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Article in *Journal of Plastic Film and Sheeting* · December 2013

DOI: 10.1177/8756087913487542

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# The relationships between crystallization characteristics and heat sealing properties of high-density polyethylene films

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and Go Murasawa<sup>2</sup>

## Abstract

The relationships between crystallization characteristics and heat sealing properties of high-density polyethylene (HDPE) films were determined. HDPE with a 1.1 g/10 min melt flow index (MFI) (190°C, 2.16 kg) was melt mixed in a twin-screw extruder with a 0.04 g/10 min MFI HDPE. The high molecular weight HDPE (HMW HDPE) concentration was varied between 1 and 20 wt%. The HDPE blends were cast into a 50 µm thick film. The films were heat sealed at 125°C and 128°C. The seal bar pressure was kept constant at 0.13 MPa. The heat seal time was varied from 0.5 to 2.0 s. Heat-sealed strength was measured by T-peel test. The molecular structure development during the heat seal process was evaluated by differential scanning calorimeter and wide-angle X-ray diffraction. The HMW HDPE decreased the film heat seal strength when sealed at 125°C. However, at 128°C the presence of up to 10 wt% reduced the heat sealing time.

## Keywords

Film processing, heat sealing, molecular structure, crystallization, crystalline orientation, polyethylene

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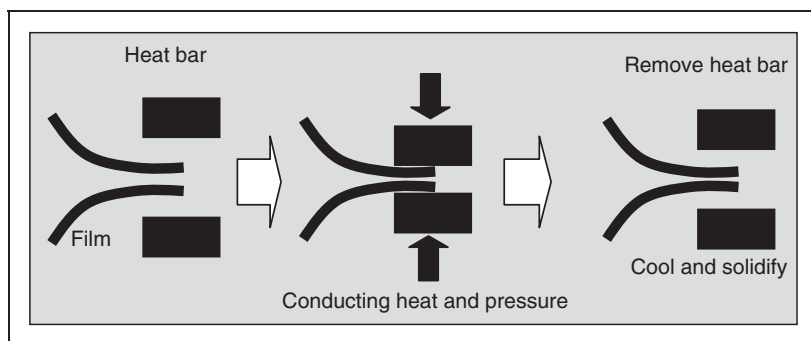
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## Introduction

Flexible laminated film packaging systems usually consist of a base film with a sealant layer and an additional functional layer. Oriented polypropylene (OPP) film, oriented poly(ethylene terephthalate) (OPET) film, or oriented polyamide (OPA) film are usually used as the base layer. These single layer base film materials do not have appropriate heat seal properties. In order to improve heat seal properties, sealant film materials such as low-density polyethylene, linear low-density polyethylene, and polypropylene co-polymerized with 2 to 5 mol% ethylene components can be laminated to the base film as a sealant layer.<sup>1</sup>

Heat seal is a conventional technique used for joining polymer films used in packaging industries. The fundamental process is firmly pressing polymer film surfaces together by heat bars where molecular chains, in a partially molten state, can diffuse across interfaces. This method is referred to as a heat bar system.<sup>2</sup> Figure 1 shows an illustration of the heat bar system. To achieve sufficient bonding, film surfaces must be adhered together with adequate pressure for sufficient time so that the polymer chains can diffuse across the interfaces and develop an interconnected molecular structure.<sup>3</sup> Adhesive strength of heat-sealed polymer film is defined as heat seal strength. It is influenced by heat seal temperature, heat seal pressure, and dwell time.<sup>4</sup> Heat seal strength is a function of heat seal temperature and relates to the polymer film melting temperature. The joining mechanism in the heat seal process has not been reported in detail. Hashimoto et al.<sup>5-8</sup> reported mechanical properties of heat-sealed part and molecular structure development. Relationship between heat seal temperature, time and pressure was reported. Morris<sup>9</sup> investigated heat sealing behavior of ionomer film. Stehling and Meka<sup>10</sup> reported that the amorphous phase fraction is a major factor for determining peeling properties. In their study, Stehling and Meka determined that seal initiation temperature corresponded to the temperature at which the amorphous phase fraction equals  $77 \pm 3$  wt%. In our earlier studies, we determined that crystalline melting enthalpy was a major factor in determining peeling properties of heat-sealed high-density polyethylene (HDPE) films.<sup>11</sup>



**Figure 1.** Heat sealing process with a heat bar.

The film surface properties are important to control heat seal characteristics. In the current study, flat film surfaces smooth on a micrometer scale were studied.

There are some important nano-scaled factors in structure development at the seal interfaces. The major factor is inter-molecular attractive force. The strong inter-molecular forces could appear due to secondary factors, such as high order structures, which are related to molecular chain entanglement, crystallization, and molecular structures.<sup>12</sup> This structure development is strongly influenced by molecular parameters, such as the time scale of molecular chain entanglement formation and the rate of crystallization. Entanglement formation is related to the time for maximum relaxation. The maximum relaxation time for common polymers, such as polyethylene and polystyrene, is between  $10^1$  and  $10^3$  s.<sup>13</sup> The molecular entanglement is an important molecular parameter for amorphous polymers.<sup>14</sup> In our study, we defined 'seal initiation entanglement number' as the polymer chain entanglement number when seal strength starts to increase. We reported that the seal initiation entanglement number was 0.02 per a polymer chain. On the other hand, polyethylene is a fast crystallizing polymer. Polyethylene crystal growth rate from melt state was  $10^6$ – $10^8$  nm/s.<sup>15</sup> For the usual sealant polymers, such as polyethylene and polypropylene, crystallization might influence heat seal properties due to the short process time, which was usually around  $10^4$  nm/s.

