In-situ Crack Propagation Observation of a Particle Reinforced Polymer Composite Using the Double Cleavage Drilled Compression Specimens

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In this study, we investigate the feasibility of in-situ crack propagation by using a double cleavage drilled compression (DCDC) specimen showing a slow crack velocity down to 0.03 mm/s under 0.01 mm/s of displacement control. Finite element analysis predicted that the DCDC specimens would show at least 4.3 fold delayed crack initiation time than conventional tensile fracture specimens under a constant loading speed. Using DCDC specimens, we were able to observe the in-situ crack propagation process in a particle reinforced transparent polymer composite. Our results confirmed that the DCDC specimen would be a good candidate for the in-situ observation of the behavior of particle reinforced composites with slow crack velocity, such as the self-healing process of micro-particle reinforced composites.

Key Words: Double Cleavage Drilled Compression (DCDC) Specimen, Crack Propagation, Stress Intensity Factor, In-Situ Observation, Self-Healing

1. Introduction

Delayed failure in composites containing reinforcing materials or voids is interesting due to the possibility of observing crack propagation and crack growth behavior (Lee and Tomozawa, 1999; Yoshida et al., 2001). A crack front with slow matrix crack growth can be directly observed in a double-cleavage-drilled compression (DCDC) specimen (He et al., 1995; Janssen, 1974; 1980; Jenne et al., 2003; Kagawa and Goto, 1998; Lee and Tomozawa, 1999; Michalske et al., 1993; Yoshida et al., 2001).

The double cleavage drilled compression (DCDC) specimen was developed by Janssen to obtain a specimen with a crack length independent \( K_1 \) (Janssen, 1974; Janssen, 1980). DCDC specimens have many advantages including resistance to compressive loading, mid-plane crack stability, and auto-precracking (Janssen, 1980). DCDC specimens were used to initiate slow crack growth (Kagawa and Goto, 1998; Lee and Tomozawa, 1999) and to directly observe crack front shape in-situ (Kagawa and Goto, 1998). With a double cantilever cleavage (DCC) geometry, stress intensity increases as the crack length increases under a constant applied load (Crichton and Tomozawa, 1999).
1999), and thus crack velocity accelerates throughout the measurement (Weiderhorn, 1967). Unlike the DCC specimens, the DCDC geometry features a stress intensity that decreases with crack growth under a constant applied load. Thus, DCDC geometry is more stable since crack propagation is retarded (Crichton and Tomozawa, 1999). And Janssen’s double cleavage drilled compression (DCDC) specimen was recommended by Yoshida et al. (2001) as a good specimen for observing crack propagation in a very brittle glass.

This study attempts to observe crack growth in a particle-reinforced composite by using the DCDC specimen. For direct observation of crack propagation, we adopted a transparent epoxy as the matrix of the DCDC specimen containing ceramic particles. We clarify the terms related to the double cleavage drilled (DCD) specimens as follows. The double cleavage drilled compression (DCDC) specimen was defined as the DCD specimen whose crack propagation is derived by externally applied compression (Janssen, 1974), while the double cleavage drilled tension (DCDT) specimen as the DCD specimen whose crack propagation is derived by externally applied tension. Based on the results of this study, we discussed the possibility of using the DCDC specimen to observe the self-healing mechanisms in situ, such as, the autonomic polymerization process of self-healing materials.

2. Materials and Methods

2.1 Experimental specimens

Commercially available CZC particles made of ZrO$_2$ (Cenotec Co., Masan, Korea) were embedded in a commercially available transparent epoxy MS–200 (Nippon Steel Chemical Co., Japan). The material properties of MS–200 epoxy and CZC (ZrO$_2$) particles are listed in Table 1. The diameter of the CZC particle was 1 mm. Transparent MS–200 powder and 0.15% volume fraction of CZC particles were mixed into a customized mold. Then the mixture was hot-pressed to shape as a DCDC specimen for 10 minutes at pressing pressure of 10 MPa and temperature of 463 K. The DCDC specimen was a 40 mm × 40

<table>
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<th>Table 1 Mechanical properties of the used material</th>
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<td>Elastic Modulus (GPa)</td>
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<td>Tensile Strength (MPa)</td>
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Fig. 1 The dimensions of the DCDC specimen. The DCD specimen was a 40 × 40 × 10 mm$^3$ plate with a hole of 8 mm diameter and two full-thickness groove of 2 mm length.

mm × 10 mm plate with a 8 mm hole and two full-thickness grooves representing a pre-crack of length, width, and end diameter of 2 mm (i.e. $a_0=2$ mm), 0.5 mm, and 0.5 mm, respectively (Fig. 1). All the specimen surfaces including the inside of the center hole were mechanically ground and polished to remove all visible scratches.

2.2 Finite element analysis using a DCDC specimen model

The effect of DCDC testing on crack propagation was evaluated and compared with that of DCDT testing by finite element analysis. By applying different loading conditions to a double cleavage drilled (DCD) specimen model, both the double cleavage drilled compression (DCDC) mode and the double cleavage drilled tension (DCDT) mode were analyzed. Because of structural symmetry, only a quarter of the isotropic DCD plate with dimensions of was modeled to include a hole of 8 mm diameter but not the groove (Fig. 3). Because the main purpose of the finite element analysis (FEA) was to evaluate the crack propagation rate of DCDC specimens versus DCDT specimens, the particles and the