IOP Conf. Series: Materials Science and Engineering **1302** (2024) 012031 doi:10.1088/1757-899X/1302/1/012031

Development of an apparatus and process for precision measurements of cryogenic thermal contraction of materials

James Tuttle[1,4], Amir Jahromi[1], Edgar Canavan[1], Jacob Nellis[2], and Justin Scheidler[3]

[1]*NASA Goddard Space Flight Center, Code 552, Greenbelt, MD 20771 USA*
[2]*Department of Computer Science, Univ. of Wisconsin, Madison, WI 53706 USA*
[3]*NASA Glenn Research Center, Cleveland, OH 44135 USA*
[4]corresponding *author:* james.g.tuttle@nasa.gov

Abstract. NASA frequently needs thermal contraction data for materials to be used in cryogenic space flight missions. To satisfy this need, we developed an apparatus and a high-precision technique for performing such measurements using a commercial fiber-optic-based position sensor. We describe the measurement process and its verification using a copper sample. We also present data for alumina and sintered samarium cobalt, which we characterized for potential NASA use.

1. Background

On NASA space missions with cryogenic elements and in cryogenic components of some electrified aircraft propulsion systems, engineers often need to use materials that are new or otherwise insufficiently characterized in the literature. Materials are selected to optimally balance strength, stiffness, thermal conductance, and thermal expansion/contraction properties. As a result, there is a significant demand for measurement of these properties of materials as a function of temperature in the cryogenic range. We, in the Cryogenics Branch at NASA Goddard Space Flight Center, have historically performed many such measurements, but until recently we had not attempted to characterize the thermal contraction of materials.

A few years ago, we acquired an Attocube Model IDS3010 fiber-optic-based position sensor system, which we used to characterize vibration amplitudes in a magnetic cooling development effort [1]. This system provides high-precision and high-frequency measurements of the distance from each of its sensor heads to a nearby reflecting surface. The tiny sensor heads are at the end of optical fibers, and the particular heads that we purchased are compatible with cryogenic operation. Our successful use of this system on the earlier effort inspired the development of an apparatus and process for characterizing the length of a material sample as a function of temperature.

2. Measurement Technique

The measurement setup is shown in figure 1A. A cylindrical rod of the selected sample material is cut to an appropriate length, and its ends are polished. Its length, L_0, is precisely measured at 293 K using a caliper or micrometer. The rod, shown in green in the figure, is supported above a cryostat's cold-stage by a clamp atop a thermally-isolating post. Thermometers are installed near the midpoint and one

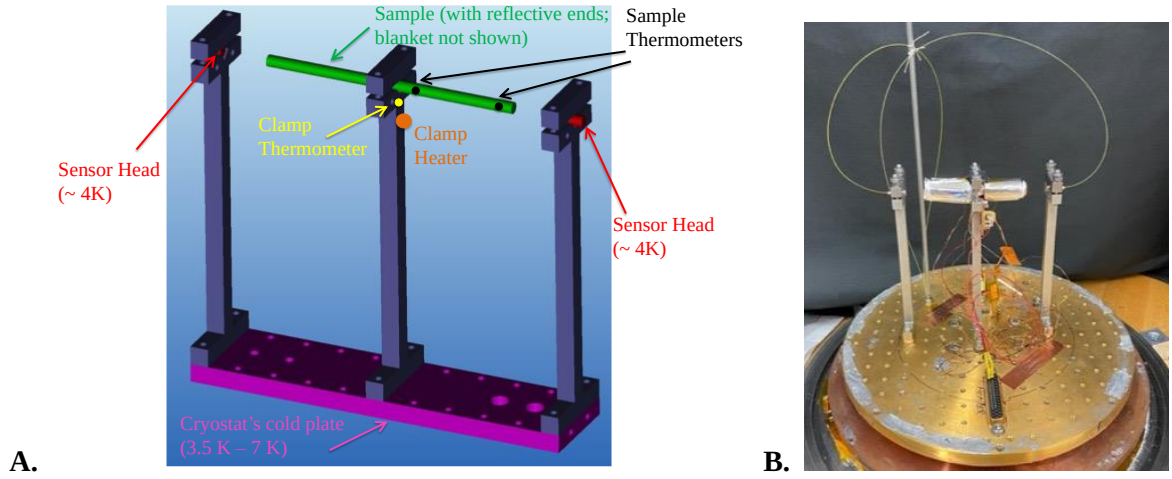

Figure 1. **A.** The test setup, indicating the locations of all components. **B.** A photograph of the apparatus with a sample ready for testing. A multi-layer blanket surrounds the sample's ends to minimize temperature gradients due to radiative heat loss.

