

Wavelength Dispersive X-ray Fluorescence Spectrometry for the Analysis of Organic Polymer Film

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1. Introduction

Recently, many studies have been focused on the thin films because there are numerous industrial processes relevant to thin films such as fuel cells, sensors, lubricants, coatings, and so on [1]. Physical and chemical properties of solid surface have been modified by ultra-thin coatings such as Langmuir-Blodgett (LB) method with a variety of types of organic functional materials for the specific purposes in many applications [2]. In addition, the layer-by-layer technique using polyelectrolyte films are now of interest as biosensors, electrochromic and electroluminescent devices, etc [3-5].

In general, several methods such as X-ray or neutron reflectivity, and quartz crystal microbalance (QCM) have been utilized for the thin film analysis [6]. These optical techniques can measure the film thicknesses up to hundreds of nanometers while X-ray photoelectron spectroscopy is widely used to study a few nanometers thick films [7-9]. Other methods such as X-ray Photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atom force microscopy (AFM) have also been used in the film analysis in spite of some disadvantages for each method.

X-ray fluorescence (XRF) has long been used as a rapid and simple analytical tool for the analysis of elemental composition of materials. XRF technique is suitable for on-line or in-line real-time monitoring because it is a non-destructive and rapid analysis with good precision and good accuracy at low cost. The aim of this work is to develop a new analytical technique for the quantitative analysis of polymer film on metal substrate. In the present study, Compton peak profile was investigated under different experimental conditions by using wavelength-dispersive XRF (WD-XRF). Compared to energy-dispersive XRF (ED-XRF), WD-XRF is more adequate in an accurate quantitative analysis of thin organic film.

2. Methods and Results

2.1 WD-XRF System Experimental Setup

XRF measurements were performed using a wavelength-dispersive X-ray fluorescence spectrometry with the rhodium (Rh) target X-ray tube, which was operated at 60 kV and 40 mA. A LiF (100) analyzing

crystal and a flow-proportional counter (FC) as well as

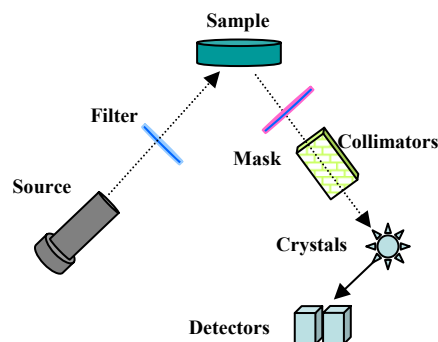


Fig. 1. Schematic diagram of a WD-XRF composed of X-ray source, filter, sample, mask, collimators, crystal and detectors

scintillation counter (SC) were used. The counting time was 0.5s. The intensities were determined for each sample by the measurement of fluorescent radiation of Rh Compton peak in the analyzed samples and the blank sample. The operating conditions of WD-XRF for the analysis of polymer film are given Table. 1.

Table 1: The operating Condition of WD-XRF

Tube Voltage	60 kV
Tube Current	50 mA.
Mask size	30 mm
Collimator	0.15°
Analyzing crystal	LiF 100
Optical path	Helium
Detector	FC + SC

Scotch® Magic Transparent Tape 810 (Korea 3M, Seoul, Korea) was cut in to a 2 cm× 5 cm piece and used as an organic adhesive film. A strip of the tape is carefully laid on the metal substrate layer-by-layer manually. One layer of the polymer film is around 50.88 μm-thick.

2.2 Result and Discussion

Many XRF spectrometers adopt Rh as an anode target material because the characteristic energies of Rh are able to excite both heavy and light elements.

Nevertheless, Rh was used in this study not because of the excitation of the element of interest in a sample but because of the quantitative analysis of organic thin films. Fig. 2 shows a WD-XRF data from the anode's Compton scattering (inelastic scattering) and Rayleigh scattering (elastic scattering) of the polymer film. Rh's $K_{\alpha 1}$ and $K_{\beta 1}$ Rayleigh scattering line are observed in the fluorescence spectrum, while Rh's $K_{\alpha 1}$ and $K_{\beta 1}$ Compton scatter lines are shifted to a lower energy compared with the main Rh's $K_{\alpha 1}$ and $K_{\beta 1}$ Rayleigh scattering line. Rh's $K_{\alpha 1}$ Rayleigh scattering line and corresponding Compton line was located at $2\theta = 17.53$ and 18.6 , respectively.

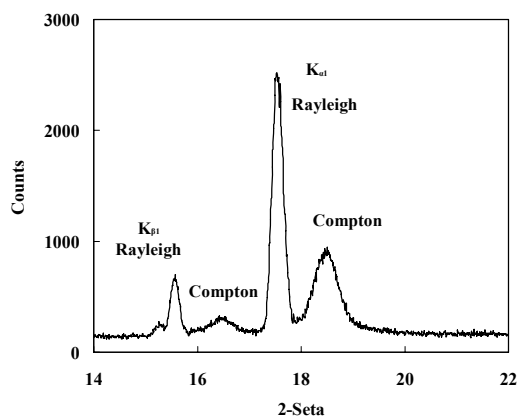


Fig. 2. A representative X-ray fluorescence spectrum of polymer film and Compton lines of Rh anode target.

An analysis of polymer film in the present study is based on the observation of the intensity of the Compton scattering peak, which is usually used as a simple and effective means of matrix correction. Our preliminary study shows that the Compton scattering peak was proportional to the number of film layer on metal plate surface. The intensity of Rh's $K_{\alpha 1}$ and $K_{\beta 1}$ Compton scattering lines increased with the increase of the number of the polymer layers.

3. Conclusions

We have proposed a new XRF technique for the analysis of polymer film by means of a wavelength-dispersive X-ray fluorescence spectrometry. It was very simple and rapid to analyze the polymer film by using the Compton line of the anode target. It did not depend on the matrix elements of the samples. Only Rh $K_{\alpha 1}$ and $K_{\beta 1}$ lines were necessary for the analysis of polymer film. It would be very promising in the analysis of the film composed of light elements such as carbon, oxygen and hydrogen.

In our WD-XRF spectrometer, the angle of x-ray incidence to the sample and the take-off angle in the spectrometer are fixed. However, if the angle settings

are optimized, performance for the quantitative analysis of the organic films can be improved by maximizing the intensity of Compton scattering line.

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