WTP-80/ISS2010

Date of submission: 2.11.2010

Date of revision: 4.2.2011

Date of accept: 18.2.2011 Physica C

In-situ strain measurements of superconducting composites by depth and layer sensitive X-ray diffraction technique utilizing synchrotron radiation

H. Okuda[a], T. Arai[b], A. Toda[b], S. Ochiai[a], M. Sato[c], W. Prusseit[d]

a Dept.Mater.Sci.Eng. Kyoto Univ. Yoshida, Sakyo-ku Kyoto 606-8501

b Graduate School of Engineering, Kyoto University, Sakyo-ku Kyoto 606-8501.

c SPring8 JASRI Kohto, Sayo 679-5198 Japan

d THEVA duennschichttechik GmbH 85737 Ismaning Germany

Abstract

Strain of the layers in Dy123-based coated superconductor composite tapes under tensile load has been evaluated by in-situ synchrotron radiation diffraction near the L3 absorption edge of Dy. In the present work, in-plane diffraction profile of the materials under tensile deformation with the scattering vector parallel to the axial load has been analyzed. The axial strain evaluated from the Dy123 peaks agreed with the average sample strain obtained from a separate mechanical test in elastic region. After Lueders deformation started, the strain of the Dy123 layer remained almost constant, in agreement with a multiple fracture model for ductile-fragile composite materials. Anomalous dispersion effect was used to identify the origin of the Bragg peak.

PACS: 74.25Ld, 74.78.Fk

Keywords: oxide superconductor composite, strain measurement, synchrotron radiation, anomalous dispersion

Corresponding author

Hiroshi Okuda, Dept. materials science and engineering, Kyoto University

Yoshida-honmachi, Sakyo-ku Kyoto 606-8501 Japan

TEL:+81-75-753-5193 /FAX:+81-75-753-4841

Email: okuda@materials.mbox.media.kyoto-u.ac.jp

Introduction

Assessment of strain in oxide superconductor composites under external loads such as applied tensile loads, bending, thermal cycling, is important to understand degradation mechanism and to assess and design reliability of the materials under operations [1-4]. In-situ assessment of strain in composite materials gains its importance, in particular because of recent rapid improvement of the superconducting performance of the materials in one hand and the complex and inhomogeneous structures that make simple mechanical assessment of the strain under field use condition difficult on the other hand. The average parameters obtained from macroscopic mechanical tests or thermal measurements sometimes turned out to be inadequate to understand local degradation processes, and sometimes the contribution of the superconducting layer of interest to the overall mechanical signal was too small for separate analysis. We have demonstrated that by choosing photon energy to have a good balance between transmission and resolution, it is possible to evaluate the strain of superconducting oxides in-situ during tensile [5-7] or bending [8] deformations at ambient temperatures. For the Ag-sheathed Bi2223 composites, it has been demonstrated that fracture of Bi2223 filaments are easily extended to the fracture of the total sample for Ag-sheathed composite, while clear multiple fracture with an almost constant strain of Bi2223 fialments was observed for the superconducting composite sandwitched by stainless steel reinforcement sheets [6]. For bending deformation, on the other hand, the fracture characteristics are quite different between the tension side and the compression side of the same sample. Early fracture of the filaments was observed in the tension side with a fracture strain close to that for tensile load. In contrast, much higher strain accumulation was observed in the compression side. Such asymmetry in the irreversible strain and fracture has been reported for oxide superconducting tapes[2] and fracture strain has been exmined for bending deformation of Ag-sheathed Bi2223 composite superconductors [8]. Owing to recent developments of Dy-based high-perfomance coated superconducting tapes[9], importance of in-situ measurements of strain for such coated materials has been also increasing. Technically, it is difficult to measure the axial strain of coated materials in the same transmission geometry as reported for the Bi2223 case[5-7], because of very thin oxide layer and relatively thick Hastelloy substrate. Another important point unique for the coated superconducting tape is that the coated layer consists of number of epitaxially grown oxide layers having different compositions but similar lattice constants. In such a case, it is also necessary to pursue technical possibility of assessing the strain of each layers separately, since the important point is the strain in the superconducting layer. In the present work, $DyBa_2Cu_3O_{6+\delta}$ (Dy123) coated superconductor composited tape has been examined. Change of diffraction pattern by the anomalous dispersion effect at the Dy L absorption edge was examined and discussed with a simple model calculation.

