

The Development of CARILON™ Thermoplastic EP Polymers for Commercial Applications. II

Impact Modification

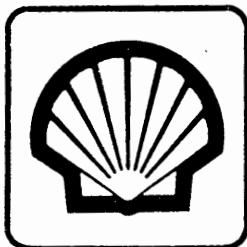
E. R. George, J. H. Coker, Jr.

Technical Progress Report WRC 8-89
Project No. 62182

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The Development of CARILON™ Thermoplastic EP Polymers for Commercial Applications. II

Impact Modification

Technical Progress Report WRC 8-89

Project No. 62182
CARILON Thermoplastic Polymer Venture Support

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REFERENCES: LR-18148, pp. 80-200; LR-18727, pp. 1-125
Based on work through June 1988

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*Under the Research Agreement between SIRM and
Shell Oil Company dated January 1, 1960, as amended.*

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ABSTRACT

CARILON™ thermoplastic polymers were impact modified effectively to produce materials suitable for a broad range of applications. CARILON™ thermoplastic polymers were compounded in binary systems with KRATON® 1901X rubber, Primacor®, and Nucrel®. Notched izod impact values of 20ft.lb/in. (equivalent to supertough nylon) were achieved with CARILON™ thermoplastic polymer 87/032 when mixed with 7 percent by weight KRATON® 1901X rubber while retaining modulus. Gardner impact resistance and melt process stability were sacrificed. Binary blends of CARILON™ thermoplastic polymer with ethylene - carboxylic acid copolymers (i.e. Primacor® and Nucrel®) lead to compounds exhibiting improved melt stability, higher elongation to break, higher notched izod impact resistance, and outstanding low temperature Gardner impact resistance (> 300 in.lb. at -30°C). Ternary systems consisting of CARILON™ thermoplastic polymer, KRATON® 1901X rubber, and either Primacor®, or Zn Surlyn® as the third component produced unexpected novel morphologies with outstanding property sets. These developments should lead to a strong patent position, and a broad range of practical applications for CARILON™ Thermoplastic polymers.

Technical Progress Report WRC 8-89

**The Development of CARILON™ Thermoplastic EP Polymers for
Commercial Applications. II****Impact Modification***by***E. R. George, J. H. Coker, Jr.****BACKGROUND**

CARILON™ Thermoplastic EP polymers are produced via the addition polymerization of carbon monoxide with ethylene and some propylene. The properties of the neat polymer are controlled by propylene content (melting point and crystallinity) and degree of polymerization (impact resistance and melt viscosity). The majority of the CARILON™ thermoplastic EP polymers provided by the MDU exhibit a melting point from 215°C to 240°C and an LVN (limiting viscosity number) of 1.4 to 2.0. Different batches are identified via a number which designates the year and the number of the batch made in that year. For example, 86/006 indicates the sixth batch made in 1986. Significant progress in the quality of the polymer was made from 1986 to 1988. The more recent batches (1988) are much cleaner, have better melt stability, and exhibit outstanding low temperature Gardner impact resistance. This report discusses impact modification of batches made from 1986 through 1988. Property sets vary due to variability of the batches.

CARILON™ thermoplastic EP polymers exhibit an increase in impact resistance with increasing LVN (i.e., molecular weight). At an LVN of ~1.9 there is a transition where the notched izod impact resistance increases significantly⁽¹⁾. However, melt viscosity increases exponentially with increasing molecular weight ($\eta \propto M^{3.4}$) and the cost of producing higher LVN polymer also increases due to longer reaction time. Thus, there is a trade-off between melt processability and cost with the maximum impact resistance polymer. Therefore, there is a practical driving force for impact modification of lower LVN CARILON™ thermoplastic polymers.

Figure 1⁽²⁾ illustrates the typical phase behavior of a semicrystalline thermoplastic. For CARILON™ thermoplastic polymers in ETP applications we desire a temperature-molecular weight combination which produces a viscous liquid. The time in the melt where $G' / G'' = 1$ (storage modulus/loss modulus) equals unity can be used as an estimate of the viscous liquid - rubber transition. Gergen⁽³⁾ and co-workers have determined that CARILON™ thermoplastic polymer melt viscosity increases

with time and that the rate of increase is proportional to molecular weight. As the quality of the polymer improved (1988 versus 1986 batches) the rate of viscosity increase decreased with time. This is attributed to cleaner polymer and improved additive packages. As a matter of fact, the ethylene-carboxylic acid copolymers which are impact modifiers in the solid state have proven to be an effective melt stabilizer as well. In conclusion, the important variables for processing CARILON™ thermoplastic polymers are time, temperature, and molecular weight. Their interrelationship is complex and any additive will change their phase behavior.

EXPERIMENTAL

All blends were prepared by first tumble mixing pellets of CARILON™ thermoplastic polymer and the added components such as KRATON® 1901X rubber, Primacor®, Zn Surlyn®, and Nucrel®. All mixtures were melt blended in 30 mm corotating twin screw extruders. Melt temperatures at the die exit were maintained at ~260°C and the zone temperatures along the barrel were maintained at ~240°C. Blends of CARILON™ thermoplastic polymer 87/032 with KRATON® 1901X rubber were done on a WP extruder with a 27/1 length/diameter ratio (L/D) at Paragon Development. Screw configurations are available upon request. All other blends were prepared on a Haake extruder with a 12/1 L/D at WRC equipped with a harsher mixing zone. Both extruders produce the same dispersed phase morphology.

Samples for testing were injection molded with a 2.2/1 compression ratio screw. The cycle time for all samples was ~30 seconds. Standard test specimens were molded in a family mold and samples were immediately placed in a dry box. All mechanical properties are reported for dry as-molded specimens.

Three distinct sets of experiments are reported here. The first set involves binary and ternary systems of CARILON™ thermoplastic polymer 87/032 with KRATON® 1901X rubber and either Primacor® 1410 or Zn Surlyn® 9520. Included in the first set of experiments were blends of KRATON® 1901X rubber with batch 87/032, 87/031, 87/015, and 87/053 all nominal 220°C melting point polymers with LVN of 1.96, 1.74, 1.57, and 1.31, respectively.

KRATON® 1901X rubber is a modified KRATON® G containing anhydride groups at about a 2 percent concentration by weight. The poly(ethylene/butylene) midblock leads to low temperature flexibility, excellent thermal, oxidative, and UV stability. KRATON® 1901X rubber has been employed to improve impact strength in several engineering thermoplastics including nylons and polyesters. We report here its outstanding efficiency in toughening CARILON™ thermoplastic polymers.

The second set of experiments employs CARILON™ thermoplastic polymer 86/006 in blends with ethylene-methacrylic acid copolymers (Nucrel®) and ethylene-acrylic acid (Primacor®) copolymers. Nucrel® grades 010, 035, 403, 535 and Primacor grades 1410 and 1430 were blended at 0.5, 1.0, and 5.0 percent by weight. The copolymers differ by acid content (%) and melt flow index (190°C) as follows;

| <u>NUCREL® GRADE</u> | <u>ACID CONTENT</u> | <u>MFI</u> |
|------------------------|---------------------|------------|
| 010 | 20 | 10 |
| 035 | 20 | 35 |
| 403 | 09 | 03 |
| 535 | 10 | 35 |
| <u>PRIMACOR® GRADE</u> | | |
| 1430 | 09 | 1.5 |
| 1430 | 09 | 05 |

The third set of experiments employ CARILON™ thermoplastic polymers 88/008 and 88/023 with Primacor® 1430 and Nucrel® 535, respectively. Gardner impact resistance is reported for these much improved base polymers.

RESULTS AND DISCUSSION

A. BINARY BLENDS WITH KRATON® 1901X RUBBER

Figure 2 shows the increase in notched izod impact resistance upon the addition of KRATON® 1901X rubber in CARILON™ thermoplastic EP polymer 87/032. At higher rubber content (>8%) the impact resistance dropped off sharply. Scanning Transmission Electron Microscopy (STEM) studies⁽⁴⁾ revealed that high izod strength occurs only when the KRATON® 1901X rubber is finely dispersed with an average diameter of ~0.3-0.6 mm. Above 7 wt% rubber the dispersed phase becomes larger and at 15 wt% co-continuous phases begin to form. Figure 3 illustrates the change in morphology of the molded test specimens with decreasing rubber content. The large increase of notched izod resistance at lower rubber content is atypical of engineering thermoplastics.

