

PDL HANDBOOK SERIES

Polypropylene

The Definitive User's Guide and Databook

**Clive Maier
Teresa Calafut**

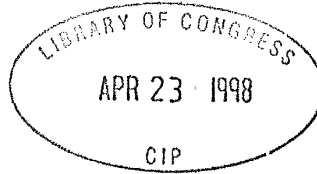


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TP 1180
P68M35
1998

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ISBN 1-884207-58-8

Library of Congress Card Number 97-076233

Published in the United States of America, Norwich, NY by Plastics Design Library a division of William Andrew Inc.

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2.3 Effect of morphology on characteristics of polypropylene

Due to the ordered crystal structure, semicrystalline polymers generally have high strength and are more chemically resistant than amorphous polymers. Semicrystalline materials are more opaque and can be used at higher temperatures, while amorphous materials are generally more transparent and have greater toughness and ductility. [772]

2.3.1 Melting point

The crystalline structure of a solid semicrystalline polymer disappears at the melting point, T_m , when the material undergoes a phase change from solid to liquid. At the melting point, physical properties of the material, such as density, refractive index, heat capacity, and transparency, change abruptly as the material becomes a viscous liquid. Melting points are commonly measured using differential scanning calorimetry (DSC). [769, 772]

The melting point of a polymer varies with the amount of crystallinity. A perfectly isotactic polypropylene resin has a theoretical melting point of about 171°C (340°F); melting points of commercial isotactic resins can range from 160–166°C

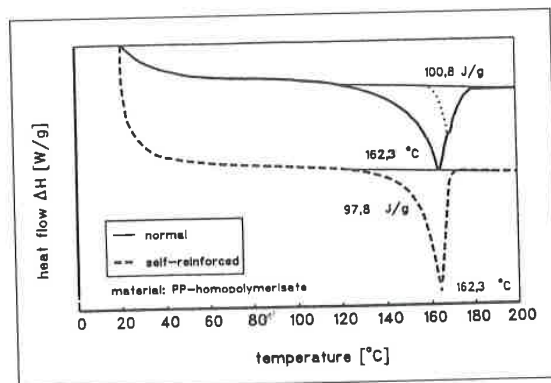


Figure 2.4 A differential scanning calorimetry (DSC) melting scan of injection molded polypropylene. In DSC, a polypropylene sample is heated, along with a reference material, and the energy required to maintain both materials at the same temperature is monitored as the surrounding temperature is increased. Heat is absorbed by polypropylene as it melts, resulting in an endothermic peak; the melting temperature given in the literature is the highest point of the peak. DSC scans of polypropylene frequently show more than one peak, due to polymorphism or crystalline changes that occur during the heating cycle. Bottom scan is normally processed polypropylene. Top scan is polypropylene with an oriented crystallization, produced with a high injection rate and pressure. A small high temperature shoulder indicates the presence of more than one morphological form. Heating rate was 10 K/min (10°C/min; 18°F/min). [789]

(320–331°F) due to atactic material and noncrystalline regions. Melting points decrease dramatically with lower crystallinity; a syndiotactic polypropylene resin with a crystallinity of 30% melts at approximately 130°C (266°F). [693]

Polymers generally melt over a narrow temperature range rather than at a distinct point. In a resin with a particular crystallinity, variations in chain lengths produce lamellae of varying thicknesses, which melt at slightly different temperatures. The melting point given for a polymer is generally the temperature at the highest point of the peak on a DSC scan (Figure 2.4) [770, 730]

The high melting point of polypropylene provides resistance to softening at elevated temperatures. Standard grades of polypropylene can withstand continuous service temperatures of over 107°C (225°F) and over 121°C (250°F) for short periods of time. The superior heat resistance of polypropylene makes it suitable for sterilization by autoclaving at temperatures of over 121°C (250°F) and for hot-fill applications. Heat resistance is lowered for resins with lower melting temperatures. [780, 696, 642, 693]

2.3.2 Glass transition

Amorphous regions of the polypropylene resin undergo a glass transition (T_g) at a temperature between -35 and 26°C (-31 and 79°F), depending on the measurement method, heating rate, thermal history, and microstructure. The glass transition temperature is related to the amount of free volume in a polymer. Molecules and segments of polymer chains above the glass transition temperature vibrate and move in noncrystalline polymer regions. Motions include diffusion, rotation about bond axes, and translation under mechanical stress. At the glass transition temperature, free volume is restricted, and only low amplitude vibrations can occur. This movement continues down to absolute zero, at which point all movement ceases. Resins with lower molecular weight generally have lower glass transition temperatures due to increased free volume at the ends of the polymer chain and lower degrees of chain entanglement. [772, 693]

