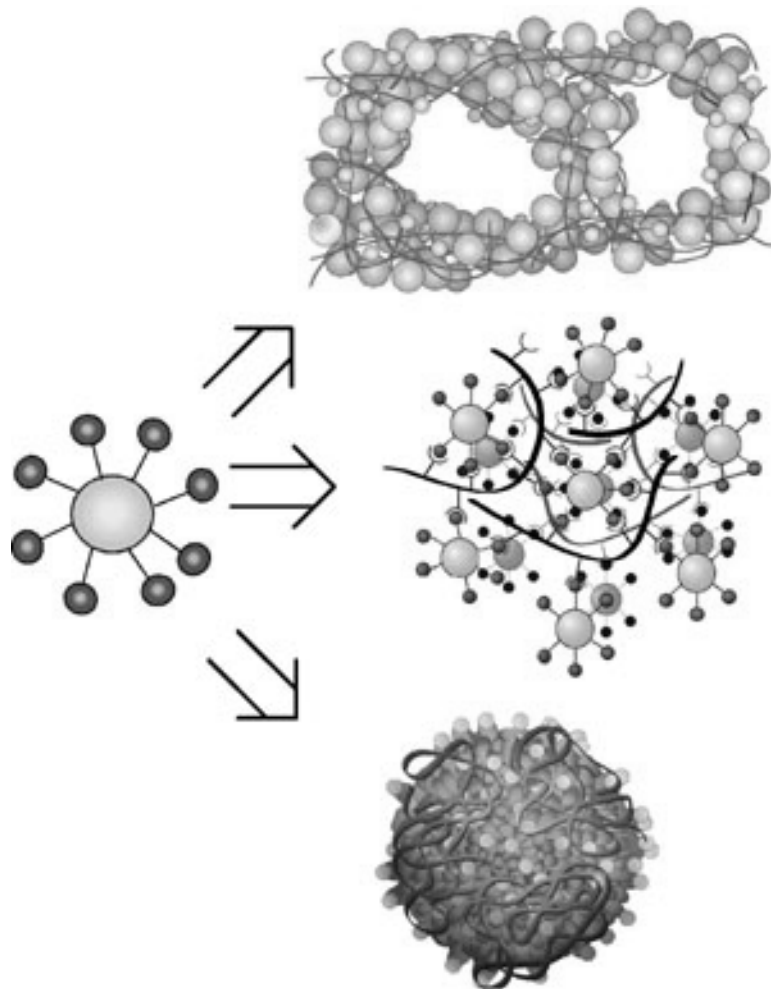


Particles 2005

Surface Modification in Particle Technology

13-16 August 2005

Sir Francis Drake Hotel, San Francisco,
California



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TABLE OF CONTENTS

Sponsors	4
International Organizing Committee	5
Exhibitors	7
General Program Schedule	9
Maps of Meeting Spaces	11
Technical Program	13
Abstracts of Oral Program	23
Abstracts of Poster Program	41
Author/Speaker/Presenter Index	83
List of Preregistered Conferees	87
Notes	103

Sponsors

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Exhibitors

Brookhaven Instruments Corporation

750 Blue Point Road
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CPS Instruments, Inc.

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Microtrac

12501-A 62nd Street North
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Surface Measurement Systems, Ltd.

5 Wharfside, Rosemont Road
Alperton, Middlesex
HA0 4PE, United Kingdom
44(0)208 797 9400
<http://www.smsuk.co.uk/>

General Program Schedule

Registration – Registration Desk (just outside of Windsor Room on Mezzanine level)

Saturday, August 13

(1800 – 1930) – Opening Reception, Mixer, & Exhibition (Franciscan Room and Mezzanine)

Sunday, August 14

(0830 – 1200) – General Session 1 (Empire Room - 2nd Floor)

(1200 – 1400) – Luncheon/Exhibition (Franciscan Room, Windsor Room, Mezzanine, and Renaissance Room)

(1400 – 1730) – General Session 2 (Empire Room - 2nd Floor)

(1800 – 1930) – Poster Session 1/Reception/Exhibition (Franciscan Room, Windsor Room, and Mezzanine)

Monday, August 15

(0830 – 1200) – General Session 3 (Empire Room - 2nd Floor)

(1200 – 1400) – Luncheon/Exhibition (Franciscan Room, Windsor Room, Mezzanine, and Renaissance Room)

(1400 – 1730) – General Session 4 (Empire Room - 2nd Floor)

(1800 – 1930) – Poster Session 2/Reception/Exhibition (Franciscan Room and Mezzanine)

Tuesday, August 16

(0830 – 1200) – General Session 5 (Empire Room - 2nd Floor)

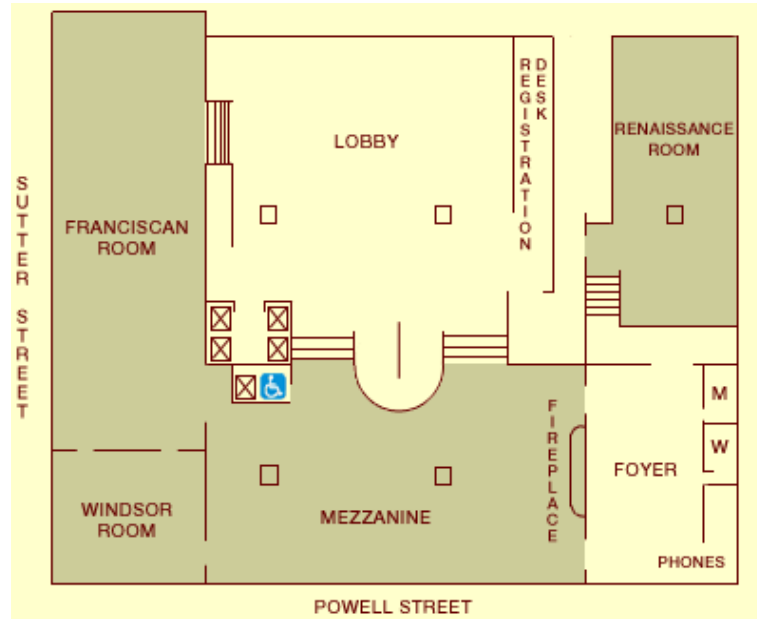
(1200 – 1400) – Luncheon/Exhibition (Franciscan Room, Windsor Room, Mezzanine, and Renaissance Room)

(1400 – 1730) – General Session 6 (Empire Room - 2nd Floor)

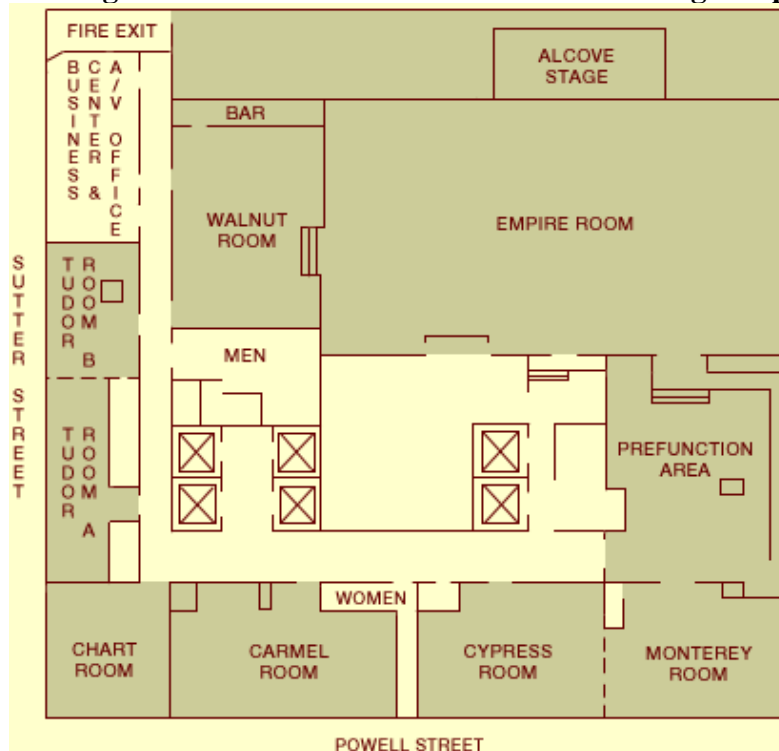
Conference Ends

Maps of Meeting Spaces

Mezzanine Level – Access via stairs from Lobby. For luncheon service, use stairs to Franciscan Room. For receptions, use stairs to Mezzanine. Registration will be in Walnut Room during General Sessions and in Mezzanine during receptions.



Second Floor - Access Empire Room through Walnut Room; registration will be in Walnut Room during General Sessions and in Mezzanine during receptions.



Technical Program

Sunday, August 24, 2003, Morning

General Session 1 (Empire Room)

Session Chair: **John Texter**, Eastern Michigan University

- 0830 **1. Helmut Schmidt**, Institute for New Materials, Saarbruecken
Chemistry based nanomaterials and their interdisciplinary processing to products on the market
- 0915 **2. Gobet Advincula**, University of Houston
Surface initiated polymerization for nanocomposite formation
- 0945 Break
- 1015 **3. T. Sudarshan**, Materials Modification, Inc.
Thin metallic coatings of particles for advanced heat transfer applications
- 1100 **4. Giulio Paciotti**, Cytimmune Sciences
Practical challenges in merging materials science and therapeutics in approaching clinical trials for nanoparticulate delivery systems
- 1130 **5. Brian Benicewicz**, Rensselaer Polytechnic Institute
RAFT polymerization from nanoparticle surfaces for nanocomposites
- 1200 End of Session

Sunday, August 14, 2003, Afternoon

General Session 2 (Empire Room)

Session Chair: **Sami Karaborni**, Merck

- 1400 **6. Angelos Kyrilidis**, Cabot Corporation
Modified carbon black for black matrix applications
- 1445 **7. John Crocker**, University of Pennsylvania
Crystallization of PEG-modified colloids using DNA-hybridization: Reducing the effects of non-specific binding
- 1515 Break
- 1545 **8. Gero Decher**, University of Strasbourg
Layer-by-layer assembly for the stabilization and functionalization of nanoparticles
- 1630 **9. Ron Cook**, TDA Research
Designing nanoparticles for functional materials and coatings
- 1700 **10. Bernie Bauman**, Fluoro-Seal International
Modification of polymer particle surfaces – From concept to multinational business
- 1730 End of Session

Technical Program (continued)

Sunday, August 14, 2003, Evening

Poster Session 1 (Franciscan Room and Mezzanine)

Session Chair: **Melanie Martin**, Particles Conference
1800-1930

- 30. Federica Bondioli**, University of Modena
Synthesis of nanoparticles in a continuous-flow microwave reactor
- 31. Li-Chen Chang**, Yuan Ze University
Influence of particle stacking structure in catalyst layers in the performance of proton exchange fuel cells
- 32. Yu-Chen Chang**, Yuan Ze University
Characteristics of silica nanoparticles derived under different combustion conditions in a low-pressure flat flame
- 33. Chao-Hung Chang**, University of Science and Technology, Taiwan
Surface modification of silica gel for removing toxic components while retaining active components in the preparation of Chinese herbal medicine
- 34. Shu-Hua Chien**, National Taiwan University
Noble metal nanoparticles on titania nanotubes: Preparation, characterization and catalytic properties
- 35. Miguel Correa-Duarte**, Arizona State University
Surface modification of carbon nanotubes by deposition of nanosized metallic and semiconductor particles
- 36. George Dougherty**, Lawrence Livermore National Laboratory
Zeta potential behavior of functionalized metallic nanorod particles in aqueous solution
- 37. Teddy Keller**, Naval Research Laboratory
Carbon nanotube formation during the carbonization process in solid compositions
- 38. Lars Kilaas**, SINTEF Materials and Chemistry
Hydrophilic particles produced by an inverse minisuspension polymerization method
- 39. Jin-Chun Kim**, Korea Institute of Machinery & Materials
Noble synthesis of Fe/SiO₂/CNT nanocomposite powders by chemical vapor condensation
- 40. Yong Seok Kim**, Korea Research Institute of Chemical Technology
Preparation and properties of polymeric nanocomposites based on high-temperature organophilic layered silicates

Technical Program (continued)

- 41. Eric Laarz**, YKI Institute of Surface Chemistry, Stockholm
Synthesis and inkjet-printing of spherical silica pigments with incorporated organic dye molecules
- 42. Woo Lee**, Stevens Institute of Technology
Synthesis of cellular catalytic structures via self-assembly of nano- and microparticles for microreactor systems
- 43. Jean-Luc Lemyre**, University of Laval
Synthesis of lanthanide fluoride nanoparticles of varying shape and size
- 44. Dongling Ma**, National Research Council of Canada
Coating of superparamagnetic nanoparticles with silica
- 45. Yoshitake Masuda**, Nagoya University
Surface modification of particles and substrates with oxides, metals or organic molecules
- 46. Harri Harma**, University of Turku
Synthesis and characterization of optimally-packed europium(III) nanoparticles for time-resolved fluoroimmunoassay of prostate-specific antigen
- 47. Michael Baksh**, University of California, Berkeley
Lipid membrane coated microspheres for label-free molecular detection
- 48. Joongho Moon**, Nomadics, Inc.
Fluorescent polymer nanoparticles: Fabrication, characterization, and application
- 49. Kohei Soga**, Tokyo University of Science
Processing of upconversion emissive particles for bioimaging
- 50. Robert Nooney**, Dublin City University
Plasmonic enhancement of fluorophores in solution using core-shell nanoparticles
- 51. Yosuke Okamura**, Waseda University
Haemostatic effects of polymerized albumin particles carrying fibrinogen g-chain dodecapeptide as platelet substitutes in vitro and in vivo
- 52. Norihito Sogoshi**, Saitama University
Magnetic colloidal crystals and their magneto-optical properties
- 53. Shane Street**, University of Alabama
Dendrimer-mediated nanoparticle formation: Photoreduction and magnetic nanoparticles

Technical Program (continued)

- 54. Dayane Tada**, Universitária São Paulo
Synthesis and photochemical photophysical properties of silica nanoparticles functionalized with methylene blue and thionin
- 55. Fang-Li Yuan**, Chinese Academy of Sciences
Preparation of surface coated zinc oxide nanoparticles
- 56. Tania Tannahill**, Clarkson University
Synthesis and characterization of mixed metal oxide nanoparticles
- 57. Nuria Tricas**, Universitat Ramon Llull
Carbon black surface chemistry modification by using plasma techniques (Low and atmospheric plasma)
- 58. Aine Whelan**, Trinity College, Dublin
Silver nanoparticle biosensors for DNA diagnostics
- 59. Helene Yockell-Lelievre**, Laval University
2D self-assembly of gold-polystyrene core-shell nanoparticles
- 60. Andrei Stanishevsky**, University of Alabama at Birmingham
Fabrication of metal nanoparticle ensembles by thermal processing of a polymer/nanoparticle composite
- 61. Jung Min Lee**, Yonsei University
Hyperdense and square lattice-free colloid crystals from highly charged monodisperse poly(styrene/NASS) particles with APTMS-modified glass substrate
- 62. B. Jeyadevan**, Tohoku University
Synthesis and surface modification of metal particles by polyol process
- 63. B. Jeyadevan**, Tohoku University
Preparation of magnetic nanoparticles for magnetic fluids hyperthermia
- 64. Matti Ben-Moshe**, University of Pittsburgh
Controlled synthesis of highly monodisperse zinc sulfide nanospheres for utilization in crystalline colloidal arrays
- 65. Takyua Tsuzuki**, Nanotechnology Limited
Application of silicone coating on nanoparticles in aqueous environment

Technical Program (continued)

Monday, August 14, 2003, Morning

General Session 3 (Empire Room)

Session Chair: **James Belmont**, Cabot Cororation

- 0830 **11. David Thompson**, Purdue University
Surface modification of Fe/Au and non-covalent nanoparticle assemblies for therapeutic applications
- 0915 **12. Andreas Fery**, Max Planck Institute for Interfaces
Layer-by-layer microcapsules: Towards controlling capsule mechanics and adhesion on the nanoscale
- 0945 Break
- 1015 **13. Ganesh Skandan**, NEI Corporation
Leveraging SBIRs and contracts for technology development
- 1100 **14. Si-Shi Feng**, National University of Singapore
Vitamin E TPGS Coated nanoparticles of biodegradable polymers for cancer Chemotherapy and medical imaging
- 1130 **15. Richard Brotzman**, Nanophase Technologies
An integrated platform of scaled nanotechnologies - Vapor phase synthesis, nanoparticle surface treatment, and dispersion - To enable commercial applications
- 1200 End of Session

Monday, August 15, 2003, Afternoon

General Session 4 (Empire Room)

Session Chair: **Yoshitake Masuda**, Nagoya University

- 1400 **16. Jim Adair**, Penn State University
Controlling particle morphology with surface adsorbed species
Modifying surfaces to direct nanocomposite formation
- 1445 **17. Karen Buechler**, ALD NanoSolutions
Particle ALD for developing novel materials through nanocoating
- 1515 Break
- 1545 **18. Sven Holger Behrens**, BASF, Ludwigshafen
Surface modification of particles and emulsion droplets with functional polymers
- 1615 **19. Robert Nick**, Cabot Corporation
Functionalization of colorants for marking applications
- 1645 **20. Emmanuel Giannelis**, Cornell University
- 1730 End of Session

Technical Program (continued)

Monday, August 15, 2003, Evening

Poster Session 2 (Franciscan Room and Mezzanine)

Session Chair: **Melanie Martin**, Particles Conference
1800-1930

66. Ciao-Cheng Chang, Yuan Ze University

Synthesis and characterization of titania nanoparticles prepared in a low-pressure flat flame

67. Yu-Chen Chang, Yuan Ze University

Detailed internal microstructure of hollow zirconia microspheres derived by sol-spray technique

68. Yu-Chen Chang, Yuan Ze University

An EXAFS study of platinum nanoparticles synthesized by a low-pressure flat flame

69. Kuei-Yuan Cheng, Yuan Ze University

Time evolution of morphology and size of hydrothermally prepared zirconia sol particles

70. Hyoung Jin Choi, INHA University

Synthesis and electroresponsive characteristics of monodisperse polymeric microbeads with conducting polyaniline shell

71. Mustafa Demir, Max Planck Institute for Polymer Research, Mainz

Synthesis of ZnO nanocrystals of narrow size distribution via esterification of zinc acetate dihydrate with alcohols

72. Jakob Dreyer, Lund University

The interaction between functionalized soft polymer particles and inorganic particles

73. Motoyuki Iijima, University of Agriculture and Technology

Surface interaction and dispersion stability control of silica nanoparticles in MEK by surface modification using silane coupling agents

74. Michael Juhnke, Clausthal University

Generation of pure nanoparticles for pharmaceutical applications by high-energy milling

75. Lidvin Kjerengtren, South Dakota School of Mines and Technology

Fillers for dimensionally stable materials

76. Anthony J. Kim, University of Pennsylvania

Engineering DNA-mediated colloidal self-assembly - System design and crystallization

77. Seong Hun Kim, Hanyang University

Technical Program (continued)

Multi-walled carbon nanotube-reinforced poly(ethylene-2,6-naphthalate) nanocomposites by melt blending

78. Kenneth Lau, MIT

Initiated chemical vapor deposition of polymers on micro and nanoparticles

79. Ingo Leubner, Crystallization Consulting

Size control of nanoparticles using surface adsorbers during precipitation

80. Maricel Marquez, University of Oklahoma

Nano and micro-patterned structures of poly(aniline) from admicellar polymerization

81. Maricel Marquez, University of Oklahoma

Effect of polymer-surfactant coatings on the rheological properties of hydrophilic particulates

82. Hanna Mouaziz, CNRS-bioMerieux, Lyon

Elaboration of hydrophilic, cationic and submicron magnetic particles for biomedical use

83. Jin-Gyu Park, Samsung Cheil Industries, Inc.

Uniform metal layer formation on monodisperse polymer particles for anisotropic conductive interconnection in packaging devices

84. Martin Piech, Sandia National Laboratories

Photoactive colloids: Light driven aggregation and deposition

85. Vitali Polonski, CSIRO, Sydney, Australia

Controlled nanoscale surface modification using optical near-field driven processes

86. Björn M. Reinhard, University of California, Berkeley and Lawrence Berkeley National Laboratory

A molecular ruler based on plasmon coupling of single gold and silver nanoparticles

87. Keisuke Sato, Tokyo Denki University

Fabrication and luminescence properties of size controlled nanocrystalline silicon particles

88. Alex M. Smirnov, North Carolina State University

Modifying the nanopore surface with lipid and protein for flow-through membrane-protein bio-chips

89. Leonard Radzilowski, Tyco Electronics

Enhanced laser mark contrast pigments from core-shell nanoparticles

90. Veronica Salgueirino-Maceira, Arizona State University

Colloidal synthesis of magnetic core-shell particles

Technical Program (continued)

- 91. Young-Seok Shon**, Western Kentucky University
Chemical and heating treatments of stable ionic monolayer-protected gold nanoparticles
- 92. Benedikt Steitz**, Ecole Polytechnique Federale de Lausanne
Modified superparamagnetic iron oxide nanobeads for single cell RNA extraction
- 93. Jacek J. Swiatkiewicz**, South Dakota School of Mines
Surface modification of nanoreactant particles used in exothermic condensed-phase reactions
- 94. Shinji Takeoka**, Waseda University
Haemostatic effects of polymerized albumin particles carrying recombinant glycoprotein Iba as platelet substitutes *in vitro* and *in vivo*
- 95. Matthias Thommes**, Quantachrome Instruments
Effect of surface chemistry on the sorption, wetting, and phase behavior of water and simple fluids in novel ordered mesoporous materials
- 96. Huan-Hsiung Tseng**, Yuan Ze University
An XPS study of platinum nanoparticles prepared by a low-pressure flat flame
- 97. Jeroen Van den Bossche**, Purdue University
Synthesis and grafting of folate-PEG-thioctic acid conjugates to Au nanoparticles for selective targeting of cancer cells
- 98. Hiroshi Yabu**, Hokkaido University
Novel preparation method of polymer nanoparticles

Tuesday, August 16, 2003, Morning

General Session 5 (Empire Room)

Session Chair: **Lee Tutt**, Eastman Kodak Company

- 0830 **21. Richard Partch**, Clarkson University and University of Florida
Nanoparticle surface effects on chemical detoxification
- 0915 **22. Atsushi Takahara**, Kyushu University
Surface-initiated nitroxyl-mediated radical polymerization
- 0945 Break
- 1015 **23. Frank Szoka**, University of California, San Francisco
Sequential assembly of targeted bioresponsive nanoparticles: Component design, synthesis and applications in drug and gene delivery
- 1100 **24. Alexey Kabalnov**, Hewlett Packard Company
Surface modification of liquid drops and microbubbles

Technical Program (continued)

- 1130 **25. Elodie Bourgeat-Lami**, CNRS, Villeurbanne
Surface-assisted nucleation and growth of polymers from organically-modified
inorganic particles
- 1200 End of Session

Tuesday, August 16, 2003, Afternoon

General Session 6 (Empire Room)

Session Chair: **John Texter**, Eastern Michigan University

- 1400 **26. Paul Alivisatos**, University of California, Berkeley
Stabilization of quantum dots for diagnostics
- 1445 **27. Christian Simon**, SINTEF
Nanocapsules for targeted controlled delivery of chemicals: Characterization and
applications
- 1515 Break
- 1545 **28. Markus Niederberger**, Max Planck Institute for Colloids, Golm
Surface-functionalized metal oxide nanoparticles as building blocks for the
fabrication of nanostructures
- 1630 **29. V. Prasad Shastri**, Vanderbilt University
Nanoengineering of drug delivery systems
- 1700 **Panel Discussion**, Helmut Schmidt, David Thompson, Paul Alivisatos
Topical issues from the conference discussions
- 1730 End of Session

End of Conference

Abstracts of Oral Program

Invited Oral Papers

General Session 1

- 1** CHEMISTRY BASED NANOMATERIALS AND THEIR INTERDISCIPLINARY PROCESSING TO MARKETED PRODUCTS
Helmut Schmidt, Leibniz-Institut für Neue Materialien, Im Stadtwald, Geb. 43 A, 66123 Saarbrücken, Germany; schmidt@inm-gmbh.de

Chemistry is a powerful tool for the synthesis of materials. However, in order to process the materials to industrial applications like components, coatings or parts, highly interdisciplinary collaboration is required in a so-called vertical approach, meaning from basics down to the market. These disciplines have to collaborate in a well-managed way and even by doing so, the added value of material sales is on the lowest level with respect to systems or devices. This leads to the fact that chemical industry with a few exceptions is strongly focused on high market volume materials, since only those are able to provide sufficient return of invest. On the other hand, nanoparticle based materials exhibit an extremely high degree of innovation, and chemical industry around the world is looking for new applications. With respect to the direct utilisation of new materials by end users, a gap between the laboratory material synthesis and the product on the market has opened, named as “death valley” recently in Japan and this death valley meanwhile has gained a lot of attention.

