Melt flow index: More than just a quality control rheological parameter. Part II

Article *in* Advances in Polymer Technology · June 1986

DOI: 10.1002/adv.1986.060060201

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Melt Flow Index: More Than Just a Quality Control Rheological Parameter. Part II*

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4. PRODUCT PROPERTY EVALUATION

Despite the fact that MFI is generally considered as a measure of the rheological behavior and processability of the polymers, it has been shown over the years to correlate with final product properties. A general outline of the effect of MFI increase can be seen from Table XIV taken from Refs. 105 and 106,† which enlists most of the common physical properties of the end product properties. In the following, a detailed discussion of the response of product properties upon changes in MFI will be done.

4.1. Tensile Strength

The tensile strength at yield as well as the ultimate tensile strength at break have a reasonably strong dependence on MFI. The changes in the tensile yield strength with MFI are shown in Figure 68 taken from Ref. 107. It can be seen that, at lower MFI values, the changes are sharper but eventually the increase proceeds in a linear manner. When plotted against temperature, the tensile yield strength of polymers with two MFI values (which are an order of magnitude different from each other) are practically the same at lower temperature and gradually merge as the temperature increases, as shown in Figure 69.

The simultaneous effect of MFI and crystallinity on the yield stress can be seen from Figure 70 taken from Crespi and Ranalli,[108] who studied the behavior of polypropylene with different isotatic index. It is seen that, with the same isotatic index that is the equivalent crystallizable part, lower MFI samples are less crystalline and hence show a lower yield stress; but a small variation of the isotatic index leads to a remarkable increase in the yield stress from samples with the same flowability or in other words, similar MFI.

The tensile strength at break is almost entirely dependent on molecular weight and hence is sharply sensitive to changes in MFI. This is demonstrated by the plots in Figures 71 and 72 taken from Refs. 109 and 110. For linear low density polyethylene films, the decrease in the ultimate tensile strengths in cross

*For Part I, refer to *Adv. Polym. Tech.*, **6**, 1–58 (1986).
†See Part I for reference citations below 105.

Advances in Polymer Technology, Vol. 6, No. 2, 125–145 (1986)

CCC 0730-6679/86/020125-21$04.00

TABLE XIV
Product Properties Affected by an Increase in the Melt Flow Index (Source: Refs. 105 and 106)[a]

Physical Property	
Tensile strength	−
Yield stress	−
Rigidity/stiffness	−
Toughness	−
Modulus of elasticity	−
Creep resistance	−
Hardness	−
Impact strength	− −
Thermal stability	x
Resistance to low temperatures	− −
Solubility and swellability	+
Permeability	+
Resistance to environmental stress cracking	− −
Gloss	+
Transparency	x

[a]Pronounced increase = + +, pronounced decrease = − −, slight increase = +, slight decrease = −, no change = x

and machine directions is approximately by the same amount for increasing values of MFI; however, for high densities while the cross direction ultimate tensile strength increases, the machine direction strength decreases for the same MFI (Figure 71). In the case of EVA copolymers, too, the decrease in the tensile strength at break is also very sharp, as can be seen from Figure 72.

4.2. Ultimate Elongation

The effect of MFI on elongation at break is not as radical as in the case of tensile strength at break, as is evident from Figure 72. The elongation at break is seen to remain fairly constant with changing MFI at constant vinyl acetate content. At least, the ultimate elongation may increase slightly with increasing MFI as can be seen from Figure 73 taken from Krassig et al.[18] for polypropylene film tape stretching. In fact, ultimate elongation is more dependent on density than MFI. Thus, in polymers that are near or above the glass transition temperature, the ultimate elongation generally decreases as crystallinity increases[111]; this is due to the decreasing mobility of the system. Whereas polymers of medium crystallinity (20–60%) will cold

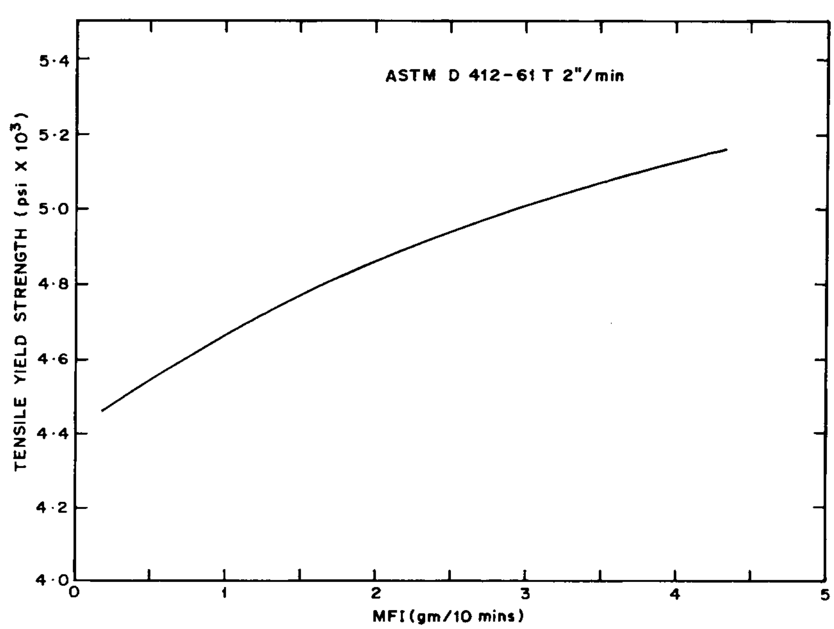


FIGURE 68
Variation of tensile yield strength with melt flow index as per ASTM D412-61T, 2 in./min (taken from Ref. 107).

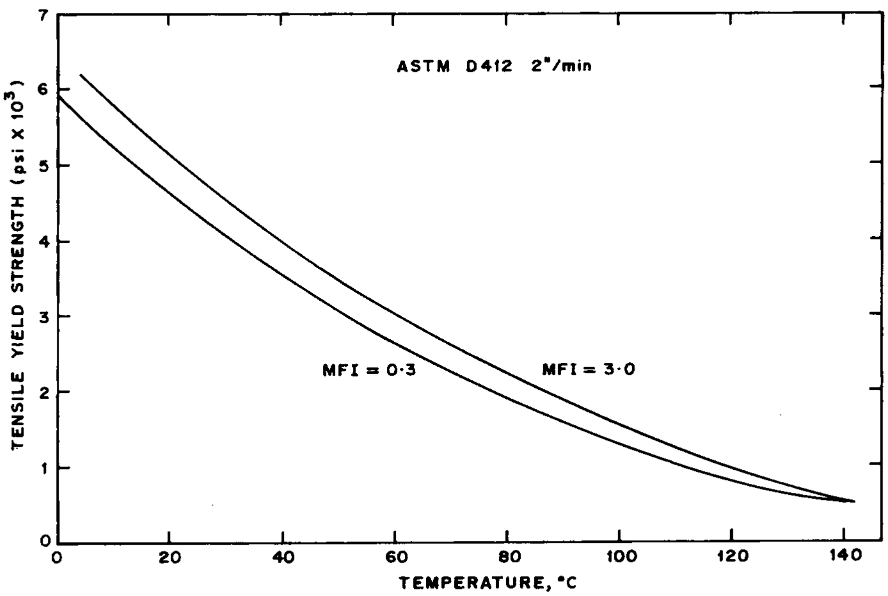


FIGURE 69 Variation of tensile yield strength with temperature for two different melt flow index samples as per ASTM D412-61T, 2 in./min (taken from Ref. 107).

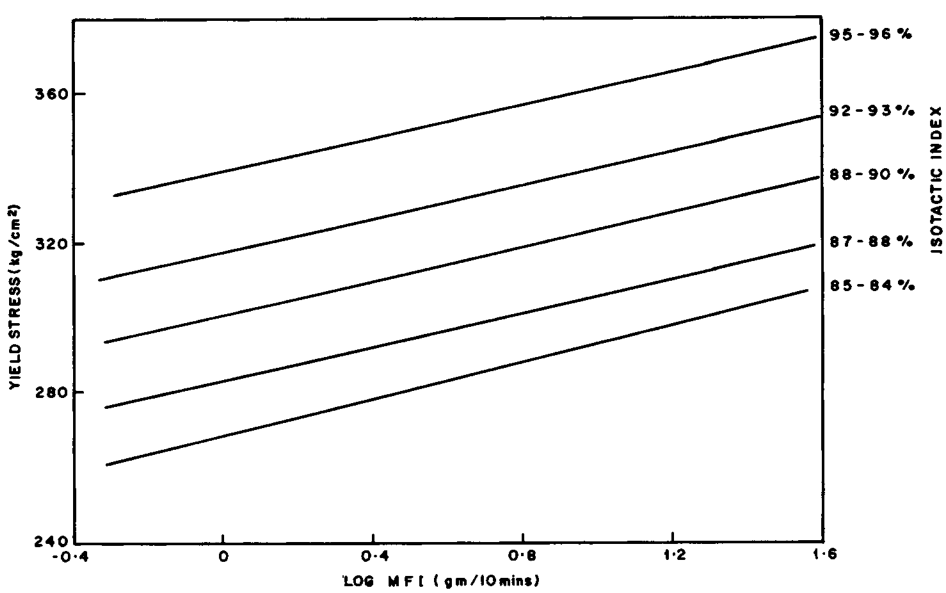


FIGURE 70 Variation of tensile yield stress with melt flow index and isotactic index of polypropylene (taken form Crespi and Ranalli[108]).

draw beyond the yield point, polymers of high crystallinity (70–90%) become brittle and break.[112] The yield point normally increases linearly with density in the case of both high and low MFI. As a general rule, the lower the MFI, the higher the stretch limit at the same density.

