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Proprietà dei materiali superconduttori e della loro interazione coi raggi X

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Premessa

Aspetti teorico-pratici collegati a questa tematica sono trattati nel corso di:

FISICA DEI SUPERCONDUTTORI - [MFN0858, Fisica]

Mutuato da:

SOLID STATE PHYSICS - [CHI0027, Scienza dei Materiali]

- 5 CFU frontali + 1 CFU lab
- In Inglese (Master Europeo MaMaSELF)
- III periodo didattico (aprile-giugno)
What about friction in the micro-world?

- Here the lack of friction is the rule!!!

Indeed different physical laws hold (*Quantum Mechanics*): The key quantity is the Planck constant $h = 6.6 \times 10^{-34}$ J s

- *Quantum Mechanics* describes the electron motion around the nucleus

Traditional picture (inadequate): ORBITS

Quantum picture (correct): ORBITALS

In each ORBITAL electron energy is CONSTANT

E.g. for the H atom: $E = -13.6 \frac{1}{n^2}$ eV

During their motion around the nucleus, electrons never lose energy, which means that they move WITHOUT FRICTION
Is it possible to observe the same phenomenon on a macroscopic scale?

- YES, in SUPERCONDUCTING materials, that are the ones which show **LACK OF ELECTRICAL RESISTANCE**

- **1911:** Superconductivity discovered in mercury by Kamerlingh Onnes (Nobel prize in 1913). $T_c = 4.2$ K $\approx -269^\circ$C.

Too cold? It depends: average T of the Universe $= 2.73$ K

- Are the temperatures always so low?

  - Maybe
  - Maybe not

1986: Bednorz & Müller (Nobel prize in 1987)
Is it really with NO FRICTION?

In superconducting loops, current lasts for years with no power supply.

Electrical resistance (if any) is $10^{17}$ times smaller than in Cu \( R = 0 \)

Another surprise: the **Meissner effect**

- Superconductors ALWAYS expel the magnetic field: the presence of BOTH phenomena defines superconductivity. \( B = 0 \)

Lorentz force induces repulsion: Magnetic levitation
How is it possible?

- Unexpected event: Due to some interaction (e.g. electron-lattice), electrons in a solid can experience a net attraction force exceeding their coulomb repulsion.

- A bound state origins for two electrons at some distance: the Cooper pair.

- In a superconductor, billions of Cooper pairs exist in the same volume overlapping and crossing each other. They are in the same *quantum state*, forming a macroscopically extended superfluid.
Cooper pair tunnelling

- Josephson effect:

- Special behaviour: DC current induces NO voltage drop; DC voltage induces an AC current (AC effect)

- This behaviour is affected by the magnetic field: this can be exploited to measure it.

Its periodicity is related to \( \Phi_0 = \frac{h}{e} \)

The SQUID nominally can measure 1fT = 10^{-15} T, i.e. 10^{11} times smaller that the Earth magnetic field.
Examples of superconducting devices
All based on the Josephson effect (both sensors and digital circuits)

*Sensors*: Superconducting QUantum Interferfence Device (SQUID): very sensitive magnetometers (typically 2 pT).

Used in *Biomedicine* for Magnetoencephalography (MEG) and magnetocardiography (MCG), even for monitoring fetal cardiac activity.

Typically about 100 sensors, which means 300 channels

Very useful for brain tumor, Alzheimer’s disease, epilepsy diagnosis. Faster than MRI (10 msec per frame)
Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ (Bi-2212): an inherently anisotropic material

\[ a = 5.407 \text{ Å}, b = 5.432 \text{ Å}, c = 30.931 \text{ Å} \]


\[ T_c = 60–95 \text{ K}, \text{ depending on } \delta \]

practical interest

Intrinsic Josephson junctions

The crystal structure of Bi-2212 can be considered as a stack of Josephson junctions (JJ’s)

Direction of current flow

\[ \text{Direction of current flow} = c\text{-axis} \]

Each JJ has atomic spatial resolution

Direct AC Josephson effect (DC voltage bias → AC current)

Emission power limited to the pW range for a single junction → Arrays of JJ’s
THz radiation emission

- Experimentally detected by Ozyuzer et al.

- Resonant frequency proportional to the inverse of the width of the mesas (max = 0.85 THz for 40-μm wide mesa)
- Power increases with the square of the junction number

Emission mechanism still unclear
- Interesting for production of THz sources
Metrological interest

Inverse AC Josephson effect:
MW excitation induces voltage steps in I-V curves

S-I-S junction

Frequency accuracy ≈ 1 part in $10^{16}$

Voltage accuracy of 1 part in $10^{10}$ affordable!

