Dustiness of Fine and Nanoscale Powders

Douglas E. Evans, Leonid A. Turkevich, Cynthia T. Roettgers, Gregory J. Deye and Paul A. Baron.

**Supplementary material**

**Appendix: Details of powders tested.**

Carbonaceous powders

The following material was donated by Southwest Nanotechnologies, Inc. (2501 Technology Place, Norman, OK 73071-11-2):

* SWeNT SWCNT—single-walled carbon nanotubes, code SG, which is currently marketed as code SG-65

The following material was purchased from CNI (Carbon Nanotechnologies, Inc., 16200 Park Row, Houston, TX 77084-5195); this material is currently manufactured by Unidym Inc. 1244 Reamwood Avenue, Sunnyvale, CA 94089.

* HiPCO SWCNT—single-walled carbon nanotubes produced by the high pressure CO process; this material is the same as that used in Shvedova et al. (2008) and was extensively characterized in that work. CNI evolved from Smalley’s team at Rice U. The HiPCO (High Pressure Carbon Monoxide) Process rapidly mixes a gaseous catalyst precursor (e.g. iron pentacarbonyl) with a flow of gaseous carbon monoxide in a chamber at high pressure and high temperature. The catalyst precursor decomposes, and nanometer-sized metal particles form from the decomposition products; these tiny metal particles serve as a catalyst. On the catalyst surface, carbon monoxide reacts to form carbon dioxide and carbon, the latter in the form of single-walled carbon nanotubes. The length of the SWCNT was 100 -1000 nm (measured by CNI using atomic force microscopy); the diameter of the SWCNT was 0.8-1.2 nm (as measured by TEM [transmission electron microscopy]). BET specific surface area is 508 m2/g (Shvedova et al. 2008).

The following material was obtained from Mitsui & Co., Ltd:

* Mitsui VII MWCNT—multi-walled carbon nanotubes; this material is the same as that used in Porter et al. (2010) and Mercer et al. (2010, 2011) and was extensively characterized in that work; this material is no longer commercially available. Briefly, MWCNT median length was 3.86 micron with a count mean diameter = 49 nm +/- S.D. = 13 nm.

The following material was purchased from Pyrograf Products, Inc. (154 W. Xenia Ave., Cedarville, OH 45314):

* Pyrograf III—carbon nanofibers, code PR-24-LDPS, with agglomerate particle diameter d ~ 0.2 µm; this code is no longer manufactured but is similar to the current code PR-24-XTPS (PS refers to the post-processing which strips the polycyclic aromatic hydrocarbons; LD and XT refer to slightly different de-bulking procedures—the fibers tend to tangle; in LD/XT the bundles are more/less tightly compact (Patten 2011).

The following material was purchased from Balentine Enterprises, Inc. (227 Somerset St., Borger, TX 79007):

* SRB B8—standard reference carbon black, approved by ASTM D24, consisting of amorphous solid carbon particles (CAS number 1333-97-4 [carbon black]; specific surface area = 124.70 – 127.90 m2/g.

The following materials were donated by Professor Günter Oberdörster (U. Rochester):

* Printex 90—HCF carbon black/furnace black. This material was manufactured by Degussa-Hüls AG (GB Sivento D-60287, Frankfurt/Main, Germany), and is now available from Evonik Industries (Rellinghausen Strasse 1-11, 45128 Essen, Germany), with a primary particle size = 14nm; specific surface area = 227 +/- 19 m2/g;
* Sterling V—carbon black. This material is available from Cabot Corporation (Specialty Carbon Blacks, Cabot Corporation, 1095 Windward Ridge Parkway, Suite 200, Alpharetta, GA 30005).

Fumed oxides

The following materials were obtained (as samples) from Degussa-Hüls AG (GB Sivento D-60287, Frankfurt/Main, Germany); these products are now available from Evonik Industries (Rellinghausen Strasse 1-11, 45128 Essen, Germany):

* Aeroxide P25—fumed titanium dioxide (CAS number 13463-67-7 [TiO2]); BET specific surface area = 50 +/- 15 m2/g;
* Aerosil 200—hydrophilic fumed silica (CAS number 7631-86-9); specific surface area = 200 m2/g;
* Aerosil 380—hydrophilic fumed silica (CAS number 7631-86-9); specific surface area = 380 m2/g;
* Aerosil OX50—fumed silica (CAS number 7631-86-9)
* Aerosil R202—hydrophobic fumed silica, consisting of fumed silica after-treated with polydimethylsiloxane;
* Aerosil R812—hydrophobic fumed silica, consisting of fumed silica (Aerosil 300) after-treated with HMDS;
* Aeroxide Alu C—fumed alumina; specific surface area = 100 +/- 15 m2/g;

The following material was donated by Delphine Bard (UK HSL, Buxton):

* Cerium dioxide—powdered nanoscale CeO2 (CAS 1306-38-3), product 1405FY (discontinued) from Nanostructured and Amorphous Materials, Inc. (16840 Clay Road, Suite 113, Houston, TX 77084), with APS particle size = 20-30 nm; specific surface area = 28-46 m2/g. While probably not manufactured in a fumed oxide process, the primary particle sizes are comparable to those of the fumed metal oxides.

