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## Evaluating Superconducting Ybco Film Properties Using Xray Photoelectron Spectroscopy

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**AFRL-RZ-WP-TP-2012-0093**

**EVALUATING SUPERCONDUCTING YBCO FILM  
PROPERTIES USING X-RAY PHOTOELECTRON  
SPECTROSCOPY (POSTPRINT)**

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## EVALUATING SUPERCONDUCTING YBCO FILM PROPERTIES USING X-RAY PHOTOELECTRON SPECTROSCOPY

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### ABSTRACT

Initial results have been recently reported that suggest a potential correlation exists between the full-width-half-maximum (FWHM) of the Y(3d) peak obtained by x-ray photoelectron spectroscopy (XPS) and the critical current density a  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  film can carry. In particular, the Y(3d<sub>5/2</sub>) demonstrated a stronger correlation. Transport currents were determined by the 4-point contact method using the 1  $\mu\text{V}/\text{cm}$  criterion. An apparent correlation was also suggested between the Y(3d) FWHM and ac loss data from magnetic susceptibility measurements. In this report, a few additional data points were acquired to further test the usefulness of the correlations. Samples were created by pulsed laser deposition of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  on  $\text{LaAlO}_3$  substrates.

### INTRODUCTION

Significant progress has been made in the development of the high temperature superconducting (HTS)  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) coated conductors.<sup>1-4</sup> These achievements have been accomplished by using a variety of techniques investigating multiple aspects of the conductor architecture and growth.<sup>5-6</sup> Successful long length development of the YBCO coated conductor will result in its availability for use in a variety of commercial applications such as power transmission cables, high field magnets, transformers, high power generators and motors, etc.

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However, many improvements can still be made in the properties of the coated conductor as well as production of longer lengths.<sup>7-9</sup> Continuing development of the coated conductor necessitates not only a detailed study of deposition techniques, source materials, deposition conditions, and substrates employed, but also characterization of the final deposited films. A technique used in this paper is x-ray photoelectron spectroscopy (XPS), especially of the YBCO layer. XPS was used to investigate the chemical and microstructural profiles of YBCO coated conductor samples.<sup>10-11</sup> Use of the technique can give consideration to interfacial issues and determine composition and chemistry at different depths in the conductor architecture.

## BACKGROUND

Comparison of the XPS spectra for the various constituents of YBCO coated conductor samples revealed that the Y(3d) photoelectron peak shape observed from the YBCO layer differed among various samples investigated.<sup>10-11</sup> This difference in XPS peak shape may indicate that there could be some difference in the atomic co-ordination between the samples. The observed difference, when compared to the particular film's properties, may also indicate the possibility of a correlation with the particular quality of the film.

In a previous study by Beyer et al.,<sup>12</sup> a difference in samples was also noted that depended on the particular substrate used. In this study, two series of samples were prepared by hollow cathode sputtering of the YBCO film, between 100 nm to 300 nm thick. One series was grown on (100) MgO and the other on (001) YSZ buffered r-plane sapphire. The  $T_c$ s were typically between 80-91 K, a wide variance by recent deposition standards. During the various treatments, the Y emission remained fairly stable, but the Ba ( $3d_{5/2}$ ) structure showed distinct differences in its lineshape in the YBCO deposited on the two different substrates. Beyer et al. noted that the differences were not caused by surface impurities but were characteristic of the samples. A structural deviation was assumed to be responsible for the difference. Even so, this does not rule out that the differences might correlate to the film's current transport properties since some substrates indeed provide better subsequent films than others.

Another possible correlation cited in the previous work is the relationship of the Y(3d) peak FWHM and the samples ac loss data from magnetic susceptibility measurements. It has been previously observed that with widening of the temperature-dependent ac susceptibility curves with increasing applied magnetic field the quality of the YBCO film generally decreases.<sup>13</sup> However, a documented study of this correlation to current transport properties using the loss component of ac susceptibility data has not been published, making it unclear how effective this correlation is.<sup>14</sup> Even so, the relationship between the Y(3d) peak FWHM and the  $\Delta T$  of the magnetic field lines reinforces the possibility. In this case,  $\Delta T$  of the magnetic field lines refers to either the difference between the temperatures at which the maxima in the ac loss occurs for the 0.025 and 2.2 Oe applied magnetic

fields (peak to peak of  $\chi''$ ) or to the FWHM of the 2.2 Oe (in particular) magnetic field data  $\chi''$  vs. T

#### EXPERIMENTAL

In this study, samples were created by pulsed laser deposition of YBCO on single crystal  $\text{LaAlO}_3$ . Previously, most samples were made in the same manner although other samples are included such as YBCO on buffered metallic tape and buffered single crystals.<sup>10</sup> The laser ablation was accomplished using a Lambda Physik LPX 305i excimer laser at the KrF transition,  $\lambda = 248$  nm with a 25 ns pulse width. Mounting of the substrates in the deposition chamber was accomplished using silver paste. Specific details of the deposition conditions were given previously.<sup>10</sup> The YBCO layers of the newly created samples were between 0.25 to 0.40 micrometers thick.

