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Structure and properties of injection-moulded nylon-6

Part 3 *Yield and fracture of injection-moulded nylon-6*

DAVID P. RUSSELL*, PETER W. R. BEAUMONT†
Department of Metallurgy and Materials Science, University of Cambridge,
Pembroke Street, Cambridge, UK

The effect of processing variables, test temperature, strain rate and exposure to water upon yield stress and toughness of nylon-6 injection mouldings have been examined. An increase in mould temperature increased the crystallinity. This resulted in an increase in yield stress and a decrease in toughness. An increase in strain rate raised the yield stress slightly and decreased the toughness significantly. Exposure to water decreased the yield stress and increased toughness. The brittle–ductile transition temperature determined from fracture toughness experiments may be equated to the α transition temperature of nylon-6.

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1. Introduction

In Part 1, we discussed the structure and morphology of nylon-6, and the effect of processing variables. Part 3 examines the yield and fracture behaviour of injection-moulded nylon-6 and the influence of crystallinity on yielding and fracture processes.

2. Experimental details

2.1. Specimen design and fabrication

Akulon K2 ZG 340, a pre-nucleated grade of nylon-6 was injection-moulded into rectangular cavities 102 mm x 13 mm x 2 mm. The injection pressure was 4 MN m^{-2} and the cycle time was 50 sec. Three mould temperatures were used, 15, 110 and 150° C; and the temperature of the melt was either 250 or 300° C. All of the specimens were stored in a desiccator for 4 weeks before testing.

2.2. Yield stress measurement

Uniaxial tensile tests were carried out to determine the yield stress of injection-moulded nylon-6 at 20 (± 2)° C using strain rates of 0.1 and 10 min^{-1}. Typical load–deflection curves are sketched in Fig. 1. The yield stress was calculated by dividing the maximum load by the initial cross-sectional area of the test-piece.

2.3. Fracture toughness measurement

Single-edge notched (SEN) prismatic bars were used to determine the fracture toughness (Fig. 2).

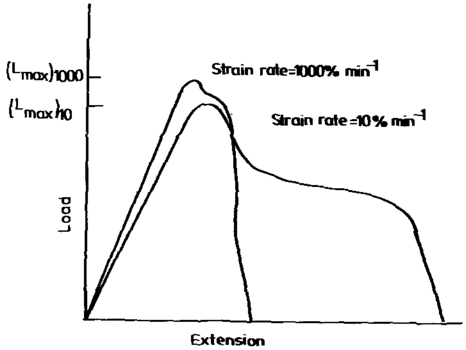

Figure 1 Typical load–deflection curves for unnotched nylon-6 in uniaxial tension.

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*Present address: Mars Limited, Slough, Buckinghamshire, UK.

†Present address: Department of Engineering, University of Cambridge, Trumpington Street, Cambridge, UK.

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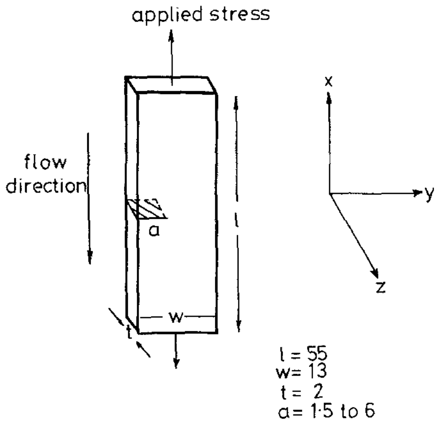

Figure 2 Specimen geometry and flow direction for a single-edge notched (SEN) specimen. (The dimensions are in mm.)

A razor blade is pushed into the root of the notch to produce a sharp crack; only those cracks which extended perpendicular to the principal axis defined by the *YZ* plane were kept for testing.

Each specimen was loaded in tension until fracture. In those cases where brittle fracture occurred, the critical stress intensity factor, K_c, was calculated using the equation

$$K_c = Y\sigma_f a^{1/2} \qquad (1)$$

where σ_f is the fracture stress, a is crack length, and Y is a geometrical constant which is a function of a/W [1].

$$Y = 1.99 - 0.41(a/W) + 18.70(a/W)^2 - 38.48(a/W)^3 \qquad (2)$$

W is the width of the specimen. The critical stress intensity factor was determined using a graphical method. Eight specimens containing a range of crack lengths between $a/W = 0.15$ and $a/W = 0.5$ were broken.

