## Introduction

The Condensed Matter Physics Department (DPMC) in Geneva has a long tradition in the studies of metallic materials with unconventional properties. In the last decade the interest of the department has been mostly focused on the understanding and developments of high temperature superconductors. The comprehension of the physical properties of these materials, dominated by strong $e^-$ -$e^-$ interactions, is today one of the most important problems in condensed matter physics. This problem is particularly important because several classes of novel key materials, including colossal magnetoresistance compounds and ferro/dielectrics oxides, are also materials whose physics is dominated by low electronic densities and electronic correlations. The DPMC in Geneva has a long standing experience to produce high quality materials in the form of single crystals, thin films and heterostructures, and tapes. Such advanced materials are essential to understand the physics of the often complex compounds studied and to prepare for their applications. Important efforts are made to characterize the materials and, in particular, superconducting tapes and wires that will be key elements for applying superconductivity. The department has also a large expertise in the study of physical properties of such materials. Several top class high quality experimental set-ups, including high magnetic field magnetotransport, transport under pressure, and specific heat measurements are installed. The department has developed these last years a novel area of expertise in local probe approaches. These local probes include Scanning Tunnelling Microscopy and Spectroscopy, STM/STS, and Atomic Force Microscopy, AFM, that allow the characterization and study of materials on a nanoscopic scale. This report represents the research carried out at the DPMC, regardless of the source of funds. Part of this research was financed by the Swiss National Science Foundation project No 20-56975.99.

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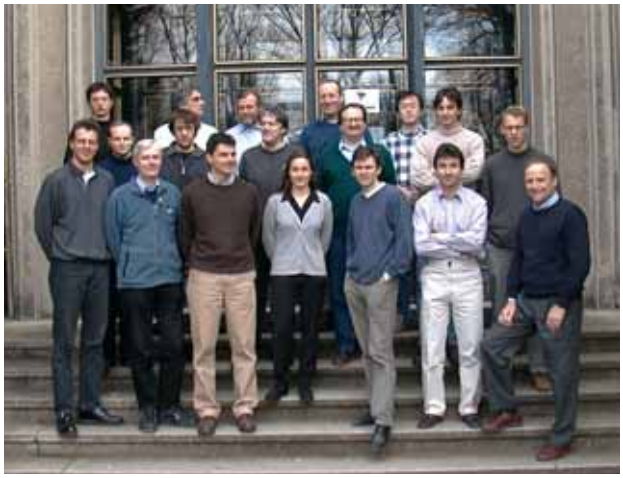
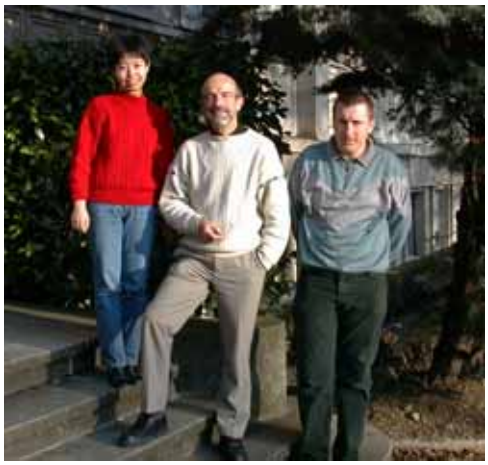


The Fischer group



The Junod group

The Flükiger group

The Sierro group

The Triscone group

The Giovannini group

Technical staff

Secretariat

## Research groups

### Nanoscopic studies of oxide superconductors and other interacting electron systems

| | |
|---|---|
| Professor | **Øystein FISCHER** |
| MER | Michel DECROUX, Alfred MANUEL |
| Postdocs | Louis ANTOGNAZZA, Morten ESKILDSEN, Benjamin GREVIN[1] Isabelle JOUMARD[2], Edmond KOLLER, Martin KUGLER, Ivan MAGGIO-APRILE, Shukichi TANAKA |
| PhD students | Laurent BESSON[3], Cédric DUBOIS[4], Bart HOOGENBOOM, Olivier KUFFER, Pascal REINERT, Serge REYMOND, Akihiko TAKAGI |
| Diploma students | Estelle DE CHAMBRIER, Daniel GUTIERREZ RIOS |
| Technicians | Paul-Emile BISSON, Jean-Gabriel BOSCH, Arthur STETTLER |

[1]until 31.10.00; [2]from 01.12.00;[3]from 01.11.00; [4]from 01.09.00

### Applications of superconductivity

| | |
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| Professor | **René FLÜKIGER** |
| MER | Eric WALKER |
| Postdocs | Emilio BELLINGERI[1],Concetta BENEDUCE[2], Marc DHALLÉ, Andreas ERB[3], Jean-Yves GENOUD, Marc LOMELLO-TAFIN[4], HongLi SUO, Pierre TOULEMONDE[5] |
| PhD students | Enrico GIANNINI, Nicolas MUSOLINO, Reynald PASSERINI, Michael SCHINDL, Grégoire WITZ |
| Technicians | Patrick CERUTTI, Simon HUGI |

[1]until 30.04.00; [2]from 01.04.00; [3]until 31.03.00;
[4]from 01.09.00;[5]from 01.09.00

### Theory and numerical physics

| | |
|---|---|
| Professor | **Bernard GIOVANNINI** |
| MER | Thomas JARLBORG |
| Postdoc | Christophe BERTHOD |

### Specific heat of oxide superconductors

| | |
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| Professor | **Alain JUNOD** |
| Postdoc | Tomasz PLACKOWSKI |
| PhD student | Yuxing WANG |

### High pressure physics of unconventional metals

| | |
|---|---|
| Professor | **Jérôme SIERRO** |
| MER | Didier JACCARD |
| Postdocs | Albin DE MUER[1], Ilya SHEIKIN[2], Heribert WILHELM[3] |
| PhD student | Alexander HOLMES |
| Technicians | Renald CARTONI, Aldo NAULA |

[1]from 01.10.00; [2]from 01.10.00; [3]until 31.08.00

### Correlated electron oxide structures : growth and electronic properties

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| Professor | **Jean-Marc TRISCONE** |
| Postdocs | Françoise LE MARREC[1] Thomas TYBELL |
| PhD students | Stefano GARIGLIO, Daniel MATTHEY, Patrycja PARUCH[2] |
| Technician | Daniel CHABLAIX |

[1]from 01.11.00; [2]from 01.08.00

## Nanoscopic studies of superconductors and other interacting electron systems

**Øystein FISCHER**

**Research summary:** The discovery of high temperature superconductivity in cuprates raised basic questions both about the superconducting state itself and about the nature of the normal state in these materials. On the other hand different phenomena observed in oxides have caught the interests of scientists like the collossal magnetoresistance in manganites. The group is carrying out research within this field using two experimental tools: Scanning tunneling microscopy and spectroscopy on the 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 very powerful tool to investigate local surface properties on a nanoscopic scale. Beside the possibility to image the sample topography with a spatial resolution reaching down to the atomic scale, it allows to probe the local density of electron energy states with an energy resolution of only a few meV, so-called scanning tunneling spectroscopy (STS). Furthermore, by applying a voltage drop on the sample, scanning tunneling potentiometry (STP) gives access to the local variation of the electric potential. Taking advantage of these exceptional possibilities, we focused our investigations on the local electronic properties of superconductors, manganites and ferroelectric heterostructures. The group has also a long standing expertise on the fabrication and study of thin films and heterostructures. In order to improve the quality of such artificial structures we have made detailed studies of interfacial strain and its effect on growth, surface morphology and physical properties. Another activity derived from our thin film studies has been to study the abrupt transition which occurs at very high current densities and to demonstrate the intrinsic nature of this phenomenon. This has important consequences for the functioning of future thin film based current limiters.

### Spectroscopy of Superconductors

A central issue to understand the mechanism of high-$T_c$ superconductivity is the origin of the pseudogap observed above $T_c$. The investigations of the local density of states (DOS) of the high-$T_c$ superconductor $Bi_2Sr_2CaCu_2O_{8+\delta}$ (B2212) performed by our group over the past years confirmed, that the pseudogap closes at $T^* >> T_c$ and revealed that it has a similar energy as the gap in the superconducting state ($\Delta$), below $T_c$. This suggested that the origin of the pseudogap might be related to superconductivity. In order to strengthen this hypothesis we investigated the parent $Bi_2Sr_2CuO_{6+\delta}$ (B2201) compound which has radically different superconducting parameters $T_c$ and $\Delta$, and studied the temperature dependence of the gap. Below $T_c$ = 10K, the spectra show a gap with well-defined coherence peaks at $\Delta \approx$ 12meV, which disappear at $T_c$. Above $T_c$, the spectra display a clear pseudogap of the same magnitude, gradually filling up and vanishing at $T^* \approx$ 68K.

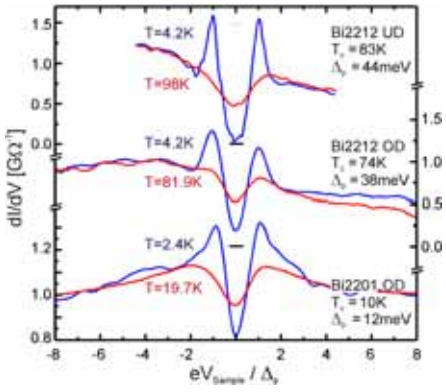



*Fig.1. Comparison between the pseudogap (red) and the superconducting gap (blue) for B2212 and B2201. The energy scale is normalized.*

Beside the remarkable observation that the general temperature dependence of the gap in B2201 is consistent with B2212, our results show that $T^*$ is scaling with $\Delta$. Furthermore, as highlighted in Fig.1, the pseudogap magnitude is similar to the superconducting gap for both compounds, although the respective $T_c$ and $\Delta$ differ by about an order of magnitude. These observations strongly suggest that the origin of the pseudogap is indeed related to superconductivity.

Another crucial issue is the doping dependence of the superconducting and pseudogap phase. Our investigations on Bi-cuprates show that the superconducting gap increases when reducing the doping level and furthermore, that the pseudogap phase is not confined to the underdoped regime, but that it also exists in the overdoped regime. In $YBa_2Cu_3O_{7-\delta}$ (Y123) this picture seems to be different. (a) The temperature dependence of the DOS of optimally doped Y123, did not show a pseudogap above $T_c$, and (b) the superconducting gap measured on underdoped Y123 appears to be smaller than on optimally doped crystals. These results rise many interesting questions which will need further investigations.

