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RPPR Final Report
as of 04-Jan-2022

Agency Code: 21XD

Proposal Number: 72489MSREP

Agreement Number: W911NF-18-1-0439

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Report Date: 29-Dec-2021

Date Received: 29-Dec-2021

Final Report for Period Beginning 30-Sep-2018 and Ending 29-Sep-2021

Title: Hybrid Plasmonics at UTSA: Investigating Plasmonic/Magnetic and Plasmonic/Biomolecular Systems

Begin Performance Period: 30-Sep-2018

End Performance Period: 29-Sep-2021

Report Term: 0-Other

Submitted By: Kathryn Mayer

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STEM Degrees:

STEM Participants: 9

Major Goals: The major goals of the project, as described in the original proposal, are to investigate hybrid plasmonic materials and to provide graduate student research training in this cutting-edge area. We have organized our research into two thrusts: Thrust 1, which is focused on plasmonic/magnetic hybrid materials, and

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Thrust 2, which is focused on plasmonic/biomolecular hybrid materials. Within each of these thrusts, our work includes synthesis/fabrication of the materials, characterization of the materials (including optical and magnetic properties), electron microscopy analysis of the materials, and computer simulation of the materials. In the realm of graduate student training, our work is focused on mentoring, professional development, and building peer cohorts.

Accomplishments: Please see attached PDF.

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Training Opportunities: Graduate Student Training:

One of the major goals of this project is to provide research training and professional development for graduate students. Within this goal, we are especially focused on creating a training program which promotes the retention and success of minority students. Our strategy to support the graduate students on the Hybrid Plasmonics project includes (1) Financial support in the form of a full tuition scholarship plus 12-month salary and health insurance stipend, (2) Mentoring from co-advisors, colleagues, and peers within their cohort, and (3) Professional development activities.

During the current reporting period, we supported a total of six Ph.D. students on the project: Joel Rigor (half-year), Emil Penafiel (half-year), Priscilla Lopez, Arturo Galindo, Rakibul Shohan, and Omar Castillo. One of the Ph.D. students, Priscilla Lopez, also completed a DOD Repperger Internship with AFRL during Summer 2021 with mentor Dr. Michael Denton.

Summer Apprenticeships:

During Summer 2021, we were awarded an AEOP Apprenticeship Program (formerly HSAP/URAP) through ARO. The undergraduate apprentices were Victoria Perez (UTSA), Richard Avila (UTSA), and William Hughes (UTSA), and the high school apprentices were Ethan Yu (Brandeis High School), Flida Etchouekang (John Horn HS), and Chelan McNear (Liberty High School). Victoria and Ethan were returning students from the previous summer's apprenticeship program. Due to COVID-19, high school students were not permitted to work in labs on campus, so the high school apprenticeships were offered virtually. The undergraduate apprentices worked in labs on campus. Program meetings were held virtually using Zoom. The apprentices were assigned in pairs (one high school student and one undergraduate student) to work on research projects with Drs. Mayer, Nash, and Brancalion. One pair investigated the effects of pulsed laser exposure on chitosan gold nanoparticle synthesis, another pair carried out docking simulations for protoporphyrin IX binding to beta-lactoglobulin, and the third pair continued last year's project on a MATLAB program to predict the optical properties of gold nanorods. In all cases, the work produced by the apprentices will be utilized and/or carried forward by the team. In addition, Dr. Ponce led a hybrid two-day electron microscopy workshop for the apprentices, in which all of the students attended online training on advanced electron microscopy, and the undergraduate apprentices were able to complete a hands-on module at the Kleberg Advanced Microscopy Center at UTSA.

Professional Development:

We conducted a summer series of professional development meetings covering the following topics:

- June 18 – Reading and analyzing journal articles
- June 25 – Literature searching, identifying reputable sources
- July 2 – EndNote tutorial
- July 9 – Tips for scientific presentations
- July 16 – Writing an abstract
- July 23 – Abstract workshop (peer critique)
- July 30 – Practice presentations

We also conducted a team retreat in August 2021, in which each of the Ph.D. students and apprentices gave a research presentation to the entire team.

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Results Dissemination: Results from our project have been disseminated through several publications, as listed below. Project-supported personnel are indicated with an asterisk (*).

Papers:

Abdul-Moqueet, M.M., Tovas, L., Lopez, P.* and Mayer, K.M.*, 2021. Synthesis and bioconjugation of alkanethiol-stabilized gold bipyramid nanoparticles. *Nanotechnology*, 32(22), p.225601.