3. Results and discussion

3.1. Yield stress, mould temperature and melt temperature

Yield stress data as a function of mould and melt temperatures are summarized in Fig. 3. The figure shows yield stress to be proportional to mould temperature but insensitive to melt temperature. An increase in strain rate by two orders of magnitude increases the yield stress by about 20%.

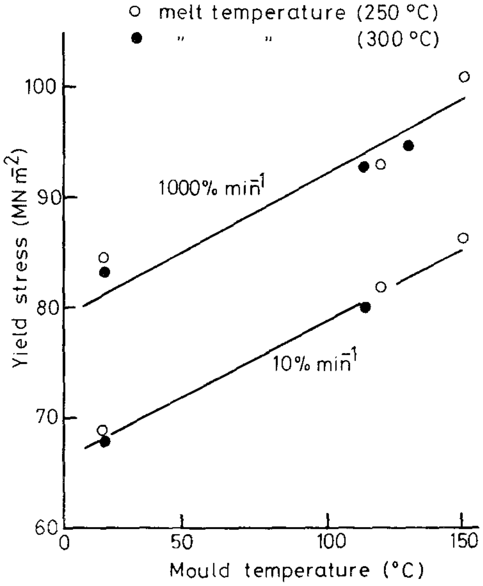

Figure 3 Yield stress, mould temperature data determined at two different strain rates at 20 (± 2)° C.

3.2. Crystallinity, mould temperature and melt temperature

Density of moulding as a function of mould and melt temperatures is shown in Fig. 4. An increase in mould temperature produced a corresponding increase in density while an increase in melt temperature from 250 to 300° C resulted in a decrease in density. Assuming a relationship of the form

$$\alpha = \frac{\rho - \rho_a}{\rho_c - \rho_a}$$

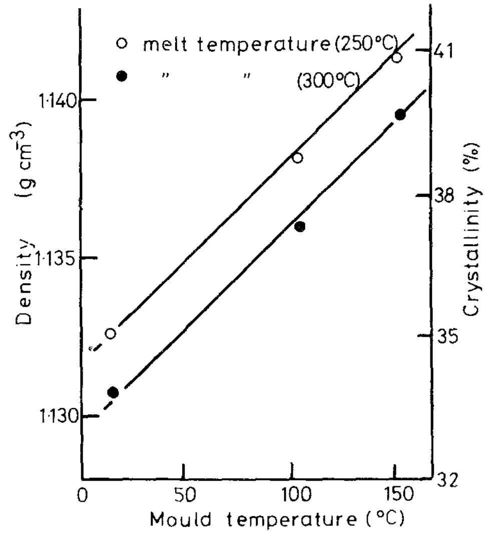

Figure 4 Density (crystallinity) of nylon-6 as a function of mould temperature, for two different melt temperatures.

where α is a degree of crystallinity, ρ_a is density of amorphous nylon-6 and ρ_c is density of the crystalline unit cell, then the experimental data can be replotted as crystallinity content. The crystallinity of a moulding made using a melt at 250° C increases from 35% to 41%, approximately, by increasing the mould temperature from 15 to 150° C. The crystallinity of nylon-6 is essentially insensitive to melt temperature.

3.3. Yield stress and crystallinity

The yield stress of injection-moulded nylon-6 increases linearly with average crystallinity (Fig. 5). Over the range of crystallinity content studied, an increase in crystallinity of about 1% raises the yield stress of the moulding by 3 MN m^{-2}.

