Initiation of Carbon Nanofiber Growth on Polycrystalline Nickel Foam under Low Ethylene Pressure


The initiation of carbon nanofiber (CNF) growth on polycrystalline Ni foam was investigated by using a combination of ex situ and in situ methods, which include SEM, XRD, and Raman spectroscopy. Experiments were performed at a low hydrocarbon partial pressure to slow down the initiation process. Very little to no CNFs were observed on reduced samples, which is caused by diffusion of C to the bulk of the Ni foam. This prevents the formation of Ni3C as a precursor of Ni nanoparticles at a low hydrocarbon partial pressure from acting as active particles for CNF formation. CNF growth was significant on oxidized samples and the initiation was slowed down by using an extremely low ethylene pressure. Ni nanoparticles are able to catalyze CNF growth, provided these are isolated from the Ni bulk by unreduced NiO, which results from the incomplete reduction of the NiO layer.

Introduction

Carbon nanofibers (CNFs) and carbon nanotubes (CNTs) are a new class of materials that are studied for various applications such as hydrogen storage,[1] heat transfer,[2] electrodes for fuel cells,[3] hydrophobic surfaces,[4] and catalyst supports.[5] Although CNTs consist of rolled-up sheets of graphitic carbon, CNFs can consist of amorphous carbon or stacks of graphitic carbon in which graphitic layers are arranged in so-called fishbone or platelet structures.[6]

CNF and CNT growth is generally achieved using arc discharge,[1, 7] catalytic chemical vapor deposition (C-CVD),[8] and plasma-enhanced chemical vapor deposition.[8f, 9] Depending on the growth conditions, that is, temperature and pressure, as well as the catalyst (type of metal and morphology[10]) and the reactive gas used as the carbon source,[10, 11] the formation of CNFs or CNTs is favored.

C-CVD on catalysts with preformed metal nanoparticles has been studied in detail for different metals,[11] that is, Ni, Fe, and Co.[6, 12] Additionally, studies on thin metal layers on flat model supports have been reported[8k, 13] in which the thin layer first fragments to form metal nanoparticles just before the CNFs start to grow. In situ TEM[14] experiments have been reported on CNF growth on preshaped transition-metal particles as well as on thin layers of transition metals. From these observations, a generally accepted picture of CNF growth was developed that involves three main steps[10] that basically consist of the decomposition of the carbon that contains gas on the metal catalyst particle, carbon diffusion through or over the surface of the metal particle, and finally, carbon precipitation at a specific side of the particle.[6, 10]

In contrast to the rich literature on CNF and CNT growth on small metal particles, insight into the formation, and especially the initiation, of CNF growth on polycrystalline bulk metal samples is lacking to an important extent. CNF growth on bulk metal has been studied on, for example, Ni,[11] Fe,[8d] and stainless steel,[8d] with different shapes and morphologies, which include foams,[15a] filters,[5a] and foils.[8d] A wide range of parameters have been studied for these materials, which includes the type of carbon-containing gas (C2H4, C2H2, CH4, C3H8, CO+H2),[15] the growth temperature (440–1000 °C),[5b] and pre-treatment of the material (oxidation, reduction, combinations of these).[8d]

CNF and CNT growth on polycrystalline bulk metal catalysts is essentially different from the case of supported nanoparticles because the micrometer-sized grains of the polycrystalline surface have to first break up into smaller nanoparticles to enable CNF/CNT growth. In the case of growth at atmospheric pressures, it has been shown that carbon diffuses into the Ni bulk and accumulates at grain boundaries and defects. Precipitation-induced disintegration can occur in a relatively low-carbon-activity environment to result in particles of the same size as crystallites in the original material, which are usually still too large to catalyze CNF and CNT formation directly.[5b] Thus, further fragmentation is required to form Ni particles.
that are small enough to catalyze the subsequent growth of carbon filaments, which can be induced by an environment with higher carbon activity. Both these processes result in corrosive degradation that is known as metal-dusting.\textsuperscript{[16]}

