This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

Main highlights ARIES WP15 on Thin Film for Superconducting RF

Oleg Malyshev
ARIES WP15 coordinator
on behalf of the WP15 team

ARIES Final Review Meeting, 15th July 2022

WP15 Description

- The aim of this work package is to intensify systematic studies and development of the coating technology of superconducting materials to enable the superconducting coated RF cavities with $Q(E)$ characteristics better than for the bulk ones.
- The main emphasis is on **a systematic study of correlation** between
  - substrate surface preparation,
  - deposition parameters for:
    - superconducting material **Nb, NbN, Nb_3Sn, NbTiN and SIS**
    - deposited on **Cu** substrate,
  - film structure, morphology, chemistry, phase,
  - AC and DC superconductivity parameters:
    - such as T_c, H_c, H_{fp}, H_{sh}, RRR
  - and, finally, the behaviour at RF conditions with the test cavities at CERN, HZB and STFC.

Work distribution for WP15 Tasks

The main emphasis is on a systematic study of correlation between

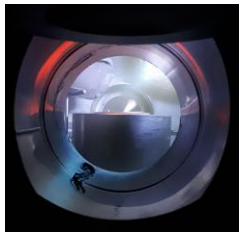
Surface preparation
- Cleaning, etching,
- Polishing, passivating

TASK 15.2

Thin film deposition
- **PVD**: DC, pulsed, HIPIMS…
- (PE)CVD, (PE)ALD
- **Nb, NbN, Nb_3Sn**, MgB_2, etc.

TASK 15.3

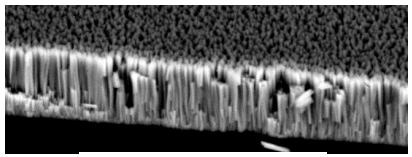
Film deposition



Film characterisation
- SEM, FIB, AFM,
- XPS, XRD, RBS, TEM…

TASK 15.4

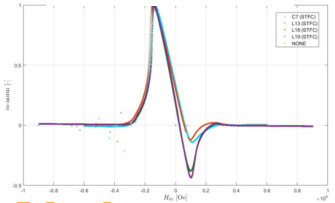
SEM image of a NbN thin film



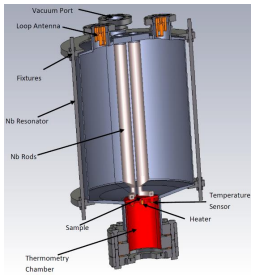
Superconducting properties measurement

RRR, H_c, H_{fp}, H_{sh}, …
DC magnetic susceptibility,
Field penetration

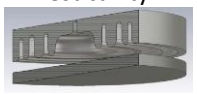
DC magnetisation



QPR



Test cavity



Superconducting RF properties evaluation
- QPR at CERN
- QPR at HZF
- HW cavity at ASTeC

Real cavity measurement
- Cavity deposition

WP15 Partners

	Participants	Leading	Participating
1	CEA (Saclay, France)		Task **4**
2	*CERN (Geneva, Switzerland)*		**All tasks**
3	IEE-SAS (Bratislava, Slovakia)		Tasks **4**
4	LNL/INFN (Legnaro, Italy)	Task 2	Tasks 1, **2** and **3**
5	Helmholtz-Zentrum Berlin (Berlin, Germany)	Task 4	Tasks 1 and **4**
6	RTU (Riga, Latvia)		Task 2 and **3**
7	University Siegen (Siegen, Germany)		Tasks **3**
8	ASTeC/STFC (Daresbury, UK)	WP and Tasks 3	**All tasks**
9	Lancaster University* (Lancaster, UK) **in-kind contribution*		Task **4**

WP coordination

- WP operates as a single team (good integration)
  - Each task includes 2 or 3 partners with similar expertise
  - Most of partners participate in 2-4 tasks
  - **WP meeting** took place every 3 month (5-8 hour long meeting with a report from each partner for each task)
    - Tasks briefing meeting took place between WP meeting when needed (every 1-1.5 months)
  - **Task 1** (coordination) meetings:
    - usually were a part of WP and Task meeting,
    - dedicated short zoom meeting,
    - emails and phone calls

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

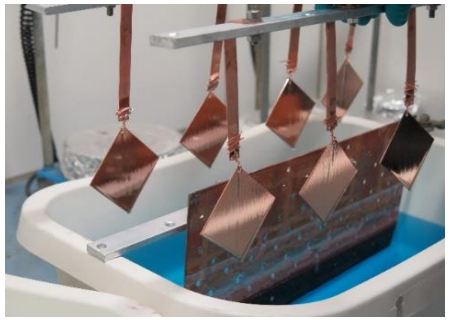
Superconducting Thin Film Development

Objectives

Thin film development on small samples (53 mm × 53 mm)

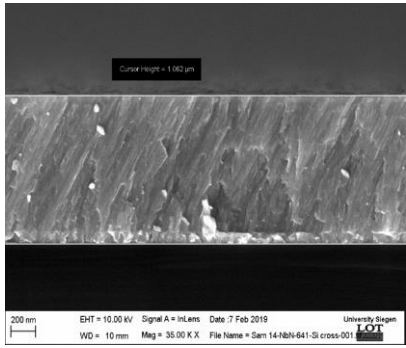
Task 15.2

- Copper substrate polishing
- with EP, SUBU5, EP + SUBU5, Tumbling (INFN)
- with SUBU5 (CERN)
- with laser (RTU)



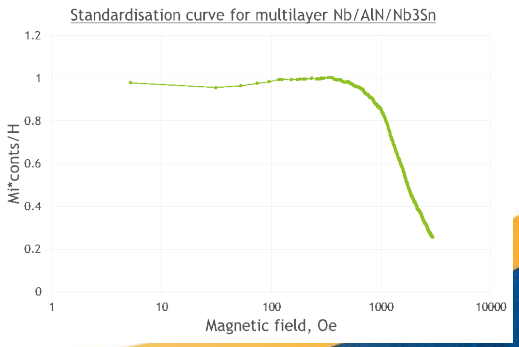
Task 15.3

- Nb film deposition (STFC, Siegen, INFN)
- Laser treatment of the film (RTU)
- NbN, Nb_3Sn, NbTiN and SIS deposition (STFC, Siegen)
- Film characterisation (STFC, Siegen, INFN, RTU)



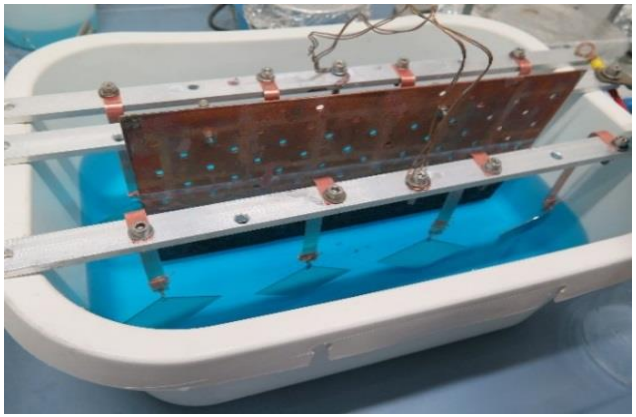
Task 15.4

- DC and AC superconductivity evaluation measurements (CEA, IEE, STFC)



Task 15.2. Substrate surface preparation

- 50 planar copper samples with a size of 53 mm x 53 mm were cut at CERN from the same copper sheet then polished with different procedures:
  - 25 samples were treated at CERN with
    - SUBU solution
  - 25 samples were treated at INFN with
    - SUBU solution,
    - Electropolishing (EP),
    - SUBU+EP,
    - Tumbling
- Based on results from 1st and 2nd year SUBU and EP were selected as most promising polishing procedures for the following WP15 work
- Results were presented
  - in IPAC and SRF conferences,
  - International Workshops on Thin Films & New Ideas for Pushing the Limits of RF Superconductivity



SUBU5 and EP treatments Courtesy of E. Chyhyrynets and C. Pira (INFN)

