

SIMULTANEOUS MASS AND DIMENSIONAL CHANGE MEASUREMENTS IN CONTROLLED ENVIRONMENTS

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Abstract

Simultaneous strain-weight measurements have many applications in the field of materials characterization, including determination of the coefficient of moisture expansion (CME), cross coupling coefficients in hygrothermoelasticity, the effects of boundary layers and in fiber characterization. Three approaches to an experimental realization of such measurements are presented and typical results with a system based on quartz spring balances, an LVDT transducer and electrical contacts are described.

1.0 Introduction

Determination of the thermophysical and micromechanical properties of materials frequently requires simultaneous measurements of several material responses such as mass, temperature and strain changes. For polymer based composites, temperature, pressure, humidity and boundary conditions and their interactions are important variables. The coefficient of expansion due to solvents such as moisture, fuels, cleaners, adhesives, paints or coatings is a frequently required design and quality control parameter. Related information includes maximum solubility, diffusion constants, permeability of coatings, and the influence of defects, temperature and/or moisture cycling, ageing, stress relaxation and postcuring.

1.1 Coefficients of Moisture expansion

Mass diffusion rates in polymers near ambient temperatures assure that determination of an equilibrium CME may take months [1]. Methods to reduce this time are outlined in [2]. The best approach is simultaneous measurement of weight and strain at temperatures just below the glass transition temperature. Changes in the ratio of strain to wt % moisture change are of special interest to aerospace applications.

1.2 Hygrothermoelasticity

Since the use and measurement of CTE and CME data involves heat and mass transfer, a stress state and their gradients and changes with time, there is the likelihood of cross coupling in their strain response and the possibility of material property changes as a result. For example, the Soret effect is a mass flux caused by a temperature gradient; the Dufour effect is heat flow caused by a concentration gradient. Determination of the various cross coupling coefficients in hygrothermoelasticity have been difficult due to lack of suitable experimental facilities with well defined boundary conditions.

1.3 Boundary Conditions

Boundary conditions must be considered in the use of weight changes during absorption or desorption to obtain diffusion coefficients. If a boundary layer of humid air, for example, is the rate limiting step, incorrect coefficients are determined. When does the rate in flowing dry air, for example, equal the vacuum dryout rate? Continuous and simultaneous weight/strain measurements also elucidate the effect of internal porosity, interfaces and surfaces on the CME.

1.4 Fiber Characterization

Several carbon fibers as well as aramid and most natural fibers absorb moisture, and some exhibit substantial negative CME values [3]. This behavior suggests studies of the effects of stress on fiber CME and also surface reactions. Tensile stresses on fibers are easily imparted using the systems to be described. (Stress application on composites may be done by using a wire to impart a flexural stress [4]).

2.0 EXPERIMENTAL DEVELOPMENT

Strain, weight, temperature and humidity must be continuously measurable in a controllable environment which must include vacuum, still or flowing gases and relative humidity. A strain range of 0-3 mm is desirable. For example, transverse CME values on unidirectional polymer matrix composites are in the 3000-8000 ppm/% Δ M range. A 300 mm long sample absorbing 2% by weight of moisture will expand about 3 mm. In general, polymer matrix composites require an accuracy and resolution on the order of 10^{-4} g and 10^{-7} m.

2.1 Weight Changes

Microgram weight changes can be detected by microbalances using quartz springs. An ability to operate during rapid changes in environment, such a change from flowing dry air to vacuum, is required. Assuming a constant buoyancy force, the spring constant k (in N/m) may be estimated [5];

$$k = P/\delta = (G D^4) / (64 N R^3)$$

P = load in newtons

δ = deflection in m

G = elastic modulus in N/m^2

D = diameter of material in m

N = number of turns

R = radius of spiral in m

An actual spiral will show a smaller k because of the horizontal arms at the spiral ends to support the spring and (below) the sample. The spring must also be able to remain elastic without fracture to support weights of 30-50 g, a typical weight for a composite laminate plate sample. A variety of useful quartz springs were fabricated with $D = 1$ mm, giving k on the order of 0.3 g/mm.

2.2 Optical Approach

Since the sample is supported by a quartz spring, any contact for strain measurement will interfere with the weight measurement. Capacitance or optical techniques are implied but there must be a range capability of several mm and insensitivity to a variable temperatures and pressures. For example, Interferometry is limited by indeterminate variations in indices of refraction.

