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# Controlled Synthesis And Characterization Of Nobel Metal

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Les Vrais Incroyables ou les Métamorphoses modernes

Synthesis, Formation, Assembly and Application

Refined Synthesis and Characterization of Controlled Diameter, Narrow Size  
Distribution Microparticles for Aerospace Research Applications

Block Copolymer Templated Nitrogen-enriched Nanocarbons

Controlled Synthesis of Chalcogenide and Halide Perovskite Semiconductor  
Nanostructures

Metal Oxide Nanoparticles in Organic Solvents

Nanoparticle Matter

Controlled Synthesis and Characterization of Metal Oxide Nanowires by Chemical  
Vapor Deposition on Silicon and Carbon Substrates

comédie en un acte et en vers représentée pour la première fois, à Lyon, le 27  
prairial an VIe par Mr. Amar, ...

Boranes—Advances in Research and Application: 2013 Edition

Nanomaterials for 2D and 3D Printing

Characterization of PmHS2 Glycosyltransferases for the Controlled Synthesis of Heparosan

Design, Synthesis and Characterization of Metal Oxide Adsorbents and Catalysts for Environmental Applications

Carbon Nanotubes and Titanium Oxide Nanowires

Controlled Synthesis and Characterization of One Dimensional Nanomaterials

The Colloidal Chemistry Synthesis and Electron Microscopy Characterization of Shape-controlled Metal and Semiconductor Nanocrystals

Shape-Controlled Synthesis for Catalysis, Plasmonics, and Sensing Applications

Controlled Synthesis, Characterization and Applications

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A Precursor of Heparin and Heparan Sulfate

Doped and Undoped Nanoparticles

Controlled Synthesis, Physicochemical Characterization and Application as Pd

Catalyst Supports in a Hydrogenation Reaction

Synthesis, Characterization, Control, and Application of YSZ, CZTS, and ZrN

Synthesis, Characterization and Aggregation Behavior of Carbon Nanotube-metal Oxide Nanohybrids

Controlled Synthesis and Characterization of Some One-dimensional Semiconductor Nanomaterials

Scholarly Brief

Graphene Nanoribbons and Their Polymeric Nanocomposites

Characterization, Properties and Applications

Bimetallic Nanostructures

Gu shi taolun ji

Synthesis and Characterization

Controlled Synthesis and Characterization of One-dimensional II-VI Nanomaterials

Metal Nanocrystals

Controlled Synthesis and Characterization of Silicon Nanocrystals

Controlled Synthesis and Characterization of Templated, Magneto-responsive Nanoparticle Structures

Controlled Synthesis of One Dimensional Nanostructured Materials and Their Applications as Catalyst Supports in Proton Exchange Membrane Fuel Cells

Synthesis, Characterization and Manipulation of Carbon Nanotubes

Design, Synthesis and Characterization of Transition Metal Oxide/sulfide-based Catalysts for Environmental and Energy Applications

*Controlled Synthesis  
And Characterization Of  
Nobel Metal*

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## **NOELLE BRADLEY**

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### **Les Vrais Incroyables ou les Métamorphoses modernes**

Science & Business Media

"Reversible-deactivation radical polymerization (RDRP), also referred to as controlled/"living" radical polymerization (CRP) has been developed over the past 20 years. RDRP promotes the synthesis of well-defined polymeric materials with controlled molecular weights and molecular weight distributions, complex topologies and functionalities. In the past decade, the Matyjaszewski and Kowalewski groups pioneered the work of synthesizing nanostructured carbon by pyrolysis of

polyacrylonitrile (PAN) containing polymeric precursors prepared via RDRP. My contribution to this topic was primarily focused on the following aspects: (1) optimization of the synthetic procedure, (2) deeper investigation of the structures, (3) exploration of the surface chemistry with particular emphasis of nitrogen functionality, (4) and energy related applications. This thesis first focuses on addressing current challenges in RDRP particularly in atom transfer radical polymerization (ATRP), one of the most robust RDRP techniques. Based on the development of a deep mechanistic understanding of RDRP's, ATRP was then used for the synthesis of PAN containing block copolymers followed by applying a series of analytical tools to provide detailed

physical characterization. Finally, these materials were utilized as precursors for the formation of nanocarbons that were evaluated in various energy related applications. The development of nanostructured carbon materials from PAN precursors is discussed in Chapter 1. Particular emphasis is placed on the rational structural design of PAN containing polymeric precursors developed in the Matyjaszewski and Kowalewski groups, while the detailed synthetic methodology will be discussed in the subsequent chapters. Controlled synthesis is the prerequisite for many applications. The successful preparation of block copolymers via RDRP requires preparation of a macroinitiator with preserved chain end functionality (CEF). Work described in Chapter 2 resulted in

the establishment of a universal rule for quantifying the CEF in all RDRPs, which is also the most important criterion for determining the "livingness" and degree of control over the polymerization. The parameters affecting the level of CEF preservation are determined. Another challenge in ATRP is diminishing the concentration of catalyst employed during the polymerization procedure in order to reduce the cost and simplify the purification steps. Chapter 3 describes the systematic study of RDRP in the presence of zerovalent copper, which offers significant advantages in this regard. The contribution of all of the potential reactions occurring in an ATRP carried out in the presence of copper zero were evaluated, and a supplemental activator and reducing

agent (SARA) ATRP mechanism is concluded to precisely describe this system. How to conduct and optimize SARA ATRP system is then demonstrated. Chapter 4 is focused on another aspect of the robust capability of ATRP : controlling the molecular weight distribution. Activator regeneration electron transfer (ARGET) ATRP was employed to prepare polystyrene-block-poly(methyl acrylate) copolymers with tunable dispersity in the range of 1.32 to 2.0 for each block. Knowledge attained from the studies discussed in Chapter 2 to 4 has been extensively utilized in the studies of nanocarbons. Chapter 5 discusses the preparation of a series of PAN containing diblock copolymers that were used as precursors for the preparation of

