Emission Monitoring in the Production of SiC Nanoparticles

by Induction Plasma Synthesis

Jelena Buha1,2, Drew Thompson3, Marc Leparoux4, Christian Jäggi4, David Y.H. Pui3 and Jing Wang1,2

1Institute of Environmental Engineering, ETH Zurich, 8093 Zurich, Switzerland;
2Analytical Chemistry, Empa, 8600 Dubendorf, Switzerland;
3Particle Technology Laboratory, University of Minnesota, 55414 Minneapolis, USA;
4Laboratory for Advanced Materials Processing, Empa, 3602 Thun, Switzerland

Safety of nanomaterials is of growing concern as it becomes more common for consumer goods to utilize them. Key to evaluating the potential health risks posed by nanomaterials is the nature of inhalation exposure to airborne engineered nanoparticles. This is not only needed for establishing safer nanomaterial work practices, toxicology studies also require doses relevant to actual workplace exposures. The few reports[1, 2] of workplace exposures suggest that engineered nanomaterials are released at high mass and/or number concentrations only under unusual circumstances. However, there still exists the need for study of exposure levels to different engineered nanomaterial types in occupational and environmental settings.

In this study aerosol emissions were monitored at a research laboratory during the production of silicon carbide (SiC) nanoparticles by inductively coupled plasma (ICP) synthesis. Regarding the emission studies, the filtration unit was used to collect small quantities of nanoparticles on the surface of a filter. The filter cartridge contained metal filters which were backpulsed to remove caked nanoparticles. This system was used to collect larger quantities of nanoparticles. Further on, real-time aerosol instruments were used to monitor particle concentration and size.
distribution. By looking for relative changes in size and concentration, while also considering particle sources, it was possible to monitor a nanoparticle release. Instrumentation that was included was: fast mobility particle sizer (FMPS), handheld condensation particle counter (CPC), nanoparticle surface area monitor (NSAM) and aerodynamic particle sizer (APS). Area sampling with track-etched polycarbonate filters was done for particle identification by scanning electron microscopy (SEM).

Figure 1. SEM image of SiC agglomerates sampled during the cleaning of the production filter with a compressed air gun

The use of direct-reading particle instruments was found to be effective. No particles were released during the synthesis of SiC nanoparticles due to the reactor being operated in a closed system under slight vacuum. However, aerosol emissions were identified in other related production tasks. A release of submicrometer particles was detected when a nanoparticle collection filter was disconnected from the reactor system. When compressed air was used for cleaning in open spaces, such as the cleaning of the nanoparticle collection filters in a ventilated
walk-in enclosure, emissions of submicrometer particles were identified and particle number concentrations exceeded 250,000 #/cm³. Particle emissions in this facility were found to be largely submicrometer and exhibited a mode size of approximately 170 nm. Observation of filter samples under scanning electron microscope confirmed that the particles released were agglomerates of SiC nanoparticles (Figure 1).


Introduction

Environmental Health and Safety of nanomaterials are of growing concern as it becomes more common for consumer goods to utilize nanomaterials. For a risk to be posed, both hazard and exposure need to be present. The hazard is determined by toxicology studies. Exposure is determined by exposure assessments. Key to evaluating the potential health risks posed by nanomaterials is the nature of inhalation exposure to airborne engineered nanoparticles. This is not only needed for establishing safer nanomaterial work practices, toxicology studies also require doses relevant to actual workplace exposures. In this study aerosol emissions were monitored at a research laboratory during the production of silicon carbide (SiC) nanoparticles by inductively coupled plasma (ICP) synthesis.

Methods

By looking for relative changes in size and concentration, while also considering particle sources, it is possible to monitor for a nanoparticle release. Following real-time aerosol instruments were used to monitor particle concentration and size distribution:

Fast Mobility Particle Sizer (FMPS) [5.6 – 560 nm];
P-Trak Ultrafine Particle Counter 8725 [1 – 1000 nm];
Handheld Condensation Particle Counter (CPC) [10 – 1000 nm];
Particle Surface Area Monitor (NSAM) [10 – 1000 nm];
Aerodynamic Particle Sizer (APS) [0.5 – 20 μm].

Additionally, area sampling with track-etched polycarbonate filters was done for particle identification by electron microscopy.

Results

In this study the synthesis of SiC nanoparticles in a prototype ICP reactor and supporting processes, such as the handling of precursor material, the collection of nanoparticles, and the cleaning of equipment, were monitored for particle emissions and potential exposure. Task based area sampling was conducted using a suite of direct-reading particle instruments. NSAM was used to measure the particle number concentration and lung deposited surface area, respectively, in the 0.5 to 1 μm size range every second. FMPS and APS were employed to measure particle size distributions. A time series approach was used to distinguish released engineered nanoparticles from the background. It was assumed that concentrations and size distributions measured during no work activity was the background. Most of the measured processes had no emission of the particles, as summarized in Table 1. However, the cleaning of the production filter was a very dusty process. Particle number concentrations measured by the FMPS/P-Trak exceeded 100,000/250,000#/cm³, respectively. The concentrations in the enclosure returned to background levels approximately 5 min after cleaning began. Size distribution measured by FMPS showed the average size of 170 nm. What appears to be a mode at 0.7 μm was thought to only be a result of APS counting inefficiencies.

Table 1. Summary of tasks monitored in the production SiC nanoparticle by ICP synthesis and respective results

<table>
<thead>
<tr>
<th>Task</th>
<th>Duration / Frequency</th>
<th>Engineering control</th>
<th>Personal protection equipment</th>
<th>Task description</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>ICP synthesis</td>
<td>1 h / weekly</td>
<td>Filtering hoods, local exhaust ventilation</td>
<td>Lab coat, nitrile gloves, safety glasses</td>
<td>Enclosing hood</td>
<td>Handling of powder precursor in enclosing hood</td>
</tr>
<tr>
<td>Reactor open and separated into two sections</td>
<td>1 min</td>
<td>Filtering hoods, local exhaust ventilation</td>
<td>Lab coat, nitrile gloves, safety glasses</td>
<td>Two valve system, LEV</td>
<td>Release of smaller particles from 2 valve system</td>
</tr>
<tr>
<td>1 min Connected to aspiration</td>
<td>Ventilated walk-in enclosure</td>
<td>Supplied air, hood type respirator, Tyvek coveralls</td>
<td>No particle emissions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 min / weekly</td>
<td>Ventilated walk-in enclosure</td>
<td>Filtering facepiece, lab coat, nitrile gloves, safety glasses</td>
<td>Release of smaller particles</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 min / yearly</td>
<td>Ventilated walk-in enclosure</td>
<td>Supplied air, hood type respirator, Tyvek coveralls</td>
<td>Release of smaller particles</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cleaning of online sampling filter w/ compressed air</td>
<td>1 min / weekly</td>
<td>Two valve system, LEV</td>
<td>Filtering facepiece, lab coat, nitrile gloves, safety glasses</td>
<td>Release of 170 nm particles from dead volume in two valve system</td>
<td></td>
</tr>
<tr>
<td>Collection of nanomaterials in online sampling</td>
<td>1 min / weekly</td>
<td>Two valve system, LEV</td>
<td>Filtering facepiece, lab coat, nitrile gloves, safety glasses</td>
<td>Release of 170 nm particles from dead volume in two valve system</td>
<td></td>
</tr>
<tr>
<td>Handling of powder precursor in enclosing hood</td>
<td>1 min / weekly</td>
<td>Enclosing hood</td>
<td>Filtering facepiece, lab coat, nitrile gloves, safety glasses</td>
<td>Handling of powder precursor released both fine (~ 200 nm) and coarse particle emissions</td>
<td></td>
</tr>
</tbody>
</table>

Conclusions

A particle release was not detected during the synthesis of SiC nanoparticles. Particles were released when nanoparticles or nanoparticle contaminated equipment were handled in the open air. Compressed air was found to resuspend nanoparticles when it was used in inadequately ventilated areas. The use of compressed air in workplaces containing nanoparticles should be avoided.

Figure 1: Left: An image of the ICP setup; Right: A photograph demonstrating that the cleaning of the production filter resuspended high concentrations of SiC particles.

Figure 2: Real-time particle measurements of the cleaning of the production filter with a compressed air gun. Top left: FMPS and NSAM number concentrations; Top right: The size distributions measured by FMPS for peak concentration and background conditions. Bottom left: Particle number concentration time series. Bottom right: Peak and background size distributions measured by APS.

The FMPS measured a size mode of 170 nm for the airborne SiC particles for both cleaning of the production filter (Figure 2, top right) and disconnection of the sampling filter (Figure 4, left) from the ICP reactor. What was believed to be a false peak at 10 nm was measured by the FMPS for both cleaning and disconnection of the sampling filter, as shown in Figure 4.