One of the key issues for handling chemically synthesized nanoparticles is the surface modification, which meanwhile, after a long period of neglectance seems to gain strong importance. Surface modification can control the particle growth, the particle surface chemistry and the aggregation. The appropriate surface chemistry leads to interesting new properties, such as cross-linking of the particles with each other (percolation) or with a matrix, thus influencing the matrix as well as the composite. Homogeneous distribution in transparent matrices leads to a new generation of optical materials because the composites show properties based on the physics of the nanoparticle as well as on the physics of the matrix. The increase of the free energy of the system due to the high surface fraction leads to an enhanced solubility in reactivity which was utilized in order to develop controlled release systems like antimicrobial surfaces or other properties, e. g. self-passivating corrosion protective coatings. Surface modification also can lead to special properties with respect to human cells: It was possible to coat superparamagnetic iron-oxide nanoparticles in a way that they can discriminate between healthy and tumor cells. A successful tumor therapy has been developed based on these findings. Moving nanoparticles along thermodynamic “slopes” were used for micropattern techniques like volume phase holography with high-refractive index nanoparticles. This was used to develop a new generation of diffuser foils for display techniques or screens for daylight projection TV. Silver nanoparticles produced in coatings during the coating fabrication in a controlled manner have been used for the fabrication of self-sterilizing pharmaceutical containers. These few examples show if basic science, surface modification, colloidal chemistry and engineering is combined in a vertical manner, many developments ready to be marketed can be developed. In the presentation, basic processing techniques and new applications which are successfully on the market will be described.

Abstracts of Oral Program

General Session 1

2 SURFACE INITIATED POLYMERIZATION FOR NANOCOMPOSITE FORMATION

Rigoberto C. Advincula, Department of Chemistry and Department of Chemical Engineering, University of Houston, Houston, TX 77204-5003

There has been wide interest in the investigation of tethered “polymer brushes” on surfaces primarily for their interesting properties as surface modifiers. While previous work has relied on physical or chemical adsorption of polymers, there has been much interest on polymers grafted by surface initiated polymerization (SIP). Recently, both scientific and technological advances have allowed a greater understanding of these polymer phenomena on a variety of surfaces where the chain density or grafting point distances can be smaller than the radius of gyration (R_g). While these systems have been widely investigated on flat surfaces, their application in particle modification is equally important and interesting. In this talk, we will review the basics and recent advances on the preparation of particles modified by SIP and their nanocomposite preparation. A number of addition polymerization mechanisms have been applied to nanoparticle modification including, free-radical, living free-radical, anionic, and cationic polymerizations. Other attempts involving the formation of hyperbranched step polymerization mechanisms have also been employed, e.g. amide type. Modification of particle surfaces including clays, silica, carbon, metal, semi-conductors, etc. have been investigated. The challenge is to understand the mechanism, provide homogeneity and efficiency, and seek technological applications for these type of materials. Our recent work includes SIP by anionic and free-radical “grafting from” the surface of the particles. We have also recently reported the attachment of conjugated polymer dendrons to metal and semiconductor nanoparticles for electro-optical applications.

General Session 1

3 THIN METALLIC COATINGS OF PARTICLES FOR ADVANCED HEAT TRANSFER APPLICATIONS

T.S. Sudarshan, Materials Modification, Inc., 2721-D Merrilee Drive, Fairfax, VA 22031

Thin coatings can provide an enhancement in physical, mechanical, thermal and optical properties on particulate materials. The coating selection is based on the desired properties. Both thick film and thin film coatings can be applied. This presentation will present an overview of the various techniques available for coating particulate materials with particular emphasis on heat transfer applications. The effect of coating parameters on the coating thickness and the resulting thermal properties will be presented.

Abstracts of Oral Program

General Session 1

4 PRACTICAL CHALLENGES IN MERGING MATERIALS SCIENCE AND THERAPEUTICS IN APPROACHING CLINICAL TRIALS FOR NANOPARTICLE DELIVERY SYSTEMS

Giulio F. Paciotti, Lonnie Myer, and Lawrence Tamarkin. CytImmune Sciences, Inc., 9640 Medical Center Drive; Rockville, MD 20850

A major thrust in cancer treatment is the development multifunctional nanotherapeutics that target cancer therapies to solid tumors. Typically these multivalent nanotherapies consist of a core particle that is bound with a tumor targeting moiety and a therapeutic payload and require agents that prevent their clearance by the reticuloendothelial systems (RES). Over the past five years CytImmune has developed a tumor-targeted drug delivery system on a pegylated colloidal gold (cAu) nanoparticle platform. The first drug developed (designated CYT-0691) actively targets and sequesters human tumor necrosis factor alpha (TNF) in solid tumors while avoiding uptake and clearance by the reticuloendothelial system (RES). The drug is comprised of TNF and thiolated polyethylene glycol (an RES avoidance molecule) that are individually, covalently bound to the surface of 26 nm cAu nanoparticles. The second generation drug currently under development is CYT-2100 a TNF targeted paclitaxel drug assembled on pegylated colloidal gold nanoparticles. To bring such technology forward for clinical evaluation requires manufacturing strategies for cost-effective production of the nanodrugs. Our discussion will highlight the technical and practical issues of bringing a metallic nanoparticle drug forward for clinical testing.

General Session 1

5 RAFT POLYMERIZATION FROM NANOPARTICLE SURFACES FOR NANOCOMPOSITES

Chunzhao Li, Sarah Lewis, Linda S. Schadler, and **Brian C. Benicewicz**, Departments of Chemistry and Chemical Biology, and Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180

Reversible Addition-Fragmentation chain Transfer polymerization (RAFT) was used to prepare silica nanoparticles coated with polymer brushes. RAFT agents anchored onto silica nanoparticles were synthesized by the functionalization of silica nanoparticles with a RAFT-silane agent. RAFT polymerization was then conducted from the nanoparticle surfaces to attach polymer brushes to the particles. Kinetics of St, n-BuA and MMA surface RAFT polymerizations were investigated. It was found that the surface graft polymerization proceeded in a controlled way, the first-order kinetics evolved in a linear fashion with time and molecular weights increased linearly with conversions. HPLC techniques were used to separate polymer grafted nanoparticles and free polymers. The amount of ungrafted polymer was proven to be minimal (< 14%). The well-defined polymer brushes attached to the silica nanoparticles were then characterized by AFM, TEM and DLS. Preliminary results of the polymer nanocomposites indicated that the molecular weight of the grafted chains should be higher than the matrix polymer chains to achieve a strong interface.

Abstracts of Oral Program

General Session 2

6 MODIFIED CARBON BLACK FOR BLACK MATRIX APPLICATIONS IN FLAT PANEL LCDS

Angelos Kyrlidis, Kao Xiong, Sean Sullivan, and Eugene Step, Cabot Corporation, 157 Concord Road, Billerica, MA 01821, angelos_kyrlidis@cabot-corp.com

Organic resin black matrices filled with carbon black are being widely adopted in the color filters of LCD flat panel displays to replace conventional chromium-based films. These thin films have unique optical and electrical performance requirements. The carbon black particles need to have high light absorption, but must not interfere with UV patterning, because modern manufacturing methods rely on patterning using photolithographic techniques. In addition, the filled system must have electrical properties (e.g. volume and surface resistivity) beyond what is possible with conventional carbon black particles.

Cabot has developed a proprietary technology, whereby organic groups can be covalently attached to the surface of carbon black particles. The process allows for the attachment of a wide range of surface functional groups including ionic, polar, hydrophobic, and polymeric groups. In this way we have produced materials for black matrix films that provide enhanced dispersion characteristics and unique electrical and optical properties. Our carbon blacks require less surfactant to disperse in photoresist formulations and provide more stable dispersions. These carbon blacks demonstrate excellent optical density characteristics and their electrical resistivity can be tuned over more than six orders of magnitude.

General Session 2

7 CRYSTALLIZATION OF PEG-MODIFIED COLLOIDS USING DNA-HYBRIDIZATION: REDUCING THE EFFECTS OF NON-SPECIFIC BINDING

John C. Crocker, Anthony J. Kim and Paul L. Biancaniello, Department of Chemical and Biomolecular Engineering, University of Pennsylvania, 220 S. 33rd St. Philadelphia, PA 19104;

The specific binding of complementary DNA strands has been suggested as an ideal method for directing the controlled self-assembly of microscopic objects. We report the first colloidal crystals formed using such DNA-induced interactions as well as the first direct measurements of such interactions between colloidal microspheres. The interactions measured with our optical tweezer method can be modeled in detail by well-known statistical physics and chemistry, boding well for their application to directed self-assembly. The polystyrene particles used in this study were densely PEG-ylated using a newly developed protocol based on reversibly swelling the particles with an organic solvent, and the DNA grafted to the terminal ends of the PEG chains. Earlier attempts at crystallization of particles formed by more conventional chemical grafting techniques were all unsuccessful. The most likely explanation is a small background of non-specific binding (NSB) that frustrates particle rolling and annealing in steric contact. Control experiments that support this hypothesis and its consequences for the use of covalently

Abstracts of Oral Program

grafted colloids for biochemical assays will be discussed.

General Session 2

- 8** LAYER-BY-LAYER ASSEMBLY FOR THE STABILIZATION AND FUNCTIONALIZATION OF NANOPARTICLES
Grégory Schneider and **Gero Decher**, Université Louis Pasteur (ULP) and C.N.R.S. Institut Charles Sadron, 6 rue Boussingault; F-67083 Strasbourg Cedex; France; decher@ics.u-strasbg.fr

The so-called Layer-by-Layer (LBL) assembly is a true bottom-up approach that leads to nano-organized multimaterial films. It is based on a straightforward concept (attractive intermolecular interaction, mostly electrostatic in nature, and combines experimental ease with low cost fabrication and environmental friendliness. Due to the broad range of materials that can be put into multilayers on substrates of almost every shape and every size, it has developed during the last 15 years from a scientific curiosity in fundamental research to an enabling technology which is in the process of being transformed into a tool for commercial mass production. Here we describe the guiding principles of LBL-assembly and how it can be used to prepare functionalized and well dispersed nanoparticles. We show that, in contrast to the existing literature, it is possible to deposit at least 20 layers of oppositely charged polyelectrolytes on native gold nanoparticles with a diameter of 13.5 nm that were prepared by classic citrate reduction. Gold nanoparticles are just used as model colloids, they present very convenient colloidal cores due to their easy synthesis, their strong plasmon absorption band and their high density. These properties facilitate enormously the preparation of coated particles, the monitoring of the adsorption process and the characterization of the resulting species. At present, we have the first indications, that the process of LBL-assembly on nanoparticle surfaces is easily extended to different functional polymers and also to different particle suspensions.

General Session 2

- 9** DESIGNING NANOPARTICLES FOR FUNCTIONAL MATERIALS AND COATINGS
Ronald Cook, Andrew Myers and Jeannine Elliott, TDA Research, Inc., 12345 West 52nd Avenue, Wheat Ridge, CO 80033

In the last decade, surface engineering of inorganic nanoparticles has advanced to the point that that it has become possible to fabricate and tune the functional properties of the nanoparticles. Intermediate in size between molecules and bulk solids, inorganic nanoparticles combine the convenience of carrying out chemical modifications in solution while retaining the properties of the inorganic core. As such, surface modified inorganic nanoparticles are ideal materials for the production of nanostructured materials and nanocomposites. When the surfaces of the inorganic nanoparticles are correctly designed, they can be fully dispersed into polymers resulting in the formation of polymer nanocomposites having exceptional thermal, mechanical and barrier properties. Finite Element Analysis shows that these properties are intimately tied to the degree of dispersion and the aspect ratios of the inorganic nanoparticles. Self-Consistent Field Theory modeling of surface engineered inorganic nanoparticles suggests that the surface density, oligomer length and the cohesive energy density of the organics attached to the

Abstracts of Oral Program

surface of the inorganic nanoparticle are important factors to consider in nanoparticle design.

In addition to using surface engineering to produce nanoparticles that can improve the mechanical/barrier properties of polymers, surface engineering can also be used to produce functional nanoparticles and by extension functional nanocomposites. At the most basic level, functional nanoparticles are surface engineered to both disperse into a host phase (e.g. a polymer) and to carry the functional agent. Additional functions such as controlled or triggered release can also be designed into the surface modification. We will present our work on developing tools to design surface engineered inorganic nanoparticles and the development of several types of functional nanoparticles. A specific example to be covered in detail is the use of surface engineered inorganic nanoparticles for triggered release of corrosion inhibitors for aluminum alloys.

General Session 2

10

MODIFICATION OF POLYMER PARTICLE SURFACES – FROM CONCEPT TO MULTINATIONAL BUSINESS

Bernard D. Bauman, INHANCE Fluoro-Seal Ltd., 16360 Park Ten Place, Suite 325; Houston TX 77084

Surface modified polymer particles represent a new dimension in material engineering, enabling dissimilar types of polymers to be combined to form novel composite materials with desirable properties. Interestingly, reactive gas surface modification was conceived 25 years ago as a way to sell more specialty gases. Instead, the development, evolution, and commercialization of this technology has led to new high performance materials, and gas usage is inconsequential. This presentation reviews the exciting journey from concept to global business. It describes some of the chemistry and other technology aspects, and suggests how this may be important in nanotechnology. The presentation also describes the author's experience in creating a start-up company and growing commercial adoption of these new materials.

General Session 3

11

SURFACE MODIFICATION OF Fe/Au AND NON-COVALENT NANOPARTICLE ASSEMBLIES FOR THERAPEUTIC APPLICATIONS

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Our lab is focused on the design, synthesis and performance of responsive nanoparticle systems that can be activated by low pH, microwave, near infrared, and UV excitation. The systems under development include Fe/Au nanoparticles, polymer micelles, and liposomes for therapeutic applications. They have been designed to exploit the enhanced permeation and retention effect as a passive targeting mechanism and to utilize folic acid surface modification for selective uptake by folate receptor positive tumor cells. Once the nanoparticles have been localized and internalized by these processes in the tumor site, activation of the nanoparticles can lead to localized cell killing via drug release, oxidative stress, thermal ablation, or a combination of these mechanisms. The performance of photo-triggered folate-PEG liposomes, 10 nm Fe/Au nanoparticles modified with folate-PEG-thioctic acid and mPEG-thioctic acid conjugates, and PEG-

Abstracts of Oral Program

vinyl ether-PLA micelles in cell culture will be described. The advantages and disadvantages of these nanoparticle methods for targeted drug delivery in cancer treatment will be described.

General Session 3

- 12** LAYER BY LAYER MICROCAPSULES: TOWARDS CONTROLLING CAPSULE MECHANICS AND ADHESION ON THE NANOSCALE
Andreas Fery, Nils Elsner, F. Dubreuil, J. Heuvingsh, Renate Mueller, Max Planck Institut f. Colloid and Interface research; Am Muehlenberg, D14424 Golm, Germany, email: andreas.fery@mpikg.mpg.de

Microcapsules made from polyelectrolyte multilayers have caught increasing attention during the last decade, since they can be formed from a variety of polyelectrolytes including biocompatible species with well defined diameter and control of the wall thickness on the nanometer level. Mechanical properties of the capsule membrane material are in the context of applications like drug delivery important for both stability and for adhesion, since capsule compliance determines the degree of deformation due to adhesive interactions.

We show that AFM force spectroscopy combined with optical techniques allows probing the mechanical properties on the single capsule level and deriving elastic constants of the wall material. Our results show that the membrane of classical lbl microcapsules is in a glassy state at room-temperature and that for some systems, the glass transition temperature T_g can be reached in water. This results in a drastic softening of the wall material. T_g can be influenced by external parameters like salt concentration. This result explains the remarkable stability of these systems and opens a way to (drastically) change their mechanical properties in a reversible fashion.

The control over mechanical properties that is possible in this system also allows to tune the adhesion behaviour. We find for capsules of identical interactions, that the capsule wall thickness has a pronounced impact on adhesion: The adhesion areas can be varied from pointlike up to large circular contact discs of diameters on the order of half the capsule diameter, which can be explained by the observed dependency of compliance on wall thickness.

General Session 3

- 13** LEVERAGING SBIRS AND CONTRACTS FOR TECHNOLOGY DEVELOPMENT
Ganesh Skandan, NEI Corporation, 201 Circle Drive, Suite 102/103, Piscataway, NJ 08854; gskandan@neicorporation.com

The road to a sustainable and profitable entity is replete with challenges, some of which include cost competitiveness, product consistency, unique product identity, and effective management. Creating a successful enterprise in the field of 'Materials' is particularly difficult because of the need for infrastructure. Small start-up companies are often torn between the disparate needs of developing a widely accepted product with a significant profit margin in a relatively short period of time, and establishing a company built on solid technological foundation with the ability to maintain sustainable competitive

Abstracts of Oral Program

advantage in the long run.

Founded in 1997 as a nanopowder producer, NEI Corporation has gone through a transformation to a developer and manufacturer of value-added nanomaterials and nanoadditives. Small Business Innovative Research (SBIR) and Small Business Technology Transfer (STTR) programs from the various federal agencies have been pivotal to NEI's evolution as a nanomaterials company. The presentation will discuss NEI's evolution as a customer and market driven company in this highly competitive field of Nanotechnology. In addition, examples of nanomaterials development at NEI will be presented.

General Session 3

14 VITAMIN E TPGS COATED NANOPARTICLES OF BIODEGRADABLE POLYMERS FOR CANCER CHEMOTHERAPY AND MEDICAL IMAGING

Si-Shen Feng, Department of Chemical & Biomolecular Engineering and Department of Bioengineering, National University of Singapore

Vitamin E TPGS could be a biocompatible as well as efficient emulsifier to prepare nanoparticles of biodegradable polymers for cancer chemotherapy. This presentation will show proof-of-concept experimental results with paclitaxel used as a prototype drug due to its excellent effects against a wide spectrum of cancers and its great commercial success. Paclitaxel-loaded poly(lactic-co-glycolic acid) (PLGA) nanoparticles were prepared by a modified solvent extraction/evaporation technique with vitamin E d- α -tocopheryl polyethylene glycol 1000 succinate (TPGS) as emulsifier. The nanoparticles were characterized by various state-of-the-art techniques, including laser light scattering for size and size distribution, scanning electron spectroscopy (SEM) and atomic force microscopy (AFM) for surface morphology, X-ray photoelectron spectroscopy (XPS) for surface chemistry, zeta-potential for surface charge, differential scanning calorimetry (DSC) for phase behavior of the drug entrapped in the nanoparticles, drug encapsulation efficiency (EE) and *in vitro* drug release from the nanoparticles by high performance liquid chromatography (HPLC). The cellular uptake of fluorescent nanoparticles was measured by employing Caco-2 cells as an *in vitro* model of the gastrointestinal (GI) barrier for oral chemotherapy. The results were found strongly depends on the particle size and particle surface coating. *In vitro* cytotoxicity of the paclitaxel-loaded, TPGS-emulsified PLGA nanoparticles was assessed by MTT and compared to that of Taxol[®]. It was found that the nanoparticle formulation could be 46, 42, 20, 10 times more effective than Taxol[®] after 24, 48, 72, 96 hour culture with HT-29 cells at 0.25 $\mu\text{g/ml}$ of paclitaxel, respectively. *In vivo* pharmacokinetics showed that the area-under-the-curve (AUC) and the characteristic time at which drug concentration dropped below the therapeutic level could be 6.21 and 2.48 times higher than those of Taxol[®] after injection of the same dose of 10 mg/kg to SD rats, and that 8.2 times higher dose tolerance could be achieved by the nanoparticle formulation.

