

MgB₂ THIN FILM STUDIES*

T. Tajima[#], H. Inoue, KEK, Tsukuba, Ibaraki 305-0801, Japan, N.F. Haberkorn, L. Civale, R.K. Schulze, LANL, Los Alamos, NM 87545, U.S.A., J. Guo, V.A. Dolgashev, D.W. Martin, S.G. Tantawi, C.G. Yoneda, SLAC, Menlo Park, CA 94025, U.S.A., B.H. Moeckly, C. Yung, Superconductor Technologies, Inc., Santa Barbara, CA 93111, U.S.A., T. Proslie, M. Pellin, ANL, Argonne, IL 60439, U.S.A., A. Matsumoto, E. Watanabe, NIMS, Tsukuba, Ibaraki, 305-0047, Japan, X.X. Xi, Temple University, Philadelphia, PA 19122, U.S.A., B. Xiao, TJNAF, Newport News, VA 23606, U.S.A.

Abstract

In order to overcome the limitation of Nb associated with its theoretical RF critical magnetic field of ~200 mT, which corresponds to an accelerating gradient of 50-60 MV/m for electron accelerators, studies of coating thin film MgB₂ have been carried out. The B_{pen}, a magnetic field at which a large number of magnetic fluxons start to penetrate into the superconductor has been measured with a DC SQUID magnetometer. An increase in B_{pen} from 500 nm to 300 nm films was observed. Also, 300 nm films showed B_{pen} significantly higher than bulk Nb at 4.5 K. In parallel, RF measurements using 11.4 GHz pulsed powers with TE₀₁₃ mode copper cavity have been carried out at SLAC. Despite the high B_{pen} measured in the magnetization measurements, the MgB₂(100 nm)/Al₂O₃(20 nm)/Nb system showed significantly lower quench fields, i.e., ~43 mT at 4 K and ~33 mT at 10 K. It was found, however, that these quenches are thermal, not magnetic. Auger depth profile showed inter-diffusion of coating components at the interfaces, which indicates the degradation of RF resistance at these interfaces. A technique to solve this problem is being developed.

BACKGROUND AND MOTIVATION

The technology to produce high-quality SRF cavities made of bulk Nb has advanced so well that more than half of 1.3 GHz 9-cell cavities have shown >35 MV/m, the current goal of the International Linear Collider (ILC). Also, a 9-cell cavity made from large grain Nb showed 45 MV/m recently at DESY [1]. These results are considered to be very close to the fundamental limit of Nb due to its critical magnetic field of ~200 mT. The purpose of this study is to evaluate MgB₂ if it can exceed this limit and can be the material for the next generation SRF cavities with >50 MV/m operating field.

Multilayer Concept

For SRF cavities, having high H_{c1} is very important since otherwise Q₀ degrades due to the flux penetration and subsequent RF losses at fields at >H_{c1}. Although most alternative materials such as Nb₃Sn, NbN and MgB₂ have lower H_{c1} than Nb, it has been observed that thin film superconductors possess H_{c1} that is much higher than that

of bulk. Based on this, Gurevich proposed multilayer coating concept to enhance the achievable gradient [2]. Figure 1 illustrates the concept of using 3 layers of 77 nm MgB₂ films. The H_{c1} of 77 nm MgB₂ is predicted to be ~550 mT using the formula in the Fig. 1 assuming the coherence length (ξ) of 6 nm and the penetration depth (λ) of 110 nm. With this coating, the surface magnetic field of 500 mT decays to 100 mT when it reaches Nb. In other words, if Nb does not breakdown up to 100 mT, this system sustains the cavity surface field of 500 mT corresponding to an E_{acc} of 125 MV/m!

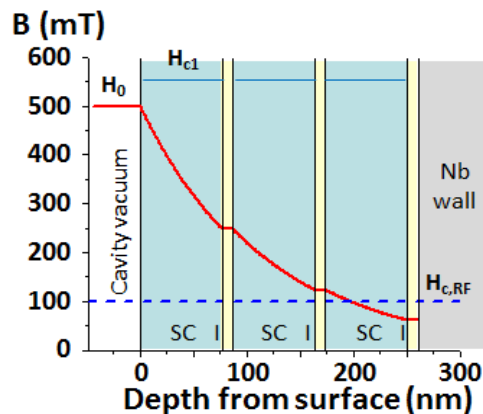


Figure 1: Concept of field enhancement with 3 layers of 77 nm MgB₂ separated by 10 nm insulator layers.

