DEVELOPMENT OF THIN FILMS FOR SUPERCONDUCTING RF CAVITIES

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Abstract

Superconducting coatings for superconducting radio frequency (SRF) cavities is an intensively developing field that should ultimately lead to acceleration gradients better than those obtained by bulk Nb RF cavities. ASTeC has built and developed experimental systems for superconducting thin-film deposition, surface analysis and measurement of Residual Resistivity Ratio (RRR). Nb thin-films were deposited by magnetron sputtering in DC or pulsed DC mode (100 to 350 kHz with 50% duty cycle) with powers ranging from 100 to 600 W at various temperatures ranging from room temperature to 800 °C on Si (100) substrates. The first results gave RRR in the range from 2 to 22 with a critical temperature $T_c \approx 9.5$ K. Scanning electron microscopy (SEM), x-ray diffraction (XRD), electron back scattering diffraction (EBSD) and DC SQUID magnetometry revealed significant correlations between the film structure, morphology and superconducting properties.

INTRODUCTION

SRF cavity technology in particle accelerators is now reaching the limit of performance achievable with bulk Nb cavities [1]. Since superconducting properties for SRF are confined to a penetration depth of less than one micron [2] then Nb thin-films can be an alternative to bulk Nb with the advantage of Cu substrate with factor 3 higher thermal conductivity than Nb [3]. Nb thin-films can have higher critical magnetic fields than bulk Nb due to greater flux pinning within the films [4] and there is the possibility of multilayer films [5] that provide greater magnetic shielding. With better thermal stability and higher critical fields it is possible to have higher accelerating gradients within SRF cavities allowing for better performance, reduced cost and reduced volumes of Nb [6].

Physical vapour deposition by magnetron sputtering has been used as a preferred process due to its high deposition rate and ease of scalability in order to synthesise superconducting thin films within SRF cavities [7]. The purpose of the present study is to determine optimum experimental parameters which will result in the best superconducting properties of Nb thin-films deposited by magnetron sputtering. Nb thin films were deposited using the Advance Energy Pinnacle + in both DC and pulsed DC mode in order to control the energy and specific flux of particles arriving to the substrate and consequently affecting the properties of the growing film. The variable parameters are deposition current, voltage, pulsed duty cycle, pulsed frequency, substrate temperature, and substrate bias. After morphological evaluation the films have then been assessed for their superconducting properties showing their suitability for use in SRF cavities.

EXPERIMENTAL SETUP

A magnetron sputtering facility has been built for the purpose of depositing thin films. Thin film samples were deposited on Si (100) substrates using Kr sputtering gas. Each substrate was cut from commercially available wafers which are prepared by cleaning in ultrasonic baths of acetone, methanol, IPA, then deionised water [8]. The deposition facility allows a number of deposition parameters to be varied, the effects of which on the film properties can be then studied. The deposition power varied between 100 and 600 W in both DC and pulsed DC settings. The relation between power, current and voltage can be seen in Fig. 1.

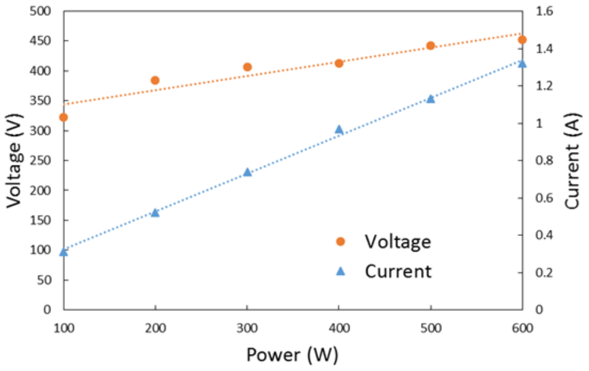

Figure 1: Current and voltage characteristics against power of the DC power supply.

When used in pulsed mode, the power supply was operated between 100 and 350 kHz with a 50% duty cycle. A bias voltage can be applied to the substrate and was varied between 0 and –150 V. The base pressure of the unbaked UHV chamber reached ~10^{-8} mbar and the Kr pressure was set to 3 mbar. The distance between the sputtering target and substrate can be varied between 10 and 25 cm. The substrate can be continuously rotated about the axis normal to the centre of the sample surface and uniformly heated from room temperature to 800 °C.

Morphological analysis was performed by SEM, XRD and EBSD. SEM analysis was used to determine the film structure and thickness and hence the growth rates. SEM images give an indication of the type of film that has been deposited *i.e.*, columnar with voids or densely packed grains. XRD analysis results show average grain size and lattice orientations within the film. EBSD analysis provides an accurate value for the grain orientation and size at the surface of the film. RRR measurements have been performed using a purpose built cryostat housing a four point probe. DC SQUID magnetic susceptibility measurements were performed using a Quantum Design MPMS, giving both the first and second critical fields, H_{c1} and H_{c2}.

