The fracture resistance of bi-axially orientated PMMA

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Abstract. The mechanical performance of the transparent polymer PMMA (polymethyl methacrylate) can be significantly enhanced by the process of bi-axial stretching during manufacture. However, few studies have reported on the fracture properties of bi-axially oriented PMMA. In this work, the fracture toughness and resulting surface morphology of bi-axially orientated PMMA has been investigated by testing single edge notch bend fracture specimens under quasi-static test conditions. The orientation of the specimens with respect to the stretching direction was systematically varied such that the effect of in-plane orientation and out-of-plane orientation was determined. The out-of-plane fracture resistance was shown to be enhanced compared to the in-plane resistance and compared to amorphous PMMA. Both out-of-plane and in-plane crack orientations had little effect on fracture resistance. Complex crack growth behavior and surface morphologies were observed in the out-of-plane direction. The results have important implications for the study of damage tolerance of transparent canopies in the aircraft industry.

1. Introduction

In an aircraft cockpit, the canopy or windows serve an essential safety critical purpose; they must withstand the pressure differential between inside and outside of the aircraft whilst retaining high optical transparency. Also, they represent a highly vulnerable part of the structure due to their exposed, forward location making them susceptible to bird-strike [1]. The need to minimize the structural mass of aircraft has led to the transparent polymer, PMMA, becoming a popular choice of material for the canopy or windows. However, due to its relative softness compared to glass, the transparent surfaces are easily scratched by impacting debris during flight and by contact during maintenance and cleaning with abrasive surfaces and or chemicals. Due to the importance and vulnerability of these transparent components, higher performance variants of PMMA have been developed by bi-axially stretching the polymer during processing. There is therefore a need to understand the damage mechanisms operating in bi-axially orientated PMMA such that their performance in cockpit canopies can be better predicted and understood. For this reason, the study of the damage tolerance of transparent materials is important [2].

A complication which arises with the bi-axial stretching of polymers is that the degree of stretching may not be uniform across a plate or structure. This can be particularly evident in the edge or corner regions and this may result in highly non-uniform properties. A first step is to study the fracture behavior of bi-axially orientated, as compared to amorphous, PMMA. There have been many studies on the fracture toughness of standard amorphous PMMA [3-6]. Many studies have focused on the effect of loading, geometry and other parameters on the fracture of PMMA [7-10]. Some studies have
focused on the in-plane properties of orientated PMMA [11,12]. In the present work, the detailed mechanical behavior of the bi-axially oriented PMMA has been investigated. Fracture specimens have been prepared according to the single edge notch bend (SENB) geometry as detailed in the international standard (ISO 13586-2000) [13]. Specimens have been machined from bi-axially orientated plates at various angles to the direction of stretch. Specimens have been obtained such that the crack growth direction is both in plane and out-of-plane.

2. Materials and experimental methods

2.1. Materials
The bi-axially orientated PMMA used in this study was supplied by China National Chemical Corporation. During the manufacture, the material was subjected to bi-axial (X-Y in-plane) stretching with extension ratio 0.7 as shown in figure 1. The degree of stretching was reported to have a strong influence of the resulting properties of PMMA [14]. An amorphous PMMA (without stretching) was also studied.

![Figure 1. Specimens at different angle.](image)

2.2. Test specimens
Single edge notch bend (SENB) specimens were machined from the manufactured plates according to the guidelines given in [ISO 13586].

![Table 1. The details of the specimens manufactured.](image)
labeled as the S-A-V specimens.

The specimens of Type S-B-H were machined at five different in-plane directions as shown in figure 1. The arrows are meant to hi-lite the presence of the biaxial tension to which the plate stock was subjected prior to specimen extraction. This was to allow the effect of in-plane crack propagation direction to be studied. Type S-B-V specimens were also machined at five different out-of-plane orientations, equivalent to figure 1, but perpendicular to the XY plane.

