Center for High-rate Nanomanufacturing at the University of Massachusetts Lowell

Test Report

Evaluation of the PCT/A1-Safetech Nanopowder Enclosure

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Process and Method of Evaluation:

The OCT/A1-Safetech powder handling enclosure was evaluated for its effectiveness when handling powders in the nanoparticle size range. The tests were performed using methods developed by Dr. Su-Jung (Candace) Tsai, as published and described below. For each experiment, 100 g of nanoalumina was either poured from beaker to beaker or transferred by spatula. The evaluation was performed at two face velocities, *i.e.,* 73 ft/min and 52 ft/min. Nanoparticle release from the enclosure was evaluated by the relative data of particle concentration change before, during and after handling nanoparticles in the enclosure. Experiments were repeated for operation at each face velocity, and the measurement at breathing zone was repeated at a single operation. The handling method was performed by transferring and pouring 100 g nanoalumina particles from beaker to beaker.

Particle Measurement Methods:

The concentration of airborne nanoparticles for diameters from 5.6 to 560 nm was measured using the Fast Mobility Particle Sizer (FMPS®) spectrometer (Model 3091, TSI), with 32 channels of resolution (16 channels per decade). The FMPS performs particle size classification based on differential electrical mobility classification. Particle concentration and size distribution were recorded every second. Three meter long conductive tubing was connected to the inlet of FMPS to reach the measuring locations.

The FMPS measurements yield detailed information about airborne particle size distribution, but give no information about particle morphology and elemental composition. To provide this information, a new nanoparticle aerosol filter sampler was used in these experiments. Transmission electron microscope (TEM)-copper grids (400 mesh with a carbon film) were taped onto 47 mm diameter polycarbonate membrane filters (0.2 μm pore size). Fiber backing filters were used to support the polycarbonate filters. Air was drawn through the filters at 0.3 L/min using a calibrated personal sampling pump, and aerosol particles deposited on the grid via Brownian diffusion.

Analytical methods:

Normalized particle number concentrations measured by the FMPS were calculated in each size channel based on the average concentration during each measurement time period. The background concentration measured before each experiment was used as the baseline to investigate the relative change in concentration during operation.

Sampled particles were characterized using TEM. TEM images of the samples were taken using a Philips EM400 TEM (Eindhoven, The Netherlands) operated at 100kV.

Location of measurement:

Measuring locations are breathing zone, source and background as shown in Fig. 1, which illustrates the nanopowder transfer operation. The breathing zone (BZ) location was measured above the sash cover, the source location including upstream and downstream sides was measured inside the enclosure as shown by the dotted circles in Fig 1. The background location was measured about 1 meter away from the enclosure.

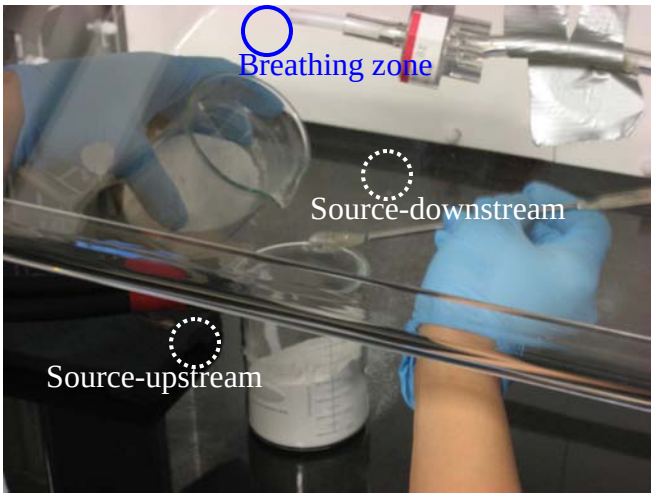


Fig.1. Location of measurement.

Results:

The measured particle release at the worker's BZ, after subtracting the background concentration, is shown in Fig. 2 for transferring and in Fig. 3 for pouring. Each curve shown in Fig. 2 and 3 is the average of two measurements at a single operation.

