

Scanning Mobility Particle Sizer™ (SMPS™) Spectrometer

Application: Nanotechnology

Bibliography

2007

Chiang, Wei-Hung; Sankaran, R. Mohan, 2007, "Microplasma synthesis of metal nanoparticles for gas-phase studies of catalyzed carbon nanotube growth," *Applied Physics Letters*, **91**, 121503.

Catalytic properties of metal nanoparticles toward gas-phase carbon nanotube (CNT) growth are presented. Narrow dispersions of iron (Fe) and nickel (Ni) nanoparticles are prepared in a direct current microplasma reactor and subsequently introduced with acetylene (C₂H₂) and hydrogen (H₂) into a heated flow furnace to catalyze CNT growth. Aerosol size classification and high-resolution transmission electron microscopy show that CNT growth occurs on Ni particles at lower temperatures than that for similarly produced Fe nanoparticles. Activation energies of 117 and 73 kJ/mol are found for Fe and Ni catalyst particles, respectively, suggesting that CNT growth occurs by carbon surface diffusion.

Holm, Jason; Roberts, Jeffrey T., 2007, Surface Chemistry of Aerosolized Silicon Nanoparticles: Evolution and Desorption of Hydrogen from 6-nm Diameter Particles," *Journal of American Chemical Society*, **129**, 2496-2503

The surface chemistry of pristine, 6-nm silicon nanoparticles has been investigated. The particles were produced in an RF plasma and studied using a tandem differential mobility analysis apparatus, Fourier transform infrared spectroscopy (FTIR), time-of-flight secondary ion mass spectrometry (ToF-SIMS), and transmission electron microscopy. Particles were extracted from the plasma, which operates at ~20 Torr, into an atmospheric pressure aerosol flow tube, and then through a variable-temperature furnace that could be adjusted between room temperature and 1200°C. DMA measurements show that freshly generated silicon particles shrink with heating, with particle diameters decreasing by ~0.25 nm between 350 and 400°C. FTIR results indicate that freshly generated particles are primarily covered with SiH₂ groups and smaller amounts of SiH and SiH₃. Spectra recorded as a function of heating temperature indicate that the amount of surface hydrogen, as measured by the intensity of modes associated with SiH, SiH₂, and SiH₃, decreases with heating. ToF-SIMS measurements also suggest that hydrogen desorbs from the particles surfaces over the same temperature range that the particles shrink.

Hsu, Li-Yeh; Chein, Hung-Min, 2007, "Evaluation of nanoparticle emission for TiO₂ nanopowder coating materials," *Journal of Nanoparticle Research*, **9**(1):157-163

In this study, nanoparticle emission of TiO₂ nanopowder coated on different substrates including wood, polymer, and tile, was evaluated in a simulation box and measured with a Scanning Mobility Particle Sizer (SMPS) for the first time. The coating process for the substrate followed the instructions given by the supply company. In the simulation box, UV light, a fan, and a rubber knife were used to simulate the sun light, wind, and human contacting conditions. Among the three selected substrates, tile coated with TiO₂ nanopowder was found to have the highest particle emission (22 #/cm³ at 55 nm) due to nanopowder separation during the simulation process. The UV light was shown to increase the release of particle below 200 nm from TiO₂ nanopowder

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coating materials. The results show that, under the conditions of UV lamps, a fan and scraping motion, particle number concentration or average emission rate decreases significantly after 60 and 90 min for TiO₂/polymer and TiO₂/wood, respectively. However, the emission rate continued to increase after 2 h of testing for TiO₂/tile. It is suggested that nanoparticle emission evaluation is necessary for products with nanopowder coating.

Keskinen, H; Mäkelä, J.M.; Aromma, M.; Ristimäki, J.; Kanerva, T.; Levänen, E.; Mäntylä, T.; Keskinen, J, 2007 “Effect of silver addition on the formation and deposition of titania nanoparticles produced by liquid flame spray,” *J. of nanoparticle Res.*, **9** (4): 569-588.

In this study, liquid flame spray (LFS) was used to produce titania, silver and silver-titania deposits of nanoparticles. Titanium(IV)ethoxide (TEOT) and silver nitrate in ethanol solutions were used as precursors and sprayed into turbulent hydrogen-oxygen flame. Production rates of 1.5-40 mg/min of titania were used with silver additions of 1, 2, 4, and 8 wt% compared to titania. Nanoparticle deposits were collected by thermophoretic sampling at six different axial distances from the flame torch head: 3, 5, 10, 12, 15, and 20 cm, of which the all but the last one occurred inside the flame. The deposit samples were analyzed by TEM and SAED analysis. The powder samples of the particles were also collected by electric precipitator to XPS and specific surface area analysis. Particle size and effective density after the flame in the aerosol were analyzed with SMPS and ELPI. The results from the previous studies i.e. controlling the particle size by setting the production rates of the particles were seen to apply also for this binary system. Characterization of the deposits showed that when the substrate is inserted into the flame, in the beginning of the flame the deposit is formed by gas phase deposition whereas further down the flame the particles are first formed in the gas phase and then deposited. The location of the transition from gas phase deposition to gas phase nucleation prior to deposition depends on chemical/physical properties (e.g. thermodynamics and gas phase interactions) of the precursor, precursor concentration in the flame and also flame temperature profile. Therefore, the deposit collection distance from the burner also affected the collected particle size and degree of agglomeration. The two component deposits were produced in two different ways: one-step method mixing both precursors in the same solute, and two-step method spraying each precursor separately. The particle morphology differs between these two cases. In one-step method the primary (d TEM) and agglomerate particle size (d SMPS) decreased with the amount of silver addition, verifying the fact that when present, the silver has a clear effect on the titania nanoparticle formation and growth.

Zhang, H.; Swihart, M, 2007, “Synthesis of Tellurium dioxide nanoparticles by spray pyrolysis,” *Chemistry of Materials*, **19** (6), 1290 -1301.

