MEASUREMENTS OF THE MATERIAL PROPERTIES OF A LAMINATED PIEZOELECTRIC STACK AT CRYOGENIC TEMPERATURES

R.P. Taylor[1], G.F. Nellis[1], S.A. Klein[1], D.W. Hoch[1], J. Fellers[2], P. Roach[2], J.M. Park[3] and, Y. Gianchandani[3]

[1]Cryogenics Engineering Lab, University of Wisconsin
Madison, WI 53706 USA

[2]NASA Ames Research Center-Cryogenics Group
Moffett Field, CA 94035 USA

[3]Solid State Electronics Lab, University of Michigan
Ann Arbor, MI 48109 USA

ABSTRACT

Future NASA missions require cooling of large structures in space. One class of thermal management solutions for providing controlled, distributed cooling would utilize actively controlled micro-scale valves that are integrated with heat exchangers and sensors in order to provide independent, local temperature control. The most attractive actuation method for these micro-valves is a multilayer piezoelectric (PZT) stack because this technology is capable of providing large force using reasonable voltages (e.g., < 100 V) with minimal power draw. In order to design a micro-valve configuration that takes advantage of this actuation technique, it is necessary to obtain information regarding the behavior of piezoelectric materials at cryogenic temperatures. This paper describes a test facility that was designed to achieve precise measurements of the coefficient of thermal expansion (CTE) and PZT stack actuator constant ($\overline{d_{33}}$) from 40 K to room temperature. The operation of the facility is validated by measuring the CTE of a copper alloy with well-known behavior. Experimental measurements are subsequently presented for a commercially available PZT stack.

KEYWORDS: Microvalve, MEMS, Piezoelectric, Coefficient of thermal expansion
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INTRODUCTION

Future space missions will require distributed cooling of larger systems such as optical assemblies or propellant depot stations. One solution is to use an autonomous distributed cryogenic cooling network that is energized by a single source of cooling. More specifically, multiple micro-scale valves that are integrated with heat exchangers and temperature sensors in order to automatically regulate the flow of a cryogen and therefore control the distribution of cooling that is provided in response to the distribution of the refrigeration load.

The use of micro-scale, locally controlled valves to distribute a centrally generated cooling load has advantages relative to temperature uniformity and efficiency. There are several specific requirements for a micro-valve that can be used within this application. The valve must be robust to large temperature changes and capable of operating from room temperature (or slightly above) down to cryogenic temperatures. The valve must be reliable and tolerant of contamination so that it has an operational life of at least 10 years. Additionally, since this is a MEMS device it should have a small overall footprint so that many valves can be fabricated on a wafer and also in order to reduce system mass and allow the integration of the valve with on-chip heat exchangers and temperature sensors. The valve must not consume significant power while it is actuated.

Initial development efforts have focused on a valve that can be used at or near 20 K in order to control the flow rate of helium. Therefore, the valve must be able to affect a large modulation (i.e., a large change in its flow area) so that large changes in the cooling can be provided and also handle a large flow rate with small pressure loss so that the input power to the circulating mechanism is minimized. This requirement suggests that the valve must generate a large flow area in a small package.

The most attractive actuator technology for this application appears to be a multilayer piezoelectric (PZT) stack. The PZT stack has the key advantages of requiring negligible power during steady operation and of generating very large forces which makes the valve robust to friction and contamination; PZT actuators are well developed and even available commercially in a wide variety of configurations and materials. However, the PZT actuator has the disadvantage of providing only relatively small displacements; typically only a few micrometers of motion can be achieved for devices that have sizes that are compatible with a 1 cm^3 footprint. The small stroke is particularly problematic for this application where a large flow area modulation is desired.

