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The study of polyimide molecular chain behavior by rubbing with Grazing Incidence X-ray Diffraction (GIXD)

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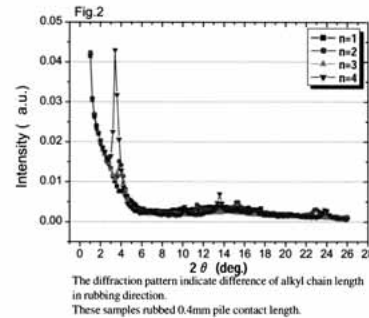Affiliation of the Author(s) Chisso Petrochemical Corporation

INTRODUCTION

Alignment film, which is ordinary formed from polyimide film, is indispensable to liquid crystal display for controlling liquid crystal direction. The grazing incidence X-ray diffraction (GIXD) method is available to know the polyimide surface crystallinity[1]. We studied the influence of polyimide chain length and rubbing strength to crystallinity.

EXPERIMENT

Polyamic acid samples, the precursors of the polyimides represented in Fig.1, were coated on the silicon substrate by a spin coater. The samples were heated at 80 °C for 5 min and subsequently imidized by heating at 210 °C for 30 min. They were rubbed under the condition that the pile contact lengths were 0.4 mm and 0.8 mm. The GIXD was performed at BL19B2 line

Fig.1

Polyimide structure within n is from 1 to 4

in SPring8 according to Hirosawa's study[2].

RESULT AND DISCUSSION

As can be seen in Fig. 2, although the reflectivity is not different upon the alkylene chain lengths of the polyimides, the diffraction patterns indicate the distinct peaks when the alkylene chain numbers are even. This result supported Yokokura's work[3], however, it is uncertain why the even numbers indicate such high crystallinity, and the mechanism is not clear.

The diffraction pattern indicate difference of alkyl chain length in rubbing direction.
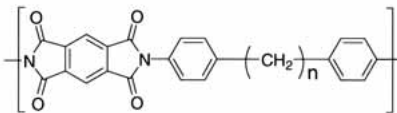These samples rubbed 0.4mm pile contact length.

Furthermore, by changing the pile contact lengths from 0.4 mm into 0.8 mm, the intensity of the scattering peak was increased remarkably with the even alkylene chain samples. This result strongly suggests that the polymer structures correlate with the rubbing acceptance.

REFERENCE

1) Michael F. Toney et al., *Nature*, **374**, p.709-711 (1995)
2) I. Hirosawa, IDW '04 Digest, p.179-182
3) H. Yokokura et al., *Mol. Cryst. Liq. Cryst.*, **225**, p. 253-258 (1993)

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Valence states of silver and tin at the surface of float glass colorized with silver colloids

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Bottom surface of float glass shows reductive characteristic compared with the inside of the glass as a result of diffusion of Sn ions in a molten Sn bath [1]. In case of Ag-printed glass on the bottom side, Ag^+ ions which diffuse into glass are reduced to metallic Ag in the surface reductive layer with the thickness of several micrometers, resulting in coloration due to Ag colloids [2]. In order to clarify the formation reaction of metallic Ag in the layer, X-ray absorption fine structure (XAFS) measurements have been conducted.

Soda lime silicate float glasses (G1 to G3) were prepared for analytical samples. Ag was printed on the bottom surfaces of G1 and G2, whereas G3 remained untreated. The glasses G1 and G2 were heated and consequently Ag was diffused into the glasses. Residual Ag-prints were removed in HNO_3 solution. Furthermore, in case of G2, most of the surface reductive layer was removed with HF etching. The colors of the glass surfaces at Ag-printed sides are deep brown for G1 and light yellow for G2, respectively. Ag and Sn K-edge XAFS spectra of the glasses were collected in a fluorescence mode with glazing angle incidence.

Figure 1 shows Ag XANES spectra of the glasses G1 and G2 in comparison with standard samples. Peak intensity at 25,550 eV in the spectrum of G1 is stronger than that of G2. This suggests that the relative amount of metallic Ag compared with Ag^+ at the surface of G1 is higher than that of G2. Figure 2 shows Sn XANES spectra of the glasses G1 and G3 in comparison with standard samples. Considering edge jump positions in the spectra, it is revealed that most of Sn ions exist as Sn^{2+} in G1 and as Sn^{4+} in G3. These results strongly suggests that diffused Ag^+ ions react with Sn^{2+} ions and converted to metallic Ag according to a following equation;

$$2Ag^+ + Sn^{2+} = 2/n(Ag)_n + Sn^{4+} \qquad (1)$$
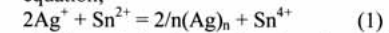
Further study is ongoing in view of the influence of Fe^{2+} ions originally existing in the glass.

References
[1] Y. Hayashi, K. Matsumoto and M. Kudo, J. Non-cryst. Solids 282 (2001) 188.
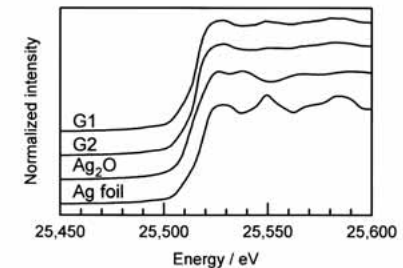[2] S. Takeda, K. Yamamoto and K. Matsumoto, J. Non-cryst. Solids 265 (2000) 133.

Fig. 1 Ag XANES spectra of G1, G2, Ag foil and Ag$_2$O.
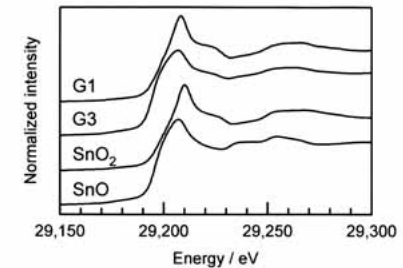

Fig. 2 Sn XANES spectra of G1, G3, SnO$_2$ and SnO.