Nb Sputtered 325 MHz QWR CAVITIES for CIADS

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Abstract. The possibility for adopting niobium thin film coated copper (Nb/Cu) quarter wave resonators (QWRs) in the low energy section of CiADS project[1] is being evaluated. Comparing with bulk niobium cavities, the Nb/Cu cavities feature a much better thermal and mechanical stability at 4.5 K. Two 325 MHz Nb/Cu QWR cavities have been fabricated at IMP, to demonstrate whether the niobium coated copper cavity technique can meet the requirements of CiADS. The cavity is coated with biased DC diode sputtering technique. This paper covers resulting film characters, vertical tests with the evolution of the sputtering process, and improvements to mitigate issues we met.

1. Introduction
The operational stability of SRF cavities is one of the foremost challenges that hinder CiADS linear accelerator from continuous running[2]. Nb/Cu cavities replace the bulk niobium cavities could be an effective solution, because Nb/Cu cavities come up with advantages in terms of both thermal stability and mechanical stability[3]. At 4.2 K, the heat conductance of high purity bulk Nb is about 75 W/(m · K), while the number is as high as 300-2000 W/(m · K) for high purity oxygen free copper[3]. The poor thermal conductivity of Nb put an upper limit for SRF cavity wall thickness, in order for the inner surface to be effectively cooled, which impairs the robustness of the cavity’s mechanical structure. The use of a copper cavity as a substrate can effectively solve the major problems of poor thermal conductivity and sensitivity to external pressure and vibration at the same time, because the thicker copper wall can provide rigid stiffening in the SRF cavity. Furthermore, Nb/Cu cavity is economical than bulk Nb cavity in terms of fabrication and processing cost. The material cost of OFHC copper is only about 4% of the price for SRF grade bulk Nb. In addition, copper is easier to anneal, polish, and machine than niobium, the cavity processing cost for Nb/Cu cavities would be much lower than that for bulk Nb cavities[3].

Nb/Cu cavities have been successfully used in LEP2 at CERN before 1999. As many as 272 Cu/Nb ellipsoid cavities were operated at 7 MV/m @4.5 K on average, well above the design value[4][5][6]. Apart from CERN experience, INFN-LNL researched sputtering technologies on complex shapes such as QWRs began in 1988[7][8]. DC diode sputtering with negative bias electrode was used for producing Nb/Cu QWRs in LNL. By 1997, four resonators with laboratory test 5.7-7.7 MV/m at 7 W had been fabricated and installed on ALPI in 1997[8] then Nb sputtered copper QWRs replaced all.
ALPI’s initial Pb/Cu cavities\[9\]. A series of studies on niobium sputtering on copper substrates have also been carried out in other laboratories and universities, such as ANU(Australia), Saclay(France) and PKU(China)\[10\]\[11\]\[12\]. The most recent and most successful example of Nb/Cu technology is HIE-Isolde injector\[13\] which was commissioned in 2015. Inspired by the success application of Nb/Cu QWRs on heavy-ion Linacs, IMP launched its Nb/Cu cavity project in 2016. Up to now, the film characters, including thickness profile along the cavity, structure and morphology tests have been performed at IMP. Two dummy QWR cavities had been produced and coated to understand and optimize the sputtering setup and process.

2. Thin film coating setup
The coating system employed in this project is modify from an existed equipment at NIN with biased DC diode sputtering\[14\] ability. Figure 1(a) showed the side view of this very system. For the purpose of R&D tests, a 325 MHz QWR dummy cavity has been designed at IMP. Different from the traditional QWR cavity, the QWR cavity we optimized has no beam line on the inner conductor to facilitate coating. The dimension of the dummy cavity is shown in Fig. 1(b). For simplicity, two dummy cavities were machined directly from OFHC ingot to avoid any difficulties brought by welding seams inside the cavity\[15\]. S.S. flanges were brazed onto the cavities. Before coating process, surfaces were treated with mechanical and electrical polishing, different from the surface preparation used by CERN for the niobium sputter coating of the HIE-Isolde superconducting cavities\[16\]. From electromagnetic simulation, the unloaded quality factor (Q\(_0\)) for an uncoated copper cavity is \(1 \times 10^5\), and is \(8 \times 10^8\) for a superconducting Nb/Cu cavity.
A QWR-like sample holder with 16 samples positions along the outer and inner conductors had been used before the actual cavity coating. This setup allows access to the film properties in different positions by small sample characterizations. Thus the thickness and Tc distribution of the coating film could be investigated from clipped samples. The surface treatment of small sized samples is similar to the dummy cavity. The samples’ locations and their distance to the bottom plate are marked on Fig. 2(b).

3. Samples Results

The thickness of the niobium film deposited on copper samples is measured by a step profiler. The critical transition temperature (Tc) is measured by Superconducting QUantum Interference Device (SQUID) through standard susceptibility measurement. Initially, the sputtering utilized a tube-target sitting right in the middle of the inner and outer conductor. Such layout generated a non-uniform growth rate distribution, which is specified in Table 1. The ratio of average growth rate between inner conductor and outer is about 10:1. The experiment also reveals that the coating rate at cavity top was one-fifth of the rate at bottom area. With a maximum ratio of 50:1, the niobium film coated on the sample at point F couldn’t be thick enough before the film at point 1 started to break-off. The thickness limit test show film starts to break when the thickness of film greater than 60 μm. Then the shape of the Nb target was re-designed to ensure a maximum deposition ratio below 10:1.

| Location    | Growth rate (nm/h) |
|-------------|--------------------|
| Outer       | Top                | 20                  |
| Conductor   | Bottom             | 100                 |
| Inner       | Top                | 200                 |
| Conductor   | Bottom             | 1000                |

With optimization between each run, 3 test runs with copper samples were performed before the cavity coating. The Tcs of selected samples are presented in Table 2. Samples along the inner conductor exhibit better Tc above 9.3 K. But samples on the top of the outer conductor are with poor properties. The film properties are improved gradually. However, point F remains a weak point of our coating. This agrees with the growth rate distribution and implies the Tc of F sample is still limited by the thickness.

Results from the last run of experiments showed an almost complete superconducting coating was achieved on the QWR-like sample holder, except for point F. Due to the limit of current deposition system size, the optimization of growth rate ratio cannot be further reduced. To solve this problem, a new deposition system with a larger vacuum chamber to tune the sputtering layout is being built.
Table 2: Summary of samples’ $T_c$s in each test run*

| Run | A | F | Bottom | 1 | 3 | Top |
|-----|---|---|---------|---|---|-----|
| 1   | 7.3 | X | 9.0     | 9.3 | N/A | #   |
| 2   | <6 | X | X       | 9.3 | 9.3 | 9.3 |
| 3   | 9.3 | <7 | 9.4     | N/A | 9.3 | 9.3 |

*: $T_c$ unit in K.

#: Large visible defect on the surface, unable to measure.

N/A: Not measured.

X: not superconducting down to 4 K.

4. Cavity Test

After the first cavity coating test, it was observed the film on the top of the outer conductor wasn’t silver shining, implying the film there could be very thin or contaminated. And particles falling from the target produced several defects at the bottom part, because the cavity is under the target. The cloudy film and defects are shown in Fig. 3.

Ultrasonic cleaning and high pressure rinsing (HPR) are used in the first coated cavity post-treatment processing. Film peeled off at a few spots on the outer conductor after ultra-sonic cleaning, with typical size 1~2 mm (Fig. 4(a)). Optical investigation suggests it may result from the fallouts from the target or contaminated substrate surface. After HPR, no more defects are detected, no matter there was or wasn’t ultrasonic cleaning before it. In order to avoid more damage to the film, ultrasonic cleaning is no longer used in the post treatment processing for the second coated cavity. An SRF grade Nb plate is used as the end-plate, sealed by indium wire, because the QWR is designed to work under isolated vacuum. In the first cryogenic RF test, the microwave power was coupled from the sides (Fig. 4(b)), and this configuration gave us a lot of problems including MP and difficulty in feeding power into the cavity, because the size of the coupling tube is too small and the tube inner surface is rough.
The vertical test of the first film cavity is very lossy at 4.2 K. The $Q_0$ cannot be measured from the damping curve. Four temperature probes were installed on the bottom and top of the outer cavity surface and near the tube. The temperature reading showed that, there was little power fed into the cavity and most of power was consumed by the MP at the coupling tube. The temperature near the tube is much higher than the top and bottom when we turned on the input RF power. The loaded quality factor ($Q_L$) of the 1st cavity is about 2.5E5 measure by FWHM, in heavily over-coupled condition. The $Q_L$ value is much higher than the OFHC copper cavity at low temperature (~ 6E4), indicating at least part of the cavity was in superconducting status.

The RF power feeding configuration for the vertical test of the 2nd cavity was switched to bottom-coupled (Fig. 5). The 2nd cavity has the same behavior: $Q_L \sim 4E5$ at 4.2 K. However, in the 2nd test, the temperature rising occurred all over the cavity. The heating was more uniform comparing with the first test, and the temperature increase was less than the maximum value observed on the first cavity. In the 2nd test, the temperature sensor near the cavity top area always heats up faster than the others, indicating the film near the cavity opening does not transfer into the superconducting state.
Calculation for the loss source was made based on the assumption that there was non-superconducting defects on the cavity wall, either (a) a $\Phi$ -1 mm exposed copper spot like the ones in Fig. 4(a), or (b) a normal conducting niobium (RRR $\sim$ 4) ring near the opening (Fig. 6). The results indicated that the loss from a type (a) defect was about the same order as the loss from a whole superconducting cavity, while type (b) defect can dramatically reduce the cavity’s Q by 2 orders of magnitude. Comparing with the measured QL, the appearance of a normal conducting Nb ring may explain why we were having a degraded RF performance. To mitigate this problem, additional efforts need to be imposed to ensure the film near the cavity opening to be good. Such efforts include extending the Nb cathode out of the cavity to get uniform coating near the cavity opening, and flipping the deposition set up upside down to avoid any particles falling from the target depositing on the cavity surface.

5. Conclusion
The results of samples tests and vertical tests of the cavity showed that we are close to the final production of Nb/Cu QWR cavities with acceptable SRF performance. With proper optimization of our deposition system, we will demonstrate the feasibility for adopting Nb/Cu cavities at the low energy section of the CiADS linear accelerator.

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