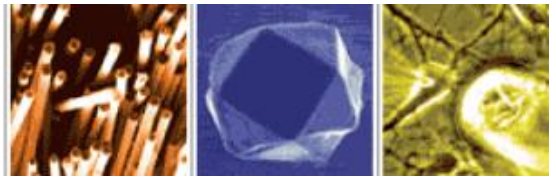


Direct-write X-ray nanopatterning for oxides

Lorenzo Mino

Department of Physics and NIS, University of Turin

X-ray induced modifications in materials: applications and challenges

Outline

- $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) properties
- Stacks of Intrinsic Josephson junctions (IJJ) and their applications
- Conventional fabrication process and related problems
- Doping change in Bi-2212 induced by synchrotron radiation
- Proof of concept of X-ray nanopatterning: fabrication of IJJ devices
- Preliminary investigation of the mechanism
- Conclusions and future perspectives

$Bi_2Sr_2CaCu_2O_{8+\delta}$ crystal structure

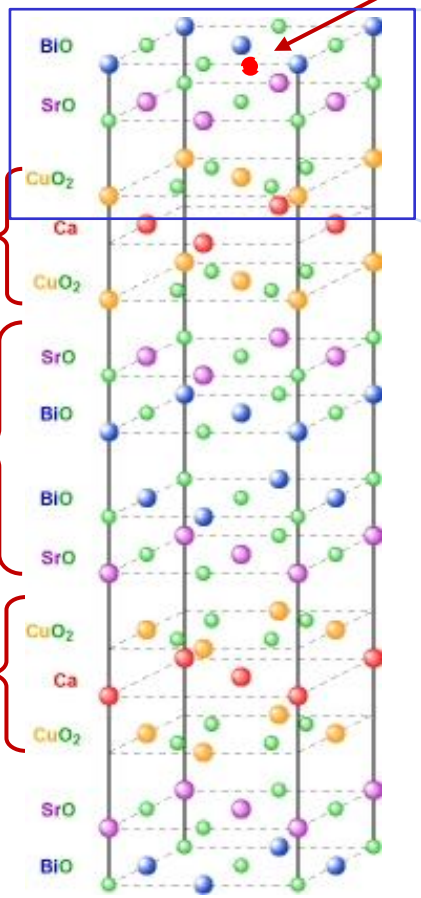
Anisotropic crystal structure

$Bi_2Sr_2CaCu_2O_{8+\delta}$:
$T_c \approx$ 80 K, depending on δ

a=5.407 Å

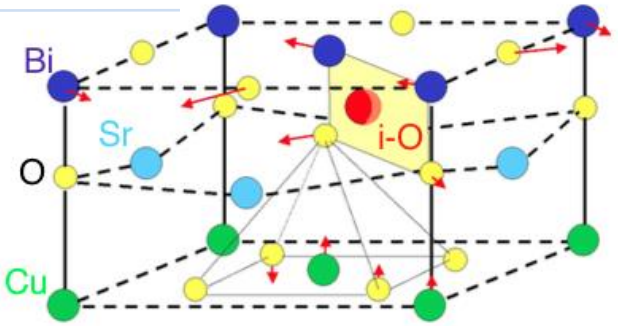
b=5.432 Å

c=30.931 Å

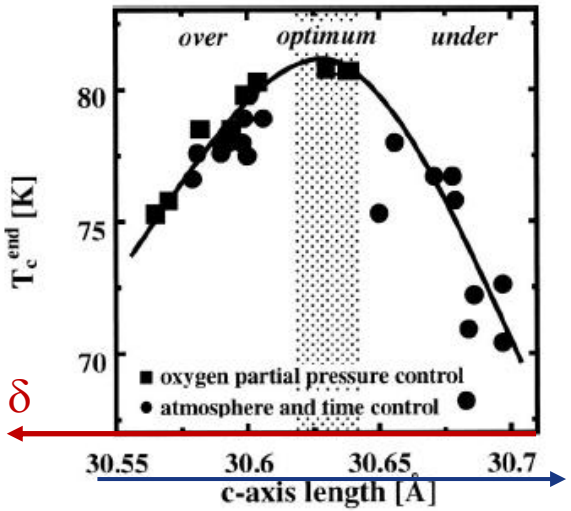


R.H. Arendt et al., Physica C, **182**, 73, (1991)

Non- stoichiometric O content



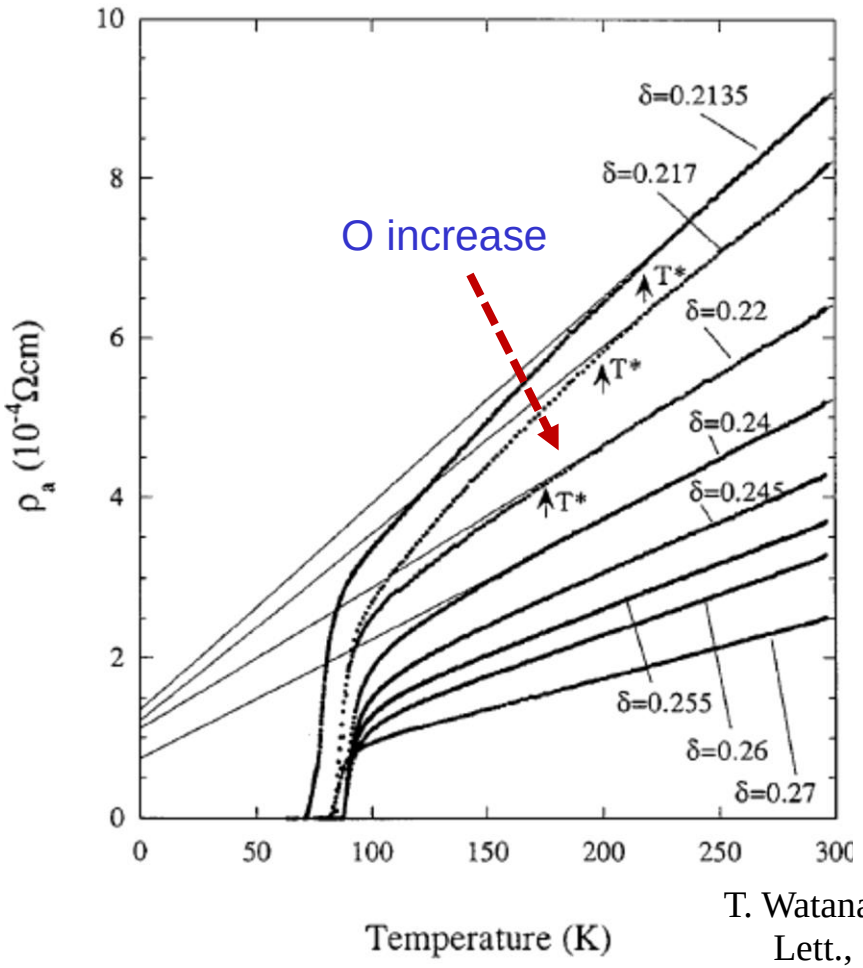
c-axis and O content correlation



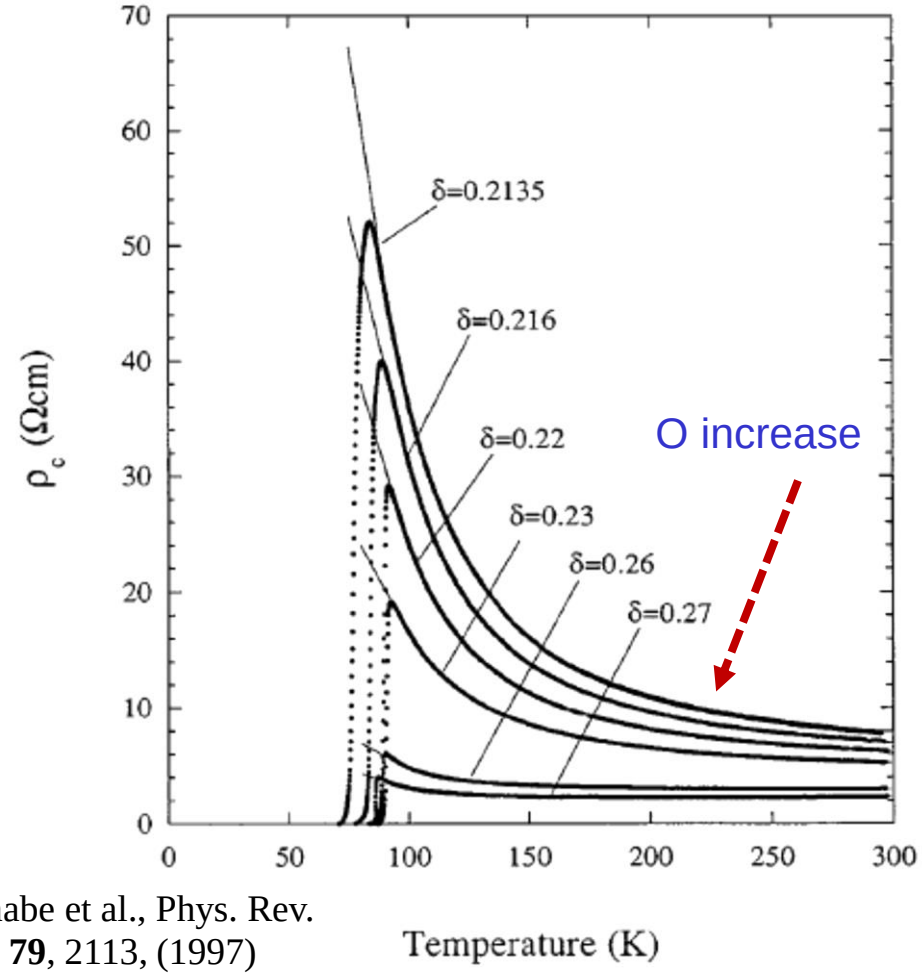
K. Inomata *et al.*, Appl. Phys. Lett. **82**, 769, (2003)

$Bi_2Sr_2CaCu_2O_{8+\delta}$ electrical properties

ab-plane



c-axis

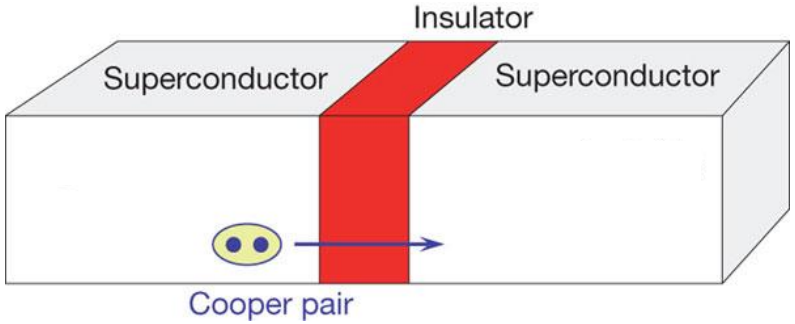


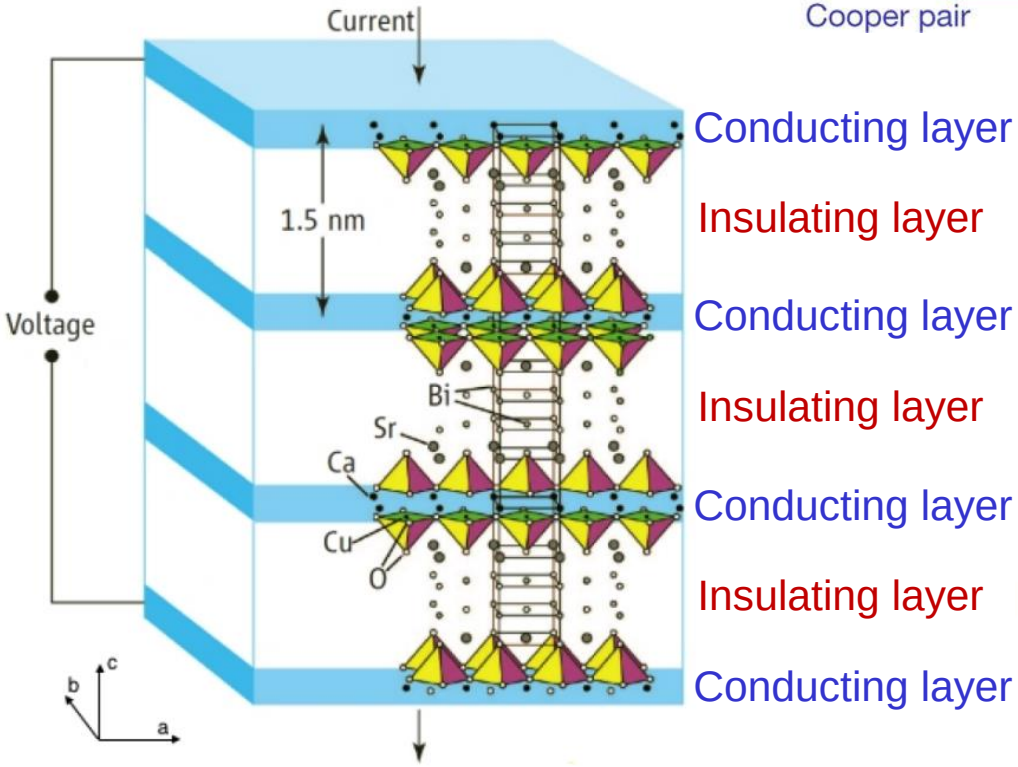
T. Watanabe et al., Phys. Rev. Lett., **79**, 2113, (1997)

