



# Main highlights ARIES WP15 team collaboration on Thin Film for Superconducting RF (TF-SRF)

Oleg Malyshev
ARIES WP15 coordinator
on behalf of the WP15 team

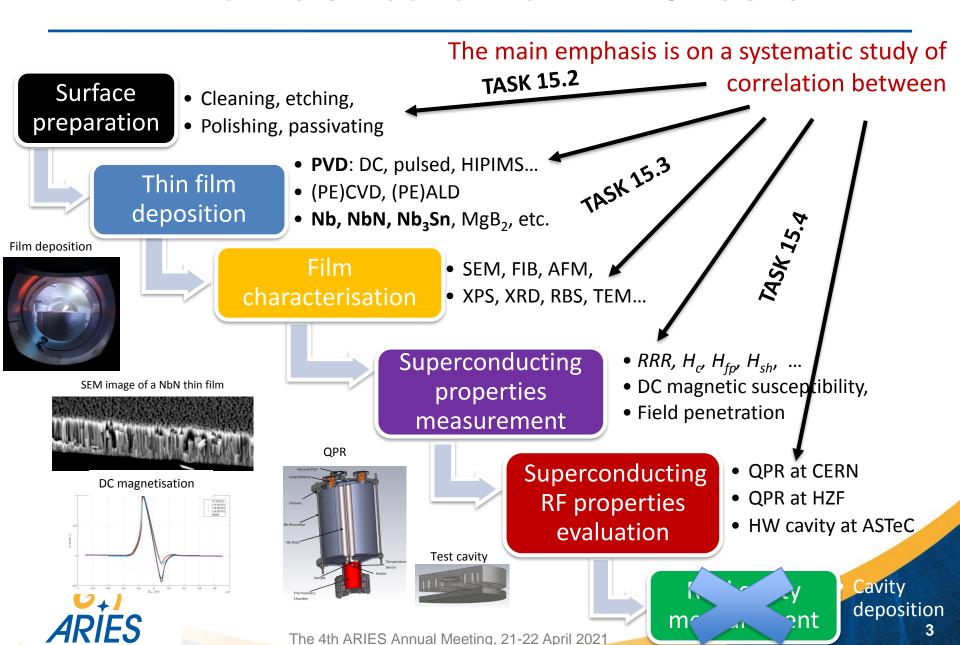
The 4th ARIES Annual Meeting, 21-22 April 2021

#### WP15 Description

- The aim of this work package was 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,
  - o deposition parameters for:
    - > superconducting material Nb, NbN, Nb<sub>3</sub>Sn, NbTiN and SIS
    - deposited on Cu substrate,
  - o film structure, morphology, chemistry, phase,
  - O AC and DC superconductivity parameters:
    - $\triangleright$  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



#### **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, <b>2</b> and <b>3</b>
5	Helmholtz-Zentrum Berlin (Berlin, Germany)  RTU  RETU  RETU	Task 4	Tasks 1 and 4
6	RTU (Riga, Latvia)		Task 2 and 3
7	University Siegen, (Siegen, Germany) UNIVERSITÄT SIEGEN		Tasks 3
8	ASTeC/STFC (Daresbury, UK)  STFC  ASTeC	WP and Tasks 3	All tasks
9	Lancaster University (Lancaster, UK)  Lancaster University		Task 4







Superconducting Thin Film Development

**Task 15.3** 

### **Objectives**

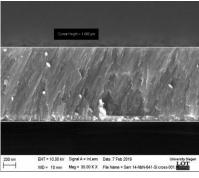
#### Thin film development on small samples (53 mm × 53 mm)

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



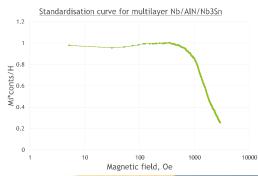
#### Nb film deposition (STFC, Siegen, INFN)

- Laser treatment of the film (RTU)
- NbN, Nb<sub>3</sub>Sn, NbTiN and SIS deposition (STFC, Siegen)
- Film characterisation (STFC, Siegen, INFN, RTU)



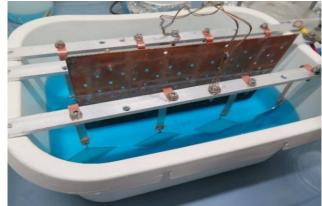
DC and AC superconductivity evaluation measurements (CEA, IEE, STFC)





#### 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 1<sup>st</sup> and 2<sup>nd</sup> 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)



#### Task 15.3. Thin film deposition

- Thin film deposition facilities are key facilities for the project
- The 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<sub>3</sub>Sn, 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.

