**ARTICLE IN PRESS** 



Available online at www.sciencedirect.com



Physica C xxx (2003) xxx-xxx



www.elsevier.com/locate/physc

# <sup>2</sup> Surface resistance measurements of single domain $YBa_2Cu_3O_x$

# Awanish K. Mishra<sup>a</sup>, N. Hari Babu<sup>b</sup>, P. He<sup>c</sup>, D. Isfort<sup>d</sup>, X. Chaud<sup>d</sup>, Altan M. Ferendeci<sup>a</sup>, D.A. Cardwell<sup>b</sup>, Robert Tournier<sup>d</sup>, David Mast<sup>e</sup>, Donglu Shi<sup>c,\*</sup>

<sup>a</sup> Department of Electrical and Computer Engineering and Computer Science, University of Cincinnati, Cincinnati, OH 45221-0012, USA

<sup>b</sup> IRC in Superconductivity, University of Cambridge, Cambridge, UK

<sup>c</sup> Department of Chemical and Materials Engineering, University of Cincinnati, Cincinnati, OH 45221-0012, USA

<sup>d</sup> Consortium de Recherches pour l'Emergence de Technologies Avancées, CNRS, BP 166, F-38042 Grenoble Cedex 9, France <sup>e</sup> Department of Physics, University of Cincinnati, Cincinnati, OH 45221-0012, USA

Received 16 June 2003; received in revised form 6 October 2003; accepted 6 October 2003

#### 13 Abstract

3

4 5 6

7

8

9

10

11

12

20

Large single domain  $YBa_2Cu_3O_x$  materials have been successfully fabricated with superb RF properties by employing the seeded melt growth (SMG) method. Grown samples were diced, polished and oxygenated. Surface resistances of various samples were measured by a cavity perturbation technique. A sapphire loaded parallel plate cavity was used. Measured results indicate that surface resistance of the SMG bulk samples approach that of high quality YBCO thin films.

19 © 2003 Published by Elsevier B.V.

#### 21 1. Introduction

22 Cellular telecommunications systems and net-23 works have expanded rapidly in recent years, 24 which require much higher efficiency of both 25 equipment components and use of available elec-26 tromagnetic spectrum. However, conventional filtering techniques do not perform well enough to 27 28 provide the level of filtering that will be required 29 by dense, heavily used RF communication sys-30 tems. More selective filters with lower insertion

<sup>\*</sup>Corresponding author. Tel.: +1-513-5563100; fax: +1-513-5561004.

E-mail address: shid@email.uc.edu (D. Shi).

0921-4534/\$ - see front matter @ 2003 Published by Elsevier B.V. doi:10.1016/j.physc.2003.10.004

losses must be designed to satisfy this rapidly in-31 creasing demand in telecommunications. The re-32 cent development of extremely low loss RF 33 components using high temperature superconduc-34 tors (HTS) such as  $YBa_2Cu_3O_x$  has succeeded in 35 achieving the extremely low surface resistance at 36 the appropriate cellular frequencies. The current 37 technologies in fabrication of HTSs for RF ap-38 plications have been primarily the thin film ap-39 can produce proaches that well-textured 40  $YBa_2Cu_3O_x$  materials with extremely low surface 41 resistance [1-6]. HTS thin films deposited by laser 42 ablation or sputtering are lithographically pro-43 cessed to implement many type of planar micro-44 wave filters. Even though films deposited directly 45 on substrates such as LaAlO<sub>3</sub> and MgO have very 46 2

47 low ohmic losses, the loaded Q of these filters is dictated by the loss tangent of the dielectric sub-48 49 strate material. On the other hand, for low loss 50 substrates such as r-cut sapphire which has an 51 extremely small  $\tan \delta$ , the presence of buffer layers 52 decreases the overall conductivity of the YBCO 53 film [7]. The recent development in seeded melt 54 growth (SMG) of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> (YBCO) has shown 55 great promise in the fabrication of RF components with superb RF properties. Not only has the sur-56 57 face resistance of the SMG reached a value com-58 parable to those of thin films, great advantages in 59 utilizing this HTS material also lie in its suitability 60 for mass production, easy control of device ge-61 ometry, and low cost.

62 One of the concerns, however, for single domain 63 YBCO is that it inherently contains second phase 64 particles such as Y2BaCuO5 (211) which is an 65 insulating phase. As RF waves travel through the 66 surfaces, great loss can take place at these 211 regions. There have been few systematic studies so 67 68 far to investigate the effects of 211 particles on RF 69 losses. Recently, several new approaches have been 70 developed to refine these 211 particles. Although 71 the purpose of 211 refinements was for an en-72 hanced flux pinning, we expect a significantly re-73 duced RF loss in these materials

In this paper, we present experimental results on
surface resistance measurement of single domain
YBCO which can be used to implement a RF
cavity resonator with very low losses.

