ROBL-CRG	**Experiment title:** In-situ XRD/XRR of thin film catalysis during carbon nanotube growth	**Experiment number**: **20_02_666**
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Results

Metal nano-particles act as catalist in the carbon nano tube CNT growth process [1,2]. We focused on catalyst formation by the dewetting of polycrystalline Ni-films with different thickeness on SiO_2 support during annealing under a reducing hydrogen atmosphere at 0.7 mbar. For the subsequent CNT growth at 550°C acetylene (55% H_2 and 45% H_2C_2 at 1.4 mbar) was added as carbon source. The synthesis process was

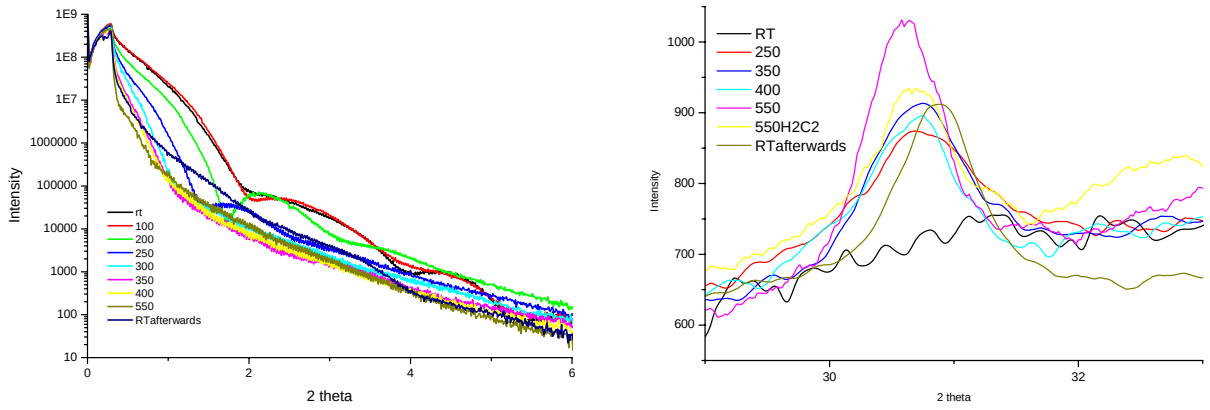

Fig.1: 2 nm Ni onto 200nm SiO_2 buffer layer on Silicon. Left: XRR data display first the reduction of the surface oxide layer and accompanying film smoothening and secondly the film dewetting. The high frequent oscillations visible in the data are due to a 200nm SiO_2 buffer layer of the system under investigation. Rigth: XRD data of the Ni (111) reflection show Ni-crystal growth during heating by crystallization.

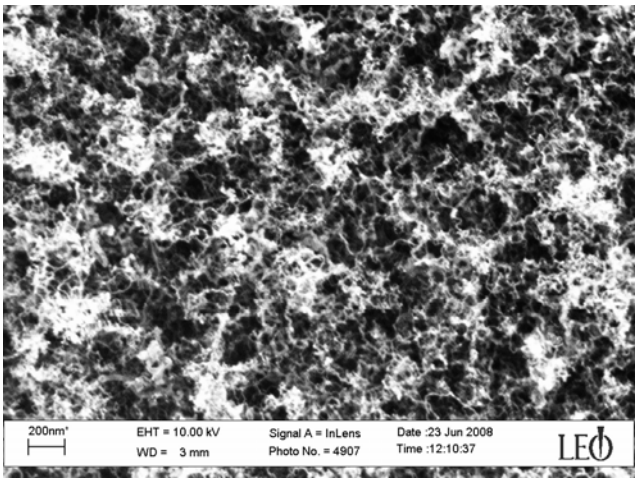

Fig. 2: SEM picture of the synthesized CNT.

observed by X-ray diffraction (XRD) for determination of the crystalline phases and X-ray reflectivity (XRR) to monitor the thin film parameter [3]. At 200°C the XRR signal in Fig 1 left displays a slightly increasing film thickness due to the reduction of an amorphous surface oxide layer of approx. 0.7 nm. Also the surface smoothens significantly proven by the more pronounced oscillations in XRR. Between 200-250°C the signal vanishes, the film has completely dewetted. At this temperature the bragg reflection of Ni is visible at first time, the increasing intensity in XRD by decreasing FWHM clearly show the ongoing nickel crystallization at higher temperatures. By this process the size of the emerging crystallites is around 9 nm. Upon acetylene exposure at 550°C XRD indicates the formation of CNT (graphite diffraction reflection at 18°2theta, not shown) and Ni_3C accounting on the Ni phase. As-grown CNT in figure 2 show an acceptable distribution in its diameter at a low yield. The dewetting temperature strongly depends on the metal film thickness. With increasing layer thickness the interface interactions with the buffer layer are less pronounced and the dewetting temperature increases. The CNT yield, thickness and morphology correlate with the metal particle size and therefore with the pristine layer thickness. Further experiments were planed to elucidate the growth mechanism of CNTs and the role of Ni_3C in it. Also open is the question if Ni_3C and not Ni act as catalyst in this growth process.

References

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