4.3. Tenacity

The effect of MFI on tenacity of film tapes can be seen from Figure 74 taken from Krassig et al.,[18] which shows that increasing MFI decreases the tenacity of the film. During film processing, in order to achieve

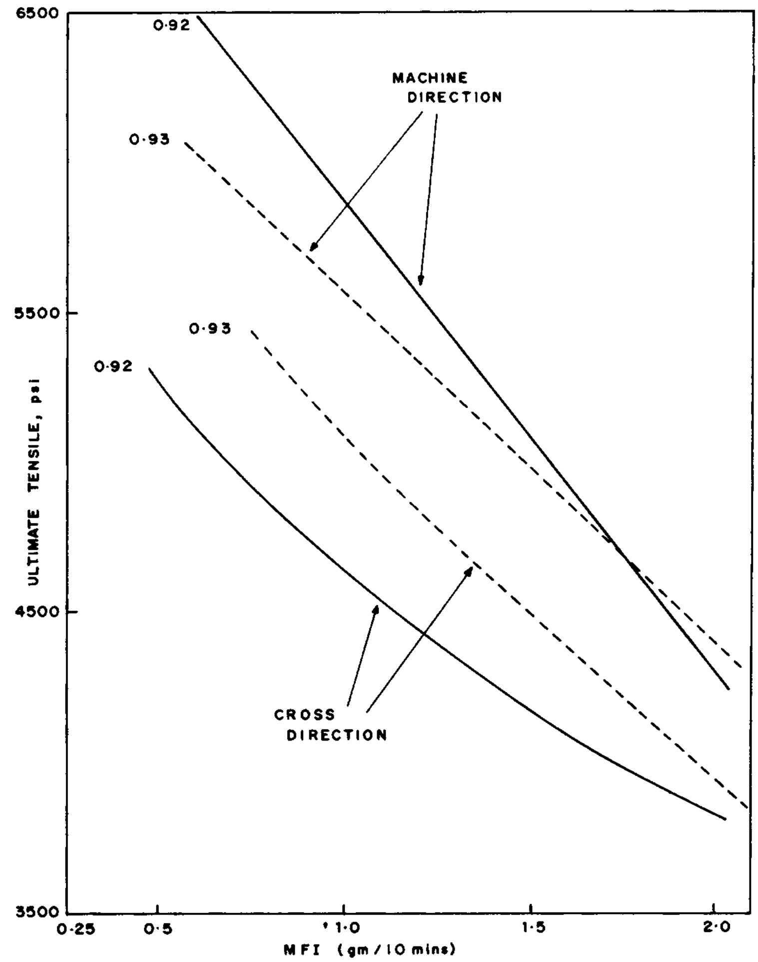


FIGURE 71 Variation of the ultimate tensile strength with melt flow index for linear low density polyethylene films (taken from Ref. 109).

high tenacity in the films the extrusion conditions must be adjusted so that the compression and metering zones are maintained at low temperatures to avoid molecular orientation while the die is maintained at a high temperature to reduce preorientation of the film in the melt form as it would prevent maximum orientation during the stretching operation. The film melt flow index indicates that for achieving the highest tenacities the MFI value has to be as low as is permissible, as can be seen from Figures 75 and 76 taken from Krassig[113] and Evans,[97] respectively. The effect of draw ratio as well as extrusion temperature becomes evident from these figures.

4.4. Elastic Modulus and Flexural Stress

The elastic modulus and flexural stress both serve as criteria for the rigidity of the material. In general, the elastic modulus is higher for lower MFI.[106] The flexural stress at maximum deflection also follows the same trend as the elastic moduli, though the values of flexural stress at maximum deflection are of the order of ten times less than those for elastic moduli.[106] Flexural modulus or stiffness increases rapidly with increasing density but not with MFI. In fact, a polymer with a MFI of 2.0 will hardly be perceptibly stiffer than the one with a MFI of 20 if the densities are the same.[114] The curve of stiffness in flexure (Figure 77) is almost a straight line. When plotted against temperature, the stiffness of polypropylene with two MFI values (which are an order of magnitude different from each other) is almost the same and, in fact, identical at higher temperatures as can be seen from Figure 78 taken from Ref. 107.

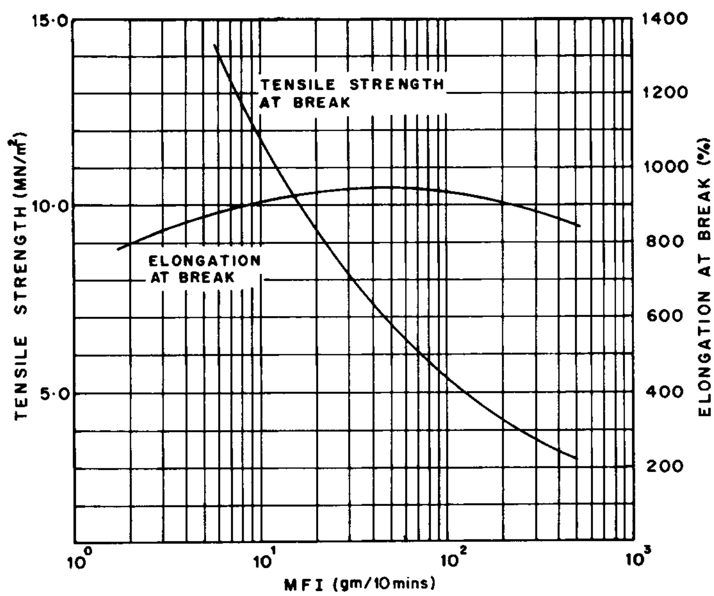


FIGURE 72 Variation of the tensile strength at break with melt flow index for ethylene–vinyl acetate copolymers at constant vinyl acetate content (taken from Gilby[110]).

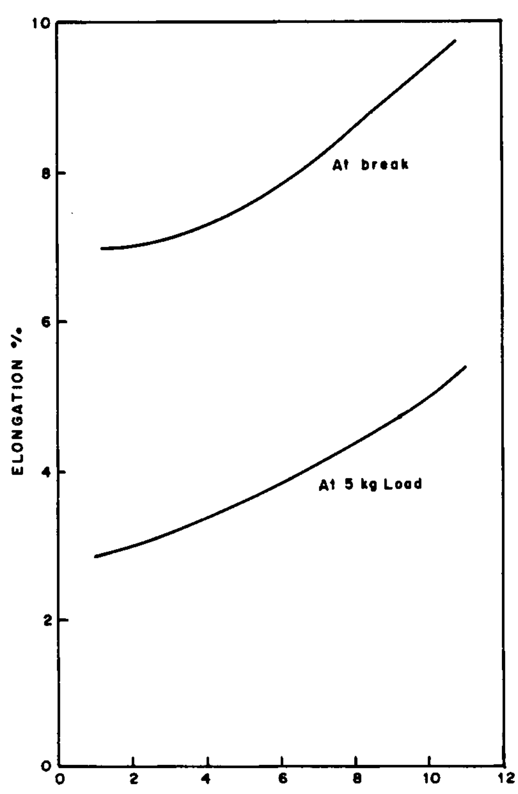


FIGURE 73 Variation of ultimate percentage elongation (130°C) with melt flow index (230°C, 2.16 kg) for polypropylene film tapes (taken from Krassig et al.[18]).

FIGURE 74
Variation of tenacity (130°C) with melt flow index (230°C, 2.16 kg) for polypropylene film tapes (taken from Krassig et al.[18]).

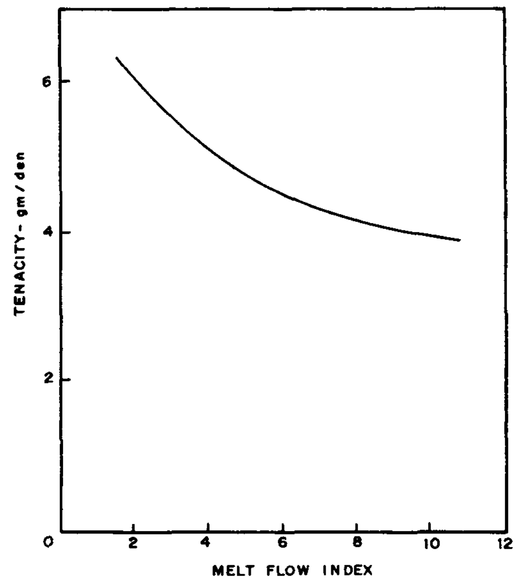
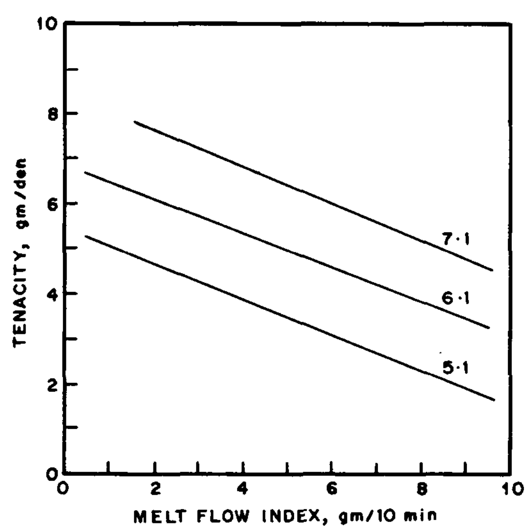


FIGURE 75
Variation of tenacity with melt flow index at different draw ratios for polypropylene film tapes (taken from Krassig et al.[113]).



4.5. Impact Strength

Impact strength could be regarded as the determination of flexural stress at a rapid rate of load increase. The impact strength is known to depend on both density as well as MFI.[109,114,115] It is highest for the lowest MFI material, but drops sharply as MFI increases and then fairly evens out at higher values as shown in Figure 79. Decreasing density also results in higher impact strength as can be seen from Figure 80. Higher density implies higher crystallinity and expectedly lower impact strength because crystallinity reduces the mobility of the segments of the adjacent amorphous polymer phase. Thus, in order to avoid brittleness, the higher density polymers must have a lower MFI.[114] Temperature changes affect lower MFI materials more as can be seen from Figure 81 wherein the differences in the impact strength behavior with temperature between materials of MFI of 1.0 and 3.0 are minor.

