Epoxy encapsulation of the CernoxTM SD thermometer for measuring the temperature of surfaces in liquid helium

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Abstract

We describe a procedure to pot a CernoxTM thermometer with the SD package in Stycast epoxy. The potting adapts the thermometer for measuring the temperature of a surface immersed in liquid helium (LHe) and other cryogenics. The technique thermally insulates the sensor chip from the cryogen while preserving the surface mounting capability of the SD package. The potting introduced <1% shift in the resistance, <0.5% shift in the calibration at 4.2 K and 77 K, and provided repeatable measurements during thermal cycles between room temperature and 4.2 K.

Keywords

Insulation, calibration, temperature sensor, potting, cryogenic, stycast

1. Introduction

In applications where one needs to measure the temperature of a heating surface immersed in liquid helium (LHe) or other cryogenics, thermally insulating the thermometer from the liquid is crucial. Not providing this insulation will yield temperature that is some combination of the surface and the liquid temperatures. We present an epoxy potting technique that makes a commercially available thermometer-CernoxTM SD package usable in such applications. The thermometer package is carefully potted in Stycast epoxy enclosed in a fiberglass (G-10) tube to engulf the entire package in the epoxy while ensuring the temperature sensing face remains clear. During a temperature measurement of a heat transfer surface in a cryogen, the sensing face will contact the surface and the epoxy pot will thermally insulate the sensor from the cryogen. These capsules can be mounted, and demounted if required, on a flat plate or even on a tube by providing a spot-face on its cylindrical surface. This paper elaborates on the potting procedure to achieve the thermal insulation and describes the effect of potting on the thermometer calibration.

2. Epoxy encapsulation of cryogenic thermometers

Miniature cryogenic thermometers of RTD (resistance thermometer detector) type typically comprise of a small resistor (heat sensing element) connected to a pair of thin lead wires. Bending of these wires due to repeated use can cause them to detach from the sensing element, which then are extremely difficult to reconnect to the sensor. One way to adapt such thermometers to heavy duty applications is to encapsulate the sensor and the lead wires in an epoxy. The epoxy on hardening will lock the sensor and the wires in a fixed position and will not allow the wires to bend at the sensor contact points. Such encapsulation, however, can introduce two problems- first, the epoxy on contracting when cooled can impose large stress on the sensor. Second, for a given excitation current, low thermal conductivity of the epoxy can cause more self-heating in the encapsulated sensor than the self-heating of the same sensor when bare. Both

these effects can shift the thermometer calibration. Süßer [1] studied epoxy encapsulation of carbon-ceramic TVO-thermometers and reported the calibration to change by as much as 0.3 K at 4.2 K. The self-heating temperature rise was 2.5 times that of the bare sensor.

The unique construction of the CernoxTM SD thermometer package helps to mitigate the above drawbacks of epoxy encapsulation. Figure 1 displays a CernoxTM thermometer as a hermetically sealed SD package (figure 1(a) [2]) and an exploded view of the SD package (figure 1(b) [3]). The sensor chip is attached to a base plate and is contained in a vacuum enclosure. The chip does not contact any face of the enclosure (the sides or the lid) other than the base. Barring heat conduction through the thin connecting wires, the vacuum ensures that the heat from a medium to the sensor principally transfers *via* the base. The challenge then is to encapsulate the enclosure fully in an epoxy and to leave the bottom of the base plate clear for mounting. The self-heating characteristics of the sensor encapsulated this way would not change significantly because the principal heat transfer path (the base) is essentially unhindered. As the epoxy does not directly contact the sensor chip, negligible stresses due to epoxy contraction would act on the chip. The calibration change due to epoxy potting is, therefore, expected to be minimal.

Figure 1. A CernoxTM thermometer (a) SD package [2] and (b) an exploded view of the SD package [3] (Image courtesy of Lake Shore Cryotronics, Inc.)

