Research groups

Nanoscopic studies of superconductors and other interacting electron systems

Professor Øystein FISCHER
MER Michel DECREOUX, Alfred MANUEL
Postdocs Louis ANTOGNAZZA, Morten ESKILDSEN, Isabelle JOUMARD, Edmond KOLLER, Olivier KUFFER, Martin KUGLER, Ivan MAGGIO-APRILE, Serge REYMOND, Shukichi TANAKA
PhD students Laurent BESSON, Cédric DUBOIS, Bart HOOGENBOOM,
Pascal REINERT, Emmanuel TREBOUX
Diploma students Estelle DE CHAMBRIER, Daniel GUTIERREZ RIOS
Technicians Paul-Emile BISSON, Jean-Gabriel BOSCH, Arthur STETTLER

Applications of superconductivity

Professor René FLÜKIGER
MER Eric WALKER
Postdocs Concetta BENEDUCE, Marc DHALLÉ, Vincent GARNIER Jean-Yves GENOUX, Enrico GIANNINI, Reynald PASSERINI, Igor SAVSYUYK, Hongli SUO, Pierre TOULEMONDE
PhD students Nicolas MUSOLINO, Michael SCHINDL, Grégoire WITZ
Technicians Patrick CERUTTI, Simon HUGI

Theory and numerical physics

Professor Bernard GIOVANNINI
MER Thomas JARLBORG
Postdoc Christophe BERTHOD

Specific heat and magnetocaloric effect of metals and superconductors

Professor Alain JUNOD
Postdoc Frédéric BOUQUET, Tomasz PLACKOWSKI, Ilya SHEIKIN
PhD student Yuxing WANG
Technician Aldo NAULA

High pressure physics of unconventional metals

Professor Jérôme SIERRO
MER Didier JACCARD
Postdocs Albin DE MUER, Ilya SHEIKIN
PhD student Alexander HOLMES
Technicians Renald CARTONI, Aldo NAULA

Growth and electronic properties of unconventional metal and oxides

Professor Jean-Marc TRISCONE
MER Didier JACCARD
Postdocs Albin DE MUER, Sarin KUMAR, Françoise LE MARREC, Thomas TYBELL
PhD students Stefano GARIGLIO, Alexander HOLMES, Daniel MATTHEY, Patrycja PARUCH
Technicians Renald CARTONI, Daniel CHABLAI
Diploma student Céline LICHTENSTEIGER

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Nanoscopic studies of superconductors and other interacting electron systems

Research summary

The group carries out research in the field of materials where strong interactions among the conduction electrons play an essential role. In this class of materials one finds superconductors, magnetic systems, and materials with other phase transitions. The group investigate such materials using two experimental tools: Scanning tunneling microscopy and spectroscopy on one hand and the fabrication and study of thin films multilayers and other artificial structures on the other. The scanning tunneling microscope (STM) is a powerful tool which offers the opportunity to perform imaging as well as spectroscopy with atomic resolution on conducting and semiconducting surfaces. Our group has a large experience in scanning tunnelling spectroscopy (STS) studies of high temperature superconductors, whereby we have uncovered some of their unusual electronic properties, such as gap values, pseudogaps features, and localised states in the vortex cores. Transport properties are also being investigated, operating the STM in scanning tunnelling potentiometry (STP) mode, to study the properties of manganites. Field induced conductivity in metal/ferroelectric heterostructures is also investigated using all these techniques. The group has also a long standing expertise in the fabrication and study of thin films and heterostructures. This year we have continued our study of the effect of interfacial strain and doping on Nd1-xBa2-xCu3O7+δ superconducting thin films. On the other hand we have continued the study of the abrupt transition at very high currents occurring in thin superconducting films. We have also investigated the use of such films in future high current limiters.

Spectroscopy of High-\(T_c\) Superconductors

When a strong magnetic field is applied to a type II superconductor, it starts to penetrate in the superconductor in the form of vortices. The behaviour of these vortices has direct consequences on the physical properties of these materials. The modification of the local electronic properties in high-\(T_c\) superconductors induced by the presence of vortices is not yet understood.

Previous investigations in our group indicated the presence of clearly discrete electronic states in vortex cores of YBa2Cu3O7-\(\delta\) (Y123), and of a pseudogap state (similar to what is observed above \(T_c\)) in vortex cores of Bl2Sr2CaCu2O8+\(\delta\) (Bl2212). More recent studies revealed weak discrete states (in addition to the pseudogap) in Bl2212 vortex cores as well.

The question is whether this unusual electronic behaviour can be understood in terms of conventional BCS theory, or if it is a direct result of the – still unknown – mechanism underlying high-\(T_c\) superconductivity. Our latest results on Bl2212 reveal a linear scaling between the energy of the electronic vortex states and the superconducting gap as shown in Fig. 1. This behaviour is rather different from the BCS theory, which predicts a quadratic relation. It further favours an interpretation in which the pseudogap plays an important role, since the latter follows a similar scaling with the superconducting gap. Adding the fact that our results are field-independent, these observations strongly question several theories which try to reconcile the observed vortex core states with a BCS scenario.


Spectroscopy of MgB2 Single Crystals

The year 2001 brought the surprising discovery of superconducitivity with a “high” \(T_c\) = 39 K in the otherwise well known compound, MgB2. Obviously, this initiated a surge of activity, which was only further increased when it turned out that MgB2 is also a two-gap superconductor.

We have performed STM/STS studies of single crystals of MgB2*. The measurements were performed on the surface of as grown crystals, at temperature between 270 mK and 30 K.
In fig. 2 we show a spectrum obtained at 270 mK, which shows a clear gap with coherence peaks at ±2.4 meV and additional shoulders at ±5.5 meV. This means that in our configuration, tunneling along the crystalline c-axis, MgB$_2$ appears essentially as a single-gap superconductor.

The manganite compound La$_{0.7}$Sr$_{0.3}$MnO$_3$ (LSMO) is well adapted for room temperature investigations ($T_c > 300$K). We use STP to map the distribution of the electrical potential induced by a current flow, and to probe the local electronic transport with a nanometer spatial resolution. Epitaxial LSMO thin films with different thicknesses deposited in situ on SrTiO$_3$ have been investigated. In all these films, we systematically obtained highly linear potential drops as shown in the image (b) of Fig.4, for film thicknesses down to 5nm.

Electronic phase separation (PS) has been predicted for hole-doped manganites, involving the coexistence of separated ferromagnetic-metallic and insulating microscopic phases. Contradictory to this, our STP measurements show an absence of electronic or chemical mesoscopic PS in LSMO at this scale. Further studies are under progress to probe the existence of a PS in other optimally doped manganites (such as La$_{0.7}$Ca$_{0.3}$MnO$_3$).

Nanosscopic Study of Ferroelectric Devices

The progress in thin thin film growth has allowed the realization of new electronic devices, such as high quality field effect devices based on complex oxides heterostructures. The use of ferroelectric materials for such applications is widely investigated because of their non-volatile and reversible polarization field.

