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PdMn and PdFe: New Materials for Temperature Measurement Near 2 K

B. J. Klemme,[1] M. J. Adriaans,[2] P. K. Day,[2] D. A. Sergatskov,[1] T. L. Aselage,[3] and R. V. Duncan[1]

[1] *Department of Physics and Astronomy, University of New Mexico Albuquerque, New Mexico 87131-1156, USA*
[2] *Jet Propulsion Laboratory, Pasadena, California 91109-8099, USA*
[3] *Sandia National Laboratories, Albuquerque, New Mexico 87185-1421, USA*

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Interest in the critical dynamics of superfluid ^{4}He in microgravity conditions has motivated the development of new high resolution thermometry technology for use in space experiments near 2 K. We have developed a magnetic thermometer using dilute magnetic alloys of Mn or Fe dissolved in a pure Pd matrix, similar to previous thermometers used at ultra-low temperatures. These metallic thermometers are easy to fabricate, chemically inert, and can have a low thermal resistance to the stage to be measured. Also, the Curie temperature can be varied by changing the concentration of Fe or Mn, making them available for use in a wide temperature range. The derivative of the magnetic susceptibility was measured for PdMn and PdFe between 1.5 K and 4 K using a SQUID magnetometer. These measurements, as well as preliminary noise and drift measurements, show them to have sub-nK resolution with a drift of less than 10^{-13} K/s.

I. INTRODUCTION

Space-based, fundamental physics experiments are being planned that require thermometry with sub-nanokelvin temperature resolution near 2.2 K.[1] Currently, paramagnetic salts such as CAB or $GdCl_3$ are used as the temperature sensing elements in magnetic high resolution thermometers (HRTs). In particular, CAB ($Cu(NH_4)_2Br_4 2H_2O$), which undergoes a ferromagnetic phase transition at 1.8 K, has demonstrated low drift (< 10 fK/s) and a temperature resolution of 0.1 nK.[2] However, paramagnetic salt crystals are difficult to grow, corrosive to most metals, and become dehydrated if kept under vacuum conditions at room temperature. These considerations have motivated the development of new thermometric

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elements for use from 2–3 K. Here we report on the properties of a dilute ferromagnetic alloy as the sensing element in a thermometer design similar to that used in previous ultra-precise measurements.

The first demonstration of the use of dilute ferromagnetic alloys as thermometers was in the mK range by Jutzler *et al.*, who showed that PdFe alloys with only a few ppm of iron follow a Curie law for T greater than the Curie temperature, T_c, with a deviation of less than one percent.[3] PdFe at these concentrations is known to undergo a spin-glass freezing transition at around 1 mK. A high sensitivity can be achieved because of the high susceptibility of the Pd matrix which creates giant localized moments of the order of 10 Bohr magnetons (μ_B), which persist well above T_c.

We report sensitivity, noise, and drift data for PdMn at concentrations ranging from 0.1 to 1 atomic percent manganese. In this concentration range, the transition is ferromagnetic with a T_c between 1.5 K and 4 K.[4] High sensitivity is obtained by exploiting the divergence of the magnetic susceptibility in the transition region where, for a "uniform" system,

$$\chi_{\text{crit}} \sim \left(\frac{T - T_c}{T_c}\right)^{-\gamma}. \tag{1}$$

Equation (1) does not accurately describe the critical behavior of these dilute random systems which have a statistical distribution of exchange interactions. Nevertheless, because of the high concentration (0.75 atomic percent) of Mn needed to create a T_c of about 2 K, the transition in that material is very sharp, and the temperature dependence of the magnetization follows a Brillouin function as one would expect for a uniform system with localized moments.[5] AC susceptibility measurements show a frequency independent susceptibility and the absence of any observable hysteresis.[6] Therefore our initial measurements have focused on PdMn as a candidate for use as a high resolution thermometer (HRT) for planned experiments on the critical dynamics of superfluid ^{4}He aboard the International Space Station,[7] and possibly in a variety of future fundamental physics experiments.

