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Polymer 47 (2006) 679–689
www.elsevier.com/locate/polymer

Temperature dependence of crack initiation fracture toughness of various nanoparticles filled polyamide 66

Hui Zhang [a], Zhong Zhang [a,b,*], Jing-Lei Yang [a], Klaus Friedrich [a]

[a] *Institute for Composite Materials, University of Kaiserslautern, 67663 Kaiserslautern, Germany*
[b] *National Center for NanoScience and Technology, No. 2, 1st North Street Zhongguancun, 100080 Beijing, China*

Received 31 July 2005; received in revised form 9 November 2005; accepted 18 November 2005

Abstract

In the present study, the crack initiation fracture toughness of various nanoparticles filled polyamide 66 was investigated in a broad temperature range (23–120 °C) by using an essential work of fracture (EWF) approach. Four types of spherical nanoparticles, i.e. two types of TiO_2 (21 nm, with/without surface modification), SiO_2 (13 nm) and Al_2O_3 (13 nm), were selected with a constant volume content of 1% in nanocomposites, which were compounded using a twin-screw-extruder. The addition of nanoparticles led to an enhanced specific EWF item at most test temperatures at the cost of the reduction of the non-EWF item. The value of the specific EWF was also estimated by a crack opening displacement method. Associated with SEM fractograph analysis, it was clear that two basic factors, i.e. crack tip blunting and net section stress, finally determined the EWF value. With the addition of nanoparticles, the item of crack tip blunting was increased at most temperature range, which may be incidental with the formation of numerous dimples and sub-dimples induced by nanoparticles; while the item of net section stress was correlated with the particle distribution, especially at room temperature, which was notably decreased in case of poor nanoparticle distribution.

Keywords: Thermoplastic; Toughening; Temperature dependence

1. Introduction

It was reported [1–3] that rigid inorganic particles are able to enhance both the toughness and stiffness of some semicrystalline thermoplastics simultaneously. The fracture toughness of thermoplastics could be dramatically increased by introducing the rigid particles with typical volume content of 10–35%. Many possible toughening mechanisms were proposed. Generally speaking, two categories can be mainly considered, i.e. the crack front bowing and the cavitation mechanisms [4]. It is worth to note that the size of aforementioned rigid particles varies usually from sub- to several microns. However, if the particle size reduces to a nanometre scale, the toughening mechanisms would be obviously different, which is likely due to the huge particle–matrix interface introduced by the nanofillers. Toughness of rigid nanoparticle filled polymers deserves a separate treatise due to some contradictory findings in literatures. Corresponding toughening mechanisms induced by nanoparticles have not yet been well understood. Lots of factors (particle size, shape, distribution, type, aspect ratio, interface, particle concentration, and dispersion, etc.) may strongly affect the toughening efficiency of nanoparticles. To our knowledge, nanoparticles with a high aspect ratio, e.g. clay, often bring negative toughening effects. It is because under external load the large aspect ratio will generate significantly high stress concentrations at the end of the particles, which lead to earlier crack initiation and propagation, and finally are adverse to material toughness [5]. Chen et al. reported that the *J*-integral value of polypropylene (PP) decreased significantly with an increase of clay filler content [6]. Bureau et al. [7] found that the addition of clay (with/without coupling agents) into PP matrix reduced the specific essential fracture work, w_e, but increased the specific non-essential fracture work item, (βw_p. The quasi-spherical nanoparticles were recognized to be more promising for polymer toughening. Chan et al. [8] conducted very tough PP/$CaCO_3$ nanocomposites by melt mixing, and the *J*-integral tests showed a dramatic 500% increase in fracture toughness. Our previous work [9] indicated also that only 1–3 vol% nano-TiO_2 can notably enhance the fracture work of resistance to crack initiation at room temperature.

* Corresponding author. Address: National Center for NanoScience and Technology, No. 2, 1st North Street Zhongguancun, 100080 Beijing, China.
E-mail address: zhong.zhang@nanoctr.cn (Z. Zhang).

0032-3861/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.polymer.2005.11.084

PA66 is a semicrystalline thermoplastic polymer used for numerous engineering applications due to its combination of high thermal and mechanical properties with easy processing and moderate cost. However, neat PA66 is notch sensitive and sometimes tends to be broken in a brittle fashion. The deficiency has somewhat limited its broad utilities [10]. Besides, many automotive parts made of PA-based composites are required to be operated at elevated temperatures [11]. Therefore, their high-temperature mechanical performances, especially fracture toughness, should be taken into consideration. In this study, temperature dependent fracture properties of PA66 and its nanocomposites filled with different types of nanoparticles will be carried out. An essential work of fracture (EWF) approach with deeply double edge notched tensile (DDENT) specimens was applied. The EWF parameters were also estimated and analyzed via a crack opening displacement (COD) method, and the fracture surfaces were observed by a scanning electron microscope (SEM). In light of the experimental results and COD method, the related fracture mechanisms were further discussed.