end of the sample, and a small resistive heater is mounted on the sample-holding clamp. Identical post/clamp assemblies hold two of the optical sensor heads (shown in red) in positions facing the sample's two end surfaces. A small multi-layer-insulating blanket is wrapped around the sample on each side of the clamp, as shown in figure 1B, to help keep the sample isothermal.

The basic measurement method is relatively straightforward. The cold stage is cooled to its lowest temperature of approximately 4 K, and all elements of the test setup are allowed to reach thermal equilibrium. The sensor system is activated, and the indicated distances between the sensor heads and the sample ends are recorded. Then, the heater is used to raise the sample temperature, and it is held at a new steady temperature using a feedback control loop. At this point, the sample has expanded in length, but the cryostat's copper cold stage has remained at nearly 4K and has not expanded significantly. Thus, the sensors remain at 4 K, and their locations have not changed from those of the initial measurement. A new pair of distance measurements is taken. The difference between the sum of these distances and the sum of the original two distances is the change in sample length, ΔL. The sample is held at many different temperatures between 4 K and room temperature, and the change in its length is measured for each step. The final measurement is done with the sample controlled at 293 K, where its precise length, L_0, is known. From these measurements the sample's $\Delta L/L_0$ relative to 293 K is calculated as a function of temperature.

Several details of this work proved more challenging than we had anticipated. Our first hurdle was to install the fiber-optic-based sensors in our available cryostat, which included a copper cold stage plate bolted directly to the cold tip of a Sumitomo 1-Watt Gifford-McMahon cryocooler. This installation was difficult, as the fibers are quite delicate and easily broken. With careful planning, fixturing and minor modifications to the cryostat shields, we were able to feed the sensor heads from their room-temperature hermetic feed-throughs to the desired locations on the cryostat's cold stage. Fortunately, we destroyed only one sensor during this process.

The next challenge was to position the sensor heads and sample relative to each other. Each head emits an infrared beam which re-enters the head after reflecting off one of the sample's ends. The amount of radiation entering a head for measurement depends on the distance to the sample, the angle relative to the end surface, and the flatness and reflectance of that surface. The amplitude for each head must fall in an acceptable range; a certain minimum signal must be exceeded, but too large a signal overwhelms the head. For modestly reflective surfaces the challenge is to maximize each signal, and the optimum surface-to-head spacing must be close to the head's focal length of 17 mm. For near-mirror-finish surfaces, the signal must be reduced by a larger spacing or intentional angular

misalignment. The signal is extremely sensitive to this reflection angle, but the cryostat's size precluded installing standard optical alignment fixtures. Thus, we used a tedious process of loosening screws, manually rotating the support posts by tiny amounts, and carefully re-tightening the screws until we achieved acceptable signal strengths at both ends. This effort took between an hour and several hours for each sample.

Next, the cryostat's thermal radiation shield was installed on its 4 K stage plate. Unfortunately, tightening the attachment screws for this shield changed the shape of the plate and significantly changed the sensor head signals. We needed to vary the tightening order and torque of these screws to maintain an acceptable signal. Finally, the cryostat was sealed, pumped out, and cooled to operating temperature. As the cold stage and sample cooled to 4 K, they both shrank, the sensor heads moved closer together, and the alignment angles changed by small, unpredictable amounts. At the same time, the reflectance of each metal surface increased at lower temperatures. The combination of these effects changed the signal strengths, sometimes moving one of them outside of the effective measurement range. When this happened, we were forced to warm up, open the cryostat, and realign the system before attempting another cooldown. Eventually this trial-and-error process resulted in the test apparatus being at approximately 4 K with two acceptable distance signals, and we were able to begin the measurement.