II. GIANT MOMENTS IN DILUTE FERROMAGNETIC ALLOYS

Giant moments arise when small amounts of Co, Fe, or Mn are dissolved in a Pd matrix.[8,5] These moments result from two sources: (1) The impurities retain their magnetic character when dissolved in the host metal and so interact with the host electrons through the exchange interaction. (2) Pd is an incipient ferromagnet. That is, correlations between the electrons in the 4d band of Pd tend to increase the extent and lifetime of spin

fluctuations. As a result, for pure Pd the magnetic susceptibility, χ_0, is enhanced by the Stoner factor so that,

$$\chi = \frac{\chi_0}{1 - U\chi_0} \tag{2}$$

where U is a measure of the repulsive Coulomb interactions. For Co, Fe, or Mn dissolved in Pd, the exchange interaction between the bare impurity moment and the easily spin polarized matrix creates a large region of spin polarization around each impurity. At large impurity-impurity distances the polarization alternates in sign (the RKKY interaction), so that the ground state is a spin-glass. For intermediate concentrations, the region of spin polarization can be described by the exchange potential

$$V(r) \propto \frac{R}{r} e^{-r/R} \tag{3}$$

where R is a measure of the radius of the polarization cloud which can be as much as several Angstroms, and r is the distance from the impurity. These "giant moments" typically range from 7–10μ_B. The overlap in space of the spin correlation clouds described by Eq. (3) results in a net ferromagnetic exchange interaction between impurities. The strength of the effective coupling constant depends on the average distance between impurities so that the Curie temperature increases with increasing concentration. For PdMn, T_c can be made to vary from the mK range to around 6 K for concentrations between 1 and 3 atomic percent.

At extremely high concentrations, direct antiferromagnetic Mn-Mn interactions compete with the ferromagnetic interactions, creating a high-concentration spin-glass state.[9,4] However, at any concentration, the statistical nature of the impurity distribution will result in some combination of all three interactions mentioned above. This complicates the development of theories applicable in the critical region, as well as resulting in an interesting domain structure and localized spin fluctuations in the ordered phase.[10] Nevertheless, PdMn is unique in that, at least for higher concentrations, it exhibits a very sharp phase transition and a temperature dependence of the magnetization that can be described by a Brillouin function. This is very much like the behavior of "uniform" ferromagnets such as iron. Magnetization measurements have shown a giant moment in PdMn of about 7.7μ_B.[11] The sharp, nonhysteretic phase transition[12] around 2 K makes PdMn an excellent candidate for the sensing element in a magnetic thermometer for use in the 2 K range where high sensitivity and low drift are of prime importance.

III. SAMPLE PREPARATION

The samples were prepared by mixing 99.9985% pure palladium powder[1] with appropriate amounts of Mn or Fe powder (99% metals based purity) to make a total sample mass of between 4 and 6 grams. The mixture was pressed at 34 MPa using a 12 mm diameter steel die. Samples where then melted under high-purity Ar on a water-cooled hearth of a tri-arc furnace equipped with tungsten electrodes. Each sample was completely melted three times, and was unloaded and inverted between each iteration. PdMn samples were prepared with nominal compositions of 0.60, 0.75, and 0.90 atomic percent Mn. One PdFe sample was prepared with 0.17 atomic percent Fe.

The samples were machined into cylinders of length 13 mm and diameter 2.3 mm. They were cleaned in aqua regia and ultrasonically rinsed in distilled water and alcohol. Finally, they were annealed under vacuum at 1000°C for 60 h.

IV. MEASUREMENT APPARATUS

For the sensitivity measurements, the samples were inserted into a BeCu holder wrapped with about 50 turns of 0.003″ diameter NbTi superconducting wire, which formed the pickup loop for a dc superconducting quantum interference device (dc SQUID). The dc SQUID, manufactured by Quantum Design, Inc., had a flux sensitivity of about $10^{-5}\,\phi_o$ in a 1 Hertz bandwidth, where ϕ_o is one flux quantum. The BeCu holder was the same length as the sample, but its diameter was such that the total volume of the sample accounted for 50% of the total volume within the holder.[13] BeCu was used because of its low electrical conductivity, thereby minimizing inductive coupling of Johnson noise into the SQUID measurement loop. This assembly screwed into one end of an approximately 2 inch long annealed aluminum post which in turn screwed into the bottom Cu platform of a low-temperature probe next to a germanium resistance thermometer (GRT) which had been calibrated earlier against the saturated vapor pressure of ^{4}He. This platform was the lower stage of a two-stage probe. The upper stage, to which it was linked through a thermal filter, was cooled by a 1 K helium pot. Both stages were surrounded by a brass vacuum can and kept under a hard vacuum for the duration of the experiment (see Fig. 1).