Experimental

The samples used in the present experiments are thin film Dy123 superconducting tapes grown on Hastelloy sheet substrate with a couple of buffer layers and capped by a thin Ag layer, supplied by THEVA thin film technologies Co. Ltd[9]. The nominal thickness of the Dy123 layer in the present measurements was 2.1 μm. The sample was fixed with Al tabs, and hooked between the pins of compact tensile test machine mounted on a Huber multiaxis diffractometer at the beamline 46XU of SPring8, Sayo, Japan, an insertion device beamline with variable wavelength. As schmatically illustrated in Fig.1, tensile load was applied to the tape specimen up to 500 N in-situ, corresponding to the condition where the Lueders band broadened from the periphery of the sample towards center of the sample. The deformation of the sample during the in-situ measurement was controlled by the applied load monitored by the load cell on the in-situ test machine. The sample showed multiple fracture of oxide layers during tensile deformations after the yield point, whose origin is Lueders deformation of the substrate Hastelloy sheet, eventually leading to abrasion of the coated layers[4,10]. After the yield stress was reached, the deformation of the sample was controlled both by the stress level monitored through the load cell and the position of the propagation front of the Lueders band monitored by a ccd camera. Since the layer of interest is a thin Dy 123 layer deposited on one side of a much thicker metal tape of about 0.1 mm, in-plane diffraction was applied to obtain the axial strain during in-situ measurements. The scattering vector is parallel to the direction of axial strain with an error introduced by a grazing angle of about 1.0 degree in the present measurements.

The photon energy used in the present measurements was about 7.290 and 7.785 keV, close to the L3 absorption edge of Dy to examine the effect of anomalous dispersion on the composite epitaxial layers of oxides. Figure 2 shows the change of the atomic scattering factor of constituent elements of the present coated samples. As shown in the inset, the sample consists of upper Ag layer, a Dy123 superconducting layer, MgO buffer layer and Hastelloy substrate. In the present experiment, the incident photon energy was chosen to be just below the L3 absorption edge of Dy, 7.885 keV, and relatively far from the edge, 7.29 keV. The atomic scattering factor for Dy alone decreases at the near-edge condition, which is useful to identify each diffraction, in particular, after abration. In-plane diffraction measurements were carried out at 0, 200, 400 and 500 N, and then after yielding, at the point where the measured point was still in elastic region and at the point after the propagation front of the Lueders band went through, and then finally at the point where abrasion of the coated layer became visible.

Results and Discussions

Figure 3 gives the stress-strain curve of the sample. As reported by preceding works [3,10], Hasterlloy substrate shows a typical deformation process exhibiting Lueders bands. The applied load corresponding Lueders deformation is about 510 N for the present sample. Before the yield stress is reached, no clear development of transverse crack was found. The arrows in the figure correspond to the condition where in-plane diffraction was measured at the two photon energies.

The Bragg peaks assigned as 110 and 113 diffraction peak of Dy123 phase and 111 peak of Ag were analyzed in the present measurements. The Dy123 peaks shifted towards lower diffraction very slightly with applied tensile load. The amount of peak shift was less than 0.1 degree, with the FWHM of the peak of about 0.5 degrees. In-plane measurement has a merit that the axial strain is directly detected, whereas the measurement using reflection (Bragg) geometry suffers from low Poisson's ratio of about 10 to 20% [11,12], which makes the detection of small strain quite difficult, in particular, for the tensile case where the fracture strain is much lower than the compression case [2,5], but technically important.

Figure 4 is a summary of the axial strain obtained from present in-situ measurements.

