SAXS/WAXD on thermally annealed nanostructured CVD-obtained TiO_2 films

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The films containing nanosized grains of titanium dioxide (TiO_2) are widely used in research of mainly optical, photovoltaic, photochromic and electrochromic devices. Besides by the size and structure of grains, their specific applications are also determined by their porosity. In most cases there is a desirable degree of porosity which leaves the outer and the inner surface of the film large enough. These morphological characteristics of TiO_2 films depend on the method of preparation but also on the subsequent processing of the material. The TiO_2 films analysed in this paper were prepared on glass substrates by chemical vapour deposition (CVD). The samples were annealed at higher temperatures in different atmospheres (O_2,H_2). We investigated the morphology of TiO_2 films using the small angle X-ray scattering (SAXS) combined with the wide angle X-ray diffraction (WAXD). The main reason for our choice of this combination of methods is that the distribution of scattered intensity over small and wide angle range allows us to estimate two relevant morphological parameters. One of them, derived from SAXS data, gives us information about the relative internal surface of grains and pores. The other, obtained by WAXD allows the estimation of the size of grains. By analysing the series of samples annealed at different temperatures, the changes in morphology can be detected and followed on the basis of dependence of parameters´ values on the annealing temperature. In previous investigations on CVD derived TiO_2 films by Raman spectroscopy the amorphous phase was recorded before anatase to rutile phase transition. In literature there are also some assumptions about the existence of an interphase during the phase transformations in nanostructured materials. We intended to check whether the behaviour of the defined morphological parameters could reveal such eventual morphological changes.

One more reason for the use of the SAXS/WAXD method is the possibility to avoid the destruction of the sample, i.e. it is not necessary to remove the films off the substrates, so samples remain in the form in which they are often used in functional applications of this material. As glass substrates strongly absorb the X-ray radiation, the standard transmission geometry mode of measurement was replaced by the grazing incidence mode. This technique of measurement and the scattered intensity big enough in sufficiently large angle range were obtained by using highly collimated X-ray radiation of strong intensity of the synchrotron source.