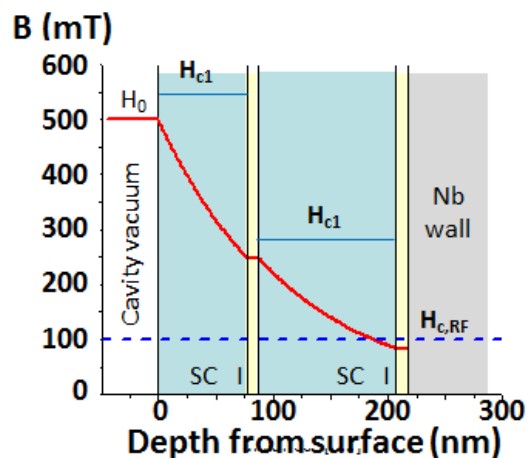


Figure 2: Tuning the thickness of superconducting (SC) layers will allow the reduction in the number of layers to achieve the same gradient.

*Work supported by US DOE Office of Nuclear Physics.

[#]tajima@lanl.gov

By tuning the thickness of SC layers, the same effect can be obtained with 2 layers of MgB₂ as shown in Fig. 2 since the H_{c1} in the second layer does not need to be as high as the top layer.

In this paper, results of DC magnetization measurements on flux penetration field (B_{pen}) with thin films and pulsed RF measurements at 11.4 GHz to evaluate RF losses and quench fields will be reported.

SAMPLES

So far, only flat samples have been used. For DC magnetization measurements, approximately 5 mm by 5 mm samples coated on *c*-plane sapphire have been used. Samples for RF measurements consisted of Nb substrates of 50.8 mm in diameter and 1 mm in thickness with thin film coatings of alumina and MgB₂. The Nb substrates were made of single-grain Nb taken from 291 mm diameter large-grain Nb sheets with RRR >300 from Tokyo Denkai Co., Ltd, Japan. After being machined to a specified size with a wire EDM, one side of the Nb substrate was polished to R_a<1 nm [3] in order to ensure that the coated surface is as parallel as possible with the external parallel magnetic field.

Based on previous experience [4] the Nb substrates were baked at 800 °C under vacuum for 4 hours in a Ti box before coating in order to improve the surface resistance. The alumina (Al₂O₃) was deposited using an atomic layer deposition (ALD) technique at 300 °C. The MgB₂ was deposited at STI using a reactive evaporation (RE) technique at 550 °C [5].

FLUX PENETRATION FIELD (B_{pen}) MEASUREMENTS

B_{pen} is defined as the field at which a large number of vortices or magnetic fluxons start to penetrate into the material. This quantity was measured using a Quantum Design Magnetic Property Measurement System (MPMS).

Figure 3 shows the magnetization curves of 200 nm MgB₂ film prepared with reactive co-evaporation technique at STI. The B_{pen} was taken as the point where slope changes drastically as shown in Fig. 3.

Figure 4 shows B_{pen} as a function of temperature for 200 nm, 300 nm and 500 nm MgB₂ films together with the data for bulk Nb and sputter-coated Nb films for comparison.

As shown in Fig. 4, it was found that the thickness effect is already observed with 500 nm and 300 nm films, i.e., 3-5 times λ (~110 nm). Also, it was successfully shown that the B_{pen} of ≤300 nm MgB₂ films prepared with RE technique possess higher B_{pen} than that of bulk Nb, which implies the increase of fundamental limit for the achievable E_{acc} of SRF cavities.

We have not evaluated the effect of surface roughness on B_{pen}, and it will be an important topic for future research toward more realistic cavity surfaces.

