ESRF	**Experiment title:** Influence of strains in the occurrence and on the dynamics of the Verwey phase transition in magnetite	**Experiment number**: HC-4565
Beamline: ID06-HXM	**Date of experiment**: from: 16 Nov 2021 to: 23 Nov 2021	**Date of report**:
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Names and affiliations of applicants (* indicates experimentalists):
Dr. Emilio Lorenzo*, CNRS – UJF Institut Neel
Dr. Guillaume Beutier*, SIMAP Laboratory
Dr. Luc Ortega*, Universite Paris Sud
Prof. Andrzej Kozlowski*, AGH UST Department of Solid State Physics
Dr. Wojciech Tabis*, AGH UST Department of Solid State Physics
Mateusz Gala*, AGH UST Department of Solid State Physics
Dr. Stephan Gepraegs, Walther-Meissner-Institute

Report:

The aim of the experiment was to determine the influence of intrinsic strains on the metal-insulator first order phase transition, the Verwey transition (VT), in magnetite. Magnetite is a canonical compound, where strong electron-electron and electron-phonon [1] interactions cause the phase transformation at $T_V \approx 124$ K. VT is manifested in anomalies of principal physical quantities, like heat capacity, dynamical susceptibility (χ_{AC}), electrical resistivity and crystal symmetry change from cubic Fd3m to low-T monoclinic Cc.

VT is actually a composite phenomenon [2] where the crystal symmetry, charge and orbital orders change at slightly different temperatures possibly creating some stress caused by those electronic and atomic inhomogeneities. The origin of these inhomogeneities is unknown, which rises the questions: are these intrinsic strains that cause subsystem separation, or, vice versa, inhomogeneities cause strain? And to what extent the strain affects the VT. For example, it is well known that hydrostatic pressure lowers T_V [3], while T_V is increased by uniaxial strain [4], so any microscopic strain certainly affects the transition. To elucidate this problem, we used dark-field X-ray microscopy (DFXM) on three single crystalline, stoichiometric, samples identically oriented (the flat part was (001) plain), but grown with different method, i.e. possibly having some other strain system. JD3 sample (and the other one, of the same batch, "Jerome", not shown

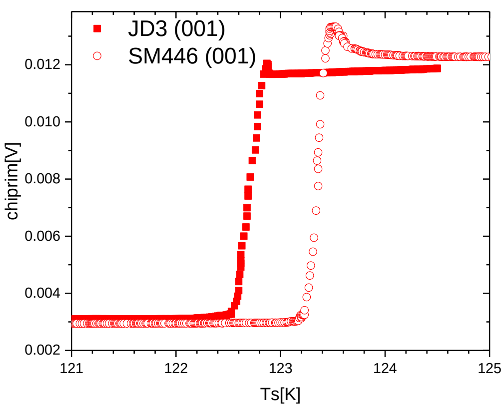
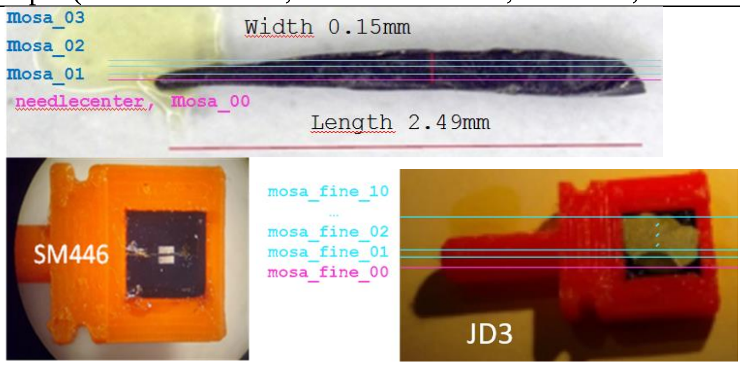


Fig. 1a. AC susceptibility for both measured samples



Samples had their surface polished with lapping paper and were mounted with GE Varnish onto 3D-printed sample holders provided on the beamline

Fig. 1b. Top: The shape of SM446 with measured slices (smz). (004) reflection and mosaicity map is for the pink position. Below, sample holders with both samples are presented.

below, but measured at 300 K) was grown using Travelling Solvent Floating Zone method, while the latter, SM446, with skull-melter technique at Purdue University, USA. Capturing the evolution of the internal texture of both samples above, in the vicinity and below T_V was a crucial part of the conducted experiment. Our plans were to first observe the crystal lattice quality (by mosaicity and strain mapping) at 300 K and correlate it with identical experiments below the Verwey transition. Although the first part was partially successful, it required long time and only initial observation was performed below T_V for only one sample. Therefore, we would like to apply for additional beamtime in future.

χ_{AC} was used to measure the Verwey temperature (see. Fig. 1a). In both cases, the VT is very sharp, with T_V for SM446 slightly greater by ~0.4 K. Both samples, with exposed (001) surface, were then observed in DFXM; beam size was 100 μm x 1 μm. Diffraction contrast was gained by the observation of the (004) peak, visible both in Fd3m and Cc phases. The results at 300 K, in the form of (004) peak profile in (μ, χ) scans (altogether 440 pictures per each of the 4 explored positions along sample height, *smz*, in case of SM446, 11 *smz* in other samples) and corresponding mosaicity maps are shown in Fig. 2a,b and 3a,b respectively. The (004) peak profiles are similar for both samples, although SM446 is more mosaic than JD3. In Fig. 2c and 3c the respective data are presented for sample JD3 below T_V.

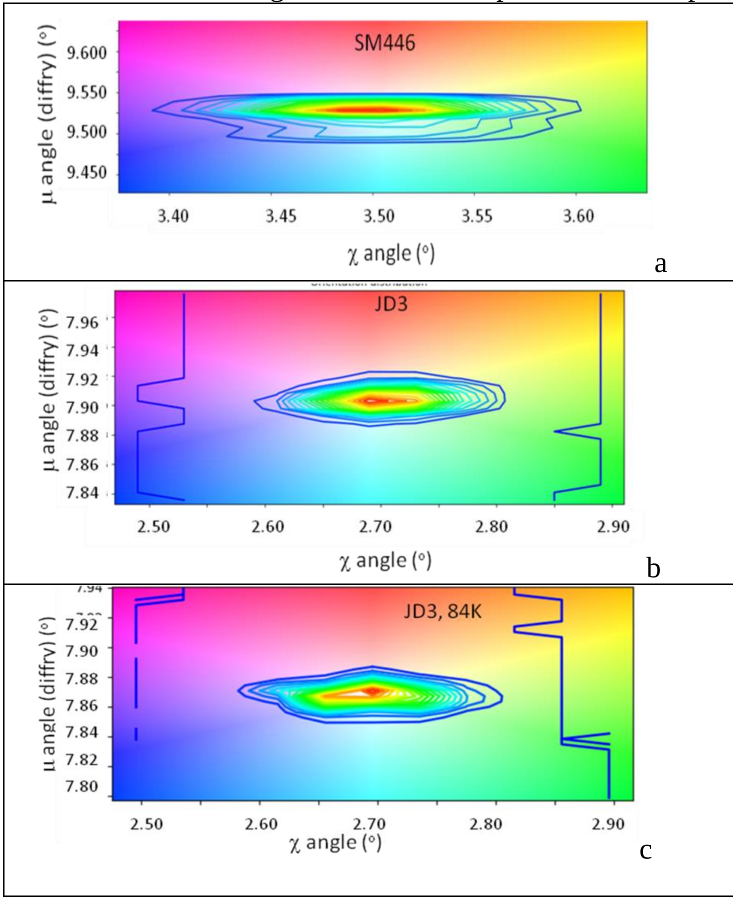


Fig. 2. (004) Rocking and rolling angle orientation distribution for (004) reflection observed for SM446 (a), JD3 (b), both at 300K, and JD3 at 84K

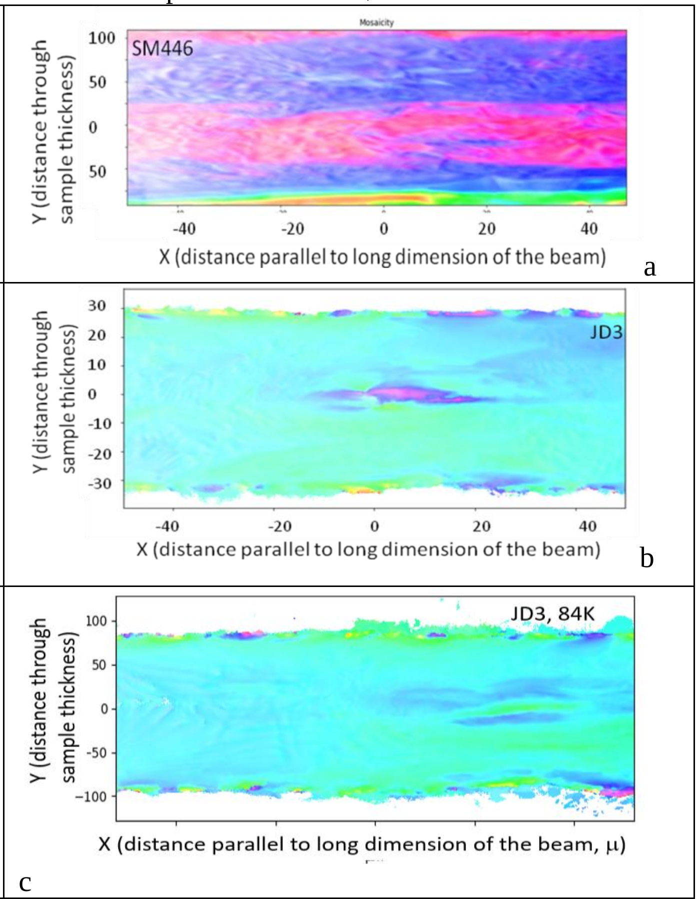


Fig. 3. Mosaicity maps for profiles presented in Fig. 2 for SM446 (a), JD3 (b), both at 300K, and JD3 at 84K samples, respectively. All distances are in μm.

Larger mosaicity in skull melter grown sample correlated with simultaneous slightly greater T_V may suggest that more defected sample increases the stability of low-T electron system increasing T_V. As already mentioned, T_V decreases with hydrostatic pressure while increases when the stress is more uniaxial, what is probably also visible here.

Departures from the ideal crystal structure are inherent in low-T phase of magnetite, where Cc structure naturally forces the occurrence of crystal domains. Therefore, the studies of VT vs. intrinsic strain/mosaicity should be done below T_V; this was our initial goal of the presented studies and this is what we want to achieve in follow-up studies.

There were good reasons that our goal was only partially achieved. First, more time than expected in a first place was spent in the Room Temperature (RT) studies. Since we needed three changes of LN_2 cryostream due to vacuum issues that prevented us from getting to the transition temperature, and one cryostream refill, several hours were dedicated to the cryostream change requiring addition a temperature ramps. Dismounting a cryostream required removing the sample from the goniometer, hence leading to the need of realignment and scanning of the RT state of the sample for reference. The GE varnish did not hold the sample as nicely as expected, leading to additional remounts and realignments. Beamline experienced pco-edege camera crashed overnight cutting some of our programmed scans that needed to be repeated, and a computer crashed on the morning of Saturday that lead to 2-3h of standy for rebooting. In the same manner some scans were interrupted by electron beam loss. All these unwanted events resulted in at least 4 shifts losts, a number that could be much larger had it not been the help of ID6 beamline Staff.

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[4]. Y. Nagasawa, et al., J. Phys. Soc. Japan 76, 110 (2007).