LOCAL STRUCTURE AND KINETICS OF DEFECT ACCUMULATION IN TITANIA FLASH EVENTS

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Understanding and utilizing flash sintering as a processing technique requires disentangling the short-lived thermal effects that occur as a result of Joule heating from the more sluggish changes in the microstructure, such as defect formation, which can change over longer time scales and persist in the finished material. Adding to the challenge is the fact that typical in-situ analysis techniques, such as those built upon Bragg diffraction, are poorly suited to examine changes aside from lattice parameters since the coarsening behavior makes the time evolution of peak intensities highly variable. We find that local structure analysis via Fourier transformation of high-*Q* data to the pair distribution function leads to increased stability in the models that can be refined to X-ray scattering data. Careful examination of the lattice expansion, atomic displacement parameters, and site occupancies reveal behavior in TiO_2 that is invisible to Bragg-only analysis. Most importantly, the defect behavior is seen to be markedly different in TiO_2 than in zirconia, with quantitative signatures of defect-related phenomena that persist over long time scales. These results indicate that the integration over large Q-ranges makes PDF measurements quite robust in the face of grain reorientation during flash events, and a suitable setup at the XPD beamline at NSLS-II is in place. We will discuss how careful analysis of on-off experiments helps to disentangle defect signatures, which can then be integrated into models that explain the power versus time evolution in flashed samples.

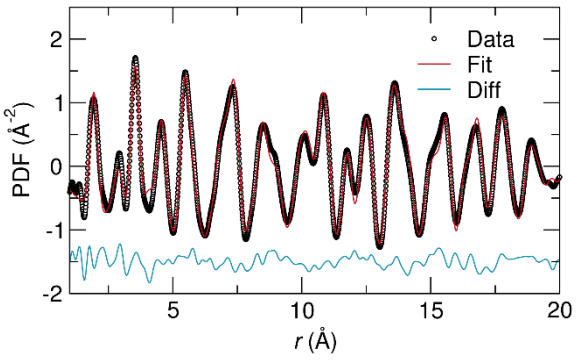

Figure 1. Representative fit to the X-ray pair distribution function of TiO_2 in an in-situ flash setup with Q_{max} = 20 Å^{-1}.

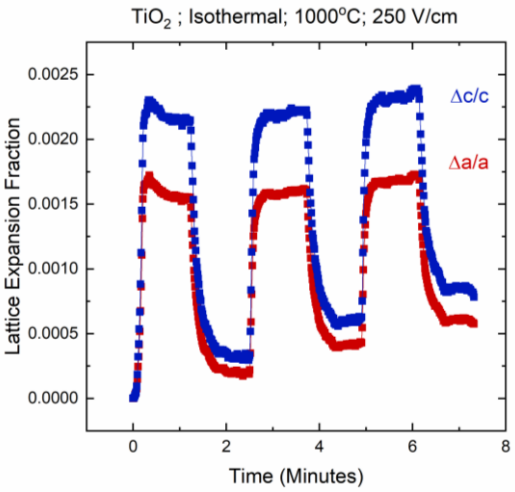

Figure 2. Changes in lattice parameters of titania over three cycles of an on-off experiment. Anisotropic expansion is seen during the on cycle as well as during the off cycle.