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Consiglio Nazionale delle Ricerche

Tl-1223 Superconducting Films for Beam Screen Coatings in FCC: Synthesis and Performance Validation

Emilio Bellingeri

FUTURE CIRCULAR COLLIDER

FCC Week 2025

OUTLINE

- *Project*

- *Why Thallium-based?*

- *Phase diagram study*
- *Precursor coating deposition and reaction*
- *Structural characterization*
- *DC and RF superconducting properties*
- *High vacuum compatibility*

- *Conclusions*

FCC beamscreen projects @SPIN

- First project: <u>Tl based HTS preparation</u>
  2016-2019
  PI Emilio Bellingeri
  Addendum FCC-GOV-CC-0049 (EDMS1580502, KE3112)

- New project: <u>New project for assessing Tl (1223) superconducting coating feasibility</u>
  2021- 2024
  PI Alessandro Leveratto
  Addendum FCC-GOV-CC-0217 (KE5072/TE)

Sample preparation, structural characterization, DC measurements, theory
Alessandro Leveratto, *Aisha Saba, Cristina Bernini, Federico Matteo, Cialone,* ***Ruggero Vaglio***

Accelerator environment compatibility, theory
Marcel Himmerlich, Bernard Henrist ,Stephanie Fernandez-Peña Mauro Taborelli, Guillaume Rosaz, ***Sergio Calatroni****, Massimo Giovannozzi*

RF measurements
Andrea Alimenti, Alessandro Magalotti, Nicola Pompeo, Kostyantin Torokhtii, Pablo Vidal García, Enrico Silva

Structural and Transport Characterization: TEM, SEM, EBSD, Jc (T, B), SHPM,…
Sigrid Holleis, Florian Semper Michael Eisterer
Andreas Steiger-Thirsfeld , Johannes Bernardi

In FCC-hh Beam screen is foreseen to work at T>50K. The surface resistance of copper at 50 k may not be sufficiently low to guarantee a safe operational margin for the fcc-beams (in particular at injection energy).
Idea: superconducting coating!

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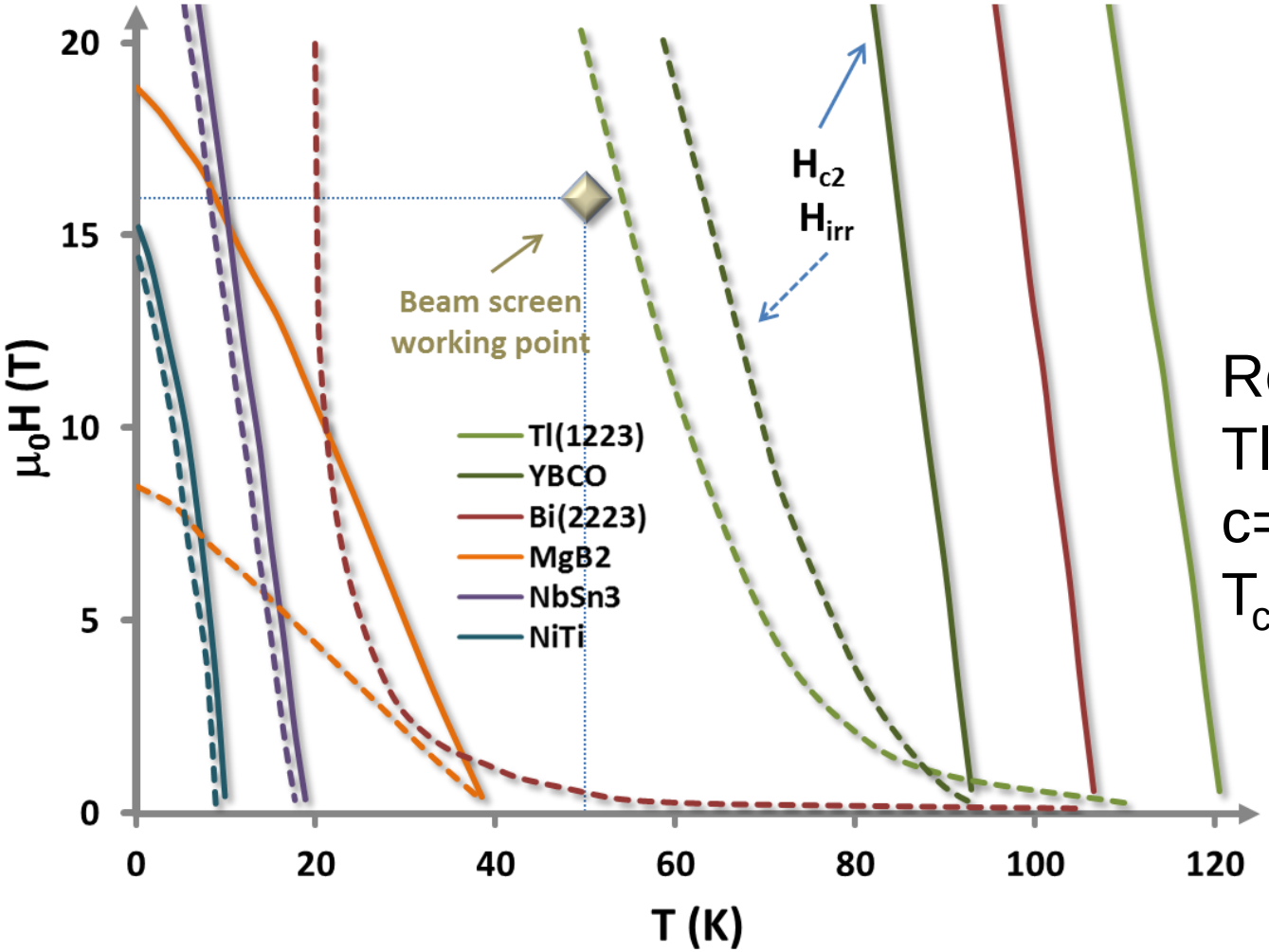
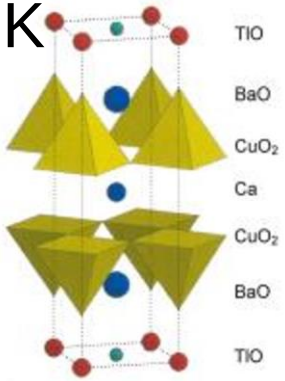
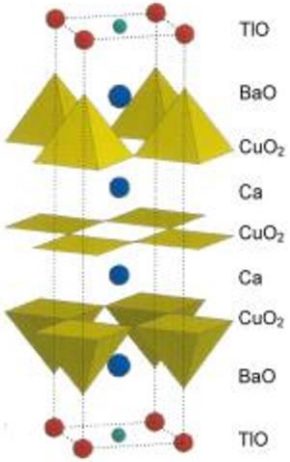


Work condition:
B=14 T (or 16 T)
T=50K (or 100 K)
Jc > 10^4 Acm^{-2}
$\nu \approx 1GHz$

ReBCO (Cu1212)
Tl1212
c=12.7Å
T_c=80 108K



Tl1223
c= 15.2Å
T_c=120K



Why Thallium-1223 ?

	ReBCO	Tl1223
PROs	• Safe • Very high Jc • Very high steep Hirr • Technologically advanced	• Very high Tc • High Jc • High Hirr • Tc and Hirr very tolerant for out stoichiometry
CONs	• Strong weak link problems • Low Tc • Very expensive and complex preparation on large scale (IBAD,..) • VERY SENSITIVE TO STOICHIMETRY	• Toxic • Tl is volatile • Few studies

- Already demonstrated the possibility to deposit via electrochemistry thanks to the high out-of-stoichiometry tolerance with respect to RE-123
- Cheap and scalable technique, which could be considered for a direct deposition on tubes or arbitrarily shaped templates
- Tc ~ 120 K

- Thallium evaporation above 700 °C
- really unstable reaction
- Tl-1212 phase thermodinamically more stable

E. Bellingeri et al. IEEE 2001

3122 IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 11, NO. 1, MARCH 2001

Electrodeposition of biaxially aligned Tl-based superconductors on Ag tapes

E. Bellingeri, HL. Suo, J.-Y. Genoud, M. Schindl, E. Walker and R. Flükiger

G. Gritzner et al. SUST 2004

INSTITUTE O... Supercond. ... ONDUCTOR SCIENCE AND TECHNOLOGY PII: S0953-2048(04)80225-2

Preparation and characterization of (Tl, Pb)(Ba, Sr)-1223 films on untextured silver

Christian Deinhofer and Gerhard Gritzner[1]

Institute for Chemical Technology of Inorganic Materials, Johannes Kepler University, A 4040 Linz, Austria

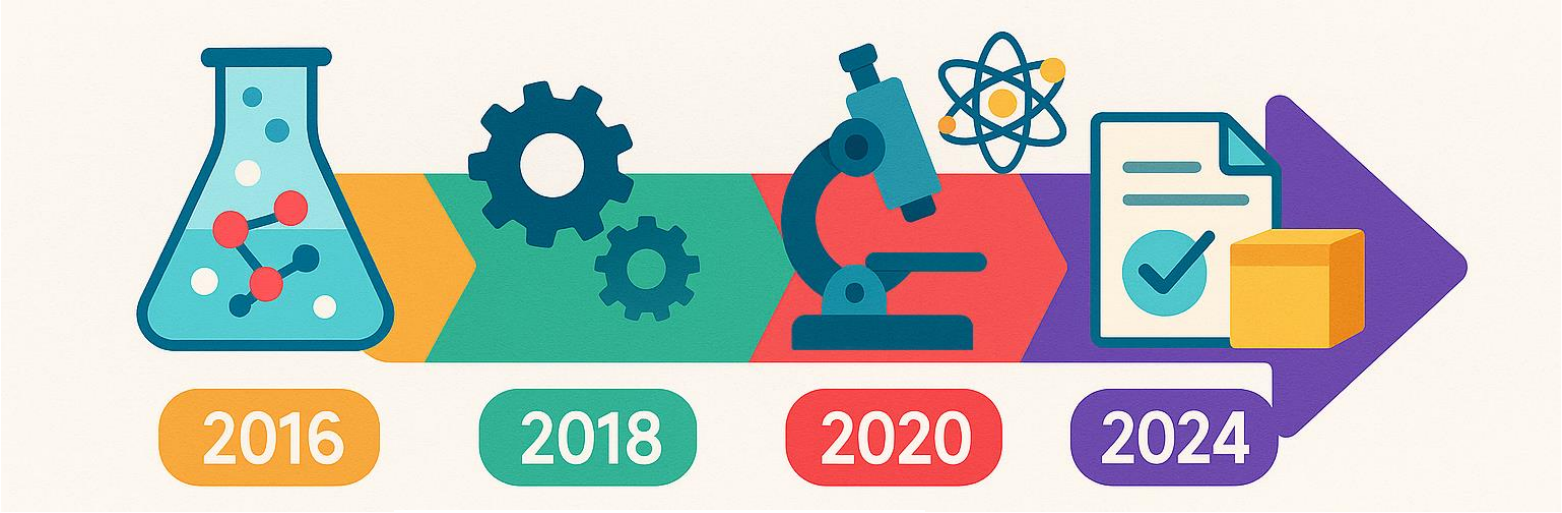
D.Y. Jeong et al SUST 2007

IOP PUBLISHING SUPERCONDUCTOR SCIENCE AND TECHNOLOGY

Supercond. Sci. Technol. **20** (2007) 1239–1252 doi:10.1088/0953-2048/20/12/026

A new process to prepare thick bufferless Tl-1223/Ag high temperature superconducting coated conductors using electrodeposition

D Y Jeong[1,3], Y H Kim[1], P M Shirage[1], S Y Kim[1], S Horiuchi[1] and J H Lee[2]



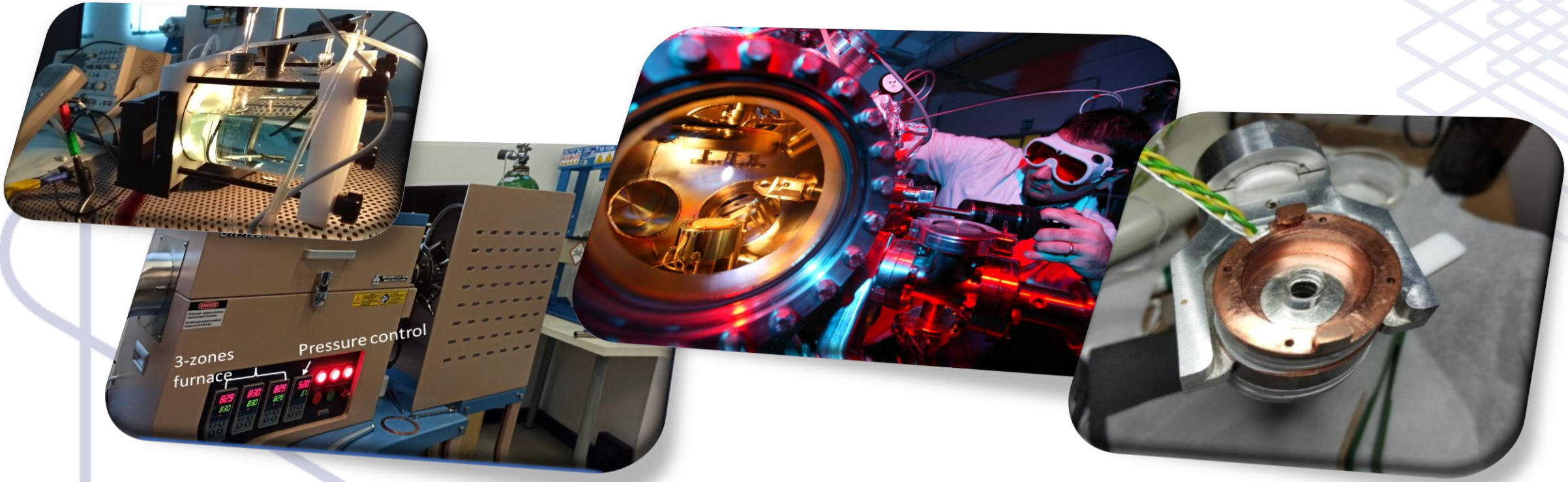
TOXIC LAB CONSTRUCTION

PRECURSOR DEPOSITION

CHARACTERIZATION

POWDER AND BULKS REALIZATION

HIGH PRESSURE REACTION

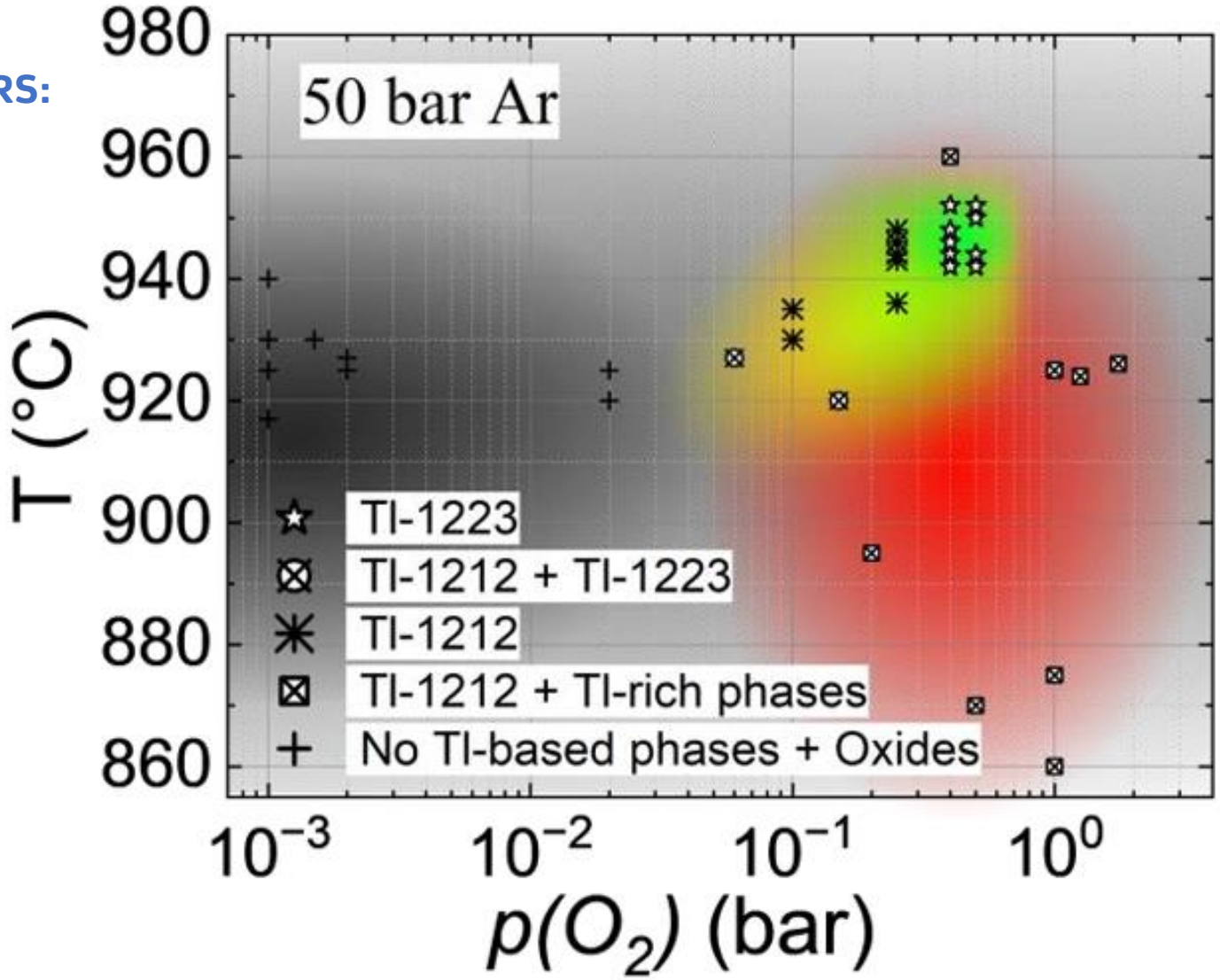


Phase diagram study

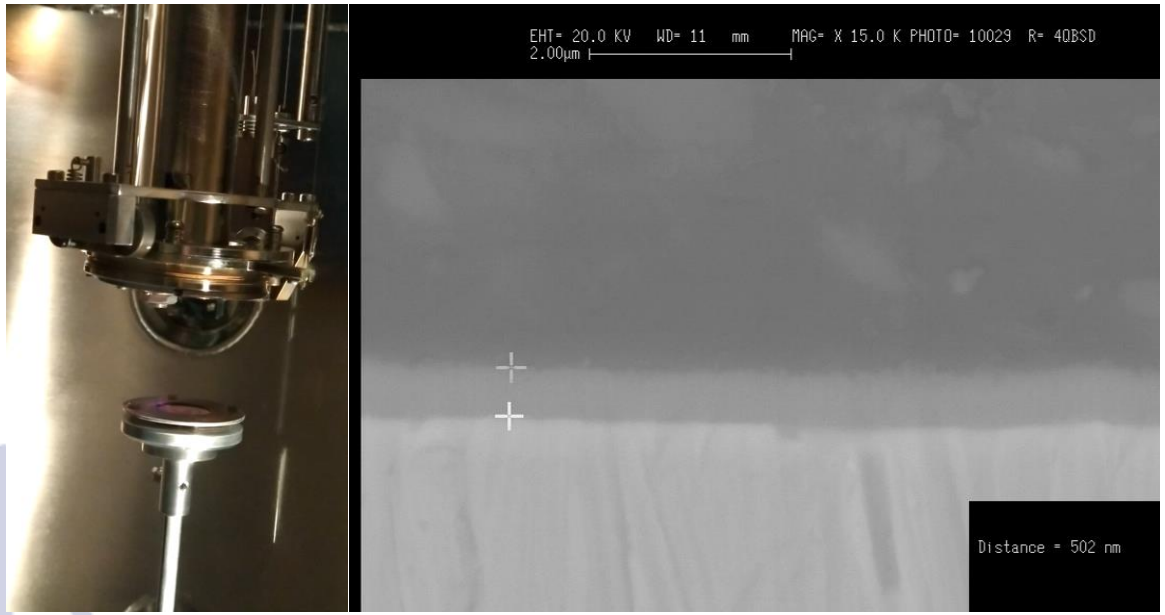
High Ar pressure syntesis in order to prevent Tl evaporation