Fortunately, the limited PZT displacement can be overcome using perimeter augmentation. The flow area for an out-of-plane valve is given by the product of the valve stroke and the perimeter of the valve seat. Rather than focusing on amplifying the valve stroke, it is possible to increase the perimeter using MEMS fabrication techniques to install a precise series of interleaved grooves. FIGURE 1 illustrates the preliminary design of a perimeter augmented valve. The valve itself consists of a perimeter augmented silicon valve die and a pyrex base plate. The PZT actuator will be installed in an energized state so that the die is pressed against the base plate and the valve is closed; the flow can be modulated by removing the voltage. The PZT actuator is surrounded by a structure that serves the dual purpose of providing a hermetic boundary and carrying the actuator force. This structural configuration is similar to the out-of-plane valves that have been described previously [1, 2]. The valve die is suspended on flexures that serve the purpose of aligning the valve die to the seat as well as providing a spring back mechanism which will allow the valve to remain fully open in the event of actuator failure. The valve die is actuated by the multilayer piezoelectric stack which is driven against a pyrex valve seat that contains the gas inlet and outlet ports. Etched into the valve disk is a pattern of raised ridges or lands,

as shown in FIGURE 1(b). The raised ridges act as the perimeter and therefore provide a large valve area for a given actuator stroke. The high pressure region in FIGURE 1(b) represents area that is etched away (like a trough) between adjacent lands and is filled with high pressure gas that enters through the valve inlet port in the valve seat die. The low pressure region is also etched away and filled with low pressure gas that is connected with very little flow resistance to the valve outlet port. The total effective flow area for the valve is therefore equal to the product of the stroke and the total length of the lands; using MEMS fabrication, the total length of the lands can be made extremely large within the confines of a small footprint.

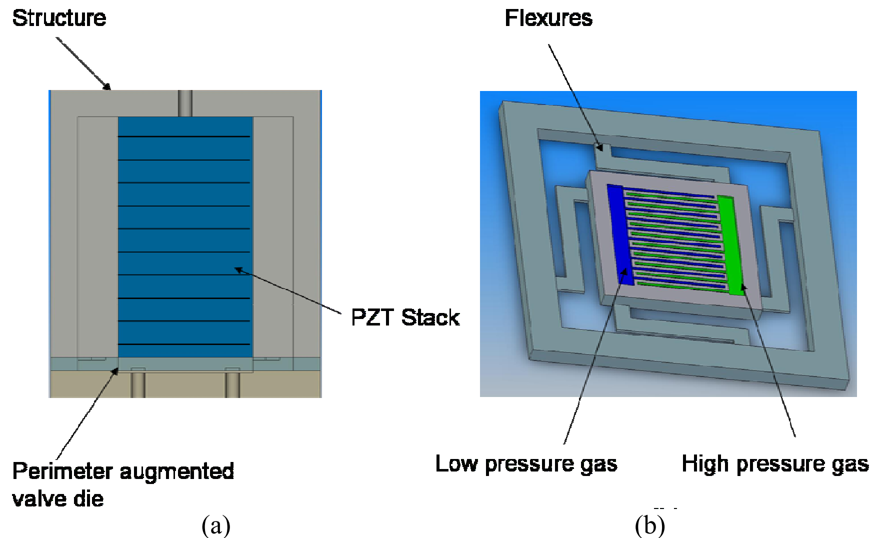

FIGURE 1. (a) Concept for the perimeter augmented valve; (b) Pattern of raised lands etched into the perimeter augmented silicon wafer die; bottom view of wafer.

Cryogenic Material Properties of PZT

One of the fundamental challenges associated with the perimeter augmented valve concept is related to distortions in the structure and actuator that are driven by temperature change. The relative motion of the PZT actuator and the structure (the outer enclosure) due to a mismatch in their coefficients of thermal expansion (CTE) can be on the same order as the motion that is produced by energizing the PZT actuator. Preliminary studies have shown that this thermal distortion effect can be compensated for by applying an appropriate pre-load at assembly; however, this can only be accomplished if the CTE of the materials involved are known precisely. In order to design the valve it is also necessary to know the actuator constant of the PZT stack at cryogenic temperatures. Some data have been published by NASA relative to the properties of various PZT crystals in the -150 to +250°C [3] and the various manufacturers of PZT stacks, such as Physik Instrumente, Morgan Electro Ceramics, and American Piezo, are able to provide only a very limited amount of information relative to the properties of their materials at very low temperatures. All of these sources indicate that the stack actuation constant ($\overline{d_{33}}$) will decrease substantially with temperature.