#### 78 **2. Sample measurements**

79 The seeded melt growth (SMG) method was originally developed for the purpose of producing 80 81 large domains for levitation applications [8–14]. In 82 SMG, based on the concept of crystal growth, a 83 small "seed" crystal of a higher melting point 84 (compared to that of the precursor) is placed on 85 the surface of the partially molten precursor ma-86 terial, and the initial growth can take place at the 87 interface between the seed surface and the liquid 88 during cooling. Furthermore, the growth assumes 89 the orientation of the seed and eventually proceeds 90 throughout the entire precursor material. In this 91 way, a large single-crystal-like domain can be obtained in the sense that the whole material possesses only one crystal orientation. 93

In this growth experiment, the samples were disc 94 shaped pellets (50 mm in diameter and 30 mm in 95 height). As precursors, 70 wt.% YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>, 30 96 wt.% Y2BaCuO5, and 0.15 wt.% Pt were used. 97 These powders were thoroughly mixed and 98 uniaxially pressed at 100 MPa into a disc shaped 99 green pellet. The green pellet was sintered in air at 100 930 °C for 24 h. A SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> single domain 101 was used as seed having a dimension of  $2 \times 2 \times 1.5$ 102 mm<sup>3</sup>. The seed was put on the top of the green 103 pellet before the growth process. The green pellet 104 was placed on an alumina plate with an interme-105 diate layer of  $Y_2O_3$  powder to avoid liquid 106 spreading on the interior surfaces of the furnace. A 107 sintered thin plate of a mixture of YbBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> 108 and  $YBa_2Cu_3O_x$  was also used between the green 109 pellet and the  $Y_2O_3$  layer. The details about the 110 YBCO single domain growth can be found in Refs. 111 [8–10]. The single domain was then sectioned into 112 a disc dimension of 15 mm in diameter and 3 mm 113 in thickness. The top of the single domain disc was 114 hand-polished down to 1/4 µm with diamond 115 paste. The polished samples were annealed in 116 flowing oxygen at 400 °C for 10 days. 117

## 3. Experimental

The surface resistance,  $R_s$ , measurements were 119 based on a dielectric cavity perturbation technique 120 [15–17]. The cavity was composed of a lower plate, 121 a cylindrical sapphire puck and a movable upper 122 plate. Fig. 1 shows the schematic representation of 123 the cavity and the measurement set-up. The cavity 124 was excited in the TE<sub>011</sub> mode. In this mode, the 125 fields decayed radially so that most of the fields 126 were concentrated around the sapphire puck. Be-127 fore a thin film sample is placed in the vicinity of 128 the resonator, the electric field  $E_1$  of the TE<sub>011</sub> 129 mode circulates around the z-axis. The corre-130 sponding magnetic field  $H_1$  is partly radial and 131 partly in the z-direction. After a sample has been 132 brought in the position, the new fields  $E_2$  and  $H_2$ 133 are assumed to retain the same symmetry. The 134 fields within the dielectric puck are hardly chan-135 ged, but the fields above the films are strongly 136

118

**ARTICLE IN PRESS** 

A.K. Mishra et al. | Physica C xxx (2003) xxx-xxx



Fig. 1. Cavity perturbation set-up and details of the cavity.

137 screened; in fact, to a good approximation  $H_2$  can 138 be taken to be parallel to the front of the sample 139 and zero behind it, and independent of the thick-140 ness and conductivity of samples.

141 The lower plate was Niobium and this plate was 142 kept at constant liquid He temperature at 4.2 K during the measurements. The upper sample plate 143 144 was connected to an external micrometer move-145 ment mechanism by a sapphire rod which was then 146 connected to a heater. A sensor monitored the 147 temperature. Heater-sensor combination set and 148 controlled the measurement temperature of the 149 sample.