$Bi_2Sr_2CaCu_2O_{8+\delta}$: $\rho_c / \rho_{ab} \approx 10^5$

Josephson junctions

Conventional Josephson junction





Conducting layer

Insulating layer

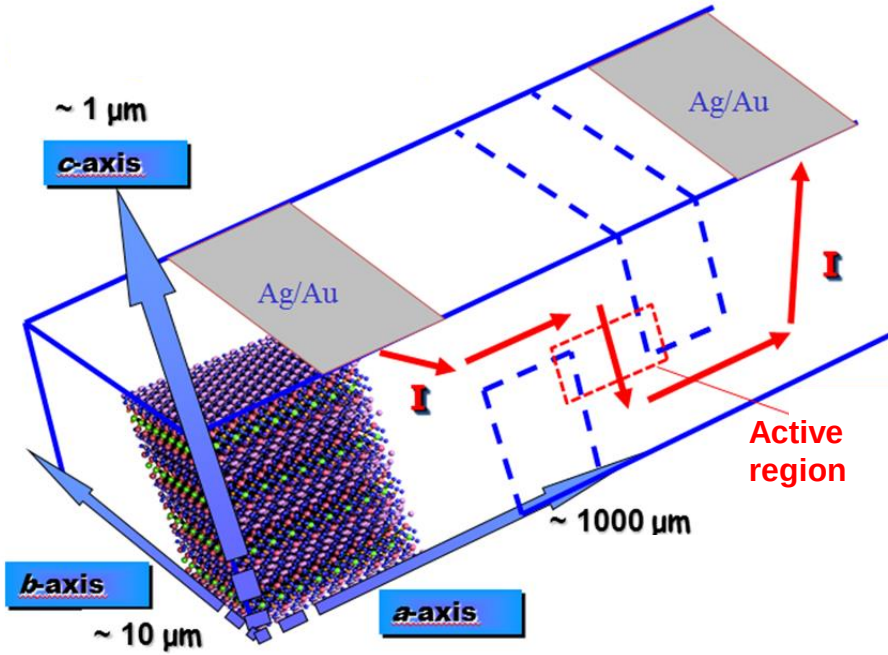
Conducting layer

Insulating layer

Conducting layer

Insulating layer

Conducting layer



Intrinsic Josephson junctions in Bi-2212

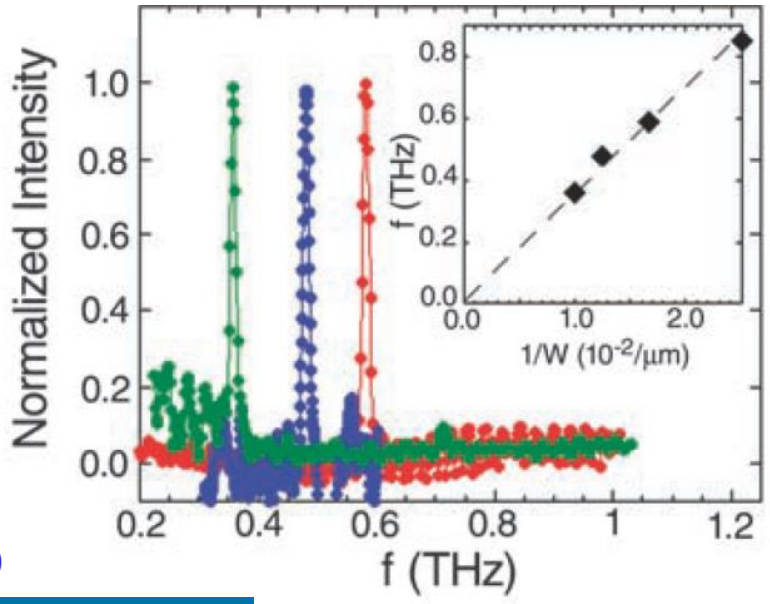
Etching at the nanoscale is required

IJJ applications

THz radiation emitters

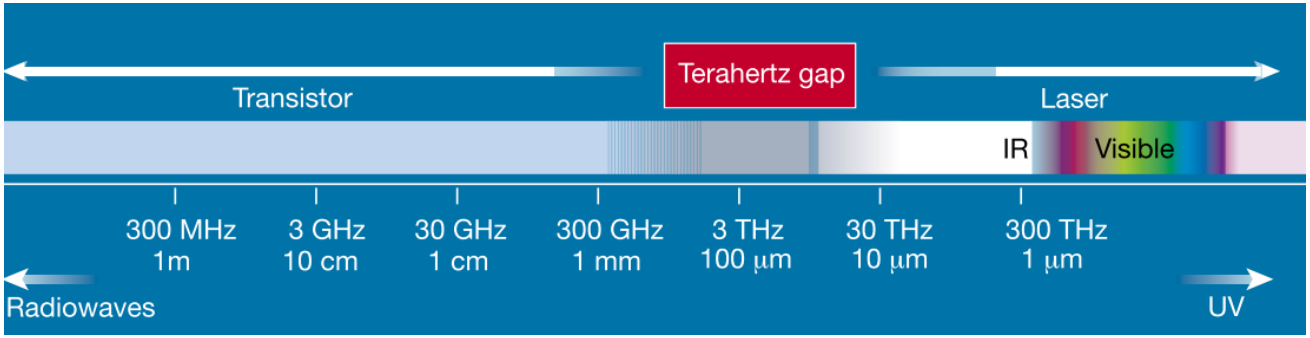
THz radiation of sizable power can be extracted from intrinsic Josephson Junctions of Bi-2212