Previously, Jarrah et al. studied CNF growth initiation on reduced bulk Ni foams as a function of exposure time to a mixture of 25\% $\text{C}_2\text{H}_4$ in $\text{N}_2$ at atmospheric pressure by using ex situ SEM and XRD.\textsuperscript{[5b, 15a]} It was postulated that CNF growth starts with the formation of metastable $\text{Ni}_3\text{C}$, which decomposes subsequently into Ni particles and carbon precipitates. The resulting Ni nanoparticles have the appropriate dimensions (20–70 nm) to catalyze CNF growth. From this, a new type of catalyst support was developed (hairy foam) that consists of a thin layer of entangled CNFs on the surface of Ni foam with an extraordinarily high porosity and low tortuosity. These support materials allow very efficient internal mass transport, which has been demonstrated with Pd supported on hairy foam for the catalytic hydrogenation of nitrite in the aqueous phase.\textsuperscript{[17]}

A similar study on growth initiation by using ex situ characterization was impossible on oxidized Ni foam because of the very rapid formation of CNFs. The presence of a NiO layer increases both the initiation rate as well as the rate of CNF formation by one order of magnitude compared to slower formation on reduced metallic Ni substrates. It was proposed that the reducing conditions for the growth of CNFs first cause the reduction of the nickel oxide layer and, consequently, the in situ formation of Ni nanoparticles, which facilitates the growth of CNFs much more rapidly than growth by the formation and decomposition of Ni$_3$C.

The goal of this work is to determine the mechanism of the initiation of catalytic CNF growth on reduced polycrystalline Ni as well as to confirm or challenge the proposed mechanism of initiation of CNF growth on polycrystalline Ni covered with a nickel oxide layer. In situ characterization during CNF growth initiation was performed by using environmental scanning electron microscopy (ESEM) in mixtures of $\text{C}_2\text{H}_4$ and $\text{H}_2$ at pressures between 10 to 100 Pa. Ex situ techniques, that is, Raman spectroscopy, SEM, and XRD, were used for characterization after the exposure of Ni foam under atmospheric pressure to highly diluted gas mixtures with similar partial pressures of $\text{C}_2\text{H}_4$ and $\text{H}_2$ as in the ESEM experiments.

\section*{Results and Discussion}
\subsection*{Growth on metallic Ni foams}
Reduced Ni shows slight morphological changes upon exposure to diluted feed under atmospheric pressure for several hours (Figure 1 b–d). The dominant change observed with the increase in exposure time is the formation of carbon deposits or precipitates, which give rise to islands of particularly dark contrast in the SEM images. A very small amount of CNFs can be observed only after prolonged exposure to ethylene after 15 min and 3 h (Figure 1 c and d).

Similar observations were made by using ESEM. The exposure of the reduced Ni foam to both pure $\text{C}_2\text{H}_4$ (Figure 2) and mixtures of $\text{C}_2\text{H}_4$ and $\text{H}_2$ (Movie S1 in the Supporting Information) at pressures between $10^{-2}$ and 100 Pa for extended times of up to several hours resulted in the formation of carbon precipitates on the surface of the foam. Carbon deposits become visible after the decrease of either the temperature or ethylene pressure, which induce the segregation of dissolved carbon to the surface of the Ni foam. No CNF formation on the reduced Ni foam was observed by using ESEM.
In summary, the exposure of reduced Ni to dilute C_2H_4 and H_2 under atmospheric conditions results in the growth of scattered CNFs after a prolonged exposure time and the formation of carbon precipitates on the surface if the exposure time is sufficiently long. This finding is in line with observations reported by Weatherup et al.\(^{[18]}\). This is in clear contrast to the results of Jarrah et al.,\(^{[5b]}\) which showed that CNF can be grown on reduced Ni at a significantly higher ethylene pressure.

**Growth on oxidized Ni foams at atmospheric pressure**

**Oxidation of Ni foam**

The effect of oxidative pretreatments on the surface morphology is shown in Figure 3. The as-received Ni foam at two different magnifications with a very thin layer of NiO on the surface that results from exposure to the ambient atmosphere is shown in Figure 3 a and b. The as-received Ni foam consists of Ni grains of approximately 1–10 \(\mu m\) in size. Significant surface structure differences are observed after oxidation for 1 h at 600 °C (Figure 3 c) and 700 °C (Figure 3 d). Oxidation at 600 °C results in a heterogeneous layer of NiO particles of approximately 30 nm estimated from XRD line-broadening (see below). This sample contains 8.5 wt\% NiO as determined by thermogravimetric analysis (TGA; Figure S1). Unfortunately, the very low surface area of the foam cannot be determined experimentally easily. Therefore, it is estimated to be approximately 0.03 m^2 g\(^{-1}\) if we assume that the foam consists of cylindrical Ni strands typically of 16 \(\mu m\). From the bulk density of NiO (7.78 g cm\(^{-3}\)), it can be estimated that this corresponds to a NiO layer of approximately 500 nm. This estimated NiO layer thickness indicates that the NiO layer is polycrystalline as the crystallite size is significantly smaller. Oxidation at 700 °C results in a more homogeneous coverage of the surface with larger and structurally more defined NiO crystals, with a NiO content of 9.2 wt\% by using TGA.

