A MINIATURE PALLADIUM-IRON THERMOMETER FOR TEMPERATURES DOWN TO 0.05 KELVIN

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ABSTRACT

Magnetic thermometers are appealing at temperatures below about 0.1 Kelvin, because they avoid the noise self-heating problems associated with resistive thermometers. In p[illegible]etallic [illegible]tronic the[illegible]eters add[illegible]ta[illegible]ical stabi[illegible]ermal c[illegible]uc[illegible], and eas[illegible]eat sinkin[illegible]work w[illegible]scribe a palla[illegible]n-iro[illegible]ermon[illegible]r w[illegible] was de[illegible]o be sma[illegible]d convenient[illegible]ackaged and [illegible]mize[illegible] use a[illegible]mp[illegible]ures do[illegible]5 Kelvi[illegible]he device sh[illegible] Curie-Wei[illegible]ehav[illegible]above[illegible].06 Kelv[illegible]a[illegible]e achi[illegible]nK/√Hz [illegible]perature reso[illegible]n at [illegible]mpera[illegible] mK. [illegible]de[illegible]be the [illegible]d opera[illegible] of this therm[illegible]eter a[illegible]presen[illegible] tes[illegible]ults.

INT[illegible]ION

Low temperature research in the range of 50 mK is complicated by thermometry problems. Traditional resistive thermometers self-heat in that temperature range due to rf noise currents if the circuits are not properly shielded and filtered. Since the same resistive thermometer can give different readings in different test setups, even correctly calibrated thermometers cannot be trusted.

For many applications magnetic thermometers are an appealing alternative. They generally operate by measuring the temperature-dependent magnetic susceptibility of a paramagnetic material. They avoid the self-heating problem, and they can be read out with a SQUID to achieve high resolution. Commonly used paramagnetic salts are chemically unstable and have low thermal conductivity. Since it is difficult to make good thermal contact to them, the packaging can be a problem. Metallic dilute electronic magnetic thermometers avoid these problems and are more convenient to use

One example is the palladium-iron thermometer. In palladium the iron atoms polarize their palladium neighbors, resulting in giant magnetic moments of about 10 μ_B per iron

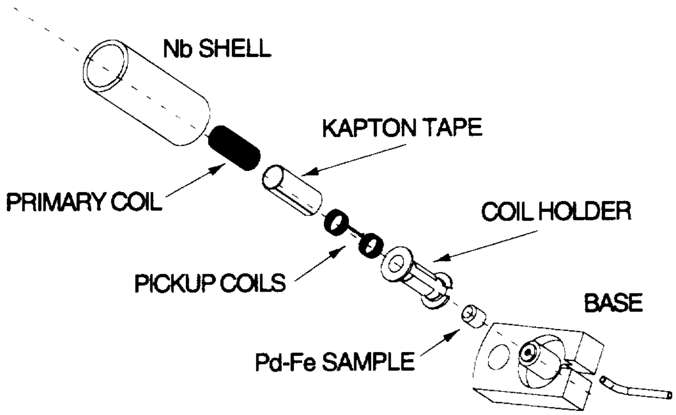

FIGURE 1. An exploded view of the Pd-Fe thermometer assembly. The tinned stainless steel tube for shielding the secondary circuit leads is shown near the base. The parts are shown to scale, and the Nb shell is 1.28 cm tall.

atom. The Pd-Fe system has been shown to exhibit spin-glass behavior at low temperatures, having a magnetic susceptibility maximum at the freezing temperature[1,2]. This temperature depends on the iron content, and above it the susceptibility obeys a Curie-Weiss law. With its large magnetic moment, such a device is quite sensitive over a wide temperature range and provides extremely high resolution near the low end of this range. It has a short thermal time constant and can is easy to thermally attach to a test stage.