$V = 1$ mV $\Leftrightarrow f_J = 0.483$ THz



L. Ozyuzer *et al.*, Science **318**, 1291, (2007)

Possible bridge for the terahertz gap

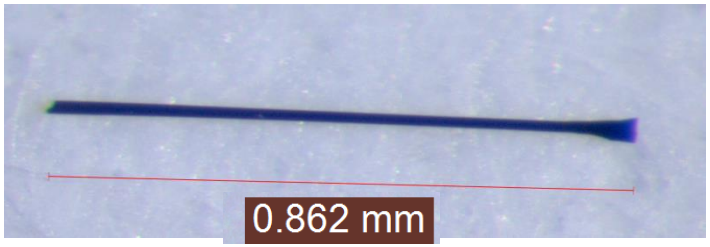


C. Sirtori, Nature **417**, 132, (2002)

Sample fabrication process

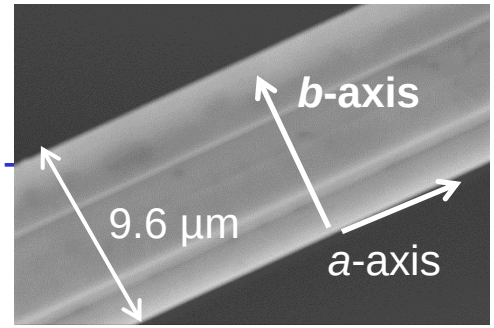
Growth of Bi-2212 whiskers

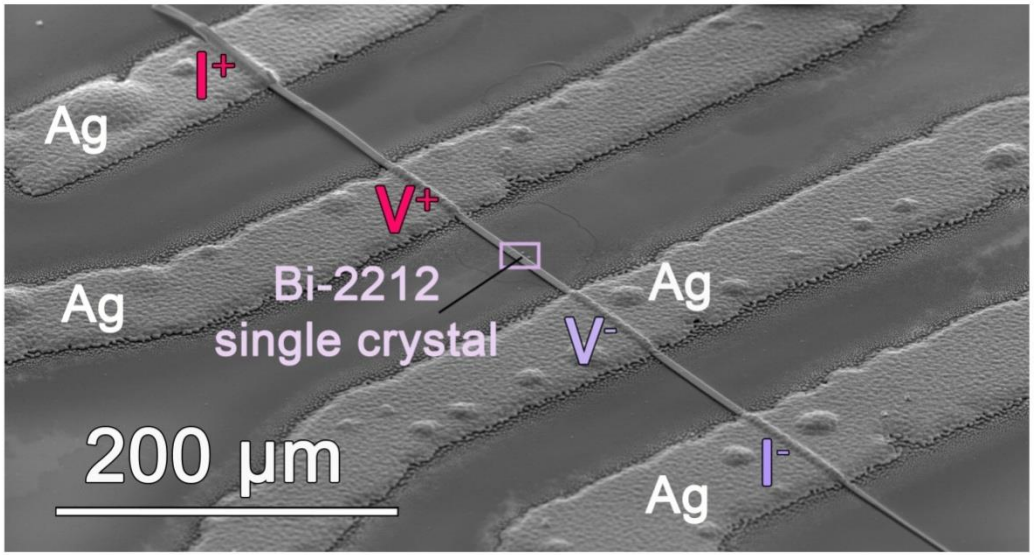
Single crystals with high aspect ratio



a-axis : *b*-axis : *c*-axis ≈ 1000 : 10 : 1

Growth:
862 ˚C for
5 days

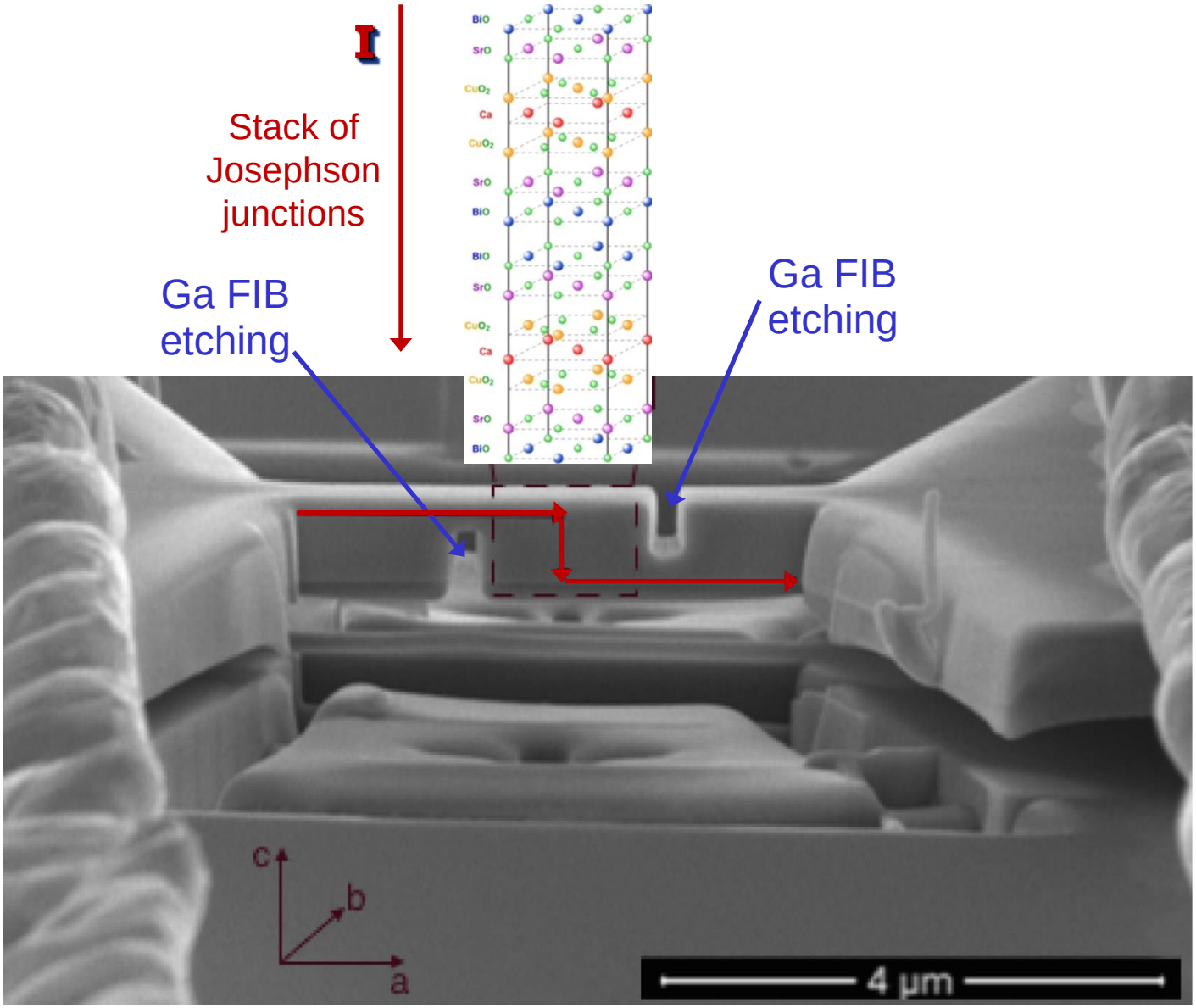




Contacting of Bi-2212 whiskers

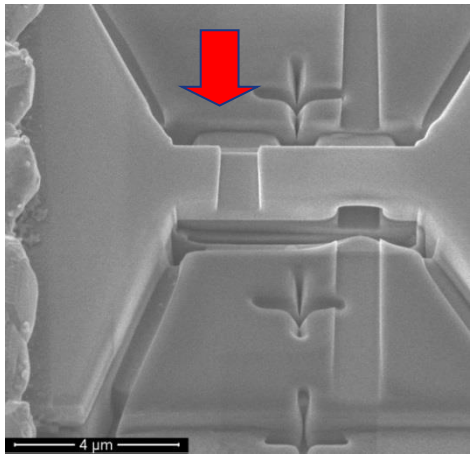
Whiskers mounted on a chip to measure the electrical properties of Bi-2212 along precisely known crystal directions

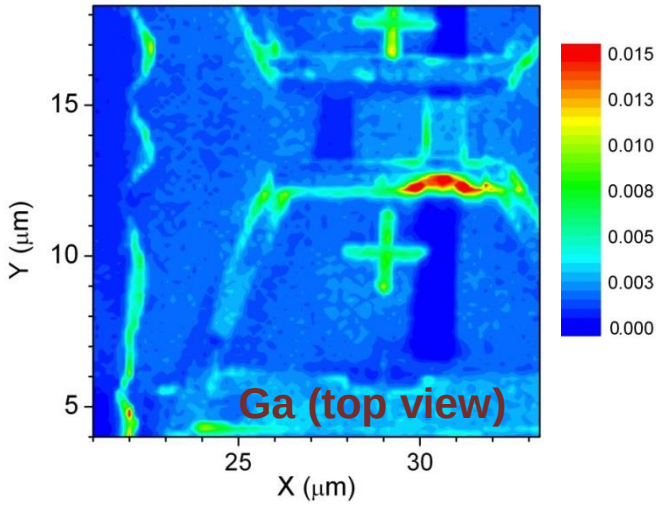
IJJ device realization

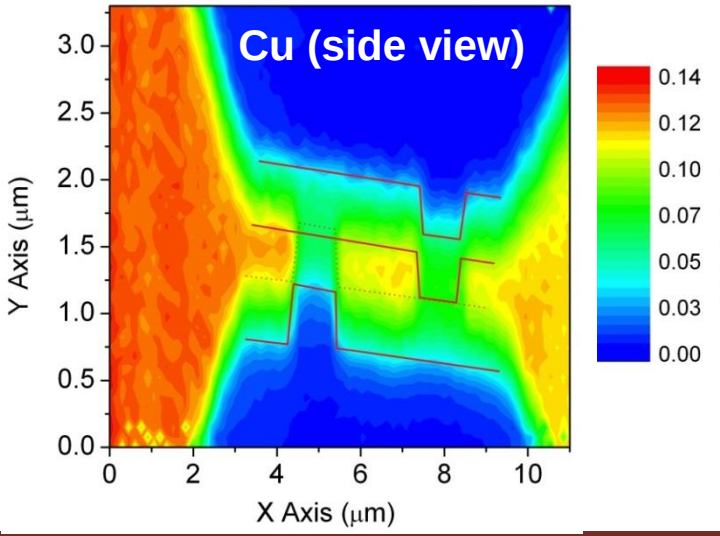


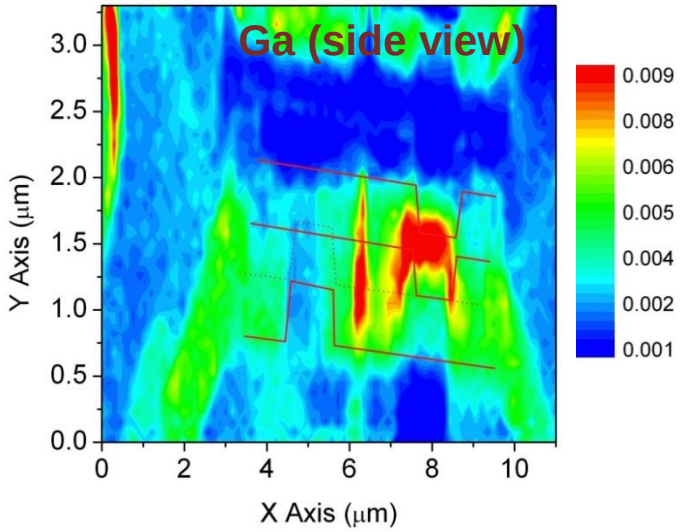
Device nano-XRF characterization

ID22, ESRF, Grenoble. Nanoprobe: 117 (v) × 116 (h) nm^2









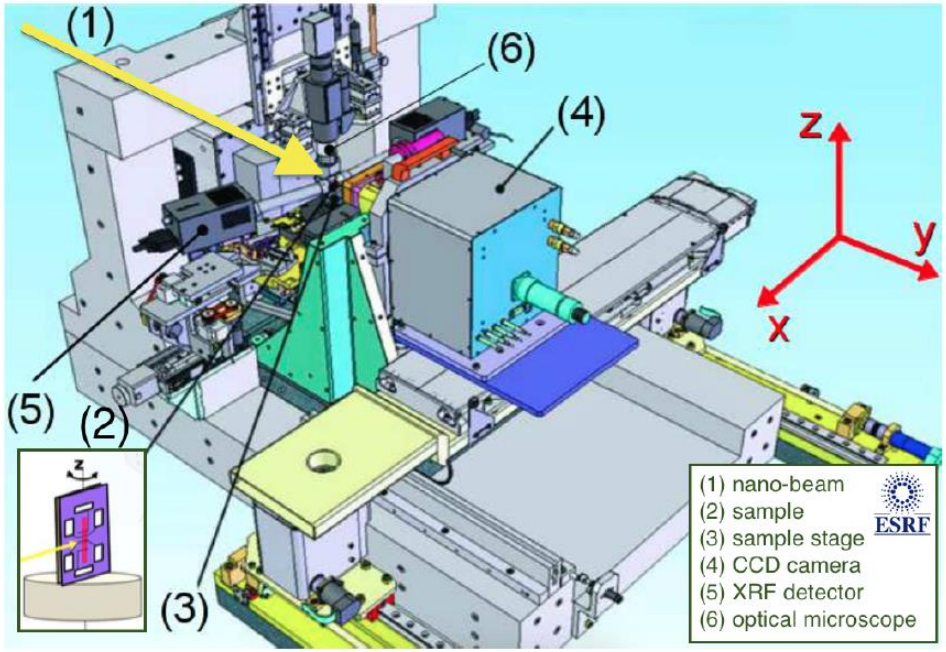
Ga contamination

Ga FIB etching issues

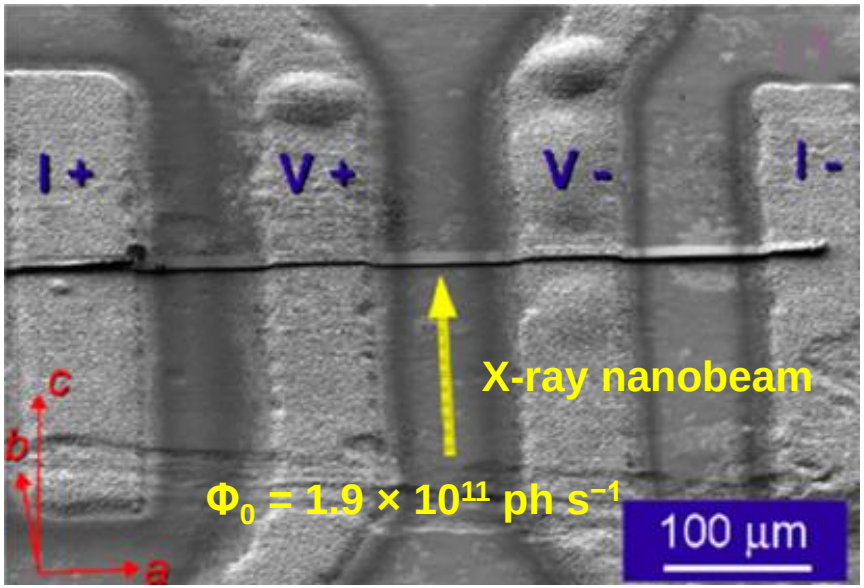
Mechanical instability

Other strategies?

