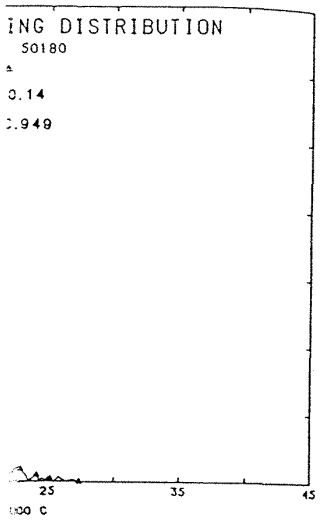
# EXHIBIT V

# PART 21

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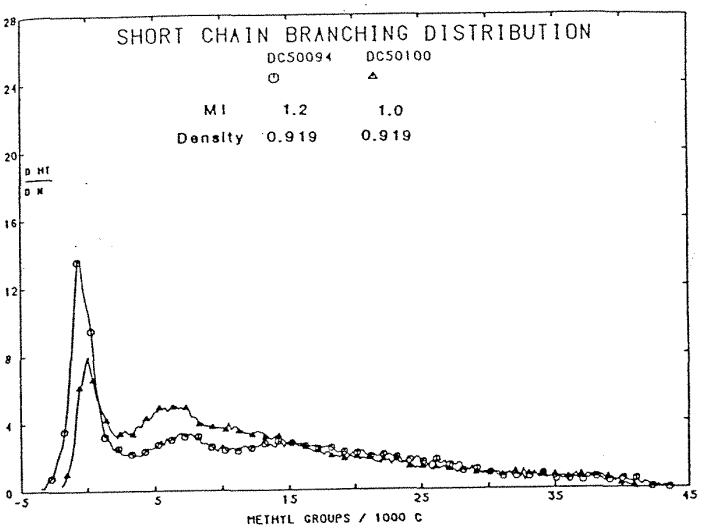


Fig. 10. SCB distributions for two commercial LLDPE resins made by different processes: (⊙) MI 1.2, density 0.919; (▲) MI 1.0, density 0.919.

ethyl acrylate are also semicrystalline and can be analyzed by TREF. In Figure 11 a comparison is made of branching distributions obtained for ethylene vinyl acetate (EVA) copolymers of 9, 14, and 18 wt % VA, as well as that for an ethylene–ethyl acrylate copolymer. As expected, increasing levels of VA show up as high levels of branching. It is interesting to note that the copolymer with ethyl acrylate is characterized by a broader, less smooth distribution compared to the corresponding EVA copolymer. This probably reflects the influence of differing reactivities of VA and EA with ethylene. It should be noted that these ethylene copolymers made by the high-pressure process also contain ethylene-type short-chain branching and that the data shown reflect the combined effects of this branching and that due to the comonomer. Whether the larger comonomer units disrupt crystallinity to different degrees than normal ethylene short-chain branching is not known at present although a study of this aspect is planned.

Throughout these series of fractionations the analytical TREF system worked well and was reasonably convenient to operate. The main experimental problem proved to be the difficulty in maintaining stable operation of the refractive index detector. Base-line drift, apparently due to room-temperature fluctuations and drafts, caused a number of runs to be aborted. Improved temperature stabilization of the detector housing and in particular of the preheat section, which conditions the eluent prior to flow through the detector block, should lead to improved operating efficiency. Under these conditions a schedule of two fractionations per day should be easily maintained. This is clearly a considerable improvement over the two to three runs a week experienced with the original preparative TREF system.

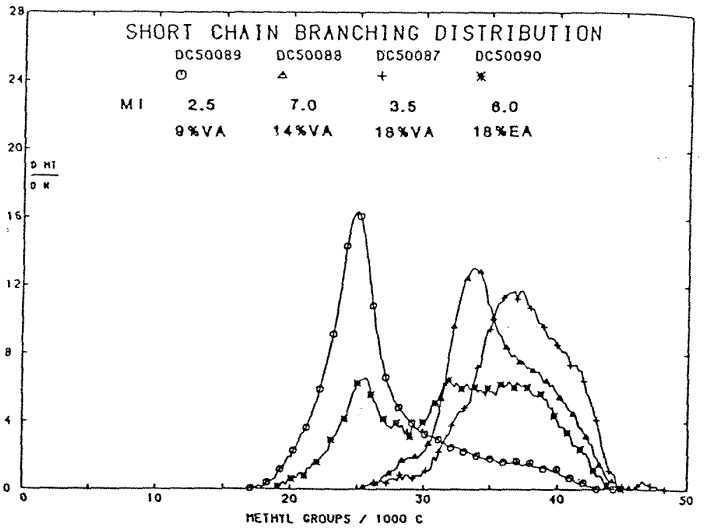


Fig. 11. SCB distributions obtained for ethylene copolymers: (⊙) MI 2.5, 9% VA; (▲) MI 7.0, 14% VA; (+) MI 3.5, 18% VA; (*) MI 6.0, 18% EA.

## CONCLUSIONS

(i) Temperature-rising elution fractionation is an effective technique for fractionating polyethylenes with respect to degree of branching, as it is relatively free of molecular weight influences.

(ii) Use of smaller sample size and an on-line detector, as in the analytical TREF system described above, increases both the operating efficiency and the quality of the fractionation.

(iii) Calibration with suitable standards allows direct calculation of meaningful branching distributions. This elminates the need for analysis of individual fractions.

(iv) Analysis of a polyethylene resin using the TREF system in conjunction with size-exclusion chromatography provides a rapid evaluation of overall resin structure. This should provide a greater opportunity for elucidating the underlying reasons for the wide variations in behavior observed for polyethylenes and semicrystalline ethylene copolymers.

We wish to express our appreciation to Cornelia Key for her analysis of branching content, to Richard Bloss for the computer programming, and to Dr. R. Ranganath for his helpful discussions during the development of the new TREF technique.

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# FLEXOMER POLYOLEFINS, A UNIQUE CLASS OF ETHYLENE COPOLYMERS FOR LOW TEMPERATURE FILM APPLICATIONS

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## INTRODUCTION

Very low density polyethylenes (VLDPEs) are produced by the Union Carbide Chemicals and Plastics Company Inc. gas - phase process (1). The primary advantage of this technology is the ability to generate a wide variety of polymers which exhibit an exceptional broad range of densities, molecular weights, and molecular weight distributions. These very low density polyolefins may be classified into two distinct families: narrow molecular weight distribution (NMWD) and broad molecular weight distribution (BMWD) products. This study will provide the basic rationale for selecting a class of VLDPE with emphasis on specific end-uses requiring low temperature properties. Depending upon the type of material to be packaged, type of packaging machines used and existent equipment for film extrusion, each family of VLDPEs may provide specific advantages for a film producer such as superior mechanical properties of the film at low temperature, good heat sealability and easy processability.

This paper will discuss the properties of both narrow and broad molecular weight distribution VLDPEs while comparing them with those of two products commonly used for low temperature applications: a typical EVA and a classical LLDPE copolymer. Furthermore, the effect of VLDPE addition to the low temperature properties of EVA film will also be shown.

## EXPERIMENTAL

The basic characteristics of the polymers studied are summarized in Table 1. All resins were extruded at 100%. Blends of VLDPEs and EVA were made to study the effect of the addition of NMWD and BMWD resins on the low temperature properties of EVA film.

Blown film was fabricated from virgin and dry blended resins on a 1.5" (3.8 cm) laboratory Sterling extruder equipped with a 3" (7.6 cm) dia. The extrusion was performed through a narrow die gap of 30 mil (0.76 mm), at 2.8:1 blow-up ratio to produce film of 1.75 mil gauge. All film samples produced contained antiblock agent and slip additive. In addition the NMWD resin contained a processing aid.

Brittleness temperatures were determined following the ASTM procedure using plaques (D746). Film properties were measured in accordance to ASTM procedures: tensile properties (D882), tear strength (D1922) and impact resistance (D1709 Method A). For the Dart Drop, Tensile Impact and Tear Resistance evaluations at -40°C, the temperature at the surface of the film was constantly monitored to ensure a maximum variation of 1°C. The method used for tensile impact is an internal test which determines the modulus of toughness and equates that number to its tensile impact strength. It is measured on a swinging pendulum apparatus. The value is obtained by determining the energy to break and dividing by the "effective volume" of the specimen. Puncture Resistance was measured using an internal test method where an Instron tensile compression tester with a 0.75" (1.9 cm) diameter head plunger traveling at 20 in/min (8.5 mm/sec) was employed. Hot tack and Heat seal strength evaluations of 1 in (2.54 cm) wide film samples, without a backing, were performed using the Packforsk Hot Tack Tester under the similar test conditions of a form-fill-seal packaging machine. Tester parameters were held at 43.5 psi (0.3 N/mm$^2$) bar pressure, 0.5 second dwell time, and 0.2 second delay time. To measure the hot tack force, the seals were peeled at a speed of 354 in/min (150 mm/sec). To measure the heat seal strength, sealed samples were aged 24-36 hours before determining the peel strength on an Instron apparatus.

The dynamic mechanical properties at low temperatures of the polymers were studied using a Rheometrics Solid Analyzer (RSA-II) with a film fixture. Film samples of five mil gauge were fixed in a holder in the MD tension mode, and submitted to a constant frequency of 1 Hz and a dynamic strain of 0.5 %. Temperatures were increased 3°C per minute from - 60°C to 20°C while samples were maintained at each temperature for 5 min.

## DISCUSSION

The purpose of this study was to evaluate the classes of VLDPE resins for performance parameters of importance for low temperature film applications such as ice bags and frozen food packaging. Major requirements for selecting a product for these applications are: superior room temperature and low temperature properties; excellent sealing characteristics including hot tack and heat seal strength; and relative ease of processability (extrudability). A representative member of each class of VLDPE resin was evaluated, relative to commonly used products, for these parameters in our R&D laboratories and/or in end use field tests.

### Film properties of VLDPEs vs. EVA and LLDPE copolymers

### Film Physical Properties

Films for end use low temperature applications such as ice bags and frozen food packaging require high dart drop impact resistance, tensile impact and tear resistance at temperatures as low as -40°C. The lower crystallinity of VLDPE resins results in energy absorbing characteristics which yield outstanding low temperature mechanical properties. Also, their lower brittleness temperature provides superior low temperature performance when compared to ethylene-vinyl acetate (EVA) and LLDPE copolymers (Table 2). The film properties of the resins studied are shown at room temperature (RT°C) and at -40°C in Table 3. For ease of comparison, the low temperature film properties of major interest, i.e., dart drop, tensile impact, and tear resistance, are graphically represented for VLDPEs, EVA and LLDPE in Figures 1, 2, and 3.

The impact resistance of VLDPEs is significantly higher at RT°C and -40°C than that of EVA and LLDPE (Table 3, Figure 1), e.g., at RT°C NMWD VLDPE yields 500 g/mil (190 KN/m) versus approximately 200 g/mil (80 KN/m) for EVA and LLDPE.

At -40°C, NMWD VLDPE exhibits an impact resistance value of 300 g/mil (115 KN/m) versus approximately150 g/mil (45 KN/m) for EVA and LLDPE.

VLDPEs yield, at room temperature and -40°C, tensile impact properties superior to those of EVA (Table 3, Figure 2). For example, at -40°C VLDPEs exhibit approximately 1000 ft.lb/cu.in (83 MJ/m$^3$) versus 300 ft.lb/in$^3$ (25MJ/m$^3$) for EVA. When VLDPEs are compared to LLDPE, the tensile impact properties are similar at room temperature; but due to their lower crystallinity, VLDPEs display higher values at lower temperatures.

At room temperature, the MD (machine direction) tear resistance of both NMWD and BMWD VLDPE is superior to EVA (Table 3, Figure 3). While this difference is extreme for the NMWD product, i.e., 400 g/mil (155 KN/m) versus 70 g/mil (27 KN/m), a smaller difference of 110 g/mil is observed between the BMWD product and EVA. Generally the BMWD VLDPEs exhibits a lower tear than the NMWD polymers. At -40°C a comparable ratio is maintained between the VLDPEs and EVA. The NMWD VLDPE tear resistance (MD) at RT°C is equivalent to that of LLDPE; however at low temperature, the NMWD VLDPE maintains a high value for tear (300 g/mil) while the LLDPE tear resistance decreases from 400 g/mil to 130 g/mil (50 KN/m).

As expected, the dart drop, tensile impact and MD tear resistance values of the resins studied decrease with decreasing temperature. As the polymer cools to -40°C, the free volume diminishes resulting in a decrease of segmental motion (4). The extent of molecular mobility is lowered, thus the energy absorption at lower temperature diminishes. An interesting trend is observed with the TD tear resistance which increases at -40°C vs. RT°C for lower density polymers, i.e., VLDPEs and EVA. This is probably due to the strong effect of orientation during film extrusion which creates an anisotropic crystalline structure. The TD tear resistance measurements suggest that for the lower crystallinity polymers, the energy absorption characteristics are higher in the TD direction where more amorphous regions are found due to a lower degree of orientation in this direction during film fabrication.

The higher average molecular weight (13 flow index) of BMWD VLDPE offers improved tensile strength when compared to the 0.5 melt index NMWD VLDPE and EVA and 1 melt index LLDPE. Also the lower density of NMWD VLDPE (0.905 g/cc) versus the BMWD product (0.912 g/cc) results in a low secant modulus, providing a film of similar softness to the EVA copolymer. The puncture resistance shows no particular trend. This is not unexpected since this property is very sensitive to the film surface (antiblock and slip levels varied for each resin tested).

### Film Dynamic Mechanical Analysis

Dynamic mechanical testing over a selected temperature range has been used as a criterion to predict the toughness and impact strength of polymers. In general, polyethylene displays three transitions in order of decreasing temperature: $\alpha$ (20°C to 80°C), $\beta$ (-30°C to 20°C) and $\gamma$ (-120°C to - 90°C) relaxations (2). For the purpose of this study, the domain of $\beta$ relaxation is of interest. The $\beta$ transition has been ascribed to the onset of diffusional motions of the polymer backbone in amorphous regions (glass transition).

The storage modulus and and the loss tangent as a function of temperature for NMWD and BMWD VLDPE versus EVA and LLDPE are shown in Figures 4 and 5. Assigning specific temperatures for the ß transition is difficult due to the very weak relaxations observed. The slopes of the storage modulus (Figure 4) and loss tangent (Figure 5) curves indicate that both types of VLDPE have very similar temperature - modulus relationships. The toughness of VLDPEs at very low temperatures, i.e., -40°C to -10°C, is almost equivalent to that of EVA as indicated by the storage modulus curves. In the same temperature range, LLDPE is more brittle than the other products tested. At temperatures higher than 0°C, the slope of the storage modulus curves versus temperature increases, indicating a decrease in stiffness in the following order: EVA is becoming the most flexible material succeeded by VLDPEs and LLDPE.

As reported by Wada and and Kasahara (3), the impact properties of polymers correlate directly with the integrated loss tangent in the temperature range of 0°K (-273°C) and 300°K (27°C). When comparing the integrated area under the loss tangent curves displayed in Figure 6, (in the temperature range of -40°C to -10°C), VLDPEs exhibit higher low temperature toughness than LLDPE. In the -30°C to -50°C domain, the VLDPEs show the highest toughness. By this test EVA copolymer appear to yield superior impact strength at temperatures higher than -30°C.

The dynamic mechanical analysis results are consistent with standard film mechanical properties at -40°C.

