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A FIRST 6 GHz CAVITY DEPOSITION WITH B1 SUPERCONDUCTING THIN FILM AT ASTeC *

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Abstract

Nb_3Sn, NbTiN and NbN are superconductors with critical temperatures of 18.3, 12.6-17 and 11.6-17.5 K, respectively, these are higher than that of Nb at 9.3 K. Hence, at 4 K, they have an RF resistance, an order of magnitude lower than that of Nb, which leads to quality factors above those of Nb. In recent years, there has been an extensive effort converting Nb cavities into Nb_3Sn. Alloying the top inner layer of the cavity using Sn diffusion at a high temperature has had some degree of success, however, the reproducibility remains a major hindering and limiting factor.

In this study, we report on the PVD deposition of NbTiN inside a 6 GHz cavity, using an external magnetic coil configuration. The deposition is done at an elevated temperature of about 650 °C.

We report on the superconducting properties, film structure and its stoichiometry and surface chemical state. The films have been characterised with SEM, XRD, XPS, EDS and SQUID magnetometer.

INTRODUCTION

500 MHz Copper cavities coated with a thin Nb film (1.5 to 5 µm), have been operational since 1980 in various particle accelerators [1]. However, their performance has only matched those of bulk Nb cavities at moderate accelerating gradient of up to 8.6 MV/m. In the past two decades, due to advancement in thin film deposition technology and better understanding of surface preparation, there has been a coordinated effort by the SRF community to push the performance of thin film, SRF cavities to a level that can compete with bulk Nb, at high-accelerating gradients of up to 20 MV/m.

Furthermore, this advancement allowed materials with a critical temperature (T_c) higher than Nb, to be synthesised on copper substrates, with superconducting properties matching their respective bulk materials.

Using materials with T_c higher than for Nb, such as NbN, Nb_3Sn, NbTiN, MgB_2, etc., as well as multilayer structures, allows us to reach parameters that are unreachable for existing RF cavities:

- Increasing the quality factor Q, reduces the heat produced, and, hence, the electricity consumption of the cryogenic system during the RF cavity operation.
- Using high T_c superconducting materials allows to operate RF cavities at 4.2 K instead of 1.9 K, used for the high-performance Nb cavities, more than doubling the efficiency of the cryogenic system.
- Increasing the cost-effective acceleration field E (at present the minimum cost is achieved at just over 30 MV/m) will result in massive saving in the infrastructure (tunnel, LHe supply and He recovery lines, electric cables, controllers, cryostats, pumps, etc), for example 20% increase in the acceleration field allows 20% reduction in the acceleration line (compare: a 4-km long tunnel for EU-XFEL instead of a 5-km long one, or 50-km long tunnel for ILC instead of 60-km).

This improvement goes by a few routes.

Other alloy forms of Nb, known as A15, such as Nb_3Sn or Nb_3Al, with a higher T_c, a potentially higher critical field H_{c2}, leading to potentially significant cryogenics cost reduction if the cavity operation temperature is 4.2 K or higher. Single crystal, high quality films have been achieved on a single crystal MgO [2, 3], sapphire and single crystal copper substrates, but more effort is needed for translating these remarkable results onto real 3D geometry cavity.

Multilayer film of Superconductor/Insulator/Superconductor (SIS) should provide a much higher E_a than a single layer film [4].

Currently, thin film SRF cavity production is based on PVD processes, where sputtering is the preferred method due to its ease of scalability, moderate conformability and above all its ability to control the film morphology and microstructures. The SRF thin film synthesis by sputtering process owes much of its success to being a single element thin film (mostly Nb). Synthesis of an alloy SRF thin film on a 3D geometry such as cavity is much more challenging.

At ASTeC (UKRI/STFC Daresbury Laboratory), alloy-superconducting material such as Nb_3Ge, Nb_3Sn, V_3Si, NbTi, NbTiN and NbN have been successfully synthesized on various flat substrates either using alloy target or by co-sputtering, i.e., by sputtering simultaneously two constituents on a temperature-controlled substrate. In co-sputtering, the achieved composition is dependent on the relative positions of the target and the substrate. The perfect stoichiometry can then be obtained by manipulating these positions. However, the control of the stoichiometry may be

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difficult over the large areas of accelerating cavities, especially if the stoichiometry range for the A15 and B1phase is narrow.

The (plasma-enhanced) chemical vapour deposition (PE)CVD of either single (Nb) or alloy superconducting material (NbTiN and NbN) has also been used at ASTeC to deposit mainly on flat substrates [5, 6]. In this process, one or more precursors, present in vapor phase, chemically react, and form a solid film, on a substrate at the appropriate temperature. The deposition rate and the structure of the film, depends on the temperature and reagent concentration. The control of the temperature and gas flow uniformity over the entire cavity surface may be difficult with complex geometries.

Combinations of the two-deposition process of PVD and CVD can overcome the shortfall of each individual process. The hybrid physical chemical vapor deposition (HPCVD) which combines physical and CVD has been shown to produce high quality MgB_2 thin film on various 2D flat substrates. The high temperature used in HPCVD favors excellent epitaxy and crystallinity, yielding RRR values in excess of 80.

Recently, we extended the HPCVD process to synthesis alloy superconducting thin film MgB_2 on a 3D geometry substrate, by using magnetron sputtering for one of the elements of the alloy (Mg), using a single element wire, and providing the remaining element of the SC alloy (B) in vapor form [7]. The plasma from the magnetron sputtering facilitated the decomposition of the precursor, and hence, allowed the chemical reaction to take place at a much lower permitted temperature onto a copper cavity.

Based on the success above, the next step is to synthesis superconducting films on to the inner surfaces of RF cavities. For this purpose, we have designed a new set up to be able to deposit inner surface of a 6 GHz copper cavity at ASTeC.

6 GHz copper cavities, made by spinning at INFN, are delivered to ASTeC, after being etched and polished and ready to be deposited on, are used for this project.

DEPOSITION FACILTY

Figure 1 shows the picture of the cavity deposition chamber (a), the closed cavity assembly flange (b and c). Due to a small aperture of the cavity iris (which is about 19 mm diameter), the magnetic field for magnetron is provided by an outer coil, see Fig. 1(a). The entire deposition chamber is confined within the coil gap. The flange assembly is consisting of a donut shaped plate, which clamp the cavity to the CF200 flange. Six halogen lamps are mounted at the outer periphery of the plate to provide heat for a high temperature deposition, a K-type thermocouple is also clamped to the cavity to measure the cavity temperature while it is being heated. A heat shield consisting of three concentric stainless-steel cylinders separated by a 1 mm gap between adjacent cylinders. A 3 mm diameter Nb rod wrapped with a 1-mm diameter Ti wire, is inserted into the centre of the cavity, which provide a deposition target. The deposition was done in a reactive environment of Kr (60%) and N_2 (40%). The deposition parameters are shown in Table 1.

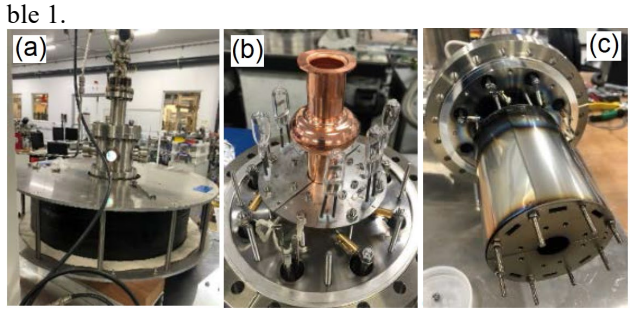

Figure 1: A 6 GHz closed cavity deposition: closed cavity deposition system (a), a cavity assembly flange without (b) and with a heat shield (c).

Table 1: Deposition Parameters for NbTiN Coating of a 6 GHz Cavity

Power	150 W
Current	0.16 A
Voltage	644 V
Pulse frequency	350 kHz
Duty cycle	1.1 µs
Deposition time	1 hour 30 min
Deposition pressure	3.6×10^{-2} mbar
Mag current	7.51 A
Deposition temperature	650 °C

The deposition was carried out using an Advance Energy pinnacle plus, in pulsed DC mode, to provide a higher concurrent ion bombardment, during deposition, and hence produce a high-density thin film.

Prior to NbTiN deposition, a thin layer of Nb was deposited. The second layer of NbTiN was deposited on Nb coated copper cavity after exposure to air for changing a target. After deposition, the cavity was cut in two section and is shown in Fig. 2. It can be seen that the cavity is uniformly coated with a golden colour, which is the signature colour related to transition metal nitride.

RESULTS

The planar and X-section SEM, of a section cut from the equator of the cavity is shown in Fig. 3. It illustrates that the film transition between each layer is smooth, and no significant damage at the Nb and NbTiN interface is observed, despite the fact that the NbTiN layer was deposited post deposition of Nb, and after the Nb layer was exposed to air. The Nb layer thickness is estimated to be about 1.1 µm and the NbTiN layer is about 2.2 µm. the crack observed in the film is due to post cutting and stretching of the sample.

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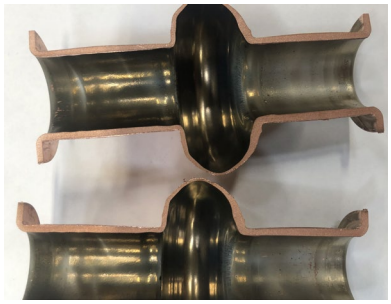

Figure 2: A 6 GHz closed copper cavity deposited with a double layer Nb/NbTiN.

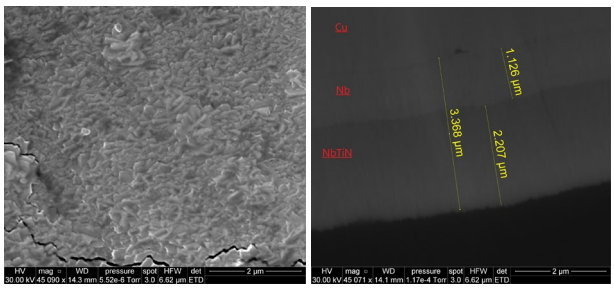

Figure 3: (a) Planar and (b) X-section SEM images of dual layer Nb/NbTiN film.

The ratio of Nb to Ti as determined by EDX analysis, varied from 0.8 to 1 for different spots on the sample. The critical temperature, determined by SQUID magnetometry was found to be around 15 K with slow transitions. This can be a consequence of non-uniform composition, as shown in Fig. 4 (b). This is lower that we achieved in flat samples, which was 17.5 K [3]. The first field of penetration is about 30 mT, and the H_{c2} is higher than the range set in the squid measurement. The magnetisation curve is free of any flux jumping, illustrating the film is free for any deep level defects, as shown in Fig. 4 (a).

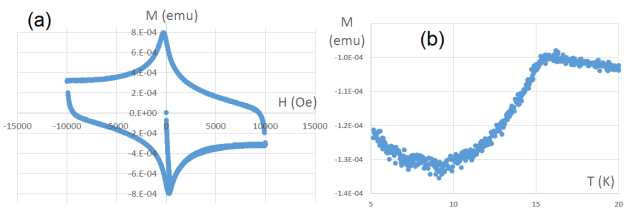

Figure 4: (a) A magnetization curve measured at 4 K and (b) magntization under contant 100 Oe for varing temperature from 20 to 5 K.

Figure 5 shows the XRD spectra carried out in grazing angle. It shows that the film is synthesised in equiaxial grains. As well as NbTiN peaks, Cu peaks are also apparent. This is due to the sample size, avoiding the edges needs a larger samples, which in this cavity area is not possible. The PDXL2 software has been used to caculate the grain size, which was estimated to be on average 15 nm, with a lattice constant of 4.327 A, which is inline with reported values in literature resulting to T_c of 14 to 15 K [4].

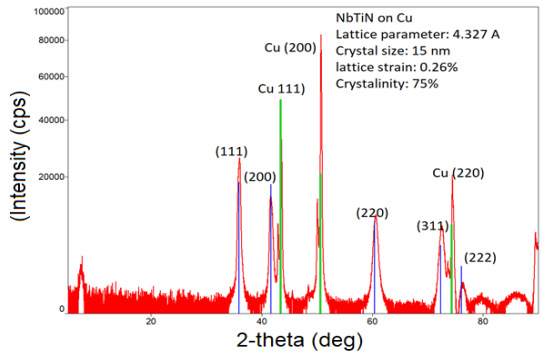

Figure 5: Grazing angle XRD of NbTiN Film.

The EDS provides bulk composition information. It does not provide information as to how the elements are related. Furthermore, it is very inaccurate in estimating the amount of N or O. NbTiN is a ternary compound and the synthesis of superconducting phase B1 is very much dependent on its adequate stoichiometry. In order to determine the chemical state of the compound, the film was examined by X-ray photoelectron spectroscopy XPS, the results are presented in Fig. 6.

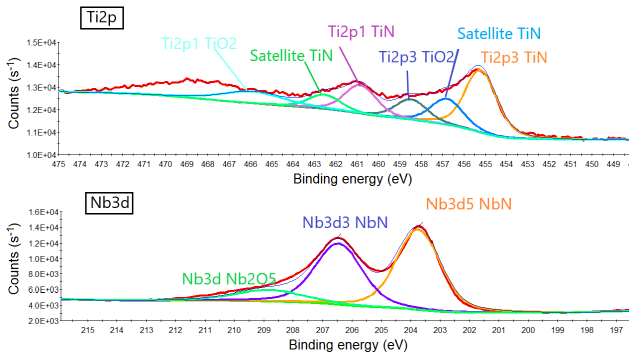

Figure 6: XPS spectra of Ti2p and Nb 3d region scan of NbTiN samples.

XPS can only reveal information from the first 10 nm of the sample surface. In this case, it depicts that both Nb and Ti in film are at their nitride state. However, in both cases, oxide state of both Nb and Ti are also present. The latter is due to surface oxidation, when surfaces are exposed to air. Nevertheless, the oxide states only exist at the surface.

Optimizing the deposition process with 6 GHz cavities allows to reduce the costs in comparison to 1.3 GHz cavity: smaller cavities require less copper, quicker production, less chemistry use, for surface polishing, smaller deposition chamber, less usage of the deposition target and less use of expensive LHe for the RF test. After defining the most promising film coating with 6 GHz cavity, these results will be applied to deposit a 1.3 GHz cavity.

CONCLUSION

The new 6 GHz cavity deposition performed well and the first B1 superconducting NbTiN cavity coating has been achieved at ASTeC. The target configuration needs some degree of optimisation, but proved to be effective where an alloy target in cylindrical form does not exist.

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