15A/2006G091

Detection of crazes in CNT/PET composites under tensile deformation

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Introduction

Crazes are often formed in the tensile deformation process of polymers. The crazes consist of fibrils elongated in the loading direction and voids surrounding the fibrils. Unlike true cracks, the crazes have load baring capability and the work of plastic deformation during formation and growth of the crazes contributes to increase the fracture toughness of polymers. If the polymer is further deformed, however, the fibrils in the crazes eventually fail and the crazes turn into true crack leading to the macroscopic fracture of polymers. Therefore, the detection of formation, growth and fracture of the crazes is important to understand the fracture behavior of polymers. There have been a number of studies on the crazes in neat polymers. In the present study, the possibility to detect formation, growth and fracture of the crazes in composites has been examined by conducting time-resolved small-angle X-ray scattering (SAXS) measurements during tensile deformation process of carbon nanotube (CNT)/poly(ethylene terephthalate) (PET) composites.

Experimental

The pellets of PET with an intrinsic viscosity of 1.07 dl g^{-1} were mixed with commercial multi-walled CNT synthesized through chemical vapor deposition, having the diameter of 20-50 nm and the length of several micrometers. The mixture was kneaded with a dual spindle kneader at 280 °C for 10 min, hot-pressed into films and immediately quenched. The films were cut into the tensile test specimens and surface notch with the depth of 150 µm was introduced perpendicularly to the loading direction. The specimens had the gage length of 10 mm, the width of 3 mm and the thickness of 300 µm. These specimens were loaded in tension at a deformation speed of 2 µm sec^{-1} at room temperature using a miniature tensile testing machine. Simultaneously, synchrotron X-ray with the beam size of about 0.2 by 0.3 mm was incident on the notched portion of the specimen and the SAXS patterns were recorded using a CCD camera and an image intensifier. Wide-angle X-ray diffraction of the specimens showed that the PET matrix was in amorphous state.

Results and discussion

With the PET film without containing CNT, cross-shaped SAXS was developed during tensile deformation. This scattering consisted of the streaks in parallel to the loading direction due to the total reflection at craze/polymer interfaces and the streaks perpendicular to the loading direction caused by the fibril/void structure in the crazes.

In the case of CNT/PET composite films, circular scattering from the CNTs and the streaks parallel to the loading direction due to the total reflection at the surface notches were produced in addition to the cross-shaped scattering observed for the neat PET films. The total reflection at the surface notch disappeared soon after the commencement of tensile deformation. After subtracting the scattering intensity from the CNTs, the integrated intensity of the streaks perpendicular to the loading direction was calculated. The integrated intensity indicates the amount of the fibril/void structure of the crazes. Figure 1 shows the load-extension curve and the variation of the integrated intensity of SAXS. The integrated intensity begins to increase during tensile deformation of the composite film and reaches maximum at maximum load. The variation of the integrated intensity differs between the neat PET and the CNT/PET composite films. This difference can be attributed to the effect of CNTs to increase the plastic work needed for the growth of the fibrils in the crazes. Further experimental study will be carried out to elucidate the effects of large aspect ratios of CNTs on the fracture behavior.

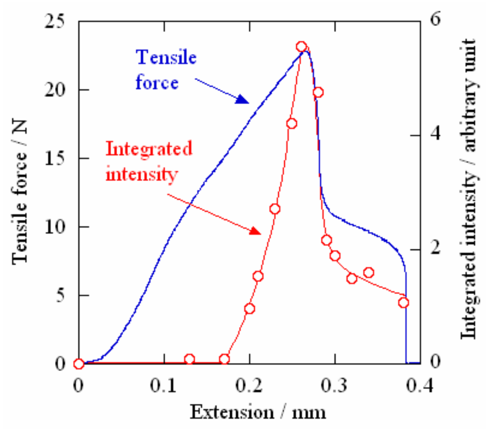

Figure 1 Load-extension curve and integrated intensity of SAXS for 1 wt% CNT/PET composite film.

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