### Effect of the addition of VLDPE to EVA copolymer

The film properties of blends of NMWD VLDPE with EVA are displayed in Table 4. The effects of the addition of VLDPE to EVA are exemplified in Figures 7 - 12. The NMWD VLDPE, at ratios of 25 and 50%, provides improved impact and tear resistance, tensile impact and tensile strength while not affecting the secant modulus. Previous results showed that the film properties of a blend of VLDPE and EVA are better when a narrow die gap is used (5). The improvement in these properties is more pronounced at RT°C than at low temperature due to the decrease in polymer molecular motion for both EVA and VLDPE resin at -40°C. By adding 50% of NMWD VLDPE to EVA copolymer, the EVA dart drop and MD tensile impact increase by approximately 80% at RT°C. These properties increase by 15% and 35% respectively, at -40°C. The EVA film MD tear resistance significantly benefits from the addition of 50% VLDPE; an augmentation of 250%, i.e., from 40 to 140 g/mil, can be observed at room temperature. At low temperature, the tear is almost double for the blend compared to virgin EVA resin, i.e., 70 vs 130 g/mil. In addition, the EVA tensile strength increases by 25% when NMWD VLDPE is blended at 50% level with EVA. Similar results were obtained with BMWD VLDPE (unpublished results).

A blend of VLDPE resin and EVA provides considerably improved physical properties at both room and low temperature as compared to virgin EVA. At the same time, EVA enhances the processability of NMWD VLDPE and improves film clarity. Industrial experience has shown that a blend of NMWD VLDPE and EVA (50:50) yields similar production rates to a BMWD VLDPE.

### Heat Sealability of VLDPE vs. EVA and LLDPE. Blends of VLDPE with EVA

High performance heat sealability is a major requirement of the flexible packaging industry. The most significant aspect of heat sealability is heat seal strength: the strength of the seal after cooling to ambient temperature. A closely related property is hot tack strength: the strength of the seal while still in the hot or molten state, immediately after the sealing operation. Hot tack is particularly important in Form-Fill-Seal applications. In addition, the ability of a film to seal through contaminants is also critical to obtain product integrity and reduced leakage rates. Figures 13 and 14 display the hot tack and heat seal strength of the VLDPEs vs. EVA and LLDPE copolymers.

The BMWD VLDPE yields outstanding heat seal strength, i.e., over 20 N/in (4.5 lbs/in), over a very broad sealing temperature range (approximately 50°C). This is due to the high molecular weight of the resin and its broad molecular weight distribution. The BMWD VLDPE shows superior heat seal strength over a significantly broader temperature range than products currently used in many flexible packaging applications: EVA and LLDPE. Narrow molecular distribution VLDPE follows the same heat seal strength versus temperature curve as the BMWD counterpart in the range of 100°C - 120°C; however, its sealing range is narrower. EVA exhibits very high seal strength at low temperature, i.e., 20 N/in at 100°C. The amorphous region created by the presence of vinyl acetate comonomer, together with the high molecular weight of this polymer provide a good balance of high seal strength and low seal initiation temperature. However under the test conditions, the sealing range of this EVA is only about 10°C. As expected, the typical LLDPE of 1 MI provides lower seal strength and narrower temperature range when compared with the other resins studied.

The BMWD VLDPE offers the highest ultimate hot tack strength (2.5 N/in) among the polymers tested due to its high molecular weight which confers the highest melt strength. EVA yields acceptable hot tack at the lowest temperatures (95 - 100°C); however the operating window of 5 to 10°C is extremely narrow. The NMWD VLDPE exhibits good hot tack between 100°C and 115°C. There is a difference of 10°C between the hot tack seal initiation temperature of the BMWD VLDPE versus its counterpart (110°C versus 100°C, respectively). The higher molecular weight and density of the BMWD VLDPE (13 FI versus 0.5 MI) increase the temperature at which the polymer flows. Also, the lower density of the NMWD VLDPE (0.905 g/cc) provides acceptable hot tack values at lower temperatures. As expected the 1 MI LLDPE exhibits the lowest hot tack values among the films evaluated.

The addition of slip additives to adjust the film coefficient of friction is widely used in the packaging industry. Slip additives act similarly to grease on the film surface sometimes imparing film seal strength. The hot tack and heat seal strength of VLDPEs remain unchanged when up to 1500 ppm of organic slip agent is added to the resin.

The heat seal strength versus temperature of BMWD VLDPE and its blend with EVA at a 50:50 ratio is shown in Figure 15. The hot tack trend for the same blend is very similar. A BMWD VLDPE - EVA blend offers the best combination of low seal initiation temperature provided by EVA with high heat seal and hot tack strength over a very broad sealing range provided by VLDPE.

### Processability of NMWD and BMWD VLDPEs

As both NMWD VLDPE and LLDPE have similar dispersity indices, they yield common extrusion characteristics. As expected, both of these types of polymers are more difficult to process on equipment designed for high pressure linear low density polyethylene (HP-LDPE).

The BMWD VLDPE resins offer flexibility in selecting general purpose extruder screws or those specifically designed for LLDPE or HP-LDPE. They can also be extruded through narrow die gaps without the addition of a processing aid. Furthermore, these polymers exhibit blown film bubble stability comparable to HP-LDPE. On some commercial HP-LDPE blown film lines, BMWD VLDPE extrudes at equivalent rates to EVA. When the two representative members of each family of VLDPE products (BMWD and NMWD see Table 1) were compared for differences in extruder productivity on a 4 1/2 inch extruder equipped with a HP-LDPE screw design, a rate increase of 35% was observed with BMWD VLDPE.

The average molecular weight of BMWD VLDPE resins is higher than that of the NMWD polymers. As expected, higher molecular weight improves the melt strength while also increasing melt viscosity. Increasing the melt strength results in better bubble stability. This allows the film producer to increase production rates, however increasing melt viscosity (molecular weight) generally results in significant and sometimes unacceptable increase in die pressure and extruder torque.

The low molecular weight species in BMWD VLDPE compensate for the high average molecular weight and act as a "lubricant" for the high molecular weight polymer chains leading to shear thinning at processing conditions. Thus enhanced processability is obtained. In many cases, extruder torque levels, die pressures and horsepower consumption are lower than those encountered with NMWD polymers. Overall, BMWD VLDPE products are significantly easier to process than their NMWD counterparts as well as standard LLDPE.

### CONCLUSION

When selecting the preferred resin for production of low temperature films, a balance of low temperature properties, sealing characteristics and processability required for a specific application must be considered in combination with the specific extrusion and converting equipment being employed.

Each family of VLDPEs, BMWD and NMWD, may provide particular advantages for a film producer or converter. Using gas-phase technology, the molecular weight distribution can be varied to obtain a large variation in processing and property characteristics. The broad molecular weight distribution resins can provide blown film bubble stability comparable to HP-LDPE; the ability to extrude through narrow die gaps without processing aid; and flexibility in selecting different screw designs without losing productivity. These polymers yield superior heat sealing characteristics: high hot tack and heat seal strength over a very wide sealing range. This may enable film converters to broaden the operating window of the sealing equipment.
The narrow molecular weight distribution resins have the fabrication characteristics of the LLDPE, while exhibiting extremely high film toughness, along with excellent low temperature properties superior to BMWD polyolefins.

Both broad and narrow molecular weight product lines of VLDPEs offer outstanding low temperature properties which are superior to typical EVA and LLDPE copolymers used in low temperatures applications. However, the low temperature and room temperature film toughness and heat seal strength of the EVA copolymers are significantly enhanced by blending them with VLDPEs. Therefore, these polyolefins are ideal choices for improved performance in low temperature applications such as ice bags and frozen food packaging.

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Table 1. Characterization of Polymers

| Resin | Resin Type | Melt - Flow Index $I_{21} - I_{21}$ | MFR $I_{21}/I_{2.1}$ | Density (g/cc) | Wt. Average MW | Dispersity Index (Mw/Mn) | Comonomer |
|---|---|---|---|---|---|---|---|
| RTS - 9069* | VLDPE BMWD | 13 FI | 75 | 0.912 | 310,000 | 12 | HAO** |
| ETS - 9066* | VLDPE NMWD | 0.5 MI | 31 | 0.905 | 140,000 | 4 | HAO** |
| HS - 7028 | LLDPE | 1.0 MI | 28 | 0.918 | 115,000 | 4 | HAO** |
| EVA | HP - LDPE | 0.5 MI | 38 | 0.931 | 210,000 | 8 | VA (7.5%) |

* Commercial FLEXOMER® Polyolefins
** High alpha olefins

Table 2. Brittleness Temperature for VLDPE vs. EVA and LLDPE

| Resin Type | Brittleness Temperature % Failure / Temperature (°C) |
|---|---|
| VLDPE BMWD | 0 / -100 |
| VLDPE NMWD | 0 / -100 |
| EVA | 60 / -100 |
| LLDPE | 10 / -100 |

Figure 1. Film Dart Drop Impact Resistance of VLDPE vs. EVA and LLDPE

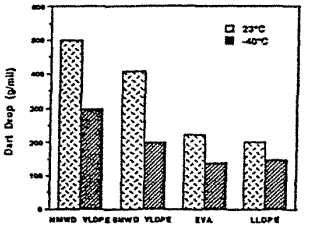



Figure 2. Film Tensile Impact of VLDPE vs. EVA and LLDPE

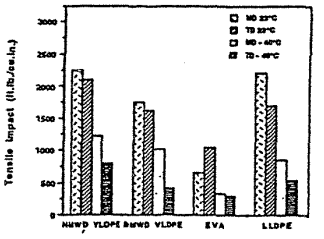



Figure 3. Film Tear Resistance of VLDPE vs.EVA and LLDPE

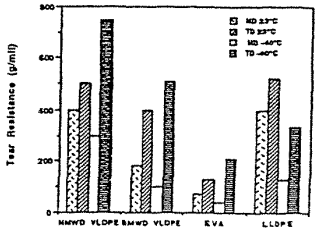



Table 3. Film mechanical properties of VLDPEs vs. EVA and LLDPE

| PROPERTY | Temp. (°C) | Orientation† | NMWD VLDPE | BMWD VLDPE | EVA | LLDPE |
|---|---|---|---|---|---|---|
| IMPACT RESISTANCE (g/mil) | 23°C | N/A | 500 | 410 | 225 | 200 |
| | -40°C | N/A | 300 | 250 | 140 | 150 |
| TENSILE IMPACT (ft. lb. /cu.in) | 23°C | MD | 2240 | 1750 | 650 | 2210 |
| | | TD | 2100 | 1820 | 1050 | 1700 |
| | -40°C | MD | 1230 | 1020 | 320 | 860 |
| | | TD | 800 | 400 | 300 | 540 |
| TEAR RESISTANCE (g/mil) | 23°C | MD | 400 | 180 | 70 | 400 |
| | | TD | 460 | 400 | 130 | 520 |
| | -40°C | MD | 300 | 100 | 40 | 130 |
| | | TD | 800 | 510 | 210 | 335 |
| TENSILE STRENGTH (PSI) | 23°C | MD | 5700 | 6000 | 3620 | 5500 |
| | | TD | 5050 | 4720 | 3130 | 5340 |
| YIELD (PSI) | 23°C | MD | 1150 | - | - | 1500 |
| | | TD | 1150 | 1300 | - | 1500 |
| ELONGATION (%) | 23°C | MD | 780 | 620 | 510 | 710 |
| | | TD | 830 | 780 | 660 | 800 |
| SECANT MODULUS (PSI) | 23°C | MD | 18500 | 22400 | 18000 | 29600 |
| | | TD | 19200 | 26100 | 16600 | 34400 |
| PUNCTURE RESIST*. (in-lb/mil) | 23°C | N/A | 11 | 11 | 4 | 10 |

† N/A = Not Applicable; MD = Machine Direction; TD = Transverse Direction
* Film additive package is different for each resin
Conversions: 0.386(g/mil)=KN; 0.0827(ft.lb./in$^3$)=MJ/m$^3$; 0.00689(PSI)=MPa; 4.48(in.lb/mil)=J/mm; 25.4(mil)=µm

Table 4. Film mechanical properties of blends of EVA with VLDPE

| PROPERTY | Temp. (°C) | Orientation† | Blends of NMWD VLDPE with EVA Blend Ratio (wt:wt) 0:100 | 25:75 | 50:50 | 75:25 |
|---|---|---|---|---|---|---|
| IMPACT RESISTANCE (g/mil) | 23°C | N/A | 225 | 340 | 400 | 460 |
| | -40°C | N/A | 140 | 150 | 160 | 220 |
| TENSILE IMPACT (ft.lb. /cu.in) | 23°C | MD | 850 | 700 | 1050 | 1550 |
| | | TD | 1050 | 1200 | 1400 | 1650 |
| | -40°C | MD | 320 | 400 | 430 | 740 |
| | | TD | 300 | 300 | 360 | 650 |
| TEAR RESISTANCE (g/mil) | 23°C | MD | 70 | 120 | 140 | 250 |
| | | TD | 130 | 230 | 300 | 420 |
| | -40°C | MD | 40 | 90 | 130 | 220 |
| | | TD | 210 | 530 | 700 | 800 |
| TENSILE STRENGTH (PSI) | 23°C | MD | 3620 | 3770 | 4500 | 5000 |
| | | TD | 3130 | 3600 | 4100 | 4550 |
| YIELD (PSI) | 23°C | MD | - | - | - | - |
| | | TD | - | 1150 | 1150 | 1150 |
| ELONGATION (%) | 23°C | MD | 510 | 550 | 670 | 749 |
| | | TD | 660 | 720 | 780 | 820 |
| SECANT MODULUS (PSI) | 23°C | MD | 16000 | 17000 | 17300 | 17850 |
| | | TD | 16600 | 18800 | 19250 | 19150 |
| PUNCTURE RESIST* (in-lb/mil) | 23°C | N/A | 4 | 4 | 6 | 6 |

† N/A = Not Applicable; MD = Machine Direction; TD = Transverse Direction
* Film additive package is different for each resin
Conversions: 0.386(g/mil)=KN; 0.0827(ft.lb./in$^3$)=MJ/m$^3$; 0.00689(PSI)=MPa; 4.48(in.lb/mil)=J/mm; 25.4(mil)=µm

Figure 4. Dynamic Storage Modulus as a function of temperature for VLDPE vs. EVA and LLDPE

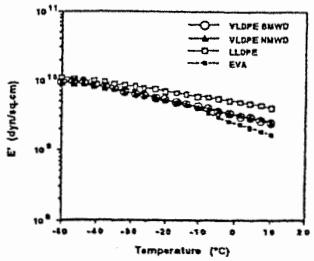



Figure 5. Loss Tangent as a function of temperature for VLDPE vs. EVA and LLDPE

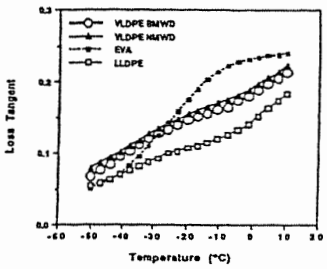



Figure 6. Comparison of integrated areas under the Loss Tangent curves for VLDPE vs. EVA and LLDPE

