EXPLORING THE EFFECT OF Al_2O_3 ALD COATING ON A HIGH GRADIENT ILC SINGLE-CELL CAVITY*

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Abstract

Encouraged by work at Argonne National Lab, we investigated the atomic layer deposition technique (ALD) for high gradient superconducting RF cavities at JLab with an ALD coating system of Old Dominion University located on the JLab site. The goal of this study was to look into the possibility of coating a dielectric layer on top of an RF niobium surface at a lower temperature of 120 °C as compared to ANL coatings at 200 °C to preserve niobium pentoxide on the niobium surface. The initial coatings showed complete, but non-uniform coatings of the surface with several areas exhibiting discoloration, which was probably due to the temperature variation across the cavity surface. The initial coating showed high RF losses, which were improved after discolored areas on the beam tubes were removed with HF rinse of the beam tubes only. The best result was $2{\cdot}10^9$ low field Q_0 and E_{acc} = 18 MV/m limited by available power.

INTRODUCTION

A group at Argonne National Laboratory pioneered the use of the atomic layer deposition (ALD) technique for RF niobium cavities. At the 2007 SRF material workshop at FermiLab, M. Pellin made a presentation that proposed to use AL_2O_3 as an insulation layer that would replace the natural niobium oxide layer and prevent impurity adsorption and diffusion. As a long term goal a multilayer development towards higher sustainable magnetic field was proposed [1]. The group followed up in collaboration with several other labs to coating of several SRF cavities, and T. Proslier reported that SRF cavities coated with Al_2O_3 has retained high quality factors and achieved high fields. In some cases after heat treatment at 300–400 °C cavities had higher quality factor by as as much as a factor of two as compared to the baseline tests [2].

Results achieved by the Argonne group with ALD coated niobium cavities encouraged us to investigate Al_2O_3 coatings onto a high gradient SRF cavity using an existing ALD coating system located on the JLab site. The coatings at Argonne were done at 200 °C, which is in the center of the Al_2O_3 ALD coating window. However, 200 °C is the temperature high enough to cause dissociation of Nb pentoxide, and heat treatments at this temperature have been shown to adversely affect niobium cryogenic RF resistance at high field [3, 4]. Instead of 200 °C, we decided to deposit Al_2O_3 with ALD at 120 °C, the temperature commonly used for so-called "mild baking" of SRF cavities. As the first step our goal is to coat a high gradient niobium cavity with Al_2O_3 by ALD without degrading cavity properties. Due to ALD system availability and other resource constraints only two Al_2O_3 ALD coatings on SRF cavity have been performed.

EXPERIMENTAL SETUP

ALD process is a chemical vapor deposition technique that has the appealing ability of producing conformal thin films with desired thickness. The physics of the process has been described in the literature and will not be reiterated here [5]. For sample experiments a 2 inch diameter tube with 2 3/4" conflat flanges was used as the sample test chamber. Heat tapes were used to establish the temperature and Al foils were used for thermal insulation. One of the flanges of the sample test chamber through a valve is connected to a kwik-flange tee. The tee is connected to the pumping line and to a pressure gauge that supplies pressure information to the Cambirdge NanoTech Inc. control unit which in turn controls N_2 flow into the chamber. The other flange of the sample test chamber is connected to a mini-conflat tee. Tee flanges are connected to a N_2 supply and through soleniod valves controlled by Cambridge conrol unit to gas supplies. For deposition of Al on activated surface Trimethilaluminum, 97%, from Aldrich was used. For oxidation of deposited aluminum, water vapors from a bottle with ultrapure water were used.

Because of degradation of niobium cavities at temperatures higher than 150 °C and since 120 °C baking is a standard procedure for ILC cavities we looked into using the benign temperature of 120 °C. As the first step we measured the depostion rate on sapphire as a function of temperature in the 100–150 °C range.

A two inch sapphire sample was broken into four pieces and coated at four different temperatures. After coating, samples' thicknesses were measured with elipsometry. Table 1 summarizes the elipsometry measurements.

As we can see from the table, although 100–150 °C

Table 1: Ellipsometry Measurement Summary

coating temperature	Al_2O_3 thickness
100 °C	196.4 A
115 °C	222.5 A
135 °C	232.8 A
150 °C	240.9 A

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is not in the temperature-independent coating window, the average non-uniformity of the film assuming 10 °C temperature spread, typically achieved with heat tapes, is 8.9 Angstrom, less than 1 nm.

After preliminary measurements on sapphire, a niobium sample was coated with alumina. After the coating the sample had a characteristic yellowish collor. The thickness of the coating could not be determined with elipsometry since the fine grain samples were too rough for elipsometry. We performed several SEM/EDX measurements. In Fig. 1 we present EDX data on a coated fine grain RRR niobium sample for two different electron beam energies, 15 and 28 keV. The data is consistent with a thin alumina layer on top of niobium.

After EDX/SEM measurements the sample was annealed at 600 °C for 10 hours. After annealing the sample still had a characteristic yellowish color. EDX data supported presence of Al_2O_3. Next the sample was

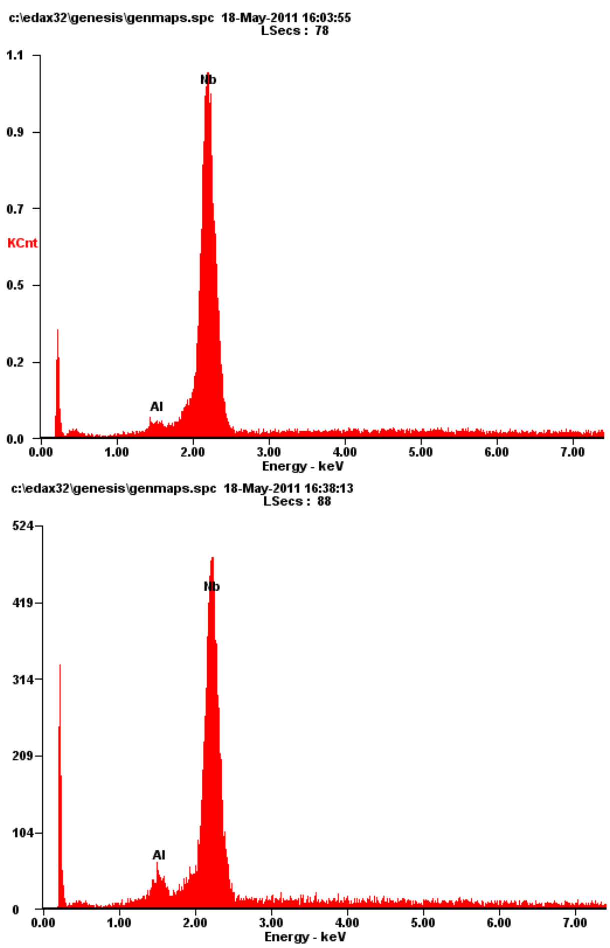

Figure 1: EDX data on Al_2O_3 coated sample. The top picture shows the EDX data for 28 keV electron beam; the estimated amount of Al is about 7 atomic percent. The bottom picture shows the EDX data for a 15 keV electron beam; the estimated amount is about 20 atomic percent for Al. The estimation dependence on the energy of the beam supports the coated alumina on top of niobium.

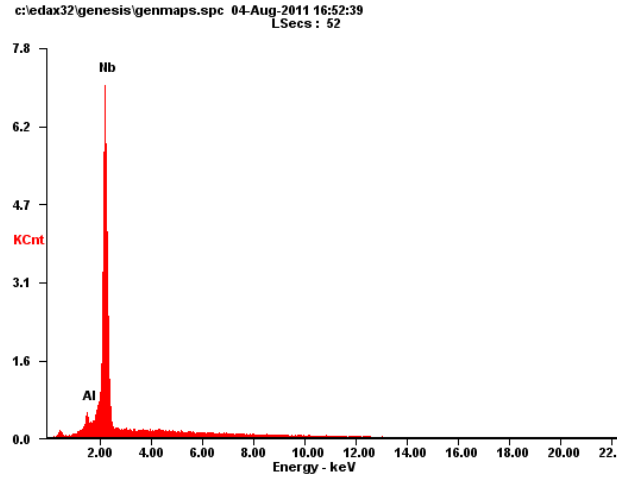

Figure 2: EDX data on Al_2O_3 coated sample after 800 °C × 2 hours heat treatment. In the picture the EDX data for 15 keV electron beam is shown. The estimated amount is about 6 atomic percent for Al.

annealed at 800 °C for two hours and was again analyzed with SEM and EDX. After the heat treatment the sample still had the same color. EDX data shows presence of aluminum supporting a layer of alumina on the surface, Fig. 2.

EXPERIMENTAL RESULTS

After samples studies a cavity has been prepared for ALD coating of alumina. TE1AES001 has been vertically electropolished in the past, heat treated at 800 °C, high pressure water rinsed, and assembled in class 10 [6]. The cavity reached above E_{acc} = 40 MV/m in the vertical test at T_{bath} = 2 K. Following the test the cavity was re-tested several times with and without thermometry, consistently reaching around E_{acc} = 40 MV/n. After the baseline the cavity was disassembled and set up for ALD coating. In Fig. 3 we show TE1AES001 placed on the support fixtures and connected to the pump down line and gas supply lines.

After the coating the cavity had a charcacteristic yellowish color on the inside, Fig. 4, although we observed on the beam tubes some discoloration that had cloudy white color.

After the coating the cavity underwent the standard cleaning and preparation procedures. During one of the standard preparation steps for high pressure rinse the cavity was degreased with micro-90. After the degreasing we observed change in the cavity color in some areas on the inside. Most of the surface had the natural niobium color without any traces of alumina. Discoloraton observed on the inside surface suggested heavy oxidation.

Since the goal of the project was to study ALD coatings in RF fields, and the treatment has produced uncontrolled varation which was not investigated on samples, we decided to remove the coating with HF rinse, re-baseline the cavity, and do the ALD coating again. After HF rinse there was no discoloration on the cavity surface.

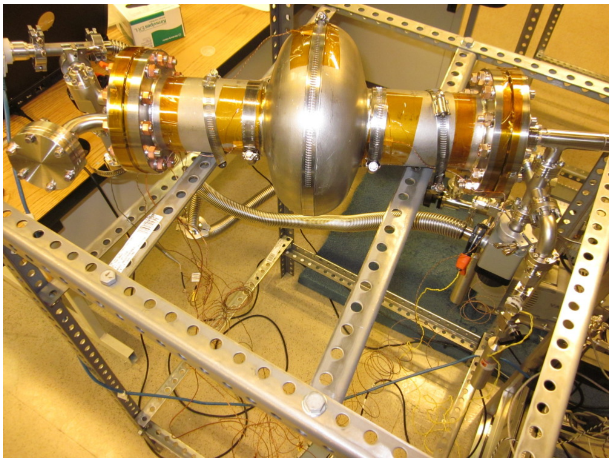

Figure 3: TE1AES001 setup for ALD coating.

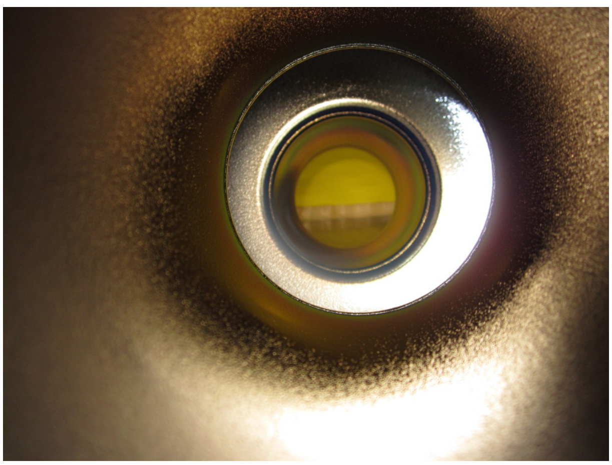

Figure 4: Cavity surface after about 20 nm Al_2O_3 coating. The surface has a characteristic yellowish color.

The cavity was assembled and tested at 2 K. The cavity had the same quality factor and was limited at the same field level as in the test before ALD coating. The cavity also had similar field emission level and lorentz detuning coefficient. After the baseline has been reestablished, the second ALD coating was done. After the coating the cavity had characteristic color. Discoloration observed in the previous run was present again on one of the beam tubes. After the coating the cavity was HPRed, assembled, and measured at 2 K. The quality factor of the cavity was measured to be of the order 10^8. Because of the significant coupling mismatch we were not able to measure quality factor as a function of field. We speculated that the discoloration that we observe on the beam tubes can be Al enriched coating that is responsible for the quality factor of the cavity. In order to improve the quality factor the beamtubes of the cavity were HF rinsed, while the RF cell was not rinsed with HF. After beam tubes HF rinsing the cavity HPRed, assembled, and prepared for RF test at 2 K. The low field Q was about 2E9 at low field. The highest field reached was 18 MV/m with Q of about 1.3E9. The test was limited by the available RF power, Fig. 5.

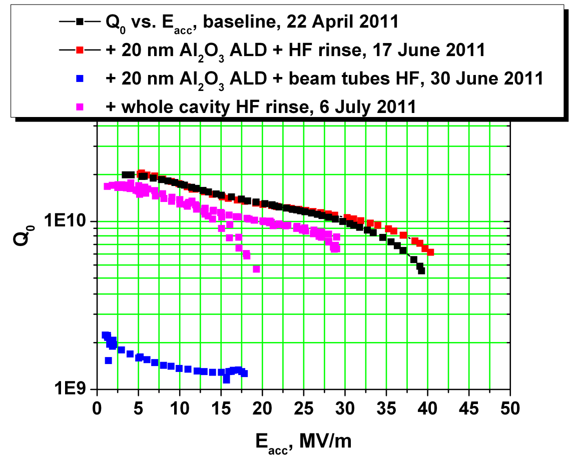

Figure 5: Summary of the test results. Black squares present baseline result. Red squares show test result after the first coating and HF rinse. Blue squares show test result after the second coating and HF rinse of beam tubes. Magenta squares show test result after whole cavity HF rinse.

CONCLUSION

Encouraged by work at Argonne National Lab, we investigated the atomic layer deposition technique (ALD) for high gradient superconducting RF cavities at JLab with an ALD coating system of Old Dominion University located on the JLab site. The goal of this study was to look into the possibility of coating a dielectric layer on top of an RF niobium surface at a lower temperature of 120 °C to preserve niobium pentoxide on the niobium surface. The initial coatings showed complete but non-uniform coatings of the surface with several areas exhibiting discoloration, which was probably due to the temperature variation across the cavity surface. The initial coating showed high RF losses, which were improved after discolored areas on the beam tubes were removed with HF rinse of the beam tubes only. After two attempts the best result was $2{\cdot}10^9$ low field Q_0 and E_{acc} = 18 MV/m limited by available power.

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