

11C.1**Flame synthesis and Characteristics of SiO₂-TiO₂**

Composite Nanoparticles. HEE-DONG JANG, Hankwon Chang, Kuk Cho, KIGAM; Daejeon, Korea; Soon-Joong Kim, Jin-Ho Park, Jeong-Woo Choi, Sogang University, Seoul, Korea

Composite nanoparticles have attracted much attention to improve the properties of single component nanoparticles such as low thermal expansion glasses, fatigue resistant optical fiber cladding, catalysts, etc. TiO₂-SiO₂ nanoparticles with various compositions for photocatalyst were synthesized from sprayed droplets of a mixture of TEOS and TTIP by flame spray pyrolysis. The effect of molar ratio between TEOS and TTIP in the mixture on the particle property such as particle morphology, average particle diameter, specific surface area, crystal structure, etc. was determined using TEM, XRD, BET, and FT-IR. A UV-spectrometer was used to characterize the photocatalytic property. As the molar ratio of TTIP/TEOS decreased by increasing TEOS concentration at the fixed TTIP concentration, the average particle diameter of the product powder increased and crystal structure changed from pure anatase to amorphous. The width of UV-absorption spectrum decreased with respect to the increase of the molar ratio. We also synthesized TiO₂-SiO₂ nanoparticles from sprayed droplets of a mixture of TTIP and SiO₂ nanoparticles by the same flame pyrolysis. As the weight fraction of SiO₂ nanoparticles in the mixture increased at the fixed TTIP concentration, the average particle diameter decreased and the crystal structure of all the product nanoparticles was found as pure anatase. UV-absorption spectrum was not changed with respect to the different fraction of SiO₂ nanoparticles.

11C.2**Flame Aerosol Synthesis of Phase-Pure Polymorphic Ceramic Oxide Particles: Effect of Particle Size.**

BING GUO, Mallika Mukundan, Texas A&M University, Zhiping Luo, Texas A&M University, College Station.

Many ceramic oxides such as Y₂O₃ are polymorphic. The different phases of a polymorphic material have significantly different material properties, and hence controlling the phase is desirable. We used a coflow diffusion flame aerosol process to study the effect of particle size on the crystal structure of Y₂O₃ nano- and micro-particles. The particles were characterized by X-ray diffraction and transmission electron microscopy. The monoclinic phase was formed when the flame temperature was sufficiently high, and when the particle diameter was below a critical size. A model based on particle melting-solidification and particle internal pressure induced by surface curvature was used to explain the findings. By removing large precursor droplets with an inertial impactor, phase-pure monoclinic Y₂O₃ particles were obtained.

11C.3

Synthesis of Nanoparticles for the Studies of Their Health Effects. MIRELLA MIETTINEN, Jorma Joutsensaari, Jorma Jokiniemi, University of Kuopio, Finland.

Nanotechnology will have an increasing impact on the every-day life of people in the industrialized countries. Thus, there are growing demands by the society for reliable and understandable information on the possible effects of engineered nanoparticles and the significance of these effects. It is essential that reliable information will be gathered before wide use of nanoparticles to avoid potential health problems of unknown nature.

Our project is a part of the NANOHEALTH research consortium, which aim is to synthesize and characterize nanoparticles, to delineate levels of nanoparticle exposure in laboratory and occupational environments, and to explore health effects (e.g. airway irritation and changes in the respiratory patterns, inflammatory responses of the lungs and pulmonary cells, and genotoxicity) of nanoparticles.

We have designed and constructed a flow reactor for controlled long term (over 5 hours) production of nanoparticles with high yield. Titanium dioxide (TiO₂) nanoparticles have been generated using titanium tetraisopropoxide (TTIP) as a precursor. Particle size distributions have been measured with ELPI, FMPS and SMPS. TEM samples have been collected for morphology studies. Up to 90 mg/m³ mass concentration with narrow size distribution and geometric mean diameter of TiO₂ agglomerates below 100 nm have been observed. The geometric mean diameter and the concentration of the agglomerates can be controlled via changing the experimental parameters.

With this flow reactor we can build up a highly controlled exposure environment for laboratory studies of health effects of engineered nanoparticles. As soon as the system is qualified with the TiO₂ particles, we will continue with other nanoparticles such as copper and cobalt.

11C.4

Synthesis of Bimetallic Noble Metal Aerosol Nanoparticles by Heterogeneous Spark Discharges. JEONG HOON BYEON, Jae-Hong Park, Ki-Young Yoon, Chul-Woo Park, Jungho Hwang, Yonsei University.

Recently, palladium-based membranes have been widely used in the separation of hydrogen and catalytic nanoparticle-deposited reactor because of their high hydrogen permeability. Pd-Ag, Pd-Pt and Pd-Au bimetallic nanoparticles have been recommended due to its better hydrogen permeability and improved mechanical strength against hydrogen embrittlement. Several preparation techniques have been used in a fabrication of the bimetallic noble metal membranes including of polyol synthesis, electroless plating, chemical vapor deposition, sputtering coating, and electrochemical synthesis. However, problems of high cost, complex chemical processes, and environmental unfriendly of among them should be remedied. In this study, the bimetallic noble metal aerosol nanoparticles prepared by heterogeneous spark discharges. The spark discharge is simple, inexpensive, environmental friendly and controllable method for stable generation of metal fine particles. The spark generation between noble metal electrodes was periodically obtained by discharging the electrodes, and produced sufficient local high temperature enough to evaporate a small fraction of the both electrodes, resulting in the generation of Pd-Ag, Pd-Pt and Pd-Au aerosol nanoparticles. The particle size distribution, morphology-element, and crystalline structure of the generated particles were observed by a scanning mobility particle sizer (SMPS), a HRTEM, and a XRD, respectively. [The authors acknowledge the supports from the Korea Institute of Environmental Science and Technology (KIEST) project under grant no. 013-071-052.]