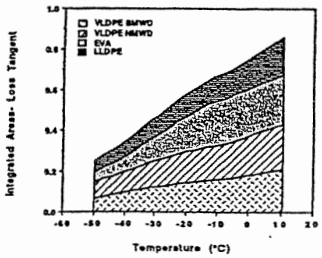



Figure 7. Effect of NMWD VLDPE addition on EVA film Impact Resistance

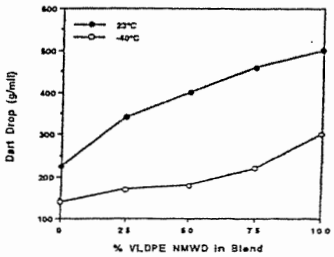



Figure 8. Effect of NMWD VLDPE addition on EVA film MD Tear Resistance

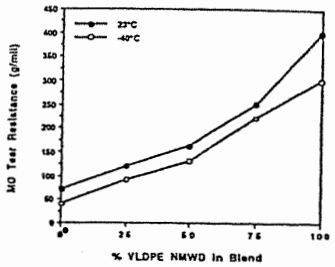



Figure 9. Effect of NMWD VLDPE addition on EVA film TD Tear Resistance

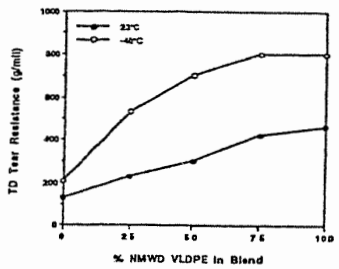



Figure 10. Effect of NMWD VLDPE addition on EVA film MD Tensile Impact

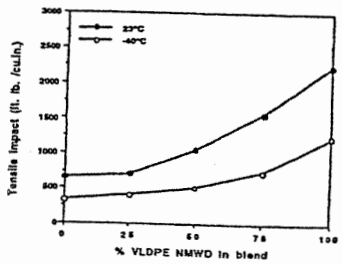



Figure 11. Effect of NMWD VLDPE addition on EVA film TD Tensile Impact

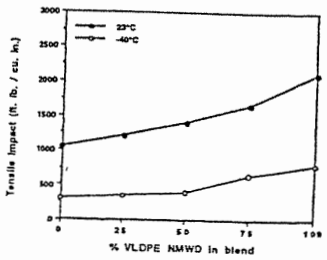


Figure 12. Effect of NMWD VLDPE addition on EVA film MD Tensile Strength

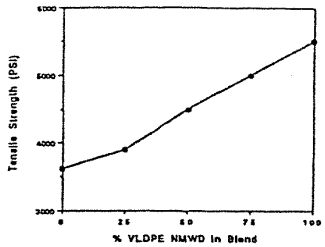



Figure 13. Film Hot Tack Strength of VLDPE vs. EVA and LLDPE

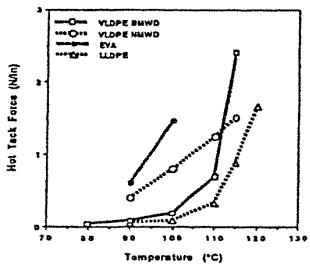



Figure 14. Film Heat Seal Strength of VLDPE vs. EVA and LLDPE

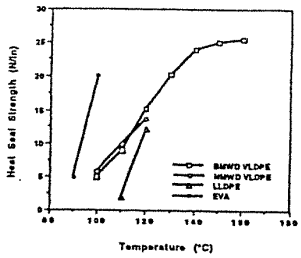
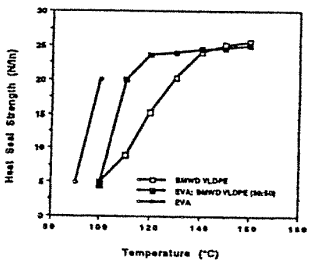



Figure 15. Effect of VLDPE on EVA film Heat Seal Strength



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# Improved Processing and Performance Balance of Polyethylene Resins Using Metallocene Catalyst Technology

by

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## INTRODUCTION

The commercialization of metallocene-based polyethylene resins has increased rapidly in the past few years and should continue to grow as the technology matures. One of the major benefits of metallocene resins is the potential for improving property performance over conventional polyethylene resins in extrusion applications. At the same time, however, metallocene resins have poor processing characteristics as compared to conventional polyethylene. The use of mixed catalyst systems enables the tailoring of resins to meet specific processing and property requirements. This paper will focus on the blown film extrusion of linear low density polyethylene (LLDPE) resins produced using metallocene/Ziegler dual-site-catalyst systems. The key factors in film extrusion and final film properties will be discussed.

## EQUIPMENT CONSIDERATIONS

In order to develop resins which perform well in any application, it is important to understand the process in which the resin will be used. Blown film extrusion of LLDPE requires specific equipment to successfully produce a high quality film.

### Extruder

The extruder melts, homogenizes, and feeds the polymer to the die. Many screw designs have been tried over the years for optimizing LLDPE extrusion. The most common design is a barrier screw, which contains a double-flighted section in the transitional region of the screw. These dual flights separate the molten from the solid polymer streams, thereby maximizing melting efficiency (see Figure 1). The result is a homogeneous melt, with high output and controlled temperatures. One indication of the processability of a resin, and an important factor for converters, is the extruder pressure. The power draw of the extruder, or drive amps, can also be used to compare the ease of extrusion of various resins.

Blown Film Die

The role of the die is to form the molten polymer approaching its final shape, while maintaining both a constant temperature and pressure for uniform gauge distribution. A low-pressure spiral mandrel die with streamlined channels is generally used for LLDPE to reduce shear and minimize degradation of the polymer (see Figure 2). The tendency of LLDPE to melt fracture necessitates the use of a wide die gap (>0.060") compared to low density polyethylene (LDPE) extrusion. A processing aid or heated die lips can be used to reduce melt fracture at narrow die gaps. Using a narrow die gap allows the drawdown ratio to be reduced, leading to more balanced film orientation and improved properties. A narrow die gap, however, may yield reduced optical properties, and extruder pressures and temperatures will increase.

Air Ring

High air velocity can cause bubble instability because of LLDPE's lack of strain-hardening as it is drawn from the die[1]. To alleviate this problem, it is important to cool the film as quickly as possible to increase its melt strength. A dual-lip air ring is generally used for LLDPE films. This type of air ring enables better control of the LLDPE bubble due to the early introduction of air to the polymer as it exits the die. Modern dual-lip air rings permit excellent distribution of air at all volumes, thus improving gauge distribution around the bubble. One important variable in determining film properties is the blow-up ratio (BUR), which is the bubble diameter divided by the die diameter. A high blow-up ratio results in more balanced orientation of the polymer chains, and thus properties are more balanced in both machine and transverse directions. LLDPE is typically extruded at between 2:1 and 3:1 blow-up ratios.

Processability

When extruding blown film, a few major factors are important to determine the processability of a resin. Measurements which can be easily monitored or calculated include power draw, extruder pressure, melt temperature, and output. Two important factors which are more subjective, and made by visual observations, are bubble stability and melt fracture. Bubble stability will affect the gauge distribution both

around the bubble and in the machine direction due to pulsing or back and forth movement of the bubble. Melt fracture, or sharkskin, is a problem in LLDPE film extrusion, especially when using narrow die gaps. This rough or wavy film surface results from a slip-stick behavior of the polymer against the die wall at a relatively low critical shear stress. Wider die gaps or heated die lips can be used to lower the shear stress and reduce melt fracture. The use of a processing aid reduces melt fracture by coating the surface of the die, thus eliminating the slip-stick behavior of the polymer against the metal surface. These six processing responses (power draw, extruder pressure, melt temperature, output, bubble stability, and melt fracture) can be used to compare the processing characteristics of a series of resins.

## POLYMER CHARACTERISTICS

Polymer properties play a key role in how resins will perform, both in the extrusion process itself and in the end product. The key factors include molecular weight, molecular weight distribution, rheology, density, and comonomer type.

### Molecular Weight/Molecular Weight Distribution

The average molecular weight of a polymer influences the flowability or processability of a resin. As molecular weight increases, the resin generally becomes harder to process (more amps needed, higher extruder pressures). However, higher molecular weight usually results in improved toughness and tensile strength, two desirable film properties. The correct balance is needed to meet the requirements for a specific application. Molecular weight, by itself, is not a reliable indicator of polymer performance. Molecular weight distribution (MWD) also influences both processing and properties of a LLDPE resin. A narrow MWD at a given molecular weight will yield improved properties (usually toughness and optics), while being harder to process than a broad MWD resin. Once again, a balance between processing and film performance must be made. A balance between film properties and processing can be obtained by combining a standard Ziegler catalyst with a metallocene catalyst using a "best of both worlds" or "BOB" approach. The Ziegler catalyst contributes good processing characteristics to the

polymer, while the metallocene catalyst, with its narrow MWD and comonomer distribution capability, contributes to improved film property performance. The relative ratios of each component can produce a wide range of polymers with various processing/property balances to suit specific needs.

Rheology

Rheological properties, for the most part, are influenced by the molecular weight and molecular weight distribution of a particular polymer. A broad MWD will result in more shear sensitivity (lower viscosity at high shear rates), which translates into improved processability. LLDPE resins tend to have poor shear sensitivity due to their relatively narrow MWD. Once again, the use of a mixed catalyst can be used to create a broader MWD for improved processability.

Density

The density of a LLDPE copolymer will influence most properties. The density can be controlled by adjusting comonomer content. Higher density provides a resin with better stiffness and lower impact and tear strength. Lower density gives better toughness and improved optics, but lower stiffness.

## DISCUSSION

For this study, a series of metallocene-based resins were characterized and blown into film. The resins included a straight metallocene LLDPE copolymer and a series of mixed metallocene/Ziegler catalyst butene LLDPE copolymers with various molecular weight distributions. Most of these resins were then extruded into blown film on typical LLDPE equipment to compare processing and properties with standard LLDPE resins. One mixed-catalyst resin, which exhibited enhanced processability, was also evaluated at a narrow die gap along with a high pressure LDPE, standard LLDPE, and a LDPE/LLDPE blend.

The equipment used for both studies included a 3.5-inch barrier-type screw with Maddock mixing section and an 8-inch monolayer, spiral mandrel die with dual-lip air ring. An extruder temperature profile of 380-440-420-400°F was used, with the exception of the LDPE resin, which was produced with a 360-400-

380-380°F profile. All films were produced at a thickness of 1.25 mils (0.00125 inches) and a blow-up ratio of 2.5:1. The die gaps for the narrow and wide die gap evaluations were 40 mils and 100 mils, respectively. The metallocene resin data are for comparison only. This resin was produced separately on a slightly different blown film line, so the values presented were estimated to account for these differences.

The metallocene-based resins were characterized for density, rheological properties (viscosity vs. shear rate), and gel permeation chromatography (GPC) for molecular weight data. This testing shows the major differences between the resins tested. Densities of the mixed catalyst resins were all 0.918 g/cm$^3$. The metallocene resin used in this study had a density of 0.901 g/cm$^3$.

It is apparent from GPC data that the metallocene resin has a more narrow molecular weight distribution (MWD) compared to the standard LLDPE resin (see Figures 3 and 4). The mixed catalyst resins can have either a narrow or broad MWD depending on the relative activities and the type of metallocene used. The location of the metallocene peak can also be varied, which can dictate processing and final product performance. Mixed catalyst resin 1 has a fairly narrow MWD, with the metallocene portion and Ziegler portion overlapping (Figure 5). Mixed catalyst resin 2 has a slightly broader distribution, with the metallocene portion toward the low molecular weight end (see Figure 6). This MWD would be expected to create a resin with excellent processability and performance similar to a conventional LLDPE resin.

Processing Results

Processing results from the blown film evaluations are given in Table 1 for the narrow die gap (0.040 inch) study, and in Table 2 for the standard LLDPE, wide die gap (0.100 inch) study. The data shows the processing improvements obtained by using mixed catalyst resin 2. Extruder pressure is dramatically reduced and melt fracture is virtually eliminated at the narrow die gap. For comparison, a blend of 70% conventional LLDPE with 30% high pressure LDPE was evaluated, which is one approach converters use to run LLDPE at a narrow die gap. The data shows that the mixed catalyst resin processes easier, at

slightly higher output, than this blend. The LDPE was produced at lower temperatures, which are typically used for this polymer. The other LLDPE resins could not be run at this temperature without excessive melt fracture.

**TABLE 1: Processing Data, Narrow Die Gap Study**

| Processing Parameter | Butene LLDPE | Hexene LLDPE | LDPE | Mixed Catalyst | LL/LDPE Blend |
|---|---|---|---|---|---|
| Barrel Pressure (psi) | 3800 | 3700 | 2100 | 2900 | 3800 |
| Drive Power (amps) | 68 | 66 | 41 | 54 | 63 |
| Melt Temperature (°F) | 418 | 418 | 386 | 415 | 420 |
| Output (lb/hr/rpm) | 7.16 | 6.16 | 5.96 | 6.74 | 6.40 |
| Melt Fracture | signif. | signif. | none | moderate | moderate |

**TABLE 2: Processing Data, Wide Die Gap Study**

| Processing Parameter | Butene LLDPE | Hexene LLDPE | Mixed Catalyst 1 | Mixed Catalyst 2 | Metallocene (estimated) |
|---|---|---|---|---|---|
| Barrel Pressure (psi) | 3600 | 3500 | 3500 | 2700 | 3800 |
| Drive Power (amps) | 82 | 82 | 81 | 63 | 83 |
| Melt Temperature (°F) | 421 | 422 | 418 | 422 | 420 |
| Output (lb/hr/rpm) | 7.9 | 7.2 | 7.8 | 7.8 | 7.5 |

NOTE: Melt fracture did not occur at the wide die gap for any of the resins.

Film Properties

Films were tested for dart drop impact strength, Elmendorf tear strength, tensile properties, secant modulus, haze, and gloss. The results are presented graphically in Figures 7 to 18. Data has been normalized for all properties, with the standard butene LLDPE resin having a value of 1.0 for each

property. The data are presented in two sets, first for the films produced at the narrow die gap, and second for the films produced at the wide die gap. The two sets will be discussed concurrently.

*Impact Strength*

Dart drop impact strength data are presented in Figures 7 and 8 for narrow and wide die gap trials, respectively. The narrow die gap results show the mixed catalyst resin 2 to have slightly lower impact strength than the butene LLDPE or LDPE resins, but similar impact strength compared to the LLDPE/LDPE blend. The wide die gap results show the same trends, but in this case the straight metallocene resin shows exceptional performance, with five times the impact strength of the standard LLDPE resin.

*Elmendorf Tear Strength*

Tear strength results are shown in Figures 9 and 10. At a narrow die gap (Figure 9), the mixed catalyst resin is seen to perform comparably to the butene LLDPE resin. This resin also shows a better balance of properties (machine vs. transverse direction) than the LDPE or LLDPE/LDPE blend. At a wide die gap (Figure 10), both mixed catalyst resins show similar tear strengths to the butene LLDPE and better than the straight metallocene resin.

*Optical Properties*

Haze and gloss properties are presented in Figures 11 and 12. At the narrow die gap the mixed catalyst resin has similar haze and gloss compared to the butene LLDPE resin. The LDPE and LLDPE/LDPE blends show improved optical properties of approximately 25-50% over the mixed catalyst resin. At the wide die gap, mixed catalyst resins show improved optical properties compared to the control. Mixed catalyst resin 1 shows a 25% improvement in both haze and gloss, comparable to the straight metallocene resin.

*Tensile Properties and Secant Modulus*

Tensile strength and elongation results showed no significant trends, with the mixed catalyst resins performing slightly worse than the standard LLDPE resin. The mixed catalyst resin 2 does outperform

LDPE and the LLDPE/LDPE blend in the narrow die gap study in terms of tensile properties (see Figures 13 and 15). At the wide die gap, mixed catalyst resin 2 showed less balanced tensile properties compared to the other resins evaluated (see Figures 14 and 16). Secant modulus results were similar to the tensile properties, as shown in Figures 17 and 18.

## CONCLUSIONS

The use of a "best of both worlds" approach, combining metallocene and Ziegler catalyst systems, can effectively tailor polymer properties to meet processing and/or property needs for LLDPE film resins. The relative activities of the two sites will determine molecular weight distribution, which influences both processing and performance. A resin can be obtained which has equivalent properties to standard LLDPE with improved processing, or with equivalent processing and improved properties. These mixed catalyst resins do not have the performance of straight metallocene resins, but bridge the gap between standard and metallocene LLDPE resins.