**Growth on oxidized Ni foams at atmospheric pressures (ex situ)**

Exposure to ethylene/hydrogen in N\(_2\) gives rise to CNF growth on the surface of the oxidized Ni foam, although no CNFs are visible after 1 min (Figure 4 b). Some CNFs are clearly visible after 15 min (Figure 4 c), whereas the structure of the Ni surface flattened slightly because of continued reduction and slight sintering. A further increase of the growth time leads to an increase of CNF growth, and the morphology of the foam surface does not show significant changes (Figure 4 d and e). The resulting CNFs have diameters that range from a few nm to approximately 35 nm.

We used CHN analysis to show that CNF growth on Ni oxidized at 600 °C results in a C content of 2.9 wt\% after 27 h of growth. Notably, the C concentration of the oxidized sample (Figure 3 c) before CNF growth is below the detection limit (\(< 0.1\) wt\%). Further experiments were performed with samples oxidized at 600 °C.

XRD patterns with a magnification of the 2\(\theta\) region centered around the main isolated diffraction peak of nickel oxide at \(2\theta = 43.3^\circ\) are shown in Figure 5. The Ni and NiO peaks at approximately 2\(\theta = 44.6\) and 51.8° overlap too closely to show
clear differences. Oxidation at 600 °C for 1 h causes the formation of a NiO layer (Figure 5, curve b) that contains particles of approximately 30 nm, as estimated from the peak width of the NiO diffraction peak using the Scherrer equation. This is in reasonable agreement with the NiO structures observed in Figure 4a. The oxidized Ni foam as well as the sample after 1 min of CNF growth contains NiO according to the diffraction peak at $2\theta = 43.3^\circ$, whereas CNF growth over 27 h reduces NiO completely, that is, to a level below the detection limit by using XRD. There is no sign of the formation of any Ni3C, which would induce diffraction peaks at $2\theta = 39.1$ and $41.6^\circ$, as was observed previously using significantly higher ethylene concentrations.[15a]

Raman spectra of the Ni foam after oxidation and after 1 and 15 min of CNF growth are shown in Figure 6. The peak at $\tilde{\nu} = 520$ cm$^{-1}$ is the most pronounced NiO peak [19] that is well separated from the peaks assigned to graphitic deposits as detected in the other two spectra. This NiO peak is clearly detectable on the sample after the growth of CNFs during 1 min, although the intensity decreased significantly. The spectra obtained after CNF growth for 1 and 15 min show double peaks at approximately $\tilde{\nu} = 1500$ and 2800 cm$^{-1}$ attributed to graphitic material and characteristic for CNFs.[20] The spectrum measured after CNF growth for 1 min demonstrates clearly that CNF growth is already initiated before NiO is completely reduced.

Although after 1 min of CNF growth, CNFs are not visible yet by using SEM (Figure 4b), Raman spectroscopy shows that there is already graphitized carbon present, indicative of CNFs. We used both Raman spectroscopy and XRD to confirm the presence of nickel oxide after the growth of CNFs for 1 min. We used Raman spectroscopy to show a clear decrease in the oxide content compared to that of initial nickel oxide sample, whereas the use of XRD shows a similar NiO content. As Raman spectroscopy is more surface-sensitive than XRD, this confirms that the surface of the NiO is reduced first, as expected, simultaneously with the formation of the first CNFs.

In situ growth on oxidized Ni foams examined by using ESEM

In situ oxidation

The use of ESEM enables the in situ observation of the oxidation process (Figure 7). Notably, the resulting surface features are similar to the surface features obtained under atmospheric pressure, although oxidation examined by using ESEM results in a more uniform coverage. This might well be caused by the difference in the O$_2$ pressure, that is, 4000 Pa in the atmospheric experiments versus 40 Pa in ESEM, or differences in the prereduction treatment in the atmospheric and ESEM experiments.

