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APPLICATION NOTE

:: APPLICATION NOTE SP003: Nano Technology

:: APPARATUS: **8000M Mixer/Mill®**

:: APPLICATION: Production of Nano Powders

Production of Nano-Crystalline Oxidized Ceramics with High-Energy Ball Milling

S. Indris, D. Bork, P. Heitjans, J. Mater. Synth. Process 8, 245 (2000)

With kind permission of Prof. P. Heitjans, Institute for Physical Chemistry and Electro-Chemistry, University of Hannover

Analyzing particle size effects in nanocrystalline materials requires a technique in which onecan adjust the particle size. In this study various nano-crystalline materials were produced usinga ball mill (8000M Mixer/Mill®, SPEX SamplePrep; equipped with alumina and zirconia vials).Ball milling is particularly suitable for this task as it is easy to use and allows the grinding of a relatively large amount of material as well as a large variety of different materials.

The analyzed media were: Li_2O, $LiNbO_3$, $LiBO_2$, B_2O_3, TiO_2 and $Li_2O:B_2O_3$ mixtures. The average particle size was determined by the grinding time and subsequently analyzed by means of X-ray Diffraction (XRD) and Transmission Electron Microscopy (TEM). The lithium containing materials were selected because of their potential use as solid electrolytes. TiO_2 is interesting with regard to its use as a photo catalyst. For hygroscopic materials the alumina grinding vial was filled in an argon atmosphere and put into an airtight stainless steel container.

Particle Sizes

The different oxides displayed different grinding characteristics, but a minimum particle size of approx. 20nm was obtained after 8 to 10 hours of grinding. Particle size was determined by XRD analysis and TEM data. The nano-crystalline samples are metastable and heating led to particle growth, which was shown by Differential Scanning Calorimetry. This was taken into account during the sintering process when a solid, compact ceramic was to be produced from the samples. Prior studies by other groups showed the particular suitability of two-step sintering with a lower temperature during the second step.

TiO_2, which was analyzed by two methods, showed partial phase transformation during the grinding process. When subjected to ball milling Rutile, comprising additional impurities, was obtained as pure Rutile (without impurities) in smaller particle sizes. Anatas, which showed the same impurities, exhibited an elevated proportion of these impurities after grinding. Therefore TiO_2 has only limited applications in high-energy ball milling experiments.

Chemical Reactions

Mixing of ceramic components and subsequent pressing produces a heterogeneous material with a multitude of different boundary layers. This network of different interfaces can be modified by varying the particle size. During analysis of a 50:50 mix of $Li_2O:B_2O_3$ chemical changes due to this chemical-mechanical procedure were detected. After a short period of time only the lines of the original compounds are detected with XRD analysis, while new lines appear after 4 hours. The newly formed product was $Li_2B_4O_7$. This indicates that the end product of the reaction does not depend on the composition of the mixture but the conditions on the boundary layers.

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Conclusion

High-energy ball milling is especially suitable for particle size reduction and the study of subsequent chemical and physical changes. The characteristics of particle size reduction and subsequent growth was similar with all analyzed oxides. Some materials showed phase transformations while others showed chemical reactions, which did not occur with the microcrystalline starting material.

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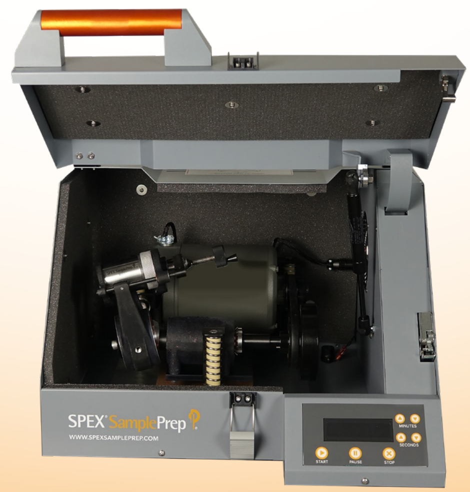

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