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ABSTRACT

The ARIES WP15 program deals with the optimization of superconducting films for the use in accelerating structures. In the present approach, all production steps necessary to arrive at a successful coating are revisited, from substrate preparation to sample handling to the actual film deposition. These steps are performed on wafer samples, and in an intermediate step, prior to doing an actual coating on cavities, on QPR-samples that allow for a characterisation under real accelerator-like conditions. In continuation of the previously reported results, sample manufacturing and DC-characterisation has progressed towards two additional systems: NbN and SIS. The procedures leading to an optimized sample are described in detail. Characterisation of the samples with standard materials characterisation methods as well as DC-magnetometry has been performed and is reported. RF-characterisation was performed on baseline Nb QPR-samples, comparing the coating results between the facilities at STFC and USI and the impact of the substrate preparation technique on the quality of the obtained film.

For more information on ARIES, its partners and contributors please see http://aries.web.cern.ch

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Delivery Slip

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TABLE OF CONTENTS

Executive summary

This report is summarizing the main achievement of the WP15 program (*the results on the evaluation of cleaning and coating procedures for highest Q_0 and E_a*)

- sample preparation, surface polishing
- Nb thin film deposition and characterisation
- NbN, Nb3Sn and NbTiN samples
- SIS multilayer structures NbN with AlN as an insulator
- DC-superconductivity evaluation
- RF testing with QPR

1. Introduction

Over four year a WP15 team followed the scope and the roadmap of WP15 programme which has been set before the ARIES project starting date. All activities of WP15 are connected, cross-correlated and integrated in one international project. The D15.1 Delivery Report summarised the WP15 progress with Evaluation of copper substrate cleaning and polishing process. Two procedure for surface preparation of Cu for Nb coating was chosen for the following WP15 activities base on various types of characterisation of copper surface and Nb film deposited on it [1]. The following activities were focused on developing the thin films (TF) based on superconducting (SC) materials different from pure Nb. The choice of materials was discussed in details in the D15.1 and D15.2 Deliverable Reports [2, 3] and based on literature survey [4-8]. The developing and evaluation of Systems 1 (Nb_3Sn) and System 2 (NbN) has been reported describing the details of deposition, surface analysis, structural analysis, and DC superconductivity evaluation. The Report D15.3 is related to the development of System 3 (NbTiN) and SIS (superconductor-insulator-superconductor) multilayer coatings [Report D15.3].

This report is a final report on WP15 programme and highlighting the main results achieved within for year. A considerable fraction of the work summarized here has been presented at various journals or conferences, see [9-15] *(Check if all papers are included including SRF2021 and IPAC2021 conferences)*.

Another significant part of the WP15 activities is focused on evaluation of SC TF at the conditions similar to ones in accelerator radio-frequency (RF) resonators. The Quadrupole Resonator (QPR) developed in earlier H2020 projects is employed for this activity [16]. Some results have been reported in D15.3 [Report D15.3]. This Report describes new QPR results obtained at the RF conditions at HZB and CERN.

2. Polishing of copper substrate before SC thin film coating on samples and an impact on the Nb film SC properties (5-7 pages)

The

2.1 POLISHING TECHNIQUES EMPLOYED IN THIS PROJECT

2.1.1 At CERN

2.1.2 At INFN

2.1.3 At RTU

The

2.1.4 Main observations

2.2 NB COATING

2.2.1 At INFN

2.2.2 At Siegen

2.2.3 At STFC

Niobium

2.3 MAGNETISATION MEASUREMENTS

2.3.1 At IEE

2.3.2 At STFC

2.4 Summary on polishing

3. Developing of SC thin film coating different from Nb on copper substrates (5-7 pages)

The consortium continues developing coatings with thin film of superconducting materials different from Nb, such as Nb_3Sn (System 1), NbN (System 2), NbTiN (System 3) as well as SIS multilayer structures. The deposition performed on the samples prepared in Year 1: flat OHFC samples with dimensions 53 mm × 53 mm × 2 mm. The samples are polished with either SUBU or EP, two polishing procedures defined as most promising in Year 1 [2]. The Report D15.2 [3] reported on Systems 1 (Nb_3Sn) and System 2 (NbN) describing the details of deposition, surface analysis, structural analysis, and DC superconductivity evaluation. The Report D15.3 [?] is related to the development of System 3 (NbTiN) and SIS (superconductor-insulator-superconductor). Below t main achievement on SC TF are summarised.

