Properties, Preparation and Requirements to Testing of Ceramic Materials

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Summary
The strength of ceramic materials is mainly defined by the defect or flaw size in the bulk as well as on the surface of the component. Defects can have various origins like raw materials, processing, moulding, sintering, and finishing. For a break through of ceramics as engineering materials the minimization of flaw size and concentration is necessary as well as the improvement of non-destructive test procedures. The test methods should be able to detect bulk and surface flaws, even in complex component shapes, to detect density fluctuations, especially in green bodies, and stresses in the final compound. They should be adaptable to industrial production processes.

Zusammenfassung

Introduction
Ceramic materials are characterized by their brittleness, potential for high strength and high temperature stability. These basic properties have led to immense efforts in research and development to introduce ceramics into modern technologies for industrial use. But despite these efforts, the break through of structural ceramics has not yet taken place. The basic obstacles are still the same for several thousand years: The ceramic materials do not have mechanisms to decrease stresses
by plastic deformation and therefore defects, at the top of which loads can create high stresses, act as fracture origins. This leads to the situation, that cracks start from defects, and under supercritical conditions catastrophic crack propagation takes place. To improve the fracture behavior (that means to improve fracture toughness, described by the $K_{IC}$ value) various strategies have been developed (fracture toughnesses of ceramics range from about 3 up to 10; steel: 50 - 100):

- to increase fracture toughness by strengthening concepts [1-3]: transformation toughening of ZrO$_2$; composites, e.g. ZrO$_2$/Al$_2$O$_3$

- reinforced ceramics (fibers, whiskers) [4]

- to avoid defects by improvement of processing and microstructure (e.g. better powders, agglomerate-free processing, clean room techniques).

All these efforts could not solve the problems until now and other questions like fabrication of large parts, high speed production technologies or finishing technologies are just at their beginning. Since defect-free fabrication is very difficult on a large scale production, one should, at least, be able to detect dangerous defects in ceramic components as early as possible during the production process with non-destructive techniques.

**Defect origins**

Defects in ceramic components can have various origins. Table 1 gives a survey over some important defect sources. The flow sheet on page 4 shows typical ceramic preparation and production procedures and the correlation to defect sources. It can be easily concluded that it is rather difficult to build up production technologies which can guarantee defect-free large scale fabrication of high performance ceramic components. Figures 1 and 2 demonstrate the formation of textures and density
Table 1. Origin of defects.

<table>
<thead>
<tr>
<th>type</th>
<th>source</th>
</tr>
</thead>
<tbody>
<tr>
<td>processing defects</td>
<td>impurities (dust, organics, grinding), inhomogeneities</td>
</tr>
<tr>
<td></td>
<td>mixing (multicomponent systems, separation, distribution of compounds, agglomerates)</td>
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<tr>
<td></td>
<td>texture (flow textures, layer formation by pressing)</td>
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<tr>
<td></td>
<td>density fluctuations</td>
</tr>
<tr>
<td></td>
<td>moulding (casting, pressing, injection moulding)</td>
</tr>
<tr>
<td>microstructural defects</td>
<td>pores, agglomerates, large crystal growth, microcracks</td>
</tr>
<tr>
<td>stresses</td>
<td>temperature differences during sintering, density fluctuations in the green body</td>
</tr>
</tbody>
</table>

![Density fluctuation](image1.png)

Figure 1. Density fluctuation from slip casting of complex shapes.

![Layer formation](image2.png)

Figure 2. Layer formation resulting from dry pressing.

fluctuations of dry pressing and slip casting processes.

Ceramic composites show special features with respect to defects. In this case artificial defects are created (microcracks, "inhomogeneities" in form of whiskers, fibers or particles,
Preparation of Ceramic Materials

melting
fiber drawing
powder technologies

grain size, distribution
agglomerates
impurities,
inhomogeneities
crystal
defects
surface defects

natural raw materials → processing
synthetic raw materials

powder processing
→ grinding
→ mixing
→ additives
→ composite-technologies

moldable materials
→ dry pressing
→ extrusion
→ injection molding

molding
→ casting
→ tape casting
→ electrophoretic deposition
→ hot pressing techniques
→ special techniques (liquid metal oxidation, coatings)

green body, preshape, endshape

sintering
→ pressureless
→ hot pressing
→ HIP, sintering and HIP
→ gas pressure sintering

end shaping

e.g. ZTA or PSZ ceramics) and are incorporated to increase fracture toughness. It is difficult to define critical defects in such systems. According to Rice [5], in BN/Al₂O₃ composites, fractography has shown, that fracture origins are only found
in $\text{Al}_2\text{O}_3$-rich areas which are inhomogeneity type defects. Defects in fiber reinforced ceramics can be based on the pore formation between the fiber and the matrix (figure 3).

![Fiber Diagram](image)

Figure 3. Defect formation during sintering in fiber reinforced ceramics.

In polycrystalline ceramic materials, the mechanical strength can be correlated clearly to the defect size. Figure 4 (after Petzow) [6], shows the correlation for several types of ceramics. The effect of flaw size was determined on $\text{Al}_2\text{O}_3$, moulded by electrophoretic deposition (plates 8 cm in diameter, 5 mm thickness, double ring bending test, figure 5).

![Strength vs Flaw Size Diagram](image)

Figure 4. Dependence of strength on critical flaw size.
Figure 5. Strength dependence on flaw size of $\text{Al}_2\text{O}_3$ (electrophoretic deposition)

Figure 6 shows a large flaw, reducing strength to about 300 MPa. One can conclude, that for high performance ceramics flaw sizes should clearly be as small as 10 $\mu$m or less.

Figure 6. Microstructure of an $\text{Al}_2\text{O}_3$ sintered body (electrophoretic deposition).

In figure 7 [7] the fracture origin of a high strength transformation toughened ZrO$_2$ ceramic is shown. The defect is due to a finishing process (grinding and polishing). This demonstrates, that surface flaws have to be controlled as well as bulk flaws. Critical flaw sizes can be a result of permanent stresses as of a fatigue process, too. Figure 8 shows this on
Figure 7. Surface flaw of a toughened $\text{ZrO}_2$ sintered body.

Figure 8. Fatigue of a natural sandstone due to cyclic loads. Acoustic sound emission versus mechanical strength.

A "natural" ceramic, a sandstone. Frost/thaw cycles are followed by acoustic emission (summarizing the energy). Bending experiments show that crack formation reaches a critical level only after ten to twenty cycles. Therefore, it is desirable to develop test procedures which can be able to control flaw
formation in ceramic components even after practical use in order to detect critical states.

Conclusions

For making better ceramics, two strategies have to be followed: Firstly, it is necessary to reduce flaw sources. One very important source seems to be processing and finishing. Therefore, the whole technology has to be improved: making better powders, improve powder processing, green body fabrication, sintering and finishing. Secondly, in order to control flaws, it is necessary to improve flaw detection procedures for bulk and surface, to detect density inhomogeneities especially in green bodies (even with complex shapes) and to detect stresses in the endshaped components. The flaw size to be detected should be less than 10 μm. The test procedures should be able to be adapted to high speed production lines. Both improvements are an indispensable prerequisite for the break through of ceramic compounds as engineering materials.

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References