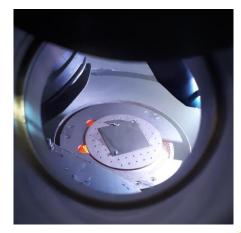
Courtesy of M. Vogel (Siegen)

LNL/INFN



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

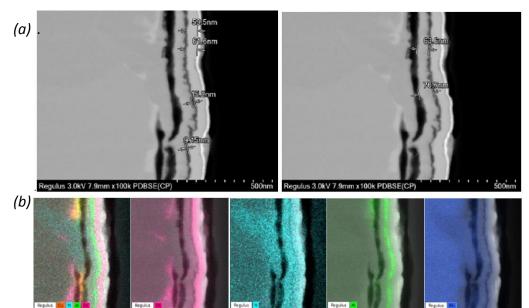
ASTeC/STFC



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

#### Task 15.3. Thin film characterisation

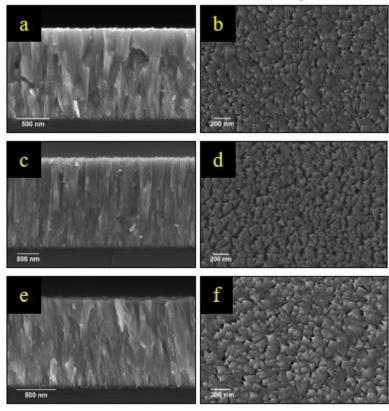
#### At STFC



- (a) High resolution SEM of ion milled X-section of SIS multilayer structure (Nb/AIN/Nb3Sn) deposited on Ta.
- (b) EDX chemical mapping of the X-section.

Courtesy of R. Valizadeh (STFC)

#### At University Siegen



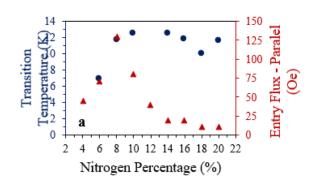
Cross sectional SEM images of NbN

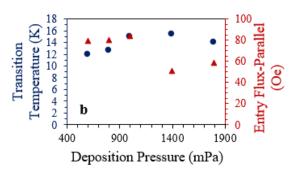
Courtesy of M. Vogel (Siegen)



# System 2: NbN thin films (University of Siegen)

	On Cu	Litera ture
$T_c[K]$	15.5	16



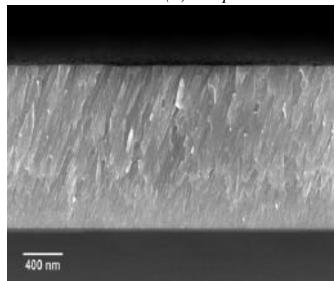


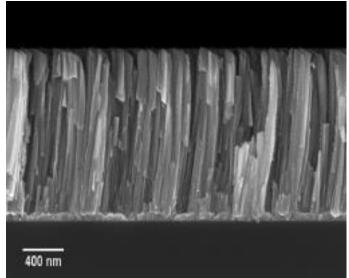
X-section SEM for NbN deposited at different pressure

(a) low pressure

and

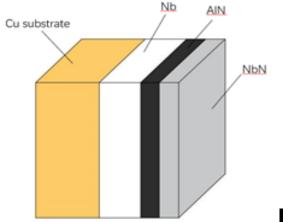
(b) high pressure





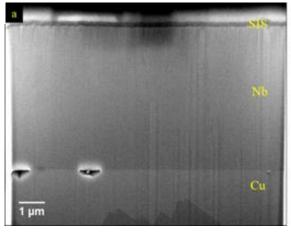


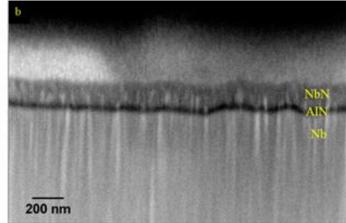
#### NbN based SIS structures (University of Siegen)



DCMS deposited SIS film structure

Nb – 4.5  $\mu$ m AlN – 10-30 nm NbN – 150-250 nm



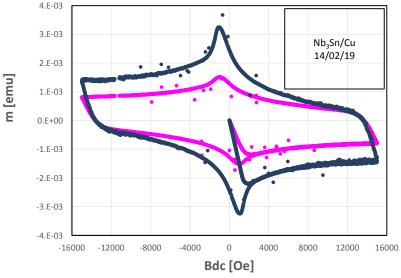


SEM images detailing a) FIB cut through full thickness of sample 927. b) Zoomed in image of SIS structure of sample 927.

