MgB₂ THIN FILMS ON COPPER, TITANIUM, AND NIOBIUM BY PULSED LASER DEPOSITION IN KEK⁺

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Abstract

We have been engaged in fabrication of high-Tc or MgB_2 thin films on metallic substrates. At the international workshop on thin films in Padova, we showed our basic idea to make an accelerating-mode cavity. In the first half of this paper, we report a subsequent development, mainly a partial success in fabricating superconducting film on a quadrant cavity of an accelerator structure. In the latter half, we describe some results concerning fabrication of films on Titanium and Niobium surfaces.

FABRICATION OF MGB FILMS ON ACCELERATING CAVITIES

As reported earlier [1],[2], we intend to make cavities of metallic material (especially of copper) covered with superconducting films. In order to develop a fabrication technique, we have been concentrated on making films on copper disks of 36 mm in diameter. There, the microwave surface resistances were measured

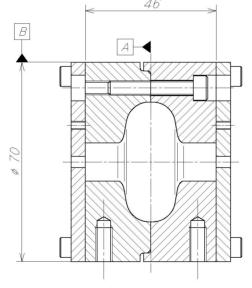


Figure 1: A 1/3 scaled-down cavity for MgB₂ films. The left part is called f-part and the right m-part in the text.

using a TE_{011} mode host cavity, in which the current does not flow through the contact plane between the host cavity and the sample disk.

We now shift to making cavities of accelerator mode, i.e., those of the TM-like field. The shape of the cavity is a 1/3 scaled-down model of the ILC superconducting cavity as shown in Fig. 1. (The original frequency is 1389.63 MHz.) Here, in comparison with simple flat disks in the preceding experiments, the cavity shape is so complicated that we need to solve two problems simultaneously; machining the cavity and forming the film. Both problems are interrelated each other.

(f-i) To avoid interception of the current flow in the longitudinal direction, a cavity could be constructed with multiple components divided in the longitudinal direction. For example, a cavity could be composed of four quadrant parts, one quadrant of which looks like something in Fig. 4. However, it is to be noted that the quadrant of Fig. 4 is composed of two parts as described in (f-ii). In this case, we could expect a rather uniform film deposition and less stress in the annealing process at the equator line. However, it is very difficult at the present state of art to machine the inner surface of the cavity by a milling technique.

(f-ii) A cavity could be assembled with two parts cut vertically to the axis as shown in Fig. 2, i.e., a part denoted by from the upper-left to lower-right slanted lines (denoted m-part hereafter) and that denoted by from the upper-right to lower-left slanted lines (f-part) in Fig. 1. In comparison with the process (f-i), we expect disadvantage in the contact plane or more precisely the possible fault of the film at the contact line on the cavity inner surface.

In this experiment, we adopt the latter process. From the experience of the X-band linear collider project, we have an amount of know how for finishing the contact plane. We use diamond bits to lathe the inner surface of the cavity and the contact planes. In the case of electron linear accelerator structure, since the mirror plane is so complete that the two parts are not easily detachable once the two parts come into contact. The contact plane around the inner surface is chamfered with 0.1^{R} to avoid swelling to the cavity inner surface.

PRECURSOR POST-ANNEALING METHOD

After assembling the cavity with the component parts, we deposit MgB_2 on the inner surface by pulsed laser

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Figure 2: Two parts machined vertical to the axis are assembled to make a cavity. The inside surface is to be covered with MgB_2 films.

deposition (PLD) and heat up to temperatures around 600 C. This procedure is called precursor post-annealing method.

This method consists of two steps.

(i) The target is a pellet with a mixture of MgB₂ and Mg powder with the stoichiometry of Mg:B=2:1. Excimer laser with λ =248 nm (KrF), 400 mJ, and 5–10 Hz is irradiated on the target in 6x10⁻⁵ Torr Ar atmosphere at room temperature for 2 hr. The thickness of the fabricated film is around 1 μ m.

(ii) Typically, the precursors are heated in 1 atm Ar gas at 550–650°C for 10 min. This process must be optimized so that magnesium evaporation should be well controlled to become MgB₂ in the phase diagram [3]. The reader is asked to refer the papers by Fukutomi *et al.* [4,5] for further details.

In the preceding experiments, we deposited chromium on the copper surface, then yttria-stabilized-zirconia (YSZ), and MgB_2 . However, we now know that if the copper oxide is completely removed from the surface, the chromium buffer layer can be omissible.

THE PULSED LASER DEPOSITION

The deposition of the mixture of the powder on the inner surface of the cavity with a satisfactory accuracy is abandoned at the present state of the experiment. We have tried two methods so far.

(p-i) A cavity is assembled from m- and f-part using copper bolts as Fig. 1 aside from the both endcap. The copper oxide layer is removed in a hydrogen furnace. A target, a rod with a cone-end of YSZ, is inserted in the middle of the cavity. Pulsed laser depositing with a laser beam scanned over the cone as shown in Fig. 3. After transposing the cavity, pulsed laser depositing on the other side. Repeating the same procedure with a target formed from MgB₂ and Mg powder. Finally annealing the whole cavity. Since this process is expensive, we tried only one for copper (and niobium) cavity, respectively.

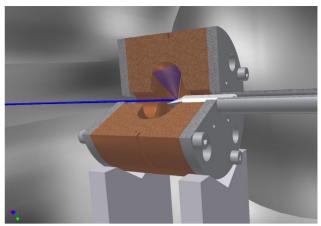


Figure 3: PLD on the inner surface of the assembled cavity.

(p-ii) After assembled, the cavity is wire-cut into four quadrants as shown in Fig. 4. (Note that this figure shows the state after YSZ is deposited, so that the color of the inner surface is not that of copper just after deoxidization.) Different from the fabrication technique described in (f-1), the contact problem between the two parts is unavoidable in this process. After removing the copper oxide layer in a hydrogen furnace,



Figure 4: A quadrant cut from the assembled cavity. The contact plane can be seen along the equator line and the cross section. Here, the inner surface is covered by YSZ.

each quadrant is separately PLDed and annealed in the same way as the case of the 36ϕ copper disks. However, since the mass of the quadrant is about thirty times as large as that of the 36ϕ disk, the same temperature regulation as that of disk is not realized yet. Finally the quadrant part was annealed. Figure 5 shows four annealed quadrants. Since annealing condition is different for each quadrant, the color of reflection looks very different each other. The upper left one showed a transition to superconductivity.



Figure 5: Four annealed quadrant. The upper left one became superconductive.

So far, including the experiments with copper disks, a brown or gold colored film becomes superconducting.

MEASUREMENT OF THE SURFACE RESISTANCE

In the case of (p-1), the microwave surface resistance, Rs, was measured as is. The number of samples made by this process is only one. Figure 6 shows the result as compared to the copper cavity of the same dimension. A weak signal of transition can be recognized at 22 K.

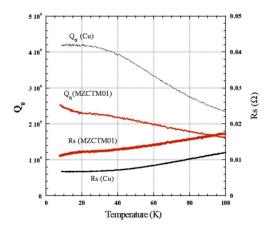


Figure 6: Q-value of the cavity fabricated with process (pi).

The sample was re-annealed at around 10 $^{\circ}$ C higher temperature. However, it was unsuccessful.

In the case (p-ii), the surface resistance of the sample is measured with a three-quadrant host copper cavity as shown in Fig. 7. First, using a copper quadrant and the three-quadrant host cavity, the whole Q-value of the copper was measured.



Figure 7: Host cavity for the measurement of a quadrant.

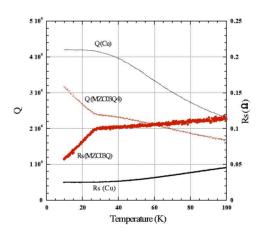


Figure 8: Microwave surface resistance of a quadrant.

Since the copper quadrant losses one fourth of the whole cavity, and from the form factor, we calculated Rs_{Cu} . Then, the copper quadrant was replaced by a sample quadrant, and the whole Q -value , $Q_{3/4Cu+MgB2}$ was measured. Since the loss of the host cavity is due to three times Rs_{Cu} , and we can find Rs_{MgB2} .

Up to now, four quadrants were fabricated. One sample among them showed a clear transition from the normal to superconducting state as shown in Fig. 8. However, the surface resistance at the lowest achievable temperature in our instrument was still higher than that of copper. The other three samples showed no indication of transition.

RF MEASURMENTS OF MGB2 ON NB AND TI

MgB2 films were formed directly on Nb disk and Ti disk by precursor post-annealing method.[6] The samples were set on host cavity made of copper as shown in Fig 9.

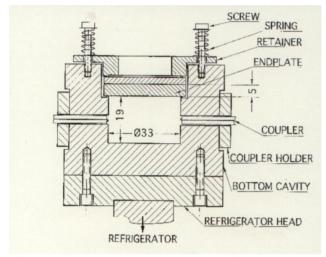


Figure 9: RF measurement cavity made of copper and the endplate was replaced with a sample.

The surface resistances Rs were measured for MgB2 film deposited Nb disc and bare Nb disk.

Fig.10 shows the measured Q_L and Q_0 of Nb and Fig.11 shows those of MgB2 film.

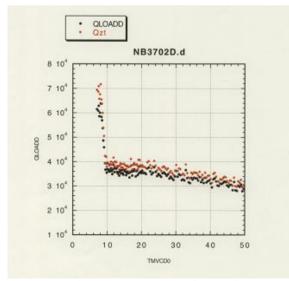


Figure 10: QL and Q0 for Nb disc.

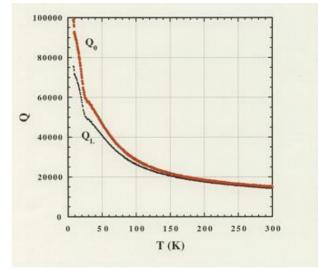


Figure 11: Q_L and Q₀ for MgB₂ film on Nb.

 Q_0 of MgB_2 film on Nb data clearly shows suddenly changes at 25 K and 9 K corresponding to critical temperature Tc of MgB2 and Nb. The Rs of each materials is calculated using that of copper host cavity .

Fig.12 show measured $\mbox{ Rs}$ of Nb and \mbox{MgB}_2 on Nb.

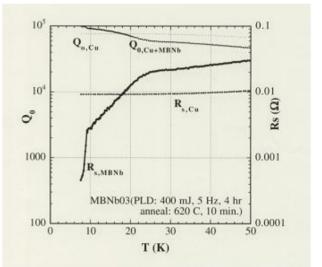


Figure 12: Rs and Q_0 of MgB₂ film on Nb and those of copper.

Rs of MgB_2 on Nb also present indication of superconducting transition of MgB2 at 25 °K. Superconducting transition also observed at about 9 °K corresponding Tc of Nb, so, if film is uniform, it is expected, MgB2 film shield RF field partially.

Rs of MgB_2 on Ti is shown in Fig 13. The transition was observed about 23 K.

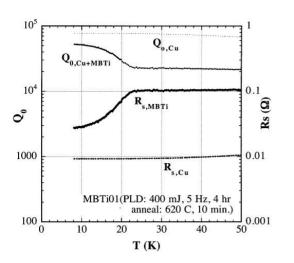


Figure 13: Surface resistance of MgB₂ on Ti.

SUMMARY

We are making films by a precursor post-annealing method on copper cavities of an accelerator structure. Some of them have already showed superconducting properties. We concentrate our effort to find the better condition to get films of better quality. We hope we can make a high power test in not-so-far future.

 MgB_2 thin films on Nb disks show partial shielding effect which is important for high field cavity.

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REFERENCES

- S. Inagaki, E. Ezura, S. Isagawa, S.Mitsunobu, H. Nakanishi,K. Saito, H. Sawa, T. Takatomi, M. Wake, M. Yoshida, M.Fukutomi, K. Kawagishi, K. Kitaguchi, Y. Kobayashi, K. Komori, H. Kumakura, and A. Matsumoto, "STUDY OF MgB THIN FILMS FOR ACCELERATOR CAVITIES IN KEK", Proc. 2006 Int. Workshop on Thin Films, Padova (2006).
- [2] Jian-Fei Liu, Kiyomitu Asano, Eiji Ezura, Sigemi Inagaki, Sigeru Isagawa, Hiroshi Nakanishi, Masao Fukutomi, Kazunori Komori and Masakazu Saito, "Precise measurement of the microwave surface impedance of a YBa₂Cu₃O_{7-δ} film on copper substrate", J. Appl. Phys. 87, 3912-3919 (2000)
- [3] Zi-Kui Liu, D.G. Schlom, Qi Li, and X. X. Xi, "Thermodynamics of the Mg-B system: Implications for the deposition of MgB thin films", Appl. Phys. Lett. 78, 3678-3680 (2001).
- [4] M. Fukutomi, S. Aoki, K. Komori, R. Chatterjee and H. Maeda, "Laser deposition of YBa Cu O thin films on a metallic substrate with biaxially textured YSZ buffer layers prepared by modified bias sputtering", Physica C219, 333-339 (1994).
- [5] M. Fukotomi, M. Ssaitoh, K. Kmori and K. Togano, "PREPARATION OF IN-PLANE TEXTURED BUFFER LAYERS FOR YBa Cu O FILM GROWTH BY MODIFIED BIAS SPUTTERING", Appl. Supercond. 4, 447-454 (1996).
- [6] S.Mitsunobu,S.Inagaki,H.Nakanishi,K.Saito,M.Wake and M.Yoshida, KEK, Tsukuba, Ibaraki, Japan, M.Fukutomi, NIMS, Tsukuba, Ibaraki, Japan, "MGB2 THIN FILMS ON NB CAVITY BY PULSE LASER DEPOSITION", Proc.SRF2007, Beijing, (2007).