Tellurite glasses (amorphous TeO₂ based materials) have two useful optical properties, a high refractive index and high optical nonlinearity, that make them attractive for a range of applications. Unlike other metal oxide nanoparticles that are prepared in large quantities by vapor-phase processes, TeO₂ nanoparticles have not been prepared by such methods. Here, we describe the vapor-phase synthesis of TeO₂ nanoparticles from an aqueous solution of telluric acid, Te(OH)₆, that is dispersed into fine droplets by an atomizer. These droplets are carried by nitrogen through a furnace, where they evaporate and decompose, resulting in nucleation of TeO₂ nanoparticles. The resulting nanoparticles were characterized by TEM, XPS, FTIR, and XRD. Particle size distributions measured online using a scanning mobility particle spectrometer demonstrated that a furnace temperature above 700°C was required for full evaporation of the precursor droplets. Thermogravimetric analysis shows that the Te(OH)₆ converts to TeO₂ without evaporating when heated at 5–100°C/min. This contrasts sharply with the complete evaporation observed in the spray pyrolysis reactor, where heating rates exceed 50,000°C/min. The effects of operating parameters on nanoparticle composition, production rate, and size were also studied. This laboratory scale process is capable of producing up to 80 mg/h amorphous TeO₂ nanoparticles with primary particle diameters from 10 to 40 nm. Particles this small exhibit negligible scattering of visible light and have potential applications in the linear and nonlinear refractive index engineering of polymer nanocomposites and as host matrices for rare-earth ions that can act as IR to visible upconverters. The method presented here allows their synthesis in macroscopic quantities from an inexpensive and environmentally friendly precursor.

Zhao, Bin; Uchikawa, Kei; Wang, Hai, 2007, “A comparative study of nanoparticles in premixed flames by scanning mobility particle sizer, small angle neutron scattering, and transmission electron microscopy,” *Proceedings of the Combustion Institute*, **31**(1):851–860

Scanning mobility particle sizer (SMPS) and transmission electron microscopy (TEM) studies were conducted for TiO₂ and soot particles. The TiO₂ particles were produced from a premixed stagnation ethylene–oxygen–argon flame ($\Phi = 0.36$) doped with titanium tetraisopropoxide. Soot was generated from a burner-stabilized premixed ethylene–oxygen–argon flame ($\Phi = 2.5$). The close agreement among SMPS, TEM, and X-ray diffraction results for TiO₂ nanoparticles demonstrates that the probe sampling/mobility measurement technique is accurate for on-line analysis of the size distribution of particles as small as 3 nm in diameter. In the case of soot, notable disagreement between the SMPS and TEM sizes was found and attributable to the fact that the soot taken from the flame studied herein is liquid-like and that upon deposition on the TEM grid, the primary particles do not retain their sphericity. This interpretation is supported by measurements with photo ionization aerosol mass spectrometry, small angle neutron scattering, and thermocouple particle densitometry.

2006

Afergan, Eyal; Epstein, Hila; Golomb, Gershon; Moise, Tamar; Richter, Yoram; Rudich, Yinon, 2006, "Number-concentration of nanoparticles in liposomal and polymeric multiparticulate preparations: Empirical and calculation methods," *Biomaterials*, **27**(4):651–659

The actual number of particles in formulations of nanoparticles (NP) is of importance for quality assurance, comprehensive physicochemical characterization, and pharmacodynamics. Some calculation methods that have been previously employed are limited because they rely on several assumptions and are not applicable for certain preparations. Currently there are no validated experimental methods for determining the particle number-concentration (N_c) of liposomal and polymeric nanoparticulate preparations (500 nm). This study examines a new empirical method for counting the number of particles in nanoparticulate formulations including drug-containing liposomes and polymeric NP. In the new method, suspended NP are nebulized to form aerosol droplets which are dried and counted using a scanning mobility particle sizer (SMPS). Experiments were conducted with three different preparations, empty liposomes (200 and 400 nm), drug-loaded liposomes (200 nm), and polymeric NP (150 nm). It was verified that no detrimental morphological or structural changes of the formulations have been induced by the SMPS technique, and that the obtained N_c values represent the original particles. It is concluded that nano-formulations with concentrations of up to 10^7 particles per 1 cm^3 air, corresponding to approximately 10^{12} particles per 1 ml solution, can be directly counted within the size range of 30–900 nm. The measured values are compared to newly developed theoretical calculations to assess the viability of these calculations.

Cho, Kuk; Biswas, Pratim, 2006, "Sintering Rates for Pristine and Doped Titanium Dioxide Determined Using a Tandem Differential Mobility Analyzer System," *Aerosol Science & Technology*, **40**(5):309–319

Sintering rates of pristine and V-doped TiO_2 were obtained using a tandem DMA system. A range of experiments were conducted to first map out the variation of mobility size of a monodisperse (by mobility) agglomerate with time at three fixed temperatures. Using relationships of the surface area to the mobility size, the sintering equation was solved to determine the activation energy and pre-exponential factor. The value of the activation energy was $236 (\pm 46)$ kJ/mol for pristine TiO_2 and $363 (\pm 1)$ kJ/mol for V-doped TiO_2 . The corresponding pre-exponential factors were 7.22×10^{19} and 2.22×10^{12} s/m⁴K, respectively. These values were then used to predict changes in mobility diameter at different temperatures, and good agreement was obtained with measurements. Possible reasons for faster sintering rates of V- TiO_2 relative to pristine TiO_2 were conjectured.

Epstein, Hila; Afergan, Eyal; Moise, Tamar; Richter, Yoram; Rudich, Yinon; Golomb, Gershon, 2006, "Number-concentration of nanoparticles in liposomal and polymeric multiparticulate preparations: Empirical and calculation methods," *Biomaterials*, **27**(4):651–659

The actual number of particles in formulations of nanoparticles (NP) is of importance for quality assurance, comprehensive physicochemical characterization, and pharmacodynamics. Some calculation methods that have been previously employed are limited because they rely on several assumptions and are not applicable for certain preparations. Currently there are no validated experimental methods for determining the particle number-concentration (N_c) of liposomal and polymeric nanoparticulate preparations (<500 nm). This study examines a new empirical method for counting the number of particles in nanoparticulate formulations including drug-containing liposomes and polymeric NP. In the new method, suspended NP are nebulized to form aerosol droplets which are dried and counted using a scanning mobility particle sizer (SMPS). Experiments were conducted with three different preparations, empty liposomes (200 and 400 nm), drug-loaded liposomes (200 nm), and polymeric NP (150 nm). It was verified that no detrimental morphological or structural changes of the formulations have been induced by the SMPS technique, and that the obtained N_c values represent the original particles. It is concluded that nano-formulations with concentrations of up to 10^7 particles per 1 cm^3 air, corresponding to approximately 10^{12} particles per 1 ml solution, can be directly counted within the size range of 30–900 nm. The measured values are compared to newly developed theoretical calculations to assess the viability of these calculations.

