Anomalous diffraction on D2AM – ESRF Grenoble

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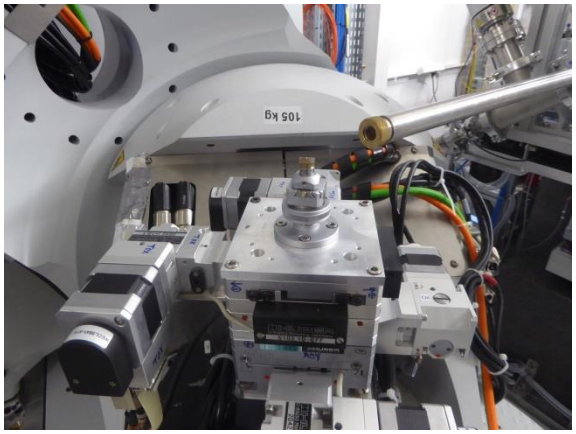
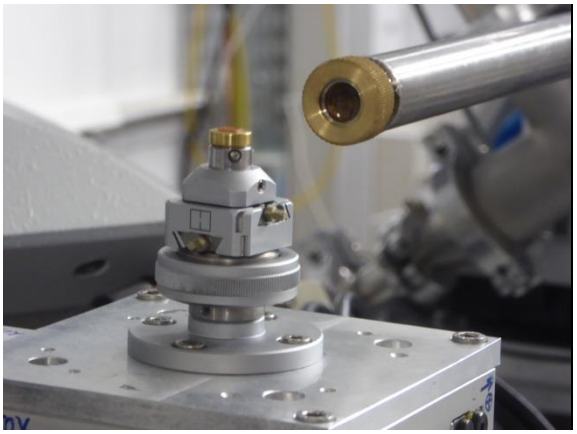
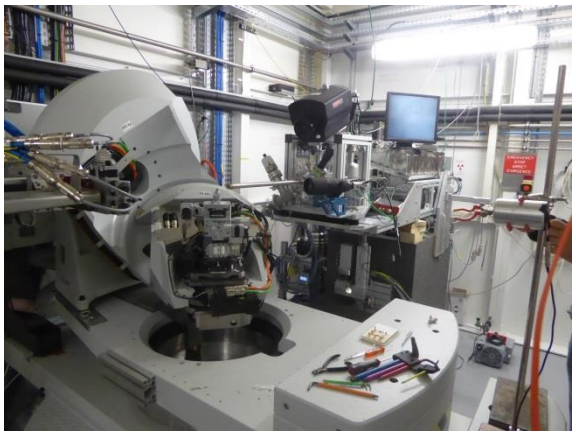
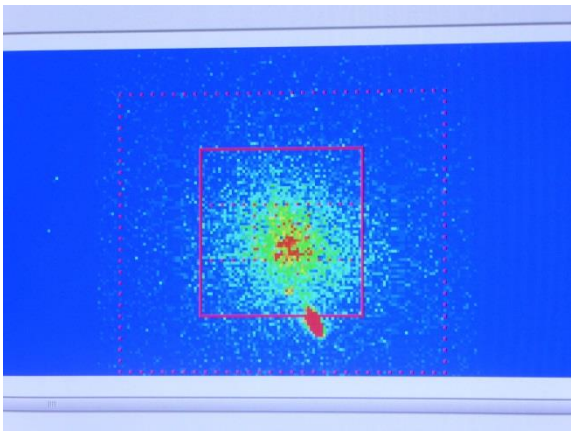
Experimental report HC 2845

Samples analyzed and objectives

CoFe2O4 and Gd-substituted CoFe2O4 spinel films on MgO
The effect of the oxidizing pressure on the cationic distribution
The position of Gd in the Gd substituted samples

