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DEVELOPMENT OF COATING TECHNIQUE FOR SUPERCONDUCTING MULTILAYERED STRUCTURE

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Abstract

In order to increase the maximum acceleration gradient of SRF cavities, S-I-S (superconductor-insulator-superconductor) multi-layered structure theory has been proposed. We focused on NbN which has a higher superconducting transition temperature than Nb. Firstly, we researched the optimal deposition condition for N_2 gas reactive sputtering of NbN by using in-house inter-back type DC magnetron sputtering equipment. The critical condition for thin film with strong crystalline orientation of NbN was identified. The superconducting transition temperature of the NbN thin film, which was coated under the best condition, was over 14 K. Secondly, we tried making S-I-S multi-layered samples that were composed of NbN/SiO_2/Nb substrate. The coating condition for the NbN layer was determined based on the research results in a single layer. The SiO_2 layer was deposited with a film thickness of 30 nm that was theoretically expected to be effective as barrier layer. We applied O_2 gas reactive AC magnetron sputtering for coating. In this article, the detailed results of the NbN single layer and multilayer film depositions are presented.

INTRODUCTION

Acceleration gradient of Nb cavities is approaching the theoretical limit because technologies to fabricate these cavities have been advanced [1-2]. For that reason, S-I-S multi-layered structure theory has been proposed, and it is expected that the thermodynamic critical field will increase according to this theory [3-5]. However, until now, multi-layered cavities that actually exceed the critical field of Nb cavities have not been realized. Even if an ideal multi-layered structure could be fabricated, it is not clear whether the value of the critical field presented in this theory is really achieved. Thus, in order to realize the multi-layered cavities, it is necessary to establish a technique for coating high quality superconducting layer and insulating layer and a technique for evaluating the superconducting characteristics of the deposited film.

In this study, we aimed to establish a basic coating technique and process. Since NbN thin-film can be coated relatively easily by reactive sputtering and it is optimal for the evaluation of the critical field, we focused on it. We investigated the fundamental characteristics of the NbN films deposited under some coating conditions. The goal in this study is to clarify the correlation between coating parameters and fundamental NbN thin-films characteristics and to make S-I-S multi-layered samples that can achieve a critical field higher than Nb.

EXPERIMENTAL

We conducted NbN and SiO_2 thin-films coating experiments by using in-house sputtering apparatus. Our sputtering experimental apparatus adopts inter-back system, and a substrate carrier passes in front of Nb and two Si targets at a regular speed. Therefore, the sputtered elements are uniformly deposited on substrate. The base pressure of the sputtering chamber was about $2x10^{-4}$ Pa. Ar and N_2 (or O_2) gasses were introduced in the sputtering chamber, NbN or SiO_2 thin-films were reactively sputtered. The Nb target was powered by DC supply, and the two Si targets were powered by AC supply. In NbN coating, DC input power was set constant at 3.0 kW or 6.0 kW, and Ar partial pressure was 0.3 Pa, 0.6 Pa or 1.2 Pa. We searched optimal N_2 gas flow rate under the some input power and Ar partial pressure conditions in order to achieve high density, low defect, strong crystalline orientation, and high T_c. On the other hand, in SiO_2 coating, AC input power was set constant at 6.0 kW, and Ar partial pressure was 0.3 Pa. We also searched and decided applicable O_2 gas flow rate in the same manner as NbN.

SEARCH FOR COATING CONDITION

We first investigated how discharge voltage change by changing N_2 partial pressure while keeping Ar partial pressure constant. As a result, we found that there was a transition zone where the reactivity between Nb and N_2 gas significantly changes (Figure 1). In this zone, some fundamental sputtering characteristics and thin-film properties such as total pressure, film deposition rate, and film resistivity also significantly changed. In particular, the film resistivity showed a unique change and had a local minimum except under the condition of high pressure (Figure 2).

N_2 content of thin-film depends on N_2 gas flow rate. Before the transition zone, although nitrogen is incorporated into the film, the amount is insufficient to perfectly generate NbN. Therefore, the crystalline is distorted, and the resistivity increases. In the transition zone, NbN is generated with appropriate content, and the resistivity temporarily decreases. After the transition zone, the resistivity increases again because nitrogen amount is too much. Figure 3 shows XRD patterns of two $Nb_{1-x}N_x$ thin-films formed before or in the transition zone. These thin-films were coated under the condition of input power of 6.0 kW and

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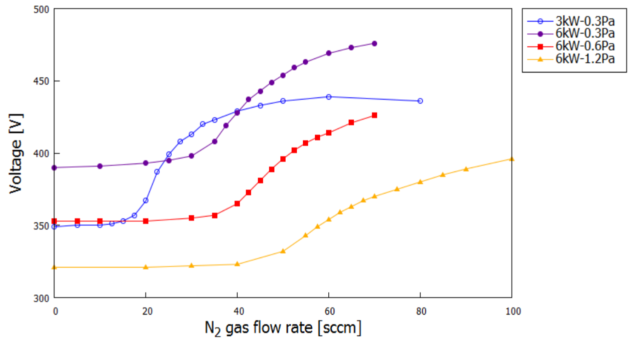

Figure 1: Discharge voltage for varying N_2 gas flow rate.

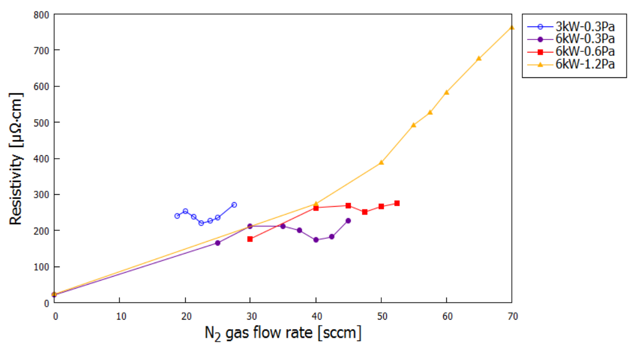

Figure 2: NbN thin-films resistivity at room temperature for varying N_2 gas flow rate.

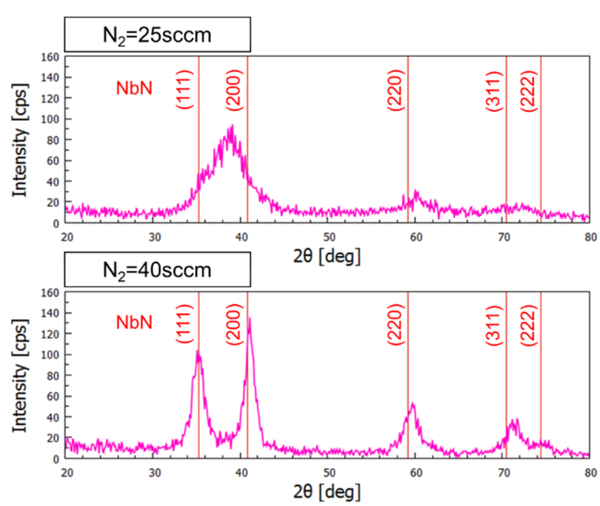

Figure 3: Comparison of two XRD patterns of NbN/glass samples measured by in-plane method. The upper is insufficient nitrogen content, and the lower is appropriate nitrogen content.

Ar partial pressure of 0.3 Pa. The peak position of the thin-film having the local minimum resistivity corresponds to NbN.

We decided the appropriate N_2 gas flow rate for other input power and Ar pressure conditions in the same way. Table 1 shows the coating conditions of the samples produced this time.

Table 1: Optimal N_2 Gas Flow Rate under Each Sputtering Parameters

	Input Power [kW]	Ar Pressure [Pa]	N_2 Gas Flow Rate [sccm]
A	3.0	0.3	22.5
B	6.0	0.3	40.0
C	6.0	0.6	47.5
D	6.0	1.2	65.0

PROPERTY INSPECTION

We coated NbN on Si wafer with a thickness of 200 nm under the conditions A to D shown in Table 1, and evaluated XRD, SEM, AFM, film density, film stress, and critical temperature. Figure 4 shows XRD patterns of these samples. The pattern of sample A particularly well matches the peak of NbN: FCC, NaCl structure. Figure 5 shows SEM images of sample B. This NbN film has a columnar structure and its surface is very flat. Table 2 shows film density, film stress, and T_c of sample A to D. Sample B is a dense film close to the theoretical value of bulk NbN: about 8.47 g/cm^3, but has a very strong compressive stress at room temperature.

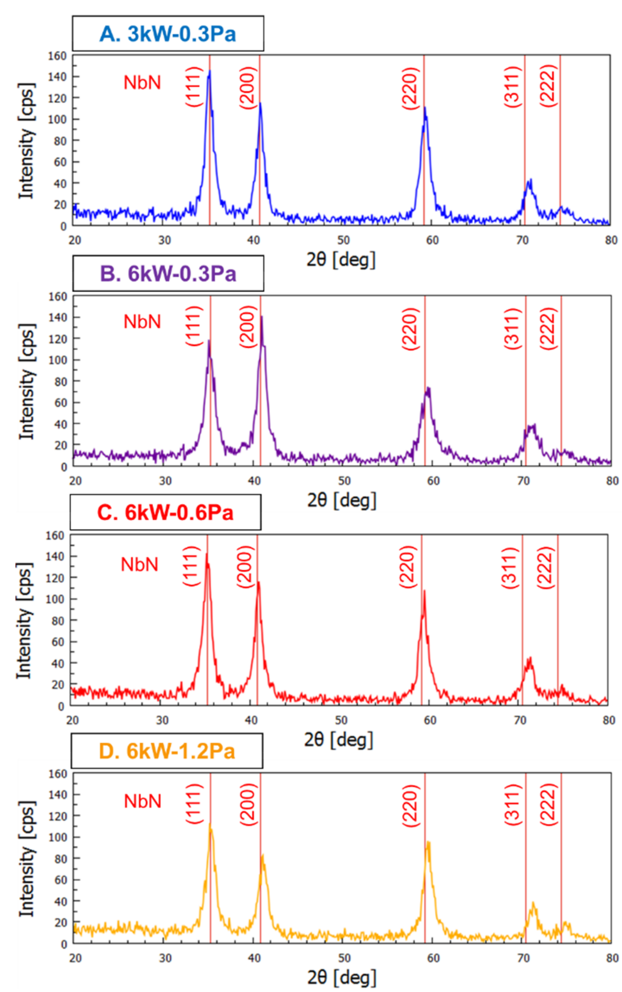

Figure 4: XRD patterns of NbN/Si samples measured by in-plane method.

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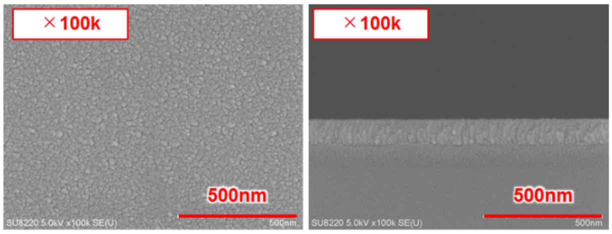

Figure 5: SEM images of the surface and the cross section of sample B. The surface roughness R_a is 1.379 nm.

Table 2: Film Density, Film Stress, and T_c

	Film Density [g/cm^3]	Film Stress [MPa]	T_c [K]
A	7.84	-492	14.4
B	8.38	-976	13.8
C	7.12	-79	13.6
D	6.15	-116	11.9

MULTILAYERED SAMPLE

We made S-I-S multi-layered samples which were deposited NbN/SiO_2 on bulk Nb substrates. Considering the inspection result of the NbN single layer, we selected the conditions A and B capable of coating a film with high T_c and low roughness. The thickness of NbN layer was about 200 nm. Table 3 shows the deposition lot number of the multi-layered samples and the NbN coating conditions. The notation "-A" or "-B" attached to the end of the lot name means that the film was coated with the same deposition lot but the Nb substrates were different from each other. SiO_2 coating condition was the same for all deposition lots, the O_2 gas flow rate was 90 sccm, and the thickness was about 30 nm. Figure 6 shows Nb substrates before and after multilayer coating. The Nb substrates were electro polished in advance. The sample after coating looks slightly darkened golden, keeping mirror surface as same as before coating.

Table 3: NbN Coating Parameters for the Multi-layered Samples

Lot No.	Input Power [kW]	Ar Pressure [Pa]	N_2 Gas Flow Rate [sccm]
180409-1-A, B	3.0	0.3	22.5
180409-2-A, B	6.0	0.3	40.0

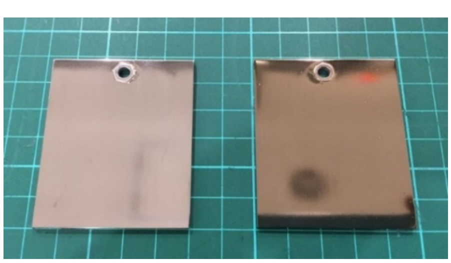
Figure 6: Electro polished Nb bare substrate (the left) and NbN/SiO_2 deposited Nb substrate (the right).

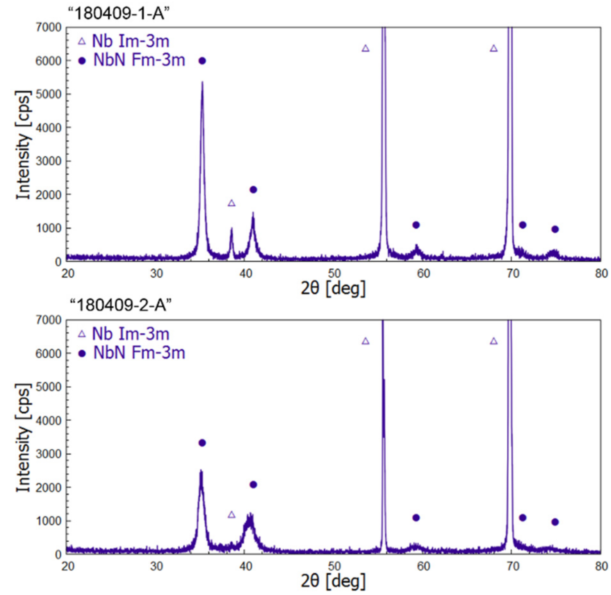

Figure 7: XRD patterns of S-I-S multi-layered samples.

Figure 7 shows XRD patterns of the NbN/SiO_2/bulk Nb samples measured by out-of-plane method. Both samples have only the peaks of Nb and NbN. The NbN peak of "180409-1-A" is sharper than that of "180409-2-A". The XRD patterns of "180409-1-B" and "180409-2-B" were hardly different from Figure 7.

CONCLUSIONS

We clarified the good conditions of NbN reactive sputtering by using in-house experimental apparatus. The critical temperature of our $NbN/SiO_2/Si$ samples achieved 14.4 K. In addition, we succeeded in making NbN/SiO_2/bulk Nb samples for measurement of critical field. The XRD patterns of these samples showed only Nb and NbN peaks.

ACKNOWLEDGEMENT

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