We developed a novel approach using STS to study the local variation of the density of states induced in the conducting channel of a ferroelectric field effect device as a function of the polarization state. We discovered a transition from semiconducting to metallic behavior in the current-voltage (IV) characteristics of the channel when the polarization is switched. This result shows that the ferroelectric field effect can control the electronic properties in the surface layer of the device.
Fig. 5. Heart-shaped ferroelectric domain written using the STM. The IV curves in- and outside the domain are shown.

The distinct behavior between each ferroelectric state allowed us to use the STM tip to write and read small ferroelectric domain as shown in fig. 5. Domains with a size down to 20nm were written using this technique.

Fault current limiter and the behavior of high $T_c$ thin films at high current densities

In our previous report on the behavior of YBCO lines at high current densities, we have shown experimental evidences that a highly dissipative state (HDS) quickly nucleates (less than 1 μs) at a new critical current $J^*$ and then propagates along the line. During this year we have pursued our investigation on two main directions. First we bring more evidences for the existence of $J^*$ by performing numerical simulation of the HDS propagation. On the other hand we have improved the performance of fault current limiter thanks to a new design based on the modulation of the standard critical current along the superconducting meander. We are also setting an experiment for the local observation of HDS.

Numerical simulation

For the simulation of the thermal and the electrical behavior of YBCO/Au lines, a complete knowledge of the I-V curve is needed. We then measured these I-V characteristics at different temperatures and, by plotting the logarithm of the voltage as a function log $(J-J_c)$, we can easily extract the critical current and a unique power dependence $U=(J-J_c)^n$ for a given temperature. These measurements also allow to map the critical current and the $n$ factor of the different lines constituting a fault current limiter.

By using all these data within a 2D finite size element software, we have simulated the propagation velocities of the HDS as a function of the applied current. We have found, as shown in the figure hereafter, that our experimental results are very well fitted if the existence of $J^*$ is supposed, unlike the case of a thermal runaway.

Fault current limiter: new design

We have previously reported that, in a voltage source configuration, the initial length of the dissipative region, varies linearly with the applied voltage. From this characteristic we can define a critical field $E_c$ of the line; $E_c$=1.5$\rho$. $J_c$ in our geometry. This field $E_c$, which mainly depends on the gold layer, is one of the key parameters for the design of the FCL; it indicates which portion $L_c$ of the FCL will instantaneously switch into the dissipative state for a given voltage $U$, i.e. $L_c= U/E_c$.

A problem which can arise and influence the performance of a FCL is the localization of all the dissipated power in one part of the meander (or wafer). This can limit the maximal power sustained by one wafer and also the possibility of having a FCL based on several wafers in series or parallel.

We have solved this problem by a new design of the meander in which the initial length $L_c$ of the HDS is split and uniformly distributed along the meander. This is done by locally decreasing the width of the line (and therefore the critical current), i.e. by including constrictions in this meander as shown in the figure.
Using this new design we have successfully tested a 3kW (300V-10A) and a 5kW (300V-16A) FCL. The results show that the transition and then the dissipated power is homogeneously distributed in all lines of the meander. This allows the wafer to sustain 5 kW for a period of 100ms.

Local Observation of HDS.

The present report underlines the importance of the HDS in superconducting lines at high current densities. The origin of this state is still not clear so it should be interesting to locally study the HDS. During this year we have developed an experiment, based on a STM technique, where the tip is used to measure the electrical potential along a line of few mm length with a spatial resolution of about 100nm. This scanning voltage probe will work at low temperature and in magnetic field.

Effect of interfacial strain and doping on Nd$_{1-x}$Ba$_2$Cu$_3$O$_{7+δ}$ superconducting thin films

As a part of our effort to investigate heterostructures of cuprates and to study c-axis transport we have continued this year our efforts to understand the influence of interfacial strains on the superconducting and structural properties of such thin films. One of the most striking characteristics of the high-T$_c$ superconductors compounds is the low density of charge carriers involved in the superconductivity. It is well known that slight variations of this carrier density cause large changes in the critical temperature (T$_c$). One common way to act on the carrier density is to change the oxygen concentration. Another way to change the superconducting properties is by pressure, modifying the cells’ parameters. Pressure can either be applied externally or obtained by growing films under strain as has been recently observed, taking advantage of the lattice constant mismatch between the film and the substrate.

Using magnetron sputtering we have grown very high quality Nd$_{1-x}$Ba$_2$Cu$_3$O$_{7+δ}$ thin films (“x=0.13”; a=3.86Å, b=3.91Å, c=11.74Å and “x=0.3”; a=3.89Å, c=11.71Å) on both SrTiO$_3$ (STO, a=3.905Å) and LaAlO$_3$ (LAO, a=3.82Å) substrates. These films have been investigated by x-ray, AFM, STM, R(T) and Hall effect measurements. We measured T$_c$ as a function of the film thickness, ranging from 24Å to 1500Å (for the same growing conditions and oxygenation).

The results are presented in figure 1. As it’s clearly seen, the behaviour of T$_c$ is different for the two substrates up to large thicknesses and also for the two types of film. For the “x=0.13” films, we found that for the same film thickness the T$_c$ is lower for the films grown on STO than on LAO. T$_c$ also reaches the bulk value at a smaller thickness in films grown on LAO than in films on STO. In contrast, for the “x=0.3” films this situation is surprisingly reversed. One explanation for this behaviour might be the strain due to the lattice mismatch between the substrate and the film and also the difference in twin formation. Therefore we performed synchrotron x-ray diffraction measurements (ESRF, ID32) on these films. One result of these measurements is shown in figure 2.

What is clearly seen is the difference between a normal twin geometry signature for the 123 compound (h-k map of the (508) reflection, left) and of a “x=0.3” film (308 reflection, right).

Fig. 2: Comparison between a synchrotron x-ray diffraction h-k map of a “x=0.13” ((508) reflection, left) and of a “x=0.3” film (308 reflection, right).
Applications of superconductivity

Research summary: A prototype device for measuring strain effects on superconducting wires and tapes of up to 1 m length and at fields ≤ 17 T is now operational. The reaction to Bi,Pb(2223) inside Ag sheathed tapes was studied under high Ar pressures (up to 1 kbar), with unchanged p(O₂) = 0.07 bar: shorter reaction times and higher densities were observed, thus leading to an increase of Jc. As an alternative, Bi(2223) filaments without Pb have been formed inside Ag sheaths: the grain size increased markedly, up to 2 m in thickness and > 500 m in length. In contrast to Bi,Pb(2223), the connections are, however, of the weak link type. A new way has been found for obtaining biaxially textured, reinforced Ag/Ni ribbons of 30 m thickness, in view of their use in buffer-free Y(123) coated conductors. Finally, filamentary MgB₂ tapes with a Fe sheath have achieved Jc(4.2K, zero T) ≈ 10⁶ A/cm², which is the highest value reported to date. Due to the anisotropy, this value is even higher than that of dense, single phase MgB₂ samples which were formed at 1000°C and at pressures up to 3.5 GPa in our cubic anvil device.

New infrastructure

High pressure DTA prototype

A prototype high-pressure DTA apparatus was built for thermodynamic studies of the system Bi-Pb-Sr-Ca-Cu-O, in oxidising atmospheres. A new concept with a horizontal, three-zone geometry and Pt heaters allows to operate up to T=1200°C and p=200 bar. After calibration, the DTA is now operating for high-pressure phase diagram studies.