nanocarbons. The block copolymers undergo phase separation and then the poly(n-butyl acrylate) serves as a sacrificial segment upon pyrolysis. Both thin film and bulk nanocarbons with diverse morphologies, resembling the original phase-separated copolymer precursors, were prepared. The carbonization of bulk copolymer precursors with branched PAN domains was of particular interest; which resulted in the formation of porous nanocarbons with large surface area and highly accessible nitrogen functionality originating from PAN. Chapter 6 illustrates how porosity and accessible nitrogen functionality in the nanocarbon introduced in Chapter 5 can be utilized for CO<sub>2</sub> capture. The main emphasis was placed on the surface area and nitrogen

content's influence on adsorption capacity and selectivity was studied. Chapter 7 discusses the application of PAN-derived nanocarbons as electrode materials for supercapacitors. Materials displaying both high energy density and high power density were achieved. This excellent performance was partially due to the mesoporous structure with high specific surface area, in combination with the pseudocapacitance originating from graphitic edge nitrogens. Evidence of electrochemical activity of the nitrogen heteroatoms provided the motivation to explore the performance of copolymer templated nanocarbon as an electrocatalyst for oxygen reduction, as described in Chapter 8. A desirable 4-electron transfer process with a low overpotential system was achieved by

as-prepared nanocarbon film with porous morphology; which again, demonstrates one of the unique properties of nanocarbons prepared from PAN containing block copolymer precursors. Finally, a summary is provided in Chapter 9 and some future directions regarding synthesis and utility of heteroatom-enriched nanocarbons are discussed."--Pages ii-v.

Synthesis, Formation, Assembly and Application John Wiley & Sons

Carbon nanotubes (CNTs) are advanced materials that have numerous novel and useful properties. Controlling the synthesis and properties of CNTs is the major challenge toward their future applications. This thesis addresses this challenge with several contributions. This thesis begins with the brief

introduction of CNTs, including the history of their discovery, their geometric structure, unique properties and potential applications. Then focus is laid on the subsequent three sections: characterization, synthesis, and manipulation of CNTs. Chapter 2 describes three characterization tools: AFM, SEM and Raman, which are commonly used to analyze CNTs and other nanomaterials. They offer both qualitative and quantitative information on many physical properties including size, morphology, surface texture and roughness. Also, they can be used to determine the structure of CNTs. Chapter 3 addresses the synthesis of CNTs, because synthesis is an important and indispensable process to study CNTs experimentally. Specifically, two

controllable synthesis techniques are realized, which are capable to produce iron catalyst nanoparticles for single-walled carbon nanotube (SWNT) growth. Iron nanoparticles of different sizes obtained from both wet chemistry and electrodeposition can be used for diameter-controlled synthesis of SWNTs. Following synthesis, two manipulation methods of CNTs are discussed in Chapter 4. Firstly, effort of electrical breakdown of CNTs is introduced. Both SWNTs and MWNTs (Multi-walled carbon nanotubes) are cut using this method. Moreover, SWNT kink is shown using AFM tip manipulation. These two manipulation methods provide us a possibility to fabricate large cavity from a MWNT for our purposes. In the end of this thesis, conclusions on my master



work in research field of CNTs are drawn and future research directions are proposed.

Refined Synthesis and Characterization of Controlled Diameter, Narrow Size Distribution Microparticles for Aerospace Research Applications  
Controlled Synthesis and Characterization of Silicon Nanocrystals  
Controlled Synthesis and Characterization of Some One-dimensional Semiconductor Nanomaterials  
Controlled Synthesis and Characterization of One-dimensional II-VI Nanomaterials  
Controlled Synthesis and Characterization of Metal Oxide Nanowires by Chemical Vapor Deposition on Silicon and Carbon Substrates  
Controlled Synthesis and Characterization of Hierarchically Structured Inorganic Materials for

Membrane Applications  
Controlled Synthesis and Characterization of Branched, Functionalized, and Cyclic Polymers  
A variety of methods were used to make polymers with different architecture and functionalities. The linking chemistry of vinyl dimethylchlorosilane (VDMCS) with poly(styryl)lithium ( $M[\text{subscript } n] = 1,700\text{--}3,000 \text{ g/mol}$ ) was studied. The average degree of branching varied from 7.5 to 9.4 with an increase in concentration of VDMCS (1.2 to 5.2 eq). The intrinsic viscosities and melt viscosities (at  $160^\circ\text{C}$ ) of the star polymers were found to be less than half of that of the corresponding linear polystyrenes.  $[\alpha]$ -Pyrrolidine-functionalized polystyrene ( $M[\text{subscript } n] = 2,700 \text{ g/mol}$ ,  $M[\text{subscript } n]$

w]/M<sub>n</sub> = 1.03, 92.5%) was successfully synthesized from [alpha]-chloromethyldimethylsilane-functionalized polystyrene (M<sub>n</sub> = 2,600 g/mol, M<sub>w</sub>/M<sub>n</sub> = 1.02) based on NMR spectroscopy, MALDI-TOF and ESI mass spectrometry. The stability of silyl hydride groups under atom transfer radical polymerization conditions was proven by copolymerizing methyl methacrylate and (4-vinylphenyl)dimethylsilane (VPDS). Tapered block copolymers of isoprene, VPDS, and styrene with narrow molecular weight distributions (1.04 and 1.05) were synthesized via anionic polymerization. Evidence regarding the topology of cyclic polybutadienes was obtained by Atomic Force Microscopy of

grafted polymers obtained by grafting an excess of silyl hydride-functionalized polystyrene (M<sub>n</sub> = 8,300 g/mol, M<sub>w</sub>/M<sub>n</sub> = 1.01) onto cyclic polybutadiene (M<sub>n</sub> = 88,000 g/mol, M<sub>w</sub>/M<sub>n</sub> = 2.0). The reactivity of polyisobutylene carbocations was compared with respect to competitive electrophilic addition to a vinyl group versus silyl hydride transfer by investigating the reaction with VPDS. Based on GPC results, and <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy, no evidence for any vinyl group addition was observed. A successful attempt was made to prepare electrospun fibers from fluoro-functionalized styrene-butadiene elastomers. The water contact angle of these surfaces was found to be 162.8°

