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STABILITY OF CERNOX ä RESISTANCE TEMPERATURE SENSORS

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ABSTRACT

Cryogenic temperature sensing devices fabricated from sputtered zirconium oxy-nitride thin films have been commercially available from Lake Shore Cryotronics, Inc. under the tradename Cernox™ Resistance Thermometers (CXRTs) since 1992. These sensors possess many qualities desired in a cryogenic thermometer and are presently widely used. To date, no long term stability data has been available in the literature. Over the past six years, thirty-nine temperature sensors from six of the initial wafer production lots have been calibrated periodically and monitored for long term calibration stability and aging effects. These devices were stored at room temperature between calibration. Stability comparisons are given in terms of elapsed time, package type, wafer lot, and sensitivity level. Analysis of calibration data over the six year period show that these devices have repeated their initial calibration to within an average of ±0.08% of temperature over the 1.4 K to 325 K temperature range. Small qualitative and quantitative differences in stability were measured with respect to wafer lot, sensor sensitivity and package.

INTRODUCTION

Resistors fabricated from sputtered zirconium nitride thin films were shown to be possible cryogenic thermometers by Yotsuya, *et al*.[1] Further work performed at Lake Shore Cryotronics, Inc. resulted in the development of commercially available devices under the tradename Cernox™ resistance temperature sensors (CXRTs).[2,3] Sputter deposition occurs in an atmosphere consisting of argon, nitrogen, and oxygen. Judicious selection of the deposition parameters allows incorporation of oxygen into the ZrN lattice. The lattice enlarges as more oxygen is incorporated into the ZrN lattice, steadily changing the electrical conductivity from metallic behavior to percolating behavior.[3] Resistance temperature sensors can thus be produced with sensitivities designed to cover desired temperature ranges in a manner similar to germanium resistance temperature sensors. These devices have many useful properties including a wide temperature range, small physical size, fast thermal response time, low magnetoresistance, and excellent resistance to ionizing radiation. Data detailing these characteristics are available either from the literature or from the manufacturer.[2,5,6,7,8,9,10] To date, however, the long term stability of these types of devices have not been quantified. The present work was undertaken to provide long term stability data for Cernox™ resistance temperature sensors.

EXPERIMENTAL PROCEDURE

Zirconium oxy-nitride films were reactively sputtered in an argon/nitrogen/oxygen atmosphere. The deposition parameters were varied to produce a series of wafers whose room temperature dimensionless sensitivity $S_T \equiv (T/R)(dR/dT)$ varied from about -0.5 to -1.5. The dimensionless sensitivity is independent of geometry and allows for quick comparison between films independent of the final sensor pattern. Test sensors were fabricated from each wafer using standard photolithography techniques to define the active sensor area and subsequent contacts. Contacts were formed in the shape of inter-digitated fingers to allow trimming sensor resistance values upward to a desirable level. **Figure 1** shows a finished sensor chip layout and scale. The lower temperature limits of these sensors are designed to cover three temperature ranges of commercial cryogenic importance as outlined in **Table 1**. For each model designation, the resistance is limited to 100 kΩ at the minimum temperature. Typical room temperature resistances are on the order of 30 to 60 Ω. **Figure 2** shows a typical resistance versus temperature curve for a sample of each wafer tested. **Figure 3** shows the typical sensitivity, dR/dT, for each sample plotted in **Figure 2**. Finally, **Figure 4** shows the dimensionless temperature sensitivity, (T/R)(dR/dT) for each of the samples shown in **Figure 2**.

To prevent film surface damage/contamination and facilitate handling, these bare chips were packaged in one of two ways. The first method utilizes a 3 mm diameter by 8 mm long gold-plated copper canister identical to those used for other common cryogenic thermometers such as germanium and carbon glass resistance thermometers. Two 50.8 µm diameter gold wires are ball bonded to the Cernox chip contact pads. The chip is then epoxied to a beryllium oxide header. The gold leads are spot welded to phosphor bronze lead wires that pass through the beryllium oxide header. The gold plated copper can and phosphor bronze lead wires are then Stycast epoxied to the header creating a hermetic seal. The can is filled with He-4 gas prior to sealing to enhance thermal connection. In the second