A reflective optical intensity integrating technique was constructed (Fig.1). A 3mm

HeNe laser beam (L) is split to provide three beams which scan 3 mm wide horizontal lines of reflective silver paint inscribed at the top of the quartz spring(Q), the spring/sample support point and near the bottom of the sample(S). As these points move, the change in luminance from the poorly reflecting sample (e.g., graphite/epoxy) to silver paint line is integrated by the optical system (lenses (L) and filters (F)) and recorded automatically by the photodetectors (PD). Relative motion of the top of the spring and top of the sample gives the sample weight change via the spring constant. Relative displacements of points on the sample give expansion coefficients. The system can be independently calibrated with an LVDT of 70 nm resolution. The nominal displacement range of 3 mm can be increased with increased laser spot size. Strain resolution is on the order of 10 microstrain. Any geometry sample that will fit in the glass chamber can be tested; currently panels 5 x 30 cm x thickness are used. Weight changes of less than 0.01 wt% can be detected. Temperature can be measured with an infrared detector or with thermocouples on an identical sample in the same system. The surrounding atmosphere can be any temperature (typically +/- 200deg C), pressure (micro to kilo- TORR) of any gas (G) or fluid, and humidity controllable from 0- 99% RH via the hot plate (HP), water bath and/or dessicant (D). This system works well provided the glass environmental container maintains thermal equilibrium, since it is included in the optical system. Consequently, this approach is not suitable when rapid temperature changes are of interest.

2.3 Transducer approach

The three reflected beam spots in the previous system can be replaced by in-line linear variable differential transducers (LVDTs). The weights of the movable LVDT cores must be added to the sample for support by the spring balance. The LVDT characteristics (voltage versus displacement) and their temperature dependence must be individually pre-calibrated. Some lag in response is incurred with sudden temperature changes due to slow LVDT temperature equilibration. In addition, unless alignment is nearly perfect, friction of the cores in the LVDTs causes sticking and requires occasional vibration prior to recording of voltage.

2.4 Electrical Contact Approach

Figure 2 shows the basic principles of a system which works well with only one LVDT and has neither rate change limitations nor sticking problems. Alignment and additional weight requirements are negligible. The precalibrated quartz spring supports a plate sample above an LVDT core which in turn is raised/lowered by a motorized actuator. A small (0.07 mm or smaller) horizontal copper wire is attached via colloidal silver paint to each end of the sample for electrical contact. (This could be part of a thermocouple to measure the temperature of the ends of the sample). As the LVDT core (typical output 0.800 V/mm) is raised, a reading is obtained at first contact equal to:

$$\delta_1 = -\delta (\Delta L \text{ due to CTE}) + \delta (\Delta W) + \delta (\Delta L \text{ due to CME})$$

Further raising causes the top of the sample to make contact with the conducting stop. This reading represents;

$$\delta_2 = -\delta (\Delta L \text{ due to CTE}) + \delta (\Delta L \text{ due to CME})$$

The difference $\delta_1 - \delta_2$ equals ΔW via the spring constant and at constant temperature, δ_2 represents the ΔL or ϵ_x due to the CME. A special circuit accounts for spring oscillations and a thermoelectric cooler keeps the LVDT at a controlled temperature. The system is supported by a quartz tripod in the environmental chamber of Fig.1.

3.0 RESULTS WITH SIMULTANEOUS WEIGHT/STRAIN

The system of Fig. 2 was calibrated with known CTE materials. The short term desorption of a unidirectional graphite fiber reinforced cyanate ester resin plate (254x50x0.75mm) sample was measured in the transverse (fiber) direction. After stabilization to the nearest 0.1 mg at 54%RH and 25° C, the sample was placed in the system of Fig. 2 at 63° +/- 1° C and held for about 4 days. Fig.3 and 4 indicate small initial scatter in the readings of $\Delta L/L_0$ and ΔM . The ratio of the linear regression lines gives the CME or β_y . The value of 3046 ppm/%M is

similar to the equilibrium value of 2560 ppm/%M obtained at 24°C on the same sample. (The difference may be due to temperature, aging or storage time (several years) and/or non-Fickian diffusion).

4.0 CONCLUSIONS

Simultaneous weight/strain measurements are required to provide valuable information on the hygrothermoelastic response of many materials. Development of a system required careful choice of sample supports, sensors and data recording techniques. Representative results for short time CME were obtained. Simultaneous measurement of weight and strain changes will also help to clarify the effects of moisture cycling and long term absorption on the CME. A potential means to study cross coupling coefficients, boundary effects and the behavior of fibers was developed.