2. Methodology of the essential work of fracture

The essential work of fracture (EWF) approach, firstly proposed by Broberg [12–14], has been used to characterize the plane-stress-state fracture toughness of ductile materials, including metals, papers, polymers, fibre composites and even foods [15]. The EWF method has gained popularity owing to its experimental simplicity, especially compared to *J*-integral technique. Many reports have indicated that the EWF parameters are affected by a number of factors, such as sample geometry [16–20], test conditions [21–25], notching method [26,27], polymer composition [28,29], filler fraction [30–32] and so on [33–36]. Recently, this method has been introduced to investigate the toughening effects of polymer nanocomposites as well [7,9,37]. A brief review of the EWF method was given in our previous paper [9], where we divided total dissipated energy (W_f) into two items, i.e. crack initiation (W_{ini}) and subsequent crack propagation (W_{prop}), respectively, according to the energy-partitioned method proposed by Karger-Kocsis [38] and Hashemi [23]. As schematically depicted in Fig. 1(a), W_f can be expressed as:

$$W_f = W_{ini} + W_{prop} \quad (1)$$

As the continuation of our previous work, the present work still concentrated on the parameter W_{ini}, since it represents the fracture energy consumed at crack initiation stage and is a crucial parameter for material design in engineering applications. Based on the EWF concept, W_{ini} is considered as a sum of essential and non-essential work of facture. The specific item (w_{ini}) can be described as:

$$w_{ini} = \frac{W_{ini}}{lt} = w_{e,ini} + \beta_{ini} w_{p,ini} l \quad (2)$$

where $w_{e,ini}$ and $w_{p,ini}$ are the specific essential and non-essential work of fracture related to crack initiation,

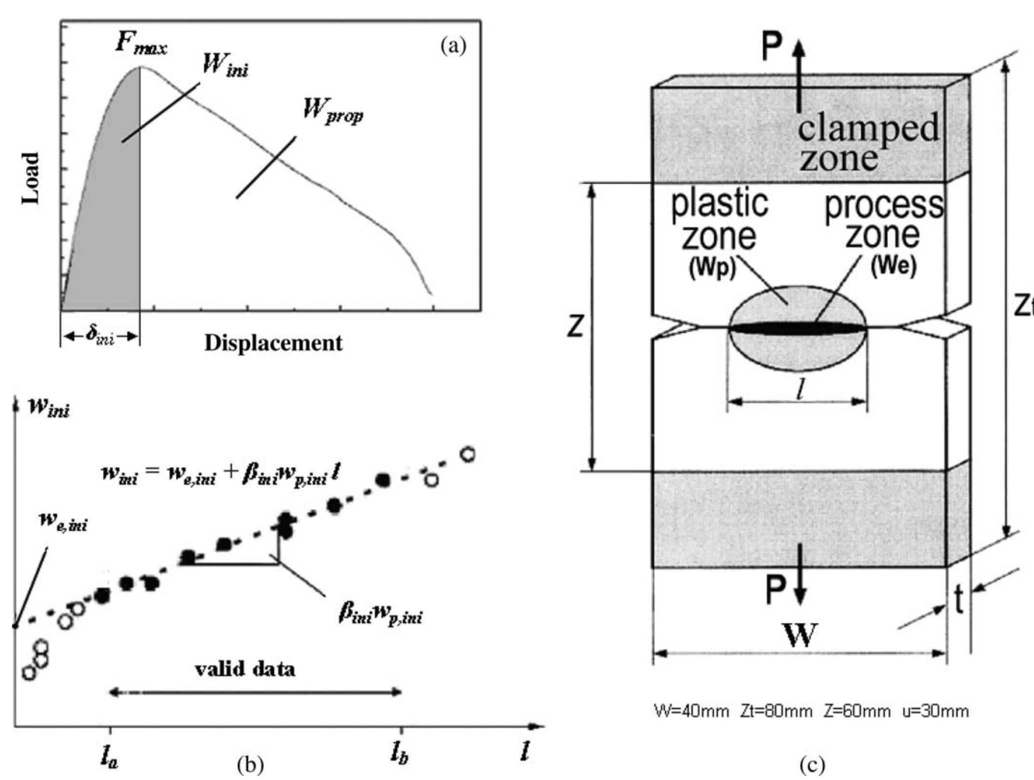

Fig. 1. Schematic diagram of the essential work of fracture (EWF) approach: (a) a typical load–displacement curve; (b) a curve of specific total work of fracture vs. ligament length; and (c) the dimensions of the deeply double edge notched tensile (DDENT) specimens.