More details in Deliverable Reports 15.1 & 15.4

Task 15.3. Thin film deposition

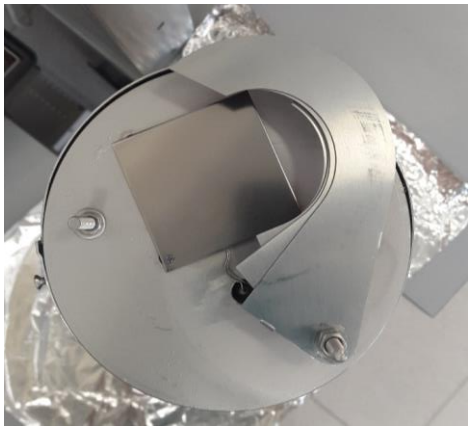
- Thin film **deposition facilities** are key facilities for the project
- Quality of Nb films deposited in Year 1 and 2 at **INFN, Siegen** and **STFC** is comparable
- Year 3 and 4: the main focus is on producing and testing the films *different from Nb*: NbN, Nb_3Sn, NbTiN, as well as SIS structures

Deposition at facilities at different institutes:

University Siegen

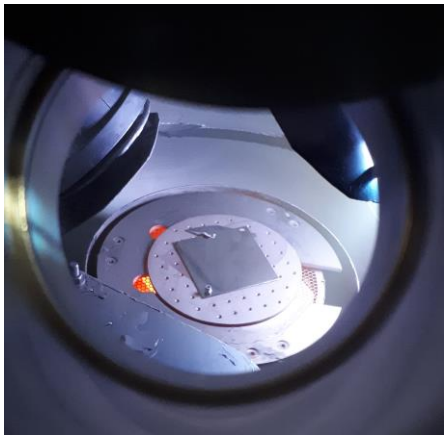
A sample and a Nb target in deposition facility at University Siegen.
Courtesy of M. Vogel (Siegen)

LNL/INFN



A sample and sutter assembly for the Nb deposition at LNL/INFN
Courtesy of C. Pira (INFN)

ASTeC/STFC

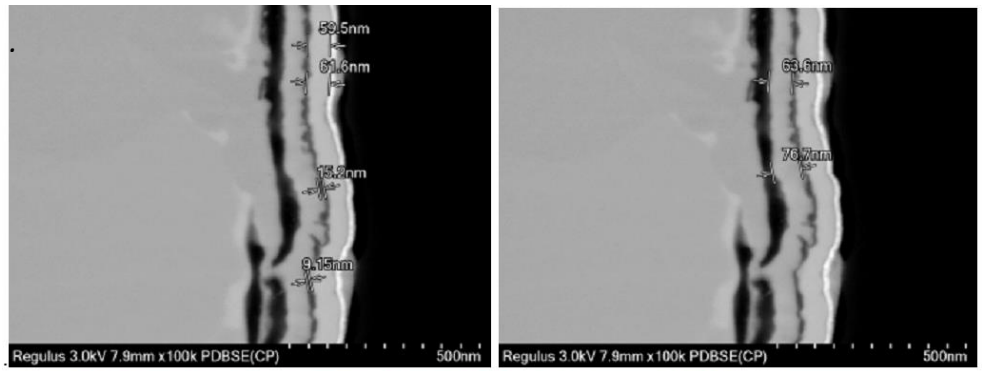


A sample during the Nb deposition at ASTeC/STFC
Courtesy of R. Valizadeh (STFC)

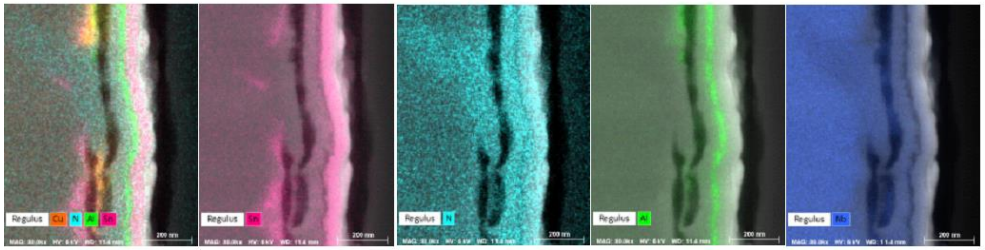
Task 15.3. Thin film characterisation

At STFC

(a)



(b)

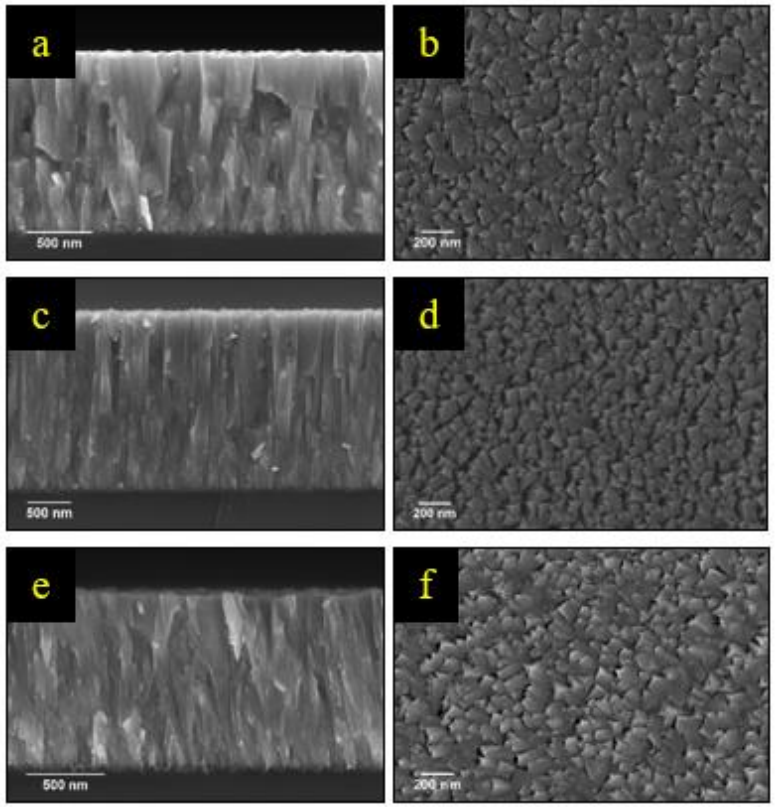


(a) *High resolution SEM of ion milled X-section of SIS multilayer structure (Nb/AlN/Nb3Sn) deposited on Ta.*

(b) *EDX chemical mapping of the X-section.*

Courtesy of R. Valizadeh (STFC)

At University Siegen



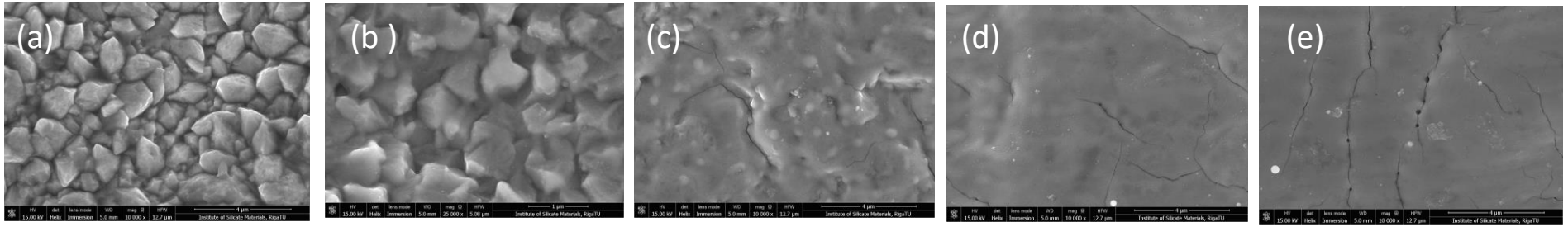
Cross sectional SEM images of NbN

Courtesy of M. Vogel (Siegen)

Task 15.3. Laser treatment Nb films at RTU

Aims:

- Increase the grain size of Nb;
- Increase the adhesion of Nb layer to Cu substrate (annealing the defects by laser radiation);
- Improve superconducting properties.