Pd-Fe thermometers have been used with different iron concentrations in different temperature ranges[3,4]. The typical iron impurity level of 10 – 20 parts per million (atomic) found in "pure" palladium samples is suitable for a thermometer sensitive down to a fe[illegible]vin. Ho[illegible]es not add[illegible]h to the co[illegible]iu[illegible] the iron [illegible]ing [illegible] desir[illegible]ve[illegible]he goal [illegible]s work w[illegible]o build and t[illegible]a Pd-Fe therm[illegible]eter [illegible] was [illegible]ntia[illegible]useful f[illegible]olling th[illegible]mperature of [illegible]e-flight dete[illegible]stage[illegible]t nee[illegible]mall an[illegible]iently pa[illegible]ed, and it ne[illegible] a high resol[illegible]n at te[illegible]ratur[illegible] mK

DESIGN

The palladium sample used in this thermometer was machined from an 8 mm diameter rod purchased from Strem Chemicals. It was tested using induction coupled plasma analysis to determine its concentration of iron and other magnetic contaminants. The iron content was 185 atomic parts per million (ppm). This was fortuitous; the sample had a higher spin-glass freezing temperature than typical "pure" palladium, and thus a higher magnetic susceptibility in the desired temperature range. It contained less than 20 ppm chromium, and the cobalt, manganese and nickel concentrations were well below 1 ppm.

The thermometer's mechanical design is shown in FIGURE 1. The parts were machined and assembled in house at Goddard Space Flight Center (GSFC). The palladium sample sits on a copper base to which it is varnished. The coil form, machined from vespel SP-1, slides down over the sample and is also varnished to the base. A niobium shell surrounds the coils and sample. This shell is varnished onto the base, which is bolted down with a #2 screw.

All of the wire used for both the primary and secondary windings is formvar-insulated copper-clad Nb-Ti with a total diameter of 63.5 μm. The two pickup coils in the secondary

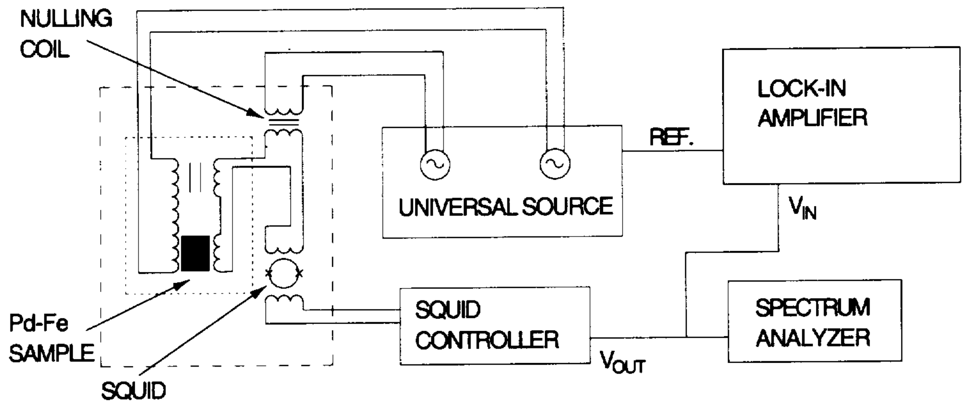

FIGURE 2. This figure will eventually be a schematic of the thermometer readout electronics, etc. This picture is just a place holder.

circuit are wound directly onto the coil form in its circumferential slots. One of these coils contains the palladium sample, and the other surrounds an empty volume. Each pickup coil has fifteen turns, and the coils are wound in opposite directions in order to cancel out the background signal. Each pickup coil has an inductance of 1 µH in order to match the A layer of 50 µm thick kapton tape is wrapped over these coils. The primary coil, consisting of five layers with 160 turns per layer, is wound onto the kapton. Its inductance is 100 µH.