Tl-based cuprated
PHASE DIAGRAM STUDY
ON VARYING MANY PARAMETERS:

- TEMPERATURE
- INITIAL PRESSURE
- FINAL PRESSURE
- O_2 PARTIAL PRESSURE

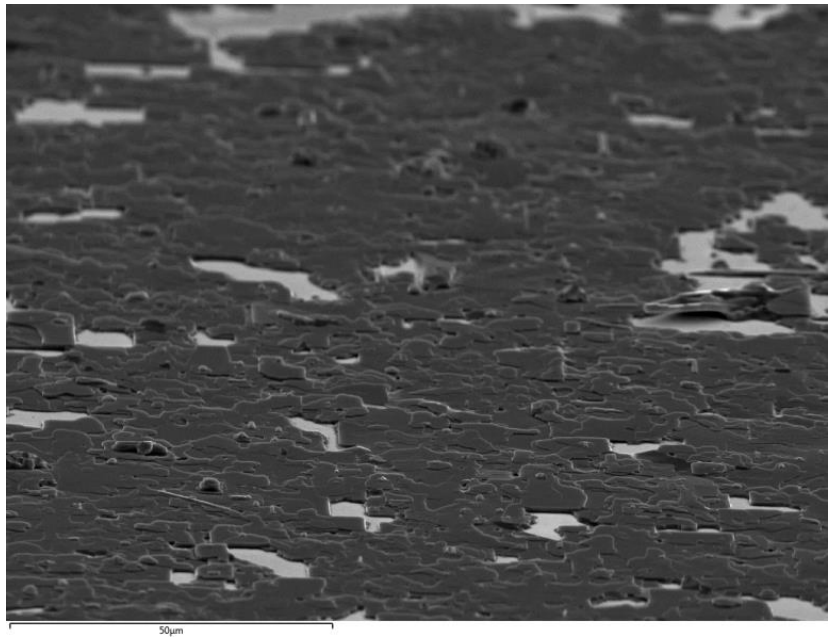


Precursos layer



Nd:YAG 1064 nm
5 Hz 90 min
Substrate LAO 001
$T_{substrate}$ = RT
p(O2) = 8.7-9.3 10^{-2} mbar

Reacted film



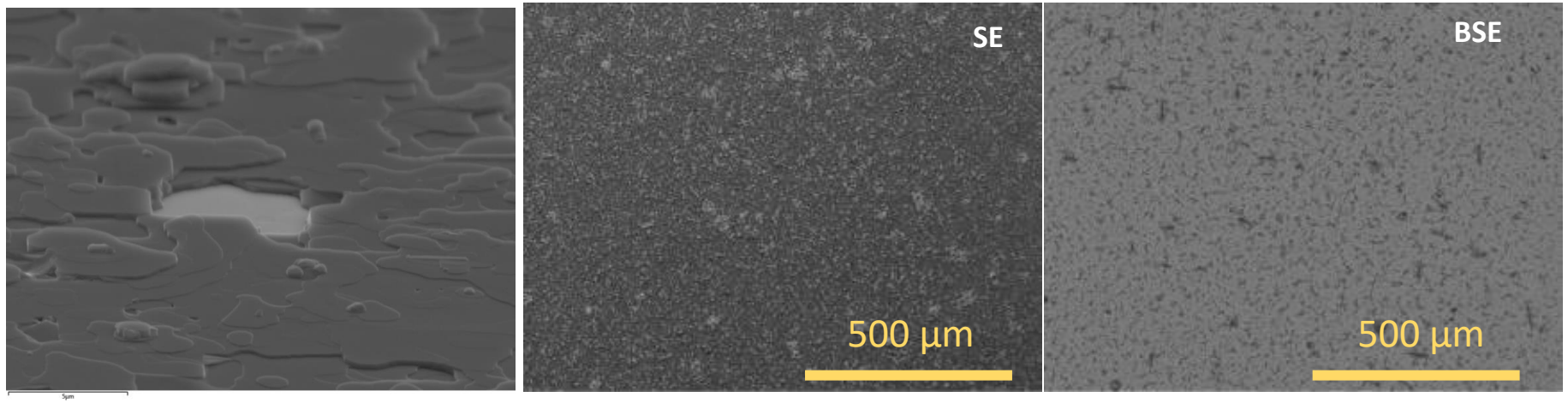
HT-HP REACTION

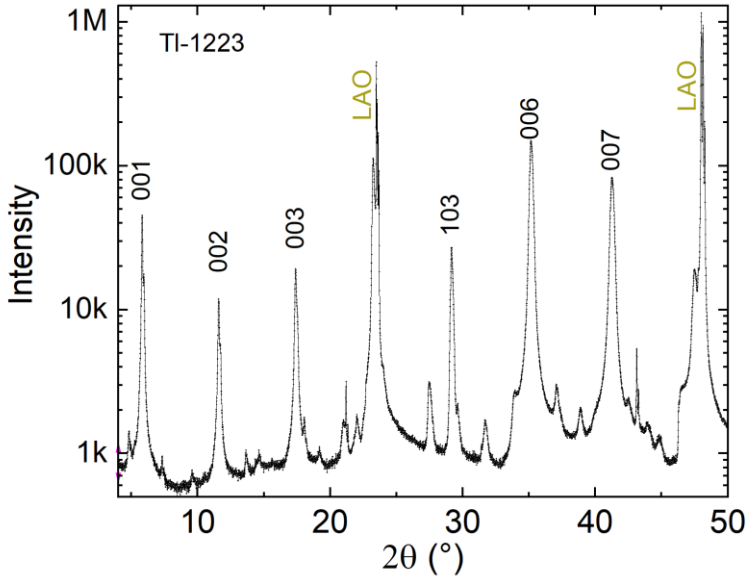
950 °C

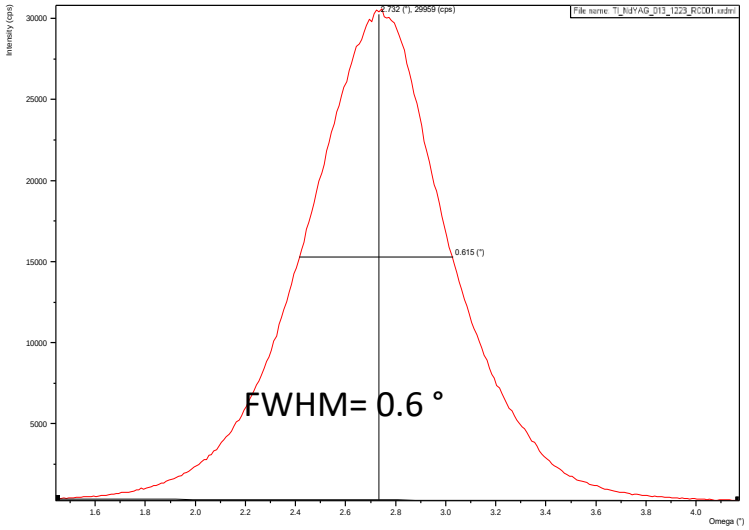
50 bar Ar+ p(O2)

