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ScienceDirect

Physics Procedia 80 (2015) 151 – 154

Physics Procedia

26th International Conference on Nuclear Tracks in Solids, 26ICNTS

Application of the ion beam graft polymerization method to the thin film diagnosis

Akira Taniike*, Raito Nakamura, Syugo Kusaka, Yuya Hirooka, Noriaki Nakanishi, Yuichi Furuyama

Kobe University, 5-1-1 Fukaeminami-machi Higashinada-ku, Kobe 658-0022, Japan

Abstract

The ion beam graft polymerization (IBGP) method was applied to diagnosis of thin film of several tens μm or less thickness. After a sample stacked on polyethylene film was irradiated with proton beam, polyethylene was graft-polymerized with acrylic acid monomer. From observation of the graft-polymerized polyethylene, information inside the sample are obtained. Demonstrations of the diagnosis method were conducted for a leaf sample and a polyvinyl acetate film contained some voids. Using imitation samples made of metal and polymer sheets, some characteristics of this method was obtained. This method is useful for diagnosis for voids in thin film.

Peer-review under responsibility of the Scientific Committee of 26ICNTS

Keywords: ion beam; graft polymerization; thin film; diagnosis

1. Introduction

Radiation graft polymerization is useful method to produce industrial materials. Generally, electron beams are utilized for this method. Ion track grafting had been developed by Betz (1995). In our laboratory, we have conducted the experiments to make a graft polymerized polymer with ion beams, such as proton, helium. The monomers react on the radicals generated by the ion beam irradiation. After polymerization reaction the graft chains are formed in the substrate. The ion beam graft polymerization (IBGP) method is useful and it can introduce the grafted chains with a functional base into the polymer at a vicinity of the surface, Taniike et al. (2011).

* Corresponding author. Tel.: +81-78-431-6298; fax: +81-78-431-6298
E-mail address: taniike@maritime.kobe-u.ac.jp

Peer-review under responsibility of the Scientific Committee of 26ICNTS
doi:10.1016/j.phpro.2015.11.095

There are many non-destructive diagnostic methods, e.g. supersonic echo, X-ray radiography among others, however these don't have sufficiently resolution for thin film analysis since interaction between sample material and probing beam is weak. On the other hand, the accelerator analyses are very powerful method for thin film diagnosis. But it is difficult to observe the voids inside of a thin film, because the analysis can only obtain the areal density. We have developed the diagnostic procedure method of a thin film with the ion beam graft polymerization. When a sample stacked on polyethylene film was irradiated with proton beam with enough energy, protons penetrate a sample and stop in the polyethylene. The range of the proton in polyethylene is a function of the size of the void that a proton passes. Thus, the polyethylene has information of the inside sample. After polyethylene is graft-polymerized with acrylic acid monomer, the information is appeared as existence of grafted polymer chains. Consequently, information inside the sample are obtained from observation of the grafted polyethylene. We examined this diagnostic method to analyze some samples made of metal and polymer. Some parameters in this method were obtained from the experiments.

2. Experimental Procedure

Samples and high density polyethylene film (HDPE) thickness is 50 μm, are irradiated with proton beams generated by the 1.7 MV tandem Pelletron accelerator at Kobe University (model 5SDH-2, NEC). Typical ion energy is 2.0 MeV, and the fluence is 2.0×10^{12} cm^{-2}, and the fluence rate is 1.2×10^{9} $cm^{-2}s^{-1}$, and the irradiation area is 1.5×1.5 cm^{2}. Fig. 1 shows schematic view of a sample irradiation a) and the cross sectional view b). Radicals are generated from the surface to ion range in HDPE. After an irradiation, the polyethylene is picked up from the holder and it is graft-polymerized by acrylic acid monomer. The IBGP procedure is described in a reference, Taniike et al. (2014). Samples, a leaf and a polyvinyl acetate resin were prepared to demonstrate this diagnostic method, and the imitation samples are prepared to measure the properties of this method.

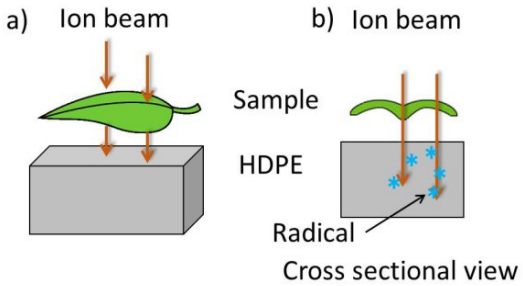

Fig. 1. An example of an irradiation. A sample stacked on a HDPE sheet.

3. Results and Discussions

3.1. An example of an observation in a sample with IBGP Method

A leaf sample and HDPE are stacked and irradiated by a 2.0-MeV proton. Then, the graft polymerization reactions are conducted to the irradiated HDPE. The HDPE has information of the inside sample. The information is obtained by HDPE observation. It looks like an ion beam radiography, and the results are shown in Fig. 2. A photograph of a clover leaf sample are shown in Fig. 2 a). The grafted HDPE was coloured in blue/green with Cu adsorption on the poly-acrylic acid. The inside structure of leaf is seen in Fig. 2 b). Polyvinyl acetate resin (bonding agent for wood) has many bubbles (voids). The bubbles in the thin film made of polyvinyl acetate could be also observed by this diagnostic method.