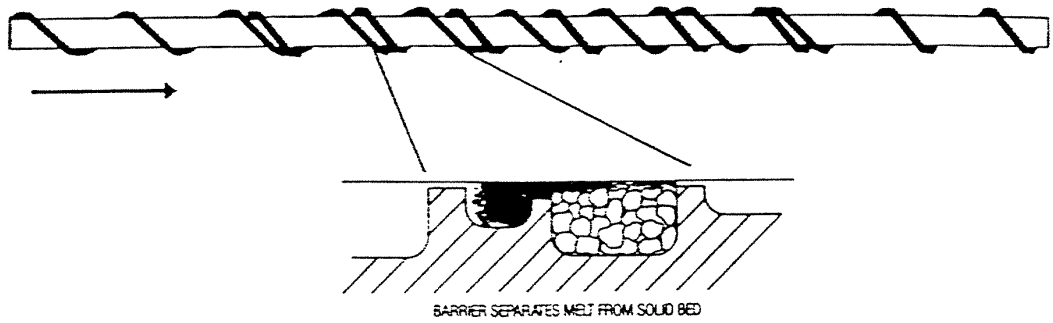
## REFERENCES

1. Bode, W.W., *TAPPI Journal*, 6:133, 1988.

2. Cooke, D.L., and Koyich, M., *Journal of Plastic Film & Sheeting*, 7:306, 1991.

3. Veazey, E.W., Barnette, T.L., and Pate, T.J., *Plastics Engineering*, 33, Sept. 1994.

## Figure 1
## Barrier Screw




## Figure 2
## Spiral Mandrel Die

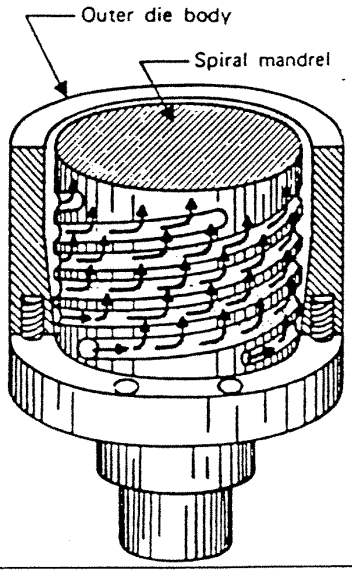


FIGURE 5
GPC DATA
Mixed Catalyst Resin #1

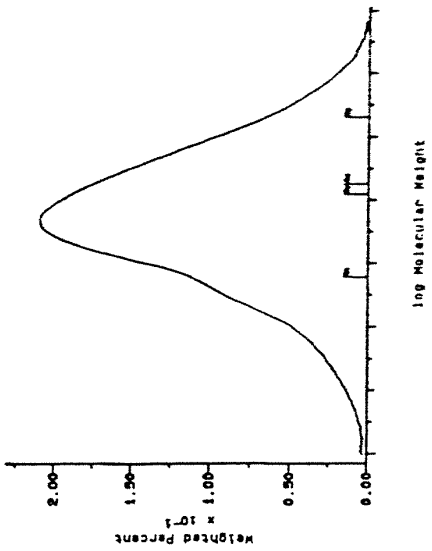



FIGURE 6
GPC DATA
Mixed Catalyst Resin #2

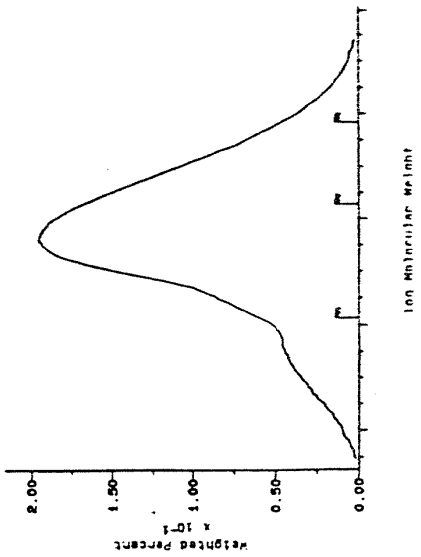



FIGURE 3
GPC DATA
Conventional LLDPE Resin

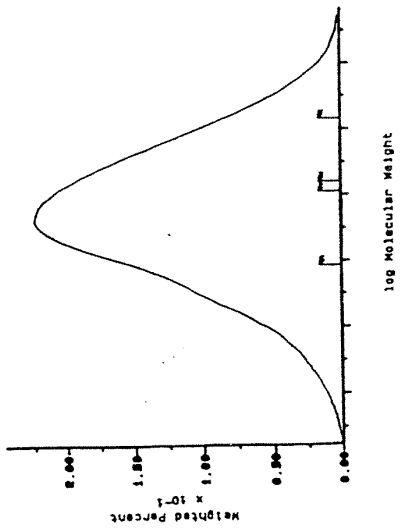



FIGURE 4
GPC DATA
Metallocene Resin

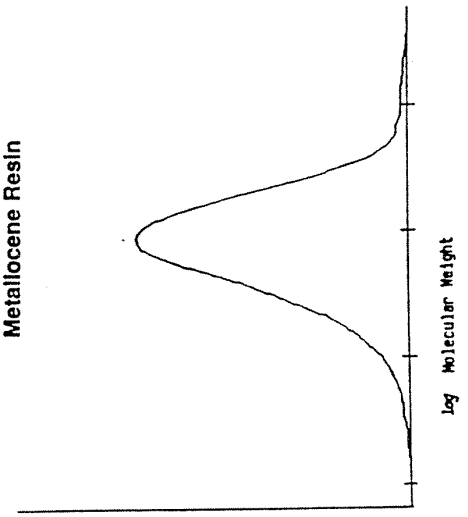


**FIGURE 7: Dart Drop**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

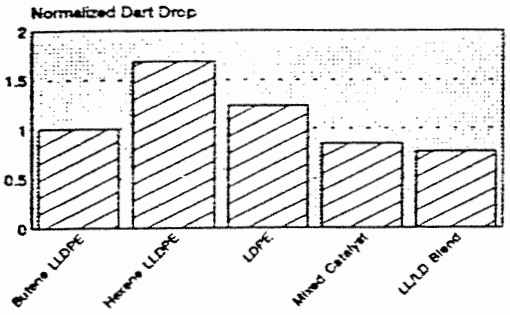



**FIGURE 8: Dart Drop**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap

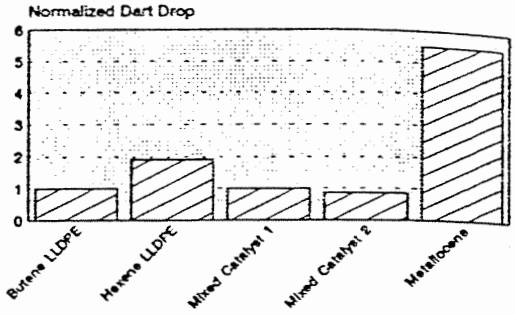



**FIGURE 9: Elmendorf Tear Strength**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

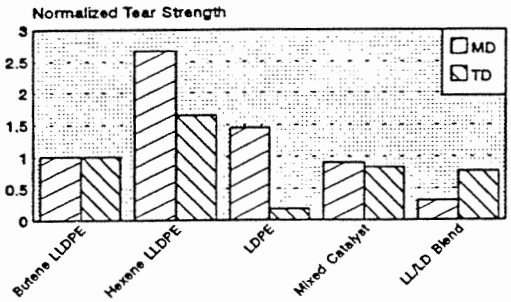



**FIGURE 10: Elmendorf Tear Strength**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap

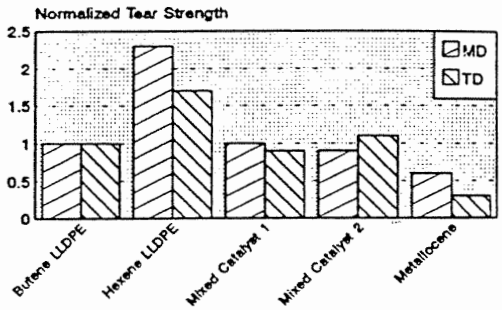



**FIGURE 11: Optical Properties**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

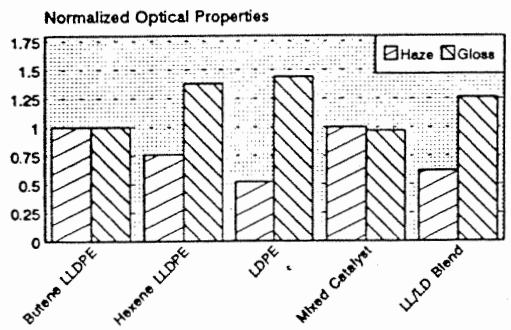



**FIGURE 12: Optical Properties**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap

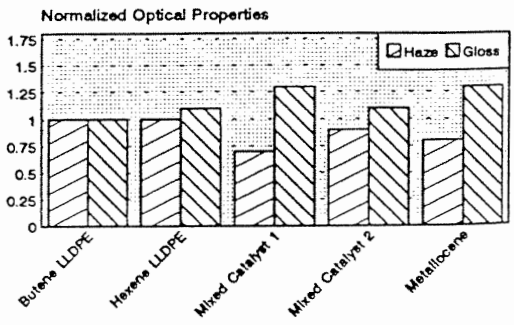


**FIGURE 13: Ultimate Tensile Strength**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

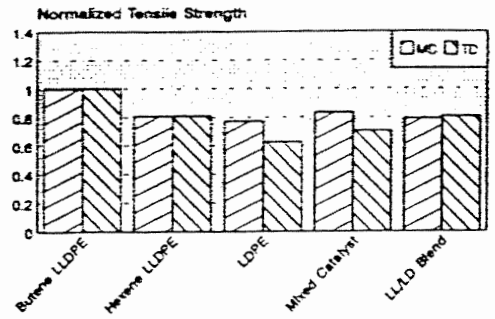



**FIGURE 14: Ultimate Tensile Strength**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap

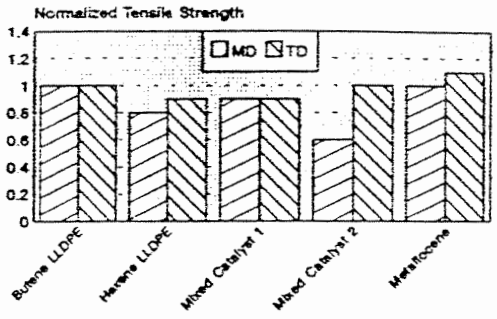



**FIGURE 15: Tensile Elongation at Break**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

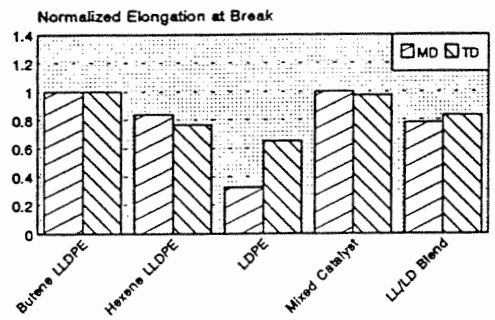



**FIGURE 16: Tensile Elongation at Break**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap

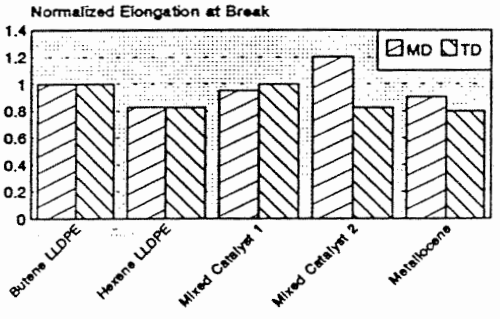



**FIGURE 17: Secant Modulus**
*Mixed Catalyst vs. Standard Polyolefins*
Narrow Die Gap

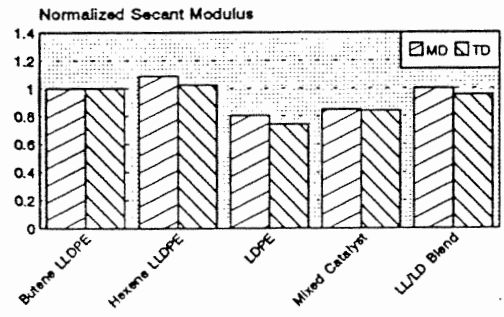
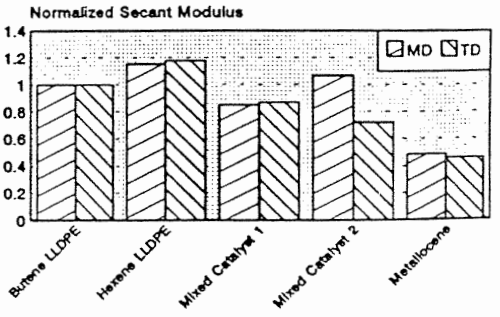



**FIGURE 18: Secant Modulus**
*Mixed Catalyst vs. Standard Polyolefins*
Wide Die Gap





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# Single-Site Catalysts Produce Tailor-made, Consistent Resins

**By David F. Simon**
Exxon Chemical Co.

**FOCUS:** The latest revolution in polyolefins is the development of metallocene, single-site catalysts used to produce consistently uniform polymer molecules. Single-site catalyst (SSC) technology has helped generate specialty ethylene polymers, which provide film and packaging producers a unique combination of properties. SSC polymer technology allows for the manufacture of tailored polymers and provides pinpoint control of molecular weight and composition distribution.

## Introduction

Not often does a new breakthrough in polyolefin polymerization technology come along. In 1933, high-pressure radical polymerization led to the first commercial production of low-density polyethylene (LDPE). In 1953, Ziegler-Natta catalysis opened the door for the first production of high-density polyethylene (HDPE) and polypropylene (PP). In the 1970s, linear polyethylenes using solution- and gas-phase technologies were introduced.

Now another breakthrough can be added to the list. Single-site catalyzed (SSC) polymers using metallocene technology are no longer a pipedream. EXXPOL[1] SSC technology has been used to produce EXACT[2] specialty ethylene polymers, commercial quantities of which were made available last year.

Using single-site catalysts, one can produce consistent, uniform distribution and incorporation of comonomer. No longer are many of the unwanted components that derive from multi-site catalyst products present. SSC products have narrowly defined molecular weight distributions and composition distributions.

Conventional catalysts have many sites of different activity levels located on their surface. The difference between the activity of each site introduces variations in the polymers they produce.

SSC catalysts also have many reactive sites, but each is identical to all the others, which eliminates variability in the polymerization reaction. This allows for a tailor-made structuring of the polymer and permits improved control of molecular weight distribution. It enables precise incorporation of comonomers and termonomers.

The benefit of SSC polymers is the absence of the high-density/high-molecular weight component, which yields an undesirably high melting point and poor optics in existing conventional multi-site linear catalyzed products. Likewise, SSC polymers eliminate the low-molecular weight, low-density component which imparts high extractables and unwanted blocking tendency in linear polymers.

The result of this technology is new and unique property combinations. For packaging film markets, this means performance improvements in the property areas of sealing, toughness/physical properties, optics and cleanliness.

## Properties

• **Sealing**—The absence of the high-molecular weight/high-density component results in lower peak melting points than are found with existing linear-based polymers, both in the traditional linear low range (>0.915d), and in lower density regions (typically 0.880-0.915d). Where Ziegler-Natta products exhibit some slight reduction in peak melting point as a function of increased comonomer/lower density, SSC products yield reduced peak melting points (Fig. 1).

