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THERMO-ELASTIC NONDESTRUCTIVE EVALUATION OF FATIGUE DAMAGE IN PMR-15 RESIN (PREPRINT)

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Thermoset polyimide resins are used as the polymer matrix in high temperature composites for aerospace applications such as engine shrouds. At these locations the components have to withstand high temperatures and significant vibration. A number of studies have investigated the effects of thermal exposure on mechanical properties of polyimide resins, and the effects of fatigue on thermoplastics have been discussed at length. However, the effects of fatigue on thermosets, in particular polyimides, have largely been overlooked. In this paper we present studies of nondestructive evaluation of fatigue damage in a thermoset polyimide resin, PMR-15, performed by measuring the changes in the evolution of heat in the samples during cyclic loading. The temperature changes are measured using a high sensitivity IR camera as a function of number of fatigue cycles. Interrupted fatigue tests were performed on four samples. The temperature rise during an increment of fatigue cycling shows two linear regions each with a different slope (region 1 and region 2). Region 1 remains constant for every increment of fatigue, while region 2 increases.

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THERMO-ELASTIC NONDESTRUCTIVE EVALUATION OF FATIGUE DAMAGE IN PMR-15 RESIN

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ABSTRACT. Thermoset polyimide resins are used as the polymer matrix in high temperature composites for aerospace applications such as engine shrouds. At these locations the components have to withstand high temperatures and significant vibration. A number of studies have investigated the effects of thermal exposure on mechanical properties of polyimide resins, and the effects of fatigue on thermoplastics have been discussed at length. However, the effects of fatigue on thermosets, in particular polyimides, have largely been overlooked. In this paper we present studies of nondestructive evaluation of fatigue damage in a thermoset polyimide resin, PMR-15, performed by measuring the changes in the evolution of heat in the samples during cyclic loading. The temperature changes are measured using a high sensitivity IR camera as a function of number of fatigue cycles. Interrupted fatigue tests were performed on four samples. The temperature rise during an increment of fatigue cycling shows two linear regions each with a different slope (region 1 and region 2). Region 1 remains constant for every increment of fatigue, while region 2 increases. The onset of region 2 occurs at the same increase in temperature due to hysteretic heating for all samples. Experimental observations are explained using a phenomenological two phase model based on crosslinking density variations in observed in other thermoset resins at microscopic scales. The results of these experiments are discussed in reference to utilizing this technique for detection and evaluation of fatigue in PMR-15 resin and composites.

Keywords: Fatigue, Thermo-elasticity, PMR-15
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INTRODUCTION

New aircraft are expected to use more composite materials. This drives the need for nondestructive evaluation (NDE) of composite materials. Current aircraft are flown beyond their design lives and have to be inspected for fatigue and other types of damage. It is reasonable to anticipate that new aircraft with large amounts of composites will need to be inspected for fatigue damage in the future. This necessitates a need to understand the NDE signal-material relationship to evaluate the component state. It is the understanding

of this relationship that will enable NDE to detect the subtle changes in the material associated with the onset of damage to be observed.

POLMER VISCOELASTIC AND HYSTERETIC HEATING

High temperature thermoset monomers have three or more reactive sites to form bonds unlike thermoplastic monomers which have only two reactive sites. These extra sites enable crosslinking and network formation as opposed to individual long chains in thermoplastics. When the reaction starts the thermoset monomers have a low viscosity, which allows them to move about freely. Gradually, large groups of reacted monomers are formed (gel balls) in a solution of monomers and small groups of reacted monomers (oligomers). As the size of the gel balls grows the viscosity increases preventing the remaining monomers and oligomers from moving freely. In a cured thermoset polymer, this leads to regions of high crosslink density (gel balls) and regions that could not reach high crosslink density due to diffusion limited motion (regions between the gel balls) [1,2]. This is clearly shown by Brill with atomic force microscopy (AFM) images in a vinyl ester thermoset [2]. Similarly AFM and ultrasonic force microscopy (UFM) images of undamaged PMR-15, thermoset used in this work, show the same type of gel ball behavior in Figure 1.

