Application Of Time Of Flight-Energy Elastic Recoil Detection For Information-Storage Media Analysis

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Abstract. The challenge of meeting the ever-increasing demands for elemental profile information with high sensitivity and improved depth resolution has prompted development of sophisticated ion beam analysis methods, including elastic recoil detection analysis (ERDA). In combination with a time of flight (ToF) spectrometry, ToF-E (energy) ERDA has been widely employed as a powerful material analysis tool in a broad range of applications. Thin-film media for magnetic and optical information storage represents one of the most difficult classes of material from an ion beam analysis viewpoint. The suitability of ToF-E ERDA to analyze commercial hard disc, CD-ROM and CD-RW structures has been investigated. A complex Co/Cr/Ni-P/Al multilayer structure taken from a standard hard disc and up to 8 elements in CD samples could be distinguished simultaneously. The results demonstrate the unique power of this technique for characterizing the composition and depth profile of the multi-layers as well as the ingress and influence of foreign species.

INTRODUCTION

Magnetic and optical information storage is today a worldwide multi-billion dollar industry. Customer demands for commercial high-density information storage products drive an intense research effort into both magnetic and optical thin-film storage media. Today, there is active commercial development in read-only (CD/DVD-ROM), write once-read many times (DVD/CD-R) and read/write (CD-RW, magnetic hard disks etc.) technologies. There are difficulties to analyze these storage media structure by conventional Ion Beam Analysis (IBA) techniques, e.g. RBS, because they are based on the ferromagnetic elements, which have near lying or overlapping isotope masses, or multi-layer films, which contain both light and heavy elements.

Time of flight-energy elastic recoil detection analysis (ToF-E ERDA)\textsuperscript{2} is one of the most powerful IBA techniques due to its ability to detect both light and heavy elements. The paper is to highlight the unique analytical potential of ToF-E ERDA for quantitative characterization of thin optical and magnetic media films.\textsuperscript{2}

EXPERIMENTAL

Typical commercially available hard disc and compact discs were investigated by the ToF-E ERDA technique. The hard disc sample is taken from a standard hard disc, and the CD samples are CD-ROM and CD-RW produced by different manufacturers.

\textbf{FIGURE 1.} Schematic illustration of the ToF-E ERDA set-up.
TABLE 1. Experimental parameters

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
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<tbody>
<tr>
<td>Projectile beams</td>
<td>48 MeV $^{35}$Br$^+$, 60 MeV $^{127}$I$^{10+}$</td>
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<tr>
<td>Flight length</td>
<td>437.5 mm</td>
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<tr>
<td>Recoil angle $\phi$</td>
<td>45°</td>
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<tr>
<td>Incident angle $\theta_1$</td>
<td>67.5°</td>
</tr>
<tr>
<td>Exit angle $\theta_2$</td>
<td>67.5°</td>
</tr>
<tr>
<td>Energy detector</td>
<td>10×10 mm Si p-i-n diode</td>
</tr>
<tr>
<td>Collimator</td>
<td>8 mm in diameter</td>
</tr>
<tr>
<td>Stopping power</td>
<td>Ziegler’s STOP code 3</td>
</tr>
</tbody>
</table>

The experimental set-up is shown in Fig. 1, and the experimental parameters are listed in Table 1. The ToF and energy data was recorded in a list mode and analysed off-line. A detailed description of the measurement system and the data analysis are given elsewhere.4

RESULTS AND DISCUSSION

FIGURE 2. Mass vs. Energy spectra of hard disc sample. The contours are drawn at 10 logarithmically spaced levels.

The ERDA spectrum from the hard disc sample using 55 MeV $^{127}$I$^{10+}$ beam is presented in Fig. 2. The corresponding film structure in Fig. 3(a) reveals a multilayer structure. The substrate is Al, on which a ~200 nm thick layer of 80% Ni and 20% P was deposited. On top of this, a ~120 nm Cr layer and a ~55 nm Co layer capped with a ~100 nm carbon-rich protective layer were deposited (assuming bulk densities). Due to limited depth resolution, the thin Co and Cr layers are not fully resolved and hence the maximum concentration does not reach 100%. The underlying Cr layer has a persistent tail that extends to the surface, which can be attributed to energy broadening and/or grain boundary diffusion, as the solid solubility of Cr in Co is negligibly small. Minor element signals from O and a heavy element W were detected as shown in Fig. 3(b). Evidently, W is incorporated during the Cr deposition at a level of only ~50 ng g$^{-1}$. The oxygen is seen to be located at the surface and the buried interfaces. These findings show that ToF-E ERDA is appropriate to characterise processes that govern stability, coercivity and flux-reversal density in magnetic storage layers such as oxidation, layer inter-diffusion and impurity migration.

FIGURE 3. (a) Elemental depth profiles for major elements in the hard disc sample from the ERDA data. (b) Depth profile for low concentration elements in the same sample.