Leandro Londoño-Calderón, C., Londoño-Calderón, A., Moscoso-Londoño, O., Galindo, A.*, Ponce, A.*, Gabriela Pampillo, L., Martínez-García, R., José Yacamán, M. and Knobel, M., Magnetic Vortex Domain Wall Observation on Polycrystalline Imperfect Iron/Cobalt Alloy Nanowires Growing on 1050 Aluminum. *physica status solidi (a)*.

García-Rosas, C.M., Medina, L.A., Lopez, P.*, Large, N.* and Reyes-Coronado, A., 2021. Magneto-plasmonic biocompatible nanorice. *Journal of Nanoparticle Research*, 23(7), pp.1-13.

Elam, D., Ortega, E., Nemashkalo, A., Strzhemechny, Y., Ayon, A., Ponce, A.* and Chabanov, A.A., 2021. Low-defect-density ZnO homoepitaxial films grown by low-temperature ALD. *Applied Physics Letters*, 119(14), p. 142101.

Gordillo-Galeano, A., Ponce, A.* and Mora-Huertas, C.E., 2021. Surface structural characteristics of some colloidal lipid systems used in pharmaceuticals. *Journal of Drug Delivery Science and Technology*, 62, p.102345.

Ponce, A.*, Aguilar, J.A., Tate, J. and Yacamán, M.J., 2021. Advances in the electron diffraction characterization of atomic clusters and nanoparticles. *Nanoscale Advances*, 3(2), pp.311-325.

Conference Proceedings:

Galindo, A.* and Ponce, A.*, 2021. In-Situ Magnetization Reversal Mechanism in Ni Nanowires Investigated by Electron Holography. *Microscopy and Microanalysis*, 27(S1), pp.330-332.

Galindo, A.*, Reyes, J.L., and Ponce, A.*, 2020. Vertical Stacking of Electrodeposited Nanorods with Controlled Dimensions and Chemical Composition. *Texas Journal of Microscopy*, 51(1), p.18.

Rigor, J.* and Large, N.*, 2021. Computational Characterization of Magneto-Plasmonic Nanowires. *Bulletin of the American Physical Society*. 66(1), M71.00319.

Elam, D., Ortega, E., Chabanov, A. and Ponce, A.*, 2021. Structural defects in ZnO thin films grown by atomic layer deposition at low temperatures. *Microscopy and Microanalysis*, 27(S1), pp.2660-2662.

Book Chapters:

Ponce, A.*, Reyes-Rodríguez, J.L., Ortega, E., Parajuli, P., Hoque, M.M. and Gazder, A.A., 2020. Large Dataset Electron Diffraction Patterns for the Structural Analysis of Metallic Nanostructures. In *Scanning Transmission Electron Microscopy* (pp. 111-146). CRC Press.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI

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Participant: Kathryn Mayer

Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co PD/PI

Participant: Lorenzo Brancaleon

Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co PD/PI

Participant: Nicolas Large

Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co PD/PI

Participant: Kelly Nash

Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Co PD/PI

Participant: Arturo Ponce

Person Months Worked: 1.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Graduate Student (research assistant)

Participant: Omar Castillo

Person Months Worked: 12.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Graduate Student (research assistant)

Participant: Arturo Galindo

Person Months Worked: 12.00

Project Contribution:

National Academy Member: N

Funding Support:

Participant Type: Graduate Student (research assistant)

Participant: Rakibul Shohan

Person Months Worked: 11.00

Funding Support:

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as of 04-Jan-2022

Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Priscilla Lopez
Person Months Worked: 9.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Joel Rigor
Person Months Worked: 9.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Emil Penafiel
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Undergraduate Student
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Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Undergraduate Student
Participant: Richard Avila
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Undergraduate Student
Participant: William Hughes
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: High School Student
Participant: Ethan Yu
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
National Academy Member: N

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Participant Type: High School Student
Participant: Flida Etchouekang
Person Months Worked: 2.00
Project Contribution:
National Academy Member: N

Funding Support:

Participant Type: High School Student
Participant: Chelan McNear
Person Months Worked: 2.00
Project Contribution:
National Academy Member: N

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ARTICLES:

Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published

Journal: The Journal of Physical Chemistry C

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Publication Identifier: 10.1021/acs.jpcc.9b12002

Volume:

Issue:

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Date Submitted: 1/31/20 12:00AM

Date Published:

Publication Location:

Article Title: Direct experimental evidence of hot-carrier-driven chemical processes in tip-enhanced Raman spectroscopy (TERS)

Authors: Rui Wang, Jingbai Li, Joel Rigor, Nicolas Large, Andrey Yu Rogachev, Dmitry Kurovski