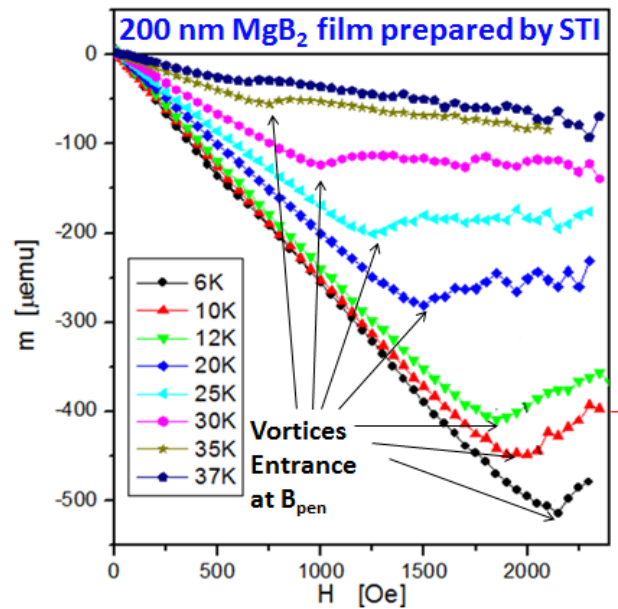


Figure 3: Total magnetic moment as a function of H_{||} for 200 nm MgB₂ film prepared by STI. The data points taken as B_{pen} are shown with arrows.

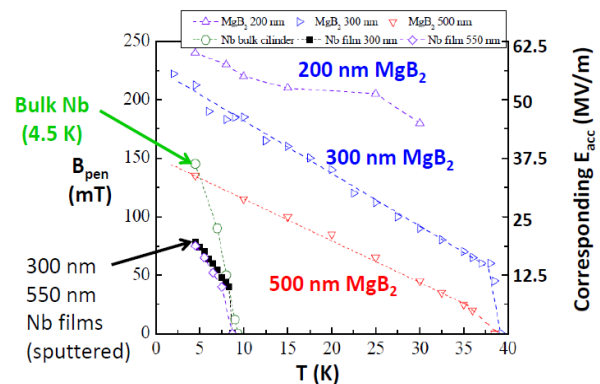


Figure 4: B_{pen} as a function of temperature for 500 nm, 300 nm and 200 nm MgB₂ films prepared by reactive co-evaporation technique at STI together with single-grain RRR>300 bulk Nb and sputter coated films for comparison.

What does B_{pen} Mean?

For Type II superconductors, i.e., κ (= λ/ξ) > 1/√2, it is known that the Meissner effect sometimes continues to above B_{c1} due to energy barrier up to the superheating field, B_{sh}. In that sense, the measured B_{pen} should be very close, or equal, to B_{sh}.

Relationship between Enhanced B_{c1} and B_{pen}

Figure 5 shows the predicted curve of enhanced B_{c1} assuming the coherence length ξ = 6 nm and C = 1.07ξ together with the measured B_{pen} in solid circles. It also shows the assumed penetration depth λ = 110 nm.

As one can see, it has been observed that the B_{pen} increases as the film gets thinner from 500 nm to 300 nm, and from 300 nm to 200 nm. All the B_{pen} data showed

higher than the predicted B_{c1} . While we have been unable to measure the B_{pen} for thinner films since the signal gets too small, we predict that the ultimate limit of B_{pen} will be the close to the thermodynamic critical field B_c calculated by the following formula.

$$B_c = \frac{\phi_0}{2\sqrt{2}\pi\lambda\xi} \quad (1)$$

Using $\lambda = 110$ nm and $\xi = 6$ nm, one gets $B_c = 353$ mT.