O doping modification by SR



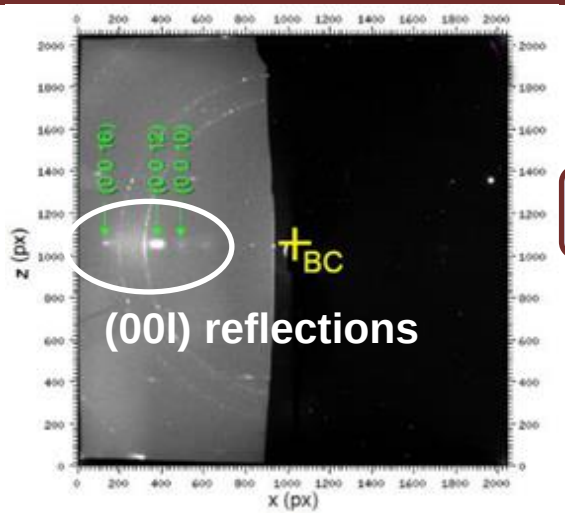
Chip for combined XRD and electrical measurements on a Bi-2212 whisker



- ID22, ESRF, Grenoble
- 17 keV nanoprobe: 117 (v) × 116 (h) nm^2
- Monochromatic beam

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

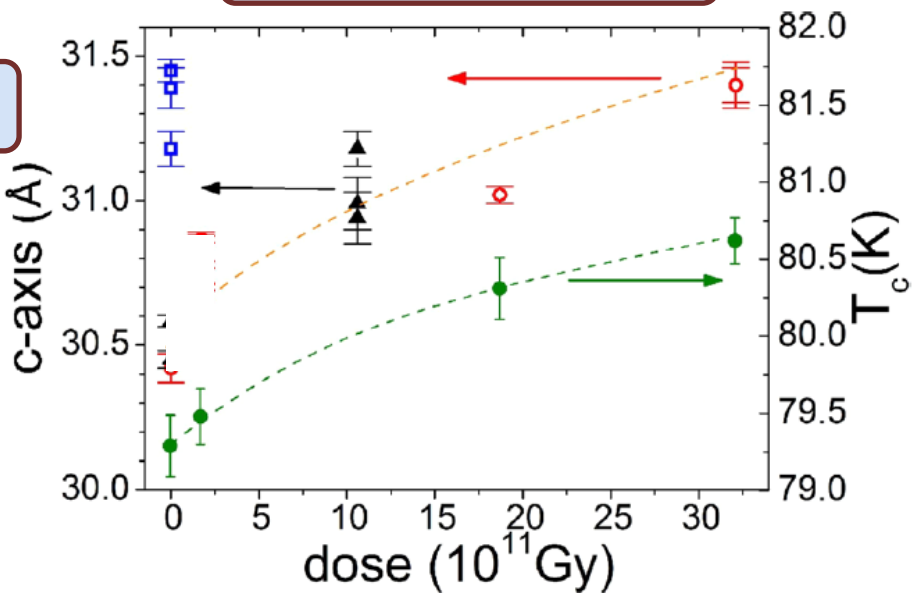
Irradiation effects



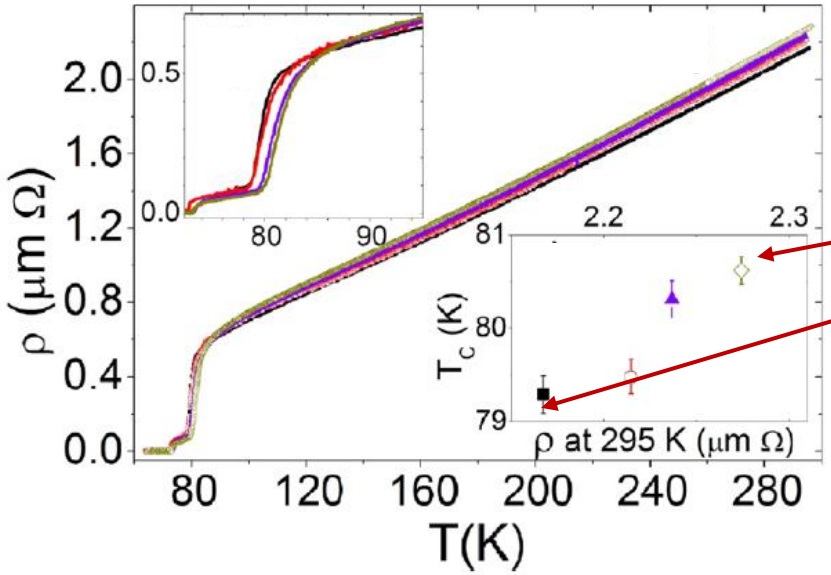
A. Pagliero, L. Mino et al., Nano Lett. **14**, 1583 (2014)

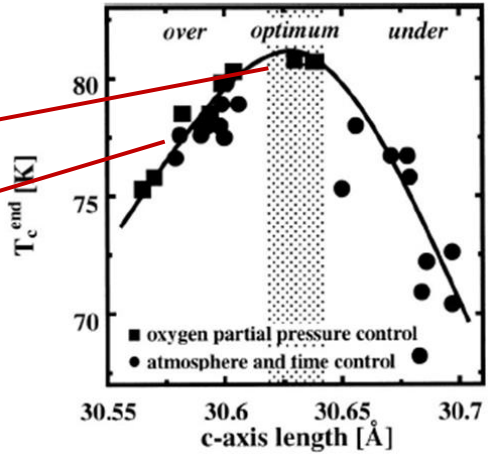
Nano-XRD

Changes in c-axis



Changes in T_c and resistivity

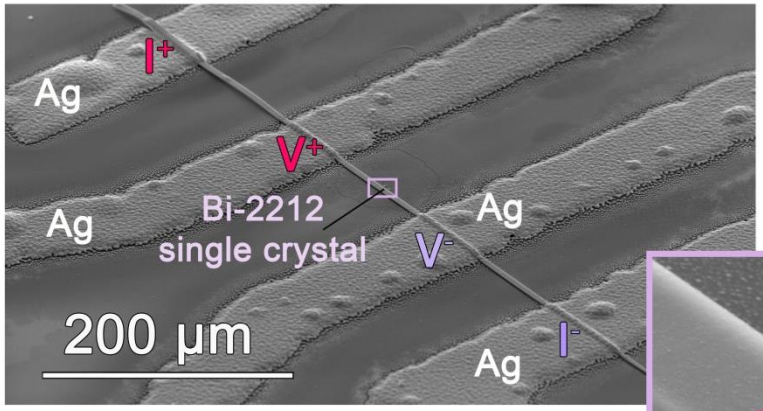


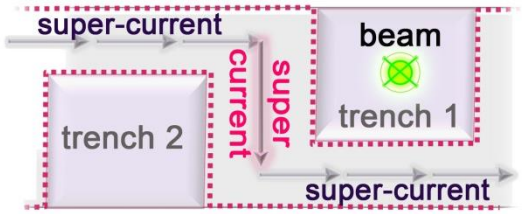


Transition from overdoped to optimally doped

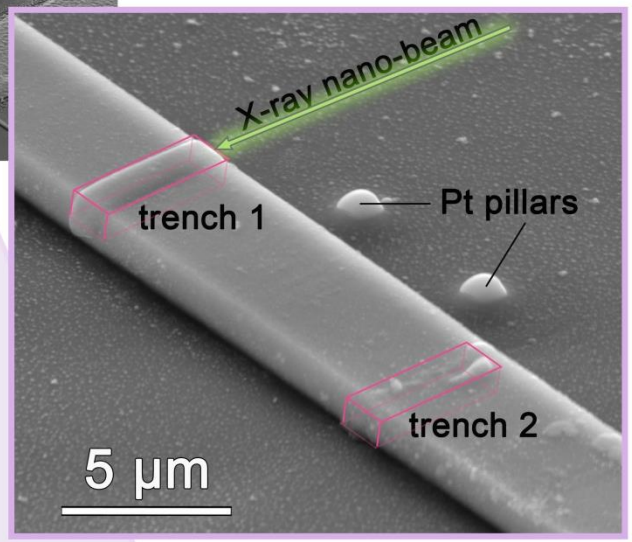
K. Inomata *et al*., Appl. Phys. Lett. **82**, 769, (2003)

X-ray nanopatterning

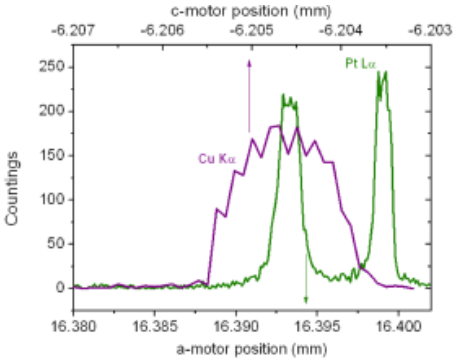


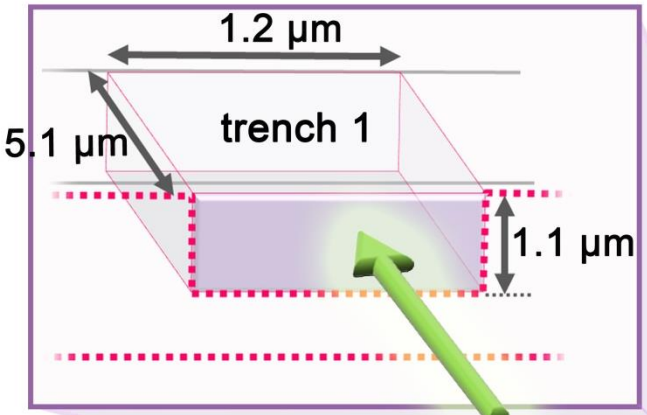


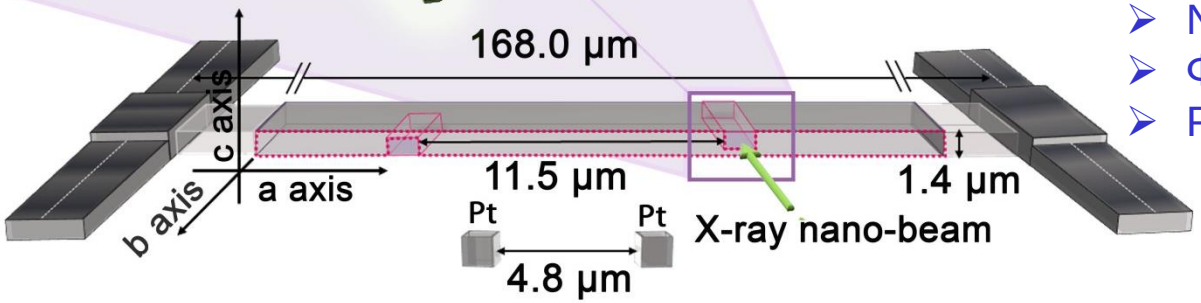
M. Truccato, A. Agostino, E. Borfecchia, L. Mino et al., Nano Lett. **16**, 1669 (2016)



