ESRF	**Experiment title:** Unraveling the peculiar internal structure of hematite microparticles	**Experiment number**: SC-5258
Beamline: ID13	**Date of experiment**: from: 30/08/2022 to: 03/09/2022	**Date of report**: 12/09/2022 *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Aicha Asma Medjahed Manfred Burghammer	

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Report:

The objective of this experiment was to investigate the internal structure of single hematite microparticles. We used the microbeam set-up at ID 13 which allowed us to completely illuminate one single particle at a time providing us with information on the average internal structure of a single hematite microparticle. We studied different shapes of hematite microparticles: cubic particles with edge lengths between 500nm-1µm (Fig 1A), ellipsoidal particles with major axes of 1.5 µm (Fig. 1B), and peanut-like particles with lengths of 2 µm (Fig 1C). These samples were measured as prepared as well as after calcination treatments at 600°C for varying times.

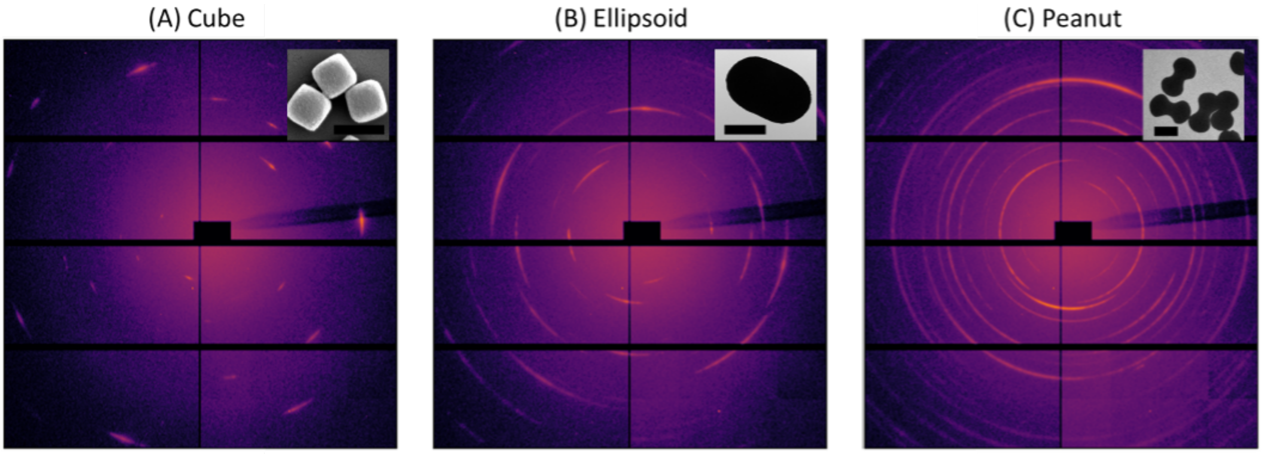

Figure 1: 2D images collected from a single (A) cubic, (B) ellipsoidal, and (C) peanut shaped hematite micro-particle. Electron microscopy images of each particle type are provided as insets. Scale bars are 1µm.

Examples of collected 2D diffraction patterns of hematite single particles with different shapes are given in Figure 1. The preliminary results show that the shape of the particle has an effect on the azimuthal broadening of the expected Bragg peaks. Cubic particles exhibit the least amount of this type of broadening suggesting that the crystallites inside a cubic hematite particle are well-aligned with each other.

Recent experiments in our lab have shown that calcination of the hematite particles at 600°C, leads to an increase in their photo-catalytic activity. Therefore, we investigated how the internal structure of the particle changed via different calcination times. Figure 2 shows 2D diffraction patterns of cubic hematite particles at 2, 4, and 10 hours calcination time. Analysis of the radial width of the diffraction peaks for different single particles could help us determine the nanocrystal size along specific crystallographic directions and, hopefully, the presence of the lattice stress. This could allow us to uncover how calcination affects the crystal structure and what lattice planes are important for hematite's photocatalytic activity. Corresponding data analysis is in progress.

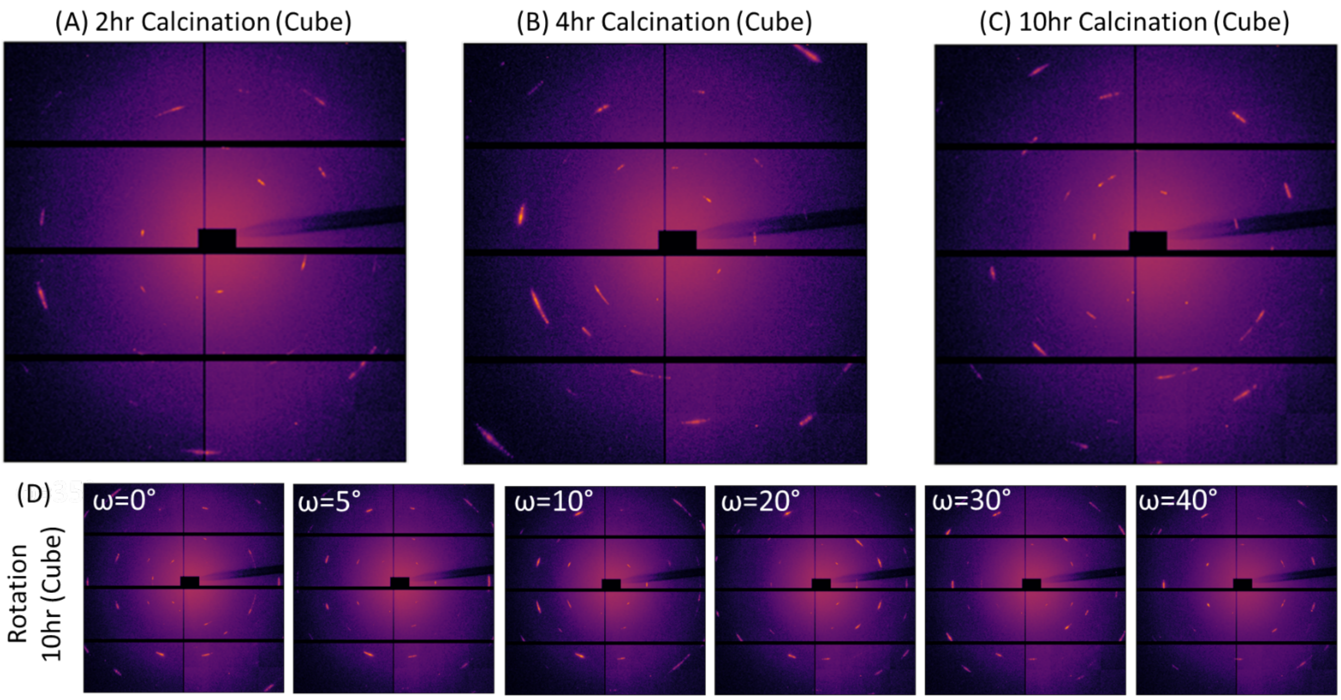

Figure 2: 2D images collected from a single cubic particle that has undergone different calcination times of (A) 2 hrs, (B) 4 hrs, (C) 10 hrs. (D) Rotational study of a single cubic particle that has been calcined for 10 hours with selected angles shown

Finally, with the set-up at ID-13 we were able to rotate our samples around the vertical axis. Figure 2D shows diffraction patterns of the same particle taken at different rotation angles. We anticipate that by performing quantitative analysis of rotating samples, we can reconstruct the 3D internal structure of each particle. Additional diffraction patterns of calcined and rotated particles have been collected also for ellipsoidal and peanut-shaped particles.

We would like to generously thank the team at ID-13, and in particular, Aicha Asma Medjahed and Manfred Burghammer for their in-depth support and assistance throughout the experiment.