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PRESS-MAG-O: a unique instrument to probe materials and phenomena under extreme conditions at Frascati

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PRESS-MAG-O is a new instrument under commission at the Laboratori Nazionali di Frascati of the Istituto Nazionale di Fisica Nucleare (INFN) designed to investigate materials under extreme conditions. The instrument, once completed, will allow combining high harmonic AC magnetic susceptibility measurements and magneto-optic experiments on samples under high pressures (HP), with a variable DC magnetic field in a wide temperature range. The system is designed to work at SINBAD, the IR synchrotron radiation beamline operational since 2001 at DAΦNE (Double Annular Φ-factory for Nice Experiments), the storage ring of the Laboratori Nazionali di Frascati of the INFN. HP will be applied up to about 20 GPa to samples inside a Cu–Be diamond anvil cell designed to allow concurrent FTIR experiments and high harmonic AC susceptibility measurements in a DC magnetic field up to 8 T and in a wide temperature range.

Keywords: high pressure; high harmonic magnetic AC susceptibility measurements; IR spectroscopy; phase transition

1. Introduction

PRESS-MAG-O is a new, original device funded by the Istituto Nazionale di Fisica Nucleare (INFN) designed to investigate materials under extreme conditions [1]. This apparatus is the result of a project funded by the Vth National Committee of the INFN [2,3]. Materials such as ferroelectric or superconducting systems, magnetic transitions and new condensed matter phases may be investigated with this device, which will allow concurrent magnetic and optical experiments.

The goal is to combine high harmonic AC magnetic susceptibility measurements and magneto-optic experiments on a sample under high pressures (HP), with a variable DC magnetic field and in a wide temperature range. In this way, it will be possible to gain unique information on

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systems and/or new phenomena that is almost impossible to obtain with standard spectroscopic methods.

The system has been originally designed to work at SINBAD, the IR synchrotron radiation beamline operational since 2001 at DAΦNE (Double AnnularΦ-factory for Nice Experiments), the storage ring of the INFN Frascati National Laboratory (LNF) [1]. However, the optical layout and the optics have also been designed to perform Raman experiments.

While for IR experiments an interferometer will be used, for the magneto-dynamic experiments a SQUID magnetometer in the 10 Hz–2 KHz frequency range has been designed and realized. HP will be applied to samples by a Cu–Be diamond anvil cell (DAC), so that the device will be able to collect FTIR spectra and high harmonic AC susceptibility data in a DC magnetic field up to 8 T and to about 20 GPa in a wide temperature range (4.2–200 K).

The aim of this work is to describe the design, the principal technical components of this system, and the status of its commissioning.

2. The instrument

The main components of the PRESS-MAG-O apparatus are summarized below. It is an original instrument; the result of significant R&D in different areas.

2.1. *The cryostat and the magnet*

The cryostat (Figure 1), manufactured by DG-Technology Service (Parma, Italy), is a compact device made by an amagnetic stainless steel vacuum vessel with two shields operating at the liquid nitrogen temperature and four access ports for optical experiments (Figure 1). Inside, there is an internal liquid helium reservoir hosting an 8 T superconducting split coil magnet manufactured by American Magnetics Inc., which can generate a DC magnetic field up to 8 T (Figure 1). It also has four radial pipes and a vertical port to insert the 'sample-insert' device. The latter has been designed to insert both the DAC cell and the magnetometer. A static sample cooling occurs via a cold finger on the bottom side. The cryostat allows concurrent magneto-optics experiments due

Figure 1. Photograph of the interior of the cryostat. Both the LN2 shield and the 8 T magnet are visible.

to two thin, wedged diamond windows of about 15 mm in diameter set along the light path. These can transmit light from the visible down to the far-IR range.

2.2. *The sample insert*

The sample holder insert is the heart of the PRESS-MAG-O system. It can be divided into two sections: an upper part that moves inside the lower, fixed section. The insert may be loaded inside the cryostat through a load-lock port installed on the upper cover of the cryostat. It allows the translation of the insert in the cryostat, still maintaining the vessel under vacuum.

The upper part of the insert can be aligned in the X–Y directions and rotated with micrometer accuracy. These motions will be used to align the sample inside the DAC to the light spot. The sample is loaded inside a CuBe gasket with a hole of ∼400 μm diameter.

The sample holder is properly shaped with some breaks to host: (1) the DAC with the Cu–Be springs to guarantee an efficient cold contact, via the ‘external fixed element’, with the cold finger of the cryostat; (2) the slider that sets the micro-gradiometer inside the DAC; and (3) the excitation coils.