SEM images of Nb/Cu structure before irradiation (a) and after irradiation by Nd:YAG laser with I_1 = 140 MW/cm^2 (b); I_2 = 170 MW/cm^2 (c); I_3 = 253 MW/cm^2 (d); I_4 = 320 MW/cm^2 (e).

Courtesy to Arturs Medvids, Pavels Onufrijevs and Jevgenijs Kaupuzs (RTU)

Main results for Nb film irradiated by laser:

- The sizes of Nb crystals can be increased
- Defects between grains (pinholes) can be eliminated
- Superconducting properties are changing

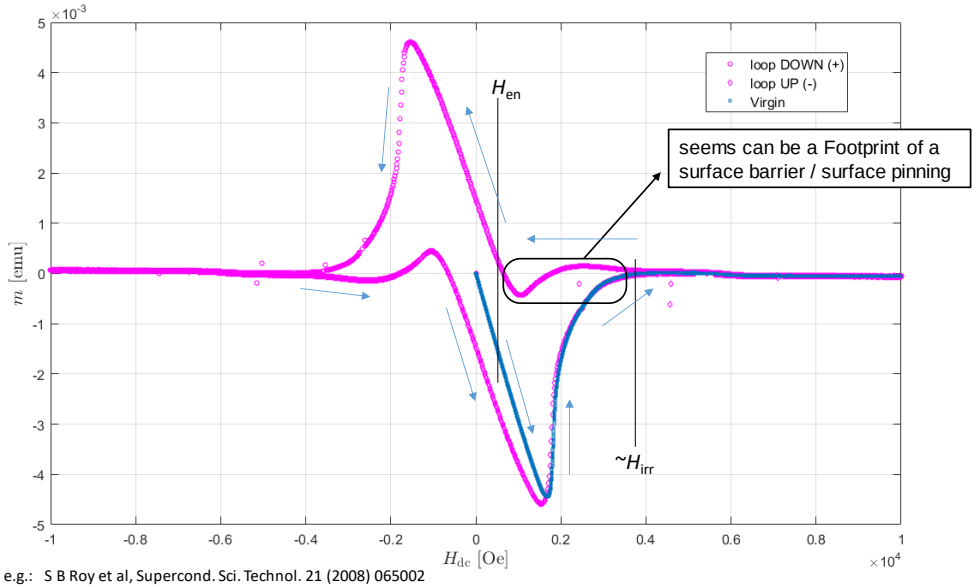
Results reported in 2 papers and at the 9th International Workshop on Thin Films & New Ideas for Pushing the Limits of RF Superconductivity, 15-18 March 2021. More details in Deliverable Reports 15.3 & 15.4

Task 15.4. DC Superconductivity evaluation at IEE

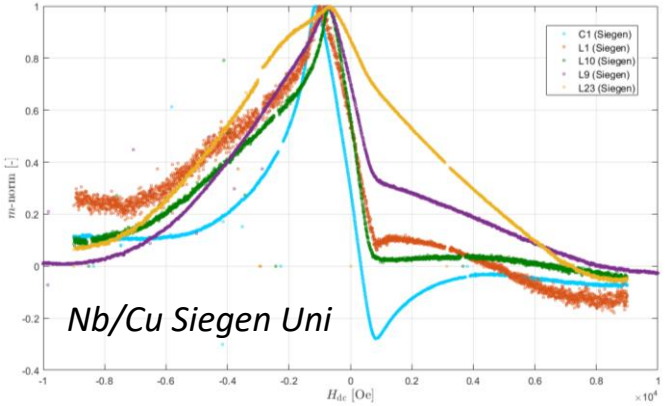
PPMS (Physical Property Measurement System)

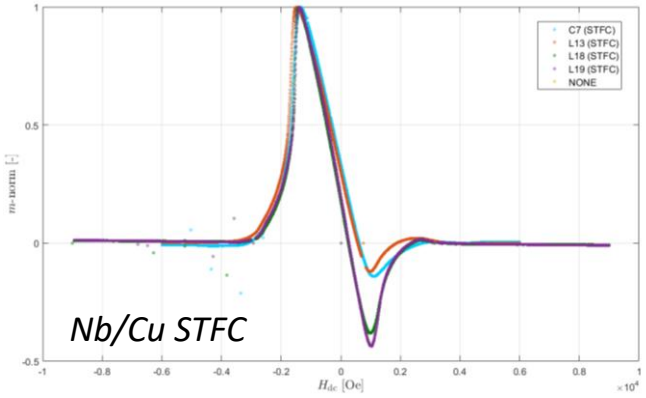
- Virgin DC magnetisation curve: $B_{en}(\sim B_{c1\ perp})$, $[B_p, B_{c2}]$

Shape of magnetization loop

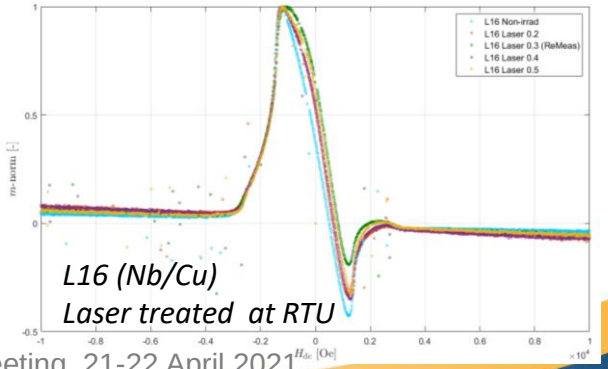


see e.g.: S B Roy et al, Supercond. Sci. Technol. 21 (2008) 065002
E H Brandt, Physica C 332 (2000) 99–107
A S Dhavale et al, Supercond. Sci. Technol. 25 (2012) 065014