Hyun, Jung Eun; Lee, Tai Gyu, 2006, "Structural effect of the in situ generated titania on its ability to oxidize and capture the gas-phase elemental mercury," *Chemosphere*, **62**(1):26–33

Structural effect of the in situ generated TiO_2 sorbent particle was examined for its ability to capture elemental mercury under UV irradiation in a simulated combustion flue gas. Titania particles were prepared by thermal gas-phase oxidation of Titanium (IV) isopropoxide (TTIP) using a high temperature electric furnace reactor. The structural characteristics of the in situ generated TiO_2 at various synthesis temperatures were investigated; size distribution and the geometric mean diameter were measured using a scanning mobility particle sizer, while fractal dimension and radius of gyration were evaluated from the transmission electron microscopy images. Results from the Hg^0 capture experiment show that with increasing titania synthesis temperature, the overall aggregate size increases and the morphology becomes more open-structured to gas-phase Hg^0 and UV light, resulting in the improved mercury removal capability.

Kim, S.H.; Zachariah, M.R., 2006, "In-Flight Kinetic Measurements of the Aerosol Growth of Carbon Nanotubes by Electrical Mobility Classification," *Journal of Physical Chemistry B*, **110**, 4555-4562

We describe an on-the-fly kinetic study of gas-phase growth of multiwalled carbon nanotubes. The methodology employs electrical mobility classification of the CNT, which enables a direct measure of CNT length distribution in an aerosol reactor. The specific experiment employs two mobility classification steps. In the first step we mobility classify the catalyst particle, in this case Ni, created by pulsed laser ablation, to generate a stream of monodisperse particles. This then determined the diameter of the CNT, when a hydrocarbon/H₂ mix is added in a heated aerosol reactor. A second electrical mobility classification step allows us to determine the length distribution of the CNTs. We found that CNT growth from ethylene required the addition of small amounts of water vapor, whereas growth from acetylene did not. We show that acetylene, which always has small amounts of acetone present when purchased, can provide the oxygen source to prevent catalyst coking. By varying the temperature of the growth, we were able to extract Arrhenius growth parameters. We found an activation energy for growth ~ 80 kJ mol⁻¹ from both acetylene and ethylene, which is considerably lower than previous works for substrate-grown CNTs (E_a) 110-150 kJ mol⁻¹). Furthermore, we observed that our aerosol CNT growth rates were about 2 orders of magnitude higher than those for substrate-grown CNTs. The dominant growth mechanism of CNT previously proposed is based upon bulk diffusion of carbon through nickel particles. However, on the basis of the lower activation energy found in this work, we proposed that the possible mechanism of gas-phase growth of CNT is correlated with both surface (E_a) 29 kJ mol⁻¹) and bulk diffusion (E_a) 145 kJ mol⁻¹) of carbon on nickel aerosol particles. Finally, the experimental approach described in this work should be amenable to other nanowire systems grown in the aerosol phase.

Ku, Bon Ki; Maynard, Andrew D., 2006, "Generation and investigation of airborne silver nanoparticles with specific size and morphology by homogeneous nucleation, coagulation and sintering," *Journal of Aerosol Science*, **37**(4):452-470

An aerosol generation facility has been characterized to produce well-defined silver nanoparticles for use as a test aerosol when evaluating instrument response to different particle morphologies within a range of sizes. The generator consists of two in-series laminar tube furnaces to produce and subsequently sinter particles, and is capable of generating spherical or agglomerated fractal-like silver particles, with corresponding projected surface (two-dimensional) fractal dimensions from 1.58 to 1.94. The morphologies of generated particles as well as size distributions at different sintering temperatures were characterized using a transmission electron microscope (TEM) and a scanning mobility particle sizer (SMPS). Mean diameters measured were significantly reduced for sintering temperatures from 100 to 300°C, but showed little variation for sintering temperatures above 500°C. TEM analysis indicated that this phenomenon was caused by sintering, followed by partial and complete coalescence of fractal-like agglomerates into spheres. Agglomerate restructuring from classical completely sintered agglomerates 500°C to spherical particles 700°C did not lead to a change in the particle mobility size distribution. The temperature at which complete sintering occurred was higher than that predicted by theory, but was in reasonable agreement with previously published experimental data. For monodisperse particles in the size range from 20 to 100 nm, a simple exponential model related sintering temperature to the diameter of coalesced spherical particles.

Lee, Tai Gyu; Hyun, Jung Eun, 2006, "Structural effect of the in situ generated titania on its ability to oxidize and capture the gas-phase elemental mercury," *Chemosphere*, **62**(1):26-33

Structural effect of the in situ generated TiO₂ sorbent particle was examined for its ability to capture elemental mercury under UV irradiation in a simulated combustion flue gas. Titania particles were prepared by thermal gas-phase oxidation of Titanium (IV) isopropoxide (TTIP) using a high temperature electric furnace reactor. The structural characteristics of the in situ generated TiO₂ at various synthesis temperatures were investigated; size distribution and the geometric mean diameter were measured using a scanning mobility particle sizer, while fractal dimension and radius of gyration were evaluated from the transmission electron microscopy images. Results from the Hg⁰ capture experiment show that with increasing titania synthesis temperature, the overall aggregate size increases and the morphology becomes more open-structured to gas-phase Hg⁰ and UV light, resulting in the improved mercury removal capability.

2005

Choi, Jong Hyun; Koshland, Catherine P.; Lucas, Donald; Sawyer, Robert F., 2005, "Photochemical interaction of polystyrene nanospheres with 193 nm pulsed laser light," *Journal of Physical Chemistry B*, **109**(50): 23905-23910

The photochemical interaction of 193 nm light with polystyrene nanospheres is used to produce particles with a controlled size and morphology. Laser fluences from 0 to 0.14 J/cm² at 10 and 50 Hz photofragment nearly monodisperse 110 nm spherical polystyrene particles. The size distributions before and after irradiation are measured with a scanning mobility particle sizer (SMPS), and the morphology of the irradiated particles is examined with a transmission electron microscope (TEM). The results show that the irradiated particles have a smaller mean diameter (similar to 25 nm) and a number concentration more than an order

of magnitude higher than nonirradiated particles. The particles are formed by nucleation of gas-phase species produced by photolytic decomposition of nanospheres. A nondimensional parameter, the photon-to-atom ratio (PAR), is used to interpret the laser-particle interaction energetics. 39 Refs.