Pt pillars for XRF alignment

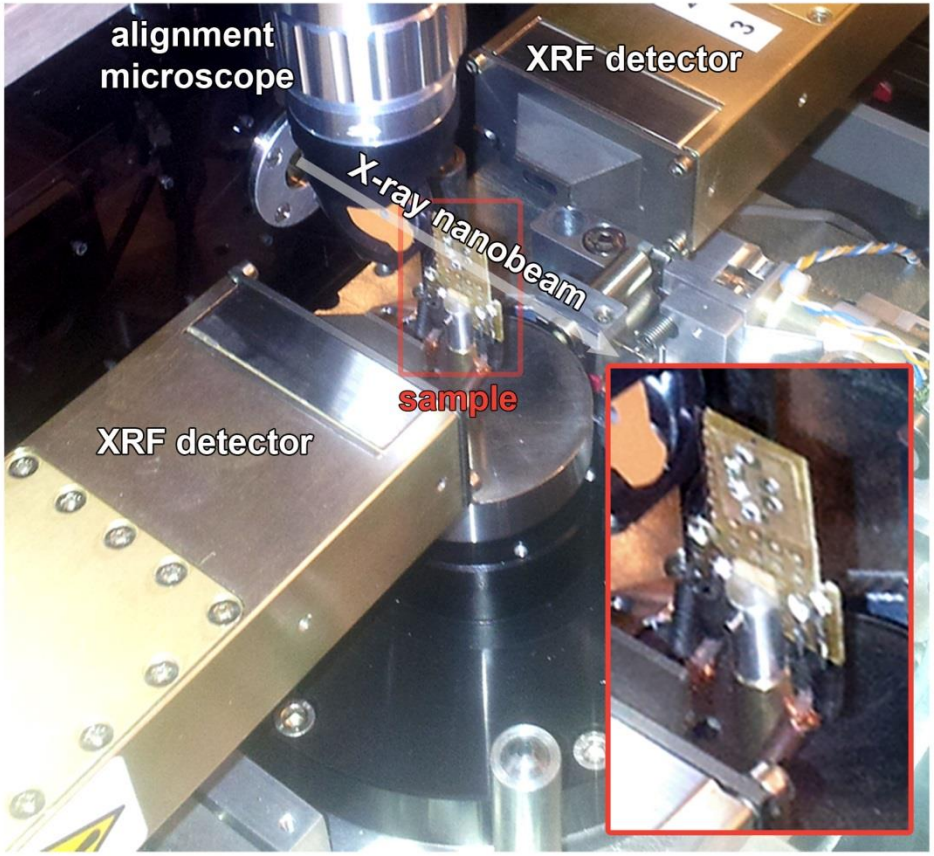






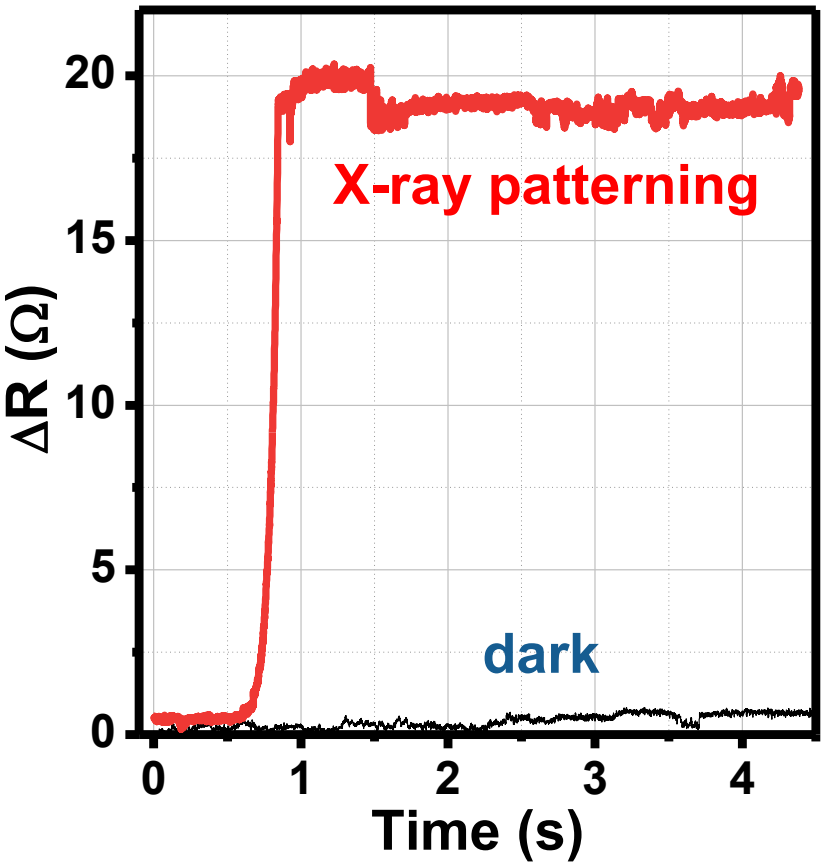
- ID16-NA, ESRF, Grenoble.
- Nanoprobe: 53 (v) × 45 (h) nm^2
- $\Phi_0 = (1\text{-}5) \times 10^{11}\ s^{-1}$, E=17.6 keV
- Pink-beam mode: $\Delta E/E \approx 10^{-2}$

In situ monitoring of nanopatterning process

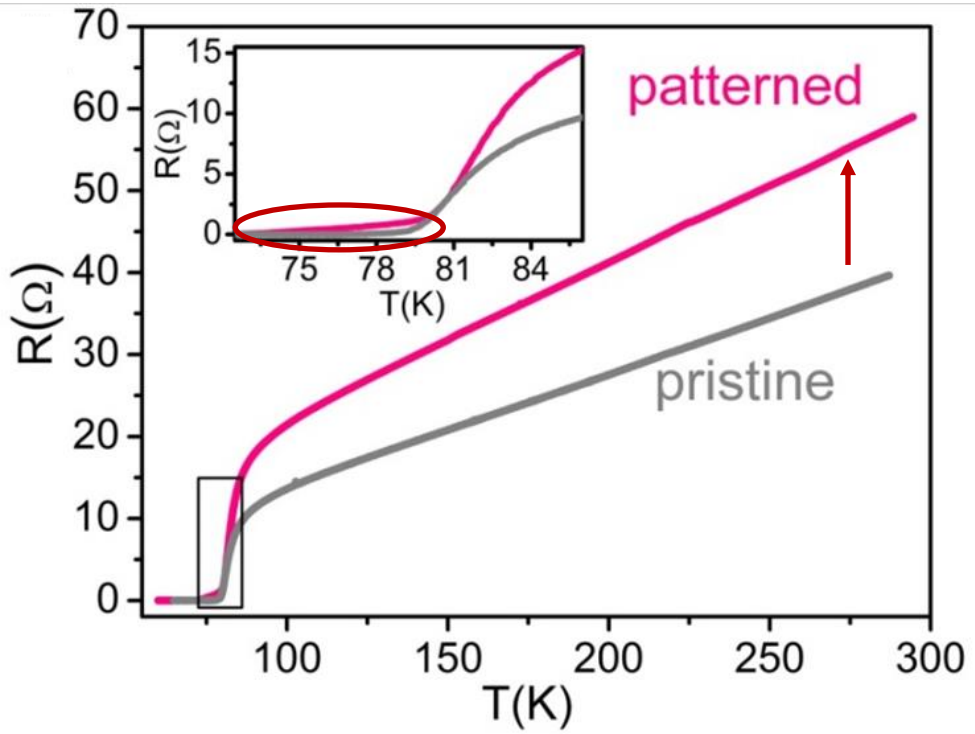


Resistance increase

oxygen loss



Electrical effects

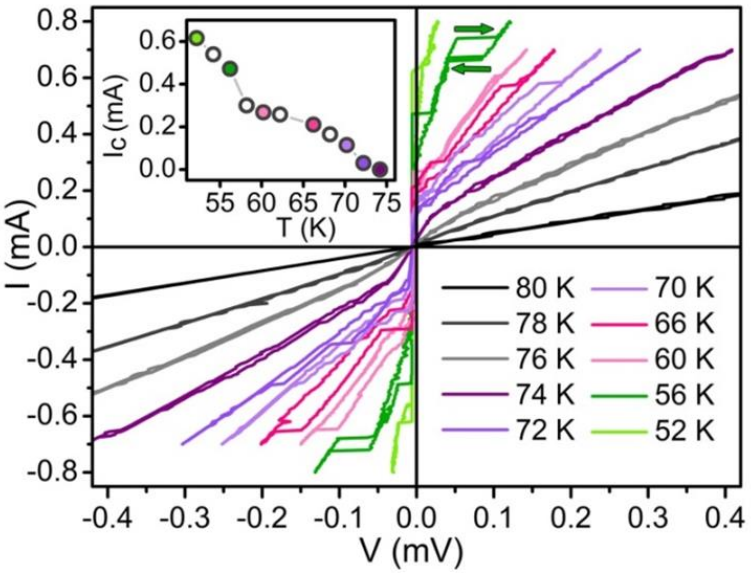


Resistance increase

oxygen loss

Resistance tail for 72 K < T < T_c

partially O-depleted region in-series

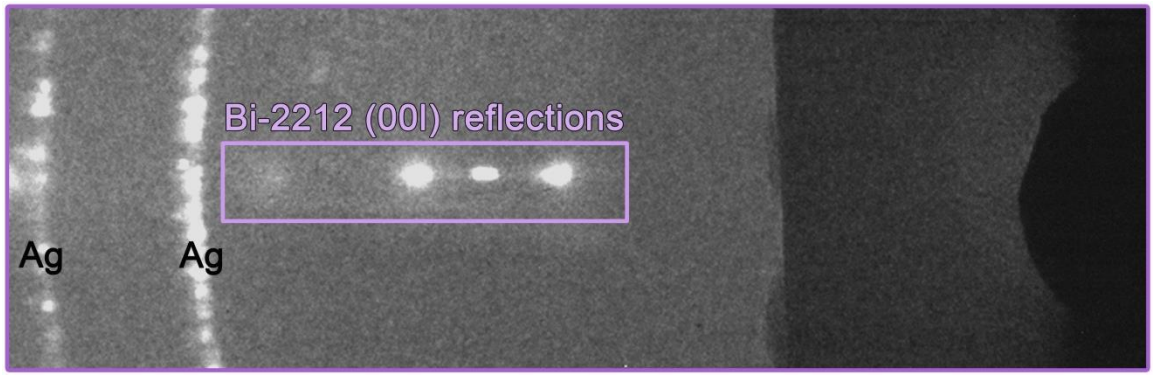


Appearance of Josephson branches for T < 72 K

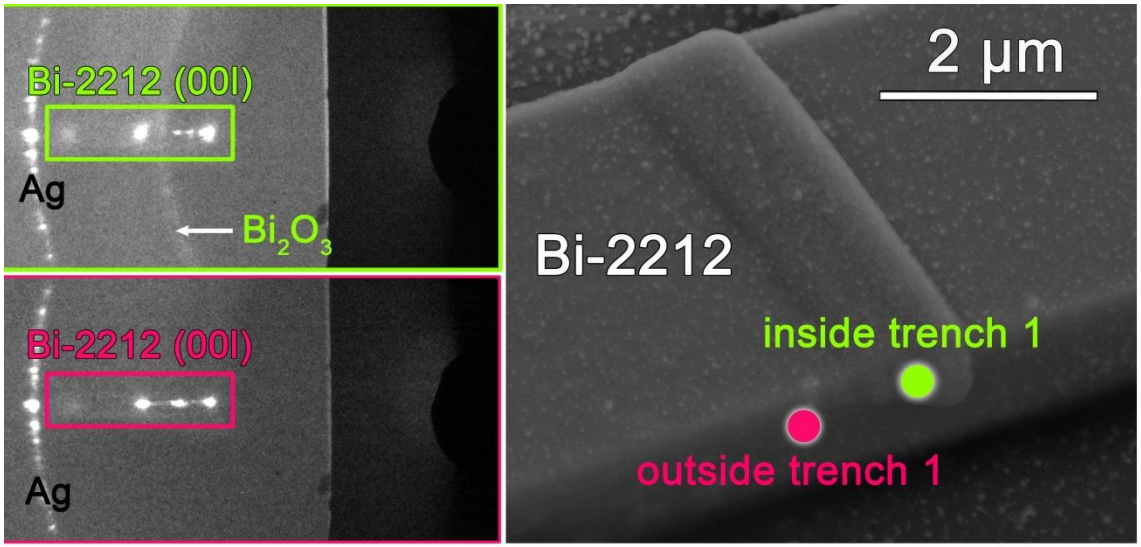
M. Truccato, A. Agostino, E. Borfecchia, L. Mino et al., Nano Lett. **16**, 1669 (2016)

