Experimental report

12/06/2020

Proposal: 1-04-146 **Council:** 4/2018

Title: Following crystallite morphology and magnetization during sintering of compacted strontium hexaferrite nano-platelets

Research area: Materials

This proposal is a new proposal

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Samples: $SrFe_{12}O_{19}$
$SrFe_{12}O_{19}$ w. Phenolic resin binder
$SrFe_{12}O_{19}$ w. Silicon resin binder
$SrFe_{12}O_{19}$ w. glass resin binder

Instrument	Requested days	Allocated days	From	To
D20	3	0		
D1B	0	3	19/07/2019	22/07/2019

Abstract:

The aim of the experiment is to investigate the changes in nano-crystallites size and morphology during high-temperature sintering of cold-pressed pellets of strontium hexaferrite. The subjects has scientific value as a crystallographic materials study, and in industrial applications of permanent magnet production. During the sintering of a pellet, the crystallites fuse together to form a more dense and sturdy pellet. This is accompanied by an unwanted crystallite growth, as the enhanced properties associated with nano-powders may be lost. To optimize the sintering process it is important to understand the changes in crystallite morphology as they occur. Neutron diffraction allows for entire pellets to be investigated, and the D20 instrument at ILL can provide the necessary time resolution to follow the sintering process in situ. The results will prove valuable for optimizing industrial manufacturing techniques.

Experimental Report for Proposal 1-04-146

Following crystallite morphology and magnetization during sintering of compacted strontium hexaferrite nano-platelets

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The experiment aimed to investigate the sintering process of a series of cold-pressed pellets *in situ*. The pellets were six series of in-house prepared pellets of strontium hexaferrite ($SrFe_{12}O_{19}$) and three commercially available pellets provided by an industrial collaborator. The three in-house prepared samples were synthesized using: a sol-gel (SG), a modified sol-gel (MSG), and a hydrothermal (HT) method.[1, 2] The samples were cold-pressed to multiple pellets with a diameter of 6 mm and a thickness of approx. 2mm. The commercial samples were cut into rods of approx. 5.5 x 5.5 x 30 mm. The samples were packed in vanadium canisters using a plastic straw (Fig. 1).

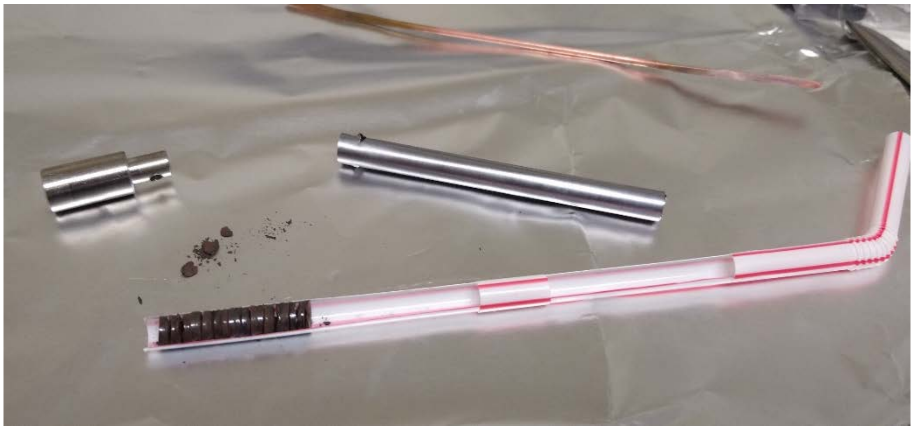

Fig. 1 – Photo of pellets being prepared for measurement.

The experiment was divided into two parts:

1) A temperature study of samples prepared from MSG powders, sintered at 600 °C, 800 °C, and at 900 °C.

2) A preparation study of pellets prepared from the three synthesis methods and the three commercial pellets, all sintered at 800 °C.

All experiments were heated with a rate of 5 K/min. An additional experiment at 700 °C was planned but had to be canceled due to issues with the first experiment (MSG@800 °C), which was instead successfully repeated.

The obtained data will be analyzed using sequential Rietveld refinement, and the nano-crystallite morphology will be determined from peak broadening effects.

Initial data analysis indicates a magnetic compensation temperature (Fig. 2), however, a more thorough refinement model is needed. This will be accomplished by combining the refinement with additional data, both from X-ray diffraction and magnetic measurements.

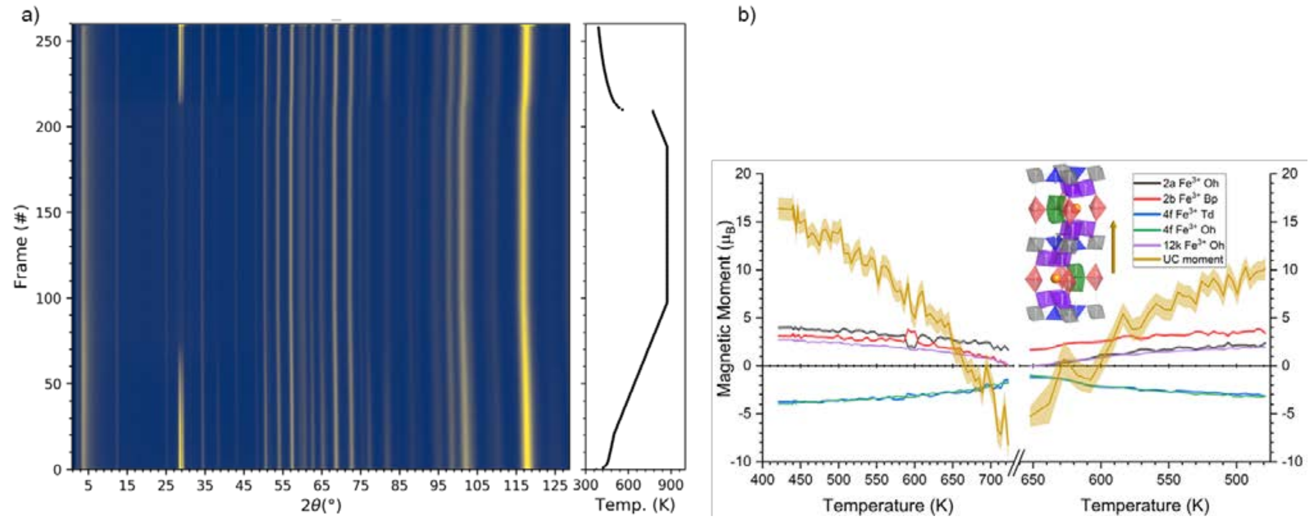

Fig. 2 – a) Frame-resolved diffraction data. The color-mapping indicating the intensity is shown on a log scale, using the perceptually linear colormap Cividis[3]. b) Initial magnetic moments obtained from Rietveld refinement. The inset shows the $SrFe_{12}O_{19}$ unit cell structure (drawn in the free software VESTA[4]).

References

[1] Saura-Muzquiz, M., C. Granados-Miralles, M. Stingaciu, E.D. Bojesen, Q. Li, J. Song, M.D. Dong, E. Eikeland, and M. Christensen, *Improved performance of SrFe12O19 bulk magnets through bottom-up nanostructuring.* Nanoscale, 2016. **8**(5): p. 2857-2866. DOI: 10.1039/c5nr07854g

[2] Eikeland, A.Z., M. Stingaciu, C. Granados-Miralles, M. Saura-Muzquiz, H.L. Andersen, and M. Christensen, *Enhancement of magnetic properties by spark plasma sintering of hydrothermally synthesised SrFe12O19.* Crystengcomm, 2017. **19**(10): p. 1400-1407. DOI: 10.1039/c6ce02275h

[3] Nunez, J.R., C.R. Anderton, and R.S. Renslow, *Optimizing colormaps with consideration for color vision deficiency to enable accurate interpretation of scientific data.* PLoS One, 2018. **13**(7): p. e0199239. DOI: 10.1371/journal.pone.0199239

[4] Momma, K. and F. Izumi, *VESTA 3 for three-dimensional visualization of crystal, volumetric and morphology data.* Journal of Applied Crystallography, 2011. **44**: p. 1272-1276. DOI: 10.1107/S0021889811038970