The increase in low temperature (-20°F) notched izod resistance is shown in Figure 4. Low temperature impact resistance approaches 5ft.lb./in. which is a requirement for automotive body panels. Since CARILON™ thermoplastic polymers exhibit a glass transition temperature around room temperature, the increase of impact strength at -20°F is significant.

Gardner impact resistance (falling dart) was evaluated at room temperature for the same series of samples. Table I shows that Gardner impact decreased at 5 weight percent rubber, however the failure was isolated at the point of impact versus catastrophic failure of the sample. Automotive compounds were prepared recently⁽⁵⁾ impact modified with KRATON® 1901X rubber. Low temperature Gardner impact was significantly improved and confirmed the change in failure mode even at low temperature. Perhaps the finely dispersed rubber phase prevents propagation of cracks and isolates the impact stress at the point of impact.

The flex modulus of this rubber modified blend series showed a similar trend to the notched izod impact resistance (Table 2). The increase in modulus can be attributed to an accelerated crosslinking reaction caused by the presence of the maleated rubber. The drop in modulus at 10 wt% rubber is due to the onset of a co-continuous phase illustrated in Figure 3. The increase in viscosity with time is shown in Figure 5 for batch 87/032 containing 5 wt% KRATON® 1901X rubber alone, and in combination with ethylene-carboxylic acid copolymers. While the acid containing copolymers improve melt stability, no improvement was observed when used in combination with the KRATON® 1901X rubber.

Tensile properties were measured for the 87/032 control and the blend containing 5 wt% rubber. The properties were almost identical but elongation at break was lower for the rubber modified polymer. The addition of 5 wt% rubber increased the stiffness (modulus) and tear strength (izod) of a CARILON™ thermoplastic polymer while sacrificing some elongation and falling dart impact resistance. These phenomena are not completely understood but chemistry and rate of reaction in the melt are likely to be important factors in the impact modification of CARILON™ thermoplastic polymers.

The effect of using different starting LVN CARILON™ thermoplastic polymers on impact strength was investigated. Figure 6 plots notched izod impact versus percent rubber for four different LVN polymers. The rate of increase in notched izod strength with rubber content is proportional to LVN of the polymer.

Effect of residence time in the extruder on notched izod impact resistance was investigated for CARILON™ thermoplastic polymer 87/015 blended with 5 wt% KRATON® 1901X rubber. Residence time was varied by passing the compound three times through the extruder. Samples were collected and molded after each pass. No change in izod strength was observed with residence time.

B. BINARY BLENDS WITH ETHYLENE - CARBOXYLIC ACID COPOLYMERS

The effect of ethylene-carboxylic acid copolymers on impact resistance, melt stability, and injection moldability in CARILON™ thermoplastic polymer 86/006 were investigated. Six different copolymers were added at 0.5, 1.0, and 5.0 wt.%. The dispersed phase morphology was investigated by STEM. The copolymers differ by acid content and molecular weight (see Experimental).

Table 3 reports the values of notched izod impact resistance for this series of ethylene-carboxylic acid copolymers blended with batch 86/006. Primacor® added at 1 weight percent yielded the highest notched izod values. Table 4 reports the injection pressure with all other molding parameters constant for this series. The injection pressure is affected only at high levels (5%) of the ethylene-carboxylic acid copolymers. However, the ease of injection is offset by a decrease in Notched Izod and Gardner Impact Resistance (Tables 3 & 5).

The dispersed phase morphology of the ethylene-carboxylic acid copolymers was investigated by STEM. Figure 7 shows that the minor component is dispersed in the polyketone matrix with an average cell size

less than 1 micron in diameter. The acid content and MFI did not affect significantly the morphology of this series of blends up to 5 wt%.

Figure 8 compares notched izod impact resistance of polyketone - KRATON® 1901X rubber binary blends to polyketone - Primacor® 1430 binary blends up to 10 wt% of the added component. All values are for a base polymer with a nominal 220°C melting point and 1.7 LVN. The KRATON® 1901X rubber promotes crosslinking in the melt (Figure 5) whereas the Primacor® can actually stabilize the melt. The sharp drop-off in impact at high rubber content is attributed to the onset of a co-continuous phase (Figure 3).

It was observed that polyketones produced in 1988 exhibited outstanding Gardner impact resistance at low temperature and particularly when modified with ethylene-carboxylic acid copolymers (8). Table 6 reports the Gardner impact values for CARILON™ thermoplastic polymers 88/008 and 88/023. The addition of 2 wt% Primacor® 1410 and 1 wt% Nucrel® 535, respectively, to these two polymers significantly increased the low temperature impact strength. These ethylene-carboxylic acid copolymers may prevent crosslinking and subsequently reduce defect concentration in the molded parts. These low temperature impact values are significantly high and these blends may be regarded as "supertough".

C. TERNARY BLENDS WITH KRATON 1901X RUBBER AND ETHYLENE-CARBOXYLIC ACID COPOLYMERS

It has been demonstrated that carboxylic acid containing copolymers improve melt stability (9) and impact resistance (7). Therefore, two different copolymers were studied in combination with 5 wt% KRATON® 1901X rubber and CARILON™ thermoplastic polymer 87/032. Figure 5 illustrates that Zn Surlyn® improves the melt stability when added at 1 wt% but had no stabilizing effect in the ternary system with KRATON® 1901X rubber. The stabilizing effect of Zn Surlyn® is similar to that of Primacor® and Nucrel®. The ineffectiveness of Zn Surlyn® in the ternary systems can be explained by the phase behavior observed in STEM (Figure 9). The Primacor® and Zn Surlyn® prefer the rubber phase over the polyketone matrix and become isolated therefore no improvement in melt stability.

The role of these distinct morphologies in impact modification is well studied (10,11). The subinclusions whether as a shell-core morphology or broken inclusions increase the effective rubber volume of the dispersed rubber phase. However, the size of the dispersed phase is critical to impact modification.

Figure 10 is a bar graph showing impact resistance versus selected blends. The addition of 5 wt% KRATON® 1901X rubber significantly increased impact strength. Primacor® 1410 at 1 wt% showed an increase in impact strength, a good increase for only 1 wt% additive. The Primacor® disperses as submicron particles similar to KRATON® 1901X rubber. The combination of 1 wt% Primacor with 5 wt% KRATON® 1901X rubber increased impact strength significantly and is attributed to a submicron dispersed shell-core morphology. High levels of Primacor in the binary and ternary system yielded impact strength similar to the control.

Figure 11 shows a similar trend for Zn Surlyn® blends, however when Zn Surlyn® was used in the same combination with the rubber no increase in impact strength was observed. Figure 9 illustrates the distinct differences in phase behavior for the two ternary systems. Zn Surlyn® is an ethylene-methacrylic acid copolymer partially neutralized with Zn. The Primacor® is an ethylene-acrylic acid copolymer with no neutralization. Perhaps the Zn plays a role in producing the dispersed subinclusions in the rubber phase.

The phase behavior exhibited by the Zn Surlyn®, Primacor®, and KRATON® 1901X combinations mixed with CARILON™ thermoplastic EP polymers can have significant ramifications. Perhaps using less rubber in combination with Zn Surlyn® can produce smaller domain size leading to improved impact resistance. Secondly, this system could be employed in many other thermoplastics all of which will require their own characteristic dispersed phase size (11). Similar considerations apply for the ternary system containing Primacor®.

