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MAIN HIGHLIGHTS OF ARIES WP15 COLLABORATION*

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Abstract

An international collaboration of research teams from CEA (France), CERN (Switzerland), INFN/LNL (Italy), HZB and USI (Germany), IEE (Slovakia), RTU (Latvia) and STFC/DL (UK), are working together on better understanding of how to improve the properties of superconducting thin films (ScTF) for RF cavities. The collaboration has been formed as WP15 in the H2020 ARIES project funded by EC. The systematic study of ScTF covers: Cu substrate polishing with different techniques (EP, SUBU, EP+ SUBU, tumbling, laser), Nb, NbN, Nb_3Sn and SIS film deposition and characterisation, Laser post deposition treatments, DC magnetisation characterisation, application of all obtained knowledge on polishing, deposition and characterisation, Laser post deposition treatments, DC magnetisation characterisation, application to the QPR samples for testing the films at RF conditions. The preparation, deposition and characterisation of each sample involves 3-5 partners enhancing the capability of each other and resulting in a more complete analysis of each film. The talk will give an overview of the collaborative research and will be an introduction to the detailed talks given by the team members.

INTRODUCTION

ARIES is a Horizon 2020 Integrating Activity, co-funded by the European Commission. An acronym of ARIES stands for Accelerator Research and Innovation for European Science and Society which started on 1st May 2017 for 4 years. ARIES includes three components: Networking, Transnational Access and Joint research activities divided in 18 WPs. Only four WPs were Joint research activities including WP15: Thin films for superconducting cavities.

The aim of this work package is to intensify systematic studies and development of the coating technology of superconducting materials to enable the superconducting thin film coated RF cavities, joining an effort of 9 partners from 7 countries brought together in this collaboration. It must be noted that 3 of them were new for accelerator related activities.

The main emphasis our team is on a systematic study of correlation between (1) substrate surface preparation, (2) deposition parameters, (3) film structure, morphology, chemistry and phase, (4) AC and DC superconductivity parameters and, finally, (5) the behaviour at RF conditions with the test cavities.

This paper reports the main results of the ARIES WP15 collaboration.

COPPER SUBSTRATE PREPARATION

For this project, small samples on copper substrate with a size of 53 mm × 53 mm were used as a standard. The objective was to investigate the effect of copper substrate polishing on Nb film. 50 planar copper samples were produced at CERN from the same copper sheet and polished with 4 different procedures: 25 samples were treated at CERN with chemical polishing (also known as SUBU5) solution and the other 25 samples were treated at INFN with SUBU5 solution, electropolishing (EP), SUBU+EP, and tumbling.

SUPERCONDUCTING THIN FILM DEVELOPMENT

Developing of Superconducting Thin Films (STF) is a core of the project. The substrates were prepared by INFN and CERN and were delivered to coating partners STFC, University of Siegen and INFN.

Nb Films

Subsequently, Nb thin films were deposited on copper samples from each procedure using direct current magnetron sputtering (DCMS). Although the deposition configuration is different from one centre to another, the deposition parameters were set to be comparable. The procedure and the applied deposition parameters in all three deposition facilities are reported in [1-3]. The results of sample evaluations with a full flux penetration field (B_{fp}) are shown in Fig. 1. Based on these results SUBU5 and EP were selected

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as most promising polishing procedures for the following ARIES WP15 work [4-7].

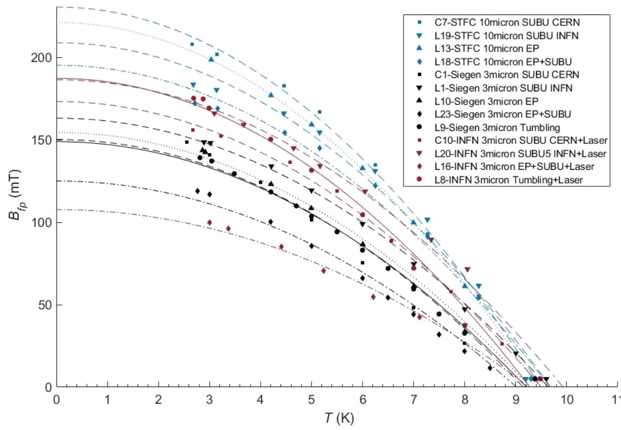

Figure 1: Overview of the field of full flux penetration (B_{fp}) for the Nb films on Cu.

