

Evaluation of Mechanical Properties and Effective Thickness of Composite Interfaces

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ABSTRACT

Polymer matrix composites (PMCs) have been increasingly used for high temperature applications in automotive and aerospace industries. Under such conditions, the materials will experience thermomechanical degradation which can cause premature failure of the composite structures. The desire to predict the damage and lifetime of high temperature polymer matrix composites (HTPMCs) and the structural durability for structural applications has been elusive. The success of the multiscale modeling and analysis relies on the experimental capability of properly characterizing the evolution of mechanical behavior of each constituent: matrices, fibers, and interfaces. This paper presents a novel technique, nanoindentation, to identify the interfaces between dissimilar materials and subsequently to evaluate the physical and mechanical properties across the interfaces. It is proposed to use a nanoindenter equipped with small spherical tip, <40 nanometers in radius, to indent across the interfaces of dissimilar materials. The proposed method has been validated by conducting a large number of virtual experiments through 3-dimensional finite element simulations, by varying the properties of the two dissimilar materials, including various combinations of modulus (E_1/E_2), yield strength (σ_{y1}/σ_{y2}), hardening index (n_1/n_2), interface sizes (R/T), Poisson's ratio (ν), etc. The mechanical properties across the interfaces have been obtained, and a quantitative model for predicting the interface sizes has been established.

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INTRODUCTION

Polymer matrix composites (PMCs) have been increasingly used for high temperature applications in automotive and aerospace industries. Example applications include the automotive engine covers and turbine engine exhaust structures [1,2,3,4]. The matrix systems in the high temperature PMCs are predominantly thermoset polymers. These polymers are chosen since they have highly cross-linked structures that form during the curing process. Typical thermosets include polyimides, epoxies, unsaturated polyesters, vinyl esters, etc. One of the major concerns in using polymer matrix composites at high temperature environments is the thermo-oxidative degradation of the polymer matrix and the matrix-fiber interfaces. Exposed to elevated temperature, the free surfaces of PMCs are susceptible to oxidation. When exposed to thermo-mechanical loading, the result is accelerated degradation and ply cracking which in turn introduces new free surfaces. Ultimately, the thermo-oxidative degradation reduces the life and durability of the composite system. Thus, the ability to fully understand and characterize the physical and

mechanical responses resulting from thermo-oxidative processes is paramount to the continued development and increased use of high temperature PMCs in the industry. The degradation of polymer resins used in high temperature applications can be a result of either physical aging or chemical aging, or both. A dominant chemical aging process for high temperature PMCs is thermo-oxidative aging. A three-region model has been proposed by Tandon, et al [5], to explain the mechanism of the thermo-oxidative degradation in polymers. According to this model, the surface oxidative layer is separated from the unoxidized polymer with an active reaction zone (Figure 1). The oxygen (O_2) in air diffuses through the polymer and then consumed by the oxidation reaction. The parameter ϕ shown in Figure 1 is called the polymer availability state variable and the parameter ϕ_{ox} characterizes the completely oxidized polymer. Once a region is fully oxidized (called the Oxidized Layer), the oxidation reaction is terminated and oxygen can diffuse through it. Then, oxygen begins to react in the adjoining region (called the Transition Region), where $\phi_{ox} < \phi < 1$. In the region far from the exposed surface (called the Unoxidized Interior), no polymer has been oxidized ($\phi=1$).



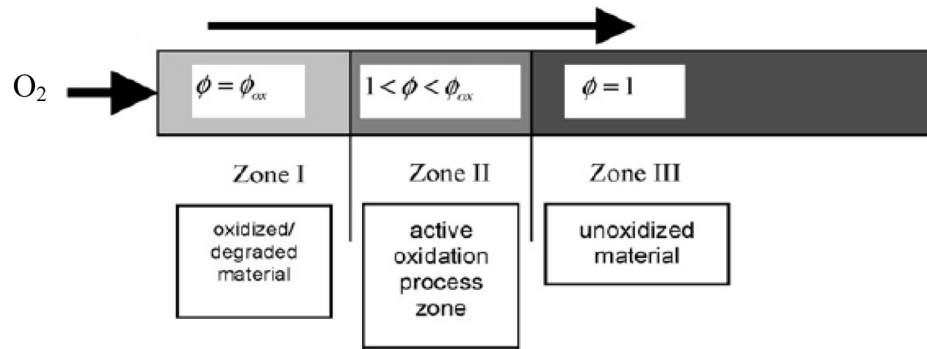


Figure 1. Schematic of the three-zones in thermo-oxidation of polymers [5].