CONCLUSION

CARILON™ thermoplastic polymers were effectively impact modified with ethylene-carboxylic acid copolymers and KRATON® 1901X rubber. The addition of 1 wt% ethylene-carboxylic acid copolymer to engineering polyketones significantly improved melt stability, impact strength, and other mechanical properties. CARILON™ thermoplastic polymers were impact modified by dispersing KRATON® 1901X rubber as a minor component in submicron dimensions. The binary system can be modified by the addition of Zn Surlyn® or Primacor® as a third component to increase the effective rubber volume. These novel morphologies can be produced in other thermoplastics thus representing a general approach. This work demonstrated how engineering polyketones may be tailored to meet the requirements of a host of practical applications.

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TABLE 1
GARDNER IMPACT RESISTANCE OF CARILON™ THERMOPLASTIC POLYMER
BLENDED WITH KRATON® 1901X RUBBER

| <u>SAMPLE</u> <u>(WT.% RUBBER)</u> | <u>GARDNER IMPACT</u> <u>(IN.LBS.)</u> |
|---------------------------------------|---|
| 87/032 (Control) | 315 |
| 2% | >320 |
| 5% | 125 |
| 7% | 38 |

TABLE 2
FLEX MODULUS VERSUS KRATON® 1901X RUBBER CONTENT
IN CARILON™ THERMOPLASTIC POLYMER 87/032

| <u>SAMPLE (% RUBBER)</u> | <u>FLEX MODULUS</u> |
|--------------------------|---------------------|
| 87/032 | 260,000 |
| 2 | 260,000 |
| 5 | 313,000 |
| 7 | 290,000 |
| 10 | 247,000 |
| 15 | 182,000 |
| 20 | 131,000 |

TABLE 3
NOTCHED IZOD IMPACT OF ETHYLENE-CARBOXYLIC ACID COPOLYMERS
BLENDED WITH CARILON™ THERMOPLASTIC POLYMER 86/006

| <u>SAMPLE</u> | <u>0.5%</u> | <u>1.0%</u> | <u>5.0%</u> |
|----------------------------|-------------|-------------|-------------|
| Nucre [®] 010 | 5.6* | 6.8 | 6.0 |
| Nucre [®] 035 | 6.4 | 5.8 | 7.2 |
| Nucre [®] 403 | 5.6 | 5.9 | 6.3 |
| Nucre [®] 535 | 5.6 | 7.0 | 7.2 |
| Primacor [®] 1410 | 6.6 | 9.8 | 7.1 |
| Primacor [®] 1430 | 7.4 | 9.2 | 6.5 |

Control 4.8

* All values ft.lb./in. measured on 1/8" injection molded test specimens.

TABLE 4
INJECTION MOLDING PRESSURES OF ETHYLENE-CARBOXYLIC ACID COPOLYMERS
BLENDED WITH CARILON™ THERMOPLASTIC POLYMER 86/006

| <u>SAMPLE</u> | <u>0.5%</u> | <u>1.0%</u> | <u>5.0%</u> |
|---------------|-------------|-------------|-------------|
| Nucrel®010 | 1250 | 1100 | 900 |
| Nucrel®035 | 1000 | 1100 | 950 |
| Nucrel®403 | 1100 | 1100 | 850 |
| Nucrel®535 | 1100 | 1100 | 725 |

| | | | |
|---------|------|--|--|
| Control | 1100 | | |
|---------|------|--|--|

* All values psi of Injection Pressure

TABLE 5
GARDNER IMPACT RESISTANCE OF ETHYLENE-CARBOXYLIC ACID COPOLYMERS
BLENDED WITH CARILON™ THERMOPLASTIC POLYMER 86/006

| <u>SAMPLE</u> | <u>0.5%</u> | <u>1.0%</u> | <u>5.0%</u> |
|---------------|-------------|-------------|-------------|
| Nucrel®010 | > 320 | > 320 | 20 |
| Nucrel®035 | > 320 | > 320 | 30 |
| Nucrel®403 | > 320 | > 320 | 20 |
| Nucrel®535 | > 320 | > 320 | 25 |

| | | | |
|---------|-------|--|--|
| Control | > 320 | | |
|---------|-------|--|--|

* All values (in.lb.)

TABLE 6
GARDNER IMPACT RESISTANCE OF 1988 CARILON™ THERMOPLASTIC POLYMERS
BLENDED WITH ETHYLENE-CARBOXYLIC ACID COPOLYMERS⁸

| <u>SAMPLE</u> | <u>GARDNER IMPACT (IN. LB.)</u> | |
|----------------------------|---------------------------------|-------------------------|
| | <u>R.T.</u> | <u>-20⁰F</u> |
| 88/023 (Control) | >400 | 220 |
| 88/023 + 1% Nucrel® 535 | >400 | 360 |
| 88/008 (Control) | >320 | 250 |
| 88/008 + 1% Primacor® 1410 | >320 | >320 |

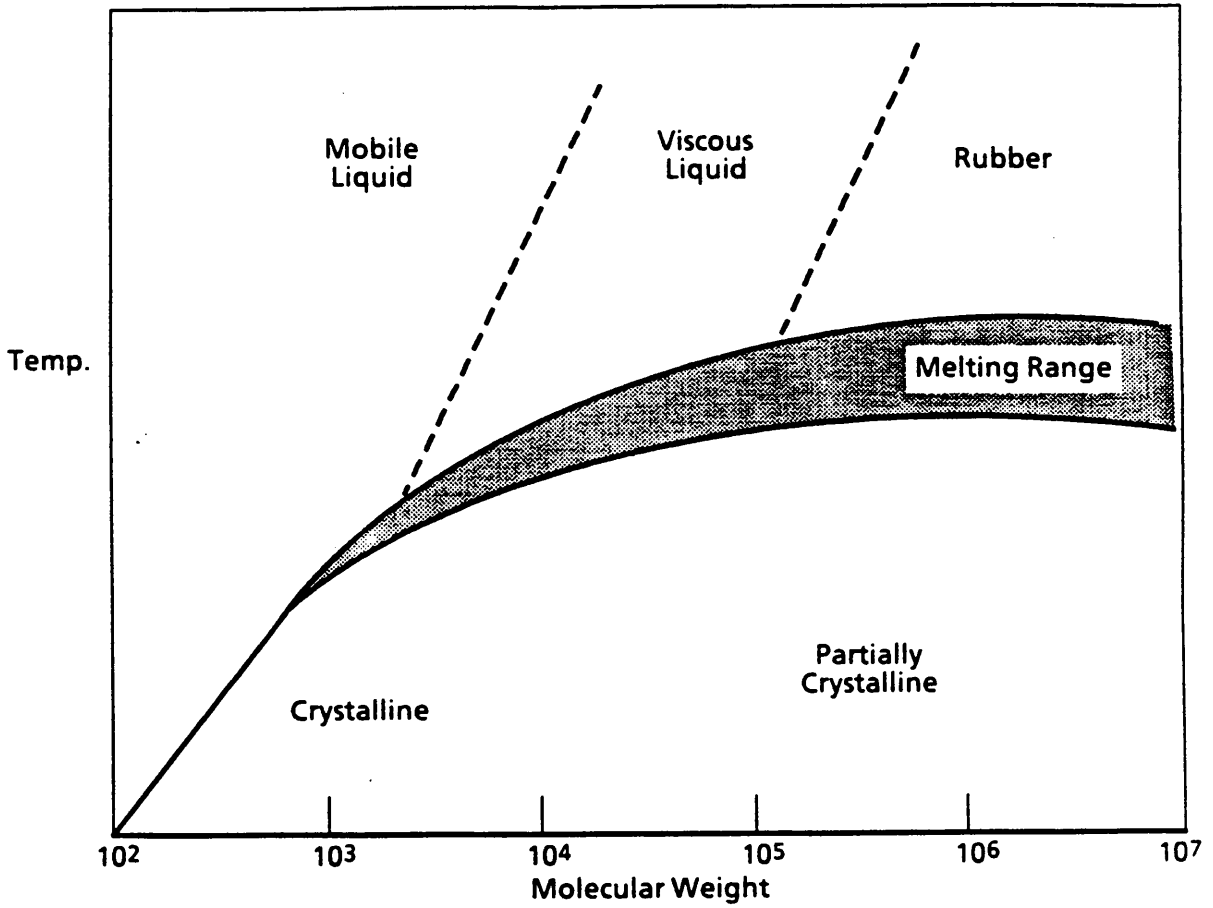


Figure 1. Phase Behavior Typical of Semicrystalline Thermoplastics (2)

- STEM of 5% KRATON® 1901X Blend shows a finely dispersed rubber phase ~0.3–0.5 μm diameter.
- Impact modification is possible at low KRATON® 1901X content (~5%) this should be more economical than impact modified nylon.
- Modulus was retained and even higher in the 5% impact modified blend.