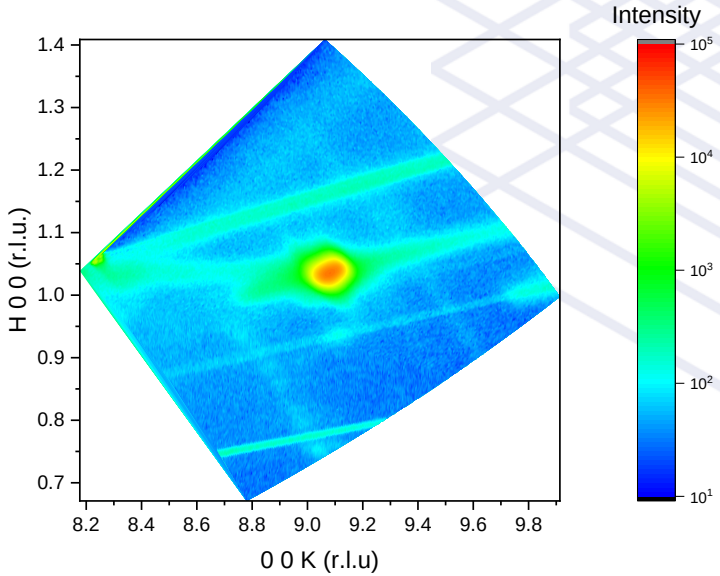
3 h

Structural properties



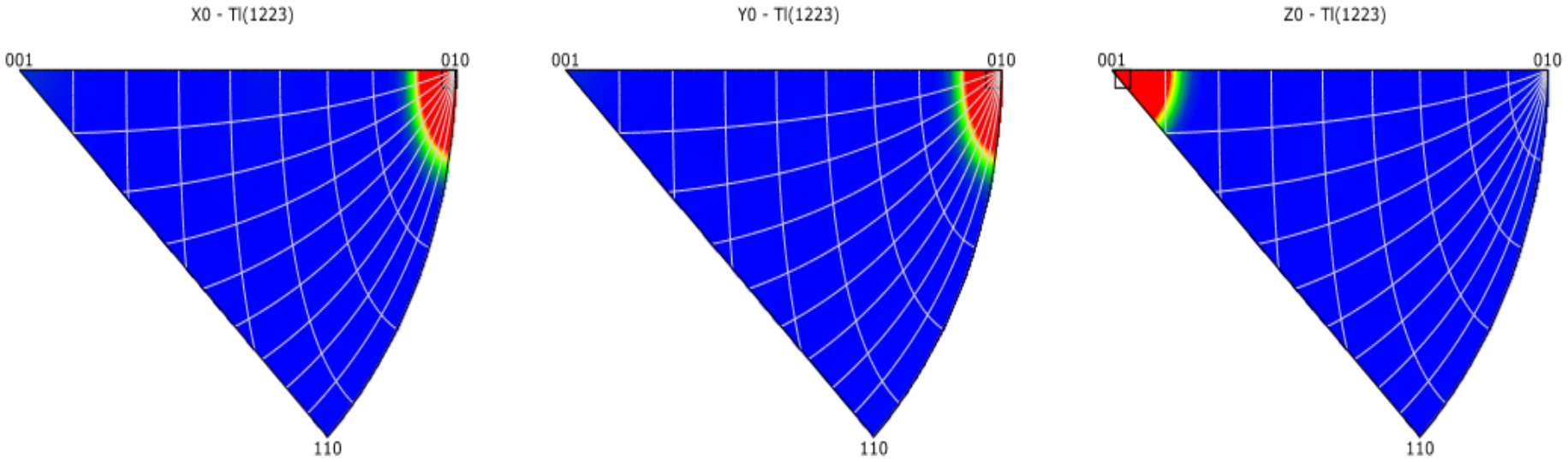


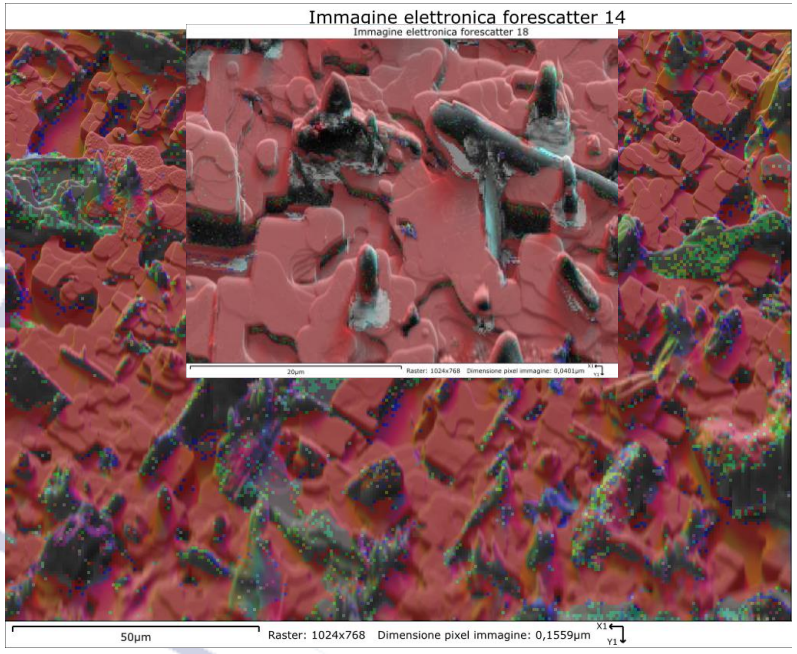


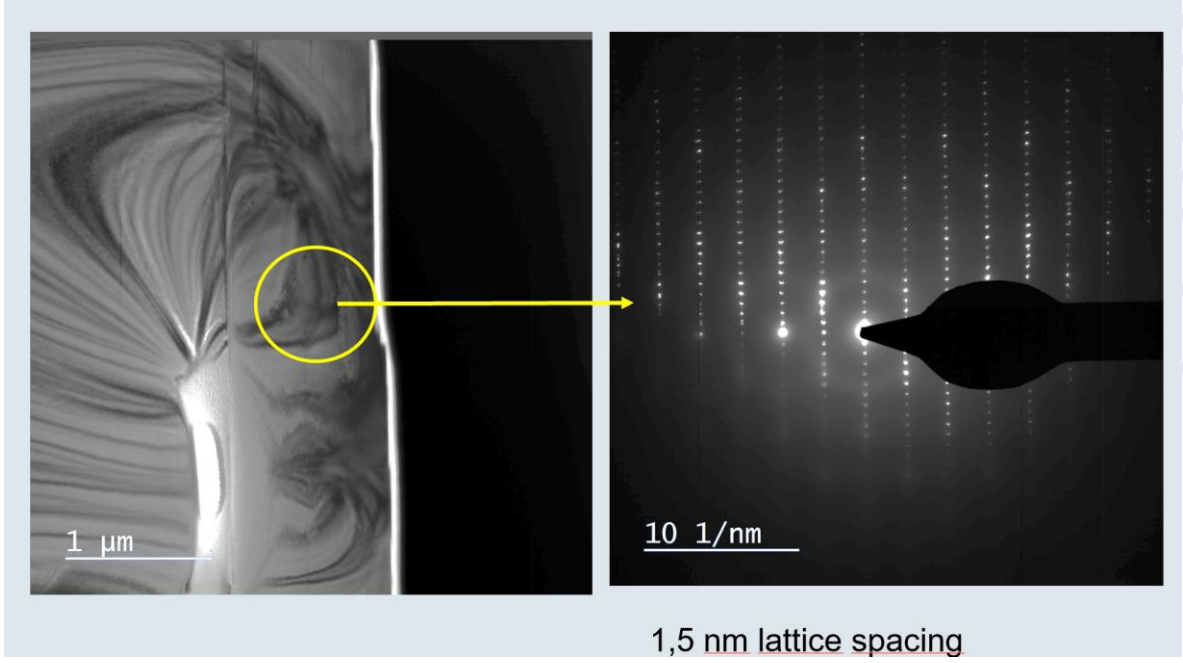


Very pure Tl(1223) phase, policristalline but fully (c and ab) oriented

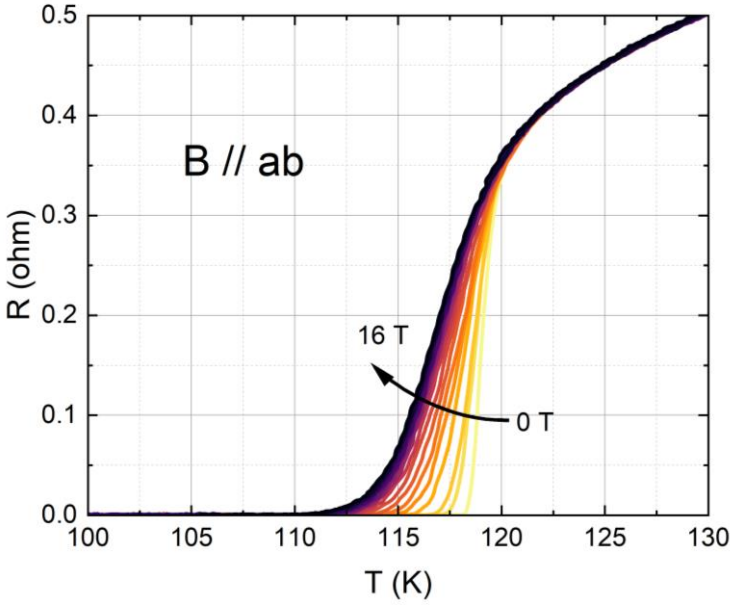
❖ Microscopy–SEM EBSD TEM

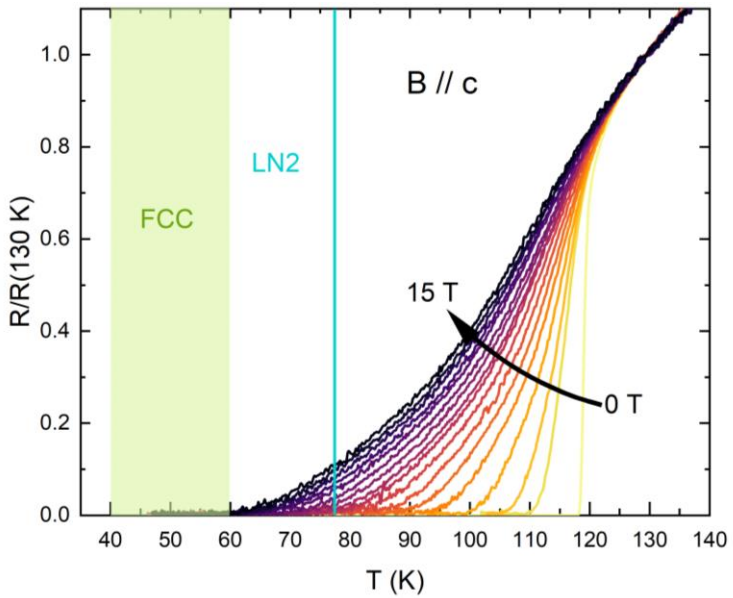


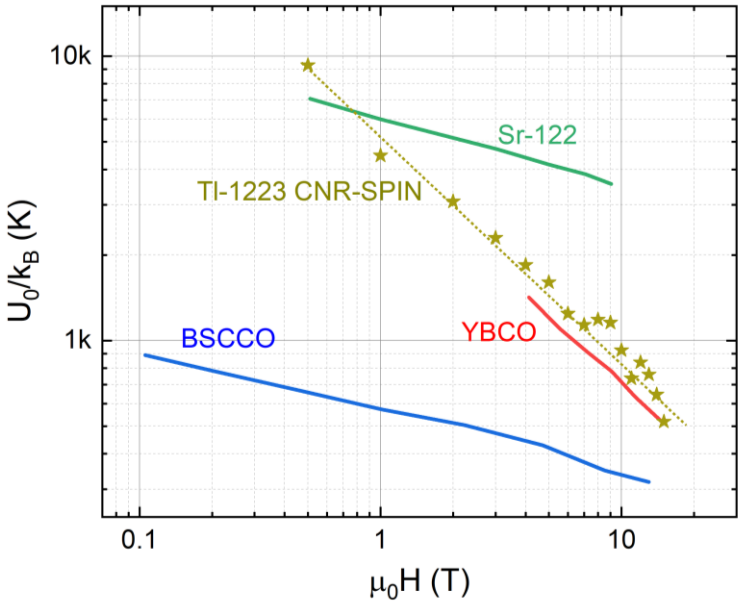


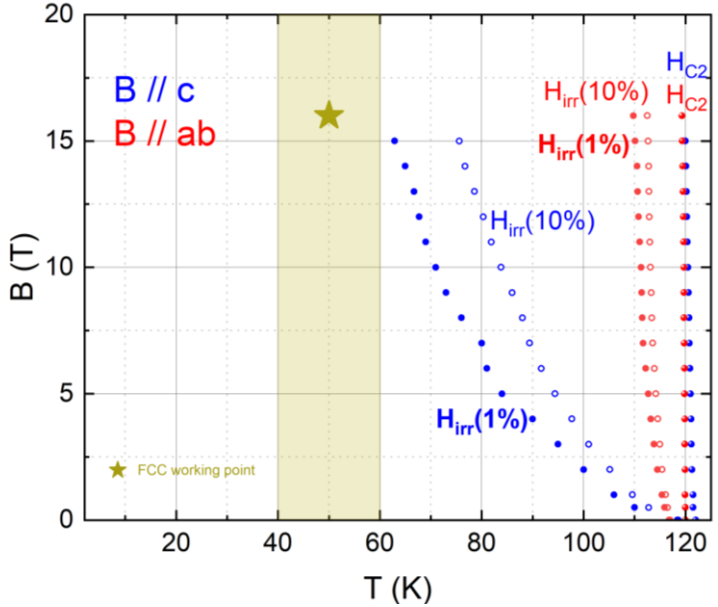


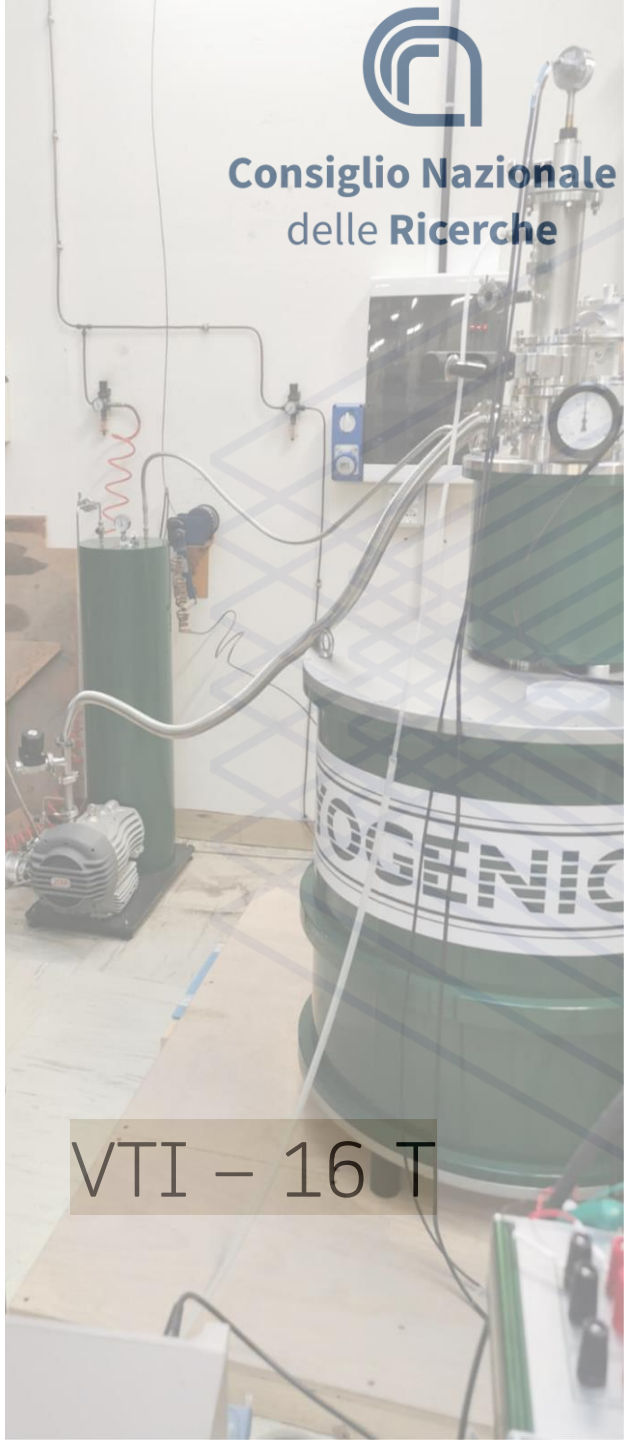
R(T. B) > H_{C2}, H_{irr}


B // ab
16 T
0 T
R (ohm)
T (K)


B // c
FCC
LN2
15 T
0 T
R/R(130 K)
T (K)


Sr-122
Tl-1223 CNR-SPIN
BSCCO
YBCO
U_0/k_B (K)
$\mu_0 H$ (T)


B // c
B // ab
H_{irr}(10%)
H_{C2}
H_{irr}(1%)
FCC working point
B (T)
T (K)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delle Ricerche
VTI – 16 T

Critical current density J_c

Transport current measured 4 wire on patterned samples to reduce Ic

Nominal channel geometry

Length l_{V+V-} = 300 µm

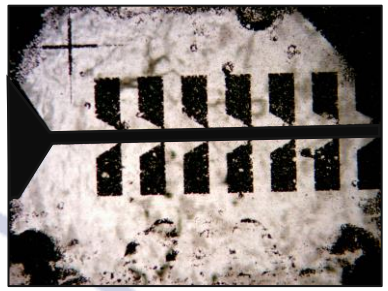
Width d = 100 µm

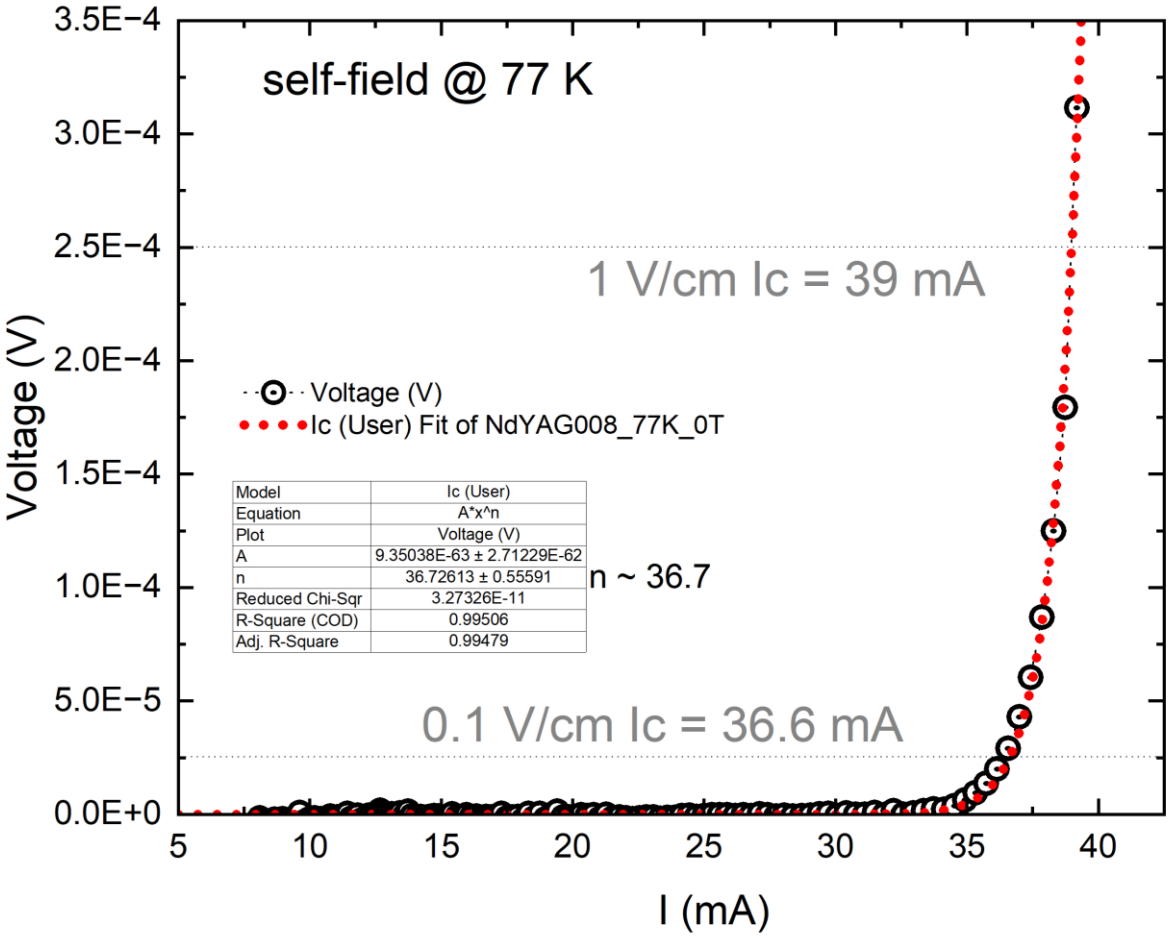
Thikness z = 0.5 µm

s = 50 10^{-8} cm^2

J_c = 2 10^5 A/cm^2

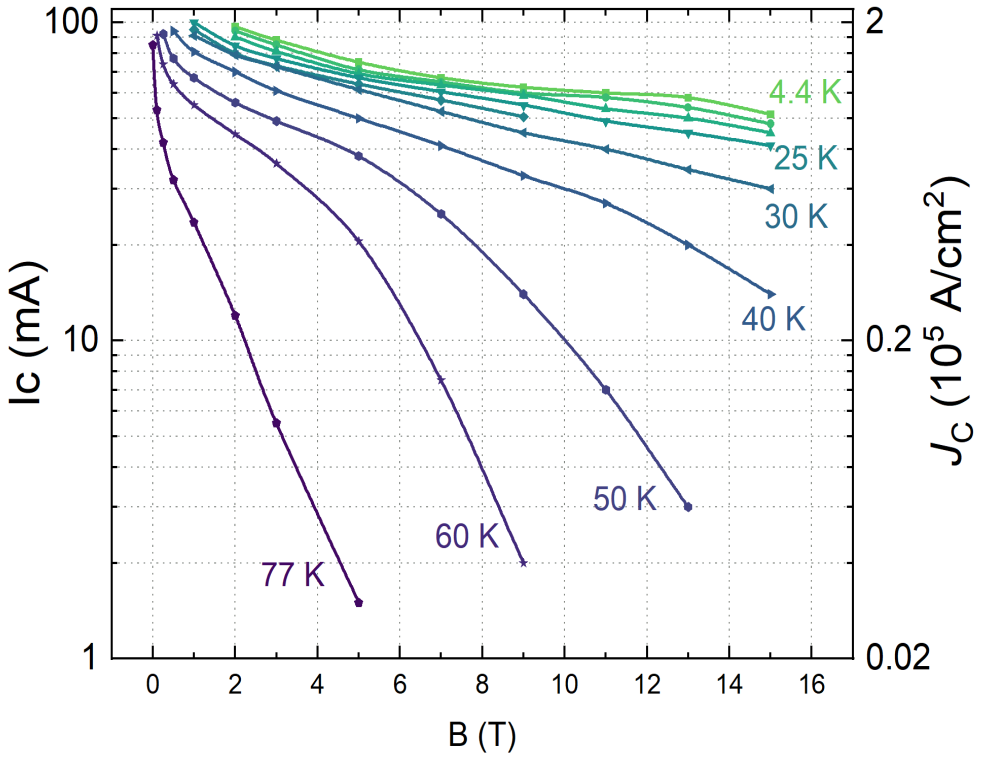
H_3PO_4 8.5%

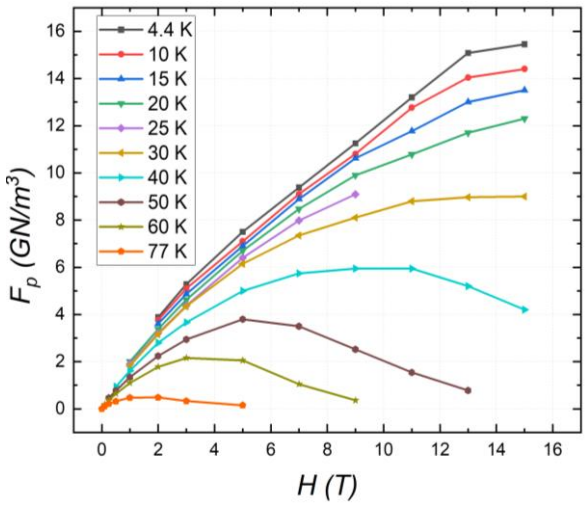


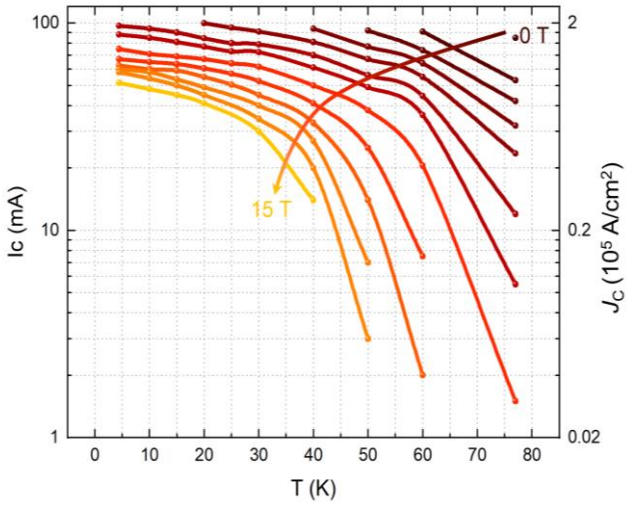


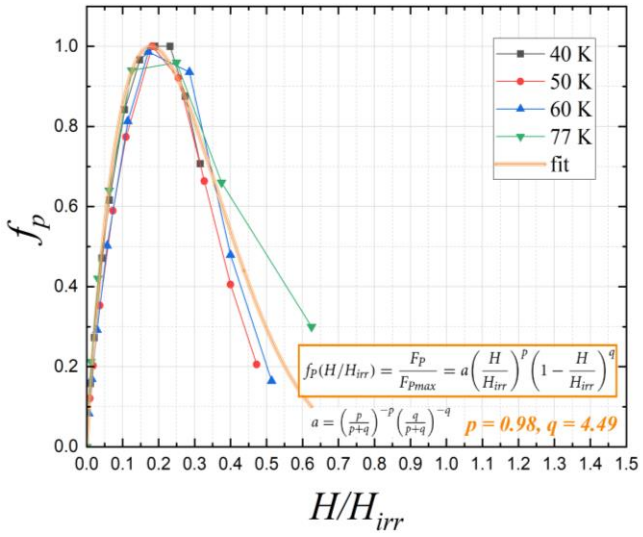
Model	Ic (User)
Equation	A*x^n
Plot	Voltage (V)
A	9.35038E-63 ± 2.71229E-62
n	36.72613 ± 0.55591
Reduced Chi-Sqr	3.27326E-11
R-Square (COD)	0.99506
Adj. R-Square	0.99479

Critical current density J_c





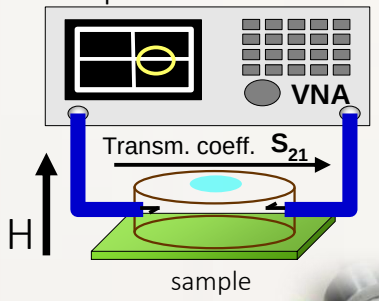


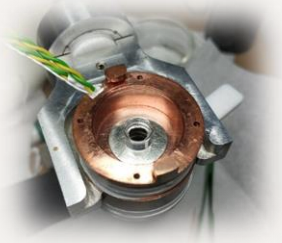


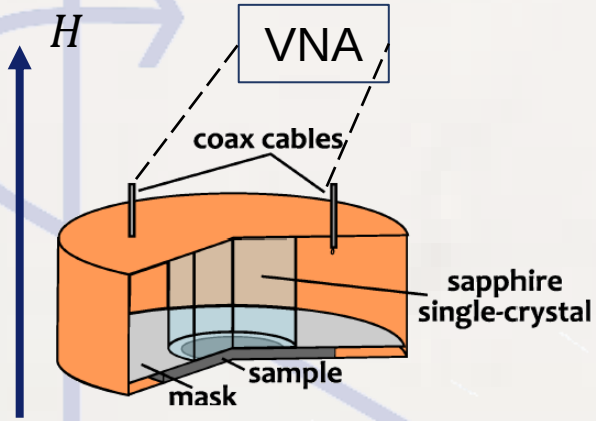
Microwave characterization

Measurement technique

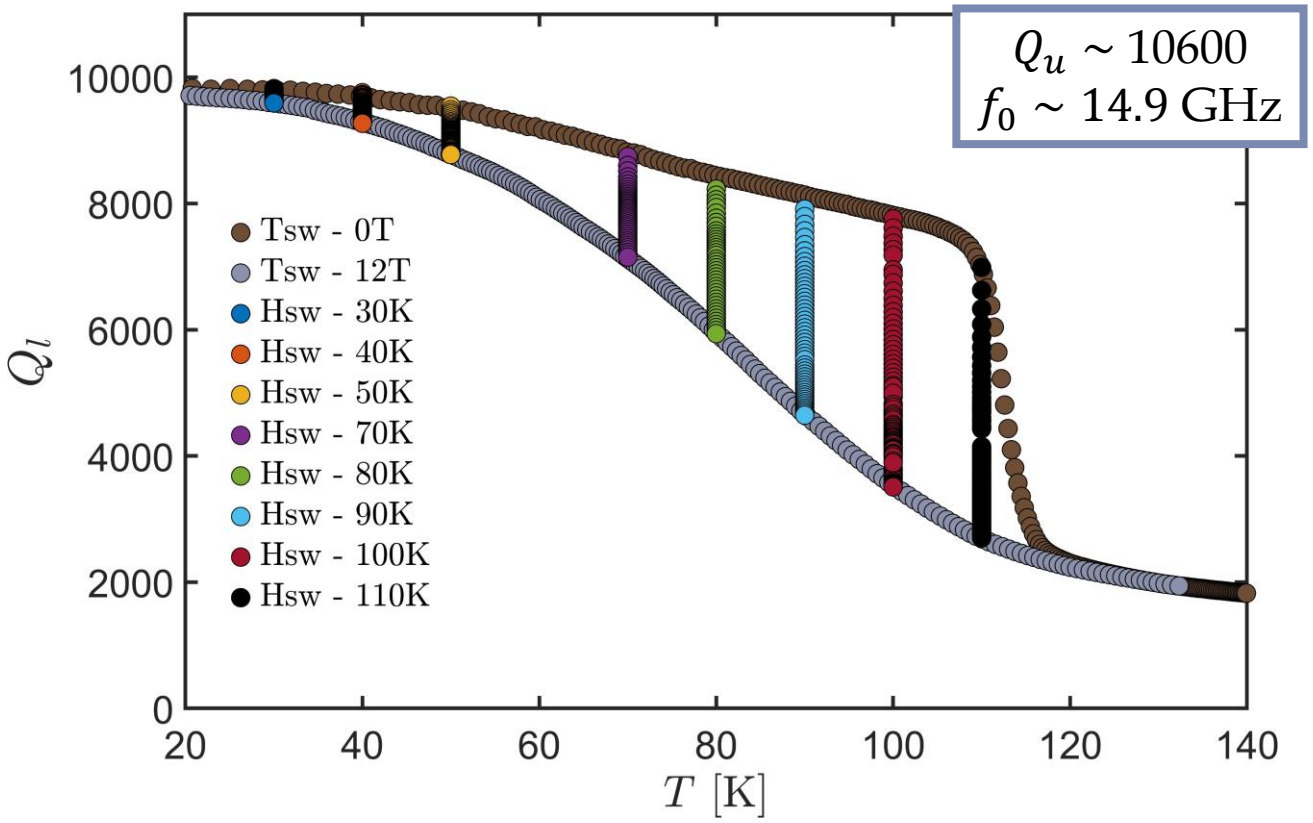
Dielectric-loaded resonator
Surface perturbation method