Heating and cooling conditions are important parameters in the heat seal process. The heater bar temperature is precisely controlled. The cooling conditions are difficult to precisely control since the heated film cools freely down to ambient temperature in air. During the cooling, heat seal characteristics are usually dictated by crystallization and sealant film layer solidification. Fast crystallizing polymers are usually selected for most sealant film layers.

## Experimental

### *Materials and film processing*

Two commercial HDPE grades from Prime Polymer Co., Ltd, Japan were used in this study. Both had a density of  $0.950\text{ g/cm}^3$  with a  $130^\circ\text{C}$  melting temperature. The first HDPE grade, 3300F, had a  $1.1\text{ g/10 min}$  melt flow index (MFI) ( $190^\circ\text{C}$ ,  $2.16\text{ kg}$  load). The higher molecular weight HDPE 7000F, had  $0.04\text{ MFI}$  ( $190^\circ\text{C}$ ,  $2.16\text{ kg}$  load). Throughout this report, these HDPE grades are referred to as 3300F and 7000F. The 7000F in the prepared blends was set at 0, 1, 3, 10, 20, and  $100\text{ wt}\%$ .

3300F and 7000F were dry mixed and fed to a twin-screw extruder equipped with a T-die (KZW-30; Technovel Co., Ltd.). The screw length to diameter ratio (L/D) was 45. The extruder barrel temperature was set at  $190\text{--}210^\circ\text{C}$ . The T-die temperature was set at  $210^\circ\text{C}$ . The extruded film thickness was controlled to  $50\text{ }\mu\text{m}$  by adjusting the chill roll speed and the screw RPM. The chill roll speed was set at  $0.45\text{ m/min}$  with the screw RPM at  $100\text{ rpm}$ , in order to keep a constant throughput rate. The chill roll surface temperature was set to  $40^\circ\text{C}$  to repress crystallization. The film samples were aged for more than  $48\text{ h}$  at room temperature before performing the heat seal tests.

### *Heat seal tests*

Extruded film samples were cut to  $100\text{ mm}$  long and  $15\text{ mm}$  wide specimens. The specimens were heat sealed in a heat seal tester (TP-701-B; Tester Sangyo Co., Ltd.). Two films were thermally sealed at near their melting temperature. The heat seal bar temperature was set at  $120^\circ\text{C}$ ,  $125^\circ\text{C}$ ,  $128^\circ\text{C}$ , and  $130^\circ\text{C}$ . The seal bar thermal capacity was enough to keep the temperature constant. Before the heat seal test the seal bar surface temperature was checked. The seal pressure was kept constant at  $0.13\text{ MPa}$ . The heat seal time was varied between  $0.2$  and  $5.0\text{ s}$ .

### *Characterization*

Heat seal strength was evaluated in a universal tensile tester (Instron3342, Instron Co., Ltd.) ‘T-peel’ test machine. The heat-sealed films were peeled at  $300\text{ mm/min}$ . For each condition, the ten specimen arithmetic average was recorded as the heat seal strength for that condition.

HDPE blend films were initially heat sealed at temperatures from  $120^\circ\text{C}$  to  $130^\circ\text{C}$ . However, sufficient seal strength was not obtained at  $120^\circ\text{C}$ . At  $130^\circ\text{C}$  the heat-sealed film melted and deformed due to high heat bar temperature.

In our study, we were able to obtain acceptable heat-sealed films at 125°C and 128°C.

The crystal structure of the films heat sealed at 125°C and 128°C was investigated by wide-angle X-ray diffraction (WAXD). The WAXD traces were recorded with a Rigaku/RINT RAPID diffractometer (Rigaku Corporation). Monochromed CuK $\alpha$  radiation source at 30 kV and 20 mA was used. Two-dimensional digital diffraction data were recorded using an imaging plate. Diffraction intensity distribution on the equator, the meridian and azimuthal direction for (110) at  $2\theta = 21.7^\circ$  and (200) at  $2\theta = 24.1^\circ$  were evaluated. In this study diffraction intensity on (200) plane was enough to analyze. Azimuthal intensity distribution for (200) will be discussed in detail.

Crystallization of the heat-sealed films was analyzed with a differential scanning calorimeter (Diamond DSC; PerkinElmer, Inc.). In DSC tests, the sample was heated from 30°C to 150°C at 20°C/min under a nitrogen atmosphere. The endothermic peak, peak temperature, and the change in endothermic enthalpy ( $\Delta H$ ) were determined.

