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Experimental techniques for pulsed magnetic fields

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Abstract

Recent developments at the K.U. Leuven pulsed field laboratory are reviewed. This includes new 60 T user magnets, pulse shaping, decentralized data recording for multiple experiments, low temperature cryogenics, vibration damping, rotatable sample holders and sensitive magnetization measurements. Experimental results (on semiconductors, superconductors and organic conductors) are shown to illustrate the improvements in the experimental techniques.

1. Magnets

The K.U. Leuven pulsed field facility [1] has been further developed to provide higher fields and improved experimental techniques. Magnets with optimized glass fibre reinforcement [2] now generate 60 T in a 15–22 mm bore with 10 ms pulse duration for daily use in experiments, with a life expectancy of the order 500–1000 pulses. The failure mode of these strong magnets is mostly soft, i.e. with limited damage to the surroundings.

A new generation of magnets is developed with CuNb microfilamentary conductor made in cooperation with the A.A. Bochvar Institute (Moscow), and with CuAg wire obtained from the Showa company. The CuAg coils are made in cooperation with the Tsukuba magnet laboratory and with the NPML at Sydney, Australia. These coils incorporate internal and external reinforcement with glass and carbon fibre composites and with pearlitic steel wires. By monitoring the inductance during the training of the magnets, it was found that repetitive plastic cycling (which is identified as the turning point where the inductance levels off) occurred at 60 T.

A critical issue in the design and behaviour of magnets with glass fibre reinforcement remains the initial yielding of the conductor which is required to preload the glass fibre reinforcement. The associated deformation of the order 3–4% is not well matched to the total deformation which the strong but brittle conductors can handle. For this reason the development of user magnets is pursued in the direction of stiffer reinforcement (carbon fibre, pearlitic steel). A prototype magnet with 12 mm bore, made from CuAg conductor and carbon fibre as internal reinforcement, was successfully tested up to 70 T. To cope with insulation problems caused by the conductivity of the carbon fibre, additional glass fibre insulation was inserted between layers and at the end of the layers.

The cooldown time of the magnets is about 30 min, therefore it is possible to run several experiments simultaneously on the same capacitor bank. The co-ordination between these experiments is done by a central computer in a multitasking environment. The computer takes care

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of the switching between different experiments, the charging/firing of the capacitor bank, the inductance measurement and the recording of the experimental signals. This allows running up to 3 experiments simultaneously.

To measure small signals with high sensitivity while protecting the electronic equipment, mobile Faraday cages are used. The signals from the probe are relayed to the upper part of this cage, which is a screened box containing a $\pm$ 15 V DC power supply for the amplifiers. In this box, the signals are processed and amplified. These signals are then relayed to the fast data recorder, which is inside the lower compartment of the Faraday cage; the recorder is linked to the central computer via an optical IEEE interface. Fast lock-in amplifiers are mounted in the same compartment. Although this mobile Faraday cage is very close to the pulsed magnet, no interference has been observed.

Magnetization measurements on high T_c superconductors are very sensitive to the sharp increase of the field at the beginning of the pulse, as explained below. To eliminate this discontinuity, a coil with ferromagnetic core is inserted in series with the pulsed field coil [3]. As the inductance of this coil is much larger than the inductance of the pulsed field coil, the current increases slowly until the ferromagnetic core becomes saturated. In our design, the initial inductance of the coil with ferromagnetic core is 10 times higher and its saturated inductance 10 times lower than the inductance of the pulsed field coil which is 0.9 mH. The core saturates at roughly 300 A. The initial dH/dt is reduced by a factor of 10. The generated maximum field is reduced by only 3% due to the insertion of the coil with ferromagnetic core.

2. ^{3}He cryogenics

Recently, some radical improvements have been made to the ^{3}He cryostat in Leuven which has enabled an extensive variety of different experiments to be performed at higher magnetic fields, and with greater sensitivity. Currently this apparatus is being used to study a range of materials including organic conductors, high temperature superconductors and heavy fermions.

The first improvement has been to replace the old ^{3}He insert with a new slim, plastic equivalent. In particular this has enabled greater flexibility in changing between different samples and probes which can be changed on a day to day basis. Given the simplicity in the design, a new fridge can be quickly assembled in the event of a breakage.