[plus or minus] 3.8° for the fibrous mat of the fluorinated polymers as compared to 151.2° [plus or minus] 2.4° for the analogous fibrous mat of the non-fluorinated polymers. In-chain functionalization of tapered styrene butadiene rubber using chloromethyldimethylsilane was quantitatively done via a hydrosilation reaction. Pyrrolidine-functionalized styrene butadiene rubber was obtained in 71% yield after reacting pyrrolidine with chloromethyldimethylsilane-functionalized styrene butadiene rubber. In-chain, silyl hydride-functionalized, deuterated polystyrene ( $M_w/M_n = 2,100$  g/mol,  $M_w/M_n = 1.01$ ) was functionalized with allyl cyanide in the presence of Karstedt's catalyst to obtain

in-chain cyano-functionalized, deuterated polystyrene (45% based on the mass of in-chain, cyano-functionalized deuterated polystyrene obtained). Controlled Synthesis and Characterization of One Dimensional Nanomaterials Carbon Nanotubes and Titanium Oxide Nanowires The Colloidal Chemistry Synthesis and Electron Microscopy Characterization of Shape-controlled Metal and Semiconductor Nanocrystals Solution methods of materials synthesis have found application in a variety of fields due to the diversity of products accessible, facility of process scalability, and the ease of tuning their properties through prudent selection of reaction conditions. Control of experimental variables during the formation of colloidally stable

nanoscale solids within a liquid matrix allows for tailoring of the particles' characteristics, including shape, size, composition, and surface chemistry. In this dissertation, I will discuss how the manipulation of reaction chemistries can be used to synthesize shape-controlled metal and semiconductor colloidal nanocrystals. Further, I will elaborate on the mechanisms by which these particles form from molecular precursors and describe how their properties can differ from their bulk analogues through extensive characterization, especially using transmission electron microscopy. These studies contribute to the continued development of chemical routes to nanocrystals and their application as functional materials. First, I will review recent advances in the

synthesis and characterization of shape-controlled nanocrystals, as well as highlight their promising applicability in a number of emerging technologies. These principles will then be leveraged to the specific case of catalytically-active rhodium nanocrystals, which can be synthesized with morphological and dimensional control using a polyol solution-mediated strategy. I describe an innovative shape-controlled synthesis to monodisperse colloidal rhodium icosahedra, cubes, triangular plates, and octahedra using this route. Additionally, new insights into the important role of the polyol reducing solvent on the synthesis of these nanocrystals are revealed, and how these might be exploited to engender superior reaction control and novel products. Next, I will

describe how a crystallization mechanism was established for the synthesis of numerous morphologies of noble metal nanocrystals. I present a thorough analysis of the synthesis of shape-controlled rhodium nanocrystals, using extensive transmission electron microscopy characterization, and relate these findings to one of the primary synthetic levers available in the polyol synthesis: the anionic ligands present. Further, I show that the crystallization process proceeds by a nonclassical mechanism in which cluster particles serve as a stable intermediate between molecular precursors and the final product. I then apply these principles to the shape-controlled synthesis of other noble metal nanocrystals before expounding a generalized formation

mechanism in the polyol synthesis of colloidal metal nanocrystals. Finally, I will highlight my efforts in the designed synthesis and characterization of colloidal tin(II) sulfide (SnS) semiconducting "quantum dot" nanocrystals. I describe a route for the solution synthesis of monodisperse colloidal SnS nanosheets, nanocubes, and nanospherical polyhedra in high yield. Further, detailed crystallographic characterization of these nanocrystals using transmission electron microscopy indicates that their atomic structure possesses a previously-unreported nanoscale deviation from the bulk phase. Additionally, I show that their electronic and photocatalytic properties of these quantum dots are both shape-dependent and distinct from bulk SnS. Controlled

Synthesis, Characterization, Growth Mechanism of Lead Sulfide Nanocrystals  
 Controlled Synthesis and Characterization of Templated, Magneto-responsive Nanoparticle Structures (Cont.)  
 A method of electrospinning was used to encapsulate magnetic nanoparticles in a polymeric matrix to create field responsive nanofibers for various applications. The magnetization properties of the nanofibers were also characterized and their behavior under an applied magnetic field was modeled.  
 Controlled Synthesis, Molecular-level Characterization and Chemical Transformation of Hybrid Organosilazane/silylamine Pre-ceramic Telechelic Oligomers  
 Controlled Synthesis of Nanoparticles in Microheterogeneous