11C.5**Water Droplet Formation in Humidified Nitrogen under Irradiation of 20 MeV Proton Beam and Corona Discharge.**

MASASHI IMANAKA, RIKEN (The Institute of Physical and Chemical Research); Shigeo Tomita, Suguru Kanda, Mitsuteru Fujieda, Shigeo Tomita, Hiroshi Kudo, University of Tsukuba.

Water droplet formation by ion-induced nucleation was studied by irradiation of 20 MeV proton beam and corona discharge. The experiments were conducted at the Tandem Accelerator Center, University of Tsukuba (UTTAC). The sizes of charged water droplet formed inside the humidified nitrogen were measured by a differential mobility analyzer (DMA) equipped with a Faraday cup electrometer (FCE). The droplets with 10 nm diameters are observed for 20 MeV protons, although they are not found for the corona discharge. This fact implies importance of the presence of both positive and negative ions, which attract each other by coulomb interactions. As a result, the enhancement of collision frequency allows in the formation of 10 nm droplets.

11C.6**Measurement of nascent charge distribution of nanoparticles and its manipulation in flame aerosol reactors.** JINGKUN

JIANG, Pratim Biswas, Washington University, St. Louis.

Nanoparticle charging is of great importance in both fundamental and applied aerosol research, as charged nanoparticles are utilizing in nanofabrication, aerosol measurement instrumentation and nanoparticle control technologies. Nanoparticles synthesized in high temperature aerosol reactors often carry certain charges, which can be further utilized in the electrostatic manipulation, measurement and control of such particles. The objective of this study was to measure and manipulate the charge distribution of nanoparticles synthesized in a flame aerosol reactor. The measured particle charge distribution was compared to the numerical results from a nanoparticle charging model (Jiang et al., 2007a & 2007b) considering both diffusion and thermionization mechanisms. To manipulate the nanoparticle charge distribution, soft X-ray irradiation was added in the flame environment. Soft X-ray photons knock out electrons from particles, resulting in positive charged particles. For this case, photoionization charging was included in the charging model to calculate the resultant particle charging distribution. The particle charge distribution was also altered by passing through a unipolar charger after they left the flame aerosol reactor, where diffusion charging was the main mechanism. The implications of these studies on nanoparticle synthesis and nanofabrication will be discussed.

References:

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11C.7

Micro/nano Patterning by Electrostatic Atomization with Controlled Frequency by Applying AC Superimposed on DC Fields. JOONGHYUK KIM, Hyun Cheol Oh, Sang Soo Kim, KAIST, Korea.

Micro/nano patterning technology has been considerably interested in many research groups for NT, IT and BT, for example, circuit and micro/nano electronic device for semiconductor, protein chip, quantum dot, and so on. Currently patterning technology has been developed by electron beam lithography, nanoimprinting and etc. However, these methods are passive process so that pattern does not have complicated structures, or difficult and time-consuming process. Therefore, the electrostatic atomized pattern of ultrafine droplets including nanoparticles easily and actively has been studied in this study. The existing electrospray technology is difficult to spray on demand. To overcome this problem, droplets were generated in the microdripping mode of electrospray with specific frequency by applying AC or pulsed voltage to the stainless nozzle. The experimental liquid was carried out with ethanol as conducting liquid dispersed polystyrene latex particles. The flow rate was controlled through hydrostatic pressure. The velocity and size of spray were measured by CCD camera. The pattern adhered nanoparticles was generated by sliding the ground substrate controlled by computer. This pattern was observed by microscope and scanning electron microscope. In the experimental results, pulsed droplets in the microdripping mode were dominated by AC superimposed on DC fields. The desired size and shape of pattern were observed by residual nanoparticles in the substrate where droplets were evaporated. It is expected that the new types of micro/nano and bio structures are developed by applying this technology.

11C.8

Particle Deposition for Nanopatterning Controlled by Highly Charging of Silver Nanoparticle Using Condensation and Evaporation Method. JOONGHYUK KIM, Sang Soo Kim, KAIST, Korea.

The research of nanoparticle charging and deposition has been basically considered to apply nanopatterning technology for semiconductor circuit, nanoelectronic device and so on. Mainly used nanopatterning methods, electron beam lithography and nanoimprinting are top-down process, and they have complicated processes or make only simple structures. To overcome these problems, the bottom-up process, such as build-up to control nanoparticles electrically like Lego blocks has been considered. However, nanoparticle is not charged easily so that it is difficult to control or pattern. In this study, basic pattern was generated by the highly charged nanoparticle adhesion to substrate using the condensation and evaporation method. Silver nanoparticles were grown into microdroplets through the condensation in the ethylene glycol as condensable vapor. The diameter of condensed droplet was about 2 micrometer enough to charge highly by indirect corona-based charger. These droplets were evaporated through the thermal evaporator, and charges remained at nanoparticles. Nanoparticles with the diameter of several tens nanometer had 30-50 charges. Highly charged nanoparticles adhered to substrate to charge the opposite polarity, and they are observed by scanning electron microscope. The basic patterns consisted of nanoparticles were developed. The manufacture of nanoparticle pattern and nanostructures is expected by applying this technology.

11C.9

Gas-Phase Organic Passivation of Aerosolized Silicon Nanoparticles: Mobility Diameter Growth and Chemical Characterization. JASON HOLM, Jeffrey T. Roberts, University of Minnesota.

Silicon nanoparticles may soon be found in devices rivaling the performance of II-VI or III-V semiconductor nanoparticle-based devices. Before silicon nanoparticle applications become widespread, however, methods must be developed to control the reactivity and promote the desirable characteristics of freshly-produced particles. One approach to controlling that reactivity and to potentially impart further functionality to the particles is by bonding organic molecules to particle surfaces.

In this work, silicon nanoparticles are synthesized via gas-to-particle conversion in a non-thermal plasma, and particle surfaces are modified *in flight* within an aerosol flow tube reactor immediately downstream from the plasma. In addition to producing high quality crystalline particles, the plasma production method provides several features which lend themselves well to organic processing in the aerosol state. First, particle agglomeration is significantly reduced due to the charged particle nature inherent to the plasma technique. This allows modification of discrete particles. Second, when silane is used as the particle precursor gas, the freshly-produced particles are hydrogen-terminated. This provides a convenient starting point for imparting organic passivation and functionality.