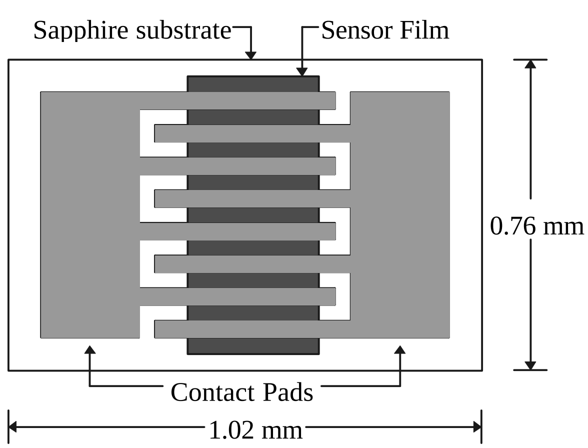

Figure 1. Chip layout and scale.

Table 1. Cernox™ RTD Temperature range and model designations.

Wafer ID	Temperature range	Model Designation	Cryogenic Purpose	Number of samples
2Z184A	0.3 K - 325 K	CX-1030	Pumped He-3 system	4
2Z243B	1.4 K - 325 K	CX-1050	Pumped He-4 system	13
2Z245A	4 K - 325 K	CX-1070	Unpumped He-4 system	16
2Z251A	4 K - 325 K	CX-1070	Unpumped He-4 system	3
2Z255A	0.3 K - 325 K	CX-1030	Pumped He-3 system	1
3Z392A	1.4 K - 325 K	CX-1050	Pumped He-4 system	2

method, the Cernox chip is soldered into the cavity of a 3 mm long by 1.84 mm wide by 0.98 mm tall ceramic package. The base of the package is sapphire while the sides and top are alumina. Electrical connections are made by ball bonding one 25.4 μm diameter gold wire from each chip contact pad to the package electrical feedthrough bond pad. After packaging, the devices are thermally cycled from room temperature to liquid nitrogen temperature 200 times for conditioning.

Temperature calibrations from 1.4 K to 325 K were performed in Lake Shore Cryotronics' commercial Temperature Calibration Facility. Temperature and resistance was measured using standards grade platinum and germanium thermometers in conjunction with a Keithley Model 224 current source, Hewlett Packard Model 3456A DVM, and Guildline Model 9330 standard resistors (resistance values from 10 Ω to 100 kΩ in decade steps). The device under test was placed in series with a standard resistor of comparable value. The current was varied to a minimum of 0.1 μA to maintain a nominal 2 mV signal level across each sensor during calibration. The voltmeter was used in a ratiometric form with readings taken across both the standard resistor and device under test. Current reversal was performed to eliminate thermal EMFs. Approximately 84 temperature - resistance data points were taken for each sensor over the 1.4 K to 325 K temperature range. Two points beyond each extreme were taken for curve fitting purposes. After each calibration, the data for each sensor was smoothed using a combination of a least squares fit and a