## Systems

A variety of methods were used to make polymers with different architecture and functionalities. The linking chemistry of vinyl dimethylchlorosilane (VDMCS) with poly(styryl)lithium ( $M_n = 1,700-3,000$  g/mol) was studied. The average degree of branching varied from 7.5 to 9.4 with an increase in concentration of VDMCS (1.2 to 5.2 eq). The intrinsic viscosities and melt viscosities (at 160°C) of the star polymers were found to be less than half of that of the corresponding linear polystyrenes.  $\alpha$ -Pyrrolidine-functionalized polystyrene ( $M_n = 2,700$  g/mol,  $M_w/M_n = 1.03$ , 92.5%) was successfully synthesized from  $\alpha$ -chloromethyl dimethylsilane-

functionalized polystyrene ( $M_n = 2,600$  g/mol,  $M_w/M_n = 1.02$ ) based on NMR spectroscopy, MALDI-TOF and ESI mass spectrometry. The stability of silyl hydride groups under atom transfer radical polymerization conditions was proven by copolymerizing methyl methacrylate and (4-vinylphenyl)dimethylsilane (VPDS). Tapered block copolymers of isoprene, VPDS, and styrene with narrow molecular weight distributions (1.04 and 1.05) were synthesized via anionic polymerization. Evidence regarding the topology of cyclic polybutadienes was obtained by Atomic Force Microscopy of grafted polymers obtained by grafting an excess of silyl hydride-functionalized polystyrene ( $M_n = 8,300$

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analogous fibrous mat of the non-fluorinated polymers. In-chain functionalization of tapered styrene butadiene rubber using chloromethyldimethylsilane was quantitatively done via a hydrosilation reaction. Pyrrolidine-functionalized styrene butadiene rubber was obtained in 71% yield after reacting pyrrolidine with chloromethyldimethylsilane-functionalized styrene butadiene rubber. In-chain, silyl hydride-functionalized, deuterated polystyrene ( $M[\text{subscript } n] = 2,100 \text{ g/mol}$ ,  $M[\text{subscript } w]/M[\text{subscript } n] = 1.01$ ) was functionalized with allyl cyanide in the presence of Karstedt's catalyst to obtain in-chain cyano-functionalized, deuterated polystyrene (45% based on the mass of in-chain, cyano-

functionalized deuterated polystyrene obtained).

*Block Copolymer Templated Nitrogen-enriched Nanocarbons* John Wiley & Sons  
A novel synthetic method of polymer brushes using polymer single crystals (PSCs) as solid-state templates is introduced in this study. PSC has a quasi-2D lamellae structure with polymer chains fold back-and-forth perpendicular to the lamellae surfaces. During crystallization, the chain ends are excluded from the unit cell onto the lamellae surfaces, which makes the material extremely versatile in its functionality. Such structure holds the unique capability to harvest nanoparticles, or being immobilized onto macroscopic flat surfaces. After dissolving PSCs in good solvent, polymer



brushes are chemically tethered on either nanoparticles or flat macroscopic surfaces. Because the chain-folding structure can be conveniently tailored by changing the molecular weight of polymer and the crystallization temperature, the thickness, grafting density and morphology of resulted polymer brushes can be precisely controlled. As a model system, poly( $\epsilon$ -caprolactone) with thiol or alkoxy silane terminal groups was used, and polymer brushes were successfully prepared on both nanoparticles and glass/Au flat surfaces. The structure-property relationships of the as-prepared polymer brushes were studied in detail using multiple characterization techniques. First of all, when functionalizing nanoparticles, by engineering the chain-

folding structure of the PSCs, interesting complex nanostructures can be formed by nanoparticles including Janus nanoparticles and nanoparticle dimers. These unique structures render hybrid nanoparticles very interesting responsive behavior which have been studied in detail in this dissertation. When grafted onto a flat surface on the other hand, not only the molecular weight and grafting density can be precisely controlled, the tethering points of a single polymer chain can also be conveniently tailored, resulting polymer brushes with either tail or loop structures. Such difference in brush structure can significantly alter the properties of functional surface. By using atomic force microscopy based force spectroscopy (AFM-FS) and macroscale

shear adhesion measurements, it is thus demonstrated that when polymer loops are grafted, the surface could exhibit much stronger adhesion compared with regular polymer tails when free-dangling polymer chains are allowed to interact with the surface, which is believed to mimic the Velcro-like behavior where polymer loops can withhold strong entanglement with free chain ends upon breaking of the physical bonding.

**Controlled Synthesis of Chalcogenide and Halide Perovskite Semiconductor Nanostructures**

American Chemical Society

Because of their structural and dynamical properties, microheterogeneous systems have been employed as solvent and reaction media both to synthesize and stabilize

nanoparticles. Following this route, inside their nanometer-sized heterogeneities the nanoparticles of many different substances have been incorporated. The book shows the distinct advantages of this synthetic strategy over that of many other methods. Moreover, it furnishes to the reader a collection of theoretical and experimental facts allowing him to reduce the number of trial and errors necessary to arrive at an optimal synthetic protocol.

**Metal Oxide Nanoparticles in**

**Organic Solvents** Springer Science & Business Media

Flow visualization using polystyrene microspheres (PSL)s has enabled researchers to learn a tremendous amount of information via particle based

diagnostic techniques. To better accommodate wind tunnel researchers needs, PSL synthesis via dispersion polymerization has been carried out at NASA Langley Research Center since the late 1980s. When utilizing seed material for flow visualization, size and size distribution are of paramount importance. Therefore, the work described here focused on further refinement of PSL synthesis and characterization. Through controlled variation of synthetic conditions (chemical concentrations, solution stirring speed, temperature, etc.) a robust, controllable procedure was developed. The relationship between particle size and salt concentration,  $MgSO_4$ , was identified enabling the determination of PSL diameters a priori.

Suggestions of future topics related to PSL synthesis, stability, and size variation are also described.

