Fracture Measurement of a Light-Cured Resin Composite under Impact Tensile Loading*

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Abstract

The fracture behavior of a light-cured resin composite under impact tensile loading was studied using single-edge-cracked specimens fabricated by packing the composite between two rectangular plates of polymethyl methacrylate (PMMA). The impact load and displacement were measured with a piezo sensor and a high-speed extensometer, respectively. The load and displacement diagram, *i.e.* the external work applied to the specimen was partitioned into three parts: the elastic energy left in the fractured specimen, the nonelastic energy due to viscoplastic deformation and the fracture energy for creating new surfaces. The energy release rate G_f was then estimated using the fracture energy in the composite and PMMA specimens. The results indicated that, although the value of G_f in both materials increased with the fracture load, they showed different increasing behaviors.

Key words: High Polymer Material, Light-Cured Resin Composite, Impact Load, Fracture Energy, Elastic Energy, Nonelastic Energy, Fracture Load, External Work, High-Speed Extensometer

1. Introduction

Light-cured resin composites have been introduced as alternatives to traditional metals in dental treatments due to their ease of handling and aesthetic. However, compared to metals, they are not as suitable in mechanical properties such as strength and fracture toughness. Hence, developing stronger and more reliable composites through material processing is necessary. In particular, the fracture behavior of the composites needs to be carefully assessed to ensure their mechanical reliability. Fracture toughness is usually evaluated in terms of elastic fracture mechanical theory by measuring the crack length and the load or external work applied to a tensile-stressed or bending specimen. Several different types of specimen geometries and experimental methods have been proposed for fracture toughness evaluation of light-cured resin composites ⁽¹⁾⁻⁽⁴⁾. However, problems exist in both the light curing procedure as well as the inherent brittleness of the cured material that results from the large content of inorganic powders and fillers, that make it difficult to fabricate standard-sized composite specimens as are required for fracture toughness evaluation. In addition, most polymers exhibit some nonelastic effects from viscoelastic and plastic deformation ⁽⁵⁾. Brittle fracture also causes dynamic or inertia effects when a crack propagates dynamically within the specimen, as is commonly the case ⁽⁶⁾⁻⁽²⁵⁾. These effects also need to be taken into account when evaluating the fracture toughness of brittle materials. Nonetheless, quantitative appraisals of both effects on fracture toughness have seldom been undertaken due to the difficulties inherent in the experimental techniques currently used.

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Fig. 1 Specimen geometries.

Fig. 2 Impact tensile loading method.

We have explored these issues by measuring brittle fracture in polymers using a tensile loading device we have developed⁽²⁶⁾⁻⁽³⁰⁾. This device consists of a high-speed extensometer that allows quantitative measurements to be made of the static and dynamic displacements near the crack position in single edge-cracked specimens. The elastic energy stored in the specimens was evaluated by determining the kinetic behavior of the half specimen following fracture. The dynamic response and residual deformation of the specimen were then measured to evaluate the dynamic and nonelastic effects. The external work applied to the specimen was partitioned into three components: the elastic energy, nonelastic energy, and fracture energy required to create a new fracture surface. The energy release rate for crack propagation was similarly evaluated by using these parameters and indicated that the dynamic and nonelastic effects play important roles in understanding brittle fracture behavior⁽²⁶⁾⁻⁽³⁰⁾.

In this study, we evaluated the proposed experimental procedure using a light-cured resin composite. As outlined above, this material presents a problem in fabricating standard-sized specimens due to both light curing procedures and resulting brittleness. To obviate these difficulties, we fabricated a novel type of single edge-cracked specimen by packing the composite between two rectangular PMMA plates. The nonelastic energy E_n arising from viscoelastic and plastic deformation was then measured using a high-speed extensometer that consisted of an optical fiber and a position-sensing detector (PSD). The elastic energy E_e was calculated from the kinetic energy of the split specimen by measuring the oscillation following fracture. The external work U_{ex} was determined from the load and displacement applied to the specimen, and the displacement near the crack was obtained from the PSD. By subtracting E_e and E_n from U_{ex} , we could obtain the fracture energy E_f required to create the new fracture surface. For a fracture surface A_s , the energy release rate was evaluated using $G_f = E_f/A_s$ and correlated with the fracture load of the specimen. This paper discusses the applicability of the experimental method to light-cured resin composites by comparing results with those of reference PMMA specimens.

