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Structure and fracture toughness of thin-wall polypropylene moulded at different injection speeds

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ABSTRACT

Thin-wall injection-moulded parts have received a great deal of attention because of their lighter weight, which offers beneficial environmental and economic savings. However, research on the fracture toughness of thin-wall injection mouldings has been limited. In this study, thin-wall injection-moulded polypropylene (PP) parts were prepared at three injection speeds to reveal the relationship between their internal structures and mechanical properties. These characteristics were investigated by polarized optical microscopy, laser Raman spectroscopy, differential scanning calorimetry, wide-angle X-ray diffraction, and small-angle X-ray scattering. Fracture toughness was characterized by the essential work of fracture (EWF) method. Overall, the results revealed that the thicker, molecularly oriented skin layer obtained at a low injection speed led to higher fracture toughness in the thin-wall PP moulded specimen.

1. Introduction

Injection moulding is a widely used method for making resin products. Parts formed by this technique have been used in transportation equipment, electric appliances, and commodity goods, among many other products. Recently, a drive to decrease the weight of resin products while retaining their strength and function has been become popular, since weight saving has been recognized as a very important factor in terms of resource conservation, reduced CO_2 emissions, good design principles, and lower costs. One of the easiest strategies for decreasing product weight is thin-wall injection moulding. During injection moulding, the injected resin is rapidly cooled by the mould, so that the moulded product's internal structure consists of skin and core layers, which strongly influence its bulk properties [1–7]. The skin-core structure is formed even with thin-wall injection moulding; thus, it is quite essential to understand the relationship between the internal structure and properties of the part.

For thin-wall injection-moulded products, the influence of β-nucleation on the mechanical properties of polypropylene (PP) injection mouldings [8] and the effect of a high injection speed on the structure and properties of injection-moulded polyethylene [1,5,9–11] have been reported. Additionally, the structure-property relationships of thin-wall injection-moulded composite materials have been studied [12,13]. However, despite this extensive research on thin-wall injection mouldings, little is known about fracture toughness (i.e. resistance toward crack propagation) of PP, even among normal injection-moulded products, because of its elastoplastic character. Indeed, in the case of thin-wall products, fracture toughness characteristics are not known at all.

Fracture mechanics were first conceptualized by A. A Griffith ("Griffith theory") [14], who considered crack propagation in a brittle material in terms of the energy balance before and after crack propagation. In the 1950's, Irwin improved upon Griffith theory and evaluated fracture toughness as a stress intensity factor *K,* which was applicable mainly for metal, glass, and several other brittle materials [15]. The fracture toughness for ductile materials such as rubbers and resins is measured using the J-integral method [16–18]. However, this method is applicable only for thick-walled specimens which can satisfy the plane stress conditions under tensile testing, and is therefore limited to measuring the fracture toughness in thin, ductile specimens. In contrast, the essential work of fracture (EWF) method [19–26] can be used to measure the fracture toughness in thin-walled specimens such as metals and resins because there is no relationship between the plane strain condition and the fracture toughness in this method. Furthermore, the J-integral and EWF methods have been used to investigate the fracture toughness of elastoplastic materials, while the EWF method

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has been used to measure that of thin PP films [20,21,27]. However, few studies have examined the relationship between the internal structure and fracture toughness evaluated by EWF for thin-wall injection mouldings. Here, we focus on the relationship between the skin-core structure and fracture toughness of injection-moulded thin-wall PP. To reveal the effects of internal structure and molecular/crystalline orientation, thin-wall PP specimens were moulded at three different injection speeds. The relationships are discussed based on results of polarized optical microscopy, differential scanning calorimetry, X-ray scattering, Raman spectroscopy, and mechanical testing.

2. Experimental

2.1. Material and injection moulding conditions

In this study, homo-PP (Japan Polypropylene Corp., NOVATEC-PP, Grade: SA4L) with a melt flow rate (MFR) of 5 g/10 min was used. Thin-wall specimens were prepared by injection moulding with the following parameters: resin temperature, 200 °C; mould temperature, 40 °C; packing pressure, 50 MPa; holding pressure, 30 MPa; and cooling time, 20 s. To change the internal structure, thin-walled samples were prepared at three injection speeds: 10, 100, and 300 mm/s. The specimen dimensions were 100 × 20 × 1 mm^3.

2.2. Structure

2.2.1. Polarized optical microscopy (POM)

The skin-core morphology of the thin-wall injection-moulded specimens was examined by POM using a BX-51 microscope (Olympus Corp.) with a 530-nm-sensitive colour plate. Thin-sectioned samples of approximately 50 μm thickness were prepared by polishing the thin-wall injection-moulded PP specimens. The samples were inserted between the polarizer and analyser, and set to 45° and −45° directions for the polarizer. The direction of the observed cross-section corresponds to the flow direction–normal direction (FD-ND) cross-section.

