NbTiN SUPERCONDUCTING THIN FILMS PREPARED BY MAGNETRON SPUTTERING.

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Abstract

A prospective study of new materials for making superconducting cavities is presently undertaken at CE Saclay. Coatings of the B2 intermetallic compound $Nb_{(1-x)}Ti_xN$ with $0.30 < x < 0.64$ have been elaborated by a reactive magnetron sputtering method. Structural, chemical and electrical characterizations of the deposits have been made in order to correlate the deposition parameters and the superconducting properties of the films. One of the main goals is to minimize the RF surface resistance of the coatings. For this purpose, a cylindrical TE011 cavity made out of massive niobium, with a dismountable end plate, has been developped in order to test the NbTiN films deposited on copper substrates. The best surface resistance of the deposit we have obtained at 4 GHz and 4.2 K is lower by a factor of 3 than for niobium.

1. INTRODUCTION

Superconducting cavities for electron accelerators are being studied at CE Saclay. These cavities, usually made out of massive niobium, benefit of the knowhow accumulated over the years in many laboratories and display remarkable performances : quality factor $Q_0 = 10^{10}$ and accelerating field 15 MV/m for monocellcavities. The chosen frequency (1.5 GHz) and the superconducting properties of Nb (Tc = 9.2 K) dictate an operating temperature below 2 K in order to get a reasonable RF surface resistance (typically 20 nΩ).

In order to reduce the cost of fabrication of these cavities, as well as the cost of the cryogenic installation, it may be interesting to replace massive niobium by thin films with better superconducting properties. The compound NbTiN is a good candidate for this purpose : first, its high critical temperature (Tc = 17 K) should permit for cryogenics the use of boiling Helium at atmospheric pressure (i.e. 4.2. K) ; second, previous results obtained by R. Di Leo et al. [1] indicate that this material may have a low residual surface resistance ; last, the elaboration technique (reactive magnetron sputtering) is compatible with the cylindrical geometry of the cavities [2]. It is thus possible to deposit the superconducting thin film inside the cavity made out of a good thermal conductivity material (copper), in order to evacuate readily the dissipated power into the helium bath.

We present in this paper a prospective study of NbTiN coatings. We describe their characterization on samples and their RF surface resistance measurements on discs with a TE011 niobium cavity.

2. ELABORATION AND SAMPLE CHARACTERIZATION OF NBTIN COATINGS.

The films are prepared by planar magnetron sputtering. Their thickness measurement is made with a Taly-Step gauge. The chemical analysis uses an electron microprobe and ion beam techniques (RBS, NRS) [3]. X Ray diffraction, using a (θ-2θ) goniometer, enables the determination of the cristallographic structure of the surface (phase, texture). Resistivity and superconducting critical temperature measurements are performed with the "4 points" method. The superconducting behaviour of the films was also investigated by a.c. susceptibility measurements at low frequency (35 Hz).

The first step of the study consisted, with a first magnetron $Nb_{0.36}Ti_{0.64}$ target, in the determination of the sputtering parameters which gives the best superdonducting properties of the films. In the second step, with two other targets, we analyzed the influence of the Nb to Ti composition ratio on the film properties.

With the first composition $Nb_{0.36}Ti_{0.64}N$, the highest Tc values obtained is 13.9 K and the lowest resistivity at room temperature is 41 μΩ.cm.

The critical temperature of the coatings increases with the niobium concentration, as it is reported in [1], and the maximum value we have obtained is 16.08 K for $Nb_{0.70}Ti_{0.30}N$ films. For this last composition, resistivity at room temperature is about 90 μΩ.cm.

3. RF CHARACTERIZATION OF THE SUPERCONDUCTING FILMS.

We use a cylindrical TE011 niobium cavity (RRR = 180) with dismountable end plate to determine the RF surface resistance of superconducting coatings deposited on a copper disc [4,5]. We also obtain their critical temperature and penetration depth (if the film is thick enough) from quality factor and resonance frequency measurements as a function of the temperature in the 10 K to 17 K range.

