Retrofit of an Commercial Dilatometer for Cryogenic Thermal Expansion Measurements

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Abstract:
While high-temperature dilatometer systems are commercially available, systems that can measure at high-temperature as well as low temperatures are rare. An existing conceptual design was implemented to enable a commercial dilatometer to make measurements at cryogenic temperatures. Data in this paper suggests that the principles used are sound and that further improvements (not possible within the timeframe of this project) will allow for accurate measurements to 4 K.

Objective
To implement NHMFL’s conceptual design of a commercial dilatometer for usage at cryogenic temperatures.

Introduction:
Thermal expansion is the measurement of length change due to change in temperature.

\[ \% \text{Expansion} (T) = \frac{L(T) - L(293 \, K)}{L(293 \, K)} \]

A pushrod dilatometer is an instrument that operates on the concept that the sample platform and the pushrod are made of the same material to measure the differential displacement of a sample. Ideally if no sample was present the differential displacement would be zero as the pushrod and platform would expand at the same rate.

Initial Configuration
The dilatometer was set up to measure thermal expansion in a furnace. The sample would be placed in the quartz tube as seen in Figure 2 with the quartz pushrod resting on top of it. It then would be placed into the furnace where the displacement would be measured through the pushrods.

Changes
• The bell jar (see Figure 5) moved over to the cold temperature setup, where a Invar tube was pushed into place at the bottom of the bell jar.
• Quartz pushrods were replaced with Invar pushrods.
• Copper plates were soldered to the Invar probe to help stratify the temperature gradient when running tests inside the dewar. (Figure 4)
• New Mitutoyo indicators were added to allow reading of the displacement by a Labview program.
• A Lakeshore Cernox sensor was for accurate cryogenic temperature measurements.
• E-type thermocouples added to monitor the thermal gradient within the system.

PROCEDURE:
• The dewar was filled up to a level of 1 foot with liquid nitrogen
• The probe and the sample were then immersed in this dewar for 5 minutes, allowing the system to reach thermal equilibrium
• The dewar was removed and emptied before reattaching the dewar.
• The sample was allowed to warm up to room temperature.

Calibration
• Followed described procedure while measuring copper, our reference material.
• The resulting displacement vs. temperature curve had the displacement of the dilatometer in it. The correction curve was found by subtracting the expected displacement of the copper sample from the curve to get the displacement of the dilatometer
• This correction curve is applied by subtracting it from the raw displacement

Analysis:
• Measured another standard, aluminum to examine the accuracy of the correction curve we found
• We were able to reproduce the reference % expansion curve of aluminum above 130 K.

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FUTURE WORK:
• Develop methods to control the atmosphere of the dewar to reduce effect of exchange gases and humidity as well as a more repeatable warm-up curve below 130 K.
• Utilize non-contact displacement sensors to develop techniques of measuring fragile samples
• Measure the thermal expansion of interesting materials such as YBCO

Figure 1. A schematic of a pushrod dilatometer
Figure 2. A 50 mm tall copper sample resting on the sample platform.
Figure 3. The indicator measures displacement. Displacement is transferred through pushrod to indicator
Figure 4. The dilatometer’s Invar tube, The copper baffles soldered to the probe stratify the temperatures by creating regions of uniform temperature rather than a continuous gradient
Figure 5. The bell jar containing all the measuring equipment and electronics
Figure 6. The reference data[1] for the thermal expansion of Aluminum compared with our measured data