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Supporting information for article:

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Reduced Ilmenite**

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Experimental

Laboratory Pair Distribution Function Analysis

Samples were loaded into quartz capillaries (0.5 mm, Charles Supper Company) and data collected using a Bruker D8 Discover, which incorporated a Ag X-ray tube operated at 50 kV and 30 mA. The data were collected from 5-140° 2 θ while rotating the capillary at 15 rpm using a step size of 0.05° 2 θ . A variable counting time (VCT) method was employed to compensate for intensity loss at higher angles due to Lorentz-polarisation, the form factor and thermal vibration (Madsen & Hill, 1992, 1994). The incident beam was defined using a 0.6 mm slit, and both incident and scattered beam divergence was controlled using 2.5° axial soller slits. Data were also collected for an empty quartz capillary under identical conditions, to enable background removal from the data collected for the RI samples. Data collected for silicon (NIST 640C line position standard) were used to establish to the instrument parameters ($Q_{\text{damp}}=0.0198$ and $Q_{\text{broad}}=0.0309$), which were subsequently applied the sample refinements. The PDFs were generated with a $Q_{\text{max}}=19 \text{ \AA}^{-1}$ and refined using the same method as for the synchrotron collected data.

Supporting Figures:

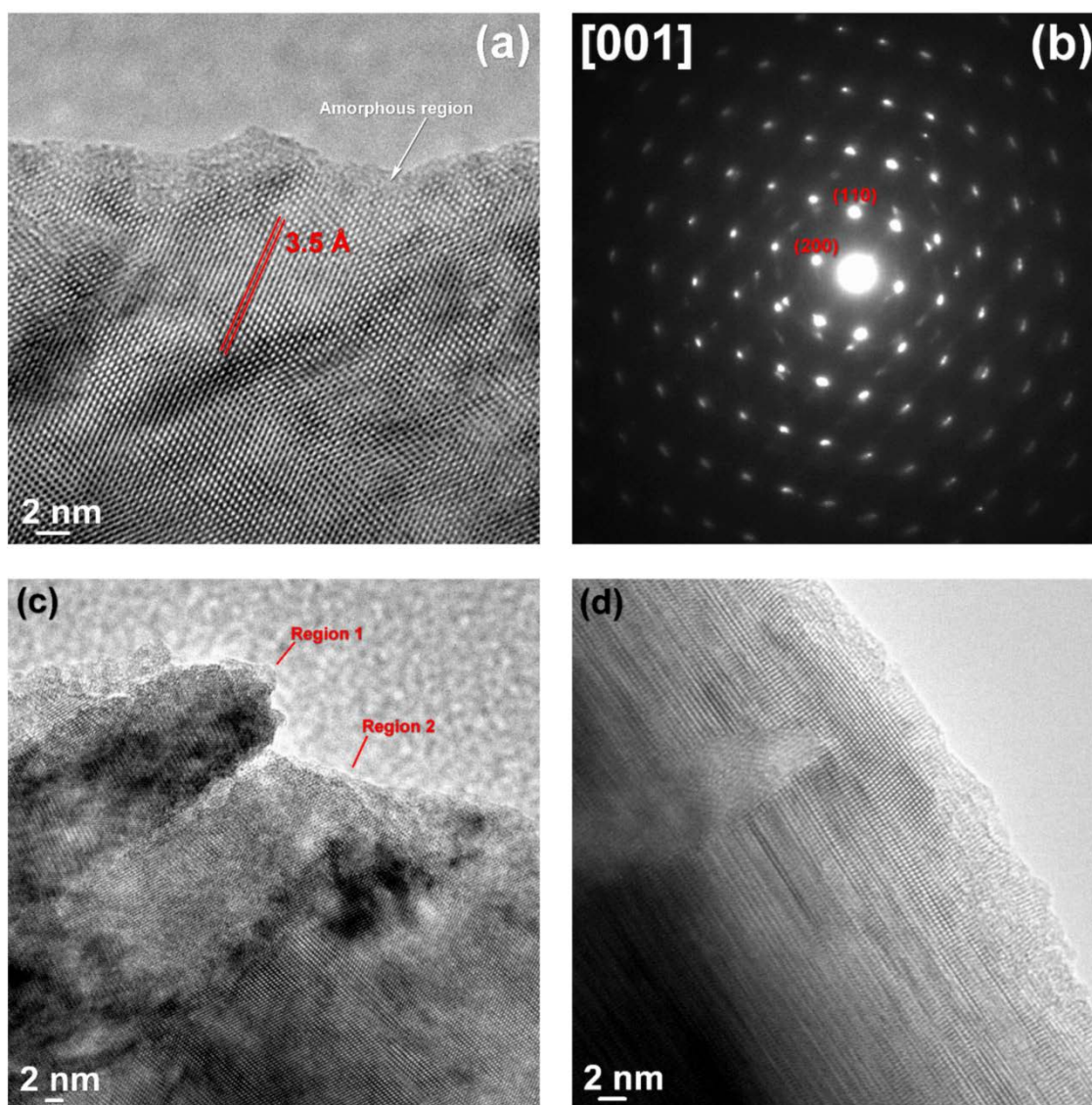


Figure S1 (a) High resolution TEM image of a rapid-cooled RI particle showing a region with minimal defects, (b) electron diffraction pattern collected from the particle in (a), (c) third particle consisting of 2 particles aggregated together, (d) image of fourth particle.

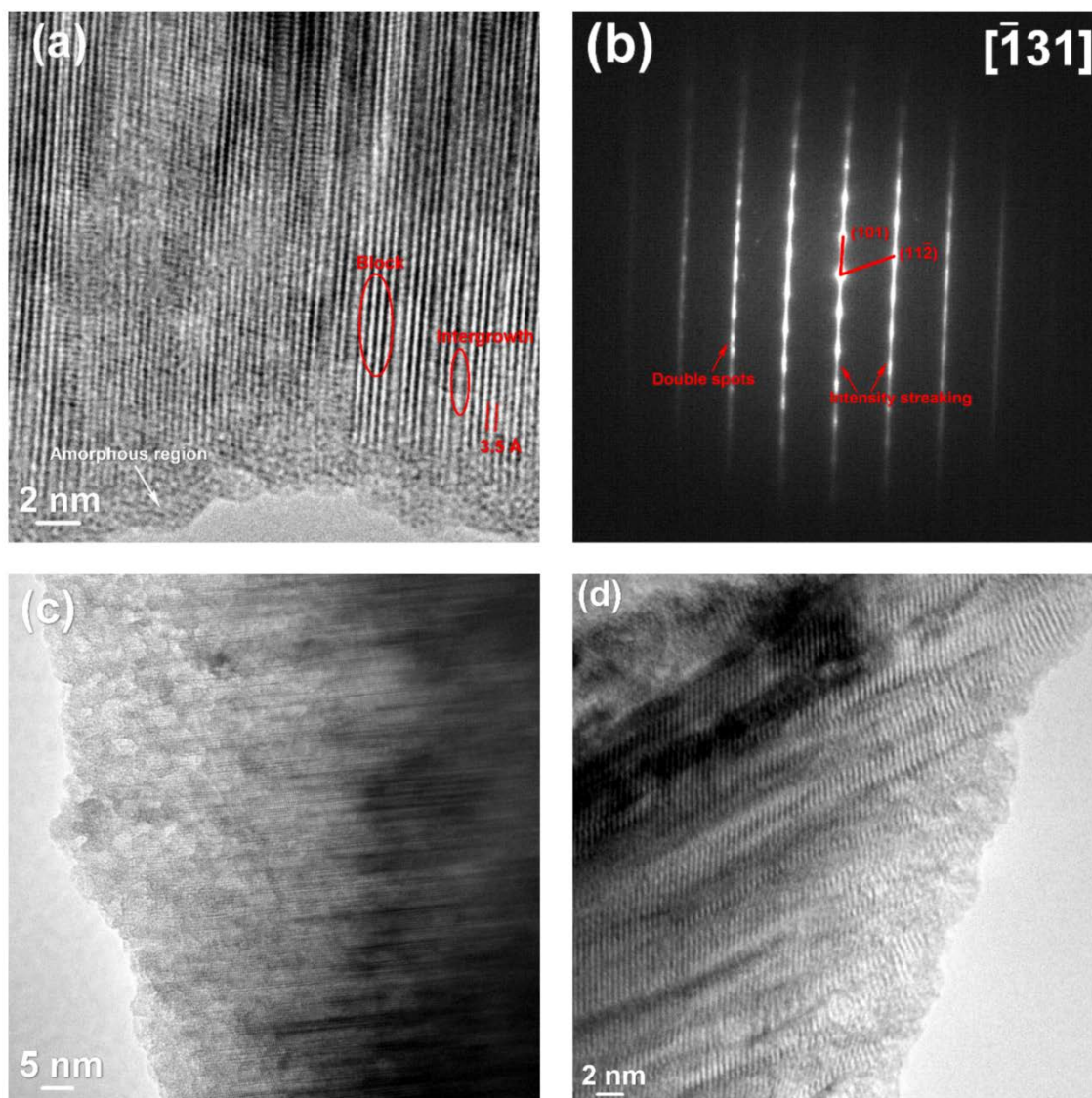


Figure S2 (a) High resolution TEM image of a slow-cooled RI particle, showing blocks of crystal planes (with spacing of 3.5 Å) and intergrowths, (b) the corresponding diffraction pattern, down the $[\bar{1}31]$ zone axis, collected from the interior of the particle, (c) image of a third and a (d) fourth particle.

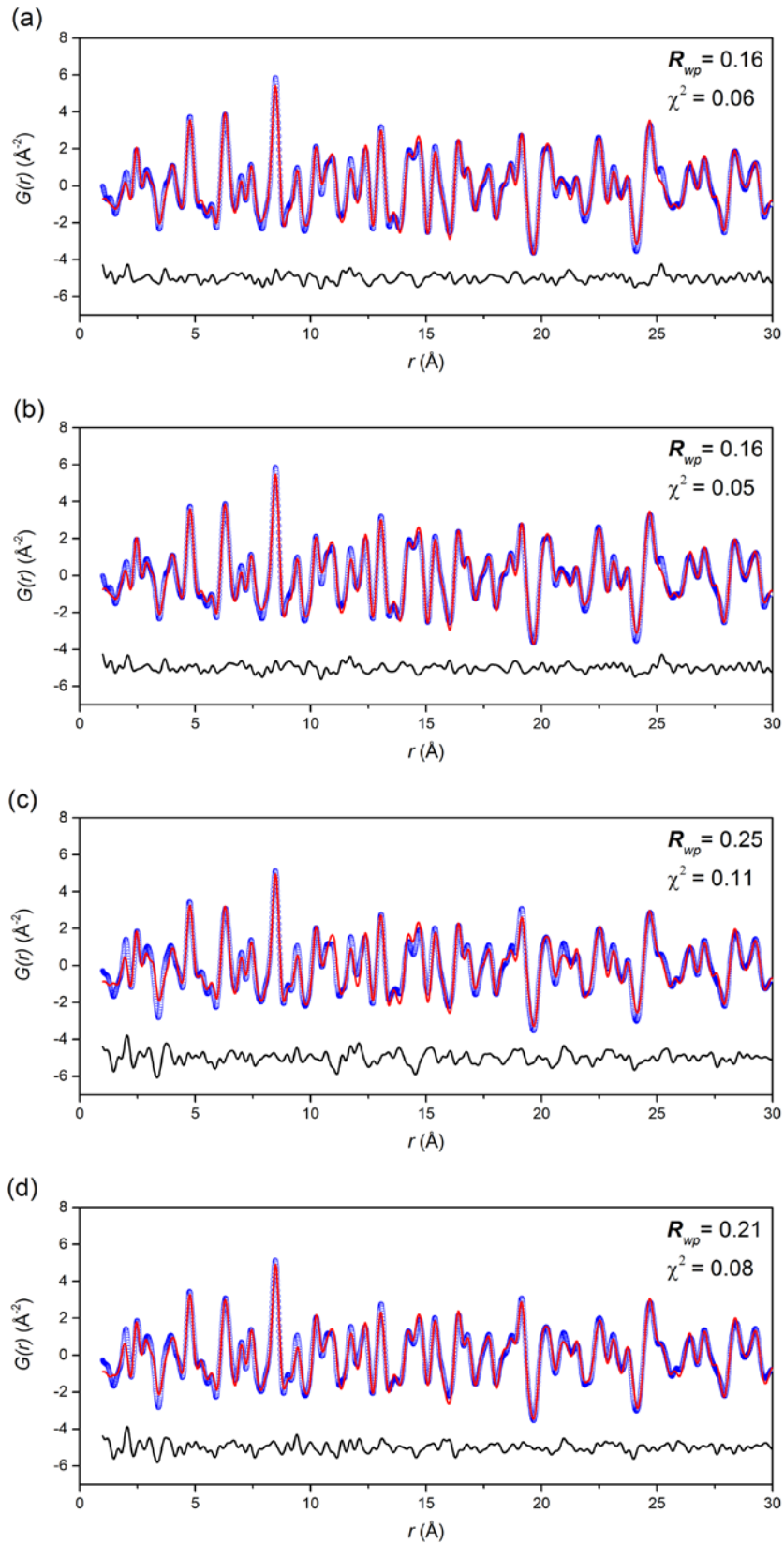


Figure S3. Fits to the PDF laboratory data for (a) rapid-cooled RI without anatase, (b) rapid-cooled with anatase, (c) slow-cooled without anatase, (d) slow-cooled with anatase. The experimental data is denoted by the blue line, the calculated pattern as the red and the difference pattern below as the black line.

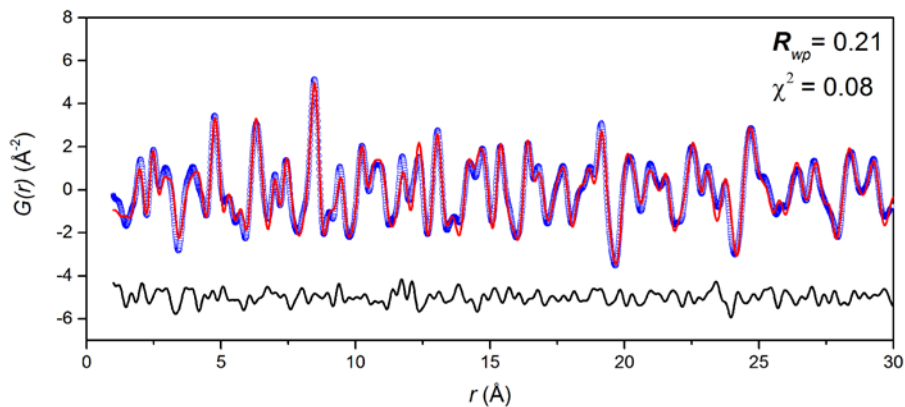


Figure S4 PDF data fit of slow-cooled RI with Ti_6O_{11} ($C2/m$) intergrowth phase. The experimental data is denoted by the blue line, the calculated pattern as the red and the difference pattern below as the black line.

References:

- Madsen, I. C. & Hill, R. J. (1992). *Adv. X-Ray Anal.* **35**, 39-47.
Madsen, I. C. & Hill, R. J. (1994). *J. Appl. Crystallogr.* **27**, 385-392.