While working out the details of this process and characterizing our first sample, we acquired the distance measurements using Attocube's proprietary software, WAVE. This program is very useful for dynamic measurements, reading at up to 500 kHz and providing real-time fast-Fourier-transform analysis of the signals. It showed that the cryocooler's motion disturbed our distance readings by up to +/- 5 μm about once per second, followed by damped resonances in the range of 10's of Hz. We initially took readings at 1 kHz and time-averaged for one minute to reduce the uncertainty of each distance measurement. The sample temperature was maintained using a proportional-integral-derivative (PID) controller box, and the temperature and control heater power were read out and plotted by another program on another computer. After making each change of the sample temperature setpoint, we watched the plots until we determined that the temperature and power were steady. Then we initiated a set of distance measurements, which WAVE stored in a data file. Then we changed the PID control setpoint for the next temperature value. The data files were loaded into spreadsheets for statistical analysis. Fortunately, the time averaging of 60,000 distances per data point enabled precise measurements despite the lack of mechanical isolation from the cryocooler cold tip. The entire process was quite labor intensive, but it produced good results, as described below.

Eventually we developed a Python routine to automate the data acquisition. This program feeds setpoints and PID parameters to the temperature control box, and it reads out the Attocube measurements at up to about 250 Hz. Experimentation showed that this is fast enough to give the same time-averaged distances measured at 1 kHz by WAVE. We allowed each sample temperature to settle for three hours before taking pairs of distance measurements at the maximum rate for 10 minutes. This settling time allowed our thermometer readings to stabilize to well within their calibration uncertainties, but it could be adjusted to accommodate future samples with longer thermal time constants. With each reading event the software stores the instantaneous sum of the two distances. This sum varies due to independent movement of the two sensors but is unaffected by low frequency motion of the sample itself. When the readings are all completed, the mean and its random uncertainty are calculated before proceeding to the next temperature setpoint. As with any automation routine, this one drastically reduces the labor involved in our measurements.

3. Verifying the Technique; Copper Between 293 K and 6 K

Our first actual measurement goal was to confirm the accuracy of our approach by testing a material that was already well characterized and documented in the literature. A very comprehensive listing of published thermal expansion data for metallic elements and alloys is found in the series "Thermophysical Properties of Matter," volume 12, edited by Y. S. Touloukian [2]. While this compendium lists its original data sources, we found that many were inaccessible through standard search methods. In addition, the series editor had carefully shifted the data points so that they all gave ΔL/L relative to the

