ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP073

SUPERCONDUCTING THIN FILMS CHARACTERIZATION AT HZB WITH THE QUADRUPOLE RESONATOR

D. Tikhonov*, S. Keckert, J. Knobloch, O. Kugeler, Y. Tamashevich
Helmholtz-Zentrum Berlin (HZB), Berlin, Germany
A-M. Valente-Feliciano, Thomas Jefferson National Laboratory (JLab), Newport News, USA

Abstract

Superconducting films have great potential as post-Nb material for use in SRF applications in future accelerators and industry. Test the RF-performance of such films in practice would require the building and coating of a full RF cavity. Deposition of thin films on such scales in test facilities are challenging, in particular when curved surfaces have to be coated. This greatly complicates their systematic research. In this contribution we report on the method we use to characterize small and flat thin film samples (Deposited onto both Nb and Cu substrates) in an actual cavity named the Quadrupole Resonator (QPR). We also summarize the latest measurement results of NbTiN films.

INTRODUCTION

Cavities, coated with different superconducting materials are good candidates for replacing bulk Nb cavities. Deposition of superconducting materials on Cu and Nb might increase maximum field, thermal conductivity and reduce costs of material. Therefore, such cavities are now perspective candidates for future accelerator projects (such as FCC), but the production procedures require optimisation.

An extensive research of RF properties of different films (supplied by various institutes) is being conducted at HZB. For this purpose we use the Quadrupole Resonator (QPR) which is a tool that is able to perform SRF characterizations at frequencies ~415, 845, 1285 MHz with RF fields using an RF-DC power compensation technique. The QPR is also able to perform measurements at wide range of temperatures (from ~2 to >20 K). For the details regarding the QPR, please, refer to [1, 2].

In this contribution we present newly produced copper and niobium QPR substrates (samples) for films deposition and report on measurements of two NbTiN films, produced by JLab with bulk (2 µm) and thin (70 nm) films. In addition to this, we performed RF characterisations of multilayer Superconductor – insulator – superconductor structure (SIS) film (for more details see [Ref. 3]).

NEW SUBSTRATES FOR SUPERCONDUCTING FILMS RESEARCH

To simplify the study of films we designed new substrates for superconducting films deposition. The substrates (QPR samples) have simplified design and reduced size, which is more suitable for coating facilities (see Fig. 1). In total, five copper and five niobium substrates have been produced at Research Instruments GmbH (RI). Those substrates were donated by the ARIES project[1] one of the goals of which is the systematic study of the copper surface preparation procedures for films deposition for applications in future accelerator facilities (such as FCC).

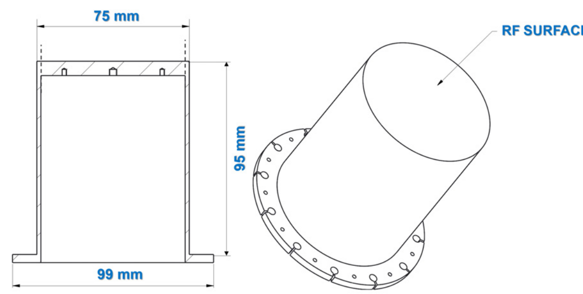
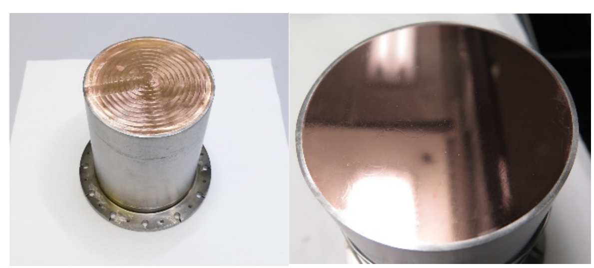


Figure 1: Samples dimensions and shape.

Copper Substrates

Five copper samples will be used to study (within the ARIES project[1]) the influence of Cu surface preparation methods on RF parameters of the coated films. As a first step, two samples will be polished with SUBU[2] (after reduction of the original roughness) at INFN LNL and then coated with NbN films at two facilities: University of Siegen and Daresbury Laboratory.



