



	Experiment title: Ferrrian Ilmenite : a potential candidate for Martian magnetic anomalies ?	Experiment number: HE-2850
Beamline: ID12	Date of experiment: from: Oct 27 th , 2008 to: Nov 5 th , 2008	Date of report: 01/02/08
Shifts: 21	Local contact(s): Andrei Rogalev	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): France LAGROIX, Institut de Physique du Globe de Paris, France Bruce MOSKOWITZ, Institute for Rock Magnetism, Minneapolis, MN, USA Marie-Anne ARRIO, Yohan GUYODO*, Claire CARVALLO*, Philippe SAINCTAVIT, Institut de Minéralogie et de Physique des Milieux Condensés, Paris, France		

Report:

Our aim was to study the magnetic properties of titanium rich ($y > 0.5$) single phases of the hematite-ilmenite ($y\text{FeTiO}_3 \cdot (1-y)\alpha\text{Fe}_2\text{O}_3$) solid solution series, hereafter referred to as ferrian ilmenite

The hematite-ilmenite solid solution have interested geophysicists for its ability to acquire a magnetization antipodal to the magnetic field present. The resurgence of interest in the hematite – ilmenite solid solution series over the last decade is intimately linked to the question: What mineral is at the source of Mars's magnetic anomaly. The complex and surprising magnetic behaviour of exsolved phases arises from the interaction of the titanium rich and titanium poor lamella. A better understanding of the magnetic behaviour of its individual parts (lamella) is crucial.

The series end-members, hematite ($y=0$) and ilmenite ($y=1$), both have rhombohedral crystal structures but ilmenite's space group has a lower symmetry than that of hematite, owing to the presence of two different ions (Fe^{2+} and Ti^{4+}) on the cationic sites. As titanium enters hematite's crystal structure two Fe^{3+} atoms are replaced by one Fe^{2+} and one Ti^{4+} cation. At high temperatures, Fe^{2+} and Ti^{4+} ions are substituted randomly on the cation sites leading to cation disorder. This yields an order–disorder phase transition (T_{OD}) at $\sim 800^\circ\text{C}$ for $y = 0.6$ and increasing roughly linearly to $\sim 1400^\circ\text{C}$ for pure ilmenite ($y = 1$). Magnetic measurements reveal very complex behaviour resulting from the heterogeneity of cation ordering within the crystal structure of single phase compositions. This heterogeneity leads to the coexistence of more than one magnetic state at a given temperature. The volume ratio of ordered versus disordered cation clusters and the size of the clusters dictate what magnetic state will be observed at a given temperature.

We used a suite composed of $y = 0.7$ single phase ferrian ilmenites quenched in liquid nitrogen from various temperatures above the cation ordering transition (1300°C and 1050°C) and below (900°C) [Lagroix et al., *Journal of Geophysical Research*, 2004]. The samples were prepared as pellets made out of powdered ilmenite and graphite. We used a magnetic field of ± 2 T or ± 6 T and temperatures

between 300 K and 10 K. The XANES and XMCD spectra for the hemo-ilmenite quenched at 1050°C sample are shown on Fig. 1, and those for the hemo-ilmenite quenched at 1300°C on Fig. 2.

Due to the ferrimagnetic nature of the long range magnetic order, the magnetization per iron ion is not large and a great number of spectra were necessary to extract a nice, almost noiseless XMCD signal. The robustness of the XMCD signal has been extensively checked and we are very confident that relative intensities of the features are correct despite their very small amplitude. One should notice the absence of a derivative-like XMCD signal in the pre-edge, indicating that magnetic iron ions are mainly sitting on one crystallographic site. Indeed all iron ions are in octahedral sites, with antiferromagnetic coupling between (001) planes.

We extended our measurements to the Ti K-edge. The measured XMCD signal is very small and might be contaminated by diffraction peaks not visible in the XAS spectra. This set of data is still under analysis before we can conclude about the existence or absence of XMCD signal.

The analysis of these spectra is currently in progress. In particular, we now want to carry out ligand-field multiplet calculations simulating the Fe K-edge in the pre-edge region (Juhin et al., Phys. Rev. B, 2008) in order to extract quantitative information. The method previously applied by Juhin is well adapted to the interpretation of XAS for cubic crystals. The spectra obtained during this run must also be interpreted together with those obtained during experiment number 2850 “Magnetic self-inversion in titanomagnetite from Ocean Drilling Program”.

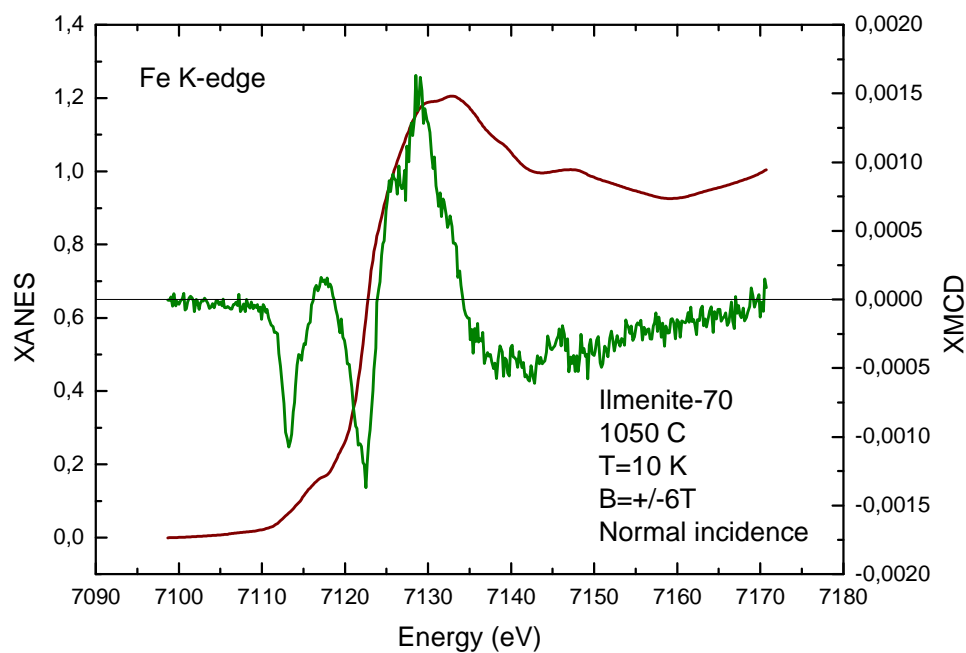


Fig. 1: Fe K-edge XANES and XMCD spectra at 6 K for the hemo-ilmenite quenched at 1050°C.

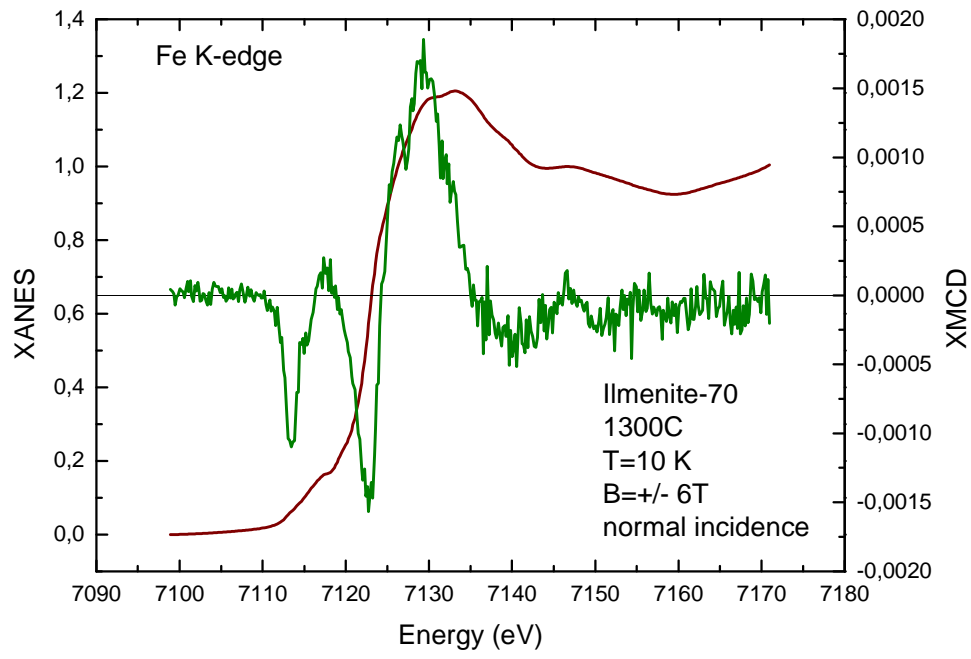


Fig. 2: Fe K-edge XANES and XMCD spectra at 6 K for the hemo-ilmenite quenched at 1300°C.