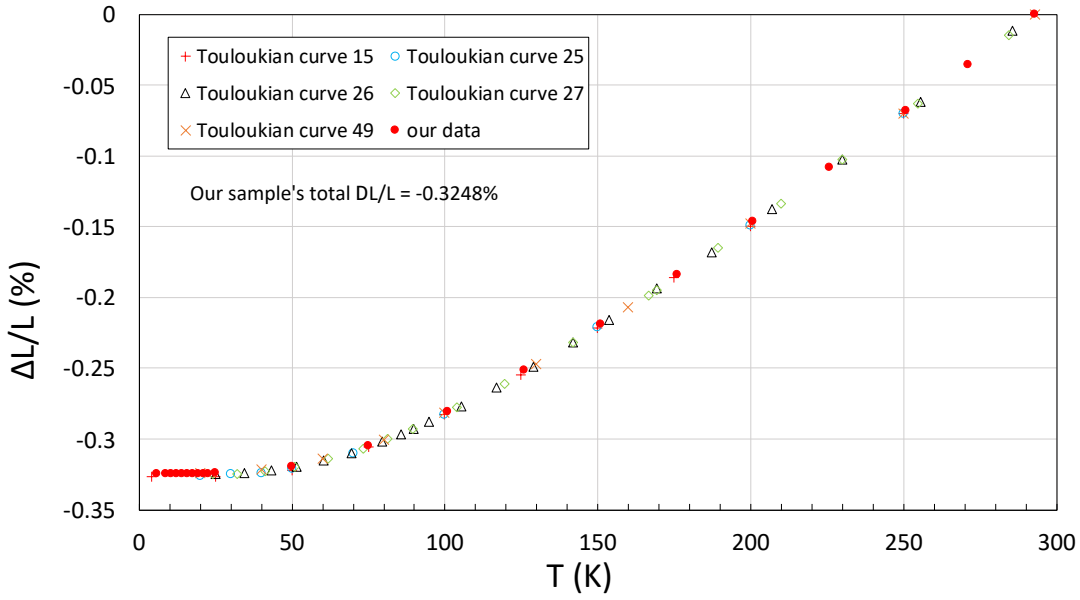

Figure 2. Thermal contraction from 293 K down to 6 K for 99.99% pure copper. The total contraction over this range was 0.3248 percent. Data from various sources in the Touloukian compilation [2] are shown for comparison.

same temperature, 293 K. For these reasons, we reference the volume, page range, and curve number in the Touloukian series for such data, rather than the original sources.

We chose 99.99% pure copper as our first sample, since multiple different researchers had found its ΔL/L to fall in a narrow range between -0.322% and -0.327% when cooled from 293 K to 4 K. For copper, the published measurements all used two basic approaches. One method, assumed to give very high precision, involved measuring changes in mutual inductance caused by relative motion of cold inductors inside the cryostat. The other method featured deflection measurements of the ends of rods extending from the cold sample out to the room temperature environment. Of the data sets in the Touloukian reference [2] covering entire cryogenic temperature range, all but one outlier matched each other to within their scatter, as shown in figure 2.

We machined a copper sample of 6.3 cm length and polished its ends to a near mirror finish. It was important to keep the ends very flat and perpendicular to the sample's main axis. When the sample temperature is varied, it's support post's temperature profile and length change, shifting the location on each end surface at which the measurement beam reflects. Any rounding or beveling of these surfaces would introduce errors in the indicated length changes. The support posts shown in Figure 1A were machined from invar 36, since its low overall ΔL/L [3] limited the sample's vertical displacement relative to the sensors when it was heated up to room temperature. In addition, its relatively low thermal conductivity [4] minimized the heater power needed to raise the sample temperature, thus reducing the temperature rise of the cryostat's cold stage. The clamp atop each post included a V-block shape to align the sample or cylindrical sensor head, and a spring-loaded rectangular block held each item in place with a modest force. The sample was clamped at its center, as shown in the figure 1A.

Preliminary testing showed that the cryostat's cold plate rose in temperature from about 3.5 K to only 7 K when the sample was warmed to 293 K. Based on published data for copper's thermal expansion in that range [2], the copper length between the sensor posts changed by less than 3 x 10^{-6} % while the copper sample expanded by 0.32 %. Thus, for materials with thermal contractions in the same range as copper, we could ignore the correction for lateral motion of the sensor posts. We controlled the sample temperature at several values between 6 K and 293 K. In figure 2, the red filled circles indicate our measured data, and the other symbols are various curves taken from pages 85 – 87 in the Touloukian

