

## Crystal structural modifications in U-rich, oscillatory-zoned hematite

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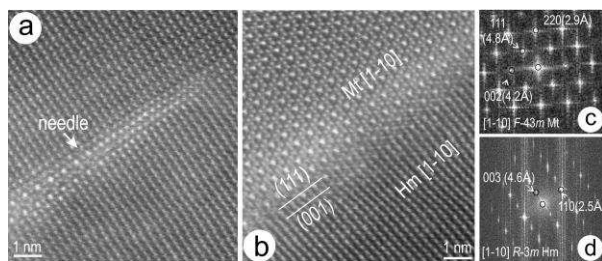
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Hematite, ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>; rhombohedral) is the most common phase among the five Fe<sub>2</sub>O<sub>3</sub> polymorphs [1]. Hematite from U-rich deposits is shown to incorporate this element up to several wt.% UO<sub>3</sub> [2,3]. This is commonly associated with a more complex signature comprising radiogenic Pb, W, Sn, and Mo and expressed in crystals with pronounced oscillatory zoning. TEM study of FIB-prepared foils extracted from high-U grains has shown crystal structural modifications interpreted as vacancy-induced superstructures [2].



**Fig. 1.** HAADF-STEM images of atom-wide (a) and tens of nm-wide (b) magnetite (Mt) needles in U-rich hematite. (c, d) FFTs obtained from hematite (Hm) and Mt in (a) and (b), respectively. Images obtained at 200 kV (Titan Themis; Adelaide Microscopy).

HAADF-STEM imaging shows modification of the crystal lattice from [1-10] rhombohedral to [1-10] cubic across domains nm- to tens of nm wide (Fig. 1). TEM-STEM mapping shows these domains are Fe-richer and O-poorer relative to matrix hematite, suggesting these represent nm-scale needles of magnetite (Fe<sub>3</sub>O<sub>4</sub>) instead of cubic ( $\beta$ -,  $\gamma$ -) Fe<sub>2</sub>O<sub>3</sub> polymorphs. Such needles are not found in high-W (up to several wt% WO<sub>3</sub>) or low-U (up to thousands of ppm U) hematite. Results suggest that such magnetite needles occur at the upper U solubility limit in the U-bearing hematite lattice. This is most significant for understanding reliability of such grains for high-precision U-Pb dating.

[1] Lee, S., Xu, H. (2016) J Phys Chem 120, 13316–13322.

[2] Ciobanu, C.L. et al. (2013) Precambr Res 238, 129-147.

[3] Verdugo-Ihl, M. et al. (2017) Ore Geol Rev 91, 173–195.