The aim of this work was to characterize a sample consisting on FeO-Fe3O4 core-shell cubic-shaped nanoparticles. Because of the similarities in the composition and effective atomic number of the core and the shell, high angle annular dark field (HAADF) imaging could not be used to resolve the structure.

As an alternative, EELS fine structure can be used to obtain information on the oxygen and iron oxidation state, thus making it possible to distinguish between FeO and Fe3O4. However, there is the limitation that EELS projects the information of the 3D nanoparticle into a 2D map.

To overcome this limitation there is the possibility to consider EELS spectrum image data-sets as suitable for 3D tomographic reconstruction, not only containing information on the chemical composition of the sample (as in [1]) but also on the oxidation state of Fe at each voxel.

A tilt series of spectrum images (SI) was acquired on a probe corrected FEI Titan. Then the images were treated with Hyperspy to obtain independent spectral components from the iron edge, with could be correlated with the different iron oxides. In order to improve the quality of the reconstruction, a new reconstruction algorithm based on the mathematical theory of compressed sensing (CS) was used. To our knowledge this is the first time that the CS algorithm has been used to reconstruct an EELS core-loss spectrum image data-set.

The CS reconstructions show a shell thickness of 9nm around the core. The 3D reconstruction proves a total shell coverage of the core and that there has been no appreciable phase mixing.

[1] Ll. Yedra et al., Ultramicroscopy 122 (2012), pages 12-18. Acknowledgement: The measurements were performed in the Laboratorio de Microscopias Avanzadas (LMA) at the Instituto de Nanociencia de Aragon (INA) - Universidad de Zaragoza (Spain). We acknowledge the support received from the European Union Seventh Framework Program under Grant Agreement 312483 - ESTEEM2 (Integrated Infrastructure Initiative - I3) and under Grant Agreement 291522-3DIMAGE.
Fig. 1: Obtained spectral components from the iron edge after performing PCA and ICA with Hyperspy.

Fig. 2: Central orthoslice from CS reconstruction corresponding to 'core' component in figure 1 showing core thickness measurement.

Fig. 3: Central orthoslice from CS reconstruction corresponding to 'shell' component in figure 1 showing shell thickness measurement.