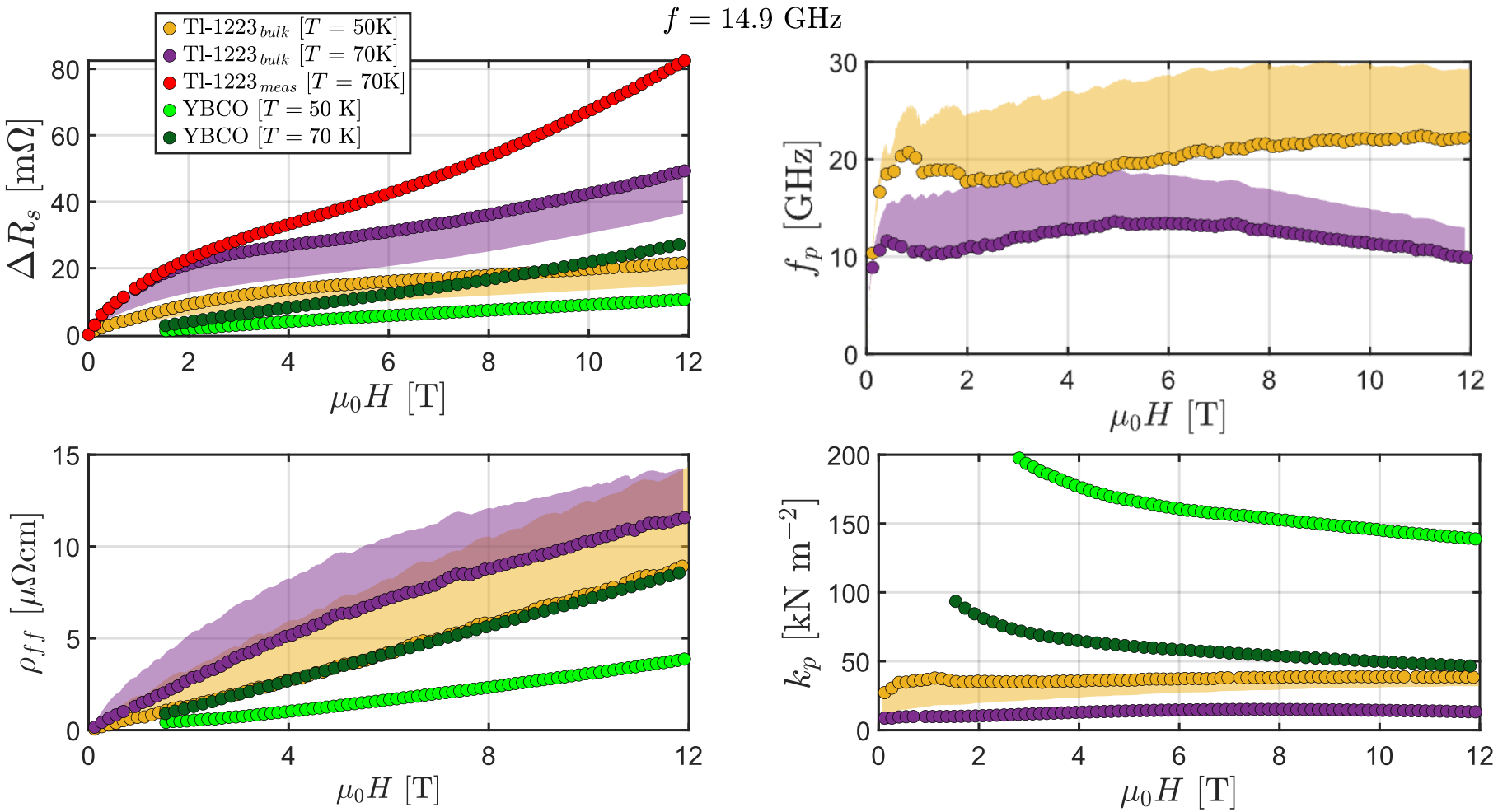




- Sample thickness ≈ 1 μm
- T_c = 116 K (Tl-1223), no signs of a second phase
- "Higher" Q-factor compared to the previous sample



Microwave characterization



Bands refer to different assumed values for $\lambda_L(0)$ = [150,600] nm

Wrt YBCO SuperPower CC:

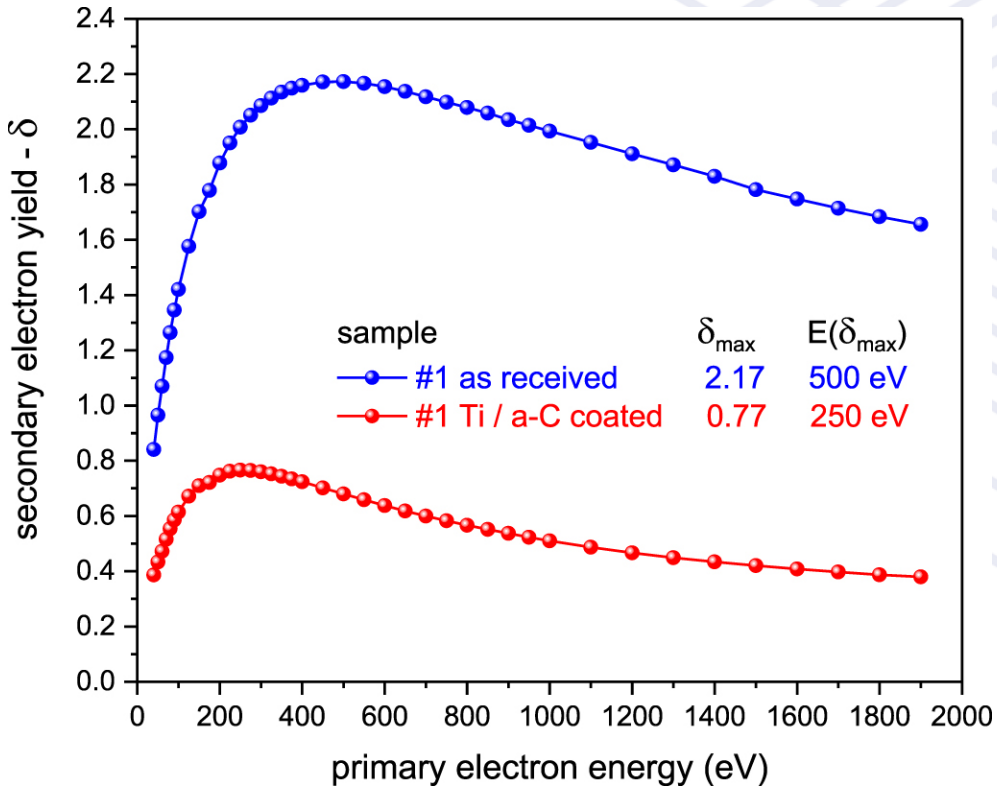
- $R_{s,bulk}$ (computed assuming a thickness of ~3 μm) within a factor of 1.5-2
- k_p is the dominant factor in R_s difference @ 70 K
- Promising results:
  - ✓ k_p constant in the T-B range reported (crucial for high B applications)
  - ✓ Ample margins for k_p optimization

Requirements inherent of the particle accelerator environment.

Vacuum: Pumping test of degassing RGA from surface after 24 h pumping are The RGA after 24 h of pumping was readily within CERN's acceptance criteria for unbaked components. In addition, XPS analysis revealed that no high atomic mass elements (Tl, Pb, Bi) are released by the pellet.

Secondary Electron Yield (SEY): the bare Tl-1223 surface has a rather high initial δmax at room temperature. The application of a thin amorphous carbon coating

(150 nm thick Ti buffer layer followed by addition of a 100 nm thick a-C top layer). dropped the SEY to 0.77, very promising result for further development of this approach.



Conclusion

A reliable method based on amorphous precursor deposition followed by a high temperature/high pressure thermic treatment to produce almost pure Tl(1223) superconducting coating has been demonstreated

Electrochemical deposition of precursors is possible but give less dense layers:To be optimized

Tl-1223 coatings approach the performances required, in terms of critical current and irreversibility field, by FCC application.

Results are on LaAlO3 single crystal:
a suitable substrate need to be optimized
(Reaction temperature is too high for pure silver)

Tl(1223) sample exhibits a vortex flux-flow resistivity (ρ_ff) that is nearly comparable to ReBCO tapes. The observed lower surface resistance is due to a lower pinning constant (k_p), (that in ReBCO tapes has been artificially increased through the introduction of controlled defects)

Pinning force shoul be increased
Thikness increased up to few micometers

Thank you for your attention

Istituto SPIN

Genova, L'Aquila, Napoli, Roma, Salerno

Film Deposition Of Thallium Based Precursors

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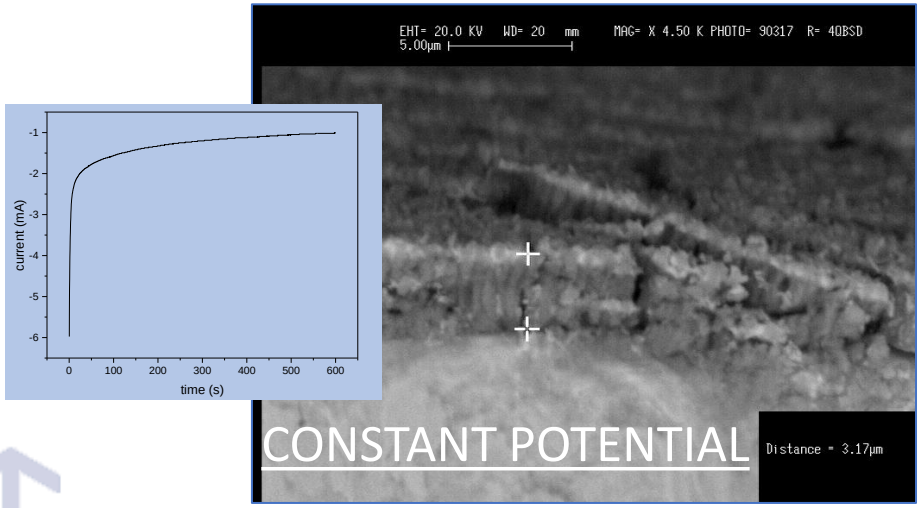
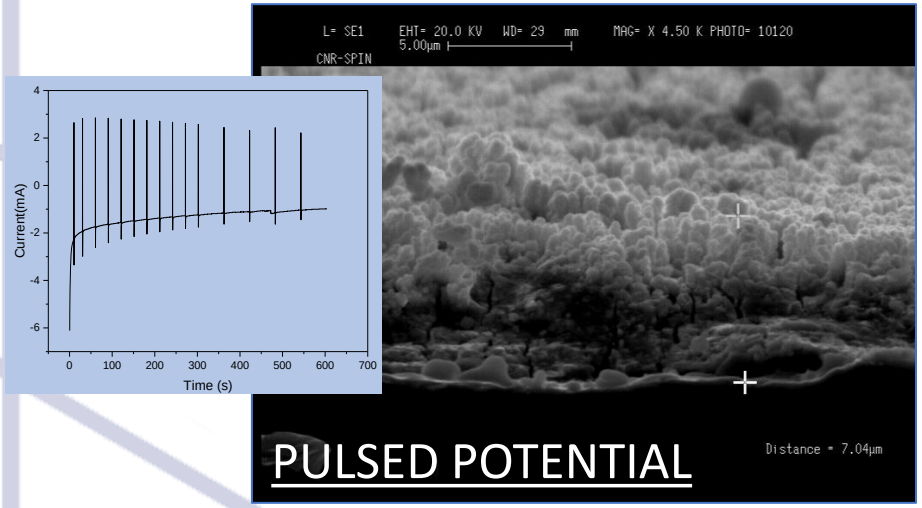
Electrochemical deposition



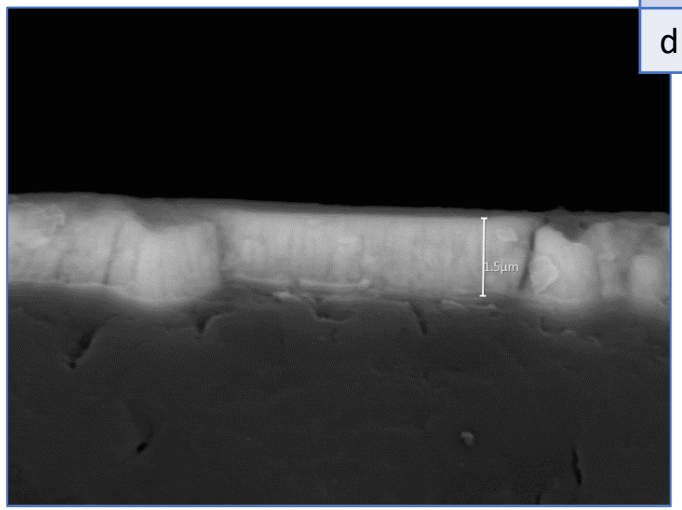


SUBSTRATES EMPLOYED

LAO (001)

STO (001)

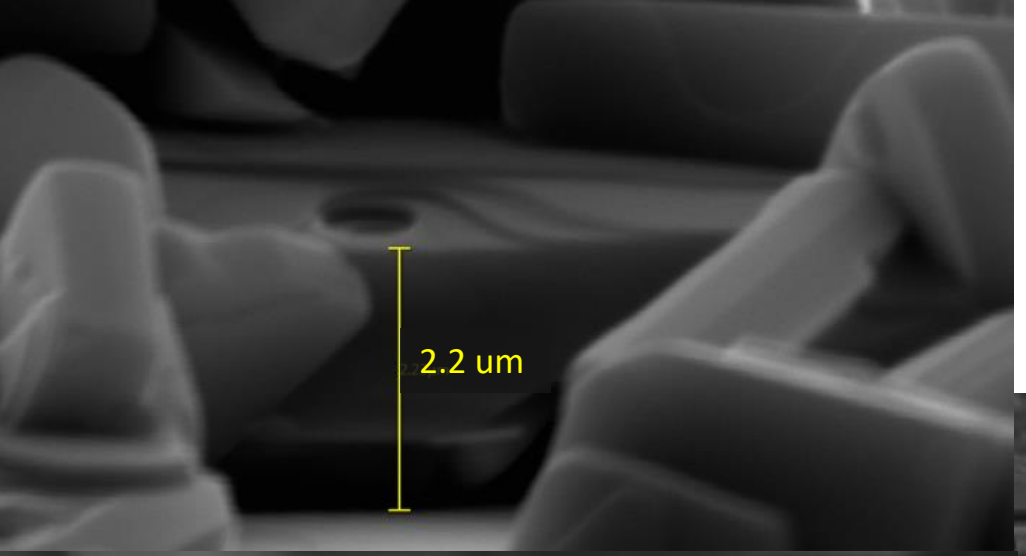
Pulsed Laser Deposition

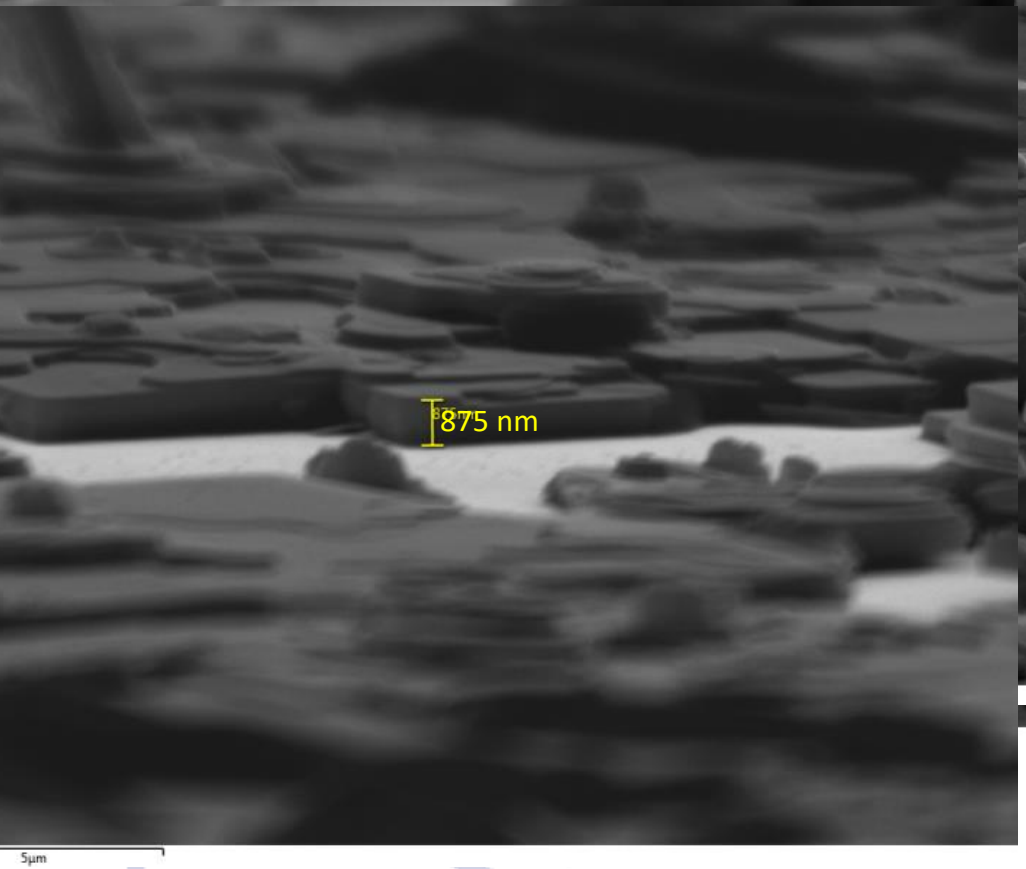


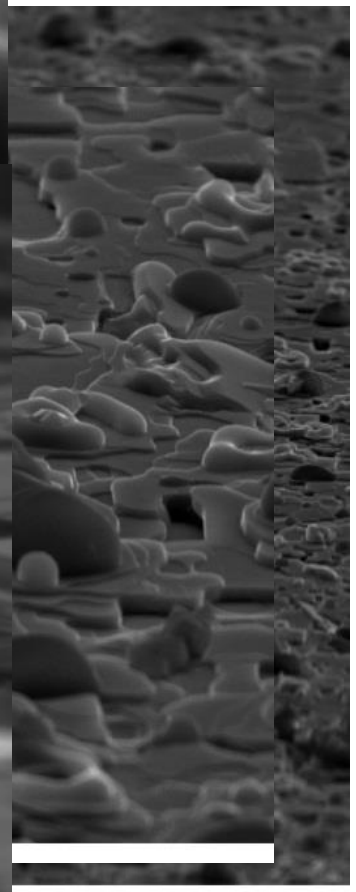
Frequency	5 Hz
Laser energy	1-3 J/cm^2
Vacuum	8 × 10^{-8} mbar
O2 pressure	10^{-2} – 10^{-1}mbar
d target - substrate	3-4 cm

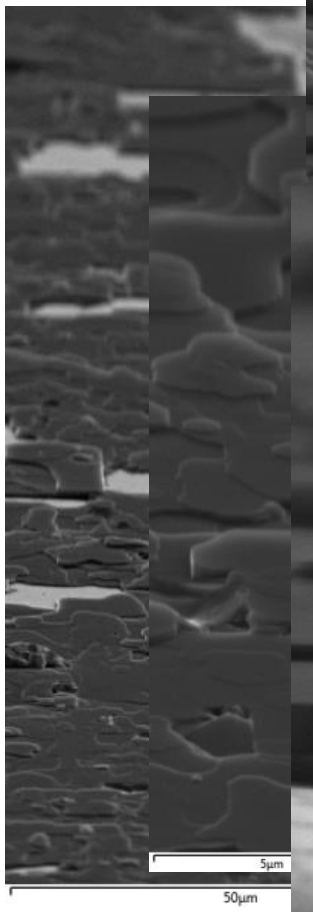
Thicker
(2 h dep.)