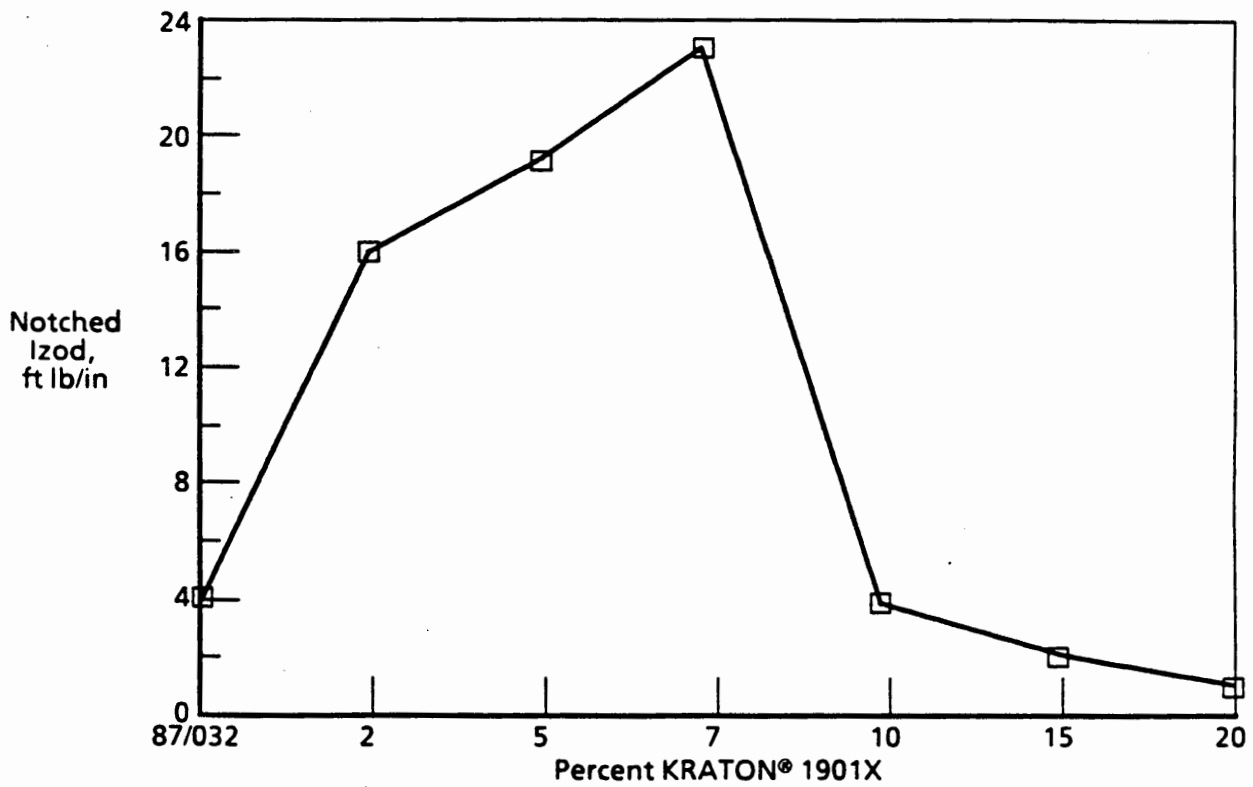
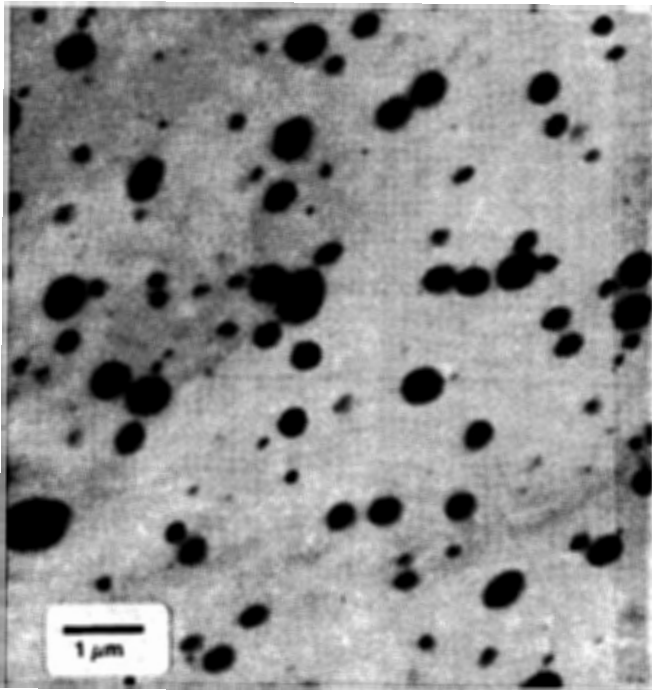
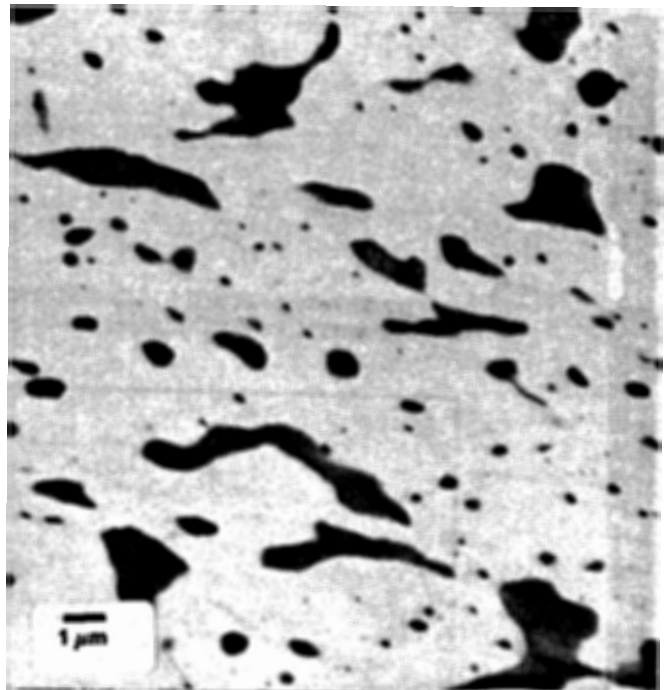


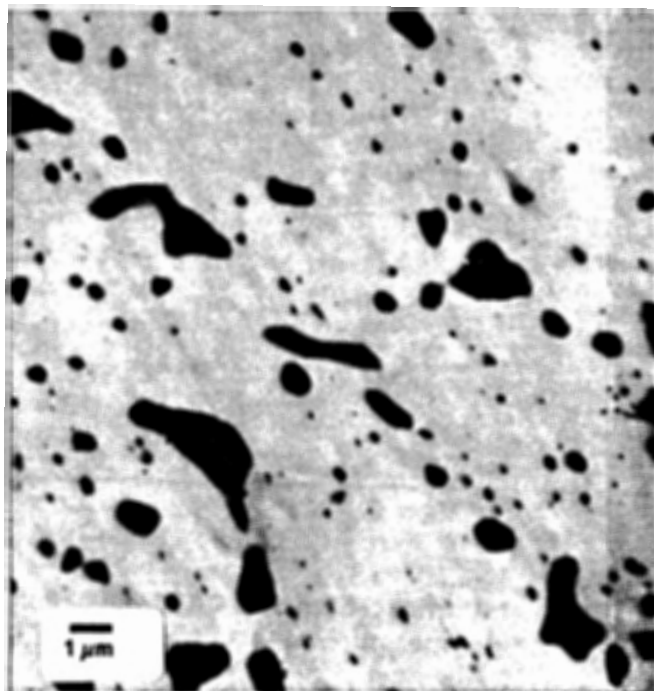
Figure 2. Impact Modified CARILON™ EP Polymer
Notched Izod vs KRATON 1901X



