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High strain rate compression of epoxy based nanocomposites

Yan Tian [a], Hui Zhang [a,*], Jun Zhao [a], Tao Li [b], Bi-Xiong Bie [b], Sheng-Nian Luo [b,c,*], Zhong Zhang [a,c,*]

[a] CAS Key Laboratory of Nanosystem and Hierarchical Fabrication, CAS Center for Excellence in Nanoscience, National Center for Nanoscience and Technology, Beijing 100190, China
[b] The Peac Institute of Multiscale Sciences, Chengdu, Sichuan 610207, China
[c] CAS Key Laboratory of Mechanical Behavior and Design of Materials, University of Science and Technology of China, Hefei 230026, China

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ABSTRACT

This work aimed to investigate the strain-rate effect (0.001–3000 s^{-1}) on compressive properties of the highly cross-linked epoxy and the epoxy sample filled with 10 wt% sol-gel-formed silica nanoparticles. As the strain rate increased, the compressive modulus and transition strength of both samples went up distinctly, the strain at break and ultimate strength decreased more or less, while the strain energy at fracture nearly did not change. Adding the sol-gel-formed silica nanoparticles can improve effectively the compressive modulus, transition strength as well as strain energy at fracture of the epoxy polymer owing to their homogeneous dispersion in epoxy matrix.

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1. Introduction

Nanoparticles can improve the key performance of polymers, such as stiffness, failure strength, fracture toughness, wear resistance, electrical conduction and insulation properties etc, due to their specific characteristics including tiny size, huge specific surface and quantum tunneling effect [1–7]. Among them, the inorganic nanosilica is frequently employed as the reinforcement for polymers, to which the stiffness and toughness are particularly important [8–11]. However, agglomerated nanoparticles in polymers induce the local stress concentration, reduce the particle-matrix adhesion, and thus weaken the load transfer efficiency at interface. Poor dispersion of nanoparticles leads to a premature failure of polymers, then reduces their strengths and failure strains. So far, the commonly-used mechanical mixing techniques prove to have a great difficulty in the preparation of agglomerate-free nanocomposites [12,13]. Moreover, incorporation of high loading nanofillers increases viscosities of polymers significantly, resulting in some processing problems. Compared with the traditional mechanical mixing methods, the sol-gel technique introducing nanoparticles into pre-polymers by chemical reaction, has been found to be effective in fabricating agglomerate-free nanocomposites, where the nanoparticles show excellently uniform dispersion, narrow size distribution and quasi-spheral shape; also, the thickening effects are relatively mild, even when the particle loading is as high as 20% in epoxy resin or other polymers or solutions [14]. Owing to these merits, the sol-gel nanoparticle/polymer systems become ideal models for elaborating the properties of the nanocomposites tested at various conditions, actually, their mechanical behaviors under static or quasi-static loadings have already been intensively investigated [15–18]. The possible fracture mechanisms of nanocomposites under quasi-static loading include nanoparticle debonding, plastic void growth and matrix shear banding, etc. [15,19–21].

The tetraglycidyl diaminodiphenylmethane (TGDDM) is a kind of epoxy resin having tetrafunctional epoxy groups on its molecule. TGDDM epoxy resins cured by aromatic amine hardeners have highly cross-linked microstructures, which yield excellent thermo-mechanical, electrical insulating properties but poor fracture resistance (The last property can be modified by adding thermoplastic particles, rigid particles or ductile difunctional epoxy resin). Owing to their excellent properties, the cured TGDDM-based materials show numerous engineering applications, especially for aviation, aerospace, transportation, construction and safety protection areas [22–24]. In these environments, high-speed collision and strike may happen to the materials occasionally, the mechanical behaviors at dynamic strain rates for these materials are, therefore, critical. As summarized in Table 1, recently, only a few works focus on the dynamic mechanical behaviors and the related strain-rate effect on epoxy composites reinforced with different fillers [25–29].

* Corresponding authors at: CAS Key Laboratory of Mechanical Behavior and Design of Materials, University of Science and Technology of China, Hefei 230026, China (S.-N. Luo and Z. Zhang).

E-mail addresses: zhangh@nanoctr.cn (H. Zhang), sluo@pims.ac.cn (S.-N. Luo), zhong.zhang@nanoctr.cn (Z. Zhang).

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Table 1
A brief review of the static and dynamic behaviors of epoxy-based composites.

Materials (epoxy/hardener)	Filler[a]	Yield strength of epoxy at low strain rate (MPa)	Range of strain rate (s^{-1})	Enhancement efficiency[c] (d, e)	Ref.
DGEBF/poly(propyleneglycol)diglycidyl ether	Silica nanoparticle (D = 140 nm)	~75[b]	0.0005–0.02, 2700–11000	9.6% (3 wt%, 2700 s^{-1})	[25]
DGEBA/piperidine	SBM nanoparticle (D = 60 nm) and CNT	~100	0.001–0.1, 1570–3500	CNT: 5.7% (0.25%, 2400 s^{-1})	[28]
DGEBA/cycloaliphatic polyamine	Graphene (T = 4 nm, L = 24 μm)	~100	0.01–10	No enhancement	[29]
DGEBA/polyoxyalkylamine	Glass particle (D = 42 μm)	<100	0.001, 300, 800–2900	22.9% (45 wt%, 2900 s^{-1})	[31]
DGEBA/Grandmer®VN–111	Diatom frustule (D = 8–15 μm, L = 3–20 μm)	52	0.001, 300–600, 1000	18.6% (15 wt%, 1000 s^{-1})	[30,32]
Fiberglass®Epoxy 2000	Alumina nanoparticle (D = 50 nm)	<66	0.0028, 3000	8.6% (5 wt%, 3000 s^{-1})	[12]

d: Optimal content of filler, at which the compressive strength reached maximum.
e: The test strain rate.
[a] D: average diameter; T: average thickness; L: average length.
[b] Obtained from the true stress-strain curves.
[c] Defined as the ratio of the increased strength of filled epoxy to the strength of neat epoxy matrix.