## Results and discussion

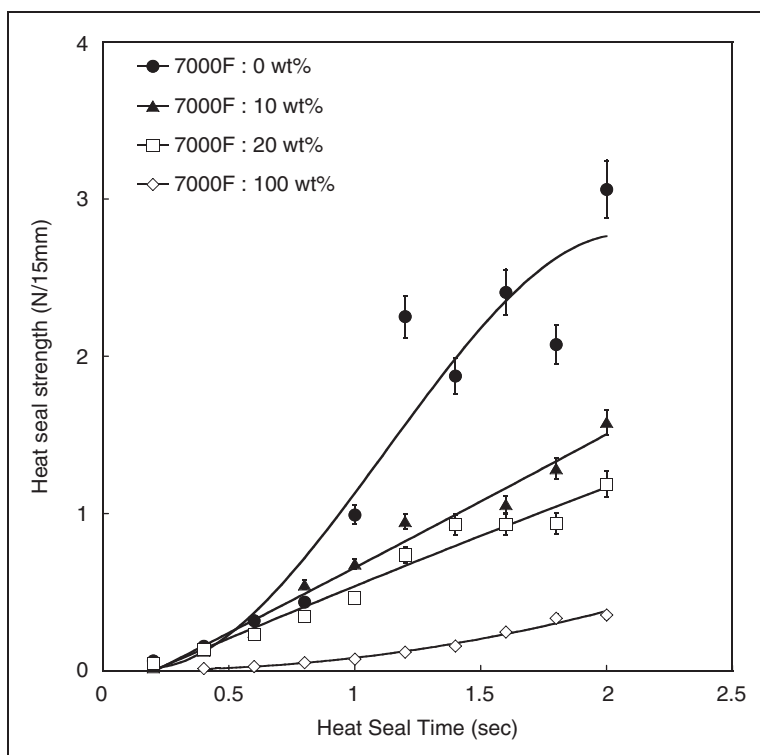
### *Heat seal strength*

Figures 2 and 3 show heat seal strength as a function of heat seal time at 125°C and 128°C. In our experiments, three failure modes were identified:

- **Adhesive** failure is when the sealant layer separated from the pre-sealed film surface.
- **Cohesive** failure is when the sealant layer was destroyed in peeling. For cohesive failure, the film surface became cloudy due to the strain induced crystallization during the sealant layer deformation.
- **Tearing** failure is when the heat-sealed film was not peeled at the sealing interface but rather, broken.

Figure 2 shows the effect of high molecular weight HDPE 7000F content and heat seal time on heat seal strength at 125°C. The results show that the film seal initiation temperature was about 125°C. Heat seal strength increased with increased heat seal times. Heat seal strength decreased with increasing 7000F content which was considered to be caused by the anticipated 7000F longer relaxation time. Note that polymers with a longer relaxation time usually need a longer heat seal time and a higher heat seal temperature to attain efficient heat seal strength. Heat seal strength was less than 3 N/15 mm (518 g/in) at 125°C (Figure 2). Note that, low heat seal strength, especially less than 1.5 N/15 mm indicated adhesive failure. For 100% 3300F heat-sealed

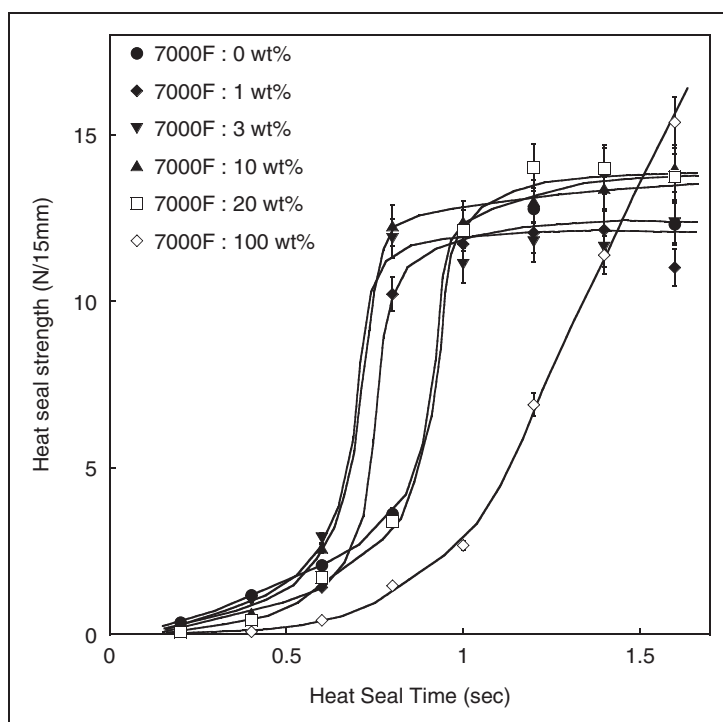
films, heat seal strength was more than 1.8 N/15 m. Thus, failure mode changed to cohesive failure at this high heat seal strength.



**Figure 2.** Heat seal strength at 125°C versus heat seal times at the studied HMW HDPE (7000F) concentrations.  
HMW: high molecular weight; HDPE: high-density polyethylene.

Figure 3 shows the effect of 7000F content and heat seal time on heat seal strength at 128°C. Film heat seal strength drastically increased at 128°C. All film heat seal strength increased with increasing heat-sealed times, which were more than 10 N/15 mm (1730 g/in.). The heat seal strength of films which contained 20 wt% 7000F drastically increased at heat seal times between 0.6 and 0.8 s and exhibited almost constant heat seal strength between 10 and 14 N/15 mm. 7000F heat seal strength started to increase at 1.0 s and was the highest at 1.5 s equal to 15 N/15 mm. Note that low heat seal strength values, less than 1.5 N/15 mm, indicated adhesive failure. The failure mode changed to cohesive failure at 1.8 N/15 mm (Figure 2). Heat seal strength drastically increased and reached a limit at heat seal times greater than 0.6 s

