within 10 ppb/K from about 10K to 300K. The standard deviation of the Yamada and Okaji data was 6.8 ppb/K.

Figures 2 and 4 compare the thermal strain and thermal expansivity data obtained in the JPL measurements with the CODATA recommended values. Figure 6 is a plot of the JPL expansivity results minus the CODATA/Lyon et al. results. Between 30K and 80K and between 165K and 305K the deviation is less than 2 ppb/K. Strangely, between 80K and 165K, near the zero-CTE temperature of silicon, the deviation increases to less than 11 ppb/K. The reason for the larger deviation between 80K and 165K is unknown but presumed to be due to the test apparatus and/or measurement technique. A final comparison of the JPL data with the CODATA/Lyon et al. recommended values is shown in Table 1.

SUMMARY & CONCLUSIONS

The single crystal silicon results from the JPL Cryogenic Dilatometer show excellent repeatability and agreement with other high precision facilities. For repeatability, over 10 separate measurements and two test samples, the standard deviation ranged from 1 to 8 ppb/K with a mean of 4 ppb/K. The JPL Cryo-Dilatometer has been compared with the Lyon et al.[4] measurements from 1976 which used a capacitance based technique, as well as, the Yamada and Okaji [5] measurements from 1999 which used an interferometric technique. The JPL results show agreement with the CODATA/Lyon et al. recommended values of better than 2 ppb/K from 30K to 80K, better than 11 ppb/K from 80K to 165K, and better than 2 ppb/K from 165K to 305K.

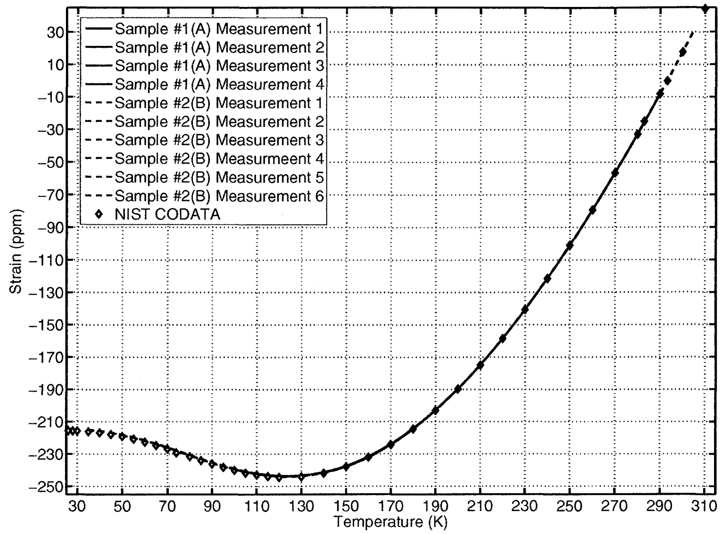

FIGURE 2. 10^{th} order polynomial curve fits of raw strain data as a function of temperature for single crystal silicon samples #1(A) and #2(B) compared with the results reported by Lyon et al. which were later adopted as recommended values by CODATA.

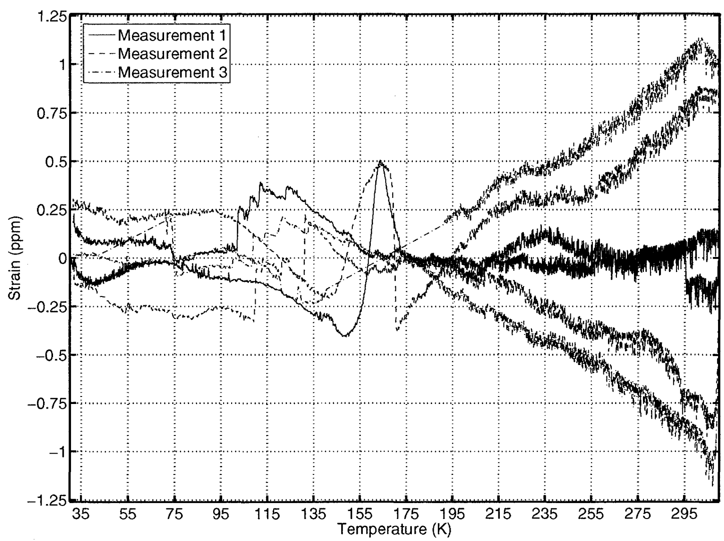

FIGURE 3. Residuals of 10^{th} order polynomial curve fits for first three measurements of sample #1(A).

