

Characterization of polyimide films for LCD by X-ray reflectometry and small angle scattering

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Rubbed polyimide(PI) films are widely used in liquid crystal (LC) displays to align LC uniformly. The surface structure of the rubbed PI are very important for control of LC alignmentability. In 2004A, we found the correlation between the degree of crystallinity on the rubbed PI surface and LC alignmentability by grazing -incidence X-ray scattering (GIXS). Not only surface structure but also internal structure of the film is important for LCD properties, because it can control electrical properties of LCD. But there are few methods to characterize the internal structure of very thin PI films (20nm~100nm). So we measured X-ray reflectometry and small angle scattering to clarify the internal structure of PI films at BL19B2 of Spring-8.

A typical PI film for monitors, cellular phone, PDA, etc. was used. A thin film (thickness;100nm) was formed on Si substrate by spin coating was performed with using a multi-axis diffractometer installed in the 2nd hatch of BL19B2. Incident angle of 10.0KeV X-ray to the sample surface was set more than the critical angle of total reflection on the polyimide surface.

Experimental curve (I_{exp}) and calculation curve by monolayer model(I_{cal}) are shown in Figure 1. We can see that I_{cal} is not fit for I_{exp}. This implies the PI may not be monolayer.

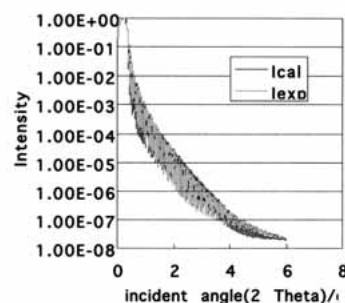


Figure 1 Intensity vs. 2 theta curve for PI film. I_{exp} is experimental curve and I_{cal} is calculation curve(by monolayer model).

We found that I_{cal} by bilayer model is fit for I_{exp} and could clarify the internal structure of the PI film by this method. From these results, we will investigate the correlation between the internal structure of PIs and electrical properties of LCD.

Partial structure analysis of Y₂O₃:Eu phosphor doped with Zn

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Purpose and Background

Rare earth doped yttrium oxide (Y₂O₃:RE) phosphors have been investigated for emitting material of electron beam excitation from initial development of color-cathode ray tubes (CRTs). Recently, field emission displays (FEDs) which are used a same excitation mechanism with CRTs have been studied actively for emissive flat panel displays of next generation. However, the excitation voltage of FED is much lower than CRT. Then, there are many problems such as charging-up on the phosphor surface, stability and resistivity, therefore it is necessary for development of new phosphors and enhancement of commercial phosphors.

Y₂O₃:RE phosphors are expected for FED phosphor because of their stability against electron beam irradiation, however, at low energy excitation, luminescence is insufficient as a result of charging-up on the phosphor surface. It was found that cathodoluminescent properties of Y₂O₃:RE phosphors were improved up to 30% by Zn addition of 10~20% in our experiment. Then, to make clear the contribution of Zn to Y₂O₃:RE phosphors for enhancement of luminescent properties, the analysis using XAFS measurements were tried.

Experimental

Zn doped Y₂O₃:Eu phosphor powders were prepared by citric acid gel method as liquid phase reaction using nitric acid yttrium and zinc and europium chloride for source material. All materials were well-solved in water then the distributions of Zn and Eu are uniformly synthesized this method. Y₂O₃:Eu powder phosphor also prepared using same method for comparison of Zn doped powder.

Results

From results of the observed Eu_L₃ and Zn_K X-ray absorption near edge structure (XANES) spectra of Zn

doped and undoped Y₂O₃:Eu, and Eu₂O₃ clearly showed that Eu atoms exists trivalent ions in Y₂O₃ host material, however Zn in Y₂O₃ exists divalent ions.

Radial structural functions (RSFs) derived from observed the extended X-ray absorption fine structure (EXAFS) oscillations of Zn doped Y₂O₃:Eu was measured. These RSFs were derived by Fourier-transformation of their EXAFS interference functions. Eu³⁺ ion in Zn doped Y₂O₃ host showed similar partial structure with Y³⁺ ion in Y₂O₃, different with Eu³⁺ in Eu₂O₃. It means that Eu³⁺ is replaced Y site in Y₂O₃.

On the other hand, Zn ions did not exist Y-site in Y₂O₃. Fig.1 shows RSFs of Zn doped Y₂O₃:Eu, ZnO, Zn metal and Y₂O₃. The nearest neighbor of Zn signal in Y₂O₃ resembles ZnO. It is thought that ZnO phases partially formed in Y₂O₃ phases. Moreover, the intensity of Zn in Y₂O₃:Eu showed that the actual concentration of Zn in Y₂O₃ is lower than a few mol%. Probably, Zn removed from Y₂O₃ easily during calcinations and only a few amounts of Zn are taken in Y₂O₃.

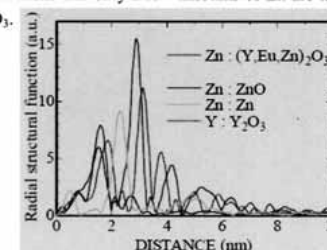


Fig.1 RSFs of Zn doped Y₂O₃:Eu, ZnO, Zn metal and Y₂O₃.

Summary

The partial structure of Zn in Y₂O₃:Eu is analyzed using XAFS measurement. It considered that ZnO phases exists in Y₂O₃ phases. Only few Zn replaced to Y-site in Y₂O₃. Moreover, it is clarified that Zn concentration is smaller than 1 mol%, around 0.1 ~ 0.5 mol%, much lower than the amount of addition during synthesis.