5.0 ACKNOWLEDGMENTS

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6.0 REFERENCES

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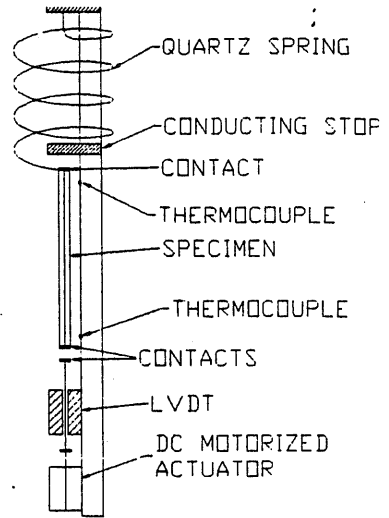
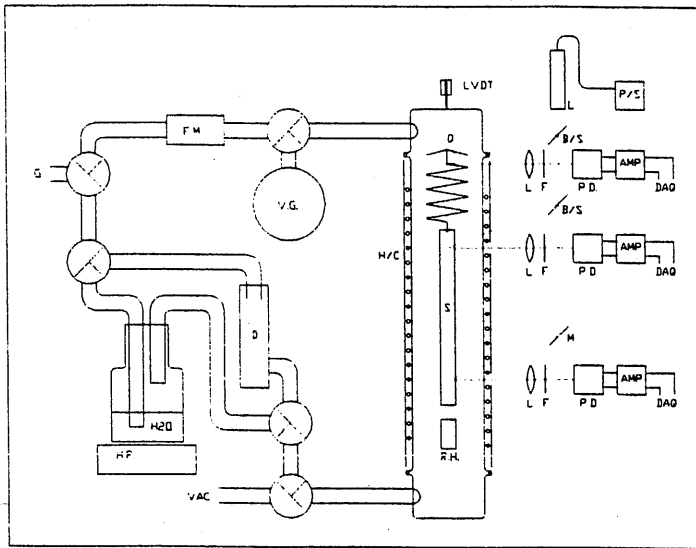


Fig.1 and 2. Schematics of simultaneous weight/strain measurement systems with controlled environments using a) luminance integration technique and b) electrical contact system.

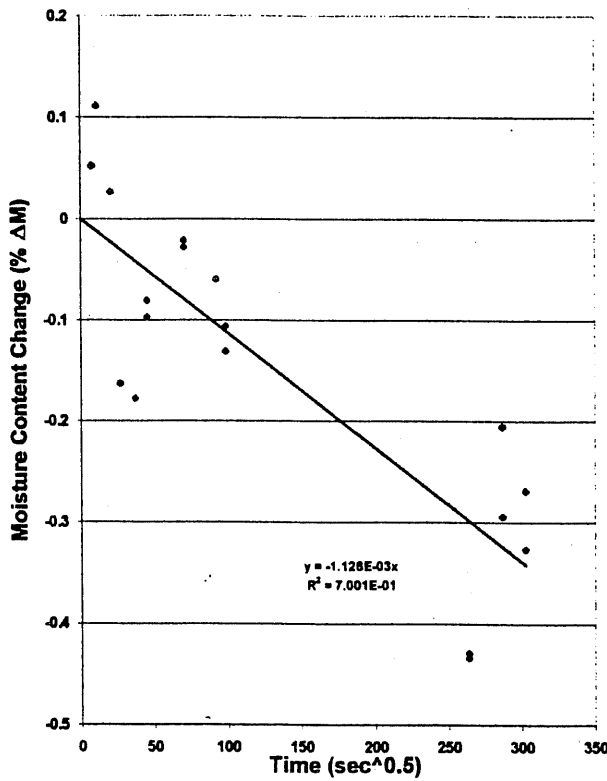


Fig.3 Moisture content change using system of Fig.2

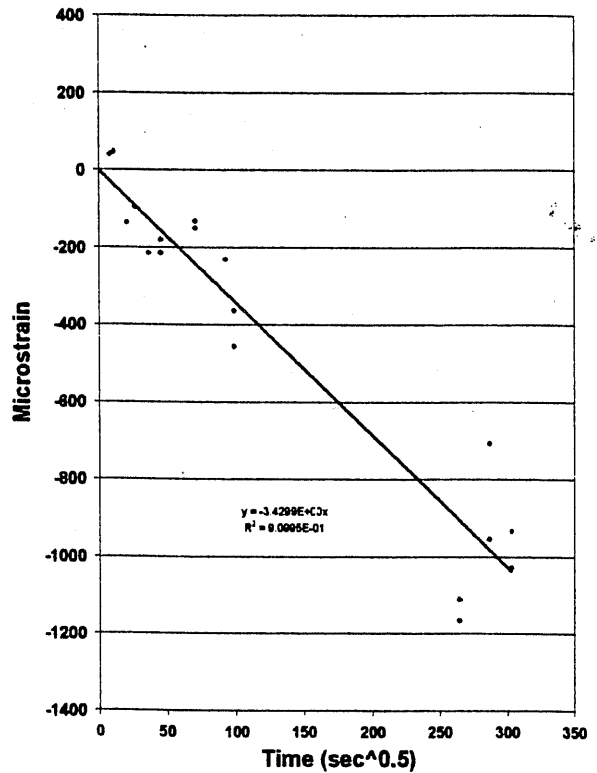


Fig. 4 Simultaneous strain with measurements in Fig.3