This results in lower seal initiation temperatures (SITs) and wider sealing range windows for packagers. Coupled with this lower seal activation temperature comes the traditionally strong hot-tack and seal strength performance for linear polymers (Figs. 2-3). Peak hot-tack

[1] EXXPOL is a trademark of Exxon Chemical Co.

[2] EXACT is a trademark of Exxon Chemical Co.

levels for higher alpha olefins (HAOs) have been measured approaching the ionomer region, while the range for the high hot-tack has also been observed to be much broader than most other "high hot-tack" alternatives.

• **Toughness/Physical Properties**—The increased comonomer content yields high toughness performance. Measured levels of puncture resistance, dart drop (Fig. 4) and spencer impact are higher than alternatives. This includes other competitive Ziegler-Natta-based very low-density polyethylene (VLDPE) polymers.

• **Optics**—The absence of the high-molecular weight/high-density component results in improved haze and gloss. Haze levels (Fig. 5) have been measured for blown film well below traditional linear polymers, and in the range of the most preferred options, namely ethylene vinyl acetane (EVA) copolymers and ionomers.

• **Cleanliness**—The absence of the low molecular weight tail yields a polymer with extremely low extractables, resulting in a very broad FDA coverage window. This coverage is much broader than other competitiive Ziegler-Natta VLDPE polymers currently available (Fig. 6). In addition, it is anticipated that the low extractables will reduce odor and off-taste.

Another attribute of SSC polymers is thermal stability. When polymers with broad molecular weight distribution are melted, their low molecular weight components tend to be oily and smoky. With the controlled molecular weight distribution, the low molecular weight components can be eliminated, thus making the "smoking" effect unlikely to occur.

## Diverse Benefits

For resin producers, reduced additivation requirements of SSC polymers are expected to contribute to a more consolidated grade slate. More importantly, the consistency and predictability of the molecular chain lengths can be achieved in any reacting environment, including gas phase, high-pressure, slurry and solution polymerization technology. This will give resin producers more flexibility in selecting the optimum cost-efficient supply option.

For film extruders and converters, SSC products offer improved control of COF/blocking, reduced draw resonance in cast embossed films, improved cutability (vs. standard linear polymers), and, perhaps most important of all, sim-

Fig. 1

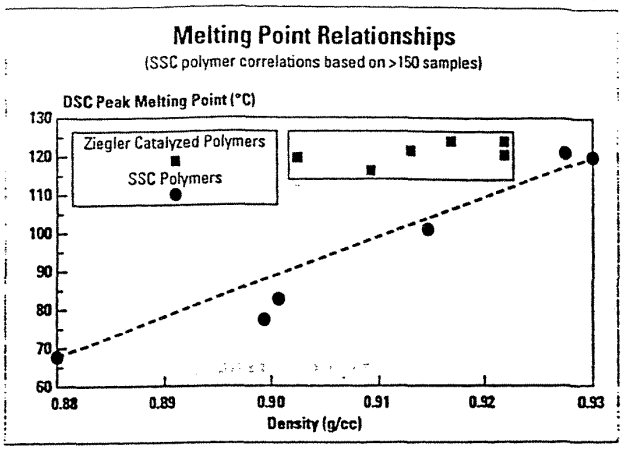



Fig. 2

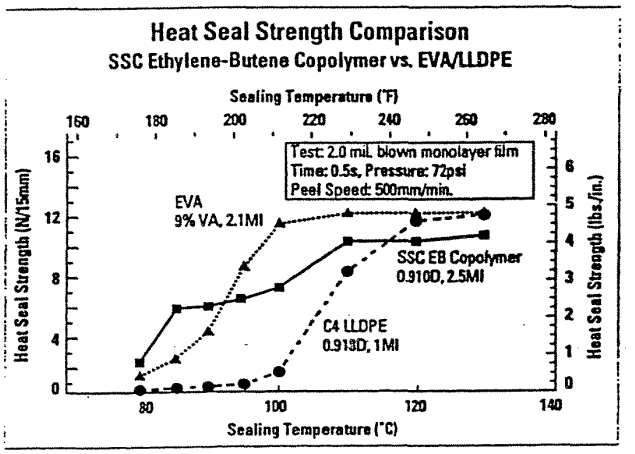
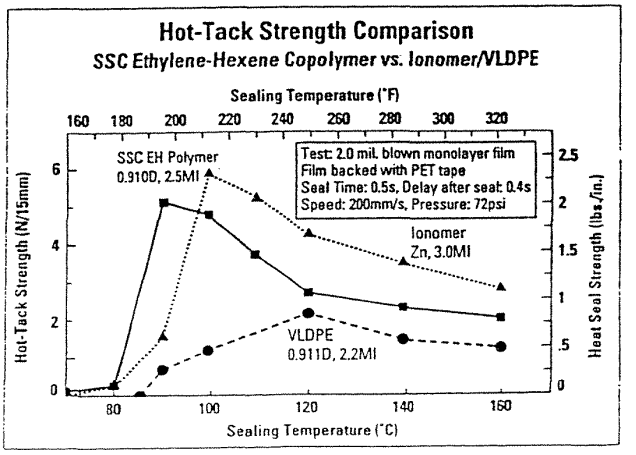
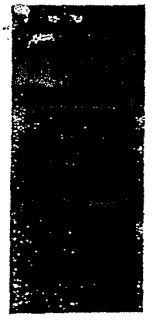



Fig. 3







plification in resin usage. No longer may two different resins, one for toughness and one for sealing, be needed. Using a reduced amount of resins/grades simplifies storage and inventorying requirements, and can eliminate the need for expensive tie/adhesive resins for coextruded structures. This must be tempered by slightly lower melt strength which will impact extruder output. For coextruded and blended options, this will likely not be an issue depending on the other components selected.

## Potential Applications

Due to enhanced sealing properties, SSC technology products will fit nicely into coextruded and/or blended heat seal layers. Anticipated applications include processed meat and poultry and seafood requiring low sealing temperatures, high hot-tack and good seal through contamination. Other applications include liquid pouch packaging requiring good sealing with toughness/abuse resistance (drop/impact), coex structures for cereal and cake mix liners and coffee pouch laminations.

In blends, SSC polymers will provide modification enhancing sealing and toughness properties. Applications such as heavy-duty sacks, consumer trash liners, barrier shrink bags for fresh meat, high energy shrink, etc., are ideal candidates. The lower density characteristic will offer opportunities to incorporate higher levels of post-consumer recycled HDPE to respond to environmental regulatory influences.

Fig. 4

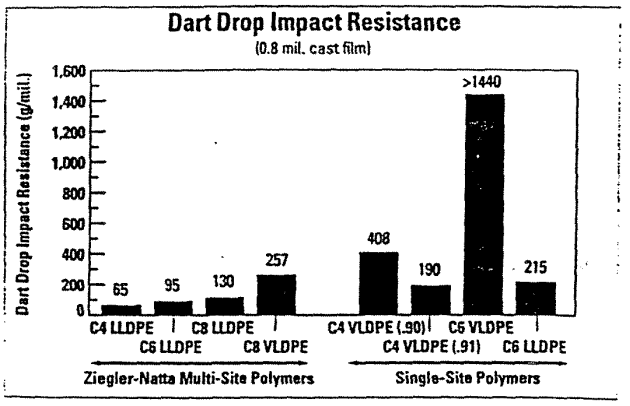
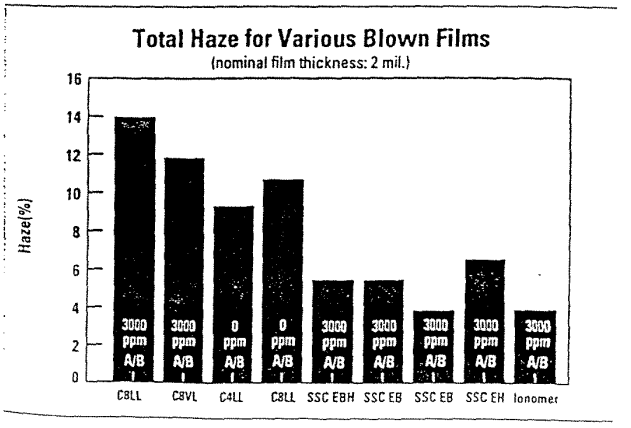



Fig. 5




## Establishing Trends

Single-site catalysts are high-activity catalysts, meaning they make many kilograms of polymer per kilogram of catalyst. A smaller amount of catalyst is needed to produce consistently uniform polymer, and less catalyst residue accumulates in the finished product. Additionally, these catalyst systems offer a wide array of metallocene cations and anionic coactivator systems, each giving unique polymers. Collectively, they offer broad polymer design capability.

Conventional Ziegler-Natta catalysts will remain in use due to their effectiveness and peoples' familiarity with them. However, the possibility exists that users of Ziegler-Natta catalysts will be able to use SSC catalysts to adjust and modify their systems for optimum efficiency, in a sort of "mixed" catalyst system.

In addition, it is likely a single reactor will use conventional catalysts and single-site catalysts at different times, and there could possibly be some intermixing of the two catalyst systems in the polymerization process.

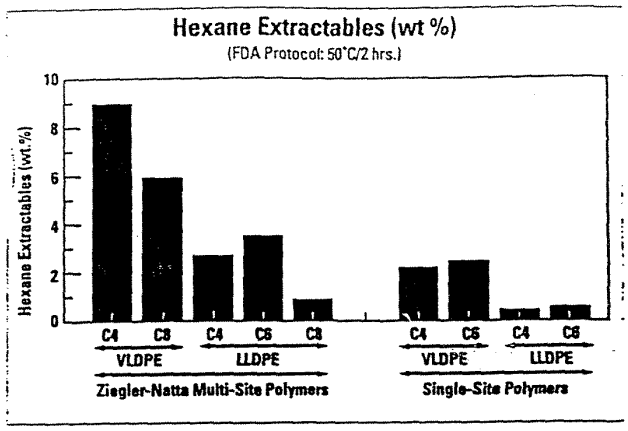
Conventional catalysts work well with smaller sized monomers, such as ethylene and propylene, but their reactivity decreases when working with larger monomers, such as the higher alpha olefins and dienes. SSC technology will make available the polymerization of monomers of varying sizes. This would enable large and small monomers to be combined into the same polymers, thereby capturing the attributes and characteristics of both. SSC technology will af-

fect polymerization processes in that, in tailoring polymers to meet customer needs, a single-site catalyst may be used in a series of reactors. During the polymerization process, another monomer or reagent could be added, or single reactors could use a mix of single-site catalysts.

Opportunities appear to be on the horizon for this next generation of ethylene polymers. These products are being designed to serve the needs of larger, more price-sensitive markets. The hope for these products is to introduce materials that process more like LDPE and have the toughness properties exceeding the best the industry can produce today. This next step will take full advantage of independent and controlled molecular weight distribution and composition distribution. ❑

For more information about this article, contact the author at (713) 870-6000.

Fig. 6






***About the Author:*** *David F. Simon is senior staff market development planner for Exxon Chemical Co.'s EXXPOL Venture. He has 15 years' experience in various polymer-related assignments since joining Exxon Chemical in 1978. Simon has a BS in Chemical Engineering from the University of Pennsylvania.*

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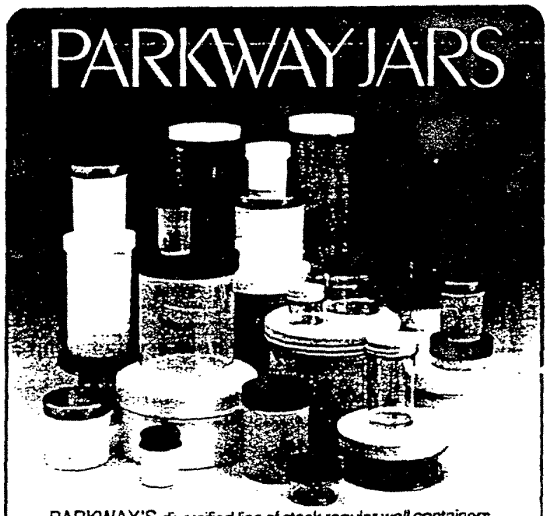

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# HIGH VALUE ADDED FILM USING AN OLEFIN BASED ELASTOMER

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# HIGHER-VALUE-ADDED FILMS USING AN OLEFIN BASED ELASTOMER


## Abstract

Multi-functional and high-performance materials are coming to be used in an increasing number of packaging materials in recent years. Especially in the field of food packaging, film having gas-barrier, moisture-barrier, easy-to-open and heat-resistant properties is begining to appear in the market. It is very difficult to realize such film with special functions by using single layer structure and generally, such film is materialized by using multi-layer structure, comprising several layers playing their respective different roles. As an example of such film in Japan, there is packaging materials for retort food which use polyamide in the barrier layer and an olefin-based polymer in the heat-sealing layer.

The items of the performance required of the olefin-based polymer used in such high-performance film include low-temperature heat sealability, low-temperature impact resistance, heat resistance, hot tack properties, transparency, easy-to-open properties, etc. It is difficult to satisfy those varied properties required of such olefin-based polymer by using one kind of polymer alone, and therefore, several polymers are usually blended to accomplish this objective.

Olefin-based elastomers (such as TAFMER) can be blended with olefin-based polymers to give various functions to film. Specifically, they can impart low -temperature heat sealability, impact resistance, easy-to-open properties, etc. to film to improve the added value of film.

The density of TAFMER ranges from 0.87 to 0.89 g/cm$^3$. Recently, LLDPE (VLDPE, ULDPE) which has the range of density as TAFMER entered the polyolefin market. The difference between TAFMER and such LLDPE are that TAFMER has a narrower molecular weight distribution and composition distribution than LLDPE. Therefor the blending effects of TAFMER to the film are different from that of LLDPE.

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# High-value-Added Film Using an Olefin-based Elastomer

## 1. Preface

Multi-functional and high-performance materials are coming to be used in an increasing number of packaging materials in recent years. Especially in the field of food packaging, film having gas-barrier, moisture-barrier, easy peel and heat-resistant properties is beginning to appear in the market. It is very difficult to realize such film with special functions by using single layer structure and generally, such film is materialized by using multi-layer structure, comprising several layers playing their respective different roles. As an example of such film in Japan, there are packaging materials for retortable pouch which use polyamide in the barrier layer and an olefin-based polymer in the heat sealing layer.

The items of the performance required of the olefin-based polymer used in such high-performance film include low-temperature heat sealability, low-temperature impact resistance, heat resistance, hot tack properties, transparency, easy-peel properties, etc. It is difficult to satisfy these varied properties required of such olefin-based polymer by using one kind of polymer alone, and therefore, several polyemrs are usually blended to accomplish this objective.

Mitsui Petrochemical Industries, Ltd. has developed an olefin-based elastomer as high-value-added olefin-based resin whose trade name is "TAFMER." The following are the details of the characteristics of this resin, the Japanese market situation and also of how TAFMER is being used for packaging materials.

## 2. Grades and Characteristics

### (1) Grades of TAFMER

There are Types A and P which are ethylene-alpha-olefin copolymers and Type XR which is a propylene-alpha-olifin copolymer. Fig. 1 shows the lineup of the types and grades of TAFMER. Types A and XR are low-crsytalline polymers, while Type P a non-cryatalline polymer. Although all these types are low- or non-crystalline polymers, they take the form of pellets as are the cases with other polyolefins. This is very important from the viewpoint of handling in that these types can be easily blended with other crystalline polyoflefins on an extruder when they are used as polyolefin modifiers. These 3 types are very transparent and odorless and moreover do not contain gel which can cause fish eyes.

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Fig. 2 shows the positions of TAFMER compared with other olefin-based resins. The horizontal and vertical axes of this figure indicate density and hardness respectively. Fig. 2 shows that Types A and P which are ethylene-alpha-olefin copolymers, are flexible and lower in density than HDPE, LDPE and LLDPE, and that Type XR which is a propylene-alpha-olefin copolymer, is flexible and has lower density than PP.