To achieve the highest possible temperature resolution, the Pd-Fe thermometer must be read out using a SQUID. In order to make a robust device that wasn't vulnerable to flux jumps, a four-wire AC susceptibility measurement scheme was selected. The wiring is show[illegible]ically in [illegible]2. The two [illegible]ter-wound[illegible]ils [illegible]dary circu[illegible] direct[illegible]QUID in[illegible]. In orde[illegible]ondary curre[illegible]or a [illegible]n exci[illegible]on c[illegible]nt, the p[illegible]coils' indu[illegible]ces are 1 µH[illegible] half the valu[illegible] the S[illegible]D's i[illegible]tor[illegible] nulling [illegible]ncluded i[illegible]s circuit to ca[illegible] out the pick[illegible]oil m[illegible]atch a[illegible]w nullin[illegible]ignal fo[illegible]solution [illegible]dy-state therm[illegible]etry. [illegible]e seco[illegible]uit is en[illegible]ly i[illegible]e a sup[illegible]ing shiel[illegible]nsisting of th[illegible]biu[illegible]s sur[illegible]di[illegible]e therm[illegible]l the S[illegible]D, a lead foil [illegible] around the S[illegible]ID a[illegible]bly, a[illegible] lea[illegible]n sold[illegible]inless [illegible] tube connect[illegible]the two. This [illegible]ngemen[illegible]s c[illegible]n to [illegible]w the [illegible]UID t[illegible] positioned o[illegible] Kelvin bath [illegible]out cond[illegible]ng [illegible]uc[illegible]eat to th[illegible]w te[illegible]ature thermo[illegible]y stage. The tubing extends up inside the thermometer's niobium shell, and it was heat sunk well near the thermometer to avoid large heat loads on the device.

EXPERIMENT

Experimental Setup

The thermometer was bolted to the cold stage of a laboratory adiabatic demagnetization refrigerator (ADR). The stage was temperature controlled using a ruthenium oxide thermometer read out with a Linear Research LR-700 bridge. The actual temperature was read from a germanium resistance thermometer (GRT) calibrated by Lakeshore Cryotronics down to 0.048 Kelvin. This thermometer was read out using a RV-Elektroniikka Oy Picowatt AVS-47 resistance bridge.

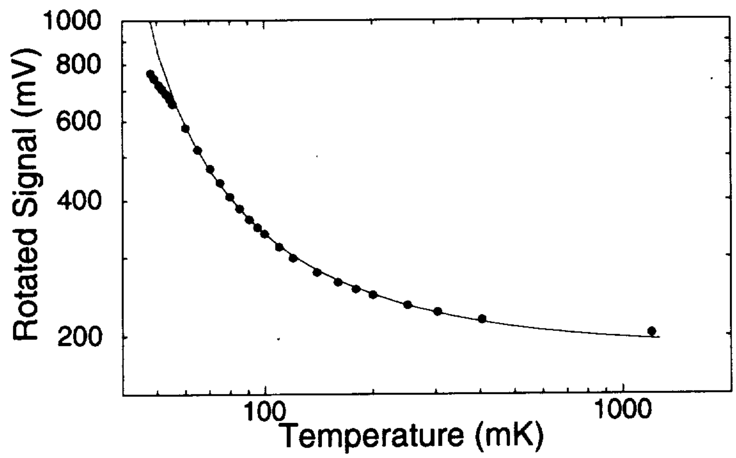

FIGURE 3. The Pd-Fe thermometer signal vs. temperature. The signal and quadrature have been rotated in phase by +22.6 degrees, resulting in a nearly temperature-independent quadrature. The solid line is a Curie-Weiss Law fit to the data above 60 mK.

In it's temperature controlling mode the ADR generates a magnetic field of up to 6 gauss at the palladium thermometer's location, but the thermometer's niobium shell was superconducting before the ADR magnet was ever operated. In this test setup four layers each of 50 μm thick vanadium permendur and 0.25 mm thick AD-mu-80 ferromagnetic shielding were wrapped around the 13 cm diameter can surrounding the cold stage to reduce the earth's magnetic field at the thermometer.

