Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602104053
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Quantitative Analysis of Yttrium Barium Copper Oxide Films on Strontium Titanate

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Superconducting thin films of $YBa_2Cu_3O_{7-x}$ (YBCO) grown on various single crystal substrates including $SrTiO_3$ (STO), $LaAlO_3$, Al_2O_3 and MgO are of great interest because of their potential application to the generation and transmission of electrical power [1,2]. Understanding how to achieve the maximum critical current density in YBCO requires establishing the relationship between film growth parameters and a variety of other parameters including film composition, morphology, thickness, grain size, resistivity, grain orientation, and phase distribution. Therefore it is not surprising that many different characterization techniques must be used to obtain this information including x-ray diffraction, scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDS), focused ion beam microscopy, analytical transmission electron microscopy and current-voltage measurements at 77 K in self-field to determine critical current density.

This study focuses on composition and thickness measurements by SEM/EDS of YBCO films on STO ranging in thickness from 0.2µm to 1.2µm, some of which gave critical current densities of as high as 2.5 MA/cm^2. Measurements were made with a LEO 1550 SEM equipped with a ThermoNORAN Vantage EDS system. At least 10^4 counts were measured for each element in five repeat runs on samples and standards. The analysis was complicated by the presence of voids, second phase particles and overlapping EDS peaks. Fig. 1 is a spectrum obtained from a 240nm thick film. The Sr $L_{\alpha1}$ (1.802 KeV) and Sr $L_{\beta1}$ (1.871 KeV) peaks from the STO substrate and the Y $L_{\alpha1}$ (1.922 KeV), Y $L_{\beta1}$ (1.996 KeV) and Y $L_{\beta3}$ (2.075 KeV) peaks from the YBCO film are seriously overlapped. Even more overlapped are the Ti $K_{\alpha1}$ (4.501 KeV) peak from the substrate and the Ba $L_{\alpha1}$ (4.459 KeV) peak from the film. These overlapping peaks were so close together that it was difficult to use a region of interest (ROI) approach after background subtraction to obtain the peak areas for the samples. Instead, we used a full peak deconvolution approach using ORIGIN [3] software. This method provides peak areas, centroids, full width at half maximum and goodness of fit parameters that were used in our subsequent calculations. Fig. 2 shows the underlying five peaks in the Y/Sr overlapping region.

Standard peak intensities were derived from measurements on a YBCO sample that had been determined to be homogeneous by electron microprobe analysis and then analyzed chemically by inductively coupled plasma spectroscopy for the cations and by thermogravimetric analysis for oxygen [4]. The program TRYZAF developed by Armstrong [5] was used to determine the intensities expected from pure element standards based on the YBCO standard measurements. Sample to standard K ratios were then input into the program GMRFILM developed by Waldo [6] to give both composition and thickness values. This program was also used to calculate K ratios for each element for a pure $YBa_2Cu_3O_{6.8}$ standard as a function of film thickness to determine the maximum thickness at which the program could be useful. As shown in Fig. 3, at 15 KV the

https://doi.org/10.1017/S1431927602104053 Published online by Cambridge University Press

calibration will not work well above about 500nm film thickness. Therefore the analysis of thicker films will require higher beam voltages if thickness is also to be determined. If thickness is not needed, then the K ratios can be used with conventional $\phi(\rho z)$ methods to determine film composition. As an example of the use of thin film method, x-ray measurements from a 240nm film (measured by FIB) gave a thickness of 218nm and a composition in atom percent of Y 8.02, Ba 11.22, Cu 23.99 and O 56.76. These can be compared to expected values of Y 7.81, Ba 15.6, Cu 23.4 and O 53.1 atom percent. This match is not as good as what would be expected from thick, flat, homogeneous samples. Nevertheless, sets of this type of data can be used to help estimate the value of this method in providing the best possible quantitative analysis in situations where surface roughness, porosity and the presence of second phase particles may detract from the results.