Figure 82 is a plot of melt flow index (under ASTM D2138 condition G: 392°F (200°C), 5 kg load) vs. impact strength (on injection molded 1/8 in. notched bars at 73°F). The product is a crystalline polystyrene containing a varying amount of SBR. This plot has been suggested for use as a quality control check for polystyrene incoming raw material or those reextruded two or more times.[32] The estimated results from this plot were found by Garcia-Barras[32] to be accurate within 10% when compared with the test results obtained by two independent laboratories. It is not unrealistic to expect similar plots when other melt flow index conditions and different impact bar sizes are used. Thus, MFI can be used as an effective quality control for impact strength.

The effect of applying load very rapidly permits measurement of the strength property related to impulses such as impact strength as discussed above. The work performed in effecting fracture has proved in practice to be a good measure of the impact resistance of a material and is generally represented as the area under the stress–strain curve for rapidly applied load. The amount of work that has to be performed is considerably affected by density and melt flow index[106]; it decreases as the density and melt flow index increases as can be seen from Figure 83.

4.6 Brittle Temperature

A typical catastrophic mechanical failure is brittle breakage under high-speed impact. When a tough rigid polymer is cooled to lower and lower temperatures, its impact strength tends to drop, and this decrease is often most marked near the glass transition temperature. When a flexible or a rubbery polymer is cooled, it reaches a temperature at which it becomes inflexible and often brittle. It is known that the brittle temperature is considerably higher than the glass transition temperature because T_g actually gives the mobility of much smaller molecular segments than those involved in embrittlement. With increasing molecular weight, the brittle temperature decreases because its increasing length of the molecules provides greater mechanical strength. This effect can be seen by plotting brittle temperature vs. MFI as given in Figure 84 for high density polyethylene.[116]

The brittle temperature depends to a great extent on the crystallinity. Crystallinity reduces the mobility of the segments and increases the temperature at which they have enough mobility to produce a glass transition. This results in an increase in the brittle temperature (Figure 85), which is higher for higher MFI.[117]

4.7. Tear Strength

In film mechanical strength properties, the tear strength is of immense importance and is related to MFI and density as shown in Figure 86. Elmendorf tear is inversely related to density as well as MFI. Cross direction tear strength is higher due to low cross direction orientation. In fact, orientation is more difficult to achieve in either direction with LLDPE resins than with conventional LDPE resins—because of the absence of long chain branching.

Kendall and Sherliker[118,119] have shown that milling carbon black into low density polyethylene results in a dramatic decrease in the tear strength of the system

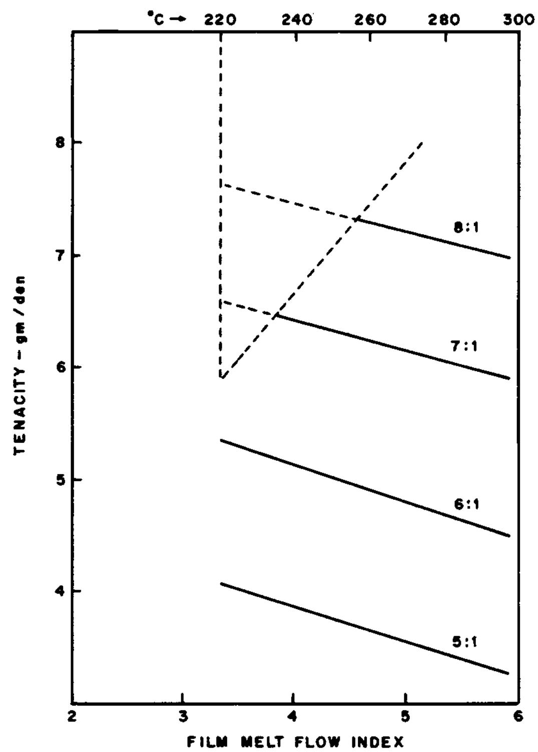


FIGURE 76 Variation of tenacity with melt flow index at different draw ratios and on the effect of extrusion temperature for polypropylene film tapes (taken from Evans[97]).

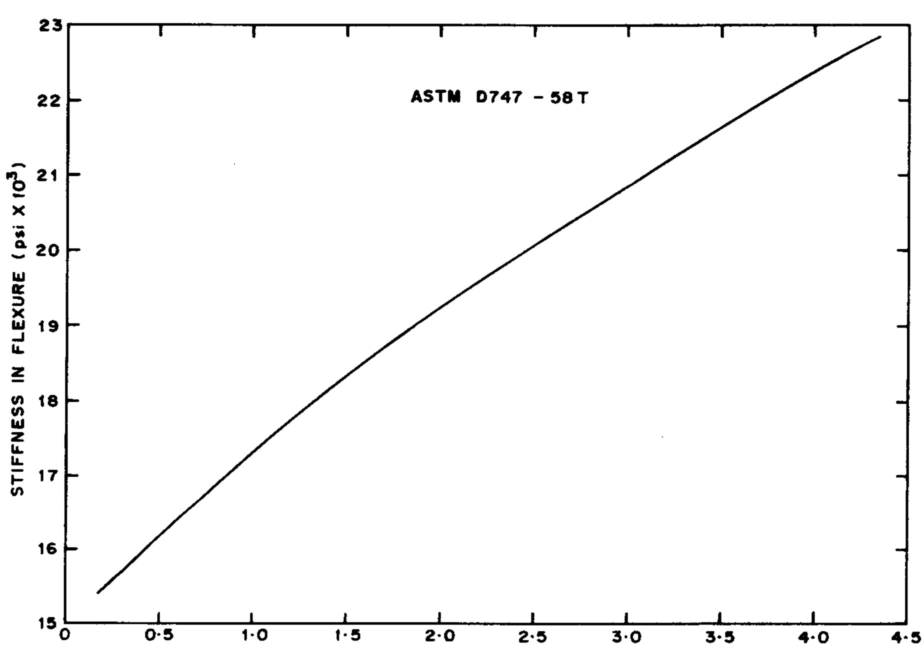


FIGURE 77 Variation of stiffness with melt flow index as per ASTM D 747-58T (taken from Ref. 107).

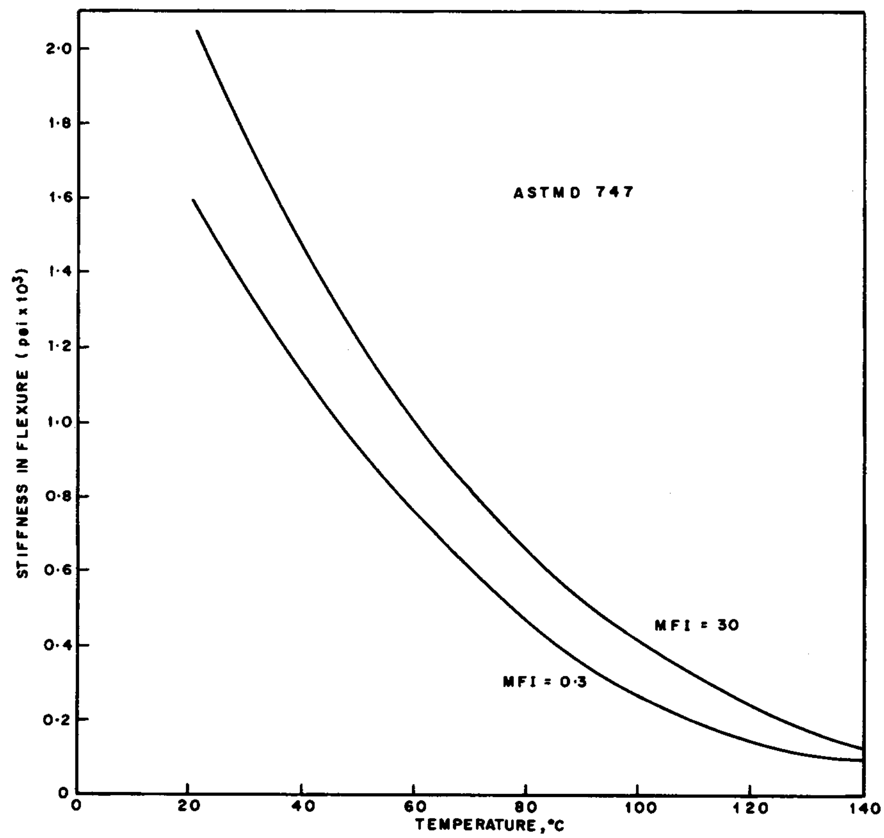


FIGURE 78 Variation of stiffness with temperature for two different melt flow index samples as per ASTM D747-587 (taken from Ref. 107).

despite the fact that the polymer molecules are bound to the filler in a layer 2–3 nm thick.[119] However, the catastrophic failure is due to the weak second interface between the bound surface layer and the remainder of the polymer matrix. Kendall and Sherliker[118] investigated the effect of molecular weight of the polymer in alleviating the brittleness of the filled system. They observed that low molecular weight polymers deteriorated badly in the presence of 10% volume fraction of the filler while the high molecular weight materials remained tough even up to a 25% volume fraction level. The transition in behavior due to the molecular weight of the polymer can be shown more obviously by plotting the tear strength at a particular volume fraction against the melt flow index as shown in Figure 87. The tear strengths on the graph have been normalized by the value of the unfilled polymer to remove the effect of molecular weight on polymer toughness and thereby isolate the influence of molecular weight on filler behavior. In Figure 87, it can be seen that the polymer becomes embrittled when filled with 10% filler around MFI equal to 1. The nature of the curve and the transition is independent of the chemical type of the filler as shown by the results for carbon black as well as silica of similar size. Figure 88 contrasts the behavior of high and low density polyethylene as well as ethylene–vinyl acetate copolymer filled with 10% silica. Above MFI equal to 0.5, both HDPE and LDPE show similar embrittlement. However, whereas filled LDPE is not toughened at high molecular weights, filled EVA shows slight reinforcement while filled HDPE shows considerable reinforcement. In the case of EVA, the transition to poorer properties occurs at such high melt flow indices that all production grades are more often than not likely to show good toughness when filled.