(2) Characteristics

Given in Table 1 are TAFMER grades and their chracteristics.

a. Mechanical properties

Fig. 3 indicates the tensile properties of TAFMER. Containing a samll amount of crystals, Types A and XR are a little higher in strength than non-crystalline Type P.

b. Chemical properties

TAFMER remains stable propertywise even when immersed in a diluted acid or alkali solution; however, it discolors in a solution of ammonium or concentrated sulfuric acid. While it has resistance to alcohols, ester, ketone, etc., it swells or dissolves in aliphatic and aromatic hydrocarbon solutions.

c. Heat deterioration properties

None of the aforesaid types has any unsaturated bond or functional group, so that they are excellent in heat deterioration properties. Lowering of molecular weight, cross-linking, or gellation does not occur during extrusion. These types can be used in the same extrusion method and under the same conditions as those intended for ordinary polyolefins.

3. Comparison with VLDPE

The following are comparisons of TAFMER with VLDPE and ULDPE placed on the market.

Types A and P are ethylene-alpha-olefin copolymers which are the same as VLDPE and ULDPE componentwise. If the density of VLDPE is defined as 0.92 - 0.90 g/cm$^3$ and ULDPE's as 0.90 g/cm$^3$ or lower, the aforesaid 2 types come under the category of ULDPE. Type A in particular is very close to ULDPE from the viewpoint of components. The physical

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properties of TAFMER, however, greatly differs from ordinary VLDPE and ULDPE due to difference in manufacturing process. Namely, compared with VLDPE and ULDPE, TAFMER is very narrow in molecular weight distribtion and also in compositional distribution.

Fig. 4 shows a conceptional figure relative to the relationship beteen Type A and VLDPE in density, crystallinity and rigidity. Despite the same density as that of VLDPE, Type A is low in crystallinity and more flexible than VLDPE.

Table 2 exhibits a comparison of Type A in physical properties with VLDPE and ULDPE commercially available. VLDPE(1) is as low as Type A in density, but is high in crystallinity, rigidity and molecular weight distribution.

Difference in compositional distribution among TAFMER, VLDPE and ULDPE can be shown in a DSC chart. Fig. 5 is a DSC chart of Type A, VLDPE and ULDPE. The compositional distribution of TAFMER is so narrow that it shows one endothermic peak in the neighborhood of 80°C. Meanwhile, several peaks are seen between 80° to 120°C in the cases of VLDPE and ULDPE.

Summerized in Table 3 is the superiority of TAFMER Type A to VLDPE and ULDPE which is attributable to its different physical properties; i.e., having low crystallinity, this type excels them in improvements in flexibility and low-temperature impact strength. Also, since Type A possesses a low melting point, films blended with this type can be heat sealed at lower temperatures. Further, Type A blended-film is superb in transparency and anti-blocking properties because it has narrow molecular weight distribution and compositional distribution.

4. Resin-modifying Effects of Olefin-based Elastomer

There are many physical properties demanded of films. Shown in Table 4 are the modifying effects obtained by blending various polyolefins with TAFMER which is olefin-based elastomer. The following are the details of such effects which are explained item by item here.

(1) Improvement in low-temperature heat-sealing properties

In general, films used for packaging are made into sacks into which goods are put or films for this purpose are used for wrapping goods in them. In both cases, they are partially heat sealed. The performance required of these films are heat-seal strength, low-temperature heat sealability, hot tack strength and sealability attained despite the existence of foreign matters. All of them are

important performance; however, low-temperature heat sealability is of importance particularly, because this feature has much to do with speeding up of a packaging line, which brings about improved productivity in the end.

A case taken up here is about the improvement of low-temperature heat sealability for which TAFMER is used. Fig. 6 gives the heat-sealing characteristics which are obtained by the use of Type XR employed for the seal layer for heat-sealable BOPP. Use of Type XR Grade 107L makes heat-seal threshold temperature 40°C lower than that of PP random copolymer while Type XR Grade 106L can lower such threshold temperature by 20°C compared with that of PP random copolymer. Also, Type XR is transparent and its surface is hard. Hence, it is possible to obtain heat sealable BOPP film which is surpassing in anti-scratch properties and transparency. The typical applications of Type XR-using heat-sealable BOPP film include wrapping film for tobacco packs and boxes of chocolates in which both sides of the film are heat sealed, and the wrapping of individual pieces of cookies and cakes in which one side of the film is heat sealed.

Fig. 7 shows the change in heat-sealing characteristics of CPP film blended with Type A or P. The heat seal temperature lowers according as the blending ratio of this resin modifier rises. CPP film to which this feature is applied, is 3-layer CPP film(PP + TAFMER/PP homopolymer/PP+TAFMER). Namely, PP homopolymer is used for the middle layer with which to give proper rigidity to the film, while Type A- or P-blended PP is used for two outer layers for the purpose of having low-temperature heat sealability. Among the applications of film having this three-layer structure is the packaging for bread and vegetables.

This improvement effect on heat-sealing characteristics can apply to LLDPE films. As seen in the case with CPP, the heat seal threshold temperature lowers according as the TAFMER blending ratio rises. This film is used for flexible packaging for dressing or the like in the structure of O-NY/Al foil/LLDPE + TAFMER A. In this case, blending of this resin modifier not only lowers the heat seal threshold temperature but brings about excellent heat sealability despite the existence of foreign matters.

2) Improvement in impact strength

The following is an instance of impact strength improved with TAFMER.

Fig. 8 shows an improvement effect on impact strength which is attained by blending PP random copolymer-based cast PP film with Type A. The impact strength is improved according as the Type A blending ratio rises. The

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improved impact strength remains intact at low temperatures. The average blending ratio of TAFMER used for improving this strength of the said film ranges from 10 to 15 wt%. Since the same effect can be obtained as to PP sheets, this manner can be applied to the impact strength improvement of cups, trays, etc. which are made of PP.

The packaging of retortable pouch is an example in which the impact strength of CPP film is improved by TAFMER blending. The film structure in this case is PET/Al foil/CPP or O-NY/CPP. Two or three layers of film having this structure are laminated in such a way that CPP layer can be arranged as an innermost layer. The food packaged with this packaging material is sterilized at 100° to 130°C and can be preserved at room temperature. With a view to, however, preventing the packages from breaking during low-temperature storage or transit, the impact strength of the CPP layer is enhanced by TAFMER blending.

(3) Improvement in flexibility

The following concerns the flexibility of TAFMER.

Fig. 9 shows the relationship between temperature and flexibility. TAFMER can maintain flexibility even at low temperatures. Young's moduls of polyolefins can be lowered by blending them with TAFMER. Cap liners and sealed containers are examples in which this effect is actually utilized. In this case, LDPE A is blended with Type A by 10 to 20 wt%.

(4) Endowment of easy peel

Shown in Fig. 10 is the control of heat-seal strength by means of the blending of PP and TAFMER. This control can be performed by varying the blending ratio of this modifier to PP. PP so modified in heat-seal strenth is in use for heat seal layers for the lids of PP cups intended for jellies and custard pudding. By controlling peel strength, the heat seal layers can be endowed with easy peel. Also, since PP is used as the base polymer for the heat seal layer, this layer can withstand the thermal treatment for sterilizing the food packaged therein.

(5) Improvement in interfacial bonding strength

The following is about the interfacial bonding strength of TAFMER in extrusion coating.

When LDPE is extrusion laminated onto CPP film, CPP film is coated with primer in most cases because of

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insufficient bonding strength bewteen PP and LDPE. This lamination process, however, can be implemented without primer coating by adopting either of the methods referred to below.

a. LDPE to be extrusion laminated is blended with TAFMER.

b. CPP film is made to be formed of multi-layers, TAFMER layer of which is arranged on the side of lamination.

Fig. 11 gives the change of bonding strength which is attaind by blending LDPE with this modifier. The said strength improves according as the blending ratio of TAFMER rises.

(6) Improvement in pinhole resistance

Given in Table 5 is the improvement in pinhole resistance of LLDPE which is achieved by TAFMER blending. Blending of TAFMER and LLDPE to be used for film production can greatly curb the occurrence of pinholes. This film is used for a bag in box.

Table 6 shows the typical structures of TAFMER-using packaging films now in use in Japan.

6. Blending Process and Improvement Effects on Compatibility

(1) TAFMER blending process

TAFMER is well compatible with polyolefins such as PE, PP, EVA, etc. When polyolefin film, etc. is extruded using TAFMER as a modifier, all we have to do is to mix polyolefin and this modifier in the form of pellets prior to having them go through an extruder. When they are mixed and extruded into pellets beforehand, better dispersion properties can be obtained.

(2) Compatibility improvement effects

TAFMER can be used as a compatibilizing agent. Mere blending of PP and PE sometimes lowers their mechanical strength or optical properties. When TAFMER is mixed with a mixture of PP and PE, it serves as a compatibilizing agent and improves mechanical strength and optical properties. In other words, when PE is dispersed in PP, the dispersed particle size of PE remains unchanged, and mixing of TAFMER with this mexture can make this size smaller. Photo 1 shows a microscopic picture of PP blended with LLDPE by 20 wt%. LLDPE particles whose size is 2 to 3 microns, are dispersed. On the other hand, Photo 2 indicates a case in which TAFMER A-4085 is added to LLDPE by 10 wt% and the dispersed particle size is made smaller to 0.5 to 1 micron.

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The following refers to the effects obtained with TAFMER used as a compatibilizing agent. Fig. 12 shows the change of the surface gloss of an injection-molded square sheets when TAFMER is added to PP already blended with LLDPE by 20 wt%. The gloss becomes better according as blending of this modifier increases. Fig. 13 shows the elongation obtained by blending 20-wt%-LLDPE-blended PP with TAFMER. A raise of this TAFMER blending ratio makes the elongation larger. These examples demonstrate that this modifier serves the purpose of betterment of compatibilization.

7. Possibility of TAFMER as Replacement for PVC

As explained so far, TAFMER which our company has developed has excellent flexibility and transparency. Type A especially possess flexibility close to flexible PVC and elasticity similar to rubber. In recent years, PVC has given rise to environmental problems inclusive of its disposal problem, and Type A, therefore, has a possibility that it will be used as a replacement for PVC. Figs.14 and 15 show stress strain(SS) curves of Type A and flexible PVC and hysteresis curves of the same respectively. Since Type A contains a small amount of crystals, its SS curve has a few inflection points but is quite close to that of flexible PVC. Also, although the hysteresis curve of Type A shows small residual strain to the same degree as that of flexible PVC, Type A has good flexibility.

Regarding this replacement, we are now in the course of studies; however, we think that we shall be able to replace the said PVC sooner or later with Type A-using olefin-based resin. Also, we are making studies of replacing intravenous solution bags made of PVC with Type A-blended LLDPE or PP.

8. Resin Modifier Market in Japan

The estimated consumption of TAFMER-like resin modifiers in 1990 in Japan is 17,000 tons/year. These modifiers are being used for PP bumpers and instrument panels of automobiles, wire coating, hot-melt adhesive, among others, as well as packaging materials.

9. Summary

With the technical renovation in our society, higher-value-added packaging materials will be requested in the future. On the other hand, such technological development as gives consideration to our environments too, will be reqiured. Under these circumstances, we anticipate that olefin-based elastomer(TAFMER) developed by our company will play an important role as a resin modifier which will further enhance the characteristics of polyolefins.

Table 1 TAFMER Grades and Properties

| Item | Testing Method | Unit | TAFMER A | | | TAFMER P | | TAFMER XR | |
|---|---|---|---|---|---|---|---|---|---|
| | | | A-4085 | A-4090 | A-20090 | P-0480 | P-0680 | XR106L | XR107L |
| elt flow rate (230°C 2.16kg) | ASTM D 1238 | g/10min | 8.7 | 6.7 | 35 | 1.8 | 0.7 | 8.0 | 7.0 |
| ensity | ASTM D 1505 | g/cm³ | 0.88 | 0.89 | 0.89 | 0.87 | 0.87 | 0.89 | 0.89 |
| ensile strength at break | JIS K 6301 | kg/cm² | 270 | 310 | 160 | 30 | 50 | 360 | 365 |
| longation at break | JIS K 6301 | % | 800 | 750 | 750 | 950 | 950 | 800 | 800 |
| orsional stiffness | ASTM D 1043 | kg/cm² | 90 | 120 | 110 | 25 | 30 | 1580 | 1080 |
| urface hardness | JIS K 6301<br>ASTM D 2240 | JIS A<br>Shore D | JIS A<br>83 | 87 | 86 | JIS A<br>60 | 63 | Shore D<br>62 | 58 |
| ica softening point | ASTM D 1525 | °C | 55 | 65 | 60 | - | - | 114 | 91 |
| rittleness temp. | ASTM D 746 | °C | <-70 | <-70 | <-70 | <-70 | <-70 | -6 | -11 |
| olume resistivity | ASTM D 257 | Ω·cm | $10^{18}$ or higher | | | $10^{17}$ or higher | | $10^{17}$ or higher | |
| Dielectric breakdown voltage | ASTM D 149 | KV/mm | 55 | | | 48 | | -- | |
| Dielectric constant | ASTM D 150 | --- | 2.1 | | | 2.3 | | 2.2 | |
| Dielectric loss tangent | ASTM D. 150 | --- | $1.3\times10^{-4}$ | | | $2\times10^{-4}$ | | $2\times10^{-4}$ | |

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Table 2 Comparison of TAFMER A in physical properties with VLDPE

| Item | | TAFMER A-4090 | VLDPE (1) | VLDPE (2) |
|---|---|---|---|---|
| Density | g/cm³ | 0.895 | 0.896 | 0.907 |
| Crystallinity | % | 25 | 30 | 41 |
| Y S | kg/cm² | - | 42 | 84 |
| T S | kg/cm² | 335 | 90 | 350 |
| E L | % | 830 | 730 | 810 |
| Young's modulus | kg/cm² | 510 | 800 | 1820 |
| Mw/Mn | - | 2.0 | 4.0 | 3.2 |
| Surface hardness | (Shore D) | 40 | 31 | 46 |

Table 3 Superiority of TAFMER A type to VLDPE

| Basic Properties | Superiority of TAFMER A |
|---|---|
| (1) Low crystallizability | Flexibility ; Low temperature impact strength |
| (2) Low melting point | Heat sealability |
| (3) Narrow molecular weight and composition distributions | Transparency<br>Blocking resistance (Film) |

Table 4 Modification Effects of TAFMER

1. Low temperature heat sealability
2. Impact strength
3. Flexibility
4. Easy peeing
5. Adhesive strength
6. Pinhole resistance

Table 5 Improved Pinhole Resistance of LLDPE Film with TAFMER Blend

| Sample | Pinhole No. after 1500 cycles |
|---|---|
| LLDPE(d=0.920) | 11 |
| LLDPE/P-0480=90/10 | 4 |
| LLDPE/P-0480=80/20 | 2.5 |
| LLDPE/A-4085=90/10 | 5 |
| LLDPE/A-4085=80/20 | 1 |

