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# Evaluation of Morphology and Crystallinity of Biaxially Oriented Polypropylene Films

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**Abstract.** An investigation on the structural and morphological variations of oriented polypropylene (PP) films processed by two different methods, including biaxial oriented cast film processing (film tentering) and film blowing process (tubular film extrusion) was conducted. Thermal properties obtained by differential scanning calorimetry (DSC) showed no significant difference in melting behaviors of machine direction oriented (MDO) film and the unoriented cast film. However, after transverse drawing (TDO) a small second melting peak was appeared on the left-hand side of the DCS thermogram which was ascribed to presence of kebab lamellae, and the appeared sharp peak was attributed to shish structures in the biaxially oriented PP (BOPP) film. On the other hand, a crystalline structure characterized by wide X-ray diffraction (WXRD) manifested an intensified peak pertinent to the formation of another crystalline structure, i.e. β-modification throughout the blown PP film, contrary to the cast films in which solely α phase existed. The topography of the surface taken by atomic force microscopy (AFM) represented an extensive fibrillar texture in the BOPP film which was in accordance with their DSC results.

**Keywords:** film processing, biaxially oriented polypropylene (BOPP), crystalline morphology
**PACS:** 81.05.Lg, 61.05.C, 81.10.Aj



## INTRODUCTION

Polypropylene (PP) is considerably used as a flexible film for food packaging applications [1-3]. Therefore, the properties of the final PP film such as haze, barrier and mechanical properties are important for such applications. To manage the required properties and produce better packaging films, it is important to follow how the processing variables influence the film morphology, crystallization and orientation; and how this morphology correlates with the film properties [4,5].

The aim of this study is to investigate and characterize the crystallinity and morphology development in the different steps of the biaxially oriented PP (BOPP) films prepared by different processing methods of tenter-frame technology and film blowing process.

## EXPERIMENTAL

### Material

A commercial polypropylene (HP 525J) supplied by Jam Petrochemical company and having a melt flow rate (MFR) of 2.6 g/10 min (under ASTM D1238 conditions of 230 °C and 2.16 kg) was used.

### Film preparation

The cast films were prepared using a twin screw extruder equipped with a slit die. The die temperature was set at 250 °C. In the MDO unit, the produced films were uniaxially stretched at 120 °C under draw ratio of 6. Afterwards, the MDO films were put into the transverse direction orienter (TDO) and were stretched at 120 °C under draw ratio of 9.

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Blown films were made using a single screw extruder with an annular die. The BUR was set at 2 and the applied draw ratio (MD) was 10.

## Film characterization

The thermal behavior of the films was characterized using a differential scanning calorimetry (DSC) Q2000, at a heating rate of 10 ºC/min.

XRD measurements were carried out using a Philips Analytical diffractometer. The generator was set up at 40 kV and 40 mA and the Cu Kα radiation (λ=0.154 nm) was used.

AFM experiments were conducted in non-contact mode using a Dualscope/ Rasterscope C26, DME microscope.

## RESULTS AND DISCUSSIONS

In order to investigate thermal characteristics of processed cast films differential scanning calorimetry (DSC) was employed. As it is conspicuous in Figure 1, there is a subtle difference in melting thermograms by increasing the draw ratio. By stretching the film along machine direction, the melting temperature shifts to higher values which can be ascribed to increasing the crystalline lamellea size after stretching at higher temperatures due to annealing process during MDO mechanism. On the other hand, melting temperature of processed BOPP film along MD and TD directions shifts to higher values and a small bump appears on the left-hand side of the thermogram. Increasing the melting temperature in BOPP film can be the result of transition of structures of stacked lamellae structures into fibrillar structures which are oriented along TD. The melting temperature of formed shish structures which is appeared as a sharp peak in the melting curve of BOPP film is enhanced about 5º C compared to crystalline spherulites/lamellae structures.

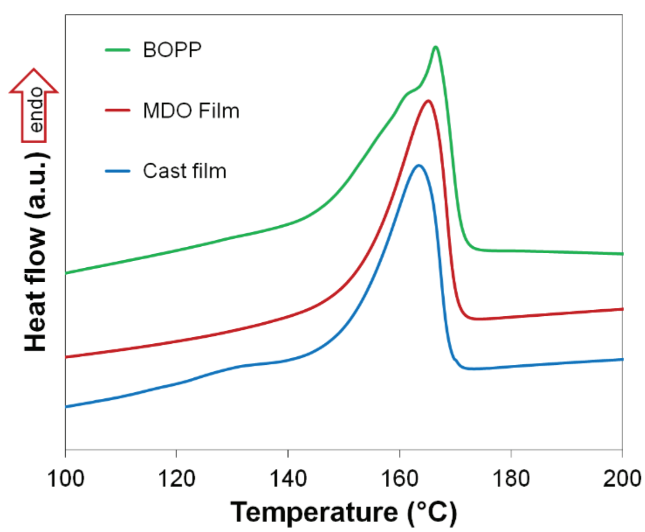


**FIGURE 1.** DSC thermograms of different PP films

Influence of various processing conditions on crystalline phases (α, β, and γ) of polypropylene films were analyzed by wide X-ray diffraction (WAXD). One dimensional WAXD patterns pertinent to cast film, stretched cast film along MD (MDO film), and BOPP are presented in Figure 2.

It is obvious that α phase exists in different films and there is not any traces of β and γ phases in the stretched cast films. It may be concluded that applied elongational forces in MDO and TDO processes have no effects on modification of crystalline phases. It should be pointed out that the intensity of peaks in WAXD patterns are under control of orientation of crystals. Comparison of WAXD graphs of MDO films and TDO films with initial cast films puts emphasis on intensifying the peaks related to TDO and MDO mechanisms.