The manner in which small amounts of colloidal filler destroy the cohesion of low molecular weight polyethylenes was thought by Kendall and Sherliker[118] to be similar to the phenomenon of environmental stress cracking, in which minor amounts of detergents or alcohol produce inordinate embrittlement of polymers, especially of low molecular weight.[120] In order to check their hypothesis, a drop of isopropanol was placed at the tip of the tear, and the fall in force at constant speed was measured. The results were plotted as a function of melt flow index as shown in Figure 89 wherein the transition in the cracking behavior was found to closely resemble the colloidal filler effect.

4.8. Environmental Stress Cracking

Environmental stress cracking (ESC) is the name given to a phenomenon by which a polymer under high stresses may crack in contact with certain active environments such as detergents, fats, and silicone fluids. There are a number of chemical and physicochemical effects involved in any given ESC phenomenon.[121] The susceptibility of a polymer to ESC decreases rapidly as the MFI is decreased and hence can be tracked quite sensitively through the MFI test as shown in Figure 90 taken from Pelagatti and Baretta,[122] which studies the cracking of polyethylene in weak organic detergents. Pelagatti and Baretta[122] concluded that water solutions of detergents are more effective for higher molecular weight whereas anhydrous detergents and small molecule organic liquids would attack preferentially lower molecular weight resins. The effect of molecular weight in terms of long-term stress crack resistance is shown in Figure 91 for low density polyethylene.[123] Experimental studies indicate that the decrease in the resistance to cracking with MFI is primarily due to extraction or leaching of the low molecular weight fraction.[123] The effect of crystallinity and MFI on environmental stress cracking is shown in Figure 92 and found to vary inversely with these two parameters.[115]

In practice, it is important not to use polymers of high MFI for applications in which they are severely stressed, especially in contact with active environments. Figure 93 shows an application in which a severe external stress is applied in service in contact with an active environment. In this case, it can be seen that a low MFI is essential and the use of the

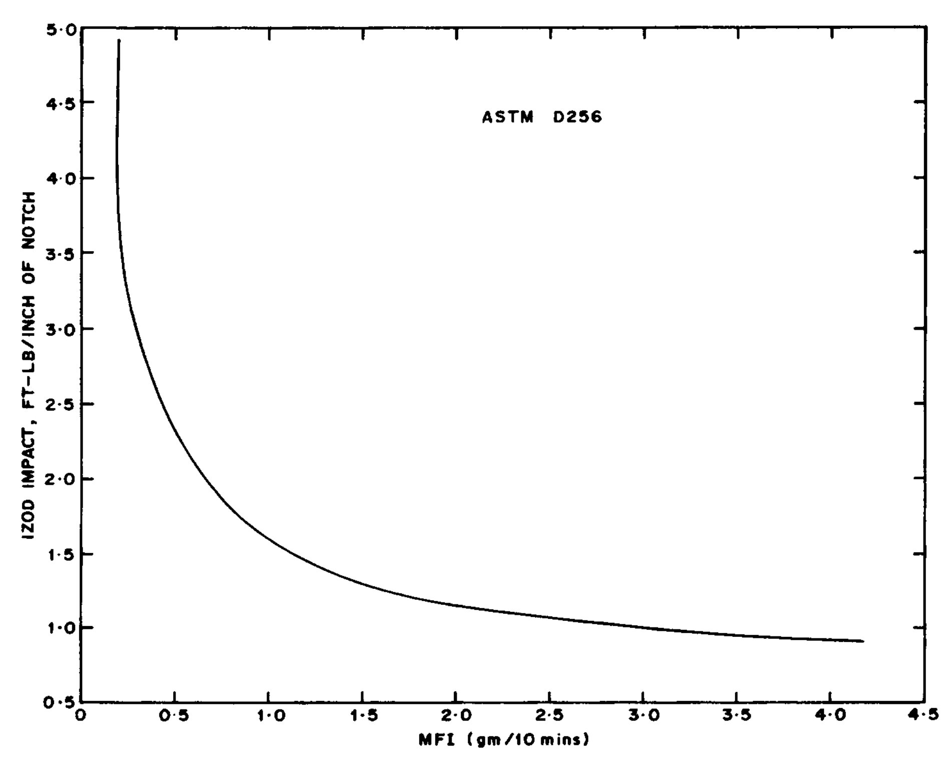


FIGURE 79
Variation of Izod impact strength with melt flow index as per ASTM D256 (taken from Ref. 107).

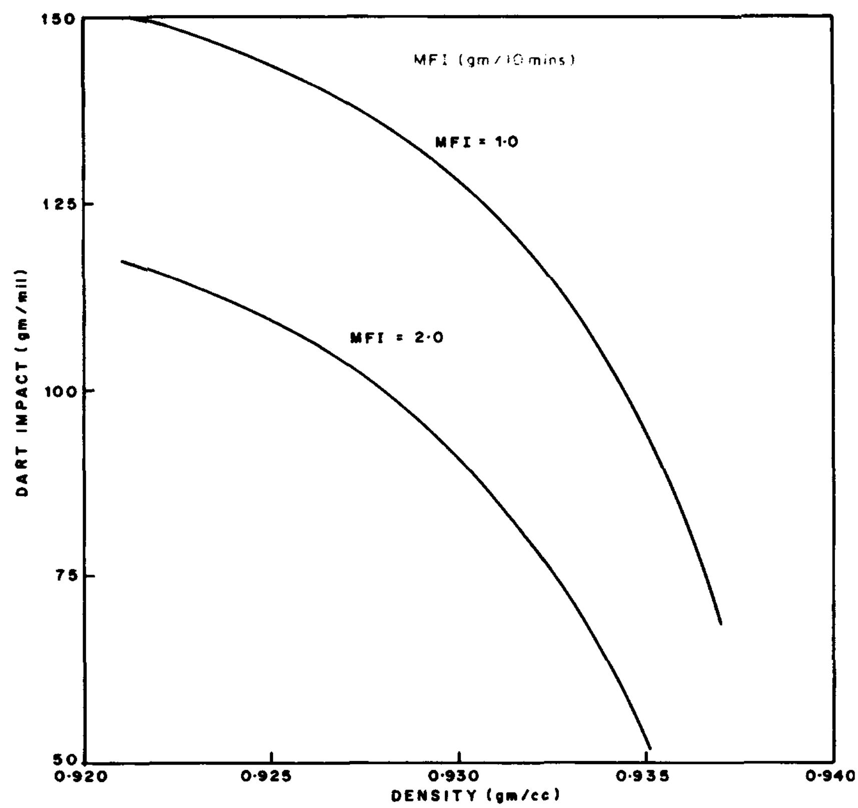


FIGURE 80
Variation of Dart impact strength with melt flow index and density (taken from Ref. 109).

best possible molding conditions will not prevent a high MFI polymer from cracking. Figure 94 shows the same two polymers, again subjected to an active environment, but in an unstressed application. Both the polymers were molded at a melt temperature of 200°C, which was suitable for the polymer of high MFI but not for the low MFI. Consequently, the molding of low MFI failed whereas that made from high MFI remained perfectly satisfactory.[114] This was because the material with lower MFI had a higher level of frozen-in strain, thereby leading to warpage.

The resistance to chemicals of any polymer can also be determined by the swelling test. Swelling is related to a decrease in strength and deterioration of the properties. This can be easily checked through MFI determination using a plot of the type shown in Figure 95 taken from Ref. 106.

4.9. Thermal Effects

Stress cracking as discussed above need not only be due to an active environment. Often a thermal stress can lead to cracking. Heat shock failure is also seen to relate rather linearly with MFI as given by Figure 96 taken from Ref. 106.

The effect of temperature is also important during film and fiber processing, for example, in determining thermoshrinkage. Low MFI enhances thermoshrinkage and there exists an optimum MFI wherein the % shrinkage is minimal as can be seen from Figure 97 for polypropylene films taken from Krassig et al.[18]

Polybutylene polymers' slow crystallization rates are helpful in forming hot melt adhesives with long open times.[124] Besides the hot melt viscosity, lap shear strength, T-peel, and open time, the shear adhesion failure temperature (SAFT) of the bonded substrate is an important property for evaluating the effectiveness of the adhesive. The polymer MFI plays an important part in determining the surface temperature at which shear adhesion failure could occur, as can be seen from Figure 98. The top curve represents the SAFT test results with a 0.5-kg load while the bottom curve shows test results with a 1-kg load. Some drop in service temperature is found as polymer MFI is increased. At the higher load, the magnitude of the drop is about 5°C over the 10–100 MFI range. At a heavier load (1 kg), the effect is more pronounced—about an 11°C drop over the same MFI range. Though the service temperature does not vary strongly as a function of MFI, Figure 98 indicates that high MFI polybutylene polymers would be preferable for preparing useful adhesive compositions.

4.10. Gloss and Clarity

The gloss of a molding article is often assessed both visually and by measuring the light reflected from the surface of the moldings under standard conditions. The effect of MFI on gloss is very significant, as can be seen from Figure 99. The higher the MFI, the lower is the temperature at which high gloss moldings can be produced.[114]

In the case of extruded films, too, gloss and clarity are directly related to MFI. Figure 100 shows the effect of MFI upon the gloss of extruded high density polyethylene film. Thus, it can be seen that low molecular weight basically favors high clarity and gloss.[125] It is also known that narrow molecular weight distribution favors high clarity while a broad distribution favors high gloss. Thus by proper balance of the molecular weight distribution and MFI, desired level of clarity and gloss can be achieved.