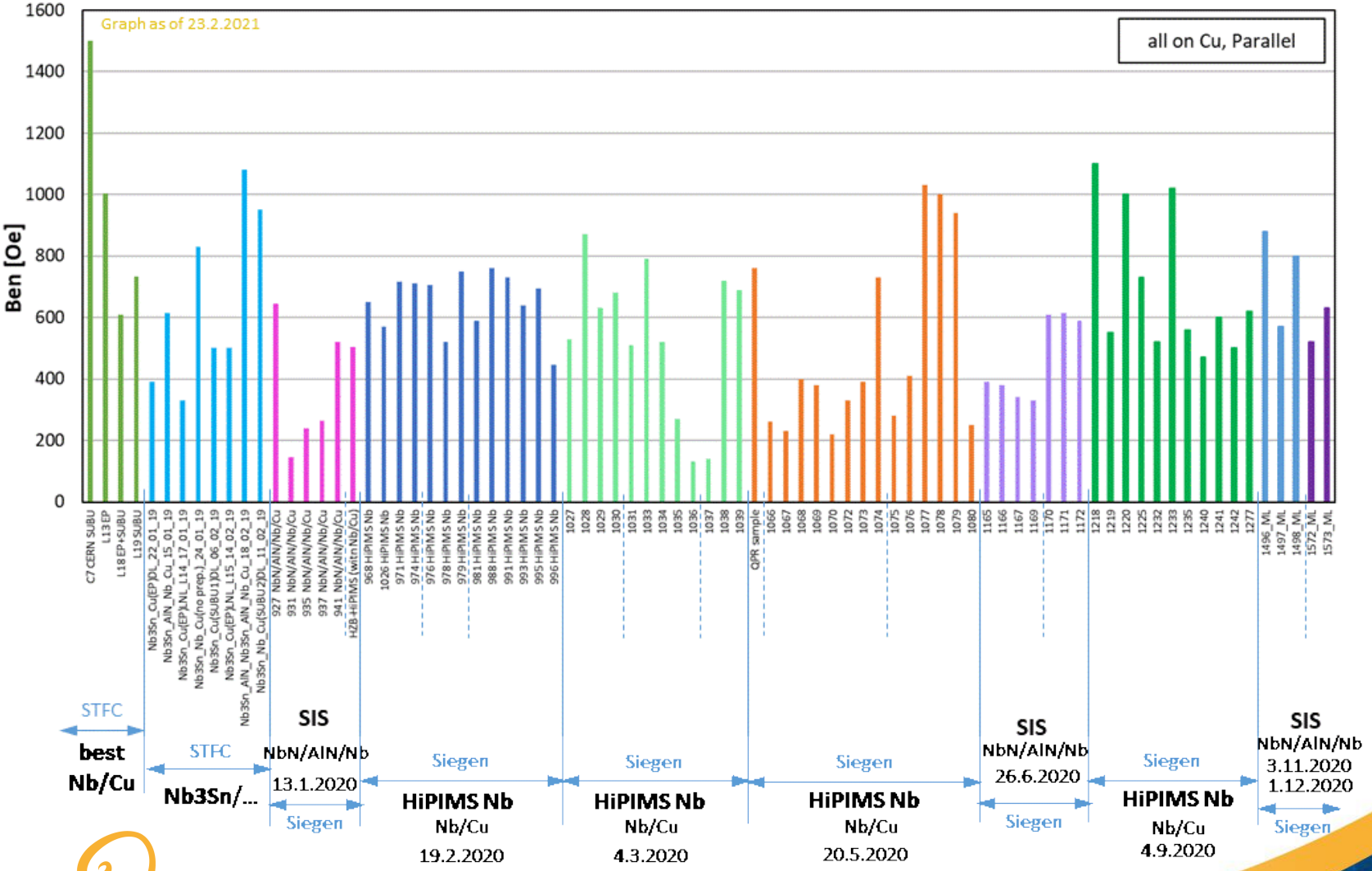


Courtesy of Eugen Seiler and Rastislav Ries (IEE)



Task 15.4. DC Superconductivity evaluation at IEE

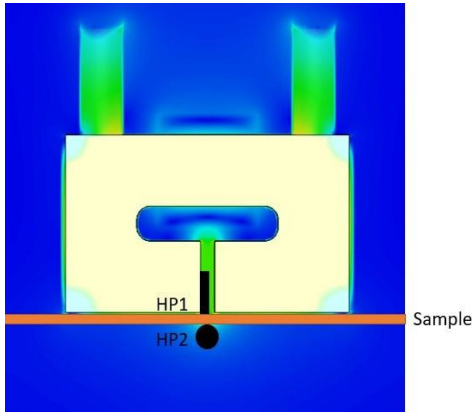
Selected films on Cu substrates:

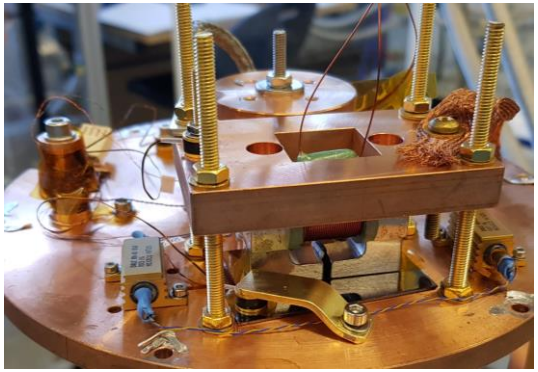


Courtesy of Eugen Seiler and Rastislav Ries (IEE)

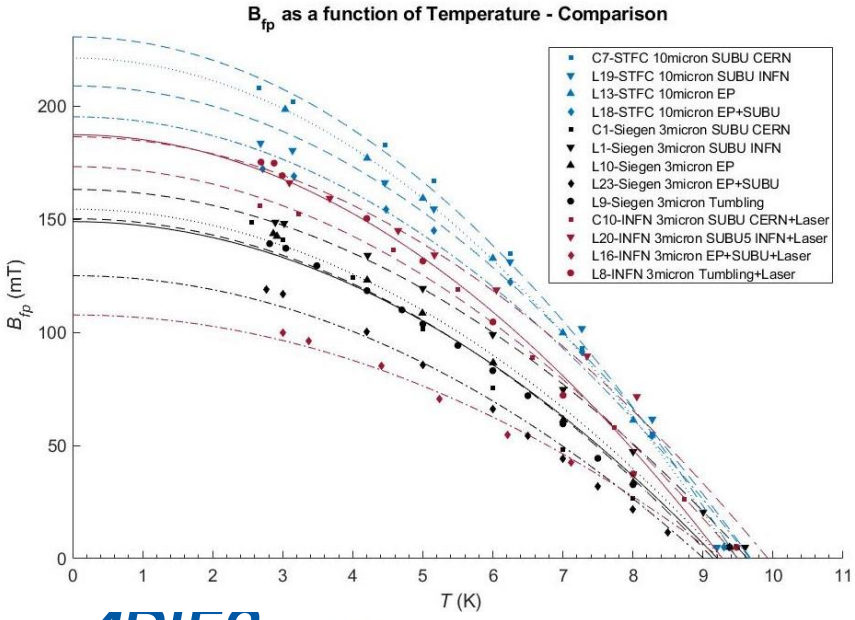
Task 15.4: AC/DC Superconductivity evaluation at STFC

- Magnetic field penetration facility for the planar samples
  - DC magnetic field parallel to the surface
  - Magnetic field applied from one side of the sample (similar to an SRF cavity)
  - Field local to the sample surface
    - Avoiding the edge effect.
    - Allows a possibility of sample scanning.
  - Applied and penetrated field measured by Hall probe sensors





Courtesy of D. Turner (LancU/STFC)

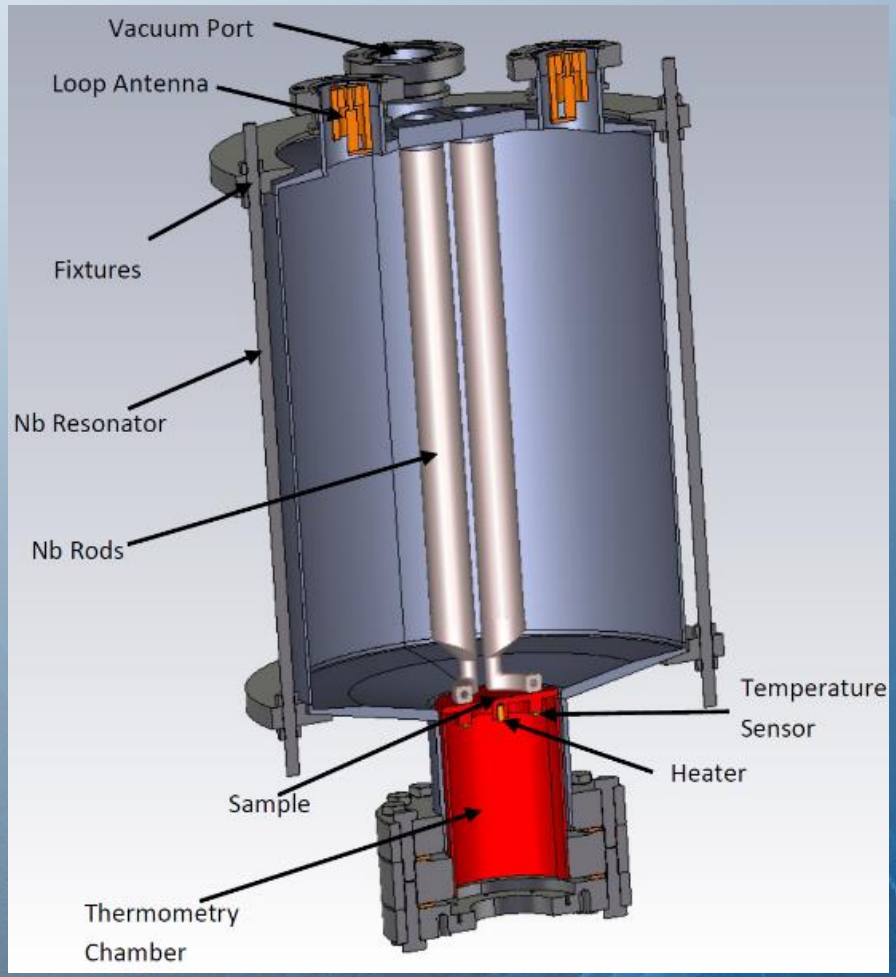


Results reported at IPAC and SRF conferences, the 9th International Workshops on Thin Films & New Ideas for Pushing the Limits of RF Superconductivity. More details in Deliverable Reports 15.4