The peaks assigned for the Dy123 layer show elongation with increased tensile load until the plastic deformation starts, and the evaluated strain in the elastic region as shown in the figure agreed with the average sample strain obtained from stress-strain curve of the sample at the respective loading shown in Fig. 3. It is reasonable since the sample deforms uniformly without bending in the elastic region. In contrast, the strain in the topmost Ag layer behaves differently. The strain obtained from Ag 111 peak scattered around almost no strain when the load increased. The left three points in Fig.4, denoted as Mult(1), Mult(2) and Abrasion, correspond to the condition that the sample was under Lueders deformation, but the front of the Lueders band does not reach the measurement point yet (Mult.(1)), the Lueders band went through the measurement point, so that the point is in the multiple fracture microstructure of the coated layer (Mult(2)), and finally, the abrasion of the coated layer started visible at the measurement point when the Lueders deformation of the sample finished and work hardening is observed (Abrasion) respectively. The load was kept at 506 N for the measurements at Lueders deformation and 528 N for abrasion condition. Under plastic deformation, the strain of Dy123 layer remains almost constant when the strain is evaluated for the most significant peak of 110, although the scatter of the evaluated strain increased. This is characteristic for multiple fracture of a brittle layer coated on ductile substrate, or of a brittle filament embedded in ductile matrix [13]. The strain of the coated layer after complete abrasion should be relaxed except for the curvature of the coating layer itself. However, in the beginning of abrasion as shown in the figure, the coated layer which is not peeled out from the substrate yet is still under strong tensile strain.

Since the layer and phase structure are generally rather complicated for composite expitaxial multilayers, having lattice parameters which are generally chosen to be close each other for excellent epitaxial growth, anomalous dispersion effect is a useful tool to identify the origin of the diffraction. In the present work, the strain of Dy123 layer during deformation is the principal interest. Therefore, the Bragg profile around the 110 and 113 peaks of Dy123 phase are examined at the two photon energies as shown in Fig. 2. The 110 diffraction profiles of Dy123 at the tensile load of 400 N are shown in Fig. 5. The position and the shape of the peaks agree each other for the two enegies, and the peak height decreased at the photon energy close to the L3 edge. When several diffractions having very close d spacing overlap in a single profile, it is difficult to distinguish the peak shift by strain of the layer of interest from that by strain of overlapping layer. From Fig.5, we may safely conclud that the strain evaluated from 110 and 113 peaks and shown in Fig.4 in the present work represents the axial strain in the Dy123 superconducting layer in the coated superconducting composite tape.

Conclusions

In-situ anomalous in-plane diffraction measurements of Dy123 coated superconductor tape under tensile load have been carried out to examine the change of axial strain in the Dy123 layer during tensile deformation. A clear transition from an elastic behavior to a multiple fracture was observed for Dy123 layers, with an almost constant tensile strain in the Dy123 layer kept during Lueders deformation. The diffractions from Dy123 layer were confirmed by using the change of the peak height using anomalous dispersion. In contrast, strain in the uppermost thin Ag layer does not agree with the axial strain in the

DyBCO layer. It might be explained in terms of surface microstructure of very thin Ag layer and/or be affected by radiation damage during diffraction measurements, but left for further examination of surface morphologies. A quantitative assessment of MgO peak was not possible in the present work due to poor statistics, and a further improved in-situ measurements on separate evaluation of DyBCO layer and MgO layer and their residual strain is now under way.

Acknolowledgments: Part of the present work has been supported by Grant-in-Aid for Scientific Research from JSPS, Japan, grant number 22360281. The synchrotron radiation measurements have been performed under proposal 2008A1895 and 2009B1860 at SPring8, Japan.

References

[1] J.E. Ekin, D.K. Finnemore, Q. Li,J.Tebrink, W.Carter. Appl. Phys. Lett., 61 (1992)858.

[2] D.C. van der Laan, J.W.Ekin Appl. Phys. Lett., 90(2007)052506.

[3] S. Ochiai, T. Arai, A. Toda, H.Okuda, M.Sugano, K.Osamura, W.Prusseit J. Appl. Phys. 108(2010)063905.

