

Supplementary Materials

Facile hydrothermal synthesis of BaTiO₃ nanoparticles studied by *in situ* X-ray diffraction

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Selected temperature profiles

Temperature profiles during heating of the capillary were measured with a thermocouple placed inside the sapphire capillary, while the capillary was filled with water and pressurized to 100 bar. Temperature profiles were measured in 50 °C intervals from room temperature (RT) to 400 °C (referring to the set-point temperature of the heat blower). The measured values were then used to calibrate the set-point temperature of the heat blower with the actual temperature inside the capillary.

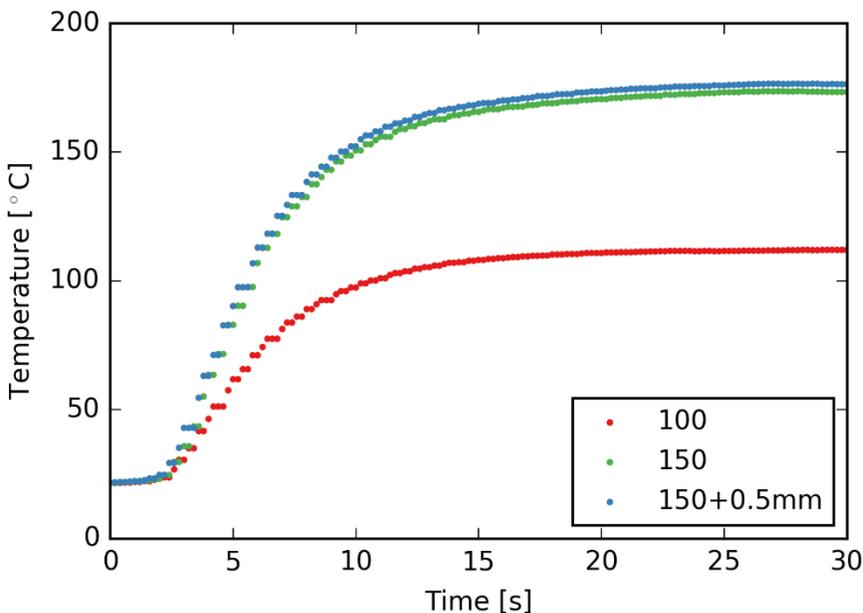


Figure S1: Heating profiles for two different set-point temperatures (red (100 °C) and green (150 °C)). Blue data corresponds to 150 °C, but with the capillary moved 0.5 mm closer to the heat blower compared to the green data showing the robustness of this setup.

Typical Rietveld refinements

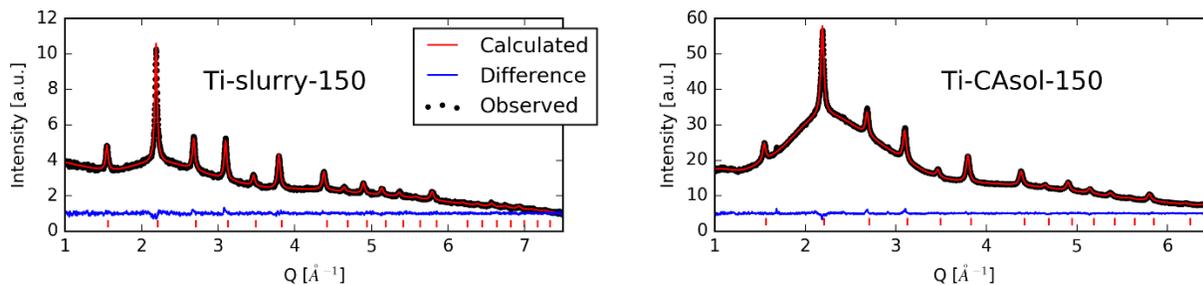


Figure S2: Last frames of experiments Ti-CASol-150 and Ti-slurry-150 showing typical refinements, with red markers showing diffraction lines of BaTiO₃ at RT from ICDD card #01-074-4539. Refined

values (R-values, lattice parameter, size, strain and atomic displacement parameters) are reported in Table S1.

Time resolved atomic displacement parameters for Ti and Ba for all experiments

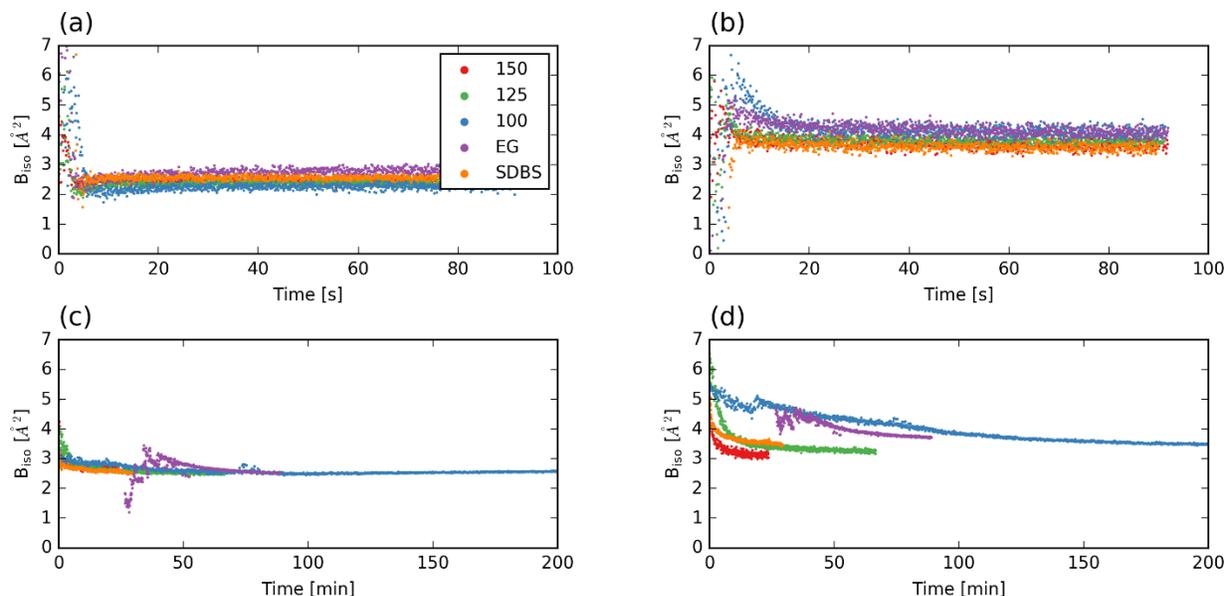


Figure S3: Refined atomic displacement parameters for (a-b) Ti-slurry, and (c-d) Ti-CAsol experiments. (a) refined B_{iso} values for Ba; (b) refined B_{iso} values for Ti; (c) refined B_{iso} values for Ba; (d) refined B_{iso} values for Ti. Color-coding is as described in (a) for all panels.

Fits to the Johnson-Mehl-Avrami equation to the normalized scale factors

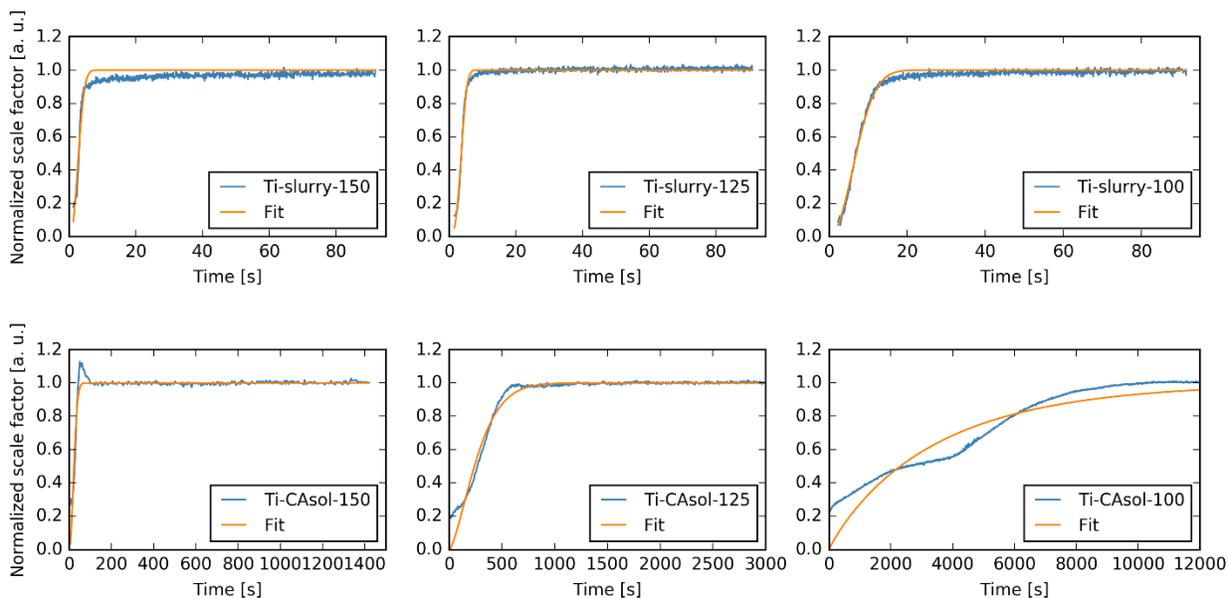


Figure S4: Normalized scale factor and fit to the Johnson-Mehl-Avrami equation for all samples used for the kinetic modeling.

Summary of the final refined values for all experiments

Isotropic Lorentzian strain and size parameter, lattice parameter, and B_{iso} for Ti and Ba for BaTiO_3 were refined. Scale factor was refined for BaCO_3 (for Ti-CAsol-100 and Ti-CAsol_EG) for the batch refinement, keeping size and lattice parameters fixed to the values refined in the last frame (frame with the most BaCO_3).

Table S1: Refined values (size, strain, lattice parameter, atomic displacement parameters for Ti and Ba and R-values) for the last frame from each experiment. Refinements were done using space group no. 221, Pm-3m, for BaTiO_3 .

Sample	Crystallite size [nm]	Strain [%]	Lattice parameter [Å]	Ti B_{iso} [Å ²]	Ba B_{iso} [Å ²]	R_{wp} [a.u.]	R_{bragg} [a.u.]	Amount BaCO_3 [wt %]
Ti-slurry-100	14.3(4)	0.127(7)	4.0612(3)	3.99(9)	2.39(6)	2.14	0.99	0
Ti-slurry-125	15.0(4)	0.125(6)	4.0603(3)	3.78(8)	2.31(5)	1.89	1.02	0
Ti-slurry-150	15.6(2)	0.094(3)	4.0587(2)	3.73(5)	2.49(3)	1.01	0.96	0
Ti-slurry-EG	8.7(2)	0.076(8)	4.0632(4)	4.23(8)	2.82(6)	1.99	0.76	0
Ti-slurry-SDBS	25.0(7)	0.060(4)	4.0531(2)	3.67(7)	2.59(4)	1.99	1.27	0
Ti-CAsol-100	13.9(3)	0.086(5)	4.0540(2)	3.48(6)	2.56(4)	1.80	1.13	12
Ti-CAsol-125	12.6(1)	0.067(3)	4.0505(1)	3.29(4)	2.54(3)	1.20	0.97	0
Ti-CAsol-150	12.5(2)	0.063(5)	4.0507(2)	3.04(6)	2.72(4)	0.90	0.67	0
Ti-CAsol-EG	13.4(2)	0.084(4)	4.0504(2)	3.68(5)	2.53(3)	1.30	0.93	5
Ti-CAsol_SDBS	10.7(1)	0.076(4)	4.0530(2)	3.48(5)	2.51(3)	1.30	0.70	0