There is a lack of consistent and reliable information regarding the cryogenic value of the actuator constant and coefficient of thermal expansion of a multilayer PZT stack and yet this information is critical to the design of the micro-valve discussed earlier. Therefore, the remainder of this paper describes a test facility that is capable of precisely measuring these quantities at cryogenic temperatures. The test facility is verified and measurements for a commercial PZT stack are presented.

TEST APPARATUS

A schematic of the test apparatus is shown in FIGURE 2. The experiment is placed within a vacuum chamber that is evacuated to below $1\text{x}10^{-6}$ torr using a turbomolecular pump. A Cryomech AL60 GM cryocooler is used to cool the sample to less than 40 K for testing. The cryocooler is thermally linked to the test platform by a compliant thermal strap that is composed of many very thin copper laminations. The test platform consists of a relatively large and therefore thermally massive copper disk that is suspended from the top flange of the vacuum vessel by three, thin-wall G10 tubes. Two fiber-optic displacement probes penetrate the bottom flange of the vacuum vessel and register the relative motion of the test platform and the sample, respectively.

The details of the test platform are illustrated schematically in FIGURE 3. The bottom of the sample is adhesively mounted to the test platform and a reflective coating is placed on the top surface of the sample as well as on the region of the test platform that forms the target for the second probe. The fiber-optic displacement probes (Model D-64 from Philtec, Inc.) measure displacement by passing the light that is emitted by an LED at 880 nm through one fiber-optic cable so that it strikes the surface to be measured. The intensity of the reflected light provides a measure of the distance between the sensor and the surface. It is possible to calibrate the sensor so that it provides accurate measurements regardless of the exact reflectance of the surface; although more reflective surfaces provide a stronger signal. The two probes are mounted on precision micrometer heads that are mechanically linked to the bottom flange of the vacuum vessel; this arrangement allows the precise control of the position of the probes relative to the sample and test platform.

Two Cernox resistance thermometers and two thermocouples are used to accurately measure the temperature of the sample during testing. These sensors are installed onto a brass shim and the sensor leads are thermally anchored to the brass using a Stycast epoxy. The sensors attach directly to opposing sides of the sample in order to provide an average temperature during testing. An additional silicon diode is attached to the test platform. The effective thermal conductivity of the multilayer stack is very low (approximately 1 W/m-K) and therefore the axial thermal resistance of the sample is quite high, approximately 500 K/W for an 18 mm long sample. Because of this large axial thermal resistance, even a relatively small (10's of milliwatts) heat leak imposed on the sample will result in a substantial thermal gradient across the sample. It is therefore important to minimize the radiation heat leak imposed on the sample from the near room surroundings and so an actively cooled radiation shield was fabricated from OFHC copper shim stock and directly linked to the cold finger. Additionally, multiple layer insulation (MLI) is used to further minimize radiation heat transfer to the sample, the platform, and the cryocooler.