Organically modified particles are characterized in terms of electrical mobility diameter growth using a tandem differential mobility analysis apparatus, and chemical composition using *in situ* Fourier transform infrared (FTIR) spectroscopy in conjunction with a hybrid gas cell developed in our group. The mobility classification method is known to be able to readily detect mobility diameter changes as small as those associated with desorption of a hydrogen layer, and has been used as a diagnostic tool to characterize monolayer formation on aerosolized silicon nanoparticles. FTIR is widely used to examine chemical bonding environments associated with organic molecules. For that reason, its application is extended as a tool to characterize *in situ* changes in the silicon nanoparticle surface chemistry as simple terminal alkenes and alkynes are adsorbed to discrete particle surfaces.

11C.10

Characterization of Diffusion Flame Synthesis of Single-walled Carbon Nanotubes. CHAD UNRAU, Richard Axelbaum, Pratim Biswas, Washington University in St Louis; Phil Fraundorf, University of Missouri-St Louis.

Single-walled carbon nanotube (SWNT) formation in ethylene and acetylene diffusion flames is analyzed to understand the role of catalyst particle composition and flame environment on SWNT growth rate by using an SMPS system. Fuel/air diffusion flames typically contain PAH and soot, and thus are not suitable for nanotube growth. By using a combination of oxygen-enrichment and fuel dilution, soot formation is suppressed and PAH formation is minimized. Furthermore, an inverse flame configuration is used to create a flame environment that is favorable for SWNT growth. Measurements were made in both ethylene and acetylene diffusion flames to determine the effect of fuel type on nanotube formation. The optimum flame conditions and location for SWNT growth as well as the optimum catalyst particle composition were determined *in situ* using a scanning mobility particle sizer (SMPS) to measure the size distribution of the flame. The optimum catalyst particle composition was determined by comparing the size distributions of three flames with the same temperature but different levels of oxygen-enrichment. Increasing oxygen-enrichment oxidized the catalyst particles from iron to iron(II) oxide to iron(III) oxide with SWNTs being produced primarily from iron(II) oxide. Thus, oxygen-enrichment can be used to eliminate soot formation and to optimize catalyst particle composition. Finally, the SMPS and flame velocity measurements were used to measure growth rate. SWNTs were found to form in the post-flame region and grow at a maximum rate of over 100 microns/s.

11C.11

Synthesis and Characterization of Doped Tin Oxide Nanocrystals for Gas Sensing Applications. GANHUA LU, Junhong Chen, University of Wisconsin-Milwaukee.

Tin oxide (SnO₂) nanocrystals are widely used as solid state gas sensing materials, catalysts, and transparent conducting electrodes. The physical and chemical properties of SnO₂ nanocrystals can be tuned by dopants due to the possible modification of the band structure. In metal-doped SnO₂, metal additives work as donor atoms and can increase the charge carrier concentration, leading to enhanced gas sensitivity and catalytic activity. In this poster, we report the synthesis and characterization of Au- and Pb-doped SnO₂ aerosol nanocrystals using a simple, convenient, and low-cost mini-arc plasma source. The nanoparticles are produced by direct vaporization of solid precursor Sn or SnO followed by a rapid quenching. The nanoparticle size distribution is measured online using a scanning electrical mobility spectrometer (SEMS). The product nanoparticles are characterized using various techniques including transmission electron microscopy (TEM), high resolution TEM (HRTEM), energy dispersive X-ray (EDX) spectroscopy, selected area diffraction (SAD), and X-ray diffraction (XRD). Gas sensing properties of as-produced doped SnO₂ nanoparticles will also be reported.

11C.12

Numerical Investigations on the Coating Uniformity of the Multiplexed Electro spray Deposition System. HYUNCHEOL OH, Kyoungtae Kim, Sangsoo Kim, KAIST, Korea.

Electrospray deposition is one of the innovative techniques for nanostructured particle films using nanoparticle suspensions and pyrolyzed thin films using precursor solutions. Compared with other spray deposition methods using ultrasonic atomization and mechanical atomization, electrospray deposition exhibits high deposition efficiency due to highly charges on droplets. Furthermore, electrospray operated in the cone-jet mode could generate quasi-monodisperse and sub-micron sized droplets.

Needs for large-area coating process are growing for industrial applications such as display and solar cell fabrications. For large-area coating via electrospray deposition, multiplexed electrospray composed of array of capillary nozzles should be used and the spray nozzles are usually scanned continuously during the coating the substrate. However, little has been investigated for the deposition characteristics using the multiplexed electrospray deposition. The important issue of films deposited by multiplexed electrospray is a coating uniformity over the large area of the substrate.

In this work, spray evolution and deposition patterns were investigated numerically for 2-nozzle electrospray deposition system of nozzle-extractor configuration with varying electric field strength. A Lagrangian physical model was developed taking drag force, electric force by external electric field, mutual electric forces between the charged droplets, the image force induced on the conductive substrate and gravity into account. Droplet size and velocity needed for simulation input data were measured with a PDPA system.

For conventional multiplexed spray deposition, the coating uniformity was deteriorated by overspray at the interface of adjacent spray. However multiplexed electrospray deposition shows good thickness uniformity compared to other conventional multiplexed spray deposition methods due to electrostatic self-dispersion effects of charged droplets.

The coating uniformity exhibits a high dependence on the electric field strength and shows best results at the optimal range of the electric field strength. The multiplexed electrospray deposition methods have a potential to be an efficient and economical means for uniform large-area deposition.