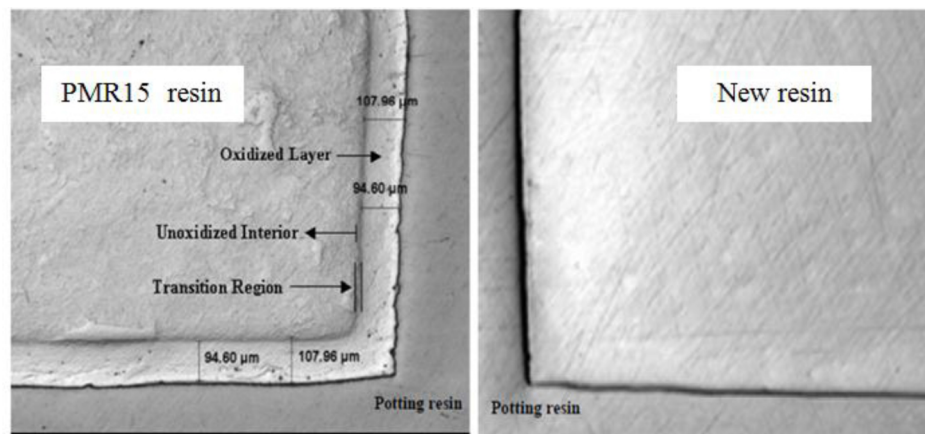


Figure 2. Photomicrographs showing the structure changes due to thermo-oxidization. (a) PMR-15 resin after 196 hrs of aging at 343°C; (b) New resin after 1200 hrs of aging at 343°C [6].

Because of the thermo-oxidative degradation, it is expected that the physical and mechanical properties will change in the polymer resins. However, depending upon the material systems, it can be rather difficult to observe and characterize such changes by using conventional techniques, such as the light microscopy, scanning electron microscopy, etc. Figure 2(a) shows a photomicrograph of PMR-15 polymer resin isothermally aged in ambient air at 343°C for a period of 196 hrs [6]. The optical micrograph clearly shows the three regions due to oxidation: oxidized region, transition region, and unoxidized region. This observation at least helps measuring and characterization of the transition region, although the exact boundaries of the transition zone are still hard to be determined. Figure 2(b) is the photomicrograph of another polymer resin aged for 1200 hrs at 343°C [6]. Unlike PMR-15, the oxidized layer could not be observed for this material by light microscopy. Thus, new technique for identifying and characterizing the interface (transition zone) is needed.

Similar issue can be extended to the interfaces in the fiber reinforced polymer composites [7]. The classical definition of interface in fiber reinforced composites is that “an interface is a surface formed by the common boundary of reinforcing fiber and matrix in contact which constitutes the bond in-

between for transfer of load. It has physical and mechanical properties which are unique and not those of either the fiber or the matrix” [8]. The interface between reinforcing fibers and matrix is believed to play an important role in the overall composite properties. However, due to the small size, it is often difficult to properly characterize the properties of the interfaces.

This paper presents a new technique, nanoindentation, to identify the interface between dissimilar materials, and subsequently to evaluate the physical and mechanical properties across the interface. It is proposed to use a nanoindenter with spherical tip to indent the interface between dissimilar material, which could represent the interface (transition face) between oxidized and unoxidized polymers and the interface between rigid fiber and polymer matrix. The nanoindentation experiments will be simulated by using the finite element method. Various interface scenarios will be considered by varying the properties of the two dissimilar materials, including various combinations of modulus, yield strength, hardening index, and interface sizes. Methods will be developed to predict the sizes (thickness) of the interfaces as well as their mechanical properties.