5. CONCLUSIONS

Through the present review of existing literature, it has been shown that MFI can no longer be considered as a mere quality control rheological parameter. It is versatile in its utility and has been shown to correlate very effectively with a number of fundamental parameters involved in the various stages of manufacturing the polymer to the final finished product.

During polymer manufacture, the quality of the material can be monitored by the reaction temperature, the catalyst activation temperature, the reaction pressure, etc. These important parameters hold a definite relationship with the MFI of the resulting polymer and hence can be adjusted quite easily to obtain the polymer grade of interest through MFI measurement. The specification of the polymer grade includes fundamental structural properties such as molecular weight, molecular weight distribution, branching, glass tran-

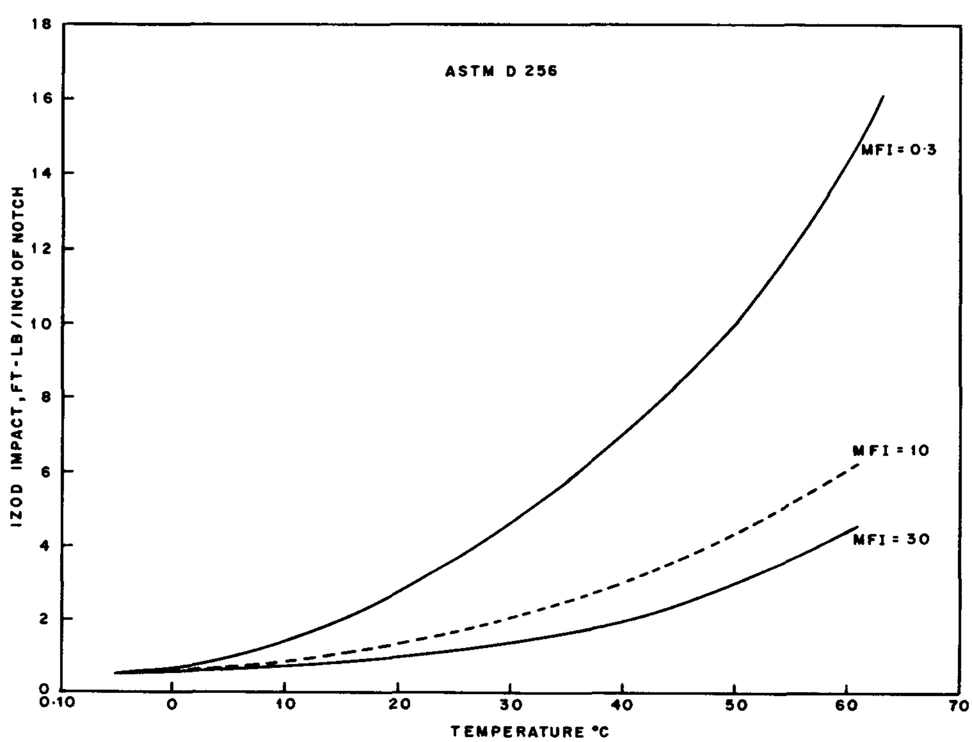


FIGURE 81 Variation of Izod impact strength with temperature for two different melt flow index samples as per ASTM D256 (taken from Ref. 107).

FIGURE 82
Variation of impact strength (1/8 in. notched bar at 73°F) with melt flow index (200°C, 5 kg) for crystalline polystyrene (taken from Garcia-Borras[32]).

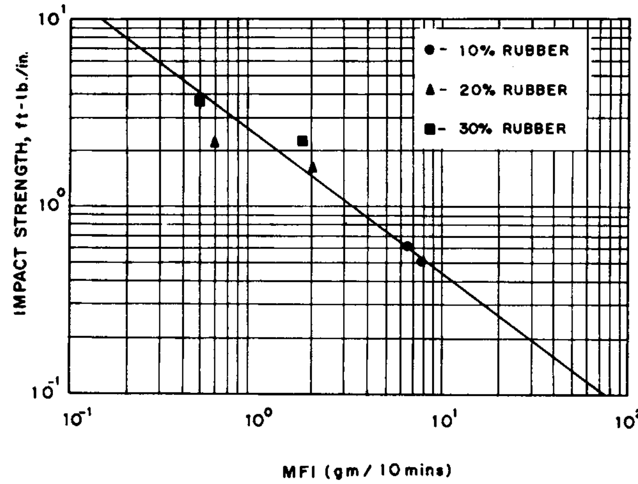
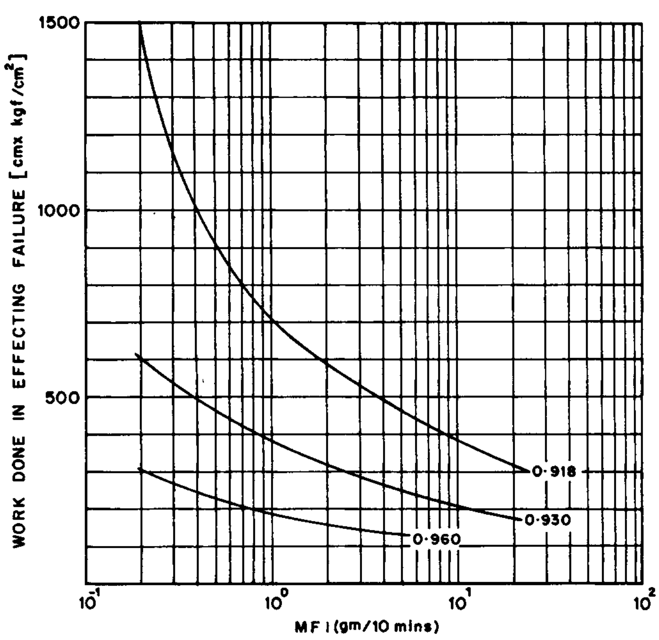


FIGURE 83
Variation of work done in affecting failure with melt flow index and density of low density polyethylene (taken from Ref. 106).



sition temperature, zero shear viscosity, etc., all of which have also been shown to relate effectively with MFI.

The processes involved in the fabrication of polymer products include injection molding, blow molding, compression molding, extrusion, thermoforming, etc. In all these processes, the polymer melt is subjected to shearing and elongational flow. A knowledge of the entire rheological characteristics is a must for process optimization and product quality control. MFI has been shown to relate shear viscosity, normal stress difference, die swell, and elongational viscosity and hence attains great importance during polymer processing. It has also been demonstrated that various important processing parameters can be estimated through MFI. For example, calculation of the minimum pressure drop during cavity filling and the minimum clamping force to prevent mold opening during injection molding, the compaction force during compression molding, the pressure losses through dies of complex cross sections during extrusion as well as the viscous heat dissipation during processing can be easily obtained just through the knowledge of MFI. Pre- and post-processing operations like crosslinking and curing can be monitored through MFI measurements, besides degradation, stabilization, and aging of the polymer.

Important product properties like tensile, flexural, tear, and impact strength as well as tenacity and ultimate elongation have been shown to relate to MFI. It has also been shown that with a proper balance of MFI and processing temperature, a desired level of clarity and gloss can be achieved. Further, the envi-

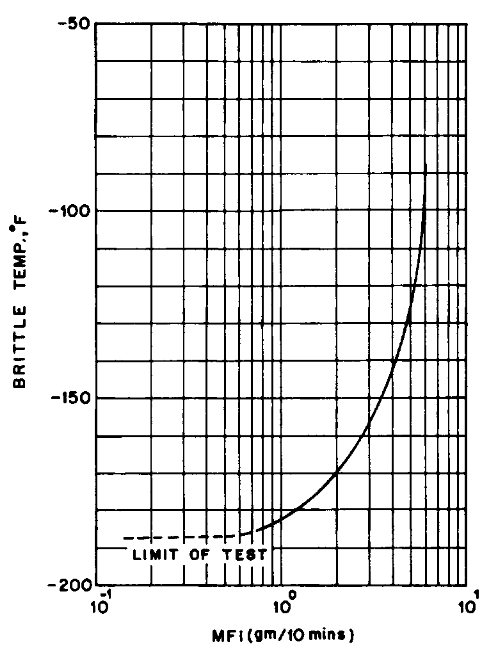


FIGURE 84 Variation of Brittle temperature with melt flow index of high density polyethylene (taken from Ref. 116).

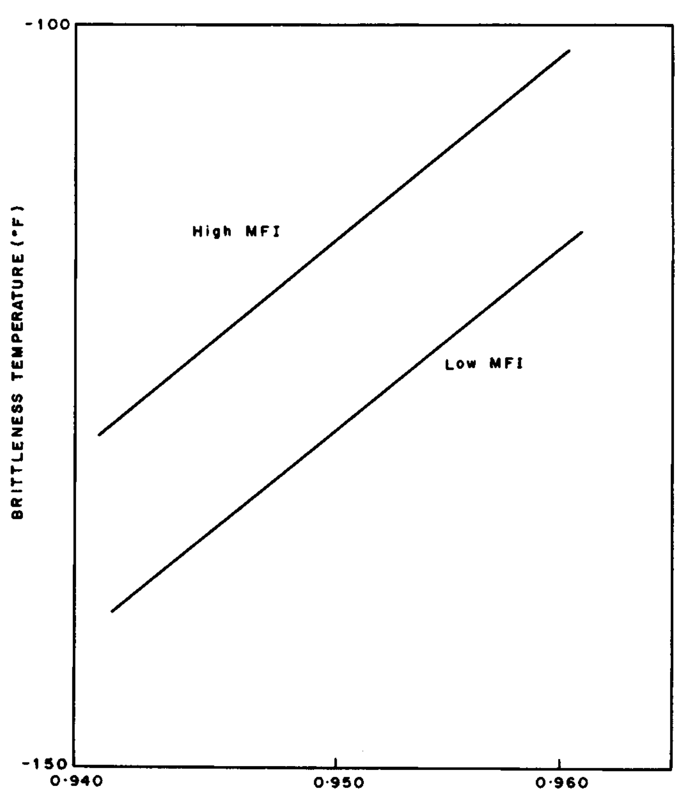


FIGURE 85 Variation of brittle temperature with density and melt flow index (taken from Ref. 117).