11C.13**Semiempirical Description For Nanosize Material**

Production. MICHAEL P. ANISIMOV, Institute of Chemical Kinetics and Combustion, Siberian Division of the Russian Academy of Sciences, Novosibirsk, Russia

The essence of nanotechnology is the ability to work at the atomic, molecular and supramolecular levels, in the length scale of about 1-100 nanometers [1]. Various technologies for nanoclusters production are based on nucleation phenomena. Nanoclusters have broad field of applications like catalysts, coatings, sensors, miniaturizing of electronic components, design of effective medication, detection of gas phase compounds etc. A number of different theoretical alternatives have been proposed. On the other hand, experimental techniques for measuring nucleation of simple molecules are very few. Promising way to get the common description of nucleation is associated with the idea of semiempirical design of nucleation rate surfaces over the phase equilibria diagrams [2].

The nucleation rate surfaces are arising from lines of phase equilibria [3]. Lines of interface equilibria can be continued through triple point to area of unstable equilibria. One gets the multifold nucleation rate surface in that case. Multi-surface nucleation rates can be met more often than we usually think. It should be avoided the interpretation of multi-surface nucleation rate data in the one-surface approximation. That will give more adequate platform for inter-comparison of the experimental results and nucleation theory. Reliable nucleation rate surfaces play the key role in the choice of nucleation conditions and can be widely used for technology development which is directed to nanosize material production.

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11C.14**Iron Oxide Nanoparticle Aerosol Gel Formation in**

Counterflow Diffusion Flames. Hector Ruiz, YANGCHUAN XING, University of Missouri-Rolla.

Iron oxide nanoparticles are synthesized in a counterflow diffusion flame reactor from thermal decomposition - oxidation of iron carbonyl. At high precursor loading, the nanoparticles form a gel-like structure at the stagnation planes. It is observed that the nanoparticles in the gels are sintered, therefore forming rigid porous aggregates of nanoparticles. Due to the strain of gas flow, the aggregates have a flat sheet structure with about one nanoparticle thickness but hundreds of particles wide. In this presentation, we will report the synthesis and characterization of such iron oxide aerosol gels.

11C.15

Fabrication of Ag Nanoparticles-Based Devices by an Aerosol Process for Bio-Sensing Applications. D.-H. TSAI, M. R. Zachariah, University of Maryland and the National Institute of Standards and Technology; S.-J. Tsai, H.-C. Kan, S.-H. Guo, and R. J. Phaneuf, University of Maryland and Laboratory of Physical Science.

We demonstrate a gas-phase process to fabricate Ag nanoparticles-based devices for bio-sensing applications. Ag nanoparticles, prepared from the gas phase are size-selected via electrostatic classification. Particle size-dependent behavior on the metal-enhanced fluorescence (MEF) is observed in our experiments, and the trend is consistent with the field-enhancement near particles by numerical simulations. The fluorescence intensity with Ag nanoparticles can be >300 times higher than the one without Ag nanoparticles.

Moreover, directed self-assembly of these Ag nanoparticles onto an electrostatic-patterned biased P-N junction substrate is also investigated. High-selectivity nanoparticle patterning can be achieved under a desirable distribution of electric field (reverse bias, the width and the spacing of P-type region). A Brownian Dynamic model is applied to simulate our experiments numerically. Our approach provides a promising tool for self-assembling other functional nanoparticles for future device applications.

11C.16

Mobility Characterization of SAM Functionalized Au Nanoparticles. D-H. TSAI, L. F. Pease III, R. A. Zangmeister, M. J. Tarlov, M. R. Zachariah University of Maryland and the National Institute of Standards and Technology.

We present results from a systematic examination of functional Au nanoparticles in the gas phase through an electrospray (ES) process of Au colloids, and characterized by differential mobility analyzer (DMA). From the mobility analysis, we identified the co-existence of salt impurities and the nominal sized Au nanoparticles in commercially available unconjugated Au colloids. Formation of self-assembled monolayers (SAMs) of thiolated aliphatic molecules conjugated on the Au surface was detected from a change in particle mobility, and the change of surface packing density of SAMs on Au nanoparticles can be traceable by DMA. A kinetics study of temperature-programmed desorption (TPD) of SAMs on the Au surface indicated that SAM

11C.17

Stability Characterization of Colloidal Gold by Gas-Phase Differential Mobility Analysis-Kinetic Study D-H. TSAI, L. F. Pease III, R. A. Zangmeister, M. J. Tarlov, and M. R. Zachariah, University of Maryland and National Institute of Standards and Technology.

Most nanoparticle material are either synthesized or are suspended in the liquid state. The nature of these particles in the colloidal state is of considerable interest. We demonstrate gas phase electrophoretic mobility analysis as a new method to investigate colloidal aggregation, and the parameters that govern it. Nanoparticle suspensions were characterized by sampling a Au nanoparticle colloidal solution via electrospray (ES), followed by differential ion-mobility analysis (DMA) to determine the mobility distribution, and thus the aggregate distribution. The ES-DMA method allows us to quantitatively monitor the distribution of n-mers. By sampling at various times, we determine the degree of flocculation and the flocculation rate, which we find to be inversely proportional to the ionic strength and proportional to the residence time. From these data we obtain a stability ratio at different ionic strengths and the critical concentration. From this we then determine the surface potential or surface charge density. This method should be a generically useful tool to probe the early stages of colloidal aggregation.

11C.18

Art Glass Colouring Using Liquid Flame Spray Generated Nanoparticles. JYRKI MAKELA

A flame based colouring method for glass items is investigated. In the method, Liquid Flame Spraying (LFS), the feedstock liquid is atomised by fuel gases at the nozzle exit. The spray mixes with the flame and nanoparticles are formed via evaporation, chemical reactions and nucleation. These nanoparticles are impacted from the aerosol phase on hot glass and they produce a coloured coating on the surface layer of the glass. Only the outermost layer of roughly 10 micrometers is coloured, and the bulk glass will remain uncoloured. For the colouring effect, one needs to control the size of formed metal or oxide particles and their deposition on the surface. Also diffusion of the colouring material inside the glass needs to be considered. Depending on the material, the colouring effect is either due to absorption of light in the ionic colouring compounds (e.g. cobalt for blue) in the glass matrix or, due to absorption and scattering from colloidal nanoparticles inside the glass (e.g. silver for yellow and gold or copper for red).

