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NOTES

In Situ SEM Observation of Fracture Processes in Thin Film of Poly(methyl methacrylate)

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Fracture mechanics change from shear yielding to crazing with increasing poly ethyl methacrylate content in epoxy resin interpenetrating networks,[1] and constrained crazing in polystyrene affected by the spherulite.[2] Although fracture mechanics of thermoplastics depend on orientation of molecular bundles and flows, void formation and coalescences and crazing, these have not yet been clarified. One of the reasons is as following; though such phenomena include dynamical processes, dynamic observation on such processes is difficult on a microscopic scale. Few works on the dynamic fracture test of polymer at microscopic scale are conducted in comparison with macroscopic order ones (like bulk materials properties).

To investigate dynamic fracture processes of the polymer on a microscopic scale (µm and sub-µm), a fracture test of the poly(methy methacrylate) (PMMA) thin film was performed with scanning electron microscope and the fracture processes of the film were *in situ* observed. Compact tensile test equipment was made and was inserted in the sample chamber of SEM (scanning electron microscope, JSM-T300, JEOL). The schematic illustration of the equipment is shown in Figure 1. The sample was PMMA (Comoglass, Kyowa gas Co. Ltd.) with molecular weights (polystylene standard) M_w and M_n were 139000 and 59900, respectively. Thermal distortion temperature of the sample was 92°C. A toluene solution of PMMA by 10% weight was made. Thin films of PMMA were produced by pulling glass microscope slides from the solution and glass slides were heated to evaporate toluene in the oven at 60°C. The films were floated on water surface and were dried in the oven at 60°C. Film thickness was $\sim 2.4\,\mu$m. Films were cut into a rectangular shape 3 mm × 10 mm and a side edge was notched by a razor. A single edge-notched specimen was chucked on the bronze blocks ((a) and (b)). The blocks were screw driven through combination gears by a baby motor, moved counter, with the displacement speed of the blocks 1.2 mm min^{-1}. Thus strain was applied to the specimen. Motion of the blocks was stopped intermittently to observe the fracture processes and to take photographs.

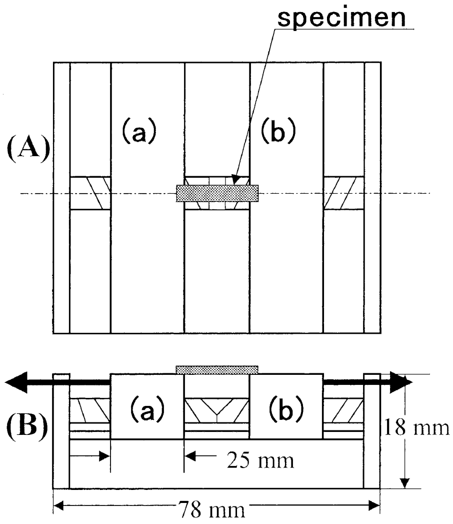

Figure 1. Schematic diagram of a compact tensile test equipment. (A) Plane view and (B) side view. A specimen is chucked on bronze blocks (a) and (b)which move in counter directions.

Figure 2 shows the results of *in situ* SEM observation. With increasing strain, many crazes were formed in the vicinity of crack tip. Craze grew in perpendicular direction to the stress direction. Contours of crazes showd straight lines and curved lines similar to those in bulk PMMA specimen (Figure 2a). With increasing strain further, a craze in front of the main crack changed to crack and thus a sub-crack was formed (Figure 2b). The main crack grew by connecting with a sub-crack in this case (Figure 2c). Clouds like crazes were also ob-