Jc vs. strain measurement on long length wires and tapes

In collaboration with B. Seeber (GAP) we have developed a new device, the so called Walters spring (WASP), for the measurement of Jc of superconducting wires and tapes under uniaxial strain and transverse fields up to 17 T. Conductors up to 1 m length, carrying currents up to 1000 A can be measured. Strain is applied by rotating one end of the spring against the other one.

Thermodynamic studies

Re-formation of Bi,Pb(2223) from the liquid (in-situ high-temperature neutron diffraction)

The reaction path commonly used to form the Bi,Pb(2223) phase inside Ag clad tapes involves a small amount of a local “transient” liquid. New reaction paths involving a larger amount of a stable liquid (closer to equilibrium) are needed to improve the Bi,Pb(2223)/Ag tape performance and to get an insight on the mechanisms occurring during the Bi,Pb(2223) decomposition. For this purpose an in-situ neutron diffraction study has been carried out. Both Bi,Pb(2223) pellets and Ag-clad tapes have been melted and cooled at different rates under various values of p(O₂). A refinement analysis has been performed on the full pattern profile. Direct crystallisation of Bi,Pb(2223) from the melt has been observed in Ag-sheathed tapes, while Jc has been partially recovered after melting and slow cooling, showing that the equilibrium formation of Bi,Pb(2223) can be performed. The appropriate conditions for Bi,Pb(2223) re-formation were found to be very sensitive to temperature, atmosphere, density of the sample and Pb mass losses.

Pb-free Bi(2223) bulk samples and tapes

The presence of Pb in Bi,Pb(2223) is known to accelerate the phase formation. Due to the Pb losses, however, the Pb-free Bi(2223) phase is still of interest, in spite of the known difficulties to synthesise it. We succeeded in forming a > 90% pure well-crystallised Bi(2223) phase in

Figure 1: Walters spring (WASP) of Ti₆Al₄V with a mounted Nb₃Sn superconductor

The conductor is soldered onto copper terminals at both ends of the WASP and can slide in a precisely machined groove. This allows measuring zero strain in the superconductor by a torque sensor.
the filament core of a Ag-sheathed tape after 100 hours reaction, starting from home-made precursor powders. Bi(2223) platelets as large as > 500 µm and 2 µm thick are found to grow at the Ag sheath interface.

![Graph](image)

**Figure 2:** Bi,Pb(2223) and Ca(2201) phase content as a function of time during partial melting and recrystallisation in Ag-sheathed Bi,Pb(2223) tapes.

**Hot Isostatic Pressure (HIP) processing of Bi,Pb(2223)/Ag tapes.**

One of the parameters of Powder In Tube (PIT) processed Bi,Pb(2223) tapes, which still leaves room for improvement, is the density of the filaments, being actually around 80-90%. To some extent this can be addressed with optimised intermediate deformation methods, such as periodic pressing, but this solution is limited by the necessity to heal the damage after deformation.

The synthesis of the Bi,Pb(2223) phase whilst applying a Hot Isostatic Pressure (HIP) has been found to be an attractive alternative route to obtain higher ceramic densities. High pressure was found to fasten the kinetics of phase formation. A thickness reduction and a density increase are obtained after HIP treatments, compared to the standard heat treatment at p = 1 bar. 20 h annealing under 100 bar improved the critical current density by 30% compared to the standard first heat treatment in the PIT process.

The positive effect of isostatic pressure was checked on tapes with different filling factors (7, 19 and 51 filaments) and different sheaths (Ag and Ag-Ni-Mg alloy).

**Synthesis of textured high Tc conductors**

**Mechanically reinforced (110)<011> textured Ag/Ni ribbons for coated conductors**

Silver is the only known metal, which does not react chemically with high temperature superconductors. Direct deposition of REBa$_2$Cu$_3$O$_{7-x}$ films on textured Ag substrates appears thus as a promising alternative method for the fabrication of coated superconductor ribbons. From the point of view of epitaxial growth, the orientation (110)<011> in Ag is the most appropriate one for coated conductor tapes. For industrial applications, Ag ribbons have in addition to be reinforced. We have produced a ribbon with a thin but textured Ag layer on a Ni or Ni-alloy core. The top Ag layer presents a pronounced (110)<011> texture. The introduction of a thin Cu foil as binding layer allows the deformation of the composite without intermediate anneals. The thickness of the outer Ag layers was as low as 7 µm, which corresponds to a reduction of the total Ag content to 25% and thus to lower overall costs. The new composites have markedly enhanced mechanical properties with respect to pure Ag ribbons. The yield strength $\sigma_{0.2}$ is 220 MPa for Ag/NiCrV ribbons, i.e considerably higher than the 30 MPa in pure Ag ribbons.

![Graph](image)

**Figure 3:** Tensile stress-strain curves for reinforced Ag composite ribbons and annealed pure Ag tape

**Y-123 films deposited by spray pyrolysis**

YBa$_2$Cu$_3$O$_{7-x}$ films have been prepared by using spray pyrolysis on both, (100) SrTiO$_3$ and (110)<011> Ag single crystal substrates at T > 850°C, using 0.3 M concentration solution of nitrates. A sharp ab plane orientation was observed in Y(123) layers deposited on SrTiO$_3$ substrates. So far, our samples exhibit $T_c = 91K$ and $J_c(77K) > 5 x 10^4$ A/cm$^2$ in self-field, calculated from VSM magnetisation loops.
using the Bean-Model. A considerable improvement of \( J_c \) is expected after further optimisation. Biaxial texturing of the deposited Y(123) film (\(< 5 \mu m \) thickness) was observed by EBSD analysis.

**High pressure/high temperature synthesis of superconductors**

The high \( T_c \) cuprate Cu-1234 is a very promising superconductor due to its high \( T_c \) value, \( > 117 \text{ K} \), its low superconducting anisotropy (\( \gamma = 1.6 \)) and its long coherence length along the c-axis (\( \xi_c = 1 \text{ nm} \)). However, its synthesis requires very high pressures. Since doping of (Cu,Tl)-1234 compounds with Ti was known to reduce this pressure, we prepared a series of samples in our multi-anvil press at 3.5 GPa and 950 – 1000 °C. The conditions for the formation of (Cu,Tl)1234 were determined.

High quality MgB\(_2\) samples for the study of physical properties were synthesized by using a high pressure/high temperature process. Dense pellets were prepared at 900 °C under a pressure of 3.5 GPa, starting from commercial MgB\(_2\) powder. Inductive measurements showed that the temperature and magnetic field dependence of the critical current density in bulk samples is almost identical to the behaviour of dispersed powders. This proves that grain boundaries do not introduce weak links, as for example in HTC cuprates. MgB\(_2\) bulk samples were also prepared by reacting fine metallic Mg and B powders in the range of 3 to 6 GPa at 1000 °C. The \( T_c \) and \( J_c \) values of the samples were compared to the behaviour of the sintered bulk samples. In collaboration with Professor Junod’s group, specific heat capacity measurements were performed to study the thermodynamically features of MgB\(_2\). Substitution of alkaline and transition elements for the Mg site were also tried. Finally, we are currently working on high-pressure growth (3 to 5 GPa, 1400 to 1700 °C) of MgB\(_2\) single crystals in BN crucible.