In earlier studies, the method has been developed and used to obtain blue and yellow. Recently, red colour in glass has gained great interest. The present work focuses on red which is not yet thoroughly established. Here, acidic gold solution is sprayed to create the colloidal particles inside the glass. In the presentation, this flame based method will be introduced and both aerosol analysis from the flame as well as morphology of the particles in the glass matrix will be described.

11C.19

Current Characterization Studies of a Candidate Carbon Nanotube Reference Material at NIST. RABIA OFLAZ SPATZ, Rolf Zeisler, and Rick L. Paul, National Institute of Standards and Technology.

Carbon nanotubes are promising candidates for numerous applications because of very unique nanostructures and possess remarkable mechanical and electronic properties. For this reason there is an urgent need for standards for carbon nanotube (CNT) manufacturers, as well as for scientific and health-related investigations done with CNTs. In response to this need, we have analyzed two groups of CNT materials. The first material consisted of a batch of about 200 g, and distributed to NIST in 0.5 g units. Instrumental neutron activation analysis (INAA) and cold neutron prompt gamma-ray activation analysis (CNPAA) were employed to characterize this material taking advantage of the techniques' non-destructive character for the determination of total element concentrations. Informative compositional values were established for 23 elements based on the calibrated analytical systems, and limits of detection were reported for an additional 22 elements sought in the material. The initial chemical analysis was somewhat impeded by unknown homogeneity of test portions prepared from the individual units. Results showed relative standard deviations ranging from a few percent for several key elements to 50 percent and more for elements that may be considered contaminants in the CNT material. A better method of preparation was found resulting in reasonable uncertainties for major matrix constituents. A second group of materials was submitted in four different diameters and lengths for characterization. One was a single-walled CNT material with 1-2 nm diameters, and the others were multi-walled CNTs with <8 nm, 10-20nm, and 30-50nm diameters. Results will be discussed in the presentation.

11C.20

Synthesis of Silica Nanopowder from Siliceous Mudstone. KUK CHO, Hankwon Chang, Hee-Dong Jang, Korea Institute of Geoscience and Mineral Resources, Korea; Jin-Ho Park, Se-Young Oh, Sogang University.

Silica nanopowder has many applications and has been produced from various precursors. This work was focused on utilizing the abundant but less used minerals such as siliceous mudstone to synthesize silica nanopowder. The process consisted of three steps. Firstly, aqueous solution of sodium silicate was made from siliceous mudstone by NaOH leaching. Secondly, aqueous solution of silicic acid was obtained from the aqueous solution of sodium silicate by using the hydrogen based cation exchange resin. Finally, silica nanopowder was synthesized from the aqueous solution of silicic acid by flame spray pyrolysis. The particle size was affected by the concentration of silicic acid and the droplet size that was dependent on the solution feed rate, the flow rate of dispersion air, and the property of the solvent. The type of solvent either ethanol or water was also an important factor because it affected the evaporation rate of droplets.

11C.21

Synthesis of Nanoparticles and Nanostructured Films Using Biological Complexes. CHRISTOPHER J. HOGAN JR., Luis B. Modesto Lopez, Pratim Biswas, Washington University in St. Louis.

Biomolecular complexes are well designed to serve specific functions. In this talk, we will present recent results on the synthesis of aerosol nanoparticles and subsequent deposition of these particles to create films from unique biomolecular complexes. The iron-protein complex ferritin was used for the synthesis of monodisperse iron oxide nanoparticles 8nm in diameter by electro spraying a solution of ferritin and subsequently volatilizing ferritin protein coats. Larger monodisperse particles were synthesized by electro spraying suspensions with higher ferritin concentration. Electro sprayed ferritin was deposited onto a heated substrate to create nanostructured iron oxide films. Chlorosome complexes from rare green-sulfur bacteria were also deposited and have the potential to act as dye sensitizers in the next generation of solar cells. It was found that chlorosomes remain active after substrate deposition and readily absorb visible light. The results presented show that aerosol processing of biomolecular complexes can be used for the production of highly functionalized materials.

16B.1

Developing a Scaling Law for Fractal Aggregate Sintering from MD Simulation. Takumi Hawa, MICHAEL R. ZACHARIAH, University of Maryland and NIST.

A simple modification to the Frenkel sintering law is developed for nanoparticle fractal aggregates, based on molecular dynamics (MD) simulations. The fractal aggregates investigated consist of up to 110 primary particles, with primary particles of 2.5 nm in diameter, and the fractal dimension of 1 (wire), 1.9 (complex), and 3 (compact). In addition simple prototype L- and T-shape aggregate were considered. We found that L-shape aggregates behaved similar to straight chains and thus did not impact the overall sintering time. By contrast T-shape aggregate sintering kinetics was controlled by the longest contiguous branch in the system (i.e. effective primary branch length). We found that sintering of fractal aggregates is a combination of local sintering processes of line-, L- and T-shape structures. As expected sintering time increases with increasing mass of the aggregate and with decreasing the fractal dimension. The sintering times normalized by the primary particle diameter showed a universal relationship which depends only on the number of particles in an aggregate and its fractal dimension. The MD results were found to be qualitatively consistent with a continuum viscous flow model, and was used as the basis from which a phenomenological sintering law for fractal aggregates could be derived. The phenomenological model is a power law modification of the Frenkel sintering equation. to include a dependence on the number of particles in a fractal aggregate and fractal dimension:

$$t = t_{\text{Frenkel}} * (N-1)^{0.68^{D_f}}$$

This relationship is amenable for use in phenomenological aerosol models that might include sintering effects.

16B.2

One step synthesis of photoactive TiO₂ nanoparticle supported noble metal catalysts (Pt/TiO₂, Pd/TiO₂ and Pt-Pd/TiO₂) in a flame aerosol reactor. JINGKUN JIANG, Pratim Biswas, Washington University in St. Louis; Vinay Tiwari, Virendra Sethi, Indian Institute of Technology (Bombay).

