Soft x-ray absorption of a buried SmCo film utilizing substrate fluorescence detection

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(Received 23 March 1999; accepted for publication 28 April 1999)

Oxygen *K*-shell x-ray fluorescence was monitored from the MgO substrate of a metallic heterostructure system containing a buried SmCo permanent magnet layer. This fluorescence was utilized as a detector to record transmission yield spectra for the SmCo film at both the Co–$L_{3,2}$ and Sm–$M_{5,4}$ absorption edges. Ordinarily, traditional transmission yield spectroscopy in the soft x-ray regime is impossible to perform with films on single-crystal substrates. The measured intensity ratios agree with simulations to confirm the thickness information. The potential and limitations of this technique are discussed in comparison to standard total electron and fluorescence yield techniques and magnetic circular dichroism.
[S0003-6951(99)04525-8]

SmCo permanent magnet films are of scientific interest for their magnetic properties, particularly in heterostructures with Fe that possess a high magnetic moment while maintaining high coercivity.[1,2] To probe such systems, x-ray absorption spectroscopy can provide a wealth of element-specific chemical information, and via magnetic circular dichroism (MCD), magnetic information as well. MCD monitors the differences in the absorption spectrum between left- and right-circularly polarized photons.[3] Accurate absorption cross sections are needed to quantify spin and orbital contributions to the magnetic moment. A problem arises however in dealing with a buried layer in the soft x-ray regime.

The three traditional methods of x-ray absorption measurement: (i) transmission yield (TY), (ii) fluorescence yield (FY), and (iii) total electron yield (TEY), all run into limitations. Saturation effects due to sample thickness and the short mean escape depth of secondary electrons makes it very difficult for TEY to probe buried layers effectively. Although there are methods for thinning samples, it is important that the qualitative x-ray absorption measurements also be carried out in a nondestructive fashion because removal of any material from the system could destroy or severely alter the key properties that are of interest. While the sampling depth of FY is sufficient to probe buried layers, corrections must be made for saturation and self-absorption effects.[4] Even small errors in these corrections, however, may result in large errors in the quantitative analysis of MCD data.[5] The TY method is the most accurate of the three, but it also is limited by the soft x-ray penetration depth. One way to overcome this is by preparing free-standing films less than 200 Å thick or by preparing a polycrystalline sample on a relatively transparent substrate. This is often not practical because many interesting structural and magnetic properties derive from the epitaxial growth of thin-film structures on specific suitable substrates that are relatively thick.[6]

In this letter, we demonstrate an alternative nondestructive method for acquiring x-ray absorption spectra from a multilayer that overcomes many of the problems inherent in the other techniques. In essence, we acquire a transmission yield spectrum in an indirect fashion because the photons that are transmitted through the layer of interest (in our case a SmCo film) are then absorbed by the substrate layer (MgO), resulting in the production of oxygen *K*-shell fluorescent x rays. The outgoing fluorescence x rays are detected by an energy dispersive x-ray fluorescence spectrometer set to detect photons in the oxygen window around 524.9 eV. As the incoming photon energy is scanned across either a Co or Sm absorption edge, the number of photons absorbed by the MgO layer (and hence the number of O–K fluorescence photons produced) varies. In this energy window, the absorption coefficients of elements other than the Co or Sm are relatively constant, as is the attenuation of fluorescence x rays exiting the sample. Hence this ''fluorescence transmission'' (FT) experiment should accurately measure the Co or Sm photoabsorption cross section if the film thicknesses are well known.

