

## Measurement of the $T_g$ , as providing information on the degree of cure, through a range of methods

### Objectives

The aim of the project is to propose internationally accepted test methods, traceability and methodologies for the evaluation of the cure state of fibre reinforced composites. It is envisaged that this work will support new or revised international standards.

### Background

The measurement of  $T_g$  - the glass transition temperature, which is a materials property but reported as a temperature value, can be determined through a wide range of measurement techniques, including:-

- Differential Scanning Calorimetry
- Thermo Mechanical Analysis
- Dynamic Mechanical Analysis

The value of  $T_g$  is often used in materials qualification and specification requirements, as  $T_g$  is related to service temperature capability. A Standard Qualification Plan was developed at NPL for aerospace pre-impregnated composite materials where DMA is the stated method.

The techniques mentioned are all used as secondary measures of degree of cure. However, as different methods are used, the lack of precision in some cases causes confusion and dispute when data

from alternative methods do not agree (e.g. polymer chemists involved in synthesising new materials favour DSC, while engineers as end users favour DMA).

In addition, some of these techniques are meant for off-line, rather than on-line measurements and a change from initial development of an epoxy resin to a final product manufacture of a carbon fibre/epoxy aerospace component forces a change in the monitoring methods for checking cure. This is a vital measurement to ensure that future performance of the component or material is as required.

### Standardization Needs

Standards exist for the thermal analysis techniques (DSC,TMA,DMA). No standards are available for  $T_g$  by other techniques (e.g. ultrasonics, dielectrics).

### Work Programme

- Fully/partially cured material will be circulated to all participants. Glass-transition temperature to be determined by a range of techniques, as above.
- A temperature reference specimen will be supplied for evaluating the DMA equipment (Indium embedded

## International Interlaboratory Trial

in a carbon-fibre epoxy resin laminate).

- Results will be analysed using statistical methods (e.g. ISO 5725)

### Deliverables and Dissemination



Recommendations / guidance sheet as to the method and procedures to obtain valid measurements of the cure state, leading to their international standardization. The initial dissemination will be via VAMAS, international conferences, scientific publications and standards committees.

### Funding

Self funded by the interested participants. Materials for the interlaboratory comparison will be supplied by NPL.

### Status

A preliminary round-robin by DMA completed. Results helped towards drafting the new standard for  $T_g$  determination by DMA (ISO 6721: Part 11– 2012).

Phase 1 of the Interlaboratory validation trial by DMA participated by 25+ laboratories worldwide. Analysis of results in progress.

Material sourced for Phase 2 exercise.

New batch of DMA reference specimens using two different carrier materials to be manufactured.

**Volunteers for participation welcome.**

**For more information on participation, please contact:**

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