Unraveling unforeseen disorders in silicates with 3D electron diffraction

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Zeolites are often synthesized as small polycrystalline powders that make their structure determination by single crystal X-ray diffraction challenging. 3D electron diffraction (3D ED) methods, especially continuous rotation electron diffraction (cRED), overcome the size limitation and can reveal structures of sub-micrometer sized crystals with similar resolution as single crystal X-ray diffraction. In this work, we have utilized cRED to reveal unprecedented disordered chains that link together the non cages \([4^{155}]\) in nonasil (NON) to form its complete 3D framework. The refinement against the cRED data in the reported \(Fmmm\) space group revealed residual peaks in the electrostatic potential maps that clearly indicate two configurations of the zig-zag chains that link the non cages together. These atoms reside on a mirror plane perpendicular to the c-axis. Another mirror plane that is perpendicular to the b-axis prevents them from relaxing into either configuration, and replacing it with a two-fold rotation axis allows full relaxation. Hence, the structure is best described by superposition of two \(Fm2m\) models, or two 50% occupant chain configurations in \(Fmmm\). Herein, with cRED and computational aid, we uncover the origin of the disorder and demonstrate that the same disorder is prevalent in all non-cage containing zeolite structures CIT-13 (*CTH), ERS-18 (EEI), EMM-25, EU-1 (EUO), ITQ-27 (IWV), and NU-87 (NES), except for ITQ-32 (IHW) and IM-12 (UTL).


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