Supported noble metal catalysts have been used in many processes due to their high activity. On the other hand, dispersing noble metal on the support surface can also enhance the catalytic activity of the support by increasing its charge separation efficiency. Conventional methods to synthesize supported metal catalysts often require many steps and make themselves industrially unfavorable. In this study, one step flame synthesis was demonstrated useful for producing TiO₂ nanoparticles supported noble metal catalysts. While flame processes have been used to synthesize Pt/TiO₂, no reports are available in the literature wherein Pd/TiO₂ and bimetallic Pt-Pd / TiO₂ have been synthesized by aerosol routes. The presentation will report results of synthesis of Pt/TiO₂, Pd/TiO₂ and bimetallic Pt-Pd/TiO₂ catalysts in a flame aerosol reactor. As produced catalysts were characterized by X-ray diffraction (XRD), nitrogen adsorption (BET), Fourier transform infrared spectrometer (FTIR) and transmission electron microscope (TEM). The agglomeration of catalyst particles in aqueous phase might affect their activity. Therefore, the surface charges of synthesized catalysts in aqueous phase were characterized using dynamic light scattering (DLS). The photoactivity of these TiO₂ supported noble metal catalysts in degrading methyl orange dye was measured. Both platinum and palladium can suppress the formation of rutile TiO₂ in the flame reactor, as a result of which, more active anatase TiO₂ nanoparticles were produced. Nanosized noble metal nanoparticles (2~4 nm) were dispersed on 30~40 nm TiO₂ support. It was found that Pt/TiO₂ had much higher photoactivity than pure TiO₂ nanoparticles, while palladium had detrimental effect on TiO₂ catalytic activity and Pt-Pd/TiO₂ also showed poor photocatalytic activity. An optimum photocatalytic activity was observed at 1% Pt loading. The size effect of TiO₂ nanoparticles on the activity was also examined.

16B.3

Predictive Modeling of Flow Reactor for Nanoparticle Generation. DAVID HESSE, Battelle Memorial Institute; Amit Gupta, Battelle Toxicology Northwest.

A conjugate heat transfer fluid dynamics model has been developed to support design optimization of a C60 nanoparticle generation system used for inhalation toxicology exposures. C60 aerosol is flash vaporized within a heated quartz laminar tube reactor. At the outlet of the reactor, supersaturated C60 vapor is diluted with compressed nitrogen to generate condensed-phase C60 nanoparticle meeting size and concentration requirements for the exposures. The first phase of this research program focused on process flow variables that can impact the physical characteristics of C60 aerosol including reactor temperature profile, dilution efficiency, residence time, and particle impaction on reactor walls. It is demonstrated how modeling results can guide design enhancements. Specific examples will include the identification of factors affecting the reactor thermal profile and flow distribution that cannot be easily measured as well as changes in a dilution chamber design that reduce overall particle residence time and increase the uniformity of the aerosol size distribution. The second phase of this study describes implementation of the particle dynamic equation for aerosol fate and transport to quantify the chemical and thermodynamic performance of the reactor. This discussion will outline the solution strategy and illustrate examples including nucleation and an inter-phase diffusion growth term in the absence of full kinetics for a heterogeneous surface reaction model.

16B.4

Nanoparticle Agglomerates Penetration: Effect of Agglomerate Structure on Filtration Efficiency. SEONG CHAN KIM, Jing Wang, Mark S. Emery, David Y.H. Pui, University of Minnesota.

Most filtration studies have been conducted with spherical or similar shape particles due to the difficulty of the filtration behavior of irregular particles. Real particles often have a complicated structure which influences their deposition behavior, so it is desirable to investigate the filtration of nanoparticle agglomerates. The filtration efficiency test was conducted with silver nanoparticle agglomerates, with the agglomerate structure controlled by an annealing furnace temperature. The mobility diameter and mass of the silver nanoparticle agglomerates were measured using the DMA-APM system, allowing for a calculation of the fractal dimension, varying from 2.07 to 2.95, for various annealing furnace temperatures, ranging from ambient to 600C. Agglomerate particles show lower penetration due to their higher diffusion coefficient and larger interception length comparing with spherical particles which have the same mobility diameters. Agglomerate filtration modeling has been investigated in terms of diffusion, impaction and interception, and the results show a good agreement with the experiment result in the particle size range of 50 to 300 nm.

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16B.5

Synthesis of Core-shell Ta₂O₅/SiO₂ Nanocomposite Based Potential Multifunctional Computer Tomography (CT) Contrast Agent. SOUBIR BASAK, Pratim Biswas, Washington University in Saint Louis; Jinda Fan, Samuel Achilefu, Washington University School of Medicine.

Abstract

Computer tomography (CT) has become an essential noninvasive tool in medical imaging to supplement X-rays and ultrasonography due to its applicability to diagnose a large number of disease entities[1]. Although a number of institutions offer full-body scans for the general population, this practice remains underutilized compared to magnetic resonance imaging (MRI) due to its lack of study to produce multifunctional engineered contrast agent. Currently, barium sulfate and iodinated organic compounds are used as X-ray contrast agents. Ta₂O₅ is a high-index, low-absorption, radio opaque and chemically inert material. It has potential application in radiography[2] and bone replacement therapy apart from its proven applications like near-UV to near-IR antireflection, multilayer filter, laser, photographic lenses, capacitors, and thin-film components. Ta₂O₅ nanoparticle coated with silica may be one of the potential contrast materials for CT due to its chemical stability and capability of attaching various bioactive ligands through surface silanol groups by covalent bonding. This study is devoted to develop an alternative CT contrast agent based on core-shell type Ta₂O₅/SiO₂ nanocomposite using furnace aerosol reactor (FuAR), similar to a method described elsewhere[3]. Emphasis is given to understand the aerosol phase formation mechanism of silica coated Ta₂O₅ nanocomposite with controlled size, shape, crystallinity and morphology. Both uncoated and silica coated Ta₂O₅ are bioconjugated with dye (cypate-II) having transmittance in near-IR region via an amide bond in DMF. SMPS, FT-IR, XRD, TEM, EDX, zita sizer, UV-visible spectroscopy are used to characterize the as produced nanopowders and their derivatives which will further in vivo studied for CT and optical imaging applications.