Nano-XRD

As grown



After X-ray nanopatterning

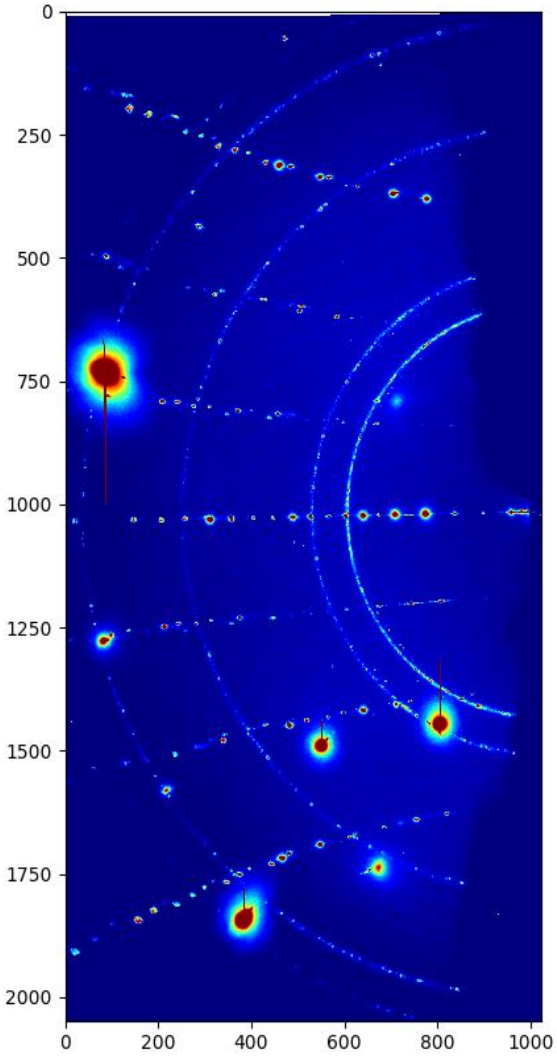


Local crystal expansion along the *c*-axis, but not crystal disruption

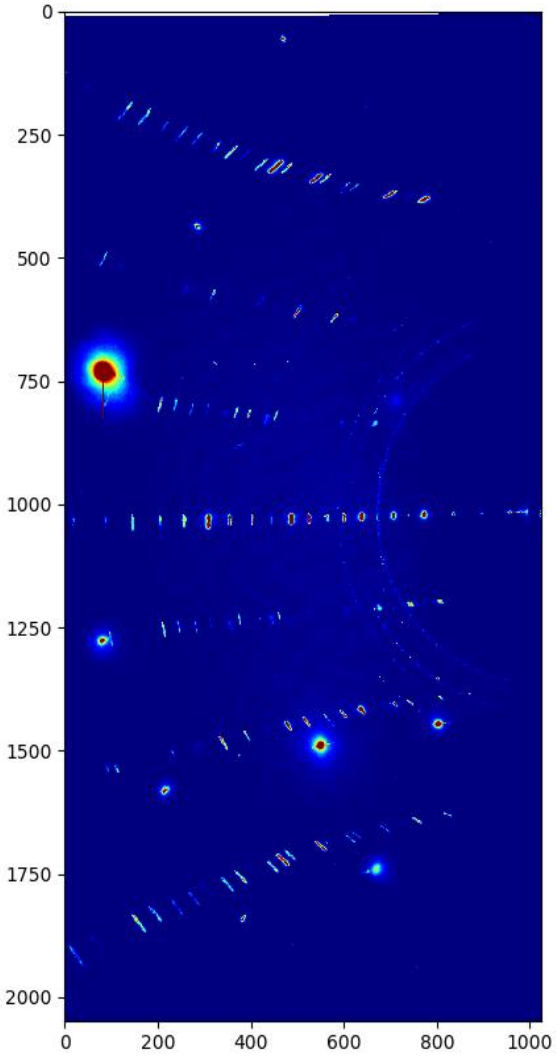
O depletion confirmed

M. Truccato, A. Agostino, E. Borfecchia, L. Mino et al., Nano Lett. **16**, 1669 (2016)

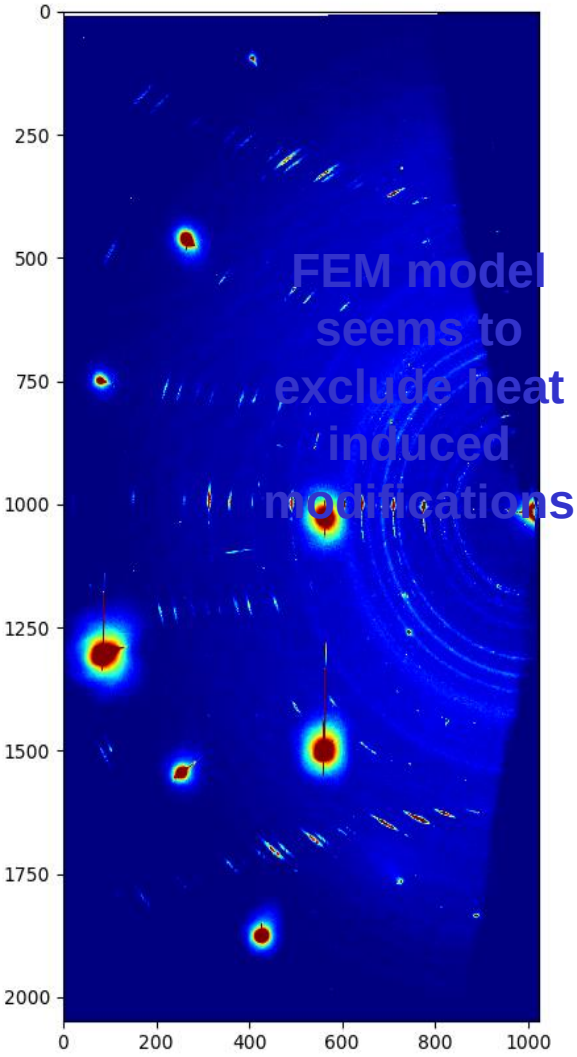
Micro-XRD @ ID13 ESRF beamline



Before irradiation



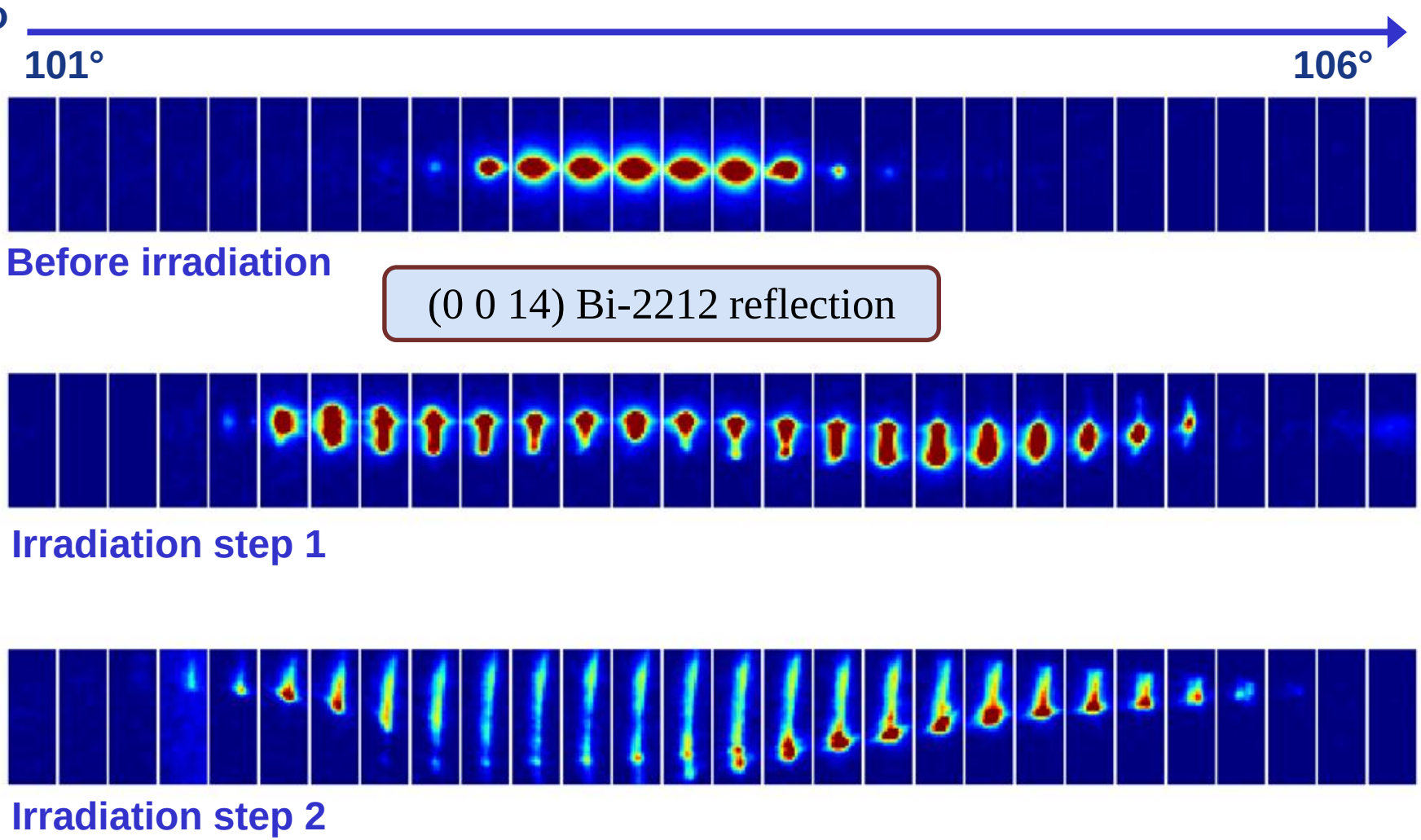
Irradiation step 1



Irradiation step 2

FEM model seems to exclude heat induced modifications

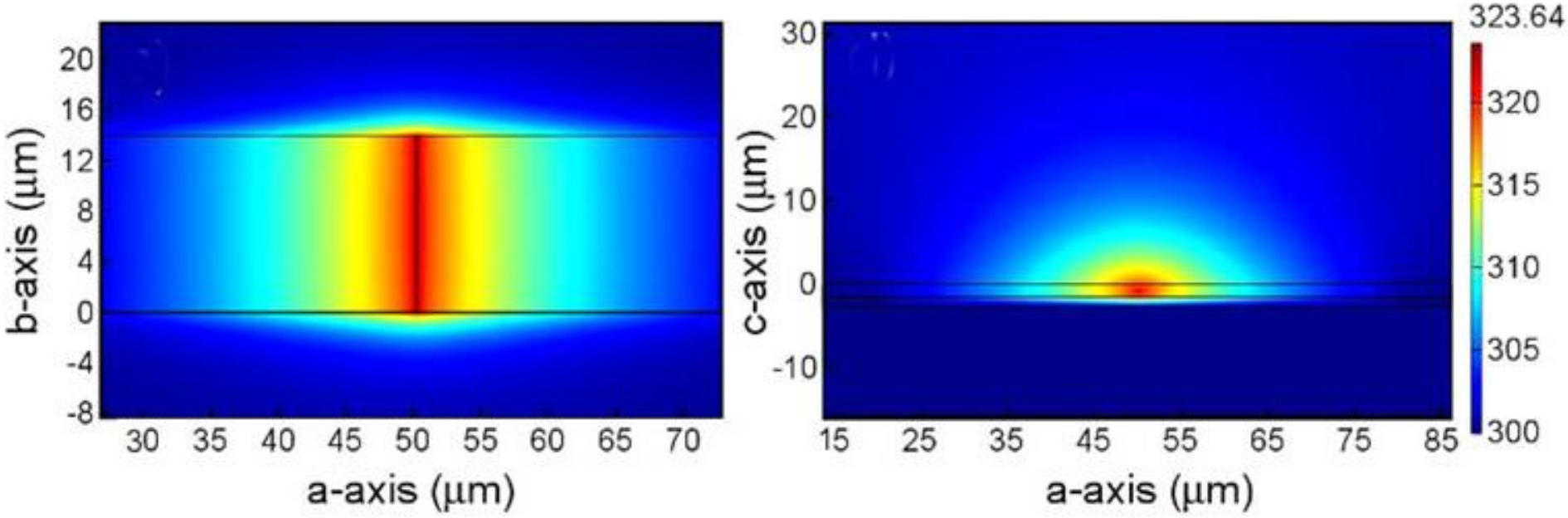
Micro-XRD @ ID13 ESRF beamline



Beam heating effect?