FIGURE 86
Variation of Elmendorf tear with density and melt flow index (taken from Ref. 109).

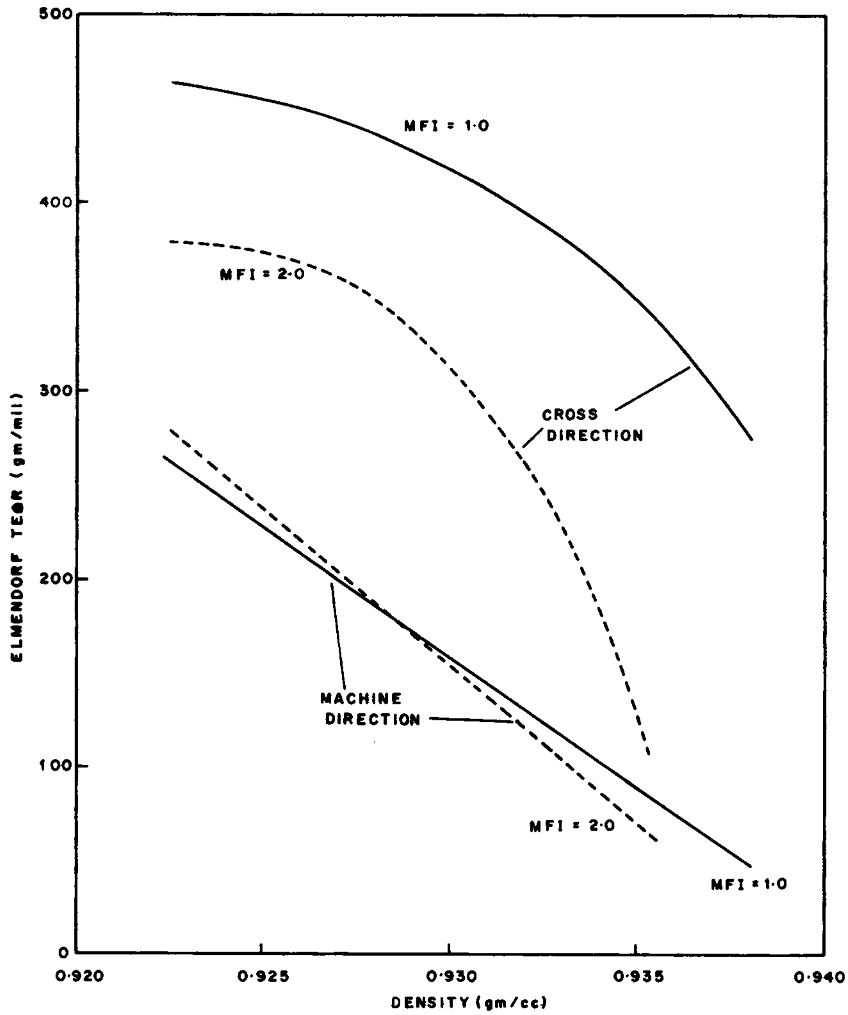
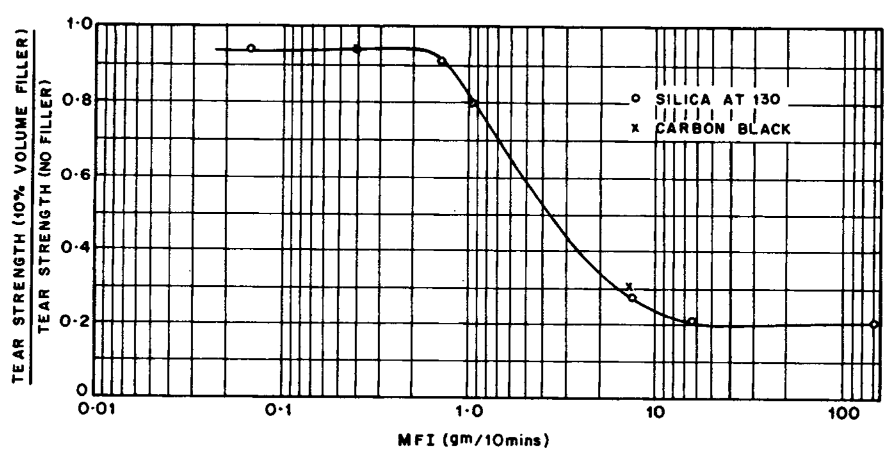


FIGURE 87
Variation of tear strength with melt flow index showing the transition between rough and embrittled behavior for low density polyethylene (taken from Kendall and Sherliker[118]).



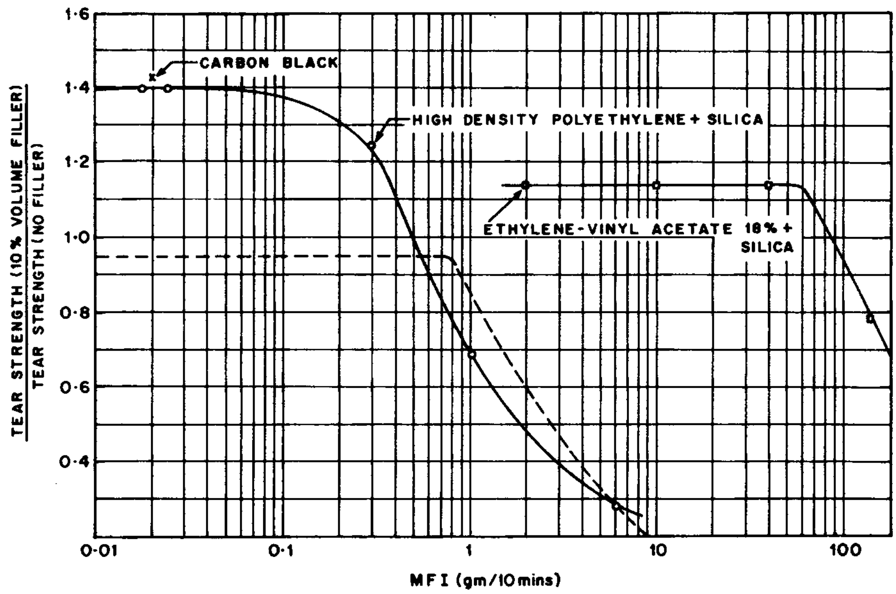


FIGURE 88 Variation of tear strength with melt flow index showing the tough–brittle transition for low density polyethylene, high density polyethylene, and ethylene–vinyl acetate copolymers at 10% volume fraction of silica (taken from Kendall and Sherliker[118]).

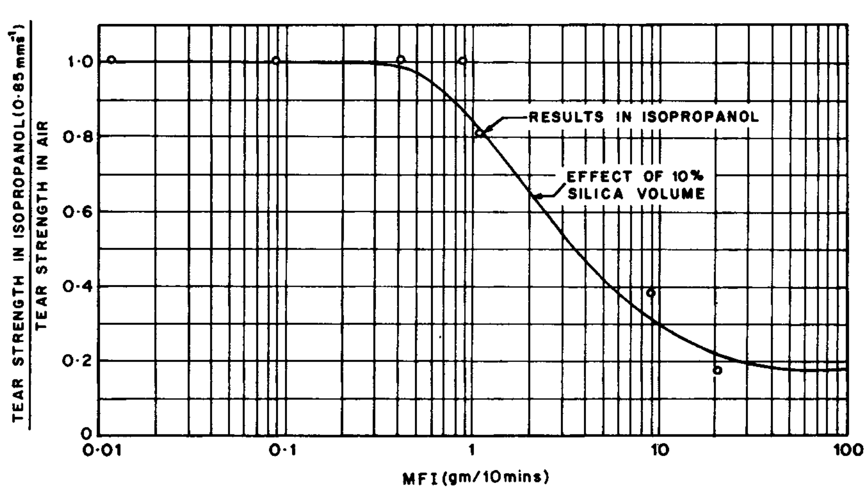


FIGURE 89 Variation of tear strength with melt flow index showing the link between the effects of colloidal silica and isopropanol on cracking of low density polyethylene (taken from Kendall and Sherliker[118]).

ronmental stress cracking susceptibility can also be adjudged through MFI values.

In summary, it has been shown that MFI is a parameter useful at all stages right from polymer synthesis, polymer processing, and final product performance. No other known parameter has such a large gamut of utility. It is important that the potential of MFI is realized by raw material manufacturers, polymer processors, and product development groups, as it truly is more than just a quality control rheological parameter.

6. A FEW WORDS OF CAUTION

Having proved that MFI is sensitive to a number of fundamental parameters in polymer synthesis, processing, and final product properties, it is absolutely essential that the measurement itself should be performed with extreme care so that the possible errors as discussed in Sec. 1 (Part I) are minimized. In order to emphasis this, we are giving below a few caution points which must be borne in mind when performing the MFI measurement.

FIGURE 90
Effect of anhydrous detergent and its water solutions on cracking times of polyethylene of various melt flow indexes (taken from Pelagatti and Baretta[122]).