Relying on the successful vortex spectroscopy obtained on B2212 and Y123 in our group, we recently focused our interest on electron doped superconductors, like $Pr_{2-x}Ce_xCuO_{4+\delta}$. In contrast to the hole doped compounds which show a d-wave pairing symmetry, it is not yet clarified if these materials are d-wave or s-wave like. Comparing the characteristics of electron doped and hole doped superconductors might shed new light on the issues stated above and help to understand the mechanism of high temperature superconductivity.

In parallel to these studies, we analyzed the tunneling spectra of B2212 using computational modelling. As a result, we are able to distinguish the characteristics which are specific for the tunneling process from those related to high-$T_c$ superconductivity.

Closely related to the electronic properties of high- $T_c$ superconductors and to the nature of the quasiparticle pairing, we investigated the possibility to fabricate nanoscale structures on Y123 single crystals using

the STM as a nanolithographic tool. Successful nano-structuring allowed to characterize the etching conditions, thus achieving a better understanding of the etching process. Our future aim is the spectroscopic study of mesoscopic structures at low temperatures.

Finally, taking advantage of our sub-Kelvin STM which allows to perform STS studies down to 275mK at magnetic fields up to 14T, we have started to investigate magnetic superconductors of the borocarbide family like $TmNi_2B_2C$, with the objective to get a better insight into the complex interaction between magnetism and superconductivity.

For more details and further reading, see Ch. Renner *et al.*, Phys. Rev. Lett. **80**, 149 (1998), T. Timusk and B. Statt, Rep. Prog. Phys. **62**, 61 (1999), I. Maggio-Aprile *et al.*, J. Electron Spectrosc. Relat. Phenom. **10**, 147 (2000), M. R. Eskildsen *et al.*, Nature **393**, 242 (1998).

## Potentiometry of Manganite Films

The compounds showing Colossal Magneto-resistance (MR) have recently gained considerable interest, because of very promising technological applications such as magnetic sensors or magneto-electronic devices. However, the microscopic mechanisms responsible for their exceptional transport properties is far from being understood.

The manganite compound $La_{0.7}Sr_{0.3}MnO_3$ (LSMO), whose high Curie temperature is of the order of 300K, is well adapted for room temperature investigations. We use scanning tunneling potentiometry (STP) to map the distribution of the electrical potential induced by a current flow, and to probe the local electronic transport with a huge spatial resolution. Two classes of LSMO thin films have been considered. Textured films, which exhibit a low field inter-grain magneto-resistance, and epitaxial thin films, which show an intrinsinc high field magneto-resistance behaviour.
In textured LSMO thin films deposited on MgO substrates, the major result of our study is the evidence for sharp potential drops closely related to the grain boundaries (Fig. 2). A significant part of the electronic transport proceeds via a percolation mechanism through “electrically well connected” crystallites. Surprisingly, potential drops are present in the case of epitaxial films on MgO textured along the 3 axes, which have MR properties similar to the ones of single crystals. Our results suggest that in these low-resistive thin-films, the electric transport proceeds via a percolation process.

In epitaxial LSMO thin films deposited in situ on $SrTiO_3$, we systematically obtained highly homogeneous potentiometric images. We thus conclude that the narrow frontier between the textured-like and epitaxial-like transport regimes may be viewed as resulting from the existence of a percolation threshold for the current through the grain network.

The possibility of phase separation in manganese oxides is another attractive field of study. Theoretical studies have predicted a novel electronic state for hole-doped manganites, involving the coexistence of

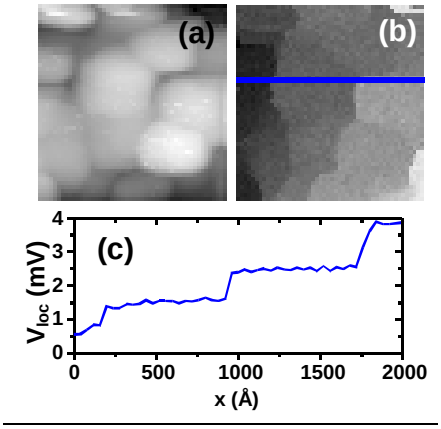



*Fig.2. Topographic (a) and potentiometric (b) 2000×2000Å images of a LSMO/MgO textured thin film acquired with an applied electric field of 17.6V/mm. gray scale: a) 18nm, b) 4mV. (c) Potential profile along the path in b).*

separated microscopic phases: one is ferromagnetic-metallic, whereas the other is insulating. Our STP measurements strongly suggest the absence of electronic or chemical mesoscopic phase separations in LSMO. Further studies are under progress to probe the existence of a phase separation in other optimally doped manganites (such as $La_{0.7}Ca_{0.3}MnO_3$.), and the possibility of strain induced mesoscopic phase separation.

For more details and further reading, see B. Grévin *et al.*, Phys. Rev. B **62**, 8596 (2000).

## Nanoscopic Study of Ferroelectric Films

The goal of this project is to investigate the feasability of studying and modifying ferroelectric domain structures in thin films on a nanometer scale by STM. We use the STM to measure the piezoelectric response of ferroelectric films and to study the polarization field effect in epitaxial bilayer heterostructures.

In a first step, we investigated the piezoelectric response in ferroelectric heterostructures. They are composed by a single crystalline ferroelectric film of $Pb(Zr_{0.2}Ti_{0.8})O_3$ (PZT) deposited on a conducting substrate and covered with an ultra-thin gold film. By applying a low frequency saw-tooth voltage to the bilayer and recording the inverse piezoelectric effect with the STM, we demonstrated the ability to measure the phase response as well as the ferroelectric switching.

The motion of the PZT layer as a function of the applied field gives a strain-field plot with a characteristic butterfly loop shape, showing both the inverse piezoelectric effect and the polarization switching of the ferroelectric material (Fig. 3a). Its numerical derivative provides a quantitative measurement of the longitudinal piezoelectric coefficient $d_{33}$ (Fig. 3b), yielding a value of 50pm/V. A striking result is that the coercive voltage and the shape of the local $d_{33}$ hysteresis loop perfectly match the macroscopic polarization hysteresis loop, although the nature of the two measurements is different (Fig. 3b).

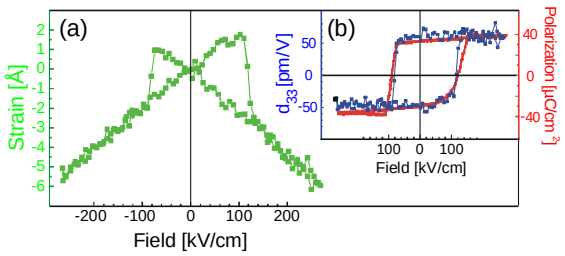



*Fig.3. (a) Strain-Field plot obtained with the STM. (b) Comparison of the $d_{33}$ and of the polarization hysteresis loops.*

In a following step, we will replace the gold electrode by a less conducting material in order to study the electronic doping of the electrode as a function of the polarization state of the ferroelectric material. Such investigations are promising for the local study of ferroelectric memory devices.

For more details and further reading, see O. Kuffer *et al.*, Appl. Phys. Lett. **77**, 1701 (2000) and the related work performed by the group of Prof. J.-M. Triscone.

## Effect of interfacial strain on $Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ superconducting thin films

In our effort to grow improved high precision heterostructures of cuprates we have concentrated this years effort on the study of interfacial strain between film and substrate on the physical properties of $Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ It was earlier found that under certain conditions this system grows without screw dislocations and it is therefore an ideal system for growing high precision heterostructures. However before studying such structures it is necessary to obtain a detailed understanding of the rôle of the inevitable strain at the interfaces.

Since the discovery of the high-$T_c$ super-conductors (HTS), there is a large effort in trying to understand the mechanisms of high temperature superconductivity. One of the most striking characteristics of the HTS 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 ($T_c$).The most common way to act on the carrier density is to play with the oxygen concentration. Recently, it has been observed that, by applying pressure, modifications in the distances between the atoms within and between the planes of these structures cause also drastic changes in the superconducting properties. Pressure can be applied externally, or obtained in a more subtle way by growing films under strain, taking advantage of the lattice constant mismatch between the film and the substrate. For example, it has been demonstrated that the $T_c$ of strained $La_{1.9}Sr_{0.1}CuO_4$ thin films could be raised far above the $T_c$ of the bulk[1].

We grow thin films of $Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$, a member of the 123 family which shows a transition from a tetragonal phase (insulating) at high temperatures or low oxygen content to a orthorhombic one (metallic and supercon-ducting) at low temperatures and high oxygen content. We synthesise these films by magnetron sputtering in an improved chamber which was recently built up. The deposition temperature is about 800°C and after the films are cooled down in a pure oxygen atmosphere to get the orthorhombic phase. In a first step we have grown films of $NdBa_2Cu_3O_7$ (a=3.86Å, b=3.91Å, c=11.74Å) on $SrTiO_3$ (a=3.905Å) and $LaAlO_3$ (a=3.82Å) substrates. For these films we measured $T_c$ as a function of the film thickness, ranging from 24Å to 1500Å [2] (for the same conditions of oxygenation).

As clearly seen in figure 1, the behaviour of the $T_c$ is different for the two substrates up to large thickness, above which the films are relaxed to the bulk values. Therefore it can be concluded that the physical properties of the thin films are changed by varying the lattice parameters. At low thickness, the 2D-dimensional character of these compounds may also play a role in the physical properties (Kosterlitz-Thouless transition). However by choosing a substrate which has a smaller mismatch with the film, the strain is expected to persist up to a thickness of about 50nm, well above the 2D-dimensional case.

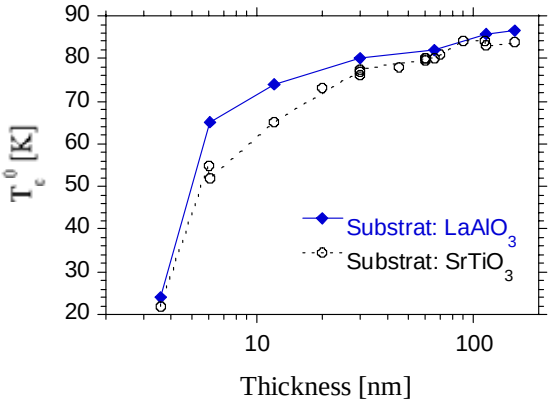



*Fig.1: $T_c$ dependence in function of the thickness of the $NdBa_2Cu_3O_7$ thin films for two substrates*

Another important point is to get more insight into the relation between strain and twins. Twins are domains with the property that the in-plane axes a and b are inverted compared to the neighbouring domains. These twins are formed during the cooling down by the symmetry breaking transition in the crystalline structure. We see by x-ray measurements that the twin geometry changes with the thickness. We are about to carry out detailed studies on the phenomena of the strain and twin formation of $NdBa_2Cu_3O_7$ thin films at the synchrotron in Grenoble.