The resonance frequencies of the TE011 and the TE012 modes on which we operate are respectively F_1 = 4.04 GHz and F_2 = 5.66 GHz. In order to localize defects of the coated disc in superfluid helium, we use sensitive fixed thermometers developped at IPN Orsay [6].

The quality factor Q of the cavity is measured by the usual decrement method. A preliminary test is first carried out with a RRR = 180 Nb end plate in order to determine the surface resistance of the cavity main body. Then, from the test performed with the coated disc, we obtain the surface resistance of the coating by substracting the contribution of the cavity body.

The validity of this method is based on the invariance of the surface resistance of the niobium cavity from one test to another one. In order to check this reproducibility after disassemblings and reassemblings without any treatment of the cavity, the first experiments were carried out with niobium discs. For three consecutive tests with different bulk niobium end plates chemically treated, the residual surface resistance at T = 1.6 K and F = 4 GHz was R_{res} = 100, 130 and 105 nΩ. For three other tests performoed with the same Nb disc we obtained successively R_{res} = 50, 70 and 70 nΩ.

With our experimental precautions we estimate the Q measurement error at +/-4%.

At T = 1.6 K the major part of the error on the coating R_s is determined by the uncertainty on the R_{res} of Nb, i.e. +/- 20 nΩ at 4 GHz, and +/- 50 nΩ at 5.7 GHz. At T = 4.2 K the R_s error results from the Q errors on each of the two measurements with Nb disc and with coated disc. It becomes very important when the coating surface resistance is lower than the Nb one.

From all the results we have obtained for 10 significant tests with coatings prepared in different conditions, one can distinguish two sorts of R_s(B) curves :

- a first one (D21 type, figs 1 and 2) with low R_s values at low field, but with a steep slope with B and quench from B = 11 to 22 mT.
- a second one (D12 type, figs 1 and 2) with higher values of R_S at low field, but with a much lower increase with B and a quench from B = 20 to 28 mT.

The correlations between the sputtering parameters, the sample characterizations, and the R_s values are still not clear, but we have observed two general tendencies :

i) The BCS surface resistance is lower for high Nb content coatings. From the two first $Nb_{0.70}Ti_{0.30}N$ coatings recently tested the measured BCS resistance was 60% lower than for the precedingt $Nb_{0.36}Ti_{0.64}N$ ones in relation with a corresponding increase of Tc from 13.4 to 15.8 K.

ii) The R_s(B) slope decreases significantly and the maximum field increases when the coatings are prepared with a negative bias voltage applied to the substrate during sputtering (between -50 and -100 V).

The maximum RF magnetic field limited by quench has been obtained with the D12 end plate of $Nb_{0.36}Ti_{0.64}N$ composition and Tc =12.95 K. Its value of 28 mT corresponds to the critical field H_{c1} for $Nb_{0.36}Ti_{0.64}N$ films reported in [1]. In all our tests of NbTiN coatings on coopper discs no particular local heating has been detected by the thermometers just before quenching (measured ΔT not higher than 350 mK). Moreover, for the two films of lowest R_s : D12 and D21 for which no RF power limitation has occured at 4.2 K, the quench field level was the same at T = 4.2 K and T = 1.6 K. This seems to reveal a magnetic quench occuring at a field level more or less close to the critical field H_{c1}, but more investigation is needed to verify such an hypothesis.

4. CONCLUSION

A surface resistance lower by a factor of 3 than the surface resistance of niobium has been obtained at 4.2 K and 4 GHz (about 1600 nΩ compared to 5200 nΩ for niobium). Significant improvment of the R_{BCS} value has been achieved after we have increased the niobium content of the coatings up to the $Nb_{0.70}Ti_{0.30}N$ composition. But the residual surface resistance is important : the lowest obtained i.e. 400 nΩ at 1.6 K and 4 GHz is much higher than the 70 nΩ niobium one.

The maximum field level attained before quenching is 28 mT, value which corresponds to the critical field H_{c1} reported in [1]. We expect to determine soon whether the quenches in our deposits are of magnetic or thermal origin.

Our work will now consist in preparing NbTiN coatings with the new sputtering set up that we have developped in our laboratory, and in completing their optimization before depositing inside cylindrical 1.5 GHz cavities.

