ROBL-CRG	**Experiment title:** In-situ XRD/XRR of thin film catalysis during carbon nanotube growth	**Experiment number**: **20_02_671**
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Results

Metal nano particles like Fe or Ni can act as catalyst in the carbon nano tube (CNT) growth process [1,2]. These metals were first deposited as thin film onto Si-wafer with SiO_2 or Al_2O_3 buffer layers and then heated up under inert or reducing atmosphere. Hereby the film dewets and the nano particles are formed. The crystal sizes of the

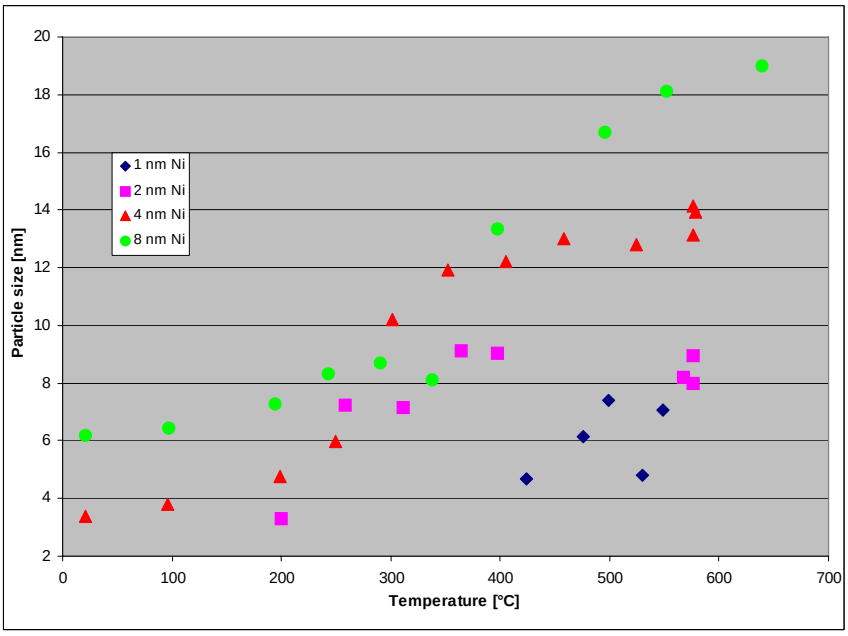

Fig.1: Temperature dependance of the Ni nano-crystallite sizes during heating and the subsequent dewetting.

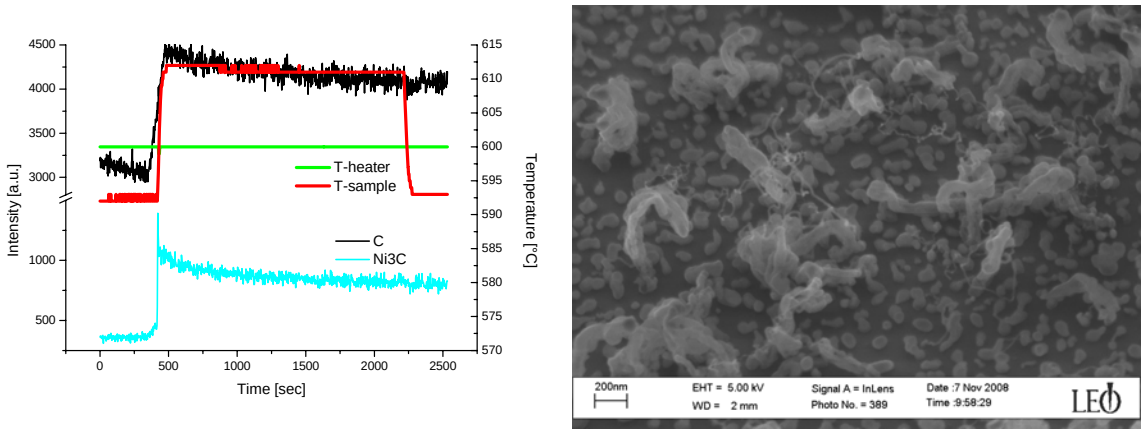

Fig. 2: Timeresolved measurement of the Ni_3C and C signal and the corresponding SEM picture (rigth).

resulting nano-particle depend on the thickness of the initial film. A crude approximation shows that the particles became three times bigger than in the film.

During the growth experiment using 2 or 4 nm thick Ni film system a second phase Ni_3C is observed. This raises the question if this could be the catalytic active material. Time resolved XRD in Figure 2 monitoring the Ni_3C and graphite signal shows the Ni_3C formation directly after the H_2C_2 injection, which act as carbon source. With a slight delay the C signal also increases. Both signal are stable after approx. 10 min. The increased sample temperature during the H_2C_2 feed indicates an exothermic reaction. Based on the corresponding SEM picture the C-signal can be attributed to the low yield CNT but also on surface coking carbon. Further experiments to elucidate this aspect were planed in future.

References

[1] Surface Diffusion: The low activation energy path for nanotube growth, Hofmann S., Csányi G., Ferrari A. C., Payne M. C., Robertson J., Phys. Rev. Lett. 95, 036101 (2005).

[2] In-situ observations of catalyst dynamics during surface bond carbon nanotube nucleation, Hofmann S., Sharma R., Ducati C., Du G., Mattevi C., Cepek C., Cantoro M., Pisana S., Parvez A., Ferrari A. C., Dunin-Borkowski R., Lizzit S., Petaccia L., Goldoni A., Robertson J., Nano Lett. 7, 602 (2007).