150 The theory of cavity perturbation shows that as 151 a result of the film, there is a change in the formal 152 resonant frequency of the cavity and is given by:

$$\Delta f = \Delta f_0 + \frac{i}{2} \Delta f_B - \left\{ \frac{i}{2\pi} \int_S H_2 \cdot Z_s H_2 \, \mathrm{d}S' \right\} / 4W$$
  
=  $\frac{i}{2} \Gamma Z_s,$  (1)

154 where the surface integral is taken over the lower 155 surface of film, W is the total energy stored in the 156 resonator. Note that real part of the formal frequency change  $\Delta f$  correspond to the shift in the 157 actual resonant frequency  $f_0$  and is associated with 158 the surface reactance of the film, while the imagi-159 nary part of  $\Delta f$  is half the change in half power 160 bandwidth, and is related to the surface resistance 161 of the film. The quantity is known as the resonator 162 constant; it is independent of the thickness and 163 conductivity of the film, but varies with its height 164 in the resonator. 165

In principal, it is necessary to calibrate the res-166 onator constant by measuring a film of known 167 surface resistance for each height of the film. 168 However, over a considerable range of heights, the 169 bandwidth changes introduced by the film are 170 proportional to the corresponding frequency 171 change  $\Delta f_0$ . This frequency change is very much 172 larger than that associated with changes in surface 173 reactance and is therefore, almost independent of 174 the thickness and conductivity of the film. 175

The movement of the upper plate perturbed the 176 cavity parameters. The changes in the resonant 177 frequency and the bandwidth as a function of the 178 upper plate movement provided the  $R_s$  data. There 179 was no need for absolute cavity calibration. The 180

3

18 October 2003 Disk used

**PHYSC 51149** 

A.K. Mishra et al. | Physica C xxx (2003) xxx-xxx



Fig. 2. Optical photograph showing SMG-processed single domain  $YBa_2Cu_3O_x$ .

181 measured data was compared to a known material,

182 in this case copper whose surface resistance was

183 already known as a function of the temperature

184 ranges used for the HTS measurements.

### 185 4. Results and discussion

186 Fig. 2 shows the typical single domain YBCO with polished top surface. The sample was char-187 188 acterized with ac susceptibility and X-ray diffrac-189 tion (XRD). The results indicate that the single 190 domain sample has a single domain structure and 191 a sharp superconducting transition near 90 K. 192 These data can be found in our previously pub-193 lished papers [8–14].

194 It was essential that  $TE_{011}$  mode was properly 195 identified among other possible modes of the 196 cavity. In order to locate the resonant frequency 197 associated with the  $TE_{011}$  mode for the dielectric 198 resonator shunted with top and bottom ground 199 planes, the structure was simulated using Ansoff 196 HFFS<sup>®</sup> software.

201 The parameters used for the simulations were: 202 Sapphire cylinder with  $\varepsilon_r = 9.4$ , diameter: 6.99 203 mm, and height: 3.42 mm.

104 In order to verify the  $TE_{011}$  mode, fields of 205 various modes were plotted. Two verifications 206 were necessary for the  $TE_{011}$  mode. First was 207 based on the premise that *E*-fields circulated 208 around the *z*-direction, i.e., in the  $\phi$ -direction, and 209 the *H*-fields were partly radial and partly in the *z*- direction. A second verification of the  $TE_{011}$  mode 210 was that the fields decayed radially away from the 211 sapphire cylinder. Simulated fields satisfying these 212 requirements are shown in Fig. 3. In these simulations, it was assumed that the top and bottom 214 plates had infinite conductivity. 215

Various simulations were made by moving the top plate. Simulated change in the resonant frequency as a function of sample separation from the top of the sapphire rod is shown in Fig. 4. 219

The  $R_s$  values of SMG YBCO samples were 220 measured using the apparatus shown in Fig. 1. Fig. 221 5 shows the changes in  $\Delta BW$  as a function of  $\Delta f_0$  222 at a fixed temperature for a given sample. The 223 slope of the HTS sample is compared with the 224 slope of a known Cu sample. 225

Fig. 6 shows the change in the Q of the cavity as 226 a function of the top plate distance away from the 227



Fig. 3. Simulated electric and magnetic field vectors for  $TE_{011}\xspace$  mode.

4





**PHYSC 51149** 

18 October 2003 Disk used

Fig. 4. Simulated and measured resonant frequency of the cavity as a function of upper plate distance.

228 top of the sapphire cylinder at 4.2 K. While the top 229 plate was moved away from the sapphire cylinder, 230 the only loss terms were due to the Niobium lower 231 plate and the loss tangent of the sapphire cylinder. 232 Fig. 7 shows the measured  $R_s$  values for three 233 SMG-YBCO samples. Two samples were annealed 234 in flowing oxygen at 400 °C for 10 days. One 235 sample was measured using He in the chamber dewar and the  $R_s$  values were measured from room 236 237 temperature dawn to 4.2 K (Fig. 7a). For the 238 second sample, liquid nitrogen was used and the  $R_s$ 239 measurements were made from room temperature 240 down to 77 K (Fig. 7b). As can be seen from these 241 plots that there is relatively small change in the  $R_s$ 242 values.  $R_{\rm s}$  value of YBCO in the superconducting



Fig. 6. Variation of cavity Q as function of top plate separation at 4.2 K.