2.3. The property of bi-axially orientated PMMA

The mechanical properties of bi-axially oriented PMMA and amorphous PMMA are shown in table 2, which can be used to calculate the fracture toughness. In this paper, the elastic moduli and tensile strengths of the material bi-axially oriented PMMA in different directions are considered to be the same.

| Table 2. Mechanical properties of bi-axially orientated PMMA. |
| PMMA | E/GPa | σm/MPa | σb/MPa |
| Bi-axially orientated PMMA (in XY plane) | 3.30 | 86 | 86 |
| Amorphous PMMA | 3.20 | 84 | 84 |

E is elastic modulus, σm is stress at the first local maximum, strength σb is stress at break

Determined in accordance with ISO527-1

2.4. The fracture tests

The fracture tests on the SENB specimens were performed according to the test conditions specified in [13]. Specimens were tested on an Instron (model 3355) universal test machine, as shown in figure 2, at a test speed of 10 mm/min and the laboratory temperature was 23°C. Prior to testing, the specimens were pre-cracked by a razor tapping technique [13]. In this technique, the machined notch tip was extended by tapping a sharp razor blade (which had been cooled in liquid nitrogen) into the notch tip and growing a 'natural crack’ ahead of the tip of the razor blade [15]. The resulting crack length was then measured optically after the fracture test by measuring at five positions evenly spaced across the specimen width using Image J software program, and then the average was calculated. The technique is shown in figure 3.

2.5. Determination of fracture toughness

The critical stress intensity factor K_{IC} was determined, according to ISO 13586 [13] as:

\[ K_{IC} = f \left( \frac{a}{w} \right) \frac{F_0}{h \sqrt{w}} \]  

(1)

where F_0 is the load at crack growth initiation, h is the test specimen thickness, w is test specimen width.
The critical energy release rate was also determined according to [13] as:

\[ G_{IC} = \frac{W_g}{h \times w \times \phi(a/w)} \]  

(2)

2.6. Scanning electron microscopy of the fracture surfaces

The fracture surfaces of the three types of specimen were examined using a scanning electron microscope (SEM). The crack direction on the orientation of specimens is shown in figure 4. A gold coating was sputtered onto the fracture surfaces make the PMMA conductive and prevent the specimen charging. The working distance was set to approx. 10 mm. An accelerating voltage of 10.0 kV was used. The magnification was set to ×200.

![Figure 4. Crack direction on the orientation of specimens.](image1)

![Figure 5. Directions diagram of specimens with notch.](image2)

3. Results

3.1. Fracture behaviour

During the pre-cracking by razor tapping, for the in-plane direction specimens S-B-H and for the amorphous specimens S-A-V, the initial cracks grew as intended, in direction 2 as shown in figure 5. However, for the razor tapping of the S-B-V specimens, a natural crack would not grow in direction 2, but rather two cracks grew at an angle between directions 1 and 2.

The crack growth behavior of specimens S-B-H, S-B-V and S-A-V are shown in figure 6 which were captures from the video record of the test.

![Figure 6. Fracture characteristic of specimens. (a) S-B-H, (b) S-B-V and (c) S-A-V.](image3)

It should be noted that all in-plane S-B-H specimens behaved in the manner shown in figure 6 regardless of the in-plane orientation of the specimen. Thus, the 0°, 30°, 45°, 60° and 90° specimens all behaved in the same manner, with a single, straight pre-crack forming in the correct direction. Thus, these specimens conformed to the standard [13] and valid values of fracture toughness could be obtained. For the S-B-V specimens, as the crack did not grow in the intended direction, a valid value of the fracture toughness of this specimen type could not be obtained according to the standard [13]. For the S-A-V specimens, the crack growth during fracture testing was all in the correct direction. It is clear that the crack growth behavior in the out-of-plane direction is severely influenced by the bi-axial orientation of the polymer.

3.2. Fracture toughness of bi-axially oriented PMMA: in-plane results

Table 3 presents the results of the S-B-H specimens machined at the various in-plane directions. For each angle, five repeat tests were performed. From the five repeated tests, average values of \(K_{IC}\) and
were determined together with the standard deviations. The standard deviations for each angle were in the range 0.10–0.15 for $K_{IC}$ and 0.13–0.19 for $G_{IC}$. Figures 7 and 8 show this data graphically, and it is clear there is no effect of in-plane angle on the value of fracture toughness. Thus, for fracture in the XY plane of bi-axially orientated PMMA, there is no influence of crack orientation.

