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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Magnetocaloric compounds of the system MnxFe5-xSi3 at high pressures and valuable temperatures	**Experiment number**: HC 1640
Beamline:	**Date of experiment**: from: 19.11.2014 to: 22.11.2014	**Date of report**: 05.09.2016
Shifts:	**Local contact(s)**: Michael Hanfland	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
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Report:

Magnetic cooling based on the magnetocaloric effect could replace conventional vapor compression cooling, as it has a potentially lower energy consumption and does not rely on environmental hazardous gases [K.A. Gscheidner Jr., et al., Int. J. Refrig. 31, 945-961 (2008)]. The intermetallic compounds in the system $Mn_xFe_{5-x}Si_3$ ($1 \leq x \leq 4$) undergo a variety of magnetic phase transitions at different temperatures depending on their iron content which is distributed on at least one mixed Mn/Fe site and one pure iron site [H. Binczyska, at al., Phys. Stat. Sol., Sect. A, 19, 13-17 (1973)] and contain only cheap non-toxic elements in comparison with other possible candidates [K.A. Gscheidner Jr., et al., Int. J. Refrig. 31, 945-961 (2008)].

This system is an ideal choice to gain a better understanding of the underlying mechanism of the MCE in multiple site driven magnetocaloric materials. To study the effect of hydrostatic pressure on the crystal lattice and structure we measured powder diffraction diagrams of two compounds of the system $Mn_xFe_{5-x}Si_3$ (x=1 and x=4) on beamline ID09A at the ESRF (λ=0.414038 Å). This facility is equipped with diamond anvil cells that can be introduced in a He-cryostat so that it is possible to measure at non-ambient conditions of pressure and temperature simultaneously.

For the x=1 compound Powder was mounted in a membrane driven diamond anvil cell modified for Boehler-Almax anvils and helium was used as a pressure medium to ensure quasi-hydrostatic conditions. A total of 52 powder pattern were taken upon compression (0.10GPa to 10.91GPa) at ambient temperature and 11 under decompression. Additional 42 datasets were taken upon compression (0.11GPa to 7.68GPa) at 373 K. Ruby luminescence method was used for pressure calibration [Mao, H. K., et al., J. Geophys. Res., 91, 4673 (1986)].

For the x=4 compound Powder was mounted in an other membrane driven diamond anvil cell modified for Boehler-Almax anvils and helium was used as a pressure medium to ensure quasi-hydrostatic conditions. A

total of 220 powder pattern were taken upon compression (0.63GPa to 10.95GPa) at 100K, 150K 200K and 250K using the instruments cryostat. Additional 24 datasets were taken upon compression (0.24GPa to 12.49GPa) at AT and a combined number 100 datasets were taken upon compression (0.12GPa to 6.16GPa) at 325K, 350K and 375K using the instruments high temperature option. Ruby luminescence method was used for pressure calibration [Mao, H. K., et al., J. Geophys. Res., 91, 4673 (1986)].

Fit2D [A P Hammersley, *ESRF Internal Report*, **ESRF97HA02T**, ``FIT2D: An Introduction and Overview", (1997)] was used to carefully mask all strong diamond reflecions and integrate the data from the Mar555 detector.

To follow the temperature and pressure dependence of the lattice parameters refinements in the Le Bail method were started [A. Lebail, et. al., Mater. Res. Bull. 23, 447-452 (1988)]. Started from the dataset measured at the lowest pressure of each temperature. All refinements were performed with the program Jana2006 [V. Petricek, et. al., **Jana2006**, Institute of Physics, Academy of Sciences of the Czech Republic, Praha (2006)]. Two profile parameters (GW and LY) and the zero shift were taken into account in the refinement. The background was manually defined.

Complementary neutron (x=4 at SPODI MLZ) and in house x-ray (x=1,2,3) powder diffraction experiments as a function of temperature were taken to ilucidate the difference between the effect of temperature to the effect of hydrostatic pressure measured at ID09A. For direct comparisson a/c ratios were plotted over the unit cell volume (see fig. 1). The temperature dependant curves show a relatively small change in volume but a strong effect on the a/c ratio while hydrostatic pressure strongly affects the unit cell volume. This clearly show that the influence of temperature is significantly stronger than the influence of hydrostatic pressure in this caloric material.

The analysis of the temperature and pressure dependend data is currently being performed.

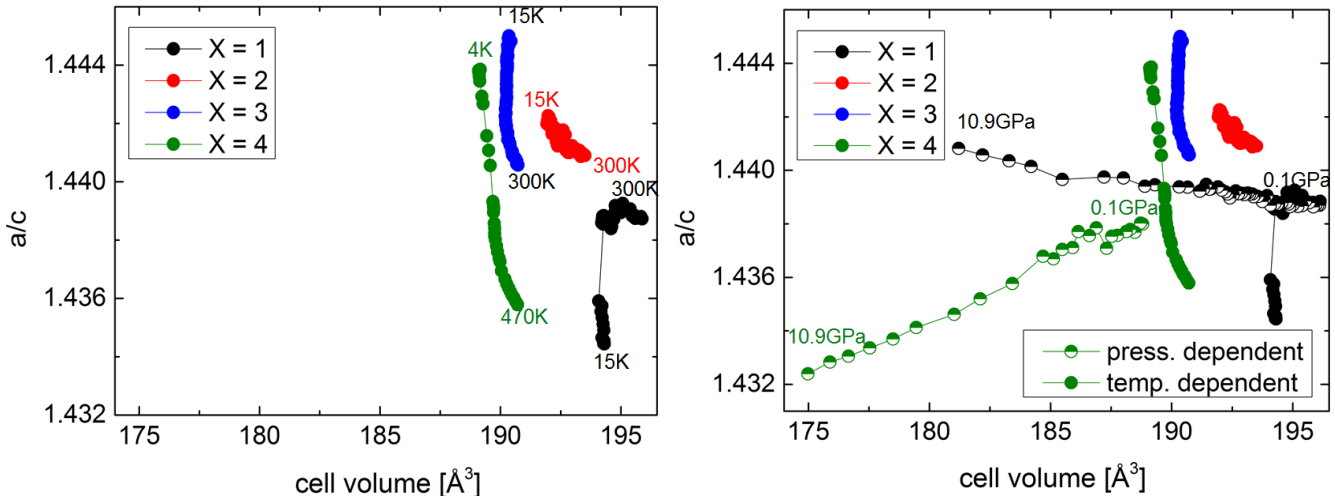

Fig. 1: Comparison of temperature dependant and pressure dependant cell volume and a/c ratio of four compounds of the series $Mn_{5-x}Fe_xSi_3$.