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Table 6 Structures of TAFMER-using Packaging films

| Film Applications | Film Structures | Function of TAFMER |
|---|---|---|
| 2 or 3-layer OPP Film<br>Wrapping Film<br>(Cigarette, Choocolate, Video Cassette) | TAFMER XR<br>PP Homopolymer<br>TAFMER XR | Low-temperature Heat Sealability<br>Transparency<br>Scratch Resistance |
| Multilayered CPP Film<br>Vegetable Packaging | PP + TAFMER A<br>PP + TAFMER A<br>PP | Low-temperature Heat Sealability<br>Low-temperature Impact Strength |
| Multilayered CPP Film<br>Bread Packaging | PP + TAFMER A<br>PP<br>PP + TAFMER A | Low-temperature Heat Sealability<br>Welding Seal Strength |
| Multilayered CPP Film<br>Snack Packaging<br>(Potato Chips, Rice Cracker) | OPP<br>LDPE(Extrusion Coat.)<br>TAFMER A<br>PP + TAFMER<br>PP | Non primer Sandwich Lamination |
| Laminated Film<br>(Soupe, Barbecue Sauce) | O-Ny<br>Al Foil<br>LLDPE + TAFMER A | Low-temperature Heat Sealability<br>Good Seal Through Contamination<br>Impact Strength |
| Laminated Film (CPP Film)<br>Retortable pouch<br>(Hamburg, Meat Ball, Boiled Beans) | O-Ny<br>PP + TAFMER A or P | Low-temperature Heat Sealability<br>Low-temperature Impact Strength |
| Laminated Film (CPP Film)<br>Retortable pouch<br>(Curry, Stew) | PET<br>Al Foil<br>PP + TAFMER A or P | Low-temperature Heat Sealability<br>Low-temperature Impact Strength |
| Laminated Film<br>Easy Peelable Lidding Film<br>(Desserts, Cheese) | PET<br>O-Ny<br>HDPE + TAFMER A | Easy Peel |
| Laminated Film<br>Bag in Box<br>(Wine, Liquor, Juice, Soy Sauce) | EVA(or LLDPE) + TAFMER A<br>tie<br>EVOH<br>tie | Low-temperature Heat Sealability<br>Pinhole Resistance |

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Fig. 1 Introduction of TAFMER Grades

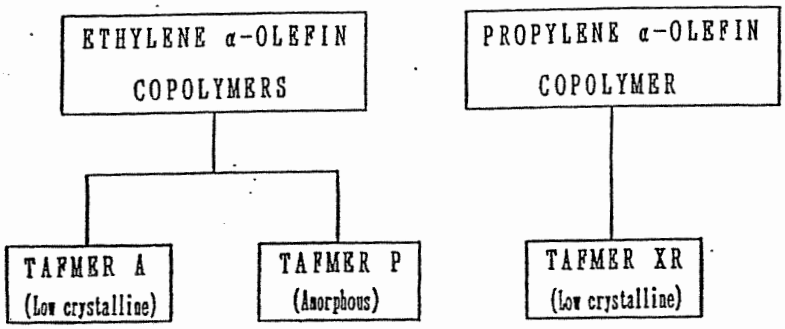


Fig. 2 Relationship between Density and Rigidity

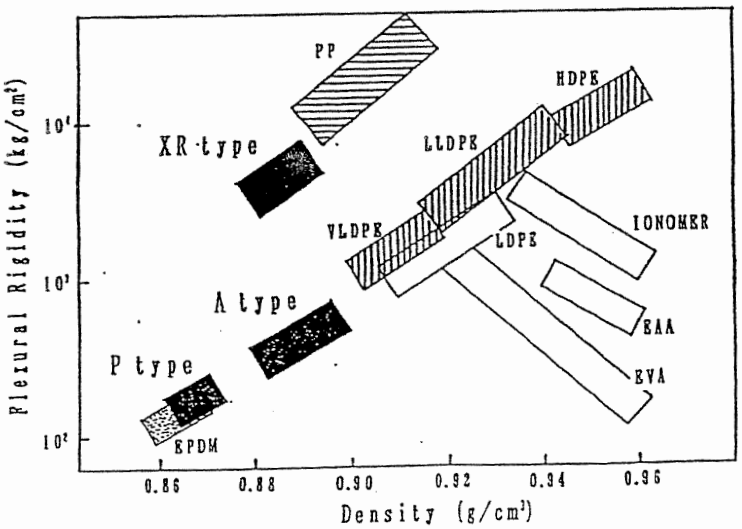

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Fig.3 Stress-Strain Curve of TAFMER

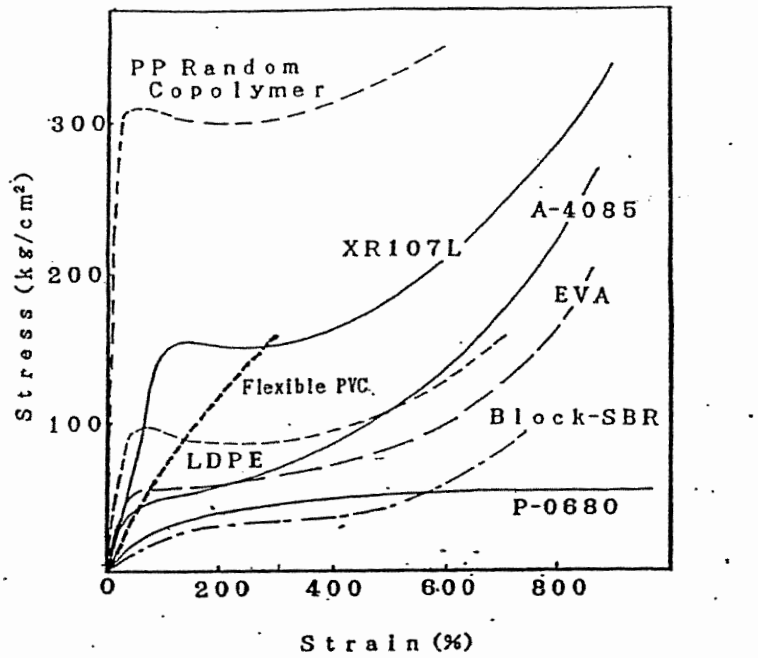


Fig.4 Relationship Between TAFMER A and LLDPE

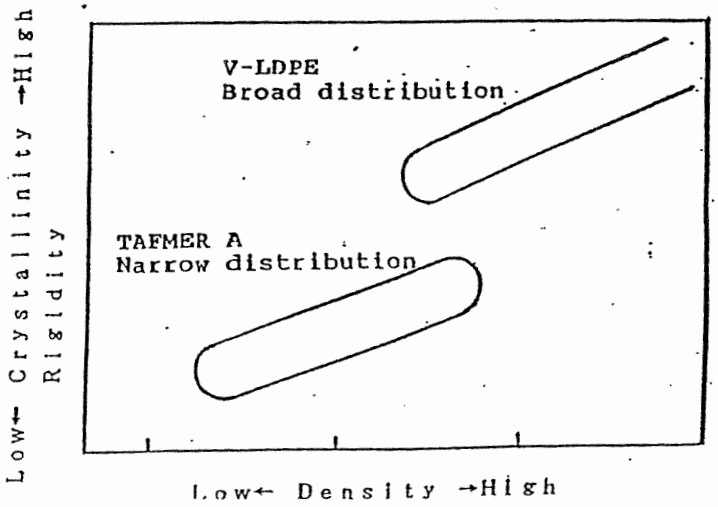

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Fig.5 DSC Chart

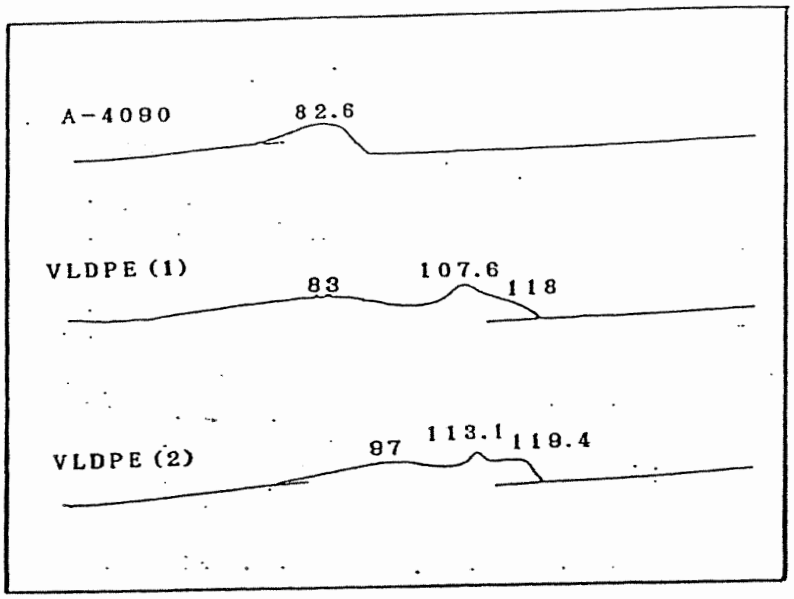


Fig.6 Heat-sealing Properties of OPP Film

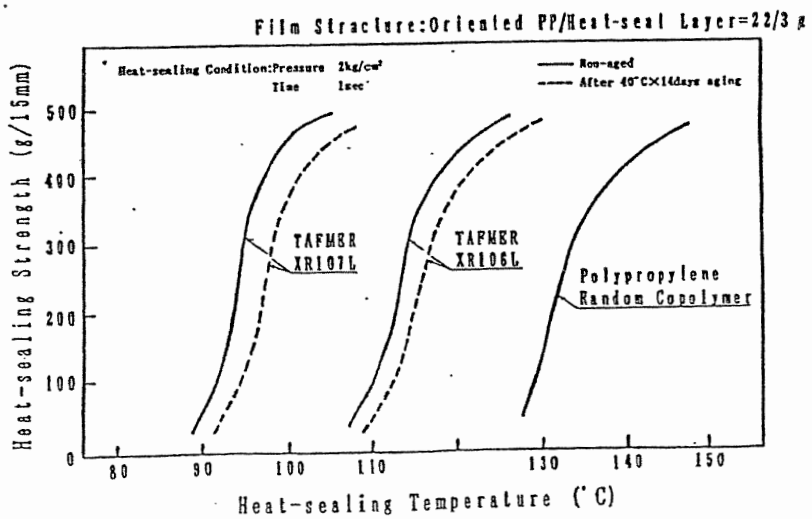

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Fig.7 Heat-sealing Properties of CPP Film

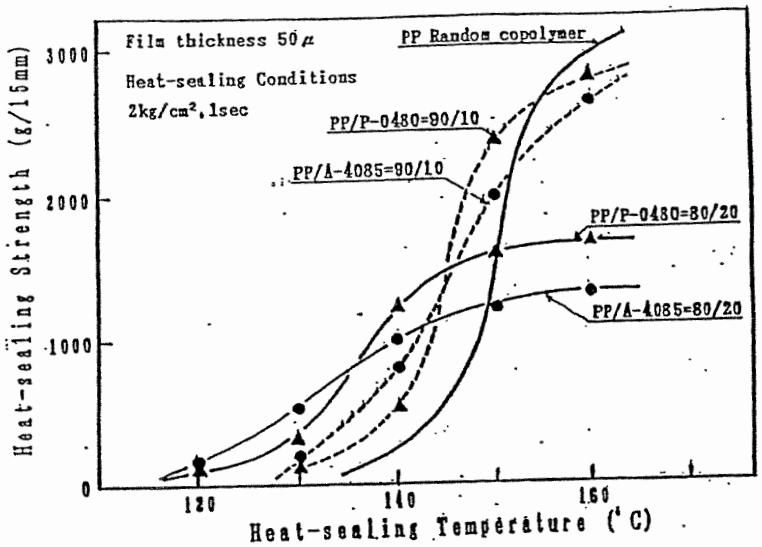


Fig.8 Improvement of Film Impact Strength

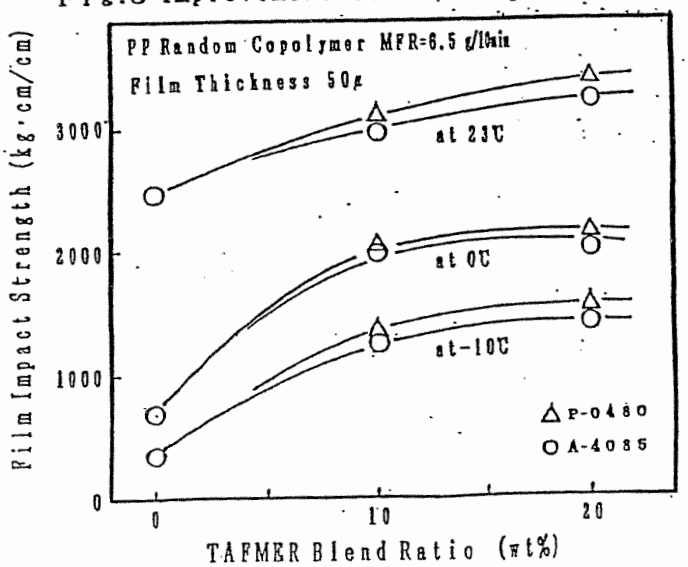

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Fig.9 Temperature Dependence of Flexibility of TAFMER A and P

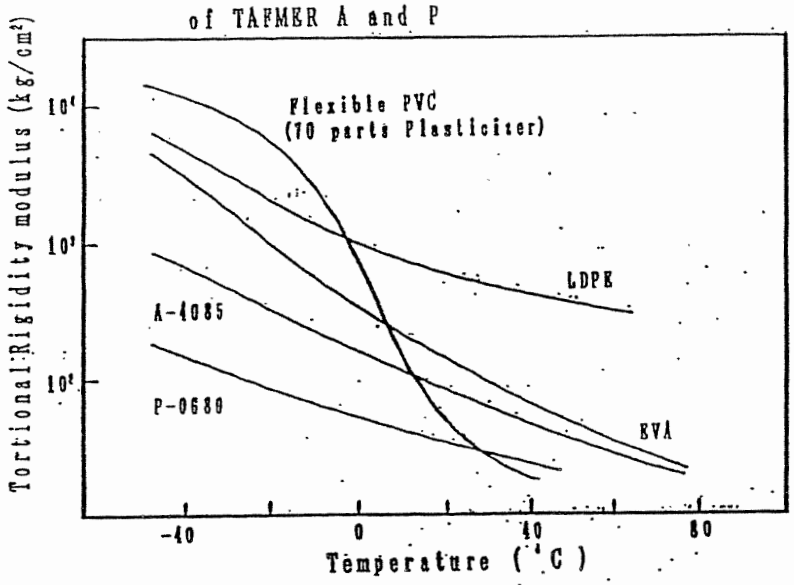


Fig. 10 Control of Heat-sealing Strength with TAFMER A

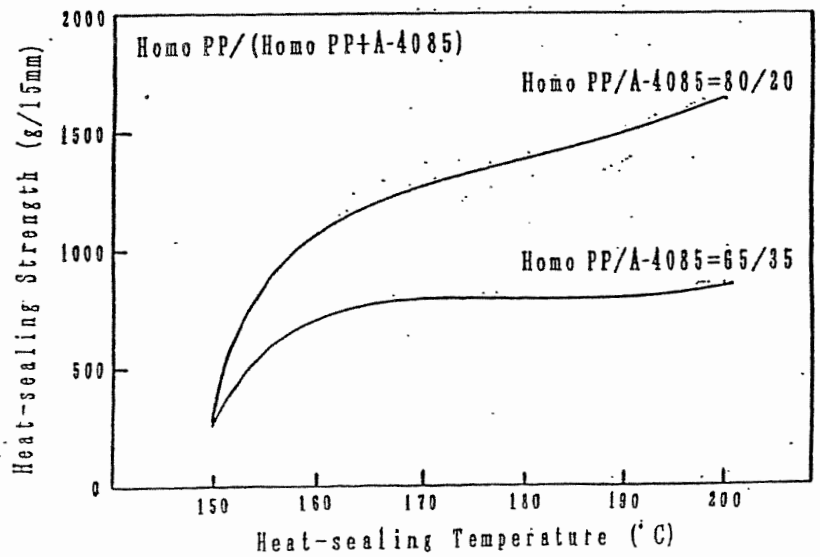

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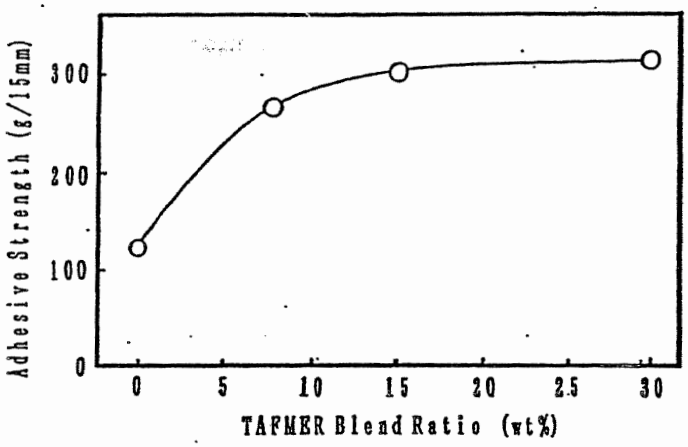
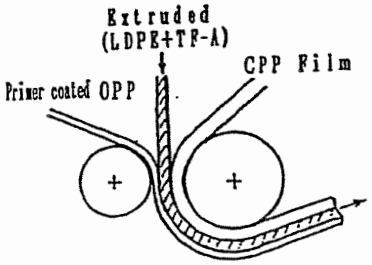
Fig.11 Adhesive Strength of Laminated Layer of LDPE Blended with TAFMER A




