Polymer 72 (2015) 59–62

Contents lists available at ScienceDirect

Polymer

journal homepage: www.elsevier.com/locate/polymer

Short communication

Charging effects in the ion beam analysis of insulating polymers

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ARTICLE INFO

Article history:
Received 26 March 2015
Received in revised form
31 May 2015
Accepted 30 June 2015
Available online 6 July 2015

Keywords:
Kapton
Charging effect
RBS

ABSTRACT

We have studied the energy shift introduced by surface charging during the course of ion beam analysis of an insulating material (a Kapton thin film) using Rutherford Backscattering Spectrometry (RBS). The potential that develops was found to be proportional to both the incident ion energy and the sample thickness. This correlation suggests a conductance mechanism that is based on the ion beam induced carriers that are generated in the material. A simple relationship between the mobility of the induced carriers and the built-up surface potential has been derived. It provides a semi-quantitative explanation for the observed spectral offset. This relationship also provides important insight into the electrical characteristics of the insulating material being irradiated. In the specific case of Kapton, we observed a split of the high energy edge in the RBS spectra, which we suggest is most likely a reflection of the presence of both amorphous and crystalline forms of Kapton. Some contribution from partial discharge of the surface potential to the surrounding is also considered.

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1. Introduction

The interactions of charged-particles with dielectric materials has scientific and technological importance in such diverse areas as thermo-isolating coatings for spacecraft [1–4], electronics [5], solar batteries [6] and in developing polymeric substitutes for human organs so as to prevent damage by penetrating ionizing radiation [7]. The charge accumulation that results from the bombardment of materials with charged particles has also been used to study the physical properties of insulating materials and/or to modify them [8–10].

The behavior of insulators under irradiation is complex and is still not well understood. RBS [11–13] and X-ray photoelectron spectroscopy [14] studies have noted distortions that can be attributed to the charging of these materials. This charging also can lead to mistakes in secondary ion mass spectrometry depth profiles [15] and to image distortion in scanning electron microscopy [16]. Ion beam bombardment of insulators also causes a huge bremsstrahlung background in the particle induced X-ray emission spectrum [17]. The spectroscopic analysis of backscattered particles under such conditions can also be disturbed by emitted light "blinding" the detector and by the sample surface potential decelerating the incoming ions and accelerating the outgoing scattered ions.

Several methods have been proposed to avoid such distortions. The use of additional electron guns can neutralize the positive charge that develops on the material surfaces [17]. Another option is to apply a thin conducting surface coating [18].

In the work reported herein, rather than removing the charge build up and its effects, we quantify it and create a model that allows us to achieve new insight into the electrical behavior of an insulator as it is bombarded by an ion beam. We use RBS [19] to study different thicknesses of Kapton [20] (a polyimide with the formula $C_{22}H_{10}O_5N_2$). Under the conditions of the RBS experiment, the Kapton is subjected to high-energy $^4He^+$ particle bombardment. The spectroscopic data obtained from these experiments as a function of sample thickness and the energy of the particles is correlated with the surface electric potential of the Kapton under such conditions.

The intrinsic resistivity of Kapton is extremely high [20] and radiation induced conductance of Kapton has been previously observed [21]. We demonstrate herein that this conductance depends on sample thickness and on the energy of the bombarding ions. It expresses itself in a systematic shift of the RBS spectral results to higher energy that provides information about the electrical and morphological properties of the Kapton.

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http://dx.doi.org/10.1016/j.polymer.2015.06.057
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2. Experimental

RBS was done using the 1.7 MV Bar-Ilan University Pelletron accelerator manufactured by NEC [22]. The experiments were carried out using $^4He^+$ ions at energies of 1.5, 2.0 and 2.4 MeV. The chamber vacuum was ~1 × 10^{-7} Torr and the normally incident beam current was ~7.5 nA collimated to 1.5 mm of diameter, with the integrated charge Q = 40 μC identical for all experiments. A secondary electron suppressor was used in front of the samples, biased at −300 V relative to the sample holder. RBS spectra were acquired with ULTRA™ Silicon-Charged Particle Detector (ORTEC) in the Cornell geometry at scattering angle 169° and having a solid angle of 2.7 msr. The data analysis was performed using the Data-Furnace software (NDFv9.6a) [23]. The electronic gain of the detection was calibrated using an Au/Ni/SiO_2/Si standard sample. This parameter was used to convert "channels" to "eV" in the data presentation.

The samples under study were Dupont Kapton HN sheets of 25, 50 and 125 μm thicknesses. Prior to ion beam bombardment, the as-received sheets were cut into 1 cm × 1 cm squares, washed with double distilled water and ethanol, and dried by a nitrogen flow. They were mounted on an aluminum holder by double sided, self-adhesive carbon tape. RBS spectra were obtained with three different thicknesses of Kapton at three $^4He^+$ beam energies with a maximum energy of 2.4 MeV to avoid the $^{16}O(\alpha,\alpha)^{16}O$ elastic resonance at 2.5 MeV [24].

