Why Cantilever?

Designing and Testing a New Material Cantilever Material For Use in Cantilever Magnetometry

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Abstract:

Cantilever magnetometry is a widely used measurement technique, and is extremely well suited for finding the critical fields of phase transitions, as well as measuring the frequency of de Haas-van Alphen oscillations. In this paper we describe our findings of the suitability of polyimide "Kapton", an inexpensive non-metallic material, for use as a cantilever in cantilever magnetometry. In addition, the construction and setup of the cantilever devices is described, an explanation of the theoretical benefits of a plastic cantilever is provided, and some advice for future testing is given.

Samples with magnetic moments respond in two ways to an applied magnetic field. There is a torque given by equation 1 that is dependent on sample orientation, and a direct force in fields with a gradient in an arbitrary z-direction, given by equation 2. If the sample is placed on a cantilever in the presence of a magnetic field, the cantilever will deflect in accordance with Hooke's Law.

$$\vec{T} = \vec{M} \times \vec{B} \qquad (1)$$

$$F = \vec{M} \bullet \frac{\partial \vec{B}}{\partial \vec{Z}} \qquad (2)$$

Measurement techniques vary according to cantilever materials. Metal cantilevers are cheap and relatively tough. They are typically of a simple 2-plate capacitance-based design (fig. 1), where only an absolute value of displacement can be measured from the change in capacitance. Torque values can be measured by placing the sample in field center, where most laboratory superconducting magnets have small field gradients. Force values can then be measured by placing the sample outside of field center where the gradient is larger.

Silicon cantilevers are useful because circuitry can be applied to them so that more direct measurements can be made. One implementation is to have two pairs of parallel capacitors, with calibrated circuitry on the top of the cantilever that makes use of negative feedback (fig. 2)[i]. The circuitry in silicon cantilevers can allow force and torque measurements to be made simultaneously. Force measurements are computed by taking the sum of the parallel signals, and torque measurements are computed by taking the difference of the signals (this also gives the sign of the torque, whereas a single pair of capacitor plates can only give the absolute value). The application of negative feedback to the circuitry of the device in figure 2 also allows the cantilever to act in a more linear fashion, as the actual deflection of the cantilever becomes orders of magnitude smaller.

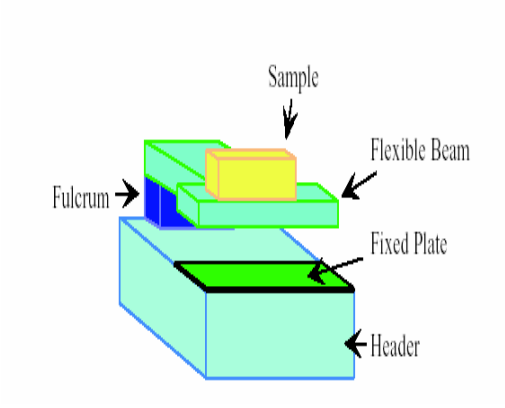

Figure 1: A simple 2-plate capacitor metal cantilever. Changes in magnetization are reflected by changes in capacitance. The metal cantilever itself forms the top plate of the capacitor, and the "fixed plate" forms the bottom

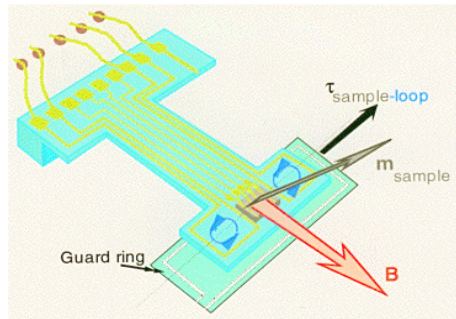

Figure 2: A silicon cantilever with circuitry plated on. The bottom of the cantilever has two parallel conducting surfaces that form the top plates of two capacitors. The small loops to either side of the sample generate a magnetic moment that balances that of the sample through a system of negative feedback. Changes in magnetization of the sample are reflected by changes in the currents through the loops.

There are several reasons why an ideal plastic cantilever would be superior to any cantilever currently in use. Metal cantilevers generate eddy currents in changing magnetic fields, and have a background magnetization. It is also difficult to add circuitry to them, since the entire surface conducts electricity. On the other hand, a single silicon cantilever typically costs $1500 and can break easily and is not suitable for materials with large magnetic moments. Plastic cantilevers would be the best of both worlds. They would be affordable, accurate, and relatively durable. The makers of polyimide claim that it is durable at low temperatures, so it appeared to be a reasonable material to test[ii].

Our measurements were made with simplest of the setups described above. We built a simple two-plate capacitor, with a small piece of brass sheet epoxied to the bottom, and the polyimide cantilever held in place by two pieces of G-10 and screws at the top. The G10 was examined under a microscope for burrs and sharp edges that might alter the flexile properties of the polyimide. A thin layer of gold (2000Å +/- 10Å thick) was sputtered to the bottom surface of the polyimide to provide a conducting surface for the cantilever. This layer is thin enough that the flexile properties of the polyimide remain unaltered, but thick enough that it doesn't rub off and its resistance is low enough that little power is dissipated from the sinusoidal excitation current (on the order of 10^{-16} watts[1]). Leads to coaxial cables were soldered to the back of the setup, and the sample itself was attached to the cantilever using GE Varnish thinned with a few drops of toluene. A diagram is given in figure 3. Measurements were taken by using an analog capacitance bridge, hooked up to a lock-in amplifier. Magnetization measurements were performed in an 18T superconducting magnet.

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[1] Given a sinusoidal input current for a parallel-plate capacitor, with current that decreases linearly along the length of the plates, the average power dissipation can be shown to be roughly equal to $(1/3)RC^2w^2V_{rms}^2L$. With R measured to be roughly 3.8 ohms, C ≈ 1pF, L ≈ 7mm, w ≈ 6283 rad/sec, and V_{rms} = 10V, average power dissipated is 1.7×10^{-16} watts.

Figure 3: One of our cantilever devices. The sample shown was used for testing the spring constant at room temperature utilizing the earth's gravitational field; it was much larger than the samples used in the magnet. The cantilevers we used were 0.383" long and 0.285" wide, stamped out by a metallic press that had been made for us. The spacers were somewhat less than 0.046" thick.

We had two sheets of polyimide to work with throughout the experiment, one .002" thick and one .003" thick, and are designated 200HA and 300HN, respectively. The suffix HA and HN correspond to different methods of manufacture; HA tends to be softer and more malleable. Both thicknesses had a slight curve to them. The 200HA had a radius of curvature between 21 and 25 mm, and the 300HN had a radius of curvature between 127 and 149 mm. The small radius of curvature of the thinner polyimide became a significant obstacle from the beginning of the experiment, and we feel it is no coincidence that we had problems with this thickness during later stages of the experiment.

The differences in types of polyimide may be important as well. DuPont claims that its Kapton® HA is softer and more malleable, whereas Kapton® HN is stiffer and considered to be the general purpose material. This is may also be an explanation for the disappointing results from the 200HA.

Our first task (having completed construction) was to get a rough estimate of the spring constants of two thicknesses of sheet-polyimide. This could be done at room temperature by using the gravitational field of the earth, attaching masses to the cantilevers, and measuring the change in capacitance between the "right side up" orientation and the "upside-down" orientation with a standard LCZ meter. The plots of values are given in figure 4, and appear to be linear. The spring constants of the different thicknesses are proportional to the inverses of the slopes of the lines drawn, such that the spring constant of the thicker polyimide was roughly 6 times that of the thinner. A derivation of an approximation for the absolute values of the spring constants is given below.

S_1 and S_2 are the initial and final deflections of the cantilever plates, and S_0 is the separation of the cantilever plates with zero deflection.

$$F = -kx = mg \rightarrow x = -mg/k$$

$$\Delta C \approx \frac{\varepsilon_0 A}{S_0 + S_1} - \frac{\varepsilon_0 A}{S_0 + S_2}, \text{ and } S_1 \approx -S_2$$

$$\therefore \Delta C \approx \frac{-2\varepsilon_0 A S_1}{S_0^2 - S_1^2} \approx \frac{-2\varepsilon_0 A S_1}{S_0^2}$$

$$\therefore S_1 = x = \frac{-\Delta C S_0^2}{2\varepsilon_0 A} = -mg/k$$

$$\therefore k = \frac{2mg\varepsilon_0 A}{\Delta C S_0^2}$$

Measuring $A \approx 2.8\text{x}10^{-5}\text{m}^2$, $S_0 \approx .00165\text{m}$, we get:

$$k \approx \frac{m}{\Delta C} \times 1.78 \times 10^{-9}$$

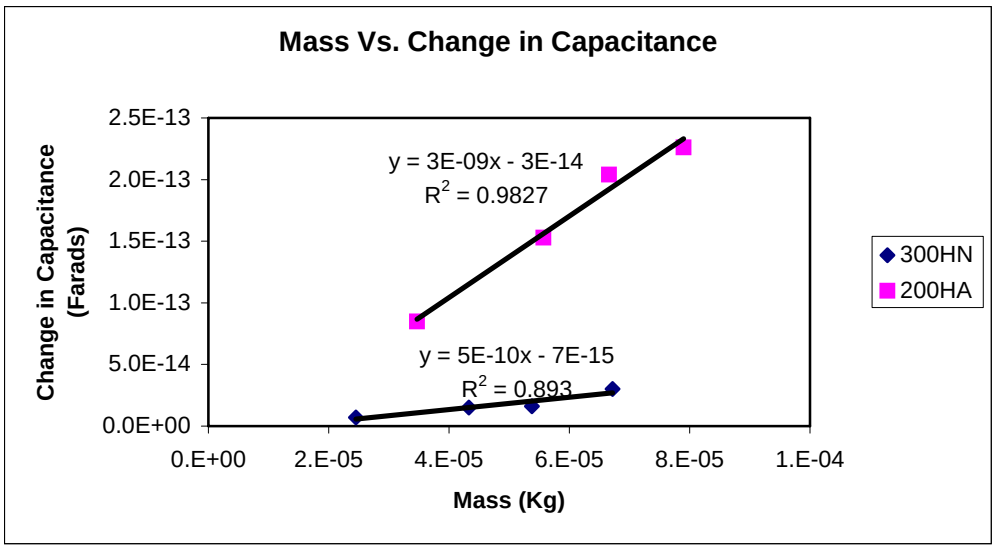

Figure 4: A plot of mass vs. change in capacitance. Linear fits are added with corresponding equations and R^2 values. Using the above derivation, we find that $k_{300} \approx 3.552$ N/m and $k_{200} \approx 0.592$ N/m.

The next step was to start taking measurements inside the magnet. $CeCoIn_5$, a material whose magnetic properties have been well mapped[iii], was used for all measurements. Its crystalline structure consists of alternating planes of $CeIn_3$ and $CoIn_2$. When the c-axis of the crystal (the axis perpendicular to these planes) is pointing parallel to the direction of the magnetic field, the transition to superconductivity occurs at its minimum field (~4.9T). When the c-axis is perpendicular to the field the transition occurs at its maximum field (~11.9 T). It is a first-order phase transition at 300mK, and a second-order transition at 1.5K.

Inside the magnet at very low temperatures (most measurements were made at roughly 0.3K) and small sample sizes, the best way to roughly determine relative spring constants is to compare transition heights (measured in units of capacitance) for the same sample at the same field on different cantilevers. Using this method of measurement, we could determine that the difference between the relative spring constants appears to have

decreased (see table 1 – the first two 200HA measurements listed should probably be discounted – see figure 7 for a plot of the first one).

TABLE 1

Thickness of polyimide	Transition Size (pf)	Transition Field (T)
300HN	0.014159292	9.39
300HN	0.015818584	8.69
300HN	0.020312	9.87
300HN	0.01957	9.17
	Average size: .0175pF	
200HA	0.001012658	11.5
200HA	0.001042753	11.13
200HA	0.07644	10.63
200HA	0.061037736	10.73
200HA	0.061641026	8.31
	Average size excluding first two: .0664pF	
	Average size including first two: .0402pF	
	Ratio of spring constants is somewhere between 2.3 and 3.8, decreased from roughly 6.	

There were several characteristics of the polyimide that we needed to check to determine its suitability as a cantilever material. These properties can be separated into three categories. First, it was important to determine that there are no fundamental material properties of the polyimide that might make it inappropriate for use. Second, it was important to determine how differences between polyimide samples (curvature, thickness, and material) might play a role in the signal-to-noise ratio. Third, we made sure that we could clearly see the phase transitions of the $CeCoIn_5$, and that our results corresponded with those previously reported by Mr. Murphy and others in the literature (see third endnote).

The very first measurements were made to determine which direction of curvature would give better responses (towards or away from the bottom plate). As expected, we found that the signal-to-noise ratio appeared to be greater when the polyimide was bent towards the bottom plate. Therefore every measurement after the first few used polyimide oriented concave down, with the curve running the length of the polyimide to reduce the possibility of a step-function response (fig 6).

Figure 6: A sketch showing the direction of curvature.

Rough initial testing of the polyimide, done by Dr. Eric Palm and Mr. Tim Murphy, pointed to the unfortunate possibility of a step-function response to applied forces. In addition to an overall loss of precision, this sort of deviation from Hooke's Law would be catastrophic when circuitry and feedback loops are applied, as the

polyimide might simply oscillate between two discrete modes. Fortunately, the 300HN polyimide never showed signs of "clicking" or a step function response during any of the data taking. Decreasing the ramp rate of the magnet (from our typical 0.5T/min to 0.2T/min) eliminated other minor deviations from expected results as well. The 200HA showed clicking responses on two consecutive runs, out of a total of 13 otherwise low-noise measurements. The effect occurred on a macroscopic level, over the course of several tesla. These two instances, of little significance on their own, hint at the overall trend of the superiority of the 300HN polyimide to its thinner counterpart.

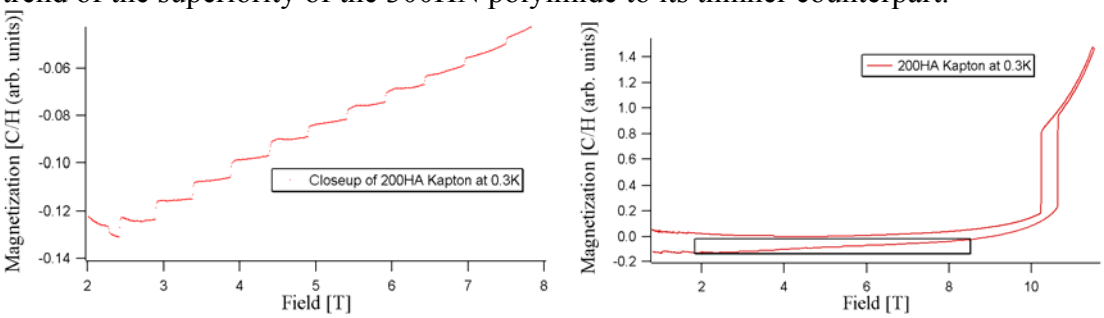

Figure 5: A possible example of "clicking" with 200HA polyimide. The graph to the left is a zoom in of the boxed region to the right. The points in the plot to the left are not connected, allowing the observer to see that this is a macroscopic effect and not noise-related.

The 300HN generally outperformed the 200HA. The thicker polyimide produced repeatable, seemingly noise-free responses (see fig 9). However, while the signal sizes for the 200HA were occasionally larger, there would be different responses each time the probe was reinserted into the magnet. For example, two of the samples were created by breaking a single crystal in half. The 300HN gave similar transition heights for both samples at 0.3K (fig 7), but the transition heights for the 200HA differed by more than a factor of 10 at similar fields (fig 8). It is important to note that different cantilevers of each type were used each time the samples were removed, making small changes due to small inhomogeneities in the sheet of polyimide possible. However, this does not seem to be a likely cause for such a large deviation.

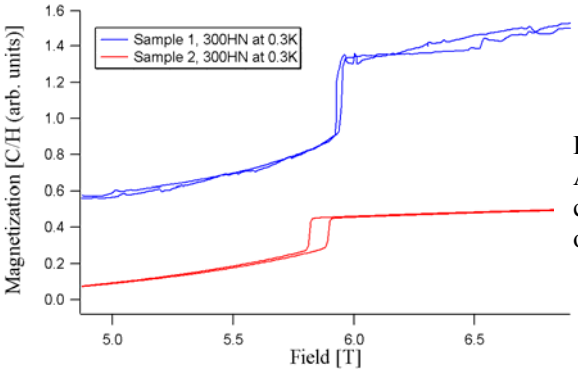

Figure 7: Two examples of 300HA at 0.3K. Although the first plot is somewhat noisy, it is clear that the transitions are of the same order of magnitude as each other.

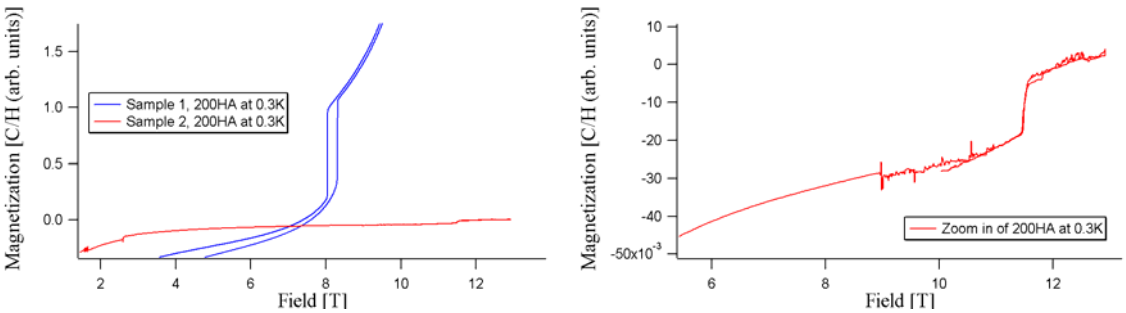

Figure 8: Two examples of 200HA at 0.3K. The graph to the right is a zoom in of the graph to the left. The same two samples are used as in figure 7, but the difference is dramatic. The smaller signal had a lot of noise near the transition, but the down sweep (stops at 10T) was done at a lower ramp rate and was much cleaner.

An additional problem with the 200HA is that we couldn't get clear signals at 1.5K, even a matter of minutes after having successfully completed smooth runs with the 300HN at that temperature. When viewed on the same plot as successful runs of the 300HN, the 200HA looks like flat lines. Closer inspection reveals only noise (fig 9). One explanation for this behavior is that the cantilever may have bent sharply up and away from the opposite plate, decreasing the signal to negligible size. The smaller transition in figure 8 was produced a few measurements later with the same cantilever at a lower temperature.

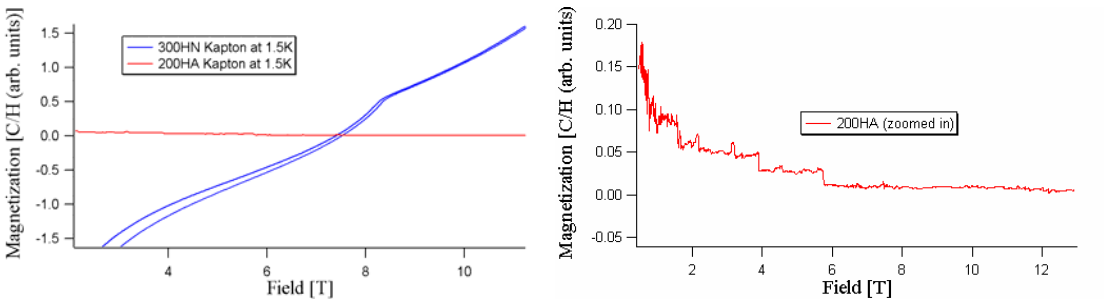

Fig 9: Results at 1.5K, done within minutes of one another

Our results with the 300HN, and in some cases the 200HA, were very good. We were able to clearly see the transition to superconductivity when it was either a first or second order transition. Some further examples are given in figure 10. Hysteresis is clearly distinguishable from sweep rate effects at 0.3K, and transitions match up well in both increasing and decreasing field intensities at 1.5K. Some trends caused by different angles of orientation can be seen in figure 11.

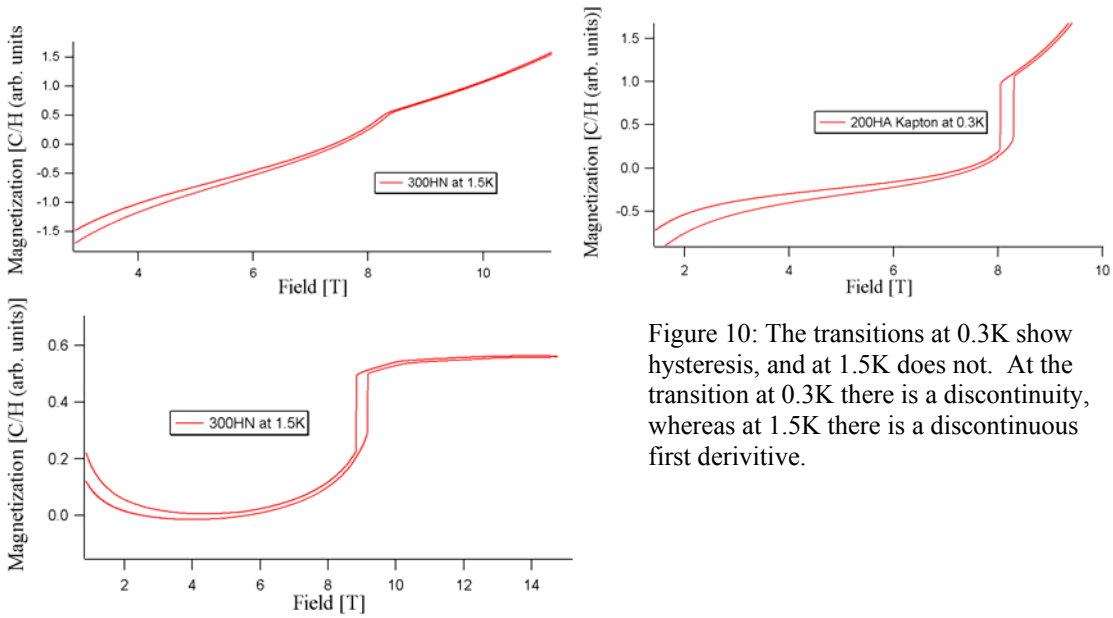

Figure 10: The transitions at 0.3K show hysteresis, and at 1.5K does not. At the transition at 0.3K there is a discontinuity, whereas at 1.5K there is a discontinuous first derivitive.

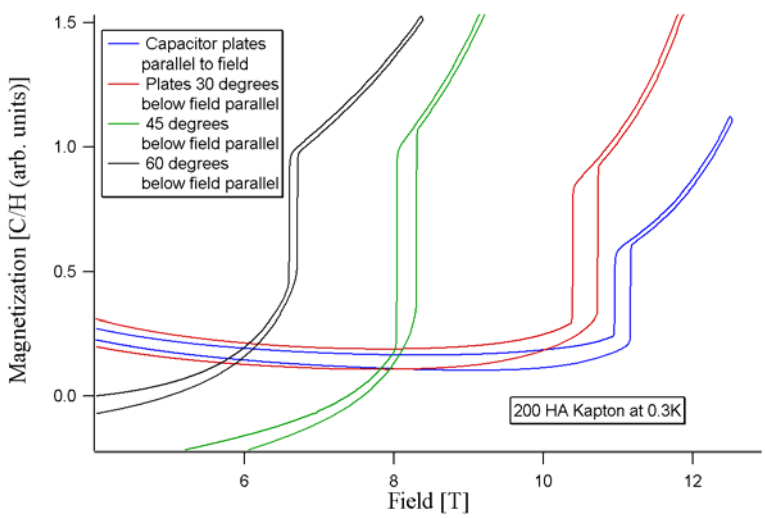

Figure 11: Several plots made with the 200 HA at different angles of orientation, at 0.3K. Changing the angle results in a change in transition field, as we expected. We had similar results with the 300HA.

There are several problems that need to be solved in all types of cantilever measurements before good data can be obtained. The first problem is to choose a sample of a size that would give an appropriate signal. This was a matter of guess and check, with good sample sizes generally on the order of a milligram. Smaller samples would also break easily, a problem that could only partially be solved by delicate handling.

The second problem involved shifting levels of ^{3}He. At 1.5K in particular, there would be strange readings that we believe were caused by changes in the dielectric constant of the capacitor plates. There were occasionally runs where sudden jumps in capacitance would occur every minute or so. We think that this was caused by droplets of ^{3}He condensing, hitting the cantilever, and dripping between the plates. Another strange effect was a large drift that would occur even at constant field. This was indicative of ^{3}He levels dropping to below the level of the top plate and continuing to change in time, and was often seen at 0.3K as well as 1.5K. Heating the sorb and fully recondensing ^{3}He solved both problems.

Future measurements should be done only with flat cantilevers (or cantilevers with large radii of curvature like the 300HN). This will allow the spacing between cantilever and bottom plate to be decreased, and the amount of relevant cantilever area to be increased, which would both increase signal size. A next step would also be to test the addition of circuitry to the polyimide cantilever, and see if it performs as it should. Overall, we feel we have made a good proof of principle of the basic suitability of polyimide cantilevers. The flatter, thicker Kapton® 300HN cantilevers worked extremely well in the qualitative measurements that we have made, and we expect them to work equally well in more complex designs in the future.

References:

[i] Figures 1 and 2 taken from Donovan Hall, "Magnetic Measurements with Metal Film Cantilevers: A User's Guide." [http://www.nhmfl.gov/users/facilities/dcfield/magnetometry/cantilever/], 1999.

[ii] http://www.dupont.com/kapton/

[iii] T. P. Murphy, Donovan Hall, E. C. Palm, S. W. Tozer, C. Petrovic, Z. Fisk, R. G. Goodrich, P. G. Pagliuso, J. L. Sarrao, and J. D. Thompson, Physical Review B, **65**, 100514 (2002).