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µ-Reactor Production using µ-CIM Technology

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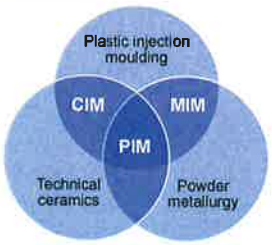
OBJECTIVE

Objective of this work is the production of ceramic µ-reactors for nanoparticle synthesis in supercritical water (sc-H_2O) using the ceramic injection moulding (CIM) technology. The µ-reactors have to be hydrothermally stable up to 500 °C and withstand high pressures of 20 MPa to be suited for precipitation reactions in sc-H_2O.

In terms of maximizing energy and material efficiency and improving process control, the synthesis reactions will be carried out in narrow flow channels with defined small-scale fluid volumes. An integrated sensor system for dielectric spectroscopy should detect the reaction progress, support process-analytical quality control and enables in-depth knowledge and precise regulation of local conditions which helps to avoid undesirable side reactions in synthesis and increase the selectivity.



PROCESS

FEEDSTOCK DEVELOPMENT

CERAMIC INJECTION MOULDING

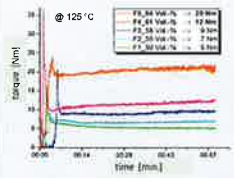
JOINING

DEBINDING

SINTERING

RESULTS

Composition screening using torque-recording mixer-kneader



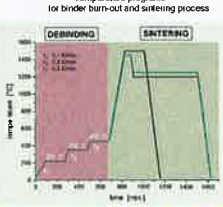
- *Hydrothermally resistent mechanoceramic: α-Al_2O_3 (d_{50} ~ 190 nm, BET ~ 8,6 m^2/g)*
- *Environmentally friendly binder system:*
  PVB (structural polymer)
  PEG 4000 (base polymer)
  Stearic acid (dispersant)
- *High filling degrees up to 64 vol.-% ceramic content*

- *Highly filled compounds processed by µ-CIM technology*
- *Injection moulded test specimens:*
  (1) Large plates
  (2) Tensile specimens
  (3) Tribology specimens (discs)
- *High moulding accuracy*
- *Significant differences in density for large specimens*

TEST SPECIMENS
with 62 vol.-% ceramic content

- *Material specific temperatures for binder burn-out determined by thermogravimetric analysis*
- *Heating rate variations for larger parts need very low heating rates (< 0,1 K/min.)*
- *Dwells at each temperature depend also on the part size (> 5h for 60x100x5 mm³ plates)*

Temperature programs
for binder burn-out and sintering process

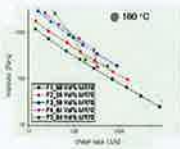


SEM micrographs of sintered specimens for structural analysis

- *Joining of two moulded parts is needed to combine the lid and the base part of each ceramic µ-reactor*
- *Joining by uniaxial pressure sintering of two stacked components was not successful*
- *Using an Al_2O_3-based paste for joining causes inhomogenities along the joining seam*
- ***Best results are achieved by ...***
  1. *Dissolving the surfaces being joined with ethanol*
  2. *Stacking the components and let them dry for 30 min.*
  3. *Outsourcing the combined stack at 80 °C and uniaxial pressure for 30 min*
  4. *Cooling the joined part down to RT before thermal follow-up treatment*

TARGET

Compound viscosity for different filler degrees from 50 to 64 vol.-%



- ***Iterative adjustment of filler** degree with focus on the reproducible moulding accuracy*
- ***Lowering the compound viscosity by fine adjustment of the PVB:PEG ratio***

- *Injection moulding of structured plates with different flow channel designs*
- *Elaborate the best process parameters*
  - *Compound temperature: 150 – 190 ° C*
  - *Mould temperature: 30 – 45 ° C*
  - *Injection speed: 30 – 100 mm/s*
- *Simulate the flow behavior during injection of the compound into the mould and image emerging density inhomogenities*

- *Binder burn-out needs to be done more slowly for larger green bodies. A purely thermal debinding process in case of a reactor size of 60 x 100 x 10 mm³ requires several days.*
- *Investigate the effect of a chemical pretreatment to elute the water soluble PEG binder content out of the green body before thermal treatment*
- *Refining the temperature program to adopt the dwell times and heating rates to the part size and avoid microcracks and defects caused by blistering*

- *The highest fracture toughness can be achieved when densification of the ceramic particles is maximum and their grain growth is minimum*
- *The 2-step sintering method seems to be a good opportunity to realize the required fine-grained microstructure and simultaneously high densification*

$$m = m_0 \exp\left[-\frac{Q_m}{kT}\right]$$

Nernst-Einstein relation
m: growth rate of grain boundaries
Q_m: thermal activation energy

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