3.4. Fracture toughness and crystallinity

The critical stress intensity factor (fracture toughness) decreases as the average crystallinity content of the moulding increases (Fig. 6). Bessell and Shortall [2] measured the fracture energy of cast nylon-6 where the crystallinity content varied between 29% and 47%. They found that the fracture energy was essentially insensitive to crystallinity but fracture energy decreased significantly at a crystallinity content above 39%; for example, increasing the crystallinity from 39% to 47% decreased the fracture energy by an order of magnitude. Our results show a similar trend to the one observed by Bessell and Shortall. It appears that a critical crystallinity in nylon-6 of the order of 40% exists. A critical crystallinity has also been reported for polyethylene mouldings [3], but as yet no satisfactory explanation is known.

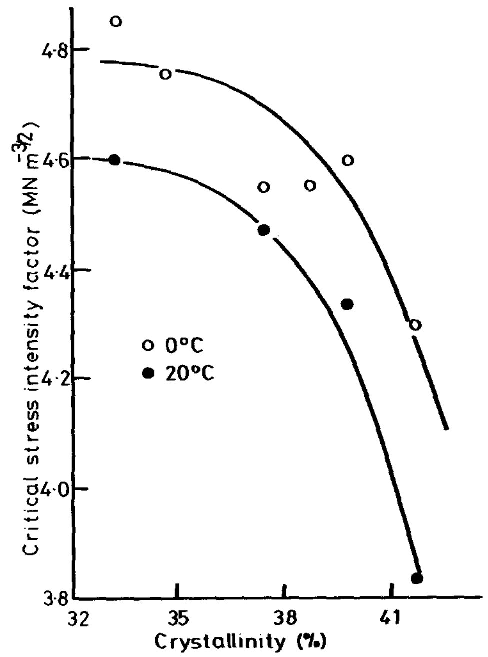

Figure 6 Critical stress intensity factor as a function of crystallinity content of nylon-6 measured at different temperatures.

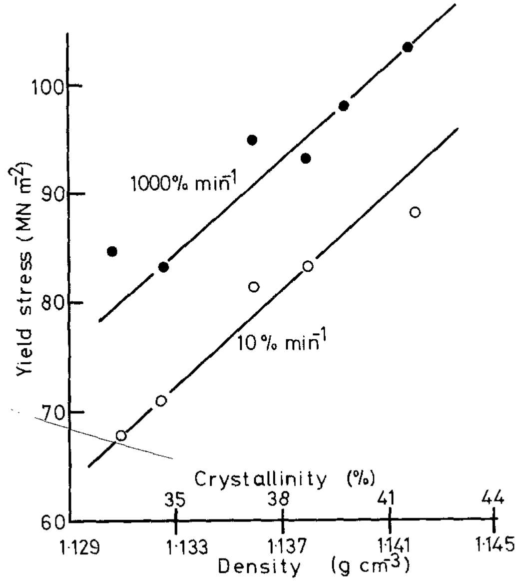

Figure 5 Yield stress of nylon-6 as a function of crystallinity content for two different strain rates.

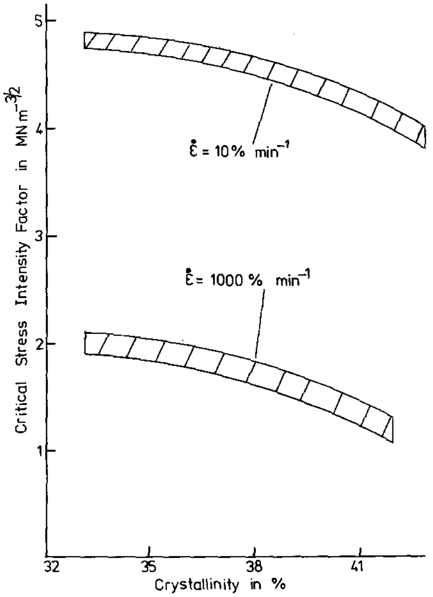

Figure 7 Critical stress intensity factor of nylon-6 measured at two different strain rates.

