OVERVIEW ON PROCEDURE FOR PCM CHARACTERIZATION

Thomas Haussmann

Fraunhofer Institute for Solar Energy Systems ISE

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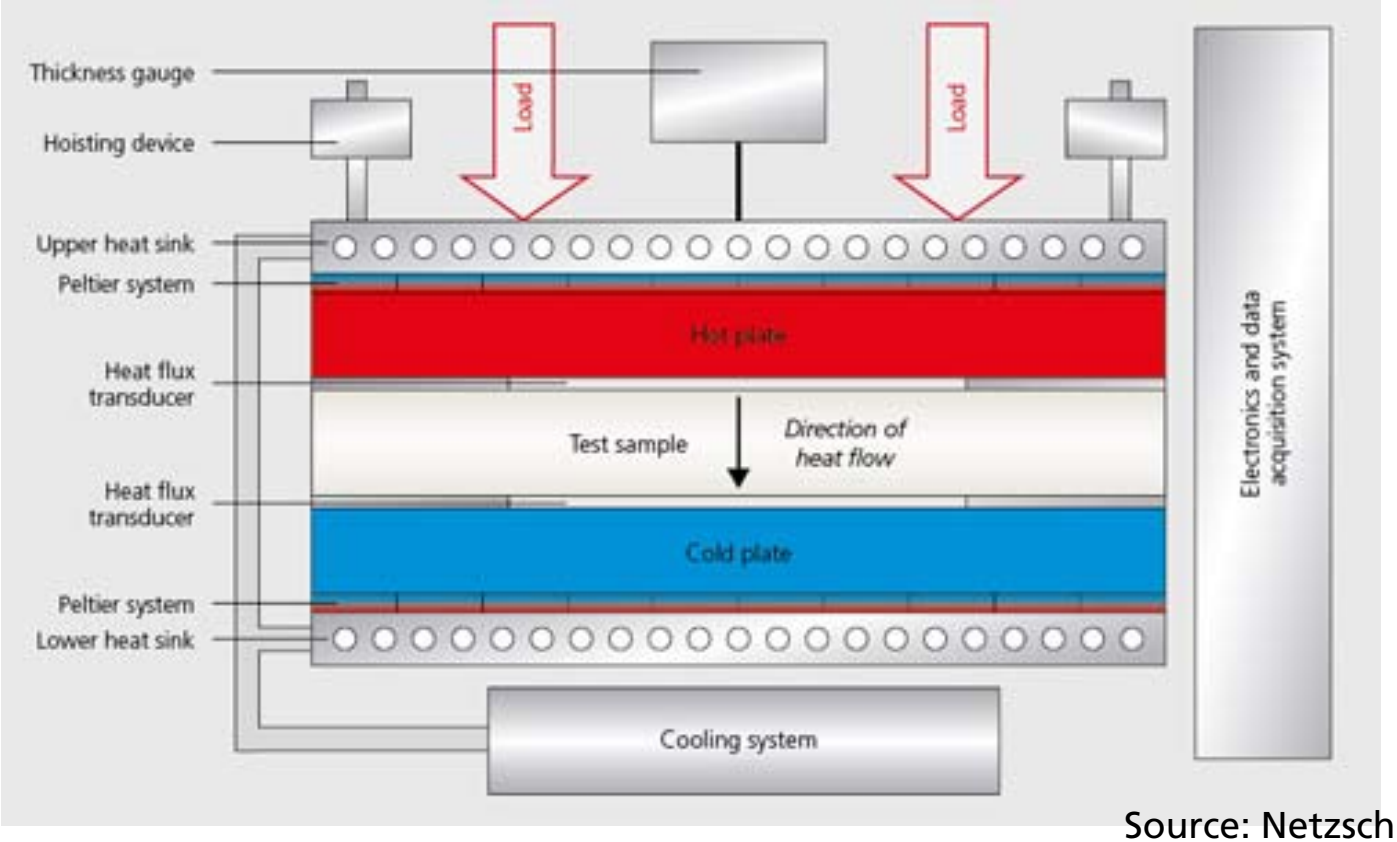
AGENDA

Measurement Standards regarding PCMs

- RAL GZ 896 (german/european quality label for PCM)
  - 3 quality criterias: Phase transition temperature and stored heat, thermal conductivity and cycling stability
  - accredited measuring methods (DSC, T-History, multilayer calorimeter…)
- ASTM C1784-13 “Standard Test method for Using a Heat Flow Meter Apparatus for Measuring Thermal Storage Properties of Phase Change Materials and Products”
  - Heat Flow Meter Apparatus
- Task 4229
  - DSC

ASTM C1784 - 13

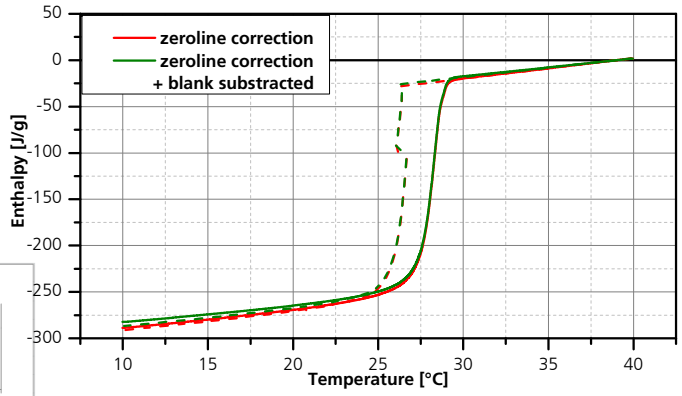
- Phase transition temperature and stored heat measured with HFMA



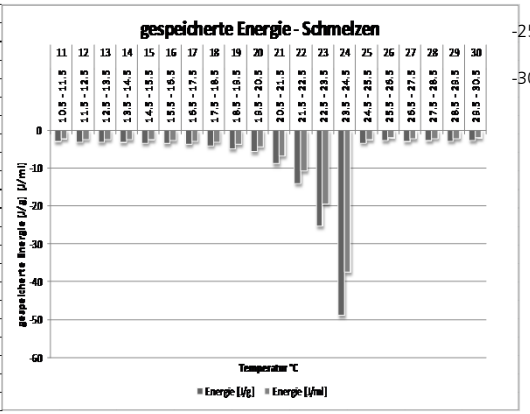
Source: Netzsch

Main Commonalities RAL/ASTM

- PCM classification:
  - RAL: PCM, PCM composites, PCM objects, PCM Systems
  - ASTM: PCM objects => PCM products
- Results presentation:
  - As H/t digram
  - Box diagram (<=1K steps)
- >= 3 measurements per sample



Temp [°C]	T-Intervall [°C]	Energie [J/g]	Energie [J/ml]
11	10.5 - 11.5	-3.13	-2.40
12	11.5 - 12.5	-3.19	-2.44
13	12.5 - 13.5	-3.25	-2.49
14	13.5 - 14.5	-3.36	-2.58
15	14.5 - 15.5	-3.48	-2.67
16	15.5 - 16.5	-3.63	-2.79
17	16.5 - 17.5	-3.83	-2.94
18	17.5 - 18.5	-4.15	-3.18
19	18.5 - 19.5	-5.02	-3.85
20	19.5 - 20.5	-5.81	-4.46
21	20.5 - 21.5	-8.99	-6.89
22	21.5 - 22.5	-14.13	-10.84
23	22.5 - 23.5	-25.39	-19.48
24	23.5 - 24.5	-49.03	-37.61
25	24.5 - 25.5	-3.41	-2.62
26	25.5 - 26.5	-2.78	-2.13
27	26.5 - 27.5	-3.05	-2.35
28	27.5 - 28.5	-2.88	-2.21
29	28.5 - 29.5	-2.93	-2.25
30	29.5 - 30.5	-2.79	-2.14



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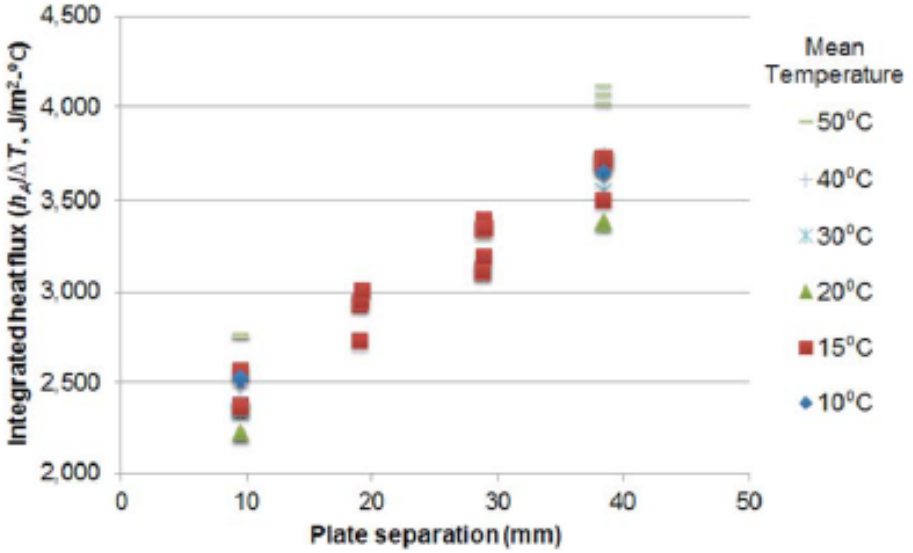
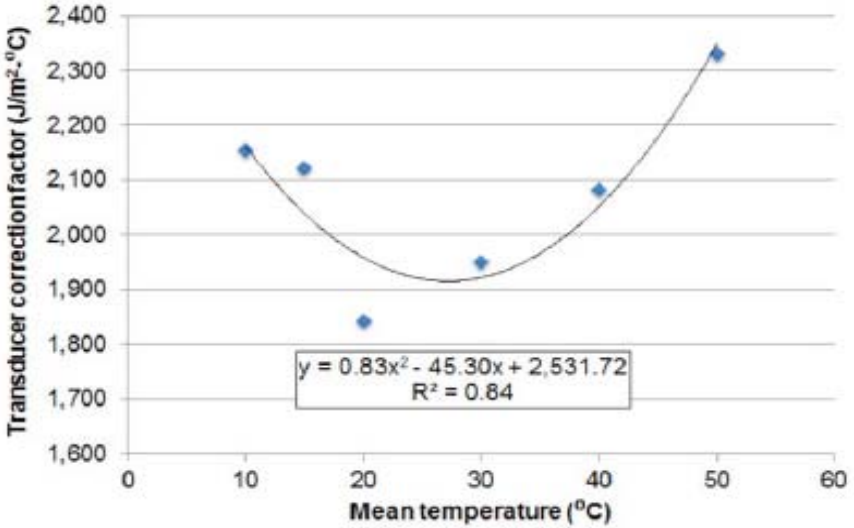
Calibration methods

- RAL: Not defined => manufacturers recommendation => Task 4229
- ASTM enthalpy calibration:
  - Capacity and temperature depending deviations of the hf-transducer are taken into account
  - Consideration of Additional material layers (e.g. for contact improvement between sample and plates)
- Temperature calibration definded in ASTM E967 ?

Calibration procedure ASTM

- >2 specimens (different sample heigts) with known and small heat capacity
- Stepwise (eg. 10°C) heating over the whole T-range
- Integral of heat flux over time divided by step temperature difference
- plot integrated heat flux vs. plate separation
- Extrapolate to 0 thickness for each mean temperature
- Plot Zero plate separation vs mein temperature to get transducer correction factor as a function of plate temperature





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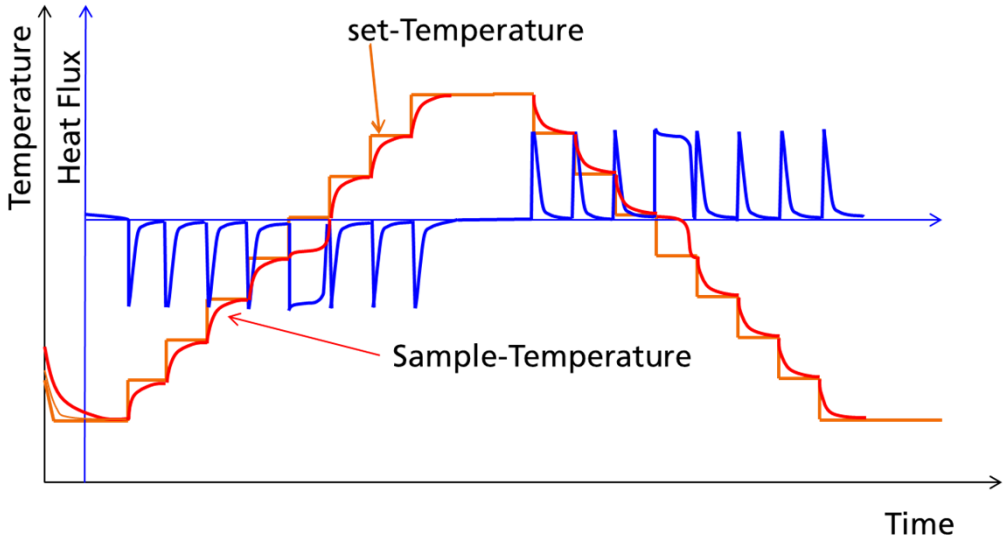
ASTM: determination of test parameters

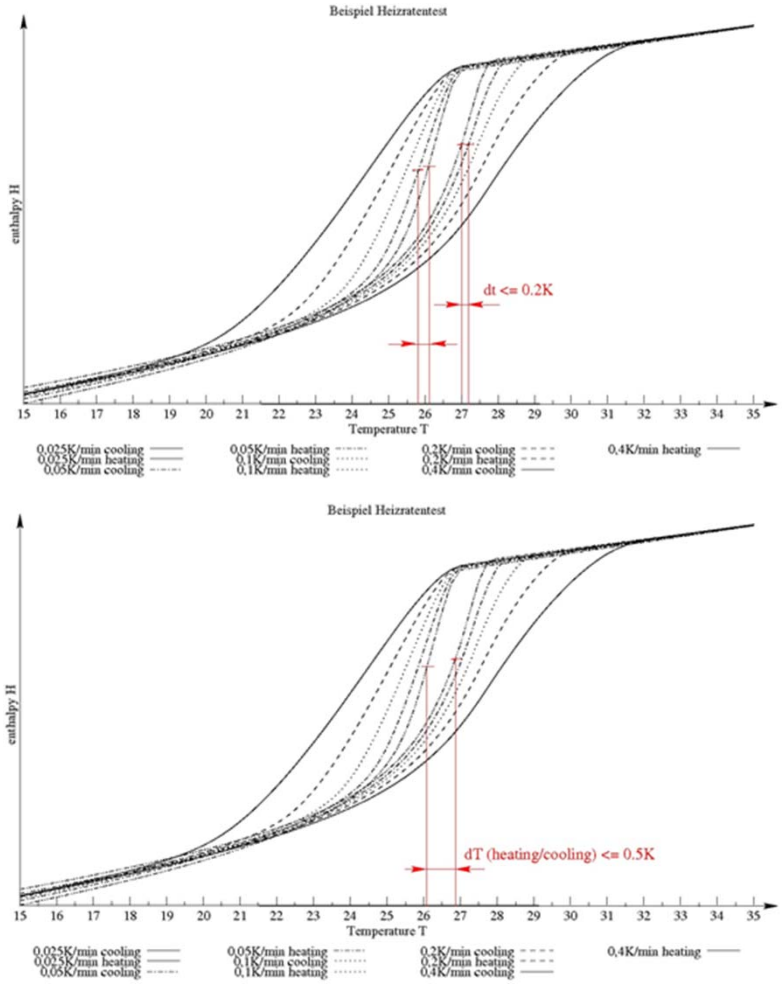
- ASTM:
  - Start 10°C below melting temperature
  - Stewise increment of temperature (1.5K+- 0.5K) >2h relaxation at each step
  - End: if energy stored returns to a small value (=> fully melted)
  - Repeat procedure for cooling

- => PCM Active Range, temperature step size, relaxation time at each step



RAL: determination of test parameters

- Heat rate test:
  - Temperature range must cover the whole melting and cristallization process
  - Halving heating rates until
    - Peak temperature differences <0,2K between two heat rates
    - Or: less than 0,5K between Peak heating and cooling



- => maximum heating rate

ASTM: sample measurement and calculations

- Similar to first test from 10K below to 10K above PCM Active Range
- 3 measurements with different starting temperature to increase precision

Definition PCM Active Range

RAL sample measurement and calculations

- With maximum heating:
  - Measure three samples with at least 6 cycles, temperature range must cover the whole cycle at least +-5K larger than offset melting and cristallization
  - first two cycles (Premelt) can be performed faster
  - 3rd cycle for determination of Phase transition temperature and stored heat
  - Cycle 3-6 used for determination of minimum Nukleation temperature

Main differences between RAL and ASTM

ASTM	RAL
Stepwise	dynamic (stepwise also allowed
	Determination of nucleation
	cycling stability

Thank you for your attention!

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Georg Hagelstein

www.ise.fraunhofer.de

georg.hagelstein@ise.fraunhofer.de