Abstracts of Oral Program

General Session 3

15 AN INTEGRATED PLATFORM OF SCALED NANOTECHNOLOGIES - VAPOR PHASE SYNTHESIS, NANOPARTICLE SURFACE TREATMENT, AND DISPERSION - TO ENABLE COMMERCIAL APPLICATIONS

Harry Sarkas, J. Piepenbrink, Glenn Judd, Daniel C. Coy, Patrick G. Murray, Roger Cayton, **R. W. Brotzman, Jr.**; Nanophase Technologies Corporation, Romeoville, IL, USA

Nanophase Technologies Corporation (NTC) has developed and integrated platform of commercially scaled technologies to deliver tailored nanoparticle products to target market applications. Nanocrystalline metal and metal oxide particles are produced by two, patented physical vapor synthesis technique – PVS and NanoArc™. These processes vaporize precursors to form plasmas, followed by rapid quenching to induce condensation and formation of metal oxide nano-crystallites. To enable most market applications, nanoparticles require surface treatment and dispersion. NTC developed proprietary and patent pending surface treatment and dispersion processes designed to provide one or more of the following properties: dispersion into fluids (aqueous, alcohol, and hydrocarbons), prevention of particle agglomeration, compatibility with formulation ingredients, chemical functionalization of the nanoparticle surface with reactive groups, and passivation or enhancement of the nanoparticle surface chemistry.

This approach to the market increases the value of products and decreases the time to market for new product introductions. Specific examples of personal care, polishing, and polymer additive products will be given.

General Session 4

16 MORPHOLOGY OF CRYSTALS GROWN FROM SOLUTION: EXPERIMENTAL & THEORETICAL CONSIDERATIONS

James H. Adair,* Nelson S. Bell,[†] Robert T. DeHoff,[#] and Rajneesh Kumar,*
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Alpha-Alumina (α -Al₂O₃, corundum) particles were synthesized under glycothermal conditions by conversion of gibbsite in 1,4-butanediol at various temperatures, stirring rates, solids loading and hold time at intermediate temperatures. The extent of degradation of 1, 4-butanediol controlled the amount of water and thus the solvent composition in equilibrium with the reactants. Development of platelet like morphology was promoted by low water concentrations in the solvent, and development of polyhedron morphology was promoted by high water concentrations in the solvent. It is proposed that platelet morphology is obtained by stabilization of the {0001} basal plane by 1,4-butanediol, and polyhedron morphology is obtained by stabilization of the $\{\bar{1}012\}$ rhombohedral plane by water.

Abstracts of Oral Program

A thermodynamic derivation has been developed for the equilibrium conditions for a particle precipitated from a liquid solvent. The expression for mechanical equilibrium agrees with the previous derivations permit the Gibbs-Wulff construction to predict the equilibrium morphology of the crystal. A theoretical model predicting morphology for particles produced under solution conditions is proposed, with the possibility of extension of the model to any particle produced under solution conditions. The extent of reaction at the solid/solution interface accounted for the generation of surface charge, and electroneutrality was maintained by the exchange of reactive species across the solid/solution interface. Generation of surface charge and predictions of isoelectric point (IEP) were made using the multisite complexation (MUSIC) model. The Gouy-Chapman (GC) model was used to describe potential decay through the diffused layer above the oxide interface. Changes in surface energies for crystal facets are related to charging at the solid/solution interface as a function of crystallographic habit by using modified Lippmann equation. A comparison is made between theoretically predicted and experimentally observed morphologies of α -Al₂O₃.

General Session 4

17

PARTICLE ALD FOR DEVELOPING NOVEL MATERIALS THROUGH NANOCOATING

Karen Buechler and John Ferguson, ALD NanoSolutions, Inc., 580 Burbank St, Unit 100 Broomfield CO 80020; Alan Weimer, Steven George and Luis Hakim, Departments of Chemical and Biological Engineering and Chemistry and Biochemistry, University of Colorado at Boulder, Boulder CO 80309; kbuechler@aldnanosolutions.com

There exist critical materials problems in almost every established industry. These problems are related to thermal management, diffusional resistance characteristics, chemical reaction characteristics, surface property mismatches, structural integrity, and electrical / magnetic / optical properties, among others. As materials are being discovered and functionalized to try to solve some of these problems, it turns out that a combination of existing materials is often the ideal solution. In many cases, a fine particle that is selectively or conformally encapsulated in an ultra- thin layer of a second material is exactly what is needed to solve the problem. The particle maintains its bulk properties while its surface properties have been modified to desirably interact with the surrounding environment. This new class of hybrid materials and the associated functionality can be economically manufactured on a nano-scale using atomic layer deposition (ALD). ALD NanoSolutions, Inc. has developed and validated proprietary technology Particle ALD™ to develop these composite materials. This technology will be described through the use of several specific material examples.

General Session 4

18

SURFACE MODIFICATION OF PARTICLES AND EMULSION DROPLETS WITH FUNCTIONAL POLYMERS

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Abstracts of Oral Program

Surface modification by functional polymers has long been recognized as a powerful tool for controlling material properties. The large inner surface of emulsions or colloidal dispersions is a prime target: here, modifications provide a handle on the particle-particle interaction, which in turn is responsible for vital application and processing properties, such as stability, flow and optical properties, to name but a few. Our understanding of particle interactions has been promoted greatly by direct interaction measurements with colloidal probe techniques. We will argue that total internal reflection microscopy (TIRM) presents a particularly sensitive way of studying colloidal forces caused by adsorbed polymers. TIRM studies reported here address the effect of charged and neutral adsorbate layers as well as their response to changes in the surrounding solution. For the case of emulsion droplets, we show that stability against coalescence and ripening can also be achieved and even fine-tuned through adsorbed polymeric species. In particular we discuss surfactant-free emulsions stabilized by pH and temperature switchable microgels, which offer unprecedented stability control.

General Session 4

19 SURFACE MODIFIED PIGMENTS IN INKJET APPLICATIONS
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Pigment use in inkjet inks is proliferating due to a market need for better fade resistance and durability of inkjet images. However, ink formulations have tenaciously held on to the dye legacy due to the lower jetting reliability, gloss, and color gamut often exhibited by pigmented inks, in spite of their advantages in UV/ozone resistance and waterfastness. In addition, continuing trends to increase inkjet printing speed have translated into lower drop weights, higher firing frequencies and the need for pigments that maintain higher levels of colloidal stability over time.

Through proprietary surface attachment technology, Cabot has expanded both the types of surface groups that can be attached to fine particles as well as the pigments that can be surface modified. Small molecule attachments have led to dispersions having superior colloidal stability in inkjet formulations with a minimal need for added dispersants. By changing the charged groups on the surface attachments, flocculation mechanisms can be controlled to boost and modulate optical density and of the resulting inkjet inks. This performance enhancement can be observed in both high and low pH systems. In addition, polymer modified pigments have improved gloss, waterfastness and highlighter resistance properties over conventionally dispersed pigments, not only for carbon black, but for an ever increasing portfolio of colored pigments. The structure vs. function of Cabot inkjet dispersions will be discussed.

General Session 4

20 MODIFYING SURFACES TO DIRECT NANOCOMPOSITE FORMATION
Emmanuel P. Giannelis, Department of Materials Science and Engineering,
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Abstracts of Oral Program

Work in polymer nanocomposites has exploded over the last few years. The prospect of a new materials technology, which can function as low-cost alternative to high-performance composites for applications ranging from automotive to food packaging to microelectronics has become irresistible to researchers around the world. In the first part of this talk I will review our research effort in nanocomposites and emphasize work on surface modification of nanoparticles to direct nanocomposite formation. In the second part I will present our recent work to synthesize and characterize “liquid” nanoparticles or *solvent-free* nanofluids. These new hybrid materials involve inorganic nanoparticles, which can flow and behave like molecular liquids in the absence of any solvent.

General Session 5

21

NANOPARTICLE SURFACE EFFECTS ON CHEMICAL DETOXIFICATION

Richard Partch^{1,2}, Evon Powell^{1,2}, Adrienne Benson¹, Manoj Varshney², Donn Dennis³, Tim Morey³, Y-H. Lee⁴, D-W. Lee², and Ron Baney². ¹Center for Advanced Materials Processing, Clarkson University, Potsdam, NY 13699-5814 USA; ²Material Science and Engineering, University of Florida, Gainesville, FL, 32611-6135 USA; ³Department of Anesthesiology, University of Florida, Gainesville, FL, 32611-6135; ⁴Department of Chemistry, Kyungwon University, Sunghnam City, Korea

The ability to reverse the effect of overdoses of chemicals once in the human blood stream is of key importance as more and more therapeutics and street drugs are being abused. An approach to create an effective and selective antidote is currently under investigation by the interdisciplinary co-authors. The focus so-far has been to determine what chemical systems and principles might apply for removal of amitriptyline and bupivacaine pharmaceuticals, and of cocaine. One system that employs principles of absorption is an oil-in-water microemulsion composed of ethyl butyrate droplets stabilized by fatty acid and pluronic co-surfactants. The results of studies on optimizing the component ratios, NMR data on how the drugs approach and become captured by the microemulsion, and in vivo removal efficiency will be presented. A second system takes advantage of covalent attachment of pi-aromatic acceptors to silica and oligochitosan carrier particles. Such moieties are shown by NMR and HPLC to also remove the three example toxins because the latter molecules have pi-aromatic donor properties. AFM data is being collected in support of the pi-pi theory.

General Session 5

22

SURFACE INITIATED NITROXIDE-MEDIATED RADICAL POLYMERIZATION

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Novel (metal oxide nanofiller/polymer) nanohybrids were prepared through direct polymer grafting reaction from the surfaces of magnetite (Fe₃O₄) (d=10 nm and d=25 nm) and titanium oxide (TiO₂) (d=15 nm) nanoparticles. The nitroxide-mediated radical

Abstracts of Oral Program

polymerization initiator (TEMPO-based alkoxyamine derivative) with the phosphoric acid group was designed and chemisorbed onto the surfaces of the metal oxide nanoparticles. This initiator system gave high-density controlled polystyrene (PS) and poly(3-vinylpyridine) (P3VP) graft layers on the metal oxide surfaces. The graft densities of polymers on the nanoparticle surfaces were estimated at 0.06-0.28 chains/nm² by thermogravimetric analysis. PS-modified and P3VP-modified nanoparticles were finely dispersed in good solvents for PS and P3VP, respectively. For instance, these modified nanoparticles were dispersed in chloroform and toluene, and P3VP-modified nanoparticles were dispersed even in acidic aqueous phase. Furthermore, by quarternization, the P3VP-modified nanoparticles were dispersed in neutral water. Fine dispersion of nanoparticles in various solvents was confirmed by dynamic light scattering and transmission electron microscopy (TEM). In the case of modified magnetite nanoparticles, the magnetic response of the particles against an external magnetic field was confirmed in the stable dispersion. The (metal oxide nanofiller/polymer) thin film was prepared from the mixture of polymer-grafted nanoparticles and matrix polymer solution. The fine dispersion of the nanoparticles into polymer matrix was confirmed by TEM and light transmittance characteristics. The fine dispersion of nanoparticles greatly enhanced the wear resistance of the hybrid polymer thin films even though the weight fraction of nanoparticles less than 0.5wt%.

General Session 5

23

SEQUENTIAL ASSEMBLY OF TARGETED BIORESPONSIVE NANOPARTICLES: COMPONENT DESIGN, SYNTHESIS AND APPLICATIONS IN DRUG AND GENE DELIVERY

Francis Szoka, Weijun Li, Zhaohua Huang & Andrew MacKay, University of California, San Francisco, CA 94143

The creation of synthetic drug and gene carriers that can efficiently access, bind to and enter a target cell requires the identification of components that can enable the various steps in the process. It also requires a method to assemble the compounds in a robust manner and in a way that permits high encapsulation of the drug or gene. Our group has investigated a variety of lipid, peptide and polymeric materials that provide the necessary functions and is working on methodologies to assemble them into a drug carrier with nanoscale dimensions. A series of thiocholesterol-based cationic lipids (TCL) have been designed and synthesized by the attachment of thiocholesterol to the cationic moiety via a disulfide bond. TCL can be incorporated into liposomes and used to package DNA into a lipoplex, thereby protecting it from DNase digestion. DNA is rapidly released from the complex in the presence of low concentrations of reducing agents. The lipoplex mediated efficient transfection activity, could deliver siRNA and had low cytotoxicity. To improve the biocompatibility of the cationic lipoplex, TCL were used as a component in the assembly of a PEG masked nanolipoparticle (NLP). The particle surface was subsequently modified by disulfide exchange to replace the cationic group with a negatively charged (glutathione, GSH) or zwitterionic (cysteine, Cys) reducing agent. A targeting ligand (TAT peptide, sequence: GRKKRRQRRRGYG) was then incorporated onto the particle surface to enhance the particle-cell recognition. The sequentially-assembled targeted NLP with a zwitterionic surface gave a larger transfection yield than the targeted

Abstracts of Oral Program

cationic NLP at all concentrations tested. At low DNA concentrations, the enhancement was eighty-fold. The disulfide cationic lipids and the sequential assembly strategy enable one to tailor the surface charge, hydrophilicity, and recognition elements of a nanosized gene carrier. This results in increased gene transfer activity in a biocompatible particle. Supported by NIH.

General Session 5

24 SURFACE MODIFICATION OF EMULSION DROPS AND MICROBUBBLES

Alexey Kabalnov* and Ernie Schutt**, *Hewlett Packard, 16399 W. Bernardo Dr., San Diego CA 92127, **12139 Ragweed Street, San Diego, CA 92129

Surface modification of emulsion drops and microbubbles, as opposed to conventional routes of stabilization, implies the irreversibility of adsorption. The approaches to attain the irreversibility are reviewed, such as the usage of high molecular weight polymers, hydrophobic surfactants, solid particles, and chemical cross-linking at the surface. Surface modification can serve various purposes, e.g., adding specific functionality to the particles (targeting), improvement in stability/lifetime in vivo (stealth), and improvement of the stability in vitro, i.e., stabilization against coalescence, ripening and dissolution. This talk will be centered around the in vitro stability aspect of the surface modification. Because the surface modification is a non-equilibrium process, the methods of microencapsulation are often non-trivial. On the working examples of emulsions for intravenous injection (fat and fluorocarbon emulsions) and microbubble dispersions for ultrasound contrast, the approaches to prepare the encapsulated systems are reviewed and the in-vitro stabilization mechanisms discussed.

General Session 5

25 SURFACE-ASSISTED NUCLEATION AND GROWTH OF POLYMERS FROM ORGANICALLY-MODIFIED INORGANIC PARTICLES

Elodie Bourgeat-Lami, Laboratoire de Chimie et Procédés de Polymérisation, UMR 140 CNRS-CPE, Bât. 308F, BP 2077, 43, Bd. Du 11 Nov. 1918, 69616 Villeurbanne.

The growth of polymers at the surface of mineral particles is a topic of current interest. In order to create strong interactions at the interface of the inorganic particles with the organic polymer and promote polymer chains formation, chemical groups, reactive in the polymerization process, need to be introduced on the mineral surface. These groups can be either monomers, initiators or catalysts. They can be introduced in various way: 1) by grafting of organosilane molecules, 2) by adsorption of polymeric compounds carrying suitable end groups or 3) by ion exchange. We will report in this talk surface modification of silica particles and clay colloids by vinyl monomers, free radical initiators and alkoxyamine compounds. The emphasis will be put on quantitative aspects of the modification reaction and on the impact of functionalization on colloidal stability. Two kinds of polymerization processes will be then envisaged: 1) controlled radical polymerization (CRP) performed in organic solutions, and 2) emulsion polymerization.

Abstracts of Oral Program

Polymerizations performed in organic solvents allows the growth of a hairy polymer layer surrounding the inorganic particles and give rise to core/corona composite particles. Emulsion polymerization allows the formation of nanocomposite colloids of various morphologies. We will show that not only does the nature of the mineral particles play an important role in the control of particles morphology but the nature of the interface is also critical. Depending on the experimental conditions, core-shell, inverted core-shell (the mineral forming the shell), raspberry-like, daisy-shaped or dissymmetrical organic/inorganic particles can be produced by this method.

General Session 6

- 26** BIOLOGICAL TAGGING APPLICATIONS OF SEMICONDUCTOR NANOCRYSTALS
A. Paul Alivisatos, Materials Sciences Division, Lawrence Berkeley National Laboratory, One Cyclotron Road, Berkeley, CA 94720

Semiconductor nanocrystals exhibit strongly size dependent emission spectra due to the quantum size effect. Further, the nanocrystals have nearly continuous excitation spectra above the threshold for absorption. As a consequence, the nanocrystals can be used as luminescent probes in biological staining experiments. The nanocrystals are in many ways superior to existing organic chromophores. Relevant applications and surface chemistry will be described.

General Session 6

- 27** NANOCAPSULES FOR TARGETED CONTROLLED DELIVERY OF CHEMICALS: CHARACTERISATION AND APPLICATIONS
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The technique of microencapsulation has been widely used to enclose various types of chemicals in a particulate material. Typical applications may be found in life science, biotechnology and food industry, cosmetic, chemical and pharmaceutical industry. Microcapsules have commonly a size in the range of 1 μm to a few μm . Nanocapsules have recently been the subject of intense research and development. Due to their smaller size, these components offer additional advantages compared with microsized capsules, like improved efficiency and reduced toxicity in drug delivery, and more homogeneous surface coverage. Also due to a particle size below a critical size, new applications could be considered in thin coatings and drug delivery where penetration through smaller capillaries was possible. The European project NANOCAPS aims in developing new technologies based on micro-encapsulation to solve industrial problems related to controlled release of chemicals. The technical objectives have been to develop nanomaterials and nanocomposite coatings for cost-efficient production of nanocapsules and, and to validate the technical and economical feasibility of the knowledge acquired in the fields of biomedical (anti-proliferating and anti-allergic agents) and metal plating (self-repair) applications. This paper will present recent developments and give some illustrations on various types of nanocapsules.

***In the name of the Partners of NANOCAPS project:** C. Simon (coordinator), SINTEF, Norway; G. Sukhorukov, Max-Planck Institute of Colloids and Interfaces, Golm, Germany; A. Foissy, University of

Abstracts of Oral Program

Franche-Comté, France; Piotr Warszynski, ICSC, Krakov, Poland; C. Kiparissides, CERTH, Thessalonica, Greece; A. Antipov, PLASMACHEM, Germany; J.J. Duprat, COVENTYA, France; J.F. Argillier, IFP, France; B. Thorstensen, KERANOR, Norway; J.M. Suau, COATEX, France; P. Swietoslowski

General Session 6

28

SURFACE-FUNCTIONALIZED METAL OXIDE NANOPARTICLES AS BUILDING BLOCKS FOR THE FABRICATION OF NANOSTRUCTURES

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In addition to the synthesis of nanoparticles with control over particle size, shape and crystal structure, the main focus of nanochemistry more and more shifts towards the use of these nanoparticles as building blocks for the fabrication of 1-, 2- and 3-dimensional superstructures. The continuing trend towards miniaturization, combined with an increase in performance and multifunctionality, is a major driving force for the exploration of such novel “bottom-up” approaches. One of the most promising strategies for the fabrication of hierarchical structures is the use of self-assembly processes, where the organization is solely determined by the interactions between the primary building blocks. Accordingly, control over the surface properties is a key factor.

In this talk, we present a strategy to synthesize nanopowders, which have their assembly behavior encoded on the surface. Powders of nanoparticles composed of surface-functionalized anatase crystals with diameters of about 3 nm self-organize into different structures upon redispersion in water. In contrast to many other procedures, which rely on the use of surfactants or block copolymers as templates, the assembly is directed by a small amount of a low molecular weight functional ligand (the “assembler”) adsorbed on the surface of the nanoparticles. The ligand functionality determines the anisotropy of the resulting structures. Multidentate ligands like trizma $(\text{HOCH}_2)_3\text{CNH}_2$ and serinol $(\text{HOCH}_2)_2\text{CNH}_2$ with a chargeable terminal group preferentially induce the formation of anisotropic nanostructures of several hundreds of nanometers in total length, whereas all the other investigated ligands (ethanolamine $\text{H}_2\text{N}(\text{CH}_2)_2\text{OH}$, glycine hydroxamate $\text{H}_2\text{NCH}_2\text{CONHOH}$, dopamine $(\text{OH})_2\text{C}_6\text{H}_3(\text{CH}_2)\text{NH}_3\text{Cl}$, tris $(\text{HOCH}_2)_3\text{CCH}_3$) mainly lead to uncontrolled agglomeration. Experimental data suggest that the anisotropic assembly is a consequence of the water-promoted desorption of the organic ligands from the $\{001\}$ faces of the crystalline building blocks together with the dissociative adsorption of water on these crystal faces. Both processes induce the preferred attachment of the titania nanoparticles along the $[001]$ direction. The use of polydentate and charged ligands to functionalize the surface of nanoparticles provides thus a versatile tool to control their arrangement on the nanoscale.