5% KRATON® 1901X Rubber



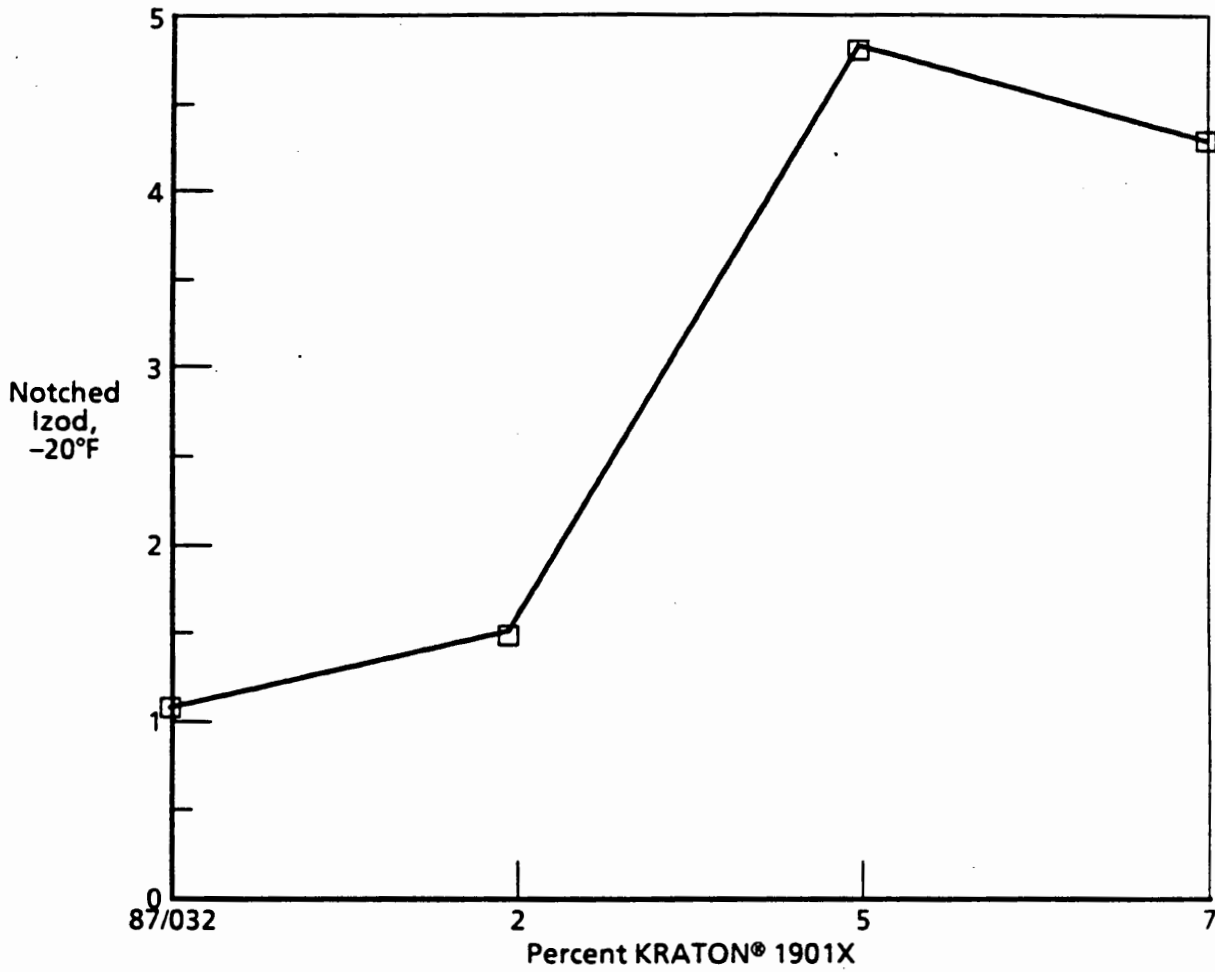
15% KRATON® 1901X Rubber



10% KRATON® 1901X Rubber

Figure 3. Change in Dispersed Phase Morphology vs. Rubber Content

- Low temperature impact approaches 5 ft lb/in which is a goal for Automotive Applications.



**Figure 4. Impact Modified CARILON™ EP Polymer
Low Temperature Impact vs % KRATON 1901X**

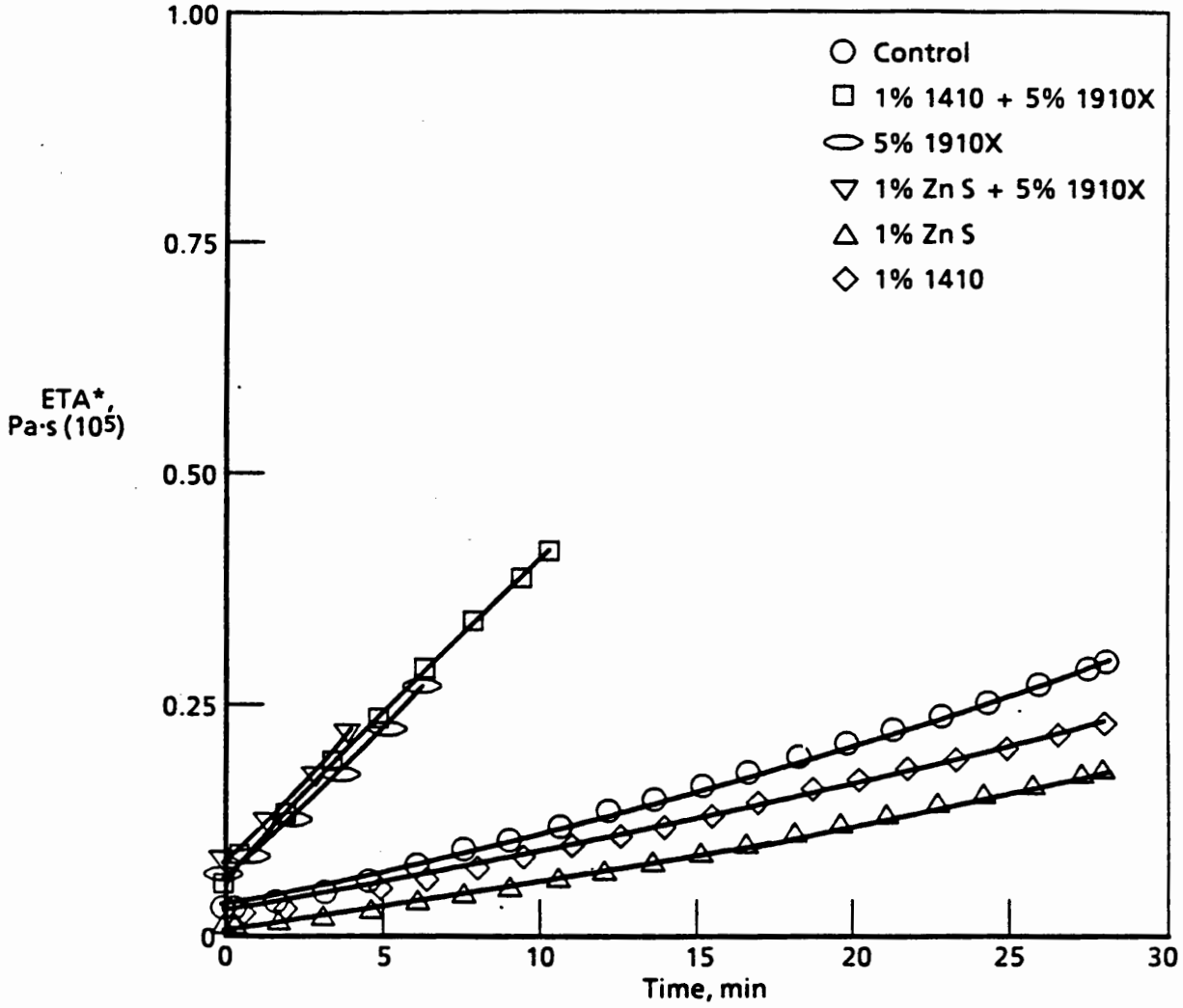


Figure 5. Melt Viscosity Versus Time for CARILON™ Thermoplastic Polymer Blends
87/032 Blends, Feb-88 -275- Dynamic Time Sweep