Post Treatment of Nb Films with a Laser

Interesting results were obtained with Nb films irradiated by a laser at RTU. It was found that the laser radiation could result in an increase of the Nb grain size, improved adhesion of Nb layer to Cu substrate (annealing the defects by laser radiation), and improving superconducting properties [4, 5].

Non-Nb Films and SIS Structures

In Year 3 and 4 of ARIES project, the main focus was shifted to producing and testing the films *different from Nb*: such as NbN, Nb_3Sn, V_3Si, NbTiN [6-13]. These superconductors have higher T_c, and B_{c1}, that should allow to reach higher Q_0 and E. In order to provide thin films with a denser structure, the deposition method was changed from DC to HIPIMS. Various surface characterisation techniques were employed by each partner to characterize deposited thin films, among them SEM, FIB, EDX chemical mapping, XPS, XRD, AFM, etc. [8-17]. For example, the X-section SEM images of Nb_3Sn and NbN deposited on Cu are shown in Fig. 2 and Fig. 3, respectively.

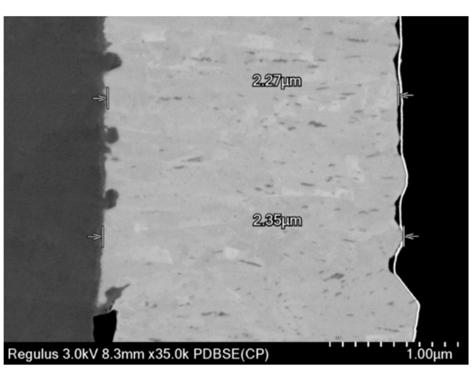

Figure 2: X-section SEM of Nb_3Sn deposited on Cu at STFC.

All samples were investigated in the DC magnetisation experiments at IEE, in order to compare their first flux entry field values B_{en}.

Comparison of B_{en} values of the non-Nb films on Cu substrate is shown in Fig. 5. The highest B_{en} values are seen among the Nb_3Sn based double layer, SIS samples and among the NbN based SIS samples deposited via HiPIMS.

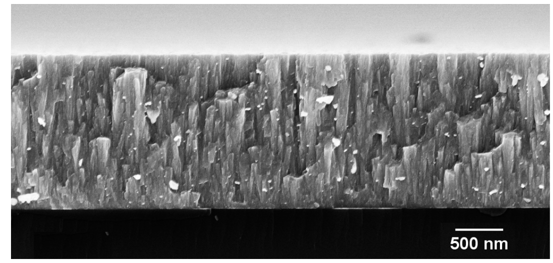

Figure 3: X-section SEM of NbN deposited on Si at high pressure at Siegen.

The progress with these films allows developing superconductor-insulator-superconductor (SIS) structures which, in theory, should allow further improvement of superconducting RF cavity characteristics [18,19]. For example, the EDS of a triple multilayer NbTiN in SIS structure (depicting each layer composition) deposited on Cu is shown in Fig. 4.

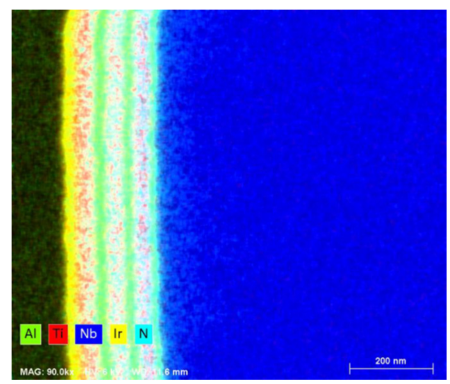

Figure 4: EDS of a triple multilayer NbTiN in SIS structure (depicting each layer composition) deposited on Cu.