A 10 cm long, 4.5 mm diameter niobium flux tube fit snugly over the entire sample/BeCu holder/aluminum post assembly (see Fig. 2). The

[1] Alfa Aesar, 30 Bond St., Ward Hill, Massachusetts 01835-8099, USA.

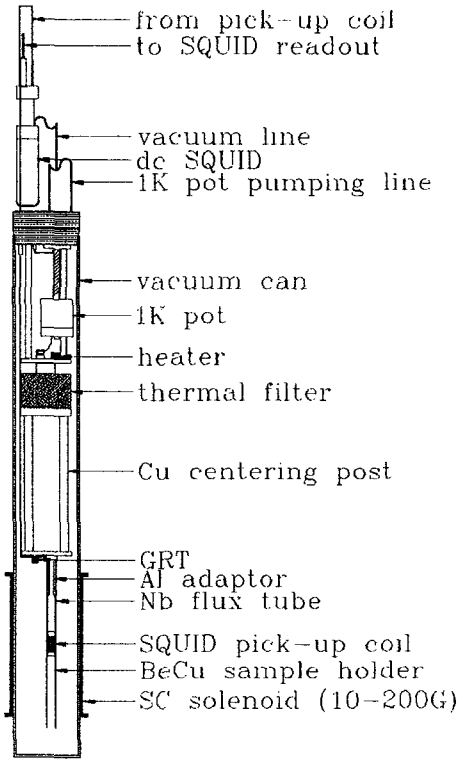

Fig. 1. Low temperature cryostat design for the sensitivity measurements.

purpose of the flux tube was to protect the sample from stray fields, as well as to trap a constant field on the sample for the measurements. For further stray field protection, the lead wires to the SQUID pick-up coil were twisted together, sheathed in teflon tubing, and fed through a 0.66 mm OD niobium capillary which went to the SQUID input. The teflon and niobium tubes were both filled with silicon based oil to prevent microphonics. The SQUID itself was located outside of the vacuum can and therefore in direct contact with the He bath. An external solenoid was used to provide a 5–200 Gauss magnetic field that had a uniformity of about 1 percent.

Noise and drift measurements were obtained using a more sophisticated cryostat equipped with a liquid He cell that provided a superfluid transition temperature "fixed point" reference. Also, temperature stability requirements for drift and noise measurements are much more stringent, necessitating the use of multiple temperature controlled stages and a radiation shield on the sample stage.[1] A third modification was that, for reasons

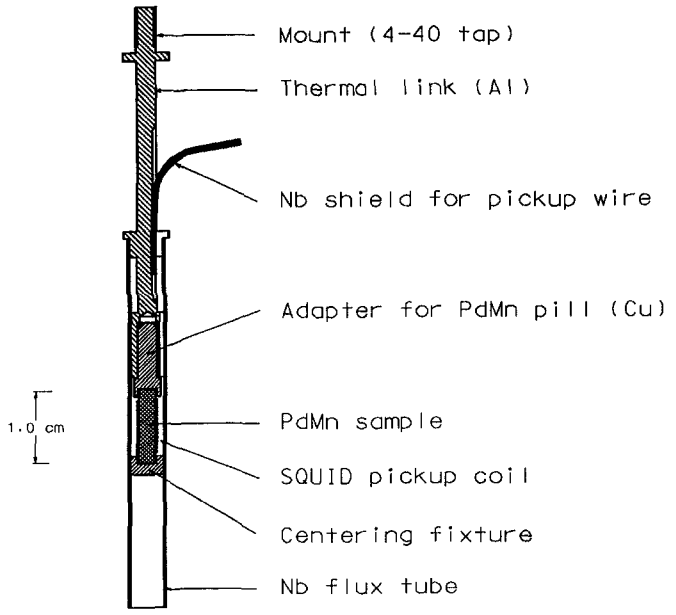

Fig. 2. Schematic of the mini-high resolution thermometer.

related to the optimization of these high resolution thermometers (HRTs) for space flight experiments, the SQUID pick-up coil was wound directly around the sample without the BeCu holder.

V. SENSITIVITY MEASUREMENTS

When a change in the magnetic susceptibility of the sample occurs, a current is induced in the SQUID pick-up coil that is detected in the SQUID input. The change, $d\phi$, in flux coupled to the SQUID by a change in susceptibility, $d\chi$ is given by[14]

$$d\phi = NAHx\, d\chi \tag{4}$$

where H is the trapped field, N is the number of turns in the pickup coil, A is the area of the pick-up coil, and x is a dimensionless coupling constant which depends on geometrical factors as well as impedance matching to the SQUID. For this experiment, the change in susceptibility resulting from a step change in temperature was measured by monitoring the output of the SQUID and the calibrated thermometer after increasing the current to a heater attached to the upper cooling stage.

The result of the sensitivity measurement, in units of flux quanta per μK, for 0.75 atomic percent Mn in Pd is shown in Fig. 3. The inset shows the sensitivity of CAB measured at 200 G with the same probe for comparison. Figures 4 and 5 show the sensitivity results for 0.90 atomic percent Mn in Pd, and 0.17 atomic percent Fe in Pd. The trapped fields used were between 10 and 200 G and, as is expected, severely broadened the transition, especially for the higher fields. For fields less than about 50 Gauss, the sensitivity peaks between 1 and 2 ϕ_o/μK in the transition region. This sensitivity measurement may be used to determine the impact of any noise source that is capable of inducing a flux change in the measurement loop, whether it be from SQUID instability, intrinsic magnetic fluctuations, or microphonics. The Quantum Design Model 5000 dc SQUID used in this experiment is stable to within $10^{-5}\,\phi_o$, where ϕ_o is one flux quanta. Using a sensitivity of 1 ϕ_o/μK, Eq. (4) gives a resolution of 10 pK in a 1 Hz bandwidth. As discussed in the next section, this is almost 2 orders of magnitude less than the spontaneous thermal fluctuations expected from a system of this heat capacity and thermal response time. Therefore the resolution will be determined from either intrinsic magnetic or thermal noise. These noise

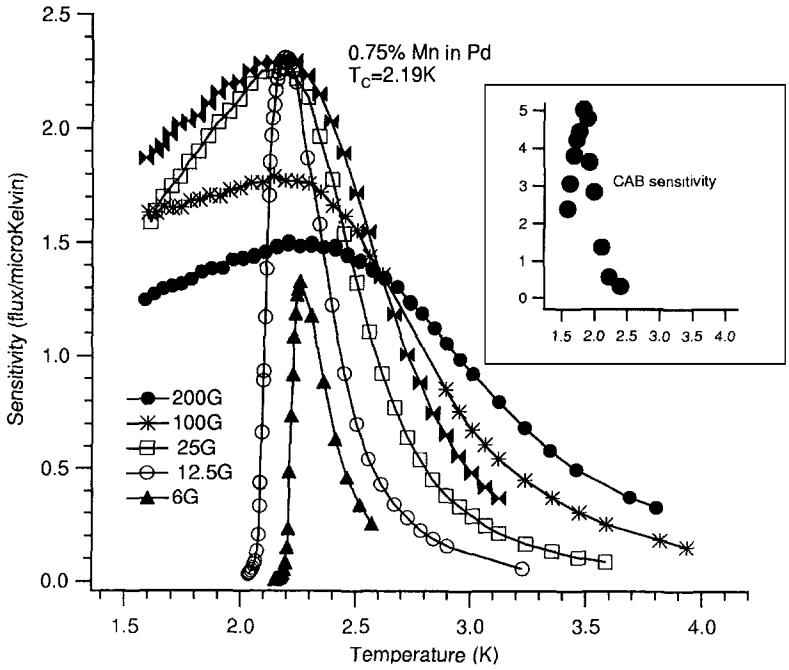

Fig. 3. Sensitivity vs. temperature in units of ϕ_o per μK for the 0.75 atomic percent Mn in Pd sample.

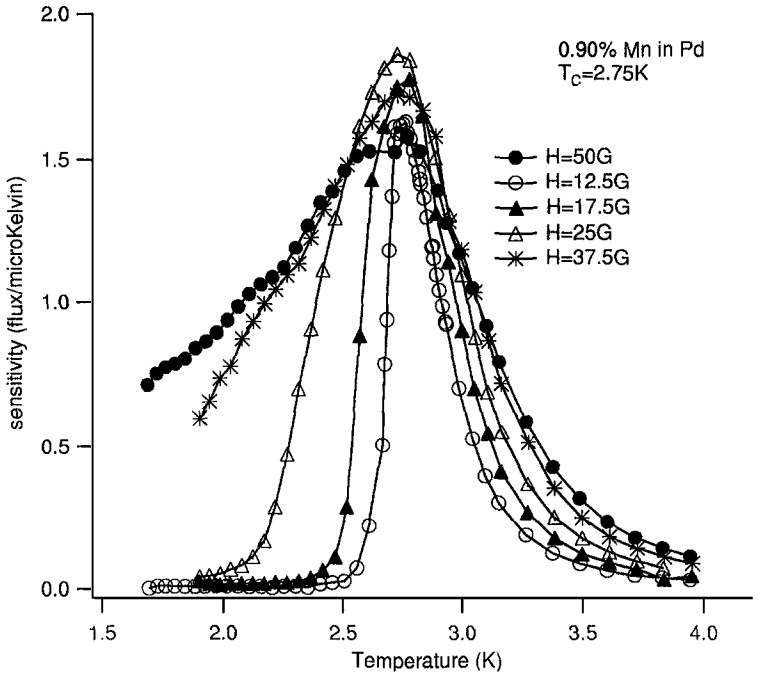

Fig. 4. Sensitivity vs. temperature in units of ϕ_o per μK for the 0.90 atomic percent Mn in Pd sample.

sources are discussed in the next section, where it will be demonstrated that the dominant contribution is from thermal noise.

Figure 6 shows the measured Curie temperature as a function of atomic percent Mn in Pd for several samples. These temperatures were determined by picking the point that corresponds to the highest sensitivity for the lowest applied field. This allowed a determination of T_c with a precision of 10 mK. It should be noted that the sensitivity maximum varied little with applied field. With this method, T_c corresponds to the temperature where a curve of the susceptibility vs. temperature would have a maximum in slope. Also in the figure are data points compiled from a review article by Nieuwenhuys.[15] The error bars for these points were not provided, and they represent data taken using several different experimental techniques and different methods for determining T_c. However, the data points seem to consistently fall on a line above those obtained from this work. The cause of this systematic error remains to be investigated. Nevertheless, the plot shows that the variation of T_c with Mn concentration monotonically increases with a slope comparable to that obtained from previous measurements.

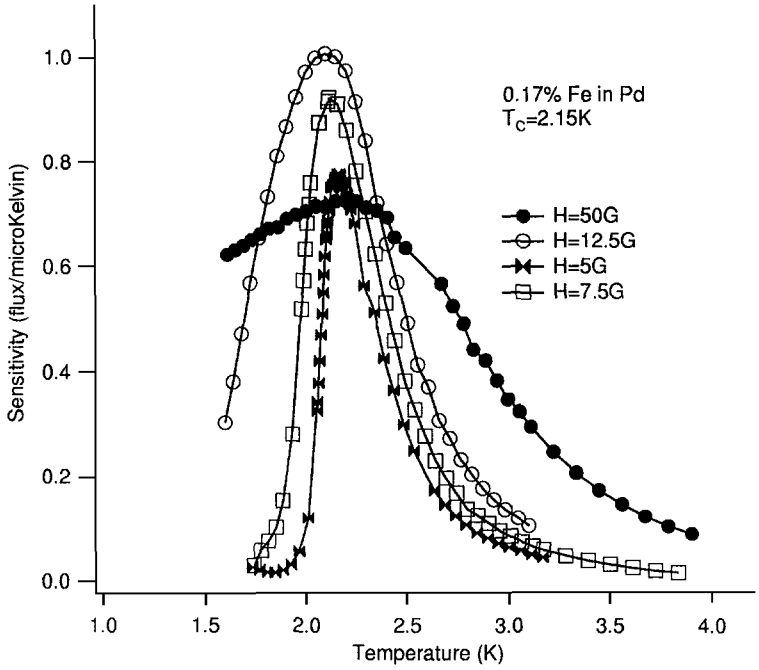

Fig. 5. Sensitivity vs. temperature in units of ϕ_o per μK for the 0.17 atomic percent Fe in Pd sample.

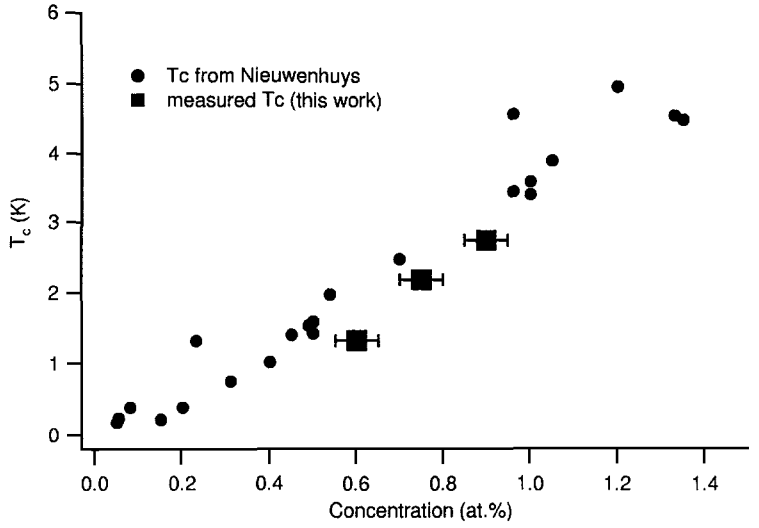

Fig. 6. T_c vs. atomic percent Mn from this work compared with that obtained by previous investigators.[15]

VI. NOISE AND DRIFT MEASUREMENTS

Noise and drift measurements were taken by analyzing the output of an HRT made with 0.17 atomic percent Fe in Pd with a spectrum analyzer, using the modified set-up described in the Measurement Apparatus section of this paper. Figure 7 shows the noise spectrum in $\mathrm{nK}/\sqrt{\mathrm{Hz}}$ for this sample, in an external field of 50 G.

Integrating the noise from 0 to 1 Hz resulted in a noise of 0.3 nK for the 1 Hz bandwidth. The thermometer time constant obtained from the roll-off frequency of the noise spectrum was 0.1 s. Similar noise measurements of the 0.60 atomic percent Mn in Pd sample were complicated by experimental difficulties, but indicated an upper bound to the noise level of $0.42\ \mathrm{nK}/\sqrt{\mathrm{Hz}}$ at 1 Hertz, and a time constant of roughly 0.3 s.

According to the fluctuation dissipation theorem, the total noise due to thermal fluctuations is given by[16–18]

$$\langle (\Delta T)^2 \rangle = kT^2/C \tag{5}$$

where C is the heat capacity. The noise for a given frequency interval is

$$d\langle (\Delta T)^2 \rangle = \frac{4\tau k T^2}{C(1+4\pi^2\tau^2 f^2)}\, df. \tag{6}$$

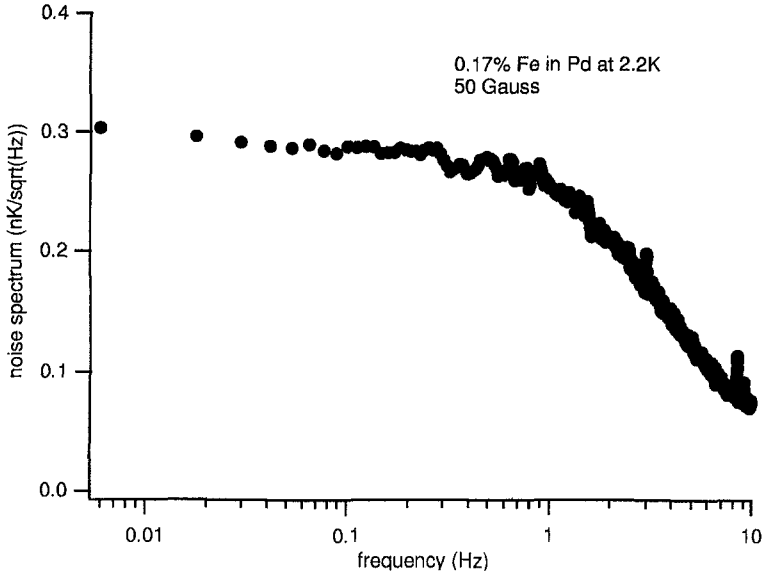

Fig. 7. Root-mean square noise as a function of frequency for the 0.17% Fe in Pd sample.

Equation (5) is obtained by integrating Eq. (6) over the entire frequency domain. It can be seen from Eq. (6) that, for frequencies small compared to the bandwidth $1/\tau$ (where τ is the thermal time constant), the noise will be inversely proportional to τ/C. Since $\tau = RC$ where R is the thermal resistance to the heat bath, the intrinsic noise of the thermometer at low frequencies depends only on the thermal resistance between the thermometer and the helium "heat bath."[16, 17]

The mean square temperature fluctuation per unit frequency interval is then given by the familiar equation:

$$\langle (\Delta T)^2 \rangle_{\Delta f} = 4RkT^2 \tag{7}$$

where $\langle (\Delta T)^2 \rangle_{\Delta f}$ is the root-mean square temperature fluctuation per measurement bandwidth. One can use the Wiedemann–Franz law to estimate the thermal resistance from the electrical resistance of the PdMn at 2.2 K. Previous measurements of the thermal conductivity of PdFe at low temperatures by Schroeder and Uher[19] have shown that it follows the Wiedemann–Franz law for Fe concentrations up to at least 0.145 atomic percent. Furthermore, applying the Wiedemann–Franz law to PdMn using published values of the resisitivity (around 1 $\mu\Omega$ cm at 2 K[15]) gives a thermal conductivity of 0.054 W/K cm, similar to values reported for PdFe. Using this value and the sample dimensions to calculate the thermal resistance of the PdMn, an estimate of the expected noise from Eq. (7) gives 1 nK/$\sqrt{\text{Hz}}$, which is in rough agreement with the measurements. As mentioned above, this is about 2 orders of magnitude larger than the SQUID noise. Also, using $C = 1$ mJ/gK[20] gives $\tau = 0.4$ s for our 0.2 g sample, which is close to the measured value.

Using a similar argument to that leading to Eq. (7), the intrinsic magnetic noise is given by,

$$\langle (\Delta H)^2 \rangle_{\Delta f} = 4\tau_M kT/\chi V \tag{8}$$

where V is the sample volume, and τ_M is the spin lattice relaxation time. To our knowledge, no measurements of the spin lattice relaxation time for PdMn have been published. Since AC susceptibility measurements show that χ is frequency independent for frequencies up to at least 100 Hz,[6] 0.01 s may be considered as an extreme estimate for the upper bound to τ_M. Combining Eqs. (4) and (8) we get,

$$\langle (\Delta T)^2 \rangle_{\Delta f} = \left[\frac{\chi}{H\, d\chi/dT} \right]^2 \langle (\Delta H)^2 \rangle_{\Delta f}. \tag{9}$$

Using $\chi < 10^{-2}$ and $d\chi/dT > 10^{-1}$/K gives an upper bound to the temperature noise induced by magnetic fluctuations of a fraction of a nK/$\sqrt{\text{Hz}}$ for a 50 G applied field. Although this is comparable to the thermal fluctuation noise, it is to be considered an absolute upper bound, especially given that the spin-lattice relaxation time could very well be orders of magnitude smaller than the value used. Using $\tau_M = 10^{-5}$ s (a more reasonable estimate for these solids at low temperature[21]), the temperature noise induced by magnetic fluctuations is around 10 pK.

Similarly, the induced current noise due to Johnson noise is:

$$\langle (\Delta I)^2 \rangle_{\Delta f} = 4k\tau T/R_{\text{el}} \tag{10}$$

where R_{el} is the electrical resistance of the sample along an axis perpendicular to the axis of the SQUID pickup coil. In principle, one could use this formula to calculate the Johnson noise using the resistivity of the sample, the sample dimensions, and the inductance of the pick-up coil, provided that the relaxation time, τ, or the capacitance of the sample are known. It is a reasonable assumption that the relaxation time of the PdMn will be similar to that of aluminum at 4 K. Measurements of the Johnson noise of an Al sample of similar dimensions in the same probe gave a flux noise into the SQUID of $10^{-3}\,\phi_o/\sqrt{\text{Hz}}$. The resistivity of the aluminum at 4 K was measured to be $5 \times 10^{-3}\,\mu\Omega$ cm. As already mentioned, the resistivity of PdMn is about $1\,\mu\Omega$ cm at 2 K. According to Eq. (10), the current noise will scale as the inverse square root of the resistivity if the sample dimensions are the same. Therefore one would expect a flux noise of about $7 \times 10^{-5}\,\phi_o/\sqrt{\text{Hz}}$ for the PdMn sample. This gives an effective temperature noise of 0.1 nK/$\sqrt{\text{Hz}}$, which is on the order of, but smaller than, the thermal fluctuation noise.

Hence the limiting factor for temperature resolution in PdMn is spontaneous temperature fluctuations given by Eq. (7). If this noise source were the only factor, then R (the thermal resistance to the heat bath) would be the only design parameter to consider. However, the heat capacity is relevant to the response time of the thermometer. This is especially applicable to space experiments where cosmic rays produce temperature spikes that are then integrated over the RC time constant of the thermometer, and hence may result in a temperature offset.[13] It is desirable, then, to design a thermometer where both C and R are as low as possible. The sensitivity measurements have demonstrated that, near the transition, $d\phi/dT$ is high enough that even an order of magnitude mass reduction won't significantly affect the resolution, since the limiting factor is spontaneous thermal fluctuations. Therefore it is advantageous to decrease the mass of the PdMn thermometric element until the magnetic or SQUID noise becomes a

significant contribution to the total noise spectrum. In any case, more design work needs to be done to minimize R and C. These measurements, although adequate for planned space experiments,[1] should be considered as an upper bound for τ and $\langle(\Delta T)^2\rangle$. Also, these results suggest that PdMn or PdFe alloys may permit ultra-low noise measurements ($\delta T < 0.1$ nK/$\sqrt{\text{Hz}}$) in configurations where R may be greatly decreased by direct contact of the thermometric element with the helium ("wet" thermometry).

Drift was measured by comparing the temperature reading of the PdMn HRT to a fixed point temperature reference[22] over a long period of time. The measured temperature variation indicates the drift of the thermometer. These drift measurements showed a drift rate of 2×10^{-14} K/s for the PdMn thermometer over a period of 24 h.

VII. CONCLUSIONS

The giant moment ferromagnets, PdMn and PdFe, show excellent potential for use in high resolution thermometers over a broad temperature range from 1.5 K to at least 3 K. Sensitivity measurements show at least one ϕ_o per 1 μK temperature change, resulting in a SQUID noise limit that is well below the limit of spontaneous thermal fluctuations resulting from the fluctuation-dissipation theorem. Also, Johnson noise and intrinsic magnetic noise do not contribute significantly to the total noise. Already these thermometers have demonstrated a time constant of less than one second and a drift rate of less than 0.1 pK/sec. Nevertheless, more work needs to be done to minimize noise by optimizing parameters such as thermal resistance to the bath (R) and thermal response time (τ). It will also be useful to examine drift and noise characteristics for a wider variety of systems and concentrations.

ACKNOWLEDGMENTS

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