**AC losses in square Bi,Pb(2223) wires**

AC losses in square Bi,Pb(2223) wires were measured by a double Hall sensor at 77 K. The losses of an untwisted wire have a hysteretic behaviour, indicating monofilament behaviour, with coupled filaments. Such saturation behaviour is not observed in wires with twist pitches within 4 to 8 mm, thus suggesting decoupled filaments. In contrast to tapes, a resistive barrier between the filaments did not significantly change the AC losses. AC losses in Bi,Pb(2223) wires were found to depend on the aspect ratio of the conductor, as predicted by theory. Our data show that square wires have much lower AC losses when compared to those of tapes (with perpendicular fields), thus proving that the wire geometry is highly favourable for using Bi,Pb(2223) in AC magnetic fields. Bi,Pb(2223) wires with a twist pitch of 2 to 3 mm and a reinforced sheath could reach an AC loss level allowing their use in many applications, e.g. cables.

**Flux pinning in heavily Pb doped Bi(2212) single crystals**

A systematic study of pinning mechanisms in Bi\(_{2-\delta}\)Pb\(_{\delta}\)(2212) single crystals has been performed. Two types of pinning centers are suspected to govern flux dynamics in such crystals: oxygen vacancy clusters as point-like defects and Pb inclusions as extended defects. The role of Pb was shown by varying the Pb concentration from \( x = 0 \) to 0.8, whereas the effect of oxygen was studied by successive annealing under various \( O_2 \) pressures.

Pb inclusions appear at the nominal lead content \( x \geq 0.4 \) and have been tracked in our samples by TEM measurement (Figure 5). They show highly oriented and periodic lamellas with typical thickness of few tenths of nm throughout the whole crystal. This microstructure unambiguously enhances the pinning properties, i.e. the values of \( J_c \) in a self-field and the temperature dependence of \( J_c \) and \( H_{irr} \). The effect of both, Pb and O, are correlated through the H-T diagram, but a better theoretical basis is needed in order to get a deeper understanding of the physics.
involved. Another important feature is the occurrence of a second peak in the magnetisation loops, regardless of O or Pb doping.

Figure 5: TEM picture of a Pb0.6 crystal. \( \beta \) denotes the Pb rich lamellas structures and \( \alpha \) the ordinary 2212 phase (from Sara Bals, EMAT).

**Mechanical properties of single filaments extracted from Bi,Pb(2223) conductors**

Young’s modulus and fracture stress of single Bi,Pb(2223) filaments after removing from the tape assembly were measured at different steps of tapes preparation, using a 3-point bending test. These data were used for determining the thermal pre-compression as well as the irreversible strain limit and the tensile failure limit in the filaments. The tensile failure limit characterises the domain of non-destructive deformation in the polycrystalline ceramics. The differences in the modulus of rupture in tapes with different \( I_c \) values further confirm the relationship between mechanical and electromagnetic weak links.

The presently measured \( \varepsilon_{irr} \) values for single filaments are in full agreement with the values calculated by our \( I_c(\varepsilon) \) model and are of practical interest because they allow predicting the irreversible strain limit \( \varepsilon_{irr} \) in any tape.

**Superconducting MgB\(_2\) Tapes**

The recent discovery of superconductivity in MgB\(_2\) at 39 K has stimulated considerable interest in the area of basic and applied research on superconducting materials. High transport critical current densities have been reported on both of MgB\(_2\) bulk samples and films. To demonstrate the feasibility of MgB\(_2\) for eventual applications, Fe- and Ni-clad MgB\(_2\) superconducting tapes were fabricated using a powder-in-tube (PIT) process. We found that both the grain size of starting powder and the appropriate heat treatment are determinant for getting higher \( J_c \) values. Various heat treatments lead to an increase of the core density, resulting in narrower superconducting transitions. At the same time, \( J_c \) was raised by a factor of 10. As shown in Figure 6, annealed MgB\(_2\)/Ni tapes yielded \( J_c \) values up to \( 1 \times 10^3 \) A/cm\(^2\) at 4.2 K in a magnetic field of 6.5 T (2.3 \( \times 10^5 \) A/cm\(^2\) at 1.5 T). The \( J_c \) values in MgB\(_2\)/Fe tapes were higher, up to \( 10^4 \) A/cm\(^2\) at 4.2 K and 6.5 T.

The high electrical resistivity of the Fe matrix is at the origin of insufficient thermal stability: monofilamentary tapes quenched at the very high currents for low field values, so that a self-field value of \( J_c \) in these tapes at 4.2 K can only be extrapolated. Our value \( J_c(4.2 \text{ K, zero T}) \) is close to \( 10^5 \) A/cm\(^2\) and is the highest reported so far. Ni showed a limited chemical reaction with MgB\(_2\), leading to the formation of Mg\(_2\)Ni reaction layers between the filament and the matrix, while Fe showed no observable reaction with MgB\(_2\) and thus appears to be an adequate sheath material for the fabricating MgB\(_2\) tapes with a realistic potential for high-current applications.

Figure 6 : Field dependence of the transport \( j_c \) values at \( T = 4.2 \text{ K} \) in both as-rolled and annealed MgB\(_2\) tapes. For comparison, the transport \( J_c \) curve of a dense hot forged bulk sample is shown.

**Selected references**


Research summary: The small theory and numerical physics group in the Department has a task of working in close collaboration with the experimental groups and providing theoretical expertise, models and calculations relevant to the interpretation of actual experimental results. The group has specialized for a long time in band calculations for various complicated systems; in addition it has focussed its interest recently on developing a phenomenological model for underdoped high-temperature superconductors, based on the phase fluctuation picture of these materials, and which leads to reasonable agreement with STM and ARPES data.

Band structure of high-$T_c$ superconductors

Density functional calculations of the electronic structure of a high-$T_c$ superconductor, HgBa$_2$CuO$_4$, show that stripe-like spin waves and phonon distortions give gaps in the one-particle density of states (DOS). The energy positions of these gaps, which are partial pseudogaps for small perturbation amplitudes, depend on the wave length of the waves. It is found that some lattice distortions will increase the exchange enhancement considerably. In addition, a spin modulation of a certain wave length is able to generate a modulation of a charge density wave of half as long wave length. A phonon mode generates a similar charge wave, suggesting important spin-phonon coupling. Two gaps appears in the DOS, the largest induced by the spin wave at $E_F$ and a weaker below $E_F$ induced by the charge wave and a phonon mode that may be coupled to the spin wave. The figure shows an example for a cell extended 8 lattice constants along [1,0,0]. These results show that strong correlation is not necessary for stripes or pseudogaps, and they are consistent with many properties among the high-$T_c$ materials. In particular, they suggest that spin fluctuations coupled to phonons are important for the mechanism of superconductivity, since the opening of the gap shows that the matrix element for coupling is large.