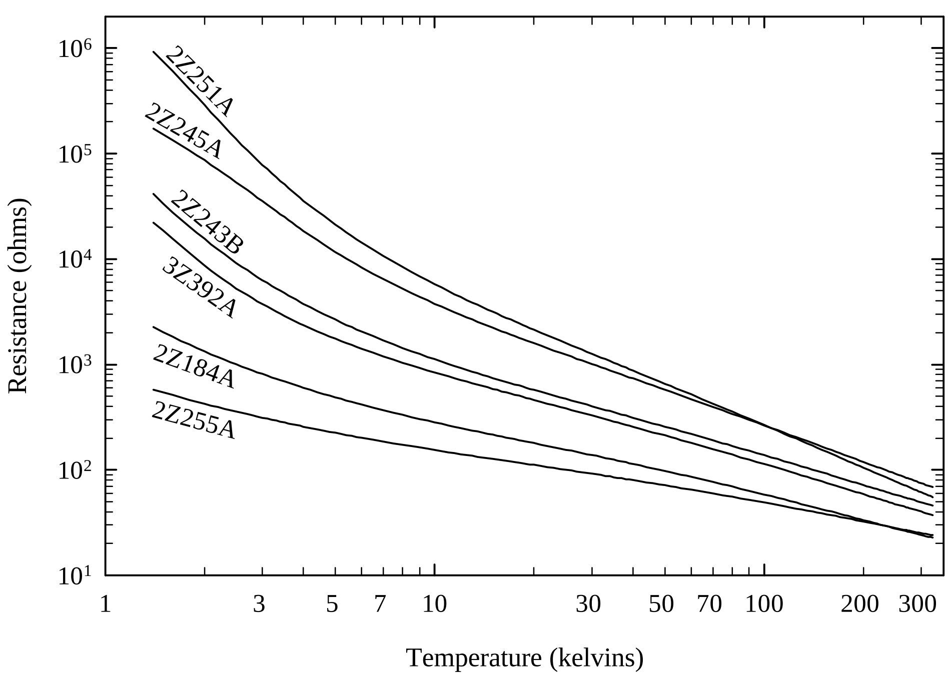

Figure 2. Typical resistance versus temperature characteristic for each wafer tested for stability.

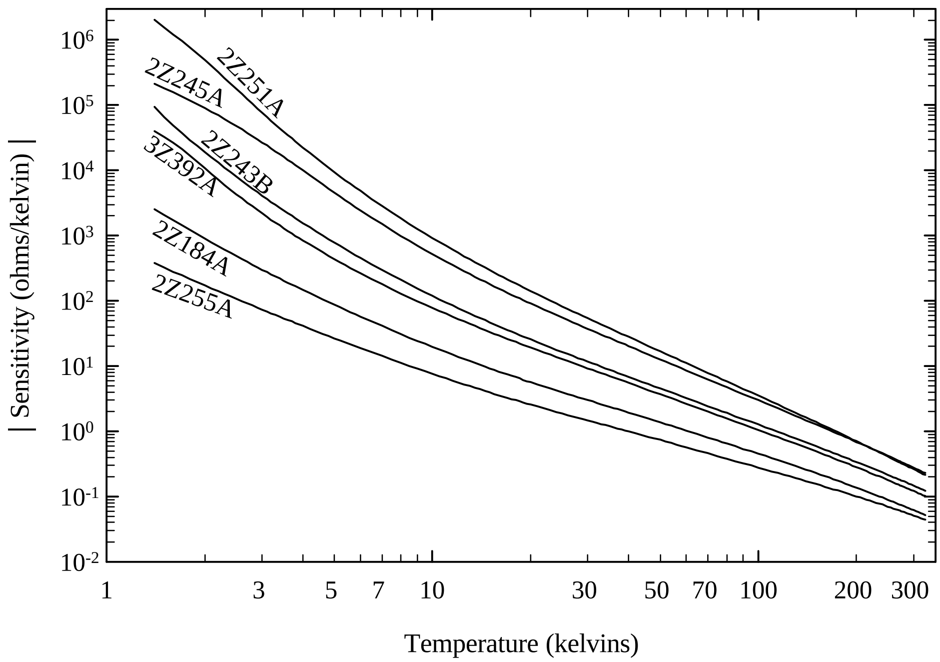

Figure 3. Typical temperature sensitivity versus temperature characteristic for each wafer tested for stability.

