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International Journal of Solids and Structures 43 (2006) 5969–5980

INTERNATIONAL JOURNAL OF
**SOLIDS and
STRUCTURES**

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Evolution of collective damage in a polyamide 6–silicate nanocomposite

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Received 17 May 2005; received in revised form 5 July 2005
Available online 30 August 2005

Abstract

The microvoiding in crack-tip plastic zone in a partly exfoliated polyamide 6–silicate nanocomposite is investigated through the experimental measurement of microvoid size distribution and the computer simulation in context of non-linear fracture mechanics. In order to analyze the effect of silicate content on fracture resistance, the evolution of the collective microvoids induced by the silicate nanolayers is characterized by the void number density (VND). The influences of the filler–matrix interaction on the yielding and hardening behaviors, as well as the total number, volume fraction, and nucleation and growth rates of microvoids are discussed in detail.

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Keywords: Fracture toughness; Microvoiding; Number density

1. Introduction

The superior properties of polyamide 6–silicate nanocomposites, such as high stiffness and strength, low permeability, and high combustion resistance, have made them attractive for a wide variety of engineering applications (Yasue et al., 2000; Masenelli-Varlot et al., 2002; Qiao et al., 2005). Over the past two decades, numerous studies have been performed for the development of processing techniques and material characterization. However, usually as a “side issue”, the addition of the silicate nanofillers would increase the level of brittleness, which could greatly limit the applicability of these materials in load-bearing components (Giza et al., 2000; Tjong et al., 2002).

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Polyamide 6–silicate nanocomposites are obtained by dispersing silicate nanolayers in polyamide 6 matrix. The small thickness of the silicate nanolayers around 1 nm and the relatively large length and width around 100–1000 nm lead to the ultrahigh three-dimensional aspect ratio. A silicate nanolayer contains several sheets of SiO_4 tetrahedral filled by Al^{3+} or Mg^{2+} (Brindley and Brown, 1980). The basic structural unit of silicate minerals is tactoid, consisting of about 100 layers. This layer stack must be disrupted during the nanocomposite processing. Usually the polyamide 6–silicate nanocomposites are synthesized through monomer intercalation technique (Kato and Usuki, 2000; Carrado, 2000). The initial basal distance of the tactoid is around 1 nm, which is smaller than the polymer chains. In polar solvents such as water, the layer stack can swell and the monomers can be intercalated into the interlayer by displacing the Al^{3+} and Mg^{2+} ions. The macromolecules can then be formed through in situ polymerization at appropriate temperature and pressure. The basal distance would be expanded to 10–20 nm and eventually the nanolayers are exfoliated.

In neat polyamide 6, the dominant mechanism of plastic deformation is cold drawing, and under compressive loading shear banding can also take place (Friedrich, 1983). With the addition of the silicate nanolayers, the crystallinity can be either reduced slightly (Wang et al., 2002) or nearly constant (Chan et al., 2002), while the spherulite size is reduced significantly (Tjong et al., 2002), which was attributed to that the nanofillers can promote crystallization nucleation and the molecular mobility in amorphous phase is considerably lowered. At a crack tip, breakdown of individual lamella or layer stacks associated with the nanolayer-induced stress concentration results in the microvoiding near the filler/lamellae–amorphous interface, causing the fibrillation quite similar with that in an amorphous polymer (Gloaguen and Lefebvre, 2001). At room temperature, the critical J -integral, J_{IC} , decreases with the increasing of silicate content, the decreasing of polymer–silicate interface strength, and the reduction in degree of dispersion (Russell and Beaumont, 1980; Nair et al., 2002). As silicate content increases, a ductile-to-brittle transition takes place in the range of 2–7 wt.%. Tjong et al. (2002) reported that the energy at break can be greatly lowered by about 75% and the elongation at break can be reduced from more than 100% to smaller than 30%.

Experimental evidences have shown that, for the polymer–silicate nanocomposites, the fracture toughness is nearly proportional to the plastic zone size, indicating that the plastic deformation mechanism is quite similar in a wide range of silicate content (Nair et al., 2002), probably due to the small void size and the rigidity of the silicate layers. However, currently there is still no satisfactory model that can relate the micromechanisms of fracture to the macroscopic properties. In order to identify the optimum microstructure, the behavior of the silicate nanolayers must be studied quantitatively. In this article we will discuss the evolution of the collective microvoids in context of nonlinear fracture mechanics, with the concept of microvoid number density being incorporated.

2. Experimental

To understand the effect of silicate content on the fracture resistance, four groups of samples were investigated: (1) neat polyamide 6 (N6); (2) polyamide 6–1.7 wt.% silicate nanocomposite (NC17); (3) polyamide 6–3.7 wt.% silicate nanocomposite (NC37); and (4) polyamide 6–5.0 wt.% silicate nanocomposite (NC5). The nanocomposites were provided by the Ube Industrial, Ltd and Dr. M. Kato in the Toyota CRD Lab, Inc. They were produced through the in situ polymerization method discussed by Kato and Usuki (2000), in which synthetic mica tactoids were first mixed with a ϵ -caprolactam solution at 80 °C and then kept at 220 °C for a relatively long time. Once the basal space expanded to about 2 nm, the temperature was raised to 260 °C. The wide angle X-ray diffraction (WAXD) data showed that the nanolayers were partly exfoliated (Qiao et al., 2005).

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