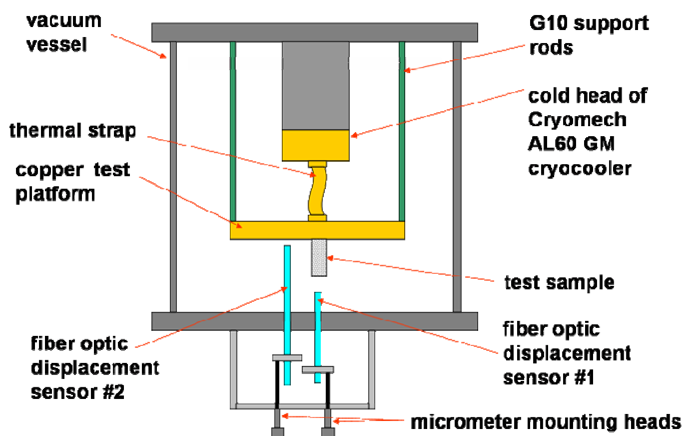

FIGURE 2: Schematic of the test apparatus

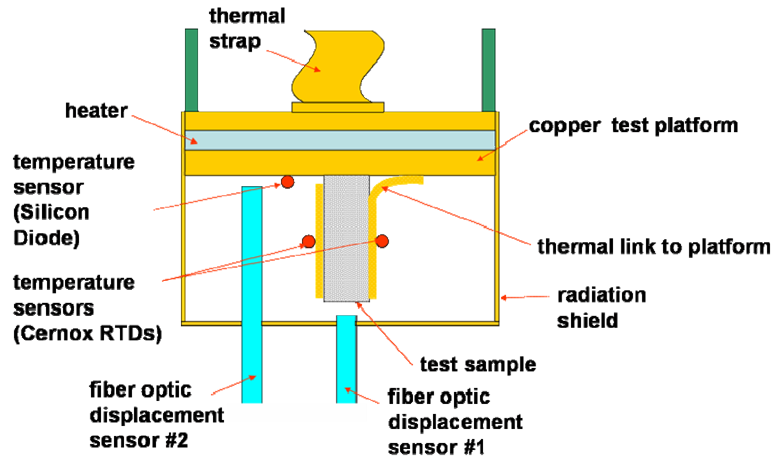

FIGURE 3: Schematic of the sample test platform.

TEST PROCEDURE

In order to measure the CTE of the sample, the cryocooler is activated and allowed to run until a steady state temperature of approximately 40 K is achieved at the sample. At this point, the cryocooler is deactivated in order to eliminate the vibration that is generated by its operation. The two fiber optic probes are calibrated and positioned for testing. The test platform is allowed to slowly warm up; the rate of temperature rise can be controlled using the heater installed on the test platform. The difference between the motion of the sample surface and the motion of the test platform corresponds to the dimensional change of the sample. It is necessary to re-position and re-calibrate the probe during the warm up if the dimensional change exceeds the useful range of the probe; however, the CTE measurement corresponds to the slope of the dimensional change plotted against the average sample temperature and therefore adjustment of the probe does not impact the measurement.

The same basic procedure is followed in order to measure the actuator constant of the sample. However, during the warm up process (which occurs very slowly, over a few hours) a voltage is applied to the sample relatively quickly (over a few seconds) in order to determine the actuation constant at a particular temperature. The displacement as a function of the applied voltage is recorded and the actuator constant is related to the slope of the displacement versus the applied voltage. A typical set of data showing displacement as a function of applied voltage is shown in FIGURE 4. Note that the PZT stack exhibits a very clear hysteretic behavior which is expected and does not pose a problem for the micro-valve application.

DATA REDUCTION

Neither the $\overline{d_{33}}$ or the CTE are measured directly; rather, both measurements correspond to a calculated slope based on a measured data set. The data reduction corresponds to identifying a subset of measurements over a limited range (of voltage or temperature) and applying a linear regression analysis. Both the dependent data (the displacement) and the independent data (temperature or voltage) are measurements that are characterized by some uncertainty. The measurement of a relative displacement is accurate to within nominally 0.07 μm, temperature measurements are characterized by an

uncertainty of nominally 0.25 K, and the voltage measurement is characterized by an uncertainty of nominally 15 mV. The uncertainty in the $\overline{d_{33}}$ and CTE measurements are obtained by propagating these individual measurement errors in the data points through the linear regression formula, as described by Taylor et. al [4].

EXPERIMENTAL APPARATUS VALIDATION

In order to verify that the experimental test apparatus described above is capable of providing accurate measurements, a copper sample in the form of a 5.0 mm square bar, 18.0 mm long was installed in the test apparatus (nominally the same dimension as the PZT sample measured subsequently). Copper is a relatively well-studied material and therefore accurate, published data is available relative to the cryogenic behavior of the CTE of copper. Published data for the CTE of copper can vary by 1-2 ppm/K depending on the source; the CTE of oxygen free high conductivity (OFHC) copper published by the National Institute of Standards and Technology and two less pure alloys were used as the basis of comparison [5]. The experimental measurements and these published results are shown in FIGURE 5.

