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Determination of the Coefficient of Moisture Expansion (CME)

ABSTRACT

A test facility for the measurement of the length variation of polymer composites due to moisture evaporation is described. The measurement method is based on commercial laser interferometers with a resolution of 10nm and working under vacuum conditions yields a total accuracy of 0,1µm. The high sensitivity and resolution of the test facility is shown by CTE experiments with copper and Invar samples. The CTE results are in the expected range although the considered temperature regime is very small. CME measurements were performed on simple lay ups – unidirectional and bidirectional material - of carbon fibre reinforced polymers, which were preconditioned in water at a temperature of 50°C. The determination of Δm is done with an online-measurement of the sample weight during outgassing under vacuum condition. The CME values for the chosen material are in the range of 5E-6/% to 1E-2/%.

1 INTRODUCTION

A high degree of dimensional stability is often required for aircraft and spacecraft components. In order to evaluate polymeric composite materials for aerospace applications the coefficients of thermal expansion (CTE) and moisture expansion (CME) have to be determined.

The swelling/shrinkage of CFRPs due to moisture uptake/release is very small - CME values of CFRPs are in the range of <5E-5/wt% H_2O in the fibre direction and about 1E-3/wt% H_2O normal to the fibres.- And therefore length variation data can be obtained with high resolution /high accuracy measurement systems only. Conventional dilatometers cannot provide the required resolution and accuracy (typically ±1µm) and the commercial facilities cannot be operated in vacuum. Special inductive systems and online external interferometric measurement systems (moisture sorption or desorption and the according length variation in the same facility) are very sophisticated and the measurement times are far off economic needs. Therefore a methodology for the determination of CME from data which are available at acceptable cost and time consumption is of great industrial interest.

The presented methodology is based on laser micro interferometers operated inside the chamber leading
a) to a surpassing accuracy and hence to reduced measuring times, since all errors introduced by external interferometric systems (ambient temperature and pressure variations, external beam path deviations,..) can be eliminated – and
b) to a simultaneous multidirectional measurement due to the use of multiple miniaturised interferometers.

CME is defined by the ratio of the length variation to the mass variation [%] due to moisture evaporation or absorption:

$$\mathrm{CME} = \frac{\Delta l}{l_0} \Big/ \left(\frac{\Delta m}{m_0}[\%] \right) \qquad (1)$$

l_0, m_0 ... initial length and mass respectively
Δl, Δm ... time dependent length/mass variation

Additionally to Δl values Δm data are needed. These data can be gained by directly weighing the sample before and after testing, but online weighing during outgassing in vacuum yields more reliable results and reduces the measurement time considerably.

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2. Measurement method and facility

2.1 Interferometric methods

The principle of the Michelson interferometer can be used for the measurement of changes of distances. The reflected beams from the two mirrors show an interference pattern (constructive and destructive interference fringes) depending on the phase shift of the beams. If one of the mirrors is moving the distance can be determined by counting the passing interference fringes. One passing fringe is caused by a mirror movement of half a wavelength. The wavelength of commonly used Lasers is in the visible light range of about 780nm. The resolution of interferometry can yield 10nm by using special detection and signal amplification methods. The difficulties and restriction of the achieved accuracies of the standard Michelson interferometers are:

- the alignment of the mirrors perpendicular to each other, while contacting one to the sample
- the refraction index of air (depends on the temperature/pressure and humidity) and of the mirrors (temperature dependence) cause measurement errors
- the operation under vacuum condition is difficult, because common lasers and detectors work only under ambient conditions, therefore the beam (in itial and interfering) have to pass windows and distances in air, which rises additional errors.

The above mentioned difficulties can be prevented by commercial instruments (micro-laserinterferometers). They work very similar to the Michelson interferometer but the complete set up is integrated to one small instrument. The moving mirror, which can be mounted to the sample, is a sphere with the refraction index of about n=2 that reflects the laser beam parallel to the initial beam.
Tests of these instruments have been performed in a vacuum facility, which was adapted for the test. It was possible to stabilise the temperature of the instruments by fluid thermostats. The preferred operation temperature is about 22°C. Further a vacuum pressure of below 5*10E-6 could be reached in this facility.

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Fig. 1: Vacuum chamber

For the test the Micro-Laserinterferometers where mounted unto micro -positioners (moveable ±5mm by micrometer screw) for their adjustment to the mirrors in the two axes perpendicular to the beam. According to the proposed measurement principle one mirror was at the front and one at the rear the side of the „specimen". The test set up consisted of one pair of lasers only , one for small distances (up to 100mm) and one for larger distances (up to 300mm). The complete set up was mounted a on a water cooled support plate and integrated in a vacuum chamber. The experiences with the instruments indicate that the important properties of the measurement facility are:

- low CTE materials for the measurement system
- additional temperature stabilising for the instrument and support plate
- vibration damping
- adjustability of the micro-interferometers
- PC control and data acquisition for long term measurement

The measurement system is integrated in a vacuum chamber and is operated at a pressure of below 1E-3Pa. Special vibration isolation is required because the interferometers are very sensitive towards vibrations. The facility which was designed and constructed inhouse is shown in Fig.1 and Fig. 2.

Temperature variation may cause mechanical strain in the sensor, the cable and the plug. yields a drift (up to 0,1µm/h) of the laser. Furthermore the thermal radiation produced by the sample heating has to be carried off. The radiation temperature will be up to about 340K and affects the accuracy of the interferometer which is calibrated for ambient temperatures. Small temperature variations of the interferometers in the regime of about 20°C-25°C are corrected by an internal Peltier element. Therefore the interferometers must be temperature stabilised and fluid channels are integrated in the supporting plate.
A temperature change in the supporting plate is lower than ±0,1K (temperature stability of the fluid is about ±0,01K!).

2.2 Measurement principle

The length variation is determined by two laser interferometers, one focused to the front and one to the rear end of the sample. Special reflectors (spherical lenses gold plated at the rear side) are fixed to both ends of the sample and the laser sensors are adjusted to these mirrors. The sensors can be moved in the z- and x-axis (laser beam in y-axis) by special micrometer screws. Additionally the sensors are temperature stabilised by a fluid thermostat which is operated at about 22°C. Samples with the dimension of approximately 200mm x 30mm can be mounted. The temperature distribution within the vacuum chamber is measured by Pt100 thermoresistors in order to estimate the effective measurement errors. The interferometer signal is detected and the length is computed by PC. The wavelengths of the sensors are calibrated and for vacuum operation a correction of the air refraction index is not required.

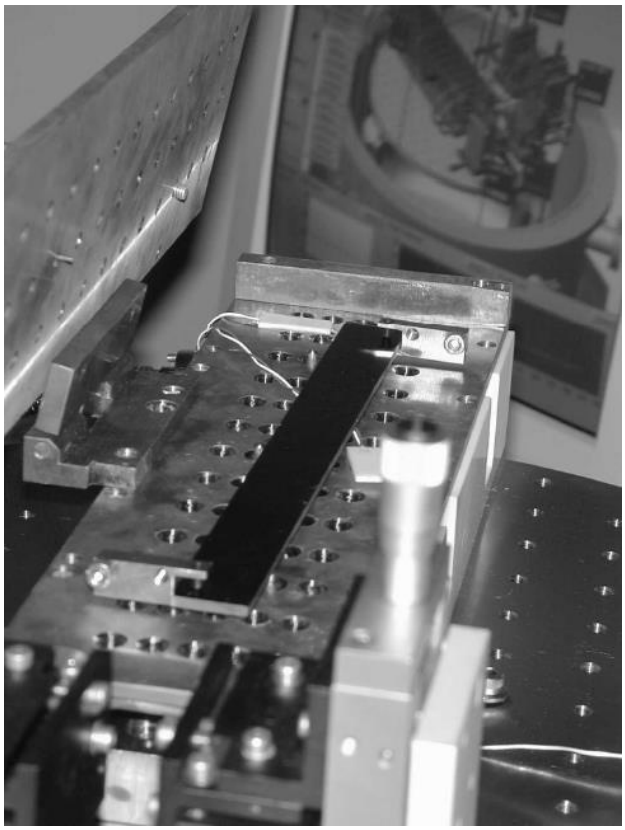

Fig. 2: Sample with mirrors

The Δm is determined by a high resolution balance operated in a vacuum chamber and recorded online. The sample size is approx. 20x50mm, which is comparable in the volume to surface ratio. This ratio affects the diffusion of moisture and therefore the Δm and Δl versus time. The Δm –measurements are also carried out in an in-house facility.