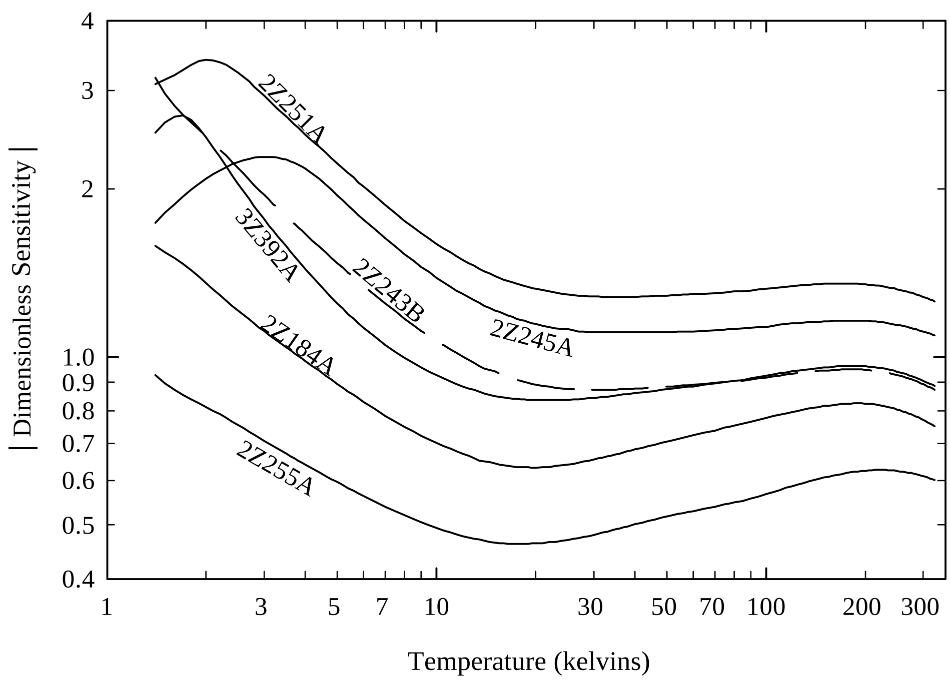

Figure 4. Typical dimensionless temperature sensitivity versus temperature characteristic for each wafer tested for stability.

Chebychev polynomial fit. After smoothing the data, a cubic spline fit was used to generate a table of resistance, temperature sensitivity, and dimensionless temperature sensitivity at a chosen set of comparison temperatures. The resulting calibration shifts were calculated from these tables as $\Delta T=(R_{final} - R_{initial})/(dR/dT)$. The calibration shift at each temperature is considered to be indicative of the stability at each temperature.

The resulting calibration accuracy is temperature dependent. The contributions to the accuracy come from stability of the current source, stability of the resistance standards, stability of the voltmeter, and accuracy of the NIST traceable thermometers. Typical values of accuracy range from ±4 mK at 4.2, ±8 mK at 20 K, ±30 mK at 100 K, and ±180 mK at 300 K. Given a worst case scenario, it is possible for the recalibration inaccuracy to be twice the specification of a single calibration. It should be noted that the resolution is typically an order of magnitude or more better than the accuracy.

EXPERIMENTAL RESULTS

Calibration shift data was analyzed in four ways: 1) by time, 2) by mounting package after 4.4 years, 3) by wafer after 4.4 years, and 4) by sensitivity (model) after 4.4 years. Comparison at the 4.4 year time mark was chosen because all sensors in the study were recalibrated at that point. Subsets were calibrated at other times. In each comparison, the offset shown is relative to the initial calibration as $\Delta T/T$ (%) versus temperature. Data for the model CX-1070 sensors are considered only over that model's designed 4 K to 325 K temperature range. Below 4 K, self heating occurs in this model as a consequence of the large resistances, obscuring the stability measurement.

Stability dependence upon time. Various subsets of the 39 sensors were calibrated at seven different elapsed times over a 5.8 year period after construction and initial calibration. These elapsed times are 0.27 years (4 samples), 1.1 years (8 samples), 1.6 years (15 samples), 2.2 years (8 samples), 3.0 years (28 samples), 4.4 years (39 samples), and 5.8 years (25 samples). The sensors tested at each point in time were varied to ensure that the total cycling effects on each individual sensor was minimized. The average calibration shift as a function of temperature at each calibration time is shown in **Figure 5**. Notable is the general trend toward a negative temperature shift at temperatures starting around 50 K and going to room temperature. Since these are negative temperature coefficient devices, this negative temperature shift is indicative of a higher resistance either in the body of the film, in the contacts, or in both. Although there is a shift toward negative temperature, the shift does not grow at a constant rate (i.e. the shift does not correlate well with time). Over the 5.8 year test cycle, the average calibration offsets are less than ±0.02% of temperature below 40 K and ±0.08% of temperature above 50 K at all times tested.