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

ARIES

QPR samples for SRF test



Objectives

The main objective is
testing the deposited thin films
at the RF conditions,

this includes:

- Sample manufacturing (HZB) — Task 15.4
- Cases for sample transfer (HZB) — Task 15.4
- Sample polishing with EP and SUBU (INFN) — Task 15.2
- Nb film deposition (STFC, Siegen, INFN) — Task 15.3
  - Laser treatment (RTU) — Task 15.3
- SIS deposition (STFC, Siegen) — Task 15.3
- SRF testing of QPR samples (HZB, CERN) — Task 15.4

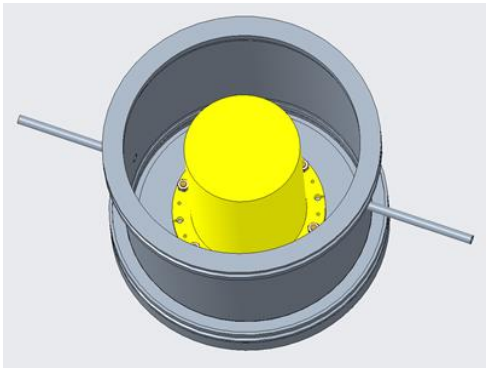
QPR samples

- QPR samples were designed at HZB, manufacured at Research Instruments and delivered in Year 2 (2018):
  - 5 OFHC/Nb samples
  - 5 Nb samples

Cu-Nb samples at HZB after production

Nb samples at HZB after production

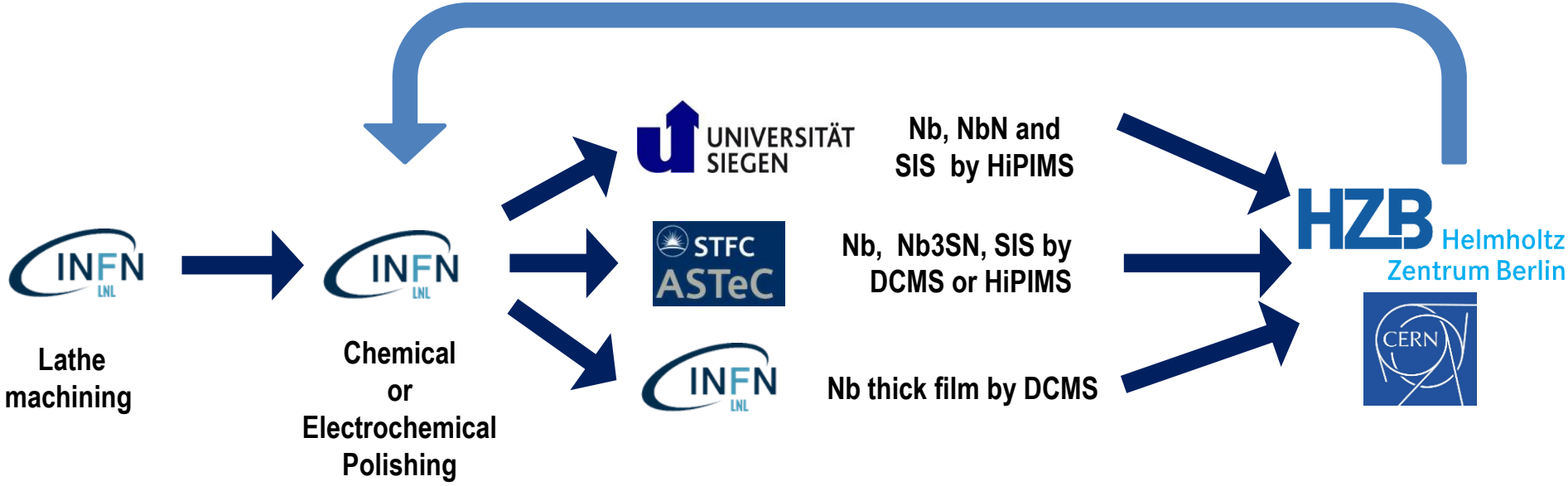
- A dedicated chamber for transporting the QPR samples under clean room conditions in vacuum or arbitrary atmospheres has been designed and manufactured:
  - It consists of ISO-KF160 standard pieces,
  - equipped with suitable fixtures for the samples,
  - and an evacuation/vent manifold.



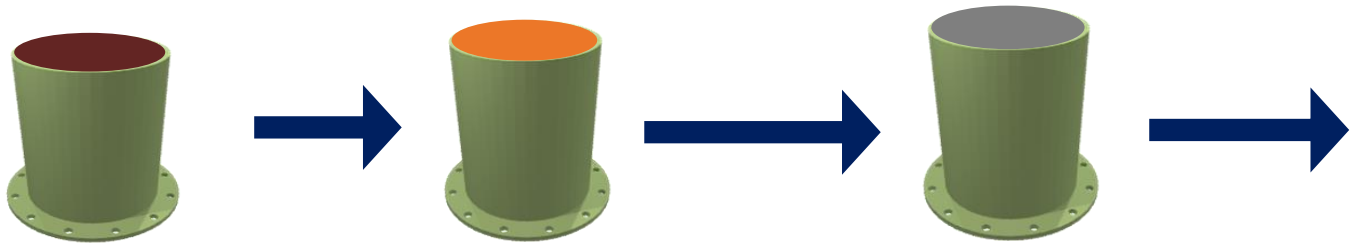
Sample transport chamber
Courtesy of Oliver Kugeler (HZB)

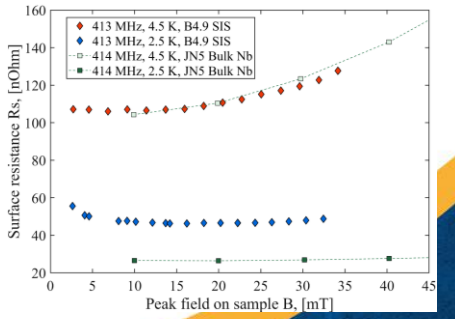
Workflow of the QPR Experiment

GOAL: Evaluate the effect of planar substrate Cu polishing on RF performance of QPR



