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Shear strength measurements of ductile polymer films with controlled normal to shear plane angles

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ABSTRACT

Shear strength of the adhesives and polymer films was usually measured by lap shear test method. However, their fracture often associates with the combination of interfacial, interfacial–cohesive and cohesive failures. To confine the shear failure exclusively in cohesion so that the reported shear strength can be regarded as an intrinsic property of the polymer films, in this study we designed a unique test fixture that is capable of controlling the normal to shear plane angle in a confined testing space. Two poly(methyl acrylate-co-methyl methacrylate) film specimens were loaded in series in the test fixture such that as one fractured the other was in a state just prior to fracture and could be used for the investigation of shear bands by scanning electron microscopy (SEM). As the normal to shear plane angle was increased from 30 to 60°, the shear bands were more concentrated on the center region owing to the higher normal compressive stress. For the fractured specimen, the fracture surface investigated by SEM showed the striations which can be related to the shear bands. As the normal to shear plane angle was increased from 30 to 45°, the concentrated shear bands caused less striations perpendicular to the fracture direction. Interestingly, it also leaded to the appearance of the striations parallel to the fracture direction. As the angle was further increased to 60°, small islands formed by the cross-over of parallel and perpendicular striations appeared in the fracture surface. A possible mechanism for the formation of this unique fracture surface was proposed.

1. Introduction

Shear strength of the adhesives and polymer films was usually measured by lap shear test methods [1–6] because it is simple for sample preparation and testing. However, the failure of the samples often associates with the combination of interfacial, interfacial–cohesive and cohesive failures [7]. Therefore, the interpretation of the experimental results relies on the morphological investigation of the fracture surface. It is incapable of regarding the measured shear strength as an intrinsic property of the samples. In view of that, to report the shear strength of polymer films as an intrinsic property, controlling failure under shear only in cohesive failure is vital.

Recently, Hsueh, et al. [8] have reported a novel method to measure the shear strength of bulk metallic glasses with controlled applied normal stresses. They designed a unique test fixture similar to the one shown in Fig. 1 (except that the bulk metallic glass samples were sufficiently large to be fully inserted in the grooves and no bonding to the tool steel blocks was required) that is capable of controlling the location of shear fracture. By defining the compressive strength σ_f as the load divided by the cross-section area (normal to the height direction) of specimens, the normal compressive stress σ_n and the effective shear strength τ_e on the fracture surface are related to σ_f and the inclined angle (φ) of the fixture as that

$$\sigma_n = \sigma_f \sin\varphi \quad (1)$$

$$\tau_e = -\sigma_f \cos\varphi \quad (2)$$

Based on the Mohr-Coulomb criterion that is often used for concrete, soil, and polymer, the effective shear strength τ_e is described by [9,10],

$$\tau_e = \tau_s - \mu\sigma_n \quad (3)$$

where τ_s is the intrinsic shear strength at zero normal stress and μ the coefficient of normal stress dependence. Substitution of Eqs. (1) and (2) into Eq. (3) yields

$$\sigma_f = \frac{-\tau_s}{\cos\varphi - \mu\sin\varphi} \quad (4)$$

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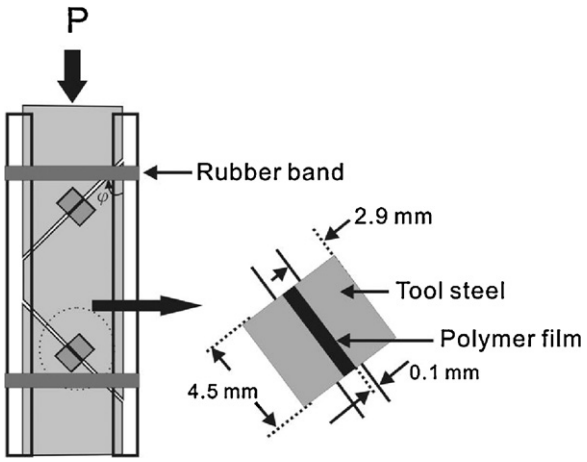

Fig. 1. Schematic illustration of the controlled-shear test fixture for polymer films.

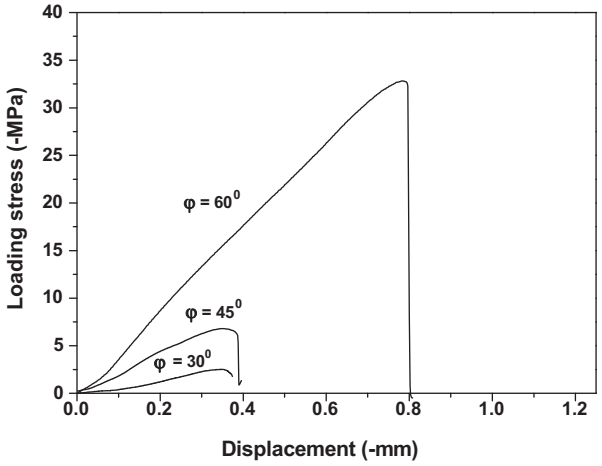

Fig. 2. Representative loading stress-displacement curves for the controlled-shear tests of P(MA-co-MMA) films with φ = 30, 45 and 60°, respectively.