RESULTS AND DISCUSSION

Forty eight samples have been deposited and analysed using the four point probe and a selection by SEM, XRD, EBSD and DC SQUID magnetic susceptibility.

The XRD analysis has shown that Nb grain sizes are in the range of 9±2 to 18±3 nm, with the larger grains being deposited at powers between 400 and 600 W. This is similar in size to the 5 to 12 nm grains produced in other studies [9]. Most films have shown the biggest peak intensity at $2\theta = 41°$ which corresponds to the (220) grain orientation (Fig. 2).

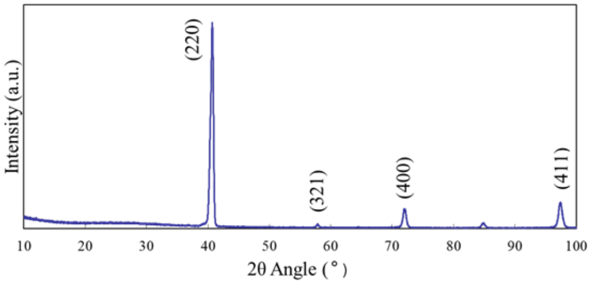

Figure 2: XRD spectrum for thin Nb film.

Three samples have also shown smaller 2θ peaks at 72° (400) and 97° (411).

High values of RRR were recorded for both orientations so there seems to be no dependence of RRR on orientation.

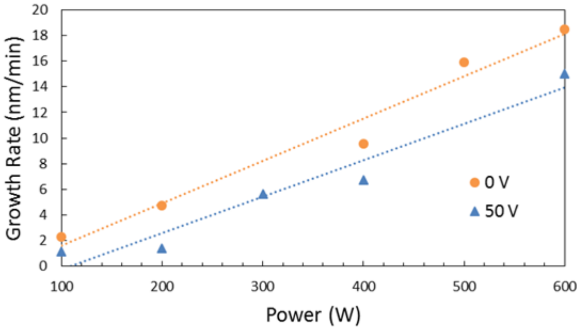

Figure 3: Growth rate as a function of power with a bias voltage of 0 or –50 V.

SEM analysis of samples has shown films ranging in thickness from 173.9 to 2768 nm with growth rates of 1.16 and 18.5 nm/min respectively. The growth rate increases steadily with increasing continuous power. Fig. 3 shows that the growth rate is reduced, relative to no bias, when the substrate is biased at –50 V. This could be the result of increased re-sputtering as ionised species within the plasma arrive at the substrate with a higher energy when the substrate is biased [10]. The reduced growth rate could also be due to the film forming with fewer voids and hence greater density. Further analysis is required using techniques such as Rutherford back scattering which determines the total number of atoms deposited per units of area allowing thickness evaluation.

Only one sample has been analysed using EBSD, see Fig. 4. EBSD data shows grains larger than 18 nm present in the sample, the largest of which are of the order 250 nm across their longest axis. The larger grains are surrounded by a matrix of smaller grains of a similar size to those described by XRD.

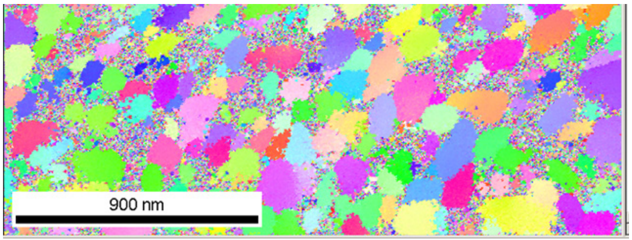

Figure 4: EBSD image showing grain size up to 250 nm across. Colours relate to different lattice orientation.

RRR values have ranged between 2 and 22. Samples with RRR ≥ 10 were deposited with $300 \leq P \leq 600$ W. In all cases there is an increased RRR when a biased substrate has been used, although a higher bias voltage does not always result in increased RRR (Fig. 5).

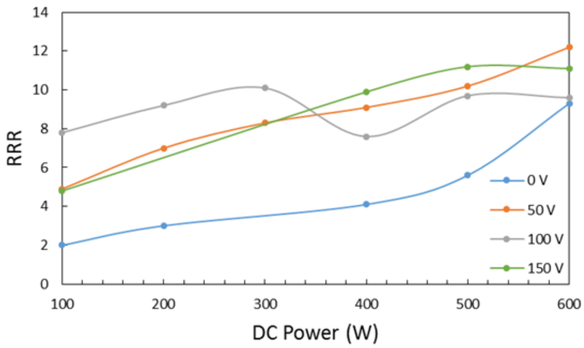

Figure 5: RRR values as a function of DC power for various bias voltages.