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16B.6**Nanostructured Particles by Aerosol Assisted Self-Assembly.**

XINGMAO JIANG, Yung Sung Cheng, Jacob McDonald, Lovelace Respiratory Research Institute; C. Jeffrey Brinker, University of New Mexico and Sandia National Laboratories.

Wide applications in nanotechnology demand good control of the architectures and economic manufacturing methods for nanoparticles. In this talk we describe aerosol-assisted evaporation induced self assembly as a means to prepare various well defined nanostructured particles. Nanoporous core shell particles in which a Ce corrosion inhibitor is encapsulated within an ordered nanoporous silica shell are fabricated. The release can be controlled effectively over a wide range by the pore size, the pore surface hydrophobicity, the salt solubility, and the pH value of the release medium. Well controlled structures and surface chemistry of self-assembled nanoporous silica materials make them of interest for 'smart' fillers that could impart to coatings and paints long term corrosion inhibition properties through controlled release.

To understand well the evaporation induced self-assembly, we develop a valid computer code to simulate the coupled heat and mass transfer associated with moving boundaries for multicomponent droplet evaporation. For ethanol-water-NaCl model droplets it is predicted that only one single crystal NaCl is formed at center each particle, which agrees well with experimental finding for single crystal NaCl/silica core-shell structured particles. Based on the simulation, drug capsules and various hollow sphere particles are prepared template-free at room temperature by controlling interfacial mass/heat transport and sol-gel kinetics. Monodisperse nanoparticles for carbon, fullerenes, FePt, and metal oxides have been generated by nanosegregation or heterogeneous nucleation within evaporating droplets. Some examples of potential applications for the nanoparticles in nanotoxicology, arsenic removal, adsorption, catalysis, disinfection, drug delivery, and wave scattering are given.

17B.1**Dual-capillary Electrospaying for Coated Particle**

Generation. FAN MEI, Daren Chen, Washington University in St. Louis.

The generation of coated particles using a dual-capillary electrospaying system was investigated in this study. At the proper operational condition the dual-capillary electrospaying can produce core-shell structured nanoparticles. The control of the microstructure of produced capsules is also possible with the studied technique.

As the demonstration example, a biodegradable polymer, poly(lactic-co-glycolic acid) (PLGA), and a serum albumin protein, bovine serum albumin (BSA), were used as the outer layer or the shell materials. Gold colloids and sucrose were used as the core materials. The size distributions of produced capsules were characterized by scanning mobility particle sizer (SMPS). Morphology and microstructure of the capsules were characterized by scanning electron microscope (SEM), transmission electron microscopy (TEM) and Fourier transform infrared spectroscopy (FTIR). When the inner solvent was miscible with the outer solvent, the core materials were partially engulfed by the shell materials. The coating condition was, however, different when the inner and outer solvents were immiscible. The core materials were coated by the shell materials. With the increase of the outer liquid flowrate and shell material's concentration the coating layer or shell thickness can be increased.

17B.2

Production of cobalt and nickel nanoparticles by hydrogen reduction. Johanna Forsman, Unto Tapper, Ari Auvinen, VTT Technical Research Centre of Finland; JORMA JOKINIEMI, VTT Technical Research Centre of Finland and University of Kuopio.

Cobalt and nickel nanoparticles were produced by hydrogen reduction reaction from cobalt or nickel chloride precursor vapour in nitrogen carrier gas. Vaporisation of the precursor from a porous pellet bed enabled continuous operation and saturation of the carrier gas flow with the precursor vapour. The evaporation rate was controlled by changing the bed temperature. The design of the facility enabled very high mass concentrations, 40 g/m^3 , and fairly high production rate, 10 g/h , of nanoparticles even with a laboratory scale system. Mixing of hydrogen with precursor vapour in a vertical furnace enabled accurate control of reaction zone. Losses inside the furnace could be reduced to below 5%. The process was monitored on-line with Fourier transform infrared spectroscopy (FTIR) for gas phase chemical reactions. The produced cobalt and nickel particles were characterised using transmission electron microscopy, selected area electron diffraction and x-ray diffraction.

The produced particles were typically single crystals (Ni) or crystals with twinning structures (Co). Both nickel and cobalt particles had cubic fcc crystal structure with very limited neck formation. Oxidation of the particles was observed to be extremely slow, probably because of a thin layer of HCl was adsorbed on the surface. Depending on the evaporation temperature of the precursors, primary particle size varied approximately between 20 and 90 nm. The size distribution of primary particles was narrow; the sample standard deviation was 18 nm for 84 nm mean diameter. Primary particles formed chain like agglomerates likely because the particles are ferromagnetic. Such agglomerates enhance the magnetic properties of the produced powder.

The process is promising for scaling up, because it is one-step process, pressure in the system is atmospheric and temperatures are much lower than for cobalt or nickel evaporation techniques. In addition, no expensive precursors or additives are required in the process.

17B.3

Flame Synthesis of Nanostructured Stabilized Zirconia for Fuel Cell Applications. Ranjan Pati, Osifo Akhluemonkhan, Hillary Sadoff, SHERYL EHRMAN, University of Maryland.