**Nanoparticle Matter** ScholarlyEditions (Cont.) A method of electrospinning was used to encapsulate magnetic nanoparticles in a polymeric matrix to create field responsive nanofibers for various applications. The magnetization properties of the nanofibers were also characterized and their behavior under an applied magnetic field was modeled. Controlled Synthesis and Characterization of Metal Oxide Nanowires by Chemical Vapor Deposition on Silicon and Carbon Substrates Walter de Gruyter GmbH & Co KG Boranes—Advances in Research and Application: 2013 Edition is a ScholarlyBrief™ that delivers timely,

authoritative, comprehensive, and specialized information about ZZZAdditional Research in a concise format. The editors have built Boranes—Advances in Research and Application: 2013 Edition on the vast information databases of ScholarlyNews.™ You can expect the information about ZZZAdditional Research in this book to be deeper than what you can access anywhere else, as well as consistently reliable, authoritative, informed, and relevant. The content of Boranes—Advances in Research and Application: 2013 Edition has been produced by the world's leading scientists, engineers, analysts, research institutions, and companies. All of the content is from peer-reviewed sources, and all of it is written,

assembled, and edited by the editors at ScholarlyEditions™ and available exclusively from us. You now have a source you can cite with authority, confidence, and credibility. More information is available at <http://www.ScholarlyEditions.com/>. comédie en un acte et en vers représentée pour la première fois, à Lyon, le 27 prairial an VIe par Mr. Amar, ... North Holland Chapter 1 provides an overview on the synthesis of graphene nanoribbons. Various types of edge structures and bottom-up synthetic strategies, on-surface and in-solution, are covered before discussing our group's third approach, solid-state. This is followed by a description of the effect of heteroatomic substitutions on electronic

properties of GNRs. Lastly a brief discussion on other semiconducting polymer structure and charge transfer properties are introduced. Chapter 2 details the synthesis of  $N = 8$  armchair graphene nanoribbons (GNRs) using a two-step solid-state method. Four diarylbutadiyne precursors undergo topochemical polymerization to four distinct polydiacetylene (PDA) polymers, which subsequently cyclodehydrogenate and undergo side chain fragmentation to afford the same  $N = 8$  armchair GNR. Various spectroscopic and imaging techniques are used to characterize this transformation, in addition to calculations of the cyclization process on a model system used to verify the mechanism. Chapter 3 describes the synthesis of GNRs with a fjord-edge

structure and site-specific nitrogen substitutions using the two-step approach above. Two dipyrindylbutadiyne precursors polymerize and cyclize to afford  $N = 8$  fjord-edge N-GNRs, with side chains still intact. Spectroscopic characterization, imaging and mechanistic calculations of a pyridyl model system verify the transformation from butadiyne to GNR. Lower the barrier of Hopf cyclizations, a step towards GNRs in our solid-state approach, through introduction of strained cycloalkenes could lead to room temperature GNR syntheses. Chapter 4 details the synthesis of two polydiacetylene synthons containing norbornadiene, a bis(norbornadienyl)1,3-butadiyne and trans-bis(norbornadienyl)enediyne. Challenges

towards synthesizing both monomer units and future applications of other trans-enediynes towards GNRs are discussed. Chapter 5 describes the synthesis of an amphiphilic semiconducting polymer, poly(cyclopentadithiophene-alt-thiophene) (PCT), according to a set of design rules aimed at straightening the polymer backbone in order to reduce polymer disorder and increase conductivity. The design rules are 1) hydrophobic polymer backbone and hydrophilic side chains, 2) alternating copolymer such that all the side chains reside on one side, 3) side chains branched off an  $sp^3$  carbon to create a 3D wedge shape, and 4) complementary bond angles between monomer units to achieve a 180 dihedral angle. The

solution phase of the polymer is characterized by small angle X-ray scattering (SAXS) and imaged using cryo-transition electron microscopy (TEM). Applications of PCT towards controlling electron donor-acceptor complexes are explored in Chapter 6. PCT and poly(fluorene-alt-thiophene) (PFT), are complexed with two electron acceptors, a charged perylenediimide and a series of charged bis-pyrrolidium functionalized fullerenes. The structure of these co-assemblies are characterized by small angle X-ray scattering and photoluminescence quenching, concluding that complementary geometries between the polymer micelle and acceptor shapes result in increased amounts of photoluminescence quenching.

**Boranes—Advances in Research and Application: 2013 Edition** BiblioGov

Semiconductor nanomaterials have become an important class of materials with great potential for applications ranging from catalytic to electronic and optoelectronic devices. For next generation catalytic, optoelectronic, and photonic applications, the synthesis of high-quality nanomaterials with uniform size, well-defined morphology, composition, and surface chemistry is of key importance, because the electrical, optical, and magnetic properties of these nanomaterials are strongly dependent on those parameters. Besides technical interests, access to defined nanoscale structures is also essential for uncovering their intrinsic properties unaffected by sample heterogeneity.

Rigorous understanding of the properties of individual nanocrystals will enable us to exploit them, making it possible to better design and build novel electronic, magnetic, and photonic devices and other functional materials based on these nanostructures. This dissertation explores both direct synthetic methods and post transformation approaches for rational synthesis of new nanomaterial systems, which are potential candidates for applications in areas of photovoltaics, non-noble-metal plasmonics, light emitting diodes, etc. And their structural, optical, and electrical properties have been investigated in detail. Chapter 1 provides an introduction to the current progress and common strategies used in rational control of the size, shape, composition, and surface chemistry of

