Nb thin films at IPJ in 2008

1. **Nb deposition on various surfaces**
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1. Roughness measurements for samples

We have studied the influence of surface roughness an the Nb film adhesion.
Surface roughness was measured with a mechanical profilometer. The tip probed the surface along straight lines in a distance of 1 cm. The tip track lines were oriented in two perpendicular directions. The accuracy in the out-of-plane direction of in a the single scan measurement was better than 0.05 μm. Detailed information is presented in Tab. 1. Due to random character of the surface roughness, differences in roughness measured for substrate scanned in two perpendicular directions were recognized as error range of roughness value originated from the statistical sense of roughness.

<table>
<tr><th rowspan="2">Sample</th><th rowspan="2">preparation methods</th><th colspan="2">substrate surface roughness rms [μm]</th><th rowspan="2">film surface roughnes s rms [μm]</th></tr>
<tr><th>→</th><th>↑</th></tr>
<tr><td>1</td><td>electro-polished</td><td>0.03</td><td>0.06</td><td rowspan="5">1.5 ±0.4</td></tr>
<tr><td>2</td><td>polished rot. speed 1200 s^{-1}0</td><td>0.03</td><td>0.08</td></tr>
<tr><td>3</td><td>electro-polished</td><td>0.10</td><td>0.23</td></tr>
<tr><td>4</td><td>polished rot. speed 900 s^{-1}</td><td>0.65</td><td>1.26</td></tr>
<tr><td>5</td><td>polished rot. speed 450 s^{-1}</td><td>1.26</td><td>1.26</td></tr>
</table>

Tab.1

2. Nb deposition on various surfaces

Niobium films were deposited on the copper plates. In order to study the influence of roughness on the Nb adhesion to Cu, the substrate was prepared for an exposure in different polishing procedures resulted in various surface roughnesses (see part 1).
The Nb films were deposited during 2-minutes arc discharges without micro-droplets filtering, but with the ion accelerating (bias) voltage of -70 V, which resulted in Nb^{3+} energies ranging up to about 400 eV. Before the deposition process, the substrates were kept at the

room temperature. During the process, they were heated up to 120 ºC in a few seconds by the 70 A arc current.

3. Pressurized water rinsing

In order to test the adhesion of deposited film a high pressure water rinsing was implemented. A standard compressor was used to provide the water flux with a pressure up to 120 bar. The Pressure at the sample surface was estimated within the accuracy of 20 % by measuring the distance between the nozzle and the sample

4 Citric acid treatment

A cleanliness of the surface was improved by applying an additional pre-treatment before the deposition. The cavities were cleaned with 15% citric acid solution directly prior to evacuation. The bath influence on the adhesion was tested with samples of polished copper plates. The Nb layer having the thickness of about 5 μm was deposited in condition described in part 2.
An adhesion was tested with a pressurized water. Results showed that bathed and polished plate can survive 100 bar water rinsing.

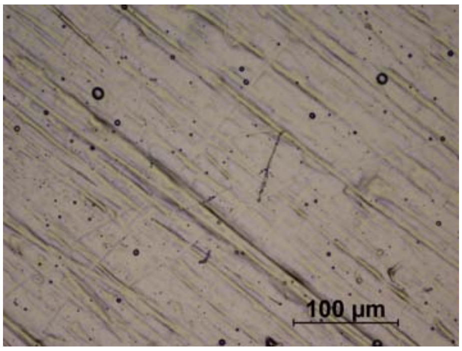

Fig. 1 Sample no 3 before HPWR

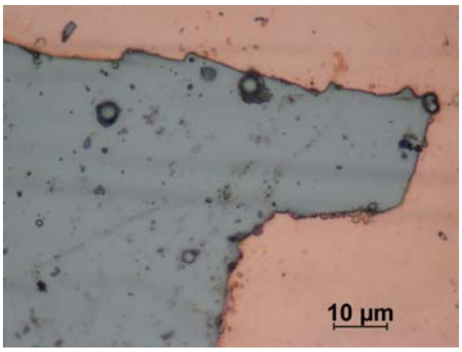

Fig. 2 Sample no 3 after HPWR at 80 bar

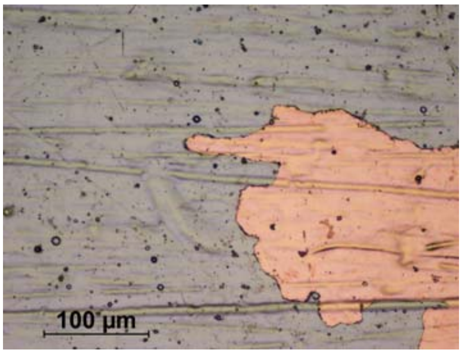

Fig. 3 Sample no 3 after HPWR at 80 bar

Fig. 4 Sample no 1 after HPWR at 80 bar

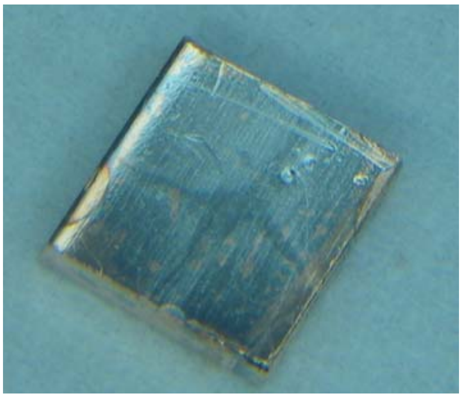

Fig. 5 Sample no 3 after HPWR at 80 bar

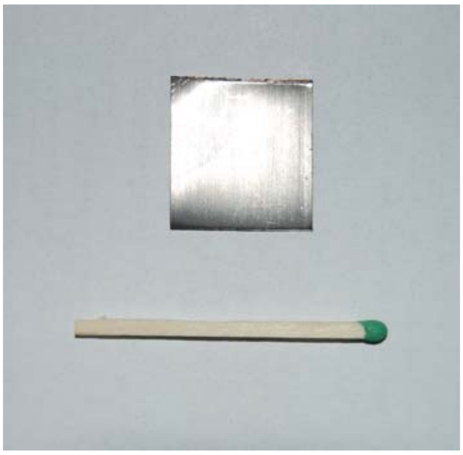

Fig. 6 Rough copper plate coated with Nb after HPWR at 80 bar.

It was observed that the substrate roughness enhances the adhesion, and that the appropriate substrate preparation affords the possibility to obtain the Nb film resistant to 100-bar rinsing for the most smooth substrates.

5. **Deposition onto dummy triple cavity**

The triple Tesla-like cavity was treated in a way similar to samples described above.
A cavity was filled with a 15% citric acid solution and kept annealed in 60 ºC to avoid the precipitation. Next, the cavity was flushed with deionised water and propanol consecutively. Such prepared cavity was further treated in N_2 atmosphere. It was mounted to the deposition facility, evacuated and 14 hours annealed. at 120 ºC – 140 ºC.
The deposition has been carried out at base pressure in the range of 10^{-9}mbar, it rised up to 10^{-6} range during the arc discharge in the chamber. A Nb layer was deposited in turns of the steering magnet, performed in the total time of .25 minuts. The cavity temperature measured at the outer side of the wall has never exceed 190 ºC.

Adhesion test performed after the deposition showed that the film peeled off, when exposed to 80 bar water rinsing (Fig.2)

Fig. 7 Inner walls of dummy triple cavity after the deposition.

6. Deposition onto Tesla-like cavity

Basing on the experience taken with the previous dummy and real cavities, we performed a deposition onto the inner walls of the Tesla-like cavity provided by DESY MHF laboratory, Before and during the deposition, the cavity was treated in a way similar to that described for the dummy triple cavity. In order to enhance the Nb penetration to the copper in the first stage of deposition, the cavity was annealed at temperature of 150 °C prior the processed started . The deposition started at this temperature. During the process, it was kept at the temperature range from 90 up to 190 °C . That was accomplished by the reduced cooling. Unfortunately, due to the strain caused by the temperature, one of ceramic insulators, broke during the deposition and the cavity has been vented. The pressure rose up to the range of 10^{-3} mbar, when the large part of cavity was in the temperature greater than 150 °C. We have found from our studies of Nb oxidation studies reported in 2007, that such condition result in formation of outer layer Nb_2O_5 on the Nb film.

7. Structural studies of the Nb layers

In order to study the crystalline structure of Nb films, a series of samples have been prepared. Films having thickness in the range 50 – 150 nm have been deposited onto single crystal sapphire and electro-polished copper substrates.

The analysis of the preliminary x-ray diffraction measurements were performed at the DORIS synchrotron in Hasylab at DESY. Obtained first results indicated the presence of two distinguished niobium phases in the film. The studies are going to be continued in the frame of dedicated project already accepted for the period of 2008 -2011.

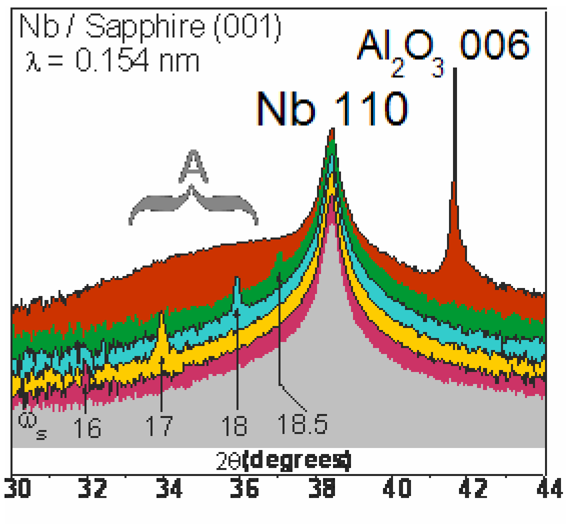

Fig. 7. Nb110 reflection measured for Nb/ sapphire film. The upper pattern represents results of symmetric ω - 2θ scan. Two components of peak profile are clearly seen. The other patterns were measured in detector scan for a fixed incidence angle showed in Figure.

8. References

[1] Structure of Nb Films Deposited by Means of Ultra-High Vacuum Cathodic Arc Technique, R. Nietubyć, J. Pełka, M.J. Sadowski, and P. Strzyżewski, Proceedings of the International Conference on Research and Applications of Plasmas, 16 – 19 October 2007, Greifswald, Germany. AIP Conference Proceedings 993, p. 415, 2008

[2] X-ray diffraction studies of the structure of Superconducting Films Deposited by Ultra-High Vacuum Cathodic Arc Technique, R. Nietubyć, M. Kuk, R. Mirowski, M.J. Sadowski, A. Trembicki, J. Witkowski, oral presentation on XXIIth IEEE-SPIE Joint Symposium on Photonics, Web Engineering, Electronics for Astronomy and High Energy Physics Experiments, 25.05 – 1.06, 2008, Wilga, Poland.

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