A Study of a Novel Test Method for Self-Healing Techniques Using Microcapsules

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SUMMARY: Recently, self-healing methods of the cracked matrix, especially from polymeric composite materials, became the center of engineering researchers. In this paper, we summarized the self-healing concept for polymeric composite materials and investigated the effect of microcapsule on the crack growth behavior in colorless and transparent matrix, by experimental observation to describe the crack propagation around the microcapsules inside epoxy matrix composite. Compression splitting test for the specimen involving microcapsules was conducted using DCDC (Double Cleavage Drilled Compression) specimen. Through the experiments, we found that the size, relative position, bonding condition and relative stiffness of microcapsules are important parameters to decide the direction of crack propagation, which is related to the rupture of microcapsule for the self-healing.

KEYWORDS: Double Cleavage Drilled Compression (DCDC) specimen, Self-healing techniques, Microcapsules, Crack propagation

INTRODUCTION

The delayed failure in a material with the cracks or the voids is very interesting research topic due to the possibility of observing the crack propagations and the crack growth behavior [1,2]. Especially for in-suit observation of recent self-healing material, the crawling crack propagation technique is very useful. In the previous researches related with a self-healing polymer composite, the research workers [3-5] inspected the fracture plane of the experimental specimen to identify the broken microcapsules in the matrix and the healing efficiency. Some investigators [6, 7] pursued to find a new method to achieve the slow crack growth in kind of brittle materials, like glasses or ceramics.

They suggested a double-cleavage-drilled compression (DCDC) specimen chosen in order to have a slow matrix crack growth and to enable direct observation of the crack front shape.

In this paper, attention has been focused on the possibility of in-suit observations which show the fracture of microcapsules in self-healing structures using DCDC specimen.

Table 1 Material Properties of MS-200 and CZC

MS-200	
Elastic Modulus (GPa)	2
Tensile Strength (kg_f/mm^2)	60
CZC(ZrO_2)	
Elastic Modulus (GPa)	110
Tensile Strength (kg_f/mm^2)	35

EXPERIMENTAL METHODS

Preparation of test samples

Commercially available micro-particles (Young's Co., Korea) were used as microcapsules in self-healing structures because the purpose of this research is in-suit observation of the crack propagation near microcapsules in the matrix. And commercially available transparent epoxy (MS-200) was used as matrix. Materials properties of MS-200 epoxy (Nippon Steel Chemical Co., Japan) and CZC (ZrO_2) micro-particles are listed in Table 1. Figure 1 shows the photos of CZC particles and MS-200.

The DCDC specimens are prepared using hot plate compression method under the commercially recommended condition from the manufacturer. The shape and the dimensions of the DCDC specimen are 40x40x10mm with an 8mm center hole and 2mm pre-cracks from the edge of the hole. All the surfaces of the specimen were mechanically ground and polished even inside of the center crack. Figure 2 shows the schema of DCDC specimen and mold.

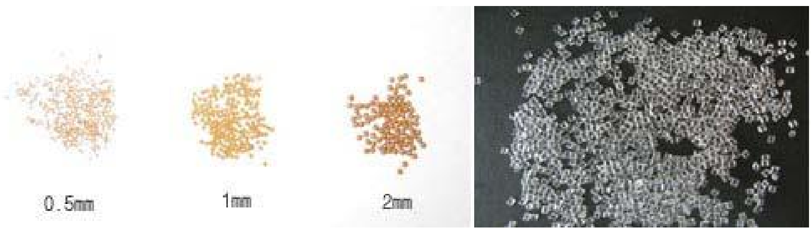


Fig. 1 The photos of CZC particles (left) and MS-200 (right)

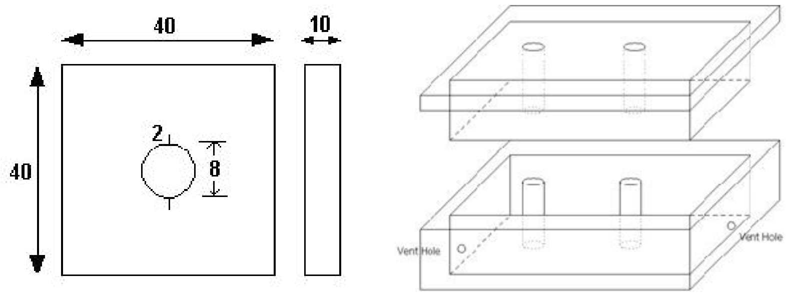


Fig. 2 The shape of DCDC and the schematic diagram of hot plate mold.

Experimental procedures

Figure 3 shows the experimental set-up of the in-suit compression splitting test. The compression test machine is Instron 8872. A digital CCD video microscope camera is used to observe the crack propagations, and the images with 100 magnifications are acquired in each 10 seconds. The load head speed is 0.01 mm/s by the displacement control.