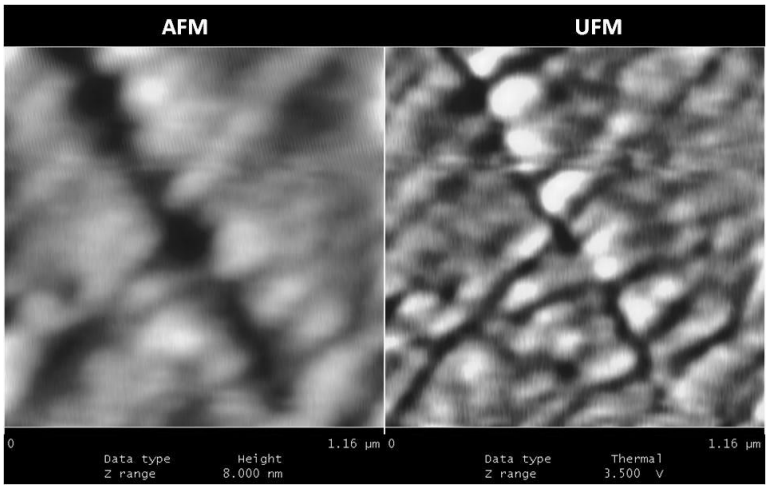

FIGURE 1. AFM (left) and UFM (right) images of PMR-15 fatigued to fracture. Images show random microstructure. "X" shape is a polishing artifact.

It is well known that crosslink density determines the physical properties of a polymer with higher crosslink density resulting in higher elastic modulus (E) and higher glass transition temperature, T_g [1]. It is expected that thermosets with regions of higher and lower crosslink density would behave like a two phase material with one phase being stiffer (gel balls) and the other being more compliant (regions between the gel balls)

Thermoset polymers are viscoelastic materials, whose mechanical behavior can be described by the Kelvin-Voigt spring-dashpot model for viscoelastic materials [3]. The two phase nature of the material and the two component nature of the model allows one to correlate the behavior of each phase to one of the components in the model. The gel balls being highly crosslinked and more stiff will behave more like the spring, while the less stiff between regions will behave more like the dashpot. The spring is assumed to be lossless and behave linearly with the stress, while the dashpot has a lossy behavior.

Under cyclic loading the deformation of gel balls and the between regions will be different. Due to higher crosslink density the gel balls are stiffer and will deform less compared to the between region. Depending on the load conditions the polymer chains might be stretched and disentangled to different extents. Since the between region is softer than the gel balls there is a possibility of rearrangement and alignment along the loading or other preferred direction, similar to slip in single crystalline materials. In tensile-tensile fatigue when the cyclic loading is interrupted, it is expected that at the peak stress the material stretches, disentangling and elongating the polymer chains. Similarly, at minimum stress the material relaxes, but not fully owing to the viscoelastic nature of the material and the fact the material is still under some tensile stress. As the cycles continue the material becomes more and more elongated since it is never allowed to fully recover. This elongation continues until the chains are fully elongated giving them the same apparent stiffness as the gel balls. This represents a transition in mechanical behavior where the material acts stiffer and less lossy than it had initially. This is expected to cause a change the hysteretic heating behavior in the material as the hysteretic heating is dependent on the materials stiffness. This is expected to result in two regions of hysteretic heating behavior, one due to chains elongating and the second driven by full extended chains.

The temperature of a polymer being cyclically loaded changes due to hysteretic heating effects. The rate of change of temperature (dT/dt) for adiabatic heating with heat loss due to thermal conduction and environment is [4]:

$$\frac{dT}{dt} = \frac{2\pi f \tan\delta}{\rho C_p}\left(\frac{\sigma_{max}^2}{2E}\right) - \frac{k}{\rho C_p}(T - T_0) \tag{1}$$

where f is the cyclic loading frequency , tan δ is the loss, ρ is the density, C_p is the specific heat, σ_{max} is the maximum stress, E is the elastic modulus, k is the thermal conductivity, T is the instantaneous temperature and T_0 is the initial temperature.

