



	Experiment title: Magnetic self-inversion in titanomagnetite from Ocean Drilling Program	Experiment number: HE-2850
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Report:

Titanomagnetites ($\text{Fe}_{3-x}\text{Ti}_x\text{O}_4$, $0 \leq x \leq 1$) carry the magnetization in submarine basalts that constitute the ocean seafloor on Earth. They are used to study the geomagnetic field behaviour and the movement of tectonic plates. During oxidation, a titanomagnetite with a dominant octahedral sublattice magnetic moment can be transformed into a titanomaghemite ($\gamma\text{-Fe}_{2-4x}\text{Ti}_{3x}\text{O}_3$, $0.0 \leq x \leq 0.2$) with a dominant tetrahedral sublattice moment, resulting in a magnetization in a direction opposite to that of the titanomagnetite. Our main goal was to investigate the microscopic origin of this self-reversal mechanism, which is not known with certainty, and to determine the occupancy of the various tetrahedral and octahedral sites by the two cations titanium and iron, by using XMCD measurements at K edges of Ti and Fe.

The thermoremanent magnetization (TRM) of monodomain titanomagnetites is the magnetization acquired after cooling through the Curie temperature under the Earth magnetic field. In most cases, titanomagnetites are oxidized into titanomaghemites. The oxidation occurs by iron removal. It is a slow process, typically on a time scale of several million years. The process of oxidation replaces the original TRM by a chemical remanent magnetization (CRM). Magnetic data and models show that CRM can undergo a self-reversal (i.e. CRM is antiparallel to the primary TRM) due to the process of ionic re-ordering. During oxidation, a titanomagnetite with a dominant octahedral sublattice magnetic moment can be transformed into a titanomaghemite with a dominant tetrahedral sublattice moment. Oxidation induces vacancies on octahedral cations sites, which may shift the balance between the magnetizations of tetrahedral and octahedral magnetic sublattices. Then a reversal of spontaneous magnetization between oxidized and non oxidized grains would take place.

Self-reversed titanomaghemites are quite common in submarine basalts. This can have an important effect on the reliability of the paleomagnetic recording. However the exact microscopic origin of the self-reversal mechanisms is not known with certainty, because the cation distribution in titanomaghemite, and the subsequent ionic reordering phenomenon have never been experimentally addressed. Classical magnetic measurements such as SQUID measurements are blind to magnetic orientations of sublattices. The goal of our experiment was to investigate the origine of this self-reversed magnetization. This behaviour has been observed in particular in 49-Myr-old basalts sampled on the Emperor Seamounts (North Pacific) during a field campaign organized by the Ocean Drilling Program.

The measurements were carried out in a field of ± 2 T or ± 6 T. The temperatures were set between room temperature and 10 K.

We first measured the XMCD at Fe K-edge of a magnetite monocystal for calibration. The XANES and XMCD spectra are shown on Fig. 1; the presented spectra are raw spectra before correction for fluorescence reabsorption and compare well with previously published spectra [Mathon et al., J. Synchrotron Rad., 2004, 11, 423-427]. This gives us confidence in the next measured spectra.

We then measured XMCD at Fe K-edge on the 49-Myr-old natural titanomaghemite. We made a pellet out of powdered basalt from which we extracted the most magnetic grains with a hand magnet. This was then mixed with graphite. The magnetization of such natural material is very weak, because of the presence of titanium and of other unavoidable spurious phases that cannot be removed in these natural samples. It was necessary to accumulate many spectra in order to obtain a good signal-to-noise ratio. The XAS and XMCD spectra at room temperature are presented on Fig. 2. The XMCD is 10 times smaller on the natural titanomaghemite than on the magnetite single crystal. This is in line with the expected total magnetization for the natural titanomaghemite. In the pre-edge region, (7110 to 7120 eV) the shape is roughly the same but with different relative intensities of the positive and negative feature. From a fingerprint approach, this can be attributed to the difference of oxidation states between magnetite and titanomaghemite. During the beamtime we could measure the variation of the XMCD spectra as a function of the sample temperature between 300K and 10 K. For completeness, we also measured the XAS signal at Ti K-edge, at 300 K, and could record a sizeable XMCD signal indicating that the 4p empty level of the supposedly “diamagnetic” Ti^{4+} are magnetically polarized by the neighboring iron ions.

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.

For sake of completeness, the spectra obtained during this run shall also be compared with those obtained during companion experiment ESRF number 2856 “Ferrian Ilmenite : a potential candidate for Martian magnetic anomalies ?”

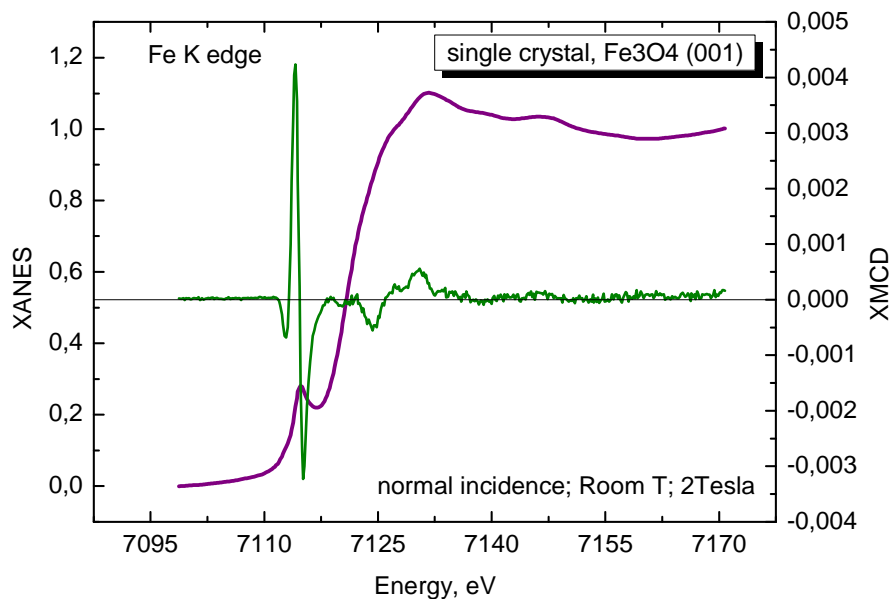


Fig. 1: XANES and XMCD spectra for a Fe_3O_4 single-crystal at room temperature.

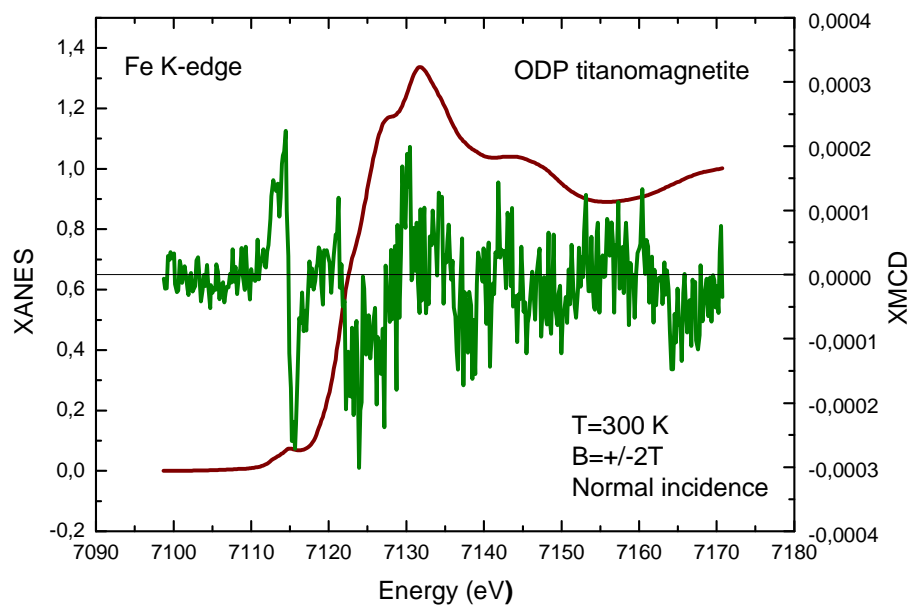


Fig. 2: XANES and XMCD spectra for the natural titanomaghemite sample at room temperature.