3.5. Fracture toughness and temperature

All of the SEN specimens tested at 0° C failed by brittle fracture while those specimens tested at 70° C failed by ductile rupture. The transition between brittle fracture and ductile rupture occurred over a narrow temperature range of 10° C, between 25 and 35° C for a strain rate of 10% min^{-1} and between 50 and 60° C for a strain rate of 1000% min^{-1}. Garbuglio *et al.* [4], investigated the α transition in nylon-6. They carried out dynamic mechanical tests, and determined a transition temperature of 30 and 50° C for strain rates of 10 and 1000% mm^{-1}, respectively. The brittle/ductile transition temperature for nylon-6 based on the fracture toughness data correlate well with the α transition temperature determined from other mechanical properties. In all cases of brittle fracture, an increase in temperature by 20 or 30° C resulted in a small decrease of K_c by a few per cent. On the other hand, fracture toughness is highly-sensitive to strain rate (Fig. 7). An increase in strain rate by one hundred times produces a decrease in K_c by a factor of 3. In contrast, yield stress is slightly dependent upon strain rate (Fig. 3).

3.6. Effect of water

An increase in the concentration of water in a moulding by up to 5% by weight produces a linear decrease in yield stress from 80 MN m^{-2} to 25 MN m^{-2}, approximately (Fig. 8). The additional absorption of water has little effect on yield stress. The plasticization effect of water on nylon-6 is well documented [5].

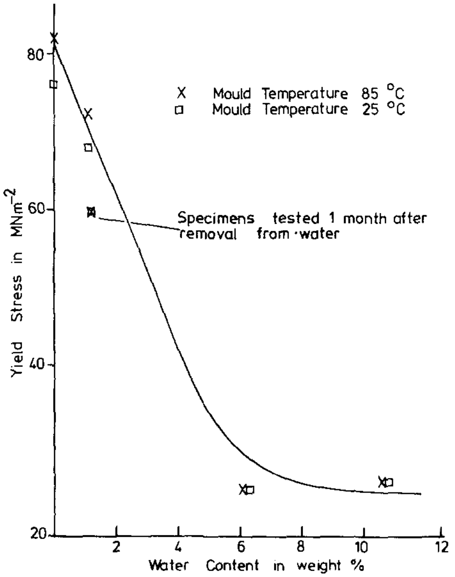

Figure 8 Yield stress of nylon-6 at room temperature as a function of moisture content.

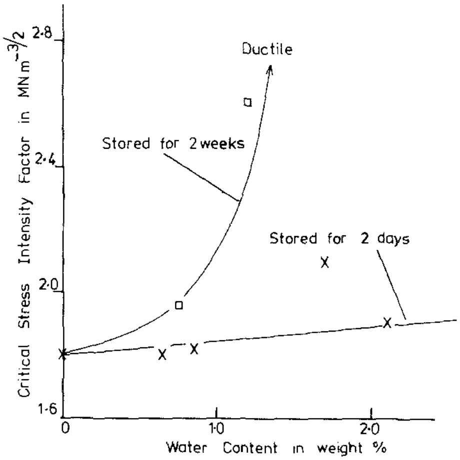

Figure 9 Critical stress intensity factor of nylon-6 as a function of water content, measured after (a) 2 days, and (b) 2 weeks.

Specimens containing about 2% by weight of water and stored for 2 days in sealed jars have approximately the same K_c value as the dry moulding; however, storing a similar specimen for 2 weeks results in an increase in toughness (Fig. 9). Eventually, after prolonged storage, brittle fracture is absent and the specimens fail by ductile rupture when tested at 20° C and a strain rate of 1000% min^{-1}. The distribution of water through the moulding is important; the amount of water at the crack tip will determine the mode of cracking and hence fracture toughness.

4. Modes of crack propagation

Nylon-6 can exhibit three modes of failure, depending on temperature and strain rate, brittle fracture, semi-brittle fracture, and ductile rupture; each mode is independent of crystallinity content. Brittle fracture usually occurred as the temperature was decreased or the strain rate increased; ductile rupture resulted at high temperature and low strain rates; and intermediate test conditions would produce a semi-brittle fracture.

4.1. Brittle fracture

The load–extension curve for brittle fracture is linear to maximum load and falls precipitously (Fig. 10). Unstable crack propagation is accompanied by crack front branching, producing a rough, stepped fracture surface with a fibrilar structure formed at the steps.