3.1 Deposition At STFC

3.1.1 System 2: Nb_3Sn thin films

achieved.

3.1.2 System 3: NbTiN thin films

achieved.

3.1.3 Deposition with TiNb alloy target:

NbTi and NbTiN were deposited from a NbTi alloy target on various diamond turned copper and various witness substrate at RT and 650 °C with deposition parameters

3.1.1 SIS structures

achieved.

3.2 DEPOSITION AT SIEGEN

3.2.1 System 2: NbN thin films

achieved.

3.2.2 Developing NbN based SIS samples on copper

3.3 DC SUPERCONDUCTIVITY EVALUATION AT IEE (1 PAGE)

Superconducting parameters were determined on flat samples with single superconducting layer as well as with multiple superconducting layers, prepared at the deposition facilities of the work-package partners.

Short summary of all results (w/o laser treated)

3.4 MAGNETIC FIELD PENETRATION MEASUREMENTS AT STFC (2 PAGES)

Short description of a facility.

Short summary of all results (w/o laser treated)

Including a comparison table to IEE results

Some discussion

4. Study of SC thin films on QPR samples (6-8 pages)

4.1 QPR SAMPLE

The main purpose of this work was.

4.1.1 QPR sample manufacturing

A total of 10 samples

4.1.2 Sample case

For the shipment of samples, a dedicated chamber has been designed and manufactured.

4.2 QPR SAMPLE POLISHING

Short summary

4.3 DEPOSITION ON QPR SAMPLES

The QPR samples have been and are being deposited at INFN, Siegen and STFC.

4.3.1 Nb coated samples

QPR-surface resistance measurements can be

4.3.2 SIS samples

QPR-surface resistance measurements can be

4.3.3 Commissioning and benchmarking of the CERN QPR 2.0

5. Exploration of new technologies for SRF

A number of new ideas were explores in WP15 for applying for SC TF characterisation and treatments

5.1 LASER POLISHING OF COPPER SUBSTRATE (0.5 PAGE)

5.2 Post deposition laser treatment of Nb films (1.5 page)

5.2.1 Small samples

5.2.2 QPR sample treatment and testing

5.3 Exploring evaluation of superconducting films with photo- and thermostimulated exoelectron emission spectra at RTU (0.5 page)

Another study performed at RTU aimed to explore the possibility of using photo- and thermostimulated exoelectron emission to superconducting thin film evaluation. The analysis of results was performed on samples obtained from the first set of 14 samples of Nb films, deposited by ARIES project partners on the Cu substrates, which were initially pre-processed by different methods. No relationships found between parameters of photo- and thermostimulated exoelectron emission spectra and Cu substrate preparation method. No correlation was found between photo- and thermostimulated exoelectron emission and surface roughness of Cu substrate and deposited Nb layers, as well as photo- and thermostimulated exoelectron emission and magnetic characteristic of the Nb films. The main conclusion was that this method is not suitable in present stay for characterising superconducting thin films.

5.4 Magnetic field penetration measurements (1 page)

Short highlight about this method

5.5 Summary for exploration of new technologies for SRF (0.5-page)

6. Conclusions and future plans

The ARIES WP15 team made significant progress with the production of superconducting thin film coatings on copper samples. System 3 (NbTiN) and SIS (NbN:AlN:Nb, as well as Nb_3Sn:AlN:Nb and NbTiN:AlN:Nb) has been deposited and characterised with surface characterisation instruments and DC magnetometry. Furthermore, the SISIS structures (Nb_3Sn:AlN:Nb_3Sn:AlN:Nb) and SS structures (Nb_3Sn:Nb, NbTiN:NbTiN, NbTi:Nb) have been investigated.