After technical evaluations and tests with an AC magnetic field generated by the excitation coils, the original Cu–Be sample holder has been manufactured in sapphire, an optimal thermal conductor and a high electrical insulator, with some components in PEEK.[1] The latter is a material highly resistant to thermal degradation as well as to attack by both organic and aqueous environments, and is characterized by a very low diamagnetic response [4].

The new DAC holder (Figure 2) allows for minimizing the ‘Foucault current’ losses still maintaining an optimal thermal contact.

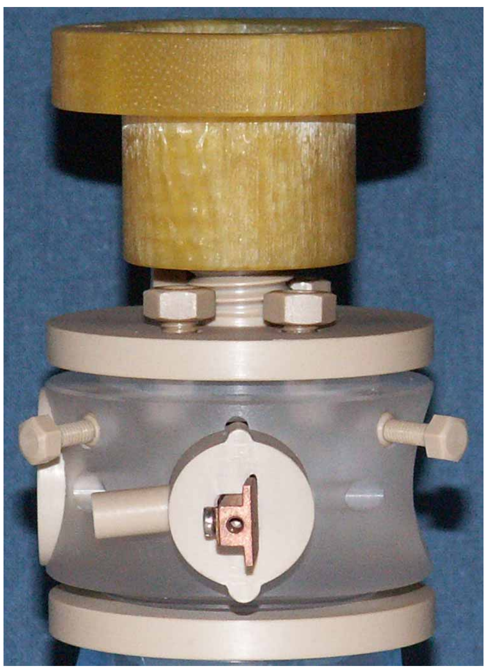

Figure 2. Photograph of the sapphire sample holder with its components in PEEK.

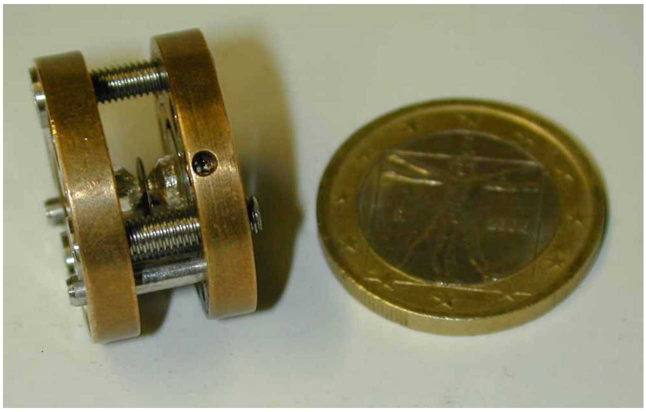

Figure 3. Lateral view of the Cu–Be DAC cell. Inside the cell the diamond anvil sets on the moissanite disks and the CuBe gasket are evident. The coin on the right is used for comparison.

2.3. *The CuBe DAC cell*

To apply pressure, we designed and manufactured a specific miniature DAC cell [5]. To minimize the magnetic contribution from the cell, it was manufactured with a non-magnetic 2% Be–Cu alloy and also its smaller parts and screws were made by non-magnetic materials. In more detail, it is assembled with two Cu–Be disks and titanium screws to apply pressure in the GPa range, two brilliant cut diamonds of type IIa for performing experiments down to the far IR range [6], and two specifically manufactured moissanite (SiC-δ) cylinders with a 400 μm conic hole to push the diamond anvils. A photograph of the Cu–Be DAC cell we developed for these experiments is shown in Figure 3.

2.4. *The AC gradiometer and the SQUID*

A micro-gradiometer coupled to a SQUID will be used to collect the magnetic response of the samples. The micro-gradiometer sensor is made with two Nb thin film coils realized by a photolithography technique on a mono-crystal silicon wafer. The AC micro-gradiometer has been designed to work inside our DAC and its geometrical configuration has been optimized to fit near the gasket position around the diamonds. In this configuration, the sample located between the anvils will be also set between the two pick-up coils. The SQUID sensor has been tested at 4.2 K with the open input coil. It showed an equivalent flux noise of $3.2\,\mu\Phi_0/\sqrt{\mathrm{Hz}}$ ($\sim 7 \times 10^{-21}\,\mathrm{T\,m^2}/\sqrt{\mathrm{Hz}}$).