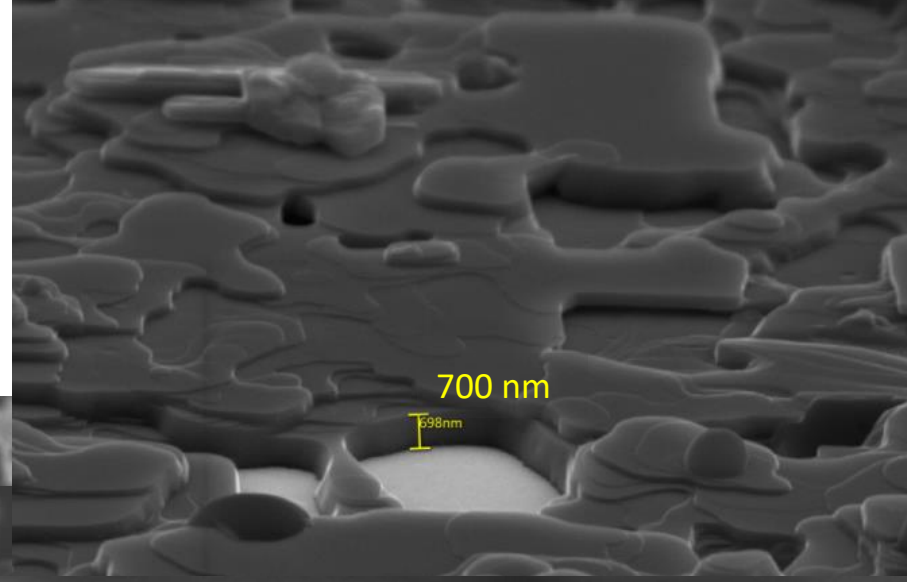
Thinner
(1 h dep.)

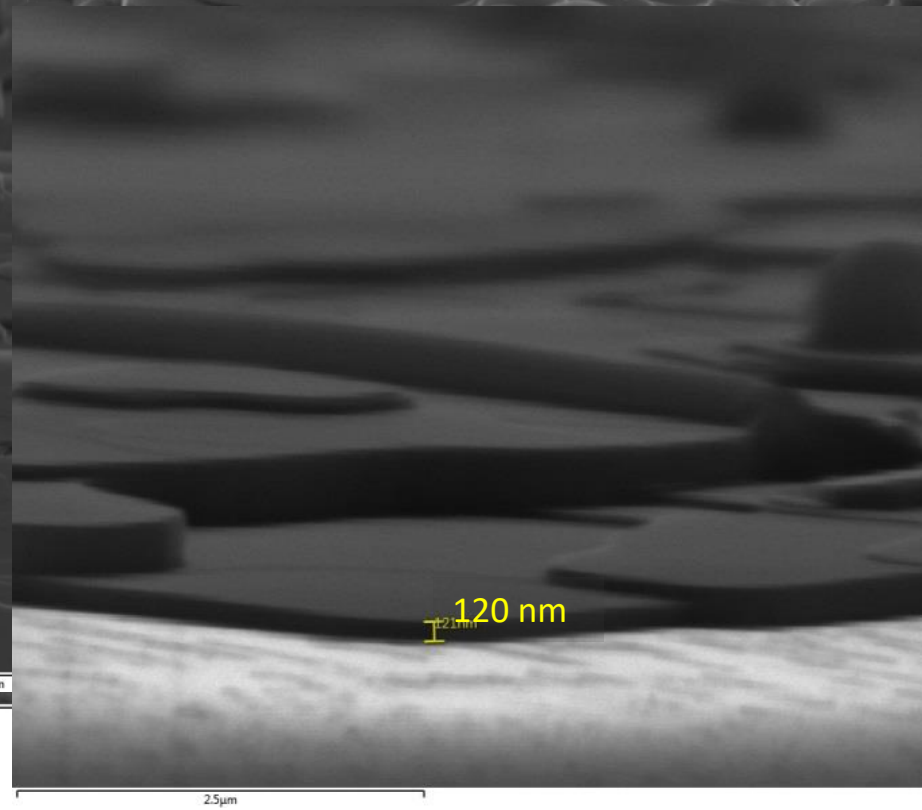




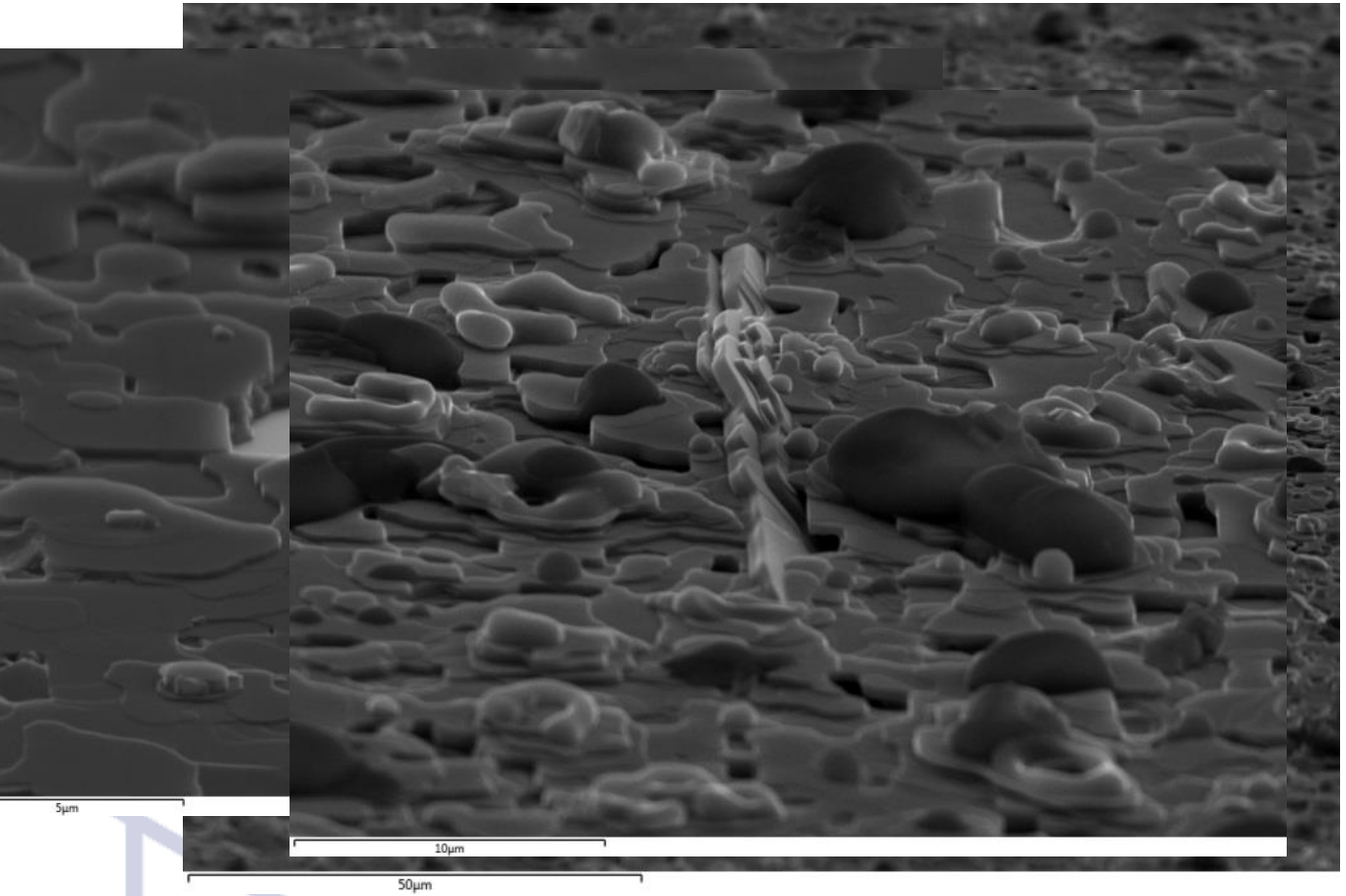




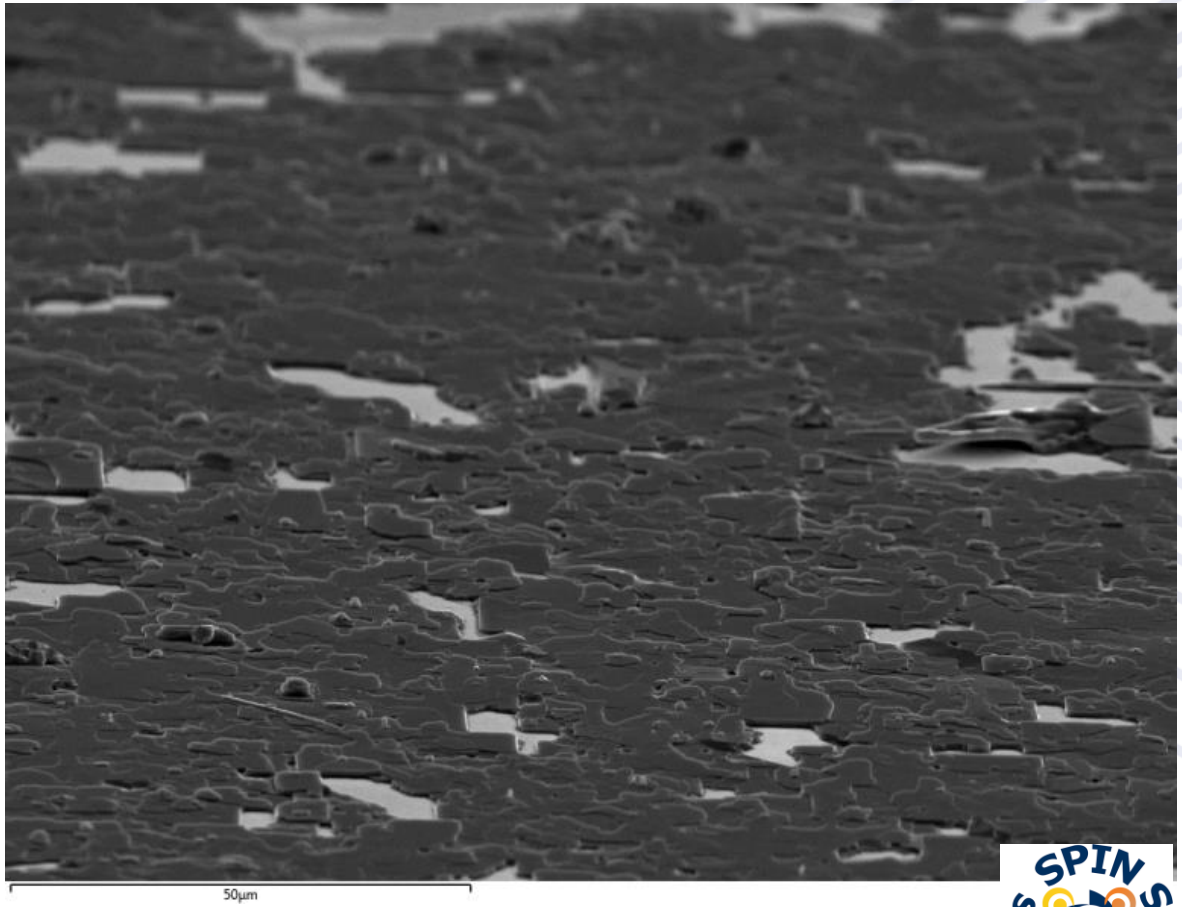




Thicker



Thinner



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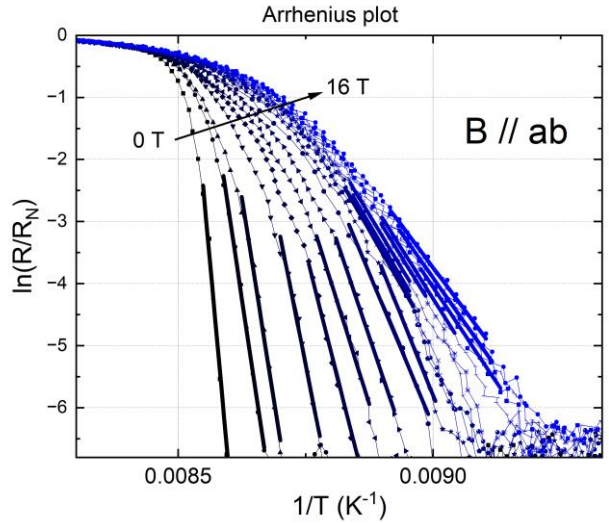
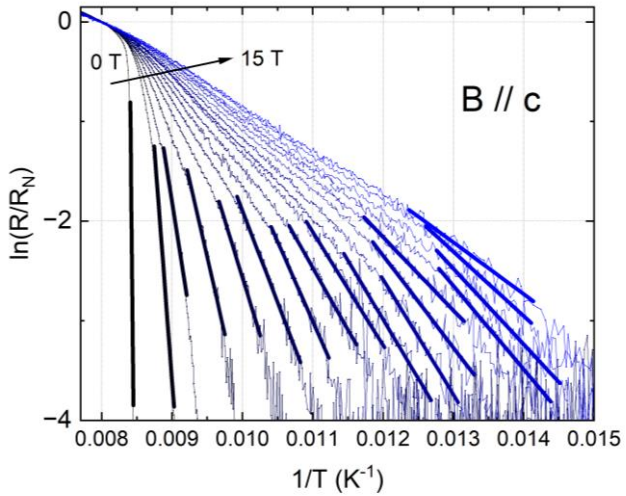
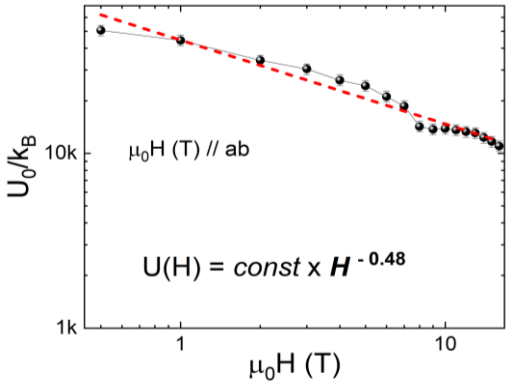
R(T, B) > U_0

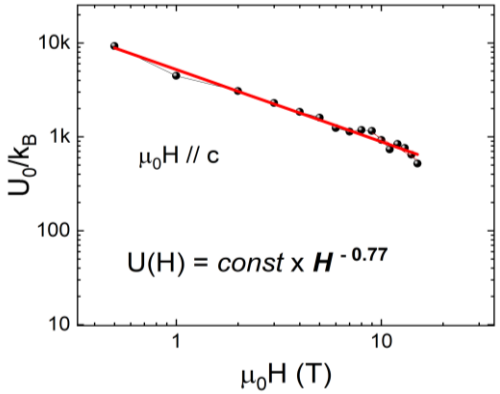




$$R = R_N\, e^{-U(H)/k_B}$$

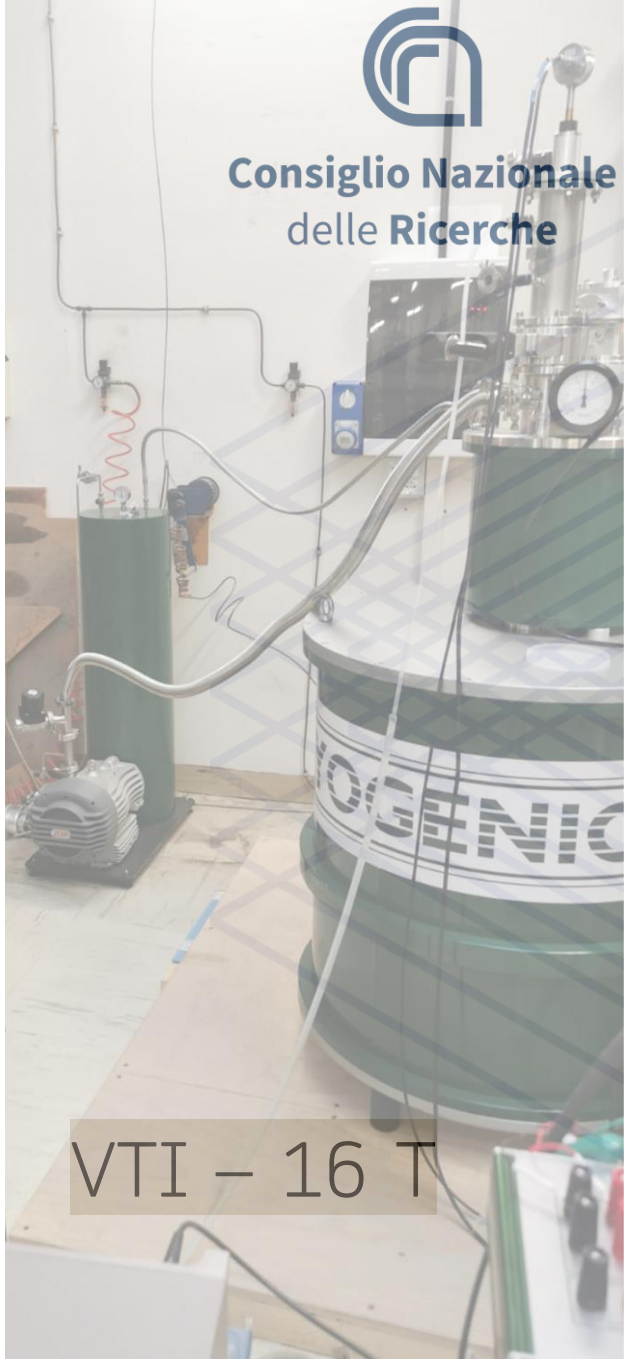
$$U(H) \sim H^{-n}$$





$\mu_0 H \,//\, ab$ with n ≈ 0.48;

$\mu_0 H \,//\, c$ with n ≈ 0.77.