Commercial optical storage films have also been studied by the ToF-E ERDA technique. In CD-ROM, the data are stored in the polycarbonate (H, C and O) substrate as pits. The pitted surface is covered with a reflecting metal layer over-coated with protective lacquer. Ingression of contaminants and metal composition may modify internal reflection at the pitted interface, which modulates the reflected laser beam used to read the pit pattern. Figure 4 shows a contour plot of the calibrated mass versus energy histogram and the corresponding mass spectrum measured from the inner surface of a CD-ROM towards the lacquer. Four 4 elements, H, C, O and Al are clearly separated.

Optical CD-RW structures are more complex. A simple drawing of the CD-RW structure is shown in Fig. 5(a). It consists of a polycarbonate substrate with deposited layers of Ge$_3$Sb$_2$Te$_5$ phase-change alloy (PC-
alloy) to store the data pattern, buffer layers, Al reflective layer and a protective lacquer film together with a printed label surface. The rewritable function is accomplished by switching the PC alloy between amorphous and crystalline states. When a moderate heat generated by the drive laser focuses on the recording layer, it changes spots in the PC-alloy from an amorphous to crystalline state. Reheating with an intense laser beam, it changes back to an amorphous state. Depending on the heat treatment, the recording material cools to an amorphous or a crystalline state that has low or high reflectivity. This reversible phase change in the GeₓSbᵧTeₗ alloy film enables any previously recorded signal to be erased and subsequently re-recorded.

To avoid the effect of non-uniform removal of the lacquer layer, the CD-RW disc was cleaved into lacquer and substrate samples as shown in Fig. 5(b) and (c), respectively, so the phase-change alloy was studied by measuring from the inner surfaces. In freshly exposed CD-RW samples shown in Fig. 6, up to eight elements, H, C, O, Al, P, Co, Cr and Sb/Te, could be identified by employing both ⁸¹Br and ¹²⁷I projectiles in the ERDA measurements. The detection efficiency of elements with mass ranging from H up to Nb in the ToF spectrometer is studied previously. The depth profile reveals that a multi-layer structure comprising lacquer/Al/PC-alloy can be discerned. The low Al concentration is attributed to beam modification that has been confirmed by analyzing the beginning of the list mode data sequence. The lacquer layer and the polycarbonate material mainly contain H and C. Higher H and smaller C concentrations were observed in the lacquer layer than in the polycarbonate substrate. It is noteworthy that in conventional ion backscattering measurements, this mixture of light and heavy elements would not be separated.

ToF-E ERDA spectrum of CD-ROM sample with 10 logarithmically spaced levels. (a) Mass vs. Energy spectrum and (b) Mass spectrum.

Elemental concentration vs. depth of (a) the lacquer sample separated from CD-RW and (b) the substrate sample. The absolute measurement uncertainty is indicated as error bars in the plots.
Figure 7. Elemental concentration vs. depth of film sample separated from CD-RW and measured after 40-day exposure to air. (a) Elemental profile with larger depth scale. (b) Elemental profile close to the surface with magnified depth scale. The absolute uncertainty is indicated as error bars in the plots.

In order to study the resistance of CD-RW to environmental heat, humidity and light, the disc was cut into 10 x 10 mm pieces and maintained for 40 days with open edges in air at room temperature with humidity around 48%. After cleaving from the thick polycarbonate substrate, the freshly exposed lacquer samples, as shown in Fig. 5(b), were analyzed, and the corresponding results are shown in Fig. 7. To minimize the beam damage and modification of the sample during the measurement, a composite spectrum with good counting statistics was measured from a number of identical samples with small fluences. Comparing Figs. 6(a) and 7(a), it is seen that the concentrations of both H and O have dramatically increased with a relative ratio of 2:1, presumably due to ingress of water. The results show that 40-day storage leads to an increase in H and O concentration of up to 40.7% and 9.7%, respectively. The C content decreases to 48.1%. It is worth noting that in Fig. 7(b), the Al concentration reaches over 90% with a small surface depletion. Notwithstanding the effect of energy broadening, (associated with energy straggling and multiple scattering etc.) on the recoil spectrum, there is apparently a well-defined plateau of Al signal besides a few at. % of H, C and O. Furthermore, a shift of the Co peak from the other alloy elements is observed in Fig. 7(b). This suggests that besides water contamination, the environment might have modified the alloy layer. The mass separating power of ToF-E ERDA may be exploited to study ingestion of species from the environment through the use of compounds enriched with low natural abundance isotopes e.g. D2O, H218O, 15NO2 etc.

Conclusions

ToF-E ERDA can deliver quantitative information of real pertinence for commercial development of sophisticated ferromagnetic and optical thin film media, such as:

- The thickness and composition of all the layers in real commercial structures with 4-8 elements distributed over 5-7 layers.
- Measurement of minor contaminants such as interfacial oxygen is possible because of the mass selectivity and high sensitivity.
- Ingression of environmental contaminants, such as H2O can be studied by simple extensions of the method, such as building up composite spectrum from sets of identical samples measured with small fluences.

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References