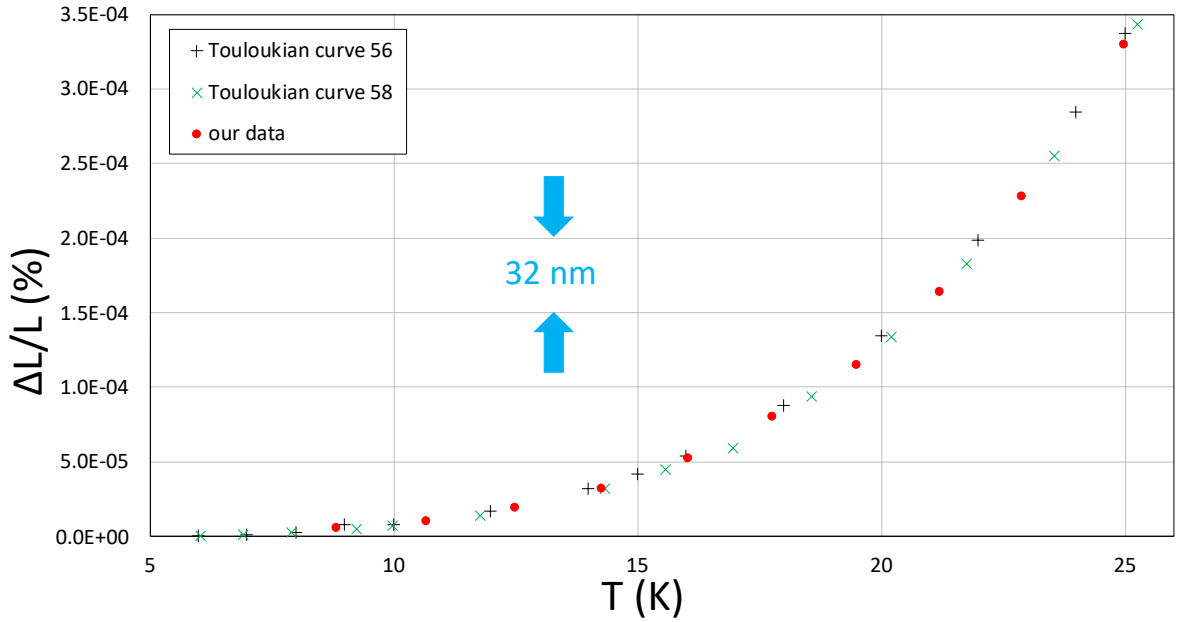

Figure 3. Thermal expansion from 6 K to 25 K for 99.99% pure copper. The random uncertainty in our data, corresponding to a length uncertainty of +/- 0.3 nm, is about 100 times smaller than the distance between the blue arrows. Data from two sources in the Touloukian compilation [2] are shown for comparison.

reference [2] spanning the range from room temperature to 20 K or below. Our total measured ΔL/L from room temperature to 6 K was -0.3248%. Our data, although slightly above the average of the published curves, fall well within their scatter. This indicates that our technique should be quite reliable for a high-thermal-conductivity sample of this type over the same temperature range.

4. Determining the Measurement Precision; Copper Between 6 K and 25 K

Another goal was to determine the limits of the precision of our thermal expansion measurements. The copper sample length shrank by about 0.2 mm when cooled from room temperature to 6 K. Despite the significant lateral motion of our sample (at times as much as +/- 5 μm) due to the mechanical cryocooler vibrations, our measurements were aided by time-averaging about 150,000 distance measurements at each end of the sample for each data point. The random uncertainty of each mean distance was found to be about +/- 0.2 nm (or +/- 3 x 10^{-7} percent of the length) at each end, which was trivial compared to the length changes measured in each temperature step between 293 K and 6 K. However, we found thermal expansion data sets in the Touloukian reference for copper between about 3 K and 30 K [2]. Over this range the relative length change is extremely small. Based on these results, we expected our sample to expand by only 0.22 μm when its temperature was raised from 6 K to 25 K. We decided that measuring the copper sample's thermal expansion in that temperature range would be a good additional test of our technique's precision.

We began at 6 K and warmed up in approximately 2 K steps to 25 K. We calculated the ΔL/L relative to the 6 K length, and the results are shown as filled red circles in figure 3. Our values match the previously published data to within their scatter. Their random uncertainty, after time-averaging, corresponds to a length uncertainty of about +/- 0.3 nm. This is significantly smaller than the red circle symbols in figure 3; it is about 100 times smaller than the distance between the blue arrows on the graph. These results indicate that our technique provides high precision and accuracy when measuring very small thermal expansion or contraction values.

