RF Critical Field Measurement of MgB$_2$ Thin Films Coated on Nb

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Abstract. Niobium (Nb) Superconducting RF (SRF) cavities have been used or will be used for a number of particle accelerators. The fundamental limit of the accelerating gradient has been thought to be around 50 MV/m due to its RF critical magnetic field of around 200 mT. This limit will prevent new projects requiring higher gradient and compact accelerators from considering SRF structures. There is a theory, however, that promises to overcome this limitation by coating thin (less than the penetration depth) superconductors on Nb. We initiated measurements of critical magnetic fields of Nb coated with various thin film superconductors, starting with MgB$_2$ films deposited using reactive evaporation technique, with the goal to apply this coating to SRF cavities. This paper will present first test results of the RF critical magnetic field of a system consisting of a 10 nm B and a 100 nm MgB$_2$ films deposited on a chemically polished 2-inch single grain Nb substrate.

1. INTRODUCTION

Having evolved over the years from accelerating gradients of 3 MV/m in the early 70s to over 40 MV/m now, niobium cavities have proved viable and useful for accelerator applications. A number of large-scale accelerator projects such as the International Linear Collider and European X-Ray Free Electron Laser are based on SRF technology and will be employing a large number of SRF cavities. Optimization of the cell shape further increased the accelerating gradient, bringing the current state of the art in niobium RF superconductivity to its fundamental limit, dictated by the RF critical field of niobium [1]. For further improvement in the accelerating gradient we need to use superconductors that have higher RF critical fields.

Since the surface resistance of a BCS superconductor, $R_s$, for $T<T_c/2$, $h\omega << \Delta$, $h\omega << k_b T$ can be approximated by $R_s \propto \sqrt{\rho_n} \cdot exp(-\Delta/k_b T)$ [2], in order to have a low $R_s$, the superconductor must have a large energy gap and a low normal-conducting resistivity just before the transition. These properties are available in some niobium compounds, such as NbN, NbTiN, and A-15 compounds such as Nb$_3$Sn or V$_3$Si. Also, MgB$_2$ unlike other high $T_c$ superconductors holds some promise due to its BCS-like behavior and low intergrain losses. So far, however, to the best of our knowledge only once has a superconducting accelerating structure made of
a superconductor other than niobium performed better than a niobium one, showing higher low-field quality factor at 4.2 K than that of niobium at 2 K [3].

In 2005, a possibility to increase the accelerating gradient was suggested based on the fact that the $H_{c2}$ in the direction parallel to the magnetic field increases if the superconductor gets thinner than the London penetration depth [4]. Our goal is to demonstrate the enhancement of the RF critical magnetic field using this theory. We decided to try MgB$_2$ first since we have studied this material for a few years and believe that it is one of the superconductors that has a high potential for accelerator cavity applications.

2. EXPERIMENTAL

2.1. SAMPLE PREPARATION

After a few unsuccessful attempts to deposit a good-quality film on polycrystalline niobium samples, we decided to concentrate our efforts on single grain niobium samples. The niobium samples are 2-inch (50.8 mm) in diameter and 1 mm thick disks with a residual resistivity ratio (RRR) $\approx$ 300. Before deposition these disks were polished with a buffered chemical polishing (BCP) solution (HF: HNO$_3$: H$_3$PO$_4$ = 1:1:2 (vol.)), the standard procedure for preparation of SRF cavities.

MgB$_2$ films were deposited at Superconductor Technologies, Inc. (STI) by the reactive evaporation technique [5]. Samples were coated with 10 nm of boron (B) on top of niobium, and then 100 nm of MgB$_2$ was deposited on top of the B layer.