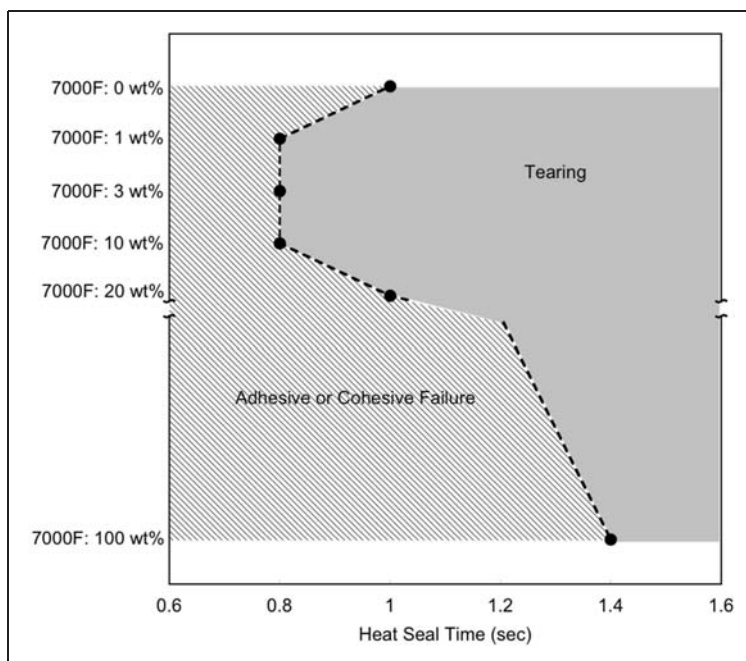
and 128°C. Therefore, the failure mode changed to tearing failure for 10 wt% 7000F at 0.8 s. Neat 7000F heat-sealed films required 1.4 s to attain high heat seal strength and exhibited tearing failure.



**Figure 3.** Heat seal strength at 128°C versus heat seal times at the studied HMW HDPE (7000F) concentrations.  
HMW: high molecular weight; HDPE: high-density polyethylene.

Figure 4 maps the failure modes as a function of 7000F content and heat-sealed times at 128°C. One clearly sees the change in the failure mode, from adhesive or cohesive failure to tearing failure mode. Incorporating 7000F influenced the failure mode. 7000F from 1 to 10 wt% transitioned from cohesive to tearing failure mode at 0.8 s. At 20 wt% 7000F the transition was at 1.0 s. For 100% 7000F this transition was at 1.4 s. From these results, the incorporating 7000F at 1–10 wt% lowered the heat seal time to 0.8 s in order to obtain tearing failure.



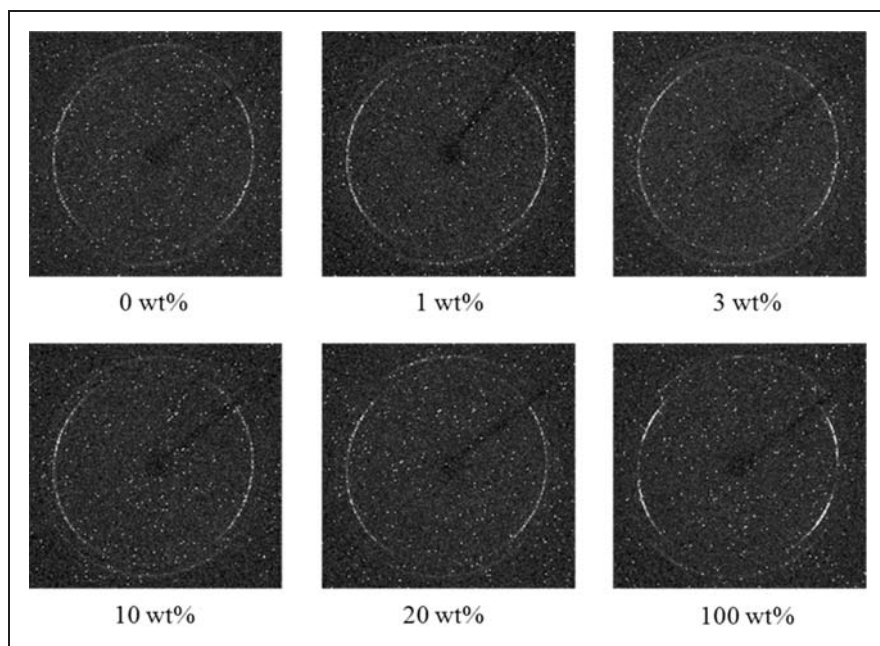


**Figure 4.** Failure mode map for HMW HDPE concentration and heat seal time at 128°C.

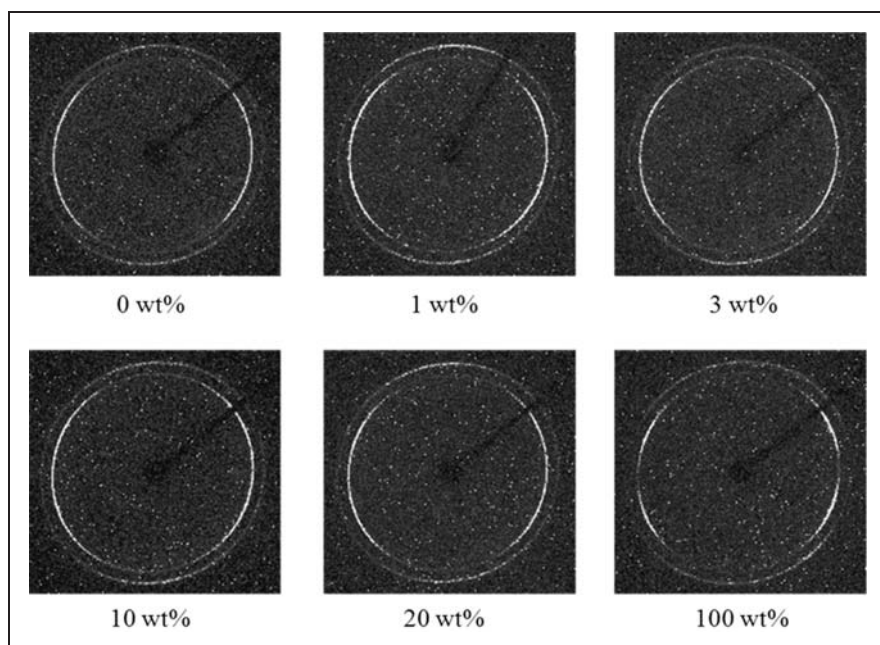
HMW: high molecular weight; HDPE: high-density polyethylene.