Abstracts of Oral Program

General Session 6

29 NANOENGINEERING OF DRUG DELIVERY SYSTEMS
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Nanoscale design offers the possibility of engineering materials that possess properties or characteristics that may not be attainable by macroscopic processing techniques. Improved understanding of assembly processes at the molecular level and structure property relationships in self-assembled systems have led to the ground-up design of materials and systems for sustained delivery of molecules. Such a design strategy can provide enhanced control over key properties such as drug stability, diffusion and localization. Delivery of bioactive or imaging agents with a high degree of tissue and cellular specificity is increasingly sought after as our understanding of disease progression improves. Among the many challenges, transport across biological barriers and targeted localization of the biological agent remain paramount. Particularly, in the treatment and imaging of tumors, localization of the agents of interest is extremely desirable, to minimize systemic toxicity and improve efficacy. Among, various factors, the nature of the particulate delivery system plays a critical role in influencing these outcomes. Our laboratory has taken a ground-up approach to address these issues by developing strategies to influence the pharmaco-kinetics of drugs and influence and control cell-nanoparticle interactions, by nano-scale engineering of microenvironments and surface chemistry. This talk will cover some recent results from our ongoing efforts.

Abstracts of Poster Program

Invited and Contributed Poster Papers

Poster Session 1

30 SYNTHESIS OF NANOPARTICLES IN A CONTINUOUS-FLOW MICROWAVE REACTOR

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Over the past two decades, significant progress were made in the development of techniques for the synthesis of well-defined colloids consisting of particles ranging from nanometers to several micrometers, of simple or composite natures and of different shapes. The most commonly employed method was precipitation from homogeneous solutions in batch systems. The procedures involved small quantities of reactants in low concentrations. A batch process is often limited to small-scale synthesis due to the low production yield of nanoparticles and the time-consuming nature of the process. However for any practical application, it is essential to obtain such dispersions in large amounts, preferably by continuous processes. In particular a microwave flow reactor can generate products on a continuous basis with faster reaction kinetics.

This work relates to a continuous microwave synthesis process of inorganic materials and more particularly to the synthesis and crystallisation of silica and titania powders. The microwave synthesis was conducted using a commercial microwave-heated flow vertical tube reactor (MLS ETHOS CFR Continuous Flow Reactor) which consists of microwave-transparent, chemically inert (polytetrafluoroethylene, Teflon, internal volume 400 cm³) vessel. This system uses 2.45 GHz microwaves and is controlled by pressure. The flow rate of the solution, and thus the irradiation time of the solution in the microwave oven cavity, was varied from 43 to 101 ml/min. The flow rate, pressure and power were computer controlled. The obtained results showed the process has the following advantages: 1) the reaction time is further shortened by several to tens of minutes for crystallisation, compared to the conventional hydrothermal reaction requiring a prolonged time; 2) the continuous manufacturing and collection processes can give access to mass-scale production of inorganic materials with relatively small facility, compared to the conventional batch hydrothermal or microwave synthesis.

Poster Session 1

31 INFLUENCE OF PARTICLE STACKING STRUCTURE IN CATALYST LAYERS ON THE PERFORMANCE OF PROTON EXCHANGE FUEL CELLS

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Fuel cell as an emerging energy generation technology of the 21st century is currently in

Abstracts of Poster Program

very rapid development in technology-advanced countries worldwide. Among the various types of fuel cells, proton exchange membrane fuel cells (PEMFCs) are being heavily studied and developed in academic and industrial settings for their potential applications in transportation and portable electronics, etc. The key component in PEMFCs, known as membrane electrode assembly or the so-called MEA, is a sandwich structure of thin film-like electrodes, one anode and one cathode, closely embracing a solid proton exchange membrane from its two sides. The electrodes are composed of a catalyst layer, which is an organic/inorganic composite nanoparticle layer. It is in this catalyst particle layer where electrochemical reaction as well as complicated two-phase transport phenomena occurs. As different particle coating techniques could result in inherently different particle stacking structures and consequently, different transport properties for liquid water produced may result. Therefore, it is the objectives of this study to investigate half-cell and single cell performances resulted from different particle coating techniques applied in catalyst layer preparation, film transfer and spraying. Other electrochemical analysis techniques are also employed to obtain further insight into the root cause of the differences in cell performance observed among different structures.

Poster Session 1

32 CHARACTERISTICS OF SILICA NANOPARTICLES DERIVED UNDER DIFFERENT COMBUSTION CONDITIONS IN A LOW-PRESSURE FLAT FLAME

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Nanoparticles such as silica prepared from gas phase techniques generally exhibit inherent characteristics such as narrow size distribution and processing advantages such as extremely short processing time, both of which are desirable for practical applications. Nevertheless, besides cost involved in nanoparticle production, the capability of the applied technique in precise tuning of particle characteristics such as particle size and morphology is without doubt one of the key factors determining the applicability of the technique in practical uses. In this study, the synthesis and characterization of silica nanoparticles under different combustion conditions in a low-pressure flat flame are presented. Analytical techniques such as transmission electron microscopy (TEM), field emission scanning electron microscopy (FESEM), etc., were employed in characterizing the as-derived silica nanoparticles. Particle characteristics as a function of combustion conditions such as fuel/oxidant ratios, flame temperature, chamber pressure, etc., were investigated. It was shown that particle size and morphology of silica nanoparticles as well as the secondary particles formed are strongly associated with flame temperature employed, which can be adjusted by operational parameters such as chamber pressure and fuel/oxidant ratios, etc.

Abstracts of Poster Program

Poster Session 1

33

SURFACE-MODIFICATION OF SILICA-GEL FOR REMOVING TOXIC COMPONENT WHILE RETAINING ACTIVE COMPONENT IN THE PREPARATION OF CHINESE HERBAL MEDICINE

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Many Chinese herbs have to be detoxified by the experienced folk medicinal experts, since they know these herbs contains not only active component(s) but also toxic component(s). In our natural products' research, we are trying to replace the traditional long and un-sanitized detoxification process with a scientific, simple and quick method based on particles' affinity to take away the toxic component(s) in the herbal medicinal preparations. But for Chinese He-Shou-Wu medicinal preparation, the modification of silica-gel particles has to be designed not only for the purpose of removing the toxic component-Emodin but also retaining the active component-Rhein. This task becomes more challenging, because the structures of Emodin and Rhein are so similar and their polarity are very close that were confirmed with the HPLC analyses by normal phase as well as reverse phase separation techniques. Finally we find the p-xylene silanized silica-gel is able to remove Emodin in a quick way while retain Rhein in the medicinal preparation that will be prepared with another modified silica-gel as controlled-released natural medicine later. This application is useful for Chinese medicinal industry to prepare non-toxic herbal medicines efficiently.

Poster Session 1

34

NOBLE METAL NANOPARTICLES ON TITANIA NANOTUBES: PREPARATION, CHARACTERIZATION AND CATALYTIC PROPERTIES

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Titania nanotubes were synthesized by hydrothermal method with TiO₂ powders immersed in concentrated NaOH solution in an autoclave at 135°C. Preparation of well dispersed noble metal nanoparticles (Pt, Rh and Au) on titania nanotubes was fulfilled by photochemical deposition method with UV irradiation on an aqueous solution containing various metal complexes and titania nanotubes. The prepared samples exhibit high surface area (>300 m²/g) with nanosize metal crystallites (2-10 nm) uniformly dispersed on TiO₂-nanotube-surfaces, which were characterized by transmission electron microscopy. Their physical and chemical characteristics were investigated by nitrogen adsorption isotherm, temperature programmed reduction, X-ray diffraction, Raman, infrared, UV-Visible, XANES, XPS and electron paramagnetic resonance spectroscopy. The experimental results showed that the characteristics of the samples with photochemical deposited metal nanoparticles are distinguished from those of the conventional impregnated ones. The prepared titania nanotubes-supported metal nanoparticles exhibit extraordinary high catalytic activities in the reactions such as CO

Abstracts of Poster Program

oxidation, NO reduction, CO₂ hydrogenation. Besides, they also showed highly catalytic activity as a photocatalyst. Details will be discussed.

Poster Session 1

35 SURFACE MODIFICATION OF CARBON NANOTUBES BY DEPOSITION OF NANOSIZED METALLIC AND SEMICONDUCTOR PARTICLES

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We discuss covering multi-walled carbon nanotubes (MWCNTs) with nanosized metallic and semiconductor nanoparticles. In the first step MWCNTs have been positively or negatively charged (depending on the nanoparticles charge) by covering with cationic poly(diallyldimethylammonium) chloride (PDDA) or anionic poly(sodium 4-styrenesulfonate) (PSS) polyelectrolyte. The following step was the deposition of nanosized metallic (Au, SiO₂@Au) particles or rods, and semiconductor (CdSe, CdTe) particles. The successive covering process was optically registered and was correlated with the structural characterization by high resolution transmission and scanning electron microscopy.

Poster Session 1

36 ZETA POTENTIAL BEHAVIOR OF FUNCTIONALIZED METALLIC NANOROD PARTICLES IN AQUEOUS SOLUTION

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Emerging applications for metallic nanoparticles as substrates for biochemical detection, or as building blocks for biochemically directed self-assembly, require an understanding of the behavior of these particles in biologically compatible aqueous solution. The zeta potential of such particles is a major determinant of their ability to resist nonspecific aggregation, to avoid adhesion to the walls of a vessel or microfluidic device, and to be manipulated using electrokinetic techniques. We have carried out an experimental study of the zeta potential of gold, silver, palladium, and gold/silver composite nanorod particles in aqueous saline solution as a function of pH. Particles were synthesized via electrodeposition in anodic alumina templates and subsequent release. Measurements were performed using the electrophoretic light scattering technique. The study explores the effect of different surface functionalization states, including several alternative self-assembled monolayers as well as conjugation with antibodies and serum proteins. The effect of varying the ionic strength of the aqueous solution was also addressed. Significant differences in behavior were observed based on each of these variables. The results provide valuable data supporting the future development of nanoparticle-based techniques in engineering and biomedicine.

Abstracts of Poster Program

Invited Paper

Poster Session 1

37 CARBON NANOTUBE FORMATION DURING THE CARBONIZATION PROCESS IN SOLID COMPOSITIONS

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Carbon nanotubes (CNTs) are formed in bulk compositions from thermal decomposition of metal-containing organometallic compounds in the presence of a multi(ethynyl)aromatic compound. Our unique method involves a precursor composition that melts and is soluble in common solvents ensuring processability into shaped components. The method permits the large-scale production of CNTs in a solid, film, fiber, or powdered form. Heat treatment of various precursor compositions, formulated from an organometallic compound and an excess amount of a multi(ethynyl)aromatic compound to temperatures up to 1400°C, results in the decomposition of the organometallic compound and the formation of metal nanoparticles in the polymeric-to-carbon nanoparticle-to-carbon nanotube compositions. The growth of the CNTs proceeds in the solid phase during the carbonization process. In the synthesis, only a minute quantity of metal nanoparticles is required to initiate the formation of CNTs in the developing carbonaceous media. The metal nanoparticles are the key to the formation of the CNTs. X-ray diffraction, Raman spectroscopy, transmission electron microscopy (TEM), and high resolution scanning electron microscopy (HRSEM) studies show the formation of CNTs in the developing carbon composition above 600°C. The shaped compositions have structural integrity, which enhances their importance for potential nanoelectronic, electrical, magnetic, and structural composite applications.

Poster Session 1

38 HYDROPHILIC PARTICLES PRODUCED BY AN INVERSE MINISUSPENSION POLYMERISATION METHOD

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Surface modifications of particles are essential for their use in different applications. In all kinds of diagnostic use, when manipulation of biomolecules like e.g. antibodies and other binding proteins, or nucleic acids are required, it is crucial that particles are well dispersed in the reaction media. Surface modification and introduction of suitable chemical functional groups will not only assure homogenous suspensions during reaction, but will also act as anchors or chemical coupling points for the different ligands to be analysed. It is often necessary to optimize amount and type of chemical groups, length of spacers for coupling and hydrophobic / hydrophilic properties of the particle surface.

In many cases the manufacturing process for particles demands certain solvents to be used and hence makes it difficult to introduce the functionality in an easy and straight

Abstracts of Poster Program

forward way. Most studied and used are preparation of polymer particles through oil in water systems. Post modifications are often necessary to introduce the proper groups, and in many cases this implies a more expensive product. However, hydrophilic particles may be produced through an inexpensive one step method – “inverse” minisuspension polymerisation. With use of suitable oils, emulsifiers and organic solvents (included water), it is possible to prepare hydrophilic particles having chemical groups as hydroxy, amine, carboxylic acid, sulfonic acid or other polar groups. Both inorganic and magnetic particles may easily be produced in this way. Example of such particles will be presented.

Poster Session 1

39 NOBLE SYNTHESIS OF Fe/SiO₂/CNT NANOCOMPOSITE POWDERS BY CHEMICAL VAPOR CONDENSATION PROCESS

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Nanocomposite powders with the size of 1-100nm have been intensively studied due to their unique physical, mechanical, electric, magnetic and tribological properties. The nanocomposite powders could be fabricated by various processes such as inert gas condensation process (IGC), plasma arc discharge (PAD) and chemical vapor condensation (CVC). Among them, the CVC process can easily produce the nanocomposite or coating powders with high purity and non-agglomeration properties. In this research, Fe/SiO₂/Carbon nanotube (CNT) nanocomposite powders were synthesized by the CVC process using the metal-organic precursors. The effects of reaction parameters on phases, microstructures, and sizes of the synthesized powders were investigated. High-purity carrier gases were fed into a heated bubbling unit containing the iron carbonyl (Fe(CO)₅) and TEOS(Tetraethyl-orthosilicate, C₈H₂₀O₄Si) precursors. The CVC experiment was conducted at 500-1100°C. The characteristics of powders were analyzed by XRD, FE-SEM, TEM, Moessbauer spectroscopy and VSM, etc. By the analysis of XRD patterns, we knew the Fe/SiO₂ powders could be produced above 700 °C. With increasing reaction temperature, the XRD peaks became clearer. The Fe powders had intricate long-stand structure due to their magnetic characteristics. TEM results revealed that the Fe powders were covered by SiO₂ layer fully or partially depending on the experimental condition. The Fe/SiO₂/CNT nanocomposite powders could produce at 1100°C under room atmosphere.

Poster Session 1

40 PREPARATION AND PROPERTIES OF POLYMERIC NANOCOMPOSITE BASED ON HIGH-TEMPERATURE ORGANOPHILIC LAYERED SILICATES

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Surface modification of clay minerals has become increasingly important for improving the practical applications of clays such as polymeric nanocomposites. The layered

Abstracts of Poster Program

silicates modified with organic chemicals are widely used as nanosized fillers. Commercially available organophilic layered silicates (OLS) with alkyl chain modifier have a critical problem that the aliphatic components limit the thermal and mechanical properties. Practically, commercially available OLS such as Cloisite series begins to degrade at temperature below 200 °C. Most of polymers, however, are processed at the temperatures above 200 °C, implying that the commercially available organophilic layered silicates are not suitable for the preparation of polymeric nanocomposites because thermally fragile modifiers in the silicates are degraded at the processing temperature. To solve these problems, we designed the oligomeric modifiers free from aliphatic components, and successfully developed a route to an amine functionalized oligomers based on oligo(ethersulfone) and oligostyrene. These modifiers having onium ion effectively ion-exchanged with Na-MMT(Montmorillonite) to form an OLS. Several type of novel OLSs showed high degree of substitution and enlarged gallery spacing and high thermal stability. By solution and melt blending nanohybridization method, an exfoliated structure of PES/OLS, and PS/OLS nanocomposites are obtained. This means that the modification on the layered silicate surface leads to the full dispersion of the clay platelets. And also, we observed the improvement of thermal stability maintaining their inherent transparency as increment of OLS content. High-temperature OLSs appear to offer excellent potential as a nanoconstruction site for the synthesis of materials as high-temperature nanocomposites.

Poster Session 1

41

SYNTHESIS AND INKJET-PRINTING OF SPHERICAL SILICA PIGMENTS WITH INCORPORATED ORGANIC DYE MOLECULES
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We used an aerosol based process for the one-pot synthesis of spherical silica pigments with tailor-made optical properties. With our synthesis method it was possible to encapsulate an organic dye in solid silica particles or, alternatively, to incorporate the dye in the organic phase of mesostructured organic/inorganic hybrid particles. By spray-drying a precursor solution containing tetraethoxysilane, amphiphilic block-copolymer and the photochromic dye spirooxazine we were able to synthesize a photochromic pigment that is easily dispersed in, for example, aqueous film-forming latex suspensions. Switching UV illumination on and off makes the latex film change from being transparent to opaque and back to transparent with a decay rate of about $K_{SO}=0.09 \text{ s}^{-1}$. The inorganic/organic hybrid mesostructure provides a mechanically and chemically stable environment for the dye, well suited to improve its protection, handling and dispersion without compromising the fast photochromic response. The production process we used is fast and continuous, with a capacity to produce relatively large quantities of mesostructured particles. Adjusting the composition of the precursor solution gives control over both the internal mesostructure and the dye content in the produced pigment. TEM microscopy and XRD characterization of calcined particles showed well-ordered internal 2D hexagonal mesostructures with pore sizes varying from

Abstracts of Poster Program

2.5 to 8.5 nm as a function of templating amphiphiles. By spray-drying dye-containing precursor solutions without the amphiphilic template material we could produce solid silica pigments with encapsulated dye molecules. Ink formulations with these pigments combine the advantages characteristic to conventional pigments with the higher color intensity of organic dyes. We found that it is possible to print water-based inks of these pigments using a commercial drop-on-demand inkjet printhead. Moreover, the pigment surface chemistry is silica-like and practically independent of the type of encapsulated dye, which benefits formulation of color tints and gives access to a larger color gamut.

Poster Session 1

42 SYNTHESIS OF CELLULAR CATALYTIC STRUCTURES VIA SELF-ASSEMBLY OF NANO- AND MICRO-PARTICLES FOR MICROREACTOR SYSTEMS

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We developed a fabrication procedure, largely based on the use of polymeric and ceramic particles, to create cellular catalytic structures containing periodically interconnected open cells of 10 to 20 μm and coated with a thin layer of about 1 μm catalyst particles for microreactor applications. In this method, polystyrene microspheres were selectively infiltrated into the microchannel space of microreactor devices from suspension. The polystyrene microspheres were partially sintered to create the inverse template of a desired cellular structure. Subsequently, SiO_2 nanoparticle suspension was infiltrated into the interstices of the template. Upon drying and sintering, the solid silica skeleton was formed and the polystyrene microspheres were removed. The resulting cellular structure was infiltrated with a thin layer of catalyst particles in the particle size range of 20 nm to 2 μm via electrostatic layer-by-layer self-assembly. The infiltrated particles were immobilized by applying a porous sol-gel oxide layer that contained pores with the size of about 3 nm. The overall procedure was refined by systematically studying the effects of major processing parameters during each fabrication step on cell size, cell interconnectivity and skeleton density of the cellular structure for optimizing the mechanical, pressure drop, and mass transfer characteristics associated with designing this type of catalytic cellular structure for several microreactor systems.

Poster Session 1

43 SYNTHESIS OF LANTHANIDE FLUORIDE NANOPARTICLES OF VARYING SHAPE AND SIZE

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Luminescent materials are of great interest for a variety of optical applications including new display technologies and lasers. A number of inorganic compounds doped with lanthanides trivalent ions are known to exhibit interesting luminescent properties. We are investigating the preparation of luminescent inorganic nanoparticles coated with organic surfactants with the objective of incorporating these luminescent inorganic materials into

Abstracts of Poster Program

organic environments such as polymer matrices. Recent scientific literature demonstrates a growing interest in new methods of nanoparticle synthesis, driven primarily by an ever increasing awareness of the unique properties and technological importance of nanostructured materials. Major issues associated with nanoparticle preparation include the control of particle size and internal structure. We have explored several synthetic routes for the preparation of nanoparticles containing rare earth elements. The fabrication of nanoparticles within reverse microemulsions has been shown to be a convenient route to monodisperse particles of controllable size. Yttrium fluoride nanoparticles of varying crystallinity, shape and size are prepared by precipitation in reverse microemulsions of water in cyclohexane stabilized with polyoxyethylene isooctylphenyl ether. YF_3 particles obtained by the classical microemulsion method are found to be monodisperse amorphous spheres, with controllable diameters between 6 and 50 nm. Furthermore, particles of the same material obtained by a relatively minor variation of this method are found to be monodisperse single crystals of octahedral and triangular shapes. The size of the crystalline particles can be varied between about 25 and 350 nm. The formation of single crystals can be attributed to the slower incorporation of the precipitant into the micelles when introduced in this fashion.

Poster Session 1

44 COATING OF SUPERPARAMAGNETIC NANOPARTICLES WITH SILICA

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Superparamagnetic nanoparticles are of intensive interest especially for biomedical applications where their fast response to a magnetic field along with the absence of remanence allows for rapid captures and releases. Surface coating is required, however, in most cases to avoid their direct exposition to bio-environments for the purpose of improving their stability and biocompatibility. Silica has been shown as one of the most effective coating material, with an additional important benefit of yielding a platform for easy incorporation of a variety of biomolecules. Our work focuses on silica coating of two kinds of representative magnetic nanoparticles: water-dispersible iron oxide and oil-dispersible iron. For iron oxide nanoparticles stabilized with surfactants in an aqueous system, a uniform core-shell structure was directly formed via a convenient sol-gel process. The silica thickness was varied from about 10 nm to over 100 nm and the surface roughness was also under a good control. For iron nanoparticles stabilized with oleic acid, two steps were employed to generate silica shells: surfactant replacement followed by a sol-gel process. In this case, the thinnest silica shell formed was around 40 nm. The structure, morphology and composition of the initial and coated nanoparticles were characterized by transmission electron microscopy, X-ray diffraction, energy dispersive X-ray analysis, and X-ray photoelectron spectroscopy. In addition, their magnetic properties were studied with both DC and AC magnetometry. The results will be presented and discussed.

