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Direct three-dimensional imaging of the fracture of fiber-reinforced plastic under uniaxial extension: Effect of adhesion between fibers and matrix

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ABSTRACT

The three-dimensional morphology and a mechanical property of a fiber-reinforced plastic (FRP) have been investigated under uniaxial extension. A custom tensile apparatus for X-ray computerized tomography was developed for this purpose. The two FRPs used in the present study consisted of nylon 6 with glass fibers. In one of the FRPs, the fiber surfaces were treated to adhere to the nylon 6. It was observed that the fibers tended to align along the extension axis, and that cavitation occurred simultaneously during the extension. In the case of the FRP with glass fibers without any surface modification (hereafter, referred as "neat" glass fibers), void formation was dominant over the whole extension range. In the case of the FRP with surface treatment, fiber alignment occurred first and was followed by void formation. A numerical simulation was carried out to study the stress concentration around a fiber with such morphological changes during extension. Through quantitative measurements of fiber orientation and void volume, together with predictions from the numerical simulations, the effect of fiber/matrix adhesion on the morphological developments and mechanical properties of the FRPs was discussed.

1. Introduction

In order to satisfy the increasing demands for high-performance (strong) polymeric materials, polymers are often mixed with inorganic or metal components [1–4]. In such composite materials, the polymers usually constitute the matrix whereas the metal or inorganic compounds are mixed to achieve certain additional properties, such as mechanical, electrical, or optical properties. In some cases, however, the special mechanical and physical properties of the reinforcing materials enhance the properties of the matrix itself. A fiber-reinforced plastic (FRP) is one of the best-known polymeric composites in use today. The matrix is a plastic (polymer) with relatively lower density, which is reinforced by stronger and stiffer reinforcing filaments or fibers. The main advantage of FRP is that it is both lightweight and strong. It is a highly efficient material for automotive, marine, and construction industries around the world [5]. Other interesting features of FRP (*e.g.*, its electrical insulating properties, radio-wave permeating properties, and corrosion resistance) are also attracting considerable attention [6]. Along with their industrial applications, FRPs are used in high-tech areas such as nuclear fusion reactors, airplanes, space equipment, and low-temperature pressure containers [6]. Although such increasing demand and widespread use testify to the high mechanical strength of FRPs, they are not without their drawbacks. These include high crack-propagation rates [7–9] and internal damage related to the detachment of matrix and fibers [10], all of which degrade the utility of strength in FRP as a structural material.

Research on the mechanical properties of FRP is categorized by

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properties [11] such as the elastic modulus [12,13], fracture toughness [12,13], fatigue-crack growth rate [14–16], fracture mechanism [14–16], and fractography [17–19]. Many of these studies emphasize the relations between fiber content and mechanical properties, fatigue strength, and crack growth behavior. Some properties, such as the strength and elasticity, are further influenced by the shape and orientation of the fibers to expansion direction within the matrix [20], and thus the values of these properties do not strictly follow a unified rule [12,13]. Similarly, fatigue strength and crack growth behavior both have their origin in the detachment of matrix and fibers, and hence the matrix/fiber interface plays a key role in the mechanical behavior of FRP.

The role of the matrix/fiber interface in the mechanical strength of FRPs has long been a focus of attention, and thus many studies have examined it [21–25]. Phenomena at the interface between an elastic material and a stiff material involve complicated physics. To clarify the relation between the interface and the mechanical strength of an FRP, it is crucial to observe the morphologies before and after fracture. It would be preferable to *directly* follow the same position inside the specimen during the fracture process in three dimensions. By performing such *in situ* three-dimensional (3D) imaging [26,27], the formation of voids could be ascertained, as could the rearrangement of fibers during the fracture. This would provide valuable insights into the source of the mechanical strength of FRPs.

Many studies utilize scanning electron microscopy (SEM) to observe the morphologies of fractured FRP cross-sections. However, morphologies obtained that way could be different from the actual internal morphology. A reasonable inference has to be made from the SEM images as to the nature of the fracture mechanism. Optical interferometry has allowed an overview of the FRP fracture process [28–33], and the cruciform-specimen method has revealed the fracture process and interfacial behavior around a single fiber [34–37]. However, a drawback of these methods is that phenomena such as the fiber arrangement, crack formation, crack growth, and specimen deformation cannot be specified.

A powerful tool for visualizing the internal 3D arrangement of micron-sized structures is X-ray computerized tomography (X-ray CT) [38]. Although laser-scanning confocal microscopy with a similar resolution to that of X-ray CT has also been used to study the static and dynamic structures of polymer materials [39–41], such 3D light microscopy cannot be used for FRP because FRP normally contains 20–30 wt % of fibers and hence is opaque. X-ray CT is the most suitable technique for the 3D visualization of an FRP [42–45]. In addition, X-ray CT installed at a synchrotron source has been used recently to investigate dynamical phenomena in polymer materials [46,47].