X-ray diffraction (XRD) $\theta$-2$\theta$ scans showed that the orientation of niobium substrate was largely (110), and the orientation of MgB$_2$ film was (002) as shown in Fig. 1. Atomic force microscopy (AFM) indicated RMS surface roughness of 1-7 nm for 10 x 10 $\mu$m$^2$ scans and 48-64 nm for 100 x 100 $\mu$m$^2$ scans on niobium substrate; after the MgB$_2$ (100 nm)/B (10 nm) coating was done, the measured RMS surface roughness was 12-18 nm for 100 x 100 $\mu$m$^2$ scans. In Fig. 2, the reconstructed 3D AFM image showing the microstructure of MgB$_2$ coating is presented.
Figure 2. An AFM reconstructed image shows the microstructure of MgB$_2$ film coated on a single crystal niobium substrate.

2.2. MEASUREMENT SETUP
The RF cavity method was employed for RF measurements. A description of the setup and measurement techniques can be found in [6] and [7]. A hemispherical copper cavity with a resonant frequency of $\sim$ 11.4 GHz for the TE$_{013}$ mode was used. The demountable plate was made to accommodate 2-inch diameter samples. There is no electric field perpendicular to the sample surface, preventing the effect of field emission. The surface magnetic field is azimuthally uniform and is in parallel with the sample surface, having a Bessel function in the radial direction with a peak at half the sample radius. The low-power RF properties were measured with a network analyzer and the high-power tests were carried out by connecting the cavity to a 50 MW pulsed Klystron. Short pulses were used to eliminate thermal quenches that could confuse the critical field data. The temperature was monitored by 4 diode temperature sensors, two on the hemispherical part, one on the RF input iris and one inserted in the copper backing plate behind the sample.

3. RESULTS
The first experiment was conducted on a fine grain niobium substrate coated with MgB$_2$. The result showed only a transition at about 9.2 K corresponding to the underlying niobium. We concluded that these poor results are due to the fact that the surface was too rough for the coating thickness, i.e., the roughness of a few hundred nm compared to 100 nm of coating. Since we do not have electropolishing capability, we decided to use single grain Nb that can have smoother surface than electropolished fine grain Nb with only chemical polishing. The second experiments were conducted with MgB$_2$ deposited on the single grain sample. Figure 3 shows the quality factor of the cavity as a function of temperature in red, together with the niobium data measured with the same setup. We observed a superconducting transition of the MgB$_2$ film around 37 K. The second transition corresponding to niobium was observed around 9 K. As the temperature goes further down, the 2 data crosses at 8 K and coated sample showed slightly lower $Q_0$ at lower temperatures, then both data levels off at 3 K with $Q_0$ = 270,000. These data suggest that the coated sample has higher RF surface resistance than Nb sample at $\lesssim$8 K and that the precise RF surface resistance measurements of samples at $\gtrsim$5 K become difficult since the total resistance gets dominated by the resistance of copper components of the cavity. The inset of Fig. 3 shows the $dQ_0/dT$ calculated from the data. The change in slope indicates the transition from normal conducting to superconducting state in MgB$_2$ film, which occurs at $T_C=$
Figure 3. Quality factor as a function of temperature for a single grain niobium sample coated with B(10 nm)/MgB$_2$(100 nm) [red circles]. For comparison the quality factor vs. temperature for niobium measured with the same setup is presented [black squares]. The inset shows $\frac{dQ_0}{dT}$ calculated from the data near MgB$_2$ transition point. These data indicate $T_C = (37.1 \pm 0.6)$ K.

Figure 4. The cavity resonant frequency as a function of temperature for a niobium sample coated with B(10 nm)/MgB$_2$(100 nm). In the inset $\frac{-1}{f_{res}(T)} \frac{df_{res}(T)}{dT}$ calculated from the frequency data (black dots) and the thermal expansion coefficient of copper from [8] (thin red line) are shown as a function of temperature.

$(37.1 \pm 0.6)$ K. Fig. 4 shows the resonant frequency as a function of temperature. The frequency variation is caused by the change of dimensions due to thermal contraction and by the change
Figure 5. The cavity loaded quality factor as a function of the peak magnetic field on the surface of the sample for the single grain niobium sample coated with B(10nm)/MgB$_2$(100nm) (red circles). For comparison the data for niobium measured in the same setup is presented (black squares). Early degradation of the quality factor for the Nb/B(10nm)/MgB$_2$(100nm) sample happened due to a quench in the niobium substrate as follows from the measurements presented in Fig. 6.