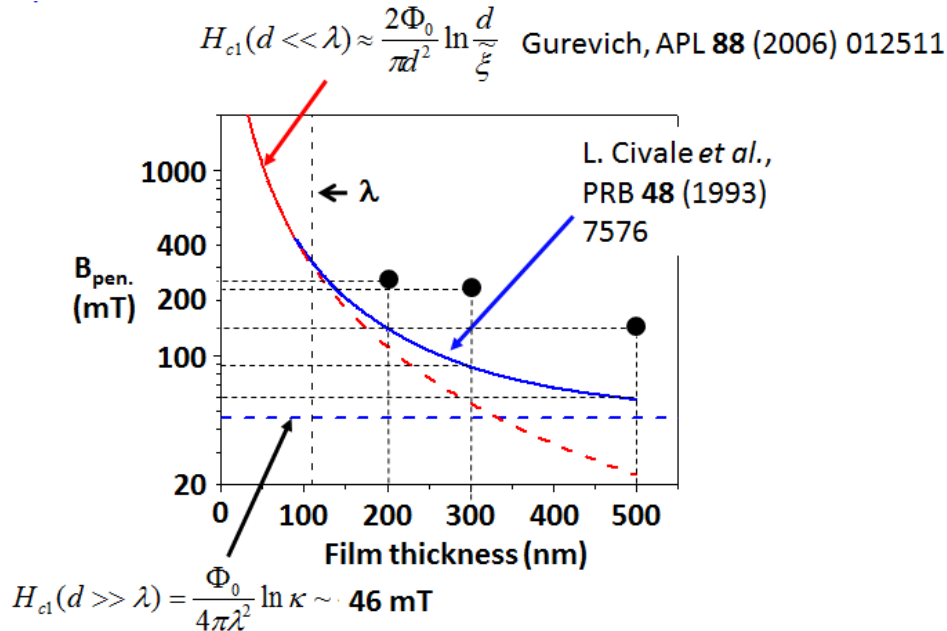


Figure 5: B_{pen} of MgB_2 films as a function of film thickness at 4.5 K. Predicted B_{c1} using the formulae shown in the references in the figure are also shown for comparison.

11.4 GHz RF MEASUREMENTS

SLAC National Accelerator Laboratory has several 11.4 GHz high-power (30-50 MW) pulsed Klystrons that can be used for materials testing at high electromagnetic fields.

A hemi-spherical TE_{013} -mode cavity made of copper was developed to test the effect of high magnetic field on the sample surface [6]. Figure 6 shows a cross section of the cavity. The magnetic field on the sample surface is parallel to the sample surface as in other TE_{01n} cavities and, as shown in Fig. 7, the magnetic field is axisymmetric and exhibits Bessel function in the radial direction with a peak at half the radius of the sample.

This system is able to reach a peak magnetic field (B_{peak}) up to ~ 400 mT with the existing configuration. Most tests were carried out with a pulse width of $1.6 \mu s$ at a repetition rate of 1 Hz.

Low-Power Tests

The quality factor Q of the cavity was measured as a function of temperature at low power. Since the parts of the cavity other than the sample are made of copper, the highest cavity unloaded quality factor Q_0 is limited to $\sim 3.5 \times 10^5$ with zero resistance on the sample. As a

result, the lowest measurable RF surface resistance is $\sim 100 \mu\Omega$.

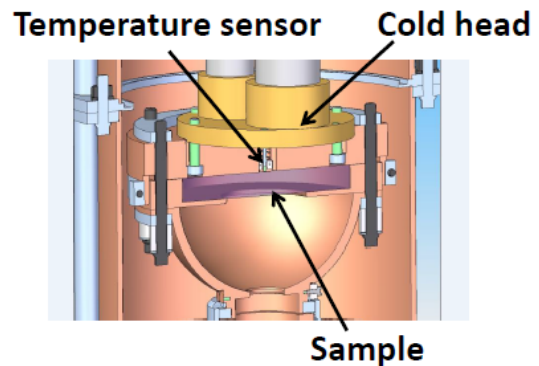


Figure 6: 11.4 GHz TE_{013} -mode hemi-spherical copper cavity at SLAC. Pulses of $1.6 \mu s$ in width and a rep rate of 1 Hz were used.

Figure 8 shows Q_0 of the cavity as a function of temperature for a sample at the following four stages: 1) as polished to $R_a < 1$ nm, 2) after UHV baking at $800^\circ C$ for 4 hours, 3) after a coating of 20 nm alumina at $300^\circ C$, and 4) after a subsequent coating with 100 nm MgB_2 at $550^\circ C$. The alumina was coated by atomic layer deposition (ALD) at NIMS, Tsukuba, Japan, and the MgB_2 was coated by RE at STI, Santa Barbara, CA, USA.

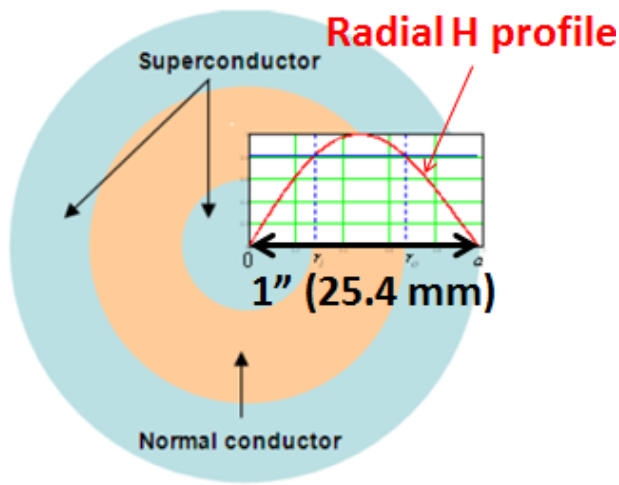


Figure 7: A schematic representing the sample surface when it partially quenched. A ring shown in beige represents the normal-conducting area due to the magnetic field being higher than the critical field shown in a blue line in the inset.

