

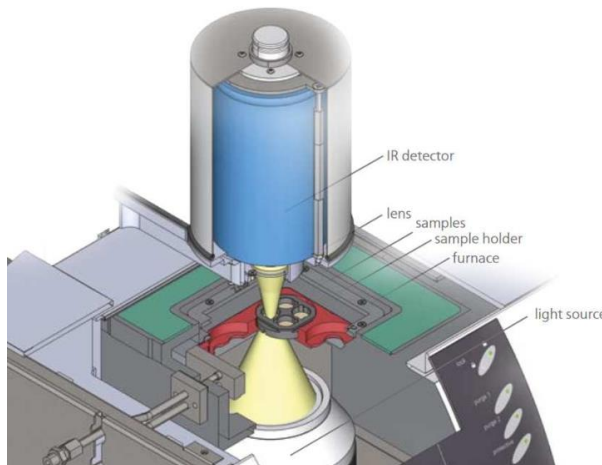
Thermal characterization of polymer and polymer composites – evaluation and comparison of measurement methods

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Polymer Competence Center Leoben GmbH

4a Technologietag 2020 - Werfenweng

4a
TECHNOLOGIETAG

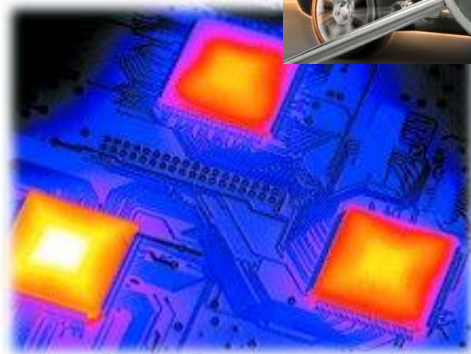


Thermal Conduction

Improving thermal management for microelectronics and power packages, by enhancing thermal conductivity of polymeric insulators



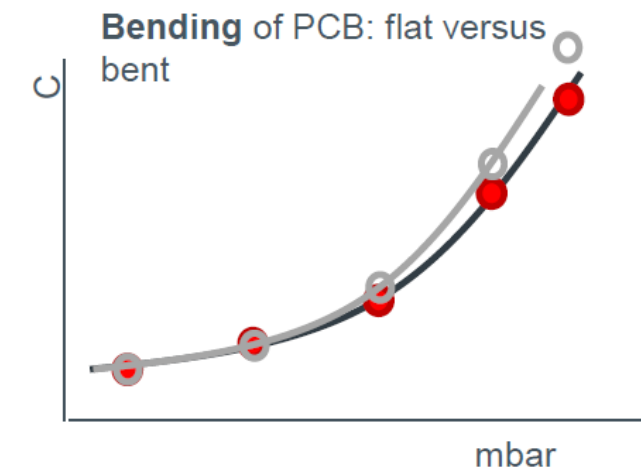
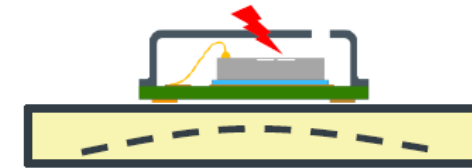
© Continental Press Release: Key Component in the Electric Drive, 2015Automotive GmbH:



Strock, J.: Does your PCB have a fever? EE-Evaluation Engineering (2000)

Thermal Expansion Behavior

Understand and adapt thermal expansion behavior to current needs for application tailored polymers.



Thermal Conduction

Three material parameters are the crucial thermo-physical properties to define heat transfers:

$$\lambda(T) = a(T)c_p(T)\rho(T)$$

Thermal Conductivity $\lambda(T)$

It links heat transfer to a temperature gradient by obeying the second law of thermodynamics.

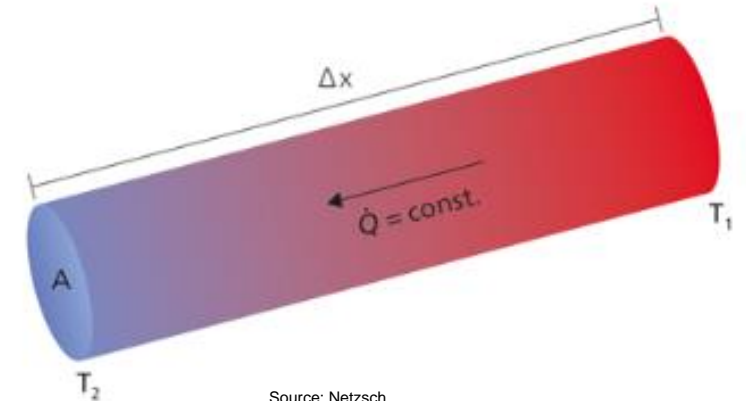
Thermal Diffusivity $a(T)$

It links an energy flux to an energy gradient, hence it is a measure for the speed of heat transfer through a body of mass.

Specific heat capacity c_p

It describes the energy consumption during heating processes.

$$\frac{dQ}{dt} = \dot{Q} = -\lambda A \frac{T_2 - T_1}{\Delta x} ; \quad \dot{q} = -\lambda \frac{\Delta T}{\Delta x}$$

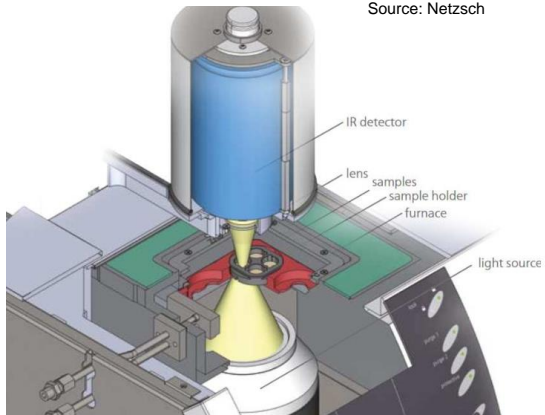


Source: Netzsch

Netzsch LFA 467



Source: Netzsch



TA Instruments DTC 300



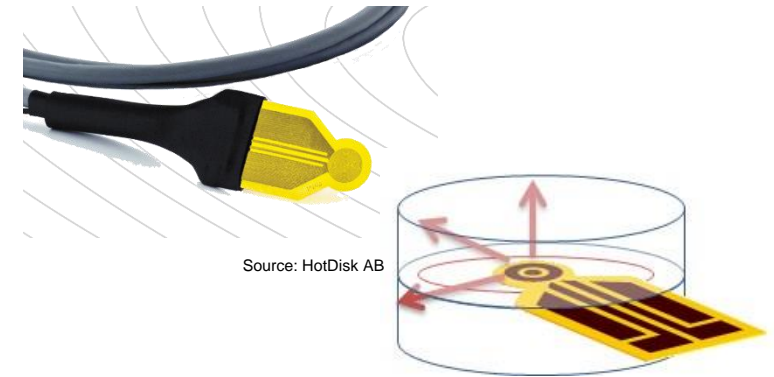
Source: TA Instruments



HotDisk TPS 2500S



Source: HotDisk AB



Source: HotDisk AB

	Netzsch LFA 467	DTC 300	HotDisk TPS 2500S
Temperature range	-100°C - 500°C	20°C - 300°C	20°C - 400°C
Specimen size	6 mm - 25.4 mm diameter 0.01 mm - 6 mm thickness	50 mm diameter > 1 mm to 25 mm thickness	> 10 mm diameter > 4mm thickness
Time per measurement	approx. 1s	10-30 min	1 to 2560 sec
Therm. Cond. Range	> 0.1 - 4000 W/(mK)	0.1 - 40 W/(mK)	0.005 - 1800 W/m/K.



Source: Netzsch

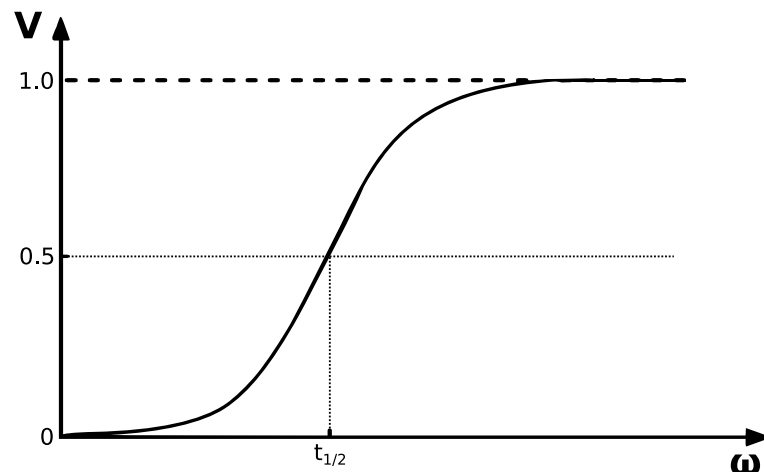


Source: TA Instruments



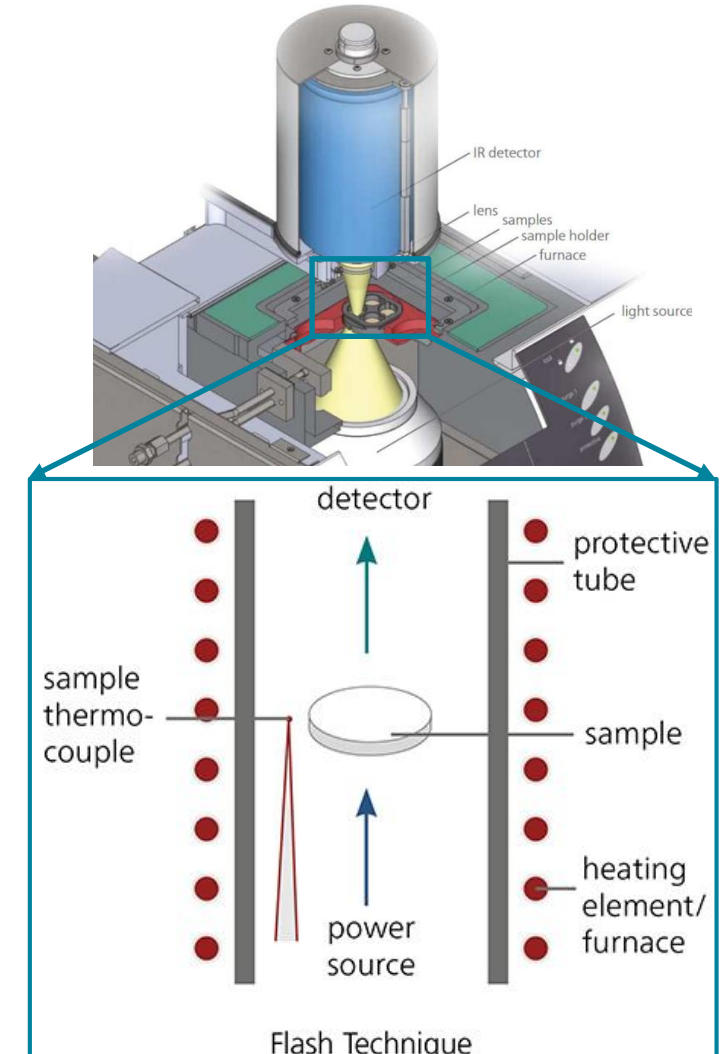
Source: HotDisk AB

- LFA is transient contactless method for the measurement of the thermal diffusivity $a(T)$.
- A short uniform heat pulse heats up the front surface of a plane-parallel specimen.
- At the rear side of the specimen an infrared detector is placed, which measures the temperature rise over time.
- Wide temperature and thermal diffusivity range as well as capability to measure thin ($>10\text{ }\mu\text{m}$) samples.

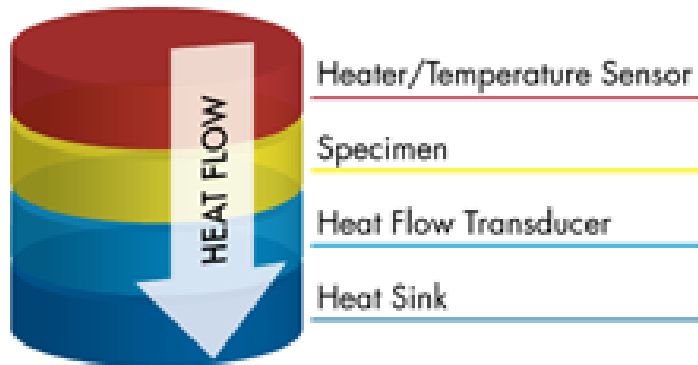


$$a = 0.1388 \frac{d^2}{t_{1/2}}$$

$$\lambda(T) = a(T)c_p(T)\rho(T)$$



- TA Instruments Guarded Heat Flow Meter DTC300
- Stationary and direct method
- Limited temperature range and precise sample dimensions necessary
- Using a steady heat flow for the determination of the thermal conductivity.
- Evaluation using Fourier's law: $\lambda = \frac{\dot{Q}d}{A\Delta T}$



Guarded Heat Flow Test Method

Source: TA Instruments

Specimen

Ø = 50mm

d ≥ 1mm



Testing Chamber

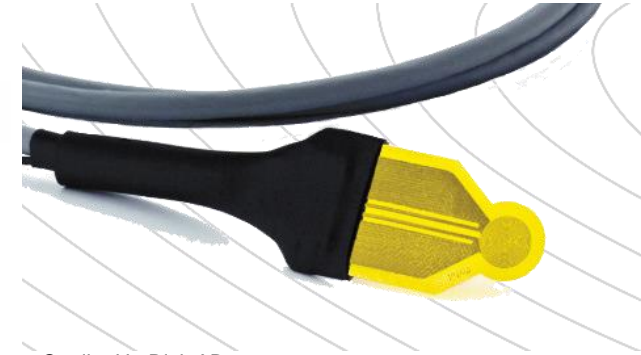


Source: TA Instruments

- Hot Disk TPS 2500S Thermal Constants Analyzer
- Transient method which determines the thermal conductivity directly.
- Relying on the Transient Plane Source (TPS) method.
- Limited temperature range, however best method for low conducting insulation materials.
- Two different measurement modes:
 - Isotropic measurement (λ)
 - Anisotropic measurement (λ_{surf} , λ_{thick})
 - $\rho(T)$ and $c_p(T)$ need to be known



Quelle: HotDisk AB

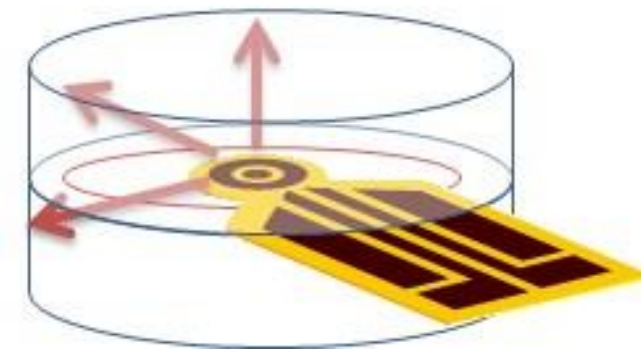
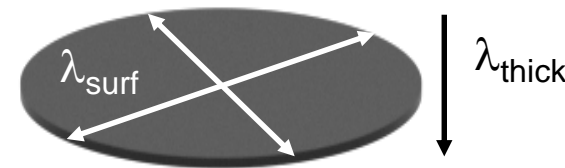


Quelle: HotDisk AB

Specimen

$\varnothing \geq 10\text{mm}$

$d \geq 4\text{mm}$



Quelle: HotDisk AB

Thermal Conductivity – Technique Comparison

- Often challenging task with polymers to define thermal conductivity.
- Material understanding required to use appropriate testing method.



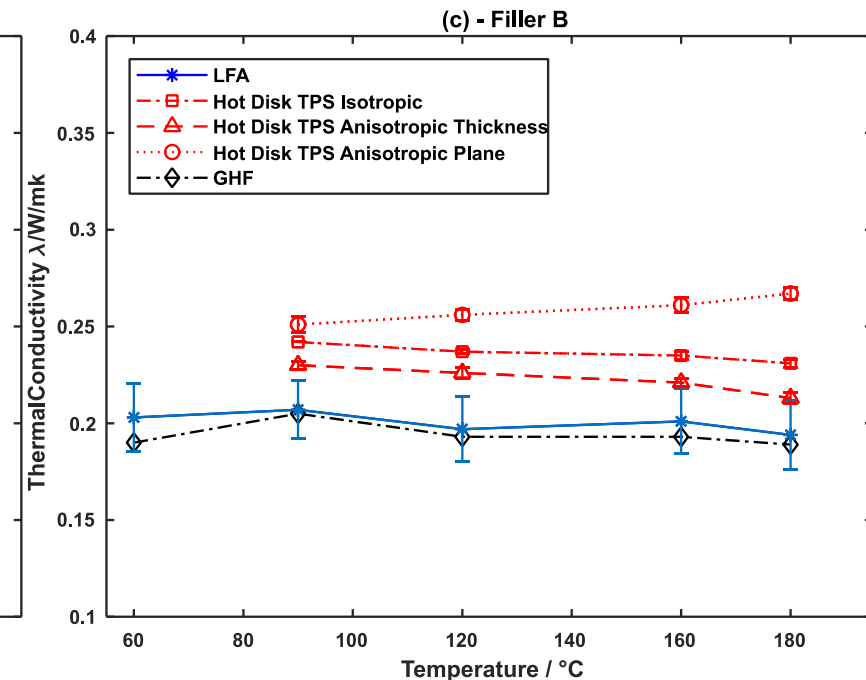
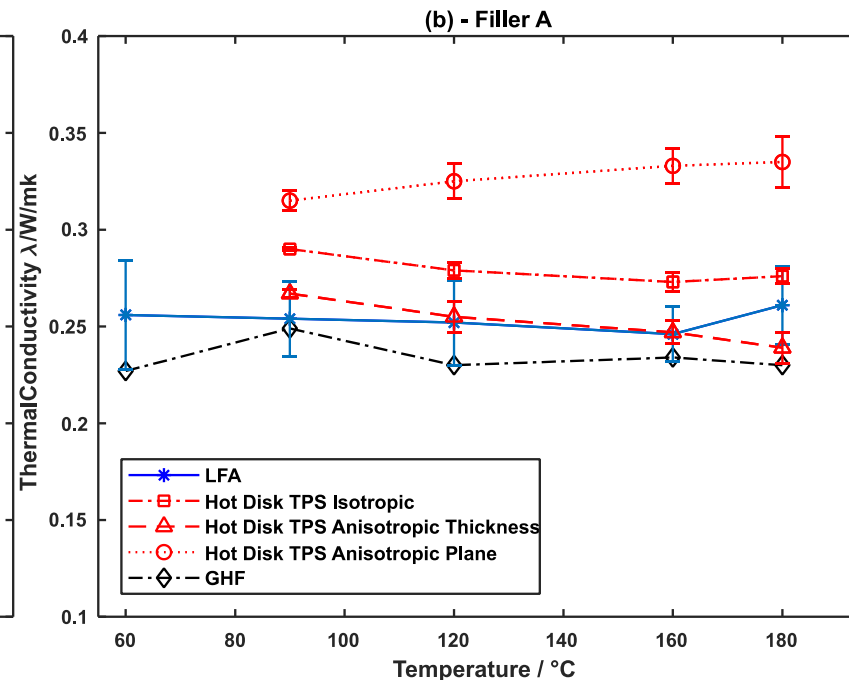
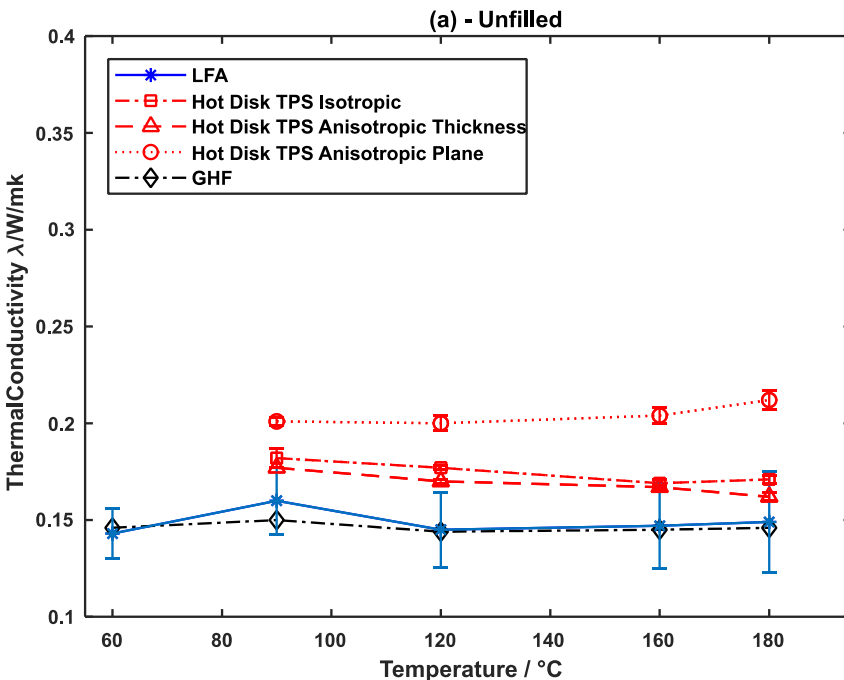
Source: Netzsch



Source: HotDisk AB



Source: TA Instruments

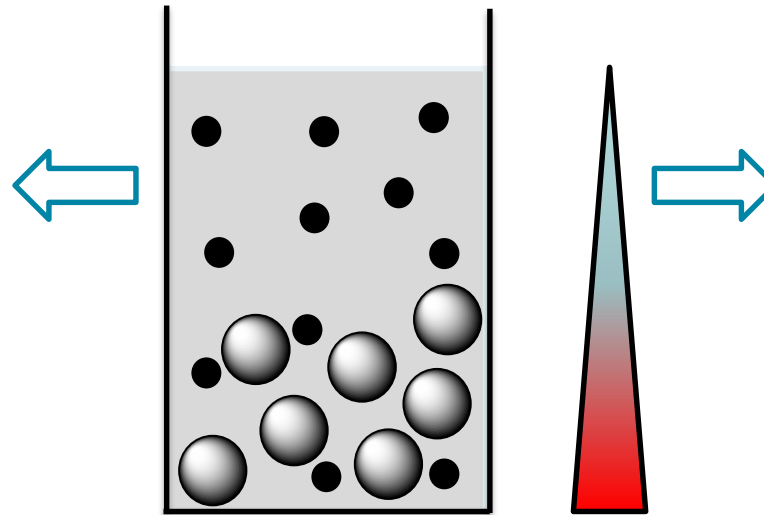


Gschwandl, M., et al. (2019). Thermal conductivity measurement of industrial rubber compounds using laser flash analysis: Applicability, comparison and evaluation, AIP Conference Proceedings 2065, 030041 (2019); <https://doi.org/10.1063/1.5088299>

Economic Aspects:

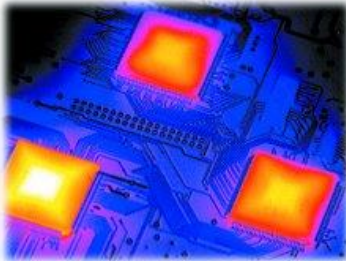
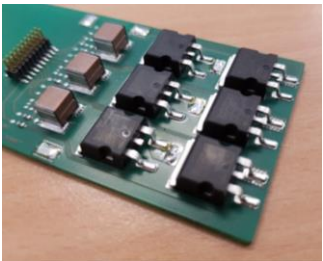
- High Thermal Conductivity where the heat is produced.
- Reduction of the overall filler content.
- **Cost reduction.**

Application Example Gradient Composites



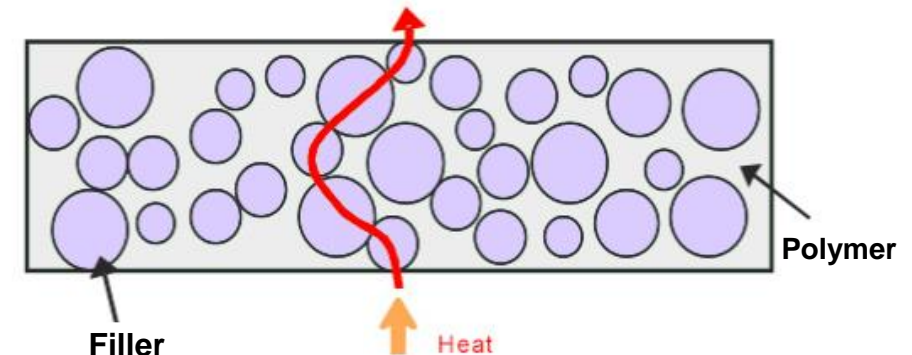
Scientific Aspects:

- Some particles are only commercially available in the micrometer range (e.g. hBN)
- Nanoparticles tend to agglomerate
- **Comparison of the heat dissipation in gradient and homogeneous materials**



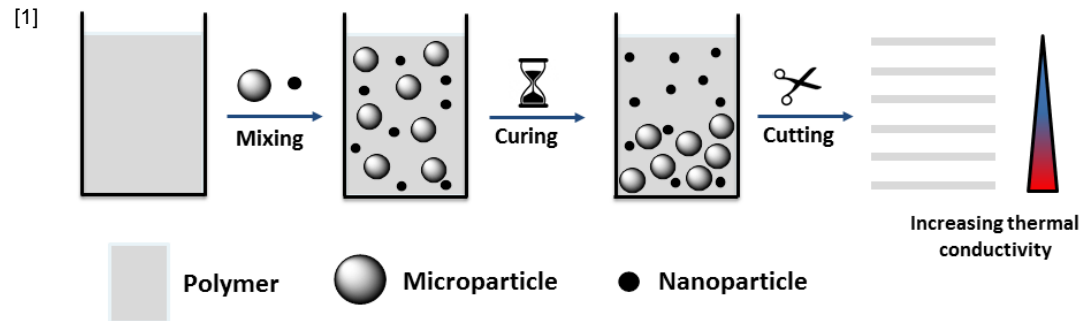
04.03.2020, mG

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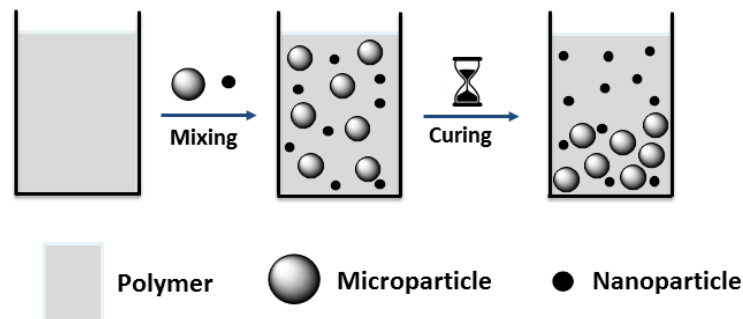
Experimental Details

1. Thinly sliced samples were tested using the Laser Flash Analysis and Differential Scanning Calorimetry.



Source: Netzsch

2. Entire specimen was tested using the Guarded Heat Flow Meter for a homogenized thermal conductivity.

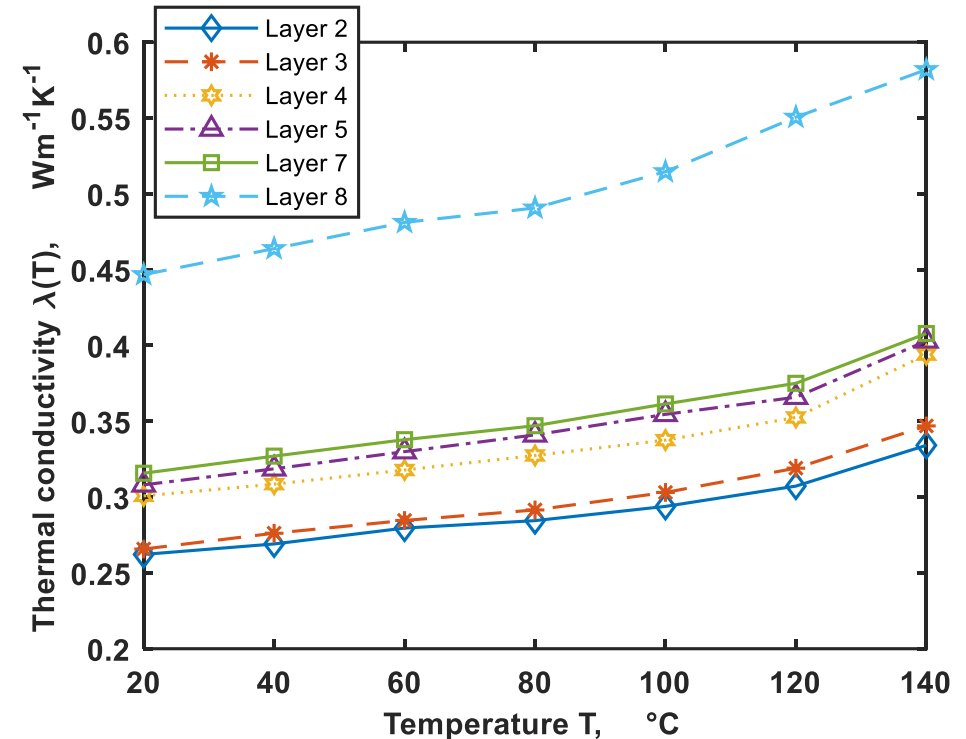
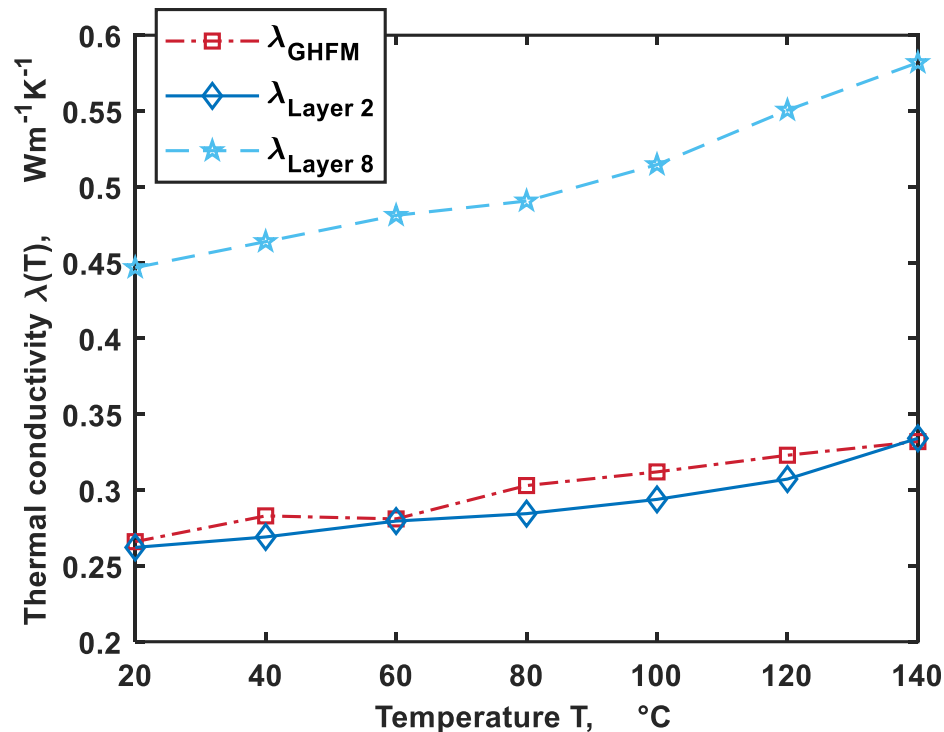


Source: TA Instruments

Morak ,M., et al. (2018). *Heat Dissipation in Epoxy/Amine-Based Gradient Composites with Alumina Particles: A Critical Evaluation of Thermal Conductivity Polymers 2018*, Vol. 10 (10), p 1131, DOI: 10.3390/polym10101131

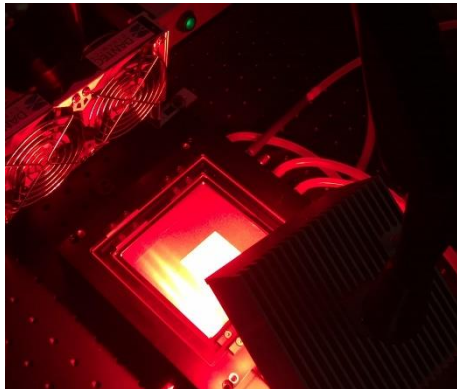
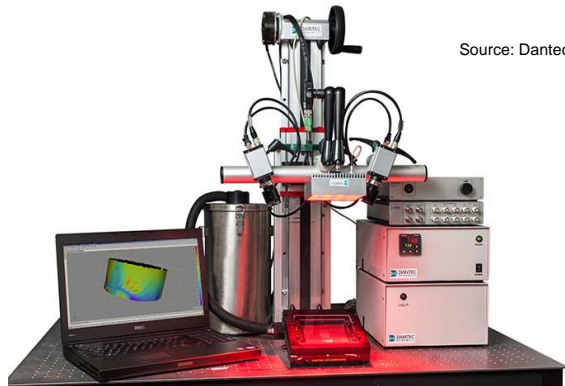
- Thermal conductivity for bulk material of $0.25 \text{ Wm}^{-1}\text{K}^{-1}$
- GHFM result slightly elevated to lowest result of LFA layer by layer measurement
- Verifies that the bulk thermal conductivity of an gradient material is limited by the lowest thermal conductivity present

Morak ,M., et al. (2018). *Heat Dissipation in Epoxy/Amine-Based Gradient Composites with Alumina Particles: A Critical Evaluation of Thermal Conductivity Polymers* 2018, Vol. 10 (10), p 1131, DOI: 10.3390/polym10101131

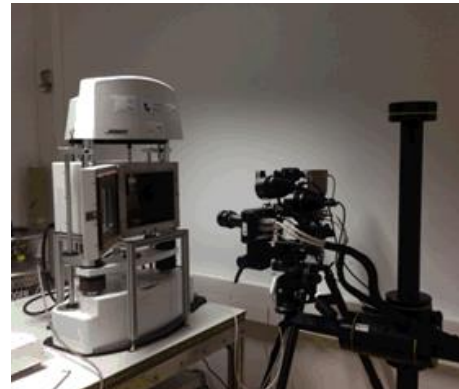


Thermal Expansion

Dantec Q400 System



Aramis 4M System



Thermo-Mechanical-Analysis

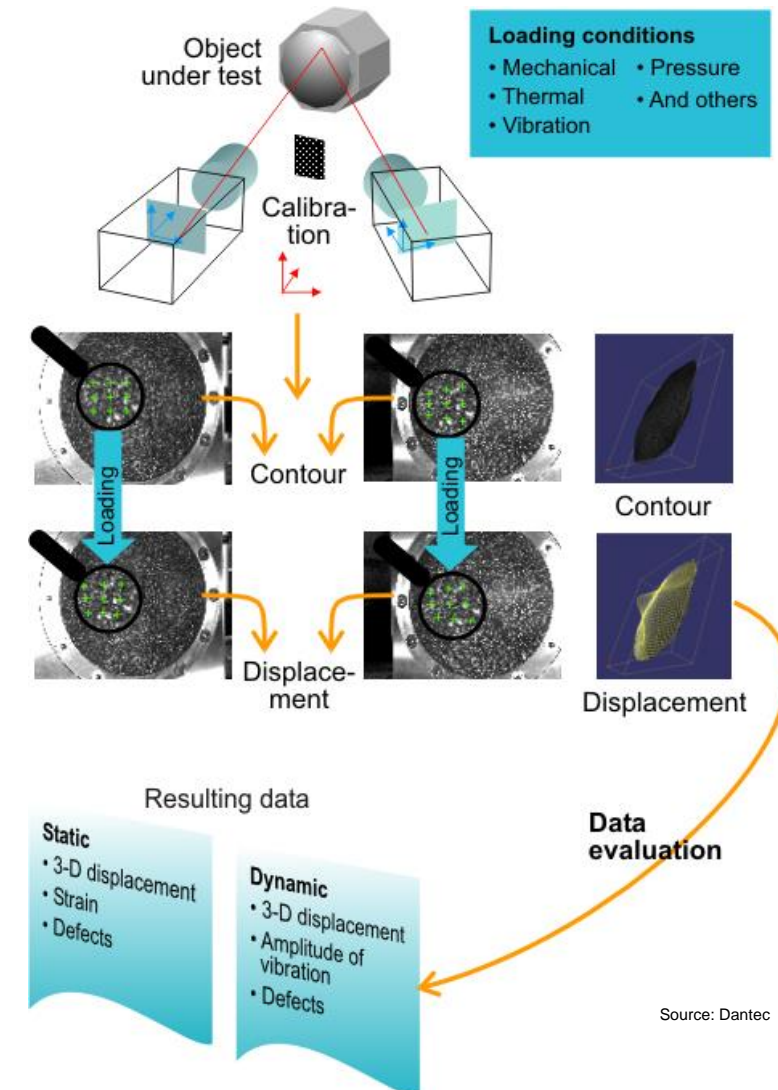


Measuring Principle

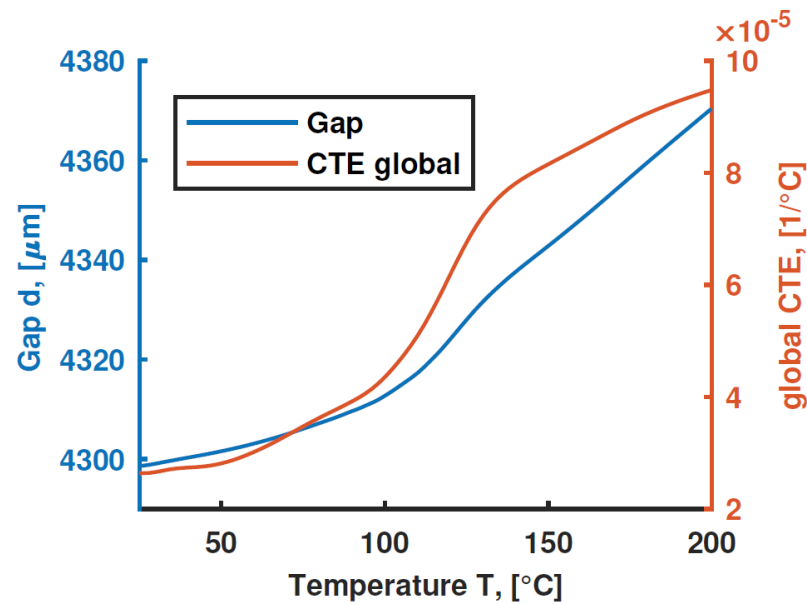
- Using stereoscopic sensors (camera) setup each object point is converted into the pixel spaces of images.
- Knowing the imaging parameters and orientation of the cameras each object point can be calculated.
- Using a stochastic speckle pattern on the object surface, the position of each object can be tracked.

Displacement measurement

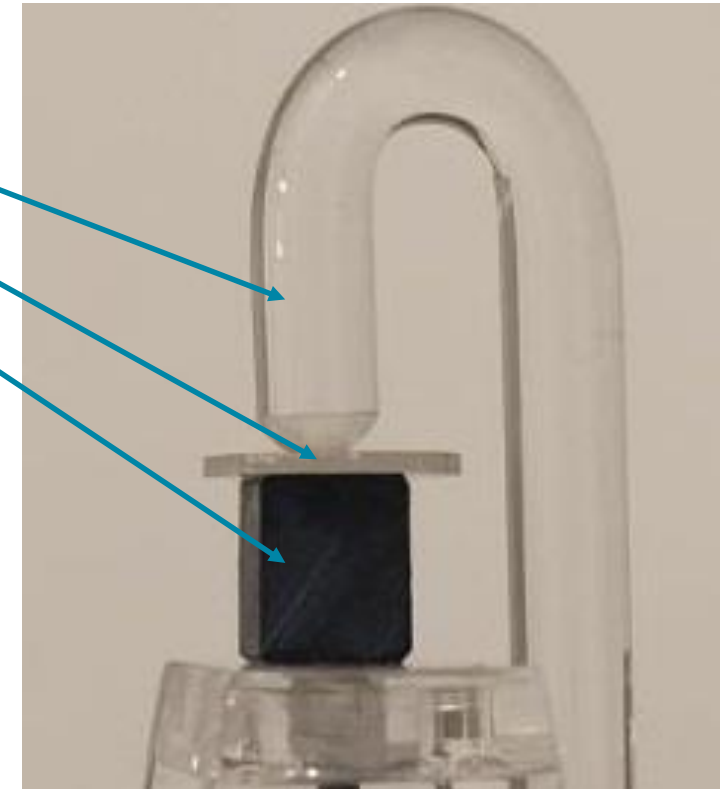
- Calculating the transformation parameters for images under different loading conditions the displacement & strain vector is determined.



- During the measurement the change of the sample length is measured constantly over the temperature rise. For smooth contact between indenter and specimen a thin glass plate is inserted.



Indenter
Glass plate
Specimen



Test Parameters

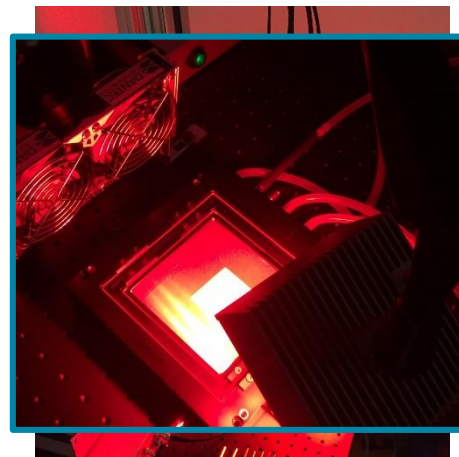
Parameters	Aramis 4M	Dantec Q400	TMA
Samples resin	1	2	2
Samples FR4	2	2	-
Temperature	30°-230°C	30°-230°C	50°-230°C
Heating rate	2 K/min	1 K/min	5 K/min

Materials

- Anisotropic FR4-Prepreg
- Corresponding isotropic epoxy resin



Aramis 4M System



Dantec Q400 System

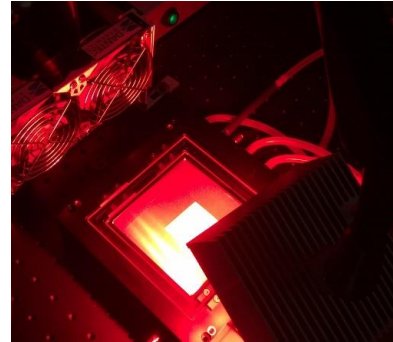


TMA

Thermal Expansion – Technique Comparison (II)

- Comparison of two DIC systems
- Isotropic resin referenced with TMA
- DIC for anisotropic thermal expansion of reinforced materials.
- Significant deviations between all systems after T_G

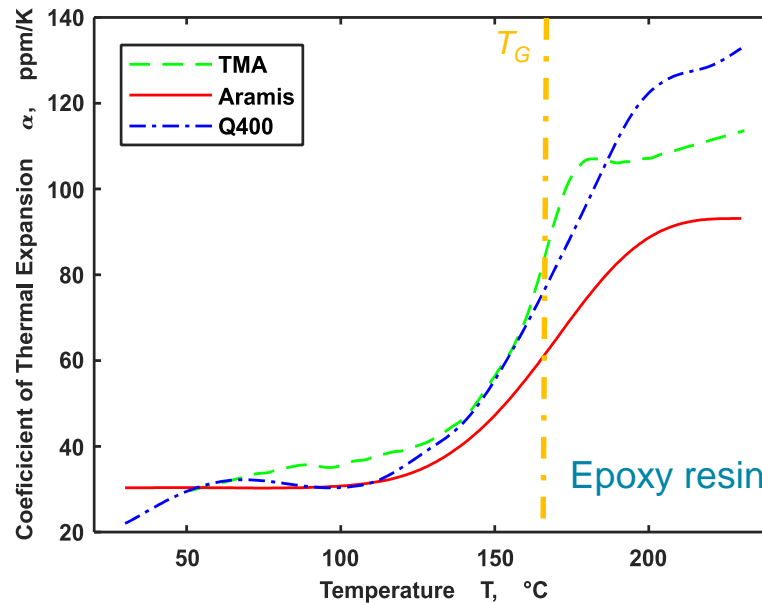
Dantec Q400



Aramis 4M

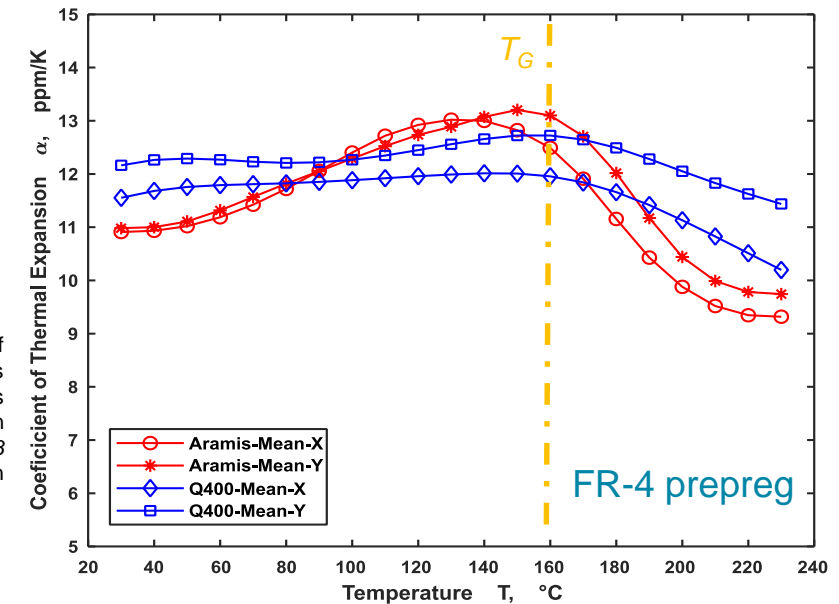


TMA



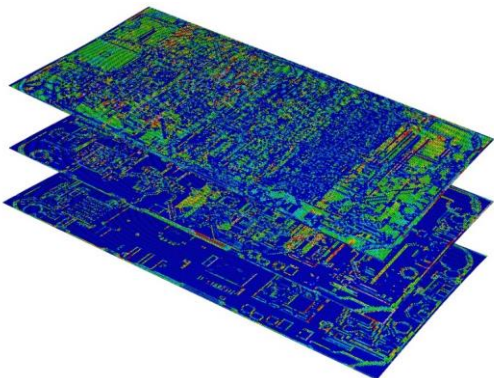
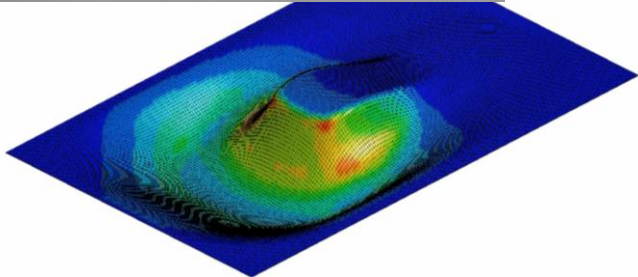
$T_G = 160^\circ\text{C}$

M. Gschwandl et al. "Evaluation of Digital Image Correlation Techniques for the Determination of Coefficients of Thermal Expansion for Thin Reinforced Polymers." 2019, 2018 20th International Conference on Electronic Materials and Packaging doi:10.1109/EMAP.2018.8660763

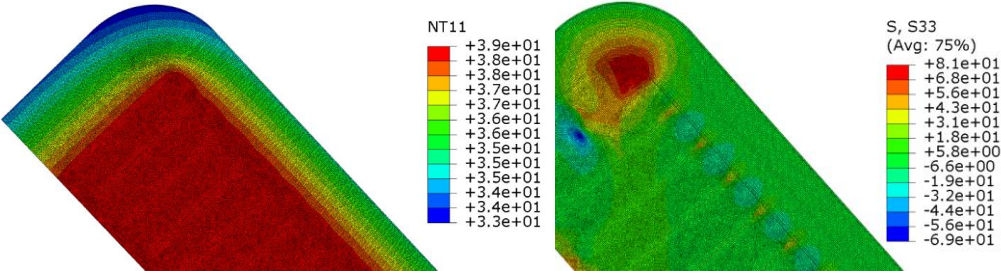
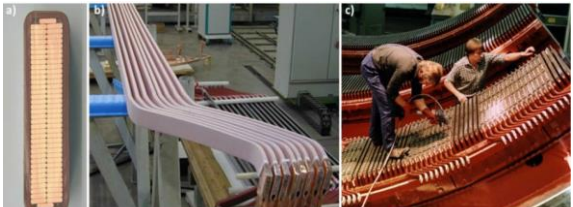
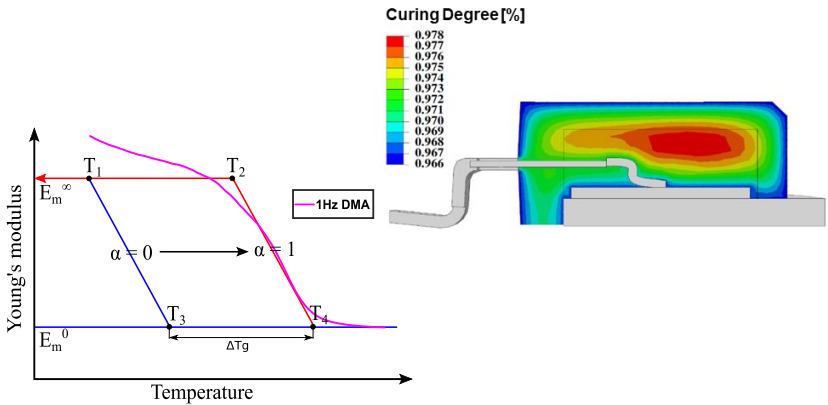
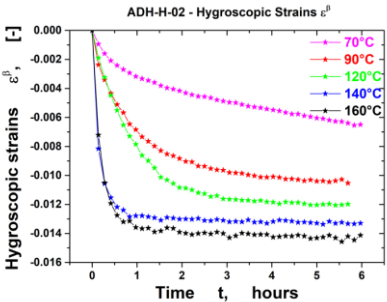
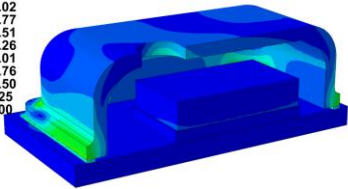
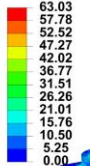


Application Examples

Application Examples



Mises [MPa]



Polymer Competence Center Leoben GmbH

Division Simulation and Modeling

„Computation Expertise Meets Polymer Science“

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