Metastable Crystalline Cobalt Iron Oxide Nano-flakes with Antiferromagnetic/Ferrimagnetic Composition Mosaicity

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Spinel-type transition metal oxides are of high interest for a variety of applications, including heterogeneous catalysis, energy conversion, and magnetic materials with fine-tuned properties. For iron containing spinels, Mössbauer spectroscopy is a powerful method for the investigation of e.g. site occupancy, degree of inversion, and phase purity. The compound Co_2FeO_4 is of particular interest, but its phase diagram contains a large immiscibility.[1] Using Mössbauer spectra recorded at low temperatures (4.3 K) and high magnetic fields (10 T), the distribution of Fe ions across tetrahedral and octahedral sites was determined (Fig. 1). This enabled characterization of the influence of different calcination temperatures from 400 °C up to 900 °C on the phase composition and miscibility, while also providing valuable insights on the temperature dependent evolution of the spectral hyperfine structure. These findings were successfully correlated with results from magnetometry, showing clear signs of a change in magnetic properties based on different degrees of intermixing, interface area, and phase separation. Exchange bias was observed for the lowest calcination temperature (Fig. 1), which is usually not found within such self-assembled nanostructures. In addition to the analysis of unique structural phenomena, the project aims to develop an *operando* Mössbauer spectroscopy setup, allowing the investigation of structural development of iron containing catalysts. Financial support by the German Research Foundation (DFG) via the CRC/TRR 247 (Project-ID 388390466, sub-project B02) is gratefully acknowledged.

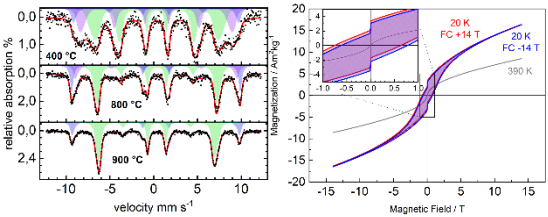

Figure 1: Mössbauer spectra of Co_2FeO_4 calcined at 400 °C, 800 °C, and 900 °C (left) and field dependent magnetization of the 400 °C sample (right).

[1] I.-H. Jung *et al.*, *Acta Mater.* 2004, **52**, 507-519, 10.1016/j.actamat.2003.09.034.