The mounting of the ^{4}He cryostat, magnet and pumping systems has been modified to include vibration isolation, mostly by home-made silicon rubber elements. A vibration-free environment is essential for certain experiments, in particular those of magnetization where it has been found to reduce the levels of experimental noise by two orders of magnitude so that sensitive de Haas–van Alphen experiments can be performed at the highest magnetic fields. As an example, in Fig. 1(a) we show an example of de Haas–van Alphen oscillations observed in the organic compound $(ET)_2KHg(NCS)_4$ [4].

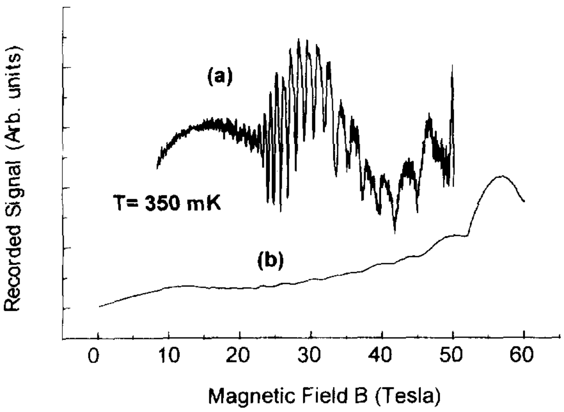

Fig. 1. (a) An example of recorded de Haas–van Alphen oscillations in α-$(ET)_2KHg(SCN)_4$ up to 50 T. (b) An example of Shubnikov de Haas oscillations measured in β-$(ET)_2AuBr_2$ in fields of 60 T.

By miniaturizing the tails of the cryostats we have been able to use magnets of smaller bore and therefore perform experiments at higher magnetic fields. As an example, we show in Fig. 1(b) an example of Shubnikov–de Haas oscillations measured in β-$(ET)_2AuBr_2$ in fields of 60 T [5]. In these experiments, a combination of an AC current source with fast lock-in techniques was used.

Several new experimental techniques are presently being investigated including field modulation in pulsed fields. This should enable magnetic susceptibility measurements to be performed with higher sensitivity, and will reveal different information than can be obtained using DC techniques. For metallic and strongly superconducting samples, methods of sample cooling are being developed, which is necessary because ^{3}He has a very poor thermal conductivity at low temperatures. By immersing the sample in a different medium within the ^{3}He cryostat, such as superfluid ^{4}He, the problems of sample heating in the pulsed magnetic fields can be overcome in many materials.

3. Magnetotransport

The ^{4}He bath cryostat, made entirely of stainless steel with a removable tail section (ID 12 mm), is mainly used

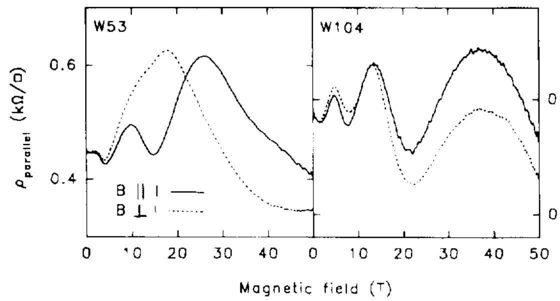

Fig. 2. Diamagnetic Shubnikov de Haas measurements in a parallel magnetic field on Si-delta-doped GaAs layers with different donor distribution widths (20 Å for W53 and 80 Å for W104). The results for two different configurations of magnetic field and current are shown [6].

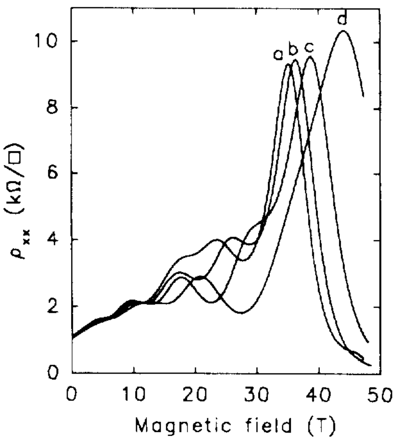

Fig. 3. Magnetoresistance measurements on a Si-δ-doped InSb structure for various angles theta between the magnetic field and the normal to the sample surface: (a) $\theta = 0°$, (b) $\theta = 15°$, (c) $\theta = 25°$, (d) $\theta = 37°$ [7].

