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Structure-activity relation of spinel-type Co-Fe oxides for lowtemperature CO oxidation

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	C1s	O1s	Co2p	Fe2p	Co/Fe	Co	Fe	0
Co _{0.9} Fe _{2.1} O ₄	43%	49%	3%	6%	0.43	0.9	2.1	4
Co _{1.8} Fe _{1.2} O ₄	37%	51%	7%	5%	1.47	1.8	1.2	4
Co _{2.1} Fe _{0.9} O ₄	35%	49%	11%	5%	2.23	2.1	0.9	4

Table ESI 1: Estimated composition at the surface of the Co-Fe thin films.

Details about the XPS analysis

The surface composition was determined by the means of X-ray photoelectron spectroscopy (XPS). XPS was done in a multi technique ultra-high vacuum instrument (Multiprobe, Omicron Nanotechnology) using a monochromated Al K α X-ray source (1486.7 eV, 280 W) and a hemi-spherical electron energy analyzer (Sphera) in constant analyzer energy mode (25 eV) and a step size of 0.05 eV. The sample was located under an angle of 13° from the surface normal to the electron detector. The base pressure of the chamber is 2×10^{-10} mbar. CasaXPS was used to analyze the spectra, and a Shirley background subtraction procedure was employed. The elemental composition was calculated using the area of the Fe2p, Co2p, C1s and O1s peaks with the according Scofield cross-sections. The peak areas of the Fe2p and Co2p signals were corrected for Auger LMM peaks that each metal has in the 2p-area of the other metal. In both cases, mainly the $2p_{3/2}$ parts of the spectra are affected. To do the correction, 2 sputter-cleaned metal oxide samples made by PSE-CVD

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were measured as references. The intensity correction of the peak area was done according to the following formula:

$$A(Fe - 2p) = \frac{M(Fe) - K(Co)M(Co)}{1 - (K(C0)K(Fe))} \qquad A(Co - 2p) = \frac{M(Co) - K(Fe)M(Fe)}{1 - (K(C0)K(Fe))}$$

Definitions:

A = Area

K = [A(LMM) / A(2p)] (Correction factor)

Measured Areas:

M(Fe) = A(Fe-2p+Co-LMM)

M(Co) = A(Co-2p+Fe-LMM)



Fig. ESI 1 Pure iron oxide (a, b) and cobalt oxide (c, d) reference sample.

Area ratio K (Fe) = A(Fe-LMM) / A(Fe-2p) = 14.3 / 84.8 = 0.148 Area ratio K (Co) = A(Co-LMM) / A(Co-2p) = 41.1 / 206.8 = 0.210 Electronic Supplementary Material (ESI) for Catal. Sci. Technol. This journal is © The Royal Society of Chemistry 2013



Fig. ESI 2 The original XPS spectra of Fe2p and Co2p with the corresponding Shirley backgrounds. In addition, the subtraction of the Auger signals was done to obtain less-disturbed iron and cobalt spectra. This was done with CasaXPS by subtracting the LMM references from the 2p data (see Fig 2a and 2b in the main text).



Fig ESI 3 Additional HIM micrographs.