FEM model for beam heating

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



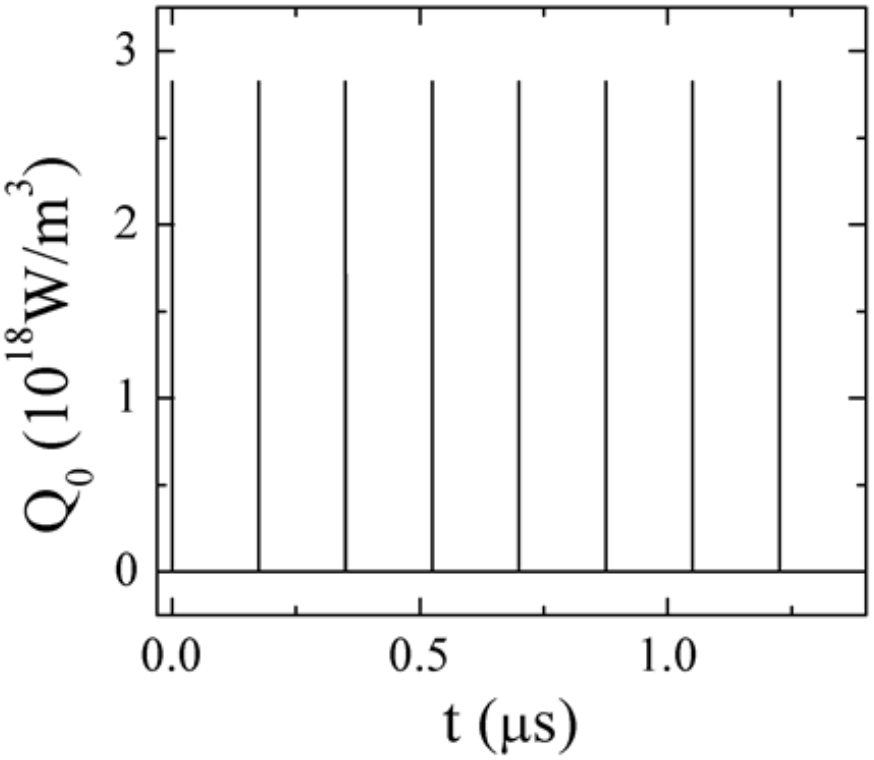
Temperature field during irradiation in steady state conditions

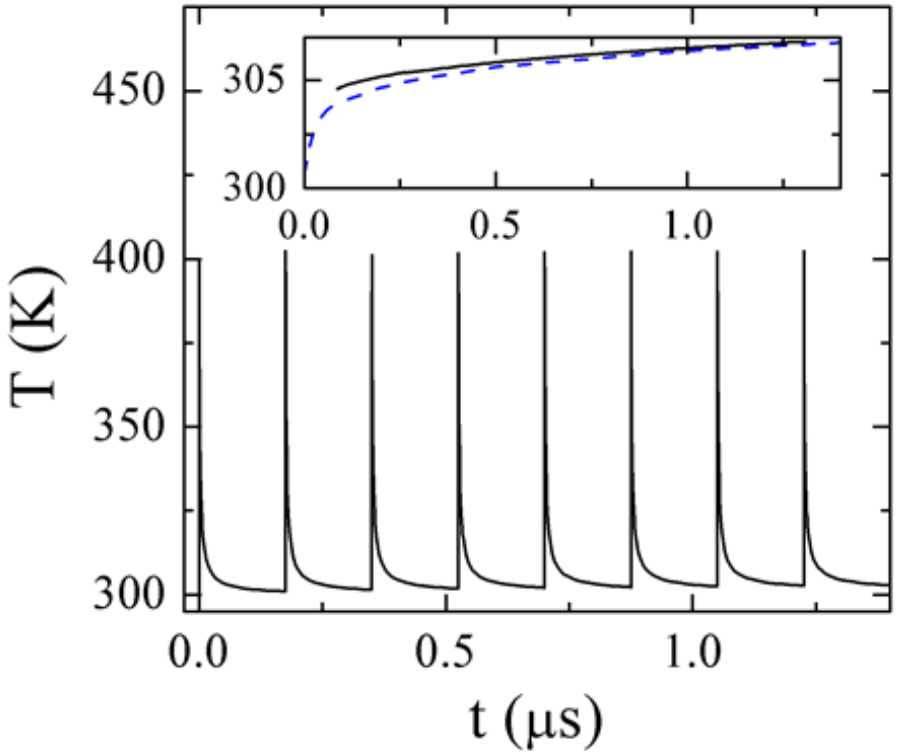
Beam size = 117 x 116 nm^2

Flux = 2 x 10^{11} photons s^{-1}

Effect of the synchrotron time structure

16-bunch filling mode



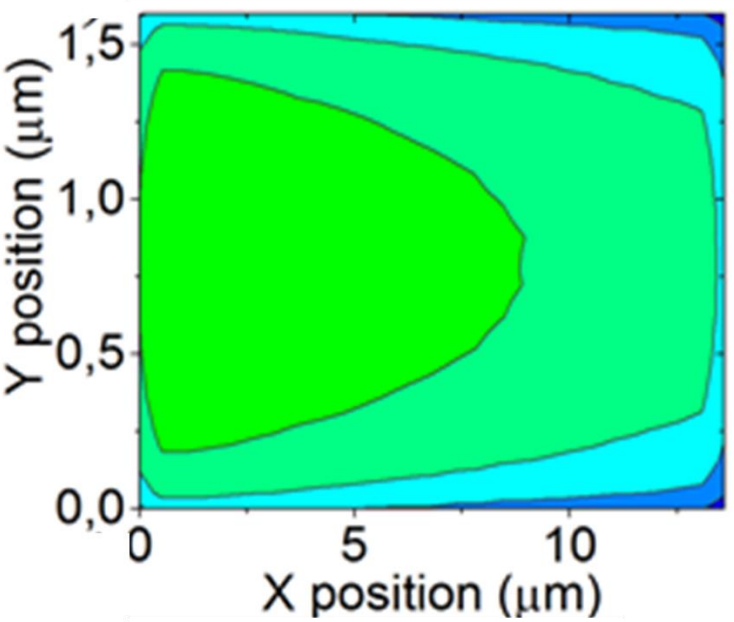


Significantly higher instantaneous temperature

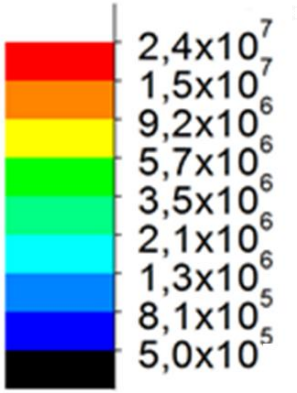
Photoelectrons effect?

Monte Carlo simulation using MCNP

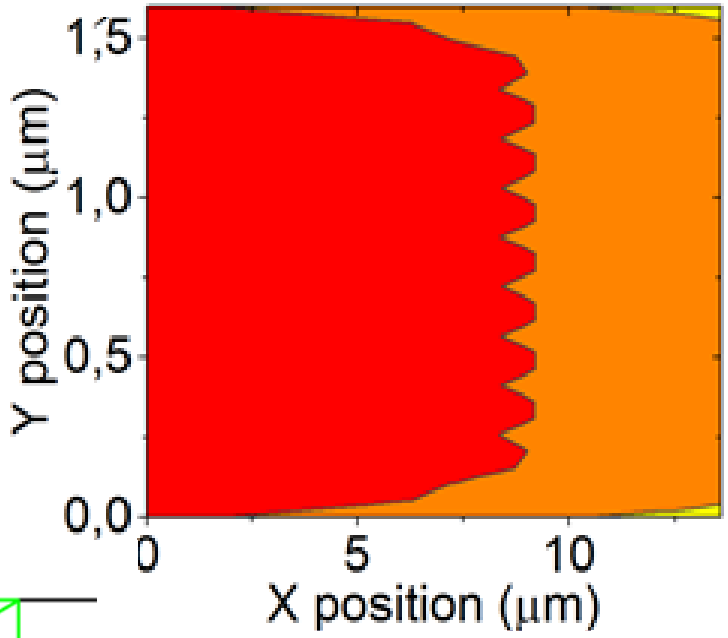
7-8 keV electrons

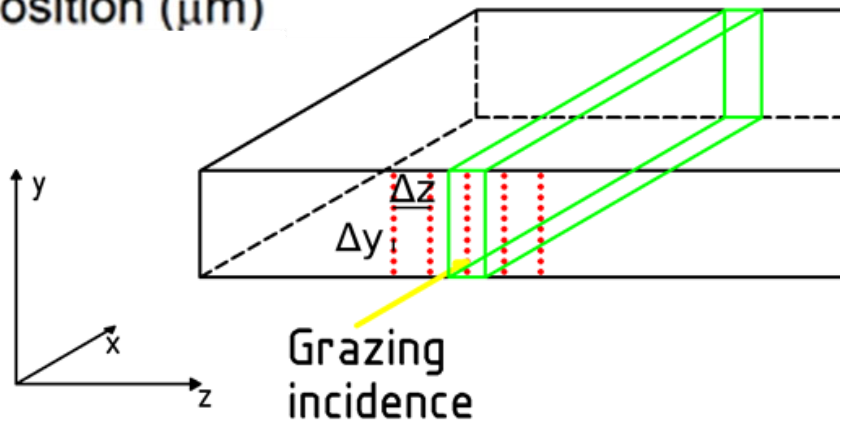


Φ_e (cm^{-2})



16-17 keV electrons





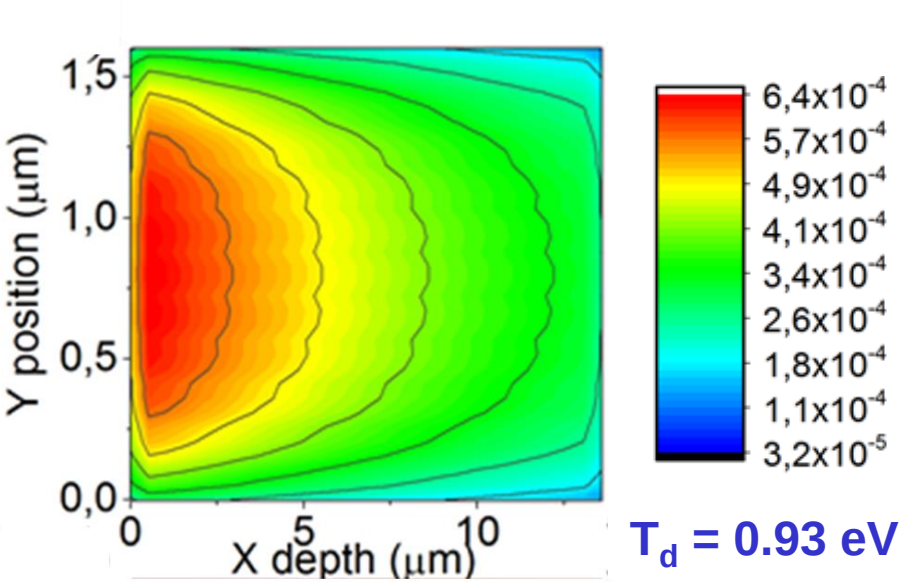
Number of displaced O atoms?

Photoelectrons effect?