As shown in Fig. 8, the R_s , which is inversely proportional to Q_0 , decreased significantly after UHV baking. However, the 20 nm alumina coating significantly increased R_s in both normal-conducting and superconducting phases. The subsequent 100 nm MgB_2 coating further increased R_s at <9 K, i.e., when Nb is in the superconducting state, although R_s decreased at >9 K.

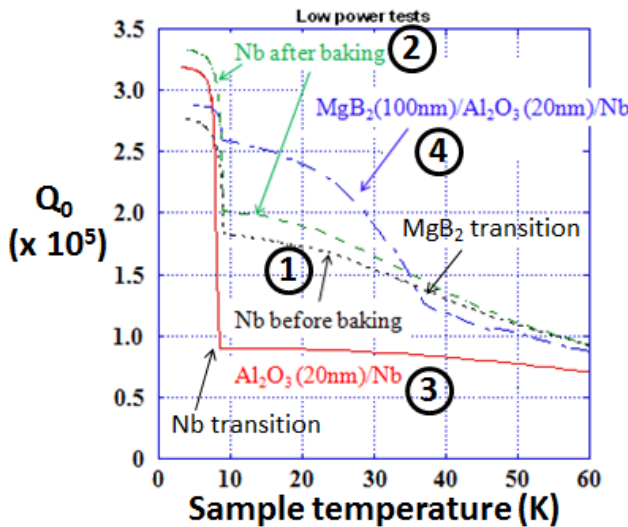


Figure 8: Q_0 as a function of sample temperature of as polished, baked, alumina-coated and MgB_2 /alumina-coated samples at low power. Due to the copper host cavity, the ultimate limit of the Q_0 is $\sim 3.5 \times 10^5$. See the detail in the text.

High-Power Tests

A unique feature of the SLAC system is its capability of producing very short ($\sim \mu s$) pulses with magnetic fields high enough to test materials that could show higher critical fields than Nb. Initially, it was anticipated that the quench that is detected in this system will be magnetic,

not thermal due to the short pulse width. However, as will be shown later, it was found that thermal quenches could occur in the case of the sample with high RF surface resistance.

Peak Power Density that Causes Thermal Quench

With the SLAC system, the following relationship between B_{peak} , Q_0 and P_{diss} (peak power density) holds.

$$Q_0 = \left[8.08 \times 10^{-10} \times \frac{P_{diss} [W/m^2]}{\{B_{peak} [mT]\}^2} + 2.87 \times 10^{-6} \right]^{-1} \quad (2)$$

Figure 9 shows two Q_0 - B_{peak} curves. The red curve is from a single-grain Nb. The blue curve is a sample after 20 nm alumina was coated on a similar Nb substrate using ALD at 300 °C. The quench is indicated as a shoulder of this curve. By taking the shoulder of the alumina/Nb sample, $P_{diss} = 1.2 \times 10^6$ W/m² was determined as the power density that causes thermal quench. With the pulse width of 1.6 μs and rep rate of 1 Hz, this translates into a time averaged power density of 1.9 W/m². The black line in Fig. 9 and the red line in Fig. 10 show the Q_0 - B_{peak} curve with this P_{diss} .

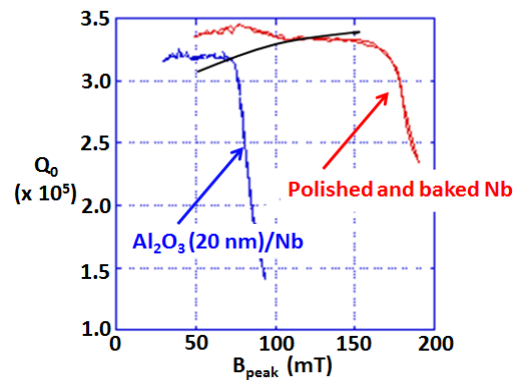


Figure 9: Q_0 as a function of peak magnetic field at 3 K for the best Nb sample that was polished to $R_a < 1$ nm and baked under vacuum, and for the sample with 20 nm alumina coated on Nb by ALD.