nanomaterials. Chapter 2 examines the Cu<sup>+</sup> cation-exchange mechanisms in CdS nanowires. A detailed transformation diagram of cation-exchange chemistry from CdS to Cu<sub>2-x</sub>S nanowires is reported. By varying the reaction time and the reactants' concentration ratio, the progression of the cation-exchange process was captured, and tunable crystal phases of the Cu<sub>2-x</sub>S are achieved. The overall process occurs in three stages: formation of discontinuous Cu<sub>2-x</sub>S islands, formation of core-shell CdS-Cu<sub>2-x</sub>S heterostructures, and complete conversion to Cu<sub>2-x</sub>S nanowires with controllable crystal phases. Detailed structural characterization reveal that the resultant Cu<sub>2-x</sub>S phases become more stoichiometric with increasing

reaction time and copper precursor concentration. This experimental result suggests a kinetically controlled process limited by diffusion. In Chapter 3, a catalyst-free, solution-phase approach has been developed to obtain single crystalline, orthorhombic CsPbX<sub>3</sub> NWs with uniform growth direction. The morphological evolution of the CsPbBr<sub>3</sub> nanostructures along the reaction has been investigated, and the reaction protocol has been optimized to achieve a high yield of monodispersed nanowires likely due to a soft template mechanism. The direct synthesized CsPbI<sub>3</sub> NWs show a room-temperature stable double-chain phase, with weak photoluminescence mainly from the trap states. Anion-exchange reaction by using the monodispersed CsPbBr<sub>3</sub> NWs as



templates can retain the favorable corner-sharing orthorhombic phase, and independently control the NW compositions, thus access to a wide range of compositions with bright and tunable photoluminescence spanning over nearly the entire visible spectrum. Meanwhile, surface treatment with the original precursors was performed to effectively passivate the surface states, and improve the quantum yield to over 10 times. In Chapter 4, a stepwise purification method has been developed to purify the ultrathin CsPbX<sub>3</sub> NWs with a uniform diameter of  $2.2 \pm 0.2$  nm. The structural and optical properties have been discussed. Aberration-corrected high-resolution TEM shows the NWs are single crystalline, absorption and fluorescence spectrum shows that those

NWs possess strong two-dimensional quantum confinement effects along with bright emission. The band gap of these ultrathin NWs can be tuned by anion-exchange reaction.

*Nanomaterials for 2D and 3D Printing*

Springer Science & Business Media

Our society depends heavily on metals.

They are ubiquitous construction materials, critical interconnects in integrated circuits, common coinage materials, and more. Excitingly, new uses for metals are emerging with the advent of nanoscience, as metal crystals with nanoscale dimensions can display new and tunable properties. The optical and photothermal properties of metal nanocrystals have led to cancer diagnosis and treatment platforms now in clinical trials, while, at the same time,

the ability to tune the surface features of metal nanocrystals are giving rise to designer catalysts that enable more sustainable use of precious resources. These are just two examples of how metal nanocrystals are addressing important social needs. Readers will have: Varied levels of familiarity with the topic of metal nanocrystals A background in chemistry, physics, biology, any number of engineering fields, or even an interdisciplinary framework. Considering this diversity of familiarity and backgrounds, as authors we put high emphasis on structure-property correlation and the emergent applications that arise from such fundamental understanding. We were inspired to contribute this book in response to the common refrain from

students that this topic or research area “looks so cool” or “seems exciting” but is quickly followed up with hesitations about whether or not they are capable of research in the field because they “lack the appropriate background”.

#### *Characterization of PmHS2*

*Glycosyltransferases for the Controlled Synthesis of Heparosan* Springer Nature Nanocatalysis, a subdiscipline of nanoscience, seeks to control chemical reactions by changing the size, dimensionality, chemical composition, and morphology of the reaction center and by changing the kinetics using nanopatterning of the reaction center. This book offers a detailed pedagogical and methodological overview of the field. Readers discover many examples of current research, helping them

explore new and emerging applications.  
Design, Synthesis and Characterization  
of Metal Oxide Adsorbents and Catalysts  
for Environmental Applications

Controlled Synthesis and  
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Controlled Synthesis and  
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Characterization of Hierarchically  
Structured Inorganic Materials for  
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Synthesis and Characterization of

Branched, Functionalized, and Cyclic  
Polymers

*Carbon Nanotubes and Titanium Oxide  
Nanowires*

Systematically summarizes the current  
status and recent advances in bimetallic  
structures, their shape-controlled  
synthesis, properties, and applications  
Intensive researches are currently being  
carried out on bimetallic nanostructures,  
focusing on a number of fundamental,  
physical, and chemical questions  
regarding their synthesis and properties.  
This book presents a systematic and  
comprehensive summary of the current  
status and recent advances in this field,  
supporting readers in the synthesis of  
model bimetallic nanoparticles, and the  
exploration and interpretation of their  
properties. Bimetallic Nanostructures:

Shape-Controlled Synthesis for Catalysis, Plasmonics and Sensing Applications is divided into three parts. Part 1 introduces basic chemical and physical knowledge of bimetallic structures, including fundamentals, computational models, and in situ characterization techniques. Part 2 summarizes recent developments in synthetic methods, characterization, and properties of bimetallic structures from the perspective of morphology effect, including zero-dimensional nanomaterials, one-dimensional nanomaterials, and two-dimensional nanomaterials. Part 3 discusses applications in electrocatalysis, heterogeneous catalysis, plasmonics and sensing. Comprehensive reference for an important multidisciplinary research field

Thoroughly summarizes the present state and latest developments in bimetallic structures Helps researchers find optimal synthetic methods and explore new phenomena in surface science and synthetic chemistry of bimetallic nanostructures Bimetallic Nanostructures: Shape-Controlled Synthesis for Catalysis, Plasmonics and Sensing Applications is an excellent source or reference for researchers and advanced students. Academic researchers in nanoscience, nanocatalysis, and surface plasmonics, and those working in industry in areas involving nanotechnology, catalysis and optoelectronics, will find this book of interest.