In situ growth on oxidized Ni foams examined by using ESEM

A switch from oxidizing conditions to conditions for CNF growth was performed by first cooling the sample to room temperature, exchanging the oxygen with 8 Pa C$_2$H$_4$/20 Pa H$_2$ mixture, and then heating back to 600 °C. As a result of the high drift of the sample during the heating step, no undistorted scanning images could be recorded during heating until the sample reached the final temperature and the drift.

Figure 5. XRD patterns of Ni foam (left) and magnification of the NiO peak at $2\theta = 43.3^\circ$ a) as-received, b) after oxidation (600 °C, 1 h, 4% O$_2$), c) after 1 min of CNF growth, and d) after 27 h of CNF growth (right). Both growth experiments were performed at 440 °C with partial pressures of 50 Pa for C$_2$H$_4$ and 100 Pa for H$_2$.

Figure 6. Raman spectra of oxidized Ni foam (600 °C, 1 h, 4% O$_2$, solid line) and after subsequent CNF growth under 50 Pa C$_2$H$_4$ + 100 Pa H$_2$ for 1 min (dashed line) and 15 min (dashed-dotted line).
stopped. The unfortunate consequence is that the initiation cannot be observed directly. Changes in the surface and the formation of Ni nanoparticles are shown in Movie S2. At the same time, some surface movement caused by CNF cluster growth can be observed. The formation of some individual CNFs is visible in real-time at intermediate magnification (Movie S3). TEM observation confirmed the growth of carbon nanofibers (Figure S2).

Part of an oxidized Ni foam is shown in Figure 8a, and the same sample after reduction is shown in Figure 8b, in which slight morphological changes can be observed. CNFs are observed in Figure 8c (top view) as well as in the side-view at a higher magnification (Figure 8f). The cross-section view, obtained by cracking the foam mechanically, reveals a brighter layer underneath the darker top-layer that is covered by a carpet of CNFs. We used energy-dispersive X-ray spectroscopy (EDX) to show that the bright layer is caused by nickel oxide.

General discussion

As expected, the extremely low ethylene concentration used in this study retarded the formation of CNFs as compared to the previous study on the same materials by Jarrah et al.\cite{15a} under much higher ethylene concentrations. Nevertheless, the enhancement of NiO on CNF formation is observed at a low concentration, similar to the previous results at high ethylene concentration. Reduced samples show CNFs at low ethylene concentration only after very long exposure time, whereas no CNF growth was observed by using ESEM (not shown).

We used ESEM to reveal that carbon diffuses into the Ni bulk (Figure 2b and Movie S1). Carbon deposits are not visible during exposure to 100 Pa ethylene within the time during which in situ observation in the ESEM was performed. However, carbon segregates to the surface after the decrease of the temperature or decrease of the ethylene pressure in the ESEM chamber, which demonstrates that exposure to ethylene caused significant carbon dissolution.

CNF growth on reduced Ni foam

CNF growth on reduced Ni foam in this study is observed after 15 min of growth; the amount of CNFs is very small and the surface contains only scattered CNFs even after 27 h. This demonstrates a slow initiation at a low ethylene concentration (50 Pa) compared to the observation of massive CNF growth under 25 000 Pa ethylene reported previously by Jarrah et al.\cite{15a}

A possible cause for this phenomenon is the lack of the formation and subsequent decomposition of Ni$_2$C as proposed by Jarrah et al.\cite{15a} based on the detection of such a metastable phase by using XRD and SEM. This result was obtained if we exposed the reduced Ni foam to 25 000 Pa ethylene. Apparently, the same mechanism does not occur significantly under 50 Pa ethylene pressure. As the in situ SEM data confirm the clear dissolution of C in Ni under the conditions used in this study, it is clear that ethylene decomposes on the Ni surface to generate C. It seems reasonable to assume that the decomposition reaction is slow at a low ethylene pressure, and we speculate that under these conditions the diffusion of C into the bulk of the Ni foam is so fast that Ni$_2$C cannot form on the surface of the Ni foam. Hence, the initiation of CNF formation is suppressed strongly.
**CNF growth on oxidized Ni foam**