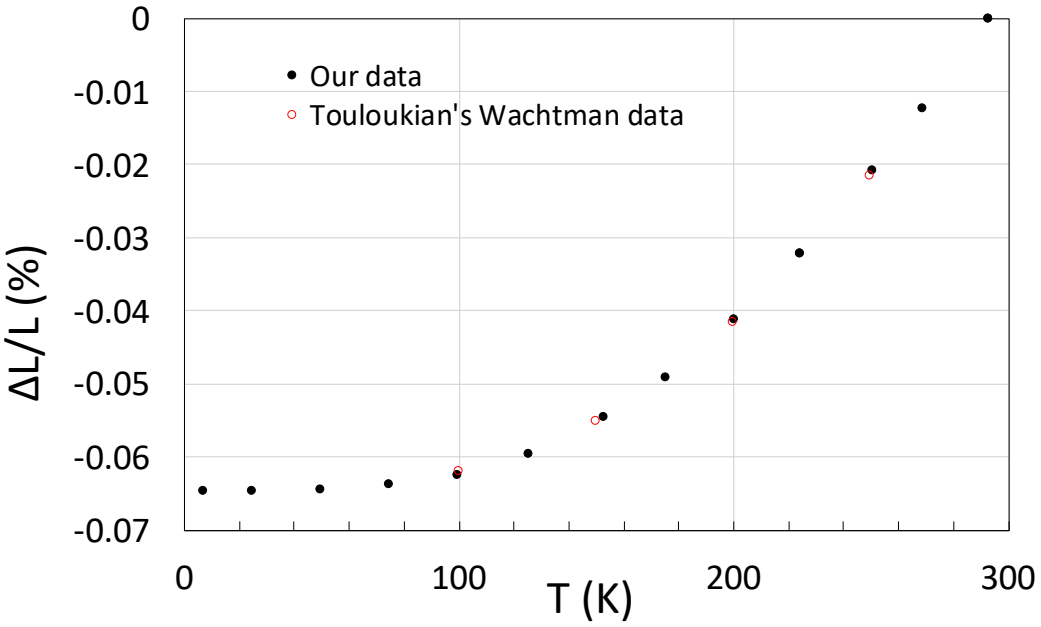

Figure 4. Thermal contraction from 293 K to 6 K for alumina (96% Al_2O_3). The filled black circles are our data, and the red open circles are Touloukian's data [5] for comparison.

5. Testing an Alumina Sample

With our apparatus and technique verified, we proceeded to perform useful thermal contraction measurements. A future NASA mission is considering the use of alumina in structural supports within magnetic actuators for space mechanisms. Engineers selected this material because it has high thermal conductivity, good strength, relatively low density, and is an electrical insulator. This last feature eliminates eddy current heating when the material is exposed to time-varying magnetic fields.

A sample was provided for a thermal contraction measurement. Due to the nature of the material, its ends could not be made reflective enough to produce readable distance measurements in the Attocube sensor heads. To solve this problem, we bonded thin polished aluminum discs onto the sample ends. The bonding agent, Stycast 2850FT epoxy, is formulated to have a thermal expansion rate matching that of aluminum in the cryogenic temperature range. We used a small vice with parallel jaws to apply a uniform axial force to the discs (through thin rubber gaskets) while the epoxy cured, resulting in very parallel reflective end surfaces. The overall sample length was measured at 293 K before and after installing these end discs. In the eventual thermal expansion data analysis, this small length difference was assumed to be due to materials having the known thermal expansion of aluminum, and an appropriate correction was made to back out the alumina sample length at each temperature. Figure 4 shows our measured data along with those given in the Touloukian series [4] for a sample described as polycrystalline alumina. Our total measured $\Delta L/L$ from 293 K to 6 K was -0.0647%, and the uncertainty of each point is about the same as that described for the copper data.

6. Testing Sintered Samarium Cobalt Samples

The same NASA project plans to use sintered samarium cobalt (SmCo) as permanent magnets in the aforementioned actuators. This material has high magnetic field strength and radiation tolerance at cryogenic temperatures. We were asked to measure its thermal contraction in two specific directions relative to its easy magnetization direction (c-axis). Two cylindrical samples were fabricated, de-magnetized for convenience in handling, and sent to us for characterization. The shorter sample had its main axis parallel to the material's c-axis, and the longer sample's axis was perpendicular to the c- axis.

