Miniature AC susceptometer

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A miniature susceptometer has been developed that is small enough to fit inside clamp type pressure cells. The design and performances of the device are described, as well as examples of measurements possibly allowed. The susceptometer is comprised of 3 coils: a primary and a pair of balanced secondaries for the pick-up coils. Measurement sensitivity is enhanced by minimizing the size of the coils choosing certain design and construction technique. Calibration work for balancing the vacuum background signal is presented, as well as susceptibility measurements of superconducting Pb and various paramagnetic salts. Upcoming use in clamp type pressure cell is discussed.

I. INTRODUCTION

Experimental studies of materials under large hydrostatic pressure is a fine way to investigate their structural magnetic properties. High pressure conditions can become particularly interesting when coupled with low temperatures, where quantum nature critical phenomena are likely to occur. Characterization of magnetic differential susceptibility in such extreme conditions thus represents an interesting tool, looking for peculiar, fully quantum phenomena.

In that sense, new methods and tools for high pressure, low temperature susceptibility measurements are certainly welcomed, in the main perspective of enhancing measurements sensitivity. Motivations for choosing the design of a miniature susceptometer have been well described in [1], and are now recalled. Neat methods for creating those high pressure conditions indeed have their limitations in the small size available for sample, feature of the low temperature cryostat devices such as He^3-He^4 dilution refrigerators. One of the methods [3] for reaching the highest pressures, clamp type pressure cells, is yet limited if using SQUID for measuring magnetic moment, as the required non-magnetic materials for building the cell do not tolerate pressures above 20 kbar. Another possible method [2] for reaching pressures of order 100 kbar, the diamond-anvil cell, also has sensitivity limitations due to the small sample to detection apparatus size ratio in case of a pick-up coils standing outside the pressure cell.

In this article, the alternative method for obtaining high sensitivity proposed in [1] consisting of a primary and a pair of balanced secondaries fitting inside the pressure cell is further enquired. First some conceptual aspects of AC susceptometers are briefly recalled, then our self-made susceptometer is presented in details. Focus is put on design, construction techniques and calibration for cancelling the vacuum signal. Last, applications of the susceptometer in its final environnement is discussed.

II. APPARATUS

A. Recall on AC susceptometers

We first recall (for details, see [1]) some general concepts for AC susceptometers. The voltage induced by a uniform magnetic field of amplitude $H_{\rm ac}$ at frequency ω, in a secondary coil containing a sample with susceptibility χ_s is given by:

$$\begin{aligned} V_{\rm total} &= V_{\rm vac} + V_{\rm s} \\ &= (1 + \chi_{\rm s} f_{\rm s}) N A \mu_0 H_{\rm ac} \sin(\omega t) \end{aligned} \tag{1}$$

where N is the number of turns, A the cross section of the coil, μ_0 the permeability and $f_{\rm s}$ a coupling constant with the meaning of sample filled volume fraction. From this, we see that the sample susceptibility $\chi_{\rm s}$ can in principle be found by suppressing the background vacuum signal $V_{\rm vac}$. This is done by oppositely connecting an empty secondary to the one containing the sample, the response of both of those necessarily as similar as possible. Furthermore, the interpretation of $f_{\rm s}$ factor as a sample/void filling ratio, valid in the case of a long solenoidal coil, tells us that it is desirable to have a coil with scale as close as possible to the measured sample size. Here lies the main favorable argument for the miniature susceptometer design: maximizing the filling factor $f_{\rm s}$. However, this formula will be mainly considered as a qualitative motivation for our enquiry, since an absolute scale fit giving the proportionality factor in $V_{\rm susc} \sim \chi_{\rm s}$ has not been achieved up to now.

B. Design

A quite simple design for the susceptometer is chosen as shown in Fig. 1. Indeed, focus is put on miniaturization of the device to achieve high sensitivity. The primary coil and the two secondaries are arranged concentrically,

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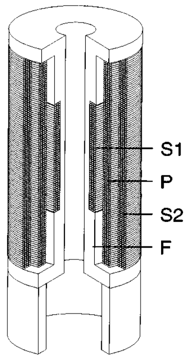

FIG. 1: Scheme of a similar miniature susceptometer, with the difference that in our case, all 3 coils have the same length, and that all support has been removed. Our dimensions are 3 mm in length and 4 mm for outer radius, inner radius or sample space is 1 mm.

the former being positionned inbetween the latter couple, in order to allow modifications out the outer secondary for calibration. An original idea for improving the ratio f_s is that the coils do not stand on any support; the whole fixation is ensured by Stycast epoxy and the Teflon-made supporting forms are removed after winding up of the coils.
In such design, calibration can only be made prior to measurements. The high simplicity of this structure can thus have the disadvantage of being an "all-or-nothing" type of device, in the sense that a background defect observed under experimental conditions cannot be corrected.

III. PERFORMANCE OF THE DEVICE

A. Calibration

From the simple concentrical design chosen, and as previously mentioned, no in-time calibration is allowed. Therefore, the best calibration is to be reached in ambiant conditions (pressure, temperature), before testing the stability of calibration in experiment conditions.
Calibration is made by modifying the outer secondary (adding/removing turns to the last layer) until reaching the best compensation, with the maximum precision of a single turn. Testings of the background signal were made in liquid Na and liquid He, ensuring similar temperature scale with future experiments. The minimal background signal was shown to be of order of magnitude comparable to low susceptibility paramagnets (such as $CuSO_4$). For reference, a measurement in liquid Na ($T \approx 77$K) gave: $U_{BG} = 3.16$ μV for an excitation $I = 1$ mA, $\nu = 1$ kHz. The imaginary part of the signal was found to be negligible here. Results given will generally be the direct signal measured, in μV. These values can in principle be scaled to give the AC susceptibility in SI units.