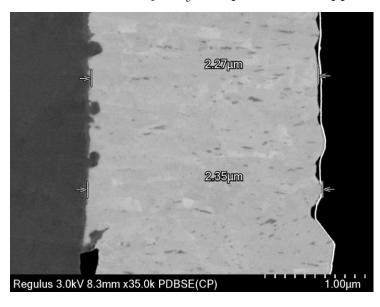


# System 1: Nb<sub>3</sub>Sn thin films (STFC)

	On Cu	On Sapphire	Literatu re
$T_c$ [K]	17.5	17.75	18.3
$B_{en}$ [mT]	90	140	



*X-section SEM of Nb*<sub>3</sub>*Sn deposited on copper* 



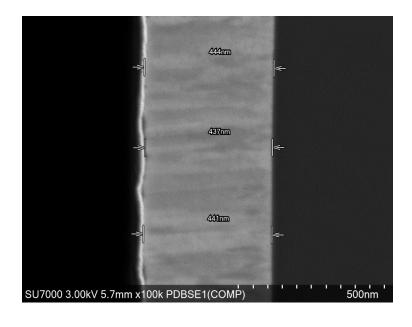
Magnetisation loop for Nb<sub>3</sub>Sn deposited on copper



## System 3: NbTiN thin films (STFC)

	On Cu	On Sapphire	Literatu re
$T_c$ [K]	17	17	16-16.6
$B_{en}$ [mT]	57	25	

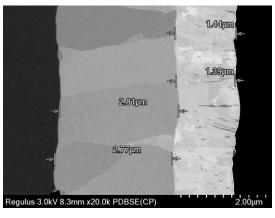
#### X-section SEM of Nb<sub>3</sub>Sn deposited on copper



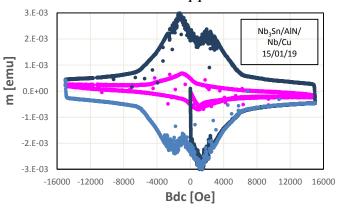


#### Towards S(I)S structures (STFC)

X-section SEM of Nb<sub>3</sub>Sn with a Nb underlayer as double structure on copper

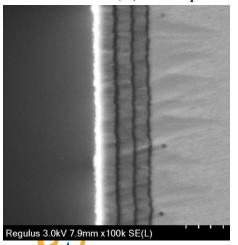


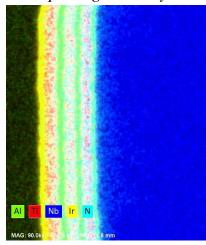
Magnetisation loop for Nb<sub>3</sub>Sn with a Nb underlayer as double structure on copper

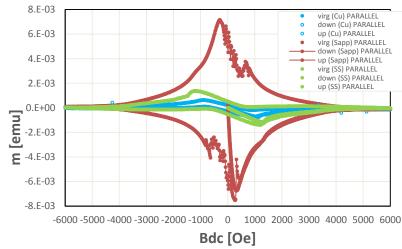


(a) X-section SEM of a triple multilayer NbTiN in SIS structure,

(b) corresponding EDS depicting each layer composition, (c) the DC magnetisation loop at  $T=4.22~\mathrm{K}$ .







#### Task 15.3. Thin film development

- The most recent results have been reported in 6 talks at the 9<sup>th</sup>
  International Workshop on Thin Films & New Ideas for Pushing the
  Limits of RF Superconductivity, 15-18 March 2021:
  - Cristian Pira (INFN/LNL), "Nb<sub>3</sub>Sn films via liquid tin diffusion for SRF application"
  - Felix Walk (STFC/CI), "Bipolar HiPIMS: Correlating plasma parameters to thin film properties"
  - Francis Lockwood Estrin (STFC/CI), "Using HiPIMS for V<sub>3</sub>Si Superconducting Thin Films Nearly all"
  - Reza Valizadeh (STFC/CI), "Synthesis of Alternative Superconducting Film to Nb for SRF Cavity"
  - Stewart Leith (Siegen Uni), "The Development of HiPIMS Multilayer SIS film coatings on Copper for SRF Applications"
  - Özdem Sezgin (Siegen Uni), "NbN Thin Film-Based Multilayer (S-(I)-S) Structures for SRF Cavities"
- The further progress with these activities will be reported in at the SRF-2021 conference, 28<sup>th</sup> June – 2<sup>nd</sup> July 2021