### WAXD and DSC analyses

The effect of 7000F on the heat-sealed film crystalline structure was investigated by WAXD patterns, which were taken normal to the plane direction. Figures 5 and 6 show the before and after heat sealing WAXD patterns for the 7000F concentrations studied. In WAXD patterns typical a-axis oriented diffraction patterns were obtained.<sup>16</sup> Reflections from (110) and (200) planes in the orthorhombic structure were clearly observed at  $2\theta = 21.7^\circ$  and  $24.1^\circ$ , respectively. The 1–20 wt% 7000F blended films WAXD patterns were similar to the HDPE 3000F film. The 7000F film shows a symmetrical 4 spot (110) crystalline reflection pattern for a-axis oriented HDPE films. The HDPE blended film WAXD patterns changed slightly due to the crystallization which occurred after heat sealing as shown in Figure 6. The (110) and (200) planes reflection intensity became strong and sharp after the heat sealing process. Therefore, the crystallization generated in the heat seal process could relate to heat seal strength.



**Figure 5.** Wide-angle X-ray patterns before heat sealing for HDPE blended films. HDPE: high-density polyethylene.



**Figure 6.** Wide-angle X-ray patterns after heat sealing for HDPE blended films. HDPE: high-density polyethylene.

Figures 7 and 8 show the azimuthal WAXD patterns of HDPE blended films before and after heat sealing. As the intensity profiles in Figures 7 and 8 are similar, quantitative analyses was required to understand if there was an effect on crystalline structure. The azimuthal intensity distribution of the orthorhombic (200) reflection at  $2\theta = 24.1^\circ$  was analyzed. For the crystalline structure of extruded PE film it is known that a- and c-axis are randomly oriented to the machine direction.<sup>17</sup> To evaluate the film crystalline orientation, reflection from (200) plane was selected by using azimuthal intensity distribution in Figures 7 and 8. The average square cosine angle,  $\langle \cos^2 \phi \rangle$ , was calculated from the azimuthal intensities  $I(\phi)$  by equations (1) and (2), where  $\phi$  is the angle between the machine direction and the normal vector of (200) plane at  $2\theta = 24.1^\circ$ .

$$\cos \phi = \cos \theta \cos \delta \quad (1)$$

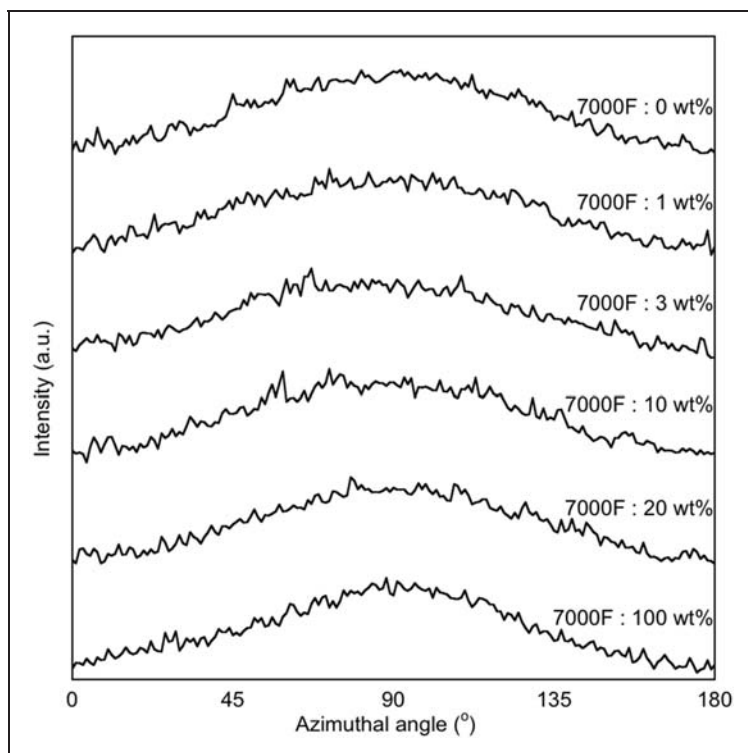
$$\langle \cos^2 \phi \rangle = \frac{\int_0^{\pi/2} I(\phi) \cos^2 \phi \sin \phi d\phi}{\int_0^{\pi/2} I(\phi) \sin \phi d\phi} \quad (2)$$

where  $\theta$  is the Bragg angle and  $\delta$  is the azimuthal angle in WAXD pattern.