(a) (b)

Figure 2: One of the Cu substrates after fabrication (a) and after first SUBU polishing (b) *[©Credits: Cristian Pira, E. Chyhyrynets and other. INFN LNL]*.

The main challenge during the production of those samples was the Cu-Nb joint on top of the sample (see Fig. 2). To create that joint the electron-beam welding method was used. Since Cu and Nb are two different materials it is impossible to weld them together (they do not create a single crystal cell structure). Therefore, this joint is rather brazed than welded. However, it has quite high mechanical strength. The welding seam on test samples was also investigated by RI. From the perpendicularly cut section (see

*dmitry.tikhonov@helmholtz-berlin.de

[1]European Unions' ARIES collaboration H2020 Research and Innovation Programme.
[2]Chemical Solution of Sulfamic acid, n-Butanol, Hydrogen per-

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP073

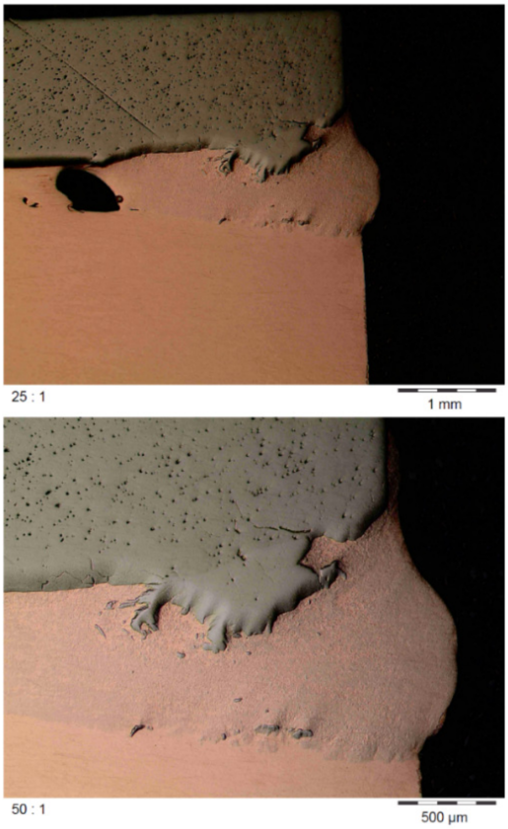


Figure 3: Cross-section of the Nb-Cu seam [©Credits: Victor Carrion, Karl-Bernhard Bolz, Michael Knaak, RI Research Instruments GmbH].

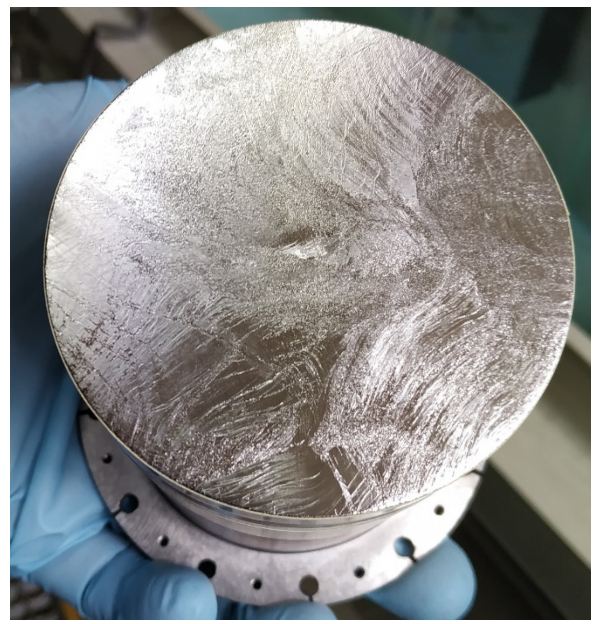
Fig. 3) the deepness of the 'welding' seam might be estimated as 2-2.5 mm. In addition, some voids were observed in in the seam, which are possible candidates for virtual leaks, therefore those samples still require testing for vacuum performance.

