MATERIALS ANALYSIS OF CED Nb FILMS BEING COATED ON BULK Nb SINGLE CELL SRF CAVITIES*

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Abstract

This study is an on-going research on depositing a Nb film on the internal wall of bulk Nb single cell SRF cavities, via a cathodic arc Nb plasma ions source, an coaxial energetic condensation (CED) facility at AASC company. The motivation is to firstly create a homoepitaxy-like Nb/Nb film in a scale of a ~1.5 GHz RF single cell cavity. Next, through SRF measurement and materials analysis, it might reveal the baseline properties of the CED-type homoepitaxy Nb films.

Literally, a top-surface layer of Nb films which sustains SRF function, always grows up in homo-epitaxy mode, on top of a Nb nucleation layer. Homo-epitaxy growth of Nb must be the final stage (a crystal thickening process) of any coatings of Nb film on alternative cavity structure materials. Such knowledge of Nb-Nb homo-epitaxy is useful to create future realistic SRF cavity film coatings, such as hetero-epitaxy Nb/Cu Films, or template-layer-mitigated Nb films.

One large-grain, and three fine grain bulk Nb cavity were coated. They went through cryogenic RF measurement. Preliminary results show that the Q_0 of a Nb film at 2 K and low rf field, produced by CED, could be close to that of the pre-coated bulk Nb surface (being CBP'ed plus a light EP); but the quality drops rapidly for increasing rf field. We are investigating if the severe Q_0-slope is caused by hydrogen incorporation before deposition, or is determined by some structural defects during Nb film growth.

INTRODUCTION

In recent years, we have extensively investigated growth of Nb films in epitaxy mode via an energetic condensation technology through a collaboration among AASC, Jefferson Lab, and some universities. [1-4].

AASC's role is to deposit Nb film on SRF cavities via a cathodic arc-discharge Nb ion deposition. Such Nb plasma ion uses a low voltage (30 V) arc discharge to generate a highly ionized plasma. The energy spectrum of ions from coaxial energetic deposition (CED) ranges from 20 eV up to 170 eV. The CED facility was running in a pulse mode with ~50 ms arc pulse width. The deposition at a given position on the substrate lasts ~1 ms/pulse, during which ~0.5 nm of Nb film is deposited.

The instantaneous deposition rate is as high as 560 nm/s. The estimated (instantaneous) ion flux is 10^{18} Nb ions/cm^2-s. The repetition rate (frequency) of the arc/pulse is 0.2 to 0.25 Hz. By considering the dead-time and frequency of the arcs, the nominal deposition rate is ~3Å/s, or roughly one atomic monolayer per second. This nominal deposition rate is similar to a conventional physical vapour deposition method, such as a magnetron sputtering source (1.67 nm/s) or the ECR Nb plasma source at Jefferson Lab (2.25 nm/s).

Our past research [1-4] indicates that the epitaxy films of Nb on copper polycrystals (Nb/Cu), Nb on magnesium oxide crystals (Nb/MgO100), Nb on different crystal planes of sapphire (Nb/A-plane Al_2O_3 , Nb/C-plane Al_2O_3), could all yield good crystal quality, close to a bulk Nb crystal.

By adopting a well-prepared crystal substrate, a heating condition for substrate, and a thickening process, high quality Nb films on small sample coupons could be reliably produced.

These experience suggested that, CED energetic condensed Nb films, growing in an epitaxy mode, might have a good SRF performance.

Albeit small samples yielded impressive results, it's earnest to up-scale such epitaxy coating technology to a SRF cavity dimension.

To provide a crystalline substrate for homo-epitaxy growth, the simplest available SRF cavity, is a 1.5 GHz single cell. These cavities are widely utilized in Jefferson Lab for SRF R&D.

In this series of work, four previously tested SRF single cell cavities received (or will do immediately) Nb film coating at AASC. Table 1 shows the name and specification of the cavities. They are made of different bulk Nb materials.

Table 1. The SRF Single Cell Cavities for this Nb-Nb Coating Work

Cavity Name	Specs.	Note
CBMM-B1	1.5GHz SRF cavity. Large grain, bulk Nb	Received 2 RF tests. Nb Film was etched away for 2nd RF test.
RDT-5	1.3Hz SRF cavity. Fine grain, bulk Nb.	Received 2 RF tests. Before 2nd RF test, it was heat treated in oven to degas hydrogen.
C1	1.5GHz SRF cavity. Fine grain, bulk Nb.	Failed. Heater melt-down.
C5-C6	1.5GHz SRF cavity. Fine Grain, bulk Nb.	Waiting for new heaters

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Fig. 1 is an illustration of Nb/Nb film coating on a bulk Nb cavity. It's a cross section view to depict interface and surface of Nb homo-epitaxy film. Nb grain size, orientation will be gauged by the underlying bulk Nb crystals. The "mold effect" of homo-epitaxy growth shall determine the microstructure of a deposited Nb film.

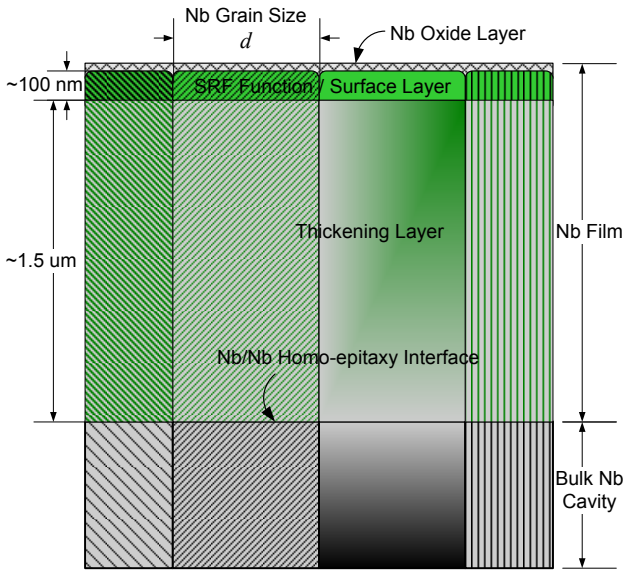

Figure 1: Illustration of proposed Nb/Nb film coating on a bulk Nb cavity. This is a cross section view to show interface and surface of Nb/Nb homo-epitaxy growth.

If a CED coating could achieve equivalent SRF performance as pristine bulk Nb cavities, in terms of Q_o and Q-slope, such evidence would strongly endorse the energetic condensation coating technology itself (opposing to a magnetron sputtering deposition) for future SRF film cavities. Good SRF Nb/Nb epi-film cavity results will also encourage more research or technical development on practical/low-cost Nb/Cu epi-film cavities. Essentially, current work on homo-epitaxial Nb/Nb film cavity is to glean fundamental knowledge for hetero-epitaxial Nb/Cu film SRF cavities.

It's known to SRF community, a Nb material will form an oxide layer naturally, when exposed to atmosphere. The oxide passivation layer is about few nanometres thick. Its microstructure has been thoroughly investigated in the past [5-8]. The outmost layer (to air side), is a glass-like randomly-networked pentoxide (Nb_2O_5) which has certain short-range orders. From air side to the deep bulk-Nb side, there are a variety of forms of NbO, NbO_2, NbO_xH_y *et al*. in sequential and complex microstructures, depending on how the oxide layer was formed. Closing to the Nb side, the Nb-O structure might be prone to be a long-range order, pesudomorphic structure of the underlying Nb lattice.

To achieve a Nb-Nb homo-epitaxial growth, one must remove the randomly-networked pentoxide (Nb_2O_5). While, for a long-range ordered NbO, which might mimic the crystal structure of underlying Nb grains, it might not be an issue as a template. As long as a Nb oxide has a crystal structure (a long range order, or a periodicity in atomic arrangement), the local pseudomorphic epitaxy of Nb/Nb or Nb/NbO/Nb may have a good structured interface to sustain a epitaxial growth mode.

To remove surface amorphous pentoxide, it was shown [5] that baking at 400^{0}C eliminates the surface oxide and all the oxygen diffuses deeper into the bulk. Figure 2 shows the removal rate, time and temperature relationship.

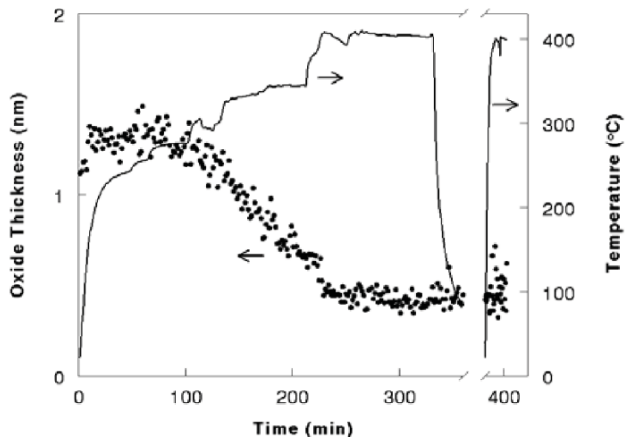

Figure 2: Nb oxide removal rate, time and temperature relationship, driven by a thermal process only [5].

In this study, we adopted the aforementioned thermodynamic principle to remove surface pentoxide. As stressed by Eremeev, baking at 400^{0}C is an ideal minimal baking temperature to largely remove some tenacious oxides. However, at the beginning of this work, AASC had no heating facility to reach such a temperature. It was limited at 350^{0}C. Thus, utilizing a H_2 glow discharge cleaning at 350^{0}C was a technical choice of expediency at that moment. A hydrogen glow discharge cleaning, plus a lower temperature baking was known as an efficient method to clean surface oxide. In retrospect, such a H_2 GDC might ruin the Nb coatings/cavities in vicious ways. For our earlier work on *CBMM-B1* and *RDT-5* cavities, a H_2 glow discharge cleaning (GDC, at $T_{cav.}$ ~350^{0}C) for 12 hrs (over night) was applied. For the work on *C1* and *C5-C6* cavities, no H_2 process is applied. AASC will be able to bake a Nb cavity at 400^{0}C.

EXPERIMENTAL RESULTS

Work on Cavity "CBMM-B1"

Cavity "CBMM-B1" single cell 1.5GHz SRF cavity is made of large grain bulk Nb material. This cavity has received the following film coating procedure at AASC.

- After mounting the cavity, pump down for ~1hr.
- Bake the cavity (at $T_{cav.}$ ~150^{0}C) for 2 days.
- H_2 GDC process (at $T_{cav.}$ ~350^{0}C) for 12 hrs. GDC condition is 500mTorr/600V/35mA.
- Coating Nb film (at $T_{cav.}$ ~350^{0}C) for 12 hrs.
- Stop heating the cavity, and start back-feeding 10%O_2/90%Ar for 2hrs.
- Take-out the cavity after cooling-down.

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Next, the cavity was returned to Jefferson Lab, and received a standard HPR cleaning. Then, the 1st round cryogenic RF test was conducted. The first RF test showed a very low Q_0 (Fig. 2) and a rapid quality factor degradation for increasing RF field.

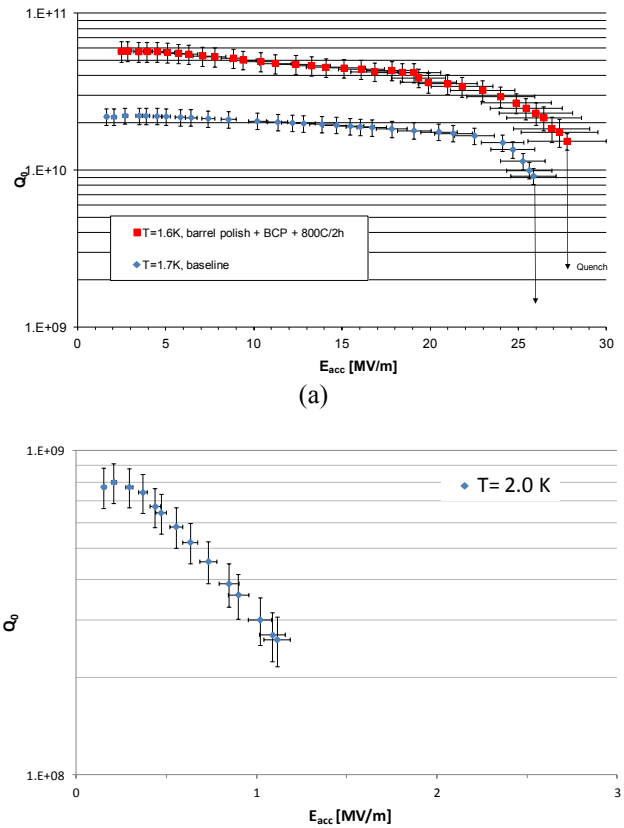

Figure 3: Q_o *vs.* E_{acc} plot of CBMM-B1 cavity, before (a) and after coating (b).

The test results also showed that the Nb film has a higher residual resistance (230 nΩ see Fig. 3), compared to that before coating (7.3 nΩ). Nevertheless, surface resistance vs. 1/*T* plot appears to still follow the BCS theory but with significantly reduced energy gap, possibly due to normal-conducting precipitates (Fig. 4).

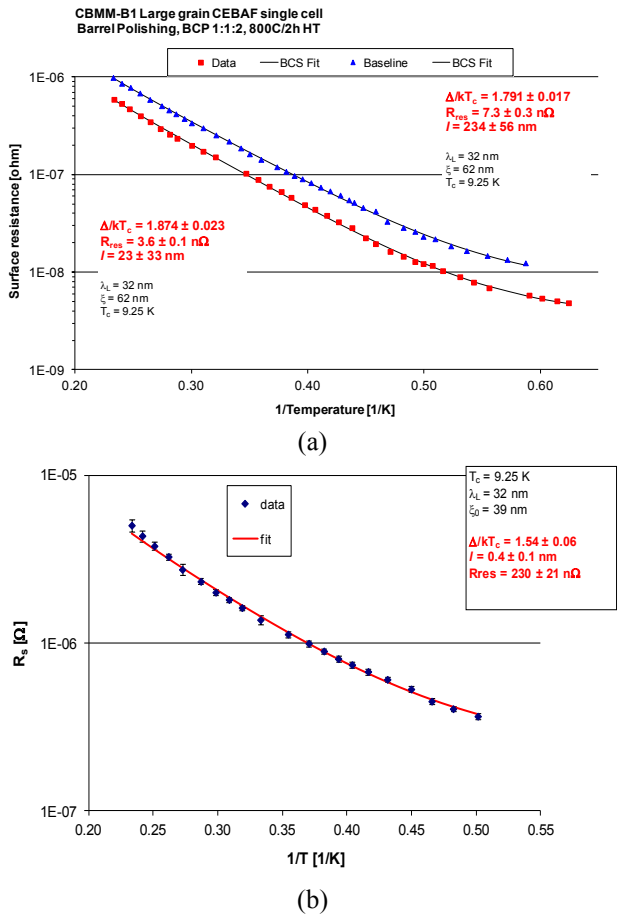

Figure 4: Surface Resistance *vs.* 1/T plot of CBMM-B1 cavity, before (a) and after coating (b).

We postulated it's either the hydrogen impurity being introduced during the H_2 GDC process, or defects of film microstructure, which caused the poor SRF performance. Thus, about 20 μm were removed from the inner cavity surface, hoping to recover a pristine BCP'ed bulk Nb surface

After the etching and cleaning procedure, the cavity received 2nd cryogenic RF test. Nevertheless, both the low Q_0 and drastic Q-slope have no improvement. Such phenomenon prompted us to believe hydrogen in-take during H_2 GDC is so deep, even the bulk Nb cavity might be fully loaded with hydrogen.

Work on Cavity "RDT-5"

Cavity "RDT-5" single cell 1.3 GHz cavity is made of a fine grain bulk Nb material. This cavity received a similar film coating procedure as "*CBMM-B1*" at AASC, except its GDC condition is 19mTorr/1.3KV/10mA

After coating, the cavity was returned to Jefferson Lab, and received a standard HPR cleaning. Then, the 1st round cryogenic RF test was conducted, and the results showed a very low Q_0 and a quick decay in Q-slope (Fig. 5).

We speculated it's also the hydrogen impurity being introduced by H_2 GDC process, which caused the poor

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SRF performance. Thus, the cavity was heat treated at 600^{0}C for 10 hrs.(with covering and caps), trying to degas hydrogen.

After the heat-treatment, the cavity received 2nd cryogenic RF test. Q_0 lifted up to a value close to the one before Nb film coating. Nevertheless, the Q still decreased quickly.

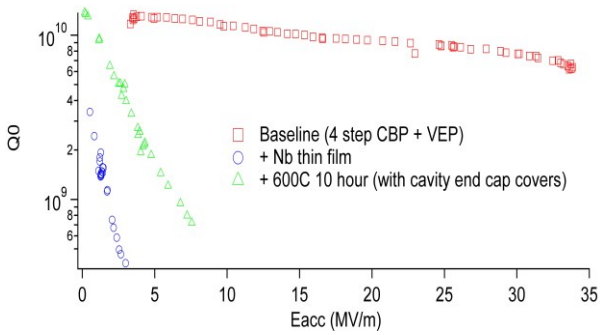

Figure 5: Q_o *vs*. E_{acc} plot of RDT-5 cavity, 1st RF Test (blue circles) and 2nd RF Test (green triangles).

Work on Cavity "C1"

Cavity "C1" single cell 1.5 GHz cavity is made of fine grain bulk Nb material. During film coating procedure (setting $T_{cav.}$ to 400^{0}C), the heater made of Aluminium materials was melt-down. The lava of Al covers up a beam-pipe's opening (flange). Such failure made the cavity impossible to receive a RF test. Furthermore, its inner wall might have embedded with Al impurities. Thus, its RF test was cancelled, but materials analysis on inner wall of this cavity might be conducted in the future.

Work on Cavity "C5-C6"

Cavity "C5-C6" single cell 1.5GHz cavity is made of fine grain bulk Nb materials. It has been shipped to AASC and is waiting to be coated. AASC is purchasing a new heater set in order to achieve mini T_{cav} up t0 400^{0}C

SUMMARY

We are working on homo-epitaxial Nb/Nb film cavities in purpose of understanding challenges in hetero-epitaxial Nb/Cu film SRF cavities. Four SRF single cell cavities are set in our work. We are gradually sorting out technical problems in order to find out a base-line SRF performance of CED energetic condensation Nb films. More materials analysis will be conducted.

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