References

[1] DOE report #GO-10098-434, July 1998.
[2] D.P. Norton, Annu. Rev. Mater. Sci. 1998. 28:299-343
[3] OriginLab Corp, Northampton, MA 01060
[4] E. Lifshin, L.A. Peluso and R. Arendt, Microbeam Anal, San Francisco Press 519-522 (1988)
[5] Provided by John Armstrong, National Institutes of Standards and Technology
[6] R. Waldo, Microbeam Anal, San Francisco Press 310, 314 (1988)

FIG. 1 EDS spectra of a 240nm thick YBCO film on STO substrate

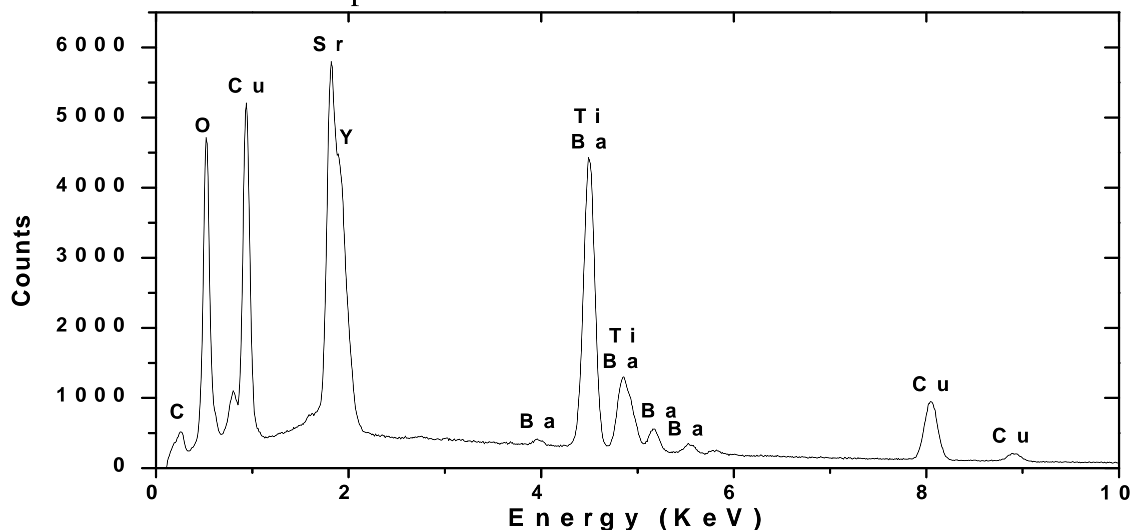


FIG. 2 Y,Sr deconvolved peaks

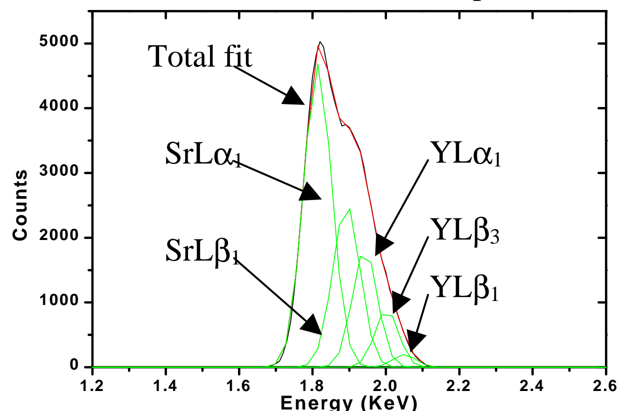
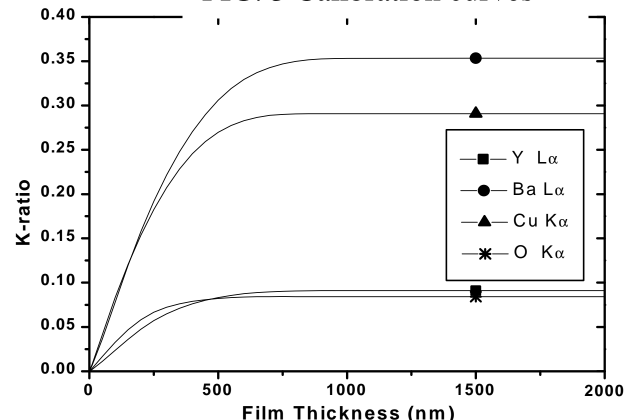


FIG. 3 Calibration curves



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