Because the twin formation depends strongly on the oxygenation of the film, we measured by x-ray the c parameter as function of temperature and oxygen pressure. The reason is that the c parameter changes sensitively with the oxygen content. All measurement were carried out on relaxed films and in pure atmosphere of oxygen or argon. We observed that the in- and out-diffusion of oxygen are quite fast (about 20 minutes) and homogenous in the temperature range of 350°C and 450°C. Furthermore by choosing the appropriate oxygen pressure we can get every

oxygen-content and so any value of $T_c$. It is important to notice that films exposed to air behave rather different from such ones cleaned at high temperature in vacuum. The air contaminated films show the deoxygenation at a temperature that is about 100°C lower. This could be explained by layer of a hydrogen or carbon compounds that pumps oxygen. The composition of this layer has to be investigated.

### References

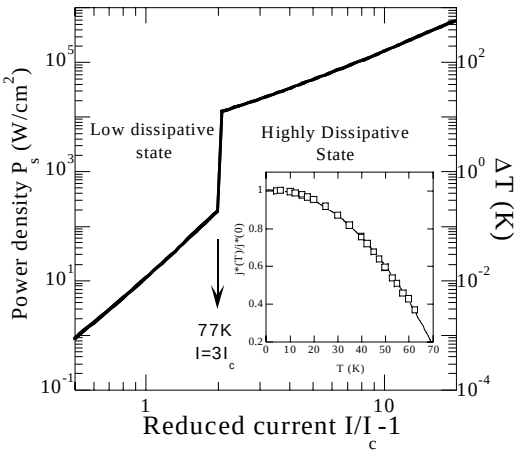

[1] J.-P. Locquet, J. Perret, J. Fompeyrine, E. Mächler, J.W. Seo & G. van Tendeloo, Letters to nature 394,453 (1998).
[2] E. Koller, P. Reinert and Ø. Fischer, to be published


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

Our previous studies carried out on the occurrence of a Highly Dissipative Domain (HDD) at large current densities have suggested that a new critical current density J* might exist. We have focused our attention on 3 important points; the existence of this new critical current, its possible origin and finally the consequences for the behaviour of a Fault Current Llimiter (FCL).

*Existence of J**

From the I-V data of a YBCO superconducting line, the power density vs current density was determined. An abrupt transition is seen at about 3 times the conventional critical current. Taking a mean thermal coupling of the YBCO film with the substrate of 1kW/cm$^2$ we have evaluated the instantaneous increase of temperature as shown hereafter. This proves that the occurrence the HDS does not result from an increase of the YBCO line temperature above $T_c$ .

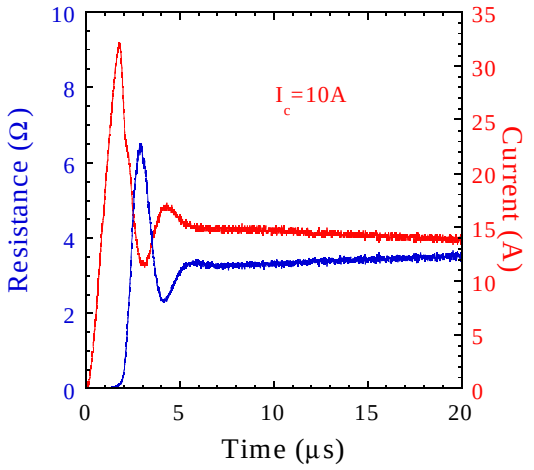



*Origin of J**

We have studied this transition as a function of the current in $NdBa_2Cu_3O_7$ and $Bi_2Sr_2CaCu_2O_8$ thin films. We have demonstrated that the breakdown of superconductivity is a two stage process: first a rapid (< 1 µs) nucleation of a small HDD and second its propagation. The HDD nucleates when the applied current exceeds the critical current j*(T). Its overall temperature dependence is shown in the inset of the figure above and can be well fitted by a power law: $j^*(T) \propto 1-(T/T_c)^{\alpha}$, with $\alpha$ ranging from 2 to 2.5. Such a law is close to what is found for the superfluid density, implying that j*(T) is related to some fundamental properties of the superconductor. In some cases, the HDD does not propagate right after its creation, and this allows us to investigate the nature of these domains. The I-V characteristics of a single HDD are non-ohmic, indicating that these domains are not in the normal state. In addition, the I-V characteristics are in agreement with a phase-slip model, developed by Scokpol et al. to explain voltage jumps in conventional superconductors. Within this framework the HDD are composed by a core where the order parameter is suppressed and a much wider region (about 7 µm long) where the quasiparticles created in the cores diffuse.

*Implications for a FCL*

As a current limiter operates in a voltage source configuration, we have studied the response of YBCO/Au lines upon voltage pulses up to 200V. We usually observe that the current is always limited after only 2-3 µs due to a fast increase of the line resistance and that the current peak never exceeds 3-4$I_c$ at 77K as shown on the figure hereafter .




After 5-10µs a quasi steady state is reached where the resistance of the line slowly increases with time. In this time domain, the resistance of the line, i.e. the length of the dissipative region, varies linearly with the applied voltage. In other word the current after 20µs does not depend on the applied voltage and is always of the order of 1.5$I_c$. This behaviour indicates that the length of the dissipative region adapts itself to limit the current to a value of 1.5$I_c$. This defines a critical electric field $E_c$=1.5.$I_c$.ρ≈25V/cm for our YBCO/Au line. $E_c$ is a key parameter to optimise the design of the FCL as it indicates the length of the FCL which will switch to the dissipative state for a given voltage. We used this knowledge to develop FCL demonstrators on 2" wafers. The design was optimised to favour the homogeneous appearance of the dissipative state across the whole wafer. A first demonstrator of a nominal power of 420W ($I_c$=6A and U=70V) has been successfully tested. This demonstrator has sustained several short circuit for periods as long as 10s. Based on the same design we then built two demonstrators of 2 and 3kW and we are currently testing the 2kW ($I_c$=10A,U=200V).

## Applications of superconductivity

**René FLÜKIGER**

### Research summary

Using a prototype high pressure DTA apparatus, we have shown that the Bi,Pb(2223) phase forms without loss at pressures of 200bar. Under these conditions, it was demonstrated for the first time that this phase can be reversibly reformed after a thermal cycle. This result is important, since it suggests that new reaction paths must exist, which may lead to substantially enhanced critical current densities. A study of the formation of Bi,Pb(2223) outgrowths inside Ag-sheathed tapes has revealed that it can be understood as a consequence of the nucleation and growth model, where a certain amount of Ag is dissolved in the transient liquid. Long, reinforced Ag ribbons with {110}<011> texture were formed, as a basis of biaxially textured Y(123) layers. A new model was developed for explaining the properties of Bi,Pb(2223) tapes under uniaxial mechanical stress, taking into account "connected-grains" domains. Strongly enhanced pinning was observed in a series of $Bi_{2-x}Pb_x$(2212) single crystals, starting already at Pb contents x=0.2. The temperature dependence of the $j_c$(H) improvements is not yet understood.

### New infrastructure

*Differential Thermal Analysis under pressure*

Some difficulties have been encountered with the DTA system under pressure (100 bar) built at the end of 1999. Under small partial pressure of oxygen the kanthal heater burned, which we solved using a platinum resistor. We also observed an anomalous melting temperature of pure gold. This anomaly depends of the pressure and nature of the gas (the deviation is smallest in pure He). We attribute this effect to the strong convection present in the actual vertical geometry of our DTA system. In the first measurements of the decomposition temperature of Bi(2223), we have corrected the observed changes using the calibration with pure gold. However, to overcome this difficulty, we have designed a new system with an horizontal geometry. The construction has started at the end of the year.

*Critical current under strain*

The study of mechanical properties of HTS tapes at the microscopic scale needed the development of a particular sample holder in collaboration with B. Seeber (GAP). The set-up is designed to be used in a split coil ($\mu_0 H < 7{,}5T$). The mechanical stress/strain parameters and the magnet are controlled with a computer. The longitudinal deformation is applied with a DC motor, stresses are measured with two force sensors near the sample and out of the cryogenic bath. The strain is given by an extensometer with four strain gauges clamped on the support (figure 1). Critical current versus field intensity, and orientation is measured with the standard four-point technique.

*Figure 1: Detail of the head of the sample holder with the extensometer clamped on the sample support*

*8K cryocooler*

In order to measure the temperature dependence of the resistivity without further having to charge our 17T He flow system, we purchased a 8K closed cycle refrigerator. The cryocooler was integrated in a 1T electro-magnet with the possibility to rotate the sample in the magnetic field.

By building the sample holder in a modular fashion, we will be able in the future to integrate new experiments within the same set-up. The first such experiment will be a Hall-sensor based local magnetometer.

### Crystal growth

*Heavily Pb-doped Bi(2212) crystals*

A serie of $Bi_{2-x}Pb_xSr_2CaCu_2O_y$ ($0<x<0.8$) single crystals has been grown by slow cooling in home-made $BaZrO_3$ crucibles. The growth process was entirely performed in a vertical 3-zone furnace with controlled atmosphere, in which the weight of the crucible is measured continuously with an accuracy of 1 mg. In this way we can detect the end of the decomposition of the starting materials and verify that we have no subsequent losses,

indicating that Pb stay in the melt. Pb evaporation is suppressed by the spontaneous formation of a (Sr,Ca)O cap layer which floats on the melt. To favor a directionnal solidification, we used a vertical temperature gradient of about 3°C/cm. Single crystals with size around 2 mm x 2 mm x 250 microns could be extracted from the solidified mass.
The measured Pb content in the crystals by Energy Dispersive X-ray analysis is in good agreement with the nominal composition indicating that lead continues to be incorporated by the crystals up to x = 0.8. Attempts to grow x = 1.0 single crystals were not successful, yielding only foreign phases.
The preliminary data of a single crystal X-ray study for x = 0.6 show that the crystal is inhomogeneous, containing inclusions with a size of at least 10 nm in agreement with the HRTEM results of Hiroi et al.