Stability dependence upon package. Sensors were packaged in either a copper can (8 samples) or in a flat, hermetically sealed package (31 samples). The average calibration shift as a function of temperature for these two groups of devices 4.4 years after construction and initial calibration is shown in **Figure 6**. There is a marked difference between long term stability indicated for the two packages. It would be expected that the copper can package would yield a more stable sensor due to decreased stresses transmitted from the package to the sensor. The data shows that sensors packaged in these copper cans have drifted to larger negative temperature shifts with ΔT going roughly as 0.10% of temperature. As mentioned earlier, these are negative temperature coefficient devices, so a negative temperature shift is indicative of a higher resistance either in the body/contacts system. On the other hand, Cernox™ devices packaged in the flat hermetic packages have small deviations around zero with the offset below ±0.05% of temperature over the 4.4 years. This difference might be attributed to better matching of thermal expansion coefficients between the Cernox chip and the flat hermetic package, yielding less stress on the sensor film

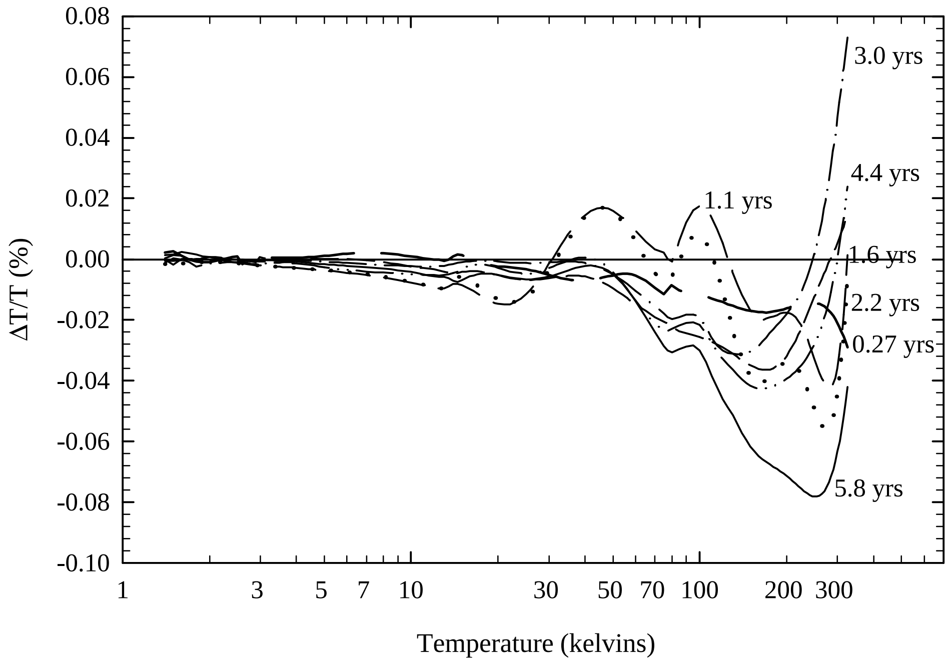

Figure 5. Average calibration shift as a function of temperature at each point in time for Cernox™ RTDs.

