Polymer Testing 50 (2016) 297–300

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Short communication: property modelling

Length-scale dependence of variability in epoxy modulus extracted from composite prepreg

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ARTICLE INFO

Article history:
Received 23 October 2015
Accepted 21 December 2015
Available online 28 January 2016

Keywords:
Polymer matrix composites (PMCs)
Mechanical properties
Statistical properties
Microstructural analysis

ABSTRACT

Property variability in conjunction with morphological variability are important sources of uncertainty in composite modeling. While image processing of experimental microstructures has enabled accurate quantification of morphological variability, the characterization of material variability is not as well established. In this study, the local material properties of epoxy extracted from a prepreg sheet was determined using nanoindentation with a spherical indenter tip with a radius of 50 μm. Indentations were carried out at four different indentation depths to evaluate the change in the variability of epoxy modulus with the sampling volume. For each length scale studied, 40 indentations were carried out to determine the variability in epoxy modulus. A significant decrease was observed in the coefficient of variation as the indentation depth increased. The corresponding modulus distributions were quantified. The results suggest that, similar to morphological variability, material variability is length-scale dependent and the appropriate variability associated with the selected length scale must be considered for stochastic modeling of composite structures.

1. Introduction

Despite large variations in microstructural features of real composites, ideal models with average constituent properties are typically used in modeling composite structures. While such models can predict the macroscopic elastic response, damage initiation and evolution is strongly influenced by microstructural variability [1,2] because the onset of damage is a local phenomenon that depends on local stresses resulting from specific microstructural features. For instance, a local cluster of fibers may cause high stress gradients, making it a potential failure initiation locus. Therefore, accurate prediction of failure and its variability at the macroscale requires accurate modeling at the microscale, taking into account microstructural uncertainties, which are present in the form of morphological and material variability [3–5].

Morphological variability such as variation in fiber volume fraction [6], relative fiber positions, fiber radius [7], fiber orientation [8], and void morphology [9] have been previously characterized via image processing. Stefanou et al. [10] investigated the effect of microstructure geometry such as inclusion shape and volume fraction on the effective properties. While characterization of morphological variability is necessary, it is not sufficient for comprehensive modeling of a composite microstructure. The variability in material properties may also serve as another important source of uncertainty. Other researchers have investigated the effect of material property variability on the failure of composites. Lacy et al. [11] incorporated fiber strength variability in their progressive failure analysis. Shang and Shi [12] investigated the effect of fiber/matrix interphase strength variability on transverse tensile properties. Fiber strength variability has also been incorporated in textile composites [13]. Sanei and Fertig [14] incorporated epoxy modulus variation in failure prediction of an idealized microstructure.

For quantifying material variability, conventional mechanical testing such as tensile testing and dynamic mechanical analysis [15] return the average modulus without any information regarding variability throughout the sample. In contrast, nanoindentation is well-suited for this purpose, as it is a robust method for local determination of properties. Nanoindentation on composites and their constituents has received much attention in recent years [16–18]. Constantinides investigated the *in situ* properties of composite constituents using nanoindentation [19]. They investigated two major indentation depths, one smaller than the reinforcement diameter to obtain constituent properties, and the other

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http://dx.doi.org/10.1016/j.polymertesting.2015.12.015
0142-9418/

much larger than the reinforcement diameter to obtain homogenized properties. Hardiman et al. [18] characterized *in situ* and bulk matrix properties of fiber reinforced polymer (FRP) using nanoindentation. In addition to fiber and matrix properties, nanoindentation has been used to determine the properties of the interphase [16,20]. Gibson has given a thorough review [17] of mechanical property determination of composites and their constituents using nanoindentation. In this work, nanoindentation is used to determine the variability in epoxy modulus as a function of sampling volume.

In addition to determining property variability, it is important to evaluate the length scale dependence of such variabilities. It has been shown that variability in a primary composite morphological feature, fiber volume fraction, is length-scale dependent such that for any length scale, there is a unique distribution associated with it. Sanei and Fertig [21] showed that, as expected, the width of the distribution of fiber volume fraction decreases with increasing length scale. Length-scale dependence of morphological variability suggests that material variability might be length scale dependent as well. To evaluate such possible length-scale dependence, indents were produced for four different indentation depths to investigate the size dependence of variability in epoxy modulus. The findings of this study underscore the fact that not only the variability in material properties must be incorporated into the model but the length-scale dependence of the variability must be accounted for in stochastic modeling of microstructures. Considering length scale dependence of the property distribution is an essential step towards addressing the discrepancies observed between simulation and experimental results in determination of statistical macroscopic properties [22].