MECHANICAL MACHINING → **POLISHING** → **COATING** → **RF TEST**

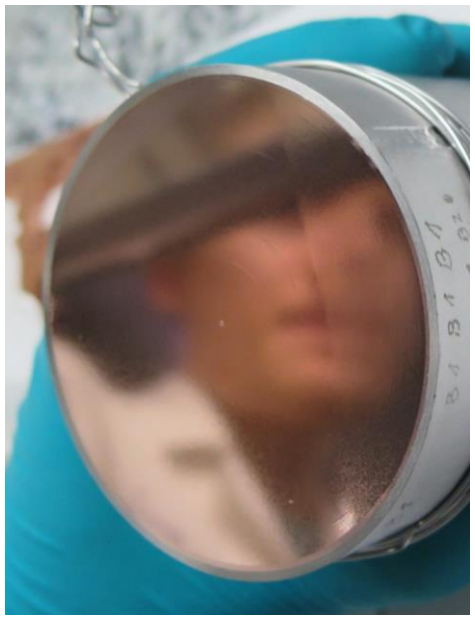


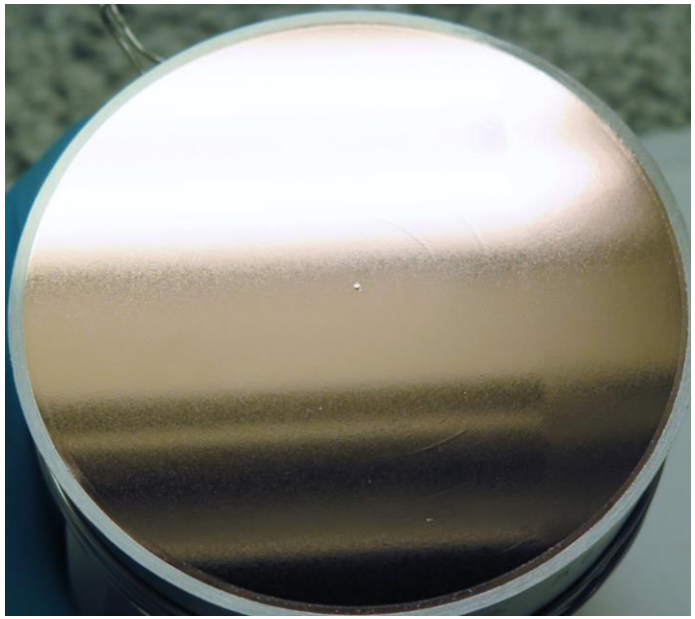


Courtesy of C. Pira (INFN) and E. Chyhyrynets

QPR substrate polishing at INFN

- SUBU5 (Chemical Polishing) -> 3 samples:
  - Sulfamic acid – 5 g/l, $(NH_4)_3Cit$ – 1 g/l, H_2O_2 – 50 ml/l, Butanol – 50 ml/l,
  - T = 73 °C
  - Average removal thickens: δ = ~6 μm
- Passivation (5 min):
  - Sulfamic acid – 20 g/l,
- EP (ElectroPolishing):
  - *Phosphoric acid 85% - 3 v.r., N-Butanol 98% - 2 v.r.,*
  - Temperature 40 °C
  - Average removal thickens: δ = ~15 μm
- Passivation:
  - Sulfamic acid – 20 g/l,



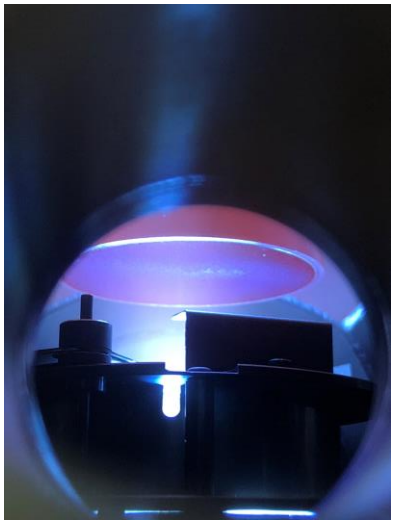


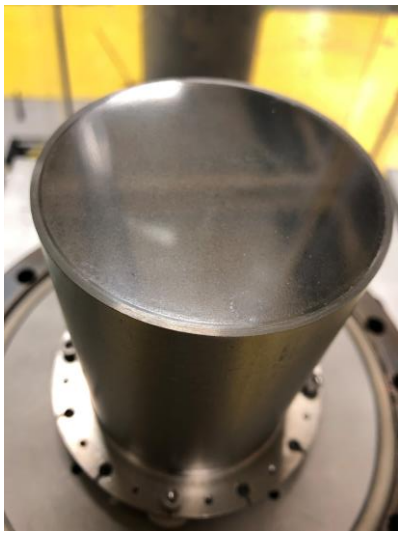
Courtesy of Eduard Chyhyrynets (INFN)

QPR deposition

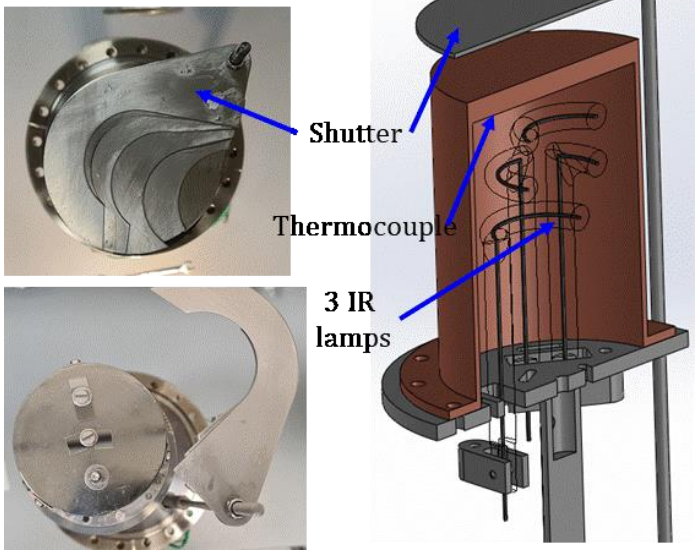
A QPR sample inside the deposition chamber at Siegen Uni. Courtesy of M. Vogel

A QPR sample (a) inside the deposition chamber and (b) after Nb film deposition at STFC/ASTeC. Courtesy of R. Valizadeh (STFC)





A QPR sample deposition facility at INFN/LNL. Courtesy of C. Pira



- **Seven QPR samples deposited with Nb at CERN, INFN, Siegen and STFC**
  - **One QPR- laser treated**
- **Three QPR samples with SIS at Siegen and STFC**

Task 15.4: RF Superconductivity evaluation with QPR

Samp							
B-1	INFN SUBU	3 µm Nb film	Cutting, Stripping, INFN **EP**	3 µm Nb film	**Laser treatment**	**Field Penetration**	
B-2	INFN SUBU	UNIVERSITÄT SIEGEN 2 µm Nb film	HZB RF test	CERN Recoat.: 7.3 µm Nb	CERN RF test	HZB RF test	Stripping, SUBU, INFN **Nb**
B-3	INFN EP	UNIVERSITÄT SIEGEN 3 µm Nb film	HZB RF test	SIS: 3 µm Nb/30 nm AlN/100 nm UNIVERSITÄT SIEGEN NbN	HZB RF test	Stripping, INFN SUBU	*TBD*
B-4	INFN EP	3 µm Nb film	HZB RF test	Stripping, INFN **EP**	UNIVERSITÄT SIEGEN **SIS**	HZB **RF test**	Stripping, INFN EP
B-5	INFN SUBU	3 µm Nb film	HZB RF test	RTU Laser treatment	HZB RF test	Stripping, INFN SUBU	**SIS**

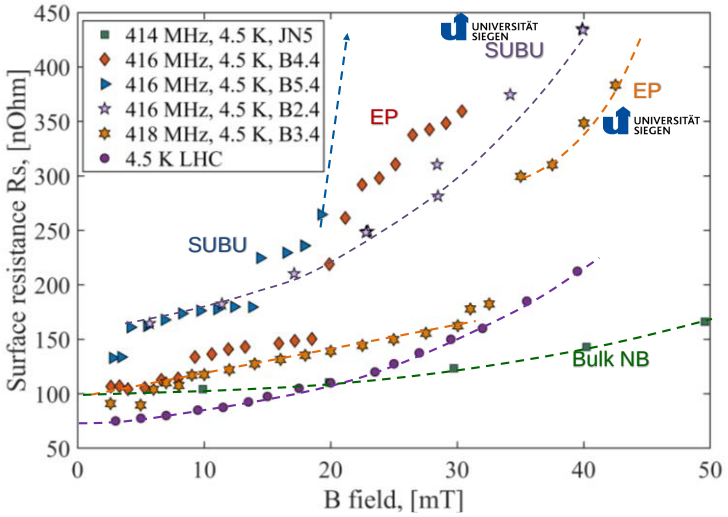
DONE **FAILED**

IN PROCESS **LASER**

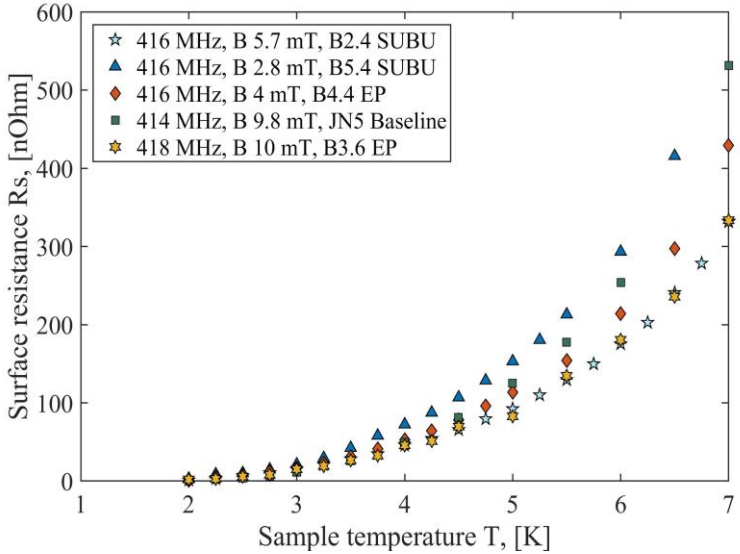
Courtesy of D. Tikhonov (HZB)