EXPERIMENTAL DATA

For the current analysis we present the experimental results of the effective PZT actuation constant normalized to length as well as the thermal expansion coefficient of the PZT stack with associated uncertainty in the measurements for a nominal temperature range of 40-300 K. The sample tested was obtained from Physik Instrumente (Model Number: P-885.50).

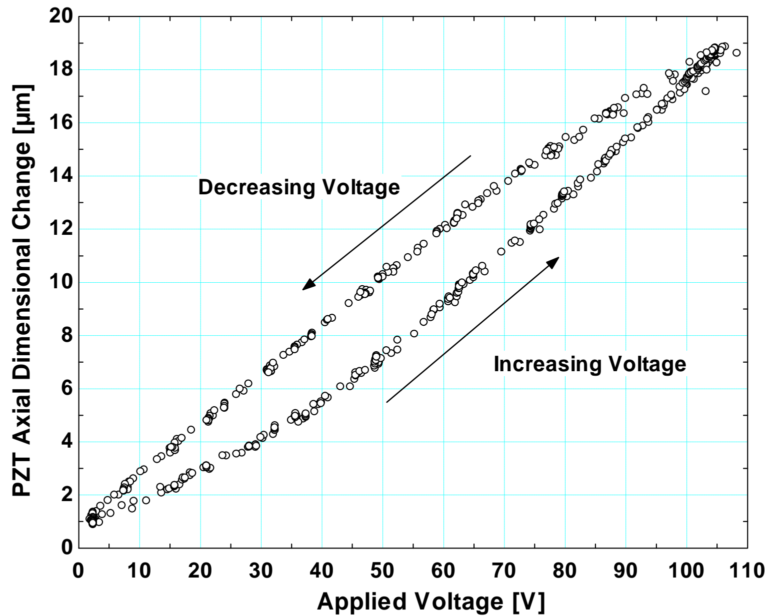

FIGURE 4: Typical measurement of displacement as a function of the applied voltage at a particular temperature. Note that the $\overline{d_{33}}$ at any temperature is related to the slope of this curve.

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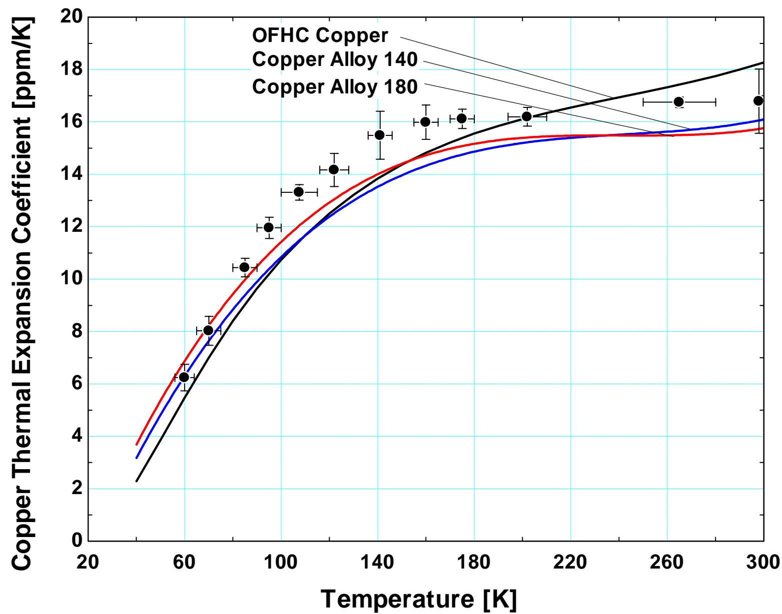

FIGURE 5: Experimental values for the CTE of copper compared to published values of the CTE of copper.

The experimental results for the PZT actuator constant (strain per unit voltage) are presented in FIGURE 6. The data is separated into two categories related to the increasing and decreasing voltage portions of the curve shown in FIGURE 4. The data was taken over an applied voltage range of 40-60 V with a median value of nominally 50 V. Note as the temperature of the PZT stack is reduced the actuation constant decreases; the nominal value of the actuation constant is reduced to approximately one third of its nominal room temperature value at 40 K.