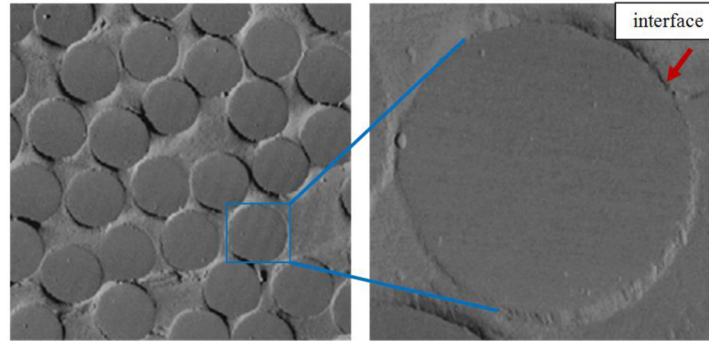


Figure 3. Photomicrographs showing the interface at the fiber reinforced polymer composites.

VIRTUAL NANOINDENTATION EXPERIMENTS

It is proposed that the quantitative nanoindentation technique be used to study the properties at the interfaces. Nanoindentation is a new method to characterize the mechanical properties of very small volumes like thin films or interfaces, coatings and surface layers including those modified by ion implantation since the layer need not be removed from the substrate. The spatial variation of the properties can also be obtained by indentation on the scales of micro, nano and pico levels with good resolution. Employing high-resolution sensors and actuators, nanoindentation can continuously monitor the loads and displacements during loading and unloading as the indenter is driven into and withdrawn from the test material. From the load-depth curves, many quantitative information such as contact area, contact depth, stiffness, hardness, and elastic modulus are obtained. For this purpose, nanoindentation is used to characterize the characteristics at the interfaces of dissimilar materials. In the present work, the finite element method is used to simulate the indentation experiments in composites. The interface could represent the region between oxidized and unoxidized polymer resins or the region between rigid fiber and polymer matrix. A general interface model can be sketched as seen in [Figure 4](#).

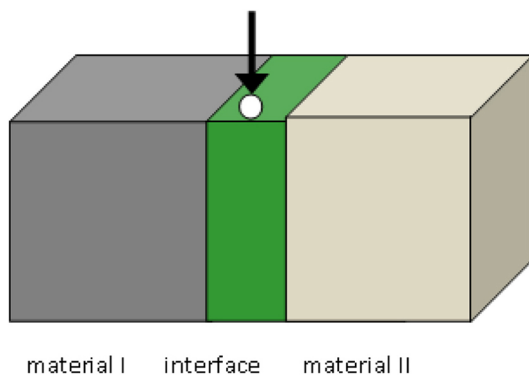


Figure 4. A generic model representing the interface of dissimilar materials.

The virtual experiments were conducted using commercial program ABAQUS [9]. Due to symmetry, only half of the structure was modeled. Much of the 3D analysis is made easy with the options available in the part module of Abaqus/Standard. Once the 2D model is built, it is rather easier to extrude, revolve or sweep about an edge or axis. As a result of which, the 3D model used in the present analysis to study the material hardness and elastic modulus by extruding the already existing 2D model in the z direction by a thickness of 500mm. The element type used for the analysis is C3D8R: Eight node brick element with reduced integration. As one or more degrees of freedom were to be arrested for a particular node, single point constraints (SPC), were used in the present analysis. In the present work it is assumed that the friction effect is negligible and $\mu=0$ is defined in all the models assuming there is no slip between the surfaces in contact. The interaction between the indenter and the specimen is modeled as contact pair without any friction. The indenter surface is defined as the 'master' surface since the indenter is rigid body. The top of the specimen is the 'slave' surface. The power law work hardening model was used to define the plastic properties. The modified uniaxial stress-strain (σ - ϵ) curve of a stress free material can be expressed as

$$\begin{cases} \sigma = E\epsilon & \text{for } \epsilon \leq \frac{\sigma_y}{E} \\ \sigma = K\epsilon^n & \text{for } \epsilon \geq \frac{\sigma_y}{E} \end{cases} \quad (1)$$

where E is elastic modulus, σ_y is yield stress, n is the work hardening exponent. When n is zero, the above equation reduces to an elastic-perfectly plastic material. To completely characterize the elasto-plastic properties of a power-law material, four independent parameters, i.e., elastic modulus E , yield stress σ_y , work-hardening exponent n , and Poisson's ratio ν , are needed. A true stress strain data spreadsheet at first is generated from the Equation Error! No text of specified style in document.-1. Then using the below equation, plastic strain (ϵ_p) is calculated.