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SINTERING AND MICROSTRUCTURE OF $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$ CERAMICS FROM BOHMITE AND ZIRCONIUM SALT

H. PASSING and P. REIJNEN

Institut für Gesteinshüttenkunde der RWTH Aachen, Mauerstrasse 5, D-5100 Aachen, F.R.G.

Abstract - The influence of powder preparation on density and microstructure was investigated. Starting powders were prepared by sol-gel process and freeze drying. To achieve high densities of compacts and homogeneous microstructures extensive milling of the powders is necessary.

I - INTRODUCTION

It has been shown that a stress-induced phase transformation can be used to increase fracture toughness of brittle materials based on $\text{ZrO}_2$ /1/ or containing $\text{ZrO}_2$ /2,3/. Retention of the tetragonal structure at room temperature or below is critically dependent on the size of the zirconia particles /4/. Maximal toughness in $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$ composites can be achieved by well-distributed small $\text{ZrO}_2$ particles in a dense fine-grained $\text{Al}_2\text{O}_3$ matrix. This aim can be reached by wet-chemical powder fabrication routes.

Powders prepared by wet-chemical methods are often highly agglomerated /5/, which strongly influences the microstructure of the powder compact and finally of the sintered body /6/. It has been found that failure of ceramics is often caused by cracklike voids, produced by differential sintering of agglomerates /7/. The scope of this work is to compare wet-chemical preparation methods for $\text{Al}_2\text{O}_3$ - 10W/o $\text{ZrO}_2$ composites in terms of agglomeration of the powders; density and microstructure of the sintered compact.

II - EXPERIMENTAL METHODS AND RESULTS

Two gel-derived powders (fig.1) were prepared by peptisizing böhmite with nitric acid, addition of a solution containing zirconiumdiacetate and small amounts of magnesium and yttrium, and gelling. Powder II was obtained by drying at 120°C, powder III by freeze-drying of the gel. This method had been used to produce highly sinterable zirconia powders /8/. Powder I was prepared by freeze-drying of a böhmite suspension containing the soluble salts (without peptisator). After preparation powder I shows the morphology of the frozen drop-
lets, powder II and III consist of gel pieces of different sizes (fig.2). All powders were ball-milled in a plastic jar with alumina balls and calcined at 850°C. After calcination all powders consist of transition $\text{Al}_2\text{O}_3$ and tetragonal $\text{ZrO}_2$. For desagglomeration wet milling in water was performed for different times. During milling some $\text{ZrO}_2$ transformed into monoclinic $\text{ZrO}_2$.

Bulk density of the gel derived powders decreases, whereas the bulk
density of powder I increases with milling time (fig.4). TEM investigations showed that powder I consists of large agglomerates that are rounded by milling so that packing is optimized. Fig.3 gives the changes of agglomerate size induced by milling of powder II. For all powders green density after compaction at 200 MPa increased with milling time (fig.4).

Powder compacts were sintered at different temperatures for 2 hours. The fraction of tetragonal zirconia in the sintered compacts was determined by X-ray diffraction of the surface /9/; it varied from 70 to 98%, depending on the sintering temperature and the compact density. Due to different amounts of tetragonal zirconia the theoretical density varies from 4.19 to 4.20 g/cm³.

As shown in fig.5 for compacts of powder I the density increased ra-

![Graph 1](image1)

powder I : ▲
powder II : x
powder III : ●

![Graph 2](image2)

green density [g/cm³]

0 0,2 0,4 0,6 0,8 1,0 1,2 1,4 1,6 1,8
milling time [h]

fig. 4

![Graph 3](image3)

sintering temperature

powder I : ▲
powder II : x
powder III : ●
monolithic gel ○

1450 °C

1550 °C

1650 °C

![Graph 4](image4)

density [g/cm³]

1,5 2,5 3,5 4,5
milling time [h]

fig. 5
pidly with milling time. The increase is lower for the gel derived powders because the densities of the untreated compacts are much higher. There is no significant difference between the densities of compacts prepared from powder II and powder III. After sufficient milling densities of 97% of the theoretical density could be achieved at 1550 and 1650°C. For all compacts densities are the same if the powders were milled for at least 24 h and sintered at 1650°C. For comparison the densities of monolithic gel pieces are shown in fig.5. Much higher densities can be achieved at low temperatures. The effect of milling on the microstructures of green and sintered compacts is given in fig.6 and 7. No significant differences could be detected between powder II and III. As shown by the micrographs

powder I
0 h milling time 48 h

compact sintered
at 1650°C
density: 30.6%
density: 33.2%

10 µm
density: 92.9%
density: 87.2%

fig. 6

powder II
0 h milling time 48 h

compact sintered
at 1650°C
density: 33.9%
density: 37.8%

10 µm
density: 94.8%
density: 92.9%

fig. 7
of the green compacts the untreated powders consist of large agglomerates that are not broken down by pressing. The agglomerates have a different content of zirconia, as can be seen by the variation of contrast. Extensive milling improved chemical homogeneity and reduced the agglomerate size so that packing is optimized. In the sintered compact of the untreated powder II dense regions are surrounded by cracklike voids. The porosity of the sintered compact of the untreated powder I is composed of small intra-agglomerate and large inter-agglomerate pores. The overall porosity of compact I sintered at 1550°C is much larger than that of the gel-derived powders. After 48 h of milling the microstructure of the sintered compacts are similar. The distribution of the zirconia particles is homogeneous and no large cracklike voids can be seen.

### III - DISCUSSION

Monolithic gel pieces or agglomerates in gel derived powders sinter to high densities at low temperature due to their homogeneous distribution of fine pores. As sintering proceeds single agglomerates reach theoretical density and separate from each other due to differential sintering. Large cracklike voids with a high coordination number are produced. Further shrinkage by elimination of pores is only possible if grain growth takes place thus reducing the pore coordination number /6/. Milling reduces the agglomerate size and thus smaller interagglomerate pores are produced with a lower coordination number which can be eliminated at lower temperatures. By freeze drying of a suspension a powder with agglomerates of a less homogeneous porosity is obtained. Internal sintering of the agglomerates is not as fast as for gel derived powders, which gives rise to a higher porosity but a smaller pore size distribution.

### IV - CONCLUSIONS

Gel-derived powders reveal an excellent internal sintering of agglomerates; yet the elimination of large interagglomerate voids needs extensive milling causing agglomerate size reduction and a more uniform microstructure.

### V - REFERENCES