Multilayer Achievements

In addition to first flux entry fields B_{en}, DC magnetometry measurements provide valuable information about flux trapping situation. Indeed, the surface of the hysteresis loop, is proportional to the number of flux lines that entered and got trapped in the material. Figure 6 shows the magnetization loops of 3 Nb_3Sn multilayer structures: a bilayer without insulating layer, bilayer with an insulating layer and finally a double bilayer. The 3 structures are deposited onto a thick Nb layer deposited itself onto a copper substrate. The fact that the hysteresis is smaller with the presence of a dielectric layer (even more when two barriers are present) shows how this structure prevents vortex penetration into the material. The individual layers present a large hysteresis [20], showing that the defects able to trap flux line are present in the layers. But thanks to the protective effect of the multilayer structure, and particularly to the presence of the dielectric layer(s), the vortices cannot enter the material in avalanche.

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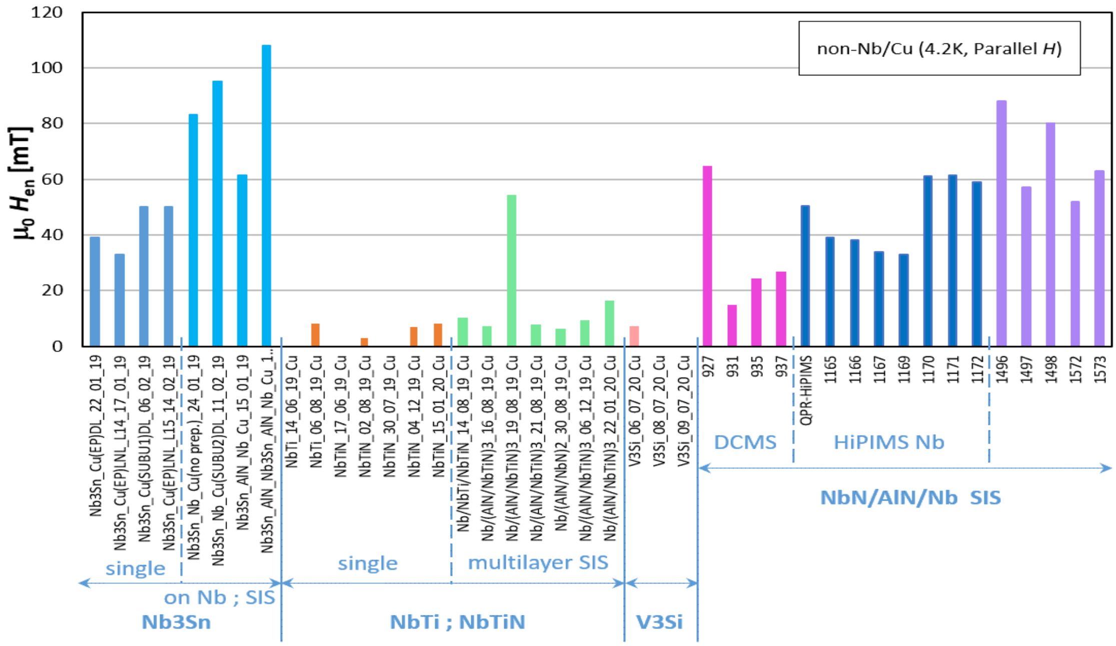

Figure 5: Overview of the first flux entry fields B_{en} for the non-Nb films on Cu substrates measured in VSM at IEE.