### Table 3. $K_{IC}$ and $G_{IC}$ measurements on PMMA (S-B-H).

| Group No. | Angle | $K_{IC}$ (MPa$m^{1/2}$) | $G_{IC}$ (kJ/m$^2$) |
|-----------|-------|--------------------------|---------------------|
| 1         | 0°    | 2.20±0.11                | 1.24±0.16           |
| 2         | 30°   | 2.31±0.13                | 1.32±0.19           |
| 3         | 45°   | 2.24±0.10                | 1.27±0.13           |
| 4         | 60°   | 2.26±0.15                | 1.34±0.16           |
| 5         | 90°   | 2.21±0.14                | 1.26±0.14           |
|           | mean  | 2.24                     | 1.29                |

**Figure 7.** Values of $K_{IC}$ as a function of crack angle for in-plane specimens (S-B-H).

**Figure 8.** Values of $G_{IC}$ as a function of crack angle for in-plane specimens (S-B-H).

### 3.3. Fracture toughness of bi-axially oriented PMMA: out-of-plane results

Bi-axially orientated PMMA S-B-V specimens were tested with five different crack orientations. These were at 0, 30, 45, 60, and 90° degrees to the axis in the x-z plane. The razor tapping of these specimens to produce the initial pre-crack as required by the standard [13] was problematic. According to the standard, if a natural crack cannot be obtained by razor tapping, then the tip of the machined notch should be sharpened by sliding a fresh razor blade across the width of the notch. This was carried out, and the fracture tests were conducted from this notch after razor-sliding.

The typical force-displacement behavior for the SENB specimens upon testing is shown in figure 9. Following crack initiation, there were subsequent points of crack arrest and reloading prior to final failure. The more commonly encountered and standard fracture behavior was observed for the bi-axial S-B-H specimens as shown in figure 10. Due to the non-standard crack propagation in the S-B-V specimens, the first and second initiation points have been used to calculate provisional $K_Q$ values of fracture toughness and $G_Q$ values of provisional fracture energy.

**Figure 9.** Typical force-displacement behavior for the S-B-V specimens, at the various out-of-plane-angles.
The values of the provisional fracture toughness and fracture energy for the S-B-V specimens are shown in table 4. The values in the first loading peak between different angles nearly same and have no correspondence with angle, the values in the second loading peak is similar considering the discreteness of the data.

![Figure 10. Typical force-displacement behavior for the S-B-H specimens.](image)

### Table 4. $K_Q$ and $G_Q$ measurements on PMMA (S-B-V).

| Specimens No. | angles | $F_1$ | $G_0$ (KJ/m²) | $F_2$ | $K_Q2$ (MPa√m) | $G_Q2$ (KJ/m²) |
|---------------|--------|-------|---------------|-------|----------------|----------------|
| 1             | 0°     | 2.14  | 1.09          |       | 2.56           | 1.98           |
| 2             | 30°    | —     | —             |       | —              | 1.34           |
| 3             | 45°    | 2.26  | 1.15          | 2.47  | 1.79           | 0.70           |
| 4             | 60°    | 2.14  | 0.86          | 2.37  | 1.37           |                |
| 5             | 90°    | 2.17  | 1.01          | 2.54  | 1.80           |                |
| mean          |        | 2.24  | 1.09          | 2.45  | 1.64           |                |

$F_1$=the loading value of first high point, $F_2$=the loading value of second high point

The values in table 4 indicate that the values of fracture toughness and fracture energy are quite insensitive to crack orientation in the plane vertical to the XY plane. However, the fracture behavior of the S-B-V specimens is very different to the fracture behavior of the S-B-H specimens. Thus, the out-of-plane fracture behavior is far more complex than the in-plane fracture behavior.

### 3.4. Fracture toughness of amorphous PMMA

Four repeat SENB specimens of amorphous PMMA were tested according to the standard [13]. The pre-cracking using the razor tapping technique had been successful, producing a natural crack in the direction perpendicular to the specimen long sides. The crack re-initiated from the pre-crack in the classic manner, resulting in a well-defined point from which to perform the analysis. From the four repeat tests, the mean value of $K_{IC}$ was 1.80 MPa√m and the mean value of $G_{IC}$ was 0.84 kJ/m².

