EFFECT OF BORON AND RARE EARTH ADDITION ON THE GRAPHITIZATION OF POLYIMIDE FILM

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INTRODUCTION

Polyimide block is known as non-graphitizing carbon. However, a thin film of polyimide such as Kapton, Upilex and Novax gives a carbon film with highly graphitizing nature[1-4]. Boron is known to affect graphitization of carbonaceous materials. Rare earth element also affects graphitization of carbonaceous materials[5,6].

In this paper, effect of boron and rare earth element addition on carbonization and graphitization of Kapton film is discussed.

EXPERIMENTAL

Polyimide films used were commercial Kapton films with 25 μm in thickness and 30 x 30 mm in size. Boron, cerium or lanthanum of 0-1.0 mg was deposited on the polyimide film by PVD method under 10^{-4} Torr. Concurrent deposition of boron and lanthanum up to 1.5 mg was also performed. The deposited films were carbonized at 1000°C in N_2 gas, and then heat-treated at 1500, 2000 or 2500°C in argon gas. Change in mass and size was measured. Interlayer spacing and crystallite size was calculated from X-ray profiles obtained from the heat-treated films.

RESULTS AND DISCUSSION

Mass change of the polyimide films was 56-60% after carbonizing, which coincides with early result[7]. Length along the film surface decreased at 22-23% against the raw film during carbonization, which also coincides with early paper[7]. It increased 12-13% against the carbonized film after heat-treating at 2500°C.

The surface of the non-deposited film heat-treated at 2500°C was flat and smooth, while the surface of the deposited films after heat-treated was rugged as shown in Fig. 1. A striation pattern was different each other depending on the element added.

Figure 2 shows a X-ray profiles of the polyimide films heat-treated at 2500 °C. We cannot find any peak in X-ray patterns obtained from the 1500°C-treated films. In the case of the non-deposited film a broad (002) peak was found at 25.6° for the 2000°C-treated film and 26.2° for the 2500 °C-treated film. In the case of the deposited film, on the other hand, a sharper (002) peak was found about 26° for the 2000 °C-treated film and 26.5° for the 2500 °C-treated film.

Figure 3 represents interlayer spacing calculated from X-ray profiles obtained from the 2000 °C-treated films. Addition of boron, lanthanum or cerium generally accelerated the structural change of the film even at 2000 °C. Concurrent addition of boron and lanthanum accelerated the structural change more rapidly. Abrupt increase in d-spacing above 2200 °C by concurrent addition[5,6] was not found in the case of the polyimide film. At 2500 °C, interlayer spacing of the film was also the same value of 3.366 angstrom.

CONCLUSIONS

Structural change of the polyimide film occurred at 2000 °C and 2500 °C. Boron and rare earth accelerated a structural change of the polyimide film. Concurrent addition of boron and lanthanum also accelerated the structural change of the polyimide film.

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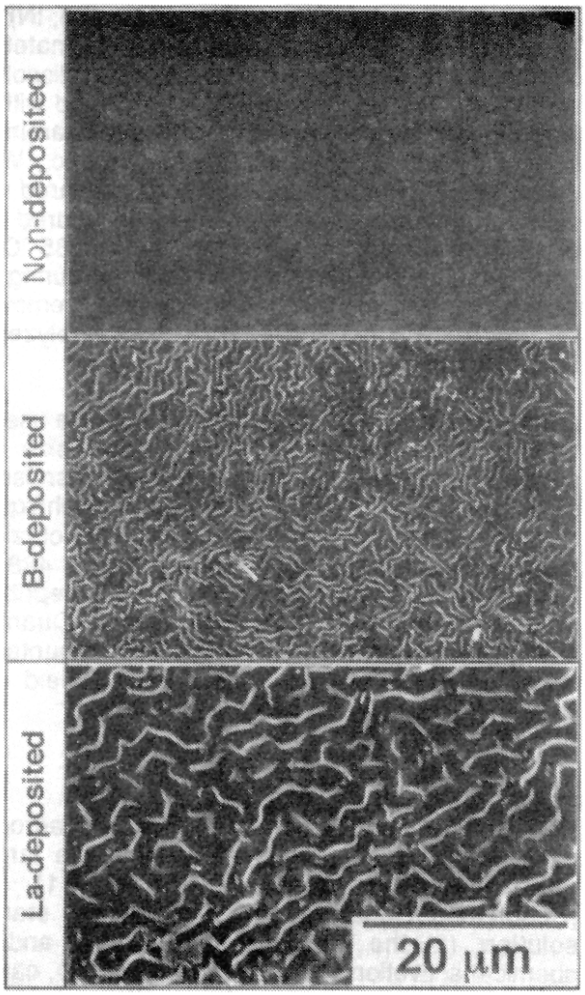

Fig.1 Suface appearances of the polyimide films heat-treated at 2500°C

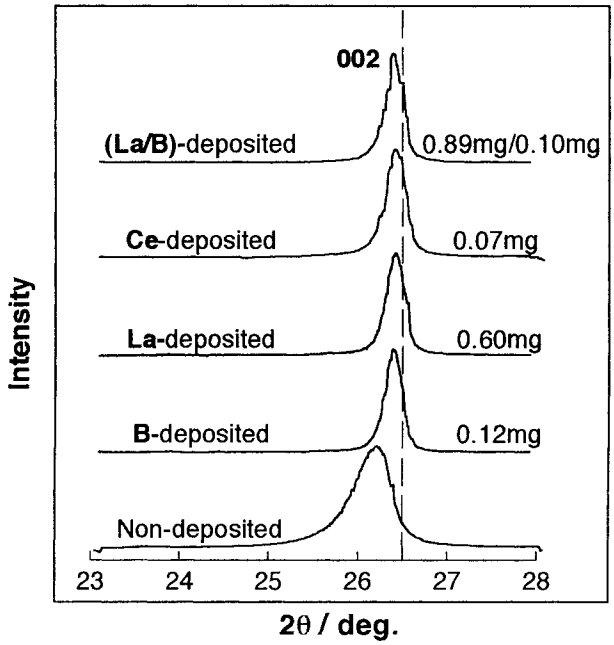

Fig.2 X-ray diffraction patterens of the polyimide films after heat-treatment at 2500°C

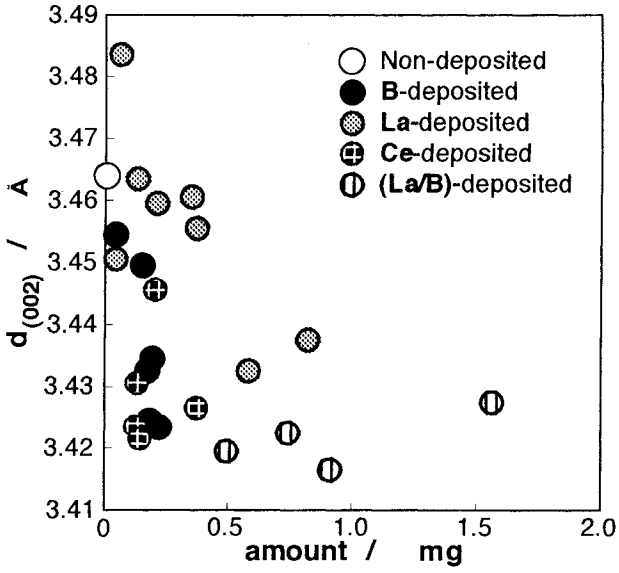

Fig.3 $d_{(002)}$ of the polyimide films heat-treated at 2000°C