2.2.2. Differential scanning calorimetry (DSC)

Crystallinity was measured via DSC (TA Instruments, Model: DSC 2920). The thin-wall injection-moulded PP specimen was embedded in epoxy resin and then sliced successively into 50 μm thick films from the sample surface to a depth of 500 μm with a rotary microtome (Leica Biosystems Ltd., RM2235) at room temperature. The samples were heated at a scanning rate of 10 °C/min from 30 to 200 °C under nitrogen atmosphere. Crystallinity was calculated using Eq. (1) and a value of 209 J/g for the crystal melt enthalpy of PP (ΔH_m^0) [28],

$$\text{Crystallinity}(\%) = \frac{\Delta H_m}{\Delta H_m^0} \times 100(\%) \quad (1)$$

where ΔH_m is the crystal heat fusion value of PP.

2.2.3. Small-angle X-ray scattering (SAXS) and wide-angle X-ray diffraction (WAXD)

X-ray analysis was conducted for the thin-wall injection-moulded products to investigate differences in crystal morphology; the lamellar orientation was measured by SAXS (Rigaku Corp., Model: MicroMax-007HF) and the β-crystalline fraction was measured by WAXD (Rigaku Corp., Model: 55R4206). These samples were prepared by the same method as the DSC film samples, i.e. with 50 μm thicknesses and sliced from the surface to 500 μm in depth. The X-ray beam permeated the film samples in the direction to thickness direction (TD). The lamellar orientation was calculated from the SAXS data using Eq. (2) [29],

$$\text{Crystalline Orientation} = \frac{180 - FWHM}{180} \quad (2)$$

where the full width at half maximum (FWHM) was calculated from the peak on 2θ angle of the 2D-SAXS pattern.

The β-crystalline fraction K from WAXD was calculated using the Turner-Jones Eq. (3) [11],

$$K = \frac{I_{\beta(300)}}{I_{\alpha(110)} + I_{\alpha(040)} + I_{\alpha(130)} + I_{\beta(300)}} \quad (3)$$

where $I_{\alpha(110)}$, $I_{\alpha(040)}$, $I_{\alpha(130)}$, and $I_{\beta(300)}$ are the integrated peak area values of the α- and β-crystalline planes at different diffraction angles from the 2D-WAXD patterns.

2.2.4. Laser Raman spectroscopy

To assess the molecular orientation along the FD, laser Raman spectroscopy (HORIBA. Ltd., Type: LabRam-HR-800) was used. The measurements were conducted on FD-ND cross-sections every 2 μm from the surface to the core layer (500 μm depth). A red laser (λ = 633 nm) beam was focused at a sample surface through the half–wave plate. The molecular orientation was evaluated by the ratio of the peak intensities (I_{813}/I_{844}) at 844 and 813 cm^{-1}, which represent the Raman bands attributed to CH_2 rocking and C–C stretching, respectively. The intensity of the C–C stretching band at 813 cm^{-1} is known to correspond to the molecular orientation in PP. Accordingly, the I_{813}/I_{844} peak ratio makes it possible to evaluate the molecular orientation of thin-wall PP injection mouldings [4,30].

2.3. Mechanical properties

2.3.1. Tensile tests

Tensile properties were measured using a universal testing machine (INSTRON Corp., Type; 55R4206). A 1-mm-thick dumbbell specimen (JIS K-7162-1BA) was used and the longitudinal direction was set along the FD. Tensile tests were performed at room temperature (RT) at a crosshead speed of 10 mm/min.

2.3.2. Essential work of fracture (EWF) test

The fracture toughness of the thin-wall specimens was evaluated by EWF tests using the universal testing machine with double-edge notched specimens (Fig. 1). The dimensions of the test pieces were 100 × 20 × 1 mm^3, and the longitudinal direction was set along the FD. A U-shaped notch with a width of 2 mm was introduced followed by a 1 mm initial crack at the tip of the U-notch. The U-notch and 1 mm initial crack were prepared by using our original crack machine in order to

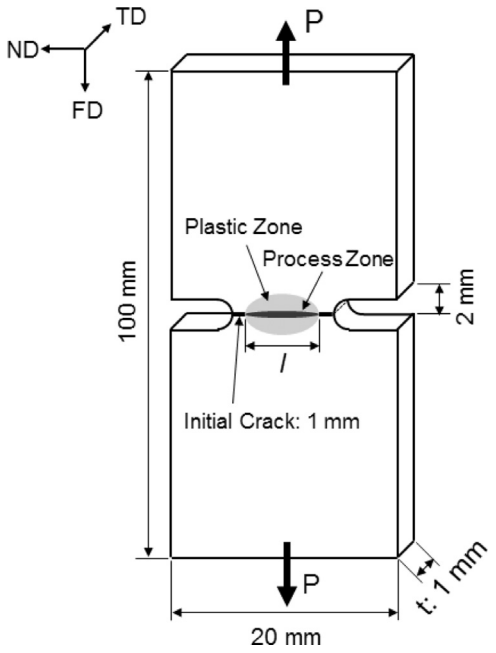

Fig. 1. Schematic drawing of double-edge notched specimen for EWF test.

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