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

Die Festigkeit keramischer Werkstoffe hängt entscheidend von der Größe der Fehler ab. Fehler resultieren aus dem Rohstoff, dem Verarbeitungs- und Formgebungsverfahren, dem Sinterverlauf und der Endbearbeitung. Zur breiten Anwendung keramischer Werkstoffe im Maschinenbau sind Fehler an der Oberfläche und im Innern von Bauteilen sicher zu detektieren. Außerdem sollten prozeßbedingte Dichteunterschiede (besonders in Grünkörpern) sowie Eigenspannungen (auch in komplexen Bauteilen) sicher nachgewiesen werden können. Die Prüfmethoden sollten an industrielle Fertigungsprozesse anpaßbar sein.

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 strenghtening concepts [1-3]: transformation toughening of ZrO_2; composites, e.g. ZrO_2/Al_2O_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).

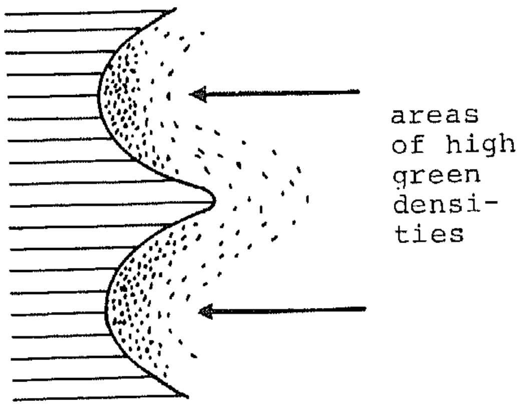
All these efforts could not solve the problems until now and other questions like fabrication of large parts, high speed production technologies or finishing technologies are just at their beginning. Since defect-free fabrication is very difficult on a large scale production, one should, at least, be 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.

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



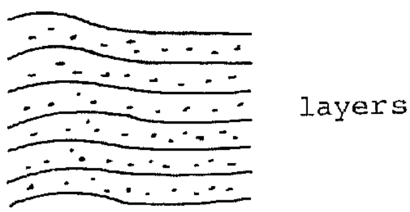
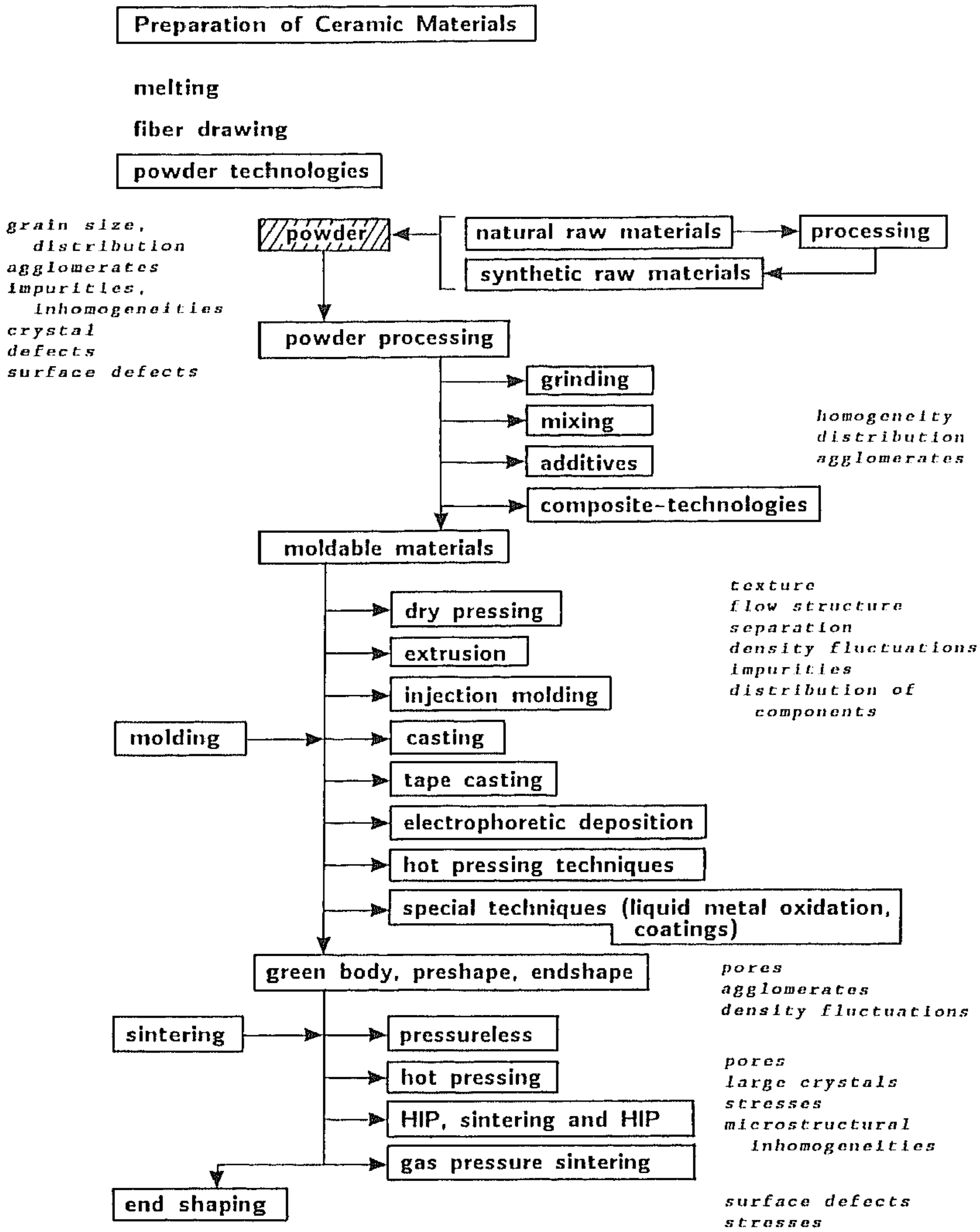


Figure 1. Density fluctuation from slip casting of complex shapes.

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,



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/Al_2O_3 composites, fractography has shown, that fracture origins are only found in Al_2O_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).

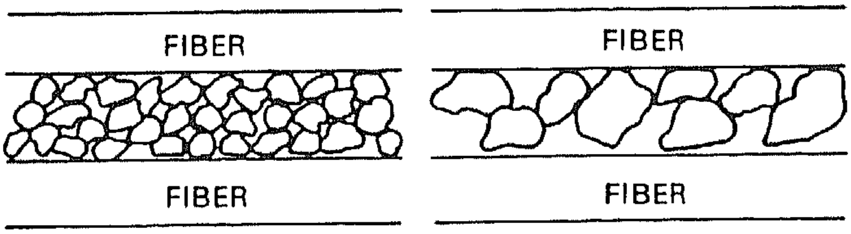


Figure 3. Defect formation during sintering in fiber reinforced ceramics.

In polycristalline 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 Al_2O_3, moulded by electrophoretic deposition (plates 8 cm in diameter, 5 mm thickness, double ring bending test, figure 5).

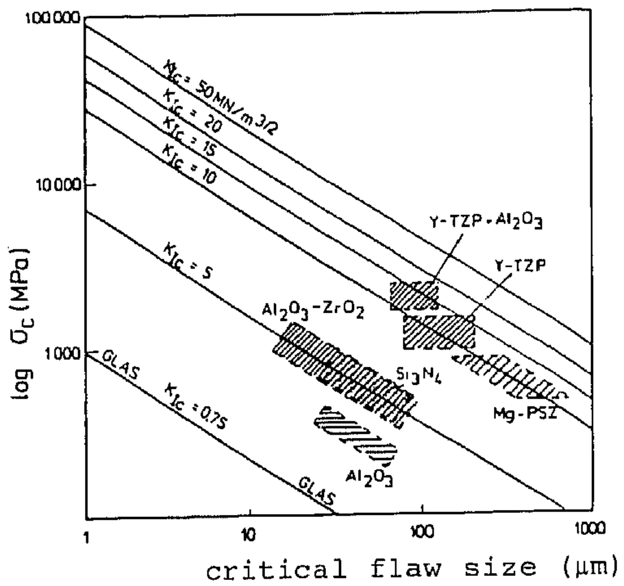


Figure 4. Dependence of strength on critical flaw size.

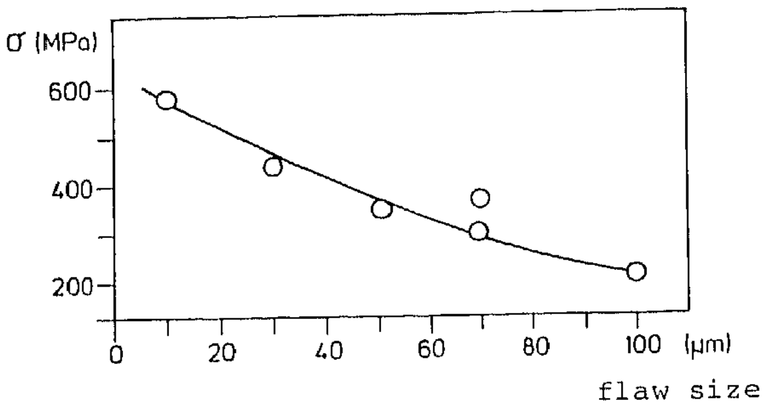


Figure 5. Strength dependence on flaw size of Al_2O_3 (electrophoretic deposition)

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 μm or less.

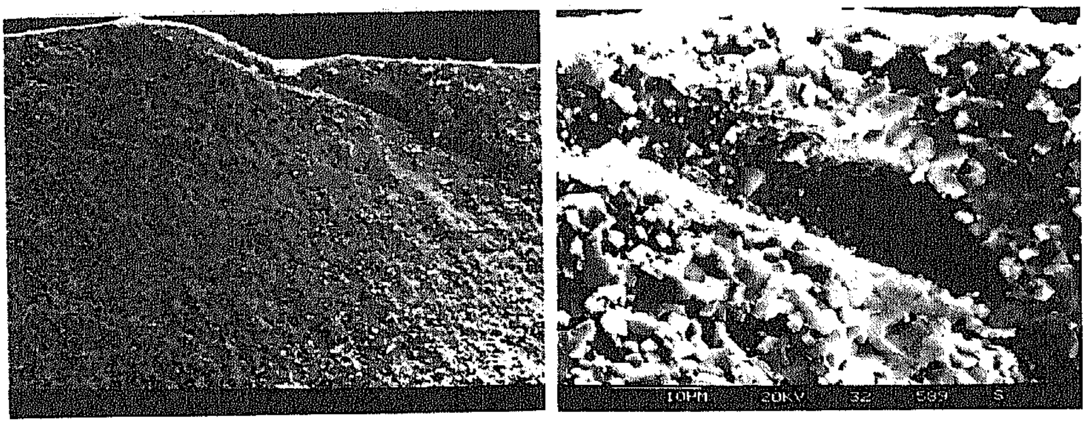


Figure 6. Microstructure of an Al_2O_3 sintered body (electrophoretic deposition).

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 fatique process, too. Figure 8 shows this on

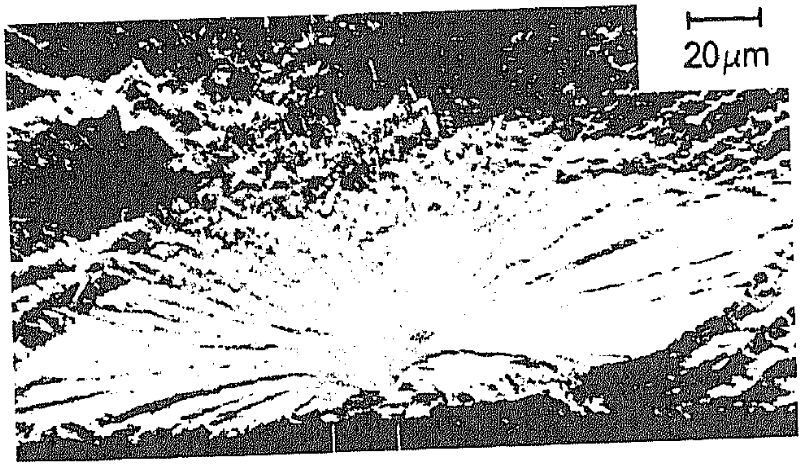


Figure 7. Surface flaw of a toughened ZrO_2 sintered body.

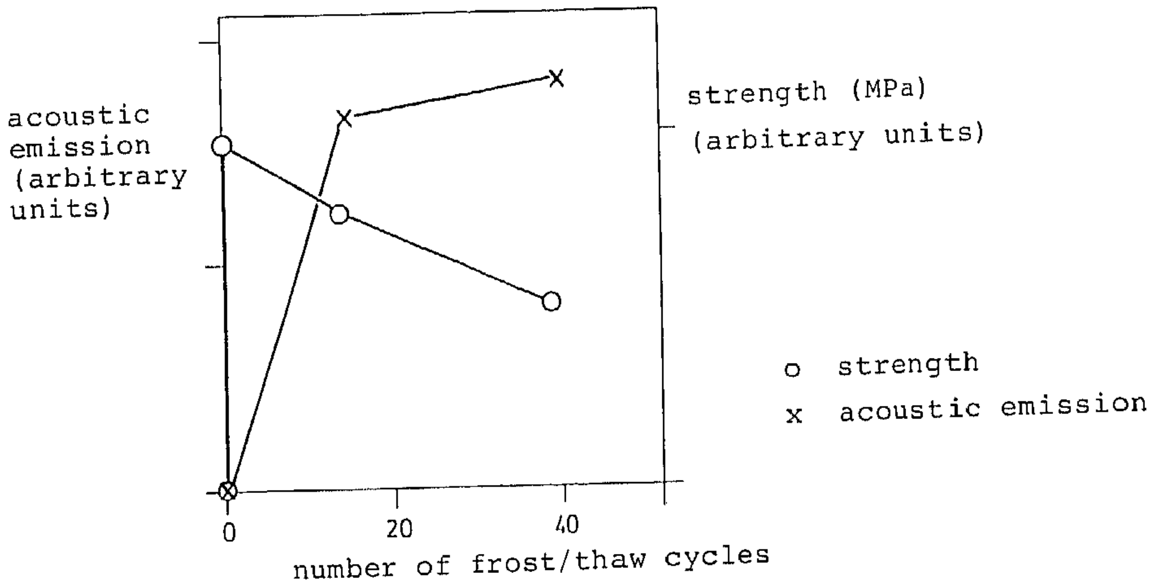


Figure 8. Fatique of a natural sandstone due to cyclic loads. Acoustic sound emission versus mechanical strength.

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 expecially 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 improvents are an indispensable prerequirement for the break through of ceramic compounds as engineering materials.

Acknowledgement

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References

[1] Garvie, R.C.; Hannink, R.H.J.; Pascoe, R.T.: Nature 258 (1975) 703.

[2] Garvie, R.C.: Structural Applications of ZrO_2-Bearing Materials, Advances in Ceramics 12. Am. Ceram. Soc., Columbus, 1984.

[3] Rühle, M.; Claussen, N.; Heuer, A.H.: J. Am. Ceram. Soc. 69 (1986) 195.

[4] Li, Z.; Bradt, R.C.: Materials Science Volumes 24 - 36 (1988) 511.

[5] Rice, R.W.: Cer. Eng. and Sci. Proc. 2 (1981) 493.

[6] Petzow, G.: Keram. Z. 40 (1988) 422.

[7] Rice, R.W.: Ceramic Fracture Features, Observations, Mechanisms and Uses, Fractography of Ceramic and Metal Failures (ASTM ST 827, 1984) 5.