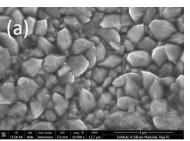


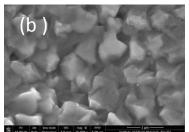
More details in Deliverable Reports 15.1 - 15.4

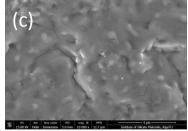
#### Task 15.3. Laser treatment Nb films at RTU

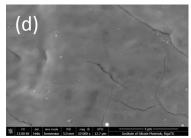
#### Aims:

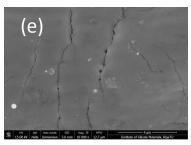
- Increase the grain size of Nb;
- Increase the adhesion of Nb layer to Cu substrate (annealing the defects by laser radiation);
- Improve superconducting properties.











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



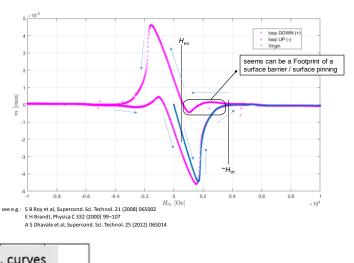
Results reported in 2 papers and at the 9<sup>th</sup> International Workshop on Thin Films & New Ideas for Pushing the Limits of RF Superconductivity, 15-18 March 2021. More details in Deliverable Reports 15.3 & 15.4

#### Task 15.4. DC Superconductivity evaluation at IEE

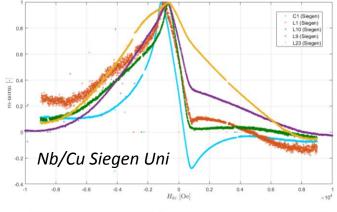
PPMS (Physical Property Measurement System)

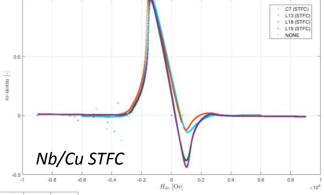
Virgin DC magnetisation curve: B<sub>en</sub>(~B<sub>c1 perp</sub>), [B<sub>p</sub>, B<sub>c2</sub>]

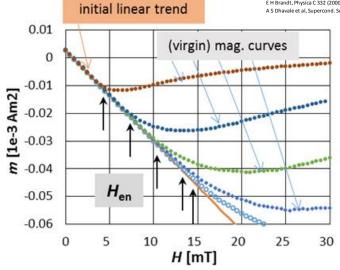
These results are an essential part in most of WP15 publications and reports

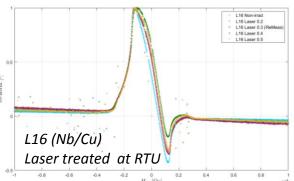


Shape of magnetization loop









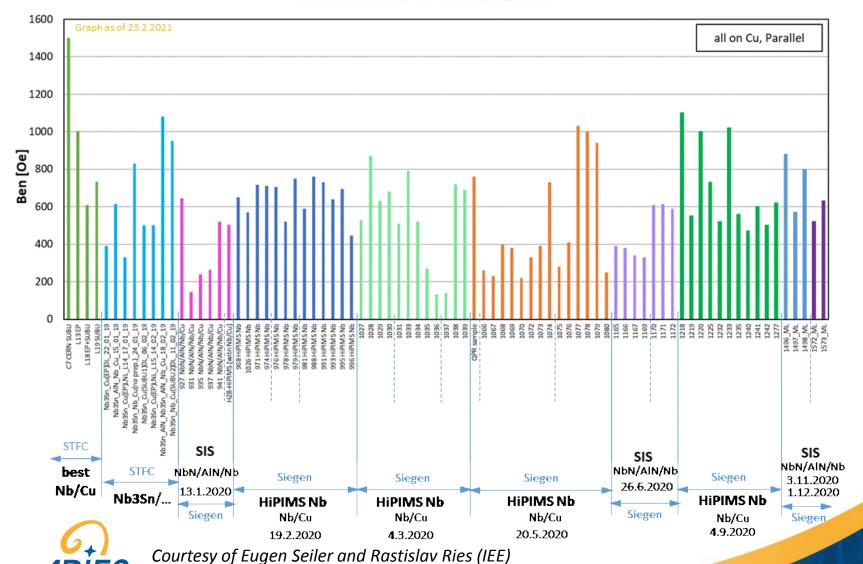
Courtesy of Eugen Seiler and Rastislav Ries (IEE)