The excitation current was provided by an HP Universal Source. Its two channels allowed independent excitation of the thermometer and nulling coil primaries with an adjus[illegible]se angle. [illegible]um Design [illegible]ID was us[illegible] ou[illegible]dary circu[illegible] The m[illegible]QUID co[illegible]ler's outpu[illegible] b[illegible]k-in ampl[illegible] and [illegible]pectru[illegible]aly[illegible] The SQ[illegible]ontroller [illegible] used with a 1[illegible]Hz two-pole [illegible]erwo[illegible]filter o[illegible]e o[illegible]t signal. [illegible]lter has a[illegible]tively flat am[illegible]ude pass band[illegible]o abo[illegible]0 Hz, [illegible]lts in sig[illegible]ca[illegible]hase shi[illegible] low fre[illegible]cies. In all o[illegible] testi[illegible]escrib[illegible]work, th[illegible]ck-[illegible]mplifier [illegible]ngle wa[illegible] at zero, so th[illegible]nal a[illegible]uadra[illegible]re[illegible]the filt[illegible]ift.

Curi[illegible]aw Test

The first testing of the Pd-Fe thermometer was to determine the temperature dependence over the entire temperature range. For these measurements the excitation current was a 19 Hz sine wave of 5 x 10^{-4} amps rms. This frequency was about as low as low as possible while still providing a bandwidth adequate for controlling nearly any cryogenic stage. The SQUID's sensitivity was set to its lowest value, 0.01 volts per Φ_0, where Φ_0 is one flux quantum. This configuration gave enough dynamic range to sweep through the entire temperature range of interest. The lock-in amplifier's phase angle was set to zero, and the in-phase signal and quadrature were measured as a function of temperature from 48.1 mK up to 1.2 Kelvin. Both the showed a strong temperature dependence, varying by 520 mV and 216 mV respectively over this range. The quadrature was plotted against the signal and found to be very linear, indicating a phase angle of –22.6 degrees for the signal's temperature dependent part. When the data were rotated in phase by +22.6 degrees, the resulting quadrature points were nearly temperature independent, varying by only 6 mV.

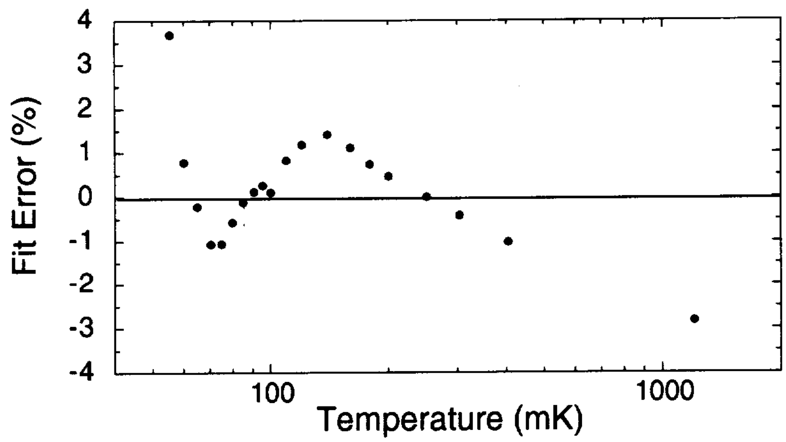

FIGURE 4. The percent error in the Curie-Weiss Law fit to the data. The values at temperatures below 55 mK are not shown.

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As in the Curie law test, both the signal and quadrature showed a temperature dependence. Again the data were rotated in phase space, this time by 56.4 degrees, to minimize the quadrature's temperature dependence. The resulting in-phase signal data are shown in FIGURE 5. The rotated quadrature changed by only 1.6 mV over this range, while the rotated signal varied by 600 mV. The solid line in the figure is a linear fit to the data, indicating a sensitivity of 369.5 μV/μK. The signal noise floor was 15 μV/√Hz near the excitation frequency, so the temperature resolution was 41 nK/√Hz. The SQUID's noise floor is about 4 μ Φ_0/√Hz, or about a factor of 40 lower than the measured signal.