Clearly, the initiation of CNF growth can be observed on oxidized Ni if the growth is tempered by decreasing the ethylene pressure. This is in clear contrast to earlier results at high ethylene pressure obtained by Jarrah et al.\[^{15a}\] who reported extremely fast CNF growth. Only massive CNF growth could be observed at a high pressure, even after a very short exposure to ethylene/hydrogen mixtures, and no information could be obtained on the initiation process. Our investigations demonstrate that CNF growth is initiated while NiO is still present. For atmospheric tests, Raman spectroscopy shows the presence of NiO as well as CNFs (graphitized carbon) after 1 min CNF growth (Figure 6). We used XRD to confirm the presence of NiO after 1 min CNF growth (Figure 5). Our ESEM results with EDX measurements show clearly that a NiO layer is still present after CNF growth was initiated (Figure 8e and f). The average thickness of the initial NiO layer is in the order of 500 nm, according to the results of TGA. This is in line with the original hypothesis\[^{15a}\] that the reduction of NiO provides a fast route to form Ni nanoparticles. The new observations allow us to further detail the effect of the NiO layer.

A first explanation assumes that NiO is responsible for the prevention of C diffusion from the Ni nanoparticles to the bulk of the Ni foam. Ni particles grow on top of the NiO layer at the external surface at which H\(_2\) is offered. If the growing Ni nanoparticles are isolated from the Ni bulk by the NiO layer, C diffusion to the bulk is not possible. Therefore, the carbon concentration in the isolated Ni nanoparticles can increase to allow the initiation of CNF growth.

Alternatively, it can be assumed that the presence of a NiO layer prevents the sintering and merging of in situ formed Ni nanoparticles with the polycrystalline bulk by the separation of the Ni nanoparticles from the bulk. Ni nanoparticles need to be small to enable CNF growth. The sintering and merging of the Ni nanoparticles with the bulk of the polycrystalline Ni is detrimental to CNF growth. The isolation of the small Ni nanoparticles by the NiO layer results in CNF growth similar to growth on preshaped nanoparticles supported on, for example, alumina, silica, and carbon.

The critical size of the Ni nanoparticles for CNF formation is in the order of tenths of nm based on the diameters of the resulting fibers. Apparently, a 500 nm NiO layer is able to induce CNF growth, whereas native oxide layers fail. Clearly, the thickness of the NiO layer (500 nm) needs to be significantly larger than the size of the Ni nanoparticles (typically 50 nm) that grow CNFs to isolate the nanoparticles from the bulk. Our observations support both explanations, and at this time it is not possible to decide if one of the hypotheses is dominant, or possibly both effects are necessary to induce CNF growth.

**Conclusion**

Carbon nanofiber (CNF) growth is slowed down and the initiation is retarded by using extremely low ethylene concentrations. Reduced samples show few CNFs at a low ethylene concentration after a long exposure time, or not at all by using environmental scanning electron microscopy. This is attributed to the diffusion of C to the bulk of the Ni foam, which prevents the formation of Ni\(_3\)C as a precursor in the formation of Ni nanoparticles. On oxidized samples, CNF growth is initiated if NiO is still present to isolate the Ni nanoparticles that form during the reduction of the NiO layer from the bulk Ni. This isolation prevents C diffusion to the bulk and/or inhibits the sintering of the Ni nanoparticles with the polycrystalline Ni in the foam.

**Experimental Section**

**Materials**

The Ni foam used for this study was obtained from RECEMAT bv.\[^{21}\] This foam consists of hollow strands of Ni that are typically 15 mm thick (Figure 9). The foam is highly porous (typically 95 %) with typical pore sizes of 0.4 mm. The specific surface area of the Ni is 5400 m\(^2\) m\(^{-3}\). The Ni foam is 99.5 % pure and contains traces of Fe (0.2 %), Cu (0.1 %), and Zn (0.1 %). Cylinders with a diameter of 4.3 mm and length of 5 mm were cut from the as-received foam sheet by using electrical discharge machining.

Ethylene/nitrogen (1000 ppm C\(_2\)H\(_4\) in N\(_2\), Praxair), H\(_2\) (99.999 %, Linde), compressed air (in-house production), and N\(_2\) (99.999 %, Linde) were used for carbon nanofiber growth and pretreatment of the foam.

**Pretreatment**

As-received Ni foam was cleaned in acetone by ultrasonication for 15 min. In the case of atmospheric growth experiments, metallic foams were additionally pretreated by in situ reduction at 440 °C.

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Figure 9. a) Optical and b, c) SEM images of the as-received metallic Ni foam sample at different magnifications.
for 1 h in 20 vol% H₂ in N₂, right before switching to carbon nanofiber growth. For the growth experiments on oxidized foams, a treatment at 600 °C for 1 h in 4 vol% O₂ in N₂ at ambient pressure was applied. For ESEM experiments, the same Ni foam cylinders were used.