*Growth of RE(124) crystals*

In collaboration with Professor Junod's group (see present report), pure $YBa_2Cu_4O_8$ single crystals in the mm range were grown in $BaZrO_3$ crucibles under 1300 bar of pure oxygen.

## Thermodynamic studies

*High pressure thermodynamic investigation of the Bi-Pb-Sr-Ca-Cu system*

Transport properties of the Bi,Pb(2223) tapes do not yet reach their expected high potential, because of the difficulty to control the non-equilibrium route standardly used for processing. We have undertaken several experiments to better understand the high-temperature phase diagram and to try to form the phase from a stable liquid.

The formation of the Bi,Pb(2223) from the melt has been obtained after zone-melting of a pure Bi,Pb(2223) syntered sample and subsequent heat-treatment at 850°C in air, without any intermediate grinding. We thus demonstrated that Bi,Pb(2223) is an equilibrium phase. The parameters for its reversible formation have been established.
One of the reason for the previous failures in trying to re-form the Bi,Pb(2223) on cooling from the melt is the escape of some volatile elements at high temperature.
In the new High Pressure DTA and in a HIP furnace we performed a systematic study of the effect of the isostatic pressure on the decomposition temperature and on the Pb losses (figure 2).

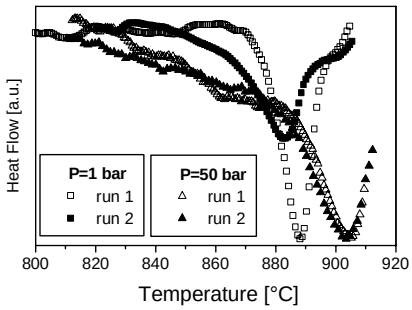



*Figure 2: DTA traces of two subsequent temperature ramps melting a Bi,Pb(2223) sample. A pressurised atmosphere renders the process reversible.*

The use of the high pressure was found to help to achieve a thermodynamic equilibrium in forming the Bi,Pb(2223) phase. Under high pressure the mass losses detected on heating above the decomposition temperature were very small (figure 3). The high isostatic pressure was found to prevent Pb from evaporating, and to keep the actual stoichiometry close to the nominal one. The temperature-dependent Pb-solubility in the Bi(2223) single phase region of the phase diagram has been elucidated.

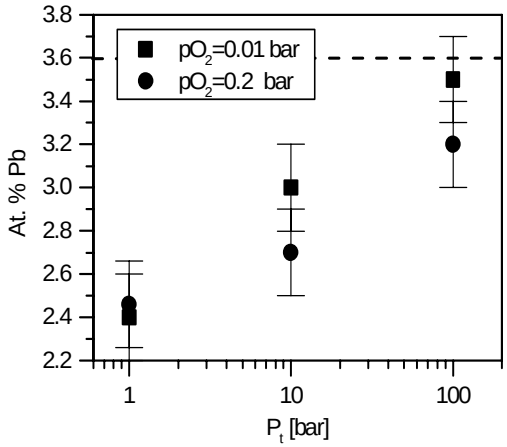



*Figure 3: Pb-content of Bi,Pb(2223) after heating above the decomposition temperature, plotted against the gas pressure in which the samples were treated.*

*In-situ neutron diffraction study of the phase transformations occuring in Bi,Pb(2223) conductors*

In order to understand the reactions involved at high temperature during the decomposition of Bi,Pb(2223), an *in-situ* neutron diffraction study was carried out.
Both Bi,Pb(2223) pellets and Ag-sheathed tapes were melted under different heating conditions and neutron diffraction patterns have been acquired all the time whilst. The pattern analysis will show how the melting of Bi,Pb(2223) occurs. The work has been done at the SINQ source (PSI – Villigen).

*The formation mechanism of outgrowths in multifilamentary Bi,Pb(2223) tapes*

Bi,Pb(2223) tapes are typically prepared in multi-filamentary form, both for enhanced mechanical strength and improved AC loss properties. Although the crystallites within the filaments show a high degree of c-axis texture, some of the grains commonly "stick out" of the filaments at relatively high angles (figure 4). When these outgrowths are long enough they can reach neighbouring filaments. Such filament bridging leads to super-current leakage between filaments, which can drastically enhance AC losses.

We studied the formation mechanism of such outgrowths, observing how their number, length and orientation evolve during the first heat-treatment of the PIT process. This micro-structural study showed that a) outgrowths are Bi,Pb(2223) platelets, b) they do not necessarily follow grain boundaries in the Ag matrix and c) they nucleate at fixed sites and then simply grow larger. The data strongly suggest that ceramic/Ag interface roughness determines their number, and the availability of transient liquid their size. All these observations are understood if one assumes that this transient liquid dissolves Ag.

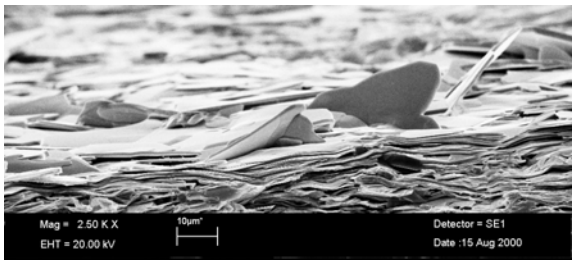



*Figure 4: SEM micro-graph of a break-surface of an isolated filament, showing both c-axis texture of the Bi,Pb(2223) platelets within the filament and outgrowths sticking out of the filament surface.*

## Synthesis of textured high Tc conductors

*Preparation of textured Ag substrates*

To get an enhanced critical current density in the Y(123) and Tl(2223) systems, a biaxial texture is mandatory. There is a commensurability between the Ag surface and the superconducting cell that allows a biaxial epitaxial growth on Ag. To be of industrial interest, long lengths of textured Ag ribbons have to be produced. We have shown that it is possible to control the texture formation of {100}<001>, {110} <112> and {110}<011> Ag ribbons. Preventing any oxygen contamination in the Ag ingot before the deformation produces a sharp {110}<011> texture after recrystallisation at 800°C with a misorientation angle of 10° as measured from EBSD maps.

For industrial applications, Ag ribbons have to be reinforced and the Ag thickness has to be as small as possible. Figure 5 below shows a composite ribbon with 8µm Ag layers on a 60µm NiCrV core. The interest of the process is to get a reinforced non magnetic core with a thin but textured Ag buffer layer by a standard deformation and recrystallization process.

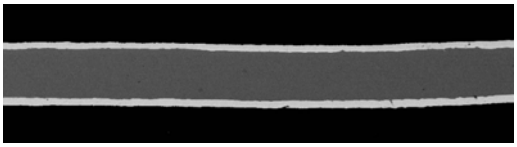


*Figure 5: {110}<011> textured Ag/NiCrV/Ag ribbon. The total thickness is 60µm.*

*Ultrasonic spray pyrolysis of $REBa_2Cu_3O_{7-x}$ conductors*

Spray pyrolysis is used in many industrial coating processes. The stoichiometry of the nitrate solution and the deposition parameters have been optimised in order to get pure 2-5µm thick Y(123) on $SrTiO_3$ substrates and {110}<011> Ag ribbons. As shown in figure 6, a sharp biaxial orientation is found in the superconducting films ($T_{c,on}$=92K) .

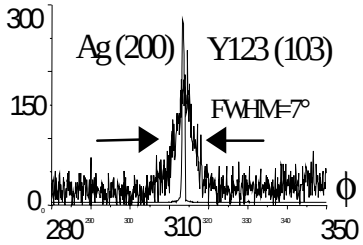



*Figure 6: $\phi$ scan of a Y123 film obtained by spray pyrolysis at 870°C on a {110}<011> textured Ag ribbon.*

*Optimised deformation control for Bi,Pb(2223) conductors by two-axes rolling under tension*

We further developed the new deformation technique of two-axes rolling under tension, which offers increased control of the ceramic density.

Optimising the parameters involved led to an increase of 25% in critical current density (tension 50 MPa) compared to a tape deformed without tension. When the tension is higher than 50 MPa, the deformation is no more homogeneous, resulting in a decrease of $j_c$. The application of tension doesn't change the final texture inside the filaments.

## Low temperature characterisation of high $T_c$ superconductors

*Magnetic texture measurement of Bi,Pb(2223) conductors at intermediate deformation stages*

By comparing VSM magnetisation loops measured using different field orientations with respect to the tape, it has been possible to determinate the average texture of the Bi(2212) grains in the precursor powder of multifilamentary Bi,Pb(2223) tapes at different stages of the deformation process. In particular, we studied the texture development during two axial rolling under tension. Contrary to expectations, we found no correlation between the texture development and the precursor density.

*The influence of Pb-doping on the pinning properties of Bi(2212) crystals*

We reproducibly prepared a series of $Bi_{2.2-x}Pb_x(2212)$ single crystals (x = 0.0 to 0.8) by slow cooling from a melt (see *Critical current under strain*). The jc(H,T) dependence of these crystals was inductively measured using a VSM. For *all* Pb-doped crystals we observed enhanced flux pinning, starting already at x = 0.2 and achieving quite drastic levels at x=0.6 and 0.8 (figure7). At these high doping levels the improved flux pinning increases the self-field $j_c$ value by a factor of at least 40 over the whole temperature range. Improvements in the field dependence of $j_c$ become only obvious above T ≈ 35K, reaching enhancements of $H_{irr}$ by a factor 50 at T = 70K. This distinct temperature dependence of the $j_c(H)$ improvements is at present not yet understood.

