Detection of Airborne Carbon Nanotubes via Embedded Nickel Catalysts in Quasi-Real Time

N. Neubauer[1] and G. Kasper[1]

[1]Karlsruhe Institute of Technology (KIT), Institute for Mechanical Process Engineering and Mechanics, Karlsruhe, 76131, Germany
Keywords: carbon nanotubes, nickel, CAAM, quasi-real time
Presenting author email: nicole.neubauer@kit.edu

Workers involved in the production and handling of carbon nanotubes (CNT) are likely to be at a high exposure risk. As CNT are supposed to be harmful to human health, their detection in the workplace air is highly desirable. In a recent publication (Neubauer et al., 2011) we introduced a new measurement technique for the material-specific detection of catalytically active nanoparticles (e.g. nickel) with a high sensitivity. Based on this concept we have developed a compact and portable laboratory prototype which we call the Catalytic Activity Aerosol Monitor (CAAM; Figure 1, left side) (Neubauer et al., 2013). In this study, this new type of detector is investigated with respect to its capability to detect airborne CNT aerosols via embedded nickel catalysts.

Experimental

As a catalyst for the generation of CNT, a nickel aerosol was produced by spark discharge. The addition of acetylene and hydrogen at 600°C led to an on-line formation of CNT by aerosol chemical vapour deposition (aerosol-CVD). For the detection by the CAAM, defined volumes of both the CNT and the nickel aerosol were sampled onto filters. After sampling was completed, pre-mixed and pre-heated gaseous reaction educts were added initiating a catalytic reaction. As model reactions for the detection of CNT via embedded nickel catalysts, the hydrogenation and the oxidation of CO were chosen. The composition of the reaction gases was measured by an IR sensor, hereby providing a measure for the catalytic activity of the nickel particles. In addition, both the nickel and the CNT aerosol were characterized by electrical mobility spectrometry, electron microscopy (TEM) and thermogravimetry.

Results

Electron microscopy analysis resulted in a median primary particle size of 3.5 nm for the nickel nanoparticles. They are arranged in agglomerates with a mode of the mobility equivalent diameter of 74 nm. Figure 1 (right side) shows a TEM image of CNT with embedded nickel catalyst nanoparticles.

While the mass of the pure nickel particles was stable during thermogravimetry (20-950°C), the CNT evaporated at a temperature of 420°C. However, this temperature is higher than the required reaction temperature of 350°C for both the CO-hydrogenation and the CO-oxidation. With this the CNT did not evaporate during CAAM analysis.

The catalytic experiments showed a proportional dependence of the catalytic activity on the sampled volume for the pure nickel nanoparticles. As no catalytic signal could be detected for the CNT aerosols, the embedded nickel nanoparticles seemed to be not accessible for catalysis. However, if the CNT were burnt away in an intermediate step by adding a mixture of oxygen and nitrogen at 500°C to the sampled CNT, the nickel nanoparticles could be detected again by the CAAM.

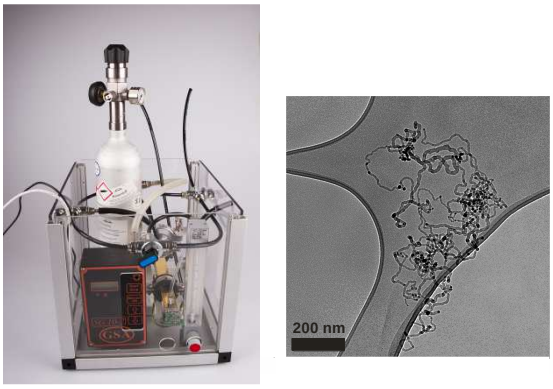

Figure 1. Left side: Catalytic Activity Aerosol Monitor (CAAM), Right side: TEM image of CNT with embedded nickel catalyst nanoparticles

Conclusion

An intermediate step of CNT burning between aerosol sampling and the catalytic reaction enables the detection of formerly embedded catalyst nickel nanoparticles by the CAAM. With this, the CAAM technique also allows the detection of CNT aerosols in workplace air in quasi-real time.

References

Neubauer N, Weis F, Binder A, Seipenbusch M and Kasper G. (2011) *Journal of Physics: Conference Series 304 (1)*

Neubauer N, Seipenbusch M and Kasper G. (2013) *Annals of Occupational Hygiene (accepted)*

The research leading to these results has received funding from the European Commission's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 211464-2 (Nanodevice).