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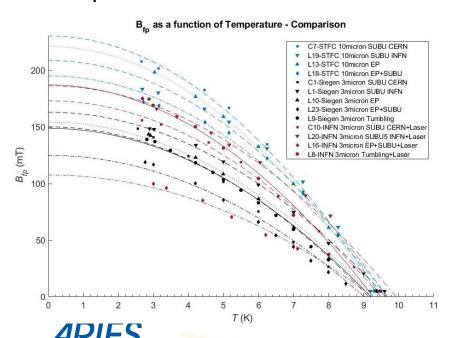
## Task 15.4. DC Superconductivity evaluation at IEE

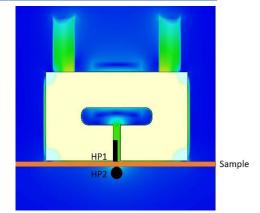
#### Selected films on Cu substrates:

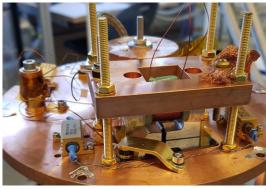


#### Task 15.4: AC/DC Superconductivity evaluation at STFC

- 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







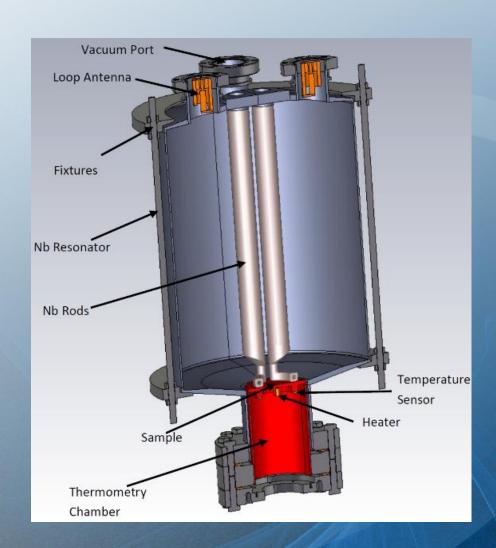
Courtesy of D. Turner (LancU/STFC)

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





QPR samples for SRF test



#### **Objectives**

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

#### this includes:

<ul> <li>Sample manufacturing (HZB)</li> </ul>	Task 15.4
<ul> <li>Cases for sample transfer (HZB)</li> </ul>	Task 15.2
<ul> <li>Sample polishing with EP and SUBU (INFN)</li> </ul>	
<ul> <li>Nb film deposition (STFC, Siegen, INFN)</li> </ul>	Task 15.3
<ul><li>Laser treatment (RTU)</li></ul>	Task 15.4
<ul> <li>SIS deposition (STFC, Siegen)</li> </ul>	103K 1314



SRF testing of QPR samples (HZB, CERN)

