HZDR HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF ROBL-CRG	**Experiment title:** In-situ investigation of the dewetting process of thin iron film in the CNT growth process	**Experiment number:** **20_02_682**
Beamline: BM 20	**Date of experiment:** from: 21.4.2009-28.4.2009	**Date of report:** 3.2.2011
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Results

Carbon nanotubes (CNTs) can be grown from gaseous precursors by the use of metal nanoparticles as catalysts. These catalyst particles are commonly prepared by dewetting of metal thin films at elevated temperature under hydrogen atmosphere (to reduce thin oxide layers).[1] The size of the resulting particles strongly depends on the film thickness of the pristine material (approx factor 3, see report 20-02-671). The system under investigation is Fe, due to Fe being the most commonly used nanotube catalyst.[2,3]

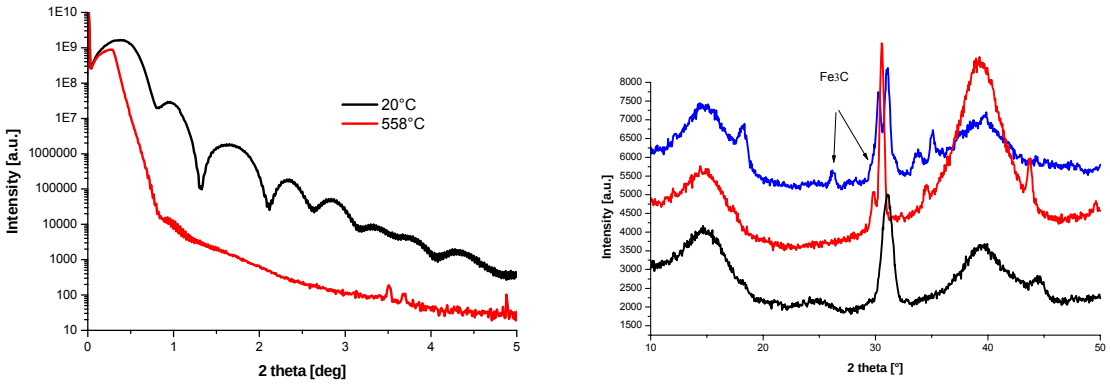

Fig. 1, left: XRR curve of the pristine material and the spitted film. Right: XRD pattern of the catalyst at different stages of the reaction; before (black line, after heating (red line and during CNT growth.

The pristine material is pure α-Fe in the thin film (shown in Fig. 1 right side, black line) with an amorphous oxide layer seen by an additional oszillation in the XRR curve in Fig. 1 left side, on a Si-wafer with a 200 nm SiO_2 buffer layer. By heating the film splits and a

phase transformations into a mixture of α- and γ-Fe (Fig.1, right side red line) as catalyst are observed. When applying H_2C_2 as carbon source gas this α/γ-ratio is shifted towards the γ-Fe phase. The formation of CNTs starts immediately (signal at 18° 2theta in Fig. 1, right hand, blue line) and additionally crystalline Fe_3C is observed. The growth parameters were 560°C surface temperature, 170 mbar pressure by 30 scccm Ar, 10 sccm H_2 and 1 sccm H_2C_2 gas flow.

We succeeded in establishing that the carbide is not necessary for CNT growth by growing CNTs only from metallic Fe, when carbide formation was very extensivly avoided. But further time resolved experiments are needed to elucidate the question, if the carbide is also an active catalyst or only a side product.The reaction itself shows a very high yield of CNTs, as shown in Fig.2.

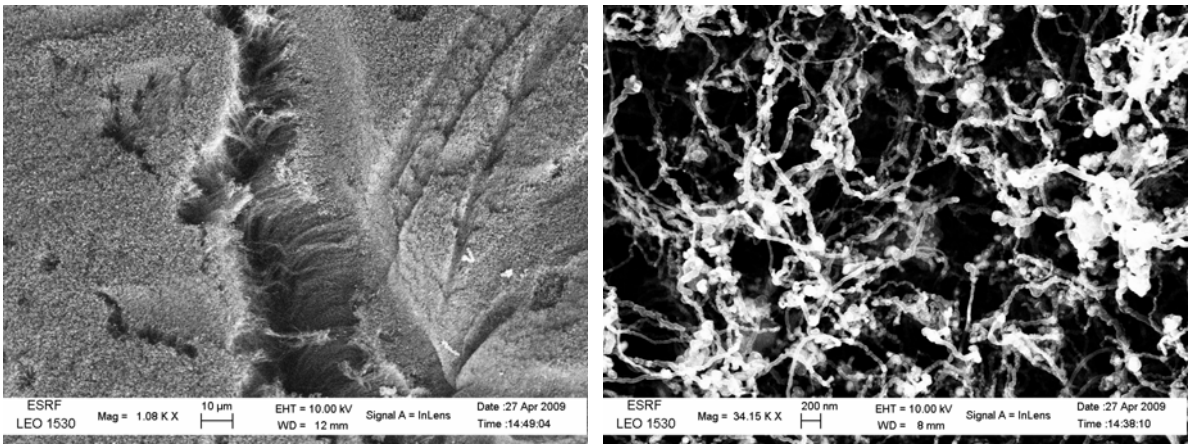

Fig.2: SEM picture of the CNT grown with fe-catalyst.

The preparation of the results for publication is in progress.

References

[1] Mattevi et al. J. Phys. Chem. C 112, 12207 (2008).
[2] Wirth et al. ACS Nano 3, 3560 (2009).
[3] Hofmann et al. J. Phys. Chem. C 113, 1648 (2009).