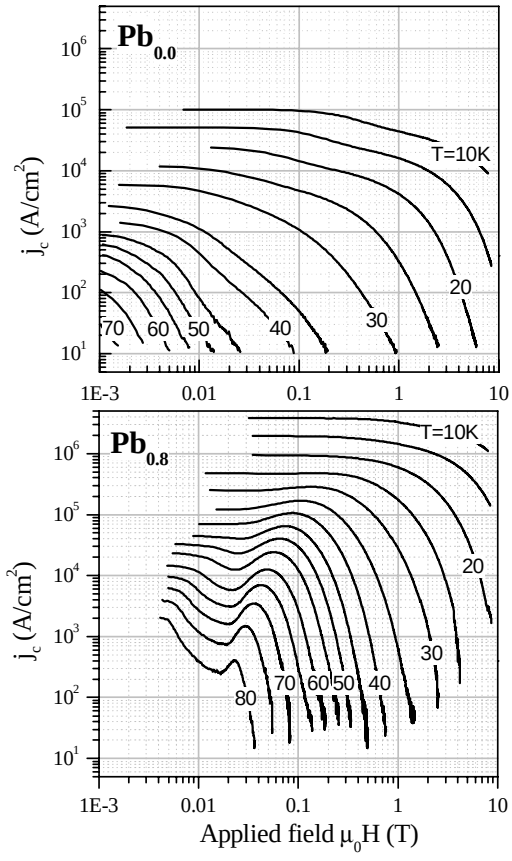



*Figure 7: Critical current density of a Pb-free (top panel) and Pb0.8 crystal (bottom panel) plotted against external magnetic field. Different curves within a panel represent different temperatures. Note that the logarithmic axes are the same for both panels.*

*The strain dependence of the critical current density in Bi,Pb(2223) conductors*

The mechanical properties of Bi,Pb(2223) tapes in longitudinal tensile experiments have been studied in order to enhance our understanding of this $I_c(\varepsilon)$ behavior, especially at low tensile strains ($\varepsilon < \varepsilon_{irr}$) where a strain insensitive plateau appears in the $I_c(\varepsilon)$ characteristic. High $j_c$ tapes with different filling factors have been measured and $\varepsilon_{irr}$ is compared to a detailed numerical calculation of pre-compression endured by the ceramic core. We proposed a modification to the standard "Irreversible $I_c$ Reduction Model" to take into account "connected-grains" domains in the $I_c(\varepsilon)$ behavior (figure 8).
Besides the theoretical determination of precompression we performed ultrasonic fracture experiments at room temperature and 77K to estimate qualitatively the stresses endured by the ceramic filaments at these temperatures.

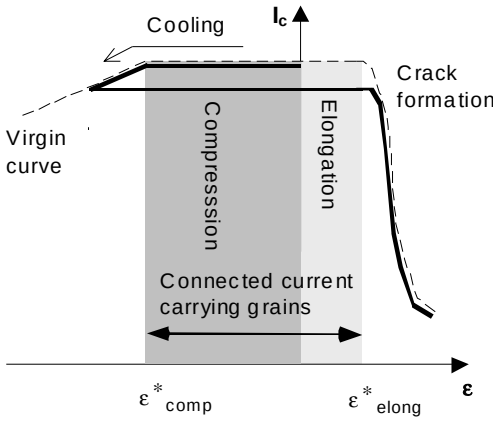



*Figure 8: Modified model proposed to take into account "connected-grains" domains in the $I_c(\varepsilon)$ behavior*

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## Theory and numerical physics

**Bernard GIOVANNINI**

### 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 developping 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.

### Theoretical studies on high-temperature superconductivity

#### Calculation of spin-waves and electron-phonon coupling in oxides

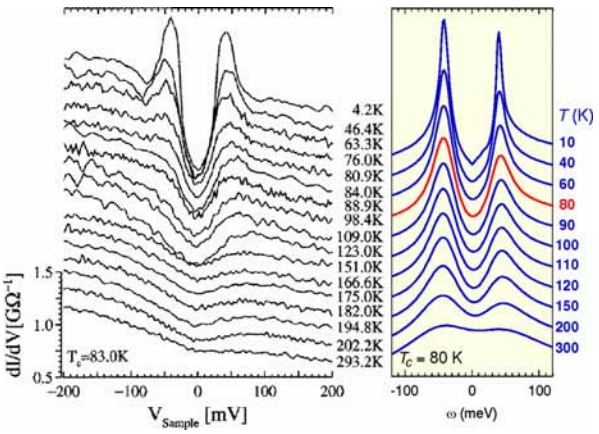
The roles of phonons and/or dynamic stripes in the mechanisms of High Temperature Superconductivity (HTS) are still very much debated. Our previous studies on high-$T_c$ materials indicated that some lattice distortions are relatively interesting for increased electron-phonon coupling, especially at large distortion amplitudes. This fact shows up in different changes of the Fermi surface topology when the lattice includes disorder. These studies continue with considerations of more realistic phonons, where "frozen" long-range disorder is introduced in supercells containing a large number of atoms. We also study supercells in calculations of static spin-waves by application of staggered fields. Preliminary results indicate that the sensitivity of the bands are similar for similar distributions of either distortions or fields, so that spin-fluctuations and strong electron phonon coupling may compete for the same wave lengths.

#### Phenomenological Gorkov equations for HTS

The various competing theories for HTS are still some way off the capacity of detailed calculations, especially in inhomogeneous situations, like vortex cores or the neighbourhood of impurities. We have therefore, based on the Kadanoff-Martin theory of superconductivity, developed a semi-phenomenological Gorkov equation relating the properties of HTS to the properties of the Cooperon propagator, and applied this theory to the calculation of the density of states (DOS) in the pseudo-gapped state and in the vortex cores below $T_c$.

The single particle DOS of the underdoped cuprate superconductors is depressed near the Fermi energy at temperatures well *above* the critical temperature $T_c$. The origin of this pseudogap is the subject of an active debate. A possible interpretation is that superconducting domains form locally above $T_c$, but the phases of these domains remain incoherent until the transition temperature is reached. We have translated this qualitative description into a phenomenological form for the Cooperon propagator, which treats the effects of strong local correlations and long-range phase fluctuations on an equal footing. With this theory, the excitation spectrum of a uniform *s*-wave superconductor was calculated numerically. We found that the sharpness of the coherence peaks in the DOS and the zero energy excitations are related to the strength and range of the correlations. The doping dependence of these correlations could explain the broadening of the quasiparticle peak observed in ARPES experiments as a function of underdoping. Our calculations also show that the ARPES and STM determinations of the pseudgap can provide apparently different results for the same system; such discrepancies were indeed found experimentally. We have also extended our approach to the case of a *d*-wave superconductor. The calculated spectra show a smooth evolution of the superconducting gap into a pseudogap across $T_c$, in good qualitative agreement with the experimental results (figure).

The pseudogap phenomenon manifests itself in many physical properties of the cuprates, in particular in the temperature dependence of the Hall coefficient and in the Josephson tunneling




Tunneling spectra across $T_c$. Left: experimental data from Renner *et al*., Phys. Rev. Lett. **80**, 149 (1998). Right: theoretical result based on the Cooperon propagator description of *d*-wave superconductivity.

spectra. We are currently elaborating our theory to investigate these properties, which will provide a crucial test for the validity of our interpretation of the pseudogap.
Describing the electronic excitations at the center of a magnetic vortex is a challenge for any theory of superconductivity. The calculations reported so far were all based on mean-field formalisms and failed to reproduce the experimental results in the underdoped cuprates. For inhomogeneous systems, microscopic calculations which include correlations beyond the mean-field level (e.g. with a Hubbard interaction) face considerable computational difficulties. With our approach, based on real-space correlations functions, the calculation for a vortex becomes tractable and a code has been developed for this purpose. Our preliminary results show that the effect of the strong local correlations is to suppress the zero-bias peak in the DOS at the vortex center, giving rise to a pseudogap-like excitation spectrum. This is consistent with the STM results in underdoped BSCCO.

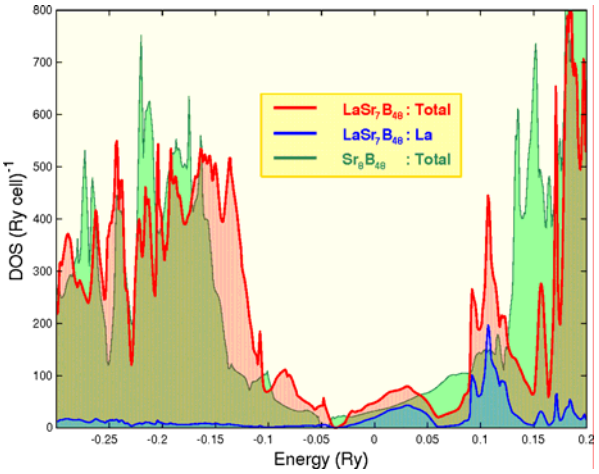
**Band calculations in other systems**

## Fermiology and calculation of momentum densities

Several of the manganite systems are so-called collossal magneto-resistance (CMR) materials, and they have been studied intensively in the recent years. There are reports that $La_xSr_{1-x}MnO_3$ shows a pronounced pseudogap in photo-emission experiments, suggesting that it has no Fermi surface (FS). We have performed local density calculations showing that the material is a metal. Compton profile and positron annihilation experiments, made by A. Alam and S. Dugdale at the University of Bristol, are in very good agreement with our calculated momentum densities, giving a clear indication of the FS. Among other systems that we have studied together with the Bristol-group are $LuNi_2B_2C$ and alloys of $Gd_{1-x}Y_x$. The comparison between experiment and theory has established the FS-sheets in these materials, and their importance for nesting and magnetic order.

## Magnetism in slightly doped systems

We have investigated whether band theory can explain the appearance of magnetic moments in La doped hexaborides, by doing density functional calculations for supercells of $M_xSr_{1-x}B_6$ systems with small amounts of dopants M. A minimal basis-set is used in order to study supercells containing up to 189 atoms. The band results show no hint of conventional magnetism, since the DOS is small and the Stoner factor is below the




limit for a ferro-magnetic instability. However, calculations for small $x$ (0.125 and 0.037) of M=La-doping, show that the La-*d* band becomes very localized, impurity-like. The DOS near $E_F$ changes significantly when one La is added as an impurity, as shown in the figure. Temperature and a spin-splitting will induce charge transfer effects. It is shown that Coulomb energies associated with the charge transfer, make a substantial contribution to the Stoner factor in the direction of a magnetic transition. The spin-polarized results confirm the stability of a small moment of the order 0.1 $\mu_B$ localized near to the impurity. A Sr-vacancy or substitutions with M=Al or In show no similar effect. The Al-*p* band hybridizes well with the Sr-band, leading to a rigid-band like situation, with no localization near the impurity. Other systems can be found that show localized states near the dopant in a host with low DOS. Some substitutions in oxides show a locally high DOS near the impurity, and supercell calculations indicate that a magnetic state is stable.