As is well known, the mechanical properties of the polymeric nanocomposites are dependent on many factors, such as the strain rate, filler fraction, filler morphology, filler orientation, dispersion level of filler and filler-matrix interface [25,30]. Due to the viscoelasticity of polymers, increasing the strain rate often increases the mechanical properties of epoxy-based composites [12,25,30,31]. For example, the compressive ultimate strength of epoxy resin can be enhanced by more than 300% at a high strain rate of 2900 s^{-1}, compared with that tested at quasi-static condition [31]. The filler fraction seems to have complicated effects on the mechanical properties of epoxy composites, no monotonous correlation is established between them [25]; optimal filler fraction regions at which the ultimate strengths of composite materials reach maximum are present, as reported in many works [12,31,32], a possible reason of which is due to the fact that the dispersion levels of fillers change with increasing filler fraction. In the literature, some models, such as Mulliken-Boyce and Ree and Eyring model, have been proposed to clarify the relationship between strain, strain rate, test temperature and flow stress for neat epoxy polymers, and the calculated values fit the experimental data well [27,28,33]. It should be noted that the works mentioned-above (Table 1) mainly focus on the bisphenol-A and bisphenol-F epoxy polymers, which have only medium cross-linked densities and common mechanical performance, little attention has been devoted to the highly cross-linked and high-performance TGDDM epoxy systems, and the mechanical response of epoxy in the strain rate ranging from 0.01 to 10000 s^{-1} has been rarely studied (see Table 1). In addition, whether the 'nano-effect' is still working at high strain rates for the TGDDM epoxy is unclear up until now.

Based on such a background, this work aims to investigate the strain-rate effect on the TGDDM epoxy polymer and its nanocomposites with sol-gel-formed silica nanoparticles. Special attention was paid to the mechanical behaviors of the materials at very high strain rates. To achieve the various test strain rates (0.001–3000 s^{-1}), different techniques (i.e. the common compression test and the split Hopkinson pressure bar impact test) were utilized respectively. The related failure mechanisms of the samples were discussed according to the morphologies of their fracture surfaces.

2. Experimental

2.1. Materials and preparation

A mixture of tetraglycidyl-4,4′-diaminodiphenylmethane (TGDDM, Shanghai Research Institute of Synthetic Resins, China) and bisphenol-A epoxy resin (DGEBA, Nanoresins AG, Germany) was used as the matrix at a weight ratio of 5:1. An aromatic amine, 4,4′-diaminodiphenyl sulphone (DDS, AVIC BIAM, China), was used as the hardener. A masterbatch containing 40 wt% of sol-gel silica and 60 wt% bisphenol-A epoxy resin served as the source for nanoparticles (Nanopox E470, Nanoresins AG, Germany). The specific equivalent weights of the TGDDM, DGEBA, DDS and masterbatch) were 125, 185, 62, 295 g/equiv., respectively.

The epoxy-based nanocomposite sample was fabricated by mixing the Nanopox E470 with an appropriate amount of the aforementioned epoxy resin at a rotation speed of 4000 rpm for 1 h in a heated oil bath of 100 °C. A stoichiometric ratio of the DDS was then added to the mixture and stirred at a lower mixing speed of 300–400 rpm at 135 °C for 0.5 h. Afterwards, the mixture was degassed in a vacuum oven, cast into release-agent-coated aluminium moulds and cured at 180 °C for 2 h, post-cured at 200 °C for another 2 h. The cured sample contained 10 wt% silica nanoparticles. For comparison, the cured neat epoxy sample was also prepared by the same procedure.

2.2. Compression tests at various strain rates

Cylindrical samples ($\Phi 5 \times 5$ mm) were used for all strain-rate experiments, unless otherwise stated.

2.2.1. At low strain rates

The compression tests at low strain rates (0.001–0.1 s^{-1}) were performed using a Zwick universal testing machine with a load cell of 20 kN. The cylindrical samples were lathed and polished to achieve smooth parallel ends perpendicular to the cylindrical axis. Then they were lubricated with petroleum jelly to minimize friction and avoid resin cracking near the loaded ends. The compliance of the testing machine for compression was performed based on a technique proposed in Ref. [34]. Then the actual deformation of the sample was calculated by subtracting the non-sample displacement of the testing fixture from the total displacement recorded by the actuator, and the compressive modulus of the sample was calculated between corrected zero point and 1% compressive strain by linear curve fitting. At least six specimens were tested for each sample at each strain rate, allowing evaluation of the test reproducibility and obtaining an average value.

2.2.2. At intermediate strain rates

The compression tests at intermediate strain rates (1–35 s^{-1}) were performed using an Instron-8801 universal testing machine.

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