From this equation the variables that could be expected to change during fatigue are E, tan δ, ρ, C_p, and k. When the temperature changes due to hysteretic heating are small, significant changes in ρ, C_p, and k are not expected since the atomic and electronic configurations of the material remain constant. For the rate of change of hysteretic heating to decrease E must increase, tan δ must decrease, or a combination of the two. A modulus increase implies the material becomes stiffer, and a decrease in tan δ implies the material has become less lossy.

When thermoset polymers are cyclically loaded the disentanglement and rearrangement of the structure is expected to have significant impact on the hysteretic heating. As the number of cycles is increased the material becomes more and more elongated since it is never allowed to fully recover. This stretching continues until the chains are fully elongated, giving them the same apparent stiffness as the gel balls. It is expected that a transition will be observed in rate of hysteretic heating where the material acts stiffer and less lossy than it had initially creating two distinct regions of hysteretic heating behavior each with a distinct slope.

Continuing with the tensile-tensile interrupted fatigue example, stopping the fatigue process periodically would allow the material to fully relax between each increment, assuming sufficient time between increments. This would allow the chains to re-entangle, which would be evidenced by the reappearance of region 1 at the beginning of each new increment of fatigue and a new transition between region 1 and region 2. Region 1 should stay the same from one increment of fatigue to the next. However, region 2 is

anticipated to increase in slope as fatigue continues gradually reaching approximately the same slope as region 1. This is hypothesized to be an effect of fatigue damage on the network structure on the thermoset. As the material is fatigued the crosslinks gradually break down making the material less stiff and more like the less crosslinked areas.

DESCRIPTION OF EXPERIMENTS

PMR-15 resin is a high temperature thermosetting polyimide. It is used for aerospace composites where high temperature strength is required. This resin is rarely used by itself and usually used as the matrix material of a composite with glass or carbon fibers. In this work PMR-15 resin was used alone so that hysteretic heating effects would not be complicated by contributions from both the resin and a fiber if a composite had been tested. To observe the changes in the behavior of hysteric heating under cyclic heating dogbone shaped specimens with 25.4 x 12.5 x 3 mm gage section were used. They were fatigued with a servo-hydraulic test machine with a stress ratio of 0.1 and a frequency of 3 Hz. Fatigue was stopped every 75,000 cycles for 30-60 min to allow the sample to return to ambient temperature. An infrared camera was used to record the sample heating behavior, and it was calibrated to have a high sensitivity about room temperature. A computer controlled data acquisition system recorded the sample temperature. To observe the changes in the microstructure due to fatigue AFM and UFM measurements were performed on samples extracted from a fatigue fractured sample.

RESULTS

Figure 2 shows the changes in rate of hysteretic heating in interrupted tensile-tensile fatigue test. The fatigue testing was interrupted every 75,000 cycles until the sample fractured. Between the interruptions the sample was allowed to cool down to room temperature (30-60 minutes). In each of the interruptions, a clear transition in slope from a steeper initial slope to a shallower one is observed. This is indicative of a change in the heating behavior of the material. This experimental observation supports the two phase deformation and hysteretic heating behaviors described in an earlier section where the material effectively becomes stiffer once the polymer chains have been untangled and extended.