TABLE 1. Comparison of JPL CTE Data with the CODATA/Lyon et al. recommended values.

Temperature (K)	CODATA (ppm/K)	JPL CTE Data (ppm/K)	JPL - CODATA (ppm/K)	JPL Standard Deviation (ppm/K)
35.00	-0.1030	-0.10869	-0.00569	0.005977
40.00	-0.1639	-0.16820	-0.00430	0.002131
45.00	-0.2289	-0.23151	-0.00261	0.003768
50.00	-0.2927	-0.29376	-0.00106	0.004142
55.00	-0.3507	-0.35214	-0.00144	0.001819
60.00	-0.3999	-0.40066	-0.00076	0.001235
65.00	-0.4376	-0.43749	0.00011	0.001835
70.00	-0.4624	-0.46232	0.00008	0.002802
74.00	-0.4725			
75.00		-0.47492		0.002371
80.00	-0.4720	-0.47490	-0.00290	0.001720
85.00	-0.4570	-0.46195	-0.00495	0.002723
90.00	-0.4290	-0.43614	-0.00714	0.003568
95.00	-0.3900	-0.39812	-0.00812	0.003844
100.00	-0.3390	-0.34961	-0.01061	0.004286
105.00	-0.2790	-0.28693	-0.00793	0.006660
110.00	-0.2100	-0.22107	-0.01107	0.005956
115.00	-0.1380	-0.14782	-0.00982	0.005398
120.00	-0.0570	-0.06733	-0.01033	0.005431
125.00		0.01877		0.005899
130.00	0.1190	0.10873	-0.01027	0.006176
135.00		0.20119		0.006049
140.00	0.3060	0.29523	-0.01077	0.005660
145.00		0.39083		0.006073
150.00	0.4970	0.48623	-0.01077	0.005379
160.00	0.6890	0.68358	-0.00542	0.005589
170.00	0.8770	0.87850	0.00150	0.005735
180.00	1.0610	1.06000	-0.00100	0.007750
190.00	1.2380	1.23550	-0.00250	0.005828
200.00	1.4060	1.40470	-0.00130	0.001813
210.00	1.5650	1.56170	-0.00330	0.003103
220.00	1.7150	1.71050	-0.00450	0.002944
230.00	1.8550	1.85090	-0.00410	0.002622
240.00	1.9860	1.98270	-0.00330	0.002947
250.00	2.1080	2.10620	-0.00180	0.004201
260.00	2.2230	2.22190	-0.00110	0.004175
270.00	2.3300	2.32980	-0.00020	0.001934
280.00	2.4320	2.42930	-0.00270	0.005558
283.00	2.4610			
290.00	2.5270	2.52480	-0.00220	0.005691
293.15	2.5550			
295.00		2.57170		0.001749
300.00	2.6160	2.61230	-0.00370	0.004715
305.00		2.65000		0.011858