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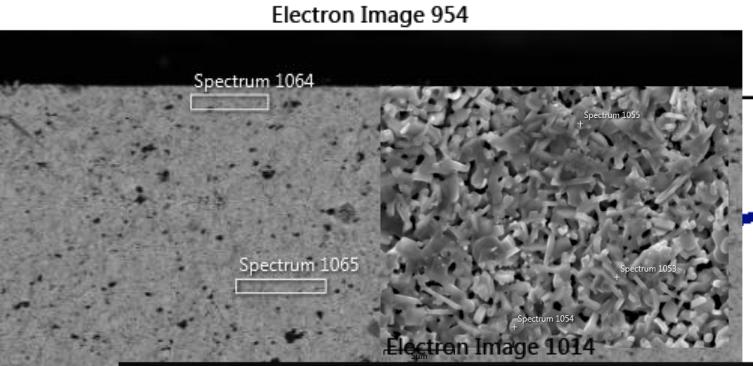
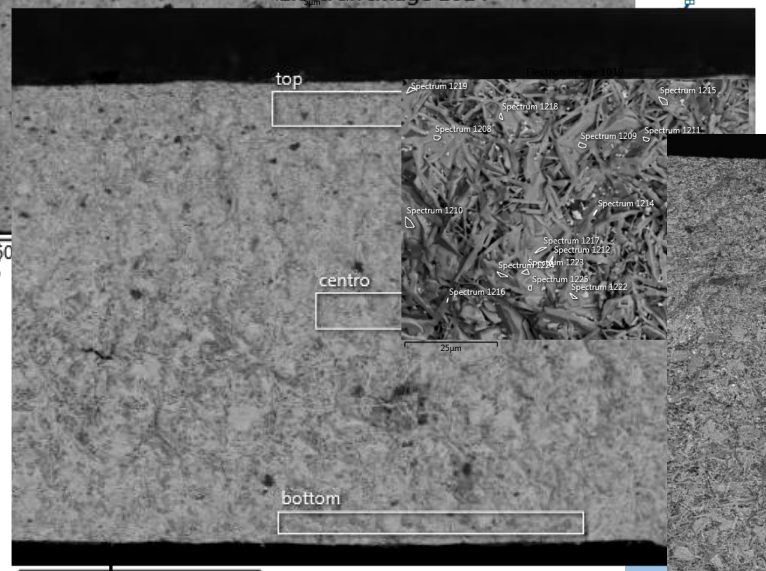
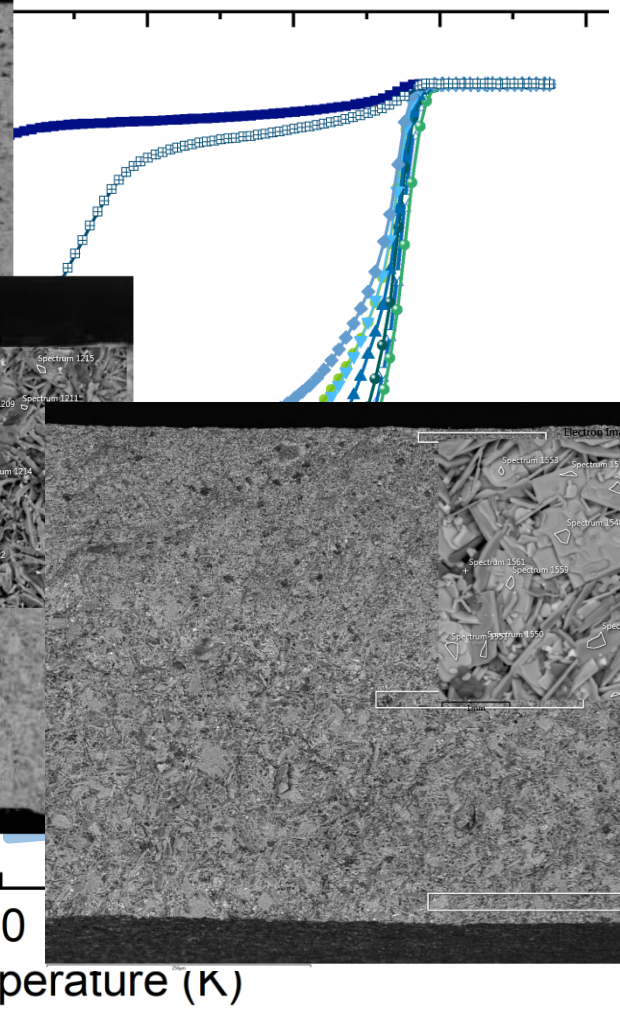
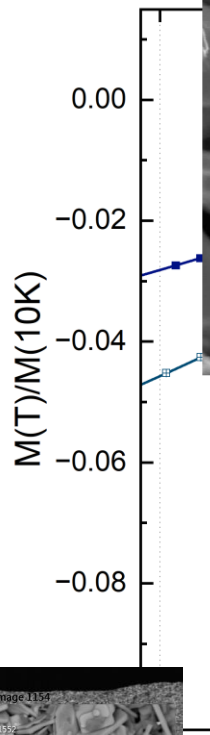
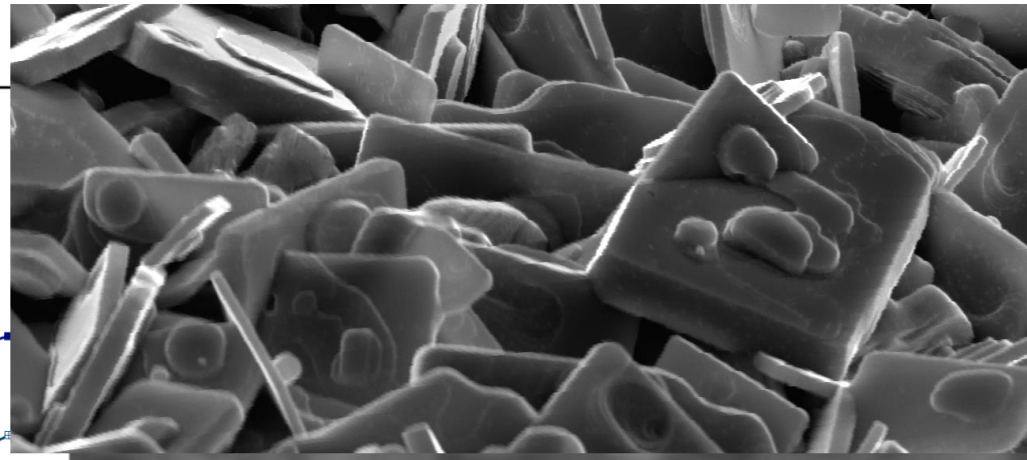
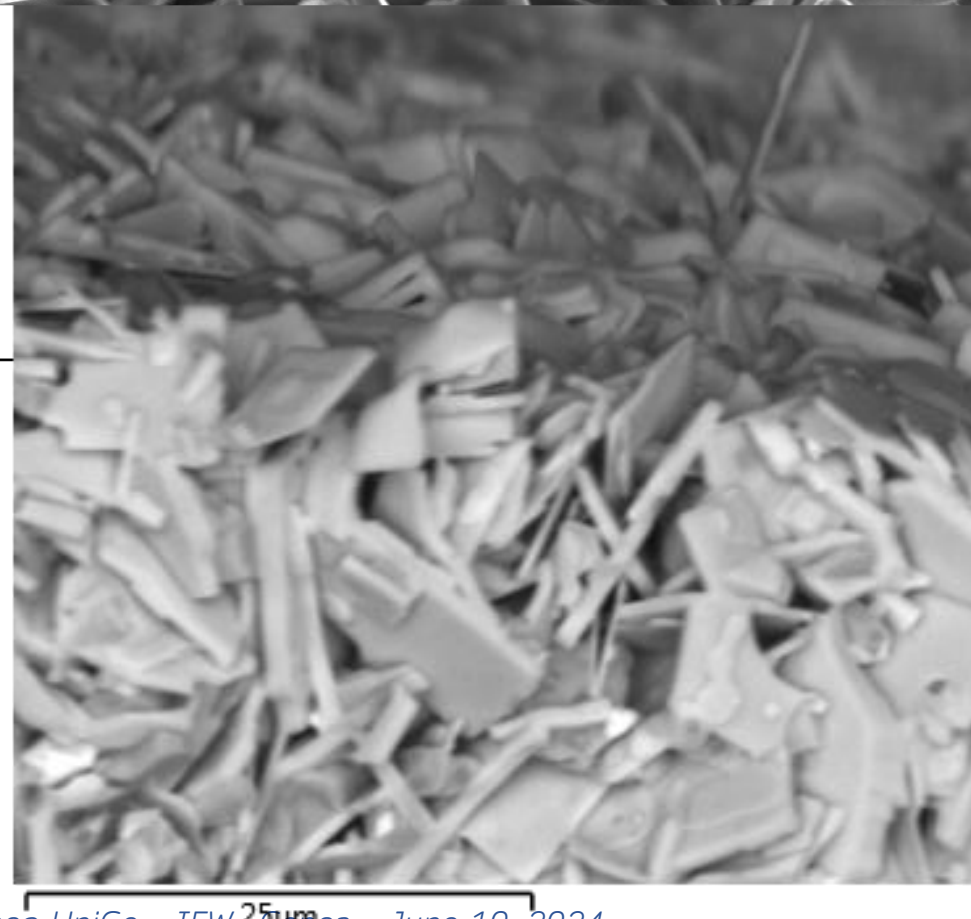






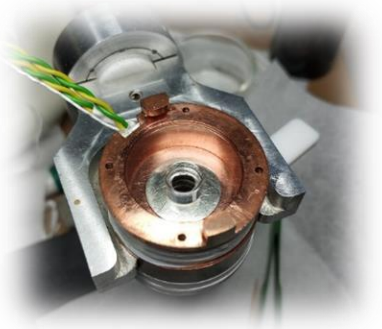




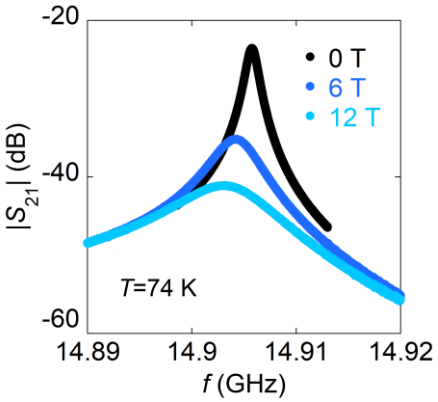


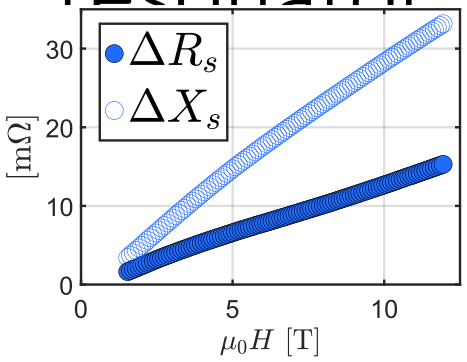
Measurement technique – dual mode dielectric loaded resonator

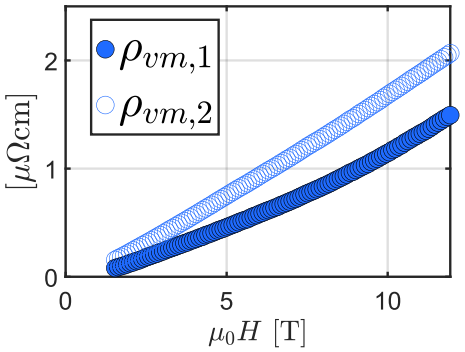
Dielectric loaded resonators
$f_{1,2}$ = [8; 14],[16; 27] GHz

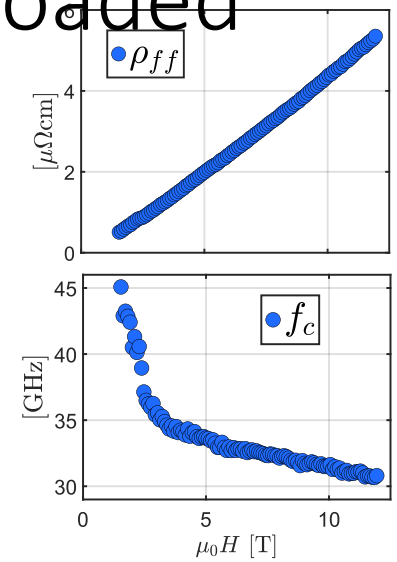


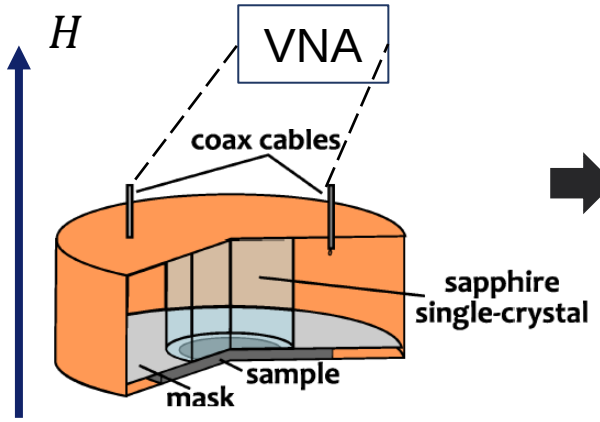
TE$_{021}$ - mode











Q-factor
Res. frequency

Q_1, f_1, Q_2, f_2 at T, H

Modified Lorentzian fit
TMQF - fit

Effective surface impedance

$\Delta Z(T, H, f_1)$
$\Delta Z(T, H, f_2)$

e.m. simulation
geometrical factors
G_i estimation

Vortex motion resistivity

$\tilde{\rho}_{vm}(T, H, f_1)$
$\tilde{\rho}_{vm}(T, H, f_2)$

Classical electromagnetism
+ vortex motion

Flux flow resistivity
Pinning frequency

$\rho_{ff}(T, H)$
$f_p(T, H)$

Single vortex physical model

N. Pompeo et al., Measurement, 184, 109937, 2021
A. Alimenti et al., IEEE Instrum. Meas. Mag., 24(9), 2021

K. Torokhtii et al., Measurement: Sensors 18, 2021
A. Alimenti et al., Meas. Sci. Technol, 30(6), 2020

K. Torokhtii et al., Acta IMEKO 9(3), 2020
K. Leong et al., IEEE Trans. Microw. Theory Techniq., 50(9), 2002

Measurement technique

Measurement technique
= Dielectric-loaded cylindrical resonator.
Note: this is essentially a **bulk** technique, meaning that the surface impedance (a kind of AC electrical resistance) is measured as an average over the **entire sample**, both in terms of surface area and thickness.

Superconducting sample
= Placed inside the resonator, between one of its end plates and the internal dielectric crystal (see schematic in slide 2).

Resonator
= Acts as a source of electromagnetic radiation → induces RF currents on the sample surface, allowing us to measure its resistivity.

The measurement
= A **differential surface impedance measurement** (ΔZ_s), calculated from variations (typically induced by the magnetic field) in the **quality factor Q** and **resonant frequency f** of the resonator with respect to a reference value*.

Theoretical/physical models
= Allow us to extract vortex parameters (such as the **flux-flow resistivity ρ_ff**, **pinning frequency f_p**, etc.).

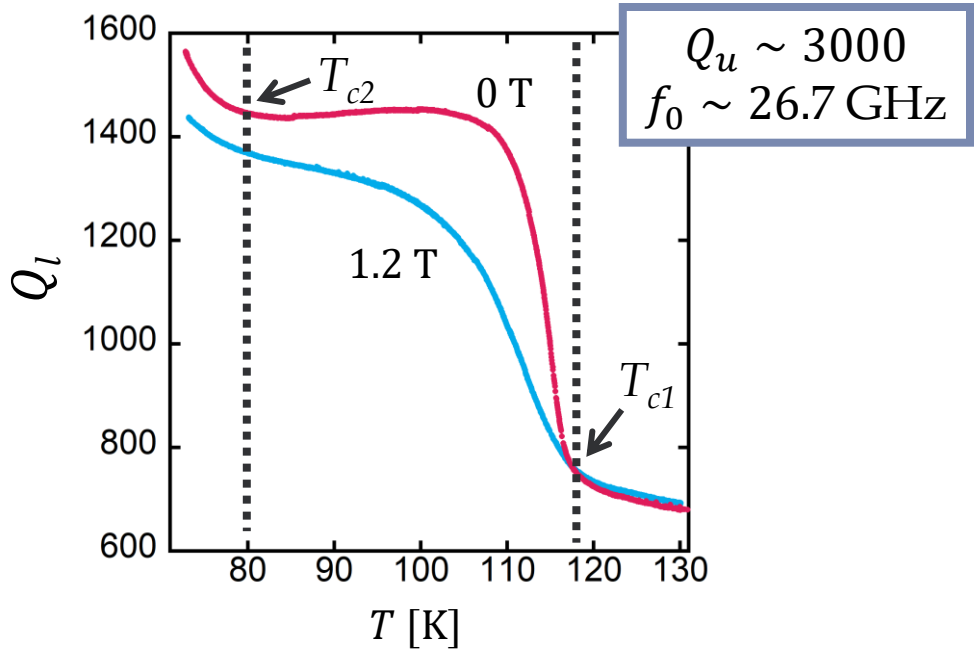
Measurements
= Performed at a fixed frequency; either at constant magnetic field and varying temperature ("transitions"), or at fixed temperature and varying magnetic field ("field ramps", used to extract the vortex parameters mentioned above).