Since the previous study contained data representing a limited number of low quality samples, i.e.  $J_c < 10^6$  A/cm<sup>2</sup>, the three samples used here were particularly chosen for this study due to their poor film quality. One sample was unintentionally made with low quality and the other two were intentionally made so by specifically reducing the deposition temperature in the PLD chamber by  $\sim 100$  °C. The critical transition temperature ( $T_c$ ) of YBCO was measured by ac magnetic susceptibility and the critical current density ( $J_c$ ) by four-contact transport current measurement.

The composition and chemistry of each sample was measured by X-ray photoelectron spectroscopy (XPS) using a Kratos AXIS Ultra. The monochromatic Al  $K_{\alpha}$  x-ray line was used for enhanced spectral resolution. The analysis spot size was approximately 110  $\mu\text{m}^2$ . An electron flood charge neutralizer was used during analysis to avoid charge build-up differences between different surfaces (if any). Ion beam sputtering was performed using a mini-beam ion gun.  $\text{Ar}^+$  ions were used at an energy of 3 keV. In the raster setting used, the sputtered area was approximately a 1 mm x 0.5 mm elliptical region, several times larger than the spot size analyzed by XPS. Spectroscopic analysis was performed on as received surfaces and after subsequent sputtering to avoid the influence of surface contamination or any chemical reactivity with air.

#### RESULTS AND DISCUSSION

The  $J_c$ s of the samples were  $1.5 \times 10^5$  A/cm<sup>2</sup> for the unintentionally made poor sample,  $2 \times 10^5$  A/cm<sup>2</sup> for the one low deposition temperature sample and  $< 10^4$  A/cm<sup>2</sup> for the other low deposition temperature sample. All three YBCO samples in this study had a reduced  $T_c$ . Refer to Figure 1 for the low temperature deposition sample with the higher critical current density. The  $T_c$  for the sample made with the normal deposition temperature had a  $T_c$  of  $\sim 86.1$  K. Since samples were exposed to air the top surface was removed by sputtering. The before and after sputtering values obtained by XPS are largely uncorrelated as evidenced by

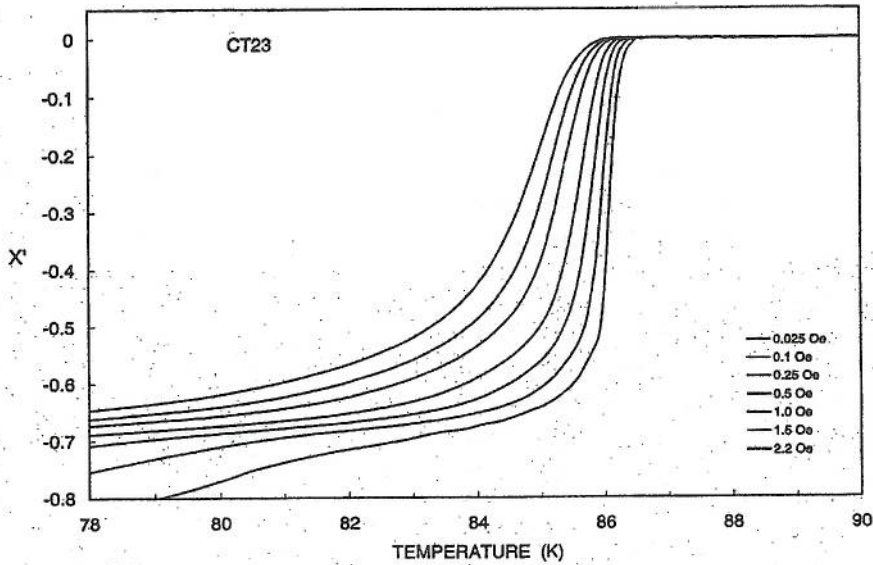


Fig. 1. The ac susceptibility data for sample CT23 made with the low deposition temperature but had a nominal  $J_c$  of  $2 \times 10^5$  A/cm<sup>2</sup>. The different curves result from the different applied fields listed in the legend—the field increases from right to left.