3. Potting procedure

The objective of potting, here, is to make the CernoxTM SD usable for measuring temperature of heat transfer surfaces immersed in liquid helium. The photographs in figure 2 describe the potting procedure. The thermometer and the epoxy are encased in a short piece of a fiberglass (G-10) tube, whose ends are faced to make them flat and perpendicular to the tube axis. The procedure starts by placing a double-sided adhesive tape on a smooth surface such as a flat plywood table-top. The G-10 section is affixed on one of its ends to the tape as depicted in figure 2(a). The double-sided tape firmly holds the G-10 to the flat table-top. Wire extensions are soft-soldered to the thermometer bare leads and the soldered connections are electrically insulated using fine plastic tubing. Figure 2(b) shows the lead wire insulation. Additional support to the lead wires is provided by wrapping kapton tape around the wires. The thermometer is then placed inside the G-10 case as depicted in figure 2(c). In this step, slight pressure is applied on the thermometer lid (the face opposite to the base) to make sure that the thermometer base firmly sticks to the tape and there is no room for the epoxy to creep under the base. Stycast epoxy (2850 FT with 23 LV catalyst) is then carefully poured into the G-10 case till the entire SD package is submerged in the epoxy (figure 2(d)). This setup is allowed to stand for ≈ 12 hours without disturbing for the epoxy to cure.

Figure 2. The thermometer potting procedure (a)-(e)

Figure 2(e) shows the cured pot with the thermometer completely encapsulated in the epoxy except the base that remains flat and clear of the epoxy. This encapsulate with its flat face (carrying the thermometer base) mounted on a heater surface in a cryogen will measure the surface temperature. The epoxy pot around the thermometer body will thermally insulate the base and the sensor chip from the cryogen. A method to install this encapsulate on a heat transfer surface has been elaborated in [4].

4. Effect of potting on the thermometer performance

a) Testing procedure

Eight Cernox™ 1050-SD thermometers were potted following the procedure described in Section 3. All the thermometers were calibrated before encapsulation over the 4.2-80 K range using an in-house calibration rig. A factory calibrated Cernox™ thermometer served as the reference. The resistance vs. temperature data were curve fitted with Chebyshev polynomials following the procedure described in [5]. Column A of Table 1 shows the non-dimensional sensitivity (defined as $T/R \cdot dR/dT$, where T is temperature and R is resistance) obtained from the calibration. The sensitivities agree very well with those documented in [2]. These encapsulates were then used in a series of experiments [6,7] (column B of Table 1), which involved several thermal cycles between 295 K and 4.2 K. After these experiments, we performed systematic tests to study the shift in calibration due to potting. Columns from C to F of Table 1 show the chronology of these thermal tests. The thermometer resistance was measured in open baths of LN₂ (column C) and LHe (column D). The thermometers were then subjected to 200 thermal shocks from 295 K to 77 K *via* dip-tests in an open LN₂ bath (column E). Finally, the thermometers were dip-tested in an open LHe bath (column F) to determine the effect of the repeated thermal shocks on the calibration at 4.2 K.

Table 1: Details of the calibration and thermal cycling tests (The tests proceed from A to F)

Thermometer	A Non-dimensional sensitivity ¹		B 295 - 4.2 K cycles ² (#)	C LN ₂ dip test ³ (#)	D LHe dip test ⁴ (#)	E LN ₂ dip test ³ (#)	F LHe dip test ⁴ (#)
	77 K	4.2 K					
1	-0.94	-1.23	26	1	1	200	1
2	-0.99	-1.34	26	1	1	200	1
3	-0.93	-1.24	26	1	1	200	1
4	-0.95	-1.27	26	1	1	200	1
5	-0.88	-1.25	9	1	1	200	1
6	-0.88	-1.23	9	1	1	200	1
7	-0.98	-1.42	9	1	1	200	1
8	-0.96	-1.39	9	1	1	200	1

¹Defined as $(T/R) \cdot (dR/dT)$ and obtained by calibration before potting

²Experiments with potted thermometers in 4.2 K LHe [6,7]

³Dip test in an open LN₂ bath

⁴Dip test in an open LHe bath

indicates the number of cycles

b) Instrumentation

We measured the resistance of each thermometer by a DC four-wire apparatus consisting of a Lake Shore 120 CS constant current source (accuracy 0.1% or 0.05% depending on the current) and a Keithley® 2700 multimeter (typical voltage accuracy 0.07% for our measurements). By removing the contribution of the lead-wire resistance to the resistance measurement, the four-wire apparatus helps to reduce systematic error. The systematic error was further reduced by removing any thermal voltage that may be present in the measurement circuit. To remove the thermal voltage, we obtained the voltage across a thermometer twice during each measurement with reversed current polarities and then averaged the absolute values of the two voltages. The random error in the resistance measurement due to the instrument accuracy is 0.17% at 4.2 K, 0.12% at 77 K, and 0.17% at 295 K. These roughly translate to 6 mK at 4.2 K, 0.1 K at 77 K, and 0.6 K at 295 K. Note that these values are only representative and the exact values depend on the sensitivities of the individual thermometers (see Table 1).

