SUPERCONDUCTING RF CAVITY WITH REACTIVELY SPUTTERED NIOBIUM NITRIDE SURFACES

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Abstract

A technique to make a superconducting RF cavity with niobium nitride (NbN) surfaces has been developed. Niobium nitride is deposited on inner surfaces of Al and Nb cavities by the reactive dc sputtering method. The sputtering is performed by applying high voltage of about 5.2 kV between a Nb cathode and a Cu or Nb anode in an atmosphere of N_2 and Ar. The deposited NbN film is about 5 μm thick and shows a critical temperature of about 15°K. A preliminary Q measurement for a C-band NbN cavity of TE_{011} type gives $Q = 0.9 \times 10^7$ at 4.2°K.

Introduction

Aiming at the construction of superconducting linear accelerators, considerable amount of theoretical and experimental works have been done to develop a superconducting surface. By these works niobium has been found to be the most favorable material, but a technique to form stable surfaces has not yet been established. Recently niobium nitride (NbN) has attracted special attentions on stabilizing Nb surfaces. For example it is formed by nitriding Nb surfaces and expected to work as a very thin protecting layer.[1,2,3] However, NbN itself is also a very interesting superconductor.[4,5,6] There are many phases of niobium nitride and some of them show very high transition temperatures as given in Table 1. Although superconductor with high T_c has not always low RF surface impedance, higher T_c is desirable. RF characteristics of NbN films are not clear now.

To investigate a use of NbN as a stable RF superconducting material, we have developed a reactive dc sputtering technique to form thick NbN layers on inner surfaces of Al and Nb cavities. In this paper we describe details of the NbN deposition method and a preliminary result on the Q measurement for a C-band TE_{011} cavity with NbN surfaces sputtered on Nb substrates.

Deposition of NbN on Cavity Surface

Reactive dc Sputtering Method

In the reactive dc sputtering method, glow discharge is built up in an atmosphere of N_2 and Ar between a Nb cathode and a Cu or Nb anode. Niobium is sputtered from the cathode and reacts with N_2 on a substrate surface. The apparatus is schematically shown in Fig. 1. The sputtering process is done as follows. First of all the system is pumped to $\sim 10^{-6}$ Torr. Then N_2 and Ar gases are introduced and the discharge is turned on. Initially the substrate is covered by an Al shutter for 2 ~ 3 hours. This presputtering process is necessary to clean the surface of the Nb cathode and to warm up and bake out the apparatus. Finally the shutter is removed and deposition on the substrate is carried out. Small pieces of glass plate are placed for reference at the side of the substrate to know the thickness and critical temperatures of the deposited films. The deposited film is required to have high T_c, thickness of about 1 μm and uniformity over the substrate surface. Sufficient film thickness (much larger than the penetration depth) is necessary since the initially deposited part of film contains a great deal of impurities than the later deposited one.[7,8,9] Superconducting properties of the film depends greatly on the gas pressures and discharge voltage. Moreover, the geometrical disposition of the electrodes and substrate must be carefully arranged to obtain uniform film over the cavity surface. We have made systematic studies of the substrate materials and sputtering techniques.

Materials for the Substrate

This method was originally developed for the deposition on small glass plates. We must use metal substrate to make RF cavities. It must have
(1) low vapor pressure at deposition temperature (200 ~ 500°C),
(2) high thermal conductivity at liquid helium temperature, and
(3) no residual magnetization.

Copper, aluminum and niobium satisfy these conditions. However, we found that NbN film deposited on copper came off like blisters when it was taken out of the vacuum and left in the air (Fig. 2). The poor adhesion between NbN and Cu substrate will be due to the difference of thermal expansion coefficients. When aluminum is used for the substrate, some chemical reaction seems to occur on the aluminum surface under the deposited film. A number of white spots appear uniformly on the surface. In microscope these spots look like transparent glass beads, and we can observe slow growth of these spots. It seems that they come from the oxidization of aluminum through pin holes in NbN film. The growth of white spots can be inhibited by anodizing surfaces of Al substrates before deposition of NbN. The proper thickness of the anodized layer is found to be about 3 μm. A good NbN surface has been obtained for Nb substrate. In the case of Nb substrate the deposition temperature can be made as high as 500°C. The high deposition temperature is one of the essential conditions to make high T_c NbN films.[9,11] For NbN layers formed on Nb material, we have not found such problems as described above. Figure 2 shows a photograph of NbN films sputtered on Cu, Al and Nb substrates.

Film Deposition on Plain Substrate

Figure 3 shows the apparatus to sputter NbN on plain substrates. The distance between cathode and substrate surface is taken to be about 3 cm.[10] Substrates were heated up to 350°C with a stainless steel wire heater before presputtering, and kept at about 400 ~ 500°C during discharge. Our sputtering conditions are as follows.

Presputtering time	~4 hrs.
P_{N_2}	$\sim 9.5 \times 10^{-5}$ Torr
P_{Ar}	$\sim 5.3 \times 10^{-2}$ Torr
Target potential	~5.2 kV
Discharge current	~35 mA
Deposition time	~3 hrs.

For NbN films sputtered on the Al plain substrate, we have measured transition temperature (T_c), transition temperature width (ΔT_c), resistance ratio (β =

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$R_{300°K}/R_{77°K}$), and film thickness (t) as a function of the distance along the radial direction from the center of the disc substrate. An example of the data is shown in Fig. 4. Thickness, T_c and β tend to decrease and ΔT_c slightly increases as the distance increases. Critical temperatures of the films are close to, or as high as those of the bulk materials; the highest value is T_c = 14.8°K and the dispersion is rather small. The fact that the onset of a plateau is located near the wall position of our cavity is most favorable.

Film Deposition on Inner Surface of Cylindrical Substrates

The substrate is a hollow cylinder with an inner diameter of 60 mm and a length of 65 mm. In this case the coaxially disposed electrodes were used (Fig. 5). The cylindrical substrate was used as the anode at earth potential and a niobium rod of 10 mm in diameter was used as the cathode. Figure 6 shows the data of the films deposited on Al substrates by this method at the following pressure and voltage conditions.

No. of curves	1	2	3	4
Presputtering time (hrs.)	4	3	3	3
P_{N_2}	0	4.5×10^{-5}	9.6×10^{-5}	9.4×10^{-5}
P_{Ar}	5.5×10^{-2}	5.5×10^{-2}	5.5×10^{-2}	8×10^{-2}
Target potential (kV)	4.6	5.4	5	2
Discharge current (mA)	18	18	15	18
Deposition time (hrs.)	$4\frac{1}{4}$	4	4	$4\frac{1}{4}$
Substrate temperature (°C)	335	335	325	325

Curve 1 corresponds to the data for sputtering of Nb in a pure Ar atmosphere. The degradation of T_c of the curve 4 is probably due to the low target potential.

Preliminary Results on Q Value of the NbN Cavity

Using the reactive dc sputtering method described in the previous section, we have constructed a C-band NbN cavity of TE_{011} type. The NbN layer of about 5 μm thick is formed on the inner surfaces of the Nb cavity. The cavity consists of three parts, a hollow cylinder (inner diameter = 60 mm, length = 65 mm) and top and bottom plates. A photograph of the cavity is shown in Fig. 7. To investigate RF superconducting properties of the NbN cavity, the Q value has been measured at 6.512 GHz by using the decrement method. In the preliminary experiment an unloaded Q value of 0.9×10^7 has been obtained at 4.2°K in the low RF power region. Improving the deposition technique and also the microwave and low temperature systems to measure Q values, we are making further experimental studies on NbN cavities.

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Table 1 Stable Phases of NbN_x and T_c

NbN_x	Crystal Structure	T_c (°K)
NbN	whiskers	10 - 14.5
NbN	diffusion wires	16.1
NbN	film, cubic, f.c.	6 - 9
$NbN_{0.824-0.988}$	film, cubic, f.c.	14.4 - 15.3
$NbN_{0.70-0.795}$	cubic, tetragonal	11.3 - 12.9
$NbN_{0.91}$	cubic, tetragonal	~16
Nb_xNO_y	cubic, f.c.	13.5 - 17.0
Nb-N-C		~17
Nb-Ti-N		~18

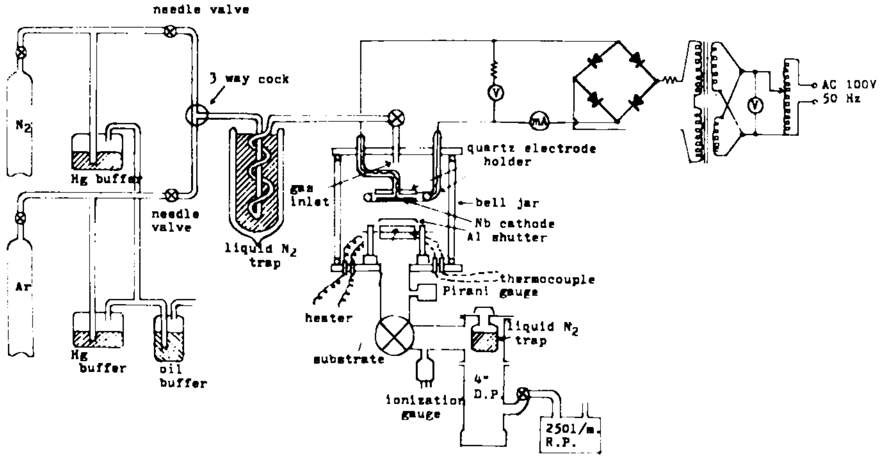

Fig. 1 Apparatus for reactive dc sputtering.

Fig. 2 NbN films sputtered on various substrates.

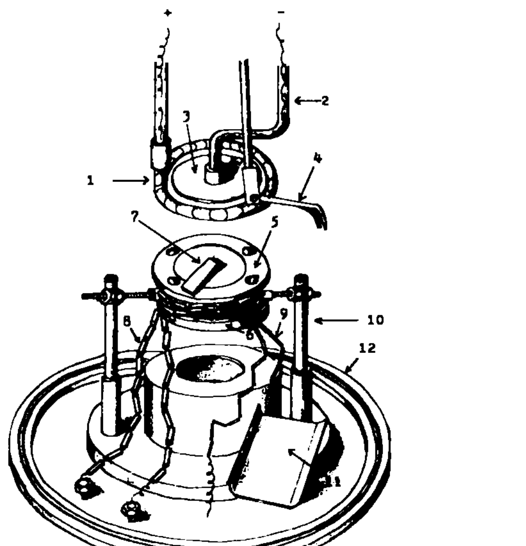

Fig. 3 Apparatus for reactive sputtering on plain substrates (1-anode, 2-cathode, 3-fused quartz cover under which Nb cathode disc is suspended, 4-magic hand, 5,6-substrates, 7-glass slide substrate for monitoring, 8-stainless steel wire heater, 9-thermocouples, 10-substrate holder, 11-Al shutter, 12-vacuum flange).

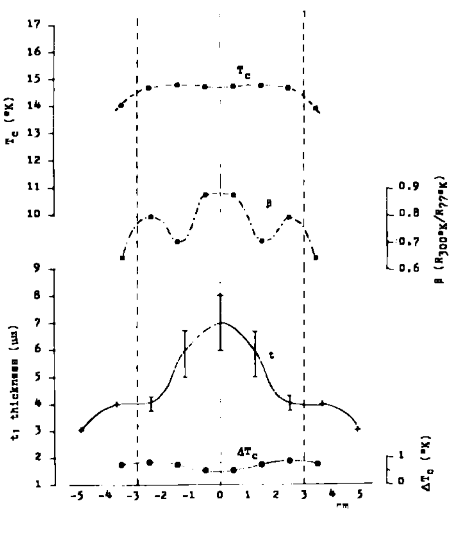

Fig. 4 Characteristics of the NbN film deposited on the Al disc substrate measured along the radial direction. Vertical broken lines indicate the boundary of the C-band TE_{011} cavity.

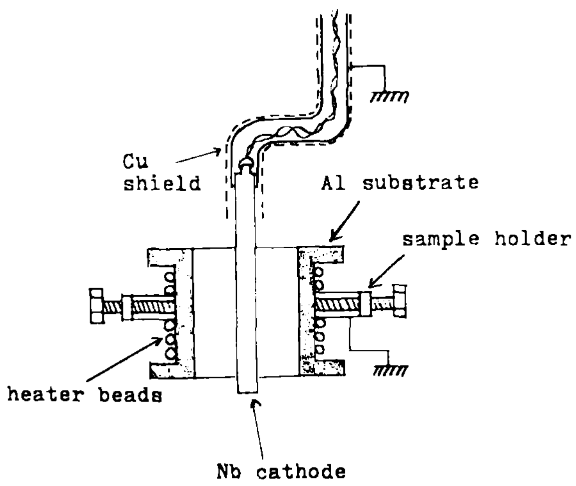

Fig. 5 Apparatus for reactive sputtering on cylindrical substrates.

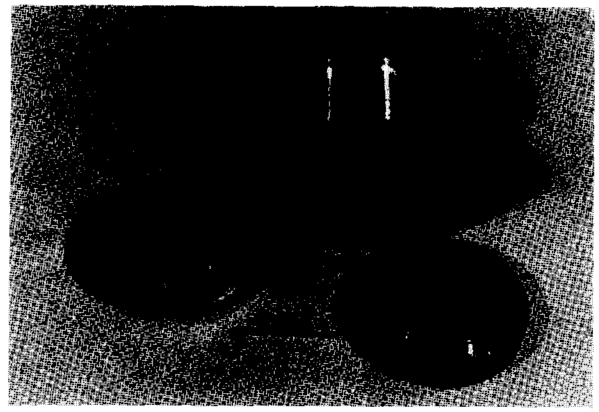

Fig. 7 C-band TE_{011} cavity with reactively sputtered NbN surfaces.

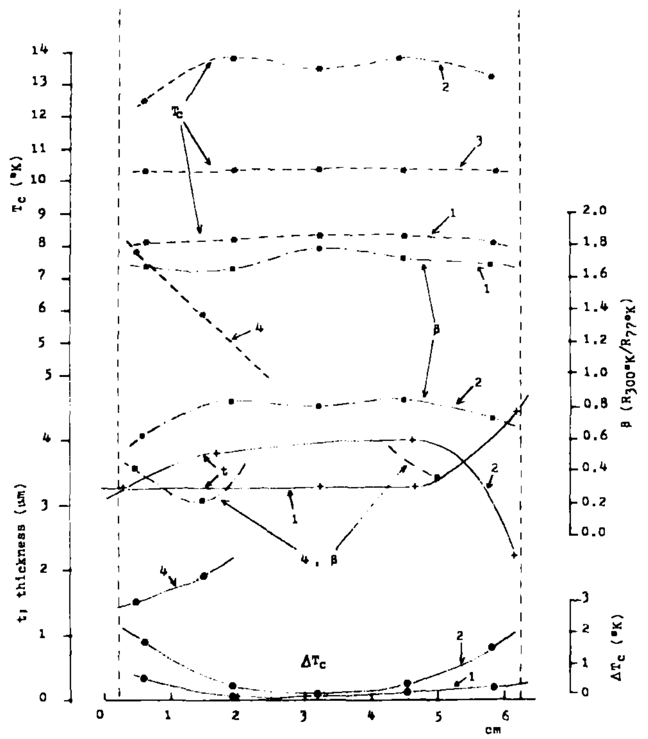

Fig. 6 Characteristics of the NbN film deposited on the inner surface of the Al cylindrical substrate measured along the axial direction. Vertical broken lines indicate the boundaries of the C-band TE_{011} cavity.