Abstracts of Poster Program

Invited Paper

Poster Session 1

45 SURFACE MODIFICATION OF PARTICLES AND SUBSTRATES WITH OXIDES, METALS OR ORGANIC MOLECULES

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We developed novel solution processes to modify surfaces of solids such as particles or substrates with metals, metal oxides or organic molecules. Self-assembled monolayers (SAM) having desired functional groups were prepared on particles or substrate to functionalize their surfaces by the immersion of solids into solutions containing SAM reagents. SAMs were connected to the substrates with chemical bonds and functional groups of SAM can be further modified to other functional groups by chemical reactions. We developed solution processes to control nucleation and growth of metal and metal oxides using SAMs. Thin films of inorganic materials such as TiO₂, Fe₃O₄, ZnO, ZrO₂, SrTiO₃, SnO₂, Ta₂O₅, HAp, Cu, Pd, etc. were deposited on particles or substrates. Crystalline anatase TiO₂, for instance, was deposited from a (NH₄)₂TiF₆ aqueous solution at 50 °C. Uniform amorphous TiO₂ was deposited from a solution containing 0.1 M titanium dichloride diethoxide at 25 °C. Pd catalyst was deposited on a SAM having amino groups from Na₂PdCl₄ aqueous solution and crystalline Fe₃O₄ was deposited on them in an aqueous solution containing Fe(NO₃)₃ and dimethylamineborane. These processes would contribute to the advancement of particle technology.

Poster Session 1

46 SYNTHESIS AND CHARACTERIZATION OF OPTIMALLY-PACKED EUROPIUM(III) NANOPARTICLES FOR TIME-RESOLVED FLUOROIMMUNOASSAY OF PROSTATE-SPECIFIC ANTIGEN

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Recent advances in fabrication and bioconjugation of nanometer-sized lanthanide(III) chelate particles have led to robust high specific activity labels. This study describes synthesis and characterization of optimally-packed lanthanide(III) nanoparticle labels and their use in a bioaffinity assay system. Europium(III) nanoparticles were prepared using extremely simple, inexpensive and fast agglomeration strategy. A core-shell type nanoparticle was synthesized from hydrophobic tris(dibenzoylmethane)mono-(phenanthroline) and tris(dibenzoylmethane)-mono(5-aminophenanthroline) europium(III) chelates with silica coat in aqueous solution. The number of europium(III) chelates within a single particle was more than 10-fold higher compared to polystyrene and silica-based europium(III) nanoparticles. The particle sizes ranged from 92 to 147 nm in diameter. The core-shell particle was further coated with a monoclonal antibody. The analytical performance of the bioconjugated nanoparticle label was evaluated in a model sandwich immunoassay of prostate-specific antigen. The detection limit of human prostate-specific antigen was 28 ng/L, 850 fM, in a microtiter plate format using time-

Abstracts of Poster Program

resolved fluorometry. The coefficient of variation ranged from 1 to 9%. The novel nanoparticle label can improve the specific activity of existing lanthanide(III) nanoparticle labels using a simplified preparation route. In addition, prepared high-density nanoparticle labels using lanthanide(III) chelates or other specific fluorochromes have potential use in number of other application fields.

Poster Session 1

47 LIPID MEMBRANE COATED MICROSPHERES FOR LABEL-FREE MOLECULAR DETECTION

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Of the variety of techniques available for particle surface modification, covalent modification seems to be most commonly used. Though well-established protocols for these modifications exist, such modifications are not always conducive to the study of systems that involve dynamic events occurring within the coating itself. Phospholipid bilayer membranes are unique two-dimensional films whose fluidity and ordered structure allow for display of multiple chemical moieties with well-controlled density and orientation. This control over density and specific orientation can be difficult to attain using covalent modification techniques. In addition, the membrane itself is a fluid environment. This is an important advantage in the study of biological systems, which rely on this characteristic fluidity for functionality. It has been shown that lipid bilayer membranes can be reconstituted, from a variety of sources, and can be used to coat certain solid supports, while maintaining these *in-vivo* aspects of ordered structure and fluidity. Interactions on or between membrane surfaces are primarily studied using fluorescent labels, with label-free techniques for study being somewhat limited either due to complexity or sensitivity of the technique. A new discovery in our laboratory of the self-assembly of membrane-coated glass beads into ordered arrangements has now given rise to a remarkably sensitive, simple, and label-free method of investigating these interactions. The membrane-coated silica particles exhibit colloidal phase transitions that are governed by membrane surface interactions. The collective phase behavior of the beads serves as a cooperative amplifier that produces a readily detectable response from a small number of molecular events on the membrane surface. As a result, binding events between ligands and membrane-bound protein(s) of interest alter the structure of the dispersion in measurable ways. Detection is accomplished using brightfield microscopy and pattern recognition software. Further statistical analysis of bead pair distribution functions enables quantitative determination of binding affinities.

Poster Session 1

48 FLUORESCENT POLYMER NANOPARTICLES: FABRICATION, CHARACTERIZATION, AND APPLICATION

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Fluorescent polymer nanoparticles (FPNs) were fabricated and modified for the sensitive

Abstracts of Poster Program

and selective detection of biological substances such as nucleic acids and proteins. FPNs are precipitated nanoparticles fabricated from amplifying fluorescent polymers (AFPs). AFPs are a class of conjugated polymers with a characteristic perylene unit that preserves superior optical properties even in solid state. The optical characteristics of AFPs are a high molar extinction coefficient, high quantum yield, and high photostability. The FPNs were fabricated by phase-inverse precipitation of the AFPs, and the particle size was controlled by modifying the chemical structure and concentration of the AFP and the salinity of solution. The FPNs were characterized by dynamic light scattering and transmission electron microscopy, and the size range of the nanoparticles was 15-500 nm. The FPNs showed efficient energy transfer to a quencher or dye, resulting in optical signal amplification. For the specific detection of dye-labeled peptides, the particle was activated by trivalent metal coordination to detect phosphorylated peptides in a homogeneous kinase/phosphatase assay. The statistical parameters of the assay, including the Z' factor, were determined in a multiwell microtiter plate. The FPN-based kinase homogeneous assay can be used for high throughput screening of small molecular inhibitors.

Poster Session 1

49

PROCESSING OF UPCONVERSION EMISSIVE PARTICLES FOR BIO-IMAGING

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Researches on bio-imaging are evolving in the research field of bio-nano-photonics, which is a key technology for bio-medical prevention, diagnosis, therapy and drug delivery system. Most of the conventional bio-imaging technologies are using protein or other organic molecules as markers, which are discolored quickly by the irradiation UV light for optical excitation. On the other hand, inorganic phosphor can restore the color under heavy irradiation. By sizing the inorganic particles into nano-size and bio-activate them by molecular modification, we can achieve long life bio-imaging. Further more, inorganic materials as rare-earth doped Y₂O₃ can emit visible light by irradiating mid-IR excitation light, which seldom damage the bio- or photonic systems. The purpose of this study is to develop bio-imaging phosphor under mid-IR excitation. As an inorganic phosphor, Er-doped Y₂O₃ was selected. The process for the targeting phosphor consists of two parts. One is a smart processing for preparing Y₂O₃ nano-particle. We developed a new laser processing for preparing single crystal Y₂O₃ particles in water. If we use conventional laser ablation technique in water, the resultant material is hydroxide that does not show any emission due to the heavy multi-phonon quenching due to the hydroxyl group. In the new process, we did not focus the laser light into on target to avoid the plasma formation in the water. The defocused light can be absorbed along with the grain boundary when with segregated ions on the boundary that absorbs the laser light. A pulsed laser irradiation generates heat that results in an impulse wave at the grain boundary. The impulse along with the grain boundary smoothly can detach a single

Abstracts of Poster Program

crystalline grain from the sintered target. By this new processing method, we could successfully create Y₂O₃ single crystalline particle from the sintered Y₂O₃. The other process is an assembling of bio-active molecule to the inorganic particles. Zeta-potential measurement showed that we need other methods than the ones used for gold or semiconductor particles.

Poster Session 1

50 PLASMONIC ENHANCEMENT OF FLUOROPHORES IN SOLUTION USING CORE SHELL NANOPARTICLES

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Surface plasmon resonance (SPR) generates an intense electromagnetic field around metal nanoparticles (NP's) and can lead to increases in both quantum efficiency and rates of radiative decay of closely associated dye molecules. Theoretically these effects can result in an order of magnitude enhancement in the fluorescence signal. When used in biochip applications these enhancements lead to improved performance with lower limits of detection, reduced reagent requirements and greater resolution. This paper focuses on the change in fluorescence when increasing the NP to fluorophore distance. The final NP consisted of a gold /silver metal core, with a silica shell of variable thickness and a final layer of fluorophores. The NP's were prepared using either the sodium citrate reduction method or a seeded growth method. To control the SPR, we used different molar ratios of silver nitrate and hydrogen tetrachloroaurate. The SPR, as a function of composition and NP size, correlated very well with theoretical predictions based on the Mie scattering theory. The dye employed here was Ru(II)-tris(4,7-diphenyl-1,10-phenanthroline). The dye bound efficiently to the NP surface in aqueous solutions via electrostatic interaction. For pure gold and pure silver NP's at short distances we observed quenching of the signal from the dye due to energy transfer from the dye to the metal surface. For gold / silver alloys with SPR at 435 and 450 nm we observed significant enhancements, due to matching of the SPR with the excitation band of the dye. Silica spacer layers of different thickness were grown on the NP surface using a vitreophilic ligand and sodium silicate solution at specific pH's. Transmission electron microscopy was used to characterise the NP shape and the thickness of the silica layer. The dependence of enhancement on the distance from NP surface was compared to a theoretical model.

Poster Session 1

51 HAEMOSTATIC EFFECTS OF POLYMERIZED ALBUMIN PARTICLES CARRYING FIBRINOGEN α -CHAIN DODECAPEPTIDE AS PLATELET SUBSTITUTES *IN VITRO* AND *IN VIVO*

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Abstracts of Poster Program

We have studied a prototype of platelet substitutes focusing on a dodecapeptide, HHLGGAKQAGDV (H12), which is specific for fibrinogen γ -chain carboxy-terminal sequence (γ 400-411). In this study, we conjugated H12 to the surface of polymerized albumin particles (polyAlb) as biocompatible carriers to produce particles having hemostatic ability, and evaluated their *in vitro* and *in vivo* effects.

Effect of H12-polyAlb (ϕ 260 \pm 60 nm) on platelet thrombus formation was evaluated *in vitro* with thrombocytopenic whole blood ($[\text{platelet}] = 2.0 \times 10^4 / \mu\text{L}$) under flow (shear rate; 150 s^{-1}). Thrombocytopenic rats were made by busulphan injection (dose: 20 mg/kg), and a 2.5 mm length x 1.0 mm depth template-guided incision (QuikheelTM) was made 1 cm from the tip of tail. The tail was immersed in a 50 mL cylinder of saline and the time taken to stop bleeding was measured.

When thrombocytopenic blood in the presence of H12-polyAlb ($[\text{rHSA}] = 0.14 \text{ mg/mL}$) was flowed on the collagen-plate, the surface coverage of DiOC₆-labeled platelets was increased to 3.9 \pm 1.1 % (n=3) from 2.1 \pm 0.4 % (n=3) in the absence of the H12-polyAlb. In the same experiments, rhodamine-labeled H12-polyAlb was found to be involved in platelet aggregates by binding to the surface of activated platelets, and then to enhance platelet thrombus formation. Hemostatic effect was studied *in vivo* by measuring tail bleeding time of thrombocytopenic rats 5 min after the intravenous administration of H12-polyAlb. The bleeding times of normal rats ($[\text{platelet}] = 8.1 \pm 0.9 \times 10^5 / \mu\text{L}$) and thrombocytopenic rats ($[\text{platelet}] = 2.0 \pm 0.3 \times 10^5 / \mu\text{L}$) were 187 \pm 51 (n=6) and 609 \pm 153 s (n=6), respectively. H12-polyAlb administration at a dose of 4 mg/kg significantly shortened the bleeding time to 342 \pm 73 s (n=10), whereas the polyAlb did not show any effects (553 \pm 104 s, n=6). These results indicate that the H12-polyAlb would be a suitable candidate for an alternative to human platelet concentrates.

Poster Session 1

52

MAGNETIC COLLOIDAL CRYSTALS AND ITS MAGNETO-OPTICAL PROPERTIES

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A magnetic field can alter photonic properties of magnetic colloidal crystals. One of its possibilities is the magnetic tuning of the structural color of a colloidal crystal. Monodisperse magnetic colloidal particles composed of a γ -Fe₂O₃ core and a silica shell were synthesized by a 2 step reaction. Colloidal crystallization was achieved by thorough deionization of the colloidal suspension. The structural color changes with the external magnetic fields. The change of the structural color was also confirmed from the change of the Bragg diffraction peak of reflectance spectra. This change is attributed to the perturbation to the force balance of the gravity acting on particles and the electrostatic repulsion between particles by magnetic force. Another possibility is that the light can interact with a colloidal crystal through magnetism. Recent measurement of the magneto-optical effects will be discussed.

Abstracts of Poster Program

Poster Session 1

53

DENDRIMER-MEDIATED NANOPARTICLE FORMATION:
PHOTOREDUCTION AND MAGNETIC NANOPARTICLES

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Polyamidoamine (PAMAM) dendrimers have been used for some time to form relatively monodisperse noble metal nanoparticles formed by chemical reduction following uptake of metal ions from precursor salts in solution. We have used photoreduction to produce very regular acicular nanoparticles of other metals, including magnetic metals such as Co, with controllable size selectivity. The photoreduction process is much simpler and cleaner than the chemical reduction process, obviating the need for post-reduction dialysis to remove unwanted ions, for example. The process is kinetically slower, and this has the unusual effect of allowing the dominate morphology of the metal nanoparticle to be expressed. For example, Co nanoparticles formed via photoreduction are rod shaped, with apparent attacking faults along the short-axis hcp (0001) direction. In this case, the resulting particles are magnetic (they have a measurable non-trivial coercivity) and remain metallic for some time even though the photoreduction process is carried out in water. Shape effects are also noted for Zn, Pt, Ru, among others. We present results for these and other studies related to understanding the mechanism of photoreduction and photoreduction of metals inside patterned dendrimer structures microcontact printed onto various substrates.

Poster Session 1

54

SYNTHESIS AND PHOTOCHEMICAL/PHOTOPHYSICAL PROPERTIES
OF SILICA NANOPARTICLES FUNCTIONALIZED WITH METHYLENE
BLUE AND THIONIN

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Searching new photoactive species to be used in Photodynamic Therapy we synthesized and characterized silica-gel nanoparticles containing two photoactive molecules, Methylene Blue (MB) and Thionin (TH). It is expected that in the particles the photodynamic properties of MB and TH are preserved with the advantage of being protected from external interferences. Nanoparticles were synthesized either by entrapping the sensitizer (sil-MB and sil-TH particles) or by linking TH to the surface (Cab-TH) by using the glutaraldehyde coupling reaction. The mean diameter obtained was 70nm for sil-MB and sil-TH particles and 50nm for Cab-TH particles. No changes in maxima absorbance and emission wavelength of MB and TH were observed in the particle as a function of the solvent. MB and TH in aqueous solution were reduced in the presence of NADPH causing total loss of absorption in 660 and 600nm, respectively. In the case of nanoparticles no changes in absorption intensities were observed after

Abstracts of Poster Program

NADPH addition, showing that the dyes are not accessible to NADPH. In fluorescence quenching studies, it was observed that the sil-MB, sil-TH and Cab-TH particles were quenched by Br^- and the calculated stern-volmer constants (0,04; 0,06 and 0,32, respectively) were much smaller than those obtained for the quenching of MB (0,80) and TH (1,70) in solution. Using time resolved NIR emission, we determined lifetimes of $^1\text{O}_2$ generated from particles in different solvents. These lifetimes are similar to those generated by free MB and TH. This occurs because O_2 can diffuse through the particle, reaching the excited sensitizer and generating $^1\text{O}_2$, which diffuses through solution. Comparing the behavior of nanoparticles with free sensitizers, we concluded that dyes in nanoparticles are protected from external interferences and still are able to generate $^1\text{O}_2$.

Invited Paper

Poster Session 1

55 PREPARATION OF SURFACE COATED ZINC OXIDE NANOPARTICLES

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Zinc oxide nanoparticles have been studied because of their unique properties, such as high reactivity, low sintering temperature, and good electrical-optical properties. In this study, surface coated ZnO nanoparticles have been prepared by a sequential precipitation process. Al_2O_3 , Bi_2O_3 , and SiO_2 coated ZnO nanoparticles were prepared. A typical experiment was done as follows: ZnSO_4 solution was added to an NH_4HCO_3 solution while stirring at 40°C , and a slurry of basic zinc carbonate (BZC) as a white precipitate was obtained. Polyvinylpyrrolidone (PVP) was added to improve the dispersibility of this BZC in some experiments. Then a surface coating was precipitated by adding $\text{Al}_2(\text{SO}_4)_3$ or $\text{Bi}(\text{NO}_3)_3$ solution into the BZC slurry, along with NH_4HCO_3 solution and stirring, to prepare ZnO nanoparticles coated with Al_2O_3 or Bi_2O_3 , respectively. Na_2SiO_3 and H_2SO_4 solution were added into a slurry of BZC with stirring to prepare ZnO nanoparticles coated with SiO_2 . After filtering, washing and drying, coated ZnO nanoparticles were prepared by calcining the resulting precursor powders at $400\text{-}600^\circ\text{C}$ for 1h. TEMs showed that there were homogeneous coatings on the ZnO nanoparticles, and the coated particles were about 30-50 nm in diameter. The thickness of the coated layer ranged from 5 to 15 nm. HRTEM and XPS showed that precipitated Al_2O_3 , Bi_2O_3 , and SiO_2 were coated on the surface of ZnO nanoparticles. PVP can also play an important role in the coating process. Al_2O_3 coated ZnO nanoparticles have excellent UV blocking properties; SiO_2 coated ZnO nanoparticles can yield good dispersibility in aqueous solution; and Bi_2O_3 coated ZnO nanoparticles yield ceramics with homogeneous microstructure.

Abstracts of Poster Program

Poster Session 1

56 SYNTHESIS AND CHARACTERIZATION OF MIXED METAL OXIDE NANOPARTICLES.

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New versions of aerosol or precipitation and sol-gel processes have been investigated to synthesize several mixed metal oxide nanoparticles, including lanthanum magnesium borate. Previous methods, such as solid-state sintering, required high temperatures that decompose some other substrates in desired composite mixtures. Our focus is on employing a continuous aerosol flow process with brief calcination residence time, and separately a sol-gel method that utilizes metal alkoxide or halide precursors that enable lower temperature for formation of desired crystallite phase and size. This poster presents synthesis and product characterization information. The work is supported by US DoE award DE-FC26-03NT4295 and was carried out at Clarkson University's Center for Advanced Materials Processing, a NYSTAR facility.

Poster Session 1

57 CARBON BLACK SURFACE CHEMISTRY MODIFICATION BY USING PLASMA TECHNIQUES (LOW AND ATMOSPHERIC PLASMA)

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Carbon black is known since many years to be a very interesting material in many applications. Although its important role as a reinforcement filler is the most known, carbon black has also many other promising applications such as pigment or carbonaceous support for catalysts. In any of these applications the interaction of carbon black with its surroundings, which could be a polymer matrix, a polar or apolar solution or maybe an environment where three state contact (gas liquid and solid) is needed, present a dramatic importance. Consequently the surface chemistry of carbon black will be a critical parameter to obtain a perfect interphase interaction, which will lead to excellent performance of the final material. The scope of this work is to achieve a chemical modification of the carbon black surface by using plasma techniques in order to improve carbon black interaction properties. Plasma was thought to be have a great potential of modification due to its very reach chemistry species, also the lack of solvents make it a very good option. To accomplish this goal both a low and atmospheric plasma reactors were adapted to treat this powder material. Nitrogen, oxygen, air, and ammonia were chosen as plasma gases and the filler was modified under different conditions (time, power, distance to the plasma origin). The final chemistry composition as well as the surface properties were studied: pH, X-Ray Photoelectron Spectroscopy, acidic titration, water solubility as polar solvent test as well as adsorption isotherms of polymers as apolar medium test among others. The results show that both low and atmospheric plasma are able to modify the surface composition of carbon black without modifying

Abstracts of Poster Program

other important parameters such specific surface area or structure. Although cold plasma presents the need of vacuum as a drawback, the higher parameter control may lead to materials with more specific properties.

Poster Session 1

58 SILVER NANOPARTICLE BIOSENSORS FOR DNA DIAGNOSTICS
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Metal nanoparticles show particular promise for biosensing as the particles are small enough to interact with biomolecules and can display tunable intense colours. We have developed a synthetic method for the preparation of silver nanoparticles of a wide variety of colours. The change in the particle colour is derived from changes in the particle size and morphology.

We have already shown that the colour of the nanoparticles is sensitive to changes in the environment at the surface of the particles e.g. upon binding of anti IgG to IgG functionalised nanoparticles, a colour change from red to purple is observed. Thus, it is possible to use silver nanoparticles for the colorimetric detection of antibody-antigen interaction. We are currently investigating the use of silver nanoparticles for the colorimetric detection of specific sequences of DNA. Here we report on synthetic strategies used to functionalize the nanoparticle surface with DNA.

