



Torino, 8 Novembre 2016

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)

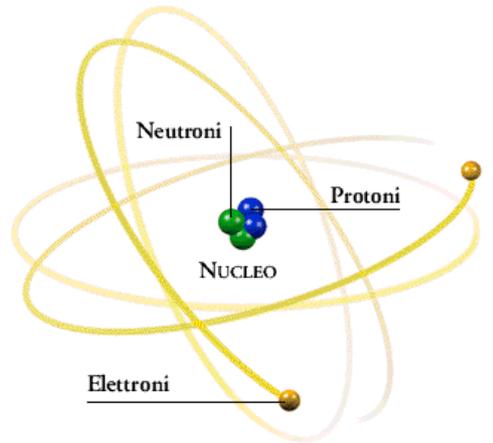


What about friction in the micro- world ?

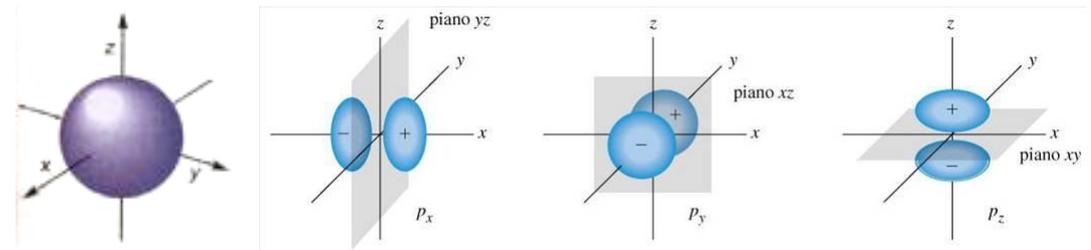
❖ Here the lack of friction is the rule !!!

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

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



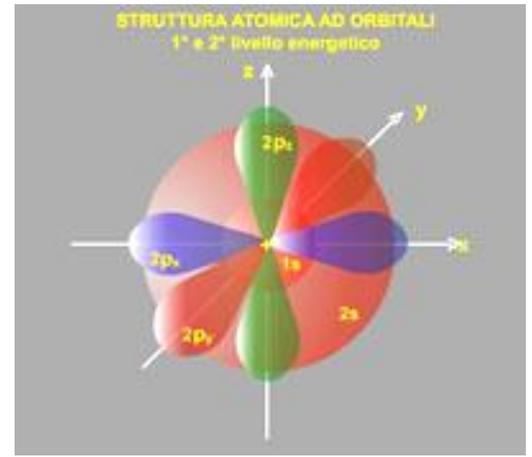
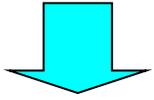
Traditional picture
(inadequate): ORBITS



Quantum picture
(correct): ORBITALS

In each ORBITAL electron energy is CONSTANT

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



During their motion around the nucleus,
electrons never loose energy, which means that
they move **WITHOUT FRICTION**



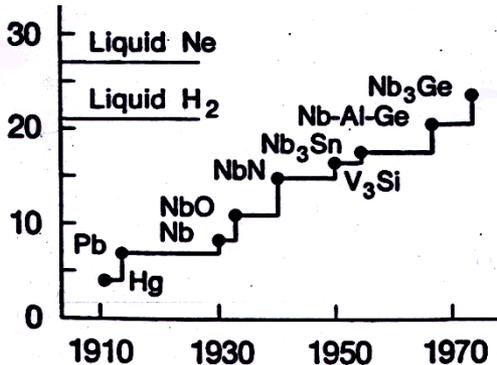
Is it possible to observe the same phenomenon on a macroscopic scale ?

❖ 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). $T_c = 4.2 \text{ K} \approx -269^\circ\text{C}$.

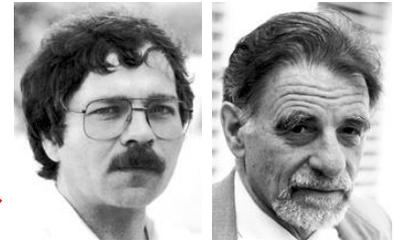
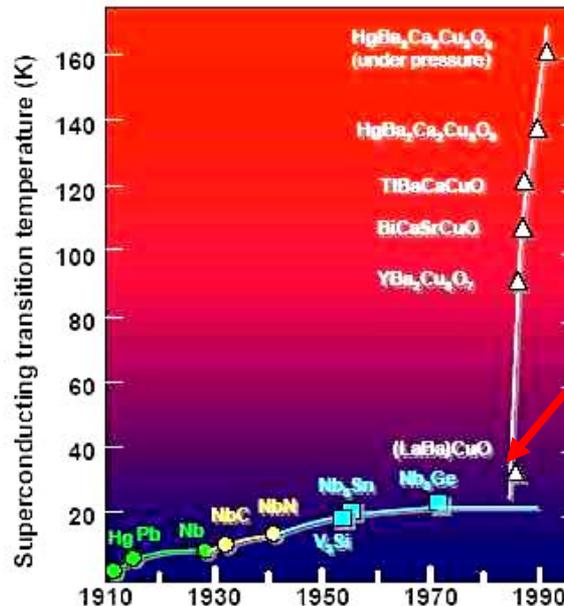
Too cold? It depends: average T of the Universe = 2.73 K

❖ Are the temperatures always so low ?

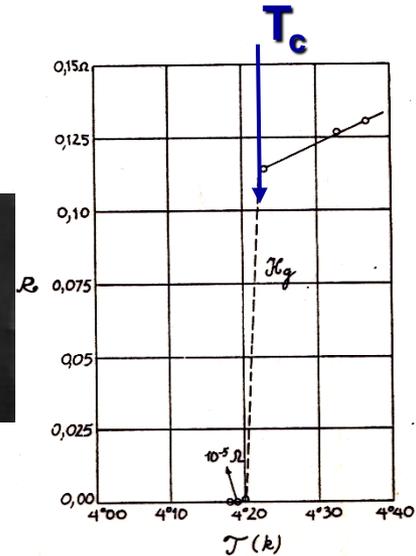


Maybe

Maybe not



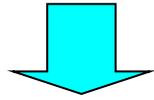
1986: Bednorz & Müller (Nobel prize in 1987)



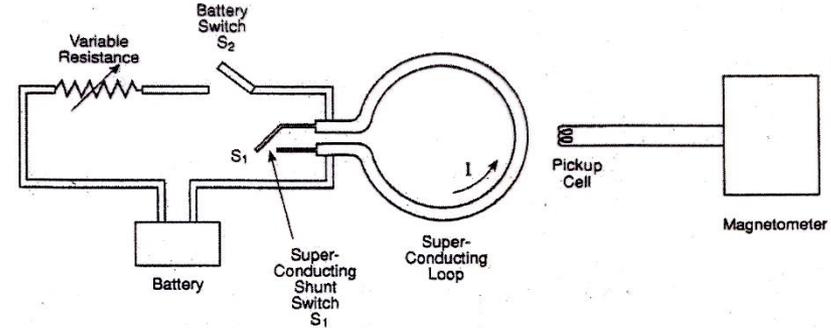


Is it really with NO FRICTION ?

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

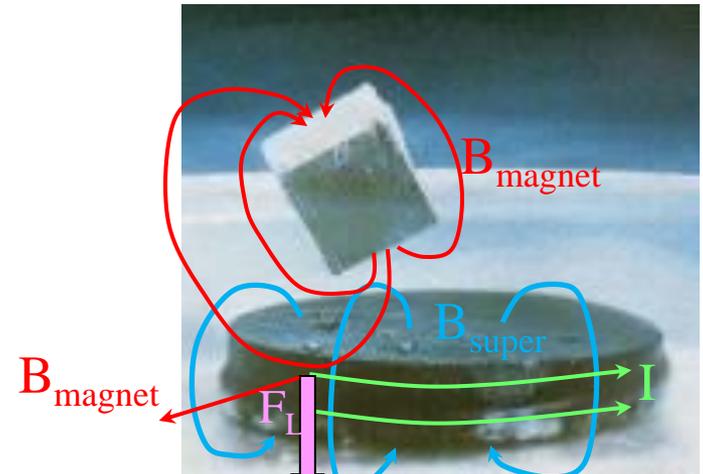
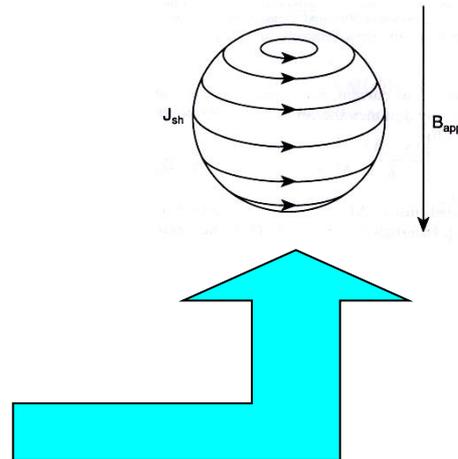
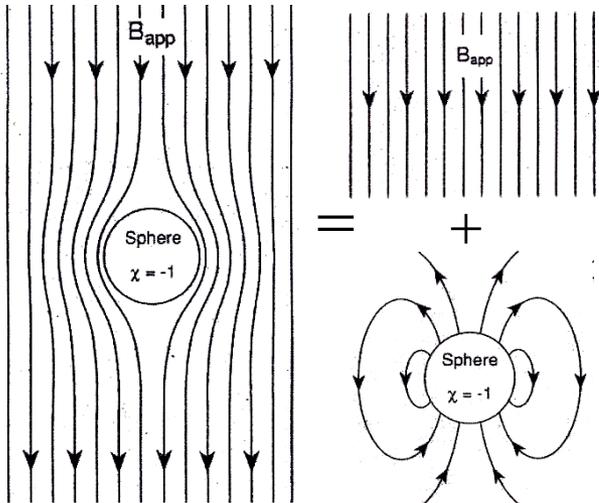


Electrical resistance (if any) is 10^{17} times smaller than in Cu $\rightarrow R=0$



Another surprise: the Meissner effect

❖ Superconductors ALWAYS expel the magnetic field: the presence of BOTH phenomena defines superconductivity. $\rightarrow B=0$

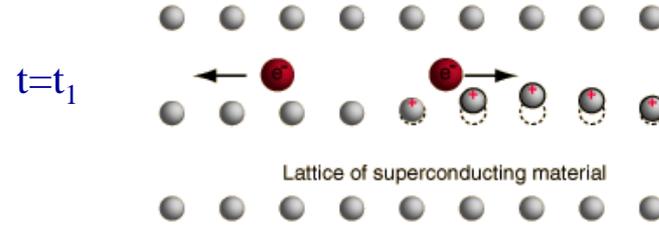
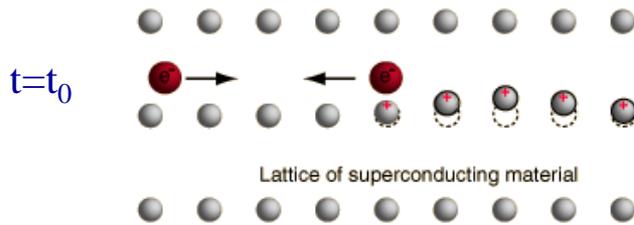


Lorentz force induces repulsion: Magnetic levitation

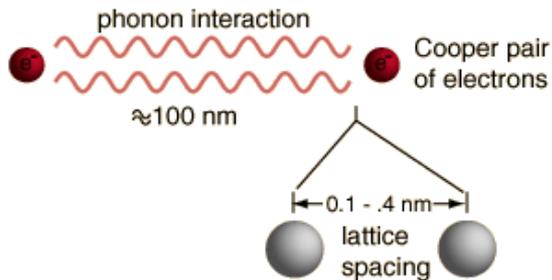


How is it possible?

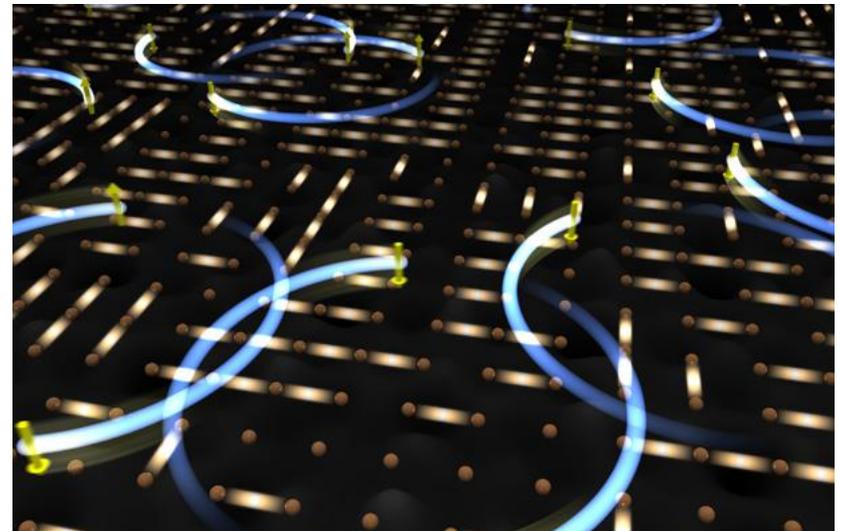
- ❖ Unexpected event: Due to some interaction (e.g. electron-lattice), electrons in a solid can experience a net attraction force exceeding their coulomb repulsion.



- ❖ A bound state originates for two electrons at some distance: the Cooper pair



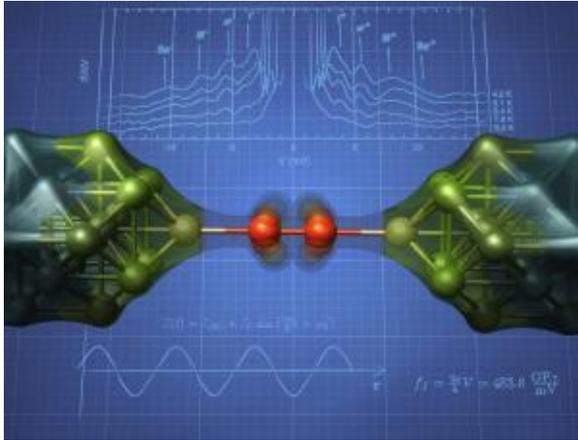
- ❖ In a superconductor, billions of Cooper pairs exist in the same volume overlapping and crossing each other. They are in the same *quantum state*, forming a macroscopically extended superfluid.



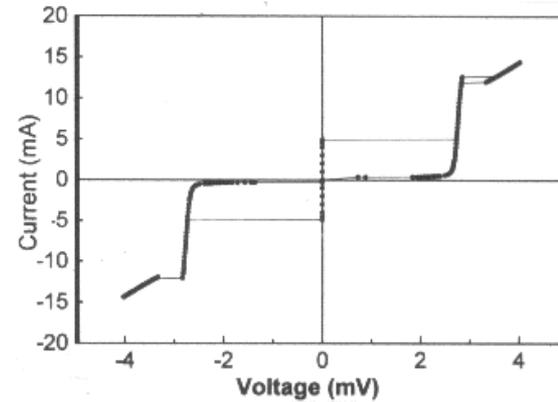


Cooper pair tunnelling

❖ Josephson effect:

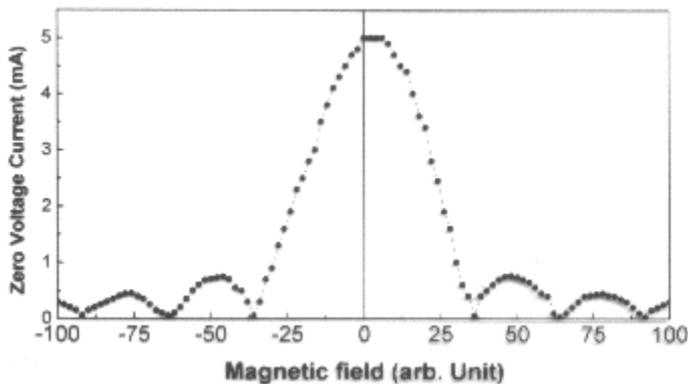


❖ Special behaviour: DC *current* induces NO voltage drop; DC *voltage* induces an AC current (AC effect)

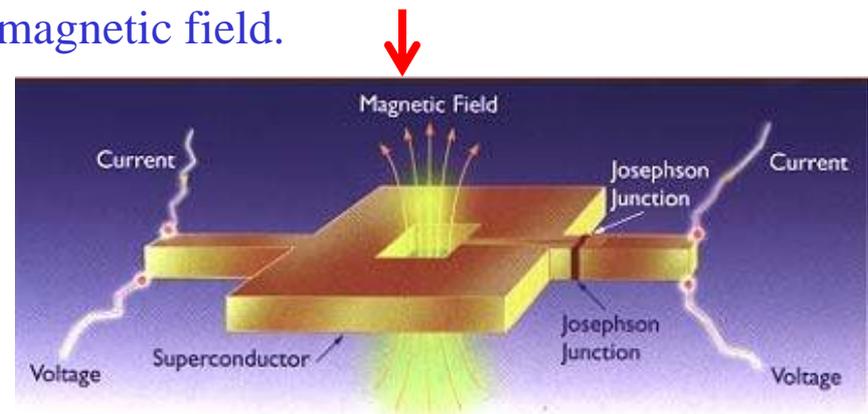


❖ This behaviour is affected by the magnetic field: this can be exploited to measure it.

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



The **SQUID** nominally can measure $1\text{fT} = 10^{-15}\text{ T}$, i.e. 10^{11} times smaller than the Earth magnetic field.



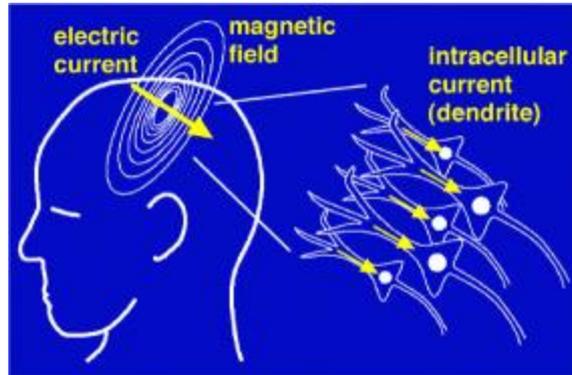


Examples of superconducting devices

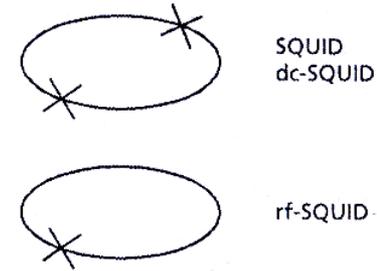
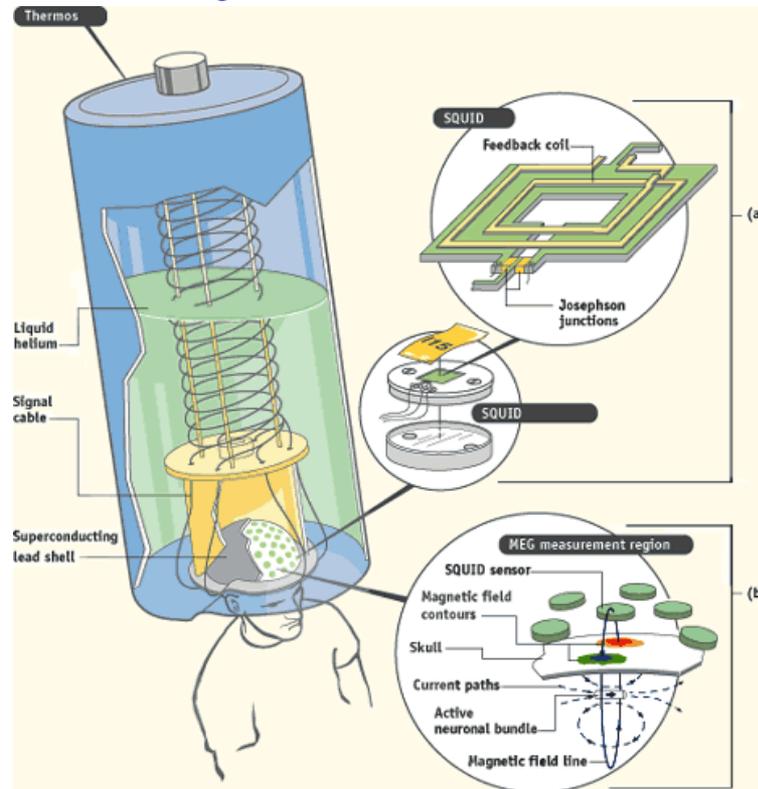
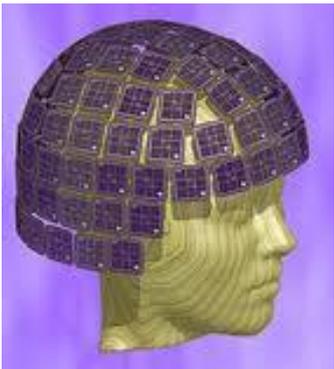
All based on the Josephson effect (both sensors and digital circuits)

Sensors: Superconducting QUantum Interference 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.

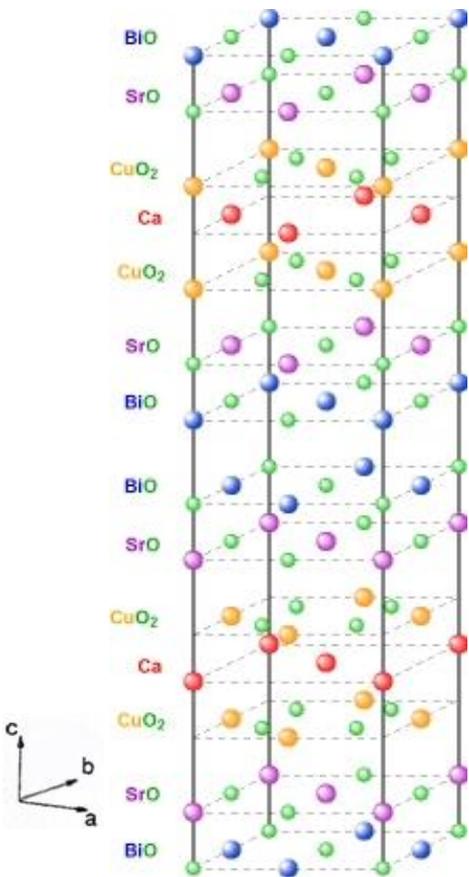
Faster than MRI (10 msec per frame)



Topic n.1: Intrinsic Josephson junctions... ..oxides...

High T_c Superconductor: BSCCO

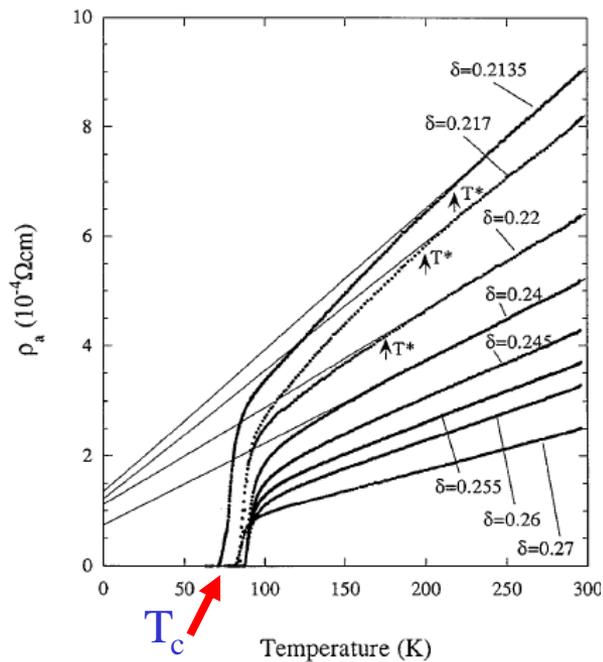
➤ $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (Bi-2212): an inherently anisotropic material



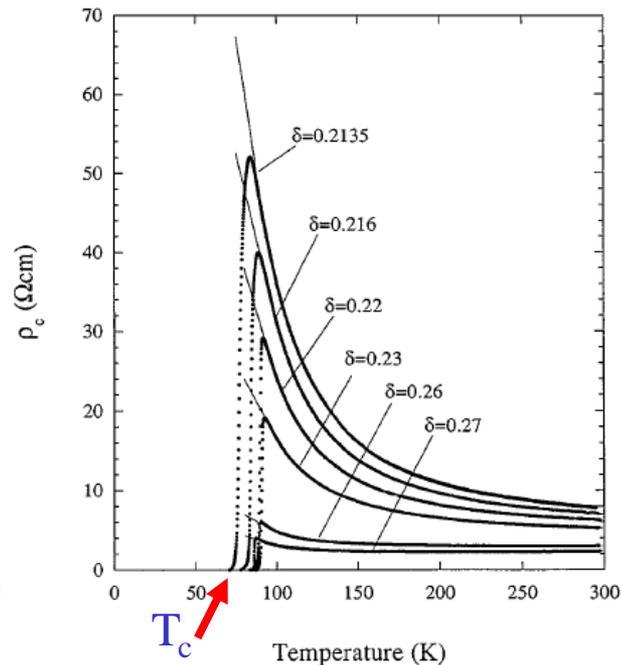
$a=5.407 \text{ \AA}$, $b=5.432 \text{ \AA}$, $c=30.931 \text{ \AA}$

R.H. Arendt et al., Physica C, **182**, 73, (1991)

ab-plane



c-axis



T.Watanabe et al., Phys. Rev. Lett., **79**, 2113, (1997)

$T_c = 60\text{--}95 \text{ K}$, depending on δ

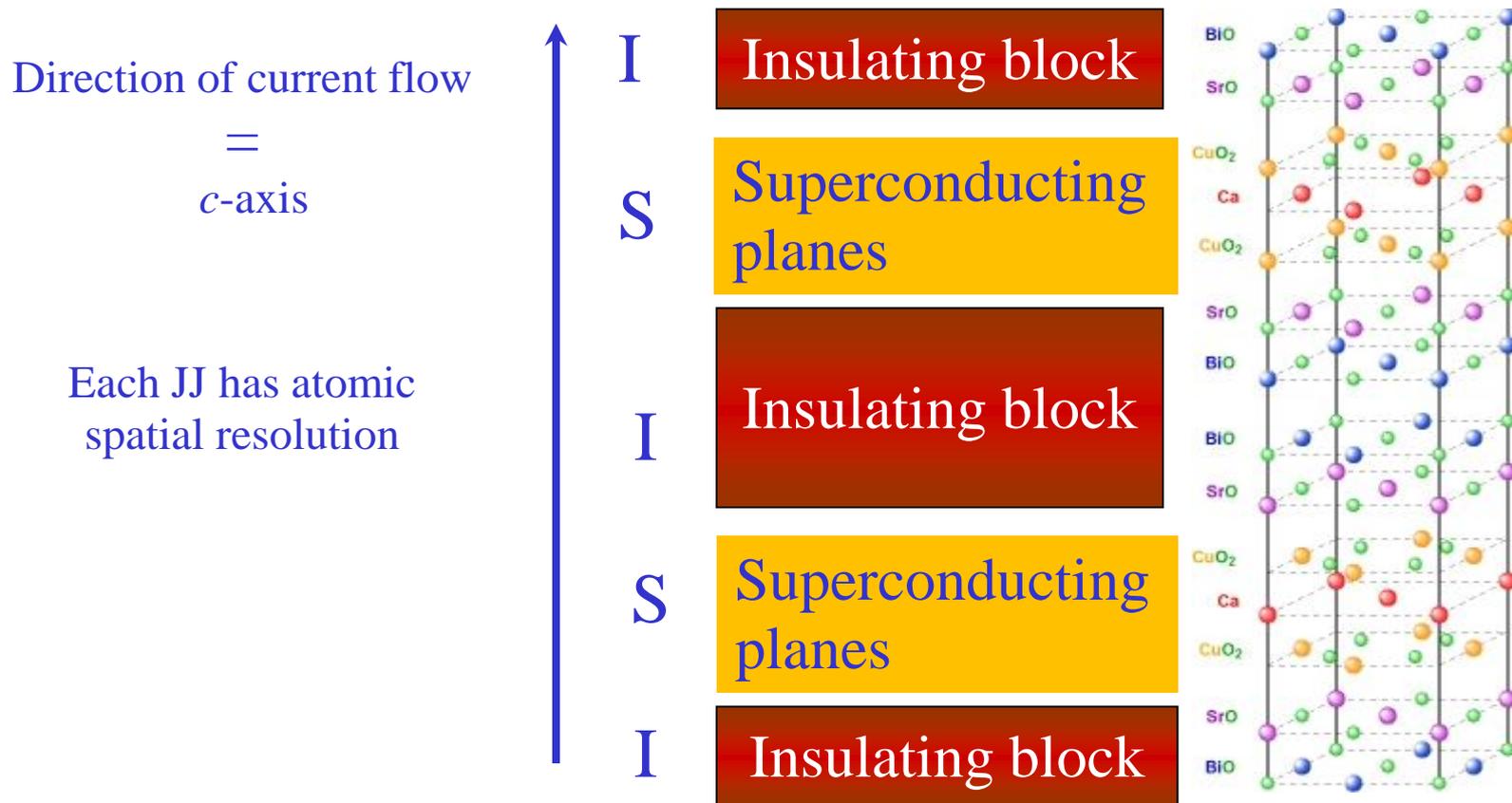


practical interest



Intrinsic Josephson junctions

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



Direct AC Josephson effect (DC voltage bias \rightarrow AC current)

Emission power limited to the pW range for a single junction \rightarrow Arrays of JJ's

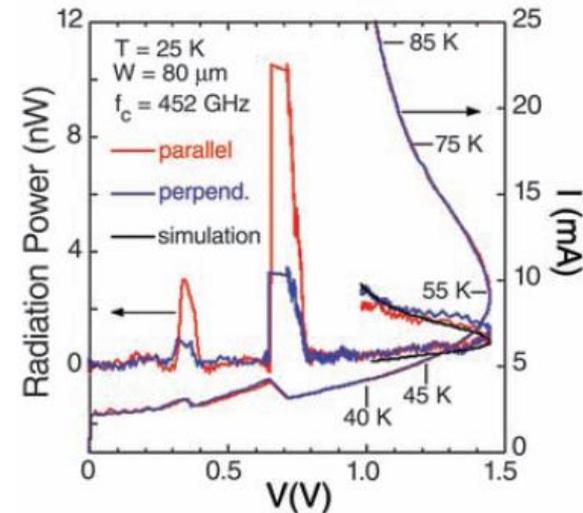
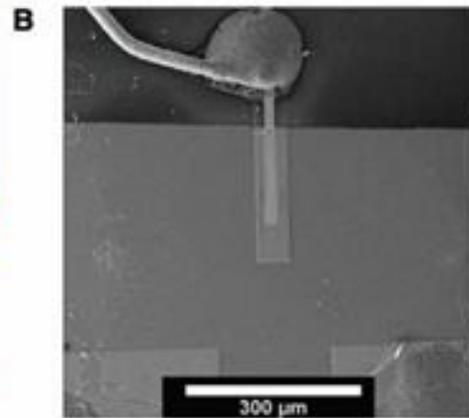
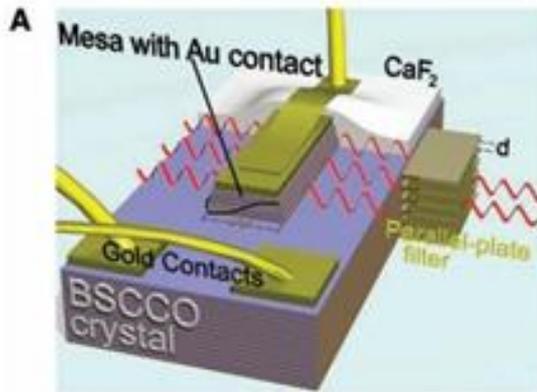


THz radiation emission

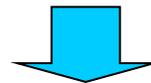
➤ Experimentally detected by Ozyuzer *et al.*



Emission of Coherent THz Radiation from Superconductors
L. Ozyuzer, *et al.*
Science 318, 1291 (2007);
DOI: 10.1126/science.1149802



- 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
- Maximum power about 610 μW (T. M. Benseman *et al.* Appl. Phys. Lett. 103, 022602 (2013))



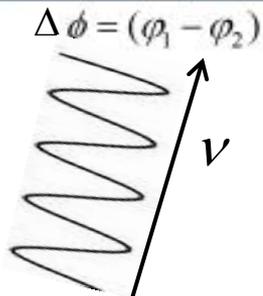
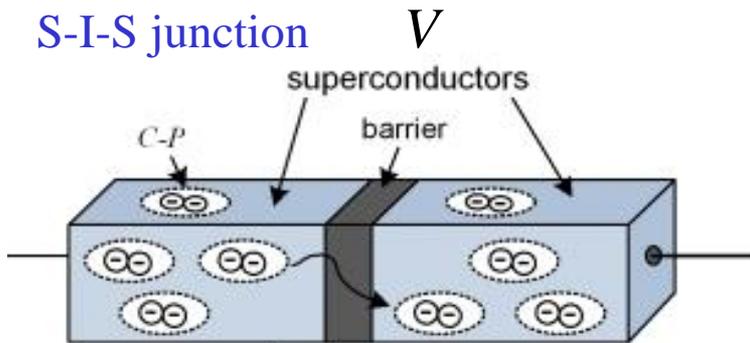
Emission mechanism still unclear

➤ Interesting for production of THz sources

Metrological interest

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

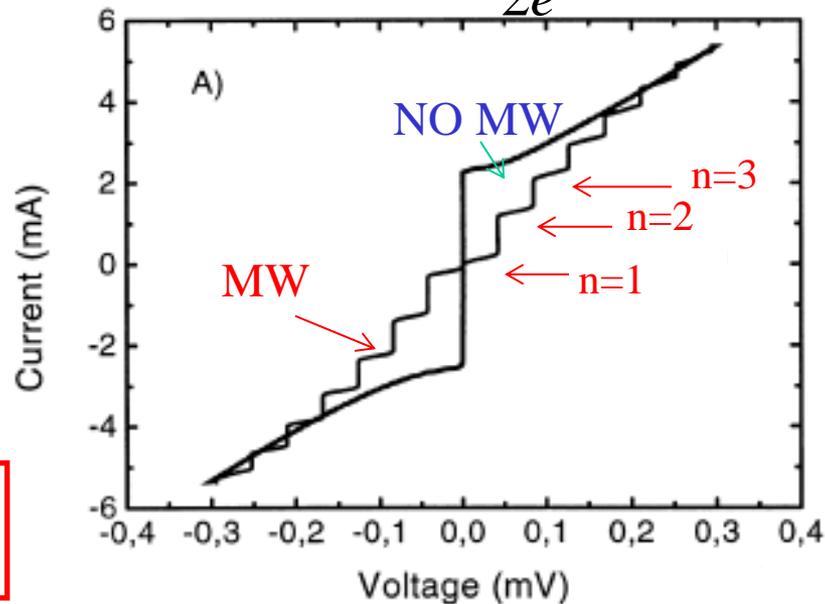
S-I-S junction



Frequency accuracy \approx
1 part in 10^{16}

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

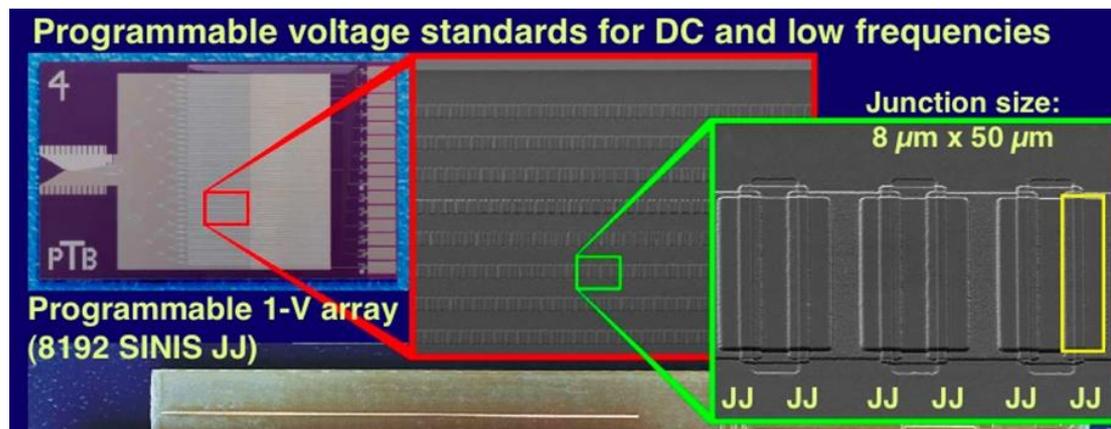
Shapiro steps: $V = n \frac{h\nu}{2e}$ per junction



present low T_c technology
(Nb-Al₂O₃-Al-Al₂O₃-Nb)



Towards a new technology
based on high T_c
superconductors ?

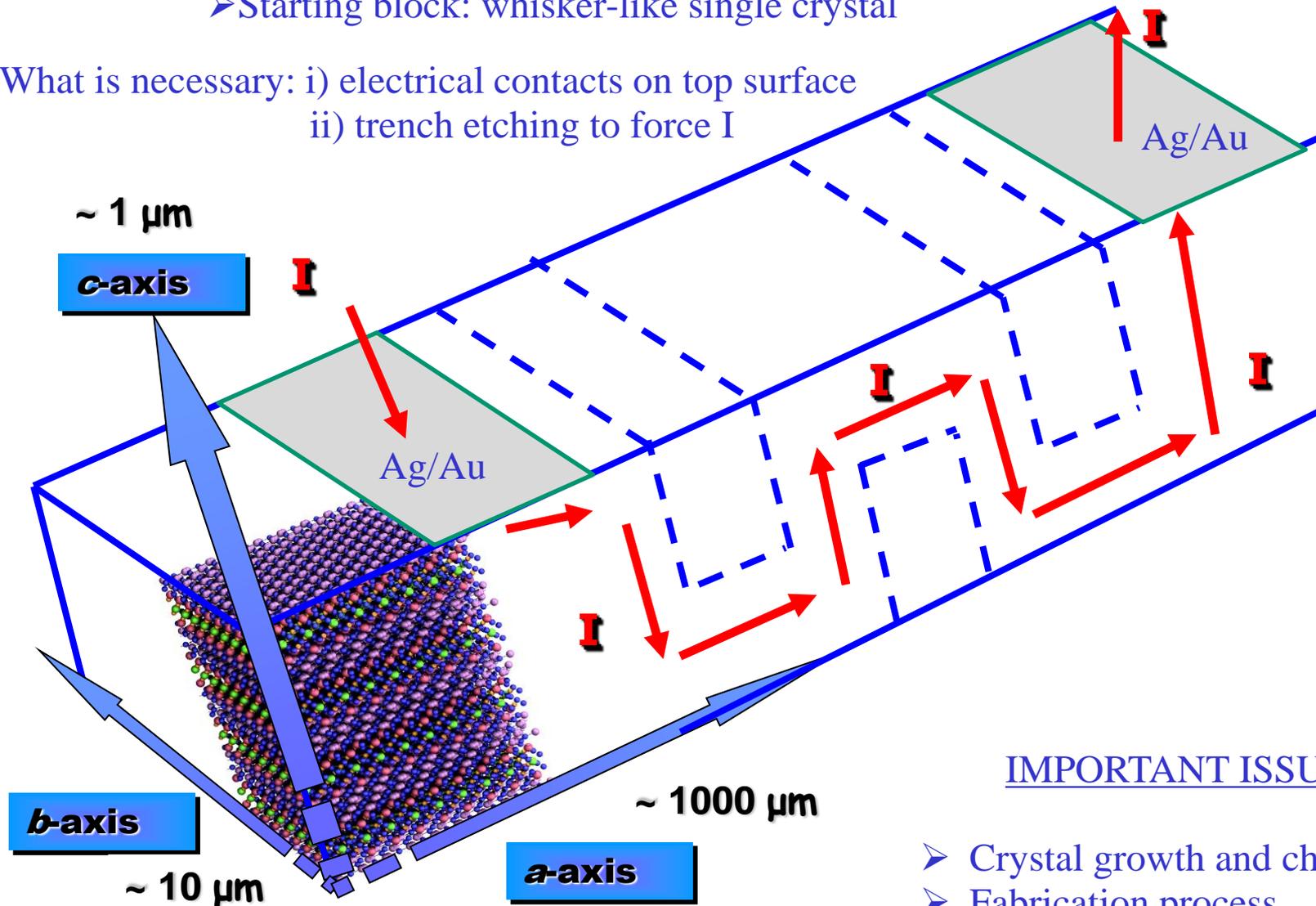




Stack fabrication principle in Bi-2212

➤ Starting block: whisker-like single crystal

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

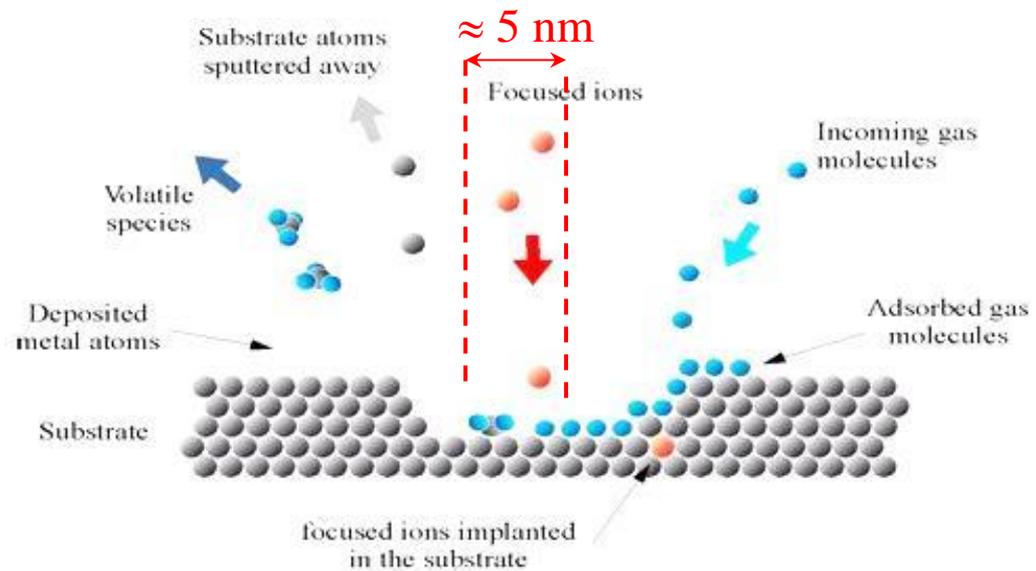


IMPORTANT ISSUES:

- Crystal growth and chemistry
- Fabrication process



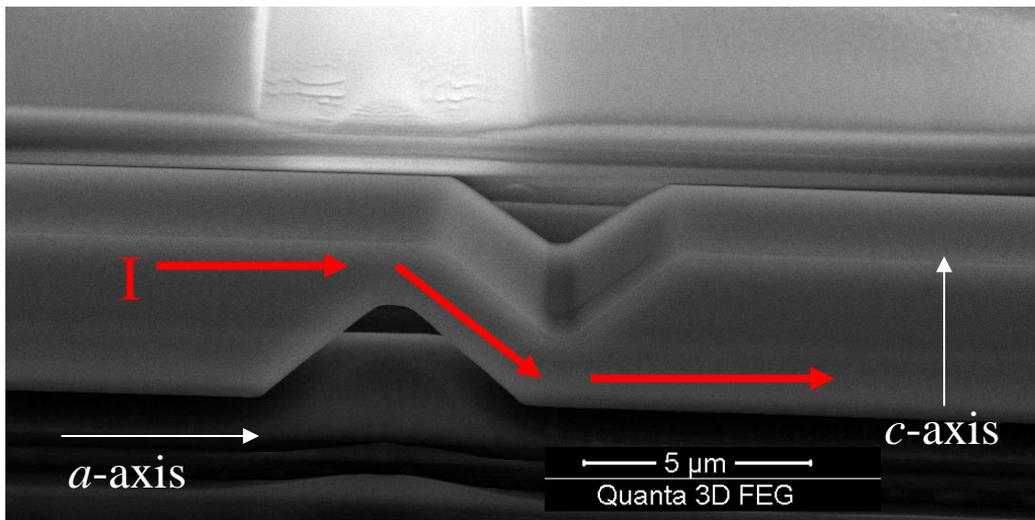
The traditional way: **FIB** etching



i) Photolithography or shadow masks

ii) Etching the trenches by FIB

1. Adsorption of the gas molecules on the substrate
2. Interaction of the gas molecules with the substrate
Formation of volatile and non volatile species
3. Evaporation of volatile species and sputtering of non volatile species



Typical parameters for Ga ion beam:

$$E = 30 \text{ KeV}$$

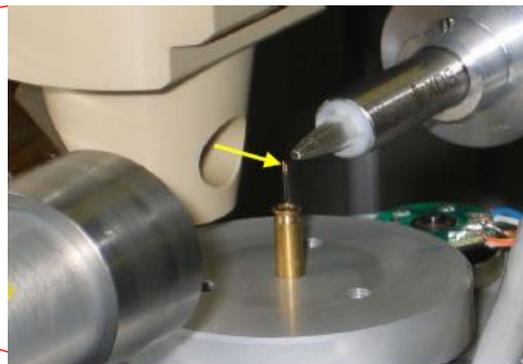
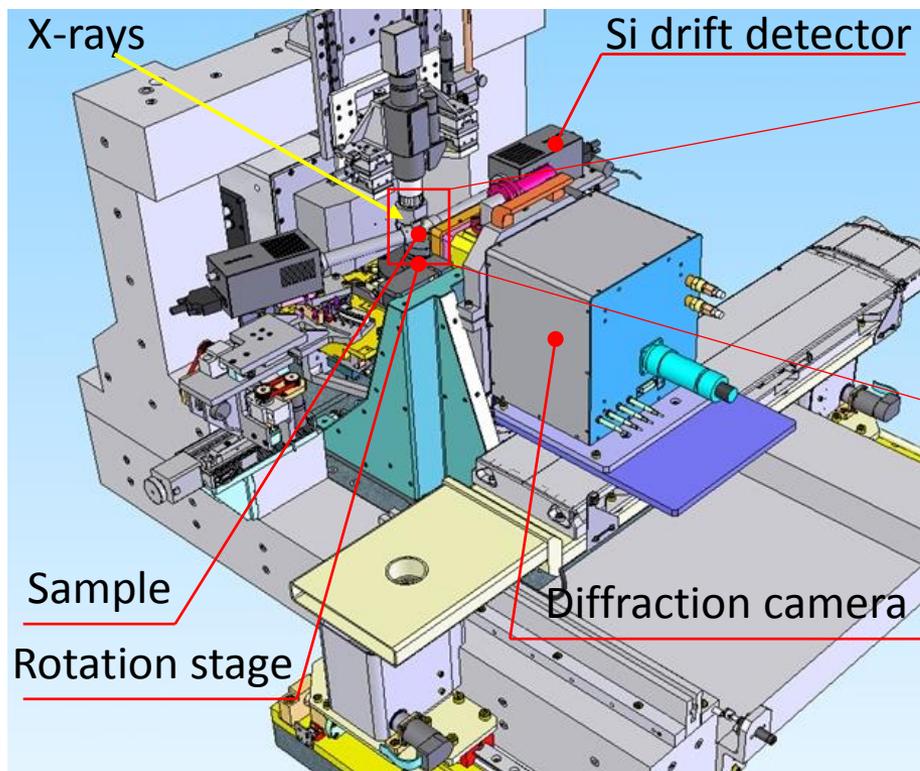
$$I = 10 \text{ pA}$$



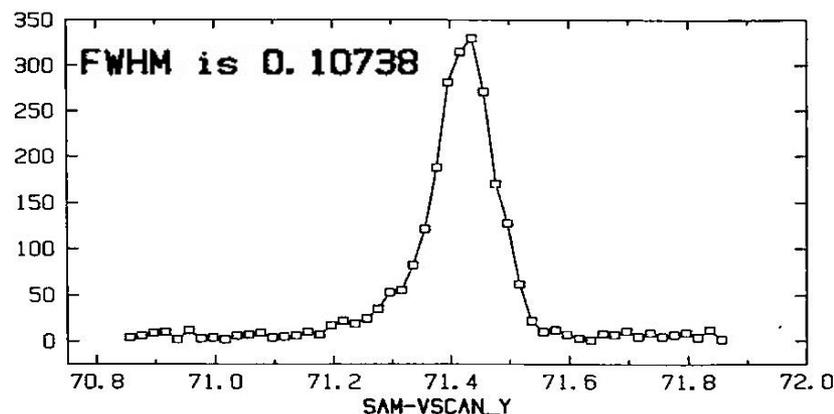
A novel emerging way:

... and X-ray nanolithography

➤ X-ray nanobeam at a synchrotron:



In collaboration with



➤ Beam size $\approx 152 \text{ nm (h)} \times 107 \text{ nm (v)}$

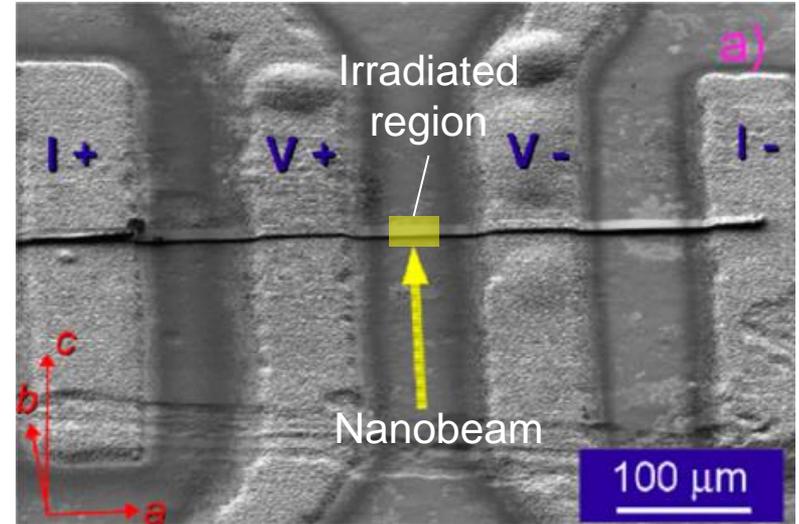
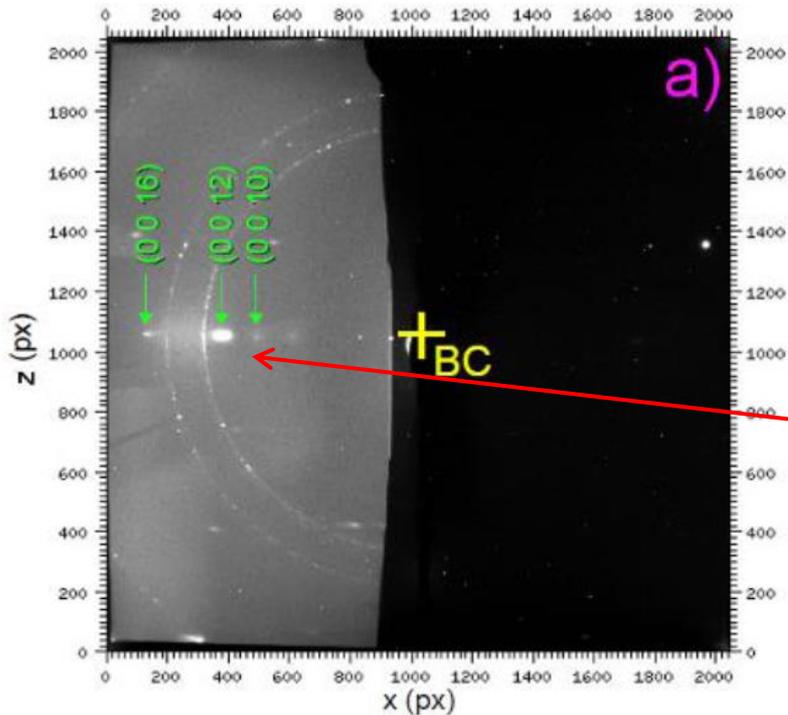
- Kirkpatrick-Baez system (two elliptical mirrors independently bent, one coated with graded multilayer)
- Flux $\approx 1.9 \times 10^{11}$ ph/s
- Energy $\approx 17 \text{ keV}$



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



A. Pagliero et al., Nano Lett. 14, 1583 (2014)

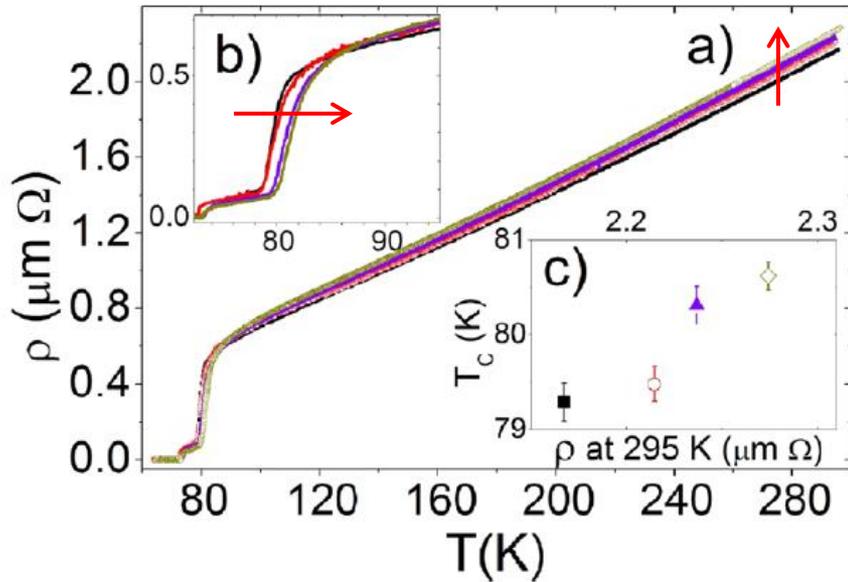
Equatorial series = (0 0 l) peaks



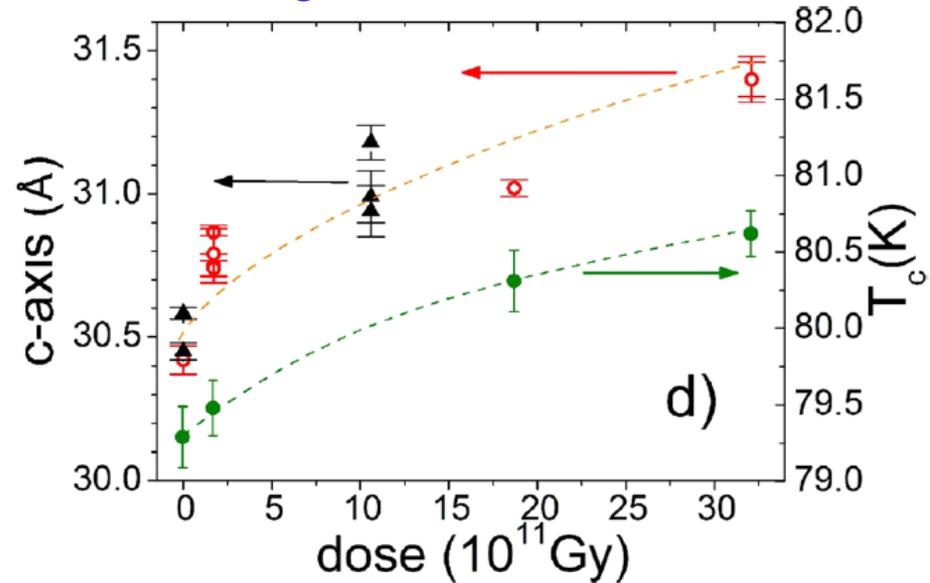
c-axis measurements



➤ Changes of T_c and resistivity

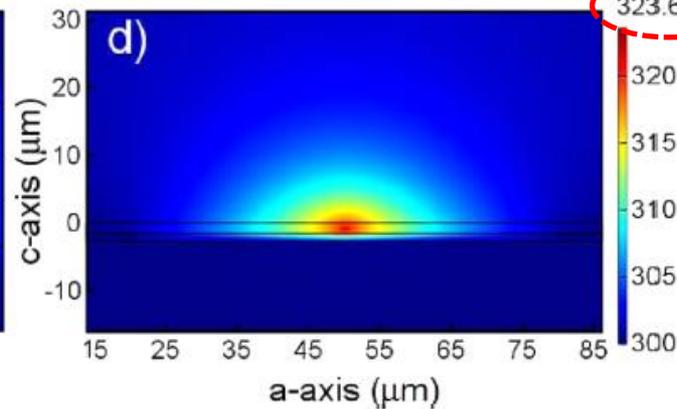
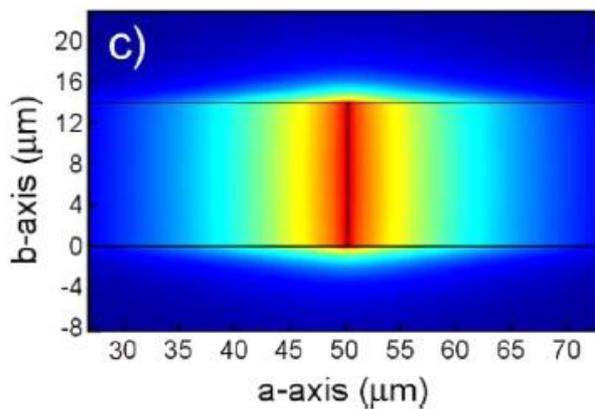


➤ Changes of the c-axis



A. Pagliero et al., Nano Lett. 14, 1583 (2014)

➤ Change in O content: thermal origin ?



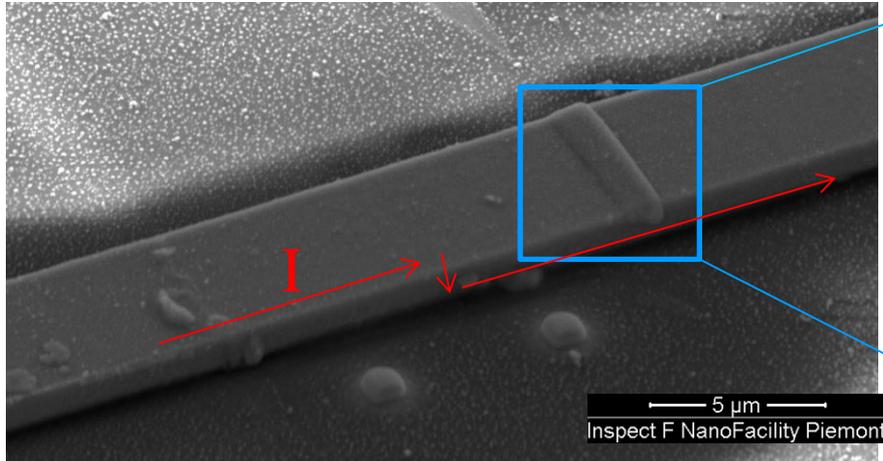
Estimated T_{max}
in DC regime

Novel possible photolithographic process for oxides

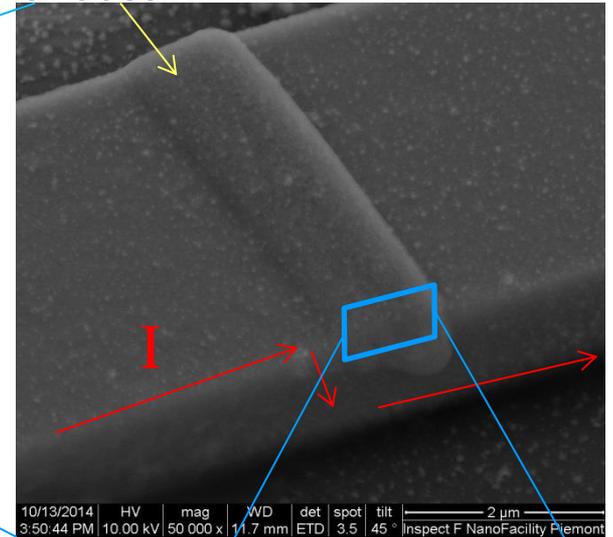


Recent experiments

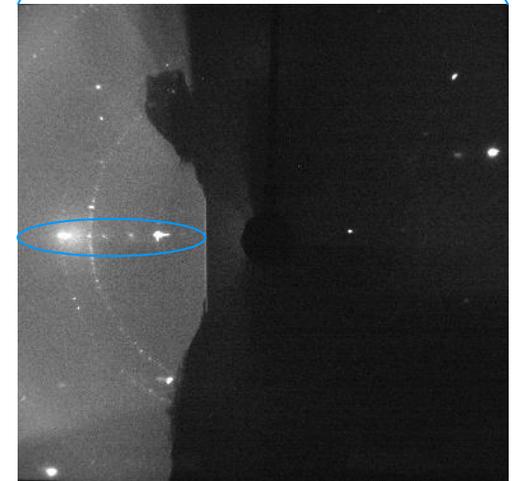
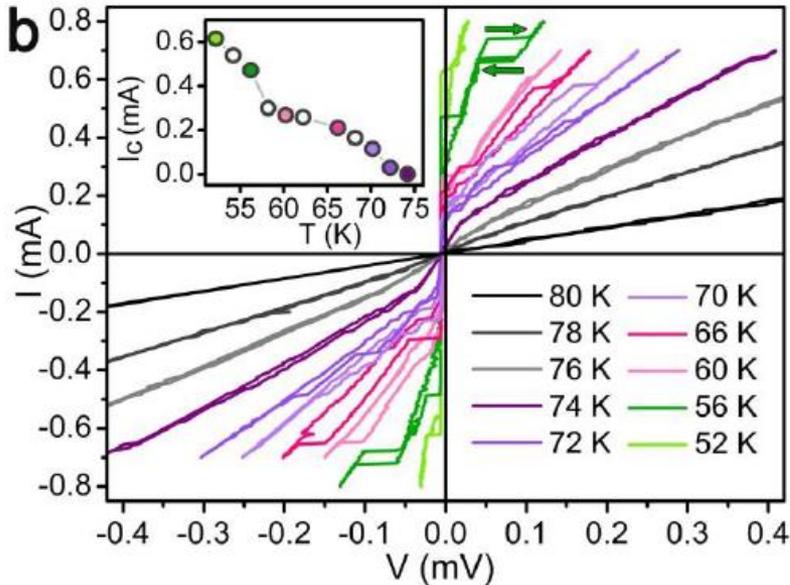
- Nano-beam: $57 \times 45 \text{ nm}^2$
 $E = 17.65 \text{ keV}$



Nanobeam



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



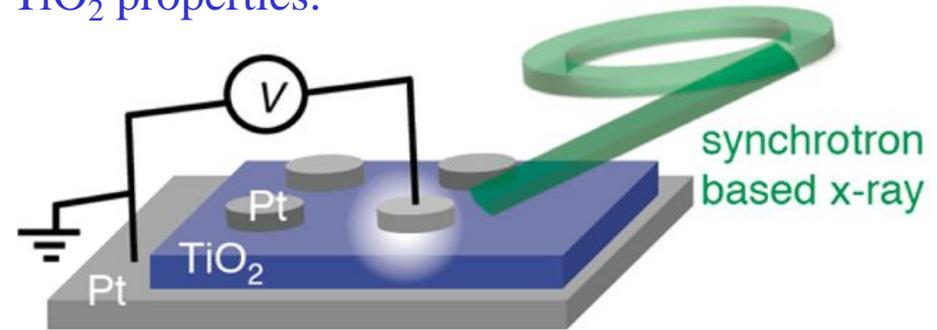
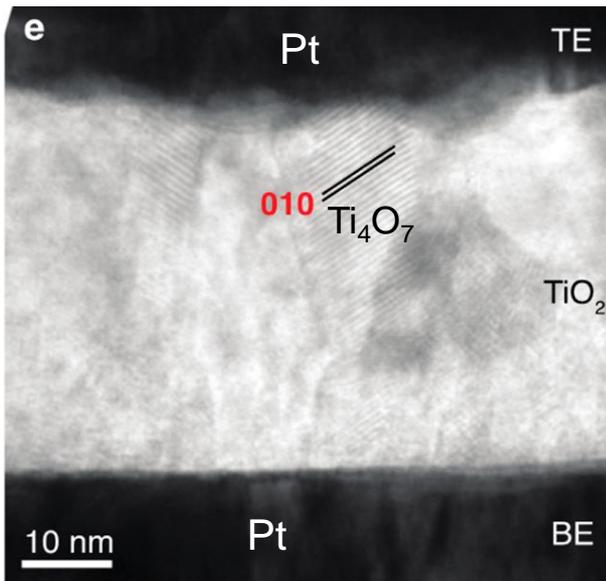


Ongoing developments

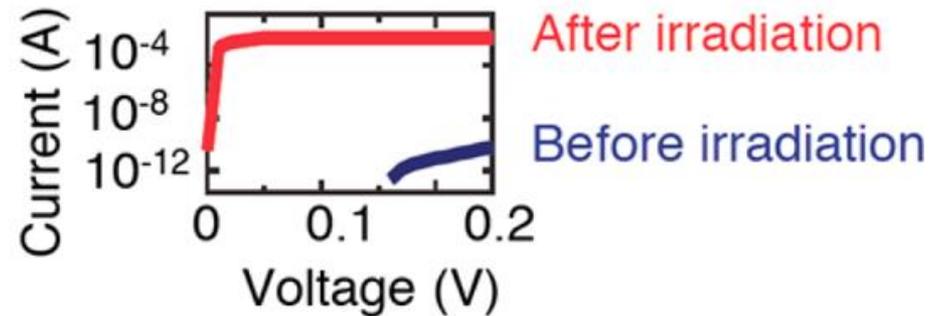
- In principle, this idea could work with many oxides

Preliminary indication: Local modification of TiO₂ properties.

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



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



We are going to test X-ray nano-lithography on TiO₂ !

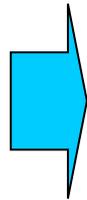
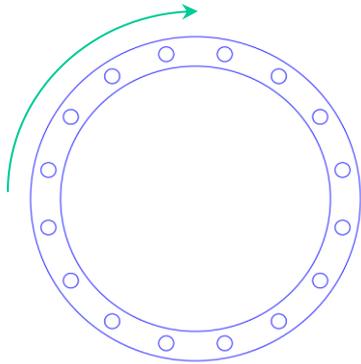


How does it work?

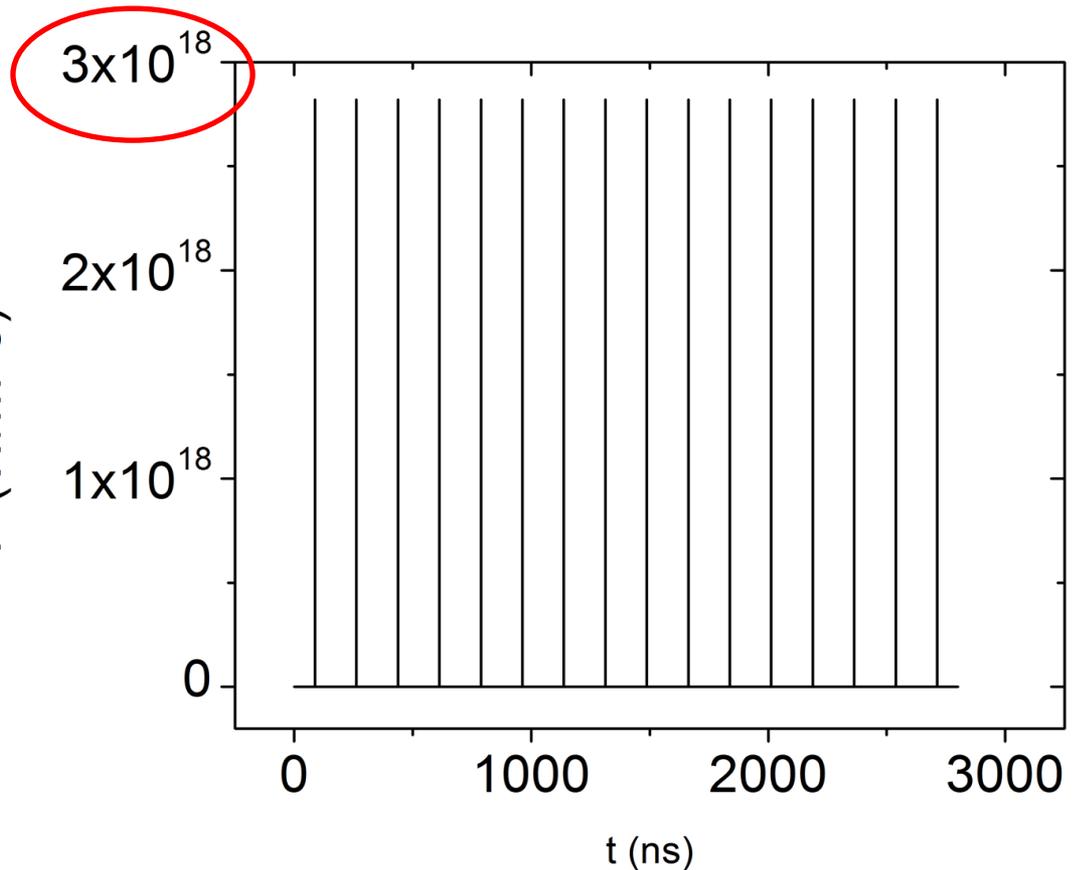
Actually, nobody knows. But:

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

E.g: 16 bunch filling mode



$P \text{ (W/m}^3\text{)}$

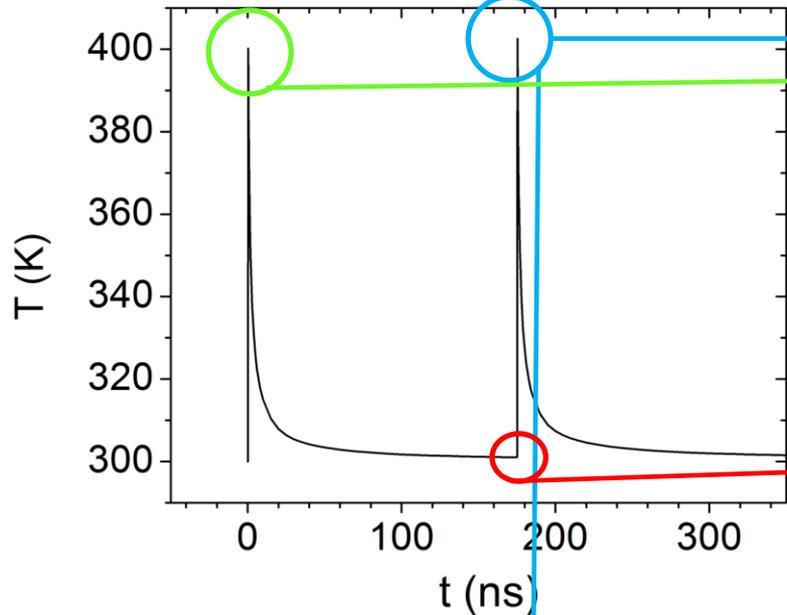


For each bunch: $20 \text{ ps} < \text{FWHM} < 48 \text{ ps}$

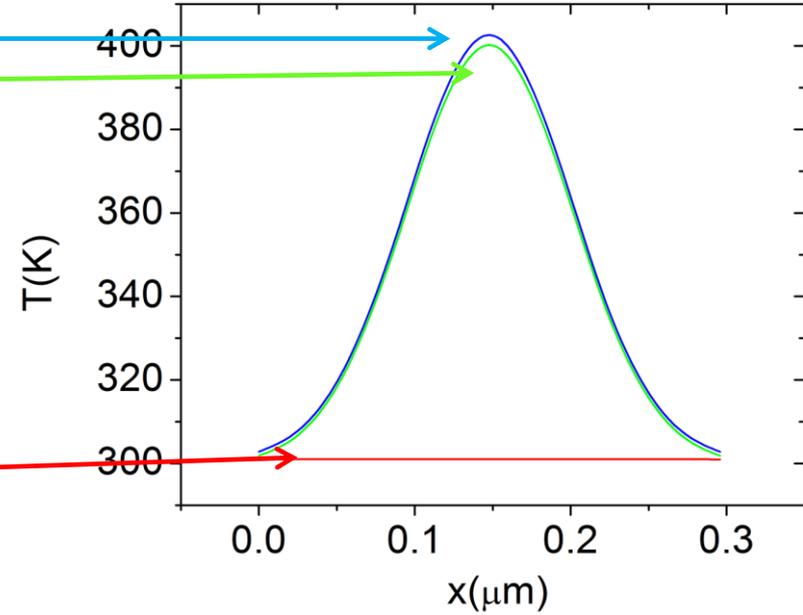


Temperature at the incidence point

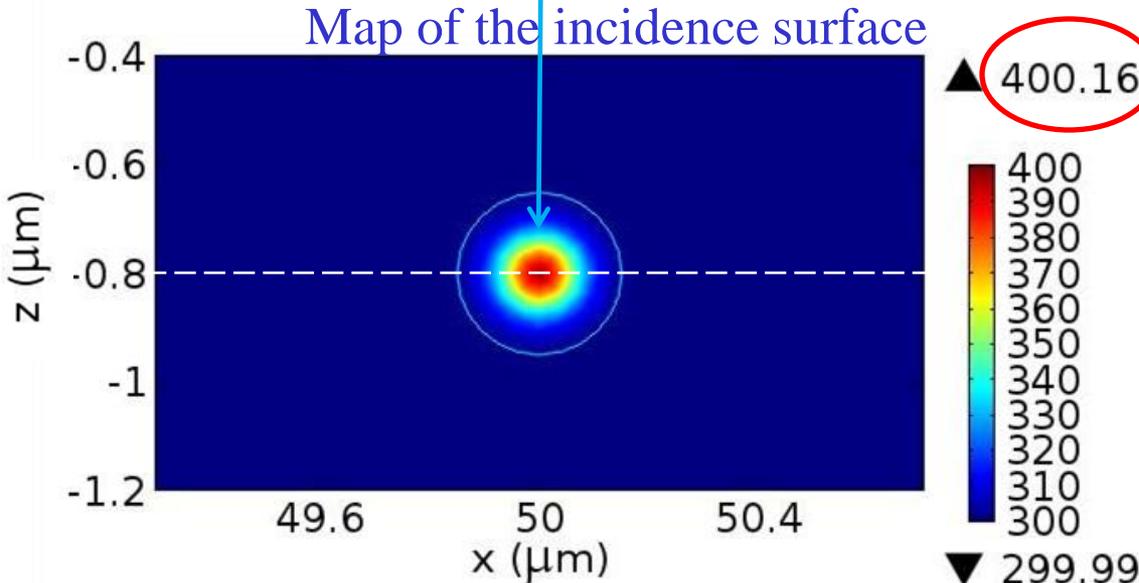
Time behaviour



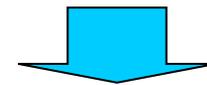
Spatial profile



Map of the incidence surface



Thermal spikes:
 ≈ 100 K for ≈ 1 ns



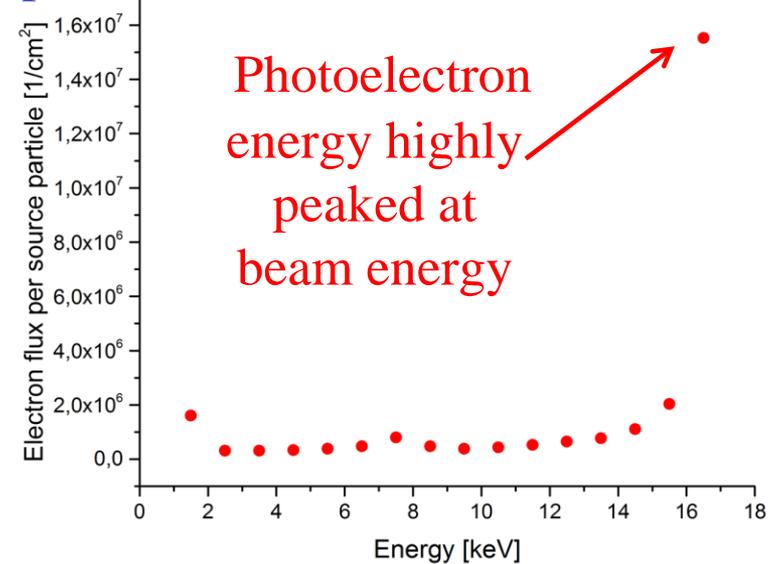
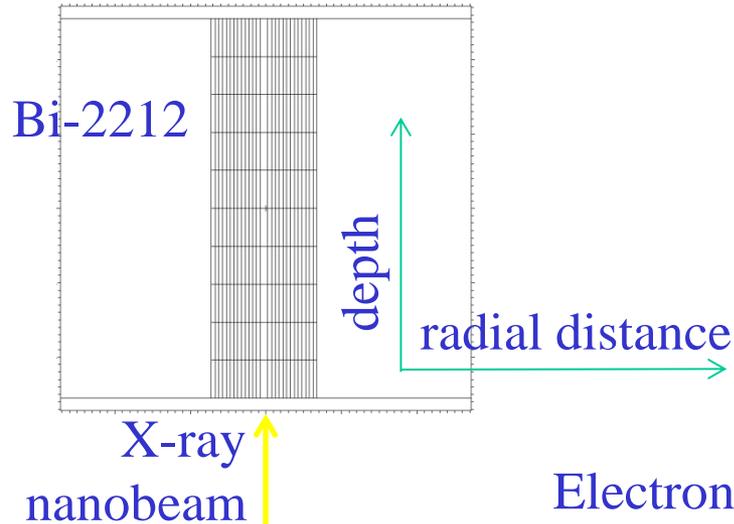
Possible role of O
diffusion ???



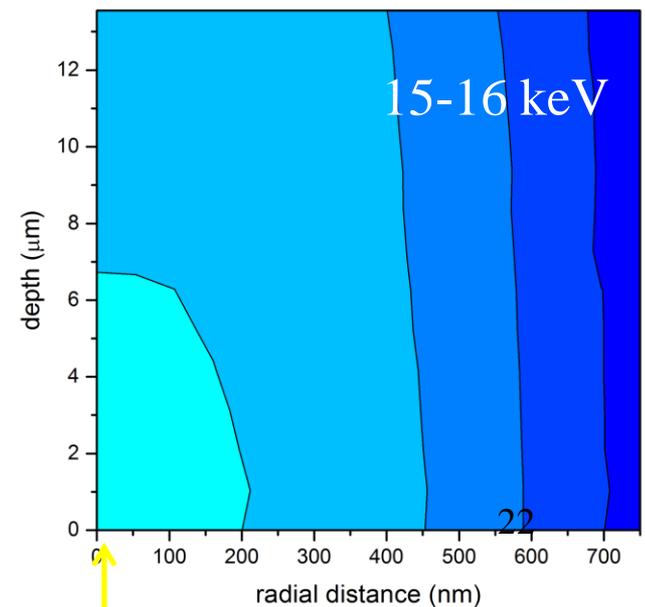
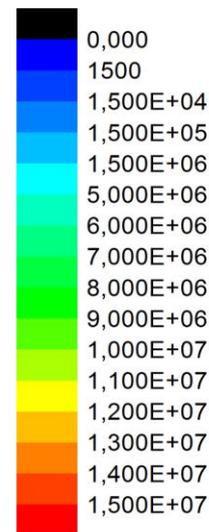
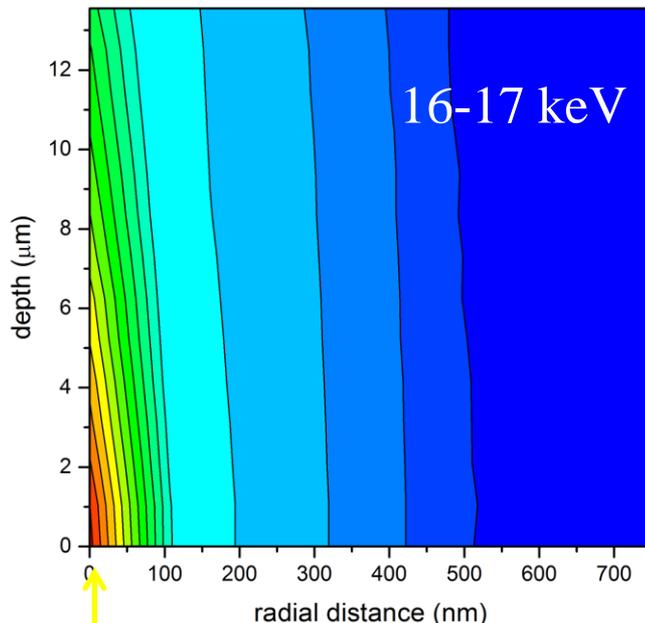
Photoelectrons

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

Cylindrical mesh with 1 keV energy bins



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





From photoelectrons to i-O knock-out

Atoms displaced:

$$N_{ad} = N_{oi} \int_{E_c}^{E_0} \sigma_d(E) \Phi_e(E) dE$$

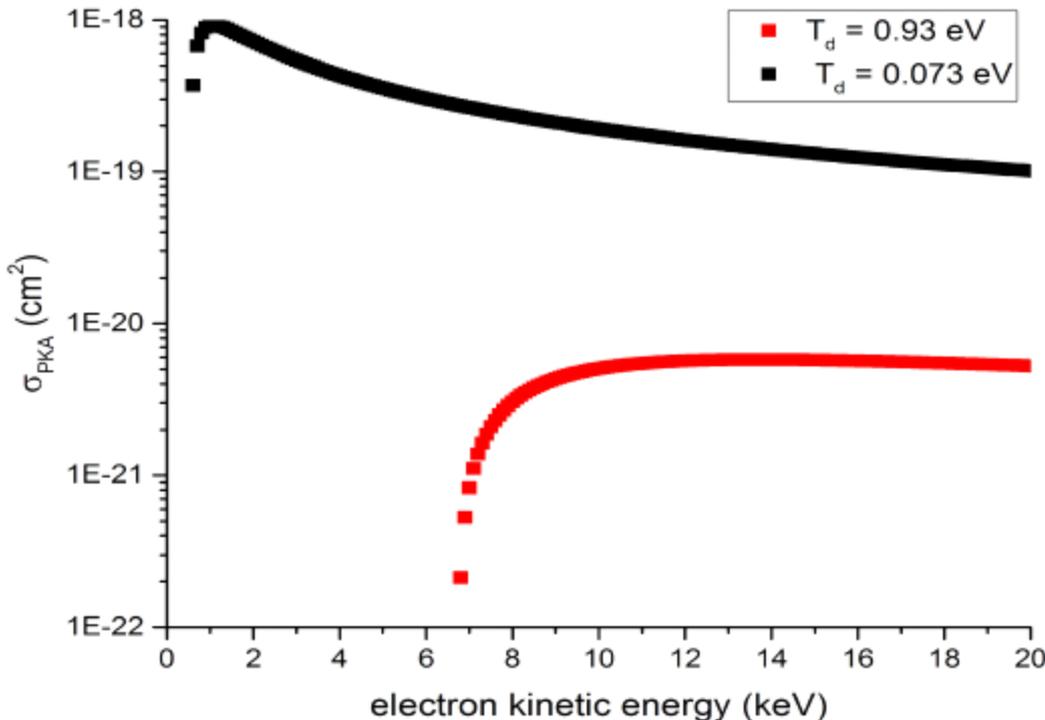
$$\text{Fraction: } f_{ad} = \frac{N_{ad}}{N_{oi}}$$

Mc Kinley-Feshbach cross section:

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

$$T_m = 2E(E + 2mc^2) / Mc^2$$

Very sensitive to T_d



T_d quite uncertain:

➤ O activation energy in the ab plane = 0.93 eV

Runde *et al.* Phys. Rev. B **45**, 7375 (1992)

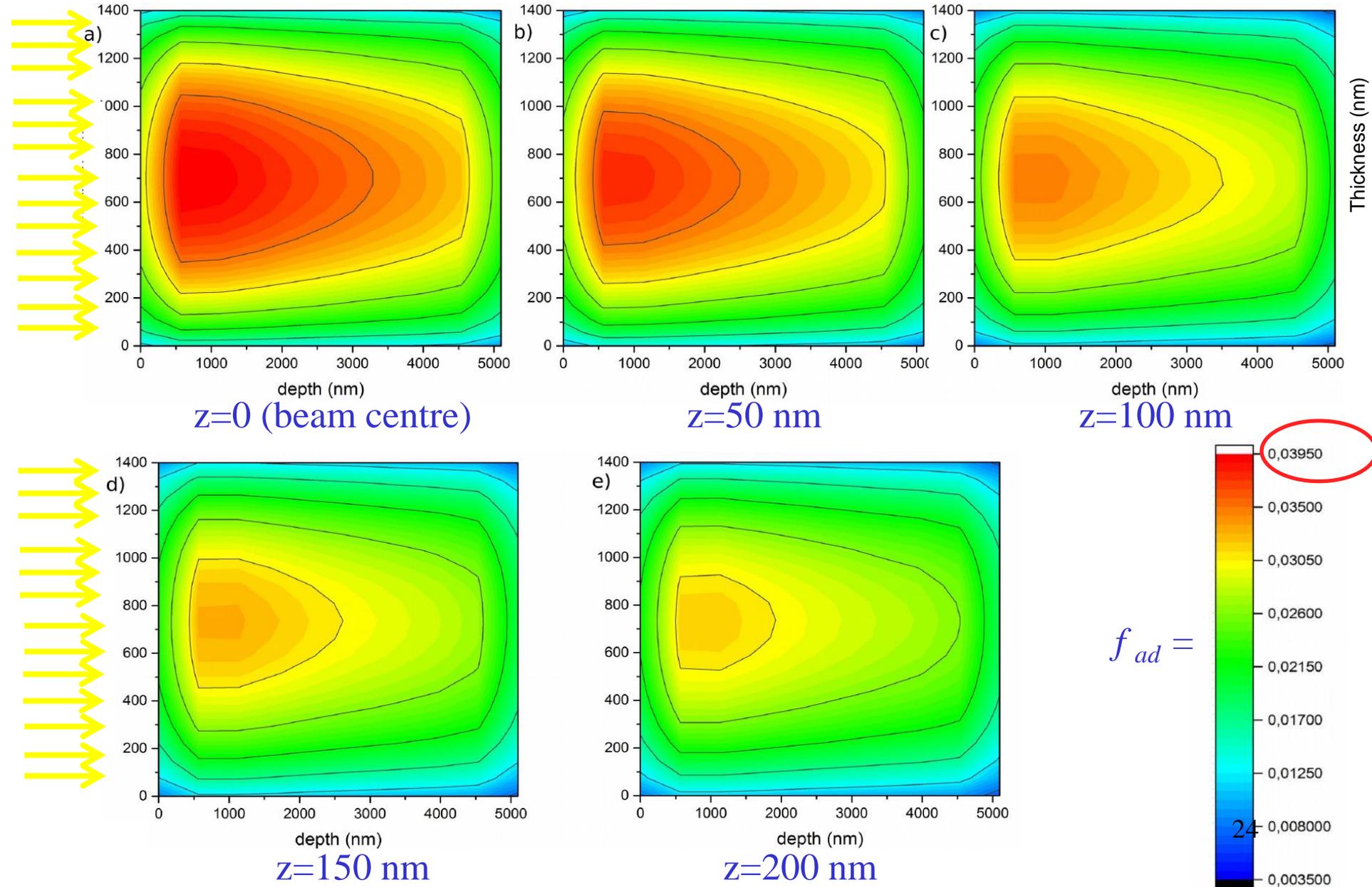
➤ binding energy of i-O atoms = 0.073 eV

Bandyopadhyay *et al.* Phys. Rev. B, **58**, 15135 (1998)



Detailed experiment simulation

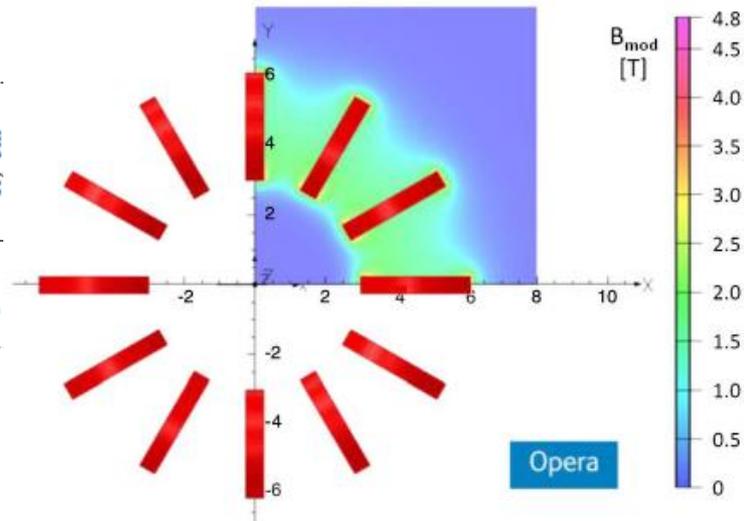
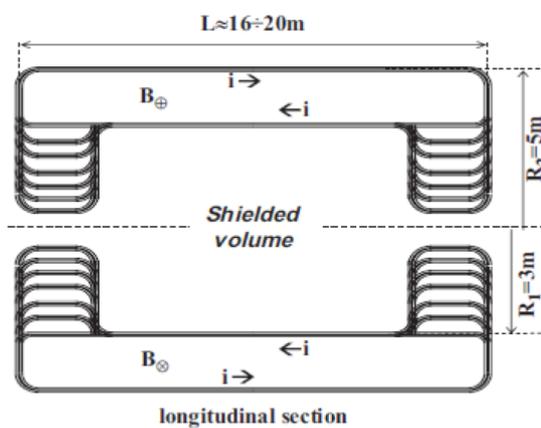
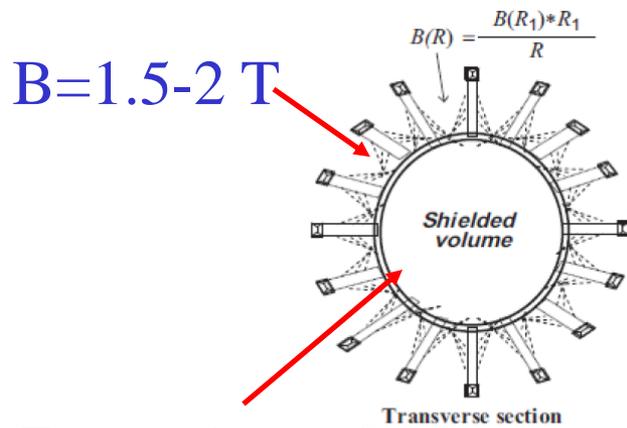
$T_d = 0.073$ eV - Crystal cross-section





Topic n.2: Magnetic shielding

Big toroidal fields obtained by means of cryogen free superconducting magnets .

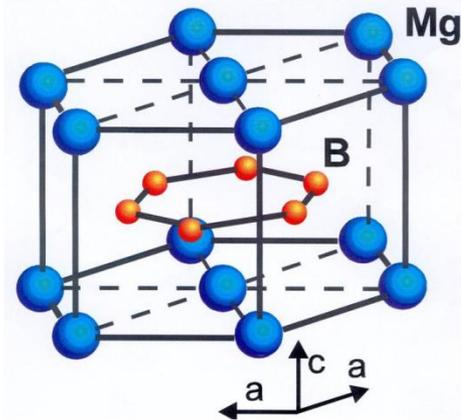


Expected stray field:
 $B \approx 0.1-0.2 \text{ T}$

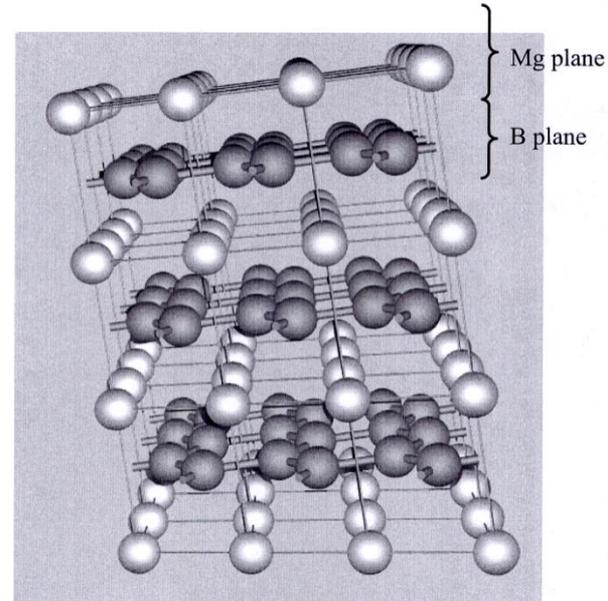
- ❖ Stray field is also a concern: to be kept below $5 \text{ G} = 0.5 \text{ mT}$
- ❖ Magnetic shielding is of interest also in MRI systems and for electronic equipment



Medium T_c SC: MgB_2



$$a=3.09 \text{ \AA} \quad c=3.52 \text{ \AA}$$



$$T_c = 40\text{K} \quad \rightarrow$$

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

Almost insensitive to grain boundaries \rightarrow

Excellent workability (lower cost compared to HTSC)

Theoretical density = $2.63 \times 10^3 \text{ kg/m}^3$ \rightarrow

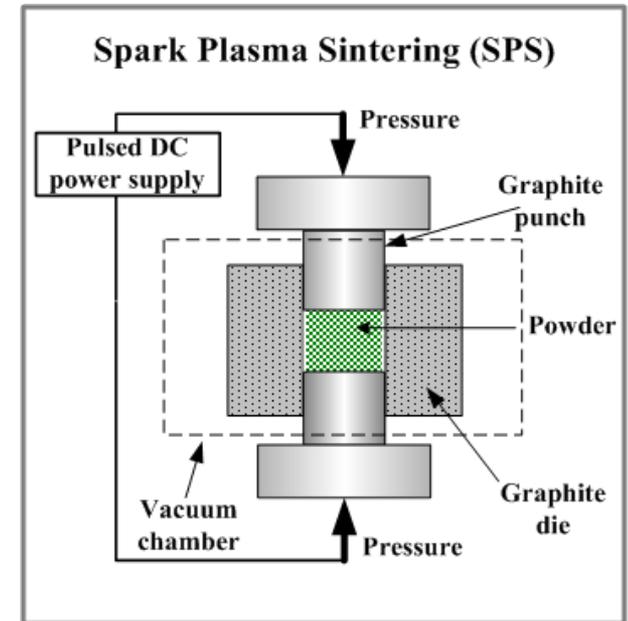
suitable for airborne applications



Sample preparation

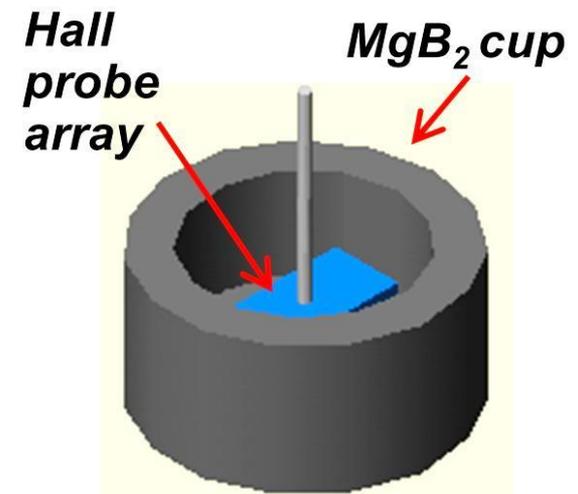
1) Spark plasma sintering (SPS):

MgB₂ 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

$T_c = 37.10$ K

Transition width ≈ 0.5 K

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

Magnetic field distribution

Experimental setup

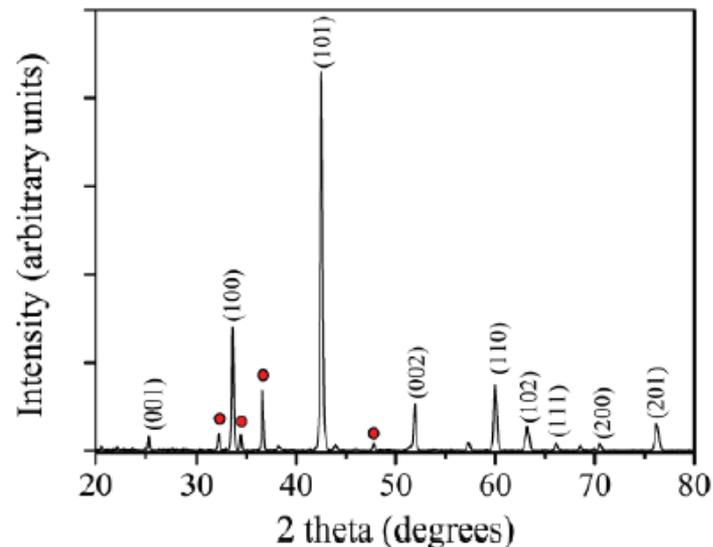
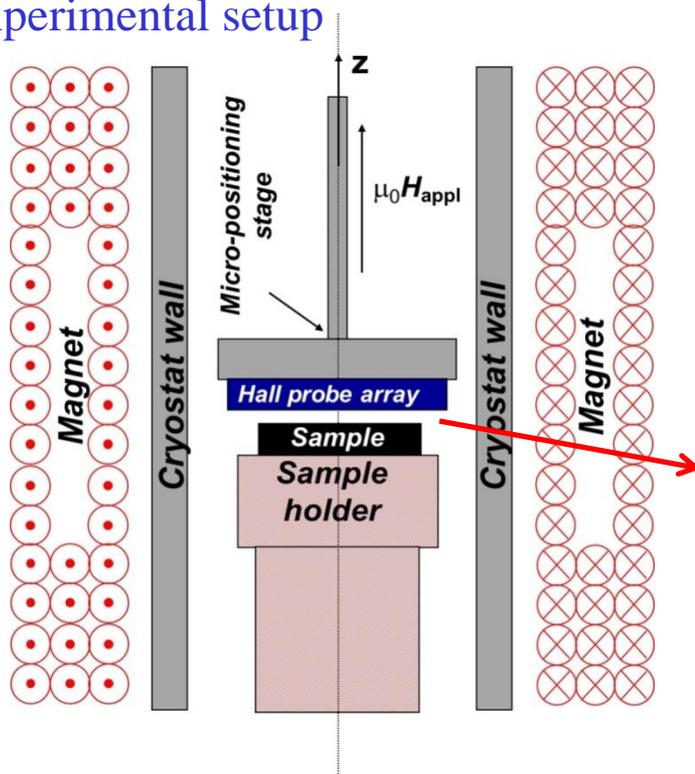
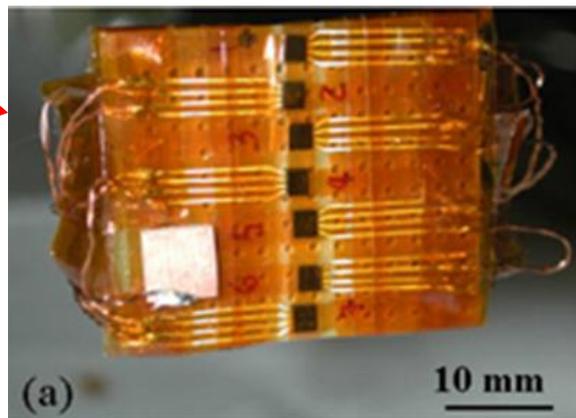


Table 1 MgB_2 lattice parameters of the synthesized samples

	Parameter value	sigma	95% conf.
a -axis (\AA)	3.08611	0.00013	0.00037
c -axis (\AA)	3.52466	0.00026	0.00073
Cell vol. (\AA^3)	29.0717	0.0026	0.0071



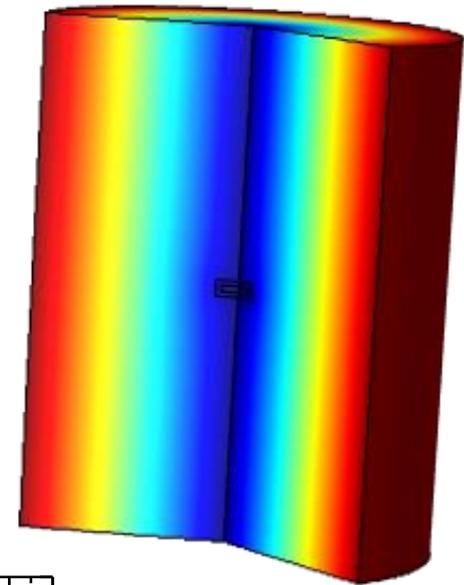
In co-operation with:





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

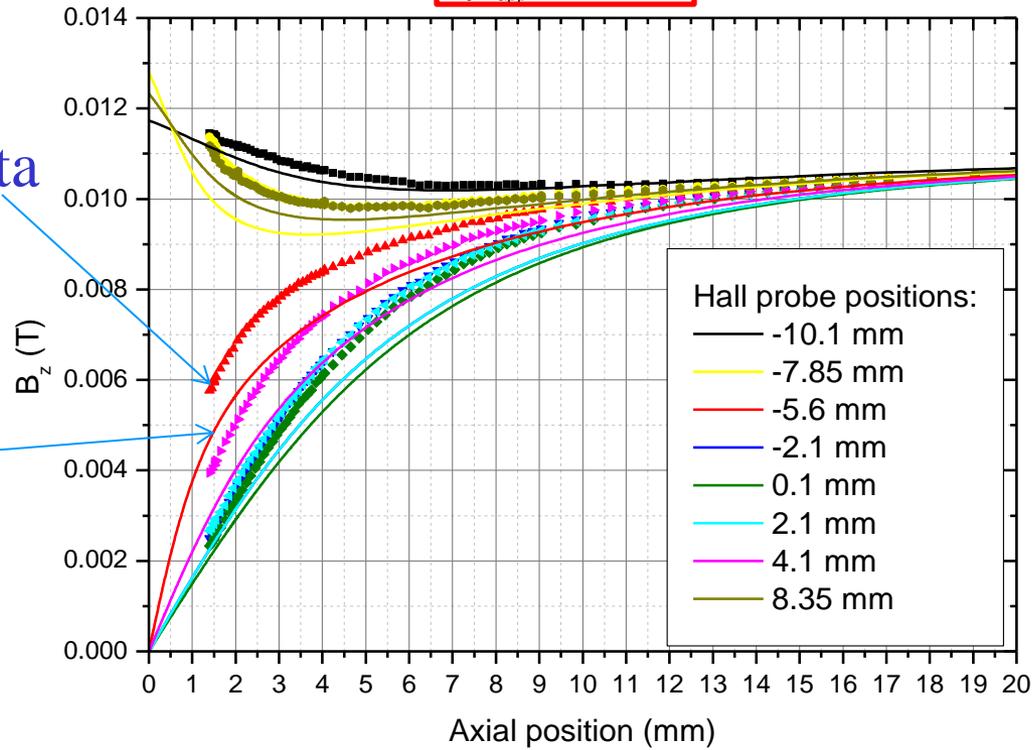


Low fields :

$$\mu_0 H_{\text{app}} = 0.0110 \text{ T}$$

Experimental data

Theoretical prediction



$$T = 20 \text{ K}$$

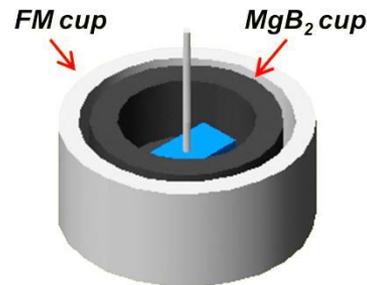
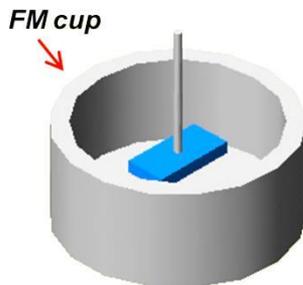
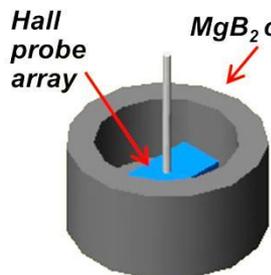


3 shield configurations:

Shielding factor

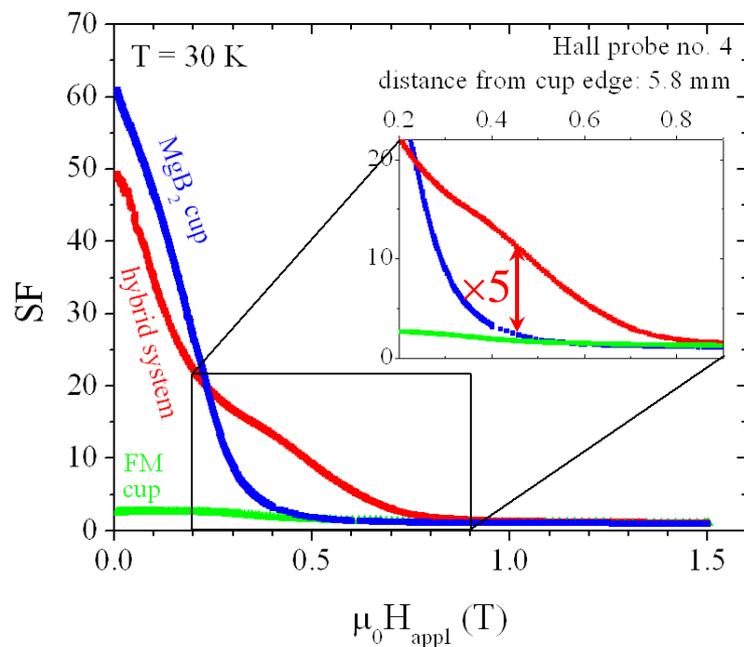
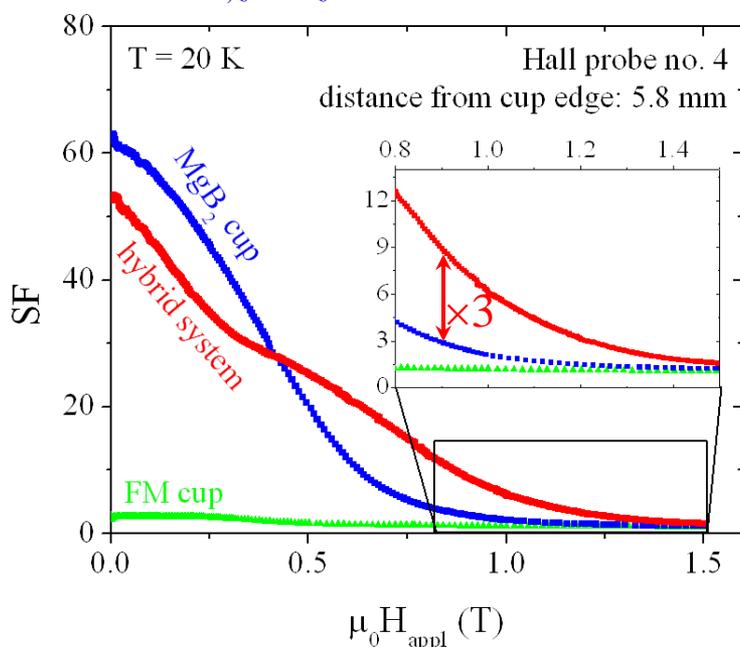
SC cup:

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

$$SF = B_{unsh,z} / B_z$$



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



Research theme

Experimental techniques for characterization and engineering of superconducting, magnetic and functional oxide materials, towards employment in innovative device applications

Tema della ricerca

Tecniche sperimentali per la caratterizzazione e l'ingegnerizzazione di materiali superconduttori, magnetici e ossidi funzionali, per impiego in dispositivi innovativi

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.

Attività di ricerca del gruppo

Il gruppo di ricerca è attivo da circa due decenni nel campo della **superconduttività sperimentale**, inizialmente nello studio degli effetti dell'**irradiazione con particelle sulla dinamica dei vortici** in superconduttori ad alta Tc, in collaborazione con INFN.

Da allora, il gruppo ha esteso la sua attività di ricerca a nuove tematiche, quali lo studio delle proprietà elettromagnetiche di **nuovi superconduttori** (attualmente quelli a base di ferro) ed **eterostrutture superconduttore/ferromagnete**, il design di **dispositivi innovativi basati su materiali superconduttori** (sensori THz, dispositivi a microonde, schermi magnetici) e l'uso di **facility di irradiazione con ioni pesanti energetici** per lo studio di meccanismi fondamentali e l'ingegnerizzazione di materiali funzionali.

Il gruppo dispone di diversi laboratori, tutti equipaggiati con sistemi criogenici. L'insieme delle tecniche (magneto-ottica e setup per caratterizzazioni strutturali, elettriche, magnetiche, a microonde e ottiche) consente di studiare efficacemente non solo materiali e dispositivi superconduttori, ma anche materiali magnetici e ossidi funzionali.

MAIN SUBJECTS for MS/PhD THESIS

ARGOMENTI PROPOSTI PER TESI MS/PhD

- 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

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www.polito.it/superconductivity



Topic n. 4: superconducting devices for basic physics research

In co-operation with :

AlbaNova
YSIK ASTRONOMI BIOTEKNIK



Typically 6 month stay in Stockholm at the nanofabrication lab.



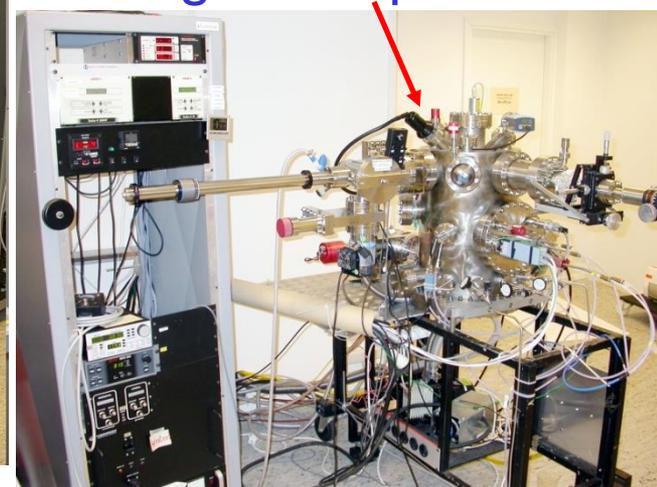
Clean room

SEM/FIB



E-gun evaporator

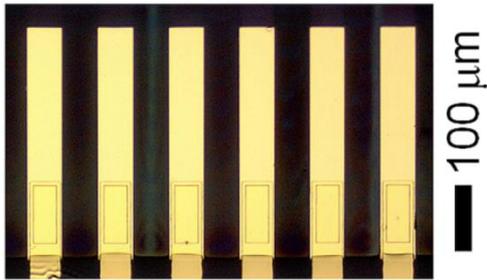
Ar milling





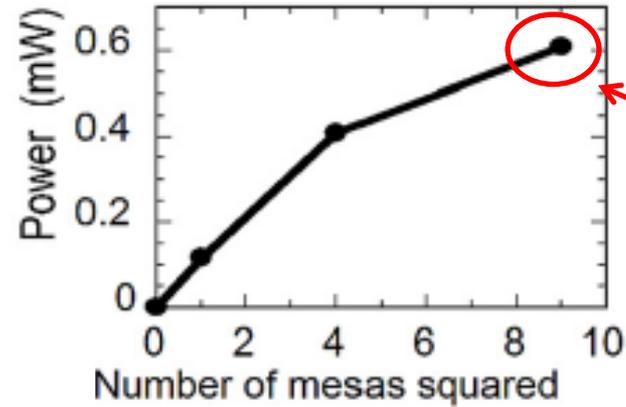
Example: THz emission

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

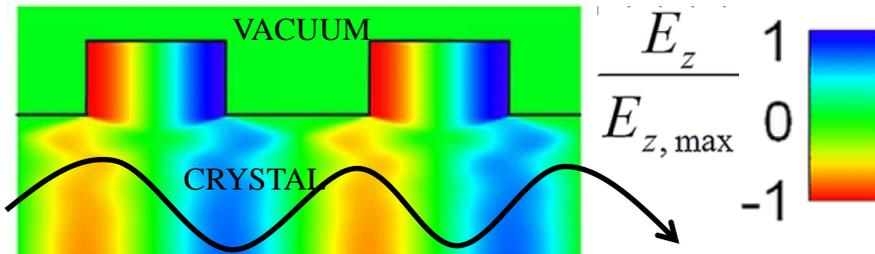


T.M. Benseman et al., Appl. Phys. Lett. **103**, 022602583 (2014)

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



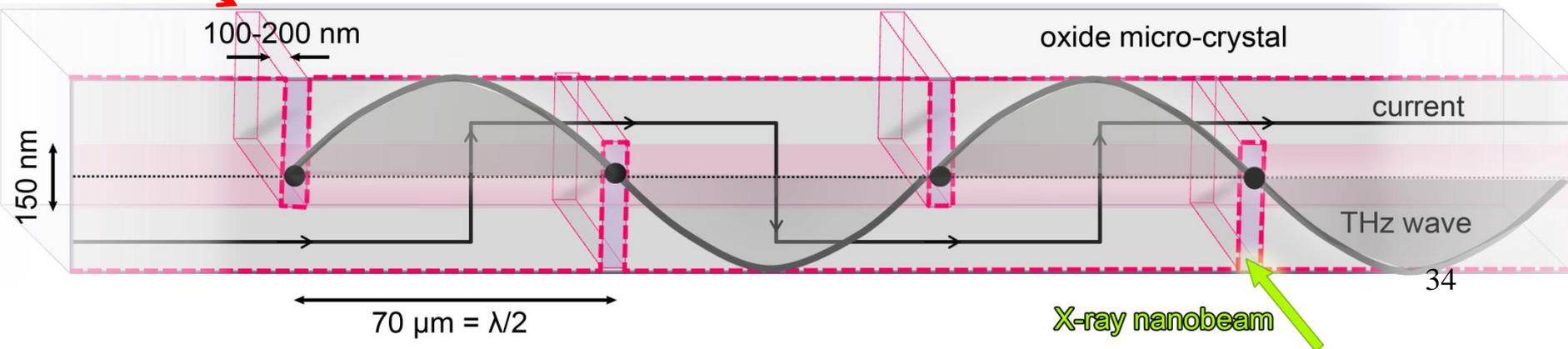
Present record



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

New design: Coupling obtained through the crystal itself

Optically transparent trench





For further information:

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room A37 – ph. 0116707374
marco.truccato@unito.it

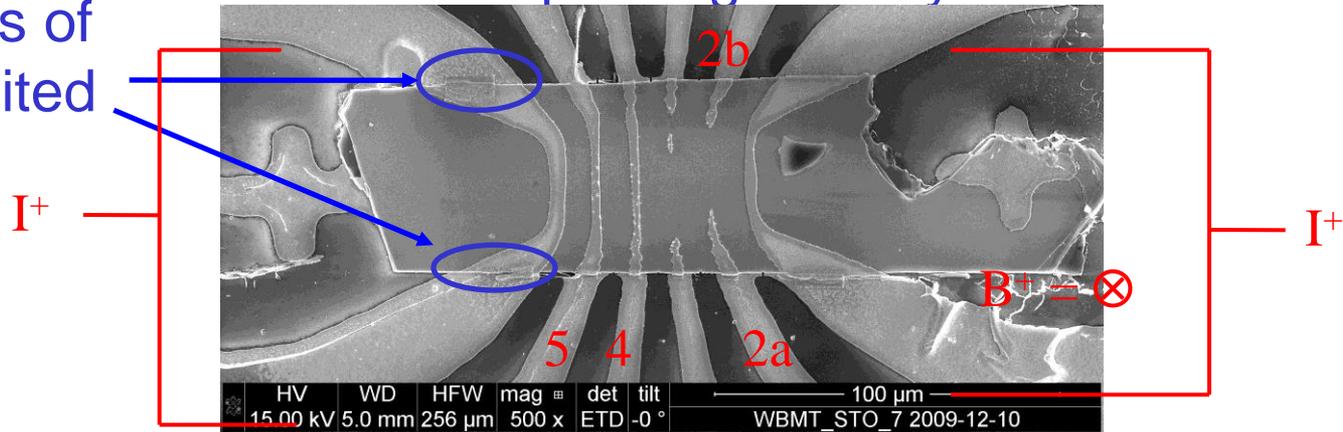




Example: study of the Hall effect in SC

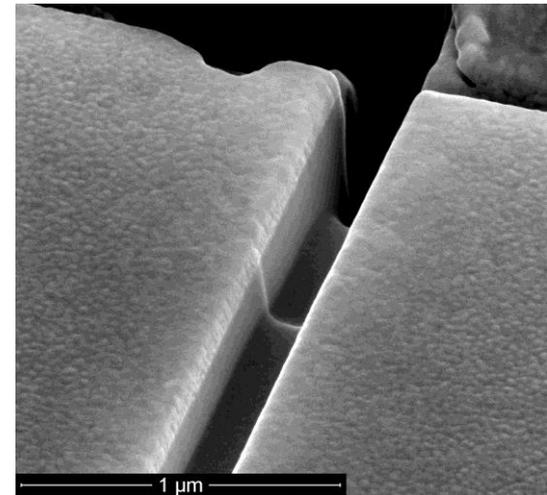
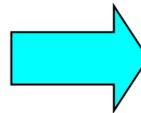
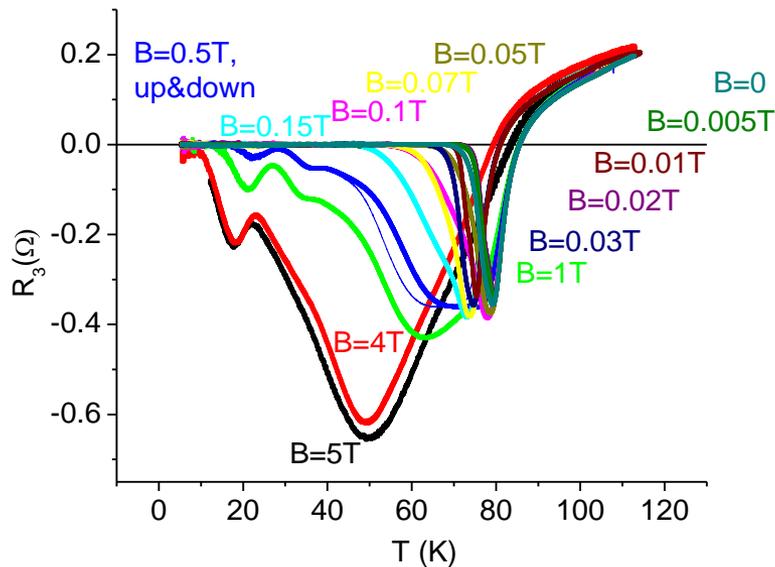
Hall probe geometry

Examples of Pt deposited by FIB



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

Transverse resistance R_3 , section 2a-2b



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