Niobium Substrates

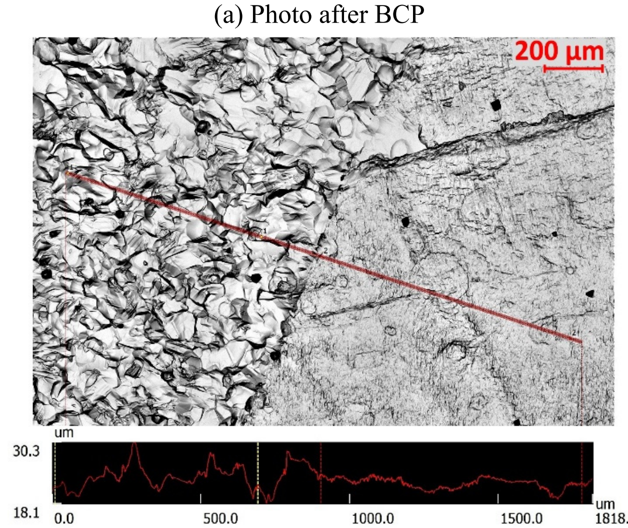
Niobium substrates have also been produced at RI (see Fig. 4). They will be used the same way: for the study of the superconducting films, deposited on Nb.

Originally, it was planned to perform standard preparation steps (polishing – BCP – annealing) as for a small-grain Nb cavity. However, after manufacturing it was found that the material grain structure was not uniform. Therefore after BCP (150 µm removed) the surface roughness was too high and not acceptable as a substrate for films deposition (R_z ~11 µm, measured with *3D Laser Scanning Microscope vk-x200,* see Fig. 5). Further surface preparations are planned.

Figure 4: Five Nb substrates after fabrication and mechanical polishing.



(a) Photo after BCP



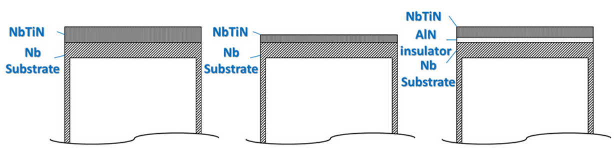
(b) Image from the *Laser Scanning Microscope vk-x200* and surface profile along the red line in µm.

Figure 5: Nb substrate after BCP.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP073

NB-TI-N FILMS MEASEREMENTS

In order to investigate the RF properties of NbTiN films, coated on Nb substrates, three samples were fully produced by JLab for characterisations with the QPR. They all have Nb base (substrate) but different NbTiN film structures (thickness). The structures of three samples shown in Table 1 as well as Fig. 6. The most recent measurement results of two samples (bulk *a* and thin *b* films) are presented in this chapter. The elaborate report of the first SIS' sample measurements with quantitative analysis using recent theoretical methods will be reported in SRF'19 proceedings [3]. Since all three films have been produced with same conditions, compared together they it is possible to investigate the RF properties of the NbTiN film more comprehensive.



(a) Bulk film (b) Thin film (c) SIS structure

Figure 6: The structure of three samples.

Table 1: Structures of NbTiN Films of Three Samples

NN	Film structure
a	Thick film: 2 μm of NbTiN on Nb substrate
b	Thin film: 70 nm of NbTiN
c	SIS structure: 75 nm of NbTiN on 15 nm of AlN insulator on Nb substrate

Surface Resistance

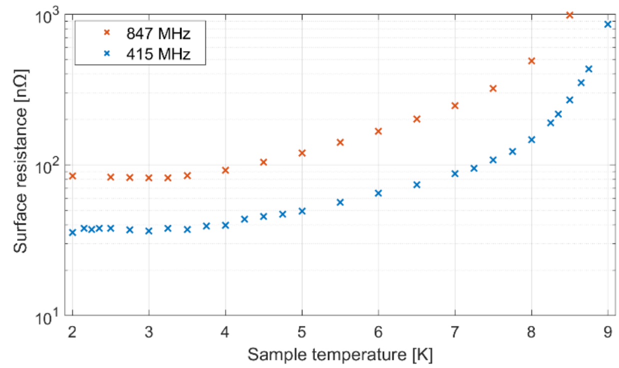
Surface resistance was measured for the bulk film (a) sample at two frequencies: 415 and 847 MHz, for the thin film sample (b) it was measured for 410, 839 and 1278 MHz. Surface resistance of two samples (*a*, *b*) shows good quality of films (see Fig. 7). For 2 K it is in the range of 20-50 nΩ at 415 MHz. The measurements were restricted by the RF quench field, which was ~55 mT and 10 mT for samples *a* and *b* correspondingly (see Table 2). Also, RF measurements where restricted by the critical temperature of Nb substrate, which is around ~9.3 K. From the surface resistance versus B field measurements (see Fig. 8 (a) and Table 2) can be seen that thin film sample *b* now shows stronger increase of R_S with B field and lower RF quench field.