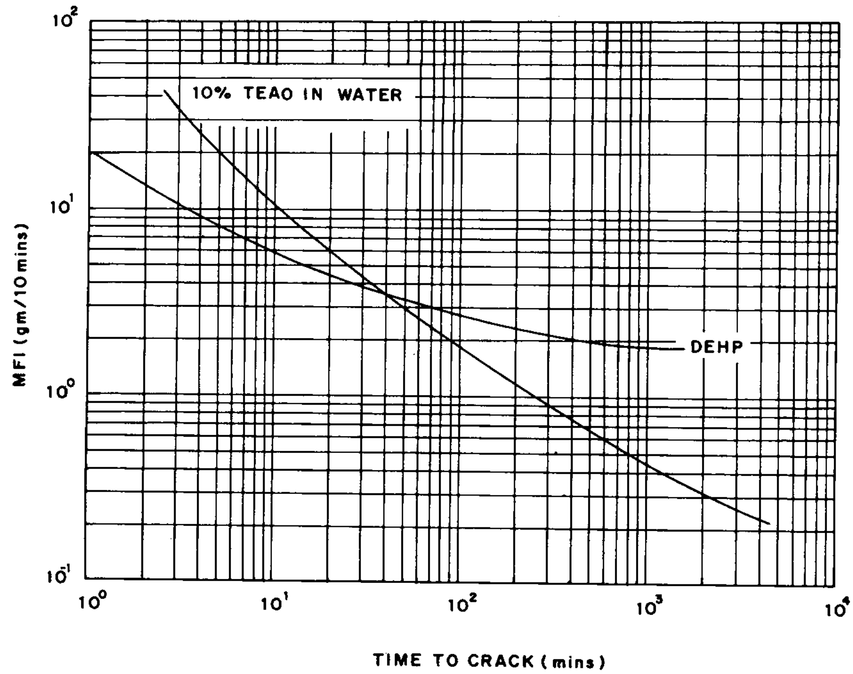
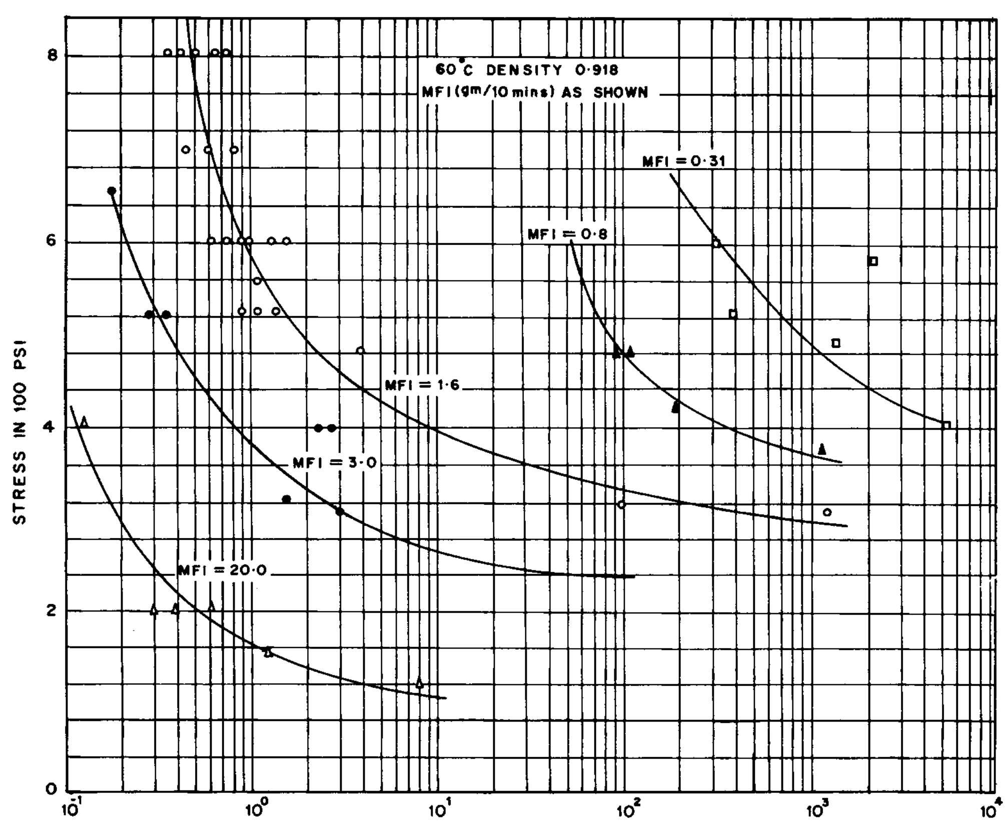


FIGURE 91
Effect of melt flow index upon the stress crack resistance of low density polyethylene (taken from Baer[123]).



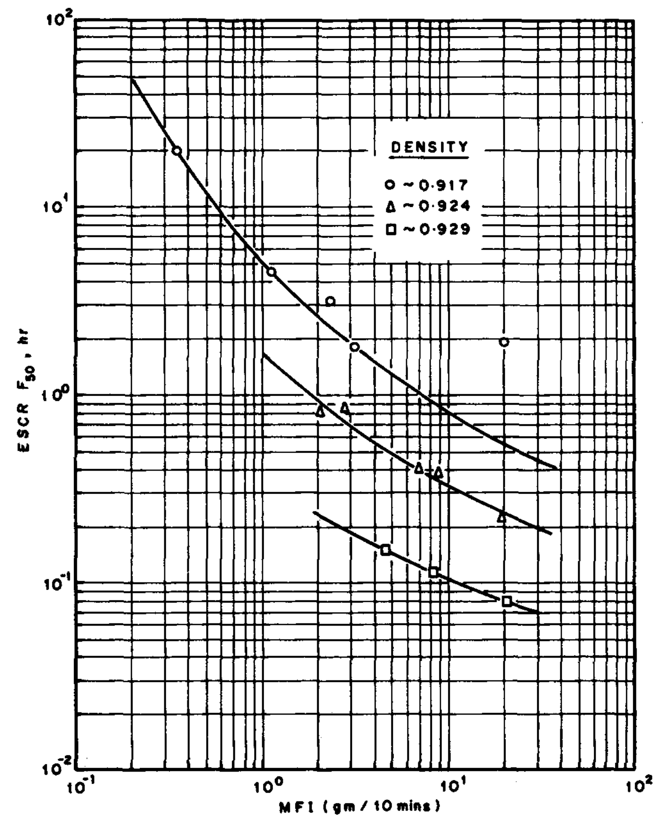


FIGURE 92
Effect of crystallinity and melt flow index upon time of failure in stress cracking of polyethylene (taken from Ref. 115).

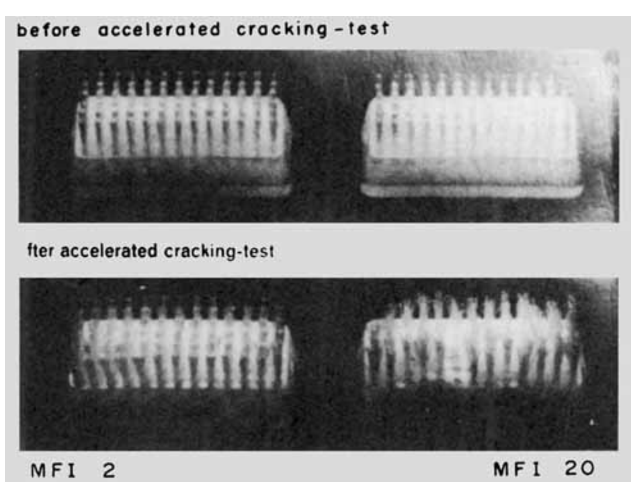


FIGURE 93
Illustration of the effect of severe external stress applied in service during contact with an active environment (taken from Dunkley[114]).

- The cylinder, piston and capillary of the melt flow indexer should be meticulously cleaned before measurement
- The measurement should be done strictly under the conditions specified in any of the standards—ASTM, DIN, BS, or ISO
- During charging of the sample, the packing should be uniform and without air gaps
- Delays between charging and packing must be kept to a minimum
- The piston height during all measurements should be kept between 50 and 20 mm
- For polymers sensitive to oxygen and moisture, the melt flow indexer should be appropriately modified so that dry nitrogen purging can be done
- For highly filled polymeric systems, higher loads must be employed and the capillary diameter modified if necessary, depending on the size and shape of the fillers
- In case two polymers of the same generic type are

FIGURE 94
Illustration of the effect of an active environment in the absence of stress during service (taken from Dunkley[114]).

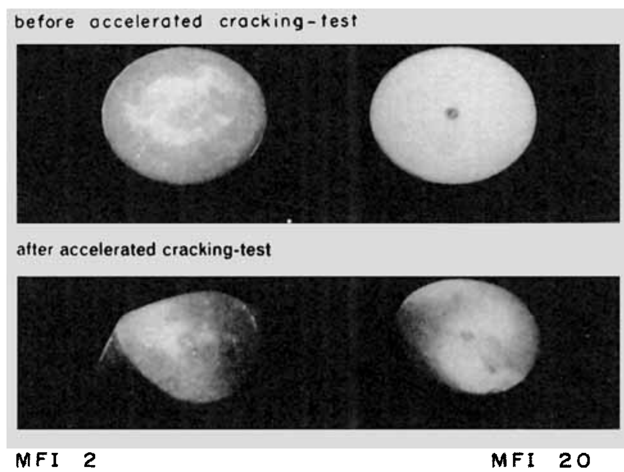
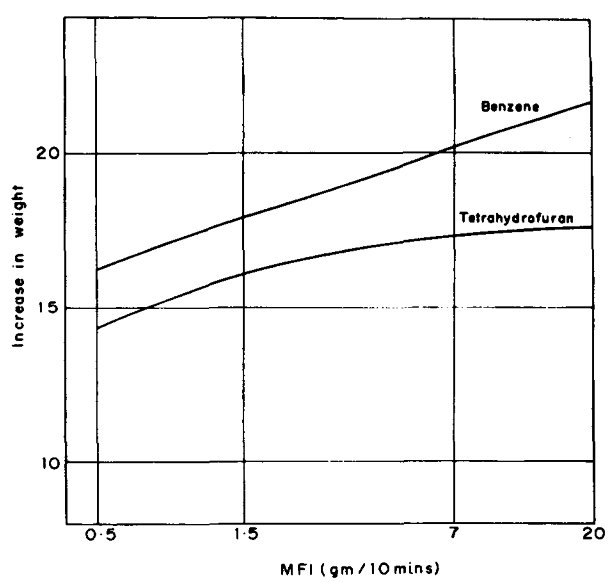


FIGURE 95
Swelling of polyethylene of various melt flow indexes in active environment (taken from Ref. 106).