[4] S.Ochiai, K.Hayashi , K.Osamura, Cryogenics 33 (1993) 976.

[5] H.Okuda, K. Morishita, S.Ochaii, D.Doko, M.Matsui, H.Fujimoto, M.Sato, Physica C 411(2004)114.

[6] H.Okuda, H.Rokkaku, K.Morishita, JK.Shin, S.Iwamoto, S.Ochiai, M.Sato, K.Osamura , A.Otto, E.J.Harley, A.Malozemoff, Scripta Mater. 55(2006)691.

[7] J.-K. Shin, S. Ochiai, H. Okuda, Y. Mukai, H. Matsubayashi, S. S. Oh, D. W. Ha, S. C. Kim, M. Sato, Supercond. Sci. Technol. 21 (2008) 075018.

[8] H. Okuda, J. K. Shin, S. Iwamoto, K Morishita, Y. Mukai, H. Mtsubayashi, S. Ochiai, A. Otto, E. J. Harley, A. Malozemoff, M. Sato, Scripta Mater. 58(2008)687.

[9] W. Prusseit, R. Nemetscheck, C. Hoffmann, G.. Sigl, A. Luemkemann,G.. Kinder, Physica C426-431 (2005) 866.

[10] C.C.Clickner, J.W.Ekin, N.Cheggour, C.L.H.Thieme, Y.Quao, Y.-Y. Xie, A. Goyal, Cryogenics 46(2006) 432.

[11] B. ten Haken, H. H. J. ten Kate, Physica C 20(1996) 21.

[12] Q.Wang, G.A.Saunders, D.P.Almond, M.Cankurtaran, K.C.Goeretta. Phys. Rev. B52 (1995) 3711.

[13] S.Ochiai, T.Tomida, M.Tanaka, M.Hojo, H.Okuda, Composite Interface 12(2005)311.

List of Figure Captions:

Fig.1 Schematic illustration of the present measurements. Sample was mounted on a in-situ tensile test machine on a Huber multiaxis goniometer. Scattering vector is parallel to the tensile loading, i.e., axial strain is directly measured in the present set-up.

Fig.2 Real part of the atomic scattering factors for the constituent elements of the sample. The scattering factor of the Dy changes near the L3 absorption edge, while the scattering factors for other elements are kept constant.

Fig.3 Relationship between applied load and nominal strain during tensile deformation of the sample. The structure of the sample is schematically shown in the inset. The thicknesses of the Ag, DyBCO, and MgO layers and the Hastelloy substrate are 0.5, 2.1, 4.0 and 90 μm respectively

Fig. 4 Axial strain evaluated from the peak shift of the diffraction peaks corresponding to Dy 123 110（DyBCO(1)） and 113 (DyBCO(2)) diffraction peaks and Ag 111 peak. By applying tensile load, peak shift of Dy123 layer was clearly observed. In contrast, the Ag upper layer does not show significant elongation during loading.

Fig.5 110 Bragg peaks for DyBCO at the two photon energies. The peak position agrees and the peak height decreased at the near-edge condition, corresponding to the anomalous dispersion effect of the Dy L3 edge. The sample is under 400 N of tension during the measurement.