Carbon nanofiber growth

Carbon nanofibers were grown directly on Ni foam cylinders in a home-built quartz reactor with a diameter of 42 mm. The reactor that contained the Ni foam was heated in a vertical furnace under a flow of 100 mL min⁻¹ N₂ with a ramp of 5 °C min⁻¹. The actual growth of CNFs was conducted at 440 °C under atmospheric pressure in a total flow of 100 mL min⁻¹ feed gas that contained N₂, 0.5 vol% C₂H₄ and 1 vol% H₂ over 1 min up to 27 h. The concentrations of C₂H₄ and H₂ were chosen such that the partial pressures of, respectively, 50 and 100 Pa were in the same range as those used in the ESEM experiments. CNF growth was stopped by flushing the reactor with N₂. Samples were allowed to cool to RT before exposition to ambient air. These experiments are termed “atmospheric” experiments.

For the real-time observation by using ESEM, a FEI Quantum 200 instrument with a field emission gun, oil-free vacuum prepumps, and a home-built laser heating stage was used. The instrument was equipped with a set of mass-flow controllers that allow the introduction of the desired amounts of gas mixtures directly into the chamber of the microscope. In the high-vacuum operation mode, the instrument reaches a base-pressure of approximately 5 × 10⁻¹³ Pa. Typically, the chamber was purged and pumped several times with N₂ after the introduction of the sample. All samples were annealed initially under 20 Pa H₂ for 15 min to 1 h at 600 °C to remove surface oxides and carbon contamination. In the case of experiments on oxidized foams, the samples were oxidized under 30 Pa O₂ for different times. Each time before the gas composition was changed, the sample was cooled to RT. After the gas atmosphere was changed, the sample was reheated to the desired temperature. The composition of the chamber atmosphere was monitored by using a mass spectrometer that is connected directly to the chamber. For CNF growth, the atmosphere was set to 8 Pa C₂H₄ and 22 Pa H₂. The sample was heated by direct illumination by using IR laser light (λ = 800 nm). A K-type thermocouple was inserted into the foam to measure the temperature of the foam directly. The temperature can be changed at relatively fast rates in the range of several 100 °C min⁻¹ because of the small mass of the heated sample. The laser heating current was controlled manually by feedback from the thermocouple. To reach the desired experimental conditions as fast as possible and to reduce the time during which observation is hindered by thermal drift, temperature changes were applied at rates of several 10 °C s⁻¹.

Characterization

The atmospheric samples were analyzed and characterized ex situ, that is, after exposure to ambient air, by using high-resolution scanning electron microscopy (HR-SEM), XRD, Raman spectroscopy, TGA, N₂ adsorption, and elemental analysis. HR-SEM pictures were obtained by using a Zeiss Merlin Scanning Electron Microscope equipped with an EDX detector. Statistical analysis of the diameters of the produced CNFs was conducted by analyzing HR-SEM pictures using ImageJ.

XRD patterns were recorded by using a Panalytical X'Pert PRO operated with a Cu source. Raman spectra were recorded by using a Bruker Senterra instrument that is equipped with an Infinity 1 camera using an excitation wavelength of 532 nm (5 mW). Spectra were averaged from five spots to compensate for any inhomogeneity of the sample with 20 individual spectra per spot and an accumulation time of 2 s. TGA was performed by using a TGA/SDTA851e (Mettler Toledo). The surface area was determined by using N₂ physisorption by using a QuantaCharm Autosorb-1 using the BET isotherm with multiple samples because of the low absolute surface area. CHN analysis was performed by using a Flash 2000 Organic Elemental Analyzer (Interscience) by repeating the measurement five times and averaging the result.

Additional characterization of samples grown in the ESEM was done ex situ by using a Hitachi S4800-SEM and a JEOl ARM transmission electron microscope. EDX was recorded by using ESEM (FEI Quantum 200) using a Bruker Si(Li) EDX detector.

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Conflict of interest

The authors declare no conflict of interest.

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Fiber initiation, fiber synthesis: The initiation of carbon nanofiber (CNF) growth on polycrystalline Ni foam is investigated by using a combination of ex situ and in situ methods. Experiments are performed under a low hydrocarbon partial pressure to slow down the initiation process. Ni nanoparticles are able to catalyze CNF growth under these conditions, provided they are isolated from the Ni bulk by unreduced NiO.