2.5. *The PRESS-MAG-O optical system*

The PRESS-MAG-O instrument has been designed to perform optical experiments such as FTIR spectroscopy in transmission or reflection modes and for Raman spectroscopy. A brilliant infrared synchrotron radiation (IR SR) source is ideal to perform HP investigations on small samples inside a DAC and this is particularly true at DAΦNE, the Frascati electron–positron collider working in topping up mode at an energy of 0.51 GeV per beam with a maximum beam current $>$2 A. SINBAD, the IR SR beamline operational since 2001 at DAΦNE [1], has been designed to work at IR wavelengths from about 10 up to 10,000 cm^{-1} and operates with a customized Bruker Equinox 55 interferometer working in vacuum coupled to a Bruker Hyperion 3000 microscope [7].

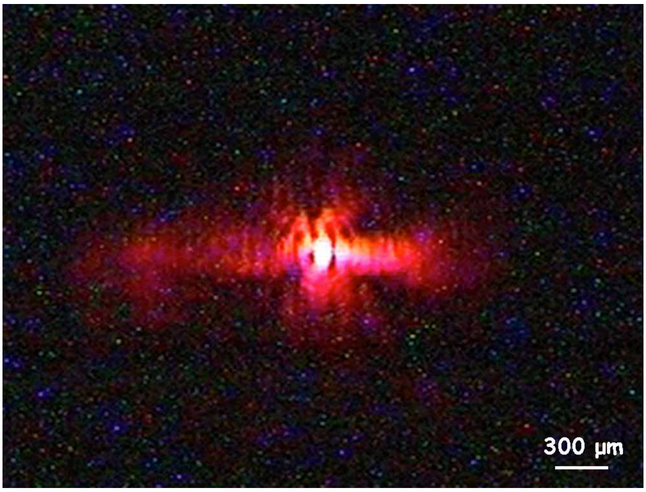

Figure 4. Image of the focal spot produced by the Cassegrain optical system, with a horizontal collimated illumination. A similar image is obtained for the vertical focus. The observed spot has a diameter <300 μm.

In more detail, the collimated beam coming out from the interferometer and entering the PRESS-MAG-O optical system will be focused inside the DAC on the loaded sample by using one of the four lateral ports equipped with optical transmitting windows. Different windows can be used, including a chemical vapor deposition (CVD) wedged diamond window, which is the best option to cover a wide IR range down to the far-IR domain [6].

A compact Cassegrain concentrator has been designed to fit the dimensions of the apparatus in order to focus the synchrotron radiation to a small-size spot (Figure 4) (the diameter of the Airy disk is $\sim$ 200 μm at the shortest wavelength) to match the aperture of the DAC. The concentrator has the maximum possible numerical aperture, fulfilling constraints imposed by the physical dimensions of the cryostat. The wavelength range of the collector goes from the visible down to the far-IR wavelength range, *i.e.* with the possibility to work down to 50 μm. The entrance beam diameter is $\sim$ 30 mm and the optical system is placed inside a tube of steel. At the end of the tube an optical window of 16 mm diameter is installed. After the first tests, the estimated spot dimensions are better than 300 μm in the horizontal direction.

For the IR transmission experiments, the beam will exit through a second window placed on the opposite lateral port and will then be focused to the detector position by a symmetrical Cassegrain lens. For Raman experiments with the DAC cell or for experiments performed in the reflection geometry, as in a microscope confocal optical layout, the beam will be collected by half of the Cassegrain optics used for the incoming beam in the transmission geometry.

3. PRESS-MAG-O measurements

To prove what kind of unique experiments can be performed with PRESS-MAG-O, we show in Figure 5 an example of multi-harmonic magnetic susceptibility measurements made on a bulk sintered $FeSe_{0.88}$ sample of dimension $3.2 \times 2 \times 2\,mm^3$. This is a superconductor with the simplest crystal structure among Fe-based superconductors (space group: $P4/nmm$). Measurements were performed with a zero field cooling experimental set-up at ambient pressure as a function of the temperature, heating the sample at a rate of 0.43 K/min in the temperature range 4–9 K.

