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Nanoscale evaluation of multi-layer interfacial mechanical properties of sisal fiber reinforced composites by nanoindentation technique

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ABSTRACT

The multi-layer structure of plant fibers has been revealed qualitatively by qualitative microscopic characterization and associated with multi-stage failure behaviors of plant fiber reinforced epoxy composites. To quantitatively evaluate the nanoscopic mechanical properties of sisal fiber (a typical plant fiber) reinforced epoxy composites (SFRCs) involving the unique structural characteristics, elastic modulus and hardness of the epoxy matrix and cell wall layers of sisal fiber along with interfacial mechanical properties were measured by applying the nanoindentation technique. A series of indents were conducted at selected positions from the matrix to each layer of the fiber cell walls to ascertain transition zones of the multi-layer interfaces. Single-step and multi-step nanoindentation methods were respectively employed on the multi-layer interfaces of SFRCs to present their distinct mechanical properties in terms of modulus and hardness, energy dissipation, crack initiation and propagation upon compressive loading. This study measures the transition zones of the multi-layer interface and the interfacial failure load, which consequently facilitates a quantitative analysis of fracture mechanisms for SFRCs with a multi-scale and multi-layer structure.

1. Introduction

To date, plant fibers derived from natural resources have been widely used in civil and automotive industries and became promising reinforcements due to their numerous advantages including abundant availability, renewability and interesting mechanical properties [1–3]. In order to promote applications of plant fiber reinforced composites (PFRCs) in engineering practice, researches regarding optimizations of plant fiber yarn structures (fiber twist and yarn linear density) and manufacturing processes (curing pressure, time and temperature) have been widely carried out by scholars to obtain high-performance PFRCs by integrating the process of design and manufacturing [4–8]. Besides the mentioned aspects, mechanical performances of composite materials including PFRCs are also largely dependent on the interfacial adhesion properties, which are correlated to stress transfer efficiency at the interface. However, rare reported works were found to comprehensively quantify the interfacial properties of PFRCs in the nanoscopic perspective.

Previous researches have concluded that single plant fiber possesses a multi-scale and multi-layer structure which is distinct from synthetic fibers with a homogeneous structure [9]. Plant fibers were reported to be depicted as a composite structure from macro-scale to nano-scale. The elementary fibers are glued together by pectin (is termed as CML, Compound Middle Lamellae) to form a single plant fiber (also called technical fiber), as shown in Fig. 1. A hole located in the center of the elementary fiber is called lumen. Such a hierarchical organization produces multi-interphase regions with different morphological characterizations. Without loss of generality, the interphase transition regions are usually small, which induces challenges to achieve an accurate evaluation of the nanoscopic interfacial properties. Therefore, it is of necessity to develop a reliable method to characterize the interfacial properties of plant fibers and their reinforcing composites at the nanoscopic level.

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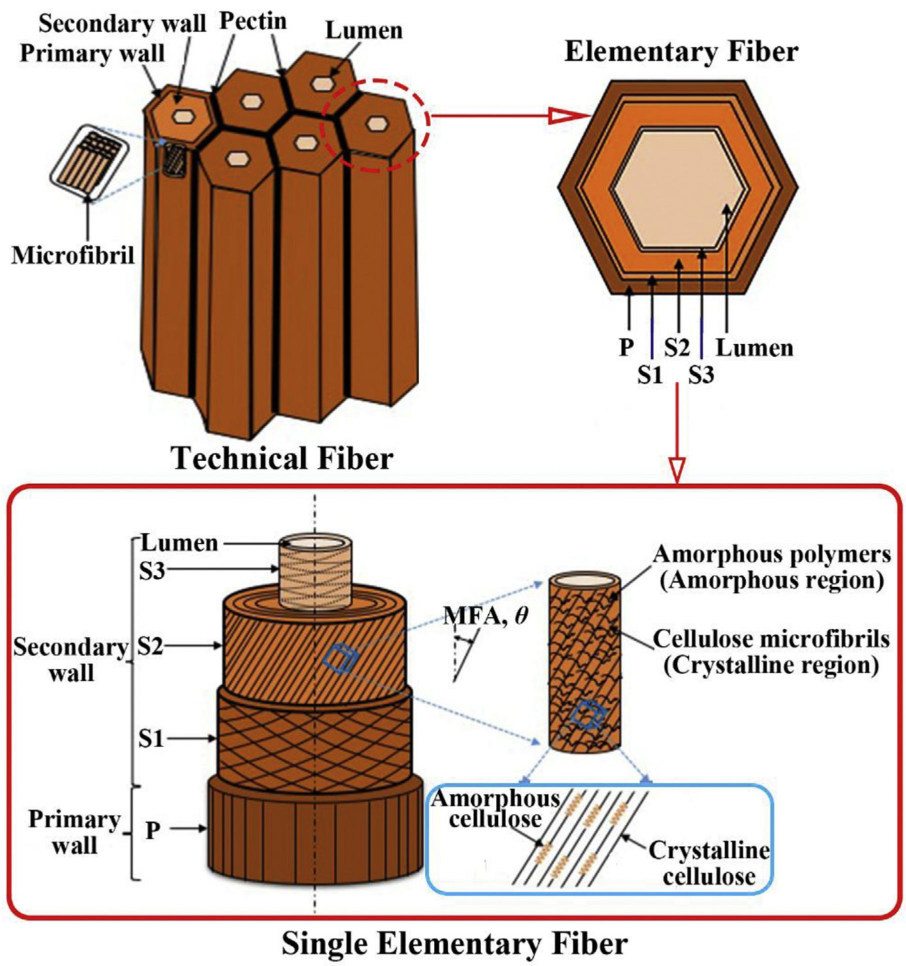