Ion bombardment occurs when the substrate is biased and will cause modifications to the film texture resulting in a denser film [10] (Fig. 6). RRR was also investigated for various pulsed DC power settings at frequencies between 100 and 350 kHz and was found to increase when the substrate was biased but with no significant differences to values observed for continuous DC.

Table 1: A Selection of DC SQUID Magnetic Susceptibility Measurements for the Samples Deposited at 500 W Power

RRR	Temp (°C)	Bias (V)	H_{c1} ⊥ (mT)	H_{c1} ∥ (mT)		H_{c2} ⊥ (mT)	H_{c2} ∥ (mT)	
			6 K	6 K	1.8 K	6 K	6 K	1.8 K
22	800	0	5±3	40±20	-	>1000	>800	-
10	30	–100	30±10	200±20	160±10	600±20	500±20	900±20
10	30	–50	-	-	-	-	450±20	900±20

The samples with the highest values of RRR ≈ 20 have been deposited on heated substrates, while without sample heating the highest RRR = 12. This shows a definite correlation between increasing substrate temperature and increasing RRR.

When the substrate is biased or heated during film deposition the arriving atoms on the surface are more mobile. Mobile atoms are able to re-orientate themselves closer to neighbouring atoms and form a denser film with higher RRR. Results have shown that increasing substrate temperature will lead to more mobility than bias alone.

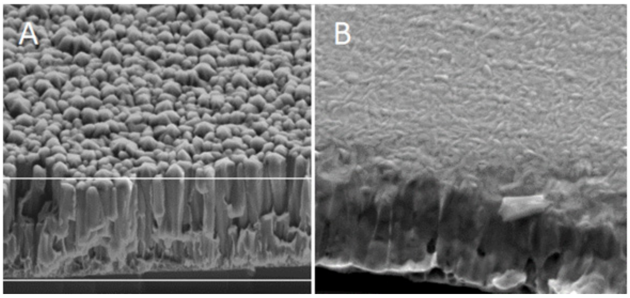

Figure 6. Two SEM images of samples deposited (A) without a bias and (B) with a –50 V bias.

This work has aimed to produce the highest RRR values. However increased vortex pinning is important for SRF cavities and it has been reported that this does not always follow from high RRR [3].

DC SQUID magnetic susceptibility measurements and analysis have been performed on a selection of samples, an example of which is given in Fig. 7. Each sample was aligned such that its surface was either perpendicular or parallel to the applied magnetic field with a precision of better than 1 degree. The parallel field measurements are more relevant to understanding how the films will perform in SRF cavities. The value of H_{c1} is determined as the point where the magnetic field begins penetrating into the sample. The value of H_{c2} is defined as the point where the magnetic field supresses superconductivity completely [4].

H_{c1} values have been recorded for only two samples measured in both parallel and perpendicular orientation, both samples show greater H_{c1} when the magnetic field is parallel to the film plane. H_{c2} was measured for seven samples and found to be greatest for films deposited with a substrate heated to 800 °C and no bias. Films deposited at room temperature and with a substrate biased at –50 or –100 V had lower H_{c2} than unbiased substrate heated to 800 °C. Measurements were made at 6, 4 and 1.8 K showing increasing H_{c2} with lower temperature. A selection of the results is shown in Table 1.

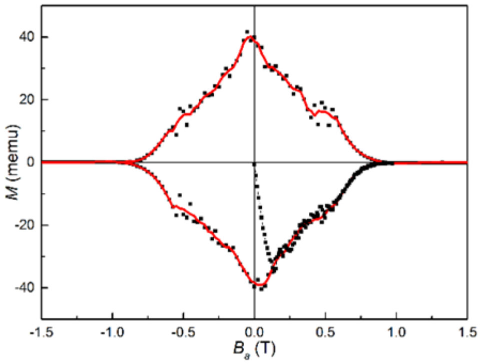

Figure 7: DC magnetic susceptibility measurement for a sample with RRR=10 deposited at 500 W and –100 V bias for applied field parallel to the plane of the film.

CONCLUSIONS

The study has concluded that among the conditions tested the most favourable condition during Nb thin film deposition was a heated substrate of 800 °C, both to achieve a higher RRR value and higher H_{c2}. The effect of DC deposition rate as a function of power has been thoroughly investigated finding that increased power increases film growth rates. When a bias is applied during deposition, the growth rate decreases but RRR increases. Increased substrate temperature during deposition results in films with higher RRR and higher H_{c2}. Further analysis is needed of more samples to make firm conclusions regarding H_{c1}.

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