The left panel of Figure 5(a) shows the real part of the first harmonic (χ_1') versus temperature for three different frequencies of the applied H_{ac} magnetic field. The curve describes the magnetic

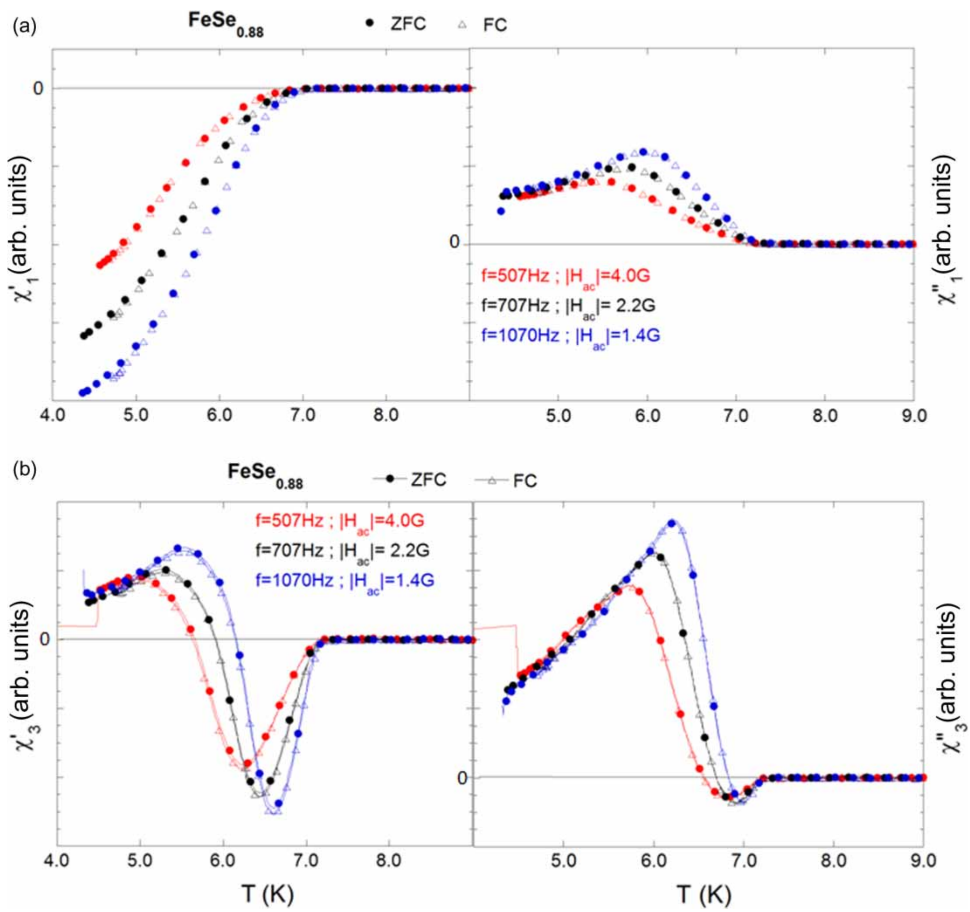

Figure 5. AC multi-harmonic magnetic susceptibility measurements of the $FeSe_{0.88}$: (a) real and imaginary parts of the first harmonic; (b) real and imaginary parts of the third harmonic. For comparison, all graphs are plotted to the same scale.

behavior of a characteristic superconductor sample with a pure negative diamagnetic signal. The first harmonic imaginary part (χ_1''), shown in the right panel of Figure 5(a), is correlated to the magnetic energy absorbed during the time of the magnetization (H_{ac}) cycle and it is connected to the superconducting critical current value. High harmonic components (Figure 5(b), left–right) can be regarded as the Fourier coefficients [8] of the steady magnetization cycles in the presence of an external oscillating magnetic field. These coefficients are affected by the magnetic flux entering and leaving the sample and are connected to the nonlinear flux dynamics. They describe the complex interaction between the flux vortex array and the extrinsic properties such as the pinning due to the disorder present in the material.

4. Conclusions

When completed, PRESS-MAG-O will represent a unique and extremely versatile instrumental facility capable of investigating systems in a wide range of DC magnetic field ($\leq$8 T), temperature (4.2–200 K) and pressure (0–20 GPa), allowing concurrent or simultaneous FTIR spectroscopy and AC magnetic multi-harmonic susceptibility measurements (10 Hz–20 KHz). The instrument based on a novel cryostat design and an ultra-sensitive AC micro-gradiometer will also allow probing of the magnetic response of small samples loaded inside a Cu–Be DAC. Both magneto-optical experiments versus pressure and versus magnetic field under a DC high magnetic field will be possible using the IR SR emission at SINBAD, the beamline operational at Frascati.

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Note

1. The acronym PEEK refers to polyetheretherketone, a polyaromatic, semicrystalline thermoplastic polymer with excellent mechanical and chemical resistance properties that are retained in a wide range of temperatures.

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