Nb coated QPR samples testing at HZB

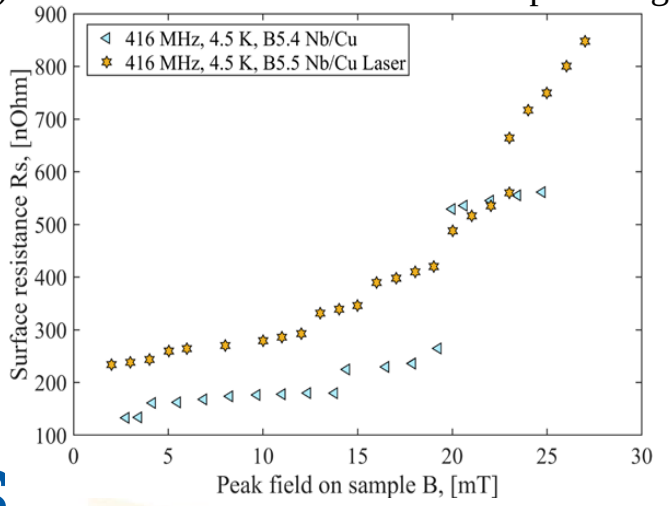
$R_s(B)$ at 415 MHz and T = 4.5 K



Measured BCS part of the surface resistance as a function of temperature at low field



$R_s(B)$ at 415 MHz and T = 4.5 K – laser polishing

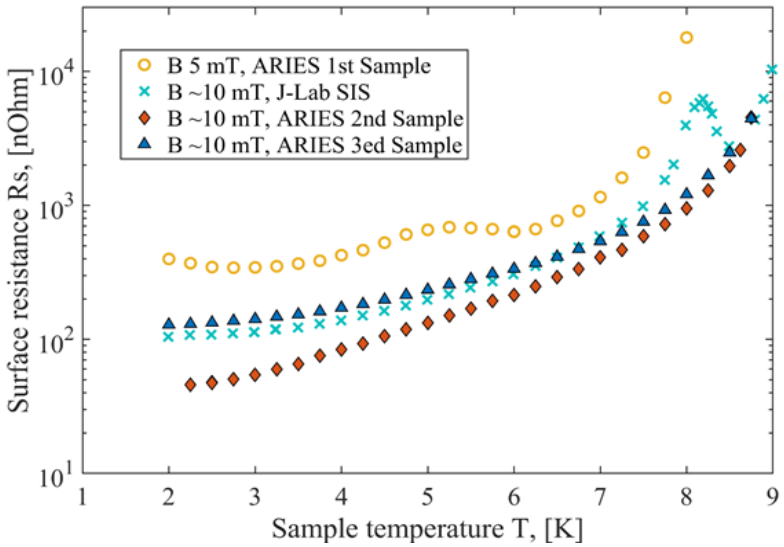


More details in Deliverable Reports 15.3 - 15.4 and future publications

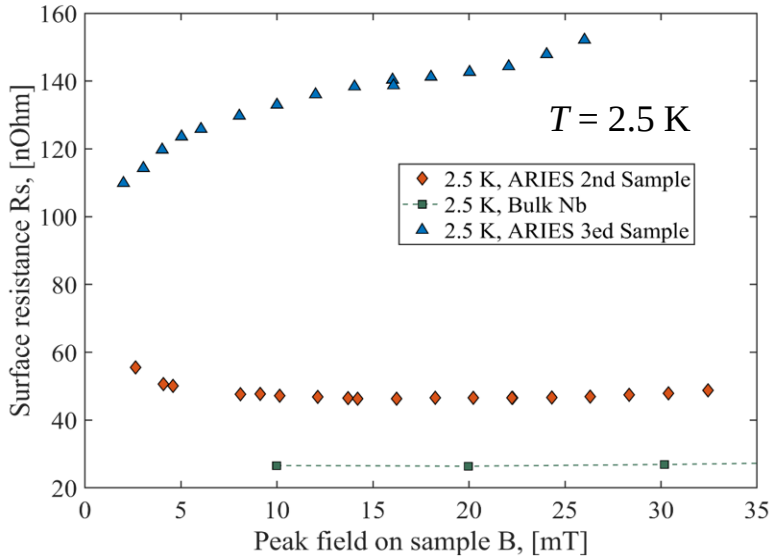
Courtesy of O. Kugeler and D. Tikhonov (HZB)

SIS coated QPR samples testing at HZB

	SIS films tested	Structure	Baseline	Production
JLab SIS	NbTiN – AlN – Nb(bulk)	75nm – 15 nm – bulk Nb	Yes	JLab, DCMS
ARIES 1st SIS	NbN – AlN – Nb(film)/Cu	197nm – 35 nm – 3 µm Nb	Yes	Siegen, DCMS
ARIES 2nd SIS	NbN – AlN – Nb(film)/Cu	180nm – 8 nm – 4 µm Nb	No	Siegen, HiPIMS
ARIES 3ed SIS	NbN – AlN – Nb(film)/Cu	180nm – 24 nm – 4 µm Nb	No	Siegen, HiPIMS



Measurement results for Q1 ~ 415 MHz



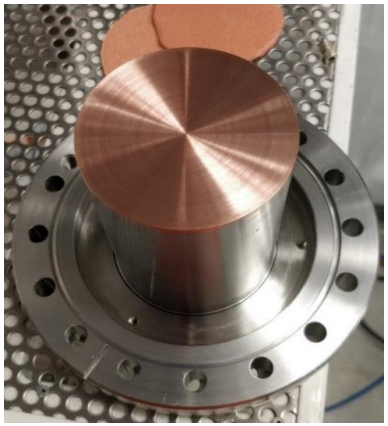
Courtesy of O. Kugeler and D. Tikhonov (HZB)

Samples from STFC and INFN still to be measured within 2 months and analysed. Results were presented at the 9th International Workshops on Thin Films & New Ideas for Pushing the Limits of RF Superconductivity and will be presented at IPAC'21 and SRF-2021 conferences,

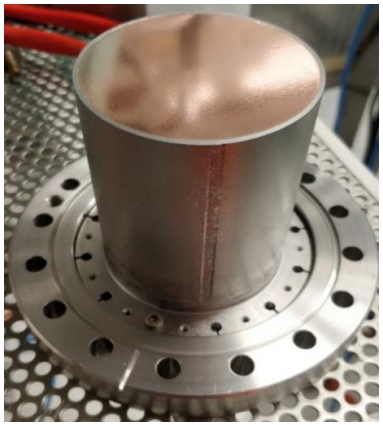
More details in Deliverable Reports 15.3 - 15.4

QPR testing at CERN

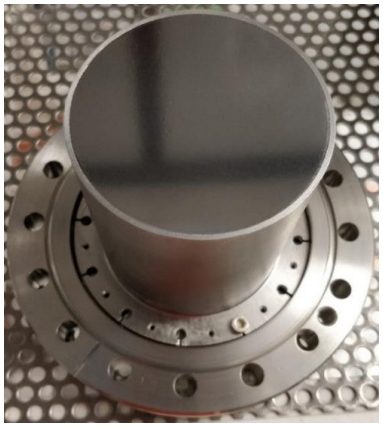
- Main purposes
  - To develop a QPR sample substrate that could be used at both CERN and HZB facilities
  - To compare the results of the same sample measured at both facilities
- *Work in progress*