#### **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
- 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.



Cu-Nb samples at HZB after production



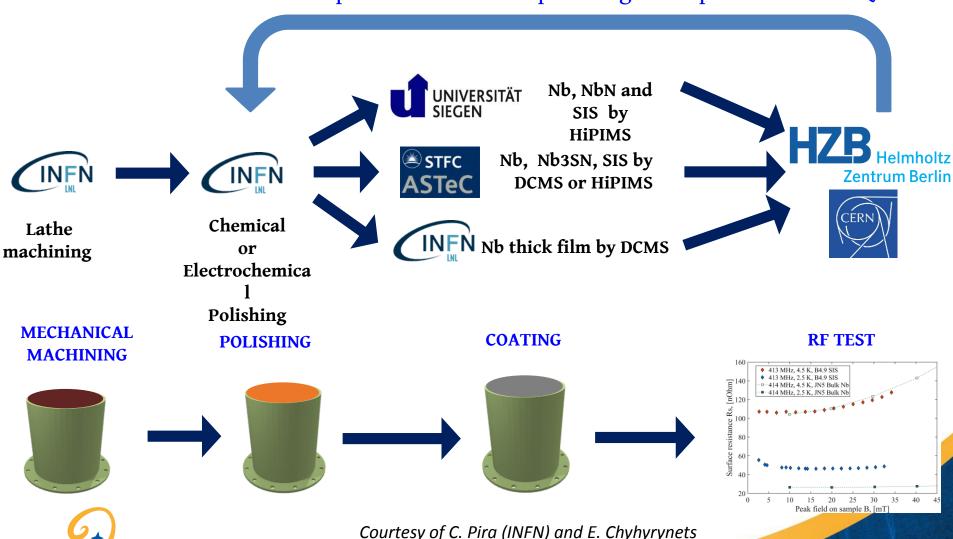
Nb samples at HZB after production

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



#### QPR substrate polishing at INFN

- SUBU5 (Chemical Polishing) -> 3 samples:
  - Sulfamic acid 5 g/l, (NH<sub>4</sub>)<sub>3</sub>Cit 1 g/l,
     H<sub>2</sub>O<sub>2</sub> 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)

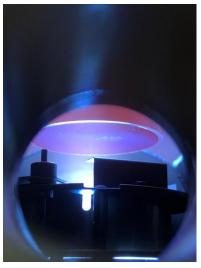


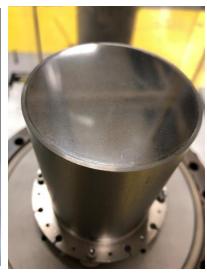
#### QPR sample deposition



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

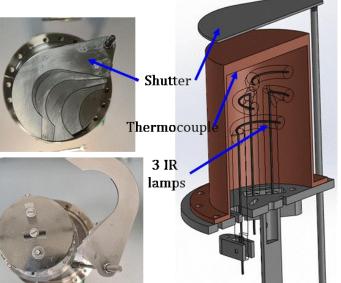
A QPR sample (a) inside the deposition chamber and (b) after Nb film deposition at STFC/ASTeC. Courtesy of R. Valizadeh (STFC)





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



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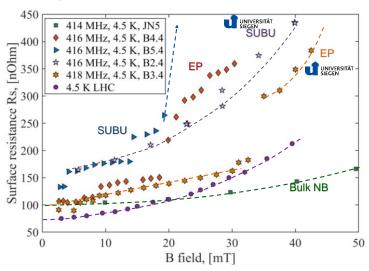
#### Task 15.4: RF Superconductivity evaluation with QPR

Samı							
B-1	SUBU	3 μ Nb fil	Strinning	Nh film	Laser treatment	Field Penetration	
B-2	SUBU	UNIVERSITÄT 2 μ SIEGEN Nb fil		Recoat.: 7.3 μm Nb	RF test	HZB RF test	Stripping, SUBU, INFN Nb
B-3	<b>EP</b>	UNIVERSITÄT 3 μ SIEGEN Nb fil	RE IEVI	SIS: 3 µm Nb/30 nm AlN/100 nm UNIVERSITÄT NbN SIEGEN	HZB RF test	Stripping, SUBU	TBD
B-4	<b>EP</b>	3 μ Nb fil	RE IDSI	Stripping, EP	UNIVERSITÄT SIEGEN SIS	HZB RF test	Stripping, EP
B-5	SUBU	3 μ Nb fil	RE IEST	Laser treatment	HZB RF test	Stripping, SUBU	SIS
	DON IN PE	E ROCESS	FAILED LASER		Courtesy o	f D. Tikhonov	(HZB)

