NANOSTRUCTURE OF METALLIC COATING ON PLASTIC FILMS

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Packaging is the largest application of polymers in all countries, at all levels of development. It is about 30-40% of total plastic consumption (1). The development of packaging materials based on plastic commodities is a very dynamic research area (1-3). Polypropylene (PP) has demonstrated a very strong growth over the past several years all around the world. In general, this growth has exceeded that of other high volume commodity polymers and it is projected to continue over the next years (4). PP is an extremely versatile material in the packaging industry. It has low density, excellent heat and chemical resistance, good mechanical properties, and a modest cost (5). However, it has a high gas permeability to be used in some applications. Vacuum vapor deposition of metals on the surface of PP films is used to improve the barrier properties. This processing technique would provide nanocrystalline materials (6). The microstructure of the metallic layer will have a strong effect on the final properties. An adequate knowledge of it would help to ameliorate these properties.In this work we characterized the crystalline microstructure of the aluminum layer vacuum deposited on PP films. The studies included transmission electron microscopy (TEM) and wide angle x-ray diffraction (WAXS). A methodology to analyze the size of Al crystals was applied.

Two commercial metallized PP films of $\cong$ 20 μm total thickness named Sample A and B were analyzed. The aluminum coating was removed from each polypropylene film by dissolving the polymer in hot xylene (approximately 140 °C). The aluminum coating was placed on copper grids for the TEM observation. TEM micrographs were obtained in a Jeol 100-CX electron microscope operated at 80 KV. WAXS experiments were performed directly on the metalized film samples, using an x-ray difractometer PHILIPS PW 1710 with copper anode with curved graphite monochromator. The working conditions were 45 Kv and 30 mAmp, goniometer speed 2 °/minutes. The analyzed area was 1 cm^2 and the samples were duplicated.

Figures 1 and 2 show the TEM micrograph at 80000x (1 cm=0.1μm) of sample A obtained in bright and dark field, respectively. In the bright field (BF) micrograph (Figure 1) aluminum nanograins appear darker due to the diffraction contrast (8). Clear regions could be assigned to amorphous material or crystalline region with different crystal orientation (7). In the dark field (DF) analysis (Figure 2), the clear regions obtained in BF appear bright showing the presence of nanograins of different orientation on the whole sample.

Figures 3 and 4 show BF micrographs of samples A and B, respectively at 80000x (1 cm=0.1μm). The sizes of nanograins are smaller in sample B than in sample A. The grain sizes were measured using specific software. Histograms showing the grain size distributions of both samples are shown in Figure 5. Size of crystals range from 200 to 2400 Å. Average values are 840 and 590 Å for sample A and B respectively.

The crystal size was calculated from the broadening of the x-ray aluminum peak, using the Sherrer methods, assuming that the aluminum crystallite was a cube.

$$\text{Scherrer formula}: \quad \langle D_{hkl} \rangle = K\lambda \,/\, \beta \cos \theta_{hkl}$$

where:
$\langle D_{hkl} \rangle$: average crystal size in the plane $\langle hkl \rangle$ = 111
$2\theta_{hkl}$: Bragg angle in the plane $< 111 >$ = 38.47°
K : Scherrer constant : 0.9.
λ : wave lenght of the x-rays
β: half-width of the peak.

The average crystal size obtained for samples A and B were 980 and 710 Å, respectively. These results are in good agreement with those obtained by TEM. Taking into account that the area analyzed in WAXS experiments was about 1 cm^2, it is considered to be more representative than the smaller regions used in TEM studies.

References

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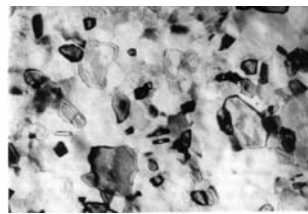[8]Sato M. et al., Science and Technology of Advanced Material, 2004, 5, 145
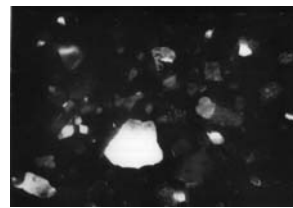
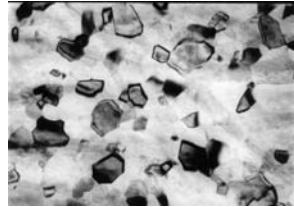**Figure 1-**Sample A, Bright Field

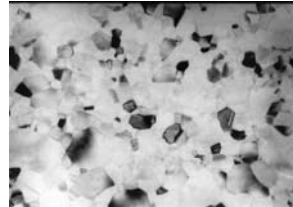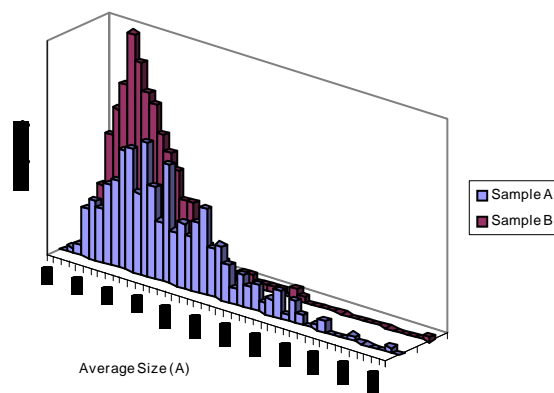**Figure 2**-Sample A,Dark Field

Figure 3 - Sample A

Figure 4 - Sample B

Figure 5- Histograms for Samples A and B