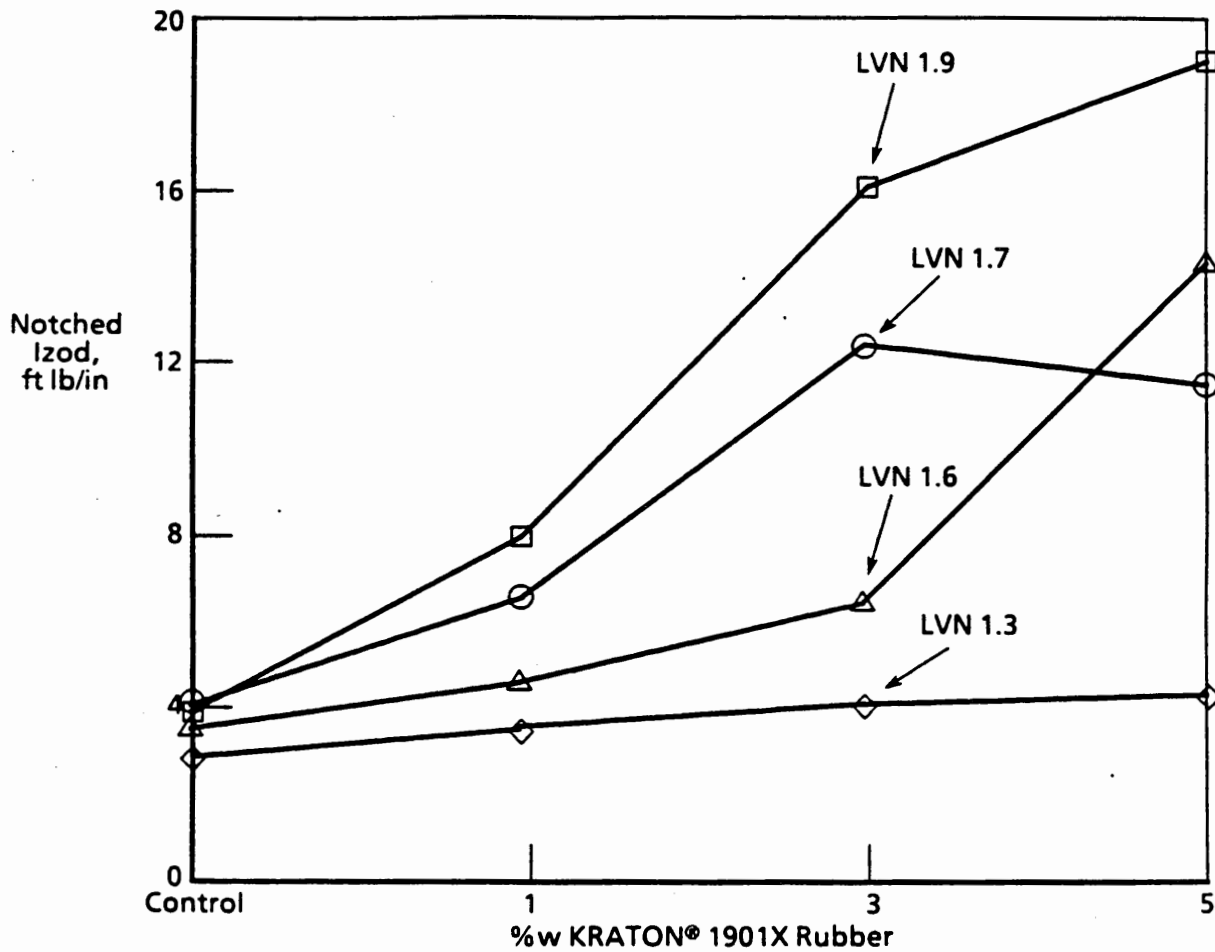
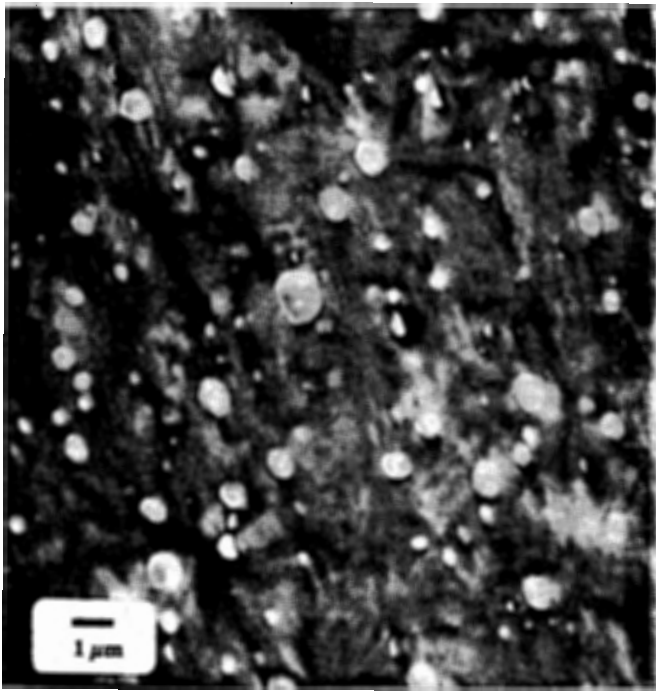
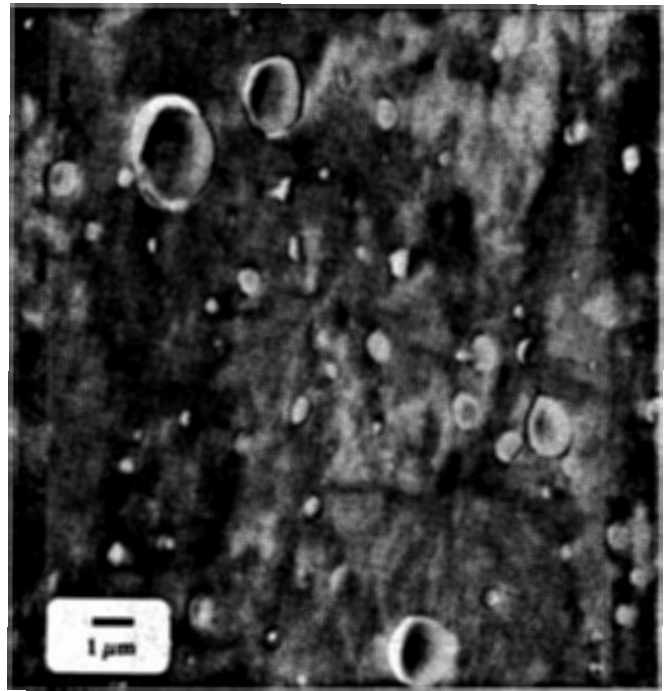


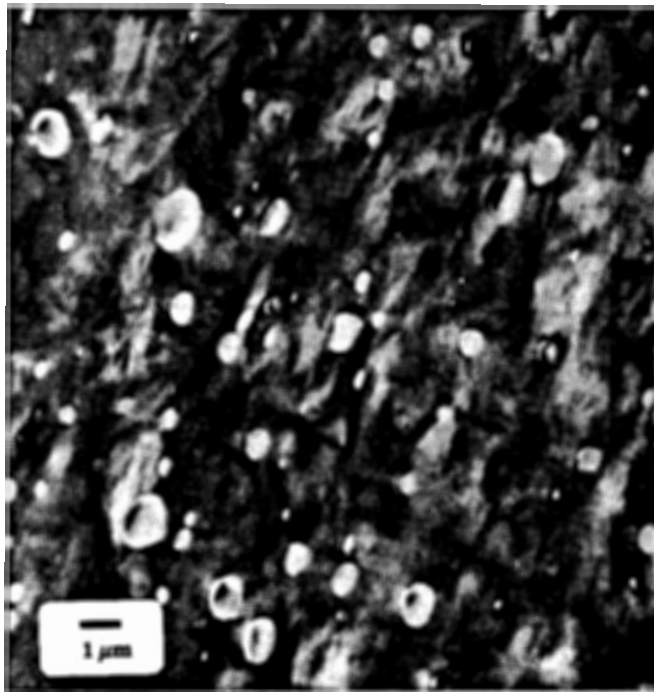
Figure 6. Impact Modified CARILON™ EP Polymers
Molecular Weight Dependence