for magnetotransport measurements on semiconductor systems, including low-dimensional III–V semiconductor structures and III–V-based semimagnetic semiconductors. For these measurements, various probes have been developed. Whereas the experiments on bulk samples are primarily performed in a magnetic field either parallel or perpendicular to the sample surface (e.g. measurements of the large negative magnetoresistance in InMnAs samples), the investigation of low-dimensional systems requires a sample holder for positioning the sample at several well-defined angles in the magnetic field. In addition to a continuously rotatable sample holder, a new probe has recently been built with a view to further miniaturization for use in a future ^{4}He cryostat with a 70 T pulsed field coil of 10 mm bore. The samples can easily be rotated e.g. for a study of the diamagnetic Shubnikov de Haas effect in a parallel magnetic field (with the magnetic field either parallel or perpendicular to the sample current, Fig. 2) or for an investigation of the angular dependence of the quantum Hall and Shubnikov de Haas effects (Fig. 3). For most of these measurements, the available high frequency lock-in technique cannot be used because of the high resistance of the samples (typically 5–30 kΩ). For this range of resistances, however, the standard DC current technique has been found adequate to perform highly sensitive experiments.

4. Magnetisation

Using a gas flow cryostat designed for far infrared transmission measurements, experiments can be performed in a temperature range 4.2–300 K, in fields up to 50 T. This cryostat was adapted to accommodate magnetotransport and magnetization measurements. Precise control and stability of the temperature can be achieved for measurements on high temperature superconductors. The cryostat consists of three concentric stainless steel tubes with dimensions 15.88/0.25, 12.7/0.25 and 12.00/0.20 mm. The helium gas flows between the middle and the inner tube which contains an exchange gas; between the middle and outer tubes a vacuum is maintained for insulation. The helium is pumped and flow is controlled by a needle valve. In the helium evaporation chamber, a 20 W heating resistor is mounted. The temperature is stabilized with a PID controller; the stability is better than 0.5 K.

An inductive magnetization sensor [8, 9] has been designed to measure thin superconducting films (of order 3000 Å). For magnetic fields in the range 25–50 T, the sensitivity of the probe is at least 10^{-2} emu, for fields below 25 T the sensitivity is better than 10^{-3} emu. Accuracy and high sensitivity were obtained by using a sample extraction system, vibration isolation and an inductive sensor with optimized flux coupling geometry. Using the sample extraction system, the magnetic moment of the empty sensor and the sensor coupled to the sample can be measured consecutively, and thus the fully compensated signal can be processed. In the case of thin film samples, a coupling of 70% is obtained by pressing the sample onto the pick-up coil. The sensor was calibrated using a Ni sample with a known saturable magnetic moment. This sensor has enabled us for the first time to perform magnetization measurements on superconducting thin films in very strong magnetic fields. An example is shown in Fig. 4 which shows the field dependence of the critical current density J_{cm} for a $Bi_2Sr_2Ca_2Cu_3O_{10}$ film at $T = 5$ K, obtained in pulsed field magnetization

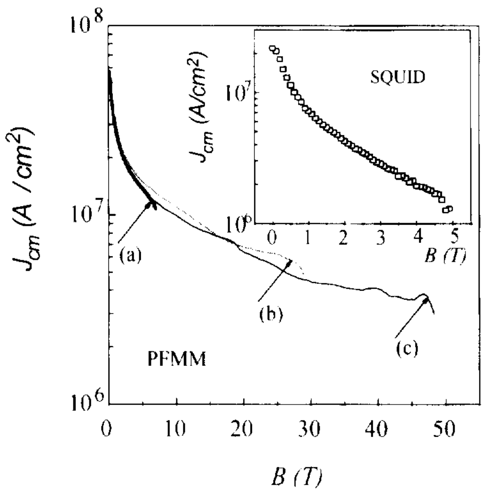

Fig. 4. Critical current density J_{cm} for a $Bi_2Sr_2Ca_2Cu_3O_{10}$ film at $T = 5$ K, obtained in pulsed field magnetization experiments with different fields B_{max}: (a) 7 T; (b) 27 T; (c) 47 T. The insert shows J_{cm} for the same sample obtained by SQUID.

