CHARACTERIZATION STUDIES OF SPUTTER DEPOSITED CERMET CRYOGENIC HEATERS

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Characterization studies of a new cryogenic heater will be presented. The material is a sputter-deposited ceramic-metal composition. It has a constant resistance to within 0.1% between 77 K and 50 mK. At liquid helium temperature, the reduced sensitivity is 2×10^{-3}. The nominal die thickness is 837 nm. The sheet resistance is 7.27 Ω/square and the room temperature resistivity is 615 μΩ·cm. The die resistance is 1000 Ω. The die size is 1.02 mm by 0.76 mm. We will show results for maximum operating power, operational life, and reproducibility due to thermal cycling. Critical current densities will be presented.

INTRODUCTION

At present, cryogenic heaters are made from winding wire alloys that have a low temperature coefficient of resistivity (Evanohm, phosphor-bronze, and nichrome)[1,2]. Heaters are wound from long lengths of small gauge wire, making for a tedious process and a relatively large heater bobbin.

Techniques used to develop the Cernox [3] sensor can be applied to develop a sputter-deposited thin-film material with an extremely low temperature coefficient of resistivity. This method allows for controlling the resistivity through die design and meander patterns. Additionally, dies can be mounted into packages used for cryogenic RTDs. These devices could be used for applications requiring a fixed resistance. This includes cryogenic heaters and fixed resistance standards.

DESCRIPTION

The new material is a proprietary ceramic-metal composition (cermet). It is reactively sputter-deposited onto a sapphire wafer. The nominal thickness is 837 nm. The sheet resistance is 7.27 Ω/square and the room temperature resistivity is 615 μΩ·cm. The electrodes were patterned to make an individual die with a target resistance of 1000 Ω, and the line width was approximately 0.0063 cm [4].

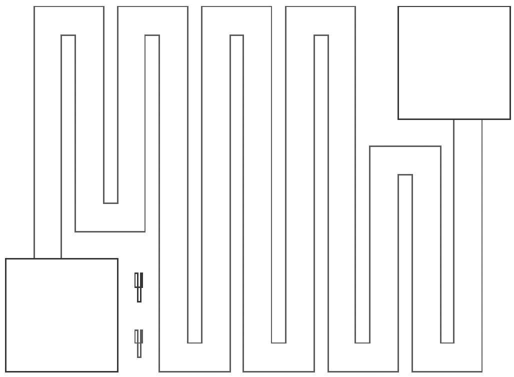

Figure 1 . Serpentine Meander Pattern for Novel Resistors.

Test resistors were fabricated from each wafer using standard photolithography techniques to define the active sensor area and subsequent contacts. The sensors were patterned in a serpentine meander. Figure 1 shows a finished sensor chip layout. The die size was 1.02 mm by 0.76 mm.

A total of nine devices were tested in a package. Three were from an original test and six additional new devices were built. The individual chips were packaged into Lake Shore standard canister packages, which are gold-plated copper canisters 3.048 mm in diameter and 8.5 mm long. The device is mounted inside the canister using a sapphire header. The device is connected by gold leads and is sealed into the canister with Stycast 2850 epoxy.

After packaging, the devices are thermally cycled from room temperature to liquid nitrogen temperature 200 times. In addition to the packaged devices, two bare dies were tested for maximum power load.

TESTING

Temperature calibrations from 0.05 K to 325 K were performed in the commercial calibration facility of Lake Shore Cryotronics. Temperature and resistance was measured using standards grade platinum and germanium thermometers in conjunction with a Keithley Model 224 current source, Hewlett Packard Model 3458A DVM, and Guildline Model 9330 standard resistors (resistance values from 10 Ω to 1 MΩ 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 0.05 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. Calibrations from 0.05 K to 1.4 K were performed in a dilution refrigerator using a Lake Shore Model 370 AC Resistance Bridge. The nominal excitation was 100 μV.

To determine their reproducibility and stability due to thermal stress, the packaged devices were thermally cycled and measured at three temperatures: 4.2 K, 77 K, and 305 K. The 4.2 K and 77 K readings were taken in an open dewar of liquid helium and liquid nitrogen. The 305 K reading was taken in a temperature-controlled oven. Readings at liquid helium were repeated 10 times for each of the nine packaged sensors.

