Investigation of structures and properties of oxide-hydroxide systems after mechanical activation

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Abstract. It has been studied Al₂O₃ and ZrO₂ with Al(OH)₃ mixtures after mechanical activation in a planetary ball mill. Measurement of the specific surface by the BET method has shown that an increase of mechanical treatment time leads to an increase in the specific surface in all studied powders. In the initial states a large agglomerate of the order of 50-150 μm and isolated particles of 0.05-0.1 μm were observed. Mechanical treatment leads to fracture of agglomerates into separate particles. It has been revealed that the amount of agglomerates decreased with the increasing of the mechanical activation time, and the number of isolated particles increased. The average size of the agglomerates of Al-based powders varies in the range of 50-15 μm. In the case of powders based on ZrO₂, it varies in the range of 10-14 μm.

1. Introduction

Porous ceramic materials based on Al₂O₃ and ZrO₂ are widely used in various fields of technology, due to the unique combination of high strength, wear resistance, thermal conductivity and heat resistance [1]. A disadvantage of the technology for producing composite ceramics based on Al₂O₃ and ZrO₂ is the presence of large agglomerates in the powder, which leads not only to heterogeneity in the material structure but also to different compaction kinetics in microvolumes [2], which in turn leads to the formation of structural defects. In the literature [3,4] were shown that the mechanical processing of the powder makes it possible to achieve uniform distribution of particles in the powder, which in turn inevitably leads to improved compaction and uniformity in the volume of the ceramic material. One of the methods of mechanical processing of powders is the treatment of the powder in a planetary mill.

The aim of this work is to study the effect of mechanical treatment in a planetary mill on the structure and morphology of Al₂O₃, Al(OH)₃, ZrO₂ (MgO), ZrO₂ (Y₂O₃) powder mixtures.

2. Experiment

Al₂O₃, Al(OH)₃, ZrO₂(MgO), ZrO₂(Y₂O₃) powders were investigated. The powders were subjected to mechanical activation for 0-600 s in a planetary ball mill.

The average particle size of the Al(OH)₃ powder was 70μm, Al₂O₃ - 60μm, ZrO₂(MgO) and ZrO₂(Y₂O₃) - 5μm. Mechanical activation of the polymer binder powder was performed in a planetary ball mill AGO-2. Determination of the crystal structures of Al₂O₃, Al(OH)₃, ZrO₂(MgO), ZrO₂(Y₂O₃)
powders was carried out on a X-ray diffractometer with CuKα radiation. The determination of the CDD size was performed using the Hall-Williamson method. Morphological studies of the powders were performed using Tescan Vega-3 scanning electron microscope.

3. Results and Discussion

On figures 1 are shows the dependence of the average particle size after mechanical activation with different time. As one can see in all cases a decreasing of particle sizes are observed and with milling time increase leads to appearance of dependences saturation up to 2-4 μm of particle sizes.

![Figure 1. An average particle sizes with increasing of mechanical activation time](image)

On figure 2 are shown specific surface of powders with increasing of milling time. The dependences for ZrO₂ powders and aluminum oxide and hydroxide are differing each others. Specific surface area of ZrO₂(MgO) and ZrO₂(Y₂O₃) reaches maximum value at 100-180 s of mechanical activation while subsequent increase of activation time leads to a slow decrease of the specific surface area.

These behavior looks like very similar to theoretical one [5] and means that on first step agglomerates are destroyed and on the next step there are formed another aggregates.

Specific surface area of Al₂O₃ and Al(OH)₃ powder only increase with milling time activation, this is probably that we didn’t reach a maximum and necessary more time for destroy agglomerates in these powders.
Figure 2 - Specific surface changes with mechanical activation time for powders

Figure 3 - Graph of CDD versus mechanical activation time.

A calculations of CDD from X-ray data, fig. 3, are shown that of Al₂O₃, Al(OH)₃, ZrO₂(MgO) and ZrO₂(Y₂O₃) powders showed that these values are decreased with increasing mechanical activation time and reached sizes about 10-20 nm which coincide with [6,7].

After comparison of the data for the average particle size, specific surface area and CDD size obtained from XRD analysis one can conclude that all studied powders has a polycrystalline structure which, in turn, became smaller during mechanical treatment.

4. Conclusions

After mechanical activation of powders particle size distribution is uni-modal. With mechanical activation time increase the average size of agglomerates decreases and size of particles in these agglomerates decreases as well.

The average size of the agglomerates of alumina-based powders varies in the range of 15-50 μm. In the case of powders based on zirconia, it varies in the range of 10-15 μm.
It has been shown that the average particle size determined from the measurement of the specific surface showed that the average particle size decreases with increasing time of mechanical activation. XRD analysis showed that the CDD size of aluminum hydroxide powder during mechanical activation decreases with increasing of mechanical activation time and all studied powders has a polycrystalline structure.

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