In the penetration depth of the sample. If we consider the cavity’s effective radius, $r_{\text{eff}}(T)$, it is expressed as:

$$r_{\text{eff}}(T) = \frac{G}{\pi \mu_0 f_{\text{res}}(T)},$$  

(1)

where $G$ is the geometrical factor of the cavity, then we can derive the change in the effective length from the change in frequency:

$$\frac{1}{r_{\text{eff}}(T)} \frac{dr_{\text{eff}}(T)}{dT} = \frac{-1}{f_{\text{res}}(T)} \frac{df_{\text{res}}(T)}{dT}.$$  

(2)

In the inset of Fig. 4 we plot $\frac{-1}{f_{\text{res}}(T)} \frac{df_{\text{res}}(T)}{dT}$, calculated from the frequency data and the linear thermal expansion coefficient of copper [8] versus temperature, which indicates that above 20 K the thermal expansion dominates the frequency change.

Following the low power measurements with a network analyzer, the cavity was connected to a 50 MW klystron. To avoid thermal effects 1 $\mu$s pulses at 10 Hz repetition rate were typically used to excite the TE$_{013}$ mode in the cavity. The loaded cavity quality factor was determined from the decay time in the reflected power signal. Figure 5 shows the quality factor as a function of field. Quality factor degradation occurred at a relatively low field of $\mu_0 H_{\text{peak}} \approx 40$ mT as shown in Fig. 5. For comparison, also shown in Fig. 5 is the result of a Nb sample measured previously under the same conditions. The key question was whether it was the niobium substrate or the MgB$_2$ film that caused the degradation at this field. To answer this question we measured the quality factor as a function of temperature for different field levels around the quench field as shown in Fig. 6. This measurement showed that the early degradation in the quality factor occurred on the niobium surface, while the MgB$_2$ film remained superconducting for all field...
levels between 18 and 42 mT. A subsequent Auger sputter depth profile measurement using 4 keV Ar ions showed the existence of Mg at the interface between Nb and MgB$_2$ layer, suggesting unsuccessful formation of clean B (insulation) layer. Also, observation of the tested surface with SEM showed some small cracks with some melting edges, although we are not sure if these cracks occurred during the testing since the surface was not checked with SEM before testing.

4. CONCLUSION
Following the theory that predicts breaking through the niobium limitation by coating thin superconductors on Nb, we initiated measurements of critical magnetic fields of Nb coated with MgB$_2$ deposited with reactive evaporation technique. We have tested a single-crystal Nb sample coated with 10 nm of B and 100 nm of MgB$_2$. The low-power Q measurement showed 2 transitions, one at 37.1 K (MgB$_2$) and the other at 9 K (Nb). The pulsed high-power test showed a low quench field of $\sim$ 40 mT caused by a quench on the Nb surface and not by the MgB$_2$. This degradation of quench field after coating of B/MgB$_2$ layers needs to be investigated and solved before the demonstration of quench field enhancement.

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References
[1] Geng R L et al. 2007 Proc. of Particle Accelerator Conference (Albuquerque) p.2337
[2] Deambrosis S M 2008 Ph.D. Thesis (Padua University); Deambrosis S M et al. 2006 Physica C 441 108113
[3] Mueller G et al. 1996 Proc. of European Particle Accelerator Conference (Barcelona)
[4] Gurevich A 2006 Appl. Phys. Lett. 88 012511
[5] Moeckly B H and Ruby W S 2006 Supercond. Sci. Technol. 19 L21L24
[6] Nantista C et al. 2005 Proceedings of Particle Accelerator Conference (Knoxville) p.427
[7] Canabal A et al. 2007 *Proc. of Particle Accelerator Conference (Albuquerque)* p.2370
[8] White G K and Collins J G 1972 *J. Low Temp. Phys.* **7** 43