## Exchange and correlation potentials

Density functional potentials in band theory lead usually to good results for ground state properties, but there are a few exceptions, the case of insulating oxides being an important one. Reasonable exchange and correlation potentials can be obtained from solutions of a two-particle Schrödinger equation with proper boundary conditions and normalizations for exchange and correlation, respectively. This formalism can be extended to an electron gas of varying density by taking into account new boundary conditions. This leads to stronger exchange where the derivatives of the density is large, which favors anti-ferromagnetism in simple oxides.

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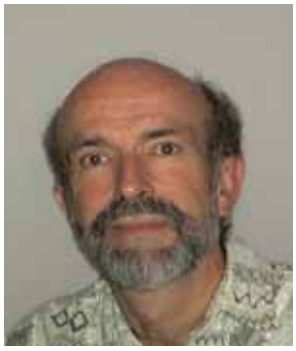
## Specific heat of oxide superconductors



**Alain JUNOD**

### Research summary

Our group specialises in thermodynamic studies of superconductors. We have built facilities allowing heat capacity to be measured in uncommon conditions: high magnetic fields, low temperatures, minuscule masses, and/or high accuracy. This equipment was used to study the melting line of the vortex lattice for two high temperature superconductors: $YBa_2Cu_3O_{6.94}$ in which oxygen vacancies are disordered in order to suppress their pinning properties, and $NdBa_2Cu_3O_7$ in which much of the usual Nd-Ba site exchange is avoided. On the technical side, we developed two new techniques in heat capacity measurements, one with very high resolution using heat-flow meters, the other aimed at thin film characterization. Finally, as a first step of a program on $YBa_2Cu_4O_8$, we succeeded in growing preliminary crystals in 1000 bar $O_2$ pressure using non-corrosive $BaZrO_2$ crucibles.

### Study of transitions in vortex phases in high magnetic fields

The field–temperature phase diagram of high temperature superconductors (HTS) has received much attention owing to the new phases formed by flux lines (or vortices). Their pinning properties are essentially different, which is of great importance for applications. The detection of the phase transition from a vortex solid to a vortex fluid by thermodynamic measurements (reversible magnetization and specific heat) allows a clear distinction to be made between the irreversibility line and the melting line, and between first-order (e.g. crystal/liquid) and second-order (e.g. glass/liquid) transitions. We investigated two superconductors, $YBa_2Cu_3O_{6.94}$ and $NdBa_2Cu_3O_x$. These projects involved a fcollaboration with laboratories in Garching/D (A. Erb, Y-123) and Karlsruhe/D (T. Wolf, Nd-123) for the crystal growth and in Grenoble/F (C. Marcenat) for fields 16-28 T. The Grenoble High Magnetic Field Laboratory (GHMFL) hosted our experiments in a 20 MW Bitter magnet (project Ref. SM3800).

### "No-fishtail" $YBa_2Cu_3O_{6.94}$ (Y-123)

We have shown how oxygen vacancies may or may not act as pinning centers in $YBa_2Cu_3O_{7-\delta}$. For a given concentration $\delta>0$, the vacancies may be clustered (ordered state) or dispersed (disordered state). In the clustered state, the pinning potential is large enough to cause a "fishtail" effect, i.e. a maximum of the critical current or magnetic irreversibility at intermediate fields $H_{c1}<H<H_{c2}$. The vortex solid is then disordered and melts with a second-order transition. In the dispersed state, the "fishtail" anomaly vanishes, and the vacancies no longer distort the vortex array, which can crystallize at low temperature and melt with a first-order transition. We observed *after* disordering this 1st-order transition with steadily increasing latent heat up to a field of 18 T, beyond which a critical point takes place and the melting transition abruptly turns to second order. This type of behavior is new, and may possibly display for the first time an intrinsic critical point which is not caused by impurity pinning. This research will be continued at the GHMFL in 2001 (project Ref. SM2101).

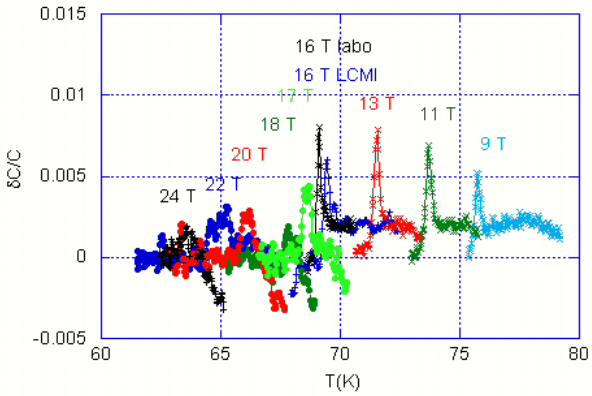



*Figure 1: Variation of the specific heat at the 1% level near the vortex melting transition in fields ≤24 T (optimally doped Y-123).*

### $NdBa_2Cu_3O_{6.97}$ and $NdBa_2Cu_3O_7$ (Nd-123)

We have detected the melting transition of the vortex lattice in $YBa_2Cu_3O_x$, $DyBa_2Cu_3O_7$ and $EuBa_2Cu_3O_7$, but $NdBa_2Cu_3O_7$ with a critical temperature of 95.5 K remained an exception, owing essentially to the pinning caused by Nd-Ba site exchange. By suitable metallurgic processes, this disorder has recently been removed to a large extent. The crystals being of a relatively large size (≈0.1 g), it was possible to observe in great detail changes of the magnetic and thermal properties in the vicinity of the vortex melting line, both in a slightly underdoped state with "fishtail" effect (x=6.97) and in an overdoped state with reduced pinning (x=7.00). A summary of extensive investigations is given by the phase diagram in the H-T plane (Fig. 2). We plotted curves assigned to the upper critical field (○), to vortex melting as detected from specific heat (●●) and from magnetization (●), and to the onset of irreversibility (◆). Together with planned thermal expansion and electrical resistivity

measurements, this set of data will help to assess the true origin of anomalies in various physical properties near the melting line.

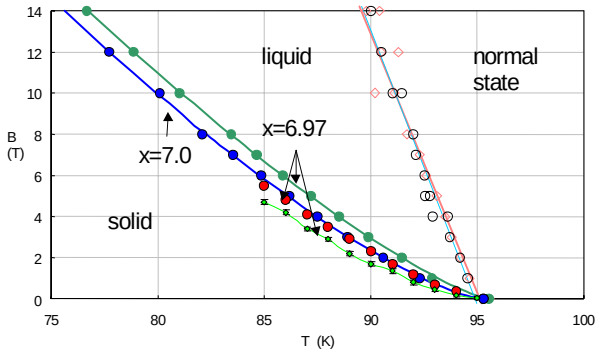



*Fig. 2: Vortex phase diagram in the field-temperature plane of $NdBa_2Cu_3O_x$ (see text for details).*

## Crystal growth of $YBa_2Cu_4O_8$ (Y-124)

$YBa_2Cu_4O_8$ is a 80-K superconductor for which the electronic doping level, an important parameter for all HTS, may be significantly changed by pressure, rather than by tuning the chemical composition, which is remarkably stable. This in principle opens up the possibility of studying the interplay between the pseudogap, the change in the dimensionality, and thermodynamic properties, using a clean reversible variable, the pressure. This project relies on recent developments in very high pressure specific heat measurements by F. Bouquet and D. Jaccard (cf. DPMC annual report, 1999). The first step is crystal growth in non-corrosive $BaZrO_3$ crucibles, which must be done at both high temperature (~1000 °C) and high $O_2$ pressure (~1000 bar). The requirements in mass and purity for specific heat measurements demanded substantial improvements of the oxygen autoclave. Examples of crystals grown up to now in collaboration with J.-Y. Genoud are shown in Fig. 3.

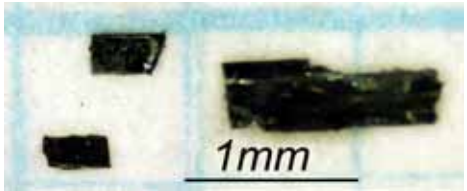



*Fig. 3: Crystals of $YBa_2Cu_4O_8$ (Y124).*

## Specific heat measurements with heat-flow detectors

There is a constant need to improve the resolution of calorimeters on both the specific heat and the temperature axes. Semi-adiabatic techniques with continuous heating, together with state-of-the art electronics limited by Johnson noise, allowed us to obtain data with ~0.01% scatter and 0.3 K spacing. These limits were improved significantly using a new concept. Rather than measuring temperature changes on the sample, we measure the heat flow to the sample with sensitive thermoelectric devices. The signal to Johnson noise ratio is improved by one decade, as well as the density of data on the temperature axis. Measurements are possible both upon heating and cooling. The sensitivity still increases in high magnetic fields. One of the differential prototypes is shown in Fig. 4.

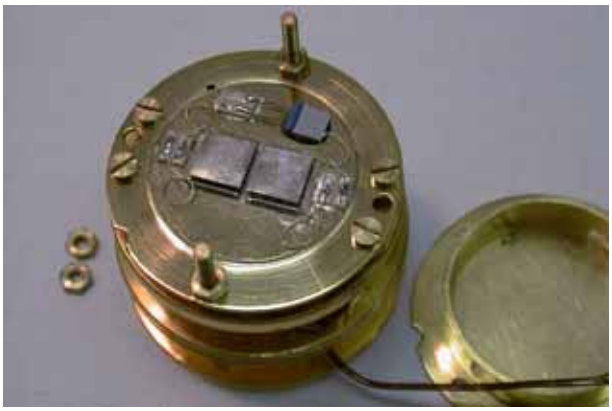


*Fig. 4: Prototype of differential heat-flow calorimeter.*

Fig. 5 presents a typical result for $GdAl_2$ exhibiting an antiferromagnetic transition at ~168 K. The insert shows the transition expanded over 2.5 K, demonstrating the high density of statistically independent points.