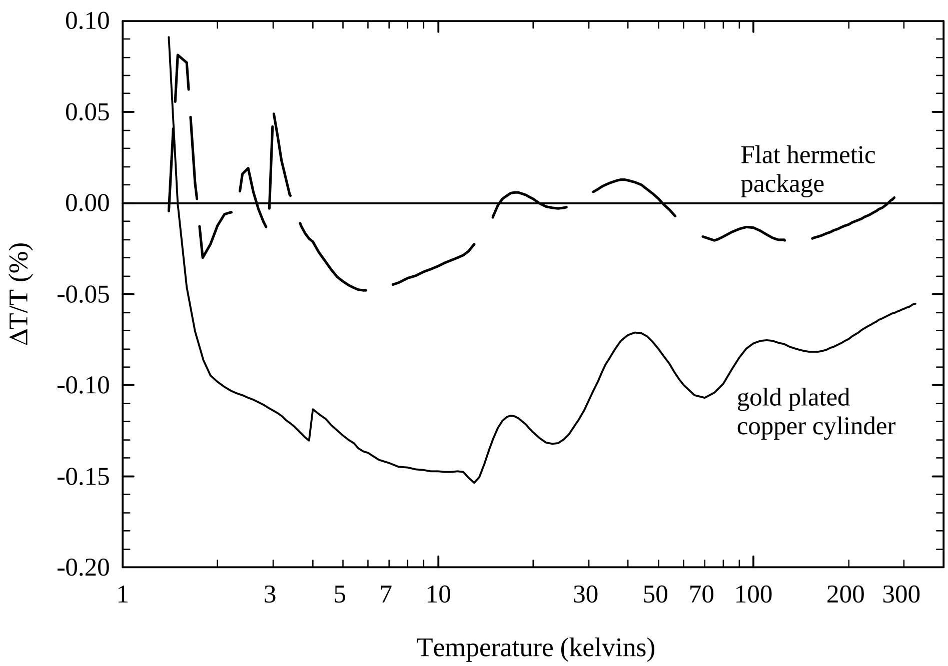

Figure 6. Calibration shift at 4.4 years as a function of temperature for two package types of CXRTs.

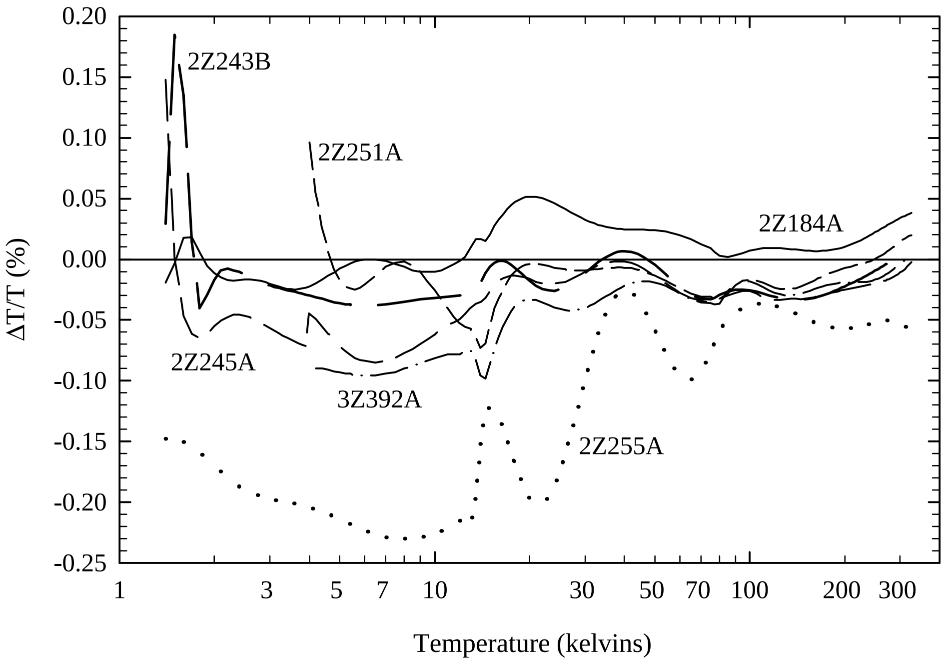

Figure 7. Calibration shift at 4.4 years as a function of temperature for each wafer used in this experiment.

upon thermal cycling. In the flat hermetic package, a solder attachment is formed between two pieces of metallized sapphire, one of which contains the Cernox™ thin film. An epoxied sapphire-to-BeO attachment is formed for the copper can. It is also possible that the flat hermetic package, with its solder ring seal, yields better hermeticity. This would keep contaminants out of the package and away from the sensor film better than the copper can with its Stycast epoxy seal.

Stability dependence upon wafer. Sensors were fabricated from six different wafers. Each wafer produces about 1300 sensors. The number of samples tested from each wafer (small in some cases) is given in **Table 1**. The average calibration shifts as a function of temperature are presented in **Figure 7** for these six groups of devices 4.4 years after construction and initial calibration. Similar behavior is noted among all of the wafers from 40 K to 325 K, where small negative temperature drifts are typical. Below 40 K, five wafers show a tendency to drift negatively in temperature. The sixth wafer, 2Z255A, also drifted in the negative temperature direction, but to a level roughly three time larger than the other five. Overall, the long term stability for all wafers is better than ±0.25% of temperature below 40 K and better than ±0.10% of temperature above 40 K over the 4.4 year period.

