High density nanocrystalline anatase TiO_2 ceramics. Part I: preparation by hot pressing

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Nanocrystalline anatase TiO_2 ceramics, with a mean grain size well below 100 nm, present remarkable electrical properties, which are not completely understood [1]. During classical sintering, the solid state diffusion process is activated by application of elevated temperatures. However, the rate of grain growth becomes comparable to the rate of sintering during the last stage of densification [2]. Furthermore, in the case of TiO_2, the rutile structure is thermodynamically favoured in comparison to the anatase structure, even if a stabilisation of the anatase phase by surface energy has been suggested [3]. Therefore, a thermal treatment at too elevated temperature can provoke the phase transition. In order to work at moderate temperature (600°C), preserving the nanometric grain size and the anatase phase, we used the hot pressing technique with application of a static uniaxial high pressure (up to 0.8 GPa) during sintering.

In this contribution, the optimisation of experimental parameters, P, T and time to obtain dense nanocrystalline (≈ 30 nm) TiO_2 anatase ceramics is presented. The pellets were characterized by X-ray diffraction, Scanning Electron Microscopy and mercury porosimetry. The size effect of TiO_2 precursor powders (between 15 and 80 nm average particle size) on the densification rate was investigated by dilatometry (see figure 1). The results are analysed in perspective of densification mechanisms, involving surface and grain boundary processes, being both proportional to the reciprocal grain size. Also, the role of alio- and isovalent doping cations (Zn^{2+}, Al^{3+}, Si^{4+}, Nb^{5+}) on the sintering process is analysed in view of point defect chemistry and solute segregation [4].

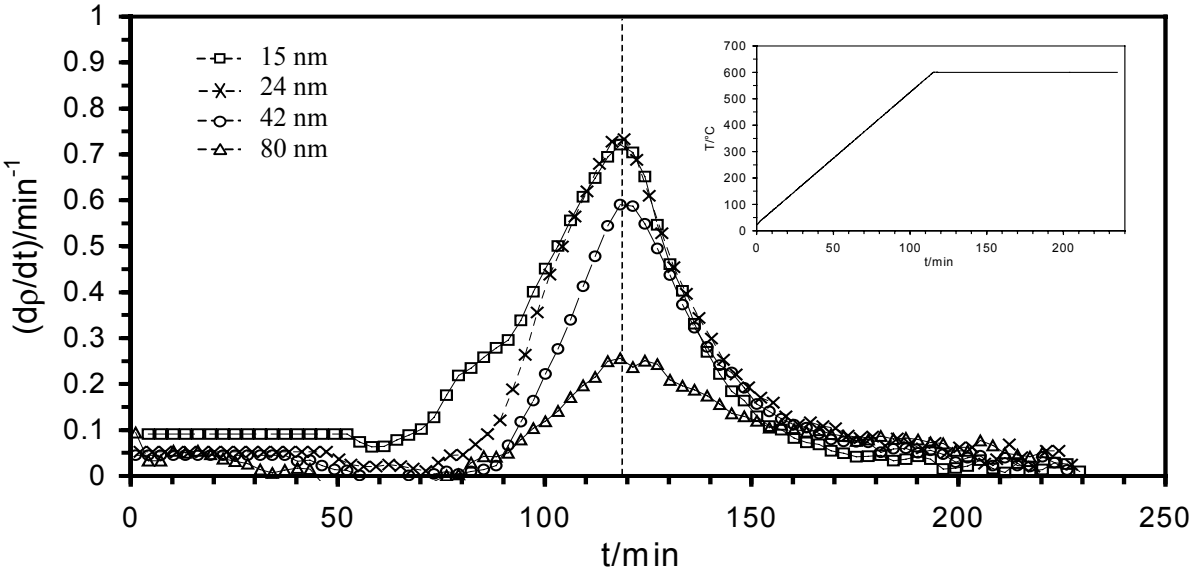

Figure 1 : Densification rate of nanocrystalline anatase ceramics as function of precursor grain size. The insert shows the temperature program during hot pressing (P = 0.6 GPa).

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[2] I.W. Chen, X.H. Wang, Nature, **404**, 168 (2000).
[3] A. Navrotsky, PNAS, **99**, 6476 (2002).
[4] R. Bouchet, A. Weibel, P. Knauth, G. Mountjoy, A. Chadwick, Chem. Mater., in print (2003).