The orientation function,  $f$ , is calculated by the following equation

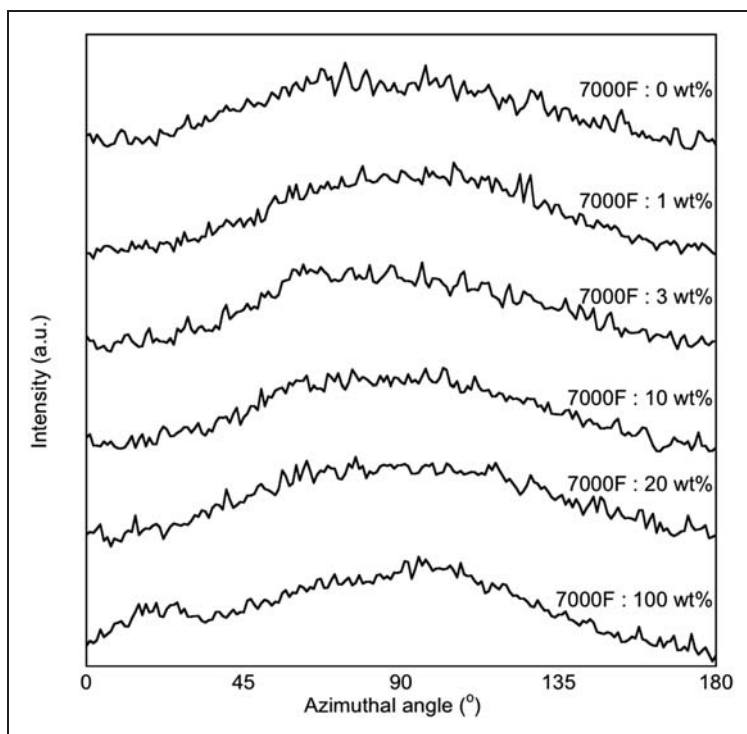
$$f = \frac{3\langle \cos^2 \phi \rangle - 1}{2} \quad (3)$$

In this study, the cast HDPE film crystals were not highly oriented. Therefore, the average square cosine angle,  $\langle \cos^2 \phi \rangle$ , in equation (2) was evaluated.



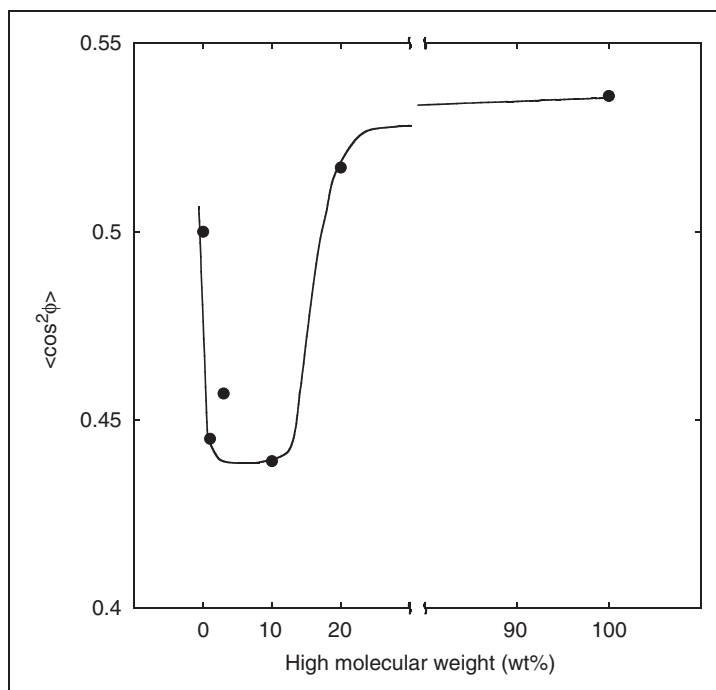
**Figure 7.** Azimuthal intensity distribution of (200) at  $2\theta = 24.1^\circ$  crystalline reflection from WAXD pattern of films before heat sealing.

WAXD: wide-angle X-ray diffraction.



**Figure 8.** Azimuthal intensity distribution of (200) at  $2\theta = 24.1^\circ$  crystalline reflection from WAXD pattern of films after heat sealing. WAXD: wide-angle X-ray diffraction.

Figure 9 and Table 1 presents the calculated  $\langle \cos^2 \phi \rangle$  as a function of high molecular weight concentration before heat sealing. The calculated  $\langle \cos^2 \phi \rangle$  before and after heat sealing was almost unchanged, which would imply that the films have similar crystalline orientation. The minimum calculated  $\langle \cos^2 \phi \rangle$  occurred from 1 to 10 wt% high molecular weight before and after heat sealing. This indicated that 1–10 wt% 7000F suppressed the crystalline orientation which corresponded to reduced heat seal time and the tearing failure as shown in Figure 4.



**Figure 9.** HDPE blended films before heat sealing (200) crystalline orientation  $\langle \cos^2 \phi \rangle$  versus high molecular weight concentration.  
HDPE: high-density polyethylene.

