Enabling mass loss measurement for controlled atmosphere experiments in a tube furnace

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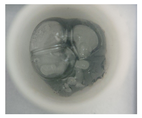
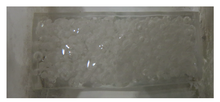
Thermogravimetric analysis

MOTIVATION

Are small scale experiments good representatives for bench and real scale experiments?
→ e.g. Bubbling effect

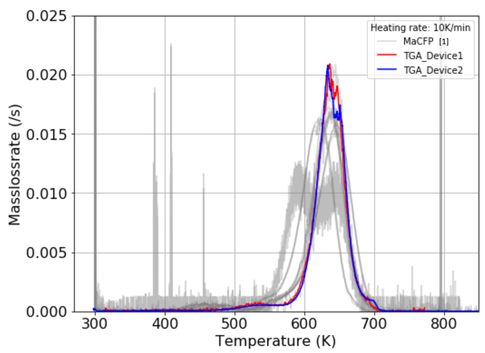


PMMA
T = 290˚C



PMMA
T = 350˚C

Small scale experiment more sensitive to fluctuations in random process.



TGA RESULTS

- 2 different devices
- Same mass (8.5 mg)
- Same material: Black PMMA
- Nitrogen atmosphere
- Same flow rate: 20 ml/min

→ Good agreement between different devices for inert atmosphere

ADVANTAGES TGA

Controllable boundary conditions

- Gas flow rate and composition
- Temperature of the furnace
- Homogeneous exposure of the sample to the heat flux

Ref fig [1]

→ Tube furnace: advantageous properties of the TGA with more reproducible sample size

→ Balance needs to be constructed for online mass loss measurements in the tube furnace

REFERENCES:
[1] IAFSS, 2020, Preliminary Summary of Experimental Measurements.
[2] *LINSEIS STA PT1600 Thermowaage Bedienungsanleitung*, Linseis-Messgeräte. Selb.

Tube furnace

SET-UP

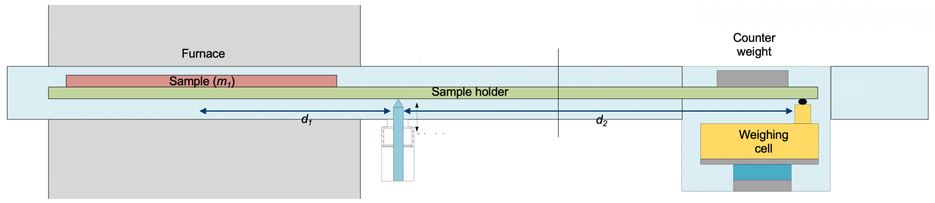
- Specimen size: 80 cm (∼ 100g for PMMA)
- Controlled atmosphere: gas species and flow rate
- Isothermal and non-isothermal experiments possible
- Max heating rate 5K/min, max temperature: 1000˚C

ANALYTICS

- Gasanalyser: CO, CO_2, O_2 concentrations
- Thermocouples: temperature measurements inside the oven
- Mass spectrometer: to be installed
- BALANCE: under construction

BALANCE

- Online mass loss measurement, both for isothermal as for dynamic experiments
- Seesaw mechanism: sample on one side, weighing cell on the other side



$$F_{balance} = \frac{m_1 g\, d_1}{d_2}$$

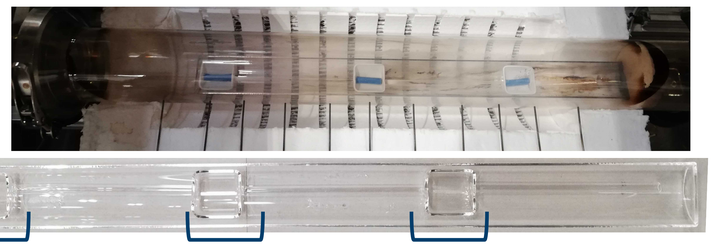
$F_{balance}$: force on the balance
g: gravitational constant

CHALLENGES:

- Buoyancy effects: correct with zero curves (weight measurements without sample or with inert sample)
- Effect of thinning of the material → Changes the centre of mass

SMALL SCALE EXPERIMENTS AS REFERENCE

- All specimens (33x15x5mm) start at same time in oven and are moved out in different time steps
  → Mass loss known for different temperatures
- Also possible with TGA sized samples
  → check reproducibility between both devices.
- Chemical analysis of all the intermediate steps
  → molecular mass, C,H,O analysis



Sample 4: in oven from T_0 to T_4
Sample 3: in oven from T_0 to T_3
Sample 2: in oven from T_0 to T_2
Sample 1: in oven from T_0 to T_1

Outlook

- Compare experimental results with FDS simulations
- Goal
  - Additional scale for material parameter determination and validation
  - Pyrolysis model improvement