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ALD-BASED NbTiN STUDIES FOR SIS R&D

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Abstract

Superconductor-Insulator-Superconductor (SIS) multilayers improve the performance of superconducting radio frequency (SRF) cavities providing magnetic screening of the bulk cavity and lower surface resistance. In this framework NbTiN alloys stand as a potential material of interest. Atomic layer deposition (ALD) allows for uniform coating of complex geometries and enables tuning of the stoichiometry and precise thickness control in sub-nm range. HERE, we report about NbTiN thin films deposited by plasma-enhanced ALD (PEALD) on insulating AlN buffer layer. Post-deposition rapid thermal annealing (RTA) studies with varying temperatures, annealing times, and gas atmospheres have been performed to further improve the thin film quality and the superconducting properties. Based upon the promising results obtained by RTA, high vacuum annealing has been also investigated in order to stablish the recipe to obtain SIS multilayer for SRF cavities by plasma-enhanced ALD and subsequent annealing.

INTRODUCTION

Over the past decades, the RF performance of bulk Nb cavities has continuously improved with material and surface developments. Meanwhile we are approaching the theoretical limits of bulk niobium and long-term solutions for further SRF performance improvements need to be pursued. A potential solution was proposed by A. Gurevich [1], namely Superconductor – Insulator – Superconductor multilayers (SIS structures). The idea is to coat the internal surface of the SRF cavities with alternating superconducting and insulating layers taking in advantage of superconductors with higher Tc than niobium (see Fig. 1).

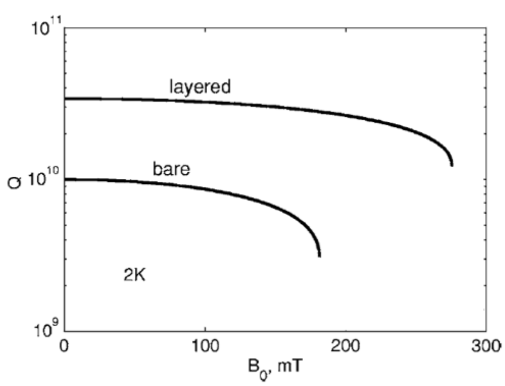

Figure 1: Q vs B curve calculated by A. Gurevich [1] for bare and layered cavity, which shows the significative improvement in the RF cavity performance due to the multilayer S-I-S coating.

The strong increase of Hc1 in films allows to operate at RF fields higher than the Hc1 of the bulk niobium cavity since the multilayers provide magnetic screening of the bulk cavity preventing vortex penetration. Moreover, the use of higher Tc superconductors reduces the surface resistance. Therefore, the SIS structures improve the SRF cavity performance, increasing the accelerating field and reducing the losses.

In this framework NbTiN alloys stand as a potential material of interest. While NbN has a high Tc of 17.3 K, it contains a high normal conducting resistivity, which is significantly reduced with the incorporation of Ti into NbN. NbTiN alloys can be deposited by physical vapor deposition (PVD), chemical vapor deposition (CVD) and atomic layer deposition (ALD). The latter provide conformal coatings on high aspect ratio structures at low deposition temperatures, which makes it particularly interesting for coating the internal surface of SRF cavities. The deposition of NbTiN by thermal ALD, generally using chlorinated precursors [2] can introduce chlorine contamination on the deposited films while plasma-enhanced ALD (PEALD) enables metalorganic precursors, improving the quality of the deposited films.

To further improve the superconducting properties of the NbTiN films post-deposition rapid thermal annealing (RTA) and high vacuum annealing have been studied.

EXPERIMENTAL DETAILS

We investigate the deposition of superconducting films of $Nb_xTi_{1-x}N$ grown on Si wafer by plasma enhanced atomic layer deposition (PEALD) using metalorganic precursors and H_2/N_2 plasma. The precursors used were (t-Butylimido)tris(diethylamino)niobium(V) (TBTDEN) and tetrakis(dimethylamino)titanium(IV) (TDMAT), which were maintained at 90 ºC and 70 ºC respectively. The deposition temperature was kept at 250 ºC and the plasma power at 300 W. The deposition process consists in an PEALD supercycle which alternates the PEALD cycles for the deposition of NbN (alternation of TBTDEN pulse and plasma exposure) with TiN cycles (alternation of TDMAT pulse and plasma exposure). Thus, the composition of the deposited $Nb_xTi_{1-x}N$ films can be modified varying the ratio of NbN cycles to TiN cycles run inside the ALD supercycle. Eight different Nb:Ti composition ratios were studied. As insulator the material selected was AlN since it enhances the Tc of NbTiN [3]. The precursor used was Trimethylaluminum (TMA) and was kept at room temperature. Both layers, AlN and NbTiN were deposited within the same PEALD process on Si wafer.

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After the deposition, rapid thermal annealing has been performed at 800, 900 and 1000 ºC varying the annealing duration between 5 and 50 min. The heating rate was 1 ºC/s and the pressure was 6.6 mbar. Different gas atmospheres (Ar/H_2 and $N_2/_{H2}$ mixtures, pure N_2 and pure H_2) were studied. The high vacuum annealing has been performed at 1000 ºC in a ceramic tubular furnace used for Nb doping studies. The heating rate was 200 ºC/h and the base pressure was 2.5 E-6 mbar mbar which reflects a standard annealing cycle already implemented for cavity treatment. The temperature was kept at 1000 ºC for 50 minutes with nitrogen flow and pressure of E-5 mbar.

RESULTS

Superconducting properties have been studied as a function of the ratio of Nb to Ti content present in the films (see Fig. 2).

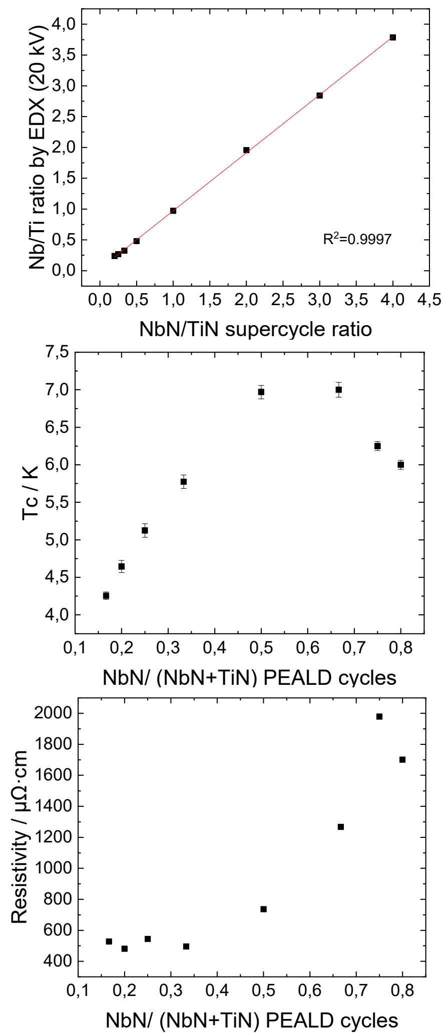

Figure 2: (From top to bottom) Linear relationship between the ratio of Nb to Ti concentration measured by EDX and the NbN to TiN ratio within the supercycle of PEALD. Variation of the critical temperature and resistivity for the different ratios of NbN to TiN supercycles during the PEALD process.

The film composition was analysed using EDX. The Nb:Ti composition ratio measured matches with the ratio of NbN to TiN within the PEALD supercycle, enabling efficient tuning of film composition and demonstrating the well-control of elemental composition of our films.

Post-deposition rapid thermal annealing (RTA) varying temperature, annealing time and gas atmospheres have been performed in order to study the influence of these parameters on the superconducting properties (see Fig. 3). Higher annealing temperatures show a larger increase of Tc. The effect of the annealing duration varies depending on the annealing gas atmosphere and needs to be studied more in detail.

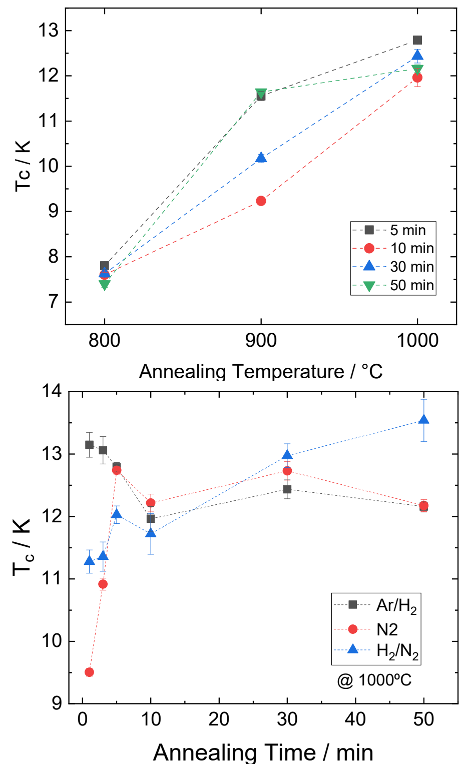

Figure 3: Effect of the post-deposition rapid thermal annealing on NbTiN film. (Top) Tc vs annealing temperature for different annealing times. (Bottom) Tc vs annealing time for gas atmospheres.

The RTA at 1000 ºC has been performed for the NbTiN layers with different compositions. Tc increased for all the films and reached a maximum value of 14.3 K for 25 nm $Nb_{0.75}Ti_{0.25}N$ films. The resistivity decreased for all the films after RTA (see Fig. 4).

Annealing in nitrogen at 1000 ºC has been studied in order to find a suitable annealing recipe for a coated cavity.

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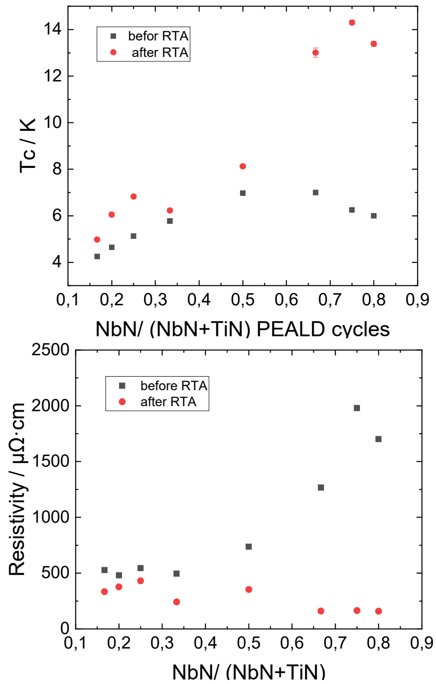

Figure 4: Variation of the critical temperature and the resistivity measured at room temperature for the different ratios of NbN to TiN supercycles during the PEALD process before and after RTA at 1000 ºC.

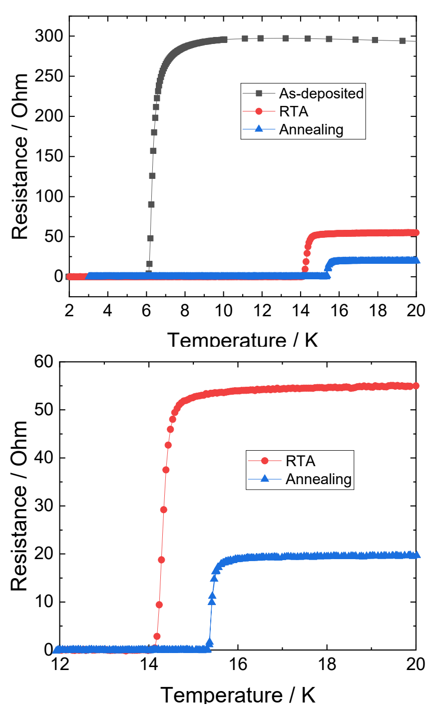

Figure 5: Superconducting transition for NbTiN films deposited by PEALD. (Top) As-deposited, post-deposition thermal annealing and post-deposition high vacuum annealing. The critical temperatures are 6.3 K, 14.3 K and 15.4 K. (Bottom) Zoom in at the RTA and high vacuum annealing. at 1000 ºC.

The results (see Fig. 5) show that slower ramping times and lower pressures continuing enhancing the superconducting properties of NbTiN layers deposited by PEALD, reaching a maximum Tc of 15.4 K for a layer of 25nm thickness.

CONCLUSIONS

Superconducting NbTiN alloys have been synthesized by PEALD and the effect on the Tc of their composition has been studied. Post-deposition annealing has been found to be mandatory in order to significantly improve the Tc of the deposited NbTiN films. Rapid thermal annealing (RTA) and high vacuum annealing with slower ramping rates have been studied. The results show an enhancement of the Tc and the resistance at low and room temperature for both kind of annealing, being better for high vacuum annealings. The foregoing results are an evidence that NbTiN layers deposited by plasma-enhanced ALD and subsequently annealed can be used for SIS multilayers SRF cavity applications.

Further analyses are being performed in order to understand the observed improvement.

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