Shapiro steps: $V = n \frac{h\nu}{2e}$ per junction

present low $T_c$ technology
(Nb-Al$_2$O$_3$-Al-Al$_2$O$_3$-Nb)

Towards a new technology based on high $T_c$ superconductors?
Stack fabrication principle in Bi-2212

- Starting block: whisker-like single crystal

What is necessary: i) electrical contacts on top surface  
ii) trench etching to force I

IMPORTANT ISSUES:
- Crystal growth and chemistry  
- Fabrication process
The traditional way: FIB etching

i) Photolithography or shadow masks

ii) Etching the trenches by FIB

Typical parameters for Ga ion beam:

\[ E = 30 \text{ keV} \]
\[ I = 10 \text{ pA} \]
A novel emerging way:

... and X-ray nanolithography

X-ray nanobeam at a synchrotron:

- Kirkpatrick-Baez system (two elliptical mirrors independently bent, one coated with graded multilayer)
- Flux \( \approx 1.9 \times 10^{11} \) ph/s
- Energy \( \approx 17 \) keV

Beam size \( \approx 152 \) nm (h) \( \times 107 \) nm (v)

In collaboration with ESRF
Synchrotron radiation in air at room temperature

Chip for combined «single-crystal» XRD and electrical measurements

- Series of: i) irradiation (XRF maps)
  ii) XRD
  iii) Electrical measurements

Equatorial series = (0 0 l) peaks

Changes of $T_c$ and resistivity

Change in O content: thermal origin?

Changes of the c-axis

Estimated $T_{\text{max}}$ in DC regime

Novel possible photolithographic process for oxides
Recent experiments

- Nano-beam: $57 \times 45 \text{ nm}^2$
  
  $E = 17.65 \text{ keV}$

- Dose effective in changing the current direction without destroying the lattice !!!

M. Truccato et al., Nano Lett. 16, 1669 (2016)
Ongoing developments

➢ In principle, this idea could work with many oxides

Preliminary indication: Local modification of TiO₂ properties.

Seo Hyoung Chang et al., ACS Nano 8, 1584–1589, (2014)

➢ $\Phi_0 = 2 \times 10^7 \, \mu m^{-2} \, s^{-1}, \quad E=8.3 \, keV$

We are going to test X-ray nano-lithography on TiO₂ !
How does it work?

Actually, nobody knows. But:

- Thermal origin should be considered in real pulsed conditions (FEM analysis of thermal load).

E.g: 16 bunch filling mode

For each bunch: \( 20 \text{ ps} < \text{FWHM} < 48 \text{ ps} \)
Temperature at the incidence point

Time behaviour

Spatial profile

Thermal spikes: ≈ 100 K for ≈ 1 ns

Possible role of O diffusion ???
Photoelectrons

- Photoelectron fluence of the beam computed with MCNP v6.0

Cylindrical mesh with 1 keV energy bins

Bi-2212

X-ray nanobeam

Electron fluence per source photon (#e\textsuperscript{-}/cm\textsuperscript{2})

- Photoelectron energy highly peaked at beam energy
From photoelectrons to i-O knock-out

Atoms displaced:

\[ N_{ad} = N_{o_i} \int_{E_c}^{E_0} \sigma_d (E) \Phi_e (E) \, dE \]

Mc Kinley-Feshbach cross section:

\[ \sigma_d = \frac{\pi r_0^2 Z^2}{\beta^4 \gamma^2} \cdot \left\{ \frac{T_m}{T_d} - 1 - \beta^2 \ln \frac{T_m}{T_d} + \frac{\pi Z}{137} \beta \left[ 2 \sqrt{\frac{T_m}{T_d}} - \ln \frac{T_m}{T_d} - 2 \right] \right\} \]

\[ T_m = 2E (E + 2mc^2)/Mc^2 \]

Fraction: \[ f_{ad} = \frac{N_{ad}}{N_{o_i}} \]

Very sensitive to \( T_d \)

\( T_d \) quite uncertain:

- O activation energy in the ab plane = 0.93 eV


- Binding energy of i-O atoms = 0.073 eV

Detailed experiment simulation

$T_d = 0.073 \text{ eV}$ - Crystal cross-section

$z=0$ (beam centre)  
$z=50 \text{ nm}$  
$z=100 \text{ nm}$  
$z=150 \text{ nm}$  
$z=200 \text{ nm}$

$\mathbf{f_{ad}} = \ldots$
Big toroidal fields obtained by means of cryogen free superconducting magnets.

\[ B = 1.5 - 2 \text{ T} \]

Expected stray field:
\[ B \approx 0.1 - 0.2 \text{ T} \]

- Stray field is also a concern: to be kept below 5 G = 0.5 mT
- Magnetic shielding is of interest also in MRI systems and for electronic equipment
Medium $T_c$ SC: MgB$_2$

$T_c = 40$K

working temperatures about 20-30 K (attainable with LNe or cryocoolers)

Almost insensitive to grain boundaries

Excellent workability (lower cost compared to HTSC)

Theoretical density = $2.63 \times 10^3$ kg/m$^3$

suitable for airborne applications

$a=3.09$ Å  $c=3.52$ Å
Sample preparation

1) Spark plasma sintering (SPS):
   MgB$_2$ powders (average grain size=2.3μm)  
   160°C/min ramp up to 1150°C for 3 minutes  
   Vacuum (30-40 Pa)  
   1600 A for 3ms, in a 12:2 pulse sequence  
   18 minutes per sample  
   only pellets feasible

2) Reactive liquid infiltration (RLI):
   Boron with graphite core and steel jacket  
   Mg/B 1.2/2 ratio in stainless steel box  
   650°C for 3 h plus 900°C for 20 h in Ar  
   Treatment at 2.45 GHz,1600 W for 30 min in Ar (optional)  
   Hollow cylinders feasible
Sample characterization

**XRD:** unreacted Mg in SPS samples

\[ T_c = 37.10 \text{ K} \]

Transition width \( \approx 0.5 \text{ K} \)

Gozzelino at al., J Supercond Nov Magn (2011)

Magnetic field distribution

**Experimental setup**

Table 1  MgB\(_2\) lattice parameters of the synthesized samples

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Sigma</th>
<th>95% Conf.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)-axis (Å)</td>
<td>3.08611</td>
<td>0.00013</td>
<td>0.00037</td>
</tr>
<tr>
<td>(c)-axis (Å)</td>
<td>3.52466</td>
<td>0.00026</td>
<td>0.00073</td>
</tr>
<tr>
<td>Cell vol. (Å(^3))</td>
<td>29.0717</td>
<td>0.0026</td>
<td>0.0071</td>
</tr>
</tbody>
</table>

In co-operation with:
Magnetic field distribution and modelling

- Numerical solution of the Helmholtz equation for the vector potential and the magnetic field
- Comparison between numerical predictions and experimental results

**Low fields:**

Experimental data

Theoretical prediction

\[ \mu_0 H_{\text{app}} = 0.0110 \, \text{T} \]

-10.1 mm
-7.85 mm
-5.6 mm
-2.1 mm
0.1 mm
2.1 mm
4.1 mm
8.35 mm

Axial position (mm)

**T = 20 K**

\[ T = 20 \, \text{K} \]
3 shield configurations:

SC cup:
- outer radius: 10.5 mm
- inner radius: 7.5 mm
- height: 10.5 mm
- depth: 7.5 mm

FM cup:
- outer radius: 14.0 mm
- inner radius: 11.5 mm
- height: 12.5 mm
- depth: 10.5 mm

$SF = \frac{B_{unsh,z}}{B_z}$

LOW FIELDS: higher penetration due to higher local field induced by FM cup

HIGH FIELDS: SF higher by a factor of 3-5, depending on $T$

In hybrid systems, the total SF can be higher than the product of the two separate SF’s.
Topic n.3: MW/THz devices by means of heavy ion lithography

EXPERIMENTAL SUPERCONDUCTIVITY GROUP
GRUPPO di SUPERCONDUTTIVITA’ SPERIMENTALE

POLITECNICO di TORINO
Department of Applied Science and Technology
and INFN, Sezione di Torino

<table>
<thead>
<tr>
<th>Research theme</th>
<th>Tema della ricerca</th>
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<tbody>
<tr>
<td>Experimental techniques for characterization and engineering of superconducting, magnetic and functional oxide materials, towards employment in innovative device applications</td>
<td>Tecniche sperimentali per la caratterizzazione e l’ingegnerizzazione di materiali superconduttori, magnetici e ossidi funzionali, per impiego in dispositivi innovativi</td>
</tr>
</tbody>
</table>
Research activity of the group

The research group is active in the field of experimental superconductivity from about two decades. It was aimed at investigating the particle-irradiation effects on vortex dynamics in high-Tc superconductors, in collaboration with INFN.

Since then, the group significantly extended its research activity to new topics, including the study of the electromagnetic properties of new superconductors (currently iron-based superconductors) and superconductor/ferromagnetic heterostructures, the design of innovative superconductor-based devices and applications (e.g. THz sensors, microwave devices, magnetic shielding solutions) and the use of high-energy heavy-ion facilities for both fundamental studies and engineering of functional materials.

We have availability of several laboratories, all equipped with cryogenic systems. The whole set of experimental techniques (magneto-optics, structural, electric, magnetic, microwave and optical facilities) allows us to efficiently characterize not only superconductors, but also magnetic materials and functional oxides.

MAIN SUBJECTS for MS/PhD THESIS

- use of high-energy heavy-ion lithography for material nano-engineering
- magneto-optical imaging techniques
- design and characterization of superconducting microwave devices
- microwave techniques for fundamental studies on unconventional superconductors
- plasmonic mechanisms and resonance of domain walls in magnetic heterostructures
- setup and test of a cryogenic scanning Hall probe facility
- study of innovative magnetic shielding solutions

CONTACT  Prof. Gianluca Ghigo  tel. 011-0907362 / 011-0907349  gianluca.ghigo@polito.it  www.polito.it/superconductivity
**Topic n. 4: superconducting devices for basic physics research**

In co-operation with:

Typically 6 month stay in Stockholm at the nanofabrication lab.

- Clean room
- SEM/FIB
- E-gun evaporator
- Ar milling
Example: THz emission

- THz emitter: 6 Bi-2212 mesa (view from top)

Mesa synchronization implies emitted power $\propto N^2$

- EM coupling supposed to take place via the crystal base.

Optically transparent trench

New design: Coupling obtained through the crystal itself

For further information:

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Hall coefficient is 100 times greater than expected. Mesoscopic phenomenon?

Forcing the current by means of FB-etched trenches (depth ≈ 200 nm)