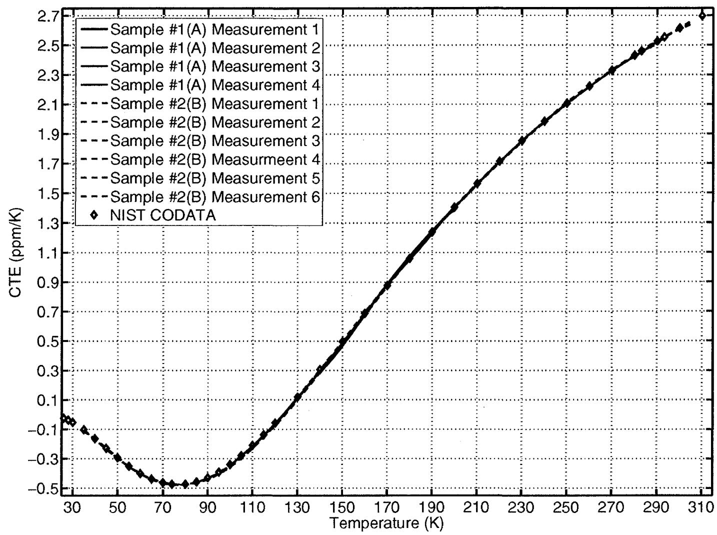

FIGURE 4. Local CTE curves derived for single crystal silicon samples #1(A) and #2(B) and the CODATA/ Lyon et al. recommended values for silicon.

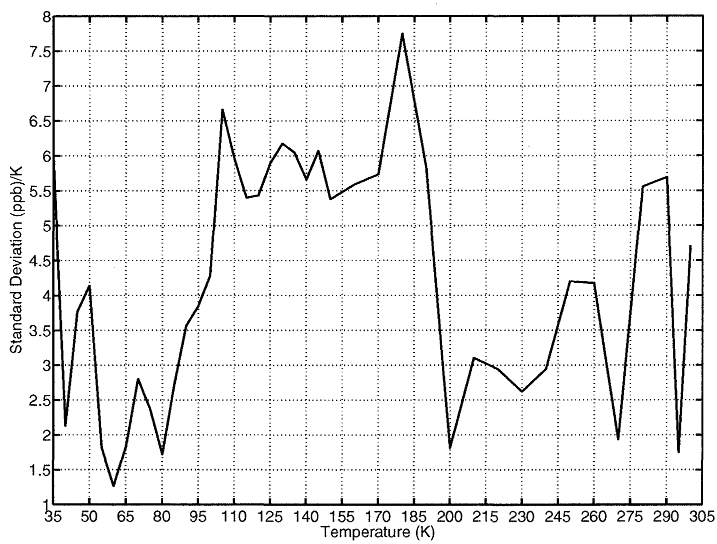

FIGURE 5. Plot of the standard deviation as a function of temperature for 10 CTE measurements made over two silicon test samples.

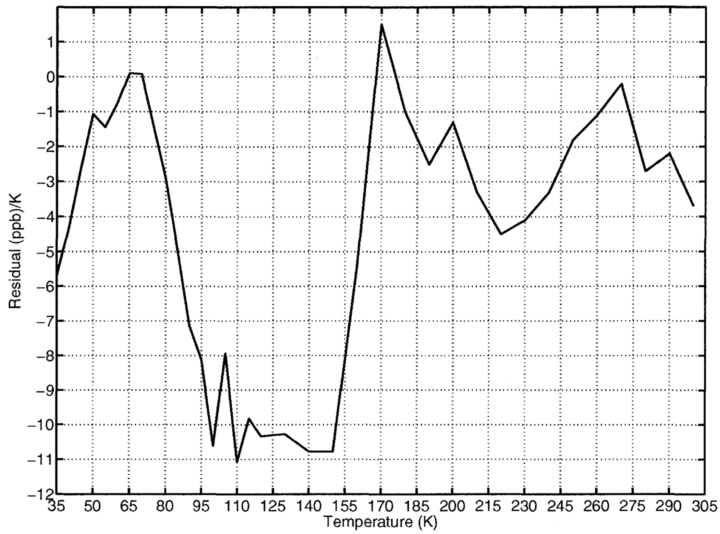

FIGURE 6. Mean deviation between the JPL CTE results and the CODATA/Lyon et al. recommended values (Residual = JPL results – CODATA recommended values).

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