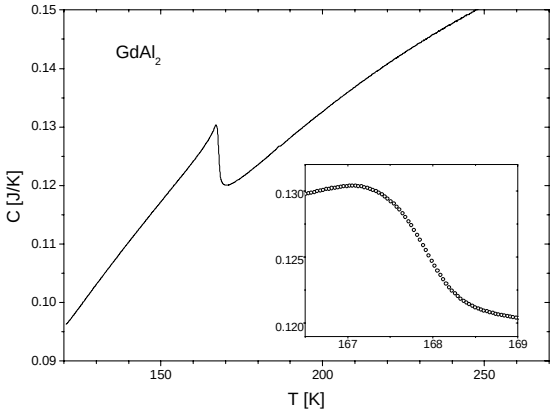



*Fig. 5: Heat capacity of a $GdAl_2$.*

## Specific heat of thin films

Physical properties of thin films attract great attention as they are often strongly modified with respect to the bulk form, opening the way to artificial materials. In collaboration with the group of J.-M. Triscone, we have performed preliminary experiments on substrate thinning, thin-film thermocouple design and laser heating.

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## High pressure physics of unconventional metals

**Jérôme SIERRO**

**Research summary**

Pressure is a powerful and clean parameter for investigating the physics of strongly correlated electron systems. To perform fundamental studies of such systems we use electrical transport and AC calorimetry as probes. A significant advance from last year concerns the new helium cell. Using our more usual technique we succeeded simultaneous measurement of the resistivity and specific heat of $CePd_2Ge_2$ up to 22GPa.

An elegant way to investigate the physics of strongly correlated electron systems is to vary the volume of a given sample by application of external pressure. In such systems, very high pressures, often of the order of 10 GPa, are needed to induce a significant change of electronic correlation. Our group has acquired considerable expertise [1] in the Bridgman anvil cell technique for generating such high pressures. Sophisticated pressure cells with up to 12 measuring electrical wires can currently be built. However, the existence of unavoidable pressure gradients in the Bridgman cell pushed us to develop a new generation of experiment with transparent diamond anvils, using highly hydrostatic solid helium as the pressure transmitting medium. Moreover, the aim is to continuously vary the pressure at low temperature even in the dilution cryostat. This will be done by using a helium pressurised bellow to transmit a force to the pressure device.

For future pressure studies, $CeNi_{2+x}Ge_{2-x}$, $CePd_{2+x}Si_{2-x}$ and $CePd_{2+x}Ge_{2-x}$ single crystals were prepared and characterised at $P$ = 0. For the Ni system, when $x$ = 0.025 the residual resistivity $\rho_0(x)$ is at a minimum, and resistive superconducting transitions were observed with $T_c^{onset}$ up to 0.2 K. But surprisingly, for constant $x$ = 0.025, complete transitions and highest $T_c$ were exhibited by samples with highest $\rho_0$. Such features agree with the charge-fluctuation scenario of superconductivity [2].

For $CePd_2Si_2$, we have studied de Haas-van Alphen oscillations at LCI, Grenoble. Experiments were carried out in magnetic fields up to 23 T and temperatures down to 30 mK. Five different frequencies were detected in the basal plane. The field dependence of the large effective mass $m^*$ is unprecedented. After the expected decrease by about 40% from 12 to 16 T, $m^*$ surprisingly started to increase, forming a pronounced maximum at 21 T. This behaviour implies a field induced phase transition, most probably of metamagnetic origin.

Let us briefly mention that we have succeeded in decreasing the substrate thickness of a thin film down to ~20 µm. Thus, pressure study of thin films should be feasible.

### Helium-filled diamond anvil cell (DAC)

Resistivity measurements have been performed in our DAC down to 1.2 K and up to 6.3 GPa in a $^4$He cryostat. At higher pressures the wires did not survive, but the cell has reached a pressure of 13 GPa. When force is applied at 4 K, the pressure inside the cell increases in a series of sudden jumps, which are absent at 300 K. These are reproducible, occurring at about the same force with several different gaskets. Remarkably, the wires are not compromised by these steps, at least at lower pressures. We have achieved a good empirical understanding of the force vs. pressure characteristics of the cell. The variation in pressure when the cell is heated from 4 K to 300 K confirms the $^4$He phase diagram [3] and indicates that helium is not lost when the temperature is cycled.

Recently, we have introduced the DAC into the dilution refrigerator. The sample shown in Fig. 1 was measured down to 50 mK at pressures up to 1 GPa. The superconducting transition was observed, with a width of 27 mK, indicating the excellent hydrostatic conditions, and the high quality of the sample. A magnetic field up to 8 T can be applied, and the ($H$,$T$) phase diagram of $CeCu_2Si_2$ has been explored to find the boundaries of the superconducting region and the A-phase at 0.2 GPa.

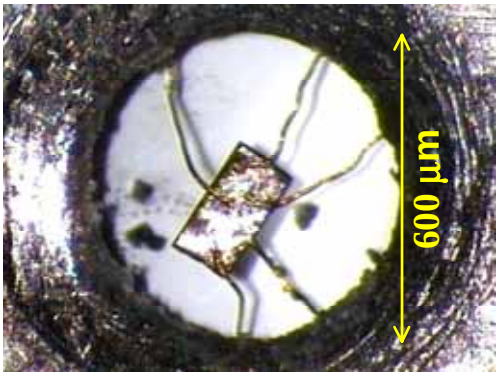


*Figure 1. DAC containing a 200×120×20 µm $CeCu_2Si_2$ crystal. Five 10µm gold wires have been spot welded to the sample, enabling two independent 4-point resistance measurements to be taken. The wires are insulated from stainless steel gasket by a layer of alumina powder and epoxy resin. Emission lines from ruby grains placed in the cell provide a direct measurement of the pressure.*

## Conventional Bridgman anvil cell

In collaboration with Prof. D. Jérome and C. Bourbonnais, we have further analysed data from the quasi-one-dimensional organic compound $(TMTTF)_2PF_6$. Our resistivity results establish the universality of the $(TM)_2X$ phase diagram (see fig. 2) with a single compound spanning all possible ground states [4]. The suppression of the spin-Peierls ground state makes the traditional phonon-mediated Cooper-pair formation unlikely to explain the existence of superconductivity at temperatures as high as $T_c$ = 2.2 K at $P$ = 4.7 GPa. The manifestation of critical SDW fluctuations above the onset of superconductivity and the close connection between their amplitude and the value of $T_c$ speak strongly in favour of an interstack mechanism mediated by exchange of these fluctuations between neighbouring stacks.

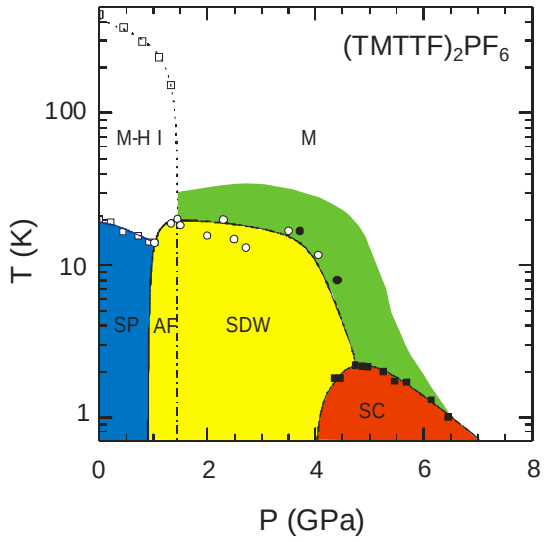


*Figure 2. (T,P) phase diagram of $(TMTTF)_2PF_6$. The spin-Peierls (SP), antiferromagnetic (AF), and the spin density wave (SDW) states are suppressed by pressure and a superconducting (SC) phase emerges above 4 GPa. AF spin fluctuations are present over a wide pressure range (green region). At high temperature, a Mott-Hubbard insulating (M-HI) and a metallic (M) state are observed. Open symbols represent data from the Orsay group. The match with our resistivity results is good.*

A success from last year is the determination of the resistivity $\rho$ and AC specific heat $C$ [5] of $CePd_{2+x}Ge_{2-x}$. In the same cell ($\phi$ = 1 mm), two crystals with $x$ = 0 and $x$ = 0.02 were connected in series for 4-point resistivity measurement. For the latter, an additional AuFe wire allowed the temperature oscillations of the sample to be read when an AC heating current was applied through the two resistivity voltage wires. Using the sample itself as a thermometer (after $\rho(T)$ measurement), calibrations of the Au/AuFe thermocouple were done at various pressures. The critical pressure $P_c \approx$ 12-14 GPa of $CePd_2Ge_2$ depends on $x$. The $\gamma$ coefficient of $C = \gamma T$ and the $A$ term of $\rho = AT^2$ show marked peaks at $P_c$. Only traces of superconductivity were detected near $P_c$.

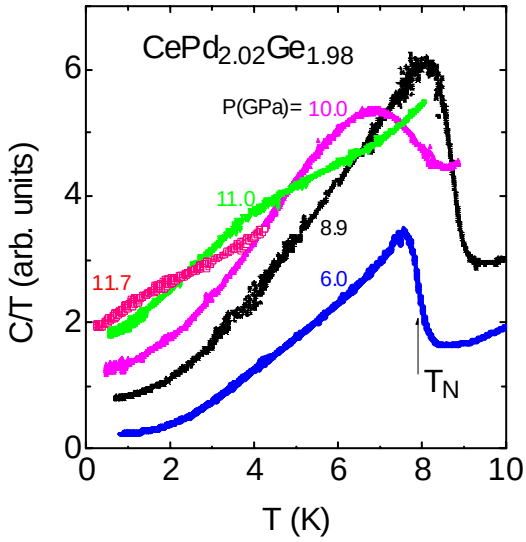


*Figure 3. The inverse lock-in voltage $V_{ac}^{-1} \propto C/T$ of $CePd_{2.02}Ge_{1.98}$ at selected pressures. The Néel temperature, $T_N$ ( 5.1 K at P = 0), reaches its maximum at 9 GPa. At higher pressure $T_N$ decreases. On approaching the magnetic quantum critical point at $P_c \approx 12$ GPa, signatures of magnetic ordering broaden. Above $P_c$, C/T becomes flat and decreases by a factor of about 5.*

In another cell, we have obtained preliminary resistivity (van der Pauw method), Hall effect, and AC specific heat data for a $CeCu_2Si_2$ single crystal. At 4.1 GPa, $\rho$ indicates superconductivity at 2 K, but the specific heat signature is observed only at about 1 K. In a magnetic field, the response of $C$ corresponds to that of $\rho$ as expected. At lower pressures results were less satisfactory, possibly due to contact problems.
Finally, we have tried to simultaneously measure the resistivity and the AC specific heat of the high $T_c$ superconductor $NdBa_2Cu_3O_7$ under pressure. The sample, characterised at P = 0 by the group of Prof. A. Junod, showed a sharp anomaly in $C$. So far, we have only tested it at 2 GPa. The resistivity revealed a broad superconducting transition with $T_c^{onset}$ = 98 K. The transition width is likely to be due to the pressure gradients and uniaxial stresses unavoidably present in a Bridgman cell. We have not succeeded in detecting a specific heat anomaly corresponding to the resistive transition. One might expect such an anomaly to be hidden within the noise level. A continuation of this work at higher pressure, where the electrical contacts will considerably improve, is an appealing prospect.