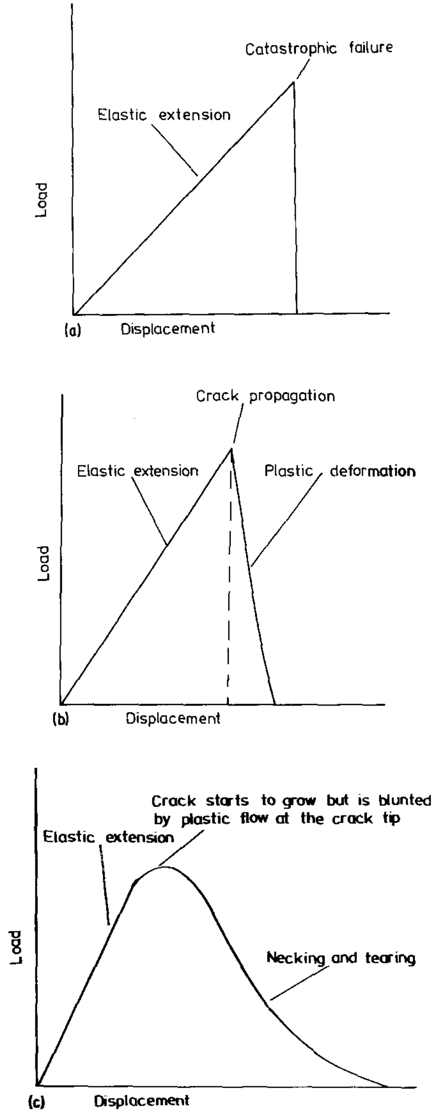

Figure 10 Typical load–deflection curves for SEN specimens: (a) brittle fracture, (b) semi-brittle fracture, and (c) ductile rupture.

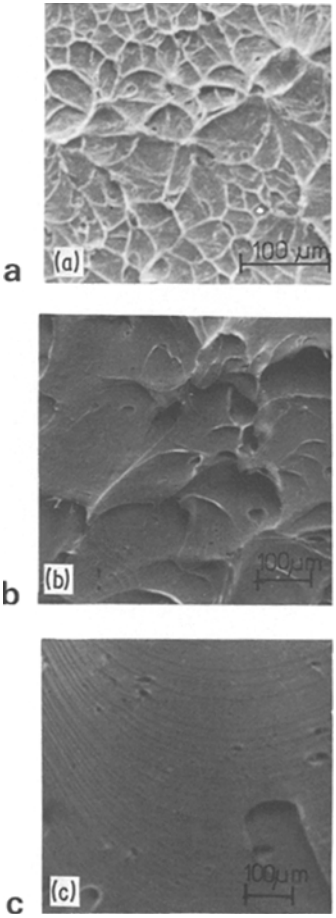

Figure 11 Semi-brittle fracture surface showing (a) dimple structure immediately ahead of the pre-crack front, (b) fracture surface some distance ahead of pre-crack front, and (c) fracture surface at far edge of specimen away from pre-crack front. (Crack has propagated from top to bottom.)

4.2. Ductile rupture

In this case, the load–extension curve is linear to peak load and falls off steadily as plastic flow occurs at the crack tip (Fig. 10). The crack tip plastic zone extends across the width of the test piece and necking and ductile rupture by tearing finally takes place.

4.3. Semi-brittle fracture

The load–extension is linear to peak load and falls off rapidly for a small amount of additional extension (Fig. 10). Some plasticity can be seen at the crack tip; failure results by the rapid propagation of the crack, the fracture plane perpendicular to the tensile stress direction. A narrow plastic zone is formed on either side of the crack path. No gross necking occurs. The fracture surface shows a "dimpled" effect, the size of the dimples depending upon the length of the crack (Fig. 11). Close to the tip of the pre-crack the dimples are of the order of 50 μm diameter. As the length of crack increases, the size of the dimple increases reaching 150 μm. The dimpled structure emerges into a smooth structure.