[Non-anchor Lamination Procedure]

(a)




(b)

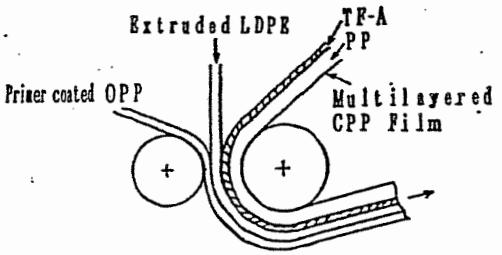


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photo 1



photo 2



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Fig.12 Relationship between Gloss and TAFMER Blend Ratio

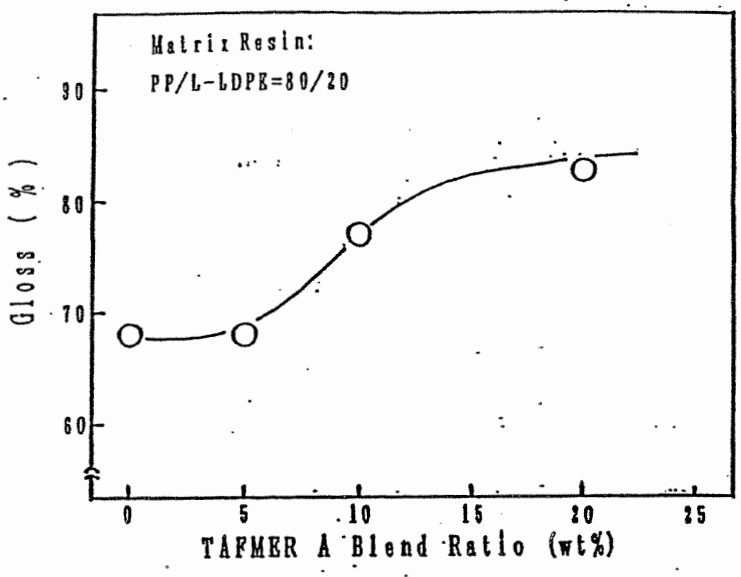


Fig.13 Relationship between Elongation and TAFMER Blend Ratio

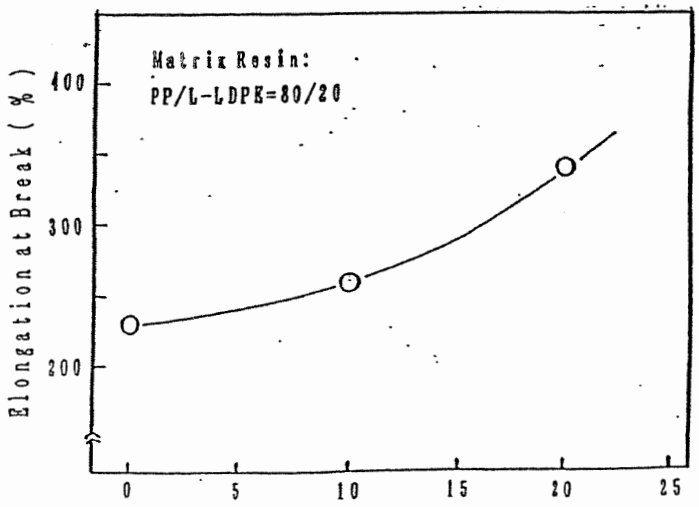

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Fig.14 Stress-Strain Curve of TAFMER and PVC

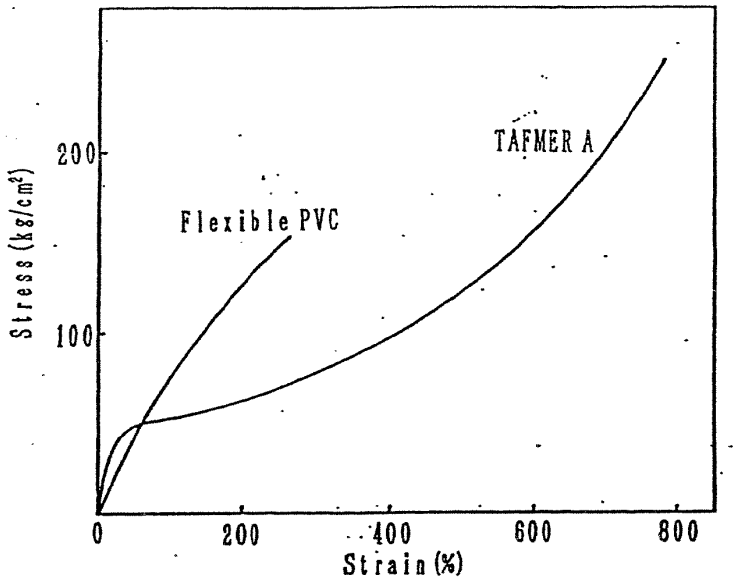



Fig.15 Hysteresis Curve of TAFMER and PVC

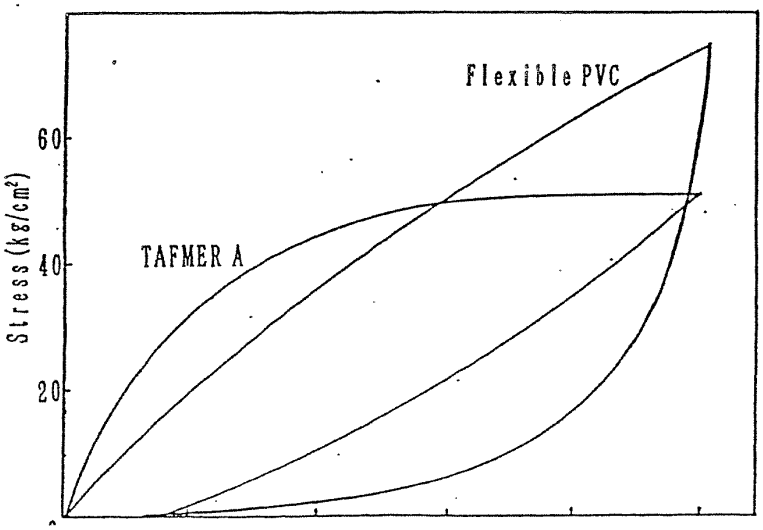
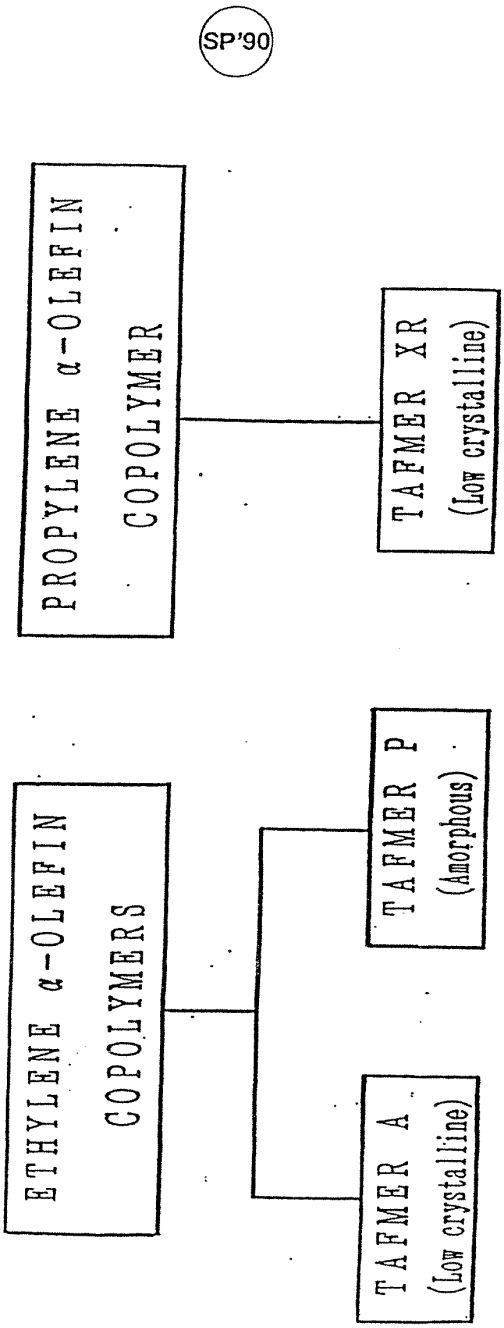


Fig. 1 Introduction of TAFMER Grades



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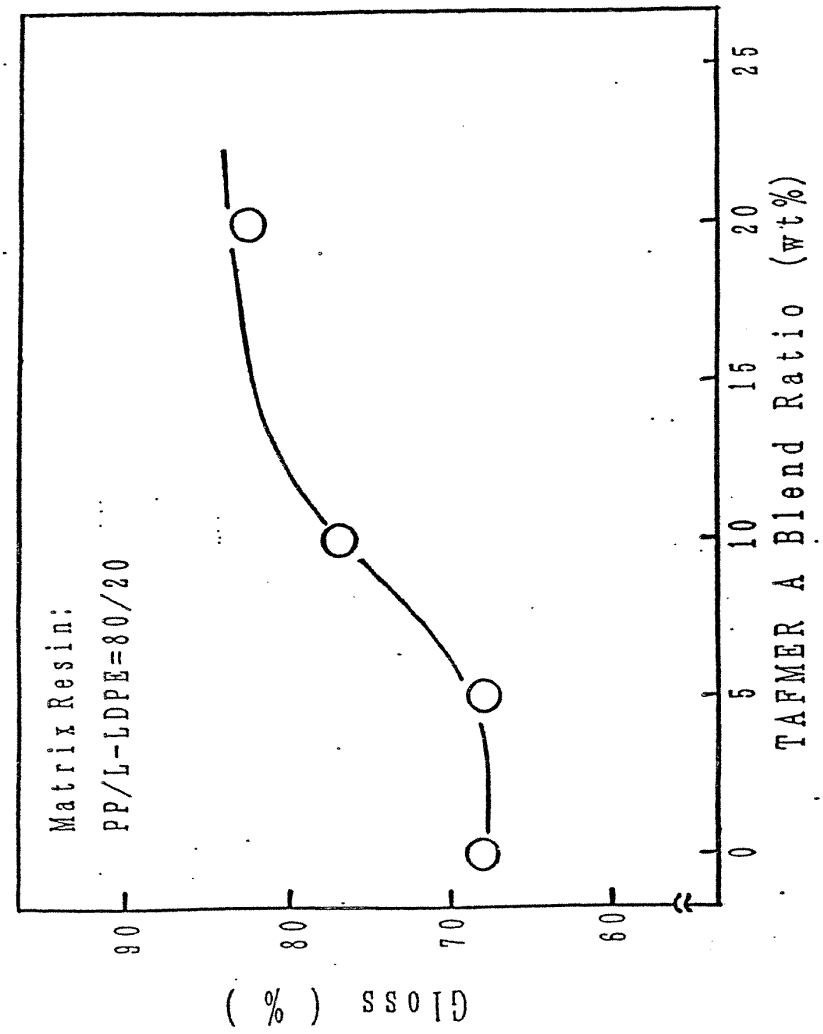


Fig. 13 Relationship between Gloss and TAFMER Blend Ratio

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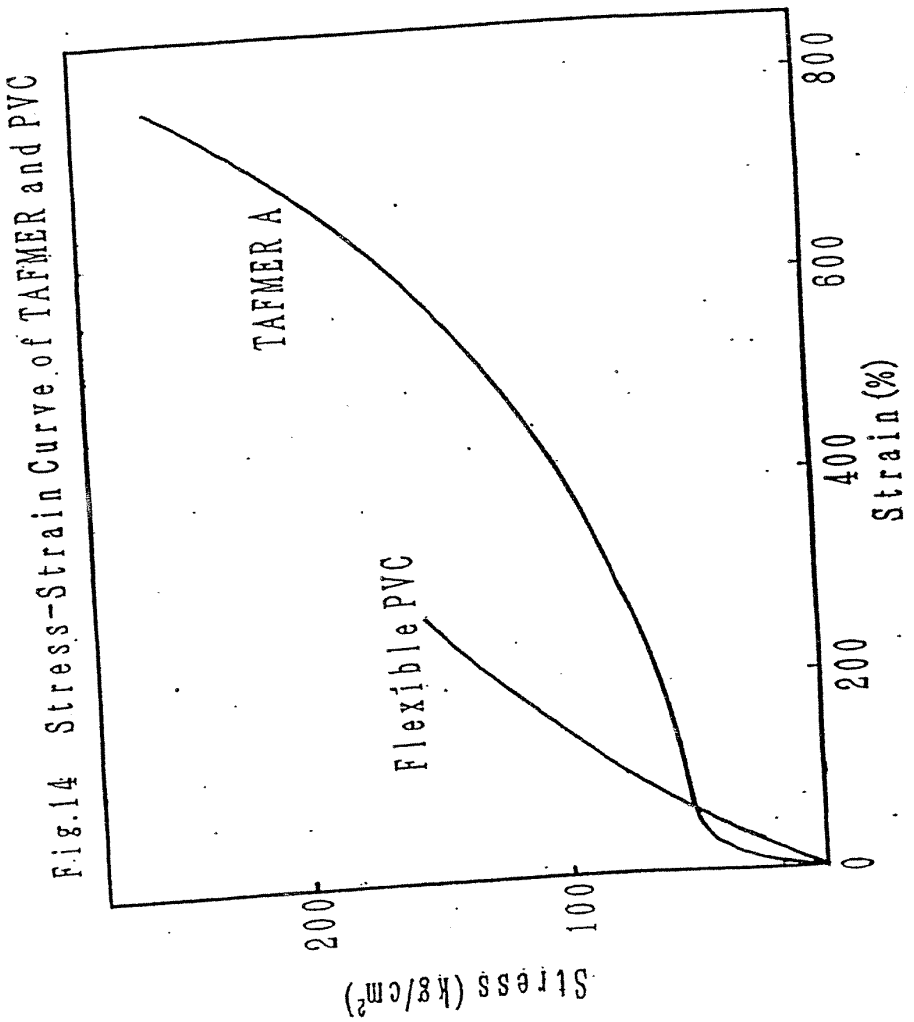


Fig.14 Stress-Strain Curve of TAFMER and PVC