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## Correlated electron oxide structures: growth and electronic properties

**Jean-Marc TRISCONE**

### Research summary

Our research relies on epitaxial structures based on oxide materials and atomic force microscopy related techniques. Beside fundamental studies of ferroelectric and superconducting oxides, and more generally strongly correlated electronic systems, we have a special interest in heterostructures that combine ferroelectric perovskites and superconducting or metallic oxides. The motivation behind this combination of materials is to exploit the defining properties of ferroelectrics (the existence of a nonvolatile polarization field and a piezoelectric response) to study ferroelectricity itself and to locally probe the electronic properties of oxide superconductors. The same approach also allows the realization of electronic micro- and nano-structures.

### Scanning probe studies of ferroelectrics

In this project, we are studying high quality epitaxial ferroelectric films using scanning force microscopy. This combination of materials and techniques allows the controlled modification of the ferroelectric domain structure over nanoscopic length scales and is a promising approach to address fundamental issues such as ferroelectricity in two dimensions. These local probe modifications of the domain structure can also lead to the development of novel nano-devices, such as high-frequency surface acoustic wave filters.

The study of finite size effects in ferroelectrics has implications for both microelectronic applications and for a fundamental understanding of ferroelectricity. Recently we have shown by using a combination of electric force microscopy and piezoelectric microscopy that a 4 nm thick film of $Pb(Zr_{0.2}Ti_{0.8})O_3$ can sustain ferroelectricity at room temperature. To pursue this study, and in order to obtain quantitative information about the ferroelectric properties in low dimensions, we are currently studying the possibility of performing ferroelectric field effect experiments using extremely thin ferroelectric heterostructures. More precisely, the idea is to measure the effect of the polarization field on the resistivity of a very thin test layer, adjacent to the ferroelectric material, to extract quantitative information about the polarization. In order to obtain reliable results, the heterostructures surface roughness and interface quality have to be particularly well controlled. To this effect, we have studied the growth of $SrRuO_3$ (a stable metallic oxide that can be used for the test layer) and $SrRuO_3$ / $Pb(Zr_{0.2}Ti_{0.8})O_3$ heterostructures onto $SrTiO_3$ substrates with controlled surfaces. As shown in Figure 1, we have demonstrated that it is possible to grow atomically smooth heterostructures (on atomically smooth prepared substrate surfaces), showing only unit-cell steps overlarge areas. These results are extremely encouraging and suggest that the high quality ferroelectric heterostructures with controlled interfaces necessary for the studies discussed above are feasible.

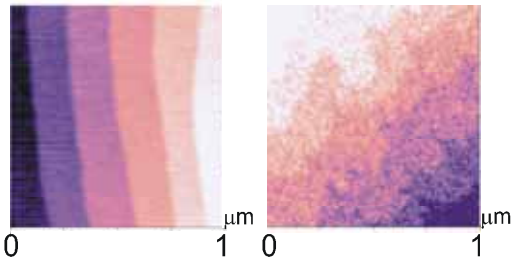


*Figure 1. AFM topographic images of, (left), an etched $SrTiO_3$ substrate showing atomically smooth terraces and, (right), a 5 nm thick $Pb(Zr_{0.2}Ti_{0.8})O_3$ film, grown onto an etched substrate and a thin metallic $SrRuO_3$ buffer layer. The height between each terrace is 0.4 nm, corresponding to one unit-cell.*

We are also studying novel ways to modify the ferroelectric polarization that should allow the control of the polarization over length scales corresponding to the minimum physical domain size.

### Evidence for a crossover in the pseudogap phase of underdoped $GdBa_2Cu_3O_{7-\delta}$ films

Among the anomalous normal state transport properties observed in high temperature superconductors, the temperature dependence of the Hall constant is probably one of the most striking: it displays a behavior which is totally different from the one observed in simple metals. Indeed, it is generally observed, in particular in $REBa_2Cu_3O_{7-\delta}$ (RE rare earth) compounds, that the temperature dependence of the Hall coefficient, $R_H(T)$, goes as 1/T over a wide temperature range and presents an ill-understood peak above $T_c$, whereas the cotangent of the Hall angle $\cot(\theta_H)=\rho_{xx}/\rho_{xy}$ (where $\rho_{xx}$ and $\rho_{xy}$ are respectively the longitudinal and Hall resistivity) varies as $T^2$ over a wide temperature range. In a series of underdoped $GdBa_2Cu_3O_{7-\delta}$ thin films, we have systematically measured the temperature dependencies of the Hall constant and Hall

angle. The main result is a well defined anomaly we find between $T_c$ and the pseudogap temperature T* : a deviation of the Hall angle from its $T^2$ behavior, that defines a new characteristic temperature T' and a new crossover line in the temperature versus doping phase diagram.
Figure 2 shows the phase diagram, temperature versus $R_H^{-1}$(100K), which summarizes the Hall effect results. The different lines correspond to the superconducting transitions (circles) (along with single crystals data from Ito *et al* (squares)) and to the observed deviations in $R_H^{-1}$(T) (triangles) and cot($\theta_H$)(T) (diamonds). The deviation in $R_H^{-1}$(T) can be related to the pseudogap temperature $T^*$, the deviation in cot($\theta_H$)(T) to the characteristic temperature T'.

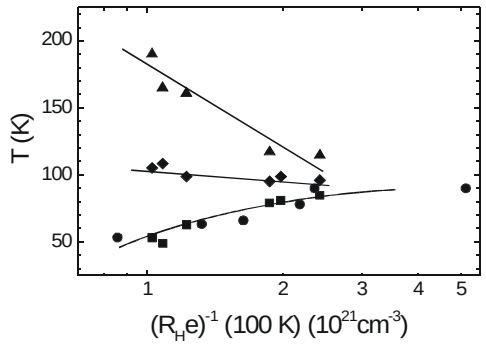


*Figure 2. Temperature versus doping phase diagram obtained from transport measurements. $T_c$ (circles), the pseudogap temperature T* (triangles) and T' (diamonds).*

The characteristic temperature T' might be related to recent reports of the presence of vortex like excitations in the pseudogap phase of $Bi_2Sr_2CaCu_2O_{8+\delta}$ and $La_{2-x}Sr_xCuO_4$ compounds. High frequency conductivity experiments and measurements of the Nernst effect have shown that vortex like excitations are found at temperatures up to 100-150 K (above $T_c$ but below the pseudogap temperature $T^*$ of these compounds). Since experimentally, the Hall angle has been shown to be sensitive to magnetic impurities, an interesting possibility is that vortex like excitations act as a parallel, temperature dependent channel for magnetic scattering modifying the Hall angle and indirectly $R_H$.

## Electrostatic modulation of the electronic properties of ultrathin films of cuprates

In this study, the idea is to use the polarization field of a ferroelectric film to modulate, in a ferroelectric/oxide superconductor bi-layer, the carrier concentration of the cuprate film. In the normal state, we pursued our studies of the Hall effect using lithographically patterned bilayer structures (100 nm $Pb(Zr_{0.2}Ti_{0.8})O_3$/ 10 nm $NdBa_2Cu_3O_{7-\delta}$). We successfully reproduced a purely electrostatic change of the carrier concentration controlled by switching the ferroelectric polarization with a metallic AFM tip. An important result is that the Hall coefficient and the resistivity are modulated with $\Delta R_H^{-1}/R_H^{-1}$ and $\Delta R/R$ essentially temperature independent. The new data additionally show that cot($\theta_H$)(T) is unchanged under polarization reversal. Taken together, these results suggest that the temperature dependence of $R_H^{-1}$ is not related to a change in carrier concentration in the 123 compound.

## Transport Properties of $La_{1-x}TiO_{3+\delta}$ doped Mott insulator thin films

In this project, in collaboration with IBM Zürich, we have studied the electronics properties of thin $La_{1-x}TiO_{3+\delta}$ (LTO) films deposited using a molecular beam epitaxy (MBE) system and a layer-by-layer deposition method. The doping level is tuned by setting the oxygen pressure during the deposition. We measured in detail the transport properties of lithographically patterned samples. The characteristic $T^2$ resistivity behavior observed in this compound is often assumed to be a result of strong electron-electron correlation. We have shown that it can also be understood if polaronic transport is considered. We find that our resistivity data can be perfectly fitted using small polaron metallic conduction as the transport mechanism. The fit to the data implies that the phonon mode coupled to the electrons has an energy of 80 K. Such a mode is indeed observed in Raman experiments on $LaTiO_3$. This soft mode is in fact characteristic of the tilt/rotation of the oxygen octahedra in perovskite materials and seems also to dominate the transport properties of other perovskite materials, such as the colossal magnetoresistance compounds. This new interpretation of the transport properties of $La_{1-x}TiO_{3+\delta}$ is in agreement with photoemission data obtained by Fujimori *et al.* [J.Phys.Chem.Solids 57,1379 (1996)], which suggest a polaronic nature for the charge carriers in LTO.

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July 2000

KUGLER Martin
*A sub-Kelvin scanning tunneling microscope and its first application of spectroscopy on $Bi_2Sr_2CaCu_2O_8$ and $Bi_2Sr_2CuO_6$*
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TAKAGI Akihiko
*Nanolithography on $YBa_2Cu_3O_{7-\delta}$ single crystals using scanning tunneling microscopy*
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TYBELL Thomas
*A scanning probe study on ferroelectrics: finite size effects & local electronic writing*
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## Diplomas

DUBOIS Cédric
*Spectroscopie à effet tunnel sur le composé supraconducteur à haute température critique $YBa_2Cu_3O_{7-\delta}$*
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## Licences

ROJAL Jean-Paul
*Spectroscopie par effet tunnel sur les supra-conducteurs BCS et HTS et recherche de l'interaction électron-phonon à partir des spectres tunnel par la procédure d'inversion de McMillan et Rowell*
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SCHMID Bernd
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