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PLASMA-ENHANCED ALD SYSTEM FOR SRF CAVITY

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Abstract

A remote PEALD (Plasma-enhanced Atomic Layer Deposition) system which would offer a high conformality and a low deposition temperature has been being developed in KEK to aim deposition of multilayer systems like a NbN/Al_2O_3 multilayer on an SRF cavity. The deposition equipment was designed to carry out ALD not only on planar silicon wafer substrates in a reaction chamber but also on inner surface of a 1.3GHz single cell cavity just by replacement of reaction chamber with the cavity. An RF plasma exciter was utilized for an ALD reactant gas in order to reduce ALD temperature. Prepared precursors were tris [ethylmethylamido][tert-butylimido] niobium (TBTEMN) and trimethylaluminium (TMA) with reactants of ammonia, hydrogen and water for NbN and Al_2O_3 deposition. Surface analyses confirmed preliminary results of NbN_x ALD with homogeneous and smooth film growth and a GPC (growth per cycle) of around 0.1 nm. However noticeable concentration of oxygen and carbon found in the film requires further improvements of the ALD system and the operation conditions.

INTRODUCTION

To enhance a breakdown magnetic field in a superconducting radio frequency (SRF) cavity, a nano-scaled multilayer system of combination of high Hc superconducting material and insulator was proposed [1-3]. A remote PE-ALD (Plasma-enhanced Atomic Layer Deposition) system [4] was developed in order to deposit multilayers on planar substrates or on an inner surface of a SRF cavity with a high conformality, a low deposition temperature and a low density of contaminants [5]. Recently PE-ALD for NbN formation was also tried with a commercially available equipment from Oxford Instruments [6, 7]. A type of remote plasma would bring a better conformality in comparison with a type of non remote plasma even for a large coating area with a longitudinal configuration like a 1.3GHz 9-cell SRF cavity. In this study, we present our remote PE-ALD system design and results of our first deposition of NbN_x on samples which were analyzed with a scanning electron microscope (SEM), energy dispersive x-ray spectroscopy (EDX) and x-ray photoelectron spectroscopy (XPS) with sputter-etching.

EXPERIMENT

Our in-house made ALD system consists of a reaction chamber, a remote plasma exciter, a precursor supply system, vacuum pumps, a quartz crystal microbalance (QCM) as a film growth rate meter, a detoxifying system and a control sequencer shown in Fig. 1 [4]. The substrate holder can be heated up to 600 ℃ and the almost whole system can be baked out up to 200 ℃. An RF frequency of 13.56MHz was used for the inductively coupled plasma exciter of a reactant gas. The whole equipment is in a draft booth for operation safety. Though the reaction chamber accepts only coupon samples including a silicon wafer substrate up to 2”, the ALD system allows to easily replace the reaction chamber with a single cell niobium cavity when ALD conditions are well found.

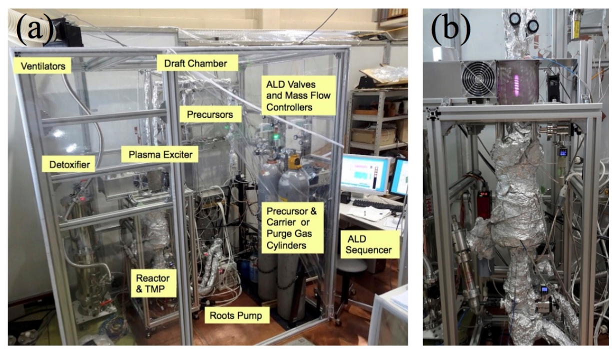

Figure 1: (a) ALD System for SRF Cavity in KEK (b) zoomed-in 13.56MHz plasma exciter and reaction chamber.

The precursors for this study are tris [ethylmethylamido][tert-butylimido] niobium (TBTEMN) with ammonia or hydrogen gas as the reactants.

Typical ALD conditions are the following:

* Precursor: TBTEMN
* Reactant: NH_3 (6 SCCM) + Ar (1 SCCM)
* RF Power: 50 W / 1 min / cycle
* Cycle: ~4 min
* Substrate temperature: 150 ºC

Deposited films on silicon substrates were analysed with SEM-EDX and XPS with sputter-etching capability to know film morphology and its atomic compositions.

RESULTS AND DISCUSSION

SEM images of a 200 cycle film and a 300 cycle film on silicon substrates are shown in Fig. 2 (a) and (b), respectively. Thickness of each film was measured to be around 20 and 35 nm with SEM observation. This results in a GPC (growth per cycle) of around 0.1 nm. It was found that the ALD films were quite homogeneous and the surfaces were smooth. To check conformity of our ALD, surfaces of a couple of silicon substrates were set in the reactor with different angles. The all samples showed a similar deposition thickness.

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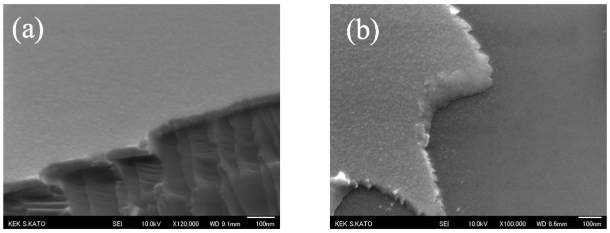

Figure 2: SEM images of 200 cycle film (a) and 300 cycle film (b) on silicon substrates.

Figure 3 shows SEM images of the 200 cycle film and the EDX spectra of the film and the film-removed substrate. While NbN_x film growth was found, intensities of carbon and oxygen were also pronounced. Even in Fig. 3(a), silicon peak is very strong. It is explainable that a large information depth of EDX causes a large signal of silicon through the very thin film of NbN_x. In Fig. 3(b), one can observe niobium peak. This would be because of remaining niobium at the interface between the film and the silicon substrate when the film was mechanically removed from the substrate.

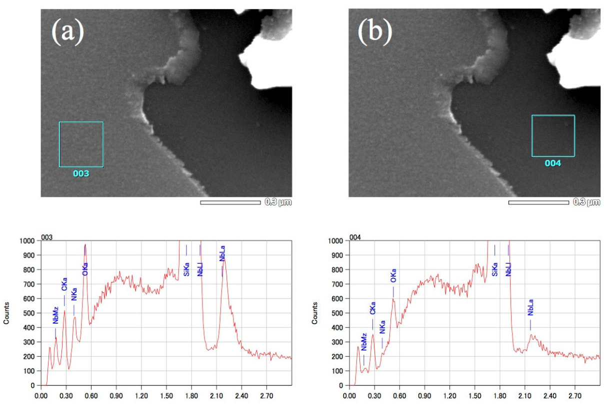

Figure 3: SEM images of the 200 cycle films and those EDX spectra which correspond to area scans shown in blue squares at the film (a) and at the film-removed substrate (b).

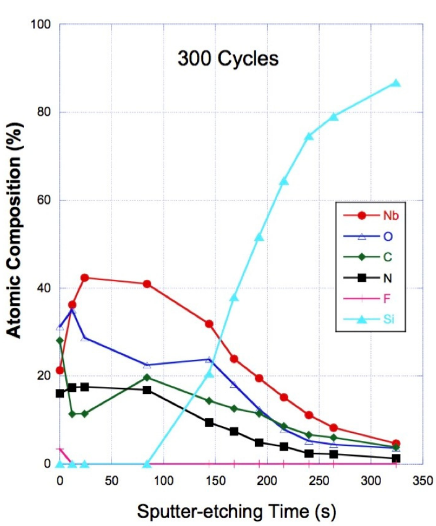

Figure 4: XPS depth profiling of 300 cycle NbN_x film.

As shown in XPS depth profiles of a 300 cycle NbN_x film (Fig. 4), a N/Nb ratio of about 0.5 in the film measured with XPS showed a nitrogen poor film since more niobium oxide and rich free carbon were found (note that 5% of carbon composition is attributed to XPS poor vacuum). Oxygen in the film might be attributed to insufficient water degassing from the reactor and the gas tubes. A wide interface between the film and the Si substrate in the XPS depth profiles is due to atomic mixing by a 5 keV Ar^+ ion beam during the sputter-etching. RGA (Residual Gas Analyser) observation during ALD implies poor removal of hydrocarbon from adsorbed TBTEMN. This can be probably improved with increase of the plasma power and the optimized substrate temperature in order to remove carbon in a deposited film.

As a next step, optimization of ALD process is mandatory to reduce oxygen and carbon concentration in the film and Tc/Hc measurements of planar samples will be performed. Then we have a plan to carry out NbN/Al2O3 multilayer formation on samples in the ALD reactor subsequently on niobium single cell coupon cavity by simple replacement of the reactor with the coupon cavity as shown in Fig. 5 and analyses of its coupons. Finally, multilayer deposition on a niobium single cell cavity will be done for its VT.

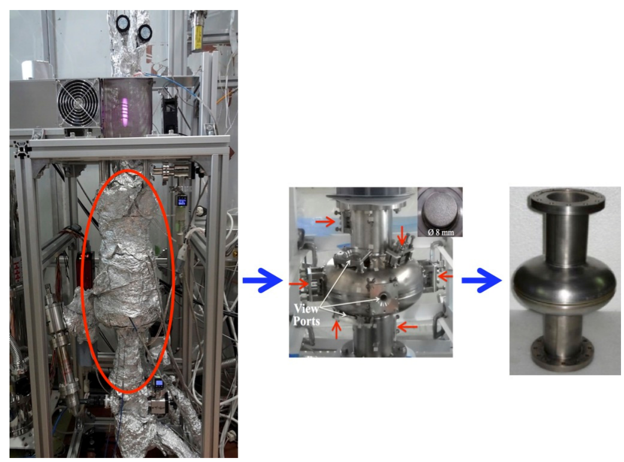

Figure 5: Simple replacement of the ALD reactor with a niobium single cell coupon cavity or a niobium single cell cavity enables ALD on those.

CONCLUSION

Remote PE-ALD system for SRF cavity was developed at KEK for the first time. Homogeneous and smooth film formation of NbN_x with TBTEMN and remote NH_3 plasma was confirmed with a reasonable GPC of 0.1 nm while the deposition condition should be further optimized to reduce oxygen and carbon and to obtain a stoichiometric NbN film.

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