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| | Experiment title: Magnetite thermal expansion and its oxidation at the Curie temperature | Experiment number: CH-1733 |
| Beamline: BM01B | Date of experiment: from: 15-4-2004 to: 20-4-2004 | Date of report: |
| Shifts: 12 | Local contact(s): Mr Hermann EMERICH | <i>Received at ESRF:</i> |
| Names and affiliations of applicants (* indicates experimentalists): Dr. Davide LEVY* – University of Torino Dr. Monica DAPIAGGI* - University of Milano | | |

Report:

The aim of this experiment was the understanding of the peculiar behaviour of magnetite thermal expansion. From a previous work [1] it has been founded out that thermal expansion slope of magnetite depend on vacuum degree used for the experiment: low vacuum gives a thermal expansion slope change at 800 K (attributed to the magnetorestriction [2]), while using high vacuum the thermal expansion is smaller and no slope change has been revealed. From these result, it has assumed that for the first thermal expansion there is a partially oxidation of the sample, while in the second thermal expansion there is any oxidation. The present experiment has been performed to understand magnetite behaviour in a reducing atmosphere (argon) at high temperature: an argon flux passed through the sample in a 0.5 mm quartz capillary. However, this set-up did not completely remove oxygen in the system oxidising magnetite to maghemite or hematite and it was impossible to study its thermal expansion in reducing condition. Despite this experimental problem, at low temperature, the sample oxidised sufficiently slow to see a shift of magnetite peaks to higher angles with time (see figure1). This shift indicates a cell contraction before magnetite oxidation. To explain this phenomenon further experiments are needed. During the beam-time, an experiment on thermal expansion of maghemite (Fe_2O_3 with a defective spinel structure) has been collected from room temperature to 723K. The result is reported in figure 2. At the moment it can be highlighted that the maghemite thermal expansion curve is in between the two magnetite thermal expansion curve.

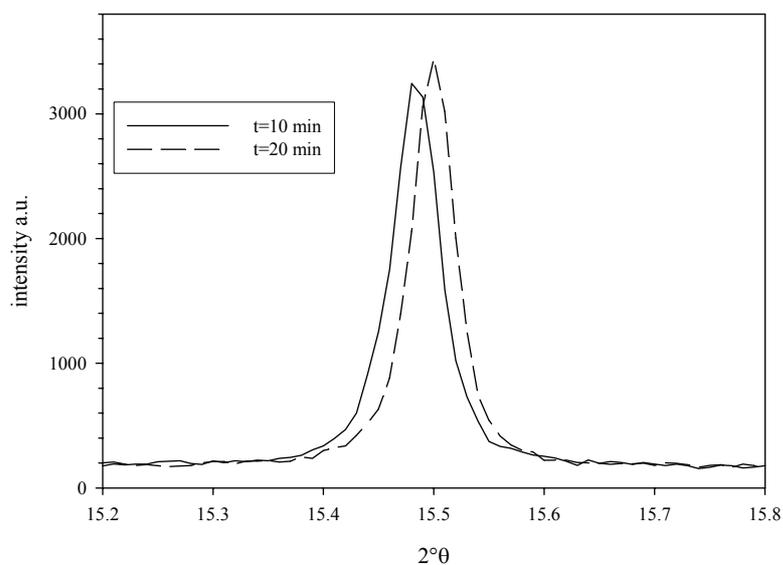


Figure 1

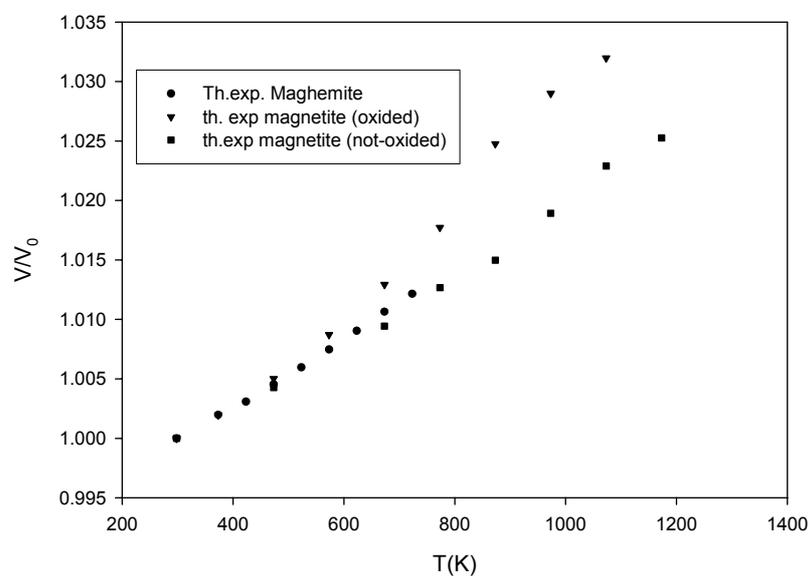


Figure 2

1. Levy, D., G. Artioli, and M. Dapiaggi, *The effect of oxidation and reduction on thermal expansion of magnetite from 298 to 1173 K at different vacuum conditions*. Journal of Solid State Chemistry, 2004. **177**(4-5): p. 1713-1716.
2. Okudera, H., K. Kihara, and T. Matsumoto, *Temperature dependence of structure parameters in natural magnetite: Single crystal x-ray studies from 126 to 773 K*. Acta Crystallographica Section B-Structural Science, 1996. **52**: p. 450-457.