MOISTURE DIFFUSION ANALYSIS FOR COMPOSITE MICRODAMAGE

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ABSTRACT

The absorption of moisture into fibrous polymeric materials has been recognized as one of the major mechanisms in the strength degradation of such materials as reported by several workshops (1-3) and publications (4-7). The objective of the work reported here is to use nondestructive evaluation (NDE) techniques to determine the moisture content (profile) within a composite by measuring the moisture diffusion rate and then subjecting the data to a statistical estimation analysis. This reveals the location of structural degradation and provides a sensitive inspection method for the serviceability of composites.

Hydrothermal aging effects on cured graphite-epoxy composites were determined using: (1) acoustic attenuation, \( \alpha \), (2) ultrasonic velocity, \( c_1 \), and (3) thickness measurements of the composites. Results showed that while ultrasonic acoustic properties, sample thickness and moisture diffusion profiles are highly sensitive to structural degradation, ultrasonic inspection becomes insensitive in areas of extensive internal damage, probably due to high acoustic attenuation which results in loss of signals. Moisture diffusion analysis (MDA), in this case, becomes highly sensitive as a quantitative detection tool. Presently, analytical methods are developed to quantify the depth profile of moisture penetration in graphite-epoxy composites. Measurement of effusion kinetics over a range of time intervals followed by application of statistical estimation theory enables the depth concentration of moisture at initial time \( t=0 \) to be calculated. For a particular model in which the sample is assumed to be exposed to periodically changing environments, the model predicts large fluctuations in moisture concentration near the surfaces while the interior concentration is relatively constant.

Moisture evolution from the composite samples are measured by the DuPont Moisture Evolution Analyzer (Model 902H) as shown below. The simplified block diagram shows the general theory of operation. The sample to be analyzed is weighed and placed in the oven. Moisture in the sample is vaporized, transported by the dry carrier gas to the electrolytic cell which is coated with a thin film of phosphorous pentoxide, and is absorbed by the \( P_2O_5 \). Electrolysis changes the water into oxygen and hydrogen, regenerating the \( P_2O_5 \). The current required (0.132\( \mu \)A per molecule of \( H_2O \)) to regenerate the \( P_2O_5 \) is integrated and displayed as total moisture content in micrograms. A.strip-chart recorder can also be attached to measure the rate of moisture release as a function of time.

The sample cell provided with the instrument is not suitable for large specimens. Therefore, external sample cells have been devised to accommodate bar specimens (sleeve type cell) and plate specimens (face plate cells). To facilitate data collection and analysis, the Analyzer has been interphased with a mini-computer via a analog-to-digital converter. Thus temperature, moisture evolution rate and moisture content can be recorded automatically, then either stored or displayed. In conjunction with the external sample cells, the unit can be portable and suitable for field inspections.

The moisture Evolution Analyzer measures the moisture release rate, \( \dot{m}_m \), as a function of time.
The three accompanying graphs illustrate the accuracy of the estimator. Starting with a fully saturated material of known dimensions and diffusion coefficients, according to Fickian diffusion, would have moisture distribution profiles represented by the solid lines, after 20, 100 and 2000 minutes of desorption.

After each time interval, a set of moisture release rates, \( J_m \), can be obtained from Fick's Law. The inversion of each set of \( J_m \) through the Estimator would give the moisture profile of the material responsible for that set of moisture release rate. These estimated solutions are denoted by the dotted lines in the graphs. It can be seen that the estimated profile agrees very well with the Fickian direct solution, thus verifying the Estimator model.

Fig. 5. Schematic representation of the estimation process.

Fig. 6. Estimated moisture concentration profile for a saturated composite, after 20 minutes of desorption.

Fig. 7. Estimated moisture concentration profile for a saturated composite, after 100 minutes of desorption.
The Estimator model can also be used in simulated actual use conditions. In this case, the material is allowed to absorb moisture for 20 or 100 minutes, and then dried for various amounts of time. This would be a dry-wet-dry cycle. As shown in the following graphs, the Estimator, taking the effusion rate values, generates moisture profiles closely correlated to the direct Fickian solutions.

In a real experiment using the Moisture Evolution Analyzer, the time span the experiment is allowed to run strongly influences the accuracy of the estimating inversion process. The larger the set of times, the better the estimation. This is illustrated by the following figures showing the moisture content as a function of exposure time. Taking the reduced time, \( t^* = Dt/L^2 \), for a typical 0.5 cm thick composite, to completely dry off the saturated material would require about 5000 minutes. A smaller set of times would give a low moisture profile at the sample core by the Estimator.
The first figure shows the arrangement for variable moisture exposure of the composite (Fiberite 934/T300) aged for about a year. Four zones are obtained on the sample bar: exposure to ambient condition, sealed from ambient environment, exposure to water vapor and exposure to water immersion.

![Schematic of variable moisture exposure of composite (Fiberite 934/T300)](image)

Fig. 13. Schematic of variable moisture exposure of composite (Fiberite 934/T300)

The extent of microdegradation of the composite bar due to moisture exposure along the length is characterized by ultrasonics (acoustic attenuation and ultrasonic velocity) through the thickness of the bar. Deformation in the physical dimension (width and thickness) can also be detected. It is seen that in areas of high moisture penetration, thus causing high levels of microdegradation, ultrasonics (attenuation and velocity) are excellent detection techniques. The damaged zones also show signs of swelling because of water penetration.

![Effects of varied moisture exposure on the ultrasonic velocity and attenuation of composite (Fiberite 934/T300)](image)

Fig. 14. Effects of varied moisture exposure on the ultrasonic velocity and attenuation of composite (Fiberite 934/T300)

Sections of the composite bar exposed to ambient and water immersion conditions can be examined by the Moisture Evolution Analyzer and the moisture profile generated by the Estimator. A separate sample, immersed in water for only 6 hours, is also examined. The rates of moisture evolution, as a function of time for these samples are illustrated in the following.

![Effects of varied moisture exposure on the width and thickness of composite (Fiberite 934/T300)](image)

Fig. 15. Effects of varied moisture exposure on the width and thickness of composite (Fiberite 934/T300)
The moisture profiles of these samples are given in the following figures. The amount of moisture, in micrograms, agrees very well with weighing determinations. One limiting factor is that the time duration of the effusion experiment is not long enough to provide a good estimation for the core of the samples.

For the saturated sample, there are two dips in the moisture profile, at \( x/L = 0.1 \) and 0.9. The \( J \) vs. time curve for this sample also shows a hump. These anomalies are due to defects in the composite, as shown below.

An enlarged view of the cross-section of the Fiberite 934/T300 used in this experiment shows layers of voids in the interlaminar orientation due to faulty layer-up manufacture. This is shown by the following photographs.
Fig. 20. Longitudinal view of composite bar showing air voids.

Fig. 21. Cross-sectional view of composite bar showing two layers of delamination.

Ultrasonic mapping techniques clearly show the layer of voids in the plane of the sample. Moisture evolution rates for the transfibrous orientation show a lower moisture concentration because the moisture is being effused at a faster initial rate along the planes of the voids, depleting the interior moisture content of the sample more rapidly than in the transverse orientation, enabling the Estimator to lower the total amount of moisture in the sample. Also, the time duration of the effusion experiment is much longer (about 1000 minutes), enabling a better estimation at the sample core.

Fig. 22. Ultrasonic mapping profile of composite bar showing regions of voids.

Fig. 23. Estimated moisture profile of composite bar Fiberite 934/T300 in transfibrous orientation.

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REFERENCES