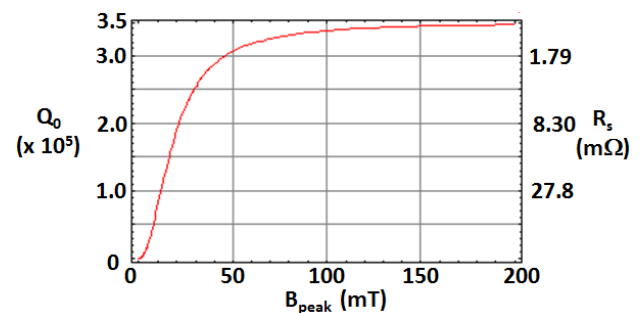


Figure 10: Q_0 vs. B_{peak} curve in the case of $P_{diss} = 1.2 \times 10^6$ W/m² in Eq. (2).

Quenches on a $MgB_2/Al_2O_3/Nb$ System

Figure 11 shows the Q_0 - B_{peak} curves for $MgB_2(100\text{ nm})/Al_2O_3(20\text{ nm})/Nb$ system at 4 K, 7 K and 10 K. The Q_0 - B_{peak} curve that causes thermal quench is also shown with a solid black line in Fig. 11. As one can see, the shoulders that indicate quench are in good agreement with this curve suggesting that these quenches are thermal, not magnetic.

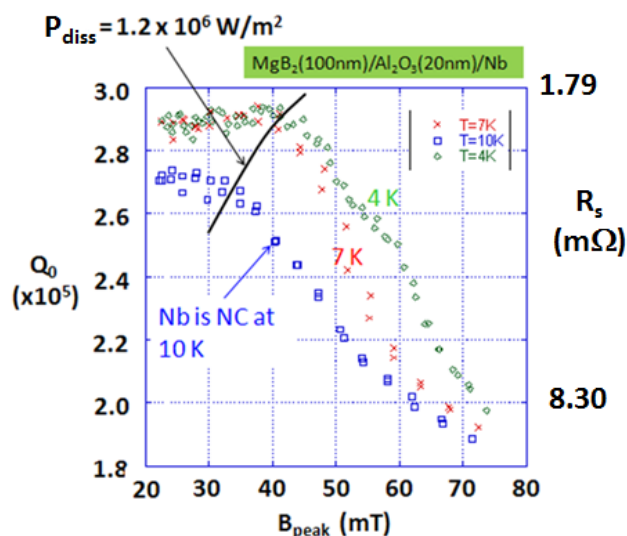


Figure 11: Q_0 as a function of B_{peak} at 4 K, 7 K and 10 K for the $MgB_2(100\text{ nm})/Al_2O_3(20\text{ nm})/Nb$ sample shown in Fig. 8. The solid line is a curve described by Eq. (2) with $P_{diss} = 1.2 \times 10^6\text{ W/m}^2$.

In Fig. 11 also shown is the corresponding surface resistance (R_s) derived from the obtained Q_0 . Considering the Nb BCS R_s of $\sim 40\ \mu\Omega$ at 11.4 GHz at 4 K, the measured resistance of $\sim 2.2\text{ m}\Omega$ before quench is 56 times higher.

INTER-DIFFUSION PROBLEM

An issue identified through this study was a significant increase in R_s after coatings of both alumina and MgB_2 . Figure 12 shows an Auger Electron Spectroscopy (AES) depth profile of the $MgB_2(100\text{ nm})/alumina(20\text{ nm})/Nb$ sample described in the previous section. A significant inter-diffusion of all the components is observed at the interfaces between MgB_2 , alumina, and Nb. These interface layers are probably responsible for the high R_s , e.g., the fact that the stable low-loss Nb_2O_5 layer on Nb decomposes into lossy NbO_x at $>250\text{ }^\circ\text{C}$ is well known [7].

Producing low-loss (interface) layers will be an important objective for the future development.

RECENT ADVANCES AT TEMPLE UNIVERSITY

Temple University took over the activities that had been developed at Penn State University led by Prof. Xi when he moved to Temple University.

After about one year of building up the facilities at Temple University, they have built nice facilities for sample studies and a small cavity coating.