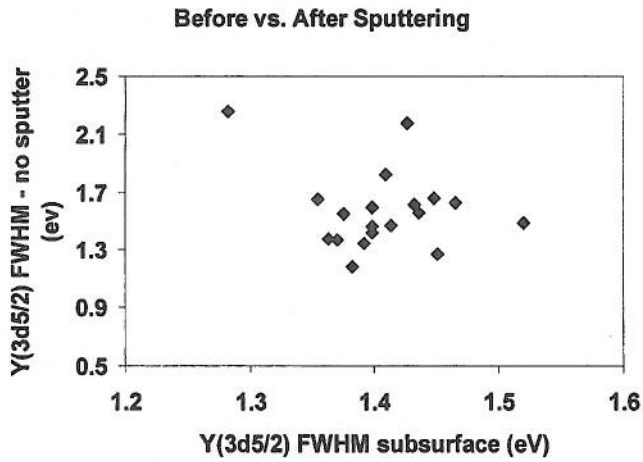


Fig. 2. Comparison of XPS data taken before and after sputtering of the surface for contaminate removal.

the scatter of the data given in Figure 2 which shows values for all samples included in the previous study, although not reported there.

In Figure 3, the relative peak intensity ratios of all samples for both studies are shown. The two stray points depicted were the two that were intentionally made poor by lowering the deposition temperature. Since all other samples had similar peak intensity ratios, it is likely that the difference caused by the low deposition temperature is due to inclusions and alternate phases incorporated during

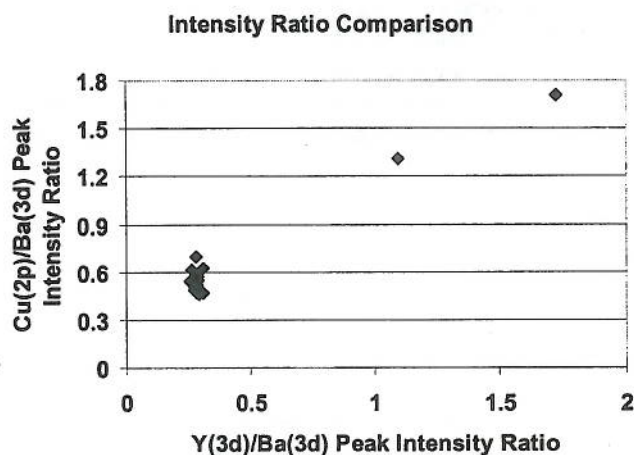


Fig. 3. Comparison of the relative XPS peak intensities of the various samples. The two compositionally stray points were made with the low deposition temperature.

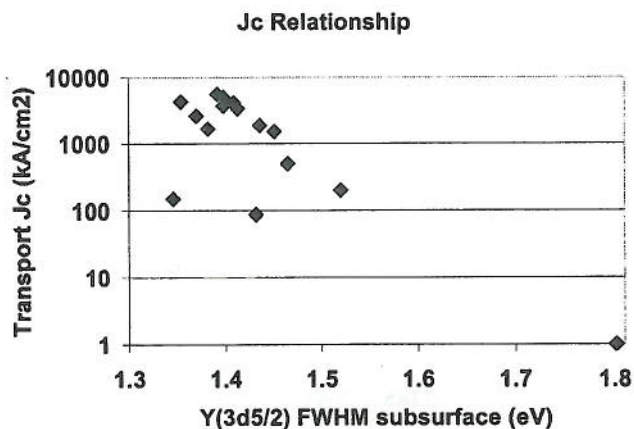


Fig. 4. Comparison of the Y(3d<sub>5/2</sub>) peak FWHM and transport J<sub>c</sub>.



deposition. As such, these compositionally stray points will be excluded from the correlation charts as being invalid for comparison.

In Figure 4 the correlation of the FWHM for the  $Y(3d_{5/2})$  peak and the critical transport current density (four point resistive) is given. The new point provided by the additional sample is located at 1.35 eV FWHM  $Y(3d_{5/2})$  with  $0.15 \text{ MA/cm}^2$ . For the given plot, this data resulted in a stray point from the other points which indicate a correlation. The point located at 1.8 eV is not stray since it fits the pattern of low  $J_c$  correlated to a larger  $Y(3d_{5/2})$  FWHM. It is not clear if the lower transport current was a result of cracks in the film which can cause a lower transport current and yet not affect the XPS spectra. However, it is interesting to note that this sample also resulted in a stray point to a greater degree on the following figure, Figure 5, which gives the relationship between the FWHM for the  $Y(3d_{5/2})$  peak and the FWHM of the critical transition temperature by susceptibility measurement.