The experimental results for the PZT stack thermal expansion coefficient (strain per unit temperature) are presented in FIGURE 7. The measurements were taken with the leads of the stack shorted in order to prevent any self energizing effects. Notice that the CTE declines rapidly when cooled below room temperature and this decline continues until approximately 120 K below which the CTE is small and less sensitive to temperature.

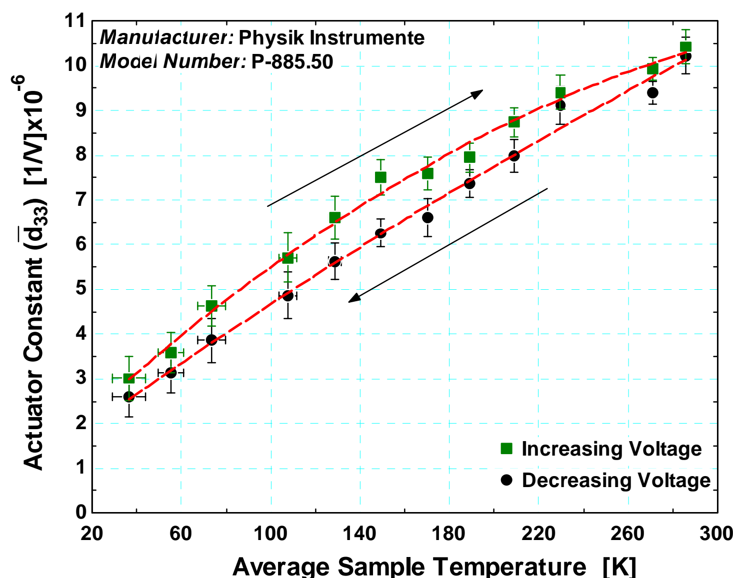

FIGURE 6: Measured effective PZT stack actuation constant as a function of temperature.

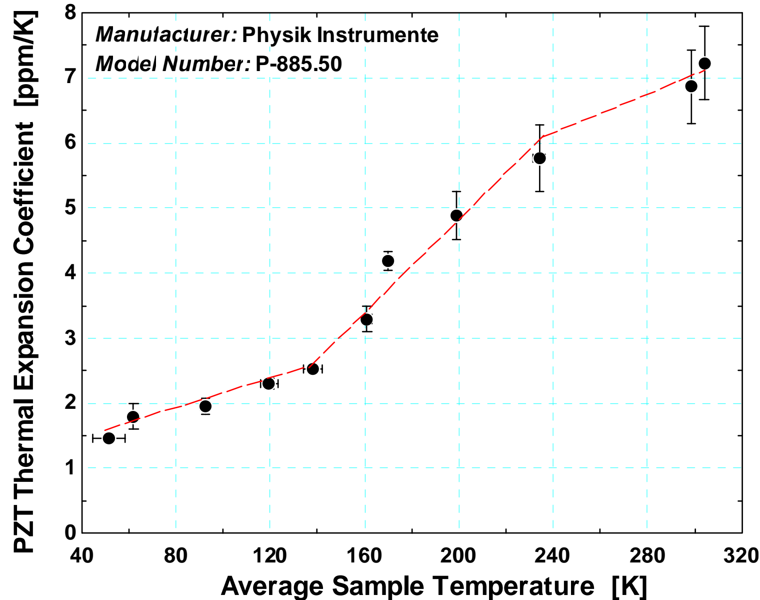

FIGURE 7: Measured effective PZT stack coefficient of thermal expansion as a function of temperature.

CONCLUSION

A design for an actively controlled cryogenic micro-valve that utilizes a PZT actuator with a perimeter augmented valve die has been discussed. The design of this micro-valve requires the measurement of the PZT stack material properties and so a test facility was designed and constructed. The test facility operation was verified and experimental results for the actuator constant and CTE were reported.

Future work with this apparatus will characterize the thermal expansion coefficient for various MEMS structural materials at cryogenic temperatures in order to facilitate the design of the micro-valve and other cryogenic MEMS applications. The facility will also be used to characterize the integrity of MEMS bonding techniques at cryogenic temperatures.

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