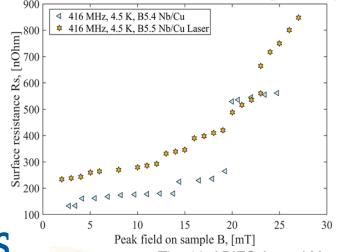


# Nb coated QPR samples testing at HZB

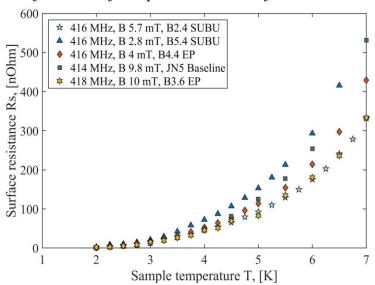




 $R_s(B)$  at 415 MHz and T = 4.5 K - laser polishing



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

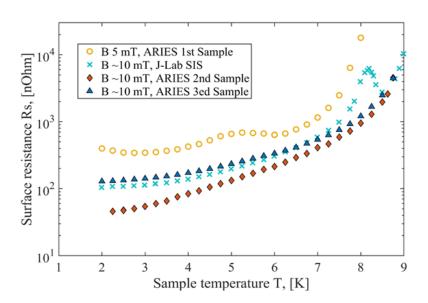


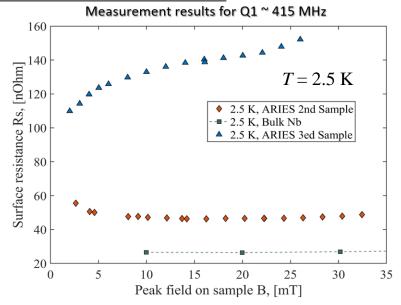
More details in Deliverable Reports 15.3 - 15.4 and future publications

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 – <mark>35 nm</mark> – 3 μm Nb	Yes	Siegen, DCMS
ARIES <b>2nd</b> SIS	NbN – AlN – Nb(film)/Cu	180nm – <b>8 nm</b> – 4 μm Nb	No	Siegen, HiPIMS
ARIES <b>3ed</b> SIS	NbN – AlN – Nb(film)/Cu	180nm – <b>24 nm</b> – 4 μm Nb	No	Siegen, HiPIMS





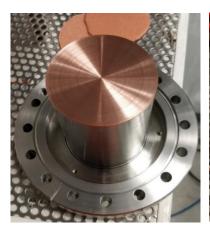
Courtesy of O. Kugeler

Samples from STFC and INFN still to be measured within 2 months and analysed. and D. Tikhonov (HZB)
Results were presented at the 9<sup>th</sup> 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,

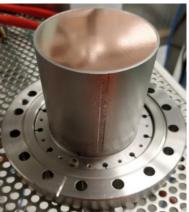
More details in Deliverable Reports 15.3 - 15.4

#### 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



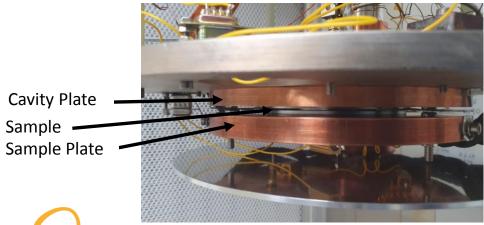
A happy ESR



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
  - $T_{cavity} = 4.1 \text{ K} \text{ and } T_{sample} = 3.8 \text{ K}.$
- RF testing with a bulk Nb sample is in progress





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



# Future: I.FAST WP9: Innovative superconducting cavities (01/05/2021-30/04/2025)

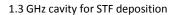
Surface preparation

- Cleaning, etching,
- Polishing, passivating

• PVD: DC, pulsed, HIPIMS...

- (PE)CVD, (PE)ALD
- Nb, NbN, Nb<sub>3</sub>Sn, MgB<sub>2</sub>, SIS, etc.
  - Nb film laser treatment

The main emphasis is on applying the result of ARIES to RF cavity deposition and testing

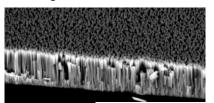




Film deposition



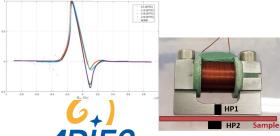
SEM image of a NbN thin film



DC magnetisation

Thin film

deposition

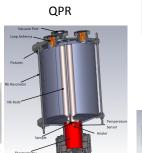


Film characterisation

- SEM, FIB, AFM,
- XPS, XRD, RBS, TEM...

Superconducting properties measurement

- RRR, H<sub>c</sub>, H<sub>fp</sub>, H<sub>sh</sub>, ...
- DC magnetic susceptibility,
- Field penetration



Superconducting RF properties evaluation

- QPR at CERN
- QPR at HZF
- HW cavity at ASTeC



Real cavity measurement

Cavity deposition

#### 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<sub>3</sub>Sn, 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/AIN/NbN and Nb/AIN/Nb<sub>3</sub>Sn,
- 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 reported at the IPAC'21 and SRF-2021 conferences within this year.

#### ARIES WP15 team:

- CERN:
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# Thanks for your attention