2. Experimental Methods

2.1. Specimen material

The light-cured resin composite used in this experiment was Clearfil AP-X, supplied by Kuraray Medical, Ltd. This material is a cross-linked acrylic resin composed of about 85 wt% inorganic powders and fillers. It is widely used as a dental restorative material in clinical practice. To overcome the problems of fabricating standard-sized specimens from

Fig. 3 Displacement measurement by PSD.

Fig. 4 Load P versus displacement δ with initial load P_0 .

this material, as outlined above, our specimens had the geometry shown in Fig. 1(a), where the composite was packed between two rectangular plates of PMMA. We used PMMA because it has similar properties to the composite and adheres readily to the composite.

The specimens consisted of two rectangular PMMA plates (Sumipex E), 3 mm thick, 20 mm wide, and 90 mm long. Clearfil Tri-S Bond bonding agent (Kuraray Medical, Ltd.) was applied to the end surfaces of the plates and irradiated using a Morita Jetlite 3000 light unit to cure the agent. The two plates were directionally aligned using two frames on a transparent 5 mm thick PMMA plate with a 10 mm clearance gap, and the composite was packed into the gap and irradiated with the light.

To prevent specimen curvature following the curing, the composite was irradiated eight times alternately from the top and bottom (four times of 5 s duration and four times of 10 s duration) followed by a further 10 s upon removal of the frames. The surface of the composite was then ground and polished flat. Each specimen was stored for 24 hrs in air at room temperature. The PMMA specimen shown in Fig. 1(b) was made to provide a comparison with the composite specimen.

2.2. Impact tensile test

Figure 2 shows a schematic diagram of a special impact-loading device that utilizes the free-fall of a weight ⁽²⁹⁾. This device consists of an electromagnet that holds the weight at an arbitrary height, loading axes for mounting the specimen, a metal frame for converting the impulse force into tension, and a piezo sensor for measuring the impact load. The specimen was clamped rigidly at its lower and upper parts to satisfy the requirements for specimen symmetry. A 3-mm-thick rubber sheet was mounted on the upper end of the frame to attenuate high-frequency vibrations caused by the impact ⁽²⁹⁾.

To change the fracture initiation load of specimens, sharp pre-cracks with lengths varying from about 2 to 7 mm were generated by forcing a fresh razor blade into a pre-machined saw-cut on the specimen's edge. All specimens were loaded by dropping a 10-kg weight from a height of 300 mm, resulting in an impact velocity of about 2.4 m/s. The piezo sensor has a frequency response of 45 kHz, permitting dynamic measurements ⁽²⁹⁾. The load P applied to each specimen was partitioned into an initial static load P_0 due to the metal frame's dead weight and a dynamic load P' due to the impact (see Fig. 2). The deformation of the loading axis was disregarded because it was much stiffer than the specimen. In this study, we excluded the experimental data obtained from the composite specimens with damages or defects on the interface between the resin and PMMA after the impact.

Figure 3 shows the configuration for measuring the displacement of the loading point and the upper region from the pre-crack position located at the center of the specimen. The

Fig. 5 Dynamic load P' versus time t .

