Controlled Synthesis And Characterization Of Nobel Metal

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דו"ח על הביקורת במועצה המקומית מזכרת בתיה
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Nanocatalysis

KADE GONZALES

Characterization, Properties and Applications

ScholarlyEditions

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 dipyridylbutadiyne 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 co-polymer such that all the side chains reside on one side, 3) side chains branched off an sp3 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.

Doped and Undoped Nanoparticles Walter de Gruyter GmbH & Co KG In this thesis, I will focus on the synthesis of transition metal oxide/sulfide-based composite materials for different types of environmental and sustainable energy applications under ambient conditions. Controlled synthesis of these catalysts with unique crystalline structures, physical, and chemical properties will be carried out to achieve an improved catalytic activity. The correlations between the material structure and catalytic activity will be investigated by various characterization techniques. Finally, the catalytic activities for the resulting materials will be evaluated for environmental friendly photocatalytic dye degradation and electrochemical water splitting reaction, respectively.

ScholarlyBrief Springer Science & Business Media (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 of Nanoparticles in Microheterogeneous Systems Springer Science & Business Media Controlled Synthesis and Characterization of Silicon NanocrystalsControlled Synthesis and Characterization of Some One-dimensional Semiconductor NanomaterialsControlled Synthesis and Characterization of One-dimensional II-VI NanomaterialsControlled Synthesis and Characterization of Metal Oxide Nanowires by Chemical Vapor Deposition on Silicon and Carbon SubstratesControlled Synthesis and Characterization of Hierarchically Structured

Inorganic Materials for Membrane ApplicationsControlled Synthesis and Characterization of Branched, Functionalized, and Cyclic Polymers Handbook on Synthesis Strategies for Advanced Materials Controlled Synthesis and Characterization of Silicon NanocrystalsControlled Synthesis and Characterization of Some One-dimensional Semiconductor NanomaterialsControlled Synthesis and Characterization of One-dimensional II-VI NanomaterialsControlled Synthesis and Characterization of Metal Oxide Nanowires by Chemical Vapor Deposition on Silicon and Carbon SubstratesControlled Synthesis and Characterization of Hierarchically Structured Inorganic Materials for Membrane ApplicationsControlled Synthesis and Characterization of Branched, Functionalized, and Cyclic PolymersA variety of methods were used to make polymers with different architecture and functionalities. The linking chemistry of vinyldimethylchlorosilane (VDMCS) with poly(styryl)lithium (M[subscript 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[subscript n] = 2,700 g/mol, M[subscript w]/M[subscript n] = 1.03, 92.5%) was successfully synthesized from [alpha]-chloromethyldimethylsilane-functionalized polystyrene (M[subscript n] = 2,600 g/mol, M[subscript w]/M[subscript 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[subscript n] = 8,300 g/mol, M[subscript w]/M[subscript n] =1.01) onto cyclic polybutadiene (M[subscript n]=88,000 g/mol, M[subscript w]/M[subscript n] = 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 1H and 13C 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 chloromethydimethylsilane 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[subscript n] = 2,100 g/mol, M[subscript w]/M[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).Controlled Synthesis and Characterization of One Dimensional NanomaterialsCarbon Nanotubes and Titanium Oxide NanowiresThe Colloidal Chemistry Synthesis and Electron Microscopy Characterization of Shape-controlled Metal and Semiconductor NanocrystalsSolution 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 NanocrystalsControlled 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 Preceramic Telechelic OligomersControlled Synthesis of Nanoparticles in Microheterogeneous Systems
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 indispensible 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 nanoparicles 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.

Metal Nanocrystals North Holland

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.

Synthesis, Characterization and Manipulation of Carbon Nanotubes BiblioGov

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/.

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

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.
Synthesis and Characterization John Wiley & Sons
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 WSi2 and Ta5Si3 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 WSi2 and Ta5Si3 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. TiSi2Ox-NCNTs and TiO2-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 ($WS_2/W_{18}O_{49}$) 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.

Characterization of PmHS2 Glycosyltransferases for the Controlled Synthesis of Heparosan American Chemical Society 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. Springer 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 CO2 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, Characterization, Control, and Application of YSZ, CZTS, and ZrN Springer Nature

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 and Characterization of Silicon Nanocrystals

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-qualify 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+ cation-exchange mechanisms in CdS nanowires. A detailed transformation diagram of cation-exchange chemistry from CdS to Cu2-xS 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 Cu2-xS are achieved. The overall process occurs in three stages: formation of discontinuous Cu2-xS islands, formation of core-shell CdS-Cu2-xS heterostructures, and complete conversion to Cu2-xS nanowires with controllable crystal phases. Detailed structural characterization reveal that the resultant Cu2-xS 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 CsPbX3 NWs with uniform growth direction. The morphological evolution of the CsPbBr3 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 CsPbI3 NWs show a room-temperature stable double-chain phase, with weak photoluminescence mainly from the trap states. Anion-exchange reaction by using the monodispersed CsPbBr3 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 CsPbX3 NWs with a uniform diameter of 2.2±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.

Bimetallic Nanostructures

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.

Controlled Synthesis of Chalcogenide and Halide Perovskite Semiconductor Nanostructures

As a material is reduced down to sub-100 nm dimensions, its interaction with light, with heat, and with other matter changes due in part to increased confinement of free charges and to an increased surface area relative to volume. In practice, different materials and their characteristics can be tuned to control bulk-system properties like optical transparency, free charge generation, electric field enhancement, and localized thermal enhancement. In this dissertation, I will discuss the controlled synthesis and characterization of three different nanoparticle material systems: yttria-stabilized zirconia (YSZ), copper-zinc-tin-sulfide (CZTS), and zirconium nitride (ZrN). I will additionally discuss the viability of using the produced materials in proposed applications, namely: YSZ as the basis material for transparent sintered ceramic disks for use as cranial implants; CZTS as the basis material for earth-abundant, inexpensive, polycrystalline thin film photovoltaics; and ZrN as a visible spectrum plasmonic absorbing material for use in light-induced localized field enhancement applications.

Nanoparticle Matter

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 and Characterization of One Dimensional Nanomaterials

A variety of methods were used to make polymers with different architecture and functionalities. The linking chemistry of vinyldimethylchlorosilane (VDMCS) with poly(styryl)lithium (M[subscript 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[subscript n] = 2,700 g/mol, M[subscript w]/M[subscript n] = 1.03, 92.5%) was successfully synthesized from [alpha]-chloromethyldimethylsilane-functionalized polystyrene (M[subscript n] = 2,600 g/mol, M[subscript w]/M[subscript 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[subscript n] = 8,300 g/mol, M[subscript w]/M[subscript n] =1.01) onto cyclic polybutadiene (M[subscript n]=88,000 g/mol, M[subscript w]/M[subscript n] = 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 1H and 13C 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 chloromethydimethylsilane 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[subscript n] = 2,100 g/mol, M[subscript w]/M[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).

Current Topics in Materials Science

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”.

Nanomaterials for 2D and 3D Printing

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(Îæ-caprolactone) with thiol or alkoxysilane 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.

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

The first book to paint a complete picture of the challenges of processing functional nanomaterials for printed electronics devices, and additive manufacturing fabrication processes. Following an introduction to printed electronics, the book focuses on various functional nanomaterials available, including conducting, semi-conducting, dielectric, polymeric, ceramic and tailored nanomaterials. Subsequent sections cover the preparation and characterization of such materials along with their formulation and preparation as inkjet inks, as well as a selection of applications. These include printed interconnects, passive and active modules, as well as such high-tech devices as solar cells, transparent electrodes, displays, touch screens, sensors, RFID tags and 3D objects. The book concludes with a look at the future for printed nanomaterials. For all those working in the field of printed electronics, from entrants to specialized researchers, in a number of disciplines ranging from chemistry and materials science to engineering and manufacturing, in both academia and industry.

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