The measurements were performed at an undulator beamline (SRI-CAT 2-ID-C) at Argonne's Advanced Photon Source.[7] This beamline utilizes a 5.5 cm period planar undu-

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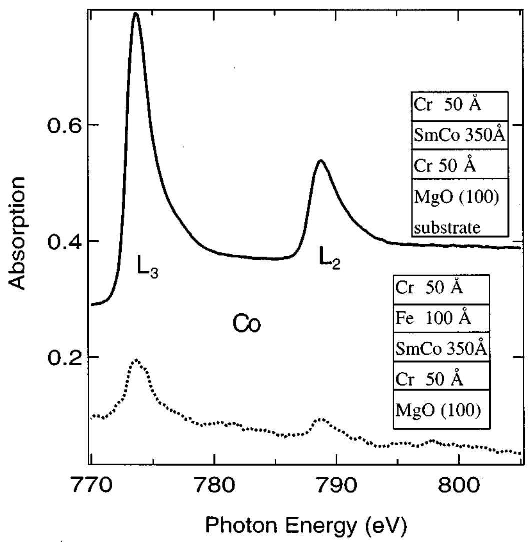

FIG. 1. X-ray absorption spectra (arb. units) acquired in total electron yield (TEY) model for a buried (dashed) and unburied (solid) SmCo film at the Co–$L_{3,2}$ edge, with schematics of samples.

lator and a spherical grating monochromator with a choice of three gratings resulting in an operational energy range from 0.5 to 3 keV, a photon resolving power of up to 10 000, and a beam spot size of approximately 2 mm×0.25 mm. The fluorescence spectrometer consists of a toroidal grating and a two-dimensional array detector, which is scanned along a Rowland circle defined by the entrance slit of the spectrometer and the grating.[8] The array detector was centered on the O–K fluorescence energy. For comparison purposes and to verify thickness information, TEY spectra of unburied standards were recorded by simultaneously monitoring the sample drain current.

The sample structure is: Cr (50 Å)/Fe (100 Å)/Sm_2Co_7(350 Å)/Cr (50 Å)/MgO (100) substrate. Such a structure is known as an exchange spring magnet[1,2] because the soft Fe layer is magnetically pinned to the hard SmCo layer. If the system is magnetized and a reverse field is applied, the Fe layer will try to follow the field but will spring back when the field is removed. A second sample of: Cr (50 Å)/Sm_2Co_7 (350 Å)/Cr (50 Å)/MgO (100) substrate was also prepared and utilized as a standard unburied layer. Both samples were prepared by direct-current (dc) magnetron sputtering. Details of the preparation and characterizations are described elsewhere.[9,10]

Figure 1 shows TEY absorption spectra at the Co–$L_{3,2}$ edge for buried and unburied SmCo. It is clear by comparison of the two spectra that TEY is of limited use with respect to the buried layer. A flat background is not achieved, and there are noise features of the same order of intensity as the true absorption features. Any analysis of this spectrum requiring integration of peaks would be wildly erroneous. Thus, this sample provides an excellent opportunity to test our proposed FT technique.

It is important to know whether our FT technique produces accurate absorption cross sections. Figures 2 and 3 show the transmission spectra at the Co–$L_{3,2}$ and the Sm–$M_{5,4}$ absorption edges, respectively, compared to simulated spectra. The simulated transmission spectra were generated utilizing TEY spectra of unburied standards and photoabsorption cross section tables.[11] The transmission, T detected by the intensity of O–K emission, is given by: $T = [1 - \exp(m_{\rm in} x)]$, where x is the thickness of the film and $m_{\rm in}$ is the total absorption coefficient for the incoming radiation of frequency $n_{\rm in}$. Note that the line ratio in the simulated and fluorescence transmission data agree well (confirming layer thicknesses) and that the backgrounds for the FT spectra are also flat. The lone problem is one of counting statistics due to weak signal. The weakness of the measured FT signal is due to several combining factors: (1) the fluorescence yield at the O–K edge is very low (0.6%), which is typical of low-Z atoms; (2) the fluorescence yield would be highest with photon energies just above the O–K edge, but we are far above it where the cross section is much smaller; (3) photons are attenuated entering and leaving the sample; and (4) the acceptance angle of the fluorescence spectrometer is small. The use of a high flux ($\sim 2 \times 10^{13}$ photons/s) undulator source[7] thus is vital to offset the low counting rate while maintaining a sufficiently high resolution. Note that any layer under the film of interest could be used as a detector provided it produces fluorescence of a lower energy than the absorption edges in the film of interest. For example, the