Fig. 6 Dynamic displacement δ' versus time t .

displacement of the loading point is determined with the tensile-testing machine, while the displacement near the pre-crack is measured using an optical fiber and a PSD (SiTek Electro Optics). Ideally, the fiber should be attached as close as possible to the pre-crack. However, in this experiment, the fiber was attached at a point 2 mm away from the pre-crack to avoid damage due to the crack propagation. Nevertheless, the errors resulting from this placement can be disregarded, as the measured value would only be a few percent smaller than what would have been obtained on the centerline of the specimen. A laser diode was used as a light source, and an amplifier and a digital wave memory were employed for recording the output signals from the PSD. Since the PSD is an optoelectronic device that converts an incident light spot into continuous position data due to the lateral photo effect and has a frequency response of 100 kHz, it enables both static and dynamic displacement measurements⁽²⁷⁾⁻⁽³⁰⁾. The displacement δ at the specimen's centerline was partitioned into an initial static value δ_o and a dynamic value δ' (see Fig. 2).

3. Definition of Fracture Energy

Figure 4 illustrates a P - δ relationship for the frame's dead weight (see Fig. 2). In this figure, P_o and δ_o indicate initial static values due to the dead weight, and P' and δ' denote dynamic values due to the impact loading. Although P - δ relationships generally differ between static and dynamic loads, to simplify the analysis, the assumption was made that they are identical. The external work U_{ex} applied to half the specimen is given by

$$U_{ex} = P_c \delta_c / 2. \quad (1)$$

Equation (1) is valid when P_o and δ_o are much smaller than the critical values P_c and δ_c , and when nonlinearity due to the material's viscoelastic and plastic deformation is not significant.

This analysis also assumes that U_{ex} can be partitioned into three regions, as shown in Fig. 4⁽²⁶⁾:

$$U_{ex} = E_f + E_e + E_n, \quad (2)$$

where E_f is the fracture energy created for half the new surfaces $2A_s$, E_e is the specimen's elastic energy, and E_n is the nonelastic energy due to the specimen's viscoelastic and plastic deformation⁽²³⁾.

E_e and E_n are determined under the following assumptions. First, E_e is converted into the kinetic energy of the split specimen after fracture. Then, as the material's viscosity can be neglected during dynamic crack propagation⁽²³⁾, the change from P_c to δ_c or δ_n is elastic (see Fig. 4). Thus, the following relationships are obtained:

Fig. 7 Dynamic load P' versus displacement δ' .

Fig. 8 Energy ratio E_e/U_{ex} versus fracture load P_c .

$$E_e/U_{ex} = (\delta_e - \delta_n)/\delta_c \quad \text{and} \quad E_n/U_{ex} = \delta_n/\delta_c. \quad (3)$$

4. Results and Discussion

The dynamic load P' applied to the specimen was measured using the piezo sensor (see Fig. 2). Figure 5 plots the value of P' as a function of time t . The value of P' increased with t , and then fell abruptly at the critical value P'_c as the crack started to propagate. No high-frequency vibration was seen due to attenuation by the rubber sheet mounted on the upper end of the frame.

Figure 6 shows the dynamic displacement δ' measured using the optical fiber and PSD (see Fig. 3). The value of δ' increased with t , and then fell abruptly as the fracture initiated at the critical value δ'_c . After fracture, δ' exhibited damped oscillation, fluctuating about δ_n . The value of δ_n was almost equivalent to the initial static displacement ($\delta_o = 0.05$ mm), which implies that no large residual displacement occurred due to viscoelastic or plastic deformation. This was the typical impact tensile fracture behavior seen in the specimen material used. Hence, the nonelastic energy E_n in Eq. (2) could be disregarded. We also assumed that the damping oscillation in Fig. 6 could be ascribed to the energy loss caused by elastic wave propagation into the loading axes from the specimen.

Figure 7 plots the relationship between P' and δ' determined from Figs. 5 and 6. Although a slight change in the P' – δ' diagram was evident, we assumed a linear relationship between P' and δ' , as shown in Fig. 4, and that the external work U_{ex} could be evaluated from Eq. (1).