Zirconia (ZrO_2) occurs in three crystallographic forms- monoclinic, tetragonal and cubic with monoclinic being the most stable form among these three. Although cubic and tetragonal phases are not the thermodynamically favored at room temperature, these desirable phases can be stabilized by the addition of various dopants including Y_2O_3 , CaO, MgO, CeO_2 , and CuO. In the present study, stabilized zirconia powders with different dopant (Y_2O_3 , CaO, MgO, CeO_2 , and CuO) concentrations are synthesized by a one step flame spray pyrolysis. In this process, aqueous solutions of precursors are atomized using a nebulizer. The atomized solution is then passed through the flame reactor. In the reactor, the precursors react in the solid phase, forming the final product. The particles are collected by thermophoresis onto a water-cooled surface. The materials are characterized by using FTIR spectroscopy, X-Ray Diffraction (XRD), transmission electron microscopy (TEM) and BET surface area. X-Ray diffraction study shows the formation of cubic and tetragonal phases of the as prepared powders. BET surface areas of the as prepared samples are in the range of 10-80 m^2/g depending on the dopants used. The tetragonal / cubic phase stabilization effect for the dopants are $\text{Y}_2\text{O}_3 < \text{CaO} < \text{CeO}_2 < \text{MgO} < \text{CuO}$, whereas the ionic radii are $\text{Ca}^{2+} < \text{Y}^{3+} < \text{Ce}^{4+} < \text{Mg}^{2+} < \text{Cu}^{2+}$.

17B.4

Production of Quantum Dots by Spray Pyrolysis. Hongwang Zhang, Sha Liu, MARK T. SWIHART, The University at Buffalo (SUNY).

Our recent work on synthesis of semiconductor nanocrystals, or quantum dots, by spray pyrolysis will be described in this presentation. In spray pyrolysis, a liquid precursor solution is delivered as an aerosol with a mean droplet diameter on the order of a micron and a broad size distribution. The precursor is to be converted into relatively monodisperse nanocrystals with a mean diameter below 10 nm. We are applying two approaches to achieve this, both of which rely on heating of the precursor aerosol in a conventional tube furnace. The first strategy is complete evaporation of both the solvent and precursor, followed by nucleation of semiconductor nanocrystals from the vapor phase. We have used this approach to produce ZnS nanocrystals from single-source precursors, zinc diethyldithiocarbamate and zinc dimethyldithiocarbamate, dissolved in volatile organic solvents. These ZnS nanocrystals exhibit efficient blue photoluminescence. We are currently extending this approach to produce CdS nanocrystals from the analogous cadmium dimethyldithiocarbamate precursor. A key challenge in this approach is avoiding contamination of the nanocrystals with organic by-products or solvent decomposition products. The second approach we describe is to adapt chemistries that have been used for solution phase synthesis of quantum dots. This requires using a high boiling point solvent that does not evaporate during the reaction, with surfactants that passivate the nanocrystal surfaces to prevent agglomeration. Each precursor-containing droplet acts as a femtoliter-scale reactor in which solution-phase chemistry occurs. These droplets can be heated and cooled much faster than macroscopic reaction vessels, and thus allow access to shorter reaction times and higher temperatures than conventional solution phase synthesis. We have produced CdS and CdSe nanocrystals using this approach. Both spray pyrolysis techniques described here provide a continuous, macroscale process for producing high-quality semiconductor nanocrystals for potential applications ranging from bioimaging and nanomedicine to photovoltaics and display devices.

17B.5

Synthesis of a Silica Aerosol Gel from the Aerosol Phase. RAJAN DHAUBHADEL, Amitabha Chakrabarti, Christopher M. Sorensen, Kansas State University.

During the aggregation process solid particles come together to form ramified aggregates with a fractal dimension of less than three. With such a dimensionality, the growing aggregates occupy a greater fraction of the available space with time and the system crosses over from cluster dilute to cluster dense and eventually gels, filling all the available space in the macroscopic volume. For a real aerosol system there is always a competition between gelation and gravitational settling of the aggregating particles. Gelation phenomenon wins only when the gel time for the system is significantly shorter than the gravitational settling time for the clusters. In previous work our group has shown that dense soot particles can gel in the aerosol phase. Here we again demonstrate the aerosol gelation phenomenon with silica nanoparticles. The aggregating silica particle system was created by instantaneous oxidation of silane with an oxidizing gas in a closed chamber. We studied the aggregation results by varying the inert background gas as well as the oxidizing gas. We have found that the background gas plays a very important role in cooling down and solidify the initially hot and molten silica nanoparticles. Our successful attempts in making silica aerosol gels also demonstrate the universality of gelation phenomenon in the aerosol phase. The silica aerosol gels were found to have very low density of 4 -6 mg/cc and high specific surface area of 300 - 500 cubic meter per gram. This aerosol gelation process may be extended to variety of materials and hence the process may find some technical values.

17B.6

Photo-Assisted Chemical Vapor Deposition of Organic Coatings on Aluminum Nanoparticles. Yuanqing He, JEFFREY ROBERTS, Steven Girshick, University of Minnesota.

Because they are pyrophoric and lightweight, aluminum (Al) nanoparticles are of potential interest as solid fuels in propulsion applications. A key technical challenge is passivating the Al surface against oxidation by ambient gases during storage. In this contribution, we describe how organic passivation layers can be deposited on Al nanoparticles in the aerosol state by photo-assisted chemical vapor deposition (photo-CVD) driven by vacuum ultraviolet (VUV) radiation. The Al nanoparticles were synthesized from aluminum trichloride (AlCl_3) in a thermal plasma torch and then extracted into an atmospheric-pressure aerosol flow tube reactor. Particle streams were mixed with a gas-phase coating precursor, generally methyl methacrylate [$\text{CH}_2=\text{C}(\text{CH}_3)\text{CO}_2\text{CH}_3$, MMA] and then exposed to radiation from an excimer lamp. Amorphous organic coatings, which for the MMA precursor have been tentatively identified as the polymer polymethyl methacrylate, were deposited on the particles through photo-assisted CVD. Tandem differential mobility analysis (TDMA) was employed to determine coating thicknesses as a function of initial particle size and precursor concentration. For Al particles of initial mobility diameters in the 30-60 nm range, coating thicknesses varied from the sub-nanometer level to 15 nm with MMA as the coating reactant, depending on MMA flow rate and the initial particle mobility diameter. Morphological and compositional analyses of bare and coated nanoparticles have been carried out using Fourier transform infrared (FTIR) spectroscopy and transmission electron microscopy (TEM).