Nucrel 035

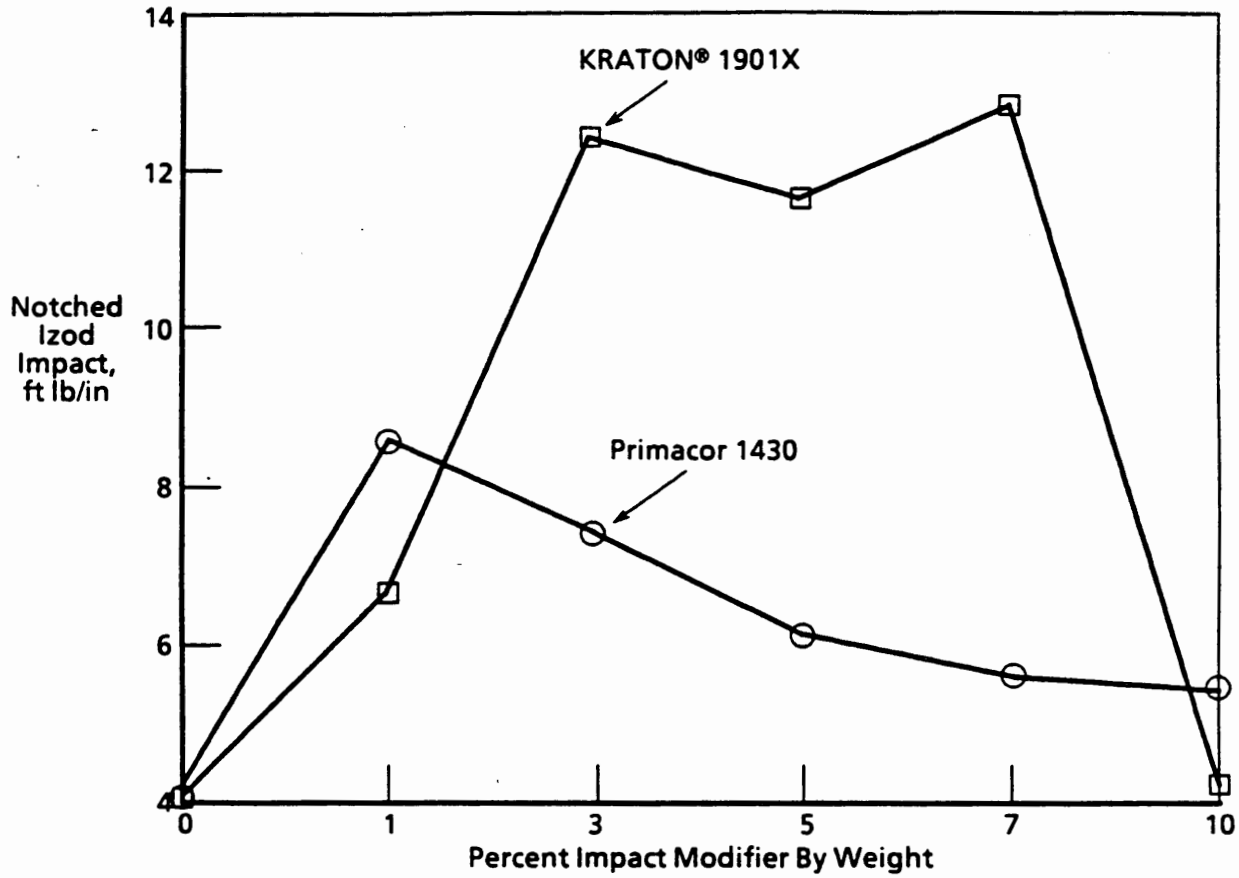


Nucrel 535

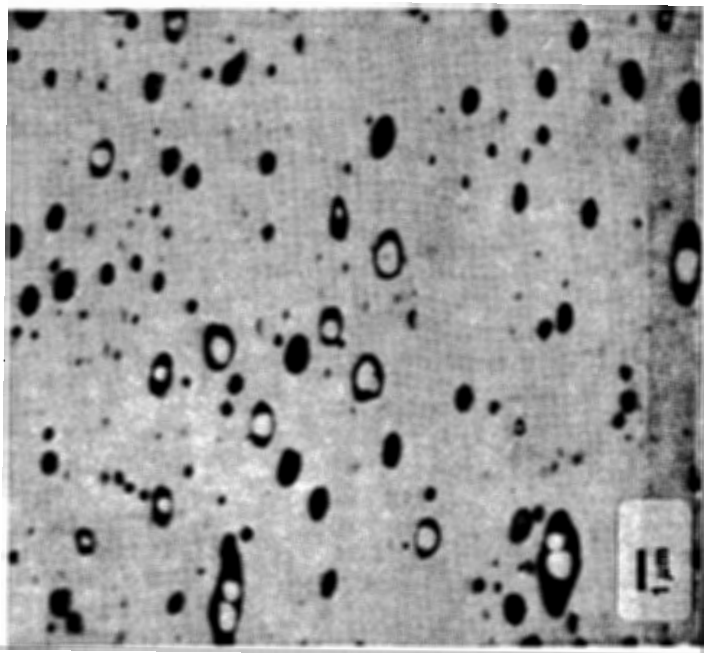


Nucrel 010

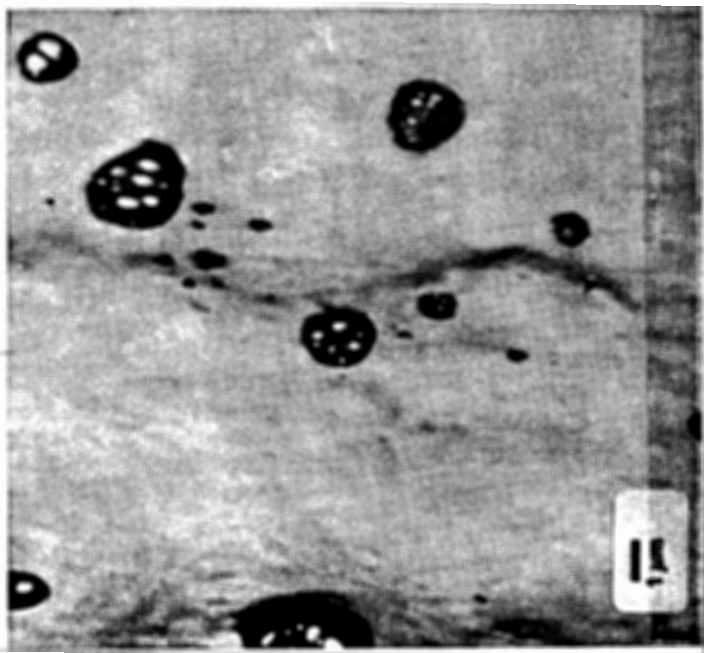
Figure 7. Dispersed Phase Morphology of Three Different Nucrels in CARILON™ Thermoplastic Polymer 86/006 at 5%w