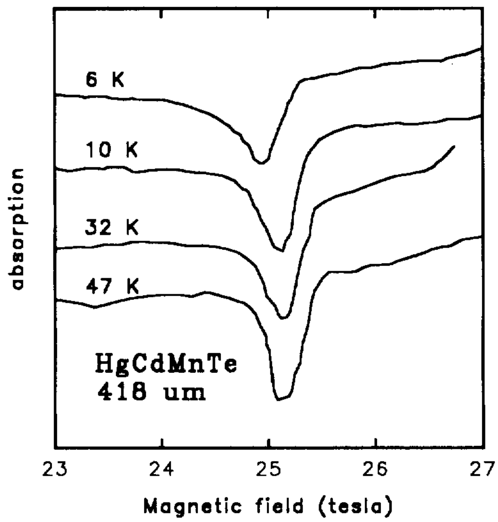

Fig. 5. Temperature dependence of the paramagnetic resonance in $Hg_{0.85}Cd_{0.03}Mn_{0.12}Te$ at 418 μm. The resonance positions shift by 0.6% when lowering the temperature due to the magnetic correlations.

experiments with different maximal fields B_{max}. The insert shows J_{cm} for the same sample obtained by SQUID. The critical current densities were calculated using the Bean model [10].

Measuring the magnetic properties of superconductors in a pulsed magnetic field is a difficult task. The superconductor generates in the pick-up coil a voltage which is proportional to $dM/dt = (dM/dH)(dH/dt)$. At the starting of the field pulse, $t = 0$, the sample is in the Meissner state and $|dM/dH|$ as well as $|dH/dt|$ have a maximum. The Meissner state lasts only for a very short time because of the high dH/dt and the low H_{c1} for superconductors. Once the superconductor is in its mixed state, induced signals become very small and require sensitive recording.

5. Far infrared

The facility is equipped with an optically pumped far-infrared laser which operates in the region between 1200 and 36 μm. A series of fast detectors (InSb, GaAs and Ge:Ga) is used to cover most of this range. The detectors are mounted in an integrating cavity at the end of a light pipe which fits a helium transport vessel. The sample holder for normal transmission experiments has a 3 mm aperture; the walls are coated with a non-conducting metal paint. For strip line transmission experiments, the light is guided to a 4 by 0.1 mm waveguide. The gold-coated tapered section has a linear horn instead of the common exponential horn; this was found to have the largest light throughput.

The strip line allows to measure the reflectance of materials in the Voight geometry. Due to the multiple reflections in the waveguide, this absorption is amplified, up to 100 times for metals. Thus ferromagnetic resonances in 3D metals could be measured with good signal-to-noise ratio. The strip line was also used to measure the dielectric response in semiconducting HgSe:Fe which has a strongly pinned Fermi level: the transmission showed Shubnikov–de Haas type oscillations similar to a magneto-transport experiment with the additional feature that the Fermi level was sampled with photons and thus showed a frequency-dependent broadening of the oscillations.

The conventional transmission experiments were recently upgraded with the possibility to measure under different angles. Angle-dependent measurements in delta-doped InSb at low fields were performed to distinguish between the different contributions of the bulk and two-dimensional (2D) electrons [11]. At high tilt angles, the two contributions are well separated and the effective mass of the lowest subband was determined. The system is now being upgraded to perform these experiments at higher field where it should be possible to discern the individual subbands.

The variable temperature cryostat operates in the range 5–300 K. This range is well suited to follow the temperature dependence of magnetically correlated systems as in e.g. $Hg_{0.85}Cd_{0.03}Mn_{0.12}Te$ (Fig. 5). The paramagnetic resonance in this diluted magnetic semiconductor shows a marked temperature dependence at low fields. Although the concentration is not sufficient to allow magnetic ordering using nearest neighbour coupling (the critical concentration for percolation is 21% in this crystal), the behaviour of the resonance shift and width with temperature is consistent with a spin glass ordering temperature of 0.5 K. The shift in the g-factor was found to be around 0.6% with a high temperature value of 2.004 [12]. The accuracy in the measurement of the g-factor was obtained by using the paramagnetic resonance of DPPH as a field marker.

Acknowledgements

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