### 3.5. Discussion of results

The mean values of fracture toughness and fracture energy (valid or provisional values) for the various specimens are shown in table 5. The mean value of $K_Q$ and $G_Q$ for specimens in the out-of-plane direction (S-B-V) is higher than the equivalent values for specimens in the in-plane directions (S-B-H). This result illustrates that the ability to resist crack growth initiating on the surface of a PMMA sheet is somewhat greater than would be the case for a crack initiating from a through-thickness hole in the PMMA sheet. This has important consequences for damage tolerance, as typical failures in molded transparencies can either result from surface defects (such as scratches) or from the stress
concentrating effects of a through-thickness hole drilled for attachment purposes.

Table 5. Comparative results for three types of specimens.

| The mean value | Three types of specimens |
|----------------|--------------------------|
| $K_{IC}/\text{MPa} \sqrt{\text{m}}$        | S-B-H       | S-B-V       | S-A-V       |
| 2.24           | 2.54(K_{Q}) | 1.80        |
| GIC(KJ/m²)     | 1.29        | 1.64(GQ)    | 0.84        |

The fracture toughness of bi-axially orientated PMMA was shown to be significantly higher than the fracture toughness of amorphous PMMA. The value of $K_{IC}$ for the amorphous PMMA was about 80% while the $G_{IC}$ value was about 65.3% of the bi-axial PMMA in-plane values.

The fracture surfaces of the tested bi-axially oriented specimens (S-B-H) at different crack orientations (0°, 45° and 90°) are shown in the figures 11 and 12. These fracture surfaces are all very similar, which is consistent with the almost constant values of fracture toughness measured.

![Figure 11. Morphology SEM of specimen with different angles (Initiation fracture surface).](image)

![Figure 12. Morphology SEM of specimen with different angles (Crack propagation surface).](image)

The fracture surface morphology for the amorphous PMMA is shown in figure 13. It can be seen that the fracture surface of amorphous PMMA is very different to the bi-axially orientated PMMA shown in figures 11 and 12. The vertical stripe features seen on the fracture surfaces of the S-B-H specimens are absent, and the surfaces show branch features. During the crack propagation stage, the fracture surface of the amorphous specimens was much smoother than the fracture surfaces of the bi-axially oriented specimens (S-B-H).

Finally, turning to the bi-axially oriented PMMA (out-of-plane) specimens, these fracture surfaces are shown in figure 14. The fracture surface morphology of these specimens is very different to both the in-plane biaxial and the amorphous specimens. At crack initiation, the out-of-plane biaxial specimens show rough transverse grooves on the surface which is scaly. During crack propagation, the surfaces show distinct transverse stripes (sawtooth-like) perpendicular to the direction of crack growth.
It is clear that the molecular orientation of the polymer has significant effects on the fracture surface morphology and fracture toughness of PMMA. Such orientations were discussed in [16,17]. In the present work, the directionality of the crack growth and the differing resistance to crack propagation in transverse directions has been highlighted, with significant consequences for studies into the damage tolerance of PMMA canopies. Further studies are being conducted to understand these failure modes in more detail, and to use the results to predict service lifetime under typical operating conditions.

4. Conclusions
The fracture behavior and fracture resistance of bi-axially oriented PMMA was studied using a fracture mechanics approach. In-plane, and out-of-plane crack orientations were investigated, and these were also compared to out-of-plane orientations in an amorphous PMMA. It was shown that bi-axially oriented PMMA has enhanced fracture toughness compared with amorphous PMMA. Also it was shown that the out-of-plane fracture resistance was enhanced compared to the in-plane fracture resistance for the bi-axially oriented specimens. Crack orientation (either in-plane or out-of-plane) had little effect on the measured values of the fracture resistance. However, very different fracture surface morphologies were shown to be present in the in-plane, the out-of-plane and the amorphous specimens. These results have important consequences for studies into the damage tolerance of transparent canopies and suggest that different defects will progress to failure at different rates, depending upon the fracture resistance in a particular direction.
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