**Controlled Synthesis and Characterization of One Dimensional**

## Nanomaterials

Nanomaterials have attracted significant interest in the past decade due to their unique structure and properties compared to their bulk counterparts. Nanomaterials-based solutions can address challenges in various technologies such as proton exchange membrane fuel cells (PEMFCs). PEMFC is an innovative energy conversion technology to directly convert chemical energy to electrical energy by using hydrogen as fuel. However, the current PEMFC system still faces significant technological roadblocks which have to be overcome before the system can become economically viable. A major impediment to the commercialization of PEMFC is the high cost of materials and manufacturing and stability, which is

primarily associated with the cost of Pt catalysts and their support in membrane electrode assembly (MEA). One approach in addressing these issues is the controlled synthesis and application of nanostructured Pt-based catalysts and their support in PEMFCs. The objective of this thesis is to synthesize and characterize various nanostructures (e.g. metal oxides and metal silicides or composites) and evaluate their performance as Pt supports used in the PEMFCs. Various advanced characterization techniques such as high resolution scanning and transmission electron microscopy, X-ray absorption spectroscopy and electrochemical characterization methods have been used to understand growth mechanism of obtained nanostructures and their

roles in PEMFCs. We also reported the synthesis of  $\text{WSi}_2$  and  $\text{Ta}_5\text{Si}_3$  heterostructures using a low pressure chemical vapor deposition (LPCVD) method. The morphologies of these nanostructures were found to be sensitive to the concentration of reactive species and silica vapor in the CVD chamber. The results indicated that the morphology of  $\text{WSi}_2$  and  $\text{Ta}_5\text{Si}_3$  nanostructures varied from nanowires, networked nanoribbons to nanosheets with the control of the oxygen concentration. A vapor solid growth mechanism based on silica sheath formation was proposed for the synthesis of these nanostructures. To take advantage of unique properties of carbon nanotubes, metal oxide and metal silicides as catalyst support, a new

method was developed for the synthesis of composite nanostructures.  $\text{TiSi}_2\text{O}_x$ -NCNTs and  $\text{TiO}_2$ -NCNTs nanocomposites were synthesized using a combination of CVD process and magnetron sputtering and their performance as catalyst supports in PEMFCs were studied. Pt nanoparticles deposited on these nanostructures showed enhanced catalytic activity compared to commercial Pt/carbon electrodes. The electronic structure of Pt on the catalyst supports was investigated using X-ray absorption spectroscopy, to obtain insight into the interaction between the catalyst supports and Pt nanoparticles. As an example of well controlled synthesis of nanostructures, one-dimensional tungsten oxide nanostructures (W18O49) have been

synthesized using a conventional chemical vapor deposition method (CVD). The morphology of the nanostructures such as diameter and length, were controlled during the synthesis process via sulfur doping. The dependence of morphology, composition and structure of tungsten oxides on the sulfur flow rate has been studied. Further, one step synthesis of tungsten sulfide/tungsten oxide nanocables (WS<sub>2</sub>/W<sub>18</sub>O<sub>49</sub>) have been achieved for the first time using tungsten and sulfur powder as the starting materials. In summary, the research work presented in this thesis aims at contributing to the development of various novel nanostructured catalyst supports and probing the correlation between synthesis approach, fine structure, and

catalytic performance of the nanostructures as well as exploring their potential applications in highly active electrocatalysts for PEMFCs.

The Colloidal Chemistry Synthesis and Electron Microscopy Characterization of Shape-controlled Metal and Semiconductor Nanocrystals

Fourth, a size-controlled synthesis of water soluble DPPH (1,1-diphenyl-2-picrylhydrazyl) nanoparticles has been developed. Importantly, these nanoparticles exhibit size-dependent absorption spectra and fast-exchange-narrowed single-line EPR spectra with linewidths of ~1.5- 1.8 G. Furthermore, the EPR linewidth can be controlled by partially reducing the DPPH radical. These water-soluble DPPH nanoparticles are a perfect standard EPR labels for

biological and biomedical systems.

### **Shape-Controlled Synthesis for Catalysis, Plasmonics, and Sensing Applications**

Extracting multifunctional benefits by combining multiple nano-scale materials has driven materials science to develop nano-heterostructures, which are known as nanohybrids (NHs). Many such composite materials have been researched for applications in the energy sector and in biomedical devices and processes. Among these NHs, carbon nanotubes combined with metal oxides (MOs) are one of the most studied materials that provide unique advantages as electrocatalyst supports, and are currently being commercialized as embedded electrodes for fuels cells. NHs are not only a new class of complex

materials but also brings in novel physicochemical properties that most likely cannot be captured by the sum of the properties of their components materials. Thus, understanding the environmental health and safety (EHS) of this new class of composite NHs is imperative. The first challenge that the nano-EHS community faces is to synthesize these materials with a range of MO loadings or composition under a controlled and comparable set of experimental conditions. In this dissertation, a set of carbonaceous-metal oxide NHs have been synthesized and characterized under comparable synthesis conditions. After synthesis, the underlying mechanisms of metal oxide formation on multiwalled carbon nanotubes (MWNT) surfaces has been



enumerated, and finally, aggregation behavior of a select NH and its components has been assessed as a function of the metal oxide loading. A modified sol-gel technique has been developed to grow TiO<sub>2</sub>, ZnO, Er<sub>2</sub>O<sub>3</sub>, and Pr<sub>6</sub>O<sub>11</sub> nanocrystals on MWNT surfaces. The novelty of this technique is that, by varying reagent composition, metal oxide content on the MWNT surfaces can be controlled, keeping all other parameters unchanged. The modified synthesis protocol has been successfully developed to produce a relatively large amount of NHs (100s of mg per batch of synthesis), adequate for systematic nano EHS studies. Following detailed characterization of the materials, underlying hybridization and MO crystal formation mechanism(s) have