3. Results

Typical RBS spectra of the Kapton films of 25 and 50 μm measured at 2.4 MeV and their simulations are presented in Fig. 1. They clearly show the shift in positions of the RBS signals of the C, N and O elemental edges to higher energies ΔE_1 for the 50 μm sample. The corresponding spectrum of the 25 μm film doesn't show any measurable shift.

RBS measurements of the thickest sample (125 μm) showed an interesting new feature, wherein the high energy edge of the spectrum shows two steps (Fig. 2). Similar doublets appear in the spectra of this thickest sample at all three He^+ beam bombardment energies (Table 1). The magnitude of the energy split ΔE_2 does not show any dependence on the incident beam energy E_0.

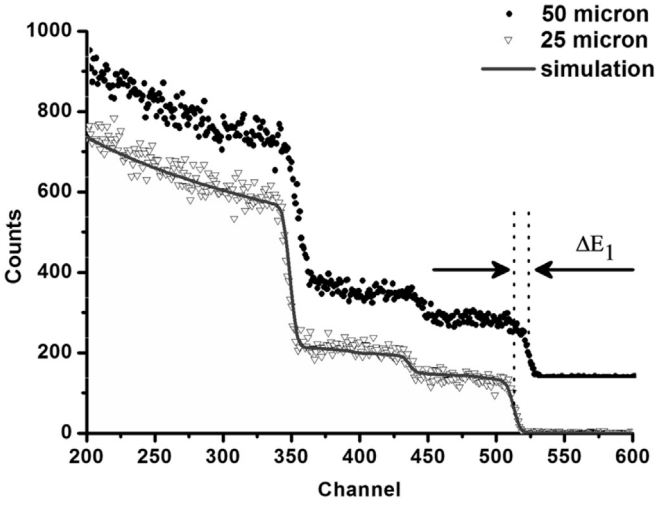

Fig. 1. RBS at 2.4 MeV of the 25 and 50 μm thick Kapton samples. The energy shift ΔE_1 between the actual and the simulated spectra is shown for the 50 μm sample. This shift is negligible for the sample of 25 μm. (The spectra are vertically separated for clarity.)

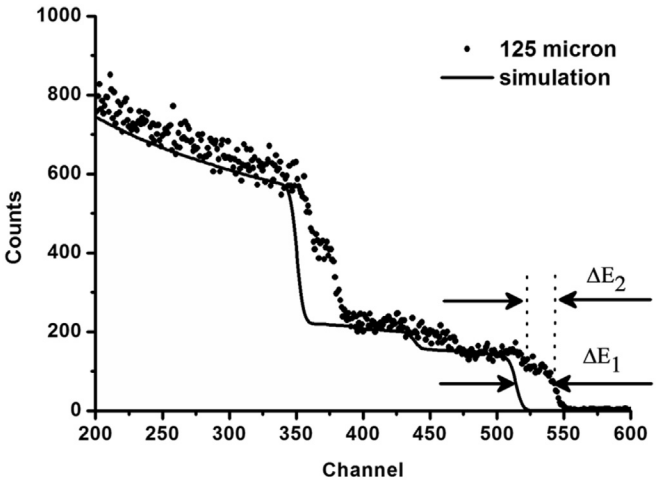

Fig. 2. RBS at 2.4 MeV of the 125 thick Kapton sample and its simulation showing their energy shift ΔE_1 and the high energy edge split ΔE_2 for the oxygen on the sample surface.

Table 1
Charging-induced energy shifts for 125 μm Kapton. Primary energy shift ΔE_1 and energetic split ΔE_2 as a function of beam energy. The systematic error of the measurements is ±3.2 keV.

Beam energy, E_0 (MeV)	ΔE_1 (keV)	ΔE_2 (keV)
1.5	39.6	28.1
2.0	44.9	28.3
2.4	49.2	27.8

The energy shifts ΔE_1 of the RBS spectra for the 50 and 125 μm samples depend almost linearly on the incident beam energy E_0 (1.5 MeV, 2 MeV, 2.4 MeV). That is, the observed shift (relative to the simulated spectra) increases with increased beam energy. However, the spectra obtained for the 25 μm sample didn't show any ΔE_1 at any of the 3 beam energies. The above results are summarized in Fig. 3 and Table 2 so as to highlight the trend in energy shift against film thickness and energy shift ΔE_1 against beam energy E_0. They show that the behavior is systematic, with a linear increase of energy shift with increased ion beam energy and increased sample thickness d.

4. Discussion

It is well known [12,13,18] that the build-up potential φ, decelerates the incident positive He ions and accelerates the back-scattered particles. Hence, the energy edge of each RBS spectrum is shifted to a higher value according to the known relationship shown in equation (1) [13].

$$E_d = k(E_0 - e\varphi) + me\varphi \quad (1)$$

E_d is the detected energy of the particles scattered from the surface oxygen atoms; e is the electron charge; m is the He ion charge state; and k is the kinematic factor of He scattered from oxygen. Using this relationship, we calculated a build-up potential φ. This calculation assumes that the scattered He particles have a charge state of +2, an assumption that is well established both experimentally and theoretically for the beam energies used in this work [25].

RBS studies of insulating materials, in general, do not relate to the question of *why one is able to measure ion current on non-conducting samples*. Clearly, the current is not provided by the

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