Stability dependence upon sensitivity (model). The sensors were purposely designed with the best sensitivities to cover specific temperature ranges. The Lake Shore model numbers designated for these sensors are CX-1030 (least sensitive, widest range), CX-1050, and CX-1070 (most sensitive, narrowest range). The number of samples of each model type is listed in **Table 1**. The average calibration shifts as a function of temperature for these three models of Cernox devices 4.4 years after construction and initial calibration are shown in **Figure 8**. Note that the shape of the offset is very consistent between the three levels of sensitivity. As might be expected, the model CX-1030 with the lowest sensitivity has the highest offset. Again, the negative temperature shift is indicative of an increase in resistance. For all three models, the offset is less than ±0.07% of temperature.

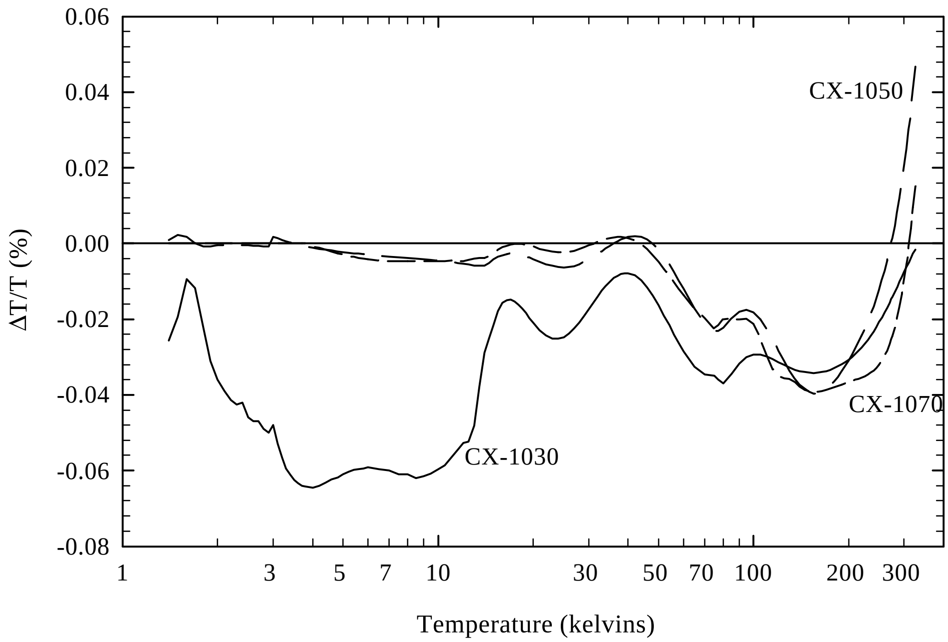

Figure 8. Calibration shift at 4.4 years as a function of temperature for three levels of sensitivity of CXRTs.

CONCLUSIONS

There does appear to be a tendency toward a higher resistance/negative temperature shift with the passage of time with the CXRT sensors stored at room temperature, but the size of the shift does not correlate well with time. Over the 5.8 year test cycle, the average calibration offsets are less than ±0.02% of temperature below 40 K and ±0.08% of temperature above 50 K at all times tested.

At the 4.4 year time mark, the CXRTs mounted in a flat hermetic package were shown to be much more stable (ΔT less than ± 0.05% of temperature) than those mounted in the gold plated can (ΔT less than ±0.1% of temperature). This might be attributed to the better matching of thermal expansion coefficients in mounting the Cernox chip in the flat hermetic package and the better hermiticity of the flat hermetic package compared to the copper can package.

At the 4.4 year time mark, similar long term stability was found among all wafers over the 40 K to 100 K temperature range. Below that temperature, one wafer showed a quantitatively different stability. Overall, the long term stability for all wafers is better than ±0.25% of temperature below 40 K and ±0.10% of temperature above 40 K.

The long term stability was measured to be very similar among all three levels of sensitivity (models) tested. The model with the lowest sensitivity showed the highest offset. For all three models, the offset is less than ±0.07% of temperature.

FUTURE STUDIES

The fabricated Cernox™ sensors for this experiment were inherently two-lead devices subject to calibration shift due to changes in contact resistance. A better design would incorporate a true four-lead measurement which would be independent of the changes in contact resistance. Future studies will incorporate appropriately designed four-lead CXRTs.

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