c) Effect of potting

Figure 3 shows the percentage shift in the resistance and in the calibration at 4.2 K, 77 K, and 295 K. In these plots, we define the shift as the value after potting minus the value before potting. The largest resistance shift, as appears in figure 3 (a), at 4.2 K is $\approx 0.6\%$ and at 77 K is $\approx 0.4\%$. The percentage shift in resistance is divided by the non-dimensional sensitivity (Table 1) to obtain the percentage shift in calibration. As calibration data near room temperature were not obtained, we have used -0.87 as the non-dimensional sensitivity at 295 K (typical for Cernox™ 1050 thermometers [2]). The maximum calibration shift, as figure 3(b) shows, is $\approx 0.5\%$ at LHe and LN₂ temperatures. In absolute terms, the calibration shift is no larger than 25 mK at 4.2 K and 0.4 K at 77 K. The deviation at room temperature is within $\pm 1\%$.

Figure 3(a) shows the thermometer resistance after potting to be lower than that before potting. The possible reason for this difference could be the change in the self-heating characteristics of the thermometers. Although the change should not be significant because all the measurements have been done by immersing in large baths of cryogenics (LHe/LN₂), the potting around the thermometer would cause finite change in the self-heating. A potted thermometer will dissipate heat less efficiently than an un-potted unit and so will report temperature slightly higher than the actual. This, in turn, will result in a slightly lower resistance value. Although thorough investigations are needed to confirm this reasoning, we emphasize that in six out of the eight thermometers, the calibration shift at 4.2 K is less than 0.25%, which in terms of temperature change is ≈ 10 mK. This shift is significantly lower than that observed by Süßer [1] in the case of potted TVO thermometers.

Figure 3. Shift in (a) resistance and (b) calibration of the eight thermometers due to epoxy potting. The shift in X is defined as $X_{\text{shift}} = (X_{\text{after}} - X_{\text{before}})/X_{\text{before}}$, where X is the quantity plotted on the y-axis and the subscripts *after* and *before* respectively denote the state after and before potting.

d) Effect of 295 – 77 K thermal shocks

Figure 4 depicts the resistance shift and the calibration shift after two hundred thermal shocks from 295 K to 77 K (column E of Table 1). The shift represents the difference between the resistance values after and before the thermal shocks, *i.e.*, the difference between the shocked and the un-shocked potted thermometers. The resistance shift at 4.2 K is less than 0.15%, which is ≈ 6 mK. At 77 K, the resistance shift is within 0.2%. This resistance shift translates to ≈ 0.15 K at 77 K. We note that these calibration shifts compare well with those documented for a CernoxTM 1050-SD thermometer that was subjected to two hundred 305 K to 77 K thermal shocks [2]. The shifts reported in [2] are 3 mK at 4.2 K and 46 mK at 77 K. We also note that, especially at 4.2 K, the calibration shift due to the thermal shocks is much less than that initially caused by potting.

Figure 4. Shift in (a) resistance and (b) calibration of the potted thermometers due to 295-77 K thermal shocks. The shift in X is defined as $X_{\text{shift}} = (X_{\text{after}} - X_{\text{before}})/X_{\text{before}}$, where X is the quantity plotted on the y-axis and the subscripts *after* and *before* respectively denote the state after and before thermal shocking.

5. Summary

We developed a simple technique to encapsulate surface mount CernoxTM thermometers in Stycast epoxy. The potting produced a robust thermometer unit without significantly affecting the calibration. Between 77 K and 4.2 K, the calibration shift due to potting is within 0.5% even after several thermal cycling between 4.2 K and 295 K. Additionally, the potted thermometers are both thermally and mechanically stable to repeated thermal shocks from 295 K to 77 K. This paper thus offers a convenient technique to produce thermometers units usable in heavy duty applications where very accurate temperature measurements are not required.

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