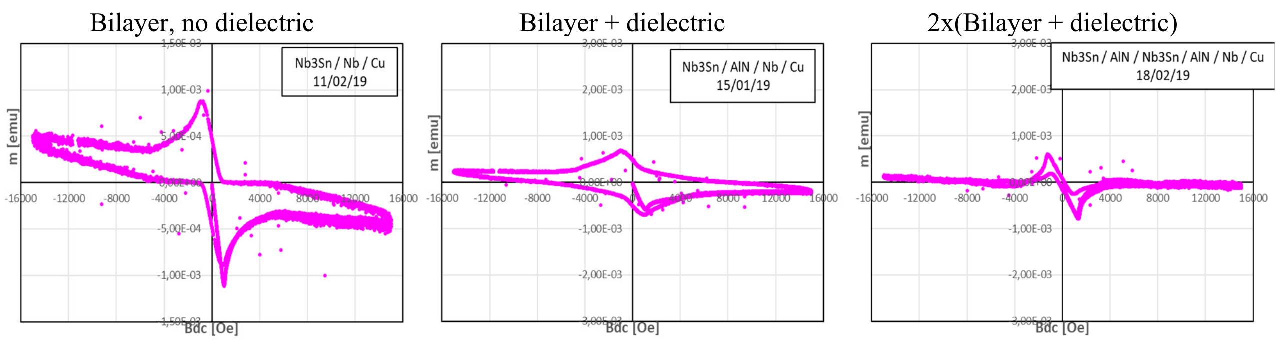

Figure 6: DC-magnetization curves of 3 Nb_3Sn multilayer structures. From left to right: a bilayer without insulating layer, bilayer with an insulating layer and finally a double bilayer. All the curves are at the same scale. The notable reduction of the hysteresis is a clear evidence of the protective effect of multilayer structures, even in presence of large quantity of defects in the material (see text).

RF TESTING WITH QPR

The SRF measurement are used to validate or disprove all the results with small samples, to demonstrate what are the correlations between the RF performance and all information obtained on small samples. Thus, the objectives for the RF testing are (a) QPR sample manufacturing, (b) cases for sample transfer, (c) sample polishing with EP and SUBU5, (d) Nb film deposition, (e) Nb film laser treatment, (f) SIS deposition, (g) SRF testing of all samples.

QPR Sample Preparation

Five QPR samples with Cu substrate for deposition were produced at the beginning of ARIES. Following the results obtained in the first period on planar samples, two polishing techniques were adapted to QPR samples: SUBU5 and EP. Before proceeding with the polishing treatments, several experiments were carried out, to solve some problems such as: enormous pitting, machining lines, point defects, (non)uniformity, oxidations, quantity of material removed and others. Then the updated protocol has been adapted to re-treat the QPR samples after the coating and RF test to produce a fresh copper substrate on the top ready for a new coating. In particular, mechanical polishing has been avoided and 3 more surface treatments have been added: indium removal, Nb chemical stripping and persulfate etching [7].

The QPR sample workflow is shown in Fig. 7. All the samples went under an identical treatment: after an initial mechanical polishing on a lathe machine, the Cu substrates were polished with EP or SUBU5 solutions at INFN [7, 8, 21, 22], then coated with superconducting thin films at INFN, Siegen or STFC; and finally, the samples were sent for the SRF testing at HZB or CERN. After completing the RF test, the sample returns to INFN for removing the film and polishing.

A great attention has been paid to provide transporting of the QPR samples under the clean room conditions. A

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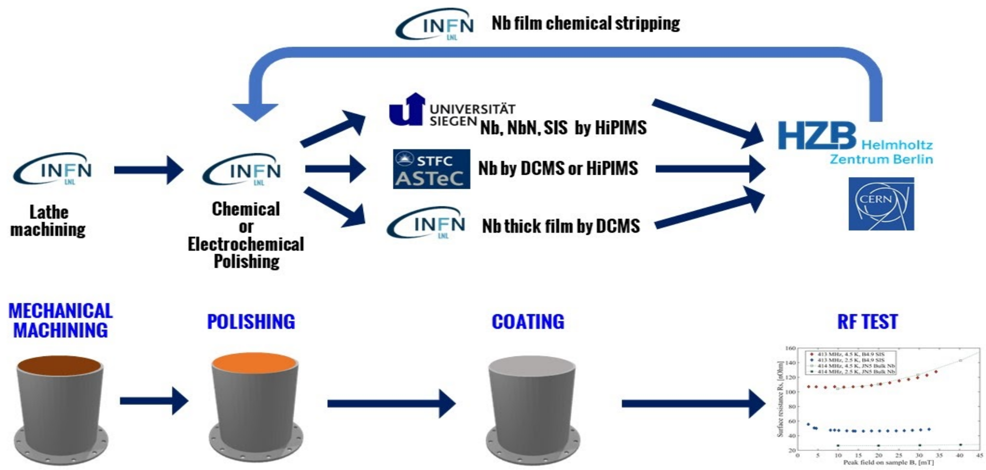

Figure 7: QPR sample workflow.

dedicated chamber for transporting the QPR samples has been designed and manufactured.

QPR Sample Deposition

Superconducting films were deposited on QPR samples at three labs after building an adapted sample holder at Siegen University, and new dedicated facilities at INFN and STFC. In total, 10 QPR sample depositions were performed within ARIES programme.