4.3.1. Crack pop-in

In some cases, crack "pop-in" precedes fast fracture and occurs under constant load conditions. The initial crack extension is confined to the centre or plane strain region of the specimen (Fig. 12). A dimple structure close to the popped-in crack is visible which is characteristic of the semi-brittle fracture process (Fig. 12b). This gives way to a smoother surface or "island" structure at the leading stage of the popped-in crack. The "island" structure is similar to the one observed on the fracture surface of polycarbonate [6]. The surface layers fail as miniature tensile specimens after crack pop-in and the fracture appearance is similar to the fracture surface of an unnotched tensile specimen (Fig. 13). Unstable crack propagation follows crack pop-in by brittle fracture and the features of the fracture surface are identical with those observed on the surface of a specimen that failed by brittle fracture.

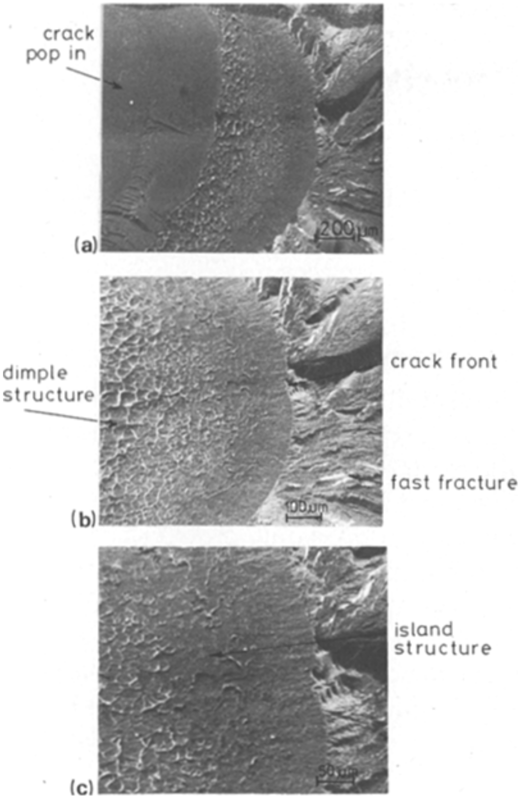

Figure 12 (a) Surface topography of the "tongue" of a crack which has "popped in". (b) "Dimpled" structure of tongue. (c) "Island" structure at tip of a "popped in" crack. (A fracture surface typical of unstable cracking is shown ahead of the "popped in" crack front.)

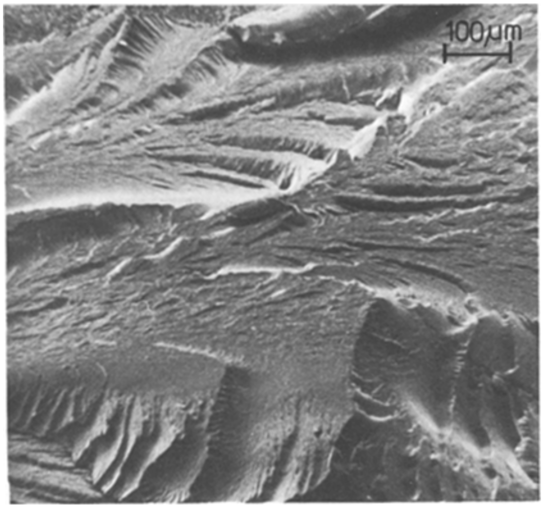

Figure 13 Fracture surface typical of a "fast" unstable fracture showing a rough, stepped topography.

5. Conclusions

The yield stress of injection-moulded nylon-6 increased linearly with increasing crystallinity, but the fracture toughness decreased with crystallinity at a crystallinity content greater than 40%, approximately. Yield stress was only slightly sensitive to strain rate while an increase in strain rate by one hundred times reduced fracture toughness by a factor of 3. A brittle–ductile transition temperature exists between 30 and 55° C depending upon strain rate. These temperatures correlate with the α transition temperature for nylon-6 determined by others.

Exposure to water of nylon-6 decreased yield strength while increasing fracture toughness and changing the mode of failure from brittle fracture to ductile rupture. It was found that the distribution of water through the thickness of a moulding, as well as the amount of absorbed water, affects yield and fracture behaviour.

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