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SURFACE MORPHOLOGY OF SPUTTER-ETCHED POLYMER FILMS

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ABSTRACT: Poly(ethylene terephthalate)(PET), nylon 6, and *p*-aramid films were sputter-etched with argon ions at a pressure of 0.1 mmHg and their surfaces were examined by scanning electron microscopy. The uniaxially stretched PET and nylon 6 films showed a number of wavy crevices perpendicular to the stretching direction, whereas biaxially stretched PET and nylon 6 films produced many protrusions. Casted *p*-aramid film also produced the same protrusions.

1. INTRODUCTION

Sputter-etching is a technique to modify polymer surface. Argon ions produced by glow discharge under a pressure of 0.1 mmHg attack the polymer surface on cathode. The treatment cause significant changes of the polymer surface [1-6]. Therefore, it seems likely that such a modification on the film surface plays an important role to the increase of wetting and adhesion in relation to polymer processing.

In this article, several kinds of films were sputter-etched, and the polymer surfaces were investigated by scanning electron microscope [SEM].

2. EXPERIMENTAL

Undrawn poly(ethylene terephthalate)(PET) and nylon 6 films were stretched to 300% for PET and 200% for nylon 6 films, uniaxially or biaxially, by using a stretching apparatus (Iwamoto Seisakusyo). The films were subsequently annealed at 110 °C for 10 min. A cast-prepared *p*-aramid film (Asahi Kasei Co.) was also used as a material [6]. The method of sputter-etching is illustrated in Fig. 1. The treatment was carried out by using a sputtering equipment VPS-020 (Kobayashi Rikakikai) with a frequency of 13.56 MHz at a pressure of 0.1 mmHg for 5 to 60 min. After the treatment the specimen was ultrasonicated by Yamato BRANSON 2200 at 60 W and a frequency of 45 KHz for 5 min. The surface morphology of the sputter-etched films was observed at magnifica-

Fig. 1. Apparatus for sputter-etching.

tion of 40000 using S-800 scanning electron microscope (Hitachi Co.). Number and height of the protrusions were investigated on the basis of the SEM photographs.

3. RESULTS AND DISCUSSION

SEM micrographs of stretched (uniaxially and biaxially) and subsequently sputter-etched PET and nylon 6 films are shown in Figs. 2-5. It has been reported that the grain structures on untreated-polymer surface are crystallites [7]. The biaxially stretched films formed many protrusions by sputter-etching. On the other hand, uniaxially stretched films formed wavy

Fig. 2. SEM micrographs of biaxially stretched and sputter-etched PET films ($\times 40000$). Time of sputter-etching was: (1) 0 min; (2) 10 min; (3) 20 min; (4) 30 min; (5) 60 min; (6) after ultrasonication of (5).

Fig. 3. SEM micrographs of uniaxially stretched and sputter-etched PET films ($\times 40000$). Arrow shows the direction of stretching. Time of sputter-etching was: (1) 30 min; (2) 60 min; (3) after ultrasonication of (2).

crevices, and their surface morphology changed with the increase in sputtering time. As is evident from Figs. 2 and 4, the number of the hand, uniaxially stretched films formed wavy crevices, and their surface morphology changed with the increase in sputtering time. As is evident from Figs. 2 and 4, the num-

ber of the protrusions decreased with increase in the treatment time, whereas the height increased considerably with sputtering time. The protrusions produced by sputter-etching were removed by subsequent ultrasonication.

SEM micrographs of the sputter-etched *p*-aramid

Fig. 4. SEM micrographs of biaxially stretched and sputter-etched nylon 6 films ($\times 40000$). Time of sputter-etching was: (1) 0 min; (2) 30 min; (3) 60 min; (4) after ultrasonication of (3).

Fig. 5. SEM micrographs of uniaxially stretched and sputter-etched nylon 6 films ($\times 40000$). Arrow shows the direction of stretching. Time of sputter-etching was: (1) 30 min; (2) 60 min; (3) after ultrasonication of (2).

films are shown in Fig. 6. Their features are almost same with those of the PET and nylon 6 films [6]. Thus, it is clear that film surface cause significant physical change by the sputter-etching. Furthermore, these micrographs show that the protrusions grow by

the sputter-etching and plasma polymerization of gases decomposed by sputter-etching, and one protrusion is made up to the top of two or three protrusions with increase in the treatment time [1,6]. The top of the protrusions formed by plasma polymerization were removed easily by ultrasonication.

Fig. 6. SEM micrographs of sputter-etched *p*-aramid film ($\times 40000$). Time of sputter-etching was: (1) 0 min; (2) 10 min; (3) 20 min; (4) 30 min; (5) 60 min; (6) after ultrasonication of (5).

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