# Particle characterization during abrasive treatment of composite material containing fibres by Cryo HRTEM

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Keywords: nano fibre composite, production emission, HRTEM, cryo.
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Composite material including nano particles and fibres is today used in production and sealing of surfaces. Highly stressed surfaces such as of cars, boats and light aircrafts need to provide properties that demand the use of new materials including nano fibres, that enhance stability. Manufacturing of these surfaces involves abrasive treatment, during which material is emitted. We characterized particles from treatment of composite material including nano fibres. Nano particles can have an adverse health effect (Donaldson et al., 2002) when inhaled by humans exposed to emissions during manufacturing and surface treatment.

Particles were collected using a micro intertial impactor set similar to the one described by Kandler (2009). Sampling was performed on 2 stages (upper cut off 0.89 µm and lower cut off 0.047 µm diameter) on Ni-TEM grids with Formvar carbon foil (Plano, Wetzlar, Germany).

Transmission electron microscopy (TEM) was used for analysis of particles on 12 samples from 6 tests in total. Overview and high-resolution (HR) microscopic images were recorded using a Tecnai T20 G2 (FEI, Eindhoven, the Netherlands) at the Center for Electron Nanoscopy, Technical University of Denmark, operating at 200kV. Due to beam sensitive material a nitrogen cooled cryo transfer holder (Gatan Inc. Ca. USA) was used to keep the sample below -80 degrees Celsius during analysis. It was accounted for possible beam damage and hydro carbon deposition by using low beam current densities and short acquisition time. Energy dispersive X-ray detection (EDS) was carried out on single particles using an Oxford 80mm silicon drift detector.

Scanning electron microscopy (SEM) was performed on samples collected from deposited material (powder) with a ESEM Quanta 200 FEG (FEI, Eindhoven, the Netherlands).

Online measurements with FMPS, ELPI, CPC and GRIMM dust monitor were conducted in parallel.

Particles in the size range from 40 nm up to 500 nm (average geometric diameter) were analysed and classified based on their structure, morphology, beam stability and chemical composition.

Particles originating from the composite proofed to be partly crystalline material (Figure 1). Few nanometer thin fibres were found in agglomerates of few hundred nanometers, parts of them sticking out of the bulk. Single fibers of 100 nm in width and several 100 nanometers in length were less abundant.

Ammonium sulfate, sodium chloride and soot particles that were found in high abundances on the substrates were classified as background particles. However, composite particles, as well as fibres were sometimes found internally mixed with background particles.

Particles revealed inhomogeneous composition and irregular shape and morphology. Size, surface area and density determination will be discussed for the different materials.

SEM investigations on deposited material revealed extreme charging of particles, especially of the fibrous material. Potential issues regarding the detection by instruments using electric mobility sizing will be critically discussed.

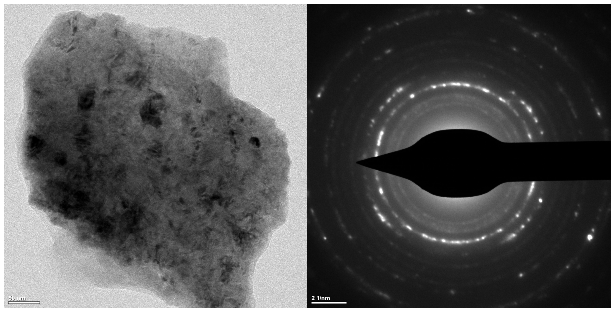

Figure 1. TEM bright field image (left) and diffraction pattern (right) of a nano particle originating from abrasive treatment of composite material.

The authors like to thank Signe Hjortkjær Hansen for technical support. We gratefully acknowledge excellent microscopy conditions at Center for Electron Nanosccopy (CEN), Technical University of Denmark and technical support by Wilhelmus Huyzer. This work was supported by the NanoSafetyCenter and Nanovalid.

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