Critical Field

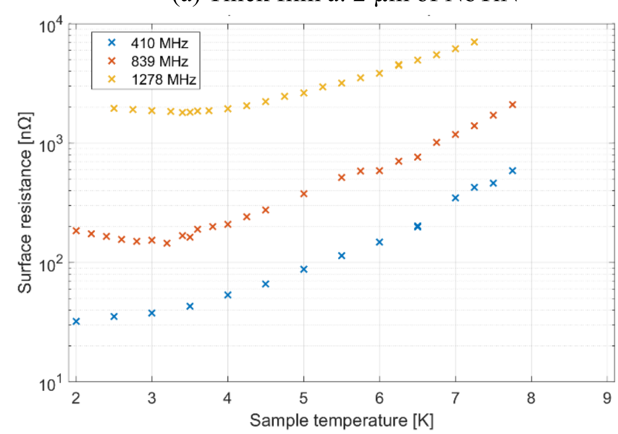
In addition the dependence of RF quench field vs temperature have been analysed for *b* sample (see Fig. 9 (a)). The measurements had been done in the single pulse regime, so thermal drift of the sample can be excluded. There is the difference around 1 mT between 3 frequencies that can also be explained by the errors in calibration parameters of the QPR system between three modes. Generally, from the measurements can be concluded that RF quench field, extrapolated to 0 K temperature, is around ~10 mT.

Table 2: Measured RF Quench B Field and Critical Temperature for Three Samples

NN	Film structure	B_{quench}, [mT] (for 3 K)	T_0, [K]
a	2 μm of NbTiN	55±1	17.0 ±0.1
b	70 nm of NbTiN	9.5±1.4	16.5 ±0.1



(a) Thick film a: 2 μm of NbTiN



(b) Thin film b: 70 nm of NbTiN

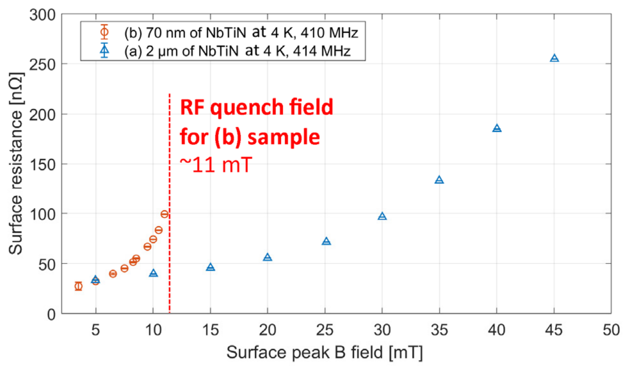
Figure 7: The measured surface resistance R_s as a function of sample temperature for *a* and *b* films. Results of the measurements of the third film are presented at [3]. Errors are not shown on the plot and have scale as 5-10% due to He bath pressure and RF system instabilities.

The empirical expression that gives the relation between critical B_c field and temperature T is (1):

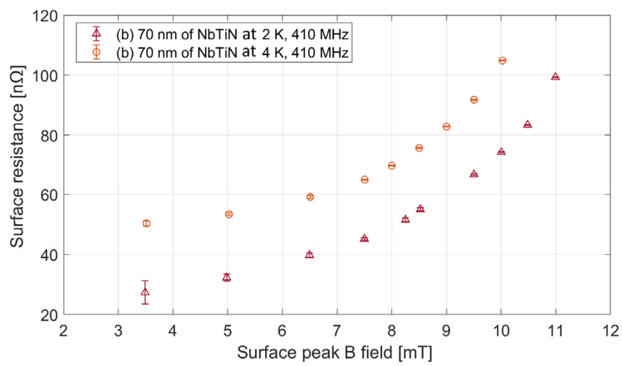
$$B_c(T) = B_0\left(1 - \left(\frac{T}{T_c}\right)^2\right). \qquad (1)$$