Choi, Jong Hyun; Koshland, Catherine P.; Lucas, Donald; Sawyer, Robert F.; Stipe, Christopher B., 2005, "NaCl particle interaction with 193-nm light: Ultraviolet photofragmentation and nanoparticle production," *Journal of Applied Physics*, **97**(12):1–9

The interaction of nanoscale NaCl particles with 193-nm photons is studied to better understand particle disintegration and production by ultraviolet photofragmentation. The particles are irradiated in a constrained air stream with laser fluences from 0.08 to 0.23 J cm² with single and multiple pulses striking the particles. The resulting particle size distributions are measured with a scanning mobility particle sizer and the morphology is analyzed qualitatively by scanning electron microscopy (SEM). Photofragmentation of NaCl particles at 193 nm produces gas-phase species as well as small solid-phase fragments without significantly heating the particles or creating a plasma. The irradiated particles have a mean diameter from 20 to 55 nm (depending on the photon energy) and a number concentration an order of magnitude higher than the 118-nm mean diameter nonirradiated particles. The SEM images before and after 193-nm irradiation reveal that the irradiated particles are less fractal and more spherical. 52 Refs.

Girshick, S.L.; Hafiz, J.; Heberlein, J.; McMurry, P.H.; Mukherjee, R.; Renault, T.; Wang, X., 2005 "System for in situ characterization of nanoparticles synthesized in a thermal plasma process," *Plasma Chemistry and Plasma Processing*, **25**(5):439–453

We have designed a particle diagnostic system that is able to measure particle size and charge distributions from low stagnation pressure and high temperature (2000–4000 K) environments in near real time. This system utilizes a sampling probe interfaced to an ejector to draw aerosol from the low pressure chamber. Particle size and charge distributions are measured with a scanning mobility particle sizer. A hypersonic impactor is mounted in parallel with the scanning mobility particle sizer to collect particles for off-line microscopic analysis. This diagnostic system has been used to measure size and charge distributions of nanoparticles (Si, Ti, Si-Ti-N, etc.) synthesized with our thermal plasma reactor. We found that the mean particle size increases with operating pressure and reactant flow rates. We also found that most particles from our reactor are neutral for particles smaller than 20nm, and that the numbers of positively and negatively charged particles are approximately equal. Inc. 33 Refs.

Kim, S.H.; Zachariah, M.R., 2005, "In-flight size classification of carbon nanotubes by gas phase electrophoresis," *Nanotechnology* **16**, 2149–2152

We demonstrate the use of gas phase electrophoresis to size classify CNTs grown in a continuous aerosol process. The separation process occurs at atmospheric pressure and involves electrostatic mobility separation which classifies fibers on the basis of equivalent projected surface area. This implies that one can, for diameter-controlled CNTs, obtain an on-the-fly determination of the CNT length distribution during CNT synthesis, or alternatively have a method for producing size separated CNTs. The method should be generic to any fiber-based material.

Ku, Bon Ki; Maynard, Andrew D., 2005, "Comparing aerosol surface-area measurements of monodisperse ultrafine silver agglomerates by mobility analysis, transmission electron microscopy and diffusion charging," *Journal of Aerosol Science*, **36**(9):1108–1124

Three methods -scanning mobility particle sizer (SMPS), transmission electron microscopy (TEM), and diffusion charging (DC) - for estimating aerosol surface area were evaluated and compared. The aerosol used was monodisperse silver particles, having morphologies that range from spherical to agglomerated particles, with corresponding fractal dimensions from 1.58 to 1.94. For monodisperse silver particle agglomerates smaller than 100 nm, the DC response was proportional to the mobility diameter squared, regardless of morphology. For particle sizes from 80 to 200 nm, the DC response varied as the mobility diameter to the power 1.5. The projected surface area of agglomerates analyzed by TEM agreed well with that estimated from particle mobility diameters for particles smaller than 100 nm. The surface area of monodisperse particles, measured by DC and SMPS, was comparable to the geometric surface area below 100 nm, but in the size range of 100-200 nm, the methods used underestimated the geometric surface area. SMPS, TEM, and DC-based measurements of surface area were in good agreement with one another for monodisperse aerosol particles smaller than 100 nm. 24 Refs.

Lee, Gyo Woo; Lee, Seung Bok; Jung, Jong Soo; Bae, Gwi Nam, 2005, "Effect of flame temperature on the characteristics of flame synthesized TiO₂ nanoparticles," *Transactions of the Korean Society of Mechanical Engineers. B*, **29**(9):1013–1021

In this work, TiO₂ nanoparticles were synthesized using a N₂-diluted hydrogen coflow diffusion flame. The effects of flame temperature on the crystalline structure and the size of formed nanoparticles were investigated. The maximum centerline temperature of the flame ranged from 1,920K for H₂-only flame to 863K for 81% N₂-diluted flame. The morphology and the

crystal structure of TiO₂ nanoparticles were analyzed by a TEM and a XRD, respectively. The particle size distribution was also measured by using a Scanning Mobility Particle Sizer (SMPS). The mean particle diameter was calculated from the TEM images depended on the flame temperature, having minimum at about 1,200K. Based on the SMPS measurements, the mean particle diameter of TiO₂ nanoparticles at flame temperatures @1,300K was smaller than that at flame temperatures @ 1,300K. From the XRD analysis, it was evident that the anatase fraction increased with decreasing the flame temperature. The portion of anatase phase in TiO₂ nanoparticles might be greater than 80% when the flame temperature was lower than 1,000K.