Spin fluctuations in superconductors

Many recent reports of superconductivity in nearly magnetic systems have appeared in the literature. We have studied MgC$_x$Ni$_3$, ZrZn$_2$ and hcp Fe. The first one is found to have a large electron-phonon coupling, consistent with the observed $T_c$ near 8 K, while for reduced $x$ the DOS increases, $T_c$ drops and the band structure becomes spin polarized. Our calculations show that superconductivity in the latter two materials can be mediated by spin fluctuations. The DOS at $E_F$ is large in ZrZn$_2$, making both those and electron-phonon couplings large, but since superconductivity is reported in the slightly FM side of the magnetic instability, it is more likely that the spin fluctuations are responsible for superconductivity. The bcc-hcp transition in Fe at high pressure is close to an antiferromagnetic instability. The theory for spin fluctuations has been extended to study two possible AFM configurations in addition to the FM one. The results show that the FM fluctuations are much more important for superconducting pairing than the AFM ones. This would also explain the disappearence of superconductivity as the pressure is increased further.

Weak magnetism and impurity states

Continued search of weakly magnetic systems has been done by turning to Nb doping in WO$_3$. As for La doping in hexaborides, we find that there is a localization of the states around the impurity, and a weak magnetism of non-Stoner type can appear if the DOS variations near $E_F$ are sufficiently large. However, in the oxide it is the localized states of the oxygens that are active magnetically, not the Nb itself. We have also studied the case of a B$_6$ vacancy in the

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hexaborides, since independent calculations have reported magnetic moments surrounding such vacancies. We find that FM in that case is of standard Stoner type, caused by a peak in the DOS of nearby B-atoms, but that the moments disappear rapidly at increased temperature.

**Momentum densities**

Other projects involve calculations of momentum densities for several compounds, also for those mentioned above, for comparison with experimental Compton profile and positron annihilation data made by the group in Bristol. These works show in general a good agreement between measured and calculated Fermi surfaces, although it is sometimes possible to pinpoint some differences. A Stoner calculation of the pressure dependence of magnetic properties in SrRuO$_3$ show that thermal disorder is important for a good account of the Curie temperature.

**Density of states in vortices**

Elucidating the nature of the pseudogap phase in high-$T_c$ superconductors (HTS) remains one of the main issues towards the understanding of these materials. The most popular interpretations are that the pseudogap originates in some precursor mechanism of superconductivity, or that it reflects a competition between superconductivity and some different order in the underdoped part of the phase diagram. While several experiments indicate that superconducting fluctuations indeed exist well above $T_c$, providing support to the first alternative, it is also becoming clear that this mechanism alone is unable to explain the whole phase diagram, especially the strong doping dependence of the pseudogap temperature $T^*$.

The study of the electronic structure inside vortices may bring new insight into this topic. Indeed, vortices behave as windows through the superconducting phase, where it is possible to catch a glimpse at the underlying normal state. Furthermore, the vortices play an important role in scenarios based on $2d$ superconducting fluctuations, where the behavior above $T_c$ is due to vortex-like excitations of the complex order parameter. Recently, scanning tunneling microscopy (STM) measurements of vortex cores became available. The local density of states (LDOS) measured by STM shows distinctive features which cannot be explained by the BCS theory, nor, at this stage, by any of the competing theories of HTS. The interpretation of these experimental findings is widely viewed as one of the keys for the validation of a microscopic model. We have found that these STM spectra can be well explained using a semi-phenomenological extension of the BCS theory for $d_{x^2−y^2}$ vortices, which takes into account strong incoherent correlations in the superconducting state (figure).

Our calculations correctly reproduce most of the experimental observations. The LDOS at the core center shows two symmetric peaks about $E_F$, whose energy is roughly proportional to the main energy gap and whose amplitude is smaller in systems with a more pronounced pseudogap. These states decay exponentially outside the core over a distance of typically 5 lattice spacings, and show no four-fold anisotropy around the vortex. These calculations are now extended to other situations where the superconducting order is locally suppressed, like near surfaces, interfaces, and impurities.

The model assumes that incoherent correlations of yet unknown origin coexist with long-range superconductivity in the ground state of HTS, and that both types of correlations contribute to the single-particle excitation gap below $T_c$. We are currently investigating a microscopic theory that may justify our model.

Specific heat and magnetocaloric effect of metals and superconductors

Research summary: Our group specializes in thermodynamic studies of superconductors and new materials. We have built facilities allowing heat capacity to be measured with high accuracy or using minuscule masses, in high magnetic fields at low temperatures. Recent developments and projects include the magnetocaloric effect, specific heat under high pressure, and specific heat of thin films. We investigated the specific heat of MgB2 with a critical temperature $T_c \approx 38$K shortly after its discovery in early 2001, demonstrating that the existence of two gaps on the Fermi surface is a bulk, fundamental, and exceptional feature of this superconductor. We succeeded in increasing its upper critical field $H_{c2}$ by 60%, using a suitable irradiation process. The sensitive heat-flow device we developed last year was used to characterize the magnetic transitions of UAs up to 14 T. In the magnetocaloric mode, it disclosed the thermal counterpart of the magnetic "fishtail" effect in the high temperature superconductor NdBa2Cu3O7.

Thermodynamics of MgB2

Cuprate superconductors set the maximum achievable critical temperature well above 100 K. The discovery of the new superconductor MgB2 with $T_c \approx 40$K nevertheless arose intense interest, as it contains no oxygen, and since its normal isotopic effect points towards classic electron-phonon coupling. However, our thermodynamic measurements revealed large deviations from classical BCS behavior, which can be interpreted in terms of two-gap superconductivity. Band calculations, various spectroscopies, and ab-initio calculations agree with this. The key observation is a large excess (1-2 orders of magnitude) of the specific heat at $T \approx T_c/5$, in addition to a reduced specific heat jump at $T_c$.

The classical BCS curve is shown in yellow.

The abnormal shape of the zero field curve can be decomposed phenomenologically into two contributions, by assuming the existence of two bands, each having a different superconducting gap. One is of the order of $k_B T_c$, the other being smaller by a factor of $\sim 3$. The contributions of these bands can be calculated assuming classical hypotheses, save non-BCS gap ratios $2\delta/k_B T_c \neq 3.5$. The quality of the fit gives bulk support to the two-gap model.

The field dependence of the low temperature specific heat of MgB2 reveals another anomaly.

In contrast to BCS isotropic superconductors, the specific heat increases non-linearly with the magnetic field at $T \rightarrow 0$ (Fig. 2). The abnormal saturation of $C/T$ above $\sim H_{c2}(0)/2$ and the crossover to a different behavior near 10 K also follow from the complex gap structure, and lead us to anticipate vortex core expansion at $T \ll T_c$.

Although the relatively small upper critical field $H_{c2}\sim 16$ T limits the use of MgB2 for applications, we found that by a suitable irradiation process, a substantial increase of $H_{c2}$ can be generated, without sacrificing more than a few degrees in $T_c$ (Fig. 3). This is a bulk effect, as determined by scaling the specific-heat at low $T$ and at $T_c$.