Fig. 1. Diagram for structure of a single sisal fiber.

Amongst the nanoscopic evaluation methods regarding material interfacial properties, nanoindentation measurement is a suitable, effective and promising technique to characterize the interphase zone and quantitatively evaluate the interfacial mechanical properties in fiber-reinforced polymer composites [10–18], especially for PFRCs with multi-scale and multi-layer interfaces. Several researchers have attempted to measure the interphase properties in carbon or glass fiber reinforced composites (CFRCs or GFRCs) [14–16]. Urena et al. [14] employed the nanoindentation method to analyze the interfacial mechanical properties of short CFRCs coated with metallic films. They found that the nanoindentation technique can achieve a complete characterization of the interfaces in the composites, which made it possible to measure the interfacial fracture and friction strengths in nano-scale. Similarly, Gao et al. [15] and Kim et al. [16] investigated the interphase nano-scale property in GFRCs and the results revealed effective interphase thickness in such composites was less than 1 μm. These pioneering works presented the availability of the nanoindentation technique on characterizing interfacial morphology (thickness of interface zone) and determining interfacial mechanical properties (elastic modulus and hardness) for the composites in nano-scale. However, limited studies have been carried out regarding evaluating interfacial properties of natural fiber reinforced composites (NFRCs) [17,18]. Lee et al. [18] evaluated the interphase mechanical properties of cellulose fiber reinforced polypropylene composites by applying a continuous stiffness technique in nanoindentation tests. The results revealed interphase property transition between the fiber and matrix and concluded that indent area played a critical role in accurately determining the mechanical properties of the interphase region. Though the results are promising, the above-mentioned two studies are mainly focused on the basic mechanical or interfacial properties between the fiber and matrix of NFRCs, ignoring the distinct hierarchical structure (i.e., multi-scale and multi-layer) of natural fibers. Thus, relevant research endeavors in extending such a technique to the quantitative evaluation of hierarchical interfacial properties and interfacial failure of PFRCs with a multi-scale and multi-layer structure are worthy of investigating and validating.

Inspired by the proven efficiency of the nanoindentation technique in evaluating interfacial properties of the artificial fiber reinforced composites in nano-scale, present study is dedicated to quantitatively measuring interfacial mechanical properties of sisal fiber reinforced epoxy composites (SFRCs) with a multi-scale and multi-layer structure by using the nanoindentation technology. To achieve this goal, a series of indents derived from matrix to each layer of cell walls of the sisal fiber (S1, S2 and S3 layer) were employed to identify the transition zones of the multi-layer interfaces. Optical Microscopy (OM), Atomic Force Microscopy (AFM) and Scanning Electronic Microscopy (SEM) characterizations were used to observe the multi-layer interface morphology of sisal fibers and the morphologies of indents. Single-step and multi-step nanoindentation measurement at various peak indentation loads