Moisala, Anna; Nasibulin, Albert G.; Shandakov, Sergei D.; Jiang, Hua; Kauppinen, Esko I., 2005, "On-line detection of single-walled carbon nanotube formation during aerosol synthesis methods," *Carbon*, **43** (10): 2066-2074

Differential electrical mobility (DMA) method for the on-line detection of single-walled carbon nanotubes (SWCNTs) formation was used for the first time. Three different gas-phase synthesis processes were used to produce SWCNTs via CO disproportionation in the presence of catalyst nanoparticles formed either by a hot wire generator method or via thermal decomposition of ferrocene or iron pentacarbonyl. The typical product measured with the DMA method was bundles of SWCNTs, which further agglomerated prior to the measurement. Despite the different product morphology and concentration, the on-line measurement was able to distinguish SWCNT formation in each experimental set-up as an increase in the geometric mean particle diameter and as a decrease in the total particle number concentration. Furthermore, information regarding the relative SWCNT concentration can also be obtained from the DMA measurement. A theoretical approach to the mobility of nonspherical particles in the electric field was successfully developed in order to convert the electrical mobility size of the high aspect ratio SWCNTs measured with DMA to the physical size of the product. Size-selected SWCNTs were studied with transmission electron microscopy in order to find the correlation between the on-line DMA measurement data and the SWCNT morphology.

Moravec, P.; Smolik, J.; Leviansky, V.V., 2005, "Preparation of Al₂O₃-SiO₂ fine particles by CVD method in tube flow reactor," *Powder Technology*, **153**(3):159-165

The aim of this study was the synthesis of mixed or coated multicomponent alumina-silica particles by chemical vapor deposition method in a tube flow reactor. The particles were produced by simultaneous thermal decomposition of aluminum tri-*sec*-butoxide and tetraethylorthosilicate in one reactor. The particle production was monitored by Differential Mobility Particle Sizer, composition of particles was analyzed by energy dispersive X-ray analysis, morphology by scanning/transmission electron microscopy and crystallinity by selected area electron diffraction. In dependence on experimental conditions, the particles produced were either alumina particles with intermixture of silica, or they were coated by silica, or it was a mixture of particles of various compositions. The particles were often agglomerates of the primary nanoparticles and were partially crystalline.

Vasiliou, J., 2005, "An evaluation of a scanning mobility particles sizer with NIST-traceable particle size standards," *2005 NSTI Nanotechnology Conference and Trade Show*, Anaheim, CA

A scanning mobility particle sizer (SMPS - TSI Model 3936-Series) was evaluated using Duke Scientific NIST-traceable particle size standards and Standard Reference Materials from the National Institute of Standards and Technology (NIST SRM's). The importance of instrument setup, electrospray operation and sample preparation for polystyrene spheres are discussed as well as the results from 14 different size reference standards. Correlations between the SMPS system and established electron microscopy and dynamic light scattering methods are also shown in tabular and graphical forms. Results show that with proper operation, the SMPS results fall within the uncertainty of the NIST traceable diameters in the range that was evaluated - 20 to 100 nanometers.

Wang, Yanmin; Forssberg, Eric, 2006, "Production of carbonate and silica nano-particles in stirred bead milling," *International Journal of Mineral Processing*, **81**(1):1-14

This paper presents the experimental results of the mechanical production of silica and carbonate colloidal particles below 100 nm using two types of stirred bead mills (i.e., DCP Superflow 12 and PML H/V). It is shown that the stirred bead mill with very small beads can be used as an efficient equipment for the production of the colloidal particles in nanoscale from the feed materials of several microns in sizes at high energy consumptions. The DCP Superflow mill with high power densities is superior for the effective size reduction and production rate, compared to the conventional PML H/V mill with lower power densities. The smaller particles could be produced by the DCP Superflow mill at the same level of high energy inputs as from the PML H/V mill. The "grinding limit" for the processes in the mills has been discussed.

The nanoparticle sizes of the ground products obtained in the mills were determined by a scanning mobility particle sizer (SMPS), an acoustic particle sizer (APS-100), a nitrogen gas adsorption method (BET), and transmission electron microscopy (TEM). The surface mean particle diameters on the same sample obtained with the different methods for the nanoparticle size analysis are varying due to the different characterization techniques. The TEM micrographs show a large amount of the primary nanoparticles below 100 nm produced after milling.

In addition, the surface and structure of the original and the ground colloidal products have been investigated with some particle/surface characterization techniques such as X-ray diffraction (XRD), nuclear magnetic resonance (^{29}Si NMR) and Fourier transform infrared spectroscopy (FTIR). It is indicated that an intense comminution of carbonate minerals in the stirred bead mills leads to a progressive loss in crystallinity of the basal planes of the crystal structure. An intensive mechanical treatment of silica gives the structural changes and the amorphisation.

Wang, X.; Hafiz, J.; Mukherjee, R.; Renault, T.; Heberlein, J.; Girshick, S.; McMurry, P., 2005, "System for In Situ Characterization of Nanoparticles Synthesized in a Thermal Plasma Process," *Plasma Chemistry and Plasma Processing*, **Volume 25**(5):439–453

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Zhao, Bin; Uchikawa, Kei; McCormick, John R.; Ni, Chao Ying; Chen, Jingguang G.; Wang, Hai, 2005, "Ultrafine anatase TiO₂ nanoparticles produced in premixed ethylene stagnation flame at 1 atm," *Proceedings of the Combustion Institute*, **30**(1):1441–1448

Particle size distribution functions (PSDFs) of incipient soot formed in laminar premixed 24.2% ethylene–37.9% oxygen–diluent (nitrogen and/or argon) flames with an equivalence ratio of 1.92 were studied by online sampling and scanning mobility particle sizer. Two series of flames were studied to quantify the effect of flame temperature on the characteristics of PSDFs. In the first series, the variation of the flame temperature was accomplished by varying the cold gas velocity. Temperature in the second series of flames was manipulated by the diluent composition from argon to nitrogen. The results show that for flames with the maximum temperature (T_{max}) around 1800 K the soot PSDFs were distinctively bimodal. As the flame temperature was increased to ~ 1850 K, bimodality faded away. The distribution was unimodal for $T_{max} > 1900$ K. The variation of the characteristics of the PSDF as a function of the flame temperature is consistent with the theoretical explanation that bimodality is the result of competition between persistent particle nucleation and particle–particle coagulation in low-temperature flames.