**Figure 8. Impact Modified Engineering Polyketones
220 MP 1.7 LVN Base Polymer**

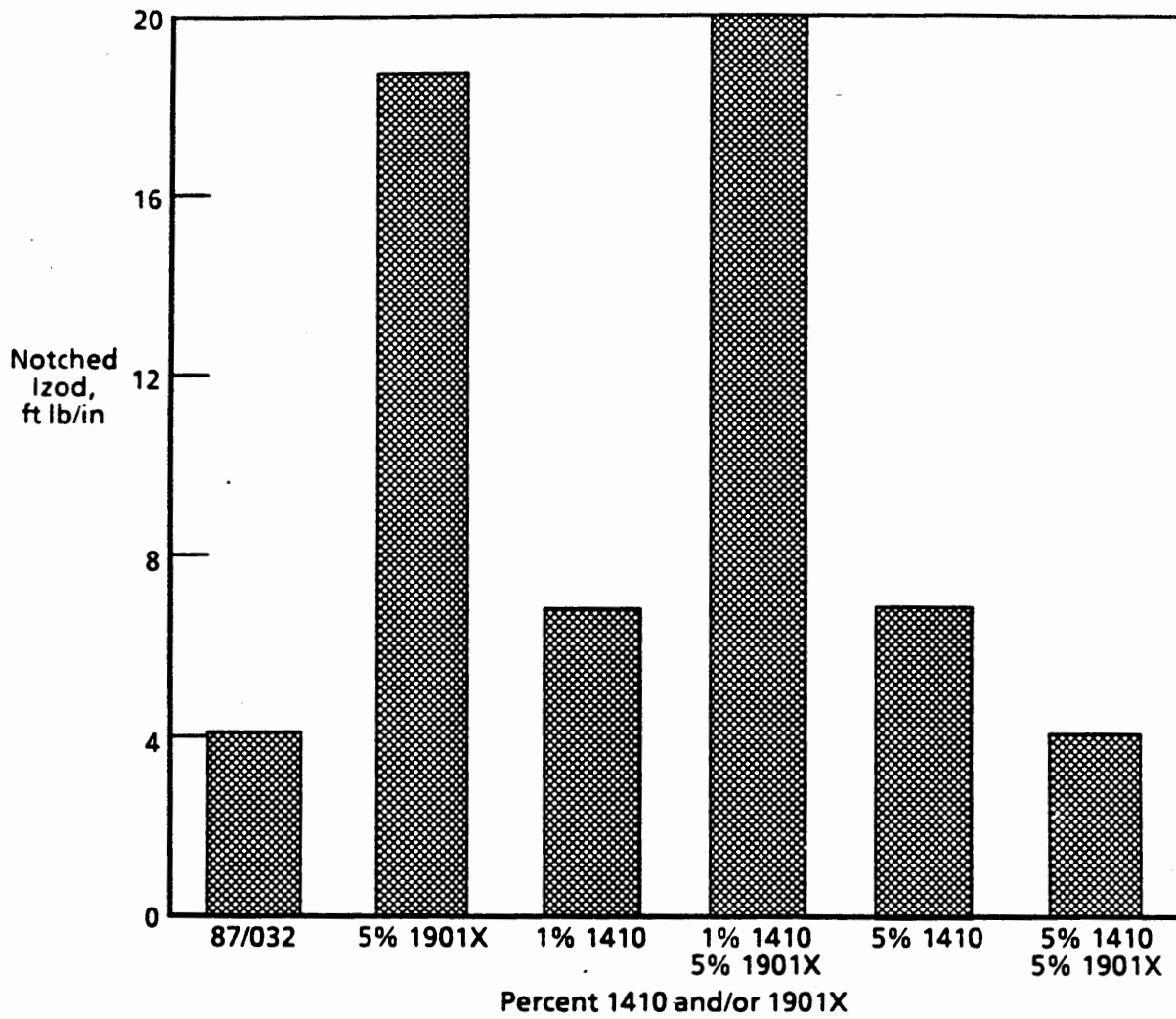


Polyketone -KRATON®
1901X Rubber - PRIMACOR®

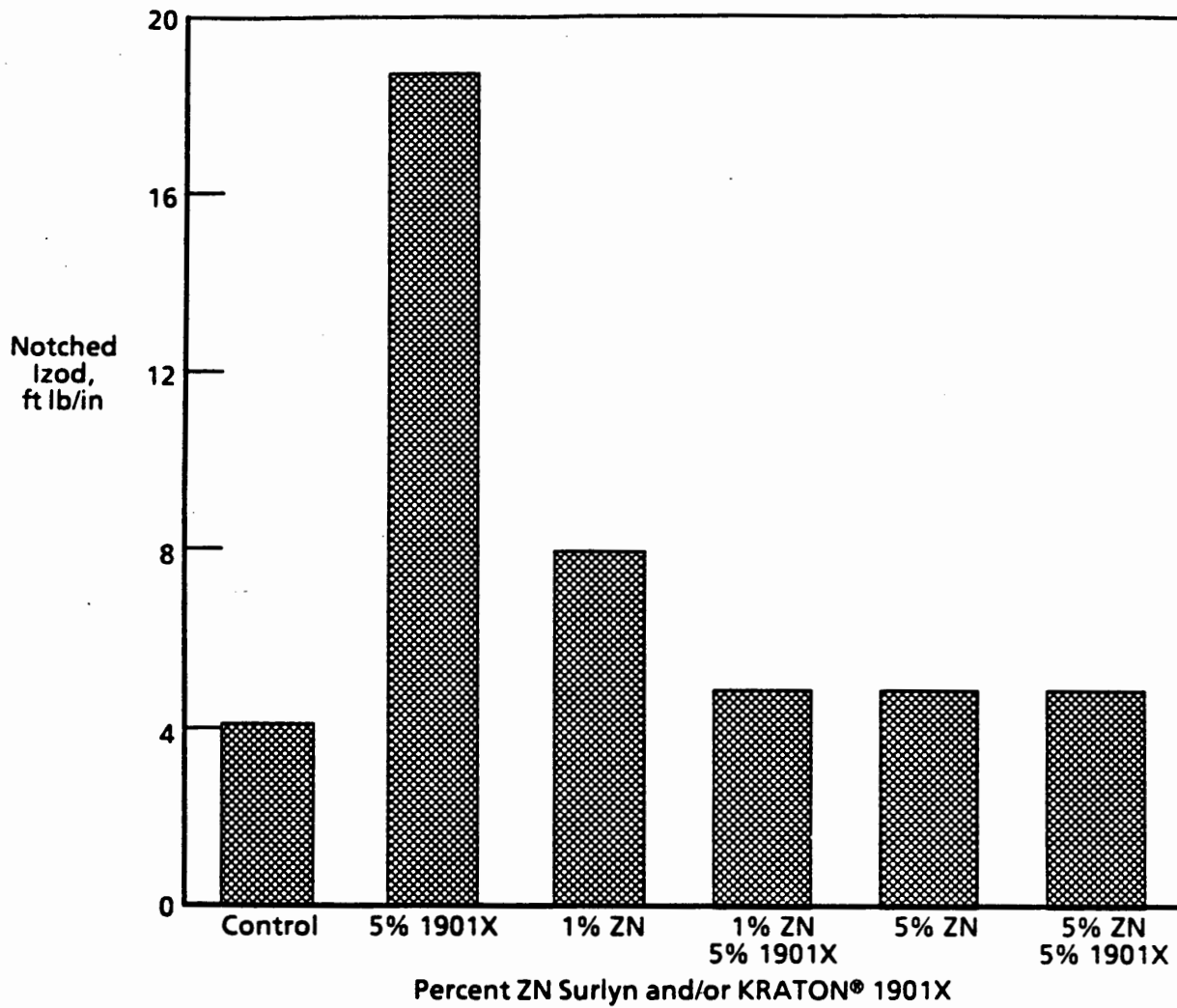


Polyketone -KRATON®
1901X Rubber - Zn SURLYN®

Figure 9. Two Distinct Dispersed Phase Morphologies for Ternary Systems Consisting of CARILON™ Polymer, 5% KRATON® 1901X Rubber Using Zn SURLYN® and PRIMACOR® as the Third Component, Respectively



**Figure 10. CARILON™ EP Polymer Blends
Primacor 1410 and KRATON® 1901X**



**Figure 11. CARILON™ EP Polymer Blends
ZN Surlyn and KRATON® 1901X**

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