Metal nanoparticles

The following materials were obtained (as samples) from QuantumSphere, Inc. (2905 Tech Center Dr., Santa Ana, CA 92705):

* QSI-Nano Nickel—nickel powder (CAS numbers 7440-02-0 [Ni], 1313-99-1 [Ni(II)O]), consisting of fcc/amorphous primary particles 10-25 nm, with oxide thickness >/ 2.5 nm; BET specific surface area = 35-65 m2/g;
* QSI-Nano Cobalt—cobalt powder (CAS numbers 7440-48-4 [Co], 13098-06-1 [Co3O4], consisting of hcp/amorphous primary particles 10-30 nm, with oxide thickness >/ 2.5 nm; BET specific surface area = 30-60 m2/g;
* QSI-Nano Manganese—manganese powder (CAS numbers 7439-96-5 [Mn], 1317-35-7 [Mn3O4], consisting of fcc/amorphous primary particles 30-50 nm, with oxide thickness >/ 5 nm; BET specific surface area = 20-35 m2/g;
* QSI-Nano Silver—silver powder (CAS number 7440-22-4 [Ag]), consisting of fcc primary particles 20-40 nm; BET specific surface area = 15-25 m2/g.

Fine oxide powders

The following material was purchased from General Motors Corp. (AC Spark Plug Division, Flint MI):

* Arizona Road Dust—‘GM Air Cleaner Test Dust, Fine’, with a particle size distribution (manufacturer quoted): 0-5 m 39 wt.%, 5-10 m 18 wt.%, 10-20 m 16 wt.%, 20-40 m 18 wt.%, 40-80 m 9 wt.%; this material is no longer available; a substitute Arizona Road Dust (ISO 12103-1 A2 Fine Test Dust Grade) is available from PTI (Powder Technology, Inc. (14331 Ewing Ave., South, Barnsville, MN 55306). The particle size distribution for the AC Test Dust is trimodal, with peaks at 1.5, 8, 40 m; the particle size distribution for the ISO Fine standard is bimodal, with peaks at 4 and 30 m ([www.powdertechnologyinc.com/test-dust-history](http://www.powdertechnologyinc.com/test-dust-history)).

The following materials were donated by Altair Nanotechnologies, Inc. (204 Edison Way, Reno, NV 89502):

* Lithium titanate spinel—powdered Li4Ti5O12, consisting of primary particles in the range 400-1000 nm; BET specific surface area = 135 m2/g (Spitler and Prochazka 2005a, 2005b);
* Kemira AFDC—uncoated anatase TiO2 pigment (CAS 13463-67-7), consisting of 170 nm crystals; specific surface area = 10 m2/g. This material is available from Kemira Chemicals, Inc. (245 Town Park Dr., Suite 200, Kennesaw, GA 30144).

Miscellaneous materials

The following materials were purchased from Micro Abrasives Corp. (720 Southampton Road, P.O. Box 669, Westfield, MA 01085):

* SiC 1200—silicon carbide powder, with a grain size distribution consistent with European FEPA grade F1200 (d3 = 11.4 m, d50 = 3.5-4.8 m, d94 = 0.2 m);
* CEO-1—cerium oxide powder, with a particle size range 1-10 m, with average particle size = 2 m.

The following materials were purchased from Nanocor, Inc. (1350 W. Shure Dr., Arlington Heights, IL 60004-1440):

* PGN nanoclay—off-white polymer-grade (G-105) montmorillonite (CAS number 1302-78-9), consisting of clay sheet-like particles with aspect ratio = 300-500; CEC = 120 meq/100g;
* PGV nanoclay—white polymer-grade (G-105) montmorillonite (CAS number 1302-78-9), consisting of clay sheet-like particles with aspect ratio = 150-200; CEC = 145 meq/100g.

The following material was donated by T. Polton (via D. Leith):

Holland lactose—powdered lactose monohydrate (CAS number 10039-26-6), most likely from LaPalma Holland B.V. (Hoorn 74-6713 KR Ede, The Netherlands), USP/BP pharmaceutical grade; 200 mesh.

**References**

Mercer RR, Hubbs AF, Scabilloni JF *et al.* (2010) Distribution and persistence of pleural penetrations by multi-walled carbon nanotubes. Part Fibre Toxicol; 7: 28.

Mercer RR, Hubbs AF, Scabilloni JF *et al.* (2011) Pulmonary fibrotic response to aspiration of multi-walled carbon nanotubes. Part Fibre Toxicol; 8: 21.

Patten, E. (2011), private communication (Pyrograf Products Inc.).

Porter DW, Hubbs AF, Mercer RR *et al.* (2010) Mouse pulmonary dose- and time course-responses induced by exposure to multi-walled carbon nanotubes. Toxicology; 269: 136–47.

Shvedova AA, Kisin E, Murray AR *et al.* (2008) Inhalation vs. aspiration of single-walled carbon nanotubes in C57BL/6 mice: inflammation, fibrosis, oxidative stress, and mutagenesis. Am J Physiol Lung Cell Mol Physiol; 295: L552–65.

Spitler TM, and Prochazka J. (2005a) Process for making nano-sized and sub-micron-sized lithium-transition metal oxides. *US patent* 6881393.

Spitler TM, and Prochazka J. (2005b) Process for making lithium titanate. *US patent* 6890510.