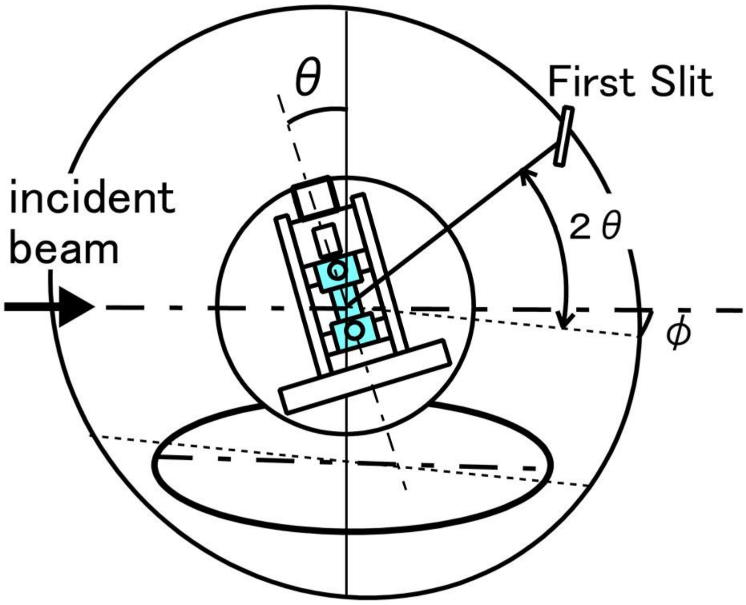

Figure.1

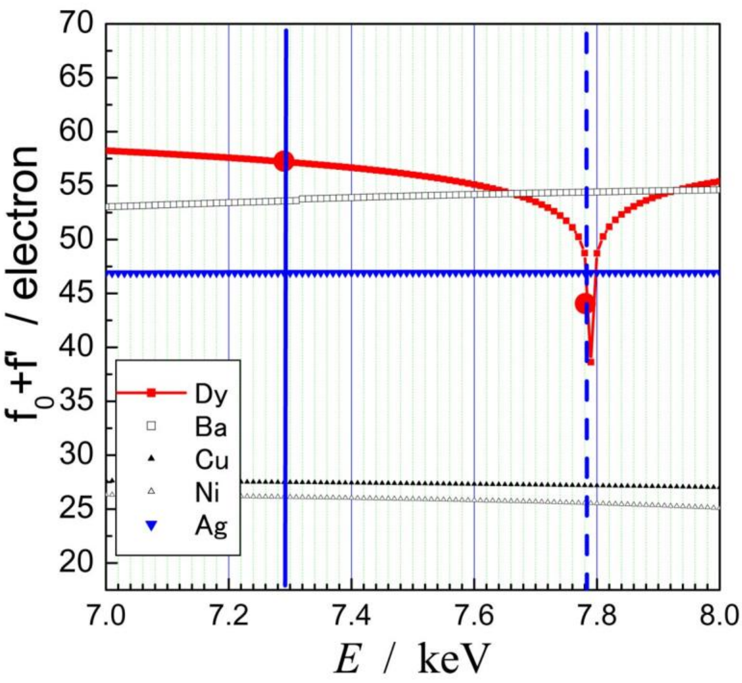
70
65
60
55
50
45
40
35
30
25
20
f_0+f' / electron
Dy
Ba
Cu
Ni
Ag
7.0
7.2
7.4
7.6
7.8
8.0
E / keV

Figure. 2

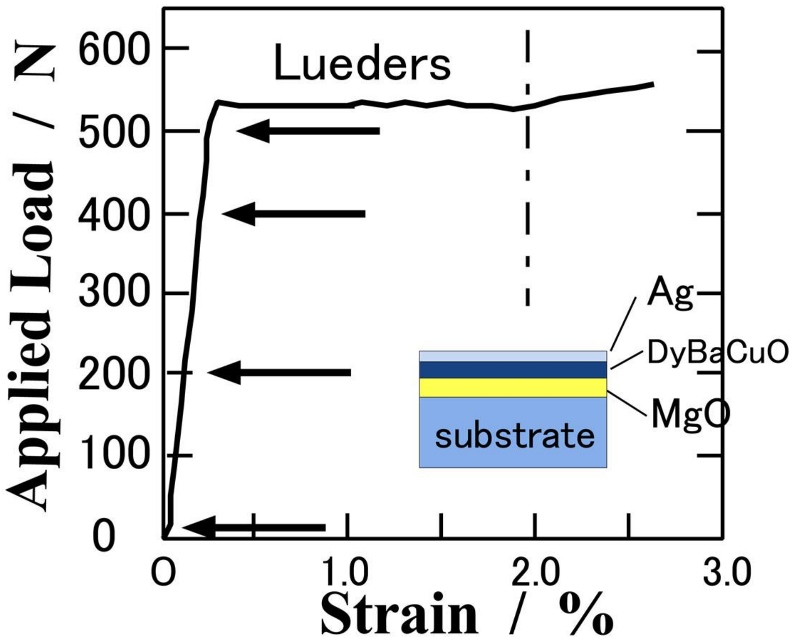
Applied Load / N
600
500
400
300
200
100
0
Lueders
Ag
DyBaCuO
MgO
substrate
O
1.0
2.0
3.0
Strain / %