Figure 3 shows AFM and UFM images of the sample very close to the fractured edge of the sample failed under cyclic loading. While the AFM images are surface topographic variations, the UFM images show elastic stiffness variations. The important features that are observed in Figure 3 are ordered spherical gel balls and a surrounding region with lower stiffness in comparison to the randomly distributed spherical gel balls in Figure 1. This observed ordering of the microstructure has occurred due to cyclic loading orienting the microstructure in a preferred direction. The UFM images in Figures 1 and 3 confirm the description of thermoset polymer as a two phase material by illustrating the stiff and less stiff regions. Furthermore, the images of the fractured sample, Figure 3, confirm the deformation behavior described based on the two phase microstructure of thermoset polymers by illustrating the alignment of the gel ball and between regions after loading.

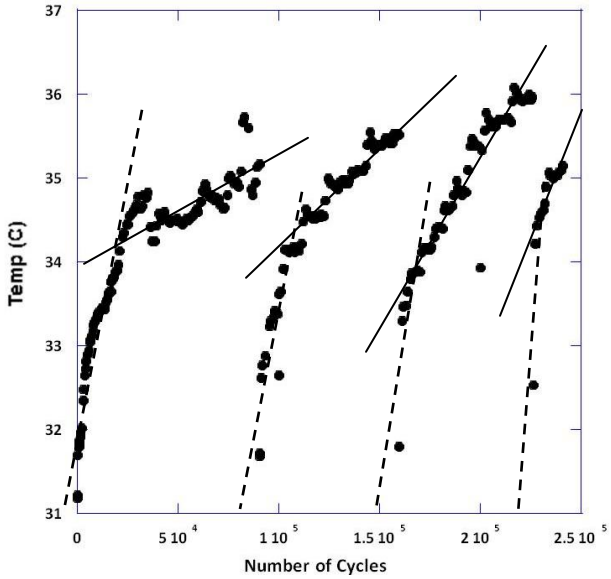

FIGURE 2. Temperature (°C) versus number of loading cycles for the fatigue of PMR-15 showing region 1 and region 2. Dashed lines mark region 1 and solid lines mark region 2.

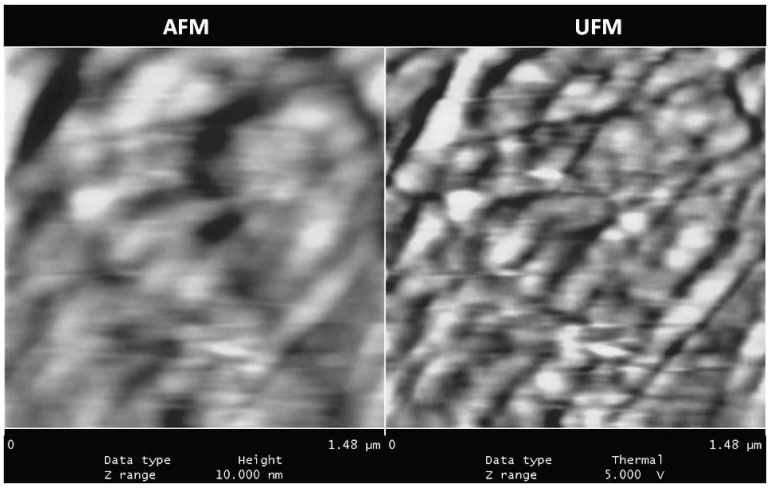

FIGURE 3. AFM (left) and UFM (right) images of PMR-15 fatigued to fracture. UFM clearly shows oriented microstructure.

CONCLUSIONS

The hysteretic heating changes observed during incremental fatigue in a PMR 15 thermoset polymer resin have been explained phenomenologically in terms of a two-phase model. The decrease in slope from region 1 to region 2 corresponds to a straightening of polymer chains in the less crosslinked regions which effectively stiffens the polymer and makes it less lossy. The increasing slope of region 2 with each increment of fatigue corresponds to a breakdown of the polymer network with fatigue. This network breakdown causes the polymer to be less stiff and more lossy making the polymer more efficient at converting mechanical energy from fatigue into heat. This change in heating behavior can be considered an indicator of accumulating fatigue damage and could be a viable means of monitoring fatigue damage accumulation in thermosetting polymers in a laboratory environment.

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