Nuclear Instruments and Methods in Physics Research A 676 (2012) 21–25

Contents lists available at SciVerse ScienceDirect

Nuclear Instruments and Methods in Physics Research A

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

A remotely controlled, semi-automatic target system for Rutherford backscattering spectrometry and elastic recoil detection analyses of polymeric membrane samples

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ARTICLE INFO

Article history:
Received 10 October 2011
Accepted 3 February 2012
Available online 13 February 2012

Keywords:
Rutherford backscattering spectrometry
Elastic recoil detection
Membranes
Thin film composite
Reverse osmosis
Nanofiltration

ABSTRACT

A new target system for Rutherford backscattering spectrometry and elastic recoil detection analysis is described which enables remotely controlled, semi-automatic analysis of multiple organic polymer samples without exceeding damaging incident beam fluences. Control of fluence at a given beam current is achieved using two stepper motors to move a thin aluminum disk loaded with polymer samples both radially and azimuthally across the beam. Flexible beam spot locations and sample irradiation times are remotely controlled in two steps via two custom LabVIEW^{TM} programs. In the first step, a digital image of the target disk is converted into precise radial and azimuthal coordinates for each mounted polymer sample. In the second step, the motors implement the user-directed sample irradiation and fluence. Schematics of the target system hardware, a block diagram of interactions between the target system components, a description of routine procedures, and illustrative data taken with a 2 MeV $^4He^{2+}$ analysis beam are provided.

1. Introduction

Rutherford backscattering spectrometry (RBS) has recently been used as an analytical technique to study the physico-chemical properties of the active layers of thin-film composite membranes for water purification [1–9]. Thin-film composite membranes used in applications such as reverse osmosis and nanofiltration consist of an ultrathin (~50–200 nm) organic active layer that lies on top of an intermediate polysulfone support (~30 μm) backed by a polyester fabric (~300 μm) [10,11]. The performance of such thin-film composite membranes, in terms of water permeation and contaminant rejection, is mostly determined by their active layer [10,11]. As a result, the development of new improved membranes significantly benefits from studying the physico-chemical properties of the active layers and their interactions with water and contaminants. The recently published [1–6,8] membrane characterization procedures that use RBS for sample analysis take advantage of the ability of RBS to resolve the active layer from their significantly thicker polysulfone and polyester supports [2,5,12].

During RBS analyses, a high-energy ion beam (e.g., 2 MeV $^4He^+$ [2,5]) is used to irradiate the target sample, and the spectrum resulting from the backscattered ions is analyzed to obtain the elemental composition of the irradiated sample with nanometer depth resolution [2,5,12]. Unfortunately, beam irradiation of organic polymers results in polymer degradation [13–17] with a corresponding emission of beam-generated small molecules [13,14] that leads to a changing elemental composition of the irradiated sample as a function of beam ion fluence [13–15,17]. Studies [13,16,17] of polymer degradation upon beam irradiation have shown, however, that induced changes in the elemental composition of irradiated samples can be neglected below fluence thresholds that depend on the polymer analyzed. Among the materials relevant to the structure of thin-film composite membranes, polysulfone has shown the lowest fluence threshold value at 3×10^{14} ions/cm^2 using a 2 MeV $^4He^+$ ion beam [17].

For RBS to be useful in the study of the properties of the active layers of thin-film composite membranes [1–9], the beam ion fluence is maintained below the threshold values where changes in the elemental composition of the sample analyzed would be detected. Given that the elements (i.e., H,C,O,N,S) that make up thin-film composite membranes have relatively small scattering cross-sections (0–342 mb/sr) [12], the only possible strategy to

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doi:10.1016/j.nima.2012.02.005

avoid exceeding fluence threshold values, while obtaining enough counts to quantify accurately the sample elemental composition, is to scan a relatively large area (e.g., a few square centimeters) with the analysis beam. One achieves this either by analyzing multiple points of the membrane sample or by continuously scanning the ion beam over the sample [2–8]. Such scanning patterns have been achieved by continual manual positioning of scattering target stages, a technique which is highly impractical for analysis of multiple samples. Active layers of thin-film composite membranes can also be studied after isolating the active layer on a solid surface by peeling off the polyester backing and dissolving the polysulfone support using organic solvents [18–21]. Isolated active layers are generally relatively small (i.e., a few square centimeters) and irregular in shape, and therefore, the RBS analysis of isolated active layers requires both irregular scanning patterns and millimeter-accurate positioning of the beam over the sample to stay within the sample boundaries.

Thus, an innovative target system is needed to implement practical analysis of multiple organic membrane samples while satisfying the fluence threshold, sample location, and scanning pattern requirements described above. Accordingly, the objective of this work was to design, fabricate, and test a scattering target system for RBS analysis, and the sister technique of elastic recoil detection (ERD) analysis, with the following capabilities: (i) remote control; (ii) irradiation of samples according to regular and irregular scanning patterns defined by the user; (iii) positioning of beam on target with millimeter-scale accuracy; and (iv) accommodation of multiple samples of any shape on the sample holder.