2004

Carter, C.B.; Deneen, J.; Gerberich, W.W.; Girshick, S.L.; Hafiz, J.; Heberlein, J.V.R.; McMurry, P.H.; Mook, W.; Mukherjee, R.; Perrey, C.R.; Wang, X., 2004, "Hypersonic plasma particle deposition of Si–Ti–N nanostructured coatings," *Surface and Coatings Technology*, **188–189**(1-3 SPEC.ISS.):364–370

Si–Ti–N coatings with various compositions were deposited on molybdenum substrates using hypersonic plasma particle deposition (HPPD). In this method, vapor phase precursors (TiCl₄, SiCl₄ and NH₃) are dissociated in a DC plasma arc and the hot gas is quenched in a rapid nozzle expansion to nucleate nanoparticles. These nanoparticles are then accelerated in hypersonic flow, causing them to deposit by ballistic impaction on a substrate placed downstream of the nozzle. Films of 10–25 μm thickness were deposited at rates of 2–10 $\mu\text{m}/\text{min}$, depending on reactant flow rates, at substrate temperatures ranging from 200 to 850°C. When the reactant gases were premixed the coatings consisted of nc-TiN, nc-TiSi₂, nc-Ti₅Si₃ and amorphous Si₃N₄. For the unpremixed reactants case, the coatings consisted of free Si, nc-TiN and amorphous Si₃N₄. Hardness of as-deposited films was evaluated by nanoindentation of polished film cross-sections. Measured hardness values, averaged over 10–15 locations for each film, ranged from 16–24 GPa. In separate experiments with the same conditions, particle size distributions were measured by placing a sampling probe at the same location as the film substrate. The sampled aerosol was rapidly diluted and delivered to a scanning mobility particle sizer (SMPS). In-situ particle size distribution measurements confirmed that the coatings were formed by impaction of nanoparticles in the 5–15 nm range, with higher reactant flow rates producing larger particles. Focused ion beam (FIB) milling was used to observe film cross-section and porosity. For as-deposited films containing pores, in-situ plasma sintering was used to densify the film without grain growth.

Heszler, P.; Landstrom, L.; Marton, Z., 2004, "Early stage of the material removal during ArF laser ablation of graphite," *Applied Physics A: Materials Science and Processing*, **79**(3):579–585

Material removal during ArF excimer laser ablation of graphite at atmospheric pressure was investigated by two independent methods; 1) by observation of the propagating properties of the shock wave generated by the carbonaceous ejecta and 2) by in situ measurement of the size distribution of carbon nanoparticles condensing in the ablation plume. This latter was carried out by a scanning mobility particle sizer system based on a differential mobility analyzer. The performed measurements indicate that the

material removal during ArF laser ablation consists of two steps at fluences above the threshold fluence. First, a thin layer of carbon (of the order of 1 nm) is removed by a quick desorption process, leading to shockwave formation. This process takes place in a ns time scale, and desorption rate estimations reveal that this can not be explained by thermal surface evaporation. Since to our knowledge there is no thermal process that could account for the estimated desorption rate, it is argued that this is a fast photochemical (i.e., non-thermal) process. The size distribution of the condensed nanoparticles related to this step shows a rising edge at diameters below 10 nm. At fluences above the ablation threshold, the majority of the material is ejected in the second phase, resulting in condensation of carbon nanoparticles, peaking at similar to 50 nm diameters in the size spectrum. Both shockwave formation and material removal are also detected well below the ablation threshold fluence, which is attributed to the photochemical process. 39 Refs.

Márton, Z.; Landström, L.; Heszler, P., 2004, "Early stage of the material removal during ArF laser ablation of graphite," *Applied Physics A*, **79**(3):579–585

Material removal during ArF excimer laser ablation of graphite at atmospheric pressure was investigated by two independent methods; 1) by observation of the propagating properties of the shock wave generated by the carbonaceous ejecta and 2) by in situ measurement of the size distribution of carbon nanoparticles condensing in the ablation plume. This latter was carried out by a scanning mobility particle sizer system based on a differential mobility analyzer. The performed measurements indicate that the material removal during ArF laser ablation consists of two steps at fluences above the threshold fluence. First, a thin layer of carbon (of the order of 1 nm) is removed by a quick desorption process, leading to shockwave formation. This process takes place in a ns time scale, and desorption rate estimations reveal that this can not be explained by thermal surface evaporation. Since to our knowledge there is no thermal process that could account for the estimated desorption rate, it is argued that this is a fast photochemical (i.e., non-thermal) process. The size distribution of the condensed nanoparticles related to this step shows a rising edge at diameters below 10 nm. At fluences above the ablation threshold, the majority of the material is ejected in the second phase, resulting in condensation of carbon nanoparticles, peaking at ~50 nm diameters in the size spectrum. Both shockwave formation and material removal are also detected well below the ablation threshold fluence, which is attributed to the photochemical process.

Young, Li-Hao; Keeler, G. J., 2004, "Characterization of Ultrafine Particle Number Concentration and Size Distribution During a Summer Campaign in Southwest Detroit," *Journal of the Air & Waste Management*, **54**(9)

This paper presents results from a study conducted in southwest Detroit from July 20 to July 30, 2002, to characterize ambient ultrafine particles ($dp < 0.1 \mu\text{m}$), and to examine the effect of local sources and meteorological parameters on the ultrafine number concentration and size distribution. The number concentrations of ambient particles in the size range of 0.01–0.43 μm were obtained from a scanning mobility particle sizer (SMPS). Meteorological parameters including ambient temperature, relative humidity, wind speed, wind direction, rainfall, and solar radiation flux were also monitored concurrently atop a 10-m tower. On average, ultrafine particles ranged from 1.4×10^4 to $2.5 \times 10^4 \text{ cm}^{-3}$, with significant diurnal and daily variations, and accounted for 89% of the total number concentration ($0.01 < dp < 0.43 \mu\text{m}$). Time-series plots of the 5-min number concentrations revealed that peak concentrations often occurred during morning rush hour and/or around solar noon when photochemical activity was at a maximum. The morning traffic-related peak coincided with the NO_x peak, whereas the photochemical-related peak correlated with solar radiation flux. On some days, the noon peak concentration was many times higher than the morning peak concentration. Although the number size distribution varied considerably over the course of the study, it typically exhibited one to three modes, with diameters around 0.01, 0.05, and 0.09 μm . Analysis of the influence of wind direction indicated that stationary sources could be one of the contributors to elevated ultrafine particle concentration. Overall, the data indicated that fossil fuel combustion and atmospheric gas-to-particle conversion of precursor gases are the major sources of ultrafine particles in the southwest Detroit area during the summer.