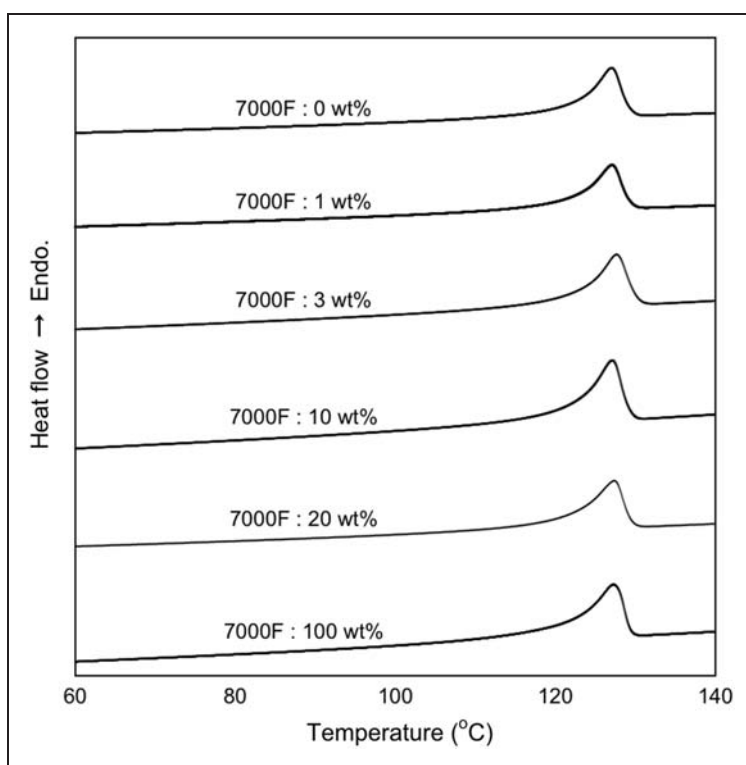
**Table 1.** HDPE blended films (200) crystalline orientation  $\langle \cos^2 \phi \rangle$  before heat sealing.

High molecular weight concentration (wt%)	0	1	3	10	20	100
$\langle \cos^2 \phi \rangle$	0.498	0.445	0.457	0.439	0.517	0.536

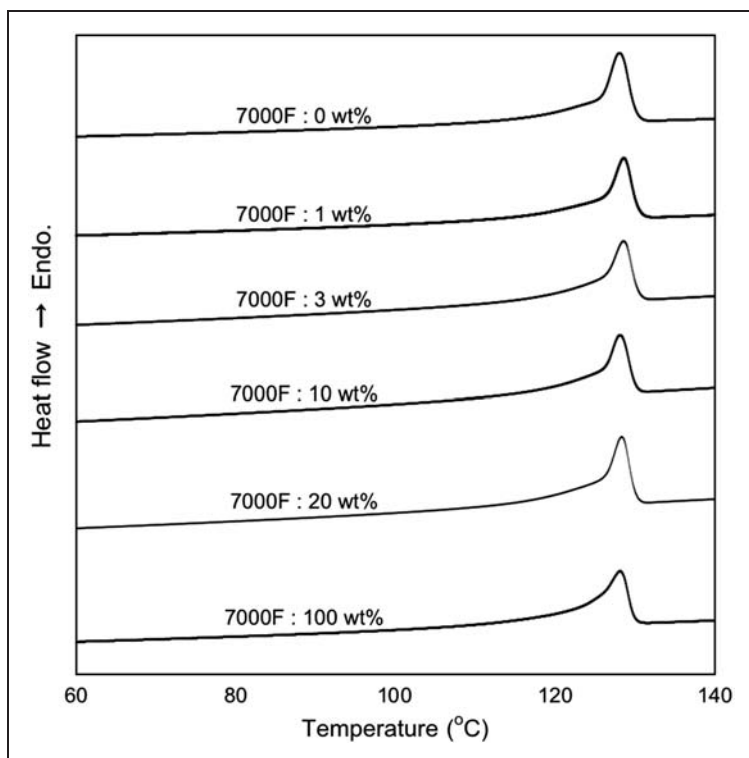
HDPE: high-density polyethylene.

Figures 10 and 11 present DSC thermograms before and after heat sealing. Endothermic crystal melting peak temperature increased slightly with increasing high molecular weight concentration. Melting endothermic enthalpies were determined to analyze the film crystallization behavior. Figure 12 and Table 2 shows the melting enthalpy change determined from DSC thermograms before and after heat sealing at 128°C for 0.8 s. Note that the melting enthalpy change is a measure for the sample crystallinity. The results revealed that the endothermic enthalpy change was low in HDPE blended films with 1–10 wt% 7000F before and after heat sealing. This indicates that they had lower degree of crystallization, purportedly with a low crystalline orientation.

The endothermic enthalpy change of HDPE blended films before and after heat sealing was plotted in Figure 13 and also shown in Table 2. The results exhibit the increment of endothermic enthalpy change after heat sealing as a function of high molecular weight concentration. Neat 7000F heat-sealed film shows the highest endothermic enthalpy increase whereas the minimum values were found in the heat-sealed films at 1–10 wt% 7000F as presented in Figure 13. The change of endothermic enthalpy increased upon blending with high 7000F concentration. These results support that the film crystallization was inhibited at 1–10 wt% 7000F, and enhanced at higher 7000F concentrations. We believe that the lower concentration (1–10 wt%) of high molecular weight HDPE (7000F) suppressed crystallization. This shortened the heat seal time and induced tearing failure of these heat-sealed films.