Figure.3

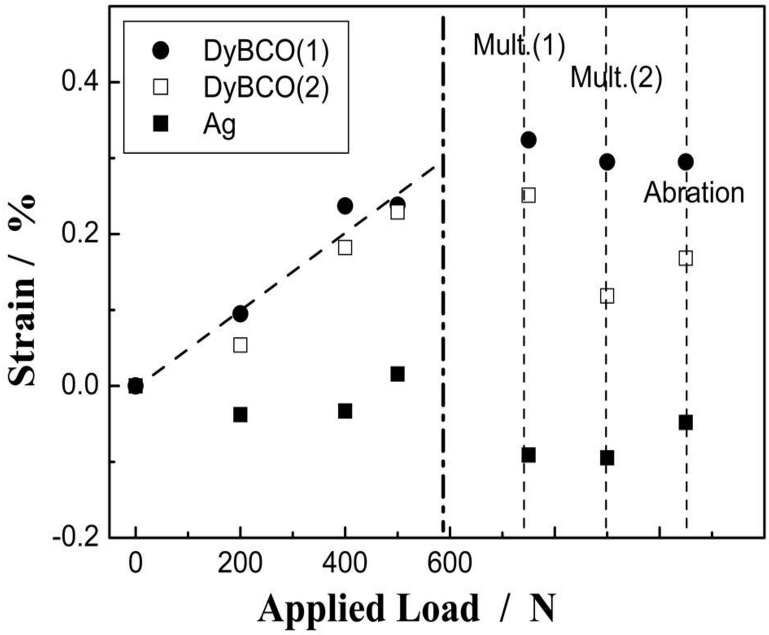
DyBCO(1)
DyBCO(2)
Ag
Mult.(1)
Mult.(2)
Abration
Strain / %
0.4
0.2
0.0
-0.2
0
200
400
600
Applied Load / N

Figure 4

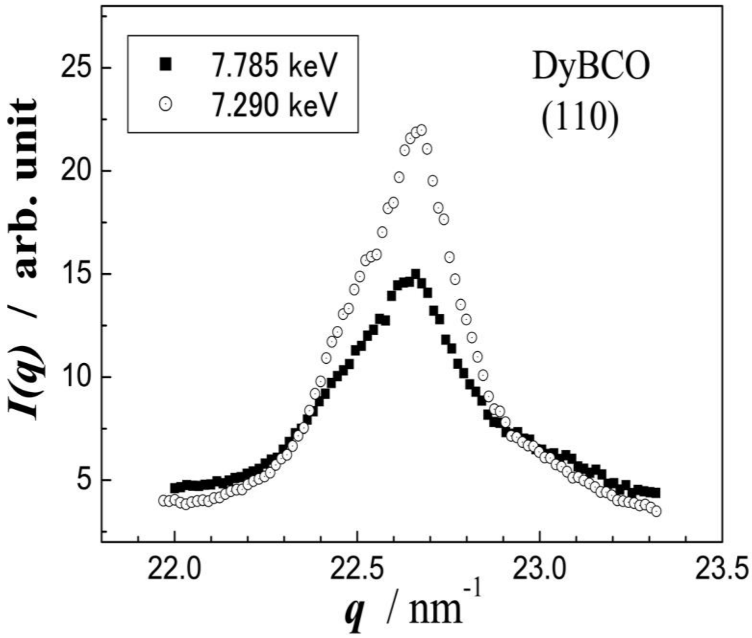
7.785 keV
7.290 keV
DyBCO
(110)
25
20
15
10
5
I(q) / arb. unit
22.0
22.5
23.0
23.5
q / nm-1

Figure 5