![Fig. 1. Normalized electronic specific heat of MgB2 vs $t = T/T_c$, experimental data (black), two-band model fit (red), contributions from the large (blue) and the small (green) gap. The classical BCS curve is shown in yellow.](image)

![Fig. 2. Specific heat of MgB2 at $T \ll T_c$, showing a non-linear increase with the field due to the quickly vanishing smaller gap. Bottom to top: $B=0, .05, .1, .2, .3, .5, 1, 2, 4, 8, 14$T.](image)

![Fig. 3 The $H_{c2}$-$T$ diagram determined by the onset of the specific heat jump at $T_c$.](image)
Magnetic transitions in UAs

We developed a heat-flow device in 2000-2001, allowing measurements of the specific heat and magnetocaloric effect to be made in the same cell. In the calorimetric mode, the distinctive features are: high density of data (~30 per K), high resolution (10⁻⁵ to 10⁻⁷), ability to measure upon increasing or decreasing temperature (typ. ±1 K/min), in high magnetic fields (0 to >14 T), with good reproducibility (0.3-0.8%). The temperature range is limited to 30-300 K. A single crystal of UAs supplied by K. Mattenberger (ETH Zürich) demonstrated the possibilities offered by this device (Fig. 4). A 1st order transition occurs at the Néel temperature ~123 K, followed by reordering at ~62 K. In a field $H//<001>$, the upper transition splits and gives rise to an intermediate ferrimagnetic phase. The phase diagram in the $H$-$T$ plane could be detailed. Information on the hysteresis and entropy jumps was obtained.

Other specific heat studies made in 2001 using this device included Tb, NbH₀.₈₄, SrRuO₃, SrTiO₃, DyBa₂Cu₃O₇, and NdBa₂Cu₃O₇.

Specific heat of CePd₂Si₂

We have measured the specific heat of a heavy fermion single crystal CePd₂Si₂ in magnetic fields up to 16 T parallel to the crystallographic a-axis. This material orders antiferromagnetically at $T_N$=9 K. The transition manifests itself by a sharp, almost step-like increase in the specific heat. When magnetic field is applied, the transition is found to shift to lower temperatures, remaining, however, as sharp as in zero field (Fig. 6). This is in contradiction with a previous study (K. Heuser et al., Physica B 259-261, 392 (1999)), where no change of $T_N$ was found up to 29 T. This is most probably due to the strong magnetic anisotropy of this compound. An analysis of the field dependence of $T_N$ points to the existence of a magnetic quantum critical point at ~40 T where $T_N$ is suppressed to 0.

Magnetocalorimetry and the fishtail effect

The same device can also detect the magnetization heat $Q$ at constant temperature, yielding the isothermal magnetocaloric coefficient $M_T = (\delta Q/\delta B)T$, which is closely related to the magnetization $M$ through $M_T = -\langle \delta M V/\delta \ln T \rangle_B$. Runs across phase transitions confirmed the phase diagram established above for UAs. Experiments with the high temperature superconductor NdBa₂Cu₃O₇ (crystal supplied by T. Wolf, FZ Karlsruhe) demonstrated distinctive features of the magnetocaloric mode: linear relation between $M_T$ and $M$ for a paramagnet, ability to detect weak transitions (e.g. flux line lattice melting), and the study of hysteresis loops. In contrast to specific heat, this experiment gives access to irreversible energy transfers. An illustration is given by the "fishtail" effect in high temperature superconductors. In

the irreversible regime, sufficiently far below $T_c$, pinning mechanisms may cause a maximum of the critical current density at some intermediate field $H<H_c2$. This effect has been widely documented by magnetization experiments. As shown in Fig. 5, strikingly similar results can be obtained by measurements of the magnetocaloric effect. However, the magnetic field can be extended much beyond that available in SQUID magnetometers.

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Growth and electronic properties of unconventional metals and oxides

Research summary

Unconventional metals and oxides, including dielectric and ferroelectric insulators, ferromagnetic metals and superconductors, are studied using standard transport, local probe, and high pressure techniques. The materials investigated include single crystals, epitaxial thin films, and heterostructures. In epitaxial ferroelectric films, atomic force microscopy is used to study domain wall motion. Heterostructures of ferroelectric perovskites with superconducting or metallic oxides allow the realization of local field effect experiments using the nonvolatile ferroelectric polarization field, while ferroelectric / dielectric structures are used to design novel materials with tailored properties. High pressures are used to tune the electronic properties of heavy fermion systems, in particular close to phase transitions. The effect of strain in epitaxial oxide thin films is studied by applying very high hydrostatic pressures.

Domain wall creep in ferroelectric films

Ferroelectric oxides have been proposed as promising materials for diverse applications, including non-volatile memories, and ultra high density information storage. Epitaxial growth of high quality ferroelectric oxides has also allowed their incorporation into oxide electronics. Such applications, however, demand a thorough understanding of nanoscale ferroelectric domain behavior in these "novel electronic materials."

In our study, ferroelectric switching and domain dynamics were investigated by atomic force microscopy (AFM) in c-axis Pb(Zr0.2Ti0.8)O3 single crystal thin films, grown by RF magnetron sputtering on (100) Nb-doped SrTiO3 substrates. Domain arrays were written in uniformly prepolarized regions by applying voltage pulses across the ferroelectric, between the conducting substrate and the metallic AFM tip, and imaged using their local piezoresponse. Measurements of domain size versus writing parameters showed a linear size dependence on writing voltage, and a logarithmic size dependence on writing times longer than ~20µs. For shorter writing times, the domain size was found to saturate at ~40nm, a non-fundamental limit governed by the AFM tip size. These data reveal a two-step growth mechanism, in which initial nucleation is followed by radial domain wall motion perpendicular to the polarization direction. The electric field dependence of the domain wall velocity fits well to:

\[
V \sim e \frac{W_0 E_o}{k_B T E}
\]

We find the activation constant \(W_0 E_o/k_B T\) to be 1.3 MV/cm, 1.3 MV/cm and 0.5 MV/cm for 290Å, 400Å, and 800Å films, typically 10 times more than the applied fields during the polarization process.

This characteristic dependence of the velocity on an external driving force, in our case, the electric field, demonstrates that ferroelectric domain wall motion is a creep process, and that the key parameters controlling domain size are the confinement, strength and duration of the electric field pulses.

Electrostatic tuning of the hole density in ultrathin films of cuprates and its effect on the Hall response

We have used the polarization field of a ferroelectric to modulate the carrier concentration of an ultrathin superconducting layer in a reversible and non-volatile manner, an approach which does not modify the background disorder. In ferroelectric Pb(Zr0.2Ti0.8)O3 / superconducting NdBa2Cu3O7-δ bi-layers, we have directly observed the change in carrier concentration \(\Delta n\), \(\Delta n= \Delta P/ed\), with \(\Delta P=(P^+-P^-)\) being the change in polarization due to polarization reversal, \(e\) the electronic charge and \(d\) the superconducting layer thickness, by measuring the change in resistivity and Hall constant. In our ferroelectric material, \(\Delta P\) is temperature independent below room temperature. The induced carrier change \(\Delta n\) is thus temperature independent. Analyses of the
temperature dependence in the normal state of the resistivity \( \rho \) and the Hall constant \( R_H \), as a function of the polarization direction, show that the resistivity curves \( P^+ \) and \( P^- \) can be rescaled over the entire measured temperature range. This rescaling is expected if the resistivity is simply proportional to \( 1/n \), as in a free electron model. The same rescaling can be performed for the Hall constant data. We note that the rescaling is not affected by the opening of the pseudogap. This analysis suggests that the Hall constant is proportional to \( 1/n \) times a function of the temperature. This behavior is clearly different from what is observed in normal metals. A natural explanation of these rescaling properties of \( \rho \) and \( R_H \) can be found in the theoretical expressions proposed by Stojkovic and Pines [Phys. Rev. B. 55, 8576(1997)]. In this model, the resistivity and Hall constant are inversely proportional to a temperature independent carrier concentration, their temperature dependencies arising from the temperature dependencies of the scattering times.