been enumerated. Furthermore, standard electron potential of the metal species (while considering electron transfer between their oxidized state to zero valent form) has been found to be the controlling factor for the formation of metal or metal oxide crystals from the precursors on MWNT surfaces, using the sol-gel synthesis technique. Self-aggregation, one of the dominant environmental processes that particles undergo upon release into aquatic environment, has been assessed for one of the most used and commercialized NHs MWNT-TiO<sub>2</sub> and its components. This study investigated the role of TiO<sub>2</sub> loading on the aggregation behavior, MWNT-TiO<sub>2</sub> NH with three different TiO<sub>2</sub> loadings. Results suggested that TiO<sub>2</sub> loading on MWNT surfaces control

aggregation behavior of the composite NHs. NHs with all TiO<sub>2</sub> loading demonstrated strong dependence on electrokinetics. Deoxygenation of the NHs with decreased TiO<sub>2</sub> loading due to the NH synthesis process appeared to be a key contributor on the electrokinetics of the NHs. The van der Waals interaction forces of the NHs decreased with decrease in TiO<sub>2</sub> loading. This study also concluded that classical DLVO theory may be inadequate to capture the aggregation behavior of the NHs. The controlled synthesis technique developed during this research, as well as the mechanisms of metal vs. metal oxide formation identified will be valuable to prepare a large set of NHs for nano-EHS studies. Aggregation behavior of the composites can be very

complex in nature and cannot be predicted from the sum of the behavior of their component materials. The deviation of DLVO prediction from the experimental aggregation data calls for further investigation on direct measurement of other complex surface properties of the NHs upon hybridization such as surface roughness and surface charge heterogeneity

### **Controlled Synthesis,**

### **Characterization and Applications**

Metal Oxide Nanoparticles in Organic Solvents discusses recent advances in the chemistry involved for the controlled synthesis and assembly of metal oxide nanoparticles, the characterizations required by such nanoobjects, and their size and shape depending properties. In the last few years, a valuable alternative

to the well-known aqueous sol-gel processes was developed in the form of nonaqueous solution routes. Metal Oxide Nanoparticles in Organic Solvents reviews and compares surfactant- and solvent-controlled routes, as well as providing an overview of techniques for the characterization of metal oxide nanoparticles, crystallization pathways, the physical properties of metal oxide nanoparticles, their applications in diverse fields of technology, and their assembly into larger nano- and mesostructures. Researchers and postgraduates in the fields of nanomaterials and sol-gel chemistry will appreciate this book's informative approach to chemical formation mechanisms in relation to metal oxides. *Controlled Synthesis, Characterization,*

*and Applications of Advanced Inorganic Nanomaterials*

This volume on iron-sulfur proteins includes chapters that describe the initial discovery of iron-sulfur proteins in the 1960s to elucidation of the roles of iron sulfur clusters as prosthetic groups of enzymes, such as the citric acid cycle enzyme, aconitase, and numerous other proteins, ranging from nitrogenase to DNA repair proteins. The capacity of iron sulfur clusters to accept and delocalize single electrons is explained by basic chemical principles, which illustrate why iron sulfur proteins are uniquely suitable for electron transport and other activities. Techniques used for detection and stabilization of iron-sulfur clusters, including EPR and Mossbauer spectroscopies, are discussed because

they are important for characterizing unrecognized and elusive iron sulfur proteins. Recent insights into how nitrogenase works have arisen from multiple advances, described here, including studies of high-resolution crystal structures.

#### Controlled Synthesis of Nanoparticles in Microheterogeneous Systems

In this thesis, I will focus on the design and synthesis of metal oxides as adsorbents and catalysts for different types of environmental applications, such as water remediation, biogas cleanup. Controlled synthesis of these materials with unique crystalline structures, physical, and chemical properties will be carried out to achieve an improved performance. Correlations between the material property and performance will

be investigated by varieties of characterization methods. In the first part, I will employ perovskite materials for catalytic wet air oxidation (CWAO) reactions for water remediation.  $\text{LaNiO}_3$ - $\delta$  (LNO) was applied for degradation of methyl orange (MO) azo dye in aqueous solutions under dark ambient conditions (room temperature, atmospheric pressure) without additional lights or chemical stimulants. The mechanism behind MO degradation by LNO under dark ambient conditions was unraveled by a series of characterization methods. Considering the large variety of perovskites in terms of constituents and composition, an excellent perovskite material should be tailorable for water remediation applications. Fuel cell performance for the double perovskite

material PrBaCo<sub>2</sub>O<sub>5</sub> (PBC) was briefly shown. In the second part, I will demonstrate a facile way for synthesizing mesoporous aluminas (MAs) with uniform and monomodal pores via a modified inverse micelle synthesis method. The effects of reaction times, surfactant chain lengths, and heat treatments on the textural properties of MA were adjusted to optimize the texture properties for biogas cleanup. The tuned MA of the large mesopore volume achieved high octamethylcyclotetrasiloxane (D4 siloxane) adsorption capacity, and maintained approximate 85% of its original adsorption capacity, demonstrating a sustainable adsorption

performance and high potential for related industrial applications. Arsenic adsorption was performed to illustrate the application of MA for heavy metal removal. The third part, I supported transition metals on optimized mesoporous alumina for methane oxidation. The texture properties were summarized and Temperature-programmed studies were used for understanding the mechanism for methane partial oxidation. Different ratios of copper supported on alumina were designed for methane combustion and exhibited improved performance with regard to the loading amount, which was explained by further characterization methods.

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