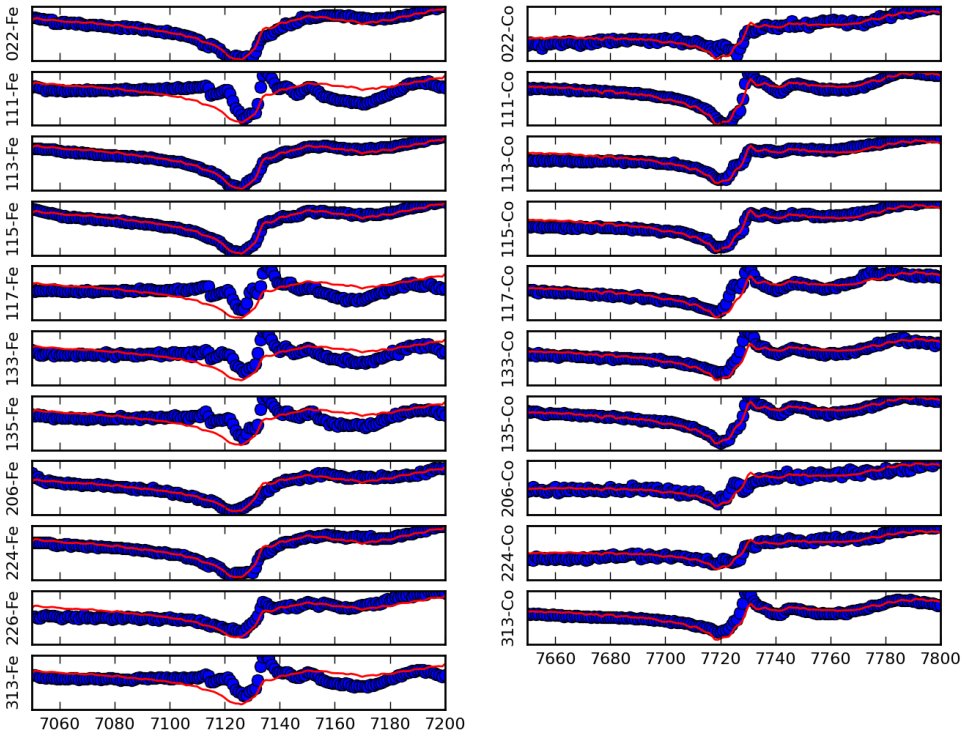
ADS35EM = GFO by sputtering + GFO by PLD
Effect of two consecutively deposition on the orientation of the unit cell

First results

CoFe2O4//MgO samples

EM10B	MgO//CoFe2O4 0.04 mbar 400°C	8.416	8.47	8.43
EM06	MgO//CoFe2O4 0.5 mbar 400°C	8.41	8.43	8.33
EM05B	MgO//CoFe2O4 0.02 mbar 400°C	8.19	8.08	8.62
EMGd01	MgO//CoFe1.8Gd0.2O4 0.05 mbar 400°C	8.36	8.39	8.60
EMGd07	MgO//CoFe1.8Gd0.2O4 0.1 mbar 500°C	8.46 8.39	8.42 8.45	8.56 8.43
EMGd09	MgO//CoFe1.8Gd0.2O4 0.02 mbar 400°C	8.44	8.42	8.62

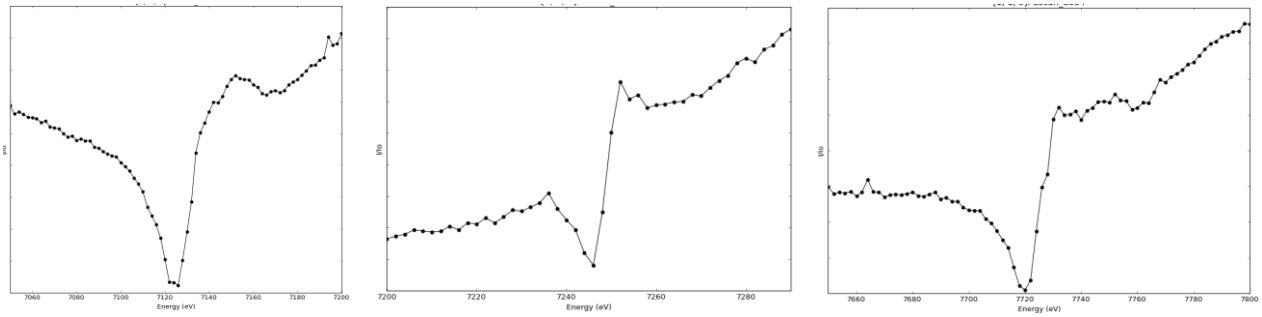
• EM10B



First refinements of EM10B using the FitREXS code is shown above. The result clearly indicates a cationic distribution $(Co_{0.1}Fe_{0.9})[Co_{0.9}Fe_{1.1}]O_4$. However, some additional rebounds are noticeable (e.g. for the 111-Fe) which have to be clarified. For the other samples (i.e. EM06 and EM05B), refinements are under process.

• EMGd01

For Gd- doped sample, spectra recorded at the Gd edge show clearly a signal indicating that Gd is in the structure.



