Ceramics Laboratory Facilities

Surface analysis
  Wettability and drop shape
  Gas adsorption analyzation
  Surface profile analysis

Furnaces
Dilatometry

Supercritical carbon dioxide equipment
Pulsed laser ablation in scCO2
Ceramics additive manufacturing
Particle size measurement
Ceramics laboratory

- For the research and processing of functional ceramics, the laboratories of DMS have a whole processing chain from powder synthesis to component sintering, e.g., sol-gel synthesis facilities, supercritical carbon dioxide (ScCO2) processing capabilities for nanopowder production, 3D-additive shapeforming and sintering in ambient and protective environments.
- In addition to the basic characterization devices, the precursor chemistry and powder synthesis is supported by special characterization equipment such as a gas adsorption/chemisorption device and a particle size analyzer. The heat treatment cycle is studied with dilatometry and several furnaces including a possibility for different atmospheres.
- The laser technology (with collaboration of production technology offers a concentrated and clean heat source for advanced processing of ceramics and modification of surfaces. The final properties of functional surfaces are evaluated, e.g., with streaming potential and surface energy measurements.
- Ceramic powders can be prepared into additive manufacturing slurries and complex geometries can be printed with digital light processing based stereolithography additive manufacturing system.
Krüss DSA100 Drop Shape Analyzer

Wettability and wetting of the surfaces can be analysed with drop shape analyzer placed in controlled room (temperature 21°C and relative humidity 50%). Many solid materials and liquids can be tested. Small sample size and liquid amounts used (a few cm/µL).

**Measured values based on image analysis of droplets:**

- Static contact angle
- Advancing and receiving angles
- Hysteresis
- Sliding angle/roll-off angle
- Surface free energy
- Surface tension

**Droplet behavior with Peltier temperature control plate**

- Temperature range -20 - +120°C
- Heating and cooling rate +/- 1K/s
- Maximum sample size (L x W x H) 90 x 90 x 20 mm
3Flex gas adsorption analyzer can be used to analyze surface area and pore size of the powder or fibrous samples by adsorption and desorption of the gases. Equipment is capable of measuring 3 samples simultaneously with different gases if needed, while pretreating 6 samples. As a result of the measurement is full adsorption-desorption isotherm, from where for example specific surface area, pore volume and pore size distribution can be determined in range of 0,35 to 500 nm

**Technical information of the device**

- 3 measurement ports, from which 2 are equipped to micro pore range (below 2nm)
- 2 sizes of sample tubes, inner diameter of the tube 6 mm and 9mm
- For powder and fibrous samples, which can be fitted inside the sample tube
- Analysis range 1,3*10^-8 - 1.0 P/P0
- Minimum measurable surface area 0,01 m2/g
- Analysis gases: nitrogen as standard, also other gases can be used if needed
**Surface analysis**

**Surface profiles and roughness**
- Topographies of the solid surfaces can be analyzed by optical profilometry (Alicona Infinite Focus G5, AT)
- Typical field area of the measurements: 0.81 x 0.81 mm in the xy-plane (20× objective magnification)
- Ra, Ry, Rz, Sa, Sy, Sz etc. values

**Structures and compositions**
- Optical and electron microscopes (OM, SEM, FESEM)
- Chemical characterization (FTIR, Bruker Tensor 27)
- Thermal analysis (TGA, Netzsch TGA209F Tarsus and DSC, Netzsch DSC214 Polyma)
Furnaces

Material Science has several furnaces for example for heat treatments. These are used to produce components with demanded strength, hardness, porosity, pore size distribution and wanted functional properties from shaped green state ceramics. There are high temperature furnaces especially for ceramic specimens. We have two air atmosphere chamber furnaces and one furnace with inert atmosphere.

**High Temperature Furnaces ENTECH SF6 (1990, 1995)**

- maximum temperature 1650-1700°C
- chamber size: 200mm x 200mm x 350mm
- maximum allowed heating rate up to 1600°C is 20°C/min
- cooling rate for empty furnace with closed door
  - 1600-1100°C 50°C/min
  - 1100-600°C 17°C/min
  - 600-200°C 7°C/min
- Limitations for Specimen
  - metallic and ceramic materials max T around 1000°C
  - maximum specimen 180 mm x 180 mm x 300 mm; Maximum weight 4 kg

**Inert atmosphere furnace ELATEC**

Three different heating element configuration and atmosphere possibilities.

- graphite heating elements:
  - Atmosphere: vacuum or inert gas up to 100 Pa pressure
- Kanthal heating element
  - Atmosphere: vacuum (1Pa-1100°C) or in inert gas up to 150 Pa pressure (1600°C).
- Metallic heating elements
  - Atmosphere: vacuum or inert gas up to 150 Pa pressure.
Furnace
Nabertherm VHT 8/22-GR

Chamber
• 8 liters (170x240x200) or with Graphite process box 3 liters (120x210x150)
• Max. Charge 5 kg
Max. Temperature 2200 C
• Heating speed max. 600 K/h min 1 K/h (empty furnace)
• Cooling speed max. appx. 1900 k/h (from 2200 in protective gas)

Vacuum & Gas
• 2-stage rotary vane pump Leybold Trivac D16B < 5x10⁻² mbar
• 2 gas connections with mass flow controllers
  • Argon and Nitrogen
  • Gas flow rate 50-500 l/h with filling time appx. 60 s
  • Partial pressure 10-1000 mbar (abs.)

Automatic controller (Siemens S7 PLC) for vacuum, gas and heating
Dilatometry is used to study length changes in ceramics, glasses, metals, composites, and polymers to reveal information regarding their thermal behavior. The samples can be solid, powders or pastes. Typically the device is used to measure the coefficient of thermal expansion from sub-zero to high temperatures or to analyze volumetric phase changes or sintering in different atmospheres with temperature or force modulation. Other uses for the device are shrinkage steps, softening point, glass transition, density changes, decomposition temperatures, anisotrophic behaviour, and thermal kinetics measurements.

**Technical details:**
- Measuring range: 50 mm (± 25 000 µm)
- Δl Resolution (over entire measuring range): 0.1 nm
- Initial sample length: 0-52 mm (diameter 12 mm)
- Temperature range: -180 °C to 1600 °C (accuracy 1 K, stability ±0,02 K)
- Heating rates: 0.001 to 100 °C/min
- Force range: 10 mN...3 N
- Gas Atmosphere: Inert, oxidizing, reducing or vacuum.
Supercritical carbon dioxide (scCO2) equipment

Used for fabrication of coatings, particle synthesis, aerogel production and extraction processes.

Specifications:

- 2 x 1,57 liter stainless steel (316L) vessels with spray nozzles on top
- Analysis cell (window d = 35 mm)
- CO₂ pump 250 g/min
- Co-solvent pump 50 g/min
- Max pressure 350 bar
- Max temperature 150 °C
Pulsed laser ablation in supercritical carbon dioxide is used:
1. For **Nanoparticle** synthesis and processing
2. For **surface patterning/modification**
3. Maximum target size 35x35x15 mm, upon which maximum scan size through 15 mm sapphire window is 8x8 mm

This set-up includes the following:

1. **Laser set-up**:
   - 70W, 250 nanosecond fiber laser at 1064 nm
   - Maximum energy per pulse = 650 µJ
   - f-theta lens 80 mm, 160 mm
   - Maximum scanning speed 2000 mm/s

2. **scCO₂ equipment**:
   - High-pressure SS vessel with heating
   - $P_{\text{max}}$ 650 bar, $T_{\text{max}}$ 150 °C
   - Vessel Temperature controller (25 °C – 150 °C )
   - PC controlled mechanical CO₂ pump, Co-solvent pump
   - Automatic back-pressure regulator
Ceramics additive manufacturing

**Stereolithography 3D-printing of ceramic materials**

- Aluminium oxide, Zirconium oxide, Zeolite, γ-Aluminium oxide, Silicon nitride

Stereolithography 3D-printing of ceramic materials is used to manufacture complex shapes and geometries for the ceramic components.

Photo-curable polymer binder network is printed around the ceramic particles. After that, the polymer binder is burned away and the ceramic particles sintered to dense ceramic component.

High density or high porosity ceramic components are achievable.
Particle size measurement

Malvern Zetasizer Nano ZS

Measurement:
- Molecular size
- Molecular weight
- Particle size (0.3nm - 10µm)
- Zeta potential

Technology:
- Dynamic Light Scattering,
- Electrophoretic Light Scattering,
- Static Light Scattering
Contacts

Professor Erkki Levänen
erkki.levanen@tuni.fi
+358408490191
K3228