In the present study, we used an *in situ* X-ray CT technique to investigate the fracture processes in FRPs during uniaxial extension. Two types of FRP composed of a polymer matrix and short glass fibers either with or without silane-coupling-agent treatment were used to understand the FRP fracture mechanisms. Particular attention was paid to the adhesion between the fibers and the polymer matrix. The stress was measured as the FRP sample was extended, and the relation between stress and morphological changes (*e.g.*, cavitation/void formation and orientation of fibers) was studied.

2. Experimental details

2.1. Specimen preparation

The specimen for uniaxial extension was prepared by mixing 1 wt % of glass fibers (Milled Fiber MF06JB1-20; Asahi Fiber Glass Co., Ltd.) with 99 wt % of nylon 6 (AMILAN CM1007; Toray Industries, Inc.). The corresponding volume fraction of the glass fibers was approximately 0.5 vol %. Two types of fiber were used, coded as gf_0 and gf_1. Type gf_0 was 10 μm in radius, 30–100 μm in length, with no surface treatment. Type gf_1 was 13 μm in radius, 30–100 μm in length, with surface treatment by the silane coupling agent 3-(2-Aminoethylamino)propyltrimethoxysilane to make the nylon 6 adhesive [48]. The two types of FRP made using gf_0 and gf_1, are referred to as FRP_0 and FRP_1. The FRP pellets were prepared by melt-kneading process. In the kneading instrument, the clearance between screw and cylinder is 100–200 μm. The wider gap of the instrument than the length of the fibers suggests it is unlikely for the fibers to be shorten by the process. The prepared pellets were melted and vacuum pressed at 230 °C, which is 5 °C above the melting point of nylon 6. The specimen was then shaped into a sheet and punched into dumbbell-shaped tensile-test specimens whose dimensions are shown in Fig. 1b. In order to keep the moisture content of the specimens as low as possible, they were stored in a box with desiccant before the extension experiments.

It is particularly important to observe the same position on a specimen as it is stretched. Therefore, because the observation volume moves during extension, particles of Cu powder (approximately 10 μm in diameter) were dispersed on the FRP surface as position markers. The volume that was subjected to 3D structural analysis was defined by the positions of two specific Cu particles, as shown schematically later in Fig. 5a.

2.2. Three-dimensional imaging under uniaxial extension

An X-ray CT instrument (Nanofocus ELESCAN, NS-SPC-00610NSF-I(4)B; Nittetsu Elex Co., Ltd.) was used for 3D imaging during the tensile tests. The X-ray wavelength was 0.71 Å(Mb was used as the target). The tube voltage and current used in the present study were 50 kV and 150 μA, respectively. The projected X-ray images were acquired after applying each strain (ε) for 3 h. A projection was taken every 0.3° at a magnification of 33× for approximately 1 h.

Together with the X-ray CT, a specially developed custom tensile apparatus that was small enough to be installed inside the X-ray CT instrument was used for 3D imaging under uniaxial extension (Fig. 1a). The tensile machine has a pair of chucks that grip the specimen by its edges, a stepping motor for extending the specimen, and a load cell to measure the load on the specimen. The specimen chucks are enclosed by acrylic tubes. The maximum stroke and force capacity are 220 mm and 10 kgf, respectively. The extension rate is adjustable in the range 0.01–1 mm/min.

Each extension experiment in this study was performed at an extension rate of 0.1 mm/min, and the associated stress was

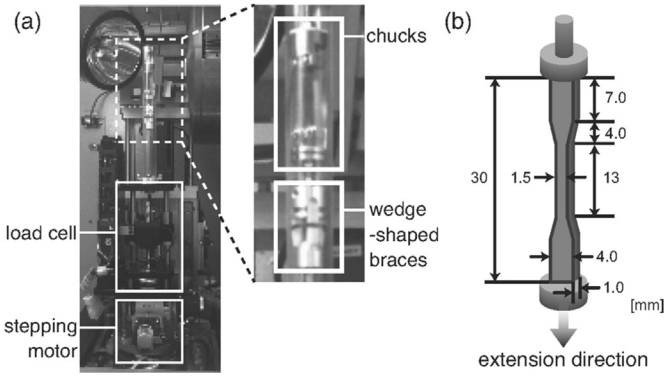

Fig. 1. (a) Photograph of tensile apparatus for X-ray computerized tomography (CT). (b) Schematic illustration of geometry of dumbbell tensile-test specimen.