2. Accelerator and scattering chamber

The new RBS/ERD target system utilizes systems associated with the tandem electrostatic accelerator at the Triangle Universities Nuclear Laboratory (TUNL). Existing hardware at TUNL includes a duoplasmatron ion source equipped with a sodium charge-exchange canal used to produce a 30 keV $^4He^-$ ion beam which is injected into the accelerator. After initial acceleration to the electrostatic accelerator terminal at 0.67 keV, the $^4He^-$ beam is stripped of its electrons in a thin carbon foil inside the terminal, and then further accelerated. The emerging 2 MeV $^4He^{2+}$ beam is momentum selected by deflection (52°) through an analyzing magnet, focused, and transported 15 m to a multipurpose, aluminum scattering chamber. The chamber is cylindrical in shape with internal diameter and depth of 59.7 cm and 26.0 cm, respectively. The beam enters the chamber through entrance slits of adjustable aperture that typically define a square 3 mm × 3 mm beam cross-section. The beam spot on target is located 6.4 cm above a bottom plate that holds several detectors of scattered or recoil particles emerging from the polymer film being used as a target. This bottom plate can be rotated while under vacuum around the vertical axis of the chamber to set the desired detector angles. Signals from the detectors are first sent through pre-amplifiers at the scattering chamber before being sent to the accelerator control room for further processing and digitization.

3. RBS/ERD target system

The new RBS/ERD target system consisting of a target rod, static stage, dynamic stage, and target wheel is shown in Fig. 1. The target rod secures and locates the rest of the target system precisely inside the scattering chamber and allows for rotation of the system around the central vertical axis of the chamber. The static stage houses a stepper motor that controls vertical

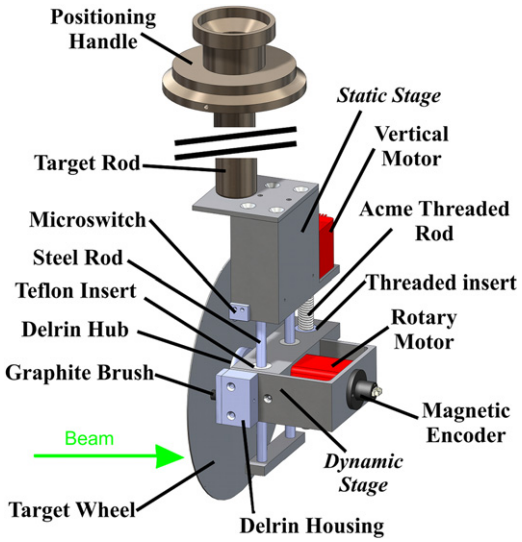

Fig. 1. Schematic of the hardware of the Rutherford backscattering spectrometry (RBS)/elastic recoil detection (ERD) target system. Not shown in the picture are a PIC18F4520 microcontroller and a TMCM-310/SG motor driver/controller. The rotational motor is connected to the microcontroller and both the rotary and vertical motors are connected to the driver/controller.

movement of the dynamic stage along two precisely located steel rods. The target wheel is attached to a second stepper motor on the dynamic stage which controls its azimuthal motion. The two stepper motors thus move the target wheel vertically and rotate it around its central axis. The target system is controlled via two custom LabVIEW™ programs. One program, WheelScan, combines a digital picture of a loaded target wheel with input from the user to establish the movement patterns of the two stepper motors. A second program, MotorLord, then uses data generated by WheelScan to control the two stepper motors, thus implementing desired movements of the target wheel. Fig. 2 illustrates the interaction among the hardware, software, and user of the target system. Detailed information about all system components is included in the following sections.

3.1. Hardware

3.1.1. Sample holder target rod

The nickel-plated brass target rod supporting the sample holder system slides vertically and rotates easily while maintaining a secure vacuum seal with the scattering chamber lid. The positioning handle of the target rod has azimuthal fiducial marks in 5° increments to indicate the angle of incidence of the beam with respect to the normal of the target wheel. The positioning handle is also pinned to a frame atop the scattering chamber to support the weight of the sample holder system and align the vertical axis of the rod with the horizontal beam axis. Using an optical transit, the horizontal rotation axis of the target wheel was confirmed to intersect both the axis of the beam and the vertical axis of the rod through the center of the scattering chamber to within 0.5 mm.

3.1.2. Target wheel

The aluminum target wheel, having 15.9 cm outer diameter and 0.8 mm thickness, is designed to be lightweight and to hold as many membrane samples as possible while fitting within the scattering chamber. The 1.59 cm inner diameter of the target wheel fits snugly over a Delrin® hub to define its axial location