Amorphous titania as a precursor to brookite-based materials obtained by hydrothermal treatment

Supplementary information

Revenko A.O.[1], Kozlov D.A.[2], Kolesnik I.V.[1], Poluboyarinov A.S.[1], Kottsov S.Yu.[2], Garshev A.V.[1,3]

[1] Faculty of Materials Science, Lomonosov Moscow State University, Moscow 119991, Russia

[2] Kurnakov Institute of General and Inorganic Chemistry of RAS, Moscow 119991, Russia

[3] Faculty of Materials Science, Shenzhen MSU-BIT University, Shenzhen 518172, China

XRD analysis of the samples

XRD patterns were registered using the external standard, which were corundum in the experiment with slightly larger than brookite crystallite sizes (about 200-500 nm). The identification of the phase composition was performed using the ICDD PDF-2 database. Full profile le Bail and Reitveld methods analysis were used to determine the integral intensities. As an example, in Fig. S1, XRD pattern of TiO_2 sample containing both brookite, anatase and amorphous phases obtained using corundum is shown. All XRD patterns analyzed in the paper had a goodness of fit (GOF) of no more than 2.0, with profile and weighted R-factors less than 7.0 and 8.0, respectively. XRD profiles were fitted using JANA2006 software.

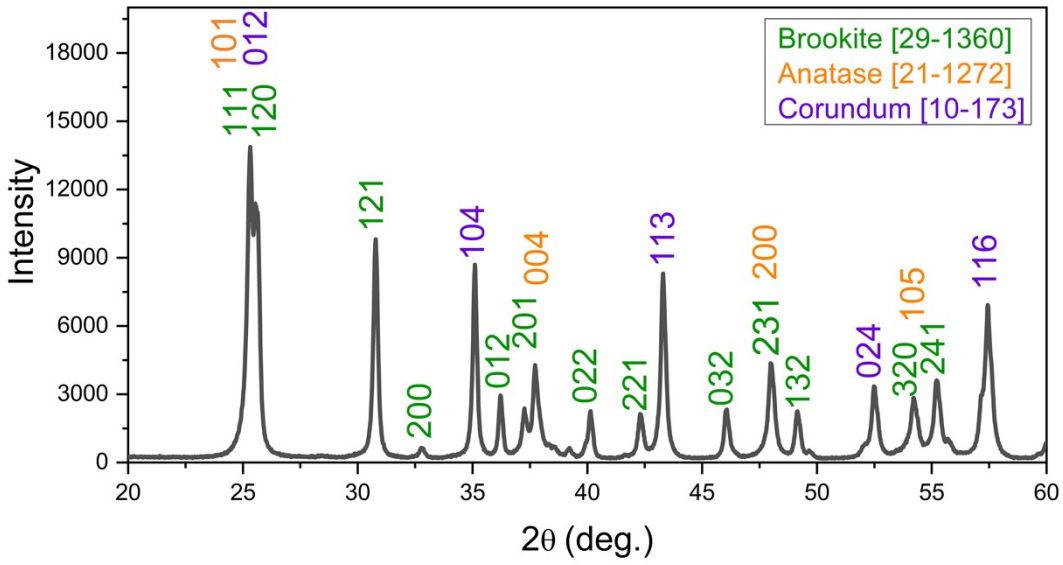

Fig. S1. A typical indexed XRD pattern of the obtained samples

Corundum was used for the quantitative determination of the phase composition of all the samples (including the content of the amorphous phase) as an external standard. The fraction of crystalline phases was determined using the integral intensities of the reflections $I(A_i)$ and Reference Intensity Ratios RIR_i (1). The fraction of the external crystalline standard measured by XRD differed from the real one due to the presence of amorphous phase in the sample ((2) and (3)).The fraction of the amorphous phase was calculated by the following equation (4).

$$\omega_{XRD}(A_i) = \frac{\frac{\overline{I(A_i)}}{RIR_i}}{\sum \frac{\overline{I(A_i)}}{RIR_i}} \quad (1)$$

$$\omega_{real}(Al_2O_3) = \frac{m(Al_2O_3)}{m(Al_2O_3) + m(Sample)} \quad (2)$$

$$\omega_{XRD}(Al_2O_3) = \frac{m(Al_2O_3)}{m(Al_2O_3) + \sum m(Crystalline\ phases)} \quad (3)$$

$$\omega_{amorphous\ phase}(Al_2O_3) = \frac{\frac{m(Al_2O_3)}{\omega_{XRD}(Al_2O_3)} - m(Sample) - m(Al_2O_3)}{m(Al_2O_3) + m(Sample)} \quad (4)$$

Particles' size was estimated using the Scherrer formula with anisotropy taken into account:

$$p_{\perp} = \frac{180K\lambda}{\pi X} \qquad p_{||} = \frac{180K\lambda}{\pi(X + X_a)},$$

where K is a parameter that depends on the shape of the particles (assumed to be 0.9), λ is the X-ray wavelength; X, X_a are refining parameters for the profile peaks (in the case of isotropic particles, X_a was assumed to be 0).

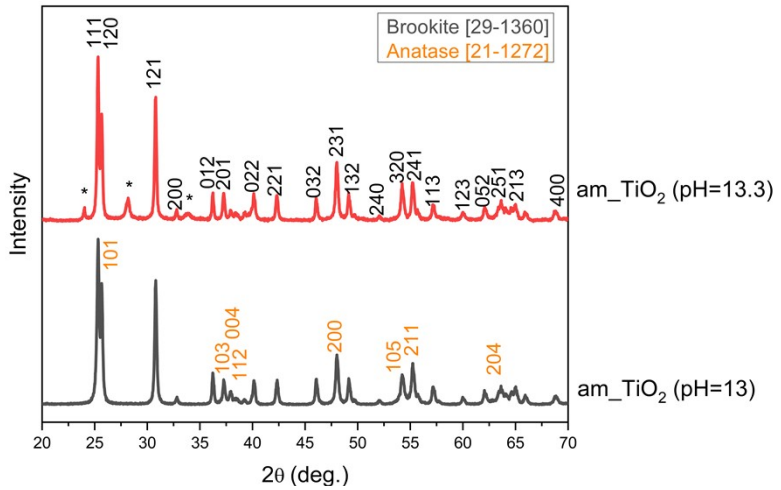

Fig. S2. XRD patterns of the samples obtained by the non-equilibrium method at high pH values

Sample	Brookite (001), nm	Brookite (≠ 001), nm	Anatase, nm
Spheres(Hla_1:1)	27	9	18.
Spheres(Hla_5:1)	24	10	18

Table S1. Sizes of particles in the samples obtained by the lactate method using titania microspheres derivied from the XRD patterns

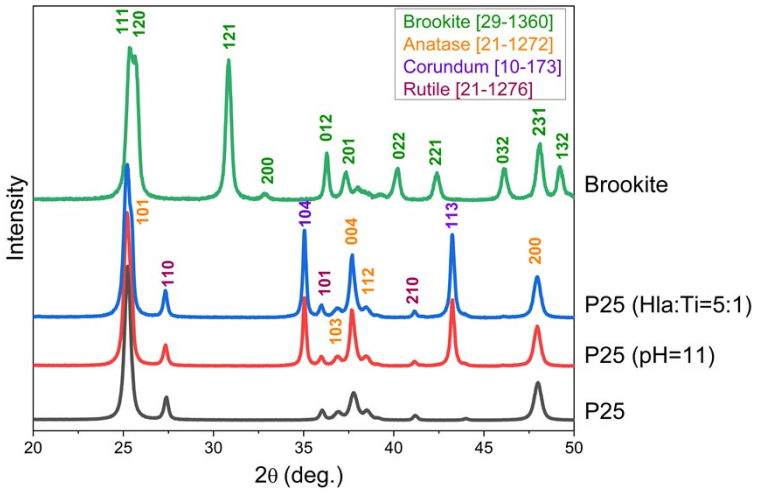

Fig. S3. XRD patterns of the samples obtained by hydrothermal treatment of crystal Aeroxide P25. The samples obtained by sodium hydroxide and lactate-involved methods were registered with the external standard Al_2O_3

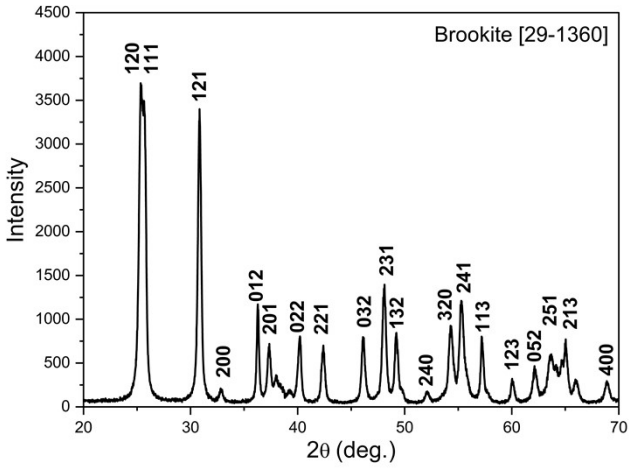

Fig. S4. XRD patterns of the obtained single-phase brookite

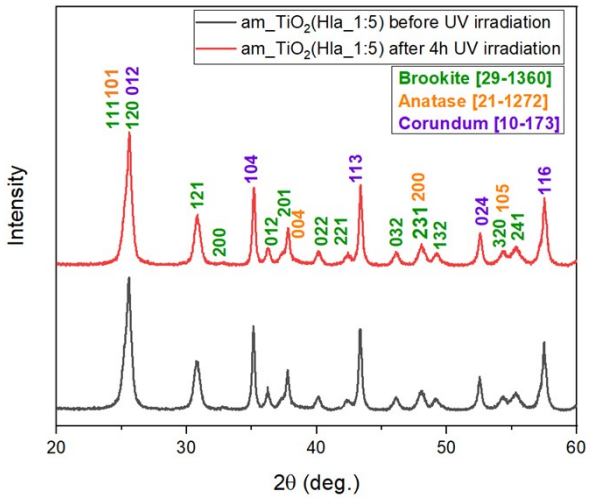

Fig. S5. XRD patterns of am_TiO_2(Hla_1:5) sample before and after UV irradiation.