Influence of Grinding and Atmosphere on the Crystallization of ZrO₂ gel

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The influence of the grinding and the surrounding atmosphere on the thermal decomposition of zirconia gel has been studied. The XRPD analysis of the products obtained by thermal decomposition of zirconia gel at 500°C has shown that pure tetragonal phase is obtained if the gel decomposition is carried out under high vacuum or dry inert atmosphere. However, monoclinic zirconia results from the decomposition of the zirconia gel either under air or inert gas saturated with water vapor. A mechanism for the thermal decomposition of zirconia gel has been proposed from the study of the variation of the crystal size of the monoclinic and tetragonal zirconia phases formed as a function of the temperature and the surrounding atmosphere.

The thermal decomposition of the zirconia gel previously ground during two hours in a centrifugal mill leads to the formation of ZrO_2 with a percentage of tetragonal phase higher than 80% irrespective of the surrounding atmosphere. The stabilization of the tetragonal phase by grinding seems to be connected with the formation of tiny cubic or tetragonal zirconia crystals that cannot be observed by XRPD. The results obtained by DSC supports this conclusion. Thus, the crystallization enthalpy determined for the as prepared gel was equal to $-128\pm$ 7 J/g, in good agreement with literature, while the crystallization enthalpy for the ground gel amounted only -12 ± 2 J/g. **Keywords: zirconia gel, crystallization mechanism, crystal size**