Figure 5 depicts the relationship between the FWHM for the  $Y(3d_{5/2})$  peak and the FWHM of the 2.2 Oe magnetic field line at the critical transition temperature by ac susceptibility measurement. As previously mentioned, the new data point on this plot (5 K for  $\Delta T$ ) also strayed from the others based on the expected relationship. Exactly why there is a corroborative stray form the original data in both graphs is not clear, but a correlation between the points apparently still exists. This clearly indicates the need for additional data to determine the effectiveness of the correlations as well as determining the exact relationship of the two plots presented here. Figure 6 shows the  $\chi''$  susceptibility data for sample CT23, from Figure 1, from which the FWHM for the  $Y(3d_{5/2})$  peak was derived. The FWHM of the 2.2 Oe magnetic field data was used, although this does not imply that this is the predominant usage.<sup>13-14</sup>

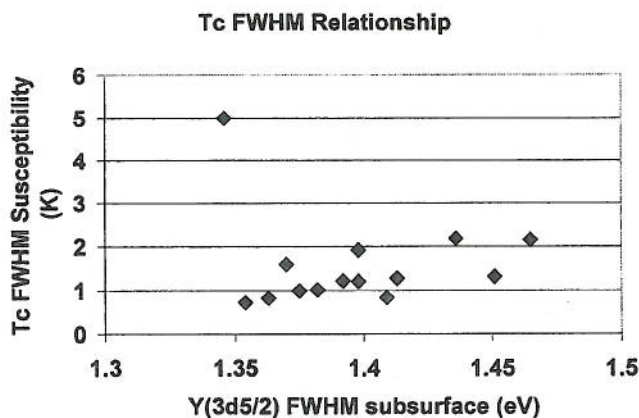


Fig. 5. Comparison of the XPS  $Y(3d_{5/2})$  FWHM and the  $T_c$  FWHM determined by ac susceptibility measurement (2.2 Oe).

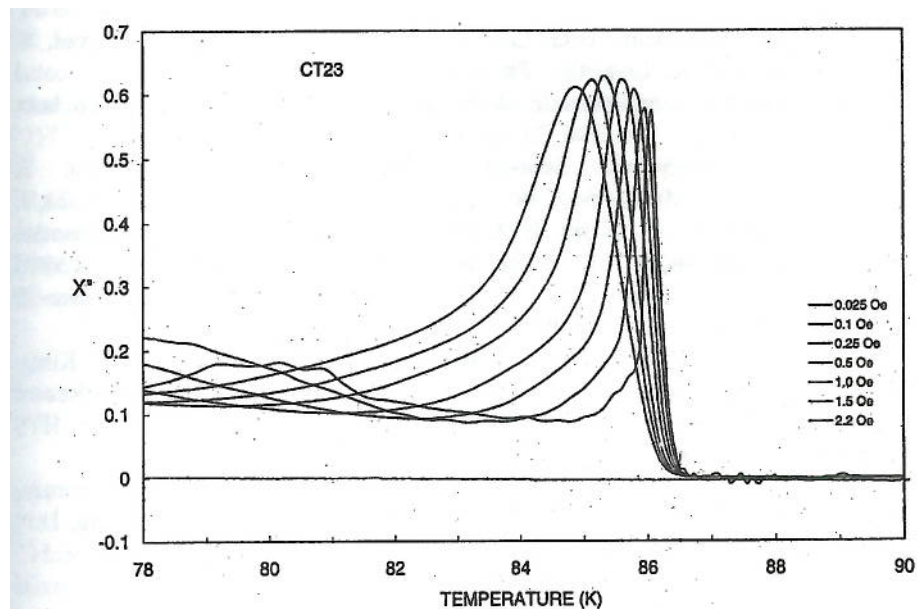


Fig. 6.  $\chi''$  versus temperature plot of the ac susceptibility data. 0.025 to 2.2 Oe magnetic field used. The different curves result from the different applied fields listed in the legend—the field increases from right to left.

#### CONCLUSION

It is seen that the FWHM of certain XPS peaks as well as XPS cationic peak ratios averaged over the analysis area can vary between YBCO samples. A possible correlation of the FWHM of the Y(3d<sub>5/2</sub>) XPS peak of YBCO to the thin film quality, specifically the critical transport current density ( $J_c$ ) may exist but is not clear. This relationship is dependent upon the appropriately phased YBCO for a proper comparison and YBCO samples whose nonstoichiometric composition will lead to an alternate XPS spectra. Broadening of the FWHM of the Y(3d<sub>5/2</sub>) XPS peak can indicate alternate undesirable bonding in the YBCO. More data is necessary to fully verify these relationships.

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