2003

Boman, M.; Heszler, P.; Landstrom, L.; Marton, Z., 2003, "A comparative study of size distribution of nanoparticles generated laser ablation of graphite and tungsten," *Material Science and Engineering C*, **23**(1-2):225-228

Nanoparticles (NPs) were generated by ArF excimer laser ablation of graphite and tungsten targets in N₂ ambient at atmospheric pressure. The size distribution of the particles was monitored in situ by a scanning mobility particle sizing system (SMPS) system, based on differential mobility analyzer. The experimental conditions made possible to record the size distributions in the 7-133-nm diameter range and results are presented for different laser fluences, repetition rates and ablated areas, respectively. Material analysis was performed by photoelectron spectroscopy (XPS), Raman spectroscopy, X ray diffraction and SEM. 17 Refs.

Zhao, B.; Yang, Z.; Wang, J.; Johnston, M.; Wang, H., 2003, "Analysis of soot nanoparticles in a laminar premixed ethylene flame by Scanning Mobility Particle Sizer," *Aerosol Science and Technology*, **37**:611-620

Mobility size distributions of soot particles produced from a fuel-rich, laminar premixed ethylene flat flame were obtained by in situ probe sampling and online analysis using a nano scanning mobility particle sizer. The emphasis of the work was the development of an in situ sampling technique to follow the evolution of nanoparticles formed in flames. Particle size distribution functions were obtained along the centerline of the flame in a spatially resolved manner. Considerable efforts were made to eliminate particle losses in the sample probe. To this end, the effect of dilution on particle losses in the sample probe was systematically studied. It is demonstrated that particle losses due to coagulation and diffusive wall deposition were negligible using a dilution ratio greater than $\sim 10^4$. The sampling technique is shown to be capable of closely following the evolution of particle size distribution from the nucleation mode to mass growth mode dominated by particle coagulation and gas-surface reactions. Beyond the particle nucleation region of the flame, the size spectra were found to be distinctively bimodal, indicating sustained particle nucleation throughout the flame studied.

2002

Ullman, M; Friedlander, S; Andreas, S., 2002, "Nanoparticle formation by laser ablation," *Journal of Nanoparticle Research*, **4** (6):499-509.

The properties of nanoparticle aerosols of size ranging from 4.9 nm to 13 nm, generated by laser ablation of solid surfaces are described. The experimental system consisted of a pulsed excimer laser, which irradiated a rotating target mounted in a cylindrical chamber 4 cm in diameter and 18-cm long. Aerosols of oxides of aluminum, titanium, iron, niobium, tungsten and silicon were generated in an oxygen carrier gas as a result of a reactive laser ablation process. Gold and carbon aerosols were generated in nitrogen by non-reactive laser ablation. The aerosols were produced in the form of aggregates of primary particles in the nanometer size range. The aggregates were characterized using a differential mobility analyzer and electron microscopy. Aggregate mass and number concentration, electrical mobility size distribution, primary particle size distribution and fractal dimension were measured. System operating parameters including laser power (100 mJ/pulse) and frequency (2 Hz), and carrier gas flow rate (1 L/min) were held constant. A striking result was the similarity in the properties of the aerosols. Primary particle size ranged between 4.9 and 13 nm for the eight substances studied. The previous studies with flame reactors produced a wider spread in primary particle size, but the order of increasing primary particle size follows the same trend. While the solid-state diffusion coefficient probably influences the size of the aerosol in flame reactors, its effect is reduced for aerosols generated by laser ablation. It is hypothesized that the reduced effect can be explained by the collision-coalescence mechanism and the very fast quenching of the laser generated aerosol.

2001

Ahn, K. H.; Lee, J. S.; Yu, J. H., 2001, "In situ characterization of TiO₂ nanoparticle in chemical vapor condensation reactor," *Scripta Materialia*, **44**(8-9):2213-2217

The effects of oxygen flow rate on the particle coagulation in the chemical vapor condensation (CVC) reactor were investigated using Scanning Mobility Particle Sizer. With a decrease of oxygen flow rate, the titanium dioxide particle size increased and the size distribution broadened with a longer residence time and a higher particle concentration. These characteristics in size were dominantly controlled by the ratio of oxygen flow rate in the CVC reactor. 8 Refs.

Ahn K.H.; Jung C.H.; Choi M.1; Lee J.S., 2001, "Particle Sampling and Real Time Size Distribution Measurement in H₂/O₂/TEOS Diffusion Flame," *Journal of Nanoparticle Research*, **3**(2–3):161–170

Growth characteristics of silica particles have been studied experimentally using in situ particle sampling technique from H₂/O₂/Tetraethylorthosilicate (TEOS) diffusion flame with carefully devised sampling probe. The particle morphology and the size comparisons are made between the particles sampled by the local thermophoretic method from the inside of the flame and by the electrostatic collector sampling method after the dilution sampling probe. The Transmission Electron Microscope (TEM) image processed data of these two sampling techniques are compared with Scanning Mobility Particle Sizer (SMPS) measurement. TEM image analysis of two sampling methods showed a good agreement with SMPS measurement. The effects of flame conditions and TEOS flow rates on silica particle size distributions are also investigated using the new particle dilution sampling probe. It is found that the particle size distribution characteristics and morphology are mostly governed by the coagulation process and sintering process in the flame. As the flame temperature increases, the effect of coalescence or sintering becomes an important particle growth mechanism which reduces the coagulation process. However, if the flame temperature is not high enough to sinter the aggregated particles then the coagulation process is a dominant particle growth mechanism. In a certain flame condition a secondary particle formation is observed which results in a bimodal particle size distribution.

Hansen, J. P.; Jensen, J. R.; Johannessen, T.; Livbjerg, H., 2001, "Synthesis of ZnO particles in a quench-cooled flame reactor," *AIChE Journal*, **47**(11):2413–2418

The quench cooling of a flame by injection of cold air was studied in a flame reactor for the formation of ZnO particles in a premixed flame with a precursor jet. A rapid temperature drop downstream from the temperature peak is advantageous for the attainment of a large specific surface area. Computational fluid dynamics simulations were used to design a quench ring with nozzles directed slightly upward and at a small tangential angle from the direct line to the center. This novel design avoids distortion of the flow pattern below the quenching plane and effectively cools the flame immediately above. At the highest tested production rate, the specific surface area of the ZnO particles increases from 20 to 60 m²/g when quenching is employed. The particles are characterized by BET surface area measurements, TEM images, and the size distributions of particle aggregates are measured by a Scanning Mobility Particle Sizer.