First five samples were deposited with Nb [7, 8, 14, 23]. This study allows to build a confidence in Nb deposition and compare the result to the leaders and move on to multilayer coating on QPR samples. The following samples were used for depositing SIS structures and the SRF tests. Three QPR samples were coated at Siegen University with different SIS structures to investigate the effects of the AlN layer thickness [24].

RF Test at QPR

The majority of samples were tested at HZB. The measurement setup of the HZB Quadrupole Resonator is comprehensively described at [25]. After several RF tests of Nb samples new properties of films were observed (such as Q-switches etc.) which cannot be seen with standard DC experiments. Some of the samples, showed comparatively good results (in regards to existing cavities and bulk Nb). Highlighted measurement results are presented in Fig. 8. After the series of RF tests, EP of the Cu surface was chosen as preferable surface preparation method for further coatings.

Another interesting and promising result was obtained from the first real tests of the SIS multilayer structures (NbN-AlN-Nb). Some of them have shown unexpected behavior on the surface resistance versus temperature parameter space and another presented quite good RF properties: low residual resistance and low "*Q*-slope" or field dependent increase of surface resistance, equivalent to bulk Nb. Due to the higher T_c of the top NbN film lower temperature dependent component of the surface resistance was also obtained (about 20 n␣ lower at 4.5 K compared to Nb with same residual resistance) [24].

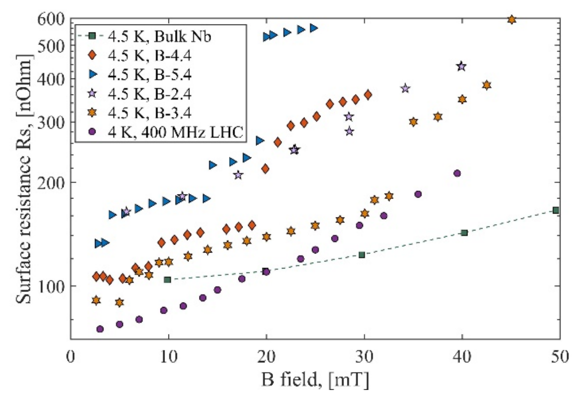

Figure 8: Nb/Cu samples: Surface resistance measured for 417 MHz at 4.5 K. The R_s for bulk Nb taken from earlier (best) QPR test and for typical LHC coating [26] are plotted for comparison.

RF TESTING OF PLANAR SAMPLES

For the SRF measurements at STFC the emphasis was made on a fast turn-around time (~2 days per sample) at low power measurement. A 7.8 GHz chocked cavity and a cryostat have been updated to operation with a closed-cycle refrigerator. The RF testing with a bulk Nb sample is in progress [8, 27, 28].

CONCLUSIONS

The main achievement over 4 years of ARIES are:

- Five polishing techniques for Cu have been tested with Nb films,
- Development of superconducting films on small samples establishes a capability of depositing NbN, Nb_3Sn, NbTiN films as well as SIS structures,
- Evaluation of Nb films at the RF conditions enables: Routine sample transport between the labs; QPR sample polishing developed and applied to the samples at INFN; A number of QPR samples has been deposited at INFN, Siegen and STFC with Nb and SIS structures; Comparative testing of QPR facilities at CERN and HZB;

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- ARIES enabled developing new technologies for the thin film SRF: Laser treatment of Cu substrate and Nb films and Magnetic field penetration facility;
- Finally, ARIES helped to set up more intense and coordinated collaboration, involving new partners, enhancing capabilities of every partner, frequent discussions and joint publications.

ARIES WP15 went to the end by 30th April 2021. A new H2020 funded collaboration has started on the 1st May 2021 for another 4 years. Its acronym is IFAST: Innovation Fostering in Accelerator Science and Technology. Our team has successfully applied for further development of superconducting thin films for SRF applications. The team has grown to 15 partners from 9 Countries. It will continue all activities started with ARIES on further development of various superconducting thin films. In addition to Physical Vapour Deposition methods, an Atomic Layer Deposition will be explored. However, the main emphasis will be shifted on applying the result of ARIES to the deposition and testing of the half-wave RF cavities at 6, 3 and 1.3 GHz.

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