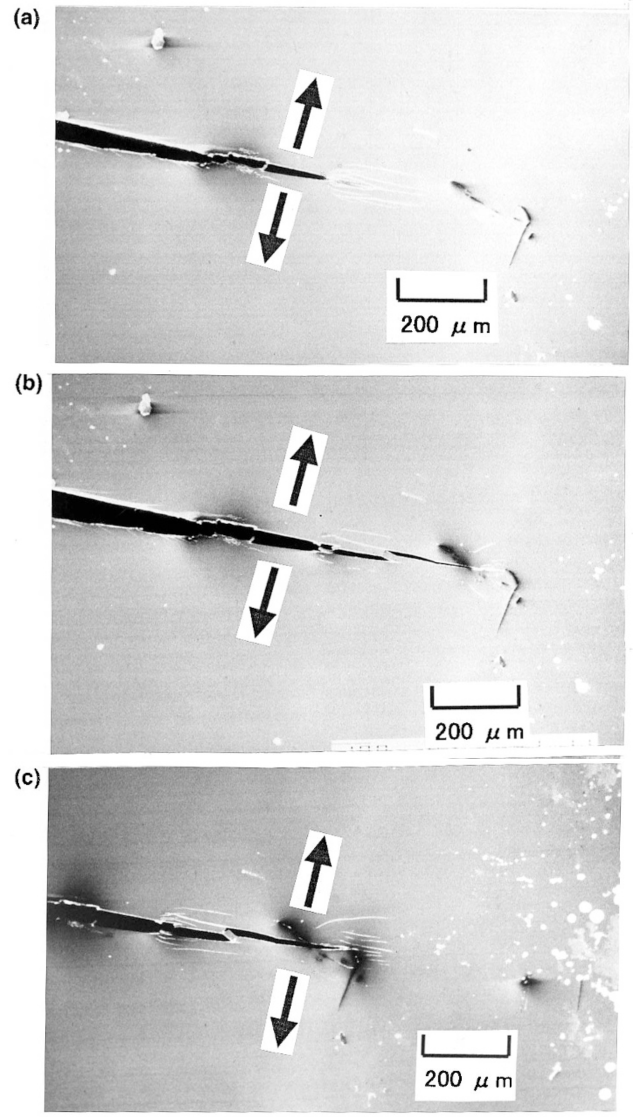

Figure 2. *In* situ SEM observation of fracture processes of thin film of PMMA. Arrows in photos show stress directions. (a) With increasing strain, many crazes are formed in the vicinity of crack tips. (b) With increasing strain further, a craze in front of the main crack changes to crack and a sub-crack is formed. (c) The main crack grows by connecting with the sub-crack.

served around the main crack (Figure 3). From the morphology of crazes and cracks, fracture of the present specimen was found to proceed with repeating craze generation, growth, and transition to cracks.

In situ SEM observation indicated that such a sub-crack is formed from a craze ahead of a main crack, growth of main crack occurs by connecting with sub-cracks, and fracture proceeds with repeating of craze generation, growth, and transition to crack. These fracture processes were similar to those in the bulk specimen (the thickness was larger than 1 mm).[3]

Concerning the dependence of the morphology of crazes on the film thickness, it was reported that PS craze behavior of film thinner than 110 nm differed drastically from thicker ones.[4] Since crazing in PS occurs at smaller stress than that in PMMA,[5] craze in PS is produced in thinner film compared with that in PMMA. PMMA craze behavior is estimated to change in the thicker film compared with PS film, but change of behavior was not observed for the present PMMA film. Since the thickness of the present film (a few μm) was much larger than 110 nm, the fracture behavior may be similar to that of a bulk specimen.

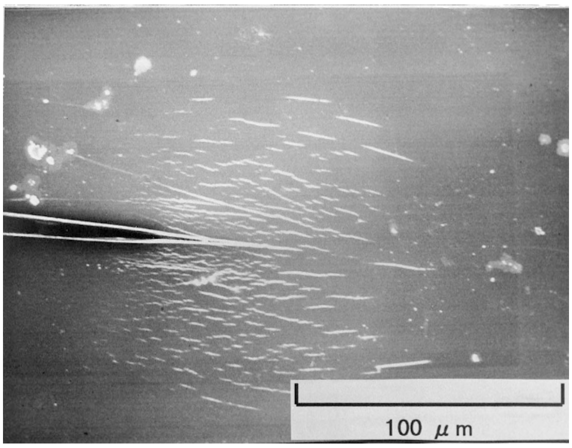

Figure 3. Clouds like crazes around the main crack.

With the equipment, we observed the fracture processes of PMMA fillm *in situ* in SEM. It was possible to observe dynamically fracture processes of PMMA film on a microscopic scale. Crazes with various morphologies were found. This means that the crazes are formed differently depending on conditions like sample preparation (thickness, homogeneity, residual stress, and etc.) and test conditions (temperature, strain rate, atmosphere, and etc.). Change of the craze to crack and craze generation were observed dynamically. Therefore, crazes have important roles for fracture processes in this film. Detailed observation of craze generation and change to cracks is planned in order to analyze fracture mechanics.

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