Nakaso, Koichi; Fujimoto, Toshiyuki; Seto, Takafuimi; Shimada, Manabu; Okuyama, Kikuo; Lunden, Melissa M., 2001, "Size Distribution Change of Titania Nano-Particle Agglomerates Generated by Gas Phase Reaction, Agglomeration, and Sintering," *Aerosol Science & Technology*, **35**(5):929–947

In the manufacturing of nanometer-sized material particulates by aerosol gas-to-particle conversion processes, it is important to analyze how the gas-phase chemical reaction, nucleation, agglomeration, and sintering rates control the size distribution and morphology of particles. In this study, titania particles were produced experimentally by the thermal decomposition of titanium tetrakisopropoxide (TTIP) and oxidation of titanium tetrachloride (TiCl₄) using a laminar flow aerosol reactor. The effect of reaction temperature on the size and morphology of the generated particles was investigated under various conditions. The size distributions of agglomerates were measured using a DMA/CNC system. The size distributions of primary particles were measured using TEM pictures of the agglomerates sampled by a thermophoretic aerosol sampler. In order to model the growth of both agglomerates and primary particles simultaneously, a two-dimensional discrete-sectional representation of the size distribution was employed, solving the aerosol general dynamic equation for chemical reaction, agglomeration, and sintering. Qualitative agreement between the experimentally observed results and the simulation are satisfactory for the large variations in reactor temperature explored.

2000

Martin, S. T.; Han, J.-H., 2000, "An aerosol chemical reactor for coating metal oxide particles with (NH₄)₂SO₄-H₂SO₄-H₂O. Manipulation of the sulfate coating," *Journal of Crystal Growth*, **219**(3):290–299

One important chemical and morphological class of atmospheric particles consists of mineral dusts contained as inclusions in crystalline and aqueous sulfate particles. The mineral dust surface plays an important role in regulating the sulfate phase by providing a surface for the heterogeneous nucleation of the crystal from supersaturated aqueous sulfate phases. In principle, the efficacy of mineral dusts in promoting phase transitions can be investigated through laboratory process studies. To do so, however, a particle generator is necessary. The present work describes the generation of metal oxide particles (viz. α -Fe₂O₃, α -Al₂O₃, Al₆Si₂O₁₃, and amorphous SiO₂) coated by (NH₄)₂SO₄. The diameter of the oxide core and the thickness of the sulfate coating are regulated from 30 to 400 nm and 13 to 81 nm, respectively. Sizing is based upon aerosol particles collected by electrostatic precipitation and subsequently imaged by transmission electron microscopy. Depending on reactor conditions, the fraction of particles containing a metal oxide core varies from 0.01 to 0.86, as inferred from number size distributions measured

with a Scanning Mobility Particle Sizer. The single most important reactor variable affecting fraction of particles containing a metal oxide core is the total sulfate loading. No dependencies on the effects of different chemical surface properties of the metal oxide particles on the coating process have been observed in our laboratory to date. Rather, the number size distribution of metal oxide core particles appears important without regard to chemical surface properties.

Signorell, R.; Kunzmann, M. K.; Suhm, M. A., 2000, "FTIR investigation of non-volatile molecular nanoparticles," *Chemical Physics Letters*, **329**(1–2):52–60

A new approach for the spectroscopic investigation of non-volatile molecular nanoparticles with diameters in the range between 10 and 100 nm is proposed. The nanoparticles are produced in an electrospray with subsequent solvent evaporation. The number size distribution of the generated aerosol is determined with a Scanning Mobility Particle Sizer. Fourier transform infrared spectroscopy is used to study the vibrational dynamics of the nanoparticles. As an example, we have investigated sucrose nanoparticles with six different diameters between 36 and 82 nm. From a comparison with different bulk phase spectra, we conclude that sucrose aerosols are formed in a solid amorphous state.

1994

Somers, J.; Caperan, Ph.; Richter, K.; Fourcaudot, S., 1994, "Agglomeration of a titanium dioxide aerosol in a high intensity sound field," *Journal of Aerosol Science*, **25**(S1):355–56

The paper investigates the influence of a high intensity field on the agglomeration of droplets in the 0.02–0.5 μm size range. Results show that acoustic treatment could enhance the rate of decrease in concentration of droplets in the size range 0.08–0.5 μm . Measurements of the influence of acoustic waves on a solid aerosol were necessary to confirm these findings. Also results corroborate the measurements on droplet coagulation in this size range and the unexpected efficiency of acoustic agglomeration for particles with diameter as small as 0.08 μm is confirmed.

1991

Akhtar, M. Kamal; X., Yun; P., Sotiris E., 1991, "Vapor synthesis of titania powder by titanium tetrachloride oxidation," *AIChE Journal*, **37**(10):1561–570

Formation of titania particles by vapor-phase oxidation of titanium tetrachloride was studied in an aerosol reactor between 1,200 and 1,723 K. The effect of process variables (reactor residence time, temperature, and reactant concentration) on powder size and phase characteristics was investigated using the differential Scanning Mobility Particle Sizer, electron microscopy, and X-ray diffraction. Titania particles were primarily anatase though the rutile weight fraction increased with increasing reactor temperature. The geometric number average diameter of the particles was between 0.13 and 0.35 μm , and the geometric standard deviation of the particle size distribution was about 1.4. The average particle size increased with increasing temperature, inlet TiCl_4 concentration, and residence time. The observed changes in the particle size distribution were compared with those predicted by solving the aerosol dynamic equation by a sectional method and accounting for coagulation and first-order chemical reaction. While variations in the process variables resulted in discernible changes in the size of the particles, the spread of the distribution remained rather unaffected.

Graham, G.M.; Alam, M.K., 1991, "Experimental Study of the Outside Vapor Deposition Process," *Aerosol Science & Technology*, **15**:69–6

Outside vapor deposition has been studied experimentally, and the deposition process parameters have been examined. The system consisted of an industrial-type burner and a cylindrical bait rod. The following aspects of this process were studied: 1) the temperature profile of the bait rod surface, 2) the deposition efficiency measured in terms of the mass deposited SiO_2 , and 3) the size distribution of the deposited particles.

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