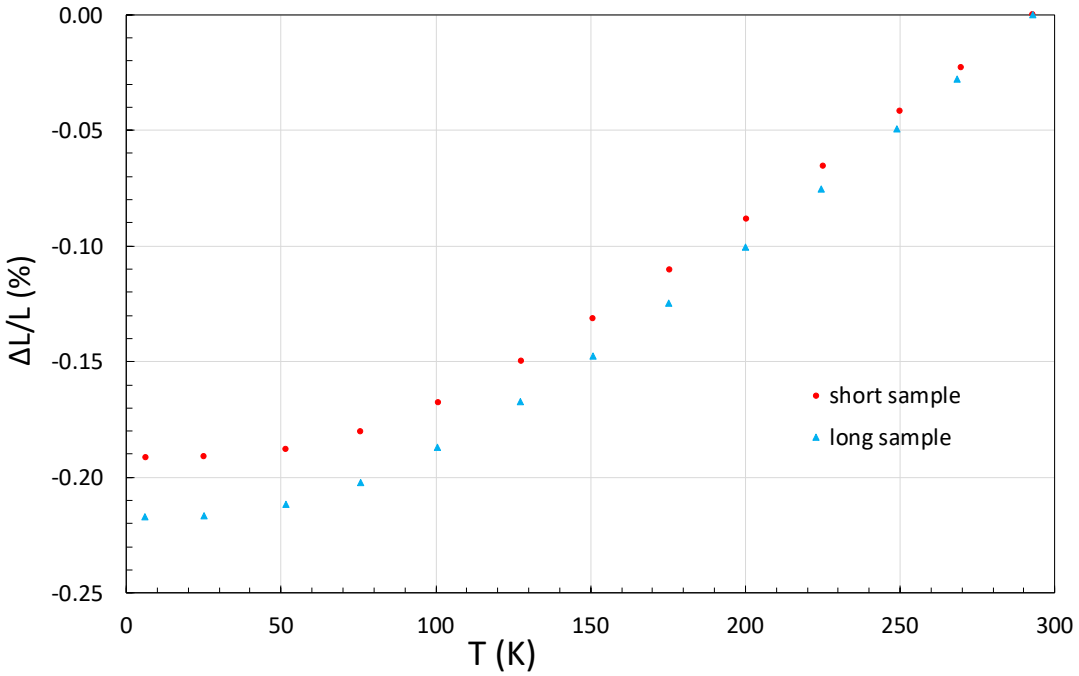

Figure 5. Thermal contraction from 293 K to 6 K for two different sintered SmCo samples. The shorter sample had its main axis parallel to the material's c-axis, while the longer sample's axis was perpendicular to the c- axis.

As with the alumina sample, we installed the polished aluminum end reflectors before performing measurements on the samples. Figure 5 shows our measured results. The short sample's total thermal contraction between 293 K and 6 K was -0.1916%, while that of the long sample was -0.2170%. Again, the uncertainty matched that of the copper data. A literature search failed to find any other published data for sintered SmCo.

7. Conclusion

We have developed an in-house apparatus and method for measuring the cryogenic thermal contraction of materials, demonstrated its accuracy and precision, and used it to characterize two different materials of interest to future NASA missions. The initial alignment of a sample, relying as it does on trial-and-error, is tedious. With sufficient future interest and funding, we would seek to improve this process. However, the actual thermal contraction measurement is straightforward due to our automation software and to the impressive capability of the Attocube system itself. We currently have a workable system available to perform such measurements for NASA customers.

8. References

[1] Tuttle J *et al.* 2020 IOP Conf. Series: Materials Science and Engineering **755** 012015
[2] Y Touloukian ed 1975 *Thermophysical Properties of Matter* vol 12, pp 77-91
[3] Y Touloukian ed 1975 *Thermophysical Properties of Matter* vol 12, p 1175
[4] Pobell F 1996 *Matter and Methods at Low Temperatures* (Berlin: Springer-Verlag) p 57
[5] Y Touloukian ed 1977 *Thermophysical Properties of Matter* vol 13, p 188

Acknowledgements

The development and verification of this measurement system was supported by the NASA Engineering and Safety Center (NESC). The measurement of alumina and SmCo was supported by the Motors for Dusty and Extremely Cold Environments (MDECE) Project.