The attempt to fit this equation to the measured data on the plot gives the result for Tc around 11.1 K (see Table 3) and does not fit perfectly to the data (see Fig. 9 (b)). Moreover, the critical temperature T_c of this film *b* measured with a different method (using the vector network analyser for performing resonance frequency versus slowly changing temperature scan [4]) shows that $T_c \sim 16.5 \pm 0.1$ K. The results of those scans are shown in Fig. 10 (b). The deviation of the fitted curve (1) from the measurements might be explained by the structure of the sample film itself.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP073

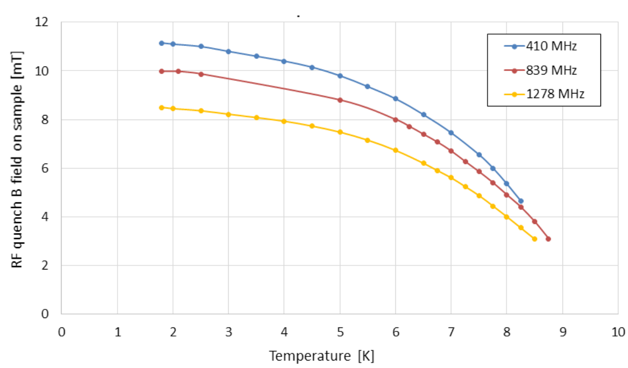


(a) Thick and thin films samples (*a* and *b*) compared

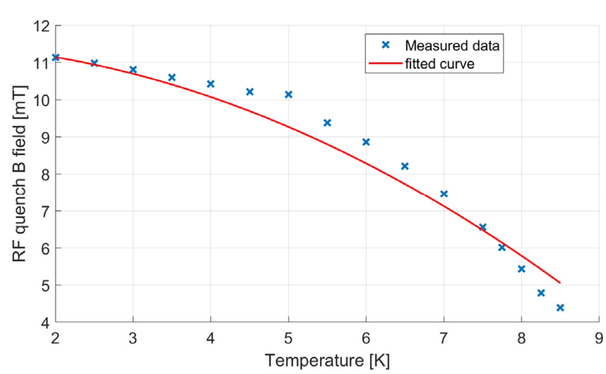


(b) Thin film: 70 nm of NbTiN

Figure 8: Surface resistance vs *B* field measurements for two (*a* and *b*) samples

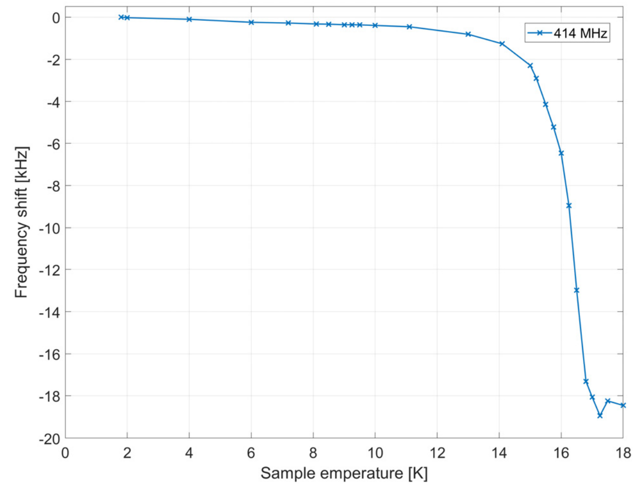


(a) measured data for three modes

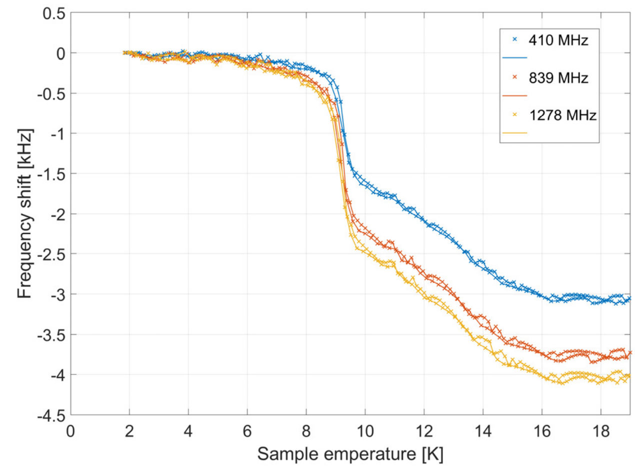


(b) Fitted curve from eq. (1) for 410 MHz data

Figure 9: RF quench *B* field measurements as a function of temperature for 70 nm thin film *b* sample



(a) Bulk 2 μm film sample



(b) Thin 70 nm film sample

Figure 10: The resonance frequency vs temperature dependence for two samples (*a* and *b*).

Table 3: Results of B_c *vs* T Data Fit to the Eq. (1)

Frequency of the mode [MHz]	**B_0, [mT]**	**T_0, [K] from fit. (1)**	**Measured T_0, [K]**
410	11.5	11.4±0.4	16.5 ±0.2
839	10.4	11.2±0.3	
1278	9.0	11.1±1.8	

Critical Temperature

Using the vector network analyser for performing resonance frequency versus slowly changing temperature scan the F_{res} vs T curves for two samples (*a* and *b*) were obtained (see Fig. 10 a, and b). From the data it can be preliminary concluded that the critical temperature for two films is 17.0±0.1 K and 16.5±0.2 K for the bulk film *a* and thin film *b* samples correspondingly. The critical temperature for the thin film sample *b* is still lower than expected (17.3K [5]), but higher then *Tc* of the SIS sample [3].

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP073

CONCLUSION

The extensive programme of superconducting film research is now undergoing at HZB. The Cu and Nb substrates (sponsored by the EU ARIES project) have been produced. They will be used for the investigation of RF properties of different films and systematic study of surface preparation procedures. The design of those substrates have been adapted to make the films deposition process easier. During the manufacturing various challenges of such design have been overcome. For ex., the Cu-Nb joint have been created using EB-welding machine and studied.

In addition, the comprehensive RF characterisations campaign of NbTiN films on Nb substrates (produced by JLab) have been conducted. The measurements have been performed at the temperature range 2 – 9 K and at frequencies ~ 415, 845 and 1285 MHz. The low surface resistance (20-50 nΩ at 415 MHz) of those films shows a good quality, however they all have far too low RF quench field $B_{RF} \lesssim 55$ mT ($B_{RF} \lesssim 10$-20 mT for 70 nm films) as compared to present Nb cavities. In addition to this the critical temperature Tc have been measured in the range of 16-17 K for two samples which is close to the theoretical value 17.3 K [5].

ACKNOWLEDGEMENT

This work is supported by EASITrain – European Advanced Superconductivity Innovation and Training. This Marie Sklodowska-Curie Action (MSCA) Innovative Training Networks (ITN) has received funding from the European Union's H2020 Framework Programme under Grant Agreement no. 764879.

The manufacture of the QPR samples received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

REFERENCES

[1] R. Kleindienst, J. Knobloch, and O. Kugeler, "Development of an Optimized Quadrupole Resonator at HZB", in *Proc. 16th Int. Conf. RF Superconductivity (SRF'13)*, Paris, France, Sep. 2013, paper TUP074, pp. 614-616.

[2] R. Kleindienst, A. Burrill, S. Keckert, J. Knobloch, and O. Kugeler, "Commissioning Results of the HZB Quadrupole Resonator", in Proc. 17th Int. Conf. RF Superconductivity (SRF'15), Whistler, Canada, Sep. 2015, paper WEA1A04, pp. 930-936.

[3] S. Keckert, J. Knobloch, O. Kugeler, D. B. Tikhonov, and A-M. Valente-Feliciano, "RF Characterization of an S-I-S' Multilayer Sample", presented at the 19th Int. Conf. RF Superconductivity (SRF'19), Dresden, Germany, Jun.-Jul. 2019, paper THFUA1.

[4] S. Keckert, Ph.D. thesis, to be submitted.

[5] A.-M. Valente-Feliciano, "Superconducting RF materials other than bulk niobium: A review", *Superconductor Science and Technology*, vol. 29, no. 11, p. 113 002, 2016. doi: 10.1088/0953-2048/29/11/113002.