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References

[1] Gurevich, A., "*Enhancement of RF breakdown field of superconductors by multilayer coating"*. Applied Physics Letters, 2006. **88**: p. 012511.

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

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

[4] Ciovati, G., P. Dhakal, and a. Gurevich, "*Decrease of the surface resistance in superconducting niobium resonator cavities by the microwave field"*. Applied Physics Letters, 2014. **104**: p. 092601.

[5] Palmieri, V., "*New Materials for Superconducting Cavities"*, in *Proc. SRF 2001*. 2001: Tsukuba, Japan. p. 162-169.

[6] Grassellino, A., et al., "*Nitrogen and argon doping of niobium for superconducting radio frequency cavities: a pathway to highly efficient accelerating structures"*. Superconductor Science and Technology, 2013. **26**: p. 102001.

[7] Adam, J.D., et al., "*Procedes de preparation de surface du cuivre compatibles acec un depot de niobium realise par pulverisation"*. 1985, CERN.

[8] Dhakal, P., et al., "*Effect of high temperature heat treatments on the quality factor of a large-grain superconducting radio-frequency niobium cavity"*. Physical Review Special Topics - Accelerators and Beams, 2013. **16**(4).

[9] 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*. 2019: Dresden, Germany.

[10] 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.

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

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

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

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

[15] 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*. 2019: Dresden, Germany.

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

[17] Kubo, T., "*Multilayer coating for higher accelerating fields in superconducting radio-frequency cavities: a review of theoretical aspects"*. Superconductor Science and Technology, 2016. **30**(2).

[18] Abdulsattar, Z. and W. Al-Ashtari, "*Effects of Laser Parameters on Solidification Cracks Formation during LSM Treatment for AA6061"*. International Journal of Applied Engineering Research, 2018. **13**(8): p. 5614-5617.

[19] Kang, M., H.N. Han, and C. Kim, "*Microstructure and Solidification Crack Susceptibility of Al 6014 Molten Alloy Subjected to a Spatially Oscillated Laser Beam"*. Materials (Basel), 2018. **11**(4).

[20] Medvid, A. and P.M. Lytvyn, "*Dynamic of Laser Ablation in SiC"*. Materials Science Forum, 2004. **457-460**: p. 411-414.

[21] Aull, S., et al., "*On the understanding of the Q-slope of niobium thin films"*, in *Proc. SRF 2015*. 2015: Whistler, Canada.

[22] Bauer, S., et al., "*Production of Nb/Cu sputtered superconducting cavities for LHC"*, in *Proc. SRF 1999*. 1999: Santa Fe, New Mexico.

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

Annex: Glossary

Acronym	**Definition**
AFM	Atomic Force Microscope
CEA	Saclay Nuclear Research Centre - Commissariat à l'Energie Atomique
CERN	European Council for Nuclear Research
EDS	Energy Dispersive X-ray Spectrometry
EP	Electropolishing

HIPIMS	High power impulse magnetron sputtering
HZB	Helmholtz-Zentrum Berlin
IEE	Institute of Electrical Engineering Slovak Academy of Sciences, Bratislava
INFN-LNL	Italian Institute of Nuclear Physics - Legnaro National Laboratories
OFE	Oxygen Free Electronic copper
OFHC	Oxygen-Free High thermal Conductivity copper
PPMS	Physical Property Measurement System
QPR	Quadrupole Resonator
QWR	Quarter Wave Resonator
RF	Radio Frequency
RTU	Riga Technical University
SC	Superconductivity
SEM	Scanning Electron Microscope
SRF	Superconducting Radio Frequency
STFC	Science and Technology Facilities Council - Daresbury Laboratory
SUBU	Chemical Polishing of Cu with a solution of Sulphamic Acid and Butanol
T_c	Critical temperature of superconducting transition (thermodynamic)
TF	Thin films
USI	University of Siegen
XRD	X-Ray Diffraction