Poster Session 1

59 2D SELF-ASSEMBLY OF GOLD-POLYSTYRENE CORE-SHELL NANOPARTICLES
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The surface plasmon absorption of metallic nanoparticle arrays is of considerable interest for photonic applications because of the tunable nature of the resulting extinction spectrum. In order to study the optical properties of such arrays, we have prepared gold nanoparticles capped with either alkane thiol or thiol-terminated polystyrene. These core-shell nanoparticles self-assemble into two dimensional hexagonal lattices when solvent cast onto a solid substrate. The self-assembly of the particles on the surface of an optical waveguide allows the extinction spectrum to be recorded in real time during the self-assembly process. Furthermore, this experimental setup permits both the transverse and the longitudinal coupled plasmon modes to be measured separately by the use of polarised light in the perpendicular (TE) and parallel (TM) orientations. The characteristic frequencies of these coupled modes are known to vary as a function of interparticle separation. In the case of the core-shell nanoparticles employed in this study, the spacing between the gold cores can be tailored by varying the length of the

Abstracts of Poster Program

adsorbed polystyrene chain. Measured spectra are compared with theoretical curves calculated using the Discrete Dipole Approximation (DDA).

Poster Session 1

60 FABRICATION OF METAL NANOPARTICLE ENSEMBLES BY THERMAL PROCESSING OF A POLYMER/NANOPARTICLE COMPOSITE

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Metal nanoparticles (NP) and their aggregates exhibit physical and chemical properties attractive for catalytic, optical, and biomedical device applications. This drives the development of the processes for novel nanoparticle 2D- and 3D-architectures on a solid support. We fabricated disperse and aggregated Ag and bimetallic Ag/Au NP structures with particle surface density of $(2 - 20) \times 10^{10} \text{ cm}^{-2}$ on glass using spin-coating and thermal processing of poly(vinyl)-N-pyrrolidone (PVP)/metal NP composite thin films. These structures vary from disperse nanoparticle layers to uniform NP aggregates of different shapes. The microarchitecture of the NP aggregates depends strongly on the temperature change rate and hold time, and on the initial surface concentration of NPs. The surface plasmon resonance (SPR) maximum is usually centered at 420-430 nm for disperse Ag NP layers on glass. It is strongly enhanced and shifts to 520 nm for the aggregated NP ensembles prepared by rapid thermal processing above 400 °C. The NP ensembles are capable of gradually de-aggregating in a controlled manner, forming a disperse NP layer upon further thermal treatment. Bimetallic NP layers with various Ag/Au ratios have either double or single SPR, depending on the process parameters. Ag/Au and pure Ag NPs differ in their aggregation behavior. We discuss the ordering and stability of nanoparticles in the aggregates, as well as their fragmentation phenomena.

Poster Session 1

61 HYPERDENSE AND SQUARE LATTICE-FREE COLLOID CRYSTALS FROM HIGHLY CHARGED MONODISPERSE POLY(STYRENE/MASS) PARTICLES WITH APTMS-MODIFIED GLASS SUBSTRATE

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Monodisperse latex particles are known to undergo ordering to form a colloidal crystalline phase with a hexagonal closed packing structure at low ionic strength when the repulsion between the particles is strong. Two and three-dimensional periodic structures from monodispersed colloidal particles can be easily made from natural convective drying of latex under appropriate conditions. In order to fabricate the high-quality crystals on the solid substrate, however, it is very important to understand the nature of the colloidal particles and substrate as well as nucleation mechanism of the

Abstracts of Poster Program

crystal. In this study, for two- and three-dimensional particle array, the natural convective drying method was applied with hydrophobically modified glass substrate, which was prepared with (3-aminopropyl)trimethoxysilane (APTMS). We showed that the particle deformation was observed at 20°C (this temperature is far from the film formation temperature) and this significantly affected the packing density and array pattern in the two and three-dimensional arrays from highly charged poly (styrene/NaSS) particle and APTMS-modified glass substrate. As the results of the particle deformation, hyperdense (packing density ~ 0.78) hcp (or fcc) packing was obtained and the square lattice structure was removed from the particle arrays. We do expect that the square lattice formation, point defects, line defects and dislocations can be effectively eliminated with this system toward minimal defect colloid crystals.

Poster Session 1

62 SYNTHESIS AND SURFACE MODIFICATION OF METAL PARTICLES BY POLYOL PROCESS

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The scientists are looking for new materials, devices and systems by controlling the behavior of atoms and electron in micro space. This has resulted in considerable development in the fields of communication devices, new materials and biotechnology in the second half of last century. In such development, the vacuum technology based dry process played a very vital role. However at the beginning of this century, the limit of such processes have begun to come within sight and wet chemical processes have been proposed as an alternative. In this paper, we describe the synthesis and surface coating of metal particles by polyol process. The synthesis of metal and alloy nanoparticles requires a non-oxidizing atmosphere and this is accomplished through polyol process. Though the process itself is well established, we have made progress in the synthesis of transition metal and platinum based alloy particles with diameter as low as few tens of nanometer and different crystal structures. And also, we have used this process to modify the surfaces of these particles with a second metal layer or polyol itself. For example, coating the micron sized copper particles (supplied by T. Asano, S. SCIENCE Co., Ltd, Tokyo 100-0005, Japan) synthesized by centrifugal atomizer process with silver to prevent the oxidation. The technical details of metal particle synthesis and surface modification will also be discussed.

Poster Session 1

63 PREPARATION OF MAGNETIC NANOPARTICLES FOR MAGNETIC FLUIDS HYPERTHERMIA (MFH)

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Magnetic Fluid hyperthermia (MFH) is a cancer treatment procedure by which the heat

Abstracts of Poster Program

generated around 42 °C by magnetic nanoparticles in an ac magnetic field destroy the cancer cells, with minimal injury to normal tissues. Recently a lot of researchers have shown interest in MFH and data from systematic studies have begun to flow out. Though the use of ferro and superparamagnetic magnetic nanoparticles have been proposed, the physical properties of the magnetic particles suitable under clinical conditions are yet to be defined clearly. In this paper, we discuss the physical properties of the nanoparticles suitable for hyperthermia based on the frequency dependent magnetic characteristic of ferro and superparamagnetic particles and report a one step synthesis of PEG coated magnetite particles by using polyol process. The physical properties such as magnetic [magnetic properties is itself a physical property] and specific heating rate of the nanoparticles exposed to an alternating magnetic field of 2 kA/m under the maximum frequency of 600 kHz will also be reported.

Poster Session 1

64 CONTROLLED SYNTHESIS OF HIGHLY MONODISPERSE ZINC SULFIDE NANOSPHERES FOR UTILIZATION IN CRYSTALLINE COLLOIDAL ARRAYS

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We developed a new method to control the synthesis of highly mono disperse ZnS particles in a broad size range of nm to micron diameters. We utilized the direct precipitation of ZnS in ethylene glycol, where thioacetamide was allowed to react with traces or low concentration of acidic water to produce the reactive sulfide anion which reacts with zinc ion to form highly mono disperse zinc sulfide nanospheres. It is possible to obtain highly mono disperse spherical particles in the range of nanometers and up to microns in size by controlling the sulfide concentration during the reaction either by changing the water content or the acidic conditions. TEM images indicate the formation of highly spherical monodisperse ZnS particles. According to X-ray powder diffraction measurements the nanoparticles have a cubic zinc-blende structure.

Upon drying droplets of these highly monodispersed ZnS nanospheres dispersed in ethanol on a glass slide a closed pack array is formed which diffracts light in the visible region. These results demonstrate the potential for utilizing these monodispersed, high refractive index spheres, in Crystalline Colloidal Arrays.

Poster Session 1

65 APPLICATION OF SILICONE COATING ON NANOPARTICLES IN AQUEOUS ENVIRONMENT

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A method to apply silicone coating onto metal oxide nanoparticles in water was

Abstracts of Poster Program

investigated. It was demonstrated that polymethylsilsesquioxane coating was applied onto ZnO nanoparticles without the need to use organic solvents or external heat. It was found that the coated nanoparticles were re-dispersible in either polar liquids, such as water, or non-polar liquids, such as many organic solvents. The coating is also effective in reducing the photoactivity of metal oxides. The effects of the conditions of coating process on re-dispersibility and photoactivity of resulting nanoparticles were studied. This process enables efficient and economical process of coating metal nanoparticles with organo-silicon materials for a wide range of applications.

Poster Session 2

66 SYNTHESIS AND CHARACTERIZATION OF TITANIA NANOPARTICLES PREPARED FROM A LOW-PRESSURE FLAT FLAME

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Nanoparticles such as titania prepared from gas phase techniques generally exhibit desirable characteristics such as narrow size distribution and processing advantages such as extremely short processing time, both of which are desirable for practical applications. Nevertheless, besides cost involved in nanoparticle production, the capability of the applied technique in fine tuning of particle characteristics such as particle size, crystal phase and particle morphology is without doubt one of the key factors determining the applicability of the technique in practical uses. The purpose of this study is to synthesize and characterize titania nanoparticles derived from a low-pressure flat flame operated under different combustion/operational conditions. Analytical techniques such as transmission electron microscopy and X-ray diffraction were employed in characterizing the as-derived titania powders. Particle characteristics as a function of combustion conditions such as fuel/oxidant ratios, flame temperature, chamber pressure, etc., will be discussed. It was shown, depending on the combustion/operational conditions employed, loosely agglomerated titania nanoparticles of mixed rutile and anatase crystal phases can be derived.

Poster Session 2

67 DETAILED INTERNAL MICROSTRUCTURE OF HOLLOW ZIRCONIA MICROSPHERES DERIVED FROM SOL-SPRAYED TECHNIQUE

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A detailed study of crust formation mechanism along with crust properties and internal microstructure of zirconia microspheres prepared from a sol-sprayed technique is presented herein. Zirconia sol particles of two different sizes, 10 and 60 nm, were prepared by hydrothermal method from zirconium oxychloride solution of the same

Abstracts of Poster Program

solids content. The solutions were sprayed ultrasonically into fine droplets in the micrometer to tens of micrometer in size. Subsequent drying and pyrolysis of unconverted salt into zirconia at elevated temperatures in the gas phase resulted in zirconia microspheres of two size group categories - around micrometer to submicrometer and another one around 10 μm size range, as seen in scanning electron microscopy (SEM). Particle sizes were shown roughly the same for both starting sol sizes. However, there are more broken shells derived with the smaller sol in comparison to the larger sol particles, possibly due to greater resistance for solvent evaporation through the smaller voids between the smaller sol particles stacking the crust, or the denser crust, than that resulted from larger voids between larger sol particles, or less dense crust. Hollow nature of these particles was revealed from broken particles. Cross-sectional specimens of the sample particles were prepared for transmission electron microscopy examination and the results will be presented.

Poster Session 2

- 68** AN EXAFS STUDY OF PLATINUM NANOPARTICLES SYNTHESIZED BY A LOW-PRESSURE FLAT FLAME
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Platinum nanostructures with high specific surface area have wide-ranging applications in numerous technologically important fields such as catalysis, sensor, opto-electronics, etc. Majority of the related literatures reported the synthesis or manufacture of platinum nanostructures based on wet-chemistry based methods. As technological demands and global competition for novel materials with intriguing properties surge, a great number of research teams worldwide are currently focusing on exploring novel synthesis routes or mechanisms. The aim of this research is to synthesize and characterize nanostructured platinum microspheres by a low-pressure flat flame technique. (EXAFS) and other analytical methods were used to analyze the as-derived powders. Collectively, the results obtained from these analyses confirmed the formation of microspheres comprising closely packed metallic platinum nanoparticles of face center cubic structure. Platinum nanoparticles contained within these sub-micrometer to tens of nanometer microspheres as derived with 0.1 and 0.01 M H_2PtCl_6 solutions have a volume-averaged crystallite size of 7.4 and 14.59 nm as determined X-ray diffraction (XRD), respectively. EXAFS analyses confirmed the as-derived particles are metallic platinum.

Poster Session 2

- 69** TIME EVOLUTION OF MORPHOLOGY AND SIZE OF HYDROTHERMALLY PREPARED ZIRCONIA SOL PARTICLES
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The fundamental understanding of time-dependent growth mechanisms involved in

Abstracts of Poster Program

nanoparticle formation in hydrothermal reaction is crucial to the predication and control of particle characteristics such as particle size, size distribution and morphology. It has widely been shown in literature that particle size and morphology of zirconia sols are dependent on precursor salt concentration and other factors. However, most studies have focused on size and morphology of nanoparticles derived from precursor salt solutions of either low or medium concentration. The aim of this study is to investigate time evolution of morphology, size and size distribution of hydrothermally prepared zirconia sol particles from low to high concentrations. Transmission electron microscopy and dynamic light scattering were used side by side in the analysis of zirconia sol particles prepared from a hydrothermal reaction. Dynamic light scattering analyses of the time-dependent particle growth revealed different growth rates and patterns for low, medium and high salt concentration, possibly indicating different growth mechanisms involved with different salt concentrations employed. Transmission electron microscopy of the sample sol particles extracted at different time stage in corresponds to the dynamic light scattering measurement was performed side by side so as to provide explanation about the different growth patterns observed and possible mechanisms involved.

Poster Session 2

70 SYNTHESIS AND ELECTRORESPONSIVE CHARACTERISTICS OF MONODISPERSE POLYMERIC MICROBEADS WITH CONDUCTING POLYANIILINE SHELL

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A core/shell microbead with a poly(methyl methacrylate) (PMMA) core and a conducting polyaniline (PANI) shell was synthesized and its electroresponsive characteristics were examined, when it was dispersed in silicone oil. Initially, the PMMA particles as a core material prepared by dispersion polymerization with uniform size distribution. PANI shell was attached to the surface via both an in-situ polymerization of aniline by adding an oxidant in an aqueous acidic solution and a reaction of a reagent having two aniline moieties with glycidyl groups on the acrylic core, of which provides grafting sites during PANI polymerization. Synthesized particles were examined using various analysis methods such as SEM, TGA, ¹³C-NMR and zeta-potential meter. Electrorheological fluids are suspensions of dielectric particles in a nonconducting liquid, and exhibit remarkable change in rheological properties, including a drastic increase in apparent viscosity, as well as the formation of reversible microstructures under an applied electric field. The ER fluid dispersed in silicone oil based on our synthesized core/shell microbeads showed ER effect, with a yield stress up to 80Pa under 3.0kv/mm dc electric field.

Poster Session 2

71 SYNTHESIS OF ZnO NANOCRYSTALS OF NARROW SIZE DISTRIBUTION VIA ESTERIFICATION OF ZINC ACETATE DIHYDRATE WITH ALCOHOLS

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Abstracts of Poster Program

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Zinc oxide (ZnO) nanocrystals were prepared via esterification of zinc acetate dihydrate with ethanol and *n*-pentanol using both solvothermal and chemical precipitation routes. X-ray Powder Diffraction (XRD), Scanning Electron Microscopy (SEM), and Photoluminescence Spectroscopy (PL) were used to characterize structure and morphology of the produced ZnO. The average diameter of the particles can be adjusted between 25 and 60 nm by controlling reaction time and acidity of the solution. The crystalline particles obtained by both methods can be prepared on the gram scale. A systematic kinetic study was performed for both reaction modes. In the former route, layered zinc hydroxyacetate was found as an intermediate product during transformation of zinc acetate dihydrate into ZnO. Particle formation was dramatically promoted by using *p*-toluene sulfonic acid monohydrate (TSA) as catalyst yielding monodisperse ZnO nanocrystals at a very short growth time.

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Poster Session 2

72

THE INTERACTION BETWEEN FUNCTIONALIZED SOFT POLYMER PARTICLES AND INORGANIC SURFACES

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Polyvinylacetate-*co*-ethylene (VAE) latex particles are commonly used as binder in many applications, including paints and adhesives. Generally, a coating in which the binder is formulated using latex functionalized with e.g. silane groups is found to have superior mechanical properties such as enhanced scrub-resistance and decreased water permeability. Yet the exact mechanism by which the functionalized polymer improves the product is unknown. The drying process of paint involves adhesion of the binder particles to inorganic constituents such as CaCO₃ and TiO₂ and, in some cases, coalescence of the binder particles into a coherent film. And the functional groups of the polymer may play a role in all these processes. In order to elucidate the role of the functionalization in a coating system, we investigate the interaction of ~100 nm latex particles with varying degree of functionalization and different inorganic materials using atomic force microscopy. At $T > T_G$, adhesion onto the substrate induces vast deformation of the particles into approximately the shape of a spherical cap, similar to the case of partially spreading liquid droplets. However, measurements of the particle height as function of volume show that the contact angle depends on the size of the particle indicating deviations from Young-Dupré-theory. This deviation directly shows that elastic contributions must be included in order to calculate the energy of adhesion. We find the scaling behavior of the height of the droplets as function of their volume to depend both on the functionalization of the polymer and the degree of hydrophobicity of the substrate.

Abstracts of Poster Program

Poster Session 2

73 SURFACE INTERACTION AND DISPERSION STABILITY CONTROL OF SILICA NANOPARTICLES IN MEK BY SURFACE MODIFICATION USING SILANE COUPLING AGENTS

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Polymer-based nano-composite materials with highly dense dispersion of silica-coated iron nanoparticles can be applied to GHz electro-magnetic wave absorbers for small devices. In order to improve the dispersity of silica coated nanoparticles and mechanical performance of such polymer-based composite materials with silica coated nanoparticles, surface modification of silica by coupling agent is widely accepted method. While many researchers have been focused on the characterization of the molecular level structure at particle-polymer interfaces and its relationship with mechanical properties, few researches have been discussed the effect of the molecular level structure at the interface on the dispersion stability and surface interactions in organic solvents. Furthermore, the surface modification on synthesized nanoparticle has not been focused in previous works. The first subject of this work is to investigate the effect of particle size ranging from several ten nm to submicron meter on reactivity of coupling agent on silica surface in MEK. The amount of reacted coupling agent per unit surface area of silica decreased with the decrease of particle size down to several ten nanometers. Secondly, in order to increase the amount of bonded coupling agent on the surface of silica nanoparticles, slight amount of pH-controlled water was added into MEK during hydration and condensation process of coupling agent and silica surface. The amounts of coupling agent bonded to silica nanoparticles have been remarkably improved. Finally, based on the measurement of suspension viscosity and surface force by colloid probe atomic force microscope, AFM method, the effect of surface modification on dispersion stability and surface interaction in MEK was discussed. While 20 vol% as-received silica MEK suspension had extremely high viscosity, the viscosity of silane-treated silica suspension has reduced due to the reduction of adhesive force and appearance of repulsive force between silica surface in MEK.

Poster Session 2

74 GENERATION OF PURE NANOPARTICLES FOR PHARMACEUTICAL APPLICATIONS BY HIGH-ENERGY MILLING

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Bioavailability of poorly water soluble drugs is improved by particle size reduction, State of the art is mechanical size reduction in suspensions with stabilizers in high-pressure homogenisers or stirred media mills. A new method to generate nanoparticles from soft materials with low contaminations has been developed. A high-energy planetary mill is

Abstracts of Poster Program

operated at temperatures continuously below -100 centigrades. Grinding media are balls of water-ice of about 5 mm diameter, which have a Mohs-hardness about 6 at these temperatures. Powder of solid carbon dioxide is added to the feed of the mill and prevents re-agglomeration during milling. After grinding, carbon dioxide sublimate at temperatures above -80 centigrades. Water-ice can be removed by freeze-drying or, if an aqueous suspension is demanded, becomes liquid after warming up. In the presentation, details of the high-energy planetary mill will be described and first results to generate nanoparticles of soft substances with low contaminations will be presented.

Poster Session 2

- 75** FILLERS FOR DIMENSIONALLY STABLE MATERIALS
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Several particles exhibiting negative coefficients of thermal expansion have recently been identified. Such particles incorporated as filler in a polymer matrix could result in a composite with near or complete dimensional stability over a range of temperatures. One such filler, ZrW₂O₈, has a highly negative coefficient of thermal expansion and its cubic structure leads to isotropic thermal behavior. Consequently, ZrW₂O₈ was investigated as a functional filler for polymer matrices. The dynamic mechanical properties along with thermal behavior of the filled systems were compared. A cyanate ester/ZrW₂O₈ (no surface treatment) sample exhibited the lowest coefficient of thermal expansion (CTE), and consequently was investigated in most detail. The final samples contained various weight percentages and sizes of ZrW₂O₈. The sectioned samples allowed investigation of filler percent and how it impacts overall CTE. Decreases in CTE of ~80% were found for samples containing ~82 wt% ZrW₂O₈. The lowest value observed for composite CTE was 2.8 ppm/K at 32 °C for a sample having 83% ZrW₂O₈, which had been subject to one temperature cycle. Finally, the effect of surface treatment on the composite CTE was determined.

Poster Session 2

- 76** ENGINEERING DNA-MEDIATED COLLOIDAL SELF-ASSEMBLY – SYSTEM DESIGN AND CRYSTALLIZATION
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The goal of these experiments is the self-assembly of novel, 3-dimensional colloidal crystal structures. The specific binding of complementary DNA strands is used to create short-range attractive interactions between DNA-grafted micron-sized colloids. These colloidal interactions will create multi-component colloidal mixtures where the

Abstracts of Poster Program

interactions between each component are independently 'programmed'. In theory, this technique should be able to produce well-ordered BCC, SC and diamond structures, as well as their alloy counterparts, the CsCl, NaCl and AnS structures, all at densities near close-packing. We have synthesized the sterically stable DNA-grafted particles using the solvent swelling/deswelling technique. PEG chains provided this extra steric stabilization for these particles. The particles showed the temperature dependent phase behavior and they were also reversible with temperature. In addition, we have successfully assembled the first colloidal crystal structures using them. The crystal structures showed a faceted shape that resembles the RHCP stacked colloidal crystals. The crystals melted immediately when the temperature was raised above the melting temperature (T_m), confirming that they were formed and held together by DNA hybridization. We also find that the particle crystallization kinetics became faster as the grafted DNA density was increased. In addition, we have developed the simple thermodynamic model that predicts the phase behavior (T_m) of the system to within a few degrees.