QPR sample holder



QPR sample mounted



After coating

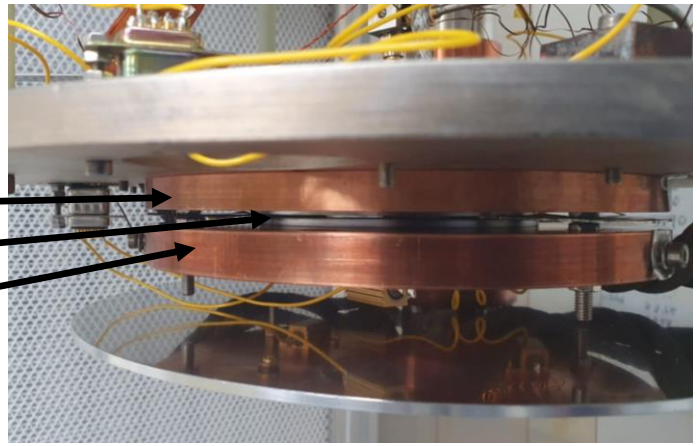
A happy ESR ☺

Courtesy of L. Vega Cid (CERN)

Task 15.4: RF Superconductivity evaluation (STFC)

- At **STFC** a radiofrequency (RF) cavity and cryostat dedicated to the measurement of superconducting coatings at 7.8 GHz has been updated to operation with a closed-cycle refrigerator.
- Low power measurements with an emphasis on fast turn-around time (~2 days for each sample).
- A cooldown demonstrated
  - T_{cavity} = 4.1 K and T_{sample} = 3.8 K.
- RF testing with a bulk Nb sample is in progress

Pill-box cavity in a new facility with a closed-cycle refrigerator in STFC



This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

Concluding

List of milestones and deliverables

Type	Delivery date	Lead beneficiary	Related task/ responsible	Description	Status
M1	m1	STFC	*Task 15.1*	1st WP meeting. Analysing outcome from EuCARD2 and current state of the field, finalising a detailed plan for WP	**Competed**
M2	m6	INFN	*Task 15.2*	First sample substrates cleaned at INFN for depositing at partners (Report to StCom)	**Completed**
D1	m12	INFN	*Task 15.2*	Evaluation of cleaning process. Report defining an optimum cleaning and polishing procedure for surface preparation for Cu and Nb substrate for Nb coating minimising the substrate effect on the final film properties.	**Completed**
M3	m14	STFC	Task 15.3, 15.4	First samples exchanged (system 1 and 2) and deposited at partners (Report to StCom)	**Completed**
D2	m24	STFC	Task 15.3, 15.4	Evaluation of systems 1 and 2. Report on deposition, surface and structural analysis, DC and RF superconductivity evaluation of systems 1 and 2 (e.g. NbN and Nb_3Sn) and Superconductor-Insulator-Superconductor (SIS) multilayer coating	**Completed**
M4	m26	HZB	Task 15.3, 15.4	First samples exchanged (system 3 and SIS) and deposited at partners (Report to StCom)	**Completed**
D3	M36	HZB	Task 15.3, 15.4	Evaluation of system 3. **Report** on deposition, surface and structural analysis, DC and RF superconductivity evaluation of system 3 (e.g. NbTiN or MgB_2) and SIS multilayer coating	**Completed**
D4	M48	STFC	Task 15.3, 15.4	**Final report** on thin film technology Report summarizing the results on the evaluation of cleaning and coating procedures for highest Q_0 and E_a	**Completed**

Conclusions

Key results: better understanding of a correlation between

- substrate preparation,
- deposition process, thin film characteristic,
- the DC and AC SC evaluation results and
- the TF behaviour at the RF conditions

- Five polishing techniques for Cu have been tested with Nb films
  - SUBU5 and EP demonstrate best results
- Development of non-Nb superconducting films on small samples:
  - NbN, Nb_3Sn, NbTiN films as well as SIS structures deposited and characterised,
  - Small sample evaluation on SC properties with VSM and MFPF;

- Evaluation of Nb films at the RF conditions:
  - New QPR sample design, transport case,
  - QRR sample polishing developed and applied to the samples at INFN,
  - Deposition of 10 QPR samples
    - at INFN, Siegen and STFC
    - *with Nb and SIS: Nb/AlN/NbN and Nb/AlN/Nb_3Sn*,
  - First QPR results for SIS structures,
  - Comparative testing of QPR facilities at CERN and HZB with the samples produced by WP15 team;
- Developing of new technologies:
  - Laser treatment of Cu substrate and Nb films,
  - Magnetic field penetration facility (MFPF);
- Collaborating, involving new partners, enhancing a capability of every partner, frequent discussions, joint publications.

Main result:

enabling progressing to the next stage: developing a real cavity prototype coated with non-Nb superconducting TF and SIS structures within I.FAST WP9

ARIES WP15 publications:

[1] Pira, C., et al., "ARIES Deliverable Report 15.1". 2018.

[2] Malyshev, O., et al., "ARIES Deliverable Report 15.2". 2019.

[3] Kugeler, O., et al., "ARIES Deliverable Report 15.3". 2020.

[4] Malyshev, O., et al., "ARIES Deliverable Report 15.4". 2021.

[6] Pira, C., et al., "Impact of the Cu substrate surface preparation on the morphological, superconductive and RF properties of the Nb superconductive coatings", in Proc. SRF 2019, Dresden, Germany, 2019.

[6] Turner, D., et al., "Characterization of flat multilayer thin film superconductors", in Proc. SRF 2019, Dresden, Germany, 2019.

[7] Malyshev, O.B., et al. The SRF thin film test facility in LHe-free cryostat. in Proc. SRF 2019, Dresden, Germany, 2019.

[8] Tikhonov, D., et al., "Superconducting thin films characterization at HZB with the quadrupole resonator", in Proc. SRF 2019, Dresden, Germany, 2019.

[9] Leith, S.B., et al., "Initial results from investigations into different surface preparation techniques of OFHC copper for SRF applications", in Proc. SRF 2019, Dresden, Germany, 2019.

[10] Leith, S., et al., "Deposition parameter effects on niobium nitride (NbN) thin film deposited onto copper substrates with DC magnetron sputtering", in Proc. SRF 2019, Dresden, Germany, 2019.

[11] Valizadeh, R., et al., "PVD deposition of Nb3Sn thin film on copper substrate from an alloy Nb3Sn target", in Proc. IPAC 2019. 2019: Melbourne, Australia.

[12] Ries R., et al, “Superconducting properties and surface roughness of thin Nb samples fabricated for SRF applications”, J. Phys.: Conf. Ser. 1559, 012040 (2020).

[13] Ries R., eta al, “Improvement of the first flux entry field by laser post-treatment of the thin Nb film on Cu”, Supercond. Sci. Technol., accepted for publication (2021).

[14] Keckert, S., "Advanced Radio-Frequency Characterization of Thin-Film Superconducting Samples", in Naturwissenschaftlich-Technische Fakultät. Universität Siegen: Siegen, 2019.

[15] Kleindienst, R., "Radio Frequency Characterization of Superconductors for Particle Accelerators", in Naturwissenschaftlich-Technische Fakultät., Universität Siegen, 2017

More results will be published in scientific journals and proceedings of the IPAC’21 and SRF-2021 conferences.

ARIES WP15 team:

- CERN:
  - A. Sublet, L. Vega Cid, G. Rosaz, P. Vidal, W. Venturini, G. Vandoni and M. Taborelli
- IEE:
  - E. Seiler, R. Ries and F. Gömöry
- INFN
  - V. Palmieri, C. Pira, E. Chyhyrynets, F. Stivanello, O. Azzolini and G. Keppel
- HZB:
  - O. Kugeler, S. Keckert, J. Köszegi and D. Tikhonov
- CEA:
  - C. Antoine
- RTU
  - A. Medvids, P. Onufrievs and A. Katashev
- Siegen University
  - M. Vogel, S. Leith and Ö. Sezgin
- STFC/CI:
  - O. Malyshev, R. Valizadeh, F. Lockwood Estrin, F. Walk, D. Turner, D. Seal, T. Sean, F. Goudket and G. Burt

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