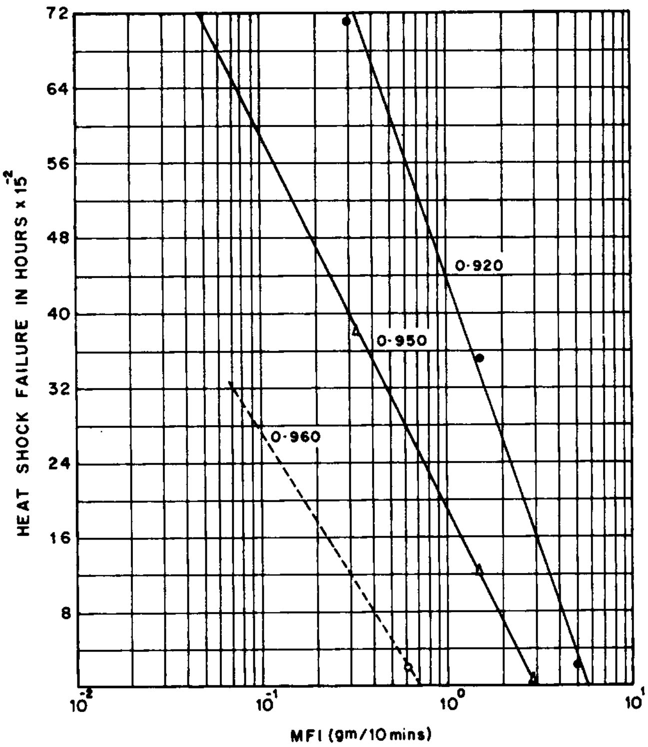


FIGURE 96
Effect of density and melt flow index upon thermal stress cracking of polyethylene wire insulation at 70°F (taken from Ref. 106).

found to give identical MFI values but show large differences in other properties, then the load condition must be altered until the MFI values are significantly different and sensitive

Any errors in MFI measurement are likely to depict amplified derogatory results when correlating with other fundamental properties. Hence it is worth using microprocessor-controlled melt flow indexers which are now available in the market, so that the highest order of accuracy and reliability can be maintained.

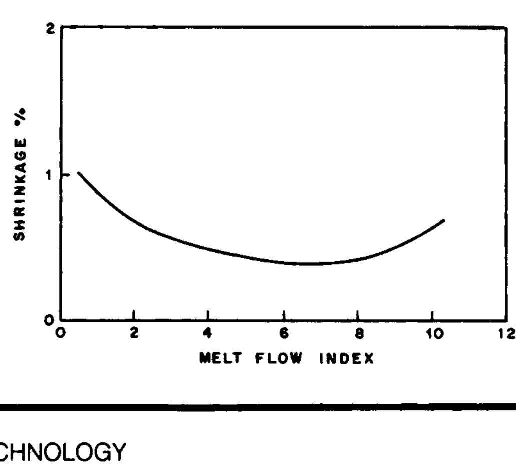


FIGURE 97
Variation of percentage shrinkage (130°C) with melt flow index (230°C, 2.16 kg) for polypropylene film tapes (taken from Krassig et al.[18]).

FIGURE 98
Relationship between surface temperature and melt flow index for a polymer/resin blend in hot melt adhesive formulation (taken from Korcz[124]).

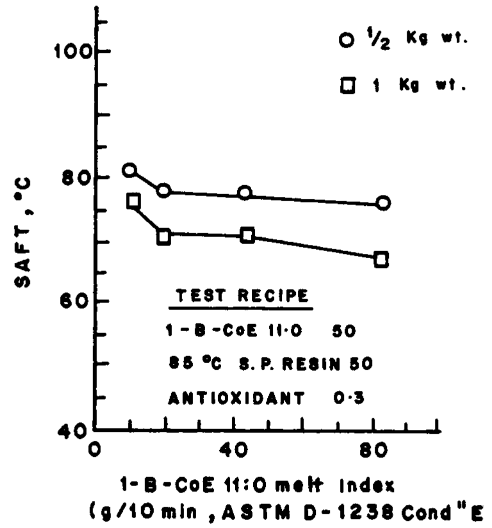
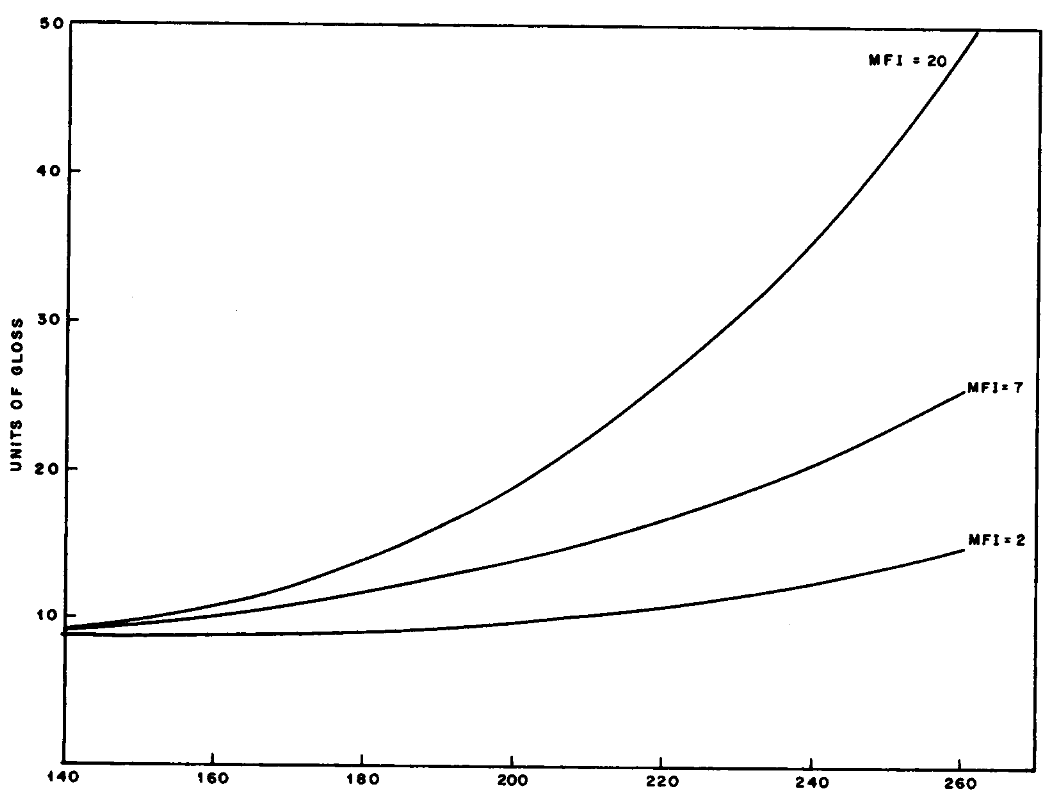


FIGURE 99
Effect of molding temperature and melt flow index on the gloss of moldings (taken from Dunkley[114]).



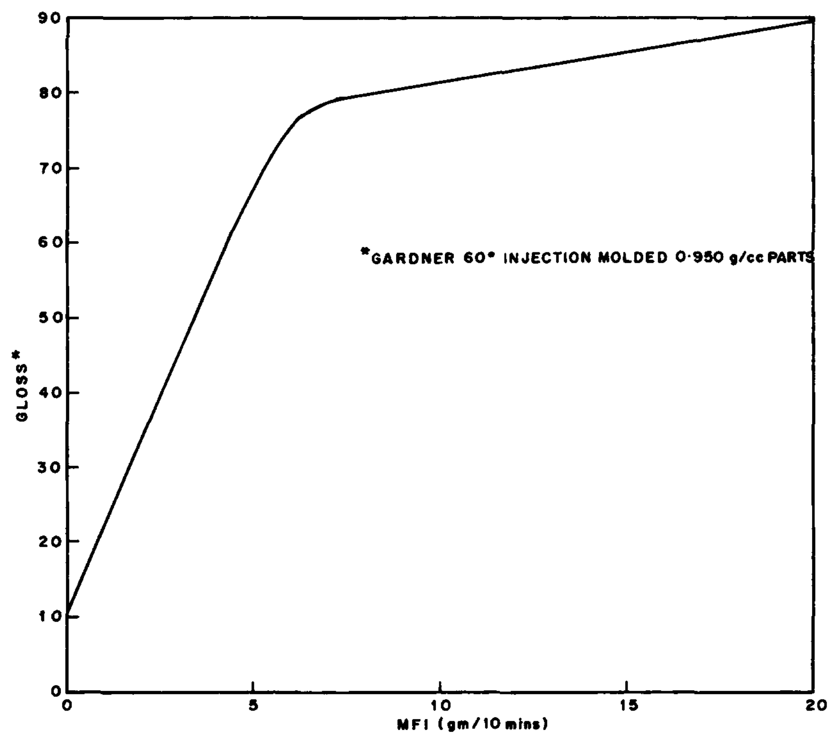


FIGURE 100 Effect of melt flow index upon the extruded high density polyethylene film (taken from Ref. 125).

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