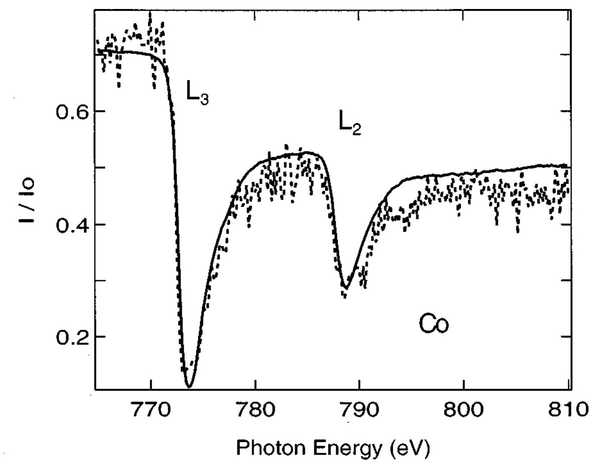

FIG. 2. Comparison of measured fluorescence transmission (FT) spectrum (dashed) for a buried SmCo film at the Co–$L_{3,2}$ edge to a simulated transmission spectrum (solid curve) for the same sample.

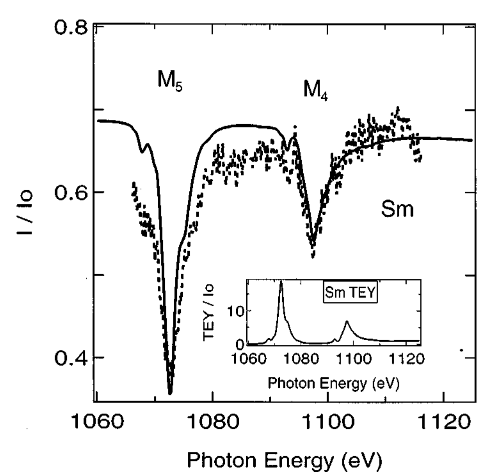

FIG. 3. Comparison of fluorescence transmission (FT) spectrum for a buried SmCo film (dashed curve) at the Sm–$M_{5,4}$ edge to a simulated transmission spectrum (solid) for the same sample. Inset is the total electron yield of a unburied SmCo film.

Cr layer could have been used as a detector. We even measured the Sm FT utilizing the Co fluorescence, although increased background noise from the fluorescence spectrometer at this energy reduced the signal-to-noise ratio. In this case the fluorescence signal is not modulated exponentially as was the case when the O–K emission was used as the detector, but rather the fluorescence intensity $I(n_{\mathrm{in}} \sim \sin[1 - \exp(-(m_{\mathrm{in}} + m_{\mathrm{out}})x]/(m_{\mathrm{in}+m_{\mathrm{out}}})$.[13] The symbols, s_{in}, and m_{out} represent the cross section at the input frequency to produce cobalt fluorescence and the total absorption coefficient for the cobalt fluorescence radiation respectively. Depending on the values of the various parameters this signal may be used to provide information about the detector atoms (Co) localized in the environment of a neighbor atom (Sm). Recent new photoemission measurements[14] have discovered multi-atom resonances and fluorescence may provide a complimentary alternate pathway to study this effect. In the future, samples could be tailored with an underlayer to act expressly as a fluorescence transmission detector.

We have demonstrated the ability to measure a transmission spectrum in the soft x-ray regime of a buried multilayer utilizing fluorescence from the sample substrate as a detector. The technique has broad applicability to acquire nondestructive, quantitative x-ray absorption measurements from buried layers. Also of note is the fact that the 2-ID-C beamline is scheduled to be upgraded with a circularly polarized insertion device ideal for the study of magnetic materials.[12]

One of the authors (J.W.F.) would like to thank Y. U. Idzerda, V. Chakarian, and C. T. Chen for insightful discussions. Work at Argonne was supported by the U. S. Department of Energy, Basic Energy Sciences, under Contract No. W-31-109-Eng-38. This work was supported partially by NSF Grant No. DMR-9017997, the University of Tennessee Science Alliance, and a DOE-EPSCoR cluster research grant DOE-LEQSF (1993-95)-03.

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