ESRF	**Experiment title:** *XAFS characterisation of new Ni-Fe catalysts for large-scale production of carbon nanofibers*	**Experiment number**: **01-01-635**
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Report:

A series of Ni-Fe bimetallic hydrotalcite (HT) catalysts has been prepared. The catalysts have been exposed to CO or ethylene to produce carbon nanofibres (CNF). Upon calcination HT will give highly dispersed cations and high surface area, while the alumina phase will stabilise the Ni and Fe against sintering or fragmentation during subsequent reduction and reaction.

Limiting the rate of deactivation is crucial for large-scale production of carbon nanofibres. It is known that the deactivation depends significantly on the catalyst properties and parameters in the catalyst preparation. Recently it has been found that the time period of catalyst storage also affects the deactivation. However, the mechanism of catalyst deactivation is not well understood. It is likely that the catalyst crystal size and the local surface structure might be key factors.

Experimental method

EXAFS data have been collected at both Ni-K and Fe-K absorption edges using the Si(111) channel-cut monochromator. Higher order harmonic rejection was achieved using double-bounce Cr mirrors at a cut-off energy of 19.5 keV.

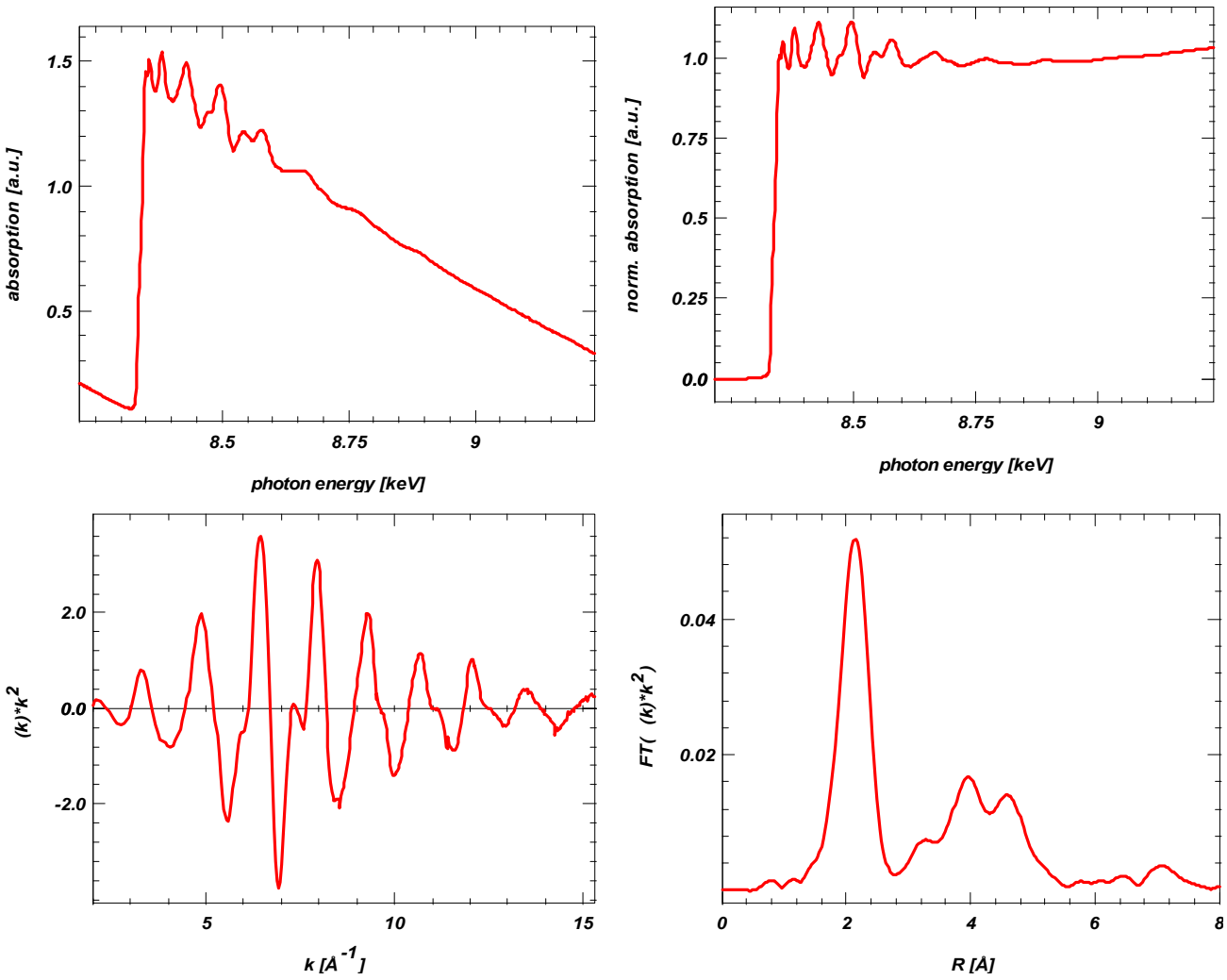

Figure 1: The Ni K-edge EXAFS spectra of a Ni-Fe-hydrotalcite catalyst after the production of carbon nanotubes. The raw spectrum, the normalised spectrum, the unfiltered chi-curve and Fourier transformation is shown.

Multiple scans of each sample (2 – 4, depending on metal loading) had to be collected and summed in order to achieve the desired signal-to-noise ratio. The EXAFS spectrum in Figure 2 shows that the data quality is very good with low noise levels as far out as 15 k.

Fresh and used Ni-Fe containing hydrotalcite catalysts with different Fe/Ni ratios have been studied. The carbon nanofibres still contain the catalysts used for producing the fibres. A milligram of catalyst can produce as much as 1000 mg of carbon nanofibres. This makes the reaction hard to study *in situ*. The vast amount of carbon produced, and the fact that the catalyst is fragmented during reaction, leads to a continuous change in sample environment. Even XAS scans with high time-resolution would be disturbed by the rapid changes. The set-up at the beamline does not have the required time resolution for monitoring such rapid transitions.

Preliminary results

Preliminary data analysis shows a clear change in particle morphology after reaction. Detailed EXAFS analysis will continue to be processed in the forthcoming months. The results will be presented at the 11th Nordic Symposium on Catalysis in Finland, May 2004.