On the other hand, upon orientation of *a*-, *b*- or *c*-axes of the crystals perpendicular to the surface of the stretched films the reflection of (h00), (0k0) or (00l) planes is intensified. Following investigation of crystalline phases in the blown films a small peak is clearly noticed at 2θ=16.1 indicative of crystalline β-structure in this sample [6]. It can be attributed to the difference in processing conditions leading to presence of β phase in the blown film.

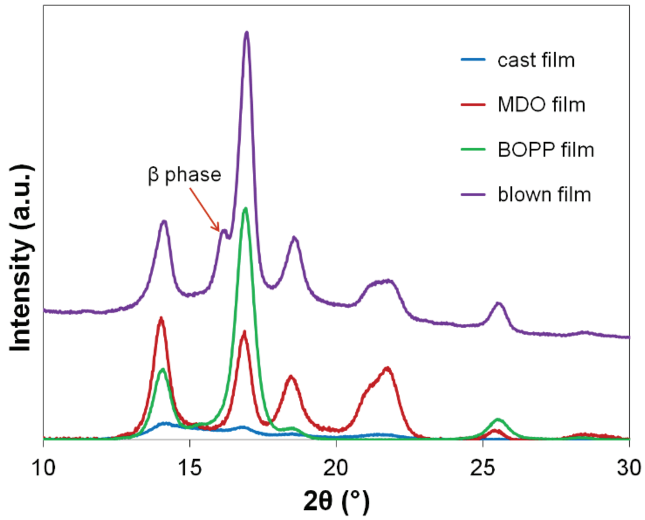

**FIGURE 2.** WAXD patterns of different PP films

In the process of blown film extrusion, applied elongational modes are performed in the melt state. However, in BOPP film preparation, elongational modes are applied in the solid state. Hence, β phase is formed as a consequence of stretching in crystallization process in the blown film.

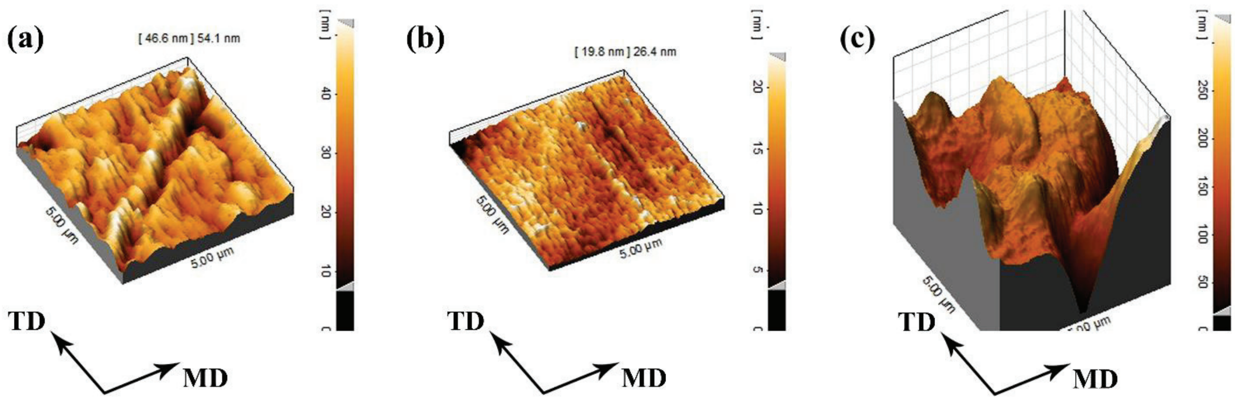

**FIGURE 3.** AFM topographies of PP films: (a) MDO film; (b) BOPP film; (c) blown film

To analyze the topography of processed films, atomic force microscopy (AFM) was utilized. According to AFM images taken for MDO film (Figure 3a), stacked lamellae structures can be observed along machine direction (MD). In the BOPP films (Figure 3b), it is obvious that crystalline fibrils are oriented along the TD. Exposure of samples to TD stretching after MDO causes to appear fibrillar structures along the TD. However, stretched film along MDO (Figure 3a) does not show any fibrils which is due to the lower draw ratio applied in comparison with the TDO draw ratio value. Figure 3c presents the AFM topography of the blown film. A random crystalline alignment is observed for the blown film which is due to simultaneous stretching in machine and transverse directions. Comparing the AFM 3D topographies of different samples as well as their mean roughness values indicates that the blown film shows a rougher surface compared to BOPP and MD films. The difference in surface-roughnesses can be related to various processing conditions ensuing sundry crystalline structures throughout the films.

## CONCLUSIONS

Different processing methods for preparation of PP films, render different morphologies and crystalline structures in the oriented PP films. Morphology of PP cast films changed to a stacked lamellar structure after stretching in MD direction (MDO film). In the BOPP film the applied stresses in the TD resulted in orientation of crystalline structures in the transverse direction and a distinct fibrillar structure was observed. However, simultaneous stretching in the TD and MD in the blown film led to a random crystalline alignment and no predominant TD direction fiber alignment was noticed. In DSC results no significant differences between the melting curve of cast film and MDO film was detected. Nevertheless, the BOPP film exhibited a small shoulder and a sharp peak which are attributed to the presence of lamellae and fibrils, respectively. The WAXD measurements for the cast film and stretched films (MDO and BOPP) showed no intensity peaks corresponding to the beta crystal form. However, melt state stretching of PP in film blowing process led to the formation of β-modification.

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