2. Procedure and methodology

2.1. Sample preparation

Nanoindentations on composite constituents have been carried out on bulk constituents and on constituents *in situ*. Debate has surrounded the accuracy of *in situ* versus bulk characterization of constituents in composites [18]. *In situ* testing of composite constituents is significantly influenced by the effects of surrounding medium such that the *in situ* resin properties are heavily influenced by the fiber constraint. Nanoindentation of *bulk* polymers may not represent the polymer *in situ* because uniform cross linking occurs across the sample, which is not likely to occur in presence of fibers. In this study, a third approach is attempted such that the epoxy sample is prepared by extraction from preimpregnated unidirectional material Hexcel Panex 35/M9.7. The extraction was performed under similar temperature and pressure conditions used in manufacturing of a laminate. The final epoxy surface was smooth without any visible defects.

2.2. Experimental procedure

Indentations were performed with an Agilent nanoindenter G200 system (Keysight technologies, Santa Rosa, CA, USA). To avoid the polymer size effect, indentations were conducted with a 50 μm radius spherical tip [23,24]. Loading and unloading time of 20 s was applied for all indentations. A holding time of 50 s at the maximum load was applied to minimize the effect of creep on the results. Forty indentations were carried out for each indentation depth. Four different indentation depths ranging from 1.2 to 10 μm were considered.

3. Analysis

The two common approaches for determining the elastic modulus via nanoindentation with a spherical tip are the Hertz [25] and Oliver and Pharr [26] methods. The presence of permanent deformation renders the Hertz assumptions invalid, whereas the Oliver and Pharr method would be a viable approach and is used in this study.

The two common intrinsic problems in nanoindentation of polymers are adhesion and surface detection [27–30]. To ensure accurate surface detection and contact area size, the stiffness limit of 100 N/m is selected, (see Ref. [31] for thorough discussion on accurate selection of stiffness limit). The load displacement curves for four applied loads are depicted in Fig. 1. The effect of adhesion is evident on the load displacement plot. Seltzer et al. [32] concluded that the presence of adhesion is manifested in a negative curvature (downward curvature) of the plot, which is evident for the highest contact radius, and the positive curvature (upward curvature) indicates the lack of adhesion which is the case in the lowest contact radius (see Fig. 1). The most common available models addressing adhesion in nanoindentation, namely, Johnson-Kendall-Roberts (JKR) [25], Derjaguin-Muller-Toporov (DMT) [28], and Maugis-Dugdale (MD) [29], are not applicable here as they are developed for viscoelastic materials in absence of permanent deformation. Maugis and Pollock (MP) [33] generalized the JKR theory to include the effect of plastic deformations in adhesive forces, however, MP modifies the hardness in a manner that is not applicable to spherical indentation.

4. Results and discussion

The results obtained indicate the presence of variability in epoxy modulus. The cumulative distributions of epoxy modulus measured at four different contact radii are shown in Fig. 2. The best fit was evaluated based on Bayesian [34] and Akaike information criteria [35]. Sixteen different distributions were considered (adopted from MATLAB Central/File Exchange directory [36]). These distributions included: Exponential, Extreme value, Gamma, Generalized extreme value, Generalized Pareto, Inverse Gaussian, Logistic, Log-logistic, Lognormal, Nakagami, Normal, Rician, t location-scale, Beta, Birnbaum-Saunders, and Weibull. For three indent depths a Weibull distribution is one of the two best fits and it fits the fourth reasonably well. Therefore, a Weibull distribution can be a reasonable approximation for epoxy modulus distribution. The

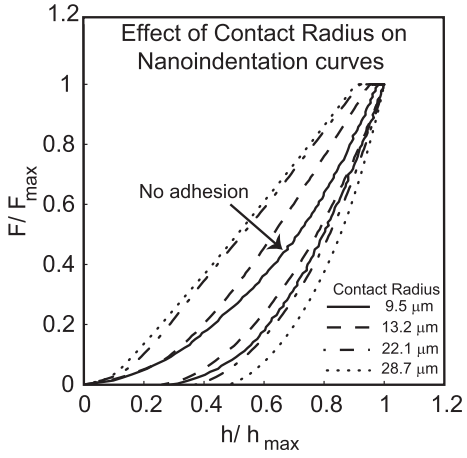

Fig. 1. Load–Displacement curves for four different applied loads showing the influence of adhesion on the curve.