Field-induced Magnetic Transition in Cobalt-Ferrite

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We present magnetostriction and magnetization measurements of a cobalt ferrite (Co$_{0.4}$Fe$_{2.2}$O$_4$) single crystal. We observe unusual behaviour in the magnetic hard axis of the single crystal which manifests in a jump of the magnetization curve at a critical field. This first order magnetization process (FOMP) which is explained as an anisotropy driven transition is visible at temperatures lower than 150 K. By analyzing the anisotropy constants we found that the higher order anisotropy constant $K_2$ dominates the anisotropy energy. In the magnetostriction measurements the FOMP is clearly visible as a huge jump in the [111] direction, which can be explained by means of a geometric model.

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INTRODUCTION

Cobalt Ferrite is under examination since more than sixty years, but still there is quite a lot of open problems. In 1988 Guillot observed a jump in the field dependence of the magnetization in pure Cobalt Ferrite with the composition Co$_{1.04}$Fe$_{1.96}$O$_4$ and also in Cd substituted Cobalt Ferrite [1]. This jump was explained as a spin-flip. Because of the rather low critical field this explanation was not conclusive. Therefore within the present work this transition was studied in more detail.

SAMPLE PREPARATION

The single crystal with the composition Co$_{0.8}$Fe$_{2.2}$O$_4$ was grown by flux method. The starting materials are 18 g Na$_2$B$_4$O$_7$ · 10H$_2$O (Borax), 2.3 g CoO (99.99%), and 6.7 g Fe$_2$O$_3$ (99.99%). After sufficiently mixing, the materials were put in a tightly closed Pt crucible and heated from room temperature to 1370 °C at a rate of 100 °C/h, and then held for a period of 6 h; slowly cooled from 1370 to 990 °C at 2 °C/h, followed by a furnace cooling by switching off the power supply [2]. The composition of the single crystal was checked by XRD and SEM investigation.

EXPERIMENTAL PROCEDURES

Magnetization was measured from 5 to 400 K in a vibrating sample magnetometer with a superconducting 9 T coil. The single crystal was oriented by XRD in a Laue setup and then transferred to the VSM sample holder. The error from transferring the single crystal from one sample holder to the other was usually smaller than 1.5°.

The magnetostriction was measured with a miniature capacitive dilatometer described in [3] using a cryostat with a variable temperature insert (VTI) and a superconducting 9 T coil.

RESULTS AND DISCUSSION

Magnetization

The degree of inversion of the cation distribution of the inverse spinel (A$^{2+}$)B$^{4+}$O$_4$ was calculated to $i = 0.625$ with (Co$^{3+}_{0.8-i}$ Fe$^{3+}_{i}$)$_4$O$_4$. The magnetic moment of $\mu = \mu_{B-Sites} - \mu_{A-Sites} = 4.1 \mu_B$ at $T = 5$ K. The value of saturation magnetization at 5 and 10 K is practically the same due to the the very high ordering temperature.

The magnetization measurements revealed that the [100] axis is the easy axis of magnetization of Co-ferrite over the whole temperature range. Below 150 K a jump in the magnetization at fields occurs as it was also found in a similar material (Co$_{1.04}$Fe$_{1.96}$O$_4$) in Ref. [1]. In figure 1 the normalized magnetization at $T = 10$ K is plotted versus the external magnetic field. The jump is clearly visible at roughly 7 T in the [111] axis. The critical field differs from that published by Guillot which can be understood regarding the different sample composition. A linear fit between 1 and 6 T was performed showing that the extrapolation to 0 T leads to a value of 10.5 T, which indicates that the magnetization vector lies in

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FIG. 1. Normalised Magnetization of Co$_{0.8}$Fe$_{2.2}$O$_4$ single crystal at $T = 10$ K

The magnetic anisotropy was determined with the integral method $E_A = \int M_s H dM$ along the measured crystallographic axis of the single crystal. Applying the formula $E_A = K_0 + K_1(\alpha_1^2\alpha_2^2 + \alpha_2^2\alpha_3^2 + \alpha_3^2\alpha_1^2) + K_2(\alpha_1^2\alpha_2^2\alpha_3^2)$, where the $\alpha_i$ are the direction cosines in polar coordinates, the anisotropy constants $K_0$, $K_1$, and $K_2$ were determined. For using the integral method it is prerequisite to saturate the sample fully. It is reported that the saturation along the intermediate and hard axis is reached at around 18 T at $T = 4$ K [1]. Our measurements were only performed up to 9 T, therefore extrapolating the M(H) curves to saturation causes a rather large error ($\pm 20\%$ for $K_1$ and $\pm 30\%$ $K_2$), but they are still in the range of reported values of $K_1$ in literature.

In figure 3 the anisotropy constants $K_0$, $K_1$, and $K_2$ of the single crystal and $K_1$ of a polycrystalline sample are plotted against the temperature. For obtaining $K_1$ for the polycrystalline sample we used the law of approach to saturation. Measuring the polycrystalline sample the maximum magnetic field was only 9 T and the sample not fully saturated. Accordingly all reported values in literature using the law of approach to saturation are not giving the correct value of $K_1$, because they estimate only $K_1$ and not higher order anisotropy constants.

It is interesting to note that no values for $K_2$ are reported in literature. Above $T = 150$ K the value of $K_2$ is $\sim 6$ times higher than $K_1$ and below the FOMP the factor is even much higher, yielding in an increased anisotropy along the [111] and [110] axes.

Magnetic Anisotropy
CONCLUSIONS

We have proved that the jump in the magnetization can be explained as an anisotropy driven transition which is called “FOMP”. The transition is caused by a rotation of the magnetization vector jumping over an energy barrier. At low temperatures the second anisotropy constant \( K_2 \) is increasing which strengthens the [100] axis as easy axis and underlines the [110] as intermediate and the [111] as hard axis. These assumptions are also supported by geometric considerations explaining the values of the \( M(H) \) curves as measured in the different crystallographic directions. Additionally we present an accurate measurement of \( \lambda_{111} \) at a temperature of 4.2 K showing also this critical transition in the magnetoelastic behaviour.

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Magnetostriktion

We are presenting the first magnetostriiction measurement at \( T = 4.2 \) K demonstrating also the effect of such a field induced transition in the magnetoelastic behaviour as shown in figure 3. Due to hysteresis effects (remanence) the magnetostrictive measurements at low temperatures are very difficult and need a special measuring procedure as will be published elsewhere. But when increasing the magnetic field above the critical field, a huge jump in the magnetostriiction occurs. At higher fields due to rotational magnetization process no hysteresis effects are observed.

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FIG. 3. Anisotropy constants of \( \text{Co}_{0.8}\text{Fe}_{2.2}\text{O}_4 \) single crystal and one polycrystalline specimen as a function of temperature

FIG. 4. Magnetostriiction measurements of \( \text{Co}_{0.8}\text{Fe}_{2.2}\text{O}_4 \) single crystal at \( T = 4.2 \)K