It has been demonstrated that the controlled shear test allows the ratio of the normal to shear stress on the shear plane to be controlled by varying the inclined angle of the fixture [8].

To measure the shear strength of ductile polymer films, we modified the inserted sample areas of the test fixture as illustrated in Fig. 1. Because the polymer film is rather thin, it was sandwiched between two tool steel blocks and adhered with epoxy adhesive, the whole set of which was inserted into the groove of the test fixture. The height of steel blocks is 0.1 mm higher than that of the grooves so that both grooves and steel blocks would not interfere with testing. In this study, a ductile poly(methyl acrylate-co-methyl methacrylate) (P(MA-co-MMA)) film was used for the test. The experimental results revealed that this controlled shear test could control the shear failure of ductile polymer films such that failure occurred only within the cohesion. Because our unique designed test fixture was loaded with two P(MA-co-MMA) film specimens in series for testing, the shear fracture always occurred only in one specimen whereas the other that did not fracture was used to provide the information about its status just prior to fracture. The shear bands prior to fracture of the specimens and the fracture surface with respect to the ratio of the normal to shear stress controlled by the inclined angle of the fixture were investigated by scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

Methylacrlyate (MA), methylmethacrylate (MMA) and potassium persulfate (KPS) were purchased from ACROS. Water used for entire study was purified to 18.3 MΩ by a Barnstead Easypure RF system. Epoxy adhesive with the trade name of Epoxy AB Adhesive was obtained from Taiwan Licorgem Co., Ltd.

2.2. Fabrication of P(MA-co-MMA) films

Fabrication of P(MA-co-MMA) films by soap-free emulsion polymerization followed the method published elsewhere [11–14]. In general, MA (16.12 mL) and MMA (3.856 mL) were added to the flask which had already been loaded with KPS (0.7704 g) in water (250 mL). The solution was heated to 70 °C with stirring under nitrogen atmosphere. After 24 h, the resulting solution was filtrated through an 80 mesh sieve to remove large aggregates and then poured into a 15 cm × 30 cm aluminum foil rectangular mold. After dried at room temperature, the cast film was removed from the mold and further dried in an oven for 24 h at 50 °C. The final form of the P(MA-co-MMA) films had a thickness of ~0.3 mm.

2.3. Shear fracture tests of P(MA-co-MMA) films

The test fixture shown in Fig. 1 is made up of two identical trapezoidal tool steel blocks and one isosceles trapezoidal block. All of the blocks have the rectangular grooves cut at the centers of each tilt surface of the blocks used for the sample insertion. Three tilt angles φ of 30, 40 and 60° were chosen for this test. The size of the grooves is 4.5 mm width × 10 mm thickness × 2.9 mm depth. The design allows for two specimens to be loaded in series. Notably, U-shaped guide rails were used to loosely cap both sides of the fixture to prevent the out-of-plane movement of the fixture and two rubber bands were used to keep the rails in place.

The specimen set consists of a P(MA-co-MMA) film sample sandwiched between two tool steel blocks. An epoxy adhesive was applied for the adhesion between the tool steel blocks and P(MA-co-MMA) film. The dimension of the tool steel blocks is 4.5 mm width × 10 mm thickness × 3 mm height and the epoxy adhesive is less than 0.1 mm in thickness. The specimen sets were then inserted into the groove of the test fixture. The purpose of this design is to ensure that the shear fracture process indeed occurs within the P(MA-co-MMA) film. Tests were conducted by using a Tian-Siang compression tester with a cross-head speed of 2 mm/min at room temperature. At least six specimens were measured for each testing result.

2.4. Morphological investigations by SEM

As soon as one specimen in the test fixture was fractured during testing, the testing was stop automatically. The other specimen in the same test fixture which was not fractured was removed from the groove. After sputter-coated with a thin layer of platinum, the surface profile of the un-fractured specimens and the fractured surface of the fractured specimens were investigated using JEOL JSM-6700F scanning electron microscope.

3. Results and discussion

P(MA-co-MMA) film has a glass transition temperature of 19.2 °C measured by differential scanning calorimetry [13]. It is a ductile material at room temperature. Its Young's modulus has been reported as 36.6 ± 7.1 MPa, yield strength 1.23 ± 0.04 MPa, yield strain 84.5 ± 31.4%, ultimate tensile strength 4.95 ± 0.29 MPa and ultimate tensile strain 1000 ± 93% [12]. Because it is highly ductile, the shear strength was difficult to obtain by using the lap shear test method, especially in cohesive failure. Therefore, the new-designed test fixture shown in Fig. 1 was attempted to measure the shear strength of P(MA-co-MMA) film under compression.

3.1. Shear strength measurements

By applying a compressive load (P) to the test fixture, it induced shear deformation in the P(MA-co-MMA) films that have been inserted in the grooves between two opposite tilt surfaces of adjacent blocks. Fig. 2 shows the representative loading stress–displacement curves of P(MA-co-MMA) films for the respective inclined angles of 30, 45, and 60°. The loading stress was defined as the applied load divided by the surface area of P(MA-co-MMA) film sample. The virtually linear increase of the loading stress with the displacement for all the samples indicates that the ductile behavior of P(MA-co-MMA) films was not observed in the compression loading. The measured compressive fracture strengths σ_f were