\[\text{[More details can be found in S. Gariglio et al., Phys. Rev. Lett. 88, 067002 (2002)]}\]

**Electrostatic modulation of superconductivity in thin NdBa\(_2\)Cu\(_3\)O\(_7\) films: the role of classical fluctuations**

In epitaxially grown ferroelectric Pb(Zr\(_{0.2}\)Ti\(_{0.8}\))O\(_3\)/NdBa\(_2\)Cu\(_3\)O\(_{7-d}\) heterostructures, we have used the reversible ferroelectric polarization to electrostatically modulate the superconducting transition of thin NdBa\(_2\)Cu\(_3\)O\(_7\) films. Reversing the ferroelectric polarization induces a change in the superconducting transition temperature of up to 7K as shown in Figure 3 (for very underdoped films, a change from superconducting to insulating behavior is observed). If the change in transition temperature is due to classical fluctuations, the physical picture is that the change in carrier concentration induces a change in superfluid density and thus a modified Kosterlitz-Thouless transition temperature in 2D. For a sample showing a 1K \( T_c \) shift, Kosterlitz-Thouless analyses of the transitions have been used to extract the Kosterlitz-Thouless temperature \( T_{KT} \) for the two polarization states. From these values, one can obtain the change in the zero-temperature superfluid density implied by the change in \( T_c \). This agrees with the magnitude of the change in resistivity and with an estimate of the change in carrier concentration produced by the field effect. These results imply that the measured data agree with the theoretical picture that fluctuations control the establishment of phase coherence in the underdoped region of the temperature-doping phase diagram.

\[\text{[More details can be found in D. Matthey et al., to appear in Physica C.]}\]

**Magnetic behavior of epitaxial SrRuO\(_3\) thin films under pressure up to 23 GPa**

Applying hydrostatic pressure to thin films is an unexplored way to study the effect of large strains on the physical properties of thin layers. The main difficulty of such an experiment is the preparation of samples with the small dimensions required for high pressure techniques, without any degradation of the film. A thin film of ferromagnetic SrRuO\(_3\) has been pressurized up to 23 GPa using the Bridgman anvil technique. Figure 4 shows a picture of the cell before pressurization. The absolute resistivity, \( \rho \), was measured by the van der Pauw method between 300 and 1.2K.
The ferromagnetic Curie temperature, $T_C$, produces a kink in the $\rho(T)$ curve. This resistivity anomaly was used to follow the decrease of $T_C$ with pressure from 150K at P=0, to 77K at 13GPa. The rate of this decrease is similar to that of the bulk compound suggesting that the bulk moduli of the substrate and film are similar. Above this pressure, a striking saturation of the magnetic anomaly is observed. In $\text{Sr}_1-x\text{Ca}_x\text{RuO}_3$, where substitution by Ca$^{2+}$ acts as a chemical pressure (reduction of the cell volume), $T_C$ falls to zero at $x=0.7$ which roughly corresponds to a pressure of 16GPa. The much larger $dT_C/dP$ observed in $\text{Sr}_{1-x}\text{Ca}_x\text{RuO}_3$ suggests that the reduction of cell volume is not the only parameter influencing the decrease of $T_C$. The complex local magnetism found below 70K in $\text{CaRuO}_3$ could be related to the persistence of an anomaly at 77K in our resistivity measurements above 13GPa.

Ferroelectric based superlattices

Epitaxial heterostructures composed of two ferroelectric materials, $\text{PbTiO}_3$ and $\text{Pb(Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3$ have been grown by RF magnetron sputtering on specially prepared substrates with atomically smooth surfaces. $[(\text{PbTiO}_3)_x/(\text{Pb(Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3)_y]_N$ superlattices were first realized on SrTiO$_3$ substrates with a total thickness of ~550 Å. The individual thickness of the two materials was varied between ~30 and 120Å, and ~40 and 160Å for PbTiO$_3$ and $\text{Pb(Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3$ respectively. The superlattices were characterized by AFM and x-ray diffraction. The AFM studies revealed atomically smooth surfaces, with an RMS roughness of about 2Å, similar to that of the single crystalline substrates used. X-ray diffraction shows satellite peaks related to the artificial modulation and finite size effect reflections arising from the finite thickness of the sample.

A second set of superlattices $[(\text{PbTiO}_3)_x/(\text{Pb(Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3)_y]_N$ with a total thickness of ~1100 Å was realized, on top of a thin film of 300 Å of SrRuO$_3$, serving as a bottom electrode. Preliminary measurements reveal a different dielectric response of the artificial structures as compared to epitaxial thin films of either PbTiO$_3$ or $\text{Pb(Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3$.

[More details can be found in "Croissance et propriétés de structures artificielles à base de ferroélectriques", C. Lichtensteiger Travail de diplôme, Université de Genève, 2001]

Strain enhancement of superconductivity in $\text{CePd}_2\text{Si}_2$ under pressure

A superconducting phase has been found in the heavy fermion $\text{CePd}_2\text{Si}_2$ between 2 and 3.2 GPa around its magnetic instability, at $P_c=2.8$ GPa, where long range antiferromagnetic order appears at 0 K. The low energy spin fluctuations, enhanced at this so-called quantum critical point, are thought to be responsible for electron pairing. A comparison of experimental measurements on $\text{CePd}_2\text{Si}_2$ with similar compounds, and with spin fluctuation models, suggests the crucial role dimensionality has to play in the physics of this system. An exceptionally pure sample was separated into two pieces with similar properties. These two samples were set in a Bridgman anvil cell with the c-axis perpendicular and parallel to the load force direction. In addition to the pressure, a weak uniaxial stress is expected to be applied along this direction. The samples were connected for resistivity measurements, with calorimetric measurement possible on one of them.

Whereas stress applied along the a-axis does not change the properties in comparison to purely hydrostatic pressure, stress applied along the c-axis extends the whole phase diagram to higher pressure and enhances the...
superconducting phase both in its pressure range and in its maximum value at \( P_c \) (+40%). This change might be related to the suppression of the quasi two-dimensional behavior evoked earlier.

Combination of resistivity and calorimetric measurements shed some light on the enhancement of the effective mass of conduction electrons at \( P_c \), and its collapse for higher pressures. Calorimetric measurements have demonstrated the bulk nature of superconductivity.

Resistivity and calorimetric measurements on CeCu$_2$Si$_2$ in a He-filled diamond anvil cell

We have extended the measurement of resistivity and AC specific heat to the very high hydrostatic pressure conditions available in a solid helium filled diamond anvil cell, studying the heavy fermion superconductor CeCu$_2$Si$_2$. We concentrated on the superconducting transition itself, which is clearly resolvable both in resistive and calorimetric measurements performed simultaneously (see Fig. 6). As well as \( T_c \), we have been able to follow the evolution with pressure and magnetic field of the superconducting specific heat jump; the electronic specific heat coefficient, \( \gamma \); the resistivity temperature coefficient \( A \); and the residual resistivity, \( \rho_0 \).