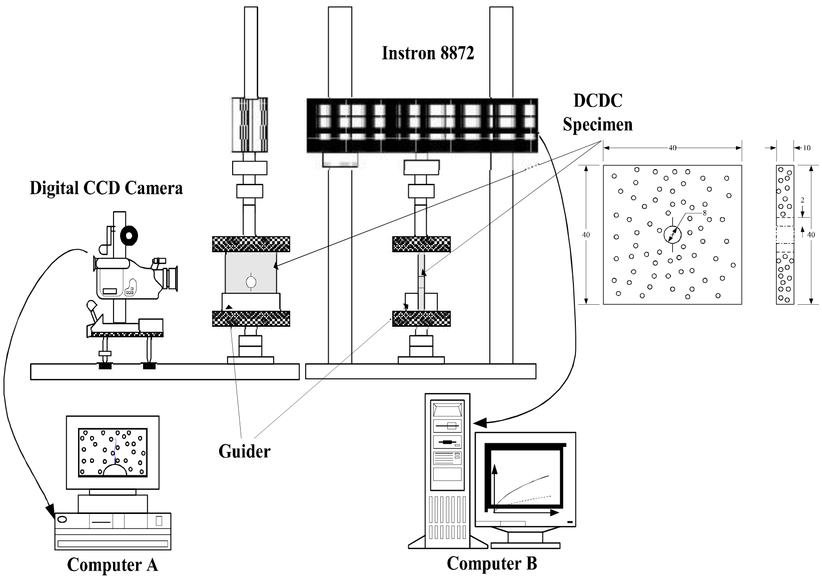
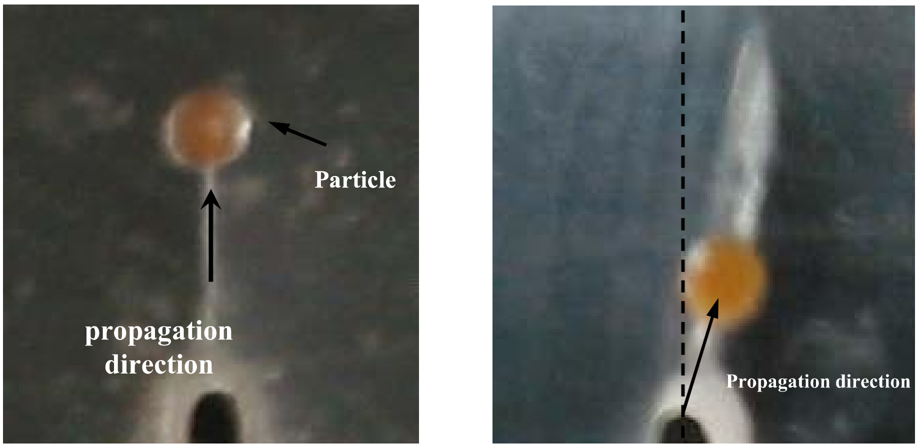


Fig. 3 Experimental set-up for in-suit observation around the microcapsule in a self-healing structure.

RESULTS AND DISCUSSIONS

Compression results

Figure 4 shows the compression test results for the microparticle inserted DCDC specimens. The specimens are in two forms; one is that the microparticle is located on the same direction of crack propagation and the other is that the microparticle is off the direction about 10^{o} degree of an oblique angle. The purpose of the specimens is the embodiment of the similar shelf-healing matrix which has the microcapsules inside to show the effects of the crack near the microcapsules. The microparticles are inserted in the matrix with the microcapsules with healing agent for the experimental convenience. Figure 4(a) shows the debonding on the boundary between the microparticle and the matrix when the crack arrived at the particle. From the experiments, it is expected that the microcapsules will be ruptured and the healing agents will be out from the capsules when the capsules are broken by the propagating crack in the matrix. Figure 4(b) shows that the crack propagation direction can be changed toward the particle in matrix. However, if the gap between the microparticle and the crack is longer than 2mm, the crack just propagates toward its direction independently of the microparticle in the matrix.



(a) (b)

Fig. 4 Two geometries of micro-particles in the matrix: (a) on the line of the crack tip (b) on the incline line of the crack tip.

Compression results

Figure 5 shows log-linear scale load-displacement curves of typical DCB specimen and DCDC specimen trough the experiment. DCB specimen's case shows that the displacement rate is high under the low load. However, in DCDC specimen's case, crack propagation is started at a high load condition. It means DCDC is more stable and has lower stress concentration near the crack tip when compared to DCB specimen. In the same load on the specimens, the displacement of DCDC specimen is within the low range and it needs large load to start the crack propagation from crack tip. And after the crack start, the displacement of the crack is in the low range.

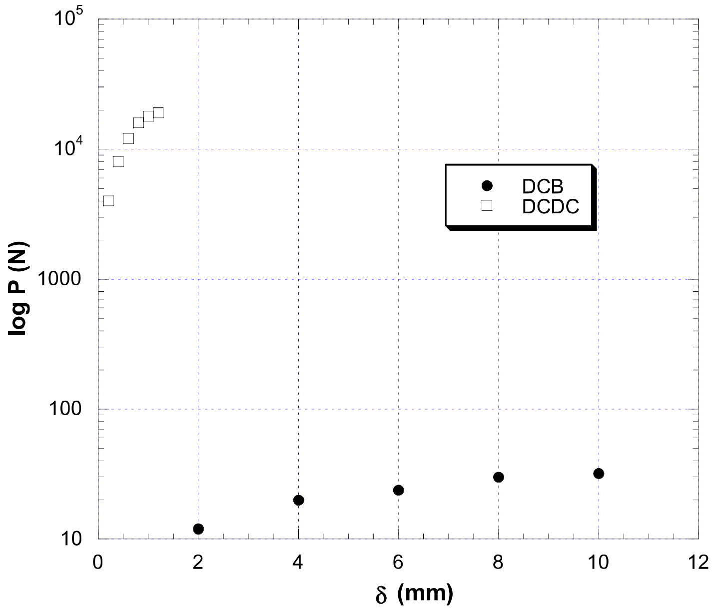


Fig. 5 Log and linear scale load-displacement curves of typical DCB and DCDC specimens

CONCLUSIONS

The most effective mechanism for fracture resistance of the matrix within this experiment is the elastic constraint effect by microparicles. The crack propagations in a microparticle-reinforced brittle matrix were observed and discussed. From the in-situ observation results, the possibilities of the application to the self-healing materials to observe the rupture of the microcapsules which heal the matrix automatically are presented.

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