Keywords: TERS; photothermal plasmonic heating; hot electrons; DFT; FDTD-FEM

Abstract: Nanoscale localization of electromagnetic fields by metallic nanostructures can catalyze chemical reactions of molecules located in the close vicinity of the metal surface. This strong confinement of the local electric field is also the undelaying mechanism for the ångström-scale spatial resolution of TERS. The question to ask is whether chemical transformations can occur upon TER imaging. To answer this question, we investigate vibrational properties of a 4NBT monolayer on a gold surface. We observed stochastic formation of 4-nitrobenzenethiolate upon TER imaging of the 4NBT monolayer by the Au tip. DFT, FDTD, and FEM calculations confirm that this chemical reaction could not occur upon thermal desorption of the molecule, which would require 2100 K in the tip-sample junction. These experimental and theoretical pieces of evidence prove that chemical transformations indeed take place in TERS and are not driven by plasmonic photothermal heating but rather by plasmon-induced hot-carriers.

Distribution Statement: 2-Distribution Limited to U.S. Government agencies only; report contains proprietary info
Acknowledged Federal Support: Y

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Journal: The Journal of Physical Chemistry C

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Publication Identifier: 10.1021/acs.jpcc.9b02901

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Date Submitted: 9/8/19 12:00AM

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Publication Location:

Article Title: Structural Analysis of Ligand-Protected Smaller Metallic Nanocrystals by Atomic Pair Distribution Function under Precession Electron Diffraction

Authors: M. Mozammel Hoque, Sandra Vergara, Partha P. Das, Daniel Ugarte, Ulises Santiago, Chanaka Kumar

Keywords: electron microscopy, electron diffraction, gold nanoparticles

Abstract: Atomic pair distribution function (PDF) analysis has been widely used to investigate nanocrystalline and structurally disordered materials. Experimental PDFs retrieved from electron diffraction (ePDF) in transmission electron microscopy (TEM) represent an attractive alternative to traditional PDF obtained from synchrotron X-ray sources, particularly for studying minute samples. Nonetheless, the inelastic scattering produced by the large dynamical effects of electron diffraction may obscure the interpretation of ePDF. In the present work, precession electron diffraction (PED-TEM) has been employed to obtain the ePDF of two different samples: lipoic acid- and hexanethiolate-capped gold nanoparticles (4.5 and 4.2 nm, respectively), randomly oriented and measured at both liquid nitrogen and room temperatures. The electron diffraction data were processed to obtain ePDFs which were subsequently compared with the PDF of different ideal structure models.

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Journal: The Journal of Physical Chemistry C

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Date Submitted: 9/8/19 12:00AM

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Publication Location:

Article Title: Synthesis, Mass Spectrometry, and Atomic Structural Analysis of Au₂₀₀₀(SR)₂₉₀ Nanoparticles

Authors: Sandra Vergara, Ulises Santiago, Chanaka Kumara, Diego Alducin, Robert L. Whetten, Miguel Jose Yáñez

Keywords: nanoparticles, nanoclusters, mass spectrometry, electron microscopy, aberration-corrected STEM

Abstract: Metallic nanoparticles display unique optical, electronic, and chemical properties compared to their bulk counterparts. These properties are influenced by the internal structure of nanoparticles. Therefore, atomic structural characterization of nanoparticles is of paramount importance in nanotechnology. In this work, we present the synthesis, mass spectrometry, and structural characterization of highly monodisperse thiolate-protected gold nanoparticles using aberration-corrected STEM. Mass spectrometry reveals the composition to be Au₂₀₀₀(SC₆H₁₃)₂₉₀. The images registered in the HAADF-STEM showed the presence of decahedral and single-crystal fcc nanoparticles as well as fcc structures with multiple planar defects. We also observed nanoparticles with an inner grain boundary corresponding to a high angle grain boundary classified as γ_9 under the coincidence site lattice notation.

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Date Submitted: 9/8/19 12:00AM

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Publication Location:

Article Title: Toward Smaller Aqueous-Phase Plasmonic Gold Nanoparticles: High-Stability Thiolate-Protected 4.5 nm Cores

Authors: M. Mozammel Hoque, Kathryn M. Mayer, Arturo Ponce, M. M. Alvarez, Robert L. Whetten

Keywords: nanoparticles, nanomaterials synthesis, electron microscopy, plasmonics

Abstract: Most applications of aqueous plasmonic gold nanoparticles benefit from control of the core size and shape, control of the nature of the ligand shell, and a simple and widely applicable preparation method. Surface functionalization of such nanoparticles is readily achievable but is restricted to water-soluble ligands. Here we have obtained highly monodisperse and stable smaller aqueous gold nanoparticles, prepared from citrate?tannate precursors via ligand exchange with each of three distinct thiolates: 11-mercaptoundecanoic acid, ?-R-lipoic acid, and para-mercaptobenzoic acid. These are characterized by UV?vis spectroscopy for plasmonic properties; FTIR spectroscopy for ligand-exchange confirmation; X-ray diffractometry for structural analysis; and high-resolution TEM for structure and size determination. Chemical reduction induces a blueshift, maximally +0.02 eV, in the localized surface plasmon resonance band; this is interpreted as an electronic charging of the MPC gold core

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Date Submitted: 9/8/19 12:00AM

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Publication Location:

Article Title: Fivefold annealing twin in nanocrystalline Au/Pd film

Authors: Prakash Parajuli, Ruben Mendoza-Cruz, J. Jesus Velazquez-Salazar, Miguel Jose Yacamán, Arturo Poi

Keywords: Five-fold twins, Annealing, Nanocrystalline, Thin films, Au/Pd alloys

Abstract: In this study, we report the experimental observation of five-fold annealing twin in the nanocrystalline Au/Pd thin film without external stress by annealing the film for 20 min at 473 K using atomic-resolution high angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) imaging, and energy dispersive X-ray spectroscopy (EDS) employing Cs aberration-corrected electron microscopy. As per the knowledge of the authors, this is the first time that five-fold annealing twin has been observed for Au/Pd thin films. Furthermore, the spectroscopic elemental analysis shows the uniform distribution of palladium in the film, indicating no palladium grain boundary segregation. Strain analyses have been performed by geometric phase analysis (GPA) across the five-fold structures. GPA method shows the nonuniform strain distribution across the various segments of the five-fold structure.

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Publication Location:

Article Title: Semiconductor behavior of pentagonal silver nanowires measured under mechanical deformation

Authors: Edgar Ochoa, Diego Alducin, John E. Sanchez, Clemente Fernando, Ulises Santiago, Arturo Ponce

Keywords: Metallic nanowires, Electric properties, In situ microscopy, High index planes . Electromechanical measurements

Abstract: In the present work, electrical measurements using in situ TEM on pentagonal silver nanowires were performed. Electrical biasing was applied to individual nanowires with and without simultaneous in situ TEM mechanical deformation. The response of the ohmic resistance was measured in the I-V curves. A reduction in the break voltage and the resistance was measured, when the nanowires were subjected to a bending deformation. In situ electric measurements on both, with and without deformation, show a typical semiconductor behavior. Surface scattering of electrons in the nanowires and movement of dislocations act as the main causes of the electrical properties reported herein. In this way, the determination of the surface morphology was carried out by using off-axis electron holography followed by a phase reconstruction and structural modeling. The high Miller-index facets were determined to be the (533) stepped surface plane on all five longitudinal sides of the nanowires.

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Publication Location:

Article Title: Growth and Structural Characterization of Doped Polymorphic Crystalline MgPc as an Organic Semiconductor

Authors: Leon Hamui, Mariana Elena Sanchez-Vergara, Rocio Sanchez-Ruiz, Cecilio Aacu

Keywords: organic semiconductors; metallophthalocyanine; thin films; Raman spectroscopy; structural analysis

Abstract: The doping and crystallization of the molecular semiconductor formed from the magnesium phthalocyanine (MgPc) and 1-(4-Methoxyphenyl)-2,2,6,6-tetramethyl-5-phenylhepta-3,4-dienedioic (MTPDA) acid was carried out in this work. The crystals obtained were characterized by using transmission electronic microscopy (TEM), Raman spectroscopy, and X-Ray diffraction (XRD), to later evaluate their optical behavior. Raman, IR, and UV-Vis results indicate that the MgPc has been doped with the MTPDA. A uniform material layer with particles is observed as a result of a two-stage process, nucleation and growth. The polycrystalline films are constituted by a mixture of alpha and beta phases with crystalline sizes of ~7 nm, 14 nm, and 20 nm average sizes. The films exhibit a preferred orientation along the [001]. The MTPDA doping does not have an important effect on the molecule planar distances indicating that the MTPDA molecule is among the equivalent MgPc plane direction.

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Publication Location:

Article Title: Misorientation dependence grain boundary complexions in γ-symmetric tilt Al grain boundaries

Authors: Prakash Parajuli, David Romeu, Viwanou Hounkpati, Ruben Mendoza-Cruz, Jun Chen, Miguel J

Keywords: Grain boundary segregation, Grain boundary complexions, Thin films, Transmission electron microscopy

Abstract: The doping and crystallization of the molecular semiconductor formed from the magnesium phthalocyanine (MgPc) and 1-(4-Methoxyphenyl)-2,2,6,6-tetramethyl-5-phenylhepta-3,4-dienedioic (MTPDA) acid was carried out in this work. The crystals obtained were characterized by using transmission electronic microscopy (TEM), Raman spectroscopy, and X-Ray diffraction (XRD), to later evaluate their optical behavior. Raman, IR, and UV-Vis results indicate that the MgPc has been doped with the MTPDA. A uniform material layer with particles is observed as a result of a two-stage process, nucleation and growth. The polycrystalline films are constituted by a mixture of alpha and beta phases with crystalline sizes of ~7 nm, 14 nm, and 20 nm average sizes. The films exhibit a preferred orientation along the [001]. The MTPDA doping does not have an important effect on the molecule planar distances indicating that the MTPDA molecule is among the equivalent MgPc plane direction.

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Publication Location:

Article Title: Synergistic photoluminescent interaction of Si and CdTe quantum dots

Authors: Janeth Alexandra Garcia-Monge, Clarissa D. Vazquez-Colon, Arturo Ponce, Gregory Guisbiers, Arturo /

Keywords: quantum dots, photoluminescence

Abstract: The doping and crystallization of the molecular semiconductor formed from the magnesium phthalocyanine (MgPc) and 1-(4-Methoxyphenyl)-2,2,6,6-tetramethyl-5-phenylhepta-3,4-dienedioic (MTPDA) acid was carried out in this work. The crystals obtained were characterized by using transmission electronic microscopy (TEM), Raman spectroscopy, and X-Ray diffraction (XRD), to later evaluate their optical behavior. Raman, IR, and UV-Vis results indicate that the MgPc has been doped with the MTPDA. A uniform material layer with particles is observed as a result of a two-stage process, nucleation and growth. The polycrystalline films are constituted by a mixture of alpha and beta phases with crystalline sizes of ~7 nm, 14 nm, and 20 nm average sizes. The films exhibit a preferred orientation along the [001]. The MTPDA doping does not have an important effect on the molecule planar distances indicating that the MTPDA molecule is among the equivalent MgPc plane direction.

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Article Title: Multiphysics Modeling of Plasmonic Photothermal Heating Effects in Gold Nanoparticles and Nanoparticle Arrays

Authors: Santiago Manrique-Bedoya, Mohammad Abdul-Moqueet, Priscilla Lopez, Tara Gray, Matthew Disiena, /

Keywords: plasmonics, gold nanorods, photothermal heating

Abstract: Induced hyperthermia has been demonstrated as an effective oncological treatment due to the reduced heat tolerance of most malignant tissues; however, most techniques for heat generation within a target volume are insufficiently selective, inducing heating and unintended damage to surrounding healthy tissues. Plasmonic photothermal therapy (PPTT) utilizes light in the near-infrared (NIR) region to induce highly localized heating in gold nanoparticles, acting as exogenous chromophores, while minimizing heat generation in nearby tissues. However, optimization of treatment parameters requires extensive in vitro and in vivo studies for each new type of pathology and tissue targeted for treatment, a process that can be substantially reduced by implementing computational modeling.

Distribution Statement: 2-Distribution Limited to U.S. Government agencies only; report contains proprietary info
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Publication Location:

Article Title: Synthesis and bioconjugation of alkanethiol-stabilized gold bipyramid nanoparticles

Authors: Mohammad M Abdul-Moqueet, Leeana Tovias, Priscilla Lopez, Kathryn M Mayer

Keywords: gold bipyramids, nanoparticles, nanoparticle bioconjugates, self-assembled monolayers, localized surface plasmon resonanc

Abstract: Gold bipyramid (GBP) nanoparticles are promising for a range of biomedical applications, including biosensing and surface-enhanced Raman spectroscopy, due to their favorable optical properties and ease of chemical functionalization. Here we report improved synthesis methods, including preparation of gold seed particles with an increased shelf life of ?1 month, and preparation of GBPs with significantly shortened synthesis time (< 1 h). We also report methods for the functionalization and bioconjugation of the GBPs, including functionalization with alkanethiol self-assembled monolayers (SAMs) and bioconjugation with proteins via carbodiimide cross-linking. Binding of specific antibodies to the nanoparticle-bound proteins was subsequently observed via localized surface plasmon resonance sensing. Rabbit IgG and goat anti-Rabbit IgG antibodies were used as a model system for antibody-antigen interactions.

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Article Title: Magnetic Vortex Domain Wall Observation on Polycrystalline Imperfect Iron/Cobalt Alloy Nanowires Growing on 1050 Aluminum

Authors: César Leandro Londoño-Calderón, Alejandra Londoño-Calderón, Oscar Moscoso-Londoño, Arturo Galir

Keywords: FeCo, Nanowire arrays, Magnetic Properties, Low Purity Aluminum, electron holography

Abstract: In this work, we report the magnetic vortex domain wall structure of polycrystalline imperfect iron-cobalt alloy nanowires growing on 1050 Aluminum by pulsed electrodeposition. The magnetic properties are analyzed using magnetometry and off-axis electron holography. The electrodeposited nanowire's arrays show homogeneous elemental composition and exhibit a structure composed of piled-up grains of small crystallites. The saturation magnetization, coercive field, and reduced remanence, measured in directions parallel and perpendicular to the nanowire's axis, are studied as a function of the temperature. Although the array of nanowires on anodized aluminum grows both straight and within an inclination angle, we notice in both cases a high shape anisotropy, which is the most predominant contribution to the magnetic behavior.

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Publication Location:

Article Title: Magneto-plasmonic biocompatible nanorice

Authors: Carlos M. García-Rosas, Luis A. Medina, Priscilla Lopez, Nicolas Large, Alejandro Reyes-Coronado

Keywords: Nanorice matryoshka, Magneto-plasmonics, Localized surface plasmon resonances, Local field intensity enhancement, Nuclear magnetic resonance

Abstract: In this work, we conduct a comprehensive computational study of the optical and photothermal properties of multifunctional γ -Fe₂O₃/Au/SiO₂ nanorice matryoshka nanoparticles using a combination of boundary element method, finite element method, and discontinuous Galerkin time-domain method. The magnetic core in the nanorice allows their use as a contrast agent in nuclear magnetic resonance technique as well as dragging and rotating the nanoparticles with an external magnetic field, while the plasmonic counterpart enables the excitation of localized surface plasmon resonances. We show that both longitudinal and transverse plasmonic resonances induced within the hybrid asymmetric nanoparticle can be sintonized into the 650–900 nm range of the electromagnetic spectrum, where the absorption of tissue is minimal.

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Article Title: Low-defect-density ZnO homoepitaxial films grown by low-temperature ALD

Authors: David Elam, Eduardo Ortega, Anastasiia Nemashkalo, Yuri Strzhemechny, Arturo Ayon, Arturo Ponce, /

Keywords: thin films, excitons, transmission electron microscopy, atomic layer deposition, photoluminescence, epitaxy, crystallographic defects

Abstract: We report atomic layer deposition (ALD) of ZnO thin films on O-polar surface crystalline ZnO substrates at the relatively low temperatures of 120, 150, and 200 C. The as-grown ZnO films are studied with aberration-corrected transmission electron microscopy and diffraction contrast, photoluminescence (PL), and surface photovoltage (SPV) spectroscopy. We find that the homoepitaxial films have a monocrystalline structure with the density of basal stacking faults comparable to that of the substrate ($\sim 10^{11}$ cm²) and that the stacking faults can induce high lattice strain due to their interaction with the inversion domain boundaries. The narrow excitonic PL linewidth (2 meV at 8K) and the sharp SPV bandgap transition confirm the high quality of the ZnO films. Despite similarities in the film properties, the growth temperature has an effect on the density and spatial distribution of intrinsic defects.

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Article Title: Surface structural characteristics of some colloidal lipid systems used in pharmaceuticals

Authors: Aldemar Gordillo-Galeano, Arturo Ponce, Claudia Elizabeth Mora-Huertas

Keywords: Density, Steric stability, Surface hydrophobicity, Zeta potential

Abstract: Surface properties of solid lipid nanoparticles (SLN), nanostructured lipid carriers (NLC), and nanoemulsions (NE) are determinant to understand their structural characteristics and their behavior as drug delivery systems; however, there is still limited knowledge in this respect. As a contribution, this paper reports the study of some surface properties of these colloidal systems which were prepared with binary mixtures of trimyristin and medium-chain triglycerides (MCT) and stabilized with Poloxamer 188 (P188). Particle sizes and polydispersity indexes decrease in the order SLN > NLC > NE suggesting that the addition of MCT to trimyristin reduces the particle asymmetry. ζ potentials range from 6 mV to 10 mV and the P188 layer is ~ 20 nm; thus, steric stabilization seems to predominate. The amount of P188 on the surface of the drops in the NE was less than that on the surface of the particles in the SLN and NLC and decreases as the proportion of MCT decreases.

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Article Title: Advances in the electron diffraction characterization of atomic clusters and nanoparticles

Authors: Arturo Ponce, Jeffery A. Aguilar, Jess Tate, Miguel José Yacamán

Keywords: electron diffraction, atomic clusters, nanoparticles

Abstract: Nanoparticles and metallic clusters continue to make a remarkable impact on novel and emerging technologies. In recent years, there have been impressive advances in the controlled synthesis of clusters and their advanced characterization. One of the most common ways to determine the structures of nanoparticles and clusters is by means of X-ray diffraction methods. However, this requires the clusters to crystallize in a similar way to those used in protein studies, which is not possible in many cases. Novel methods based on electron diffraction have been used to efficiently study individual nanoparticles and clusters and these can overcome the obstacles commonly encountered during X-ray diffraction methods without the need for large crystals. These novel methodologies have improved with advances in electron microscopy instrumentation and electron detection.

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Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published
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Article Title: In-Situ Magnetization Reversal Mechanism in Ni Nanowires Investigated by Electron Holography

Authors: Arturo Galindo, Arturo Ponce

Keywords: magnetization, nanowires, electron holography

Abstract: Increasing interest in ferromagnetic (FM) nanowires (NWs) has inspired considerable research in recent years with the rapid development of information storage technology. The miniaturized dimensions of NWs generate anisotropic magnetic properties due to a competing energy balance between the shape and crystallographic structure. For this reason, the magnetization reversal mechanism in Ni NWs has been greatly investigated as they offer unique advantages in developing high-density magnetic recording media. Consequently, the precise control of the physical properties of FM NWs is a great challenge in nanotechnology. Thus far, the magnetic state of nanomaterials is analyzed using various techniques, however, the magnetic information obtained is limited to the exterior of the nanomaterial. In contrast, a powerful magnetometry technique rests in a specialized transmission electron microscopy (TEM) mode called off-axis electron holography.

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Article Title: Vertical Stacking of Electrodeposited Nanorods with Controlled Dimensions and Chemical Composition
Authors: Arturo Galindo, Jose Luis Reyes, Arturo Poncs
Keywords: electrodeposition, nanorods
Abstract: We have developed a method designed to assemble vertical layers composed of ultra-dense arrays (1Tb/inch²) of Ni and Co nanorods. The fabrication scheme allows total control of the nanorod's dimensions (diameter and length) by using porous anodic aluminum oxide (AAO) membranes. A two-step anodization process is employed for the fabrication of AAO using Oxalic and Sulfuric electrolytes. The resulting porous network consists of nanopores with 40 and 80 nm diameters respectively.
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Journal: Microscopy and Microanalysis
Publication Identifier Type: DOI **Publication Identifier:** 10.1017/S1431927621009417
Volume: 27 **Issue:** **First Page #:** 2660
Date Submitted: 12/27/21 12:00AM **Date Published:** 7/1/21 5:00AM
Publication Location:
Article Title: Structural defects in ZnO thin films grown by atomic layer deposition at low temperatures
Authors: David Elam, Eduardo Ortega, Andrey Chabanov, Arturo Ponce
Keywords: thin films, atomic layer deposition
Abstract: In epitaxial thin films, stacking faults (SF's) on a crystal play an important role due their interaction with dislocations. Dislocations often divide into partial dislocations with the formation of a stacking fault connecting them. On the synthesized thin films SFs parallel to the samples' interface can be seen not only on the substrate but also in the film where they appear to be more common. These parallel defects can be easily spotted when acquiring dark-field images. As the thin films and images differ in thickness and size, using the number of individual defects observed on a single image can be misleading. In a way of normalizing results SF's are counted in several DF images, considering the area of the film that is enclosed, to latter extrapolated these values for comparison. In this work, ZnO layers have been grown at low temperatures, 100, 150, and 200°C by atomic layer deposition (ALD).
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Date Received: 27-Aug-2020 **Conference Date:** 11-Aug-2019 **Date Published:**
Conference Location: San Diego, CA
Paper Title: Synthesis, characterization, and computational modeling of polyelectrolyte-coated pl
Authors: Priscilla Lopez, Kathryn Mayer, Nicolas Large
Acknowledged Federal Support: Y

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Paper Title: Computational Characterization of Magneto-Plasmonic Nanowires

Authors: Joel Rigor, Nicolas Large

Acknowledged Federal Support: **Y**

Partners

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I certify that the information in the report is complete and accurate:

Signature: Kathryn Mayer

Signature Date: 12/29/21 1:52PM

Hybrid Plasmonics at UTSA: Work Accomplished Under Goals

Background Note

This is the third and final report for our project “Hybrid Plasmonics at UTSA: Investigating Plasmonic/Magnetic and Plasmonic/Biomolecular Systems,” which ran from 09/30/2018 – 09/29/2021. Below we describe our progress under each of the two main research thrusts.

During the current reporting period, the impact of COVID-19 on our project continued to be felt. Research operations at UTSA had mostly returned to normal by Fall 2020; however, administrative and supply chain delays continued throughout the year. Our summer AEOP apprenticeship program (formerly HSAP/URAP) was held in a hybrid format, with some of the undergraduate apprentices participating in person, and others online. (All of the high school apprentices participated online.) Team meetings were largely held online over Zoom.

Thrust 1: Plasmonic/Magnetic Hybrid Materials

This thrust is focused on both experimental (fabrication and characterization) and theoretical (numerical simulation) studies of hybrid plasmonic/magnetic nanowires. These materials are of interest because the plasmonic component provides the possibility of optical control of the magnetic properties of the system. By gaining an understanding of the interplay between optical and magnetic properties in these materials, we open the door to applications such as high-density data storage and memory devices, and photothermal/ hyperthermia-based medical treatments.

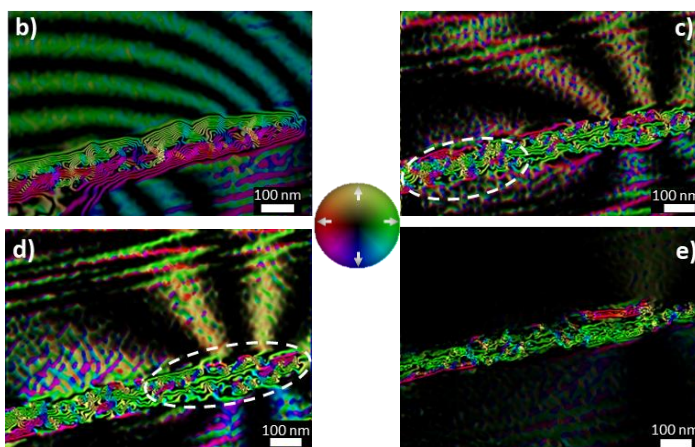


Figure 1. Off-axis electron holography showing detailed magnetic behavior of a single nickel nanowire under increasing magnetic field (b) – (e).

In the previous two reporting periods, we described fabrication methods for arrays of segmented nanowires consisting of gold and nickel segments, as well as the characterization of these plasmonic/magnetic systems using advanced electron microscopy-based methods. In the current reporting period, we continued on these themes as well as describing new electron holography techniques to reveal detailed information on the magnetic behavior of the nanomaterials.

Figure 1 shows an example of off-axis electron holography of an isolated nickel nanowire. As the applied magnetic field is increased as shown in Figure 1(c) and (d), we first begin to observe an overall magnetic flux along the longitudinal direction of the nanowire, as shown by the prevailing green color, along with several vortex cores (circled regions) rotating in the counterclockwise direction. As the magnetic field is increased further as shown in Figure 1(e), the vortex cores

disappear, along with the stray field lines outside the nanowire, indicating that the saturation state of the nanowire has been reached.

In the past year we also carried out finite-difference time-domain (FDTD) simulations of hybrid plasmonic/magnetic systems including segmented cobalt and gold nanowires, as seen in **Figure 2**. Figure 2(a) shows two spectral regions where plasmon modes exist: UV modes with distinct, narrow lines, and strongly interacting IR modes which produce a Fano resonance-like line shape. Figure 2(b) shows the electric field distributions associated with some of the IR modes for a segmented Co-Au-Co nanowire featuring persistent plasmon oscillations in the metallic segment.

Thrust 2: Plasmonic/Biomolecular Hybrid Materials

Thrust 2 is focused on the synthesis, functionalization, and characterization of hybrid plasmonic/biomolecular nanoparticle bioconjugates and the computational modeling of their optical properties, as well as the synthesis and characterization of molecular analogues to these systems which can be used to elucidate the mechanisms of photosensitizer-induced conformational changes in proteins. These systems have applications in enhanced spectroscopies, biosensing, and photothermal and photodynamic therapies; they also enable further studies of how light-induced biomolecular manipulations can influence cell signaling pathways.

In the previous two reporting periods, we described the experimental and computational characterization of polyelectrolyte-coated gold nanorods, and progress towards the preparation of photosensitizer-modified proteins. In the current reporting period, we successfully produced and characterized the photosensitizer-modified proteins, as well as preparing gold nanoparticle-photosensitizer conjugates, as shown in **Figure 3**.

Figure 3(a) shows the absorbance spectra of the protein human serum albumin (HSA) modified with several different metalloporphyrins. These systems can act as analogues to hybrid plasmonic/biomolecular systems in which the photosensitizer molecule, with its well understood photophysics, takes the place of the plasmonic nanoparticle. Figure 3(b) shows the absorbance

