Ceramic materials based on nanopowders

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Structure of Presentation:

1. The Fraunhofer Institute for Ceramic Technologies and Systems IKTS
2. Vision Nanoceramics
3. Selected Results and Experiences
4. Continuation
5. Summary
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Fraunhofer-Gesellschaft

- Fraunhofer is Europe’s largest application-oriented research organization
- more than 80 research units, including 60 Fraunhofer Institutes at different locations in Germany
- the majority of the 18,000 staff are qualified scientists and engineers

IKTS has more about 400 employees in 2 locations (Dresden, Hermsdorf)
Hermsdorf (Thuringia)  ➩  Dresden (Saxonia)

Start 2010:
- One institute / Two location
- More potential in employees and equipment
- Addition of Know-how and capacity
- Better efficiency for customers
- Completely line from research to pilot plant

The Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), Dresden and Hermsdorf, covers the complete field of advanced ceramics, from basic research to applications.
Fraunhofer IKTS in profile

Services

Development of
State-of-the-art advanced ceramic materials
Industrial powder metallurgical, wet chemical and precursor-assisted technologies
Prototypical components and systems

for applications in
Energy and environmental technology
Mechanical and plant engineering
Microsystems and medical engineering
Automotive engineering
Fraunhofer IKTS in profile
Research fields

Structural ceramics
- Materials
- Processes and Components

Functional ceramics
- Sintering and Characterization
- Energy Systems
- Environmental Engineering and Bioenergy
- Smart Microsystems
What ceramic materials are essential for in Future?

Advanced ceramics as „Enabling Technologies“ with unique attributes:
- Very hard and stable
- Corrosion- and wear resistance
- High temperature stability
- Light weight
- Biocompatibility
- Multi-Functional

Applications:
- Machine building
- Automotive
- Energy systems
- LifeScience / Health care
- Environmental
- IT / Electronic

Nanotechnology Forum, Saint Petersburg / Russia, 03 July 2012
Advanced applications need advanced ceramic materials!

Complex procedure
- Raw material (powder)
- Manufacturing process
- Materials structure

Complex relationship
- Materials structure
- Materials characteristics
- Materials properties

Complex properties
Approach:

Structure design for better material properties

Main emphasis

- Mean crystalite size
- Crystalite size distribution
- Defect free sintered body: without pores, cracks, impurities
- Homogeneous structure and minimizing grain growth
- Homogeneous structure and high final density

To minimize structure and defects:

- Development for manufacturing of bulk nanostructured ceramics
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2. Vision Nanoceramics

Main emphasis on the field of Nano Technologies:

- Characterize and processing of sub-µm- and nano-Powders
- Mixing, homogenizing and coating of nano-Powders with organic temporary additives and development of surface modified powders
- Nanocomposites and sol-gel-coatings for special applications
- Industrial processing technologies for forming and thermal technology
- Ceramic materials with improved properties: strength, hardness, reliability, optical transmission, thermal and chemical resistance
- Surface area machining and structuring

For example:
Nanoparticle characterization

**Challenge:** nano-specific material properties

- Small primary particles that are aggregated and/or agglomerated to larger particles
- High specific surface → Increased adsorption of dispersants and lower solid content in suspension feasible

**Solution:** Combination of different analysis methods and preparation techniques

- BET, electron microscopy, XRD, particle size distribution (DLS, centrifuge)
- Energy input using ultrasound, mechanical dispersers, mills
- Tailored suspension stabilization as key for any further measurements and applications

Interactions of particles among each other and with dispersants

Nanosized Al$_2$O$_3$ powder
Suspension characterization in toxicological studies

Assessment of the impact of nanomaterials on environment and human in terms of a reliable nanotechnology

**Now:** Existing methods and standards for testing chemicals, e.g. using OECD guidelines

**Future:** Adaption of these tests to the requirements of nanomaterials
- Preparation of suspensions for *in vitro* and *in vitro* testing
- Characterization of nanoparticles‘ behavior in physiological solutions or environmental relevant media
Advanced densification technologies for densification of nanomaterials

- **FAST/SPS Hybrid System** – FAST/SPS with additional inductive Heating
  - Temperature up to 2200°C
  - separate Heating SPS and Inductive heating possible

- **Hot Isostatic Pressing System**
  up to 2000°C and 2000 atm
  maximal sample dimensions:
  D 300 mm x 450 mm

- **Development of sintering strategies** for ceramic and nano powders
For instance / result:

Nano- $\text{Si}_3\text{N}_4$ ceramics with high wear resistance

Preparation and characterization of nanosized materials for wear application (e.g. Ball bearings)

Fine grained materials (grain size $\leq 100$ nm) show lower friction coefficients and higher loading capacity in dry rolling (1 % Slip)
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Manufacturing processes for ceramic nanopowders & ceramics

For nanopowder:
- Desintegration / Synthesis
- Agglomeration / Isolation
- Handling / Processing
- Characterization / Preparation

For nanoceramic:
- Shaping / Compaction
- Sintering (+/- sintering aids)
- Processing / Finishing
The Base:
1) Powder manufacturing and characterization

Main emphasis

Mean particle size
Particle size distribution
„Giant particles“
Particle form
Hard or weakly agglomerated grains

- Important to manufacture bulk nanostructured ceramics
- Basic for R&D to manufacture fully dense nanostructured ceramics with improved properties
The shaping process:

2) Powder compaction for nanoceramic materials

Main emphasis

- homogeneous, high green density
- defect free green body: without pores, cracks, impurities
- compacts without gradients of density and stresses
- technological possibilities: dry powder compacting, slip- / gel casting, plasticize / granulation

- Important to manufacture bulk nanostructured ceramics
- Basic for R&D to manufacture fully dense nanostructured ceramics with improved properties
The final process:

3) Structure design of nanoceramic materials

(Example: Sintering technology of nano-AI₂O₃)

> Giant grain growth during sintering process <

sintered at 1500°C / 2 h – theoretical density: \( \rho = 3.99 \text{ g/cm}^3 \)

- without doping
- with doping

Grain growth for 500 times (40 nm to 20 µm)!
Grain growth for 100 times (40 nm to 4 µm)!

SEM of polished surface

\( \rho = 3.89 \text{ g/cm}^3 \)
\( \rho = 3.94 \text{ g/cm}^3 \)

Development of Dopands and Sintering technology / aim at: \( d_{50} < < 300 \text{ nm} \)
Topical: Optimizing of crystal structure in Alumina with nanosized metal-fluorides additives
(Results from R&D-Project in Hermsdorf, 2009)

SEM: sintered structure

Particle Size Distribution

\[ D_{50} = 0.35 \, \mu m \] / \[ \rho = 3.94 \, g/cm^3 \] (MgF$_2$-Sol-Additive)
Example: Transparent Ceramics for optical application

(Results from R&D-Project in Hermsdorf, 2009)

Results:

- transparent polycrystalline alumina ceramic
- high hardness HV(0,1) = 3000
- high bending strength ($\sigma_{bb}$) > 700 MPa
- transmission ($\lambda=800\text{nm}$) = 63%
  (Probe thickness 0,97 mm)

View through transparent alumina
(on left site / right site without plate)
Example: Transparent Ceramics for dental application

- Commercial available TZ-3Y-Powder
- Ultrafine milling and dispersion
- Optimized shaping and thermal processing

Homogeneous sub-\(\mu\)m-structure with \(d_{95} < 180\) nm
Material characteristics

4-Point Bending Strength  \( > 1000 \) MPa
Weibull-Modulus          \( > 10 \)
Fracture Toughness       \( > 8 \) MPam\(^{1/2}\)
Microhardness HV0.1     \( > 19 \) GPa

0.5 mm thick sample

![Graph showing total transmission and in-line transmission vs. wavelength (nm).](image)
**Summary**

- **Structure design**

**Main emphasis**

Mean crystalite size
Crystalite size distribution
Defect free sintered body: without pores, cracks, impurities
Homogeneous structure and minimizing grain growth
Homogeneous structure and high final density
Development of Dopands, Compaction and Sintering technology
Reduction of sintering temperature and grain growth / aim at: $d_{50} \ll 300$ nm

- Important to manufacture bulk nanostructured ceramics
- Basic for R&D to manufacture fully dense nanostructured ceramics with improved properties
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- Testing of nanopowders of suitability for ceramic materials
- Development / Production of Ceramic nanopowders
- Processing / Compacting technologies for Ceramic nanopowders
- Further develop of nano powder and nano material examination
- Definition of advanced attributes (direction for development)
- Choose for concrete demands / demonstrate applications
- Development of ceramic materials with nano bulk structure
- Search for project partners
- Draft of cooperative R&D projects
Applications

Nanostructured ceramic materials for

- Medical implants
- Surgical instruments
- Nanostructured optoceramics for diagnostics and therapeutic techniques (antibacteria photodynamic Therapie – aPDT)
- Sensors
- Cutting tools
- Bearing
- Wear parts
- etc.
Project plan – search for partners

„Nanostructured ceramic materials with improved properties“

■ **Object:**

■ Development of technologies for manufacturing oxide ceramics with nano- and submicron bulk structures and advanced functions
■ Development of ceramic materials with high density and defect free sintered body: without pores, cracks, impurities

■ **Advanced attributes:**

   Toughness, strength, hardness, wear, reliability, optical performance
„Nanostructured ceramic materials with improved properties”

Who will participate?
Producer of Nanopowders
Manufacturer of Ceramic materials
Plants for processing of Ceramic materials
User of Ceramic parts
User with new or special demands
Research institutes, Universities

Which are other points of interest for the project?
New applications for advanced nanostructured ceramic materials
New connections of R&D and industrial partners
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The Fraunhofer Institute IKTS in Hermsdorf / Germany can refer to a long history and big experiences in ceramic materials production and R&D.

The IKTS research at new ceramic materials with improved properties, with nano- and submicron bulk structures, based on nanopowders.

The development is directed at better strength, hardness, reliability, optical transmission, thermal and chemical resistance.

We are looking for project partners from institutes and from innovative companies for a collaborative R&D project to create new ceramic materials.
With ceramics to new horizon!
Большое Спасибо!

Thank you for your attention!

For more information: www.ikts.fraunhofer.de