The AC calorimetry technique is feasible down to ~100 mK, either with AC resistive or laser heating, though the cut-off frequency varies strongly with temperature, complicating both the experimental task and treatment of the results. CeCu$_2$Si$_2$ displays a very non-monotonic variation of \( T_c \) with pressure, and our results bear this out. The peak in \( C_p \) remained extremely sharp at lower pressures, even when \( dT_c/dP \) was large. Interestingly, as \( T_c \) starts to be reduced by high pressure, the peak broadens and then collapses. The resistive transition width does not seem to bear any relation to this, but follows \( dT_c/dP \). Our results do not agree well with \( T_c(P) \) previously measured in solid helium by susceptibility [Thomas et al., Physica B, 186-188, 303 (1993)]. We observed a lower maximum \( T_c \), disappearing at lower pressure. However, resistivity measurements carried out in Geneva using the quasi-hydrostatic Bridgman technique [Vargoz et al., Solid State Commun. 106(9), 637 (1998)] showed a more similar pressure dependence. With pressure gradients ruled out by the use of helium, sample inhomogeneity remains an important factor.

Superconductivity of pure iron at 22 GPa

Recently iron was reported to become superconducting below about 2 K and 15 <\( P < 30 \) GPa [Shimizu et al., Nature 412, 316 (2001)]. A ~10% resistivity drop and tiny Meissner signal were presented as evidence.

For two samples of different origin, pressurized at 22.2 GPa, we measured (just before Christmas) complete resistivity transition with \( T_c \) onset slightly above 2K. For \( P > 15 \) GPa, iron has an hcp structure and might be weakly or nearly antiferromagnetic at low \( T \). Superconductivity, possibly mediated by magnetic spin fluctuations, develops only in very pure samples (better than 99.99%) with electronic mean free path above a threshold value.

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Collaborations

Prof. C. H. Ahn
Yale University, USA

Dr. Y. Ando,
Central Power Industry, Tokyo, Japan

Dr. B. Barbiellini
Northeastern University, Boston, USA.

Dr. J. G. Bednorz
IBM Research Laboratory, Zürich

Dr. G. Behr
Institute for Solid State and Materials Research,
Dresden, Germany

Dr. J. Bindslev Hansen
Nordic Superconductor Technologies A/S,
Brenby, Denmark

Dr. D. Braithwaite, Dr. C. Marcenat
DRFMC, Commissariat à l’énergie atomique,
Grenoble, France

Dr. P.C. Canfield, Dr. V.G. Kogan, Dr. C. Petrovic
Ames laboratory and Department of Physics and
Astronomy, Iowa State University, Ames, USA

Prof. A.D. Caplin
Imperial College, London, UK

Prof. R. Chevrel
Université de Rennes I, France

Dr. S. B. Dugdale, Dr. G. Santi, Dr. A. Alam
University of Bristol, UK.

Dr. B. Dutoit
EPFL, Lausanne

Prof. C.B. Eom
University of Wisconsin Madison, USA

Dr. A. Erb
Walther-Meissner Institut, Garching, Germany

Dr. Denis Feinberg
LEPES, Grenoble, France

Prof. J. Flouquet
CENG, Grenoble, France

Dr. J. Fompeyrine
IBM Research Laboratory, Zürich

Prof. H.C. Freyhardt
Universität Göttingen, Germany

Prof. Dr. Albert Furrer
Laboratory for Neutron Scattering,
ETH Zürich and PSI Villigen

Prof. N. Gisin
Groupe de Physique Appliquée,
Université de Genève

Dr. R.E. Gladyshevskii
Dept. of Inorganic Chemistry,
L’viv State University, Ukraine

Dr. G. Grasso
INFM Genoa, Italy

Dr. B. Grévin
Commissariat à l’Energie Atomique, Grenoble, France

Dr. J.-C. Grivel
Rise National Laboratory, Roskilde, Denmark

Dr. Y.B. Huang
American Superconductor, Westborough, MA, USA

Dr. L.A.G.M. Jansen
Laboratoire des champs magnétiques intenses,
Grenoble, France

Prof. J.-L. Jorda, Dr. Ph. Galez
Laboratoire de Structure de la Matière,
Université de Savoie, Annecy, France

J. Jun, Dr. S.M. Kazakov
Physics Department, ETH Zürich

Dr. K. Kadowaki
University of Tsukuba, National Research Institute
for Metals, Japan

Dr. J. Karpinski
Laboratorium für Festkörperphysik
ETH, Zürich

Prof. P. Komarek, Dr. W. Goldacker
Forschungszentrum Karlsruhe, Germany

Dr. K. Kwasnitza
PSI, Villigen

Prof. D.C. Larbalestier
University of Wisconsin, Madison, USA

Dr. Leghissa
Siemens AG, Erlangen, Allemagne

M. Li
Kamerlingh Onnes Laboratory,
Leiden University, The Netherlands

Dr. J.-P. Locquet
IBM Research Laboratory, Zürich

Prof. J. MacManus-Driscoll
Imperial College, London, UK

Dr. Daniele Marré
INFM, Genoa, Italy
Prof. P. Martinoli  
Université de Neuchâtel

Dr. C. Meingast, Dr. T. Wolf  
Forschungszentrum Karlsruhe, Germany

Dr. A.F. Morpurgo  
Delft University of Technology, The Netherlands

K. Nørgaard, A.B. Abrahamsen, Dr. N.H. Andersen  
Risø National Laboratory, Denmark

Dr. X. Obradors  
ICMAB-CSIC, Barcelone, Spain

Prof. Y. Onuki  
Osaka University, Japan

Dr. W. Paul, Dr. M. Chen  
ABB Research Center, Baden-Dättwil

Prof. A. Perrin, Dr. M. Guilloux-Viry, Dr. O. Pena  
Université de Rennes I, France

Prof. N.E. Phillips, Dr. R.A. Fisher  
University of California and Lawrence Berkeley National Laboratory, Berkeley, USA

Dr. C. Renner  
NEC Research Institute, Princeton, USA

Dr. M. Salluzzo  
Universita di Napoli, Napoli, Italy

Dr. B. Seeber  
Groupe de Physique Appliquée, Université de Genève

Dr. A. Semeno  
Université Catholique de Louvain, Belgium

Prof. A.S. Siri  
Universita di Genoa, Italy

Prof. P. Stadelmann, Prof. P. Buffat  
EPFL, Lausanne

Prof. S. Tajima  
Superconductivity Research Laboratory, Tokyo, Japan

Dr. B. Ten Hake  
Technical University Twente, Enschede, The Netherlands

Prof. H. Ten Kate  
CERN, Geneva

Dr. P. Tixador, Dr. L. Porcar  
CNRS, CRTBT, Grenoble, France

Prof. H.W. Weber, Dr. M. Eisterer  
Atominsttitut der österreichischen Universitäten, Wien, Austria

Prof. F. Weiss  
CNRS, Grenoble, France

Dr. H. Wilhelm  
Max-Planck-Institut, Dresden, Germany
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