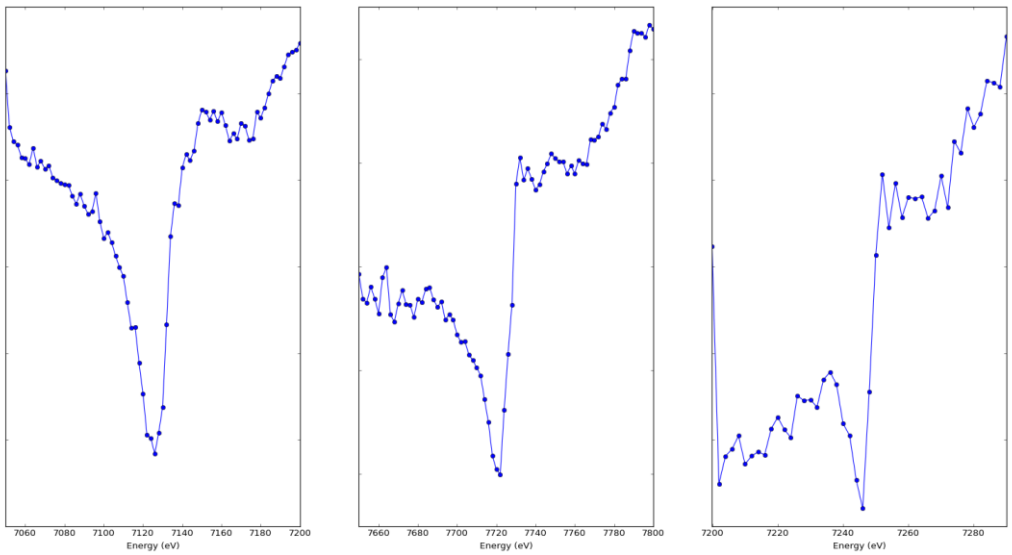
Recording spectra of the 113 of EMGd01 at the Fe edge (left), Gd edge (middle) and Co edge (right). Those spectra have been corrected of fluorescence

• EMGd07

EMGd07, deposited under 0.1 mbar at 500°C, contains 2 spinel phases. It is difficult to separate them but 2 different orientation matrices have been done and the measurement shave been performed with each of them. One of the phases has smaller parameters and it is thus expected to contain less Gd.

• EMGd09

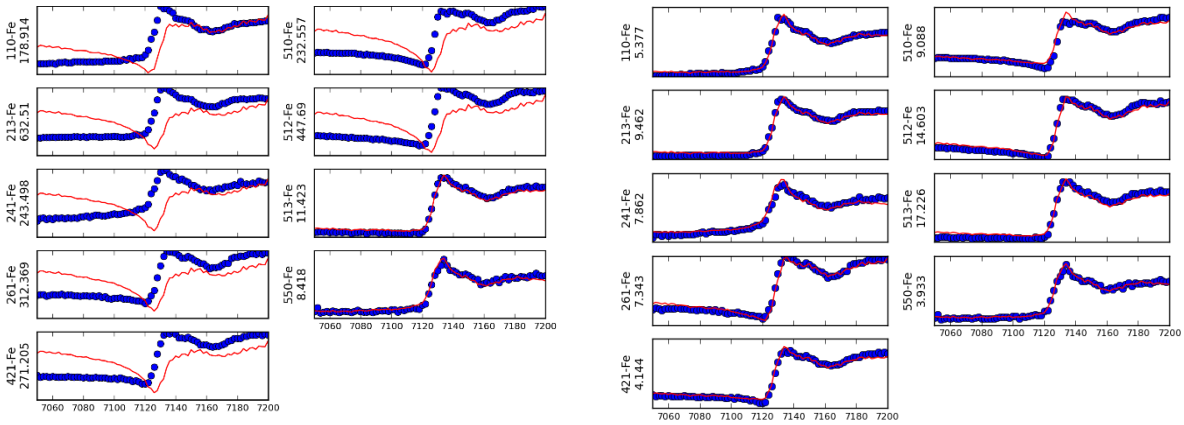
As well as for EMGd01, a signal at the Gd edge is observed indicating then unambiguously that Gd is inserted within the structure.



ADS35EM = GFO by sputtering + GFO by PLD

ADS35EM	YSZ001//sputtered GFO/PLD GFO	8.749	9.425	5.073

We already have proved that orientation of the unit cell is dependent on the elaboration method: with tetrahedron pointing toward the unit cell (PLD ; η = 0) or having an opposite direction (sputtering ; η = 0.3). The aim of this measurement was to check the orientation of a thin film that had consecutively the two elaborations and thus to answer to the following question: Has an additional layer deposited by PLD reoriented the film initially elaborated by sputtering?



The best refinement considering only the orientation of the unit cell as free parameter is shown on the left graph. It's clearly evidenced that the refinement I not optimum. Refinements of the cationic positions as well as orientation reach to the graph on the right. For this refinements, we obtain a value η = 0.3 indicating an intermediate state between PLD and sputtering deposition.