Poster Session 2

77

MULTI-WALLED CARBON NANOTUBE-REINFORCED
POLY(ETHYLEN 2,6-NAPHTHALATE) NANOCOMPOSITES BY MELT
BLENDING

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Multi-walled carbon nanotube (MWCNT)-reinforced poly(ethylene 2,6-naphthalate) (PEN) nanocomposites were prepared by a melting blending process in a twin-screw extruder to create advanced materials for possible practical applications in various industrial fields. The effect of MWCNT on the physical properties of the PEN/MWCNT nanocomposites was investigated. The incorporation of MWCNT accelerates the mechanism of nucleation and crystal growth of the PEN, this effect being more significant at lower MWCNT content. The strong shear thinning behavior observed in the PEN/MWCNT nanocomposites may be attributed to the orientation of rigid molecular chains in the nanocomposites during the applied shear deformation. The complex viscosity of the PEN/MWCNT nanocomposites slightly decreased by incorporating a very small quantity of MWCNT into the PEN matrix. The storage modulus and loss modulus of the PEN/MWCNT nanocomposites increased with increasing frequency, this increment being more significant at low frequency region. The loss tangent maximum of the PEN/MWCNT nanocomposites shifted to higher frequency region with increasing MWCNT content, implying the formation of densely network-like structures. The rheological behaviour of the PEN/MWCNT nanocomposites can be influenced by physical interactions such as nanotube-polymer and nanotube-nanotube interactions. In addition, the incorporation of MWCNT has a significant effect on the long relaxation time of the PEN/MWCNT nanocomposites.

Abstracts of Poster Program

Poster Session 2

78 INITIATED CHEMICAL VAPOR DEPOSITION (ICVD) OF POLYMERS ON MICRO AND NANOPARTICLES

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Solution-based methods for coating polymers on micro and nanoparticles often suffer from particle agglomeration due to liquid surface tension effects during drying. This usually limits the applicability of these methods to coating particles larger than 100 μm in size. We present a novel process, initiated chemical vapor deposition (iCVD), that would circumvent this barrier, enabling the coating of polymers on particles much finer than 100 μm , down to the nanometer dimension. iCVD takes advantage of a typical CVD environment to form conformal coatings on small and intricate objects. Further, iCVD combines the polymerization and polymer coating processes in a single step, making processing much simpler yet does not compromise on the design capabilities for surface functionalization. Unlike conventional CVD methods e.g., plasma enhanced CVD, iCVD is able to create clean, unscrambled polymers by relying on regulated initiation pathways to generate active radical species in the vapor phase which subsequently adsorb on a surface for polymerization. We will show examples of homopolymers and copolymers created using iCVD, providing spectroscopic evidence on the formation of well-defined polymer structures, microscopy images that show conformal coating with no particle agglomeration, and kinetic data that supports the radical polymerization mechanism. Further, we will demonstrate various uses of these polymers on fine particles, showing the formation of superhydrophobic surfaces, the binding of ligands for affinity studies, and the controlled release of particles.

Poster Session 2

79 SIZE CONTROL OF NANOPARTICLES USING SURFACE ADSORBERS DURING PRECIPITATION

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Size control of nanoparticles can be achieved by having adsorbing materials present during crystal nucleation. At low concentrations, the morphology is retained, while at higher concentrations, the morphology may change. Varying the chemical structure of the adsorbent can allow to direct the morphology. The possible variations of morphology are determined by the underlying crystal structure. Prescreening of probable candidates for the size and morphology control is achieved by evaluating their activity as growth restrainers during Ostwald ripening of the crystals (Gibbs-Thompson effect). The BNG model of crystal nucleation predicts that the crystal number increases linearly with restrainer concentration once a concentration threshold is exceeded. This prediction was experimentally confirmed for a model system, using restrainer concentrations ranging from none to 500 mg/l. Crystal size reduction was achieved between 735 and 135 nm (5.4 x) while the crystal number increased by a factor of 300. Changing the growth conditions allows further size control. This work gives quantitative guidance for the

Abstracts of Poster Program

formation of nanocrystals for systems where generally much larger crystals are obtained.

Poster Session 2

80 NANO AND MICRO-PATTERNED STRUCTURES OF POLY(ANILINE) FROM ADMICELLAR POLYMERIZATION

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Nano and micro-patterned structures of conductive poly(aniline) (PANI) on HOPG have been prepared from using adsorbed latex spheres as a mask followed by admicellar polymerization (AP). The structure formed (honeycomb-like structure vs. isolated dots) can be controlled by changing the size of the latex spheres in the masking step, as well as by adjusting the conditions for the admicellar polymerization. Sample characterization includes atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS). Structures obtained by this technique are compared to patterns obtained from masking with latex spheres coupled with adsorption of the polymer in the interstitial spaces. In addition, results on mica are presented.

Poster Session 2

81 EFFECT OF POLYMER-SURFACTANT COATINGS ON THE RHEOLOGICAL PROPERTIES OF HYDROPHILIC PARTICULATES

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A custom-designed apparatus termed the Yield Stress Adaptor has been used to evaluate the effect of polymer-surfactant coatings on the rheological properties of hydrophilic particulates (i.e. sand and nanoparticles) with the aim of reducing interparticle friction forces. High solids rheology tests show a significant reduction of interparticle friction forces in samples with adsorbed polymer and surfactant. A smaller reduction was found for polymer-only and surfactant-only coated samples. X-ray photoelectron spectroscopy was used to determine the concentration of organic species at the surface. The effect of polymer/surfactant concentration, adsorption time, and polymer molecular weight, surfactant charge and chemical structure, as well as particle size, shape, and surface chemistry are also addressed.

Poster Session 2

82 ELABORATION OF HYDROPHILIC, CATIONIC AND SUBMICRON MAGNETIC PARTICLES FOR BIOMEDICAL USE

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Abstracts of Poster Program

Magnetic particles are largely used in various applications in biomedical diagnostic such as in nucleic acids extraction and detection, cell sorting, immunoassay, bacteria and viruses isolation and biomolecules concentration. Nowadays, the main problem in biomedical diagnostic is to elaborate well appropriate functionalized magnetic particles dedicated to a given application. In fact, each application needs well-defined colloidal carrier in order to be adequately adapted in terms of dispersability, colloidal stability, biological compatibility etc.... In non-specific nucleic acids extraction, purification, concentration and enzymatic amplification, hydrophilic cationic submicron magnetic particles are of great interest. The hydrophilic property is to reduce the protein adsorption. The cationic character is to favour attractive electrostatic interaction with the DNA and RNA. The submicron size range is to avoid particles sedimentation and to favour high specific surface area.

Amino containing polysaccharides based magnetic particles are elaborated by exploring two routes: (i) adsorption of dextran onto iron oxide nanoparticles followed by dextran modification via oxidation and reductive amination and (ii) iron oxide synthesis in the presence of amino-containing dextran. The elaborated magnetic particles are characterized in terms of chemical composition, particle size and size distribution, surface charge density and magnetic properties. The elaborated functionalized magnetic particles are evaluated in nucleic acids extraction and amplification.

Poster Session 2

83

UNIFORM METAL LAYER FORMATION ON MONODISPERSE
POLYMER PARTICLE FOR ANISOTROPIC CONDUCTIVE
INTERCONNECTION IN PACKAGING DEVICE

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Polymer-metal composite particles within the size range of 1-10 μm have gained a widening interest in both chip-size packaging and anisotropic conductive interconnection. The composite particle can be prepared by several process including physical-chemical adsorption and electroless plating. The aim of our study is to prepare highly monodisperse polymer particle surrounded by continuous metal layer via electroless plating. Because of very high surface area of substrate (i.e., polymer particle), the process need some special technique to control the thickness and uniformity of metal layer, and some electrical properties. During the preparation of polymeric substrate particle, we incorporated some kind of functionality for ease of electroless plating. Moreover, we designed a new plating process that can control the reduction of metal ions. In this study, 4 μm of polydivinylbenzene particle (C.V 4.0%) was selected as a organic particle substrate and Nickel was chosen as a metal layer. A series of experimental showed that carboxyl-, thiol-, and hydroxyl group on substrate surface showed improved interaction between organic particle and Nickel layer. We also found that control of initial reduction rate of metal ion is a key factor for formation of metal layer. At a fixed condition, we could introduce a uniform Nickel layer in the thickness

Abstracts of Poster Program

range of 0.01~0.1 μm on polymer particle surface.

Poster Session 2

84 PHOTO-ACTIVE COLLOIDS: LIGHT DRIVEN AGGREGATION AND DEPOSITION

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Novel photo-active core-shell colloids were prepared by grafting spirobenzopyran methylmethacrylate-*co*-methylmethacrylate copolymers from silica particles utilizing the atom transfer radical polymerization method. Dispersions of these particles in non-polar solvents flocculated upon exposure to UV irradiation ($\lambda \sim 360$ nm). Subsequent treatment with visible light ($\lambda \sim 540$ nm) or heat, followed by mild agitation returned the dispersion to its original, unaggregated state. Application of polymer matrices provided a robust system yielding minimal fatigue and reproducible results even after a large number of aggregation/redispersion cycles. Moreover, it was possible to direct deposition of these modified colloids on a similarly modified surface into the areas patterned with UV irradiation. Synthesis, characterization, sedimentation/deposition behaviour, and rheological response will be described in the poster.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Poster Session 2

85 CONTROLLED NANOSCALE SURFACE MODIFICATION USING OPTICAL NEAR-FIELDS DRIVEN PROCESSES

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The interaction of sub diffractive light with surfaces provides additional control of particles. This feasibility study is aimed at the application of optical near-field phenomena to nanometre scale surface engineering and modification with an emphasis on non-resonant dry-etching and deposition experiments. Investigations in the field of nanophotonics are revealing that evanescent optical fields have the capacity to realize novel functions based on local electromagnetic interactions. The desired spatial confinement of such fields can be achieved by tailoring the macroscale dimensions of light propagation paths within the substrate. Optical near-fields by themselves, suitably configured in the reaction chamber, provide a tool for surface engineering and controlled nanoparticle fabrication and modification. Therefore, by varying precursors, gas-etchants, flow rates, pressure parameters and exposure wavelengths, efficient control over the growth or modification of nanoparticles is achieved. In our experiments (confined to sub-half-micron matrix cells with optical near-fields generated within them), we demonstrate the deposition, dry-etching, and non-resonant polymerization of materials. In particular, we present non-resonant deposition of metal particles from metal beta-diketonate

Abstracts of Poster Program

complexes, non-resonant polymerization of DUV material, and dry etching of organic and inorganic materials via non-adiabatic photochemical pathways. Such optical near-field driven processes can be utilized for emerging applications that require the formation of elaborate nanostructured particles or films, while maintaining a high degree of purity during the fabrication steps.

Poster Session 2

86 A MOLECULAR RULER BASED ON PLASMON COUPLING OF SINGLE GOLD AND SILVER NANOPARTICLES

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Förster Resonance Energy Transfer has served as a molecular ruler that reports conformational changes and intramolecular distances of single biomolecules. However, such rulers suffer from low and fluctuating signal intensities, limited observation time due to photobleaching, and an upper distance limit of ≈ 10 nm. Noble metal nanoparticles have plasmon resonances in the visible range and do not blink or bleach. They have been employed as alternative probes to overcome the limitations of organic fluorophores, and the coupling of plasmons in nearby particles has been exploited to detect particle aggregation by a distinct color change in bulk experiments. Here we demonstrate that plasmon coupling can be used to monitor distances between single pairs of gold and silver nanoparticles. We followed the directed assembly of gold and silver nanoparticle dimers in real time and studied the kinetics of single DNA hybridization events. These 'plasmon rulers' allowed us to continuously monitor separations of up to 70 nm for $>3,000$ s.

Poster Session 2

87 FABRICATION AND LUMINESCENCE PROPERTY OF SIZE CONTROLLED NANOCRYSTALLINE SILICON PARTICLES

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Nanocrystalline silicon (nc-Si) particles are one of the promising materials for application to bio-medical technology such as a bio-imaging with the visualization function, because they are a harmless, visible luminescent and nano-structured materials. The nc-Si particles are generally fabricated by using wet and dry processes. The luminescence color from the nc-Si particles fabricated by these processes is dependent strongly on the particle size. However, the formation quantities of nc-Si particles are not an enough amount. Therefore, it is important to control the particle size and increase the quantities of nc-Si particles in order to apply the nc-Si particles for the biotechnology applications.

Abstracts of Poster Program

Here, we report the luminescence property and formation of a large quantity of size controlled nc-Si particles using wet process.

The nc-Si particles were formed by chemical etching of Si powder with average size of approximately 1 μ m in a mixture of HF, HNO₃ and CH₃COOH as a etching solution. The etching time was 60min. The sample was estimated by photoluminescence (PL) and high-resolution transmission electron microscope (HRTEM) measurements.

The nc-Si particles with average size of approximately 2.5 nm were formed uniformly with a spherical shape. The quantities of nc-Si particles strongly depended on the mixture ratio of etching solution because the etching speed of Si powder was controlled by the mixture ratio. When the mixture ratio was HF: HNO₃: CH₃COOH =1:2:4, the nc-Si particles were formed about 40 mg per hour. The quantities of this nc-Si particles were largely improved in comparison with the nc-Si particles formed using wet and dry processes. Moreover, the nc-Si particles were stably and strongly emitted red light with a peak of 720nm.

Poster Session 2

88

MODIFYING THE NANOPORE SURFACE WITH LIPID AND PROTEIN FOR FLOW-THROUGH MEMBRANE-PROTEIN BIOCHIPS

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Substrate-supported lipid bilayers represent, perhaps, an ideal approach for substrate biofunctionalization: the resulting surface is biocompatible and has very low non-specific protein absorption. Moreover, lipid bilayers represent an ideal environment for incorporating membrane peptides and proteins and preserving their biofunctionality over a long time. Typically, substrate-supported lipid bilayers are formed on the outside surfaces of planar chips and nanoparticles but these structures are known to be short-lived and prone to contaminations. Here we show that many problems of substrate-supported bilayers could be solved by assembling these structures on the inside rather than outside surface of the nanoscale templates. Specifically, we show formation of stable lipid bilayers in a form of nanotubular structures that cover entire inner surface of the nanochannels formed inside anodic aluminium oxide. Depending on the deposition method both single bilayer and nested multiple bilayer structures can be formed. Characterization of this new class of substrate-supported bilayers with differential scanning calorimetry, EPR, and two-photon fluorescence microscopy shows that the surface covering is uniform, the lipids are well aligned, and the thermodynamics of bilayer is essentially unperturbed. These structure are stable for a period of at least a month when hydrated and essentially indefinitely when frozen. Moreover, the lipid bilayer surfaces are easily accessible to solute molecules and for binding membrane lipids and peripheral proteins. We show that these lipid nanotube arrays provide an optimal architecture for building lipid and lipid protein biochips as well as creating aligned arrays of membrane proteins for structure-function studies. Supported through grants from DOE and NIH.

Abstracts of Poster Program

Poster Session 2

89 ENHANCED LASER MARK CONTRAST PIGMENTS FROM CORE-SHELL NANOPARTICLES

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Our laboratory has developed a number of methods for encapsulating inorganic nanoparticles such as titanium dioxide in silsesquioxanes. In particular, two solution-based methods have been developed that can deposit as much as 50% wt. silsesquioxane. In one method, a silicone polymer such as polyphenyl silsesquioxane (PPSQ) is adsorbed onto the surface of titanium dioxide nanoparticles from a toluene-water dispersion. After removing the toluene, ammonium hydroxide is added to catalyze crosslinking of PPSQ and produce an insoluble shell. In a second method, a silsesquioxane shell is grown on the nanoparticle surface starting from an alkoxy silane. Titanium dioxide nanoparticles are dispersed in water with ammonium hydroxide, which creates a charged titanium dioxide surface that stabilizes the dispersion and which catalyzes the polymerization of the silane through hydrolysis and dehydrative polycondensation. Core-shell nanoparticles prepared by either of these methods can then be directly formulated into coatings. With proper choice of the silsesquioxane shell, such nanoparticles can be used, for example, as a pigment in a coating that yields irreversible black marks when irradiated with a laser.

Poster Session 2

90 COLLOIDAL SYNTHESIS OF MAGNETIC CORE-SHELL PARTICLES

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In my presentation I will discuss approaches to synthesize magnetic nanoparticles whose surface can be functionalize by adsorption of organic or inorganic shells fulfilling the requirements of possible applications. The organometallic synthesis of monodisperse magnetic nanoparticles with sizes ranging from 2 to 20 nm in organic solvents is of significant importance, because of its simplicity and control. Many different metallic and semiconductor nanoparticles, and core-shell particles consisting of an inorganic crystalline core surrounded by an organic monolayer have been synthesized.

In the case of biological applications organically synthesized particles need to be stabilized in a physiological environment. Therefore, following a basic procedure [Ref.: S. Sun *et al.*, *Science*, **2000**, 287, 1989] we demonstrate the synthesis of 3 nm $\text{Fe}_x\text{Pt}_{1-x}$ ($0.3 \leq x \leq 0.7$) nanoparticles in an organic solvent which can be transferred to aqueous solution by replacing the organic layer around the nanoparticles by another type of surfactant, tetramethylammonium hydroxide, obtaining water-based $\text{Fe}_x\text{Pt}_{1-x}$ nanoparticles [Ref.: V. Salgueirino-Maceira *et al.*, *Langmuir*, **2004**].

Abstracts of Poster Program

In addition to alloys, core-shell structured Co@SiO₂ nanoparticles (approx. 25-200 nm diameter) were synthesized and studied. Cobalt colloids have been homogeneously coated with silica using the silane coupling agent ((3-aminopropyl)-trimethoxysilane) as a primer to render the cobalt surface vitreophilic. The thickness of the silica layer can be controlled by the amount of tetraethoxysilane used in the procedure and the particles can be transferred into polar and non-polar solvents. The reversed core-shell structure can also be created using silica spheres as cores and depositing a thin shell of magnetic Co/CoO material (*Adv. Mater.* **2005**). All particles were analyzed by transmission electron microscopy and magnetic techniques for characterization.

Poster Session 2

91 CHEMICAL AND HEATING TREATMENTS OF STABLE IONIC MONOLAYER-PROTECTED GOLD NANOPARTICLES

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This poster describes the synthesis and chemical and heating treatment of stable nanoparticles protected by ionic ligands. Metal nanoparticles capped with ionic surfactants have drawn an increased interest due to several applications, which require temporary monolayer binding properties. However, their unsatisfactory long-term stability has limited the broader use of these nanomaterials. The availability of highly stable monolayer-protected nanoparticles with an ionic bonding between protecting ligands and particle surface should provide a diversification of available nanoparticulate materials and successful applications in catalysis and biosensing. Here, we present a facile synthesis of tetraoctylammonium-protected gold nanoparticles with improved chemical and thermal stabilities by introducing thiosulfate or sulfide counter anions in place of bromide anions. IMPCs are characterized using UV-vis spectroscopy, transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS). Chemical stability of the nanoparticles is examined by cyanide decomposition, which shows about two-fold increase in kinetic stability. IMPCs are stable up to 120 °C in dilute solution. Solid state heating treatment of IMPCs causes the partial removal of ionic monolayers and core size evolution of nanoparticles.

Poster Session 2

92 MODIFIED SUPERPARAMAGNETIC IRON OXIDE NANOBEADS FOR SINGLE CELL RNA EXTRACTION

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Only about 1 % of total RNA is mRNA, representing the current gene expression pattern of the cell. Typical RNA extraction is done from 10⁷-10⁸ cells. The detection of specific mRNA transcripts from a single cell is a technical challenge due to the very low amount

Abstracts of Poster Program

of mRNA. This limitation largely increases in the case of cells with a complex architecture such as neurons where the mRNA is transported to the dendrites. The dendrites of the neuron are embedded in a tri-dimensional matrix of other cells making it impossible to extract the intact neuron from the matrix for gene expression analysis.

The aim of our work is an important improvement of the single cell expression analysis by tagging the whole mRNA content of selected neurons with superparamagnetic nanoparticles. The magnetic tagging allows the isolation of the mRNA expressed in selected neurons from the rest of the cells in the tissue homogenate. For this 10 nm superparamagnetic iron oxide nanoparticles have been synthesized and consequently beads have been formed in a silica matrix with a final size of 100 nm. These beads have been tagged with avidin, capturing biotinylated poly(T). The final product as well as the intermediates have been characterized in terms of size, colloidal stability, morphology, and concentration. The magnetic beads show a fractal structure with a high protein immobilization efficiency. 100% sedimentation occurs on a permanent magnet (300 mT) within 20 min for protein modified nanoparticles. Protocols for RNA extraction and RT-PCR have been developed. It is shown that RNA extraction is possible with the developed and investigated 100 nm nanobeads.

Poster Session 2

93

SURFACE MODIFICATION OF NANOREACTANT PARTICLES USED IN EXOTHERMIC CONDENSED-PHASE REACTIONS

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Metastable nanoenergetic composites (MNC, also known as metastable interstitial composites or superthermites) are binary mixtures consisting of solid reactants in form of nanopowders. The MNCs, including Al-MoO₃, Al-WO₃, Al-CuO, and Al-Bi₂O₃, have shown excellent performance characteristics, such as impact sensitivity, high temperature output and low temperature ignition limit, which are very desirable in energetic materials applications. However, it has been found that the MNC powders are difficult to process safely due to their sensitivity to moisture, friction, and electrostatics. Therefore, mixing of powder reactants is usually carried out in a liquid suspension. Water as a benign and nonflammable liquid would be the best choice for the dispersing liquid but chemical stability of the MNC components in water suspension rise to a paramount problem, particularly for metal aluminum nanoparticles. Effective prevention of the hydroxylation reaction of the aluminum nanoparticles in liquid water is the main focus of this presentation. Aluminum nanopowders with surfaces coated by a protective organic and/or inorganic layer can effectively sustain contact with liquid water for several hours without decreasing the reactive aluminum content. This additional preparation of aluminum nanopowder complemented with dispersing additives allows for powder processing in water. Effect of surface modification of aluminum nanopowder on its decomposition rate in liquid water by monitoring pH and conductivity changes will be discussed. Examples of impact sensitivity tests for the percussion primers made from Al- Bi₂O₃ MNC will demonstrate feasibility of water-based processing of the energetic material. Recently, it

Abstracts of Poster Program

was also demonstrated that another class of exothermic reacting systems based on reactant nanopowders can be utilized in the formation of structural ceramic-intermetallic composites by using simultaneous combustion and densification process. Mixing and processing of nanoreactants participating in such combustion processes will be discussed as well.