- * *For example, in measurements with varying magnetic field and fixed temperature, the reference values Q(B=0) and f(B=0) are compared to the experimental values Q(B>0) and f(B>0).*

Measured Samples

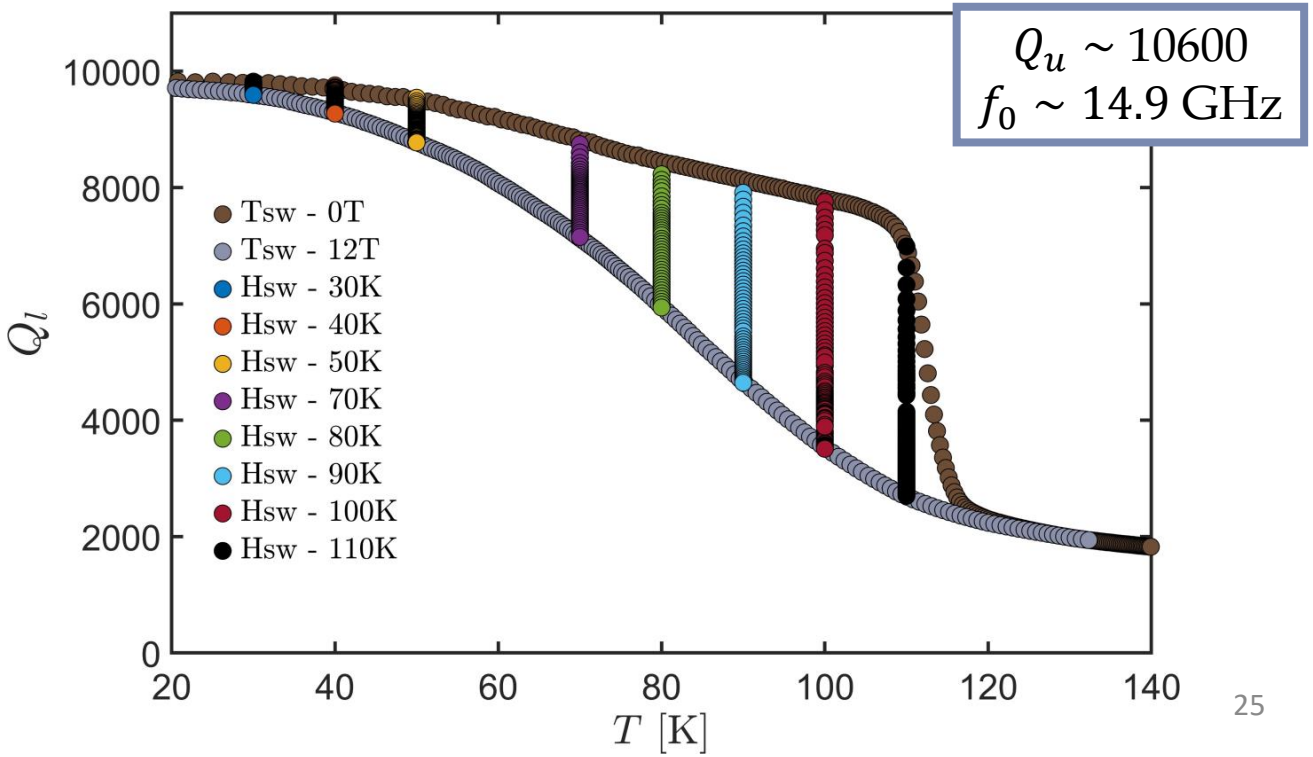
Sample NdYAG012 (03/2024)

- Sample thickness ≈ 1 μm
- RF measurements show two different T_c [116 K (Tl-1223); 80 K (Tl-1212)]
- "Low" Q-factor → large losses (high ΔR_s)

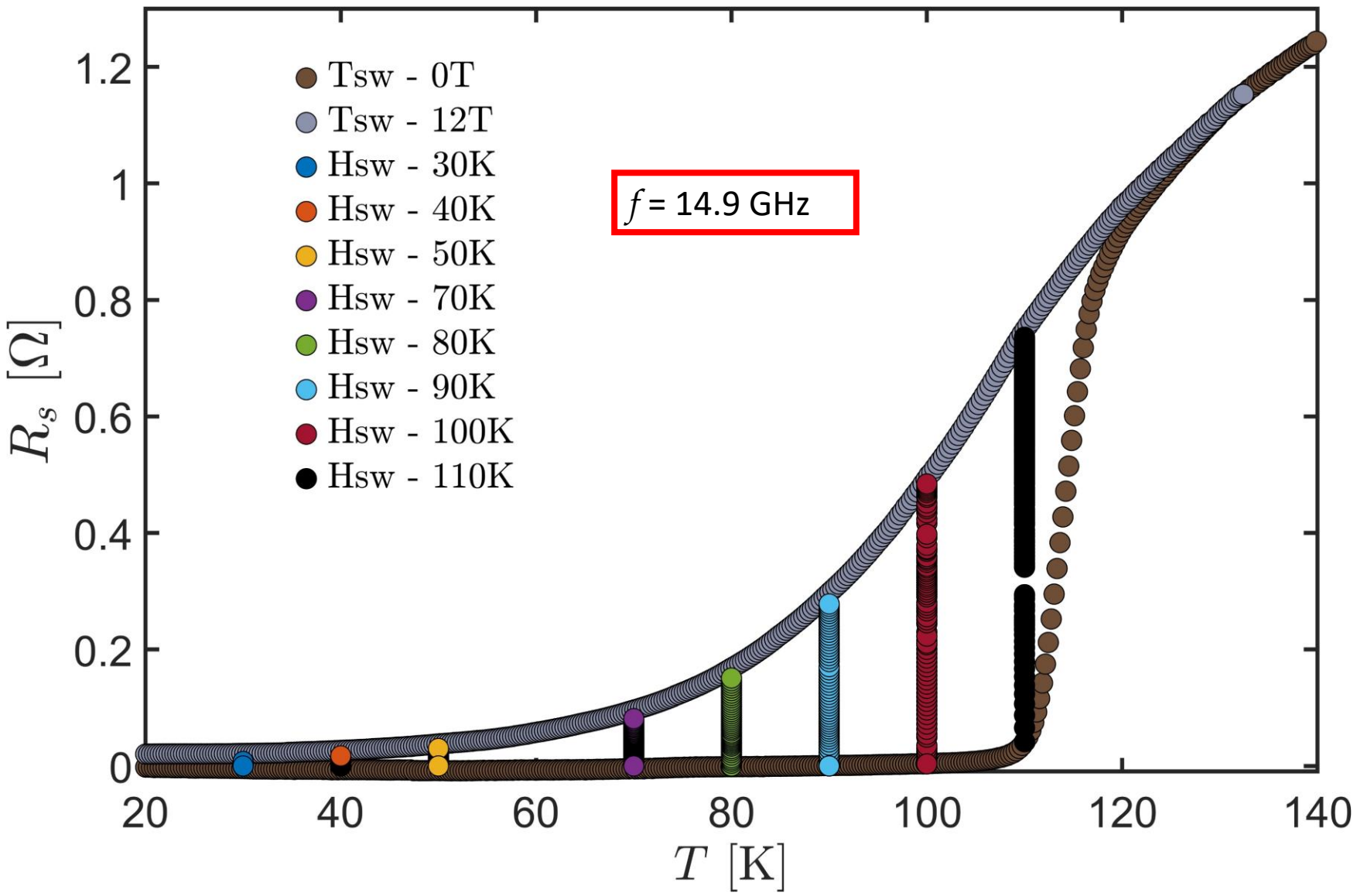


Sample NdYAG031 (12/2024)

- Sample thickness ≈ 1 μm
- T_c = 116 K (Tl-1223), no signs of a second phase
- "Higher" Q-factor compared to the previous sample



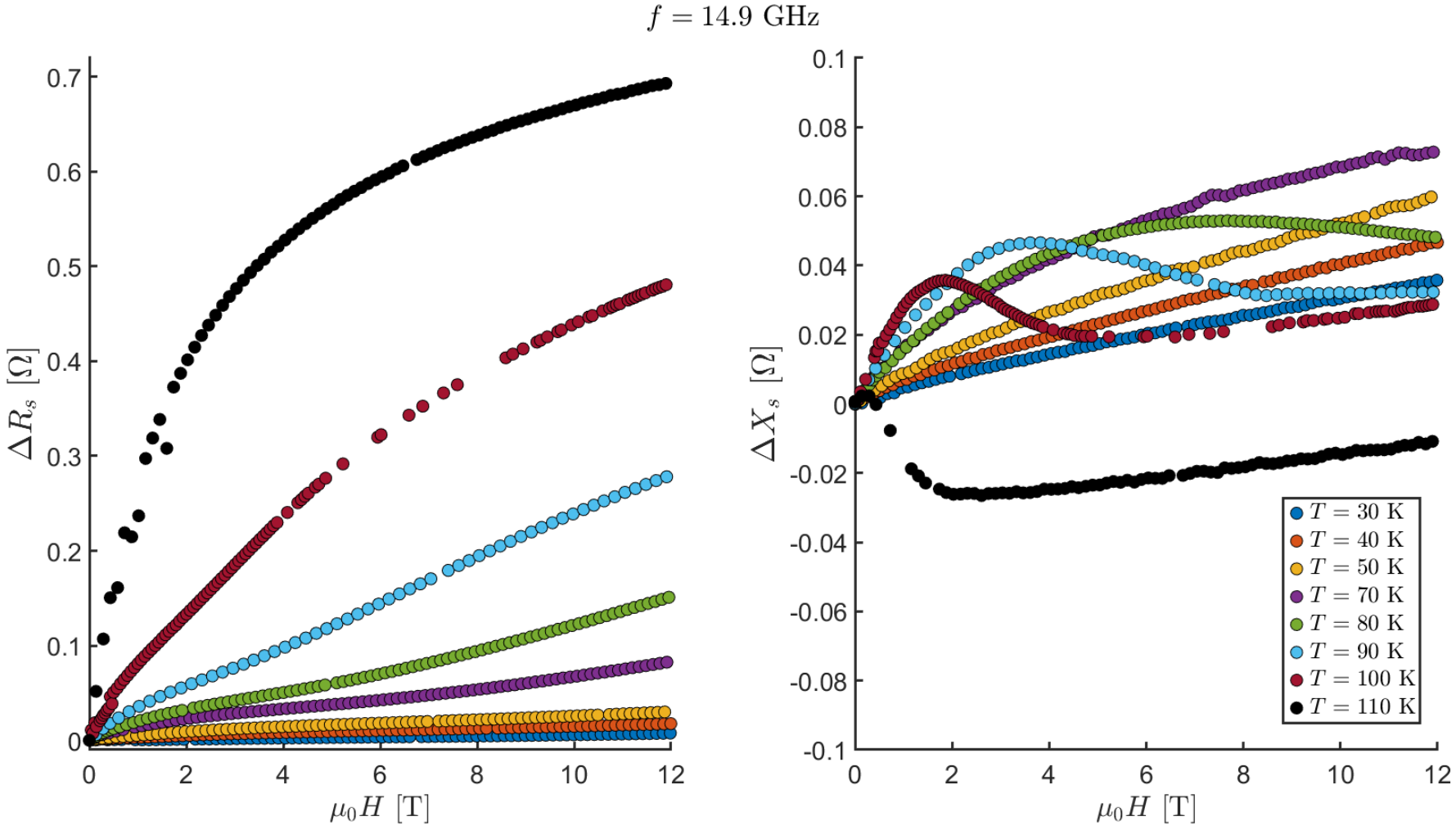
Temperature sweeps at constant B



- Estimated normal state resistivity ≈ 100 μΩ cm
- Normal state resistivity from literature [1,2,3,4] ≈ 300-500 μΩ cm

[1] G. Triscone, *Physica C* 272, 21-25 (1996).
[2] R. Awad, et al., *Supercond. Sci. Technol*,17 35 (2004).
[3] S. Isber, *Supercond. Sci. Technol*. 18 311 (2005).
[4] A.I. Abou Aly, *J Supercond Nov Magn,* 23 (2010).

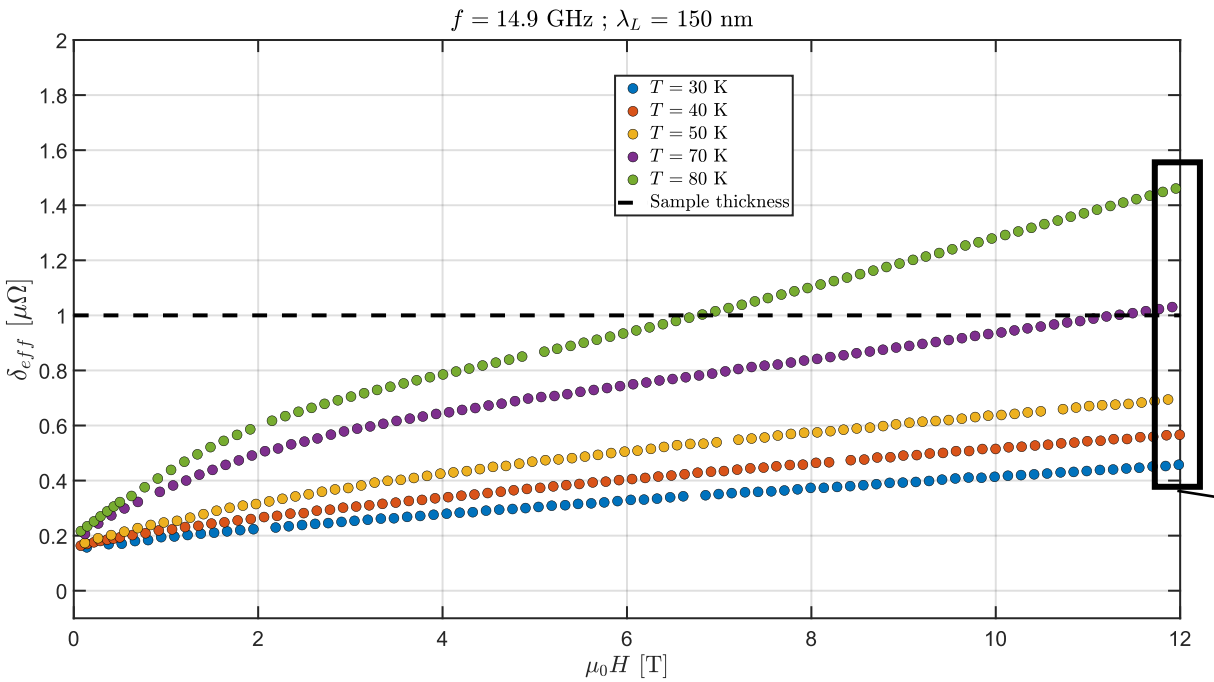
Magnetic field sweeps at constant T (Surface impedance)

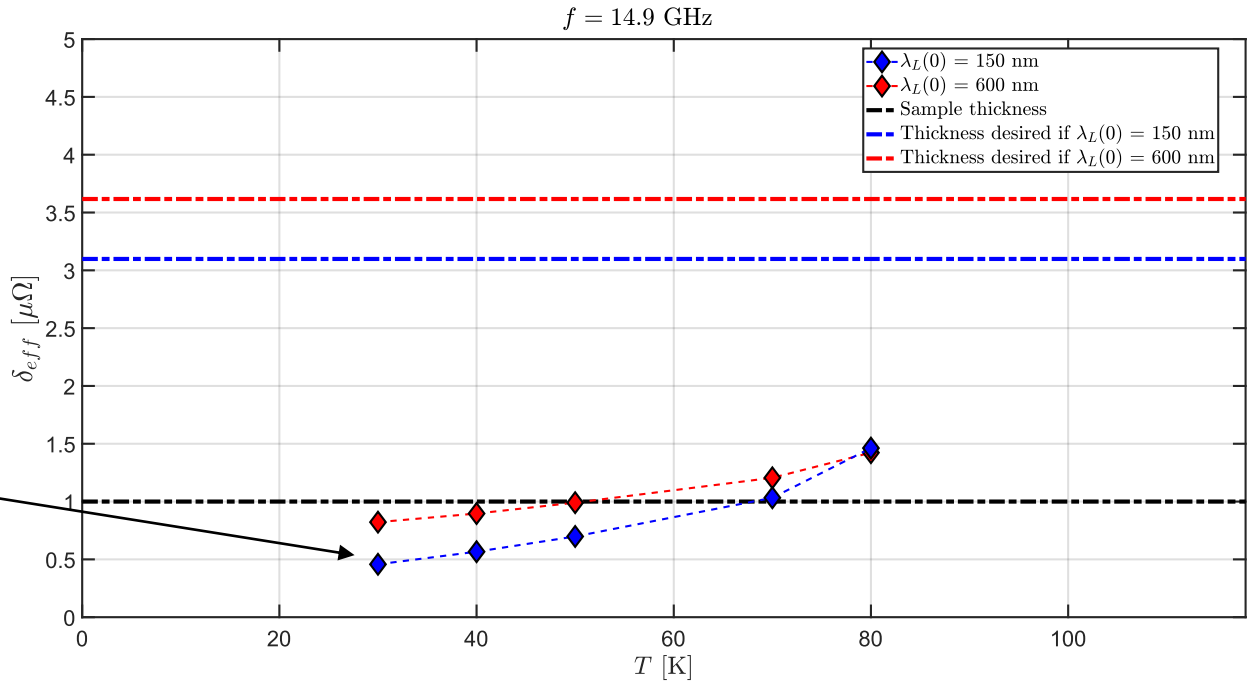


From the magnetic field sweep:

- Rs increases with field -> dissipation due to increased number of fluxons in the sample
- Rs (surface resistance) and Xs (surface reactance) allow for the determination of the vortex parameters (an estimation of of λ_L is needed)

Magnetic field sweeps at constant T (Desired thickness)





To be in the bulk regime (no substrate contribution to R_s), the required coating thickness is computed from the e.m. penetration depth.
This estimation depends on the actual λ_L

At T < 70 K, B = 12 T and f = 14.9 GHz:

- If λ_L = 150 nm → Thickness ≥ 3.1 µm

➡ At lower frequencies, a thicker sample is needed.

In the worst case (pure flux flow), with the present data, at T < 70 K, B = 12 T and f = 2 GHz:

$R_s(f_2) \approx R_s(f_1)\ \mathrm{sqrt}(f_2/f_1)$ → thickness ~ 9 µm

Magnetic field sweeps

Dimensional crossover

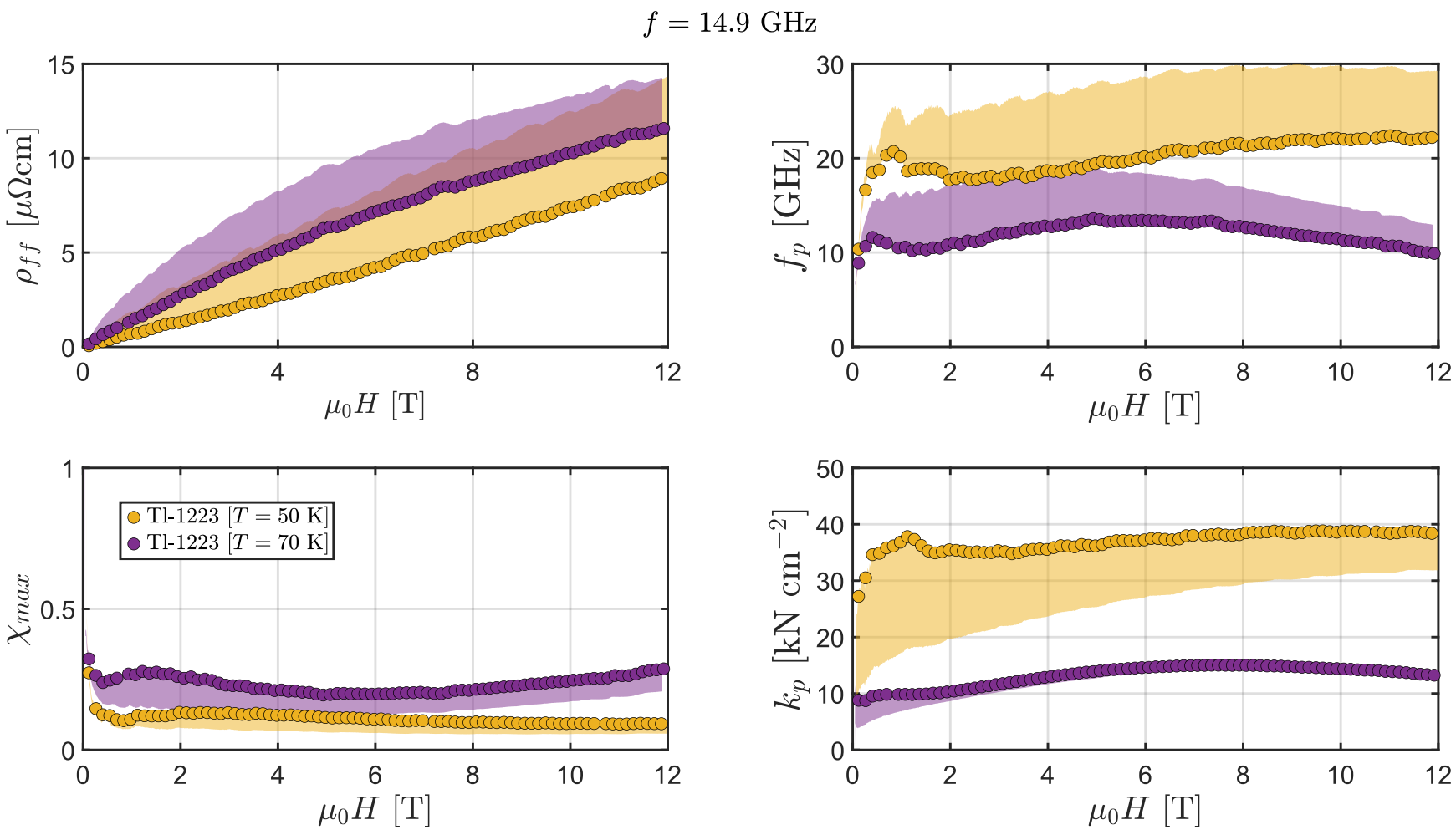
= Depending on the values of magnetic field and temperature, the sample transitions between different electromagnetic geometries (bulk, thick film, thin film).
This complicates both **data analysis** and **interpretation**, making the **London penetration depth λL** a particularly critical parameter.