In our previous study ⁽²⁶⁾, we examined the dynamic response of the PMMA specimen before and after fracture using a Cranz-Shardin high-speed camera, which permitted simultaneous records of caustic patterns and corresponding specimen-focused images. The results indicated that the elastic and nonelastic effects of the material play an important role for understanding the dynamic fracture of the material. However, the direct measurement of E_e/U_{ex} was difficult in the impact test, the elastic displacement δ_e after fracture was determined by measuring the displacement δ' beside the crack position before and after fracture (see Fig. 3). This was estimated as follows. First, the amplitudes of the initial two oscillations were measured to estimate their attenuation (see Fig. 6). Then, this attenuation was used to determine the intersection with the falling curve δ' , i.e., the dynamic value δ'_e just after fracture. Finally, δ_e was determined using the following relationship (see Fig. 4):

$$\delta_e = \delta'_e (P_o + P'_c)/P'_c. \quad (4)$$

Figure 8 shows the ratio E_e/U_{ex} as a function of the critical load P_c . The value of E_e/U_{ex} for the composite specimen was about 81% in the region of small P_c , and about 64% in the

Fig. 9 Energy ratio E_f/U_{ex} versus fracture load P_c .

Fig. 10 Energy release rate G_f versus fracture load P_c .

region of large P_c , with E_c/U_{ex} for the PMMA specimen remaining almost constant at 73% over a wide P_c range. In this analysis, we were unable to make a quantitative evaluation of the energy loss caused by elastic wave generation from the crack tip and this energy loss was disregarded. Hence, the elastic energy determined from Eq. (4) may have been underestimated.

The fracture energy E_f was evaluated from Eq. (2) by substituting the elastic and nonelastic energies ($E_n = 0$). Figure 9 shows the energy ratio E_f/U_{ex} as a function of the critical load P_c . Although there was a scattering of E_f/U_{ex} data for the composite specimen, the value was about 19% in the region of small P_c , and about 35% in the region of large P_c , with E_f/U_{ex} for the PMMA specimen remaining almost constant at 27% over a wide P_c range.

The energy release rate G_f was estimated from:

$$G_f = E_f/A_s, \quad (5)$$

where A_s is half the area of the total fracture surface. Figure 10 shows G_f as a function of P_c . The value of G_f increased in both materials with P_c , although their increasing behaviors were slightly different. This difference supports the validity of both the present testing method and of the specimen geometry employed, particularly given the difference in fracture behavior of the two materials. It is our intention to explore this effect in more detail in our future work by investigating the crack velocity during crack propagation, given that the energy release rate also depends on the velocity.

5. Conclusions

The fracture behavior of a light-cured resin composite was examined using single-edge-cracked tensile specimens fabricated by packing the composite between two rectangular PMMA plates. From the load and displacement diagram under impact tensile loading, the external work U_{ex} applied to the specimen was used to determine elastic energy E_e , nonelastic energy E_n due to viscoelastic and plastic deformation, and fracture energy E_f for creating a new fracture surface A_s . The energy release rate was then estimated using $G_f = E_f/A_s$. Energy ratios E_n/U_{ex} , E_e/U_{ex} , E_f/U_{ex} , and G_f were correlated with the fracture load P_c , and compared with those for PMMA specimens. The results showed following.

- (1) Nonelastic energy E_n for the composite specimen under the impact tensile loading condition can be disregarded since it was almost zero similar to the PMMA specimen.
- (2) E_c/U_{ex} for the composite specimen was about 81% in the region of small P_c , and about 64% in the region of large P_c , with E_c/U_{ex} for the PMMA specimen remaining almost constant at 73% over a wide P_c range.

- (3) E_f/U_{ex} for the composite specimen was about 19% in the region of small P_c , and about 35% in the region of large P_c , with E_f/U_{ex} for the PMMA specimen remaining almost constant at 27% over a wide P_c range.
- (4) The value of G_f increased in both materials with P_c , although they showed different increasing behaviors.

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