Poster Session 2

94 HAEMOSTATIC EFFECTS OF POLYMERIZED ALBUMIN PARTICLES CARRYING RECOMBINANT GLYCOPROTEIN Ib α AS PLATELET SUBSTITUTES *IN VITRO* AND *IN VIVO*

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We have developed platelet substitutes focusing on the platelet membrane glycoprotein Ib α (rGPIb α), which recognizes von Willebrand factor (vWf)¹⁻³. In this study, we conjugated rGPIb α to the surface of polymerized albumin particles (polyAlb) as biocompatible carriers¹⁾, and evaluated the *in vitro* recognition ability with a flow chamber and *in vivo* efficacy with thrombocytopenic rats.

rGPIb α was conjugated to the surface of polyAlb with *N*-succinimidyl 3-(2-pyridyldithio)propionate (rGPIb α -polyAlb). Similarly, rGPIb α was bound to poly(ethylene) glycol (PEG)-polyAlb (rGPIb α -PEG-polyAlb). We analyzed their interaction with vWf-immobilized plate under flow conditions. Thrombocytopenic rats were made by busulphan injection (dose: 20 mg/kg), and a 2.5 mm length x 1.0 mm depth template-guided incision (QuikheelTM) was made 1 cm from the tip of tail. The tail was immersed in a 50 mL cylinder of saline and the time taken to stop bleeding was measured after 5 minutes administration of rGPIb α -conjugates.

Both rGPIb α -polyAlb and rGPIb α -PEG-polyAlb adhered and accumulated on the vWf-surface in a time-dependent manner at a shear rate of 1600 s⁻¹, whereas the adhesion was suppressed in the presence of anti GPIb/IX antibody (GUR83-35), indicating that they specifically recognized to the vWf. The bleeding times of normal ([platelet]=8.1 \pm 0.9 \times 10⁵/ μ L) and thrombocytopenic rats ([platelet]=2.0 \pm 0.3 \times 10⁵/ μ L) were 187 \pm 51 and 609 \pm 153 s (n=6), respectively. rGPIb α -polyAlb administration at a dose of 4 mg/kg significantly shortened the bleeding time to 340 \pm 52 s (n=5) in comparison with control polyAlb (597 \pm 124 s, n=7). On the other hand, rGPIb α -PEG-polyAlb at a dose of 1 mg/kg reduced the bleeding time (382 \pm 122 s, n=5) due to the PEG modification. rGPIb α -PEG-conjugates would be a suitable candidate for an alternative to human platelet concentrates.

1) *Biomacromolecules*, **1**, 290-295 (2000). 2) *Biochem. Biophys. Res. Commun.*, **296**, 765-770 (2002). 3) *ibid*, **306**, 256-260 (2003).

Abstracts of Poster Program

Poster Session 2

95 EFFECT OF SURFACE CHEMISTRY ON THE SORPTION, WETTING AND PHASE BEHAVIOR OF WATER AND SIMPLE FLUIDS IN NOVEL ORDERED MESOPOROUS MATERIALS

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Significant progress has been achieved during recent years with regard to the understanding of the sorption- and phase behavior of fluids in ordered mesoporous materials. This has led to important improvements in the physical adsorption characterization of porous materials. However, the influence of the chemical nature and heterogeneity of the pore walls on adsorption/desorption isotherms is still under investigation. In order to address some of these problems we performed a systematic experimental study on the adsorption- and phase behavior of simple fluids (nitrogen, argon, krypton, carbon dioxide) and water in novel mesoporous adsorbents consisting of similar ordered and well defined pore structure, but very different surface chemistry (e.g, CMK 1, CMK 3 mesoporous carbons, various periodic mesoporous organosilicas i.e. so-called PMO's, and SBA-15 silica). A detailed analysis of our water sorption isotherms in comparison with the sorption results obtained for simple fluids on the same materials leads to a better understanding of the interplay of confined geometry effects and the strength of the adsorption forces on the sorption, wetting and phase behavior of pore fluids. Our results are therefore important for an accurate physical adsorption characterization of mesoporous materials.

Poster Session 2

96 AN XPS STUDY OF PLATINUM NANOPARTICLES PREPARED BY A LOW-PRESSURE FLAT FLAME

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Platinum nanostructures with high specific surface area have wide-ranging applications in numerous technologically important fields such as catalysis, sensor, opto-electronics, etc. Majority of the related literatures reported the synthesis or manufacture of platinum nanostructures based on wet-chemistry based methods. As technological demands and global competition for novel materials with intriguing properties surge, a great number of research teams worldwide are currently focusing on exploring novel synthesis routes or mechanisms. The objectives of this study are to synthesize and characterize nanostructured platinum microspheres by a low-pressure flat flame technique. X-ray Photoelectron Spectroscopy (XPS) and other analytical methods were used to analyze the as-derived powders. Collectively, the results obtained from these analyses confirmed the formation of microspheres comprising closely packed metallic platinum nanoparticles of

Abstracts of Poster Program

face center cubic structure. Platinum nanoparticles contained within these sub-micrometer to tens of nanometer microspheres as derived with 0.1 and 0.01 M H₂PtCl₆ solutions have a volume-averaged crystallite size of 7.4 and 14.59 nm as determined X-ray diffraction (XRD), respectively. XPS analyses confirmed the composition of the surface layer of the microspheres to be metallic platinum without the presence of other platinum compounds.

Poster Session 2

97 SYNTHESIS AND GRAFTING OF FOLATE-PEG-THIOCTIC ACID CONJUGATES TO AU NANOPARTICLES FOR SELECTIVE TARGETING OF CANCER CELLS

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This research is aimed at the creation of Au nanoparticles that are stable in aqueous solution over a broad range pH and ionic strength and are capable of selective uptake by cancer cells. For this purpose we have synthesized polyethylene glycol molecules that are functionalized on opposite ends with thioctic acid and folate. Both these folate-PEG-thioctic acid conjugates and mPEG-thioctic acid conjugates have been successfully coated onto 10 nm diameter Au particles in aqueous solution. The mPEG and folate-PEG coated nanoparticles remain unaggregated over a pH range of 2 to 12 and at electrolyte concentrations of up to 0.1 M when the particle concentrations are as high as 1.5×10^{13} particles per ml. We have tested the performance of these coated nanoparticles in cell culture and used electron microscopy to demonstrate selective targeting towards cells that overexpress the folate receptor. Understanding this process is an important step in the development of methods which use targeted nanoparticles for drug delivery in cancer treatment.

Poster Session 2

98 NOVEL PREPARATION METHOD OF POLYMER NANOPARTICLES

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Nanoparticles are receiving great interest for their potential applications in the fields of electronics, photonics and biotechnology. Here, we report a novel preparation method of nanoparticles by mixing a poor solvent into a material solution. The evaporation of the good solvent can provide particles of diameter ranging from tens nanometer to micrometer scale of the material dispersed in poor solvent. By using this method, nanoparticles can be prepared from functional materials (i.e. dyes, electro-conductive polymers, biodegradable polymers). In this report, we show the preparation of nanoparticles with unique shapes and surface structures by using this method.

We show the preparation of nanoparticles from diblock copolymers. Water was slowly

Abstracts of Poster Program

mixing into tetrahydrofuran (THF) solution of poly(styrene-*block*-isoprene) (PSt-*b*-PI). After evaporation of THF, nanoparticles from PSt-*b*-PI were dispersed in water. By scanning electron microscopy (SEM) and transmission electron microscopy (TEM), They had phase separation structures on their surface. The phase separation structures can be controlled by the segment length and solubility parameters.

Furthermore, by annealing the block-copolymer nanoparticles at 50°C, which is lower than the phase transition temperature, the phase separation structure formed on the surface of particles was disappeared. This result indicates the phase transition temperature of the block-copolymer nanoparticles is lower than its film.

The micro-phase separation structures and thermal properties of block-copolymer nanoparticles will be discussed.

Poster Session 2

99

HYDROPHOBICALLY MODIFIED N-ACYLATED CHITOSAN GOLD ANOPARTICLES FOR DNA DELIVERY

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Special emphasis has been given on the role of individual functional groups of chitosan or its derivatives for the application in biomedical field. We synthesized the chitosan incorporated gold hybrid nanoparticles (i.e. chito/gold) by reducing the gold (tetrachloroaurate) salt with sodium borohydride in presence of aqueous *N*-acylated chitosan solution. The formulation of plasmid DNA (pcDNA3.1His/Myc/LacZ) on chito/gold nanoparticles (13nm) surfaces was tethered via electrostatic interaction, and the behavior of the complex in cell culture media was studied. Addition of plasmid DNA on the surface of highly dispersed positively charged *N*-acylated gold nanoparticles markedly reduced the surface potential as well as resulted in a 20~30% increase in hydrodynamic diameters. The *N*-acylated chito/gold /DNA nanoparticles could partially protect the encapsulated plasmid DNA from nuclease degradation as shown in electrophoretic mobility analysis. The transfection efficiency of chito/gold/DNA was dependent on cell type and higher β -galactosidase activity was observed on MCF-7 breast cancer cell. Typically, this activity was 10-15 times lower than that achieved by the complex of commercially available transfection kit (i.e. lipofectamine) and DNA.

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Abstracts of Poster Program

Poster Session 2

100 GROWTH OF HYDROXYAPATITE CRYSTALS USING CARBON NANOTUBES AS A MATRIX

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Hydroxyapatite (HA), the prime constituent of teeth and bone minerals involves in the biological calcification process of teeth and bone formation. The synthesis of HA with suitable morphology and controlled size is an important aspect to determine its applications in biomedical implants that can be achieved by growing it on nano-matrix. Herein, we report the formation of plate like multilayered HA crystals on the surface of carboxyl functionalized multiwalled carbon nanotubes (MWNTs) using calcium chloride and disodium hydrogen phosphate. The crystal growth is favored by $-\text{COOH}$ group, since the carboxylate ion is an excellent binding site for Ca^{2+} ions. The HA crystals thus obtained have been characterized by various physico-chemical techniques like FT-IR, XRD, SEM, EDX, and TEM. The bands characteristic of phosphate were found in the FT-IR spectrum. XRD showed the presence of HA crystals by exhibiting the significant diffraction patterns. The multilayered morphology with uniform thickness and elemental composition of the as synthesized HA were analyzed by SEM equipped with EDX. TEM images confirmed their unique morphology. In addition, the crystallinity of HA was identified with selected area electron diffraction (SAED) patterns. The results obtained in this study revealed that MWNTs functionalized with carboxyl group served as an efficient matrix for the growth of HA crystals.

Author, Speaker, Presenter Index

(Alphabetically With Paper Number; Boldface Corresponds to Presenter)

- Adair, James H.; **18**
Adelhelm, Philipp; 41
Advincula, Rigoberto C.; **2**
Alaouie, Ali M., 88
Alberius, Peter; 41
Alivisatos, A Paul, **26**, 86
Andersson, Nina; 41
Andres, Ronald P., 97
Antonietti, Markus; 28
Aryal, Santosh, 99, 100
Asher, Sanford A., 64
Atsumi, Takashi, 63
Auweter, Helmut; 19
Bae, T.S., 83
Bahadur, Remant K.C, 99, 100
Baksh, Michael M.; **47**
Baney, Ron; 21
Baptista, Maurício S.; 54
Bauman, Bernard D.; **10**
Behrens, Sven H.; **19**
Bell, Nelson S.; 18, 84
Ben-Moshe, Matti, **64**
- Benson, Adrienne; 21
Bhattarai, Shanta Raj, 99
Biancaniello, Paul L.; 7, 76
Bondioli, F.; **30**
Borrós, S.; 57
Bourgeat-Lami, Elodie; **25**
Brennan, Margaret E.; 58
Benicewicz, Brian C.; **5**
Brotzman, Jr., R.W.; **15**
Buechler, Karen; **17**
Chakarova, Gabriela ; 36
Chang, Chiao-Cheng; 32, **66**
Chang, Li-Chen; **31**
Chang, Yu-Chen; 31, **32**, 66, **67**, **68**, 69, 96
Chang, Yu-Chen;
Chang, Chao-Hung; **33**
Chen, Blithe; 33
Chen, H.; 42
- Cheng, Kuei-Yuan, 67, **69**
Cheong, In Woo, 61
Chien, Shu-Hua; **34**
Chinnasamy, C. N., 62
Cho, Min Sung, 70
Choi, Chul-Jin; 39
Choi, Hyoung Jin, **70**
Choi, Kyung-Eun, 100
Cook, Ronald; **9**
Corradi, A.; 30
Correa-Duarte, Miguel A.; **35**
Crocker, John C.; **7**, 76
Cross, William, 75
Daniel C. Coy, ; 15
Decher, Gero; **8**
DeHoff, Robert T.; 18
Demir, Mustafa M., **71**
- Dennis, Donn; 21
Dharmaraj, N., 100
Diaz, Rudy; 35, 90
Dixit, Vivechana, 97
Dixon, J., 95
Dougherty, George M.; **36**
Dreyer, Jakob Kisbye, **72**
Dubreuil, ; F. 12
Dyrli, Anne D.; 38
Elaïssari, Abdelhamid, 82
Elliott, Jeannine; 9
Ellsworth, Mark W., 89
Elsner, Nils; 12
Fang, Fei Fei, 70
Farle, Michael, 90
Feng, Si-Shen; **14**
Ferguson, John; 17
Ferrari, A.M.; 30
Fery, Andreas; **12**
Froeba, M., 95
George, Steven; 17
Giannelis, Emmanuel P.; **16**
Gibson, Stephen, 91
Giersig, Michael; 35

Author, Speaker, Presenter Index

- Gingras, Daniel, 59
Ginot, Frédéric, 82
Gleason, Karen K., 78
Glenn Judd, ; 15
Grady, Brian P., 80, 81
Groves, Jay T.; 47
Gu, Mingwei, 60
Guan, ; Jingwen 44
Gurevich, Laura, 89
Hakim, Luis; 17
Han, Seung San; 40
Hancock, Lawrence F.; 48
Handa, M.; 51, 94
Härmä, Harri; **46**
Harry Sarkas, ; 15
Henderson, Brooks, 75
Herranz, J.; 57
Heuvingh, J.; 12
Higutchi, Takeshi, 98
Hirakuri, Kenji, 87
Hisano, S., 62
Hofmann, Heinrich, 92
Hu, Peng; 55
Huang, Shu-lan; 55
Huang, Zhaohua; 23
Huwe, H., 95
Hwang, Pyoung Han, 99
Iijima, Motoyuki, **73**
Ikeda, Y.; 51, 94
Isaacs, Steven, 91
Itri, Rosângela; 54
J. Piepenbrink, ; 15
Jagiello, J., 95
Jeyadevan, Balachandran, **62, 63**
Juhnke, Michael, **74**
Jun, J.B., 83
Kabalnov, Alexey; **24**
Kamiya, Hidehiro, 73
Karlsson, Ola, 72
Kellar, Jon , 75
Keller, Teddy M.; **37**
Kelly, John M.; 58
Keränen, Anne-Maria; 46
Kilaas, Lars; **38**
Kim, Anthony J.; 7, **76**
Kim, Hak Yong, **99, 100**
Kim, J.I., 83
Kim, Jin-Chun; **39**
Kim, Jun Young, 77
Kim, Jung Hyun, 61
Kim, Seong Hun, **77**
Kim, Sun Young, 99
Kim, Yong Seok; **40**
Kitajima, Kazutoshi; 52
Kjerengtroen, Lidvin, **75**
Kobayashi, Kazushi; 52
Koizumi, R.; 49
Kumar, Rajneesh; 18
Kuo, Ming-Chih; 34
Kyrilidis, Angelos; **6**
Laarz, Eric; **41**
Laskoski, Matthew; 37
Lau, Kenneth K.S., **78**
Ledwith, Deirdre M; 58
Lee, Il Sang, 70
Lee, Jae Heung; 40
Lee, Jung Min, **61**
Lee, Y-H.; 21
Lee, D-W.; 21
Lee, W.Y.; **42**
Leite, Carlos; 54
Lemyre, Jean-Luc; **43**
Leonelli, C.; 30
Leubner, Ingo H., **79**
Lewis, Sarah; 5
Li, Chunzhao; 5
Li, Jin-lin; 55
Li, Shao-hua; 55
Li, Weijun; 23
Lieberwirth, Ingo, 71
Lindgren, Mikael; 41
Liphardt, Jan, 86
Liz-Marzan, Luis M.; 35
Lövgren, Timo; 46
Ma, Dongling; **44**
MacCraith, Brian D.; 50
MacKay, Andrew; 23
Markram, Henry, 92
Marquez, Maricel, **80, 81**
Masuda, Yoshitake; **45**
Martin, Barry, 85

Author, Speaker, Presenter Index

- Martin, Phil J., 85
Matsuno, Ryosuke; 22
Matuura, D.; 49
McCormick, Paul, 65
McDaniel, William R.; 48
McDonagh, Colette; 50
Moon, Joong Ho; **48**
Morell, J., 95
- Morey, Tim; 21
Mouaziz, Hanna, **82**
Mueller, Renate; 12
Myer, Lonnie; 4
Myers Andrew; 9
Nagasaki, Y.; 49
Nakabayashi, Seiichiro; 52
Ngai, To; 19
Nick, Robert; **20**
Niederberger, Markus; **28**
Nooney, Robert I.; **50**
Normandin, Francois; 44
Nylander, Tommy, 72
Okamura, Y., **51**, 94
Örtegren, Jonas; 41
Otsuka, Hideyuki; 22
Paciotti, Giulio F.; 4
Pannu, Satinderpal; 36
Park, Jin-Gyu, **83**
Partch, Richard; 56, 21
Patel, Krupa, 80
Patrick G. Murray, ; 15
Penn, Sharron; 36
Petri-Fink, Alke, 92
Philippe, Aurelien; 50
Piculell, Lennart, 72
Piech, Martin, **84**
Pinna, Nicola; 28
Polleux, Julien; 28
Polonski, Vitali, **85**
Powell, Evon; 21
Puszynski, Jan A.,93
Qadri, Syed B.; 37
Qiu, H.; 42
Radzilowski, Leonard H., **89**
Rast, Lauren, 60
Reinhard, Björn M, **86**
- Ritcey, Anna M.; 43, 59
Robb, Ian, 81
Robben, Amanda, 81
Rodriguez-Toledo, Maria, 92
Roger Cayton, ; 15
Rose, Klint A.; 36
Rossi, Liane M.; 54
Salgueirino-Maceira, Verónica, **90**
Sato, Keisuke, 62, **87**
Sato, Yoshinori, 62, 63
Schadler, Linda S.; 5
Schaefers, Sarah, 60
Schmidt, Helmut; **1**
Schmidtke, David W., 80
Schneider, Grégory; 8
Schuster, R.H.; 57
Schutt, Ernie; 24
Shastri, V. Prasad; **29**
Sherman, Debbie, 97
Shimomura, Masatsugu, 98
Shinoda, Koichi, 87
Shon, Young-Seok, **91**
Simard, Benoit; 44
Simon, C.; **27**
Skandan, Ganesh; **13**
Small, Adam, 91
Smirnov, Alex, **88**
Soga, Kohei; **49**
Sogoshi, Norihito; **52**
Sönnichsen, Carsten, 86
Stanishevsky, Andrei, **60**
Starkovich, John, 75
Steitz, Benedikt, **92**
Step, Eugene; 6
Stranik, Ondrej; 50
Street, Shane C.; **53**
Sudarshan, T.S.; **3**
Sullivan, Sean; 6
Suzuki, S.; 49
Swiatkiewicz, Jacek J., **93**
Szoka, Francis; **23**
Tada, Dayane B.; **54**
Takahara, Atsushi; **22**
Takeoka, S.; 51, **94**
Tamarkin, Lawrence; 4
Tannahill, Tania; **56**

Author, Speaker, Presenter Index

Theretz, Alain, 82
Thommes, M., **95**
Thompson, David H., 97, **11**
Tohji, Kazuyuki, 62, 63
Tok, Jeffrey B.-H.; 36
Tricás, N.; **57**
Trotter, Geoff, 65
Tseng, Huan-Hsiung, 68, **96**
Tsukada, Mayumi, 73
Tsuzuki, Takuya, **65**
Vallee, Real, 59
Van den Bossche, Jeroen, **97**

Varshney, Manoj; 21
Venkatesan, Umamaheswari, 88
Veres, Teodor; 44
Veyre, Raphaël, 82
Wegner, Gerhard, 71
Weichert, Reiner, 74
Weimer, Alan; 17
Welsh, Jeffry, 75
Weyer, Wayne, 75
Whelan, Aine M. **58**
Williamson, Heather, 60
Winter, Esther M.; 47
Won, Jong Chan; 40
Wu, Hai-Ping; 33
Xiong, Kao; 6
Yabu, Hiroshi, **98**
Yamada, M.; 49
Yamamoto, T.; 49
Yanagisawa, Satoshi, 87
Yi, Ho-Keun, 99
Yockell-Lelievre, Helene, **59**
You, Wen-Yueh; 34
Yuan, Fang-li; **55**
Yuan, Shih-Jeing; 33

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