λL

= Leaving aside the strictly physical aspects, the analysis was carried out by treating **λL as a free parameter**.
As a result, the extracted microscopic parameters are accompanied by a **"confidence range"** (shaded areas) that reflects the possible variations of λL.
Clearly, the range is wide (λL up to 600 nm), yet the confidence intervals remain relatively narrow.

- **Desired thickness**
  = In view of a potential FCC application, and in order to **avoid dimensional crossovers**, the system must operate in the **bulk regime**.
  To achieve this with the current performance of the superconductor, a **minimum thickness of 4–5 µm at 70 K and 12 T** is required.
  If the material performance is improved, a thinner layer will be sufficient to stay in the bulk regime (and thus effectively shield electromagnetic radiation).

Magnetic field sweeps at constant T (Pinning Parameters)



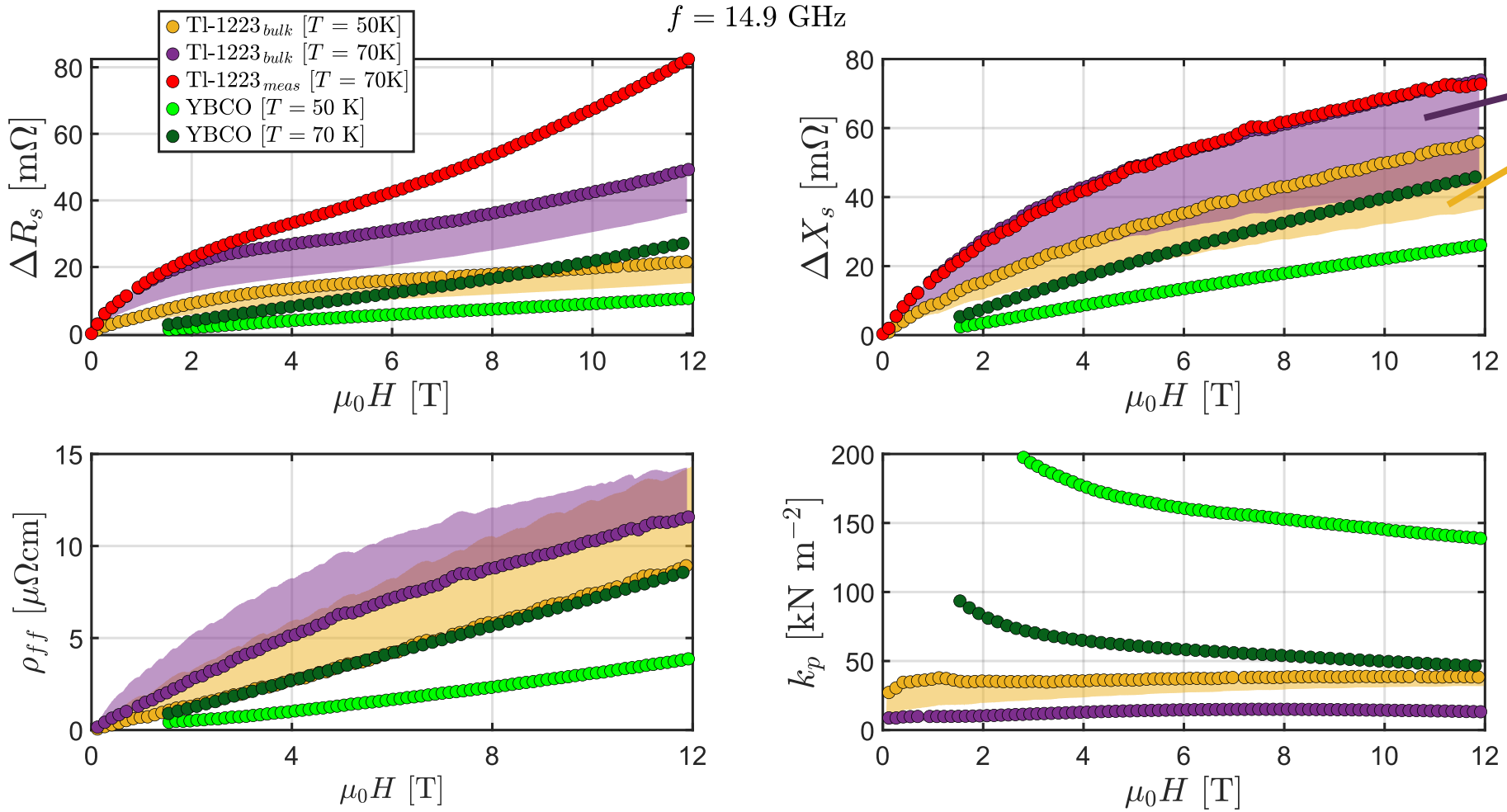
Bands refer to different assumed values for $\lambda_L(0)$ = [150,600] nm (full dots correspond to 150 nm)

Gittleman-Rosenblum pinning parameters, with the estimation of the maximum creep factor χ_{max}, see:

N. Pompeo, et al., *Physical Review B* 78.9 (2008): 094503.

Considering pinning, at T < 70 K, B = 12 T and f = 2 GHz:

- If $\lambda_L(0)$ = 150 nm → thickness ~ 4 μm
- If $\lambda_L(0)$ = 600 nm → thickness ~ 5.5 μm

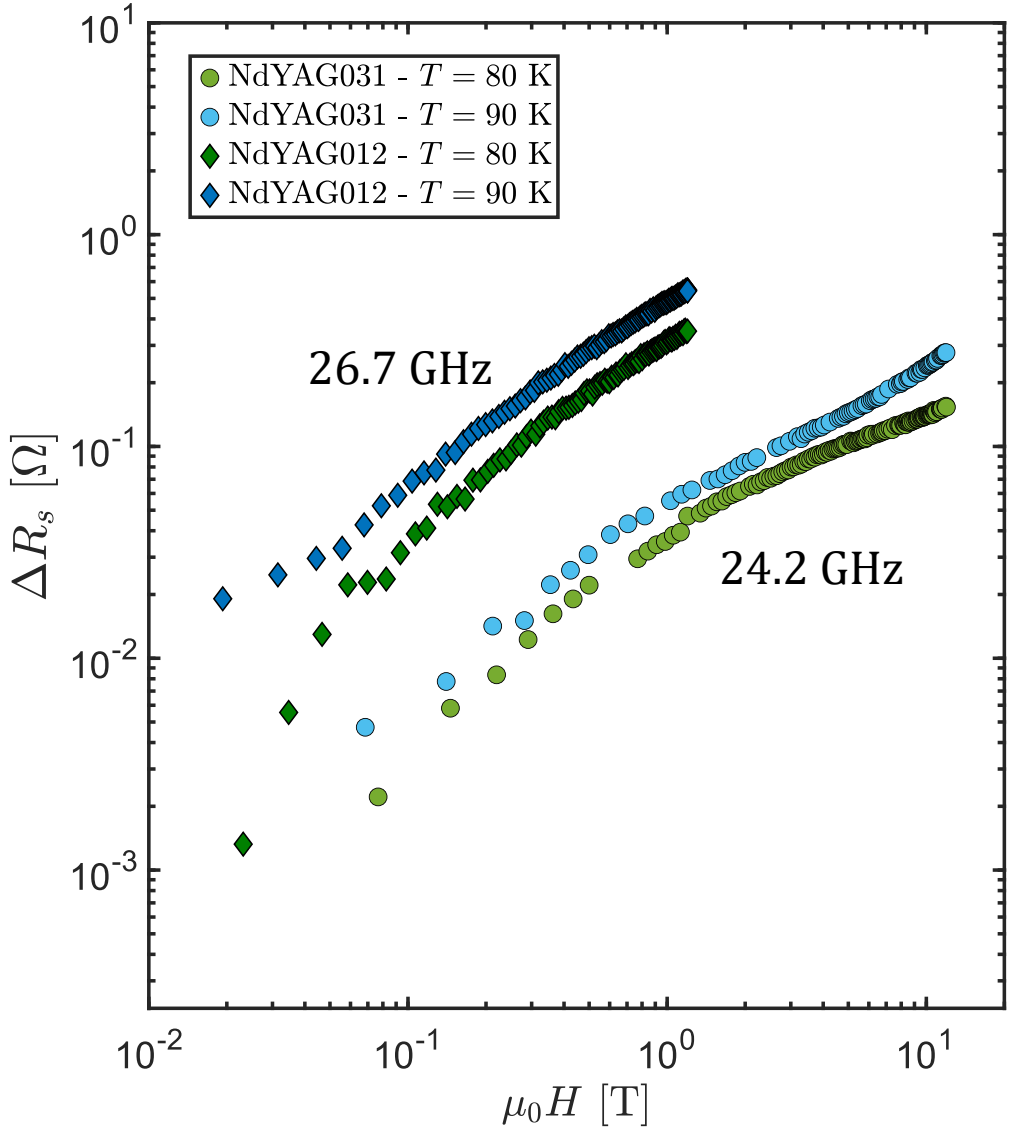


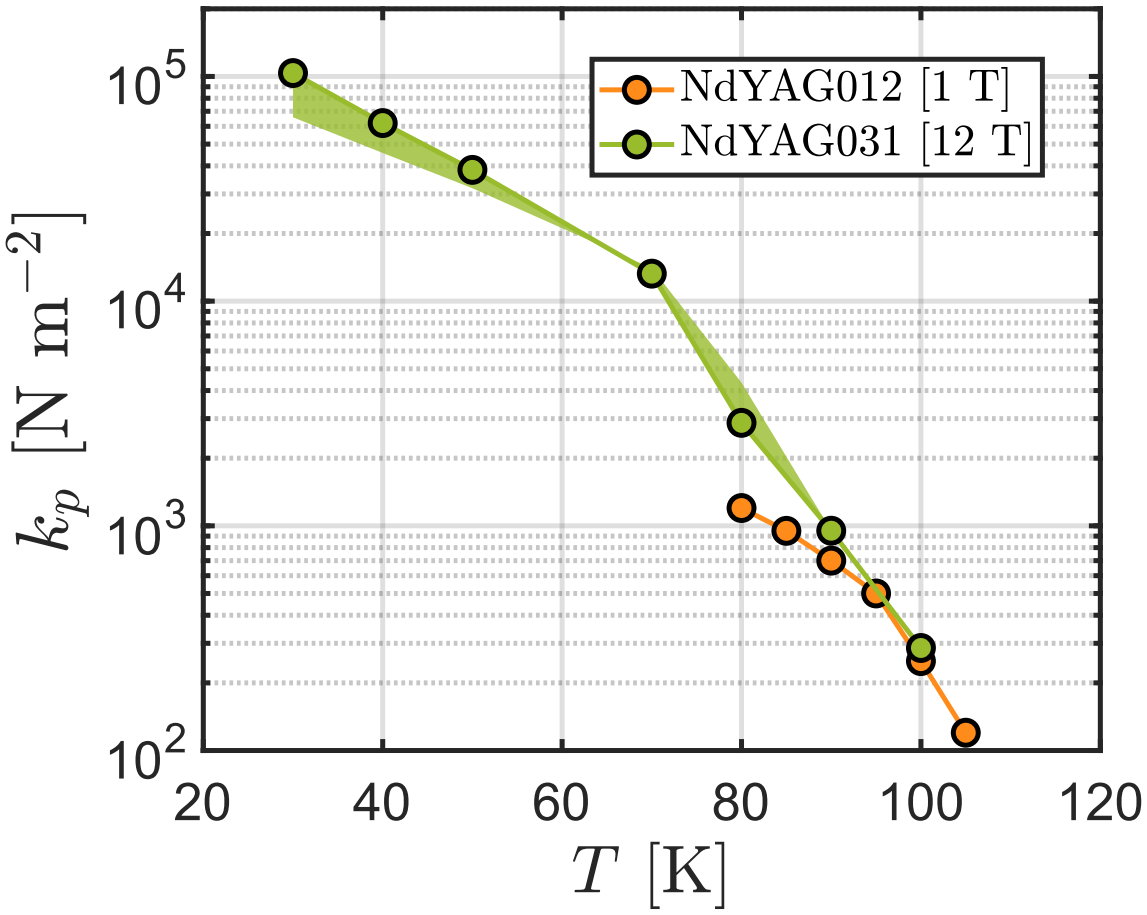
Bands refer to different assumed values for $\lambda_L(0)$ = [150,600] nm

Wrt YBCO SuperPower CC:

- $R_{s,bulk}$ (computed assuming a thickness of ~3 μm) within a factor of 1.5-2
- k_p is the dominant factor in R_s difference @ 70 K
- Promising results:
  - ✓ k_p constant in the T-B range reported (crucial for high B applications)
  - ✓ Ample margins for k_p optimization

Pinning parameters comparison, magnetic field sweeps at constant T





The recent sample (NdYAG031) clearly shows improved performances both in ΔR_s and k_p compared to the old one (NdYAG012)

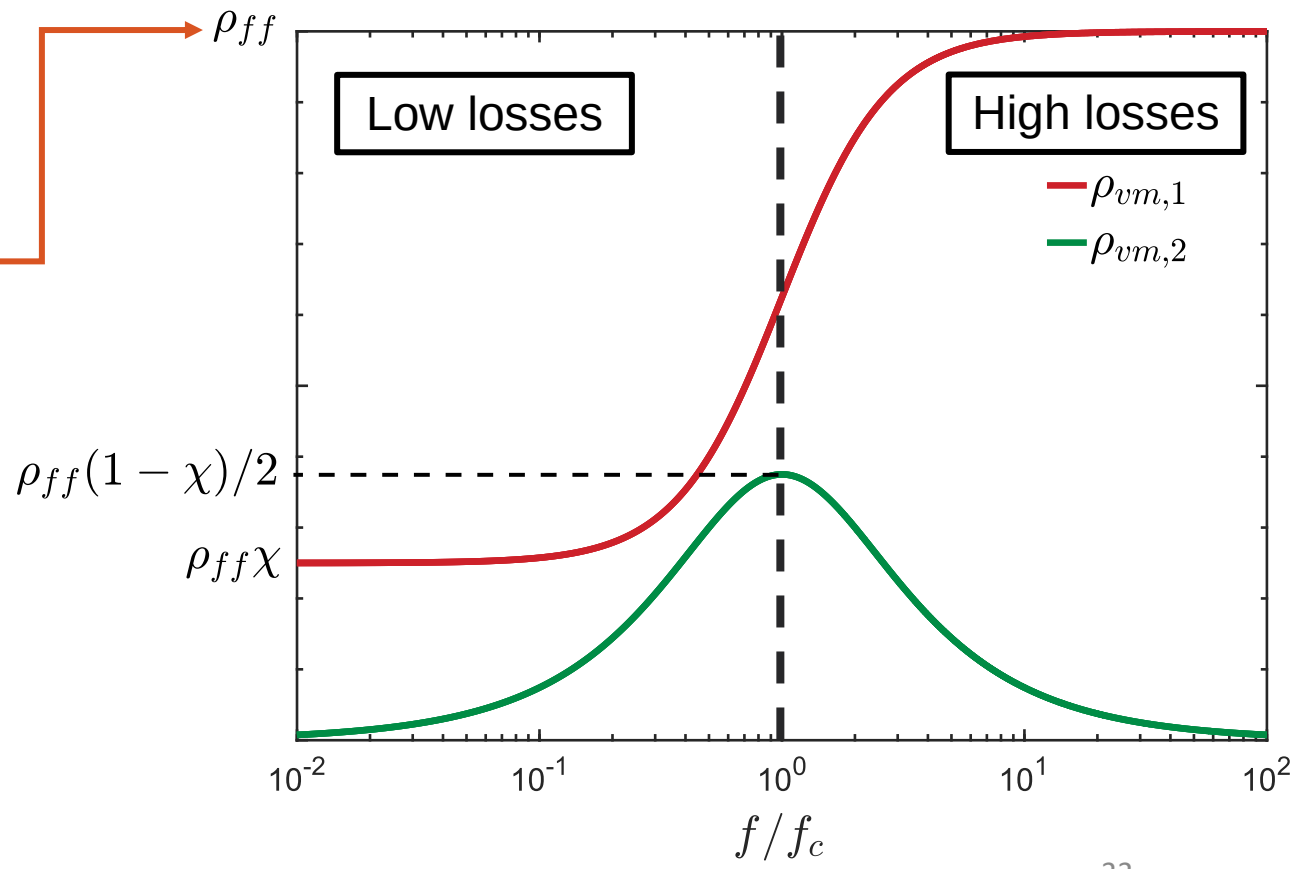
Vortex motion resistivity $\tilde{\rho}_{vm}$

Resistività di vortice (grandezza complessa)

$$\tilde{\rho}_{vm} = \rho_{vm,1} + \mathrm{i}\rho_{vm,2} = \rho_{ff}\frac{\chi + \mathrm{i}\frac{f}{f_c}}{1 + \mathrm{i}\frac{f}{f_c}}$$

- $\rho_{ff} = \rho_n B/B_{C2}$: flux-flow resistivity (dissipation)
- f_c (or f_p): pinning frequency (pinning strength)
- χ : creep factor (thermal activation)

For high−frequency applications:

- Minimize the flux-flow resistivity ρ_{ff} (dissipation)
- Increase the pinning frequency $f_c > f$ (pinning strength)

$\tilde{\rho}_{vm}$ ⇩ ➡ R_s ⇩