Normal use temperatures of polypropylene are generally between the glass transition and melting temperatures, so that strength and stiffness from the crystalline region are combined with toughness of the amorphous “tie points”. The low temperature ($\leq 5^\circ\text{C}$; 40°F) brittleness of polypropylene is

due to its relatively high glass transition temperature; as the temperature drops, approaching the glass transition temperature, the resin becomes increasingly brittle, and impact resistance becomes negligible. [693]

2.3.3 Mechanical properties

Mechanical properties of polypropylene are strongly dependent on its crystallinity. Increasing crystallinity increases stiffness, yield stress, and flexural strength but decreases toughness and impact strength. The secant flexural modulus at 1 % displacement can range from 2067–2412 MPa (300,000–350,000 psi) for polypropylene with ultra-high crystallinity but decreases to 1378–1654 MPa (200,000–240,000 psi) for general purpose polypropylene of lower crystallinity. In experiments with α -form, isotactic polypropylene, increasing crystallinity increased the growth of cracks and decreased fracture toughness. [693, 785]

Spherulite size affects the strength and ductility of the resin. In studies of fatigue strength, different crystallization temperatures were used to produce spherulites of different sizes (37, 54, and 61 μm); higher crystallization temperatures produced larger diameter spherulites. In fatigue fracture tests investigating the effects of a sharp initial crack, the resin with smaller spherulites exhibited more ductile behavior, with more damage ahead of the crack tip and crazing in the surrounding region. Larger spherulite sizes resulted in more brittle behavior, with less material damage. Slow crack growth changed to unstable crack growth at a critical crack length; this length decreased as spherulite size increased. [778]

In investigations of the fracture behavior of nucleated and non-nucleated isotactic polypropylene using the J-integral method, fracture toughness decreased with increasing crystallinity, for both nucleated and non-nucleated resins. An increase in spherulite size from 25 to 100 μm decreased fracture toughness and increased brittleness in non-nucleated polypropylene. Nucleated polypropylene, with a spherulite diameter of 2 μm , exhibited increased crystallinity and brittleness, and cracks grew faster after initiation, leading to early failure. It was proposed that this brittleness was due to the higher crystallization temperature of nucleated polypropylene. Higher crystallization temperatures increase the lamellar thicknesses and decrease tie-molecule concentrations of amorphous regions; since tie molecules provide mechanical continuity between spherulites,

lower tie molecule concentrations would make the amorphous regions of the polymer more susceptible to crack propagation. [785]

2.3.4 Haze

Transparency in semicrystalline polymers is directly related to the crystallinity. Spherulites are much larger than the wavelength of visible light (0.4–0.7 μm), and the refractive index of crystalline regions is higher than that of amorphous regions. As light rays pass from amorphous to crystalline regions, they encounter the large spherulites, resulting in light scattering; as a result, transparency is lower, and haze is produced. Due to their noncrystalline structure, amorphous materials have lower levels of haze than semicrystalline materials, and a decrease in crystallinity in a semicrystalline polymer enhances the clarity. Excessive reductions in crystallinity can result in unacceptable reductions in strength, stiffness, and resistance to softening, so a compromise must be reached that is appropriate for the application. [693, 780, 786, 774]

Nucleating agents such as dibenzylidene sorbitols reduce spherulite size to below a level that scatters visible light, resulting in a dramatic reduction in haze. Spherulite size distribution is also reduced. A higher concentration of nucleating agent can result in greater clarity. Clarity can be optimized by using a resin with low crystallinity with a nucleating agent added and by rapidly cooling the molten polymer to accelerate the crystallization rate. [693, 786]

2.3.5 Sterilization

Polypropylene resins with lower crystallinity exhibit greater resistance to embrittlement resulting from gamma radiation sterilization. Exposure to gamma radiation increases the susceptibility of the material to oxidation. Free radicals and ions, highly reactive chemical species containing free (nonbonded) electrons, are created and are then trapped in the crystalline structure of the polymer. These free radicals and ions promote covalent bond disruptions in the main polymer chain or in pendant methyl groups. The resulting chain scission and/or crosslinking of adjacent polymer chains increases the brittleness of the material. [55, 799, 798]

Radiation resistance is associated with the amount of free volume in the polymer structure, which allows greater mobility of the polymer chains. Amorphous or smectic phases have lower densities

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