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## Determination of fracture toughness of propylene polymers at different operating temperatures



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#### ABSTRACT

The influence of the temperature on the fracture parameters of three types of polypropylenes under static loading conditions has been analyzed. Single-phase polypropylene homopolymer and random ethylene-propylene copolymer were tested slightly above their glass transition temperatures (within the ductile-to-brittle transition region), and well below and above them; heterophasic ethylene-propylene block copolymers were tested on the range of temperatures from above the glass transition temperature of propylene matrix to below the glass transition temperature of the elastomer phase. Attending to the mechanical response, suitable Fracture Mechanics approaches were used. Fracture toughness increased with temperature for single-phase polymers but block copolymers were not temperature sensitive for the range of temperatures above the glass transition temperature of the elastomeric phase despite their variation on the mechanical properties.

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#### 1. Introduction

Low manufacturing costs, ease of recycling and possibility of tailoring properties are some of the reasons why isotactic polypropylene, PP, has been enjoying the fastest growth in consumption since its discovery [1]. There are three typical types of PP: homopolymers, random copolymers and block (or impact/heterophasic) copolymers. The copolymers are usually of ethylene and butane. Random copolymers usually contain up to 6 wt% of ethylene or other comonomers inserted randomly within the chain, reducing the crystallinity and the melting point by introducing irregularities into the chain. They are used where clarity, lower melting point or lower modulus is desirable. The low content in ethylene provides a continuous and monophase structure with one single glass transition temperature,  $T_{\rm g}$ , slightly below that of the PP homopolymers because of the presence of ethylene. Block copolymers, also known as impact or heterophasic copolymers, contain up to 40% ethylene–propylene rubber (EPR), uniformly dispersed within the homopolymer matrix [1–3]. They show two distinct glass transition temperatures, associated with the PP matrix, ~23 °C, and with the elastomeric phase, ~ -50 °C. As implied in the name, those copolymers are used where impact strength is important, especially at low temperatures [1]. The toughening mechanism is related to the generation of many small crazes, which absorb significant energy, and the interruption of a propagating fracture in the matrix [4–11]. This mechanism operates above the glass transition temperature of the dispersed phase, and its effectiveness is determined by one side, the nature of the elastomeric phase, which defines the deformation

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#### Nomenclature

a<sub>0</sub> initial crack lengtha crack length

ASTM American Society for Testing and Materials

B specimen thickness

DMTA Dynamic Mechanical Thermal Analysis
DSC Differential Scanning Calorimetry

E Youngs modulus

EPBC ethylene-propylene block copolymer EPR ethylene-propylene random copolymer ESIS European Structural Integrity Society

EWF Essential Work of Fracture

F(J) cumulative probability

G<sub>IC</sub> critical energy release rate

GPC Gel Permeation Chromatography

J J-integral

 $J_{Cmin}$  lowest fracture toughness  $J_{Cmax}$  highest fracture toughness

 $J_{IC}$  fracture toughness at crack initiation

 $J_{med}$  mean fracture toughness  $J_0$  threshold toughness

 $J_{0.2}$  crack resistance at 0.2 mm of the total crack growth

K<sub>Imax</sub> critical stress intensity factor LEFM Linear Elastic Fracture Mechanics

*m* shape parameter

M<sub>n</sub> number average molecular weight
 M<sub>w</sub> weight average molecular weight
 M<sub>z</sub> z-average molecular weight

NLEFM Non Linear Elastic Fracture Mechanics

NMR Nuclear Magnetic Resonance P cumulative probability PP polypropylene homopolymer

SENB single edge notched bend specimens abbreviation

 $T_{\sigma}$  glass transition temperature

W specimen width  $\Delta_a$  crack extension  $\sigma_Y$  yield stress

*χ* degree of crystallinity

3P-W three-parameter Weibull model

behaviour of the dispersed particles and their ability to absorb and redistribute the energy; and by the other side, the concentration and particle size, which establishes the probability of both initiating crazes and intercepting the fracture line.

The influence of the structural and morphological parameters (content, size and interparticle distance of the rubber modifiers [4–14], molecular weight, tacticity, crystallinity index, shape, size and distribution of crystalline regions of the PP matrix) on the fracture behaviour of PPs has been widely analyzed specially under high loading rates and at low temperature [2–4,6–17,18–25]. The main reason is that these polymeric materials, especially the block copolymers, exhibit a wide range of properties and structures with a great performance in the high strain field.

Apart from the typical high strain rate applications, the propylene polymers are also particularly suitable for hot and cold water distribution systems, heating and air conditioning for residential and industrial networks and transport of agricultural and industrial fluids as well as food substances. In service and in most of these applications, the polymers are to bear temperatures that go from  $-30\,^{\circ}\text{C}$  to even  $100\,^{\circ}\text{C}$  under static loading conditions. Previous works by Pooler [26] and Kobbe [27] illustrated vastly different behaviours over a practical in-service range of temperatures for HDPE and polypropylene formulations under compressive, tensile and flexural testing configurations, respectively. However, under quasi-static conditions there is lack of systematic studies in the literature of the evolution of the fracture parameters of bulk PPs with the temperature.

Santarelli and Frontini showed that it was very difficult to determine the fracture toughness of PP homopolymers at room temperature and under static loading conditions [28]. The fracture toughness data were highly scattered and showed a strong sensitivity to notch [19–31] and geometry dimensions [4,5,28] and strain rate [5,12,15,16,30,31]. In addition, some

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