FABRICATION OF REFERENCE STANDARD 1 OHM RESISTORS FROM EVANOHM S ALLOY

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Abstract

Reference standard 1 Ω resistors are now being fabricated that have superior characteristics to the Thomas type resistor.

A coil of Evanohm S wire is fabricated into a four terminal resistor. A program of heat soaking at temperatures below 500 °C for varying periods brings the first order temperature coefficient and the resistance correction at room temperature close to zero. Resistance adjustments are made using an electrolytic etching technique so that the required specifications are achieved. The heat treatment program also achieves a low initial drift rate.

The finished coil is fitted into a mount incorporating three hinges that support the coil without detracting from its performance as a resistor.

Introduction

A reference standard resistor should have a temperature coefficient of zero at room temperature and be close to nominal in resistance value.

The value of resistance R as a function of temperature T can be expressed by the equation:–

$$R = R_{22\cdot5}\,\{1 + \alpha\,(T - 22.5) + \beta\,(T - 22.5)^2\}$$

where $R_{22\cdot5}$ is the resistance, and α and β are coefficients at 22.5 °C.

The most widely accepted 1Ω standard resistor is the Thomas type resistor. It is made from Manganin wire for which dR/dT is zero when T is near 25 °C. This property is a function of the alloy, and β is typically –0.5 ppm/(K)^2. The NML 1Ω resistors are made from Evanohm S for which the temperature at which dR/dT is zero can be moved by heat treatment. For this alloy β is typically –0.003 ppm/(K)^2.

The new resistors have the following characteristics:

1. A value of d^2R/dT^2 that is much smaller than for Manganin resistors and a dR/dT that is zero near 22.5 °C. Thus the value of resistance is much less dependent on temperature than for Thomas type standard resistors.
2. Low long term drift rate.
3. Low thermal hysteresis of resistance.
4. Excellent transportability.

Fabrication

The resistive element consists of a coil of Evanohm S wire 2.1 mm in diameter, bifilar wound to minimise inductive effects. Potential connections are made by welding pieces of Evanohm wire to predetermined positions to produce a four terminal resistor that is self supporting. The coil is annealed in a furnace at 980 °C for 15 minutes and quenched in oil. The coil is soft at this temperature and so is supported by three serrated stainless steel clamps. The positioning of these clamps and the pitch of the serrations determines the geometry of the coil which must be regular to enable the coil to be mounted with minimal strain. After the anneal no mechanical work is done on the coil which might impart strain.

Before anneal α is about +10 ppm/K. To produce a resistor that has a value near nominal when $\alpha = 0$ the potential terminals are placed so that R is about 0.980 Ω. After anneal R is about 0.94 Ω and α is about +36 ppm/K. The changes in R and α during anneal are large and can vary by up to 10% of these values. The resistors are therefore made so that if no adjustment were made their final resistance value would be low by typically 3000 ppm.

The remainder of the coil production schedule involves heating the coil at various temperatures, commencing at 470 °C, for varying periods of time and measuring the resistance of the coil after each period. The coil at temperatures below 500 °C is self

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supporting and so in order to measure the four terminal resistance the coil is mounted on an adjustable jig. The resistance measurements are made in oil at room temperature.

The heat treatment increases the value of resistance while decreasing the value of α. For values of α within 5 ppm/K of 0, $^{\Delta R}/_{\Delta\alpha}$ is about $-1550\ \Omega$ K. As the heat treatment progresses and the final value of α is approached the resistance is adjusted by an electrolytic etching process. During etching the value of $^{\Delta R}/_{\Delta\alpha}$ is an order greater than for heat treatment, allowing both R and α to be brought to the required values.

A program of heat treatment temperatures and times brings the value of α down to zero and also brings the drift rate down to a low value. The heat treatment program involves heating the coil at a particular temperature until a steady value of $^{\Delta R}/_{\Delta t}$ is obtained, where Δt is the incremental period during which the coil was at the raised temperature. Each temperature has a corresponding steady state drift rate and this steady state drift rate is lower at lower temperatures. At 400 °C the steady state drift rate is 400 $\mu\Omega$/hour while at 200 °C the rate is typically 0.005 $\mu\Omega$/hour. The final temperature of heat treatment before mounting is 100 °C and the period of treatment at this temperature is several hundred hours.

Mounting

The heat treated coil is mounted in a support which consists of three carriers held by hinges that are injection moulded from polypropylene. Each carrier consists of two brass strips, between which is a pair of polyethylene liners which are shaped to be a clearance fit to each turn of the coil. Contact to the coil is made by diaphrams of silicone rubber which is injected between the pairs of polyethylene liners. The hinges are fixed to an inner cylinder. The mount only allows for radial expansion and contraction but resists other modes of movement.

The completed resistor is then heat soaked in an oven at 60 °C for 150 hours before characterisation measurements are commenced.

Ackowledgements

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References

[1] "New Alloy has Improved Electrical and Resistance Properties", Materials and Methods, Vol. 28, p. 62, August 1948.

[2] A.H.M. Arnold, "Nickel–Chromium–Aluminium–Copper Resistance Wire", Proc. IEE, Vol. 103B, pp. 439–447, July 1956.

[3] C. Dean Starr and T.P. Wang, "The Electrical Characteristics of a Nickel–Chromium–Aluminium–Copper Resistance Wire", Proc. IEE, Vol. 104B, pp. 515–518, September 1957.

[4] C. Dean Starr, T.P. Wang and K.A. Hofacker, "Effect of Heat Treatment and Plastic Deformation on Electrical Properties of a Modified Nickel–Chromium Alloy", Proceedings of the American Society for Testing and Materials, Vol. 61, pp. 831–837, 1961.

[5] C. Dean Starr, "Properties of Wire for Resistors", Materials Research & Standards, Vol. 6, pp.435–440, September 1966.

CuGeMn, a New Material for High Precision Cryo-Resistors

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Abstract

The electrical resistance of CuGeMn alloys was measured as a function of temperature. It shows maxima at temperatures between 1,5 K and 9,1 K. Since the temperature coefficient at the maxima is zero and due to the high value of resistivity these alloys are very suitable for the construction of cryo-resistors, which are operated in liquid helium and can be used to improve quantum Hall measurement systems.

Introduction

For the comparison of resistances with very low uncertainties cryogenic methods are in use. For instance, the comparison of a quantized Hall resistance of about 6,45 kΩ or 12,9 kΩ with an 1 Ω resistance standard can be done by means of a cryogenic current comparator. Often, this measurement is done in two steps with an auxiliary resistance of 100 Ω [1,2,3]. One serious limitation of the obtained uncertainty is the thermal noise of this auxiliary resistance, which is 1,3 nV/√Hz at room temperature. If a cryo-resistor at 4 K could be used for this purpose, its thermal noise will be of the same order as that of the 1 Ω resistance standard at room temperature, which is only 0,13 nV/√Hz.

In the past, cryo-resistors were reported with a low temperature coefficient, which had been constructed as properly designed series connections of two different materials with a positive and a negative temperature coefficient [4]. In order to avoid thermoelectric effects we looked for an alloy which should have an extremum in its $R(T)$-behaviour at temperatures in the liquid helium range. Possible candidates for this purpose are CuGeMn alloys with low concentrations of manganese. They are similiar to the commercially available alloy ZERANIN [5], which is used for resistors operating at room temperature. CuGeMn is a material with good mechanical properties which allow wire drawing and welding without difficulties. In order to investigate their properties six alloys with different concentration of manganese were fabricated at the Isabellenhütte [5], and we have hitherto investigated the temperature dependence of the resistivity of these alloys.

Experimental Results

All alloys had the same concentration of Ge (5,3 at%) leading to the same resisitivity at 4,2 K of about 17 μΩcm and a residual resistance ratio R(4,2K)/R(300K) of 0,89. The concentration of manganese varied between 0,12 and 0,39 at %. Samples were made from each alloy and their resistivity was measured between 1.5 K and 300 K. Qualitatively all six alloys show the same behaviour of its dependence on temperature. It increases at the lowest temperatures, passes the spin glass maximum, decreases to the Kondo minimum and increases afterwards nearly linear with temperature.

Whereas the Kondo minimum, which occurs at about 27 K, depends only weakly ($\propto c^{1/8}$) on the concentration c_{Mn} of manganese, the temperature T_{max} at which the maximum in the R(T)-dependence occurs, increases linearly with increasing concentration of manganese:

$$T_{max} = a \cdot c_{Mn}$$

where 26,1 K/at% is obtained experimentally for a.

Since the maxima of the $R(T)$-curves are rather sharp peaks, one has to control the temperature of the helium bath at T_{max} to better than ± 5 mK in order to keep relative resistance changes smaller than $3x10^{-8}$.

Discussion

The CuGeMn alloys under investigation have a high resistivity at 4,2 K and, depending on their concentration of manganese, a temperature between 1 K and 9 K can be chosen at which the temperature coefficient is zero. This is of great importance if a cryo-resistor of this alloy is to be used in the resistance comparison as mentioned before, since this auxiliary resistance is used with different power consumption. However, the maximum of the $R(T)$ curve is not very broad, which has the consequence that the temperature of the helium bath has to be controled precisely. The additional consequence of this sharp peaked maximum is, that the design of the cryo-resistors has to be in such a way that the self heating at different power consumptions does not exceed the limits which are determined by the desired resolution. If these boundary conditions are obeyed, we believe that cryo-resistors from these alloys are an improvement due to the obtainable accuracy of the measuring system. A 100 Ω cryo-resistor is presently constructed and will be integrated in our quantum Hall system.

References

[1] A. Hartland, R.G. Jones, and D.J. Legg,: An NPL re-determination of the quantized Hall resistance in SI units, Document CCE/88-9 submitted to the 18th meeting of the CCE.

[2] F. Delahaye, A.Satrapinsky, and T.J. Witt, "Recent determinations of R_H in terms of Ω_{BI-85}", IEEE Trans. Instrum. Meas., vol. IM-38, pp. 256-259.

[3] E. Braun, and D.Dominguez, "Quanten-Widerstandsnormal", PTB-Bericht, E-35 (1989), pp. 65-85.

CH2822-5/90/0000-0292 $01.00 © 1990 IEEE

[4] P. Warnecke, and E. Braun, "Improved Cryo-Resistors with Low Temperature Coefficients", IEEE Trans. Instrum. Meas., vol. IM-38, pp. 293-295.

[5] Isabellenhütte, Heusler GmbH KG, Dillenburg (FRG). ZERANIN is a registered trademark of Isabellenhütte.

THE ULTRA-ZENER ....
A PORTABLE REPLACEMENT FOR THE WESTON CELL ?

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Abstract

24 samples of the so-called Ultra-Zener, an integrated circuit containing a buried Zener junction, type no. LTZ 1000, have been tested since March 1988. With some circuit modification, half of the samples have been found to have an ageing rate of less than 1.5 ppm/year, and all have ageing less than 4 ppm/year.

Introduction

The so-called Ultra-Zener is an integrated circuit containing a buried Zener junction, a heater and two sensing transistors all in a small area of silicon. It has data sheets (type LTZ 1000) which promise very low noise and a very small change of voltage with time. The latter feature is about 4 times less than that achievable by certain other good reference Zeners.

There is always a demand for better accuracy, lower drift with time, a better temperature coefficient and resistance against thermal shock, and low source resistance from a reference element. The circuit suggested by the makers has been modified to reduce the controlled temperature at which the Zener junction operates and to reduce low frequency noise.

It is desired to test a group of 7V Zeners for noise at the 0.05 ppm level and voltage rate of change with time (ageing) at the 0.3 ppm/year level. This has needed a specially developed measuring system, whose readings are traceable to the National 10V level. It is equipped with a measure of "intelligence' to give confidence in the data produced.

Development of the Zener Circuit

The Ultra-Zener is assembled on a small circuit board in a slightly modified circuit, Fig.1, to that given in the makers data sheet. It is shown in the centre of the diagram with the heater resistor between pins 1 and 2, the Zener between pins 3 and 4, and the two npn transistors. The circuit to the right of the Zener together with the resistor R1 defines a constant Zener current in the region of 5mA. The circuit to the left of the Zener controls the temperature of the integrated circuit using the -2mV/$^{\circ}$C base-emitter temperature coefficient of the npn transistor as sensor. The choice of R4 and R5 as 12k and 1kohm define about 45°C for the chip. Temperatures defined by other resistor ratios are shown in Fig.2. R4 and R5 should be in one package to get best rejection of environment temperature. Together with R1, which defines the Zener current, and the op-amp type LT1013, premium quality tested components are wanted so as not to degrade the performance of the Zener.

Tests on Zener Ageing

Some investigation has started on the device ageing and how it varies firstly with Zener temperature, and secondly with the Zener either continuously powered or normally unpowered (except for 4 to 8 hours on each measurement day). Some results of these tests are shown in Fig.2.

All devices are run continuously at the start. I did not have the same value resistor pairs R4 and R5 for all of the samples tested so some devices were run cooler than others. The trend, shown dotted in Fig.2, is for the ageing doubling for each 10°C rise in temperature and this could be argued to be some sort of fit to the results.

Next some of the devices which were ageing at about 4 ppm/year were taken off continuous power and were only powered one day in ten when they are measured. Provided that they are turned on 4 hours before a measurement, negligible change of voltage over the next 4 to 8 hours is found. The much lower values for ageing are shown in Fig.2 for the devices operated like this.

The Measurement System

My computer-aided measurement system contains

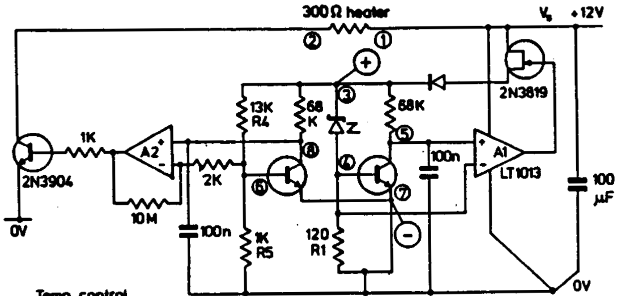

Fig.1. The Ultra-Zener circuit diagram. R1 is for current sensing; tolerance 0.01%, 1 ppm/$^{\circ}$C temperature coefficient R4:R5 = 13:1 for 65°C chip and matched to 3 ppm/$^{\circ}$C.

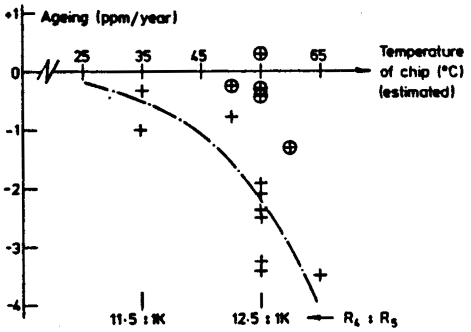

Fig.2: Ageing of 14 samples of LTZ 1000 at various chip temperatures: continuous operation except for devices denoted by ⊕ .

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at its heart a high grade voltmeter. It is simplistic to use this directly as this just compares the Zener under test with the reference in the instrument: if noise or a step change is seen, it is not certain which Zener has caused it. Instead, a known fraction of the voltage of a 10V standard is derived from resistive voltage dividers, and 7.14 or 7.0 V are available with dividers made of 7 or 10 resistors respectively. Now the voltmeter on its 0.1V range can be used to measure the difference between the Zener diode and the "divided-down standard" and it does so with a 0.1 μV resolution. The voltmeter needs very high input resistance to avoid loading errors. The 6½ decade voltmeter used for 10 years has just been changed to an 8½ decade type, whose lower noise and calibration errors, etc., should give smaller measurement uncertainty.

To ensure reliability and good measurement repeatability each divider must be regularly calibrated and all the 10V standards must be intercompared and assigned a value. This is what the rest of the system comprising a 16/way 4 pole scanner, a personal computer and a printer perform in a 3 step process using the IEEE-488 bus to interconnect the system.

Step 1. A group of eight 10V standards contain three portable units which are assigned a value traceable to the National standard of voltage about every 9 months. Four of the group of 10V standards, selected as being those of lowest noise, are intercompared continuously by the double-difference method. This allows any noise to be unambiguously traced to the unit causing it.

Step 2. Two separate resistive dividers across two of the 10V standards are calibrated at the start of every measurement session and if necessary during the session to give accurately defined 7V and 7.142V levels. The nominal output of the Ultra-Zener is about 7.1V.

Step 3. Lastly the voltage differences between each Zener reference and both calibrated dividers are taken 24 times in sets lasting about half a minute. From this is calculated a mean voltage for the Zener and a standard deviation of each reading set. The latter is typically 0.3 μV on the 7V level and contains contributions from the 10V standard, the divider and the Ultra-Zener.

The computer uses a cross-correlation program which is actually fed with the average voltage of the two dividers. After calculating the voltage of each Zener, the program calculates the mean of the group of Zeners being measured. Then the results of step 3 of the process are used in reverse to calculate back to the voltage of each divider tap. If these are not both correct to 0.5 μV, the measurement is repeated (or the DVM calibration is checked or the dividers recalibrated). This back check is an important component in giving confidence to the measured data.

Results

A line fit to the measurements will give an "ageing" rate, see Fig.3. More importantly, the errors from a constant voltage or a constant smooth change will indicate the suitability of the device to be used in a voltage standard. It is now held that no more than 1 ppm per year for the rate of change of voltage is desirable. Any errors from a smooth rate of change may of course be caused by uncertainties in the system or environment. A full uncertainty budget for the system has been determined as ±3 μV or just under 0.5 ppm at the 7V level. In the event, errors of about half of this are inferred from the scatter of points from the linear ageing trend, Fig.3.

The largest long-term error is caused by the step change every 9 months when the 10V group is traced in value to the National level at the National Physical Laboratory.

Conclusion

Initial tests indicate that the Ultra-Zener seems to have no serious disadvantages as a voltage standard apart from price and some added circuit complexity. This is a good omen for the performance of several new commercial instruments which contain it.

References

[1] B.F. Field, "A sub-ppm 1-10V-DC measuring system", IEEE Trans Instrum and Measurements, Vol. IM-34, No.2, pp.327-330.

[2] P.J. Spreadbury, "The Ultra-Zener", Proceedings of the British Electromagnetic Measurements Conference, National Physical Laboratory, 7-9 November 1989, pp.16.1-16.4.

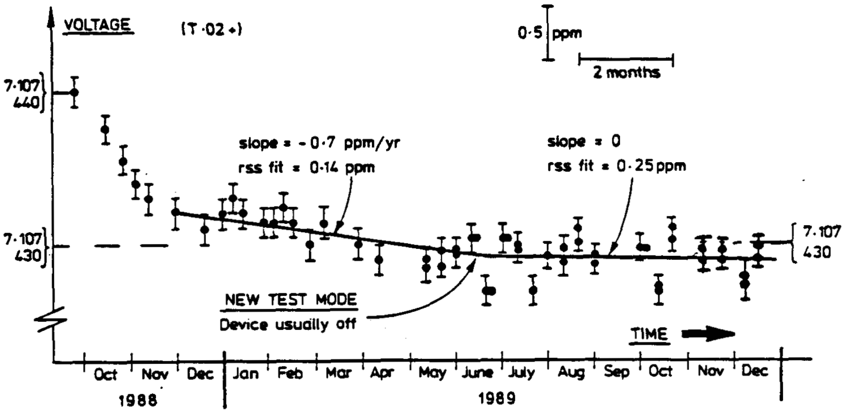

Fig.3. Measured voltage of one sample of the Ultra-Zener over a period of 14 months.