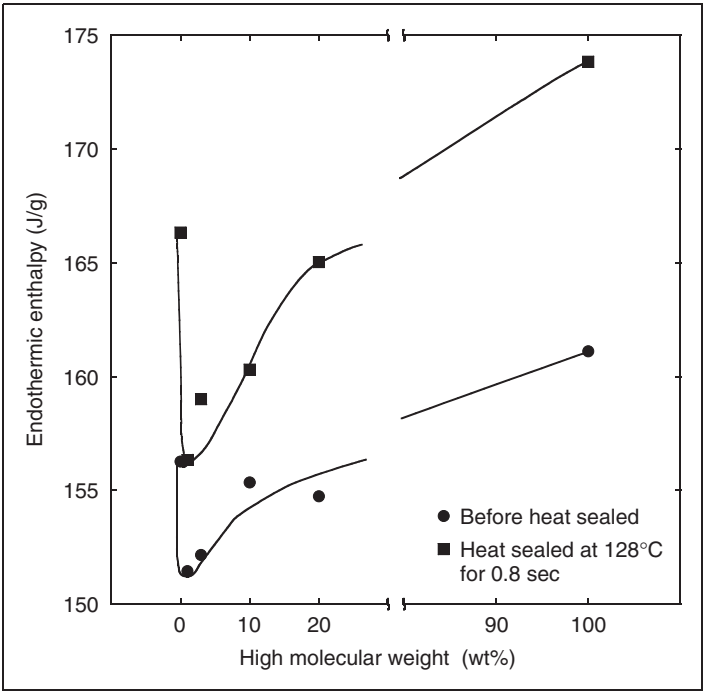


**Figure 10.** DSC thermograms before heat sealing.  
DSC: differential scanning calorimetry.

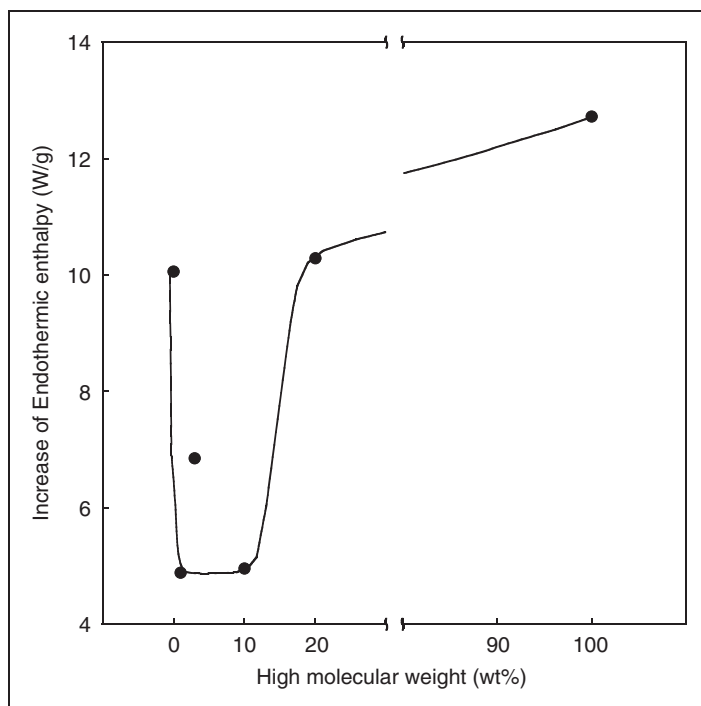


**Figure 11.** DSC thermograms after heat sealing.  
DSC: differential scanning calorimetry.





**Figure 12.** DSC melting enthalpy change versus high molecular weight concentration, before and after heat sealing at 128°C for 0.8 s. DSC: differential scanning calorimetry.



**Figure 13.** Melting enthalpy increase versus high molecular weight in the heat sealing process.

**Table 2.** HDPE blended film melting enthalpy before and after heat sealing.

High molecular weight HDPE (7000F) content (wt%)	0	1	3	10	20	100
Enthalpy before Sealing (W/g)	156.3	151.5	152.2	155.4	154.8	161.1
Enthalpy after Sealing (W/g)	166.3	156.3	159.0	160.3	165.0	173.8
Enthalpy increase (W/g)	10.0	4.8	6.8	4.9	10.2	12.7

HDPE: high-density polyethylene.

### *Relationship between heat seal time and film crystallinity*

Stegling and Meka<sup>10</sup> suggested a molecular structure formation model during heat sealing. In heat seal process, film interface melts when pressed between the heated bars. Upon melting, chains diffuse and form entanglements under heat and pressure. Upon removing the heat bar, the sealed films, cool, solidify and crystallize. In this study, HDPE blend films with 1–10 wt % 7000F

exhibited tearing failure and reduced heat seal time. This was attributed to lower crystallinity and reduced crystalline orientation in these heat-sealed films. Therefore, the films with lower 7000F concentration would require shorter heating time versus the neat HDPE film. Chains can sufficiently diffuse at film interface and form entanglements. Moreover, melted polymer film crystallized along with the chain entanglements to further improve the heat seal strength.

The film with 20 wt% high molecular weight HDPE (7000F) required longer heat seal time to melt the film interface than the films with a lower 7000F concentration. This is attributed to the higher crystallinity and higher crystalline orientation of the films with 20 wt% 7000F. Melting at the interface and chain diffusion can occur upon application of enough heat seal time. It is interesting to note that 1–10 wt% high molecular weight concentration in HDPE blend films induced a reduction in crystallinity and purportedly suppressed crystalline orientation which resulted in reduced heat seal time.

## Conclusions

Adding greater than 10 wt% high molecular weight HDPE to a lower molecular weight HDPE decreased the heat seal strength of the HDPE blend films at 125°C heat seal temperature. HDPE blend films, which contained less than 10 wt% high molecular weight HDPE exhibited excellent heat seal strength at 128°C heat seal temperature and 0.8 s heat seal time. These heat sealed films showed tearing failure mode. WAXD and DSC results confirmed reduced crystalline orientation and reduced extent of crystallinity of these films. We concluded that the HDPE film heat seal strength was remarkably affected by crystallization behavior upon adding higher molecular weight HDPE. The optimum high molecular weight HDPE content for these HDPE blend films was 1 wt%.

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## Biographies

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