RESULTS

Figure 2 shows the resistance as a function of temperature for three select devices. At room temperature, the devices range from 905 Ω to 925 Ω. From 305 K to 4.2 K their resistance changes by 2.5%, with most of this change appearing from 77 K to 305 K. From 77 K to 4.2 K the resistance changes less than 0.1%.

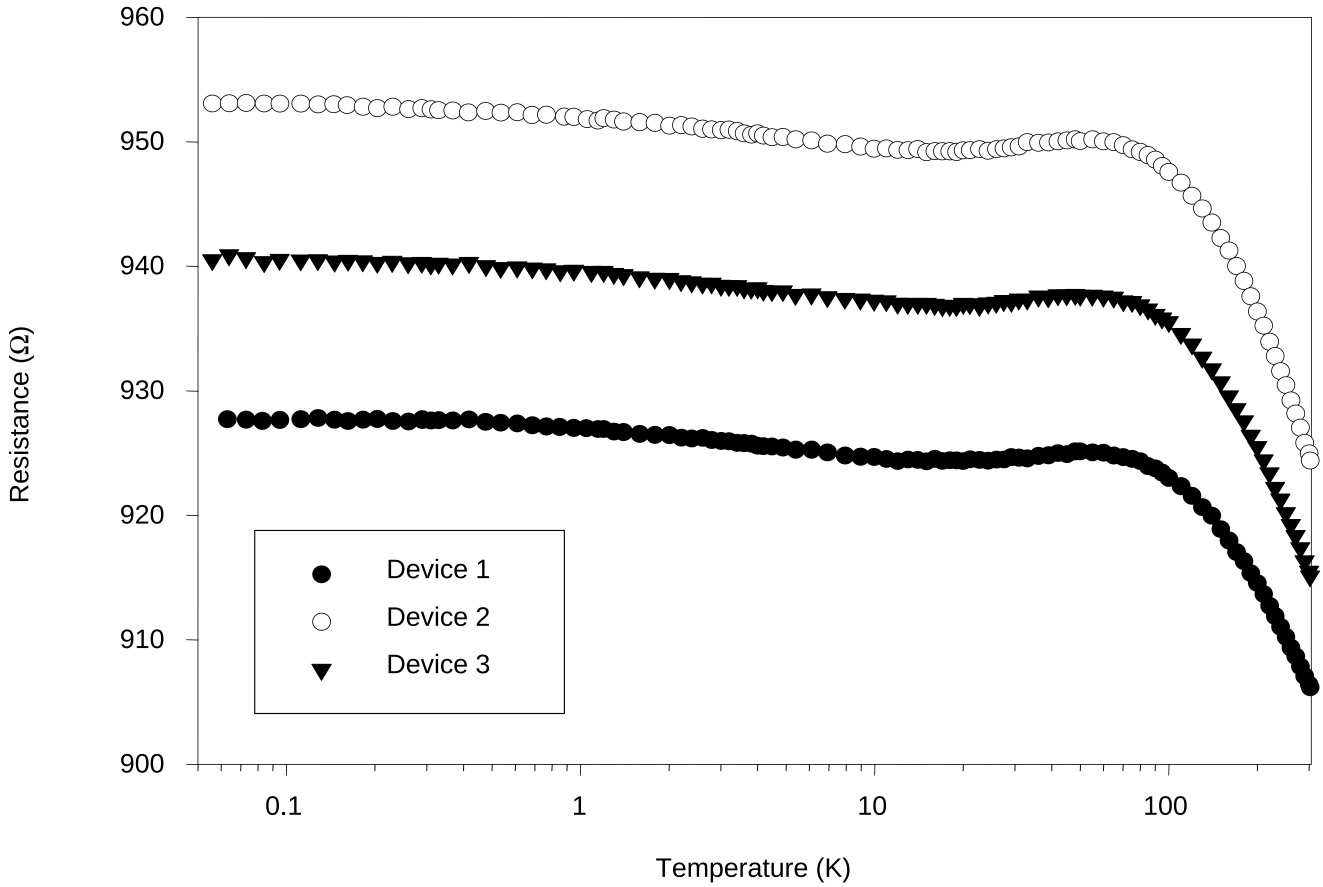

Figure 2. Resistance as a function of temperature for three selected heater devices.

The temperature coefficient of resistivity, (1/R)(dR/dT), at 4.2 K is approximately 2×10^{-3} K^{-1}.

Results from the thermal reproducibility tests are summarized in Table 1. Over the course of 10 thermal cycles into an open dewar of liquid helium, the standard deviation for all the devices was ±0.0195 Ω. Using an average resistance of 936.8 Ω, this is a reproducibility at 4.2 K of approximately 20 ppm in resistance. It should be noted that results were taken in an open dewar and the temperature was assumed to be constant during the duration of testing. For these tests, there was no attempt to correct for temperature drifts.

Table 1. Reproducibility results at 4.2 K. Results at 4.2 K based on 10 thermal cycles.
All devices come from the same wafer. Devices A – C were packaged 9 months prior to Devices 1 – 6.

	Resistance at 305 K	**Resistance at 77 K**	**Average Resistance at 4.2 K (Ω)**	**Standard Deviation at 4.2 K (Ω)**
DEVICE A	905.8	924.244	925.318	±0.0575
Device B	914.819	936.839	937.955	±0.0181
Device C	924.339	949.381	950.483	±0.0487
Device 1	924.377	946.268	947.132	±0.0133
Device 2	932.653	954.435	955.366	±0.0079
Device 3	926.201	948.925	949.880	±0.0083
Device 4	885.541	905.081	905.818	±0.008
Device 5	898.665	921.018	921.010	±0.0065
Device 6	914.762	938.346	938.339	±0.0068

Also seen in Table 1 is that the change in resistance for 77 K to 4.2 K is, on average, better than 0.1% in resistance. This confirms earlier results.

To test maximum power we used both the packaged devices and bare die devices. Two packaged devices were tested with a Keithley 220 current source and HP voltmeter. Power was incremented from 1 mA up to 100 mA to determine the maximum current. All four devices tested could sustain a constant current of 70 mA. This corresponded to approximately 4.5 W. This same device was tested in LN2 to provide a thermal shock while at maximum power. There were no damaging effects due to the thermal shock. When the current was increased to 100 mA (9 W) the device burned out and failed. This was duplicated on both packaged devices.

To confirm the failure was due to the thin film resistor and not some other component of the package, we tested bare dies to maximum power. A strip of 10 dies was cut from the original wafer. The dies were left on the cutting tape and placed in a 4-point probe station. Using a Keithley 220 current source and HP voltmeter, current was increased from 1 mA to 100 mA. Again, the devices could sustain a constant current of 70 mA without damage. At 100 mA the die under test flashed as arcing developed across the traces.

To estimate the critical current density we will use the value of 70 mA, based on a cross-sectional area of 5.3×10^{-11} m^2. This gives an average current density of 1.3×10^9 A/ m^2. The actual current density of the cermet film may be higher. The geometry of the meander pattern had sharp corners where there could be excessive current build up. In this case, the effective cross-sectional area would be smaller. To explore this further we will study different patterns.

APPLICATIONS

The results indicate these devices can be packaged as cryogenic heaters with power ratings up to 5 W. Increasing the film thickness will result in larger current densities and a larger power rating. Long term rating at maximum power still needs to be tested and alternate meander patterns will be explored.

The new cermet thin films offer many advantages over conventional techniques for cryogenic heaters. Because of the fabrication techniques, the new cermet thin-film resistors can be packaged similar to cryogenic RTDs and also as very small bare die heaters. Standard packaging allows greater flexibility in attaching and mounting the devices. A bare chip would allow the heater to be used for applications that would be impossible for wire-wrapped heaters.

Additionally, different resistances could be designed by changing the electrode mask, using a laser trimmed meander pattern, or changing the film thickness. Changing the film thickness can also change the effective power rating. The canister configuration is not ideal for a cryogenic heater and a simpler package will need to be used to best transfer heat out from the resistive element.

Another application would include a small moderately powered heater used by cryostat developers for temperature control over a wider range. The resistance is nearly constant over the whole range for easier power calculation based on room T values. Small bare die device heaters can be used in precision thermal properties studies.

CONCLUSIONS

A novel cermet material has been presented that shows a very low temperature dependence of resistivity. The temperature coefficient of resistance at 4.2 K is 2×10^{-3} K^{-1}. The material is very stable under multiple thermal cycles. The reproducibility at 4.2 K is 20 ppm in resistance or better.

This material can be packaged into standard cryogenic sensor packages and is useful as a small cryogenic heater for power rating up to 5 W.

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