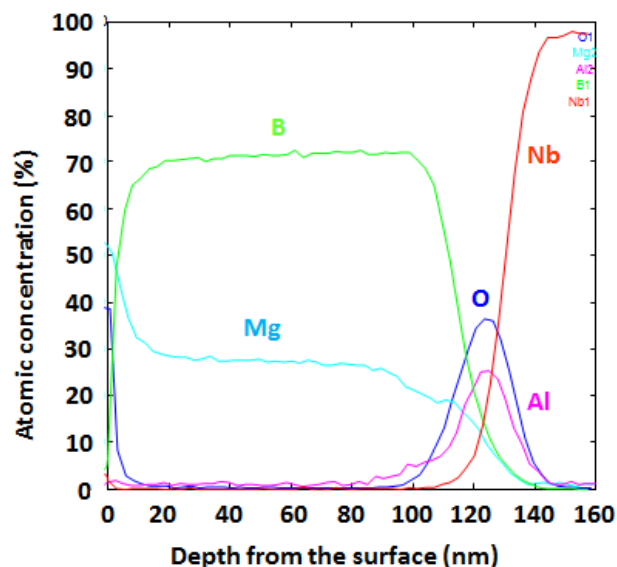


Figure 12: Auger depth profile of the $MgB_2(100\text{ nm})/Al_2O_3(20\text{ nm})/Nb$ sample shown in Fig. 8. Inter-diffusion of coating components at the interfaces is observed.

Figure 13 shows the coating furnaces for small and up to 2-inch (50.8 mm) diameter flat disks. Figure 14 shows a produced 2-inch disk and a 1 cm x 1 cm sample. Very smooth and high-quality films have been made. More details on their films are shown in their papers presented in this conference.



Figure 13: Reactors at Temple University for small size (left) and for 2-inch diameter (right) MgB_2 films.

CONCLUSIONS

The demonstration of Gurevich's proposal to enhance magnetic breakdown field by adding multilayer thin film superconductors, which in turn will lead to an increase in accelerating gradients of SRF cavities, is our goal. As a first step, to know the fundamental limit of MgB_2 films in terms of magnetic field, B_{pen} , the magnetic field at which

a large number of vortices or fluxons start to penetrate into the superconductor, was measured.



Figure 14: 50.8 mm diameter (left) and 1 cm x 1 cm (right) samples prepared at Temple University using a hybrid physical chemical vapour deposition (HPCVD) technique.

While thin films with a thickness comparable to penetration depth have not been measured due to technical difficulties, it has been found that even thicker films with a thickness 3-5 times the penetration depth show an increase in B_{pen} . For example, 500 nm and 300 nm MgB_2 films showed B_{pen} of ~ 135 mT and ~ 210 mT at 4.5 K, respectively, compared to $B_{c1} \sim 46$ mT for bulk MgB_2 calculated from $\lambda(0) \sim 110$ nm and $\xi(0) \sim 6$ nm. Also, the fact that the B_{pen} of ≤ 300 nm MgB_2 films show higher number than that of Nb implies that the fundamental limit of MgB_2 is higher than Nb, a very encouraging news.

RF measurements of a $MgB_2(100 \text{ nm})/alumina(20 \text{ nm})/Nb$ sample, however, showed quenches at ~ 42 mT at 4 K, which is significantly lower than what is expected from the magnetization measurements (>90 mT). From the data analysis, it has been found that this quench was thermal, not magnetic, due to a high surface resistance

caused by inter-diffusion of coating components during coating processes.

The key to the success of this demonstration is to develop a coating technology to reduce this inter-diffusion in order to reduce the RF surface resistance to a level usable for SRF cavities.

REFERENCES

- [1] S. Aderhold et al., "Update on Large Grain Cavities with 45 MV/m in a Nine-Cell Cavity at DESY," TTC-Report 2011-01.
- [2] A. Gurevich, "Enhancement of rf breakdown field of superconductors by multilayer coating," Appl. Phys. Lett. 88, (2006) 012511.
- [3] S. Lesiak of Cabot Microelectronics Polishing Company, private communication.
- [4] J. Guo et al., "Cryogenic RF Material Testing at SLAC," PAC 2011, New York, 28 March – 01 April 2011, TUP102; <http://www.JACoW.org>
- [5] B.H. Moeckly and W.S. Ruby, "Growth of high-quality large-area MgB_2 thin films by reactive evaporation," Supercond. Sci. Technol. 19 (2006) L21.
- [6] J. Guo et al., "A Cryogenic RF Material Testing Facility at SLAC," IPAC'10, Kyoto, May 2010, WEPEC073, p. 3049 (2010); <http://www.JACoW.org>
- [7] F.L. Palmer et al., "Oxide overlayers and the superconducting rf properties of yttrium-processed high purity Nb," Nucl. Instrum, Meth., A297 (1990) 321.