SIB 52 - THERMO Stakeholder meeting

May 16

Metrology for thermal protection materials

Challenges in thermal conductivity measurements of thin (multi-layered) thermal insulation materials

MESURES & RÉFÉRENCES
Clés de la COMPÉTITIVITÉ et d'un MONDE PLUS SÛR

Laboratoire national de métrologie et d'essais

Outline

- Introduction
- Difficulties specific to thin thermal insulation materials
- Guarded hot plate technique
- Measurement with laser flash thermal diffusivity measurement technique
- Conclusion

- Introduction

Many insulation or thermal protection materials are now produced in multi-layer and thin form.

Demands from industry for thermal conductivity data.

Challenging measurements due to low thickness and structure.

- Difficulties for thin multi-layered thermal insulation materials

Low thermal resistance → low thickness of the material → Challenging for GHPs.

Non isotropy (thermal conductivity depends on direction).

Non homogeneity (different layers).

Definition of the thickness can de difficult: porosities, fibers
→ (high relative uncertainty)

Transparency due to materials and low thickness.

The material can be rigid (structural composite) → thermal contact difficulties.

→ very difficult situation to get "accurate" and reproducible results.

Presentations of techniques

Guarded hot plate

- Measurement of the thermal resistance of a sample with a uniform thickness in steady state conditions → calculation of the equivalent thermal conductivity.

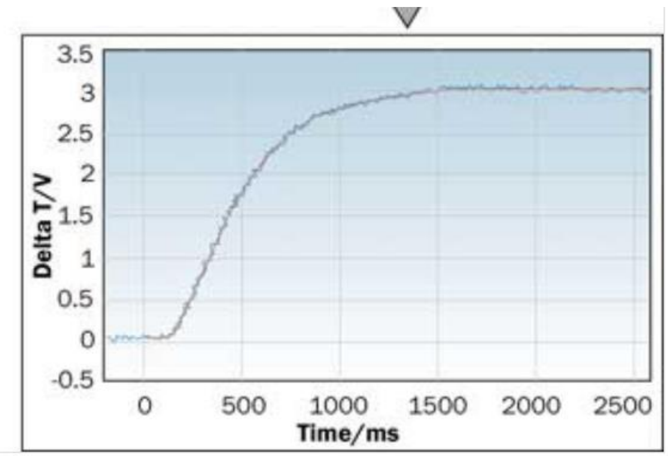
  The heat power crossing the “metering section” is measured directly (electrical power).
  Measurement of the temperature gradient: usually by measurement of the temperature of the plates and measurement of the thickness of the sample.

$$\lambda = \frac{\Phi \,.\, d}{A \,.\, \left(T_H - T_c\right)}$$

- Presentations of techniques
  - Thermal diffusivity laser flash technique (LFA)

$$a = \lambda / C_P \cdot \rho$$

- A laser impulsion very short in time irradiates the front face of the sample → deposition of a pulse of heat on the front face.
- An infrared detector measures in function of time the relative variation of the temperature of the rear face. It is not required to measure the true temperature, only the relative shape of the curve is required.
- Assuming a material homogeneous in the direction of measurement, the heat transfer equation is solved and the thermal diffusistvity can be identified by fitting the relative temperature variation curve given by the theoretical model to the experimental curve.
- The front face must be opaque at the wavelength of the laser → laser radiation is completely absorbed at the surface.
- The material must be opaque in the wavelength spectral range corresponding to the temperature of the sample so that the heat transfer theoretical model can be applied (no radiation transfers inside the material).



Advantages and limitations of the techniques

Guarded hot plate (measurement on one thickness of the material)

- Avantages:
  - Steady state → no heat capacitive effects, the measurement is not affected by the non homogeneity of thermal capacity in the material.
  - The "true" thermal resistance is measured (steady state) whatever the structure of the material (number of layers, arrangement of layers) → the "equivalent" thermal conductivity is calculated.
  - The GHP technique can be very accurate when applied in good conditions.

- Disavantages:
  - The width of the gap of the instrument must be adapted to thicknesses to be tested (ratio>10).
  - Thermal contact resistances between the two sample surfaces and the plates can be high relatively to the thermal resistance of the sample.
  - Specific techniques for measurement of temperature gradient:
    - very thin temperature sensors
    - techniques for reduction of thermal contact resistances ("conductive interfaces")
  - For rigid materials, samples must be very plane with parallel surfaces → low TCR.
  - Sometime thin insulation materials have low emissivity surfaces to increase their apparent thermal resistance → higher TCR.

- Advantages and limitations of the techniques

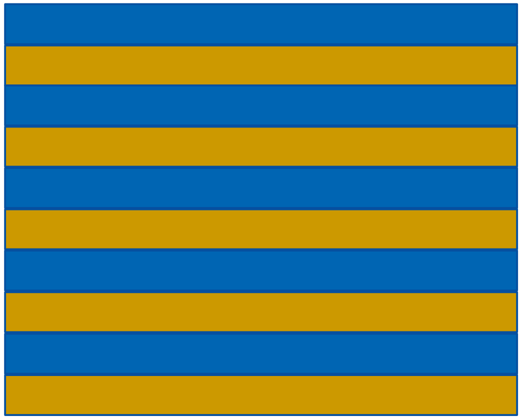
Thermal diffusivity laser flash technique

- Avantages:
  - The technique can be applied quite easily at high temperatures (the sample is heated in a furnace, no accurate temperature measurement is required).
  - The technique can be applied quite easily with samples under vacuum or inert atmosphere.
  - Samples are usually quite “small” → ∅ 7 mm to 25 mm.
  - No contact with the samples during measurement (laser, remote temperature measurement)
  - Very fast measurement (a few seconds once the sample is at the required temperature).
  - No sensitivity to thermal contact resistances.

- Advantages and limitations of the techniques
  - Thermal diffusivity laser flash technique
    - Disavantages:
      - The thickness of the material (sample) must be very well defined → difficulty for porous materials of materials with fibers (non homogeneous).
      - The thermal expansion and the density of the sample must be known at the temperature of measurement (density measured at RT + dilatometry)
      - The specific heat must be known at the temperature of measurement (measurement DSC)
      - Thickness is of the sample is limited (a few mm) → to get measurable temperature variation on the rear side.
      - The front face must be opaque at the wavelength of the laser → laser radiation completely absorbed at the surface.
      - The material must be opaque in the spectral range corresponding to the temperature of the sample (heat transfer equation can be used).
      - The material must be assimilated to an homogenous material in the direction of measurement for solving the heat transfer equation in unsteady state conditions.
      - The layers must be periodically arranged with a sufficient number of periods (number of periods depends on the differences of thermal diffusivity between layers and time constant of the layers).
      - Measurement must be performed along principal axis of the material

- Advantages and limitations of the techniques
  - Thermal diffusivity laser flash technique

    The layers must be periodically arranged with a sufficient number of layers (more than 5 or 6 periods can be required).



Other solution

When the measurement can not be performed on one thickness of the material with an appropriate GHP or thermal diffusivity can not be measured.

Recommendation :

- Measurement with a guarded hot plate of the thermal resistance of a stack of many thicknesses of the multi-layered material.
- Calculation of the thermal conductivity by dividing the thermal resistance result by the number of thicknesses in the stack.

Advantage :

- A “common” GHP can be used.
- The mean number of interface per thickness of the material is only (n+1)/n with n the number of thicknesses.

Disadvantage :

- Thermal contact resistances at the interfaces can be difficult to control.
- Uncertainty can be difficult to quantify.

Conclusions

For well controlled measurement :

- Use a GHP specific to thin materials.
- Use laser flash diffusivimeter when the material is appropriate (multilayer rigid composites).

Other cases : Measurement with a guarded hot plate of the thermal resistance of a stack of many thicknesses of the multi-layered material.