I. Transferring

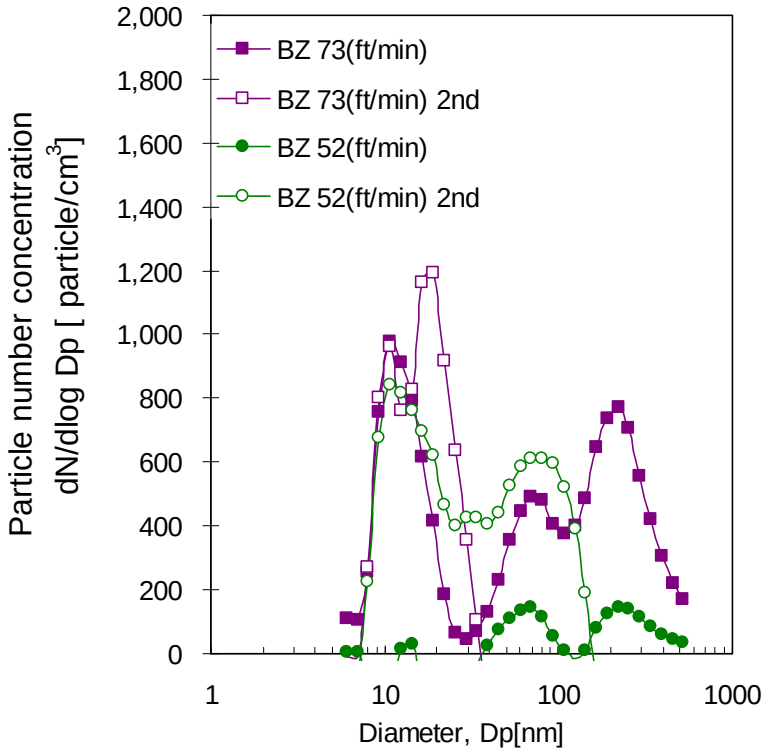


Fig. 2. Particle concentration increase at breathing zone using transferring method.

II. Pouring

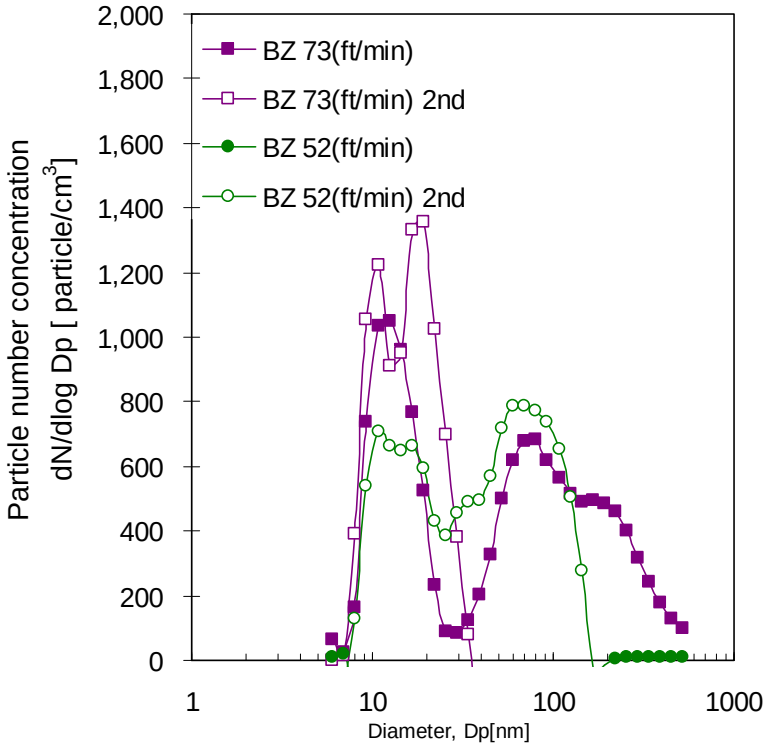


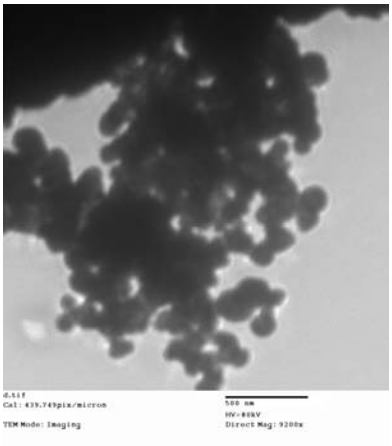
Fig. 3 Particle concentration increase at breathing zone using pouring method

For both transferring and pouring methods, operations at face velocity of 73 ft/min show higher release compared to the operations at the lower velocity of 52 ft/min. Nanoalumina particles were scattered on the enclosure work surface toward the back slot after operations (Fig. 4). More information from data of upstream and downstream show the turbulent airflow still exists in the enclosure during operation. To investigate the airflow pattern, capture the patter of airflow is important to analyze the cause of circulatory airflow. For this purpose, a visual test using laser and smoke are required to fully investigate this issue.

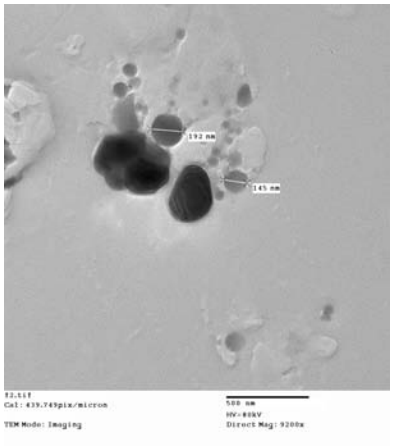
Fig. 4. Nanopowder contamination of the work surface.

III. Analysis of nanoparticles

Aerosol particles were sampled for TEM analysis at the breathing zone location; a single sample was collected during the complete experiment. Very few particles were found on the sampler when analyzed by TEM. However, nanoalumina was found among those particles as seen in Fig 5a.



a. Nanoalumina



b. other particles

Fig. 5. TEM images of particles collected during handling.

Conclusions:

The enclosure was found to be very effective at containing nanoalumina during pouring and transfer. The particle release of nanoalumina during both operations was very low at a relatively high powder use of 100 g. The measured BZ number concentrations would not be detectable on a gravimetric sample. In addition, very few particles were collected on the TEM grid. Particle release should be undetectable if gram quantities of nanopowder are handled.