Fig. 7. Measured  $R_s$  values. Lowest temperatures: (a) 4.2 K, (b) 77 K, and (c) sample annealed for a short period.



Fig. 5. Measured slope of copper and high temperature superconductor at 10 K.

18 October 2003 Disk used

**PHYSC 51149** 

**ARTICLE IN PRESS** 

6

A.K. Mishra et al. / Physica C xxx (2003) xxx-xxx

243 state decreases as the temperature decreases. The 244 minimum surface resistance value observed was 35 245  $\mu\Omega$ . As expected, above the transition temperature, 246 the values of  $R_s$  were even higher than that of the 247 Cu sample. A third sample was oxygenated for a 248 shorter period. For this sample, measured  $R_s$  val-249 ues were higher as shown in Fig. 7c.

#### 250 5. Conclusions

251 Microwave properties of seeded melt grown 252 (SMG) YBCO samples were measured using a 253 cavity perturbation technique around 18.5 GHz. 254 Well annealed samples exhibited  $R_s$  values that 255 were comparable to high quality thin film YBCO 256 samples. These SMG samples are cheaper to pro-257 duce compared to the thin films. At the same time 258 they can easily be formed into desired geometrical 259 shapes before sintering. Therefore, various cavity 260 structures can easily be processed from these low 261 loss SMG YBCO samples, thus opening up many 262 communications application areas over the mi-263 crowave and millimeter wave spectra.

#### 264 References

 265
 [1] D. Zhang et al., IEEE Trans. Appl. Supercond. 5 (1995)

 266
 2656.

- [2] M. Schmidt, W.L. Olson, IEEE Trans. Microwave Theory Tech. 39 (1991) 1475.
- [3] D. Dijkkamp, T. Venkatesan, X.D. Wu, S.A. Shaheen, N. Jisrawi, Y.H. Min-Lee, W.L. McLean, M. Croft, Appl. Phys. Lett. 51 (1987) 619.
- [4] H.C. Li, G. Linger, F. Ratzel, R. Smithey, J. Greerk, Appl. Phys. Lett. 52 (1988) 1098.
- [5] R.L. Sandstrom, W.J. Gallagher, T.R. Dinger, R.H. Koch, Appl. Phys. Lett. 53 (1988) 444.
- [6] C.B. Eom, J.Z. Sun, K. Yamamoto, A.F. Marshall, K.E. Luther, S.S. Laderman, T.H. Geballe, Appl. Phys. Lett. 55 (1989) 595.
- [7] R. Wordenweber, J. Einfeld, R. Kutzner, A.G. Zaitsev, M.A. Hein, T. Kaiser, G. Muller, Appl. Supercond., IEEE Trans. 9 (1999) 2486.
- [8] D. Shi, W. Zhong, U. Welp, S. Sengupta, V. Todt, G.W. Crabtree, S. Dorris, U. Balachandran, IEEE Trans. Magn. 5 (1994) 1627.
- [9] D. Shi, K. Lahiri, S. Sagar, D. Qu, V. Pan, V.F. Solovjov, J.R. Hull, J. Mater. Res. 12 (1997).
- [10] D. Shi, K. Lahiri, J.R. Hull, D. LeBlanc, M.A.R. LeBlanc, A. Dabkowski, Y. Chang, Y. Jiang, Z. Zhang, H. Fan, Physica C 246 (1995) 253.
- [11] P. Gautie-Picard, X. Chaud, E. Beaugnon, A. Erraud, R. Tounier, Mater. Sci. Eng. B 53 (1998) 66.
- [12] P. Cautier-Picard, E. Beaugnon, X. Chaud, A. Sulpice, R. Torunier, Physica C 308 (1998) 161.
- [13] P. Bautier-Picard, E. Beaugnon, R. Tournier, Physica C 276 (1997) 35.
- [14] D. Shi, P. Odier, A. Sulpice, D. Isfort, X. Chaud, R. Tournier, P. He, R. Singh, Physica C 384 (2003) 149.
- [15] R.J. Ormeno, D.C. Morgen, D.M. Broun, J.R. Walfram, Rev. Sci. Instrum. 68 (5) (1997) 2121.
- [16] E. Selcuki, M.S. thesis, Department of Physics, University of Cincinnati, 1992.
- [17] A. Farahat, J. Supercond. 8 (3) (1995).