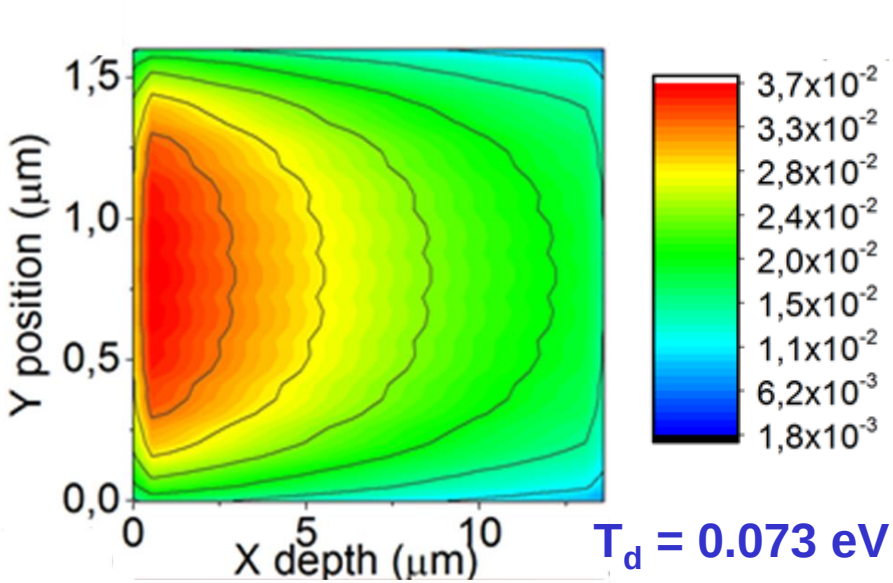
McKinley-Feshbach cross section for atom displacement by electrons:

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

threshold displacement energy



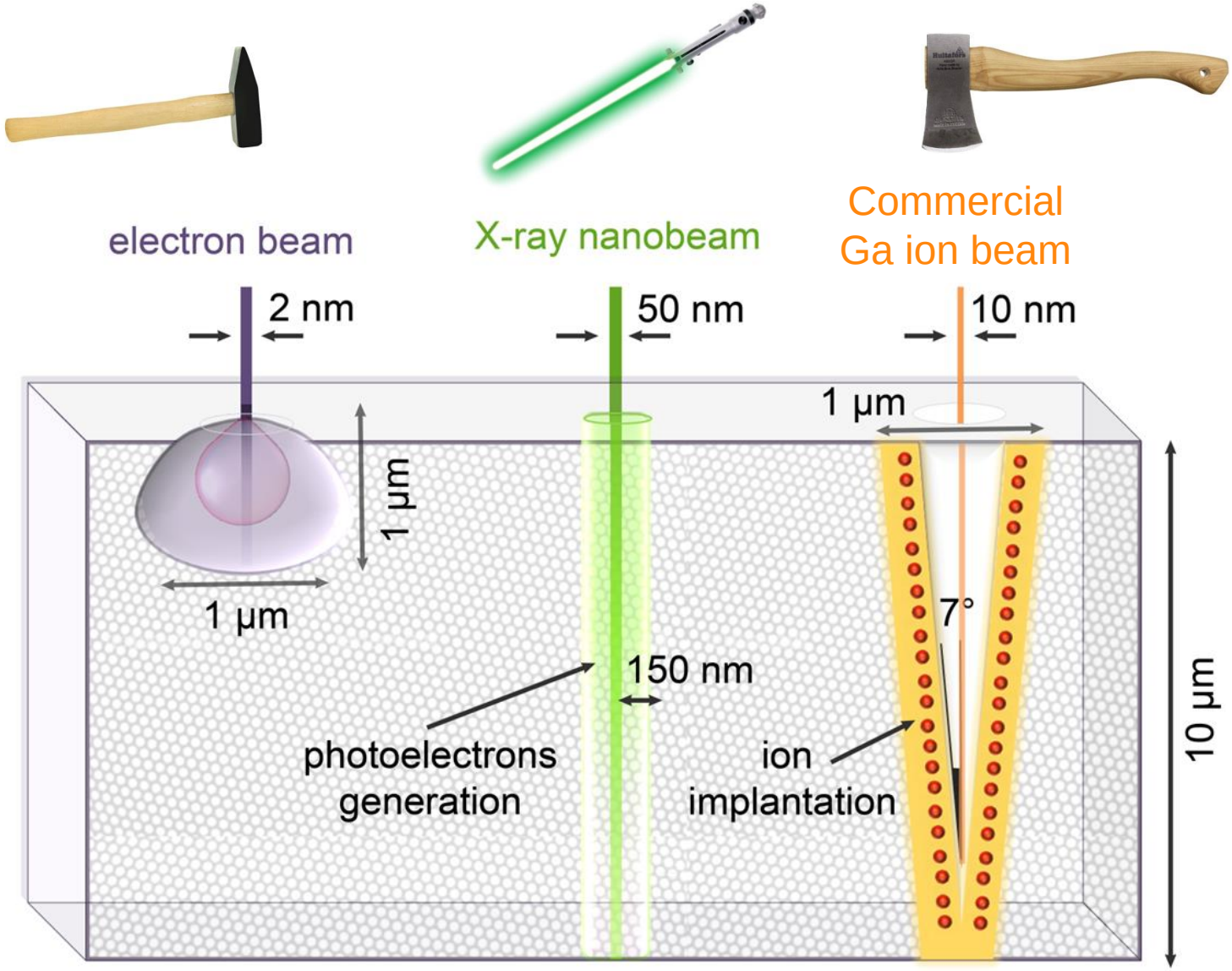
T_d = 0.93 eV



T_d = 0.073 eV

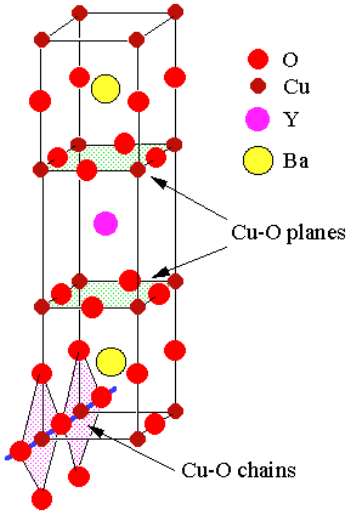
Fraction of displaced oxygens

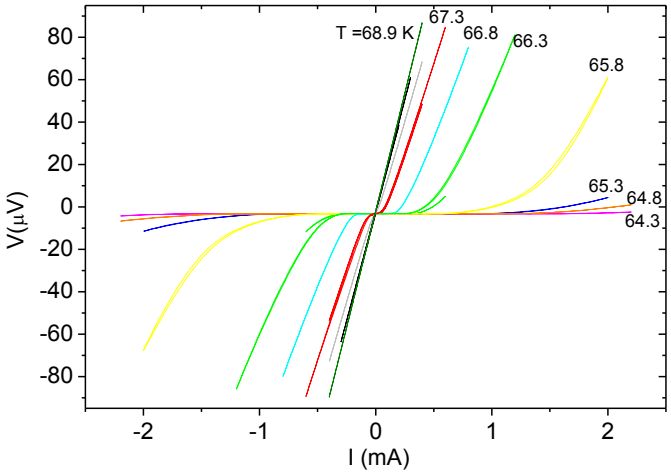
Patterning techniques comparison



Conclusions and perspectives

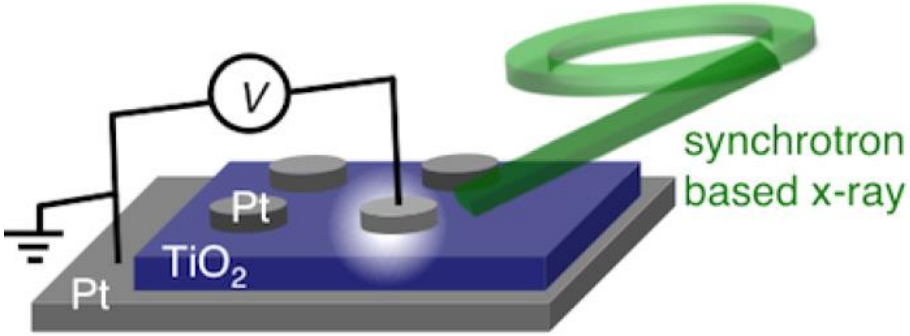
- X-ray nano-beams can be employed not only to characterize, but also to modify materials in a controlled way
- Local changes in Bi-2212 structure and resistivity can be monitored *in situ*
- IJJ devices have been produced by X-ray nanopatterning
- Preliminary investigation of the heat and photoelectrons role
- In principle, this idea could be extended to other oxides

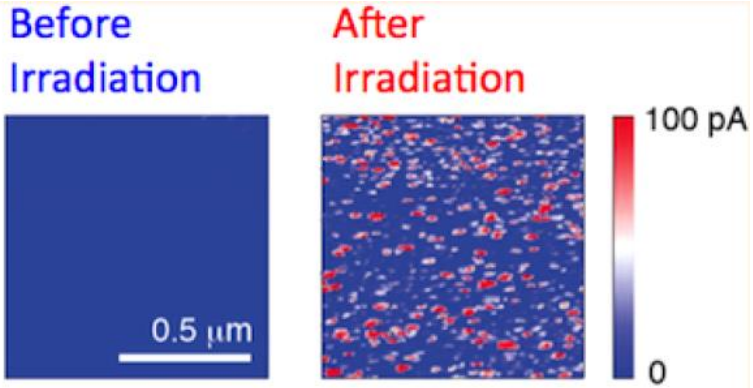


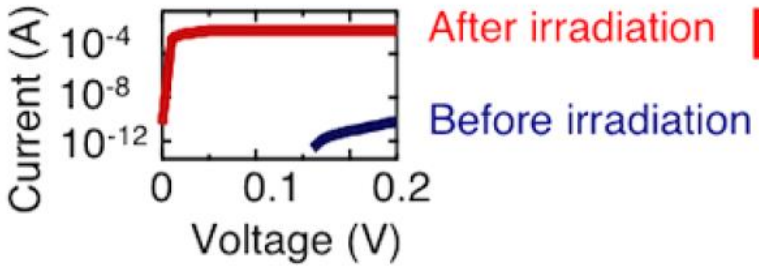


IJJ device in YBCO

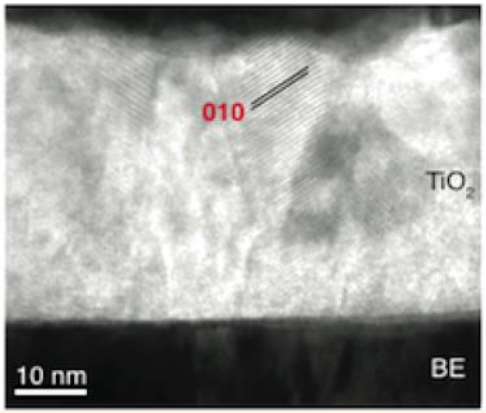
Conclusions and perspectives







Formation of Magnéli Phase Filaments



Preliminary indication:

local modification of TiO_2 properties

S. H. Chang *et al*., ACS Nano **8**, 1584, (2014)

Acknowledgments

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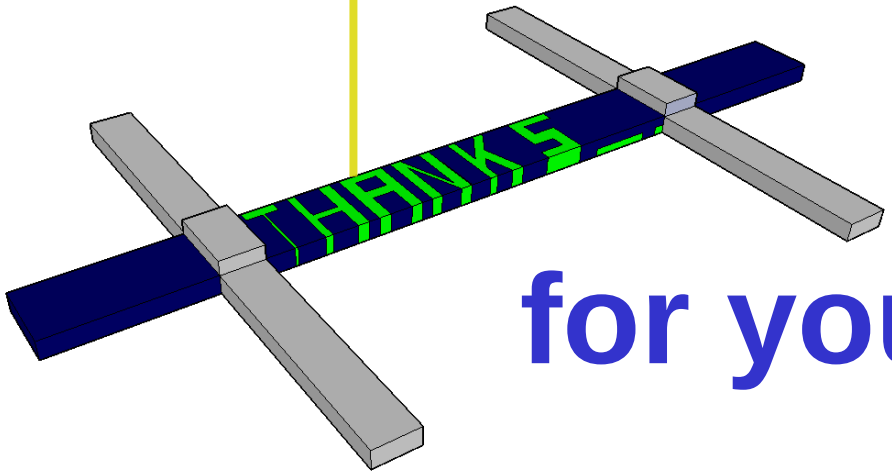

for your attention!