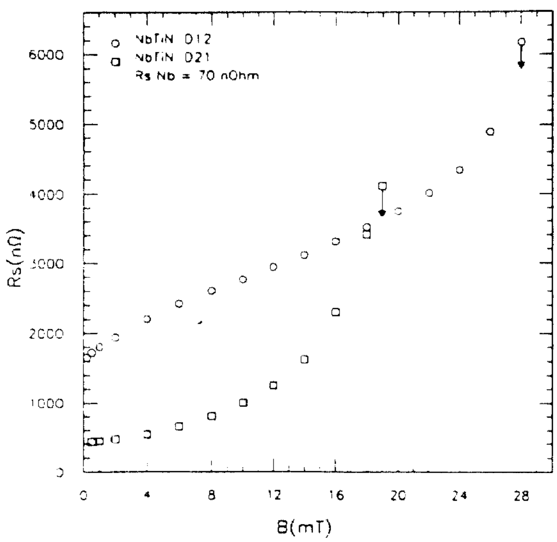

Fig.1 $R_{res}(B)$ curves of the D12 ($Nb_{0.36}Ti_{0.64}N$) and the D21 ($Nb_{0.70}Ti_{0.30}N$) coatings at 4 GHz and 1.6 K.

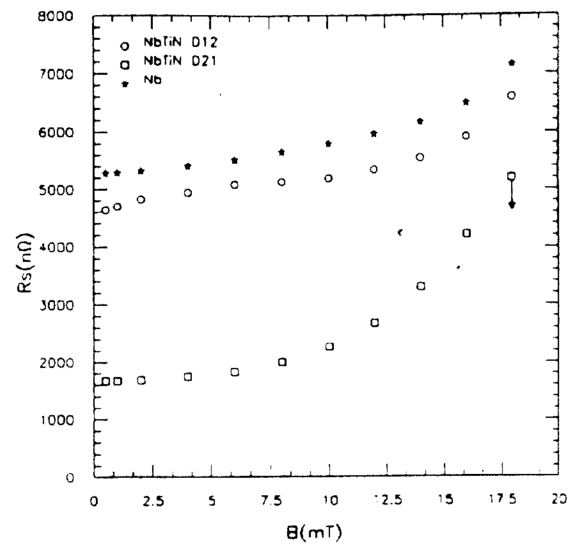

Fig.2 $R_s(B)$ curve for the D12 ($Nb_{0.36}Ti_{0.64}N$) and the D21 ($Nb_{0.70}Ti_{0.30}N$) coatings at 4 GHz and 4.2 K.

5. REFERENCES

[1] R. Di Leo, A. Nigro, G. Nobile and R. Vaglio, "Niobium-Titanium Nitride Thin Films for Superconducting RF Accelerating Cavities", Journal of Low Temperature Physics, vol. 78, n° 1/2, pp 41-50, 1990

[2] C. Benvenuti, D. Bloess, E. Chiaveri, N. Hilleret, M. Minestrini and W. Weingarten, Status Report on CERN Activities aiming at the Product ion of Sputter-Coated Copper Superconducting RF Cavities for LEP, 4th Workshop on RF Superconductivity 14-18 August 1989, Tsukuba, Japan.

[3] B. Roux, A. Chevarier, N. Chevarier, M. el Bouanani, E. Gerlic, M. Stern, P. Bosland and F. Guémas, "Ion Beam Analysis using Alpha Particles and Protons for Compositional Determination of Niobium Superconducting Compound Films". To be published in N.I.M. (Special Issue).

[4] M. Juillard, B. Aune, B. Bonin , L. Wartski, "Superconducting Surface Resistance Measurement with a TE011 Cavity". European Particle Accel. Conference, Nice, 1990.

[5] J.M. Laurent, H. Padamsee, "Demountable TE011 Cavity for RF Superconducting Surface Studies". CERN/EF/RF 83-2.

[6] M. Fouaidy, T. Junquera, A. Caruette, "Surface Temperature Measurements on Superconducting Cavities in Superfluid Helium", 5th Workshop on RF Superconductivity DESY/Hamburg-19-23 August 1991

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