Results in Physics 5 (2015) 53–54

Contents lists available at ScienceDirect

Results in Physics

journal homepage: www.journals.elsevier.com/results-in-physics

Microarticle

Sample cell for in-field X-ray diffraction experiments

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ARTICLE INFO

Article history:
Received 20 November 2014
Accepted 9 January 2015
Available online 31 January 2015

Keywords:
In-field measurements
Applied magnetic field
Permanent magnet
X-ray powder diffraction
Magnetization
Synchrotron radiation

ABSTRACT

A sample cell making it possible to perform synchrotron radiation X-ray powder diffraction experiments in a magnetic field of 0.35 T has been constructed. The device is an add-on to an existing sample cell and contains a strong permanent magnet of NdFeB-type. Experiments have shown that the setup is working satisfactory making it possible to perform in-field measurements.

Introduction

Experiments performed using synchrotron radiation X-ray powder diffraction (SR-XRPD) with an applied magnetic field usually need specialized beam-lines for extreme conditions where samples can be exposed to high magnetic fields. In this work, an existing sample-cell has been equipped with a strong permanent magnet making it possible to perform in-field SR-XRPD measurements in a magnetic field of 0.35 T.

Experimental

Polycrystalline samples of nominal composition $(Fe_{0.45}Mn_{0.55})_2P_{0.50}Si_{0.50}$ were prepared by the drop synthesis method [1] using a high frequency induction furnace in an Ar atmosphere of 40 kPa at 1623–1673 K. Stoichiometric amounts of Fe (purity 99.995%), Mn (99.999%), P (99.999%) and Si (99.999%) were used as raw materials. Prepared samples were crushed, pressed into pellets, sealed in evacuated fused silica tubes and finally sintered, annealed and quenched in water.

The SR-XRPD experiments were conducted on the I711 beam-line at the MAX-IV laboratory in Lund, Sweden and the intensities were recorded on a large area Titan CCD detector [2]. The wavelength (1.104456 Å) and sample detector distances were determined using a LaB_6 standard. Samples were placed in a quartz capillary which in turn was placed in a single crystal sapphire tube which was mounted on a custom built sample cell [3]. An add-on was constructed to fit the sample cell making it possible to perform measurements in a magnetic field of 0.35 T using a permanent magnet of NdFeB-type, see Fig. 1. The samples were heated using a tungsten filament coiled around the sapphire tube. The SR-XRPD intensities were recorded using exposure times of 20 s and the data were reduced using the FIT2D [4] software and subsequently refined by the Rietveld method [5] using the software FULLPROOF [6,7] in sequential mode.

Magnetization experiments were performed using a Quantum Design MPMS SQUID magnetometer. Magnetization vs. temperature measurements were recorded in a temperature range of 300–370 K using field cooled cooling (FCC) and field cooled warming (FCW) protocols.

Results and discussion

The integrated 2D diffractograms vs. temperature collected in a magnetic field of 0.35 T are seen in Fig. 2. A structural transition due to magnetostriction may be distinguished at about 350 K. A further illustration of the experiment is shown in Fig. 3 where the relative changes of the unit cell parameters are plotted vs. temperature on heating and subsequently cooling. The transition temperatures are found to be 355 K on heating and 348 K on cooling which also indicates a thermal hysteresis of 7 K.

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http://dx.doi.org/10.1016/j.rinp.2015.01.006
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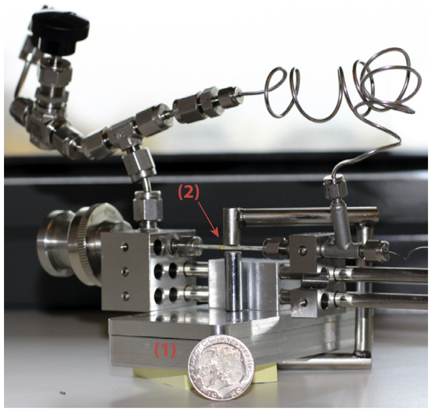

Fig. 1. Sample cell for SR-XRPD experiments in a magnetic field. A permanent magnet of NdFeB-type is placed inside the aluminum box (1). Samples placed in the gap (2) are exposed for a magnetic field of 0.35 T.

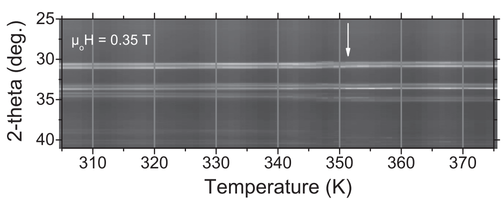

Fig. 2. Diffraction intensities vs. temperature for $(Fe_{0.45}Mn_{0.55})_2P_{0.50}Si_{0.50}$. Collected in a magnetic field of 0.35 T.

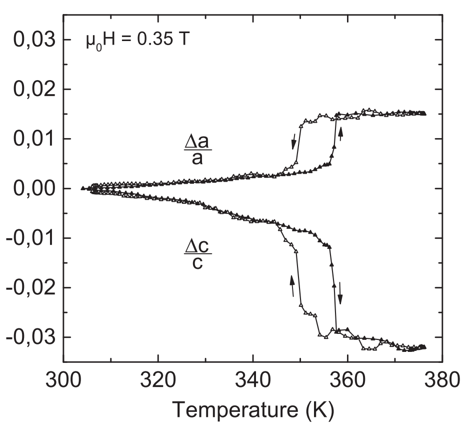

Fig. 3. The relative changes of the unit cell parameters of $(Fe_{0.45}Mn_{0.55})_2P_{0.50}Si_{0.50}$ vs. temperature. Data were collected in a magnetic field of 0.35 T.

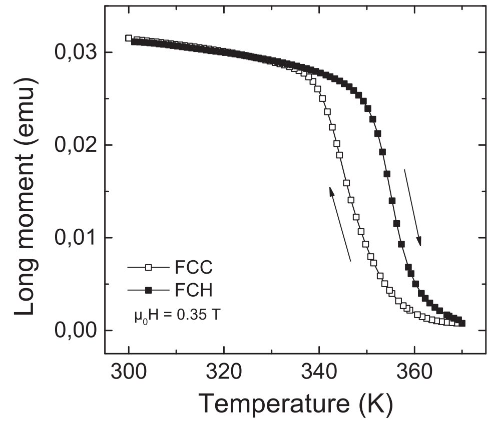

Fig. 4. Magnetization vs. temperature data of $(Fe_{0.45}Mn_{0.55})_2P_{0.50}Si_{0.50}$.

Magnetization vs. temperature measurements in the same applied field, 0.35 T, are shown in Fig. 4. A first order magnetic transition is observed at approx. 355 K on heating and 348 K on cooling which are in agreement with the transition temperatures and thermal hysteresis observed in the SR-XRPD measurements.

Conclusions

A simple set-up to perform synchrotron radiation X-ray powder diffraction experiments in a magnetic field of 0.35 T has been tested on a compound of composition $(Fe_{0.45}Mn_{0.55})_2P_{0.50}Si_{0.50}$.

Acknowledgments

This work was financed by the Swedish Research Council and the Swedish Energy Agency, which is gratefully acknowledged. The skillful assistance from Anders Lund and Jan Bohlin in construction of the add-on device is gratefully acknowledged. Mats Leijon is acknowledged for valuable discussions.

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