B. Application tests

Prior to the final application of the susceptometer, measurements are performed at atmospheric pressure, giving a first indication of the response signal of the susceptometer.
A first simple test is made with superconducting Pb, and the transition is observed through a signal jump towards a large negative value corresponding to $\chi_{SC} = -1$ [SI]. The observe critical temperature is $T_c = 7.1$ K giving a satisfactory correspondance with the theorical value: $T_c = 7.2$ K.

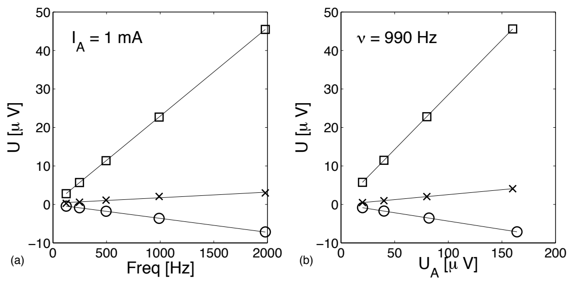

FIG. 2: Linear dependance of the signal to excitation amplitude and frequency, measured in liquid He ($T \approx 4.2$ K) and atmospheric pressure. The empty circles are the background signal with negative value, while the crosses are $CuSO_4$ and the squares gadolinium-gallium garnet (GGG), both with background signal substracted. Solid lines are the linear regression for each series. For similar sample sizes and thus similar f_s, the slope is directly linked to the susceptibility. $CuSO_4$'s signal for susceptibility is thus comparable to the background signal while GGG has a signal and thus susceptibility about 7 times larger.

Another checking measurement is the dependance of the signal in the excitation frequency ω and amplitude U_A (directly linked to the applied magnetic field amplitude H_{ac}) shown in Fig. 2(a) and Fig. 2(b) respectively. The linear relation in those parameters expected from equation 1 is well verified.

A cryostat with controlled temperature down to 2 K then has been used for obtaining the temperature dependance of the signal, for background and various other materials. The temperature dependance of background and a target material $SrCu_2(BO_3)_2$ (SCBO) is shown in Fig. 3. Evidence is found that the environnement gives an important stray signal, dominating a possible signal from the weakly paramagnetic material SCBO.
Comparison with background measurement in liquid He ($T \approx 4.2$ K) give a signal 3 times more important in the temperature controlled environnement under similar excitation, denoting an important stray signal and the difficulty of further measurements in that environnement.

Our results have shown the existence of a small complex part of the AC susceptibility. The meaning of these

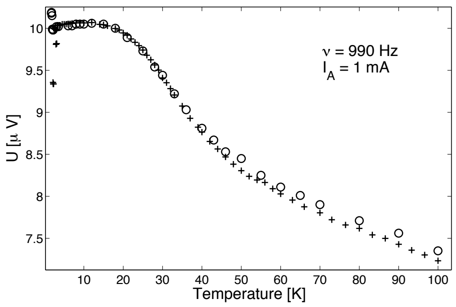

FIG. 3: Temperature dependance of the susceptibility signal at atmospheric pressure. The circles denote the background signal while the crosses are the measurements for SCBO. The latter shows to have no difference with the simple background measurement. The background measurement shows an important stray signal, increasing for decreasing temperatures and becoming constant around 20 K.

out-of-phase characteristics is that of a dissipative response to excitation and is not of interest for our AC susceptibility considerations. It has shown to be systematic enough and close to the normal $\phi = \pi/2$ in-phase response, but further occurance of it may need a deeper enquiry.

C. Further applications

With the elements of the previous sections in hand, practical application of the device can now be considered. As originally mentioned, the interest of the device lies in its use inside a clamp type pressure cell. A critical question in order is whether signal contributions from the body of the pressure cell will be important. While having no control and no possible "live-calibration" of the susceptometer, existence of stray signal can only be hoped to be small and systematic enough to still allow measurement of susceptibilities. This will be confirmed by first making background measurements inside the pressure cell simply at ambiant pressure. Another critical effect can be the modification of f_s with application of pressure. Again, this effect is hard to control if existing, but it has been discussed [1] to be generally negligible.

Various materials can be interesting to investigate under high pressure. $SrCu_2(BO_3)_2$ (SCBO) is one of the materials with which a phase transition is expected for $P \geq 10-12$ kbar. The device may also be used for enquiring the new iron pnictide $Ba(Fe_{1-x}Co_x)_2As_2$, $x = 0.08$, and namely enquiring the superconducting transition temperature T_c as a function of pressure.

IV. DISCUSSION

Different testings of our miniature susceptometer have been made, generally showing good results in expectation of future real-condition applications in clamp type pressure cells. Important stray signals have been observed which need to be controlled, thus probably requiring the use of a non-magnetic pressure cell. Some peculiar effects are observed in the dissipative, imaginary part of the signal, but which have shown a sufficient systemacy to be coped with.

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