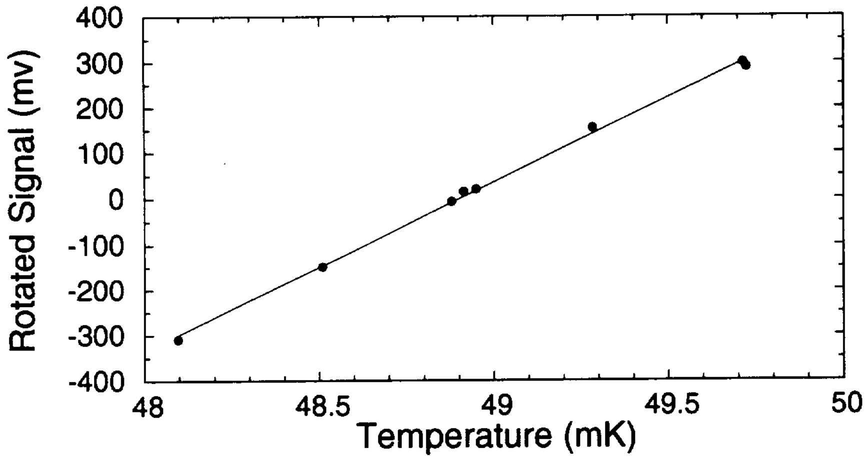

FIGURE 5. The Pd-Fe thermometer signal vs. temperature. The signal and quadrature have been nulled at a temperature of 50 mK, then rotated in phase by +56.4 degrees, resulting in a nearly temperature-independent quadrature. The solid line is a linear fit to the data.

ANALYSIS

The change in slope of the signal vs. temperature plot is not yet understood. It truly appears to be a transition to a constant slope on a log-log plot, rather than rolling over toward a constant value or a maximum. It is possible that the approach of the spin-glass freezing temperature is causing this effect. The fit of the data above 60 mK, where it does obey a Curie-Weiss law, indicates a freezing temperature closer to 36 mK. Future studies using [illegible]ive thermo[illegible]librated to lo[illegible] temperatu[illegible]lut[illegible]tor) woul[illegible]e light o[illegible]

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A third possibility is a calibration problem with the standard GRT. It is conceivable that the low temperature end of the calibration curve has changed, or that the original calibration had some problem. Since this thermometer has only been used as a primary standard, such a calibration anomaly would not have been noticed. In the future it will be checked against another calibrated standard resistor, possibly in a dilution refrigerator. This uncertainty points out one of the important motivations for developing the Pd-Fe thermometer in the first place.

Another area for improvement is in the thermometer signal's noise floor. With the large thermal mass of the ADR's salt pill, there should be no significant noise spectral density at the operating temperature of 47 Hz. Thus the noise floor must be due to thermometer readout noise rather than actual temperature noise. One possible source of disturbance is Johnson noise currents in the resistive palladium. However, calculations of the Johnson noise pickup indicate that it would be well below the existing noise floor. Most likely the situation could be improved by enhancing the shielding and filtering and by eliminating microphonics in the wiring in the presence of the earth's field and the ADR's fringing field. One of the goals of this work was to demonstrate a device that was convenient to use, so noise reduction efforts were modest in the early testing. However, more efforts will be made to limit the noise in the future.

CONCLUSIONS

The work so far has produced a Curie-Weiss law thermometer down to about 60 mK. Efforts will be made to improve the heat sinking of leads to see if the roll-off below this temperature disappears. In addition the device might be tested in a dilution refrigerator with a resistive thermometer calibrated down to lower temperatures. This would eliminate the possible effect of residual magnetic fields from the ADR and allow a characterization of the spin glass transition. The steady state control mode took advantage of the thermometer's high sensitivity. Attempts could be made to reduce the circuit noise to a value closer to the SQUID's noise floor (better filtering, magnetic shielding, and vibration reduction), but the device as-is would provide significantly better resolution than traditional resistive devices. Work is currently proceeding at GSFC to demonstrate a version of this thermometer deposited directly onto a chip.

ACKNOWLEDGEMENTS

The authors would like to thank Mike Jackson for his help with the Pd-Fe ther[illegible]design. [illegible]k was sup[illegible]ed by Go[illegible]e [illegible]er's Dire[illegible]tionary[illegible].

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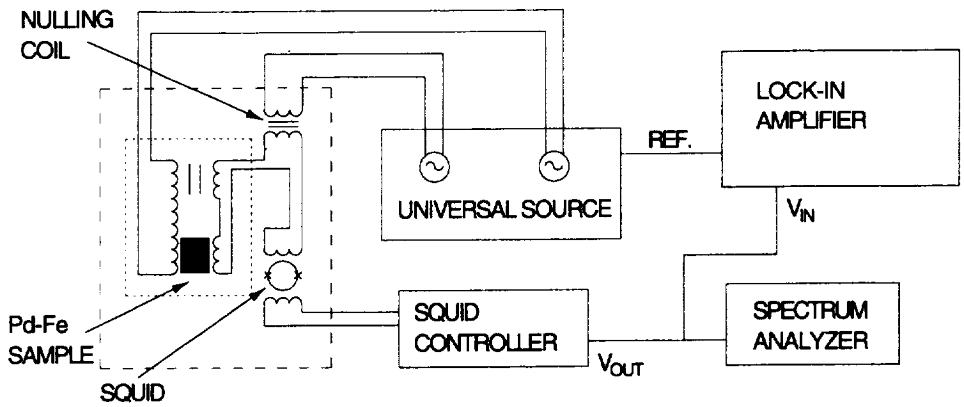

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EXPERIMENT

Experimental Setup

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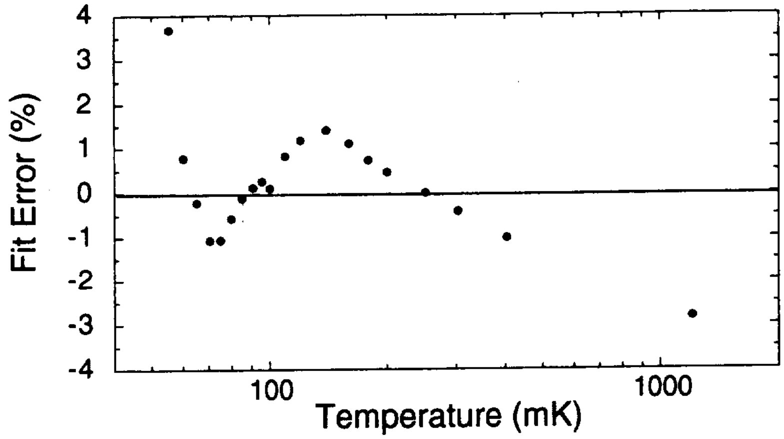

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As in the Curie law test, both the signal and quadrature showed a temperature dependence. Again the data were rotated in phase space, this time by 56.4 degrees, to minimize the quadrature's temperature dependence. The resulting in-phase signal data are shown in FIGURE 5. The rotated quadrature changed by only 1.6 mV over this range, while the rotated signal varied by 600 mV. The solid line in the figure is a linear fit to the data, indicating a sensitivity of 369.5 μV/μK. The signal noise floor was 15 μV/√Hz near the excitation frequency, so the temperature resolution was 41 nK/√Hz. The SQUID's noise floor is about 4 μ Φ_0/√Hz, or about a factor of 40 lower than the measured signal.

the Johnson noise pickup indicate that it would be well below the existing noise floor. Most likely the situation could be improved by enhancing the shielding and filtering and by eliminating microphonics in the wiring in the presence of the earth's field and the ADR's fringing field. One of the goals of this work was to demonstrate a device that was convenient to use, so noise reduction efforts were modest in the early testing. However, more efforts will be made to limit the noise in the future.

CONCLUSIONS

The work so far has produced a Curie-Weiss law thermometer down to about 60 mK. Efforts will be made to improve the heat sinking of leads to see if the roll-off below this temperature disappears. In addition the device might be tested in a dilution refrigerator with a resistive thermometer calibrated down to lower temperatures. This would eliminate the possible effect of residual magnetic fields from the ADR and allow a characterization of the spin glass transition. The steady state control mode took advantage of the thermometer's high sensitivity. Attempts could be made to reduce the circuit noise to a value closer to the SQUID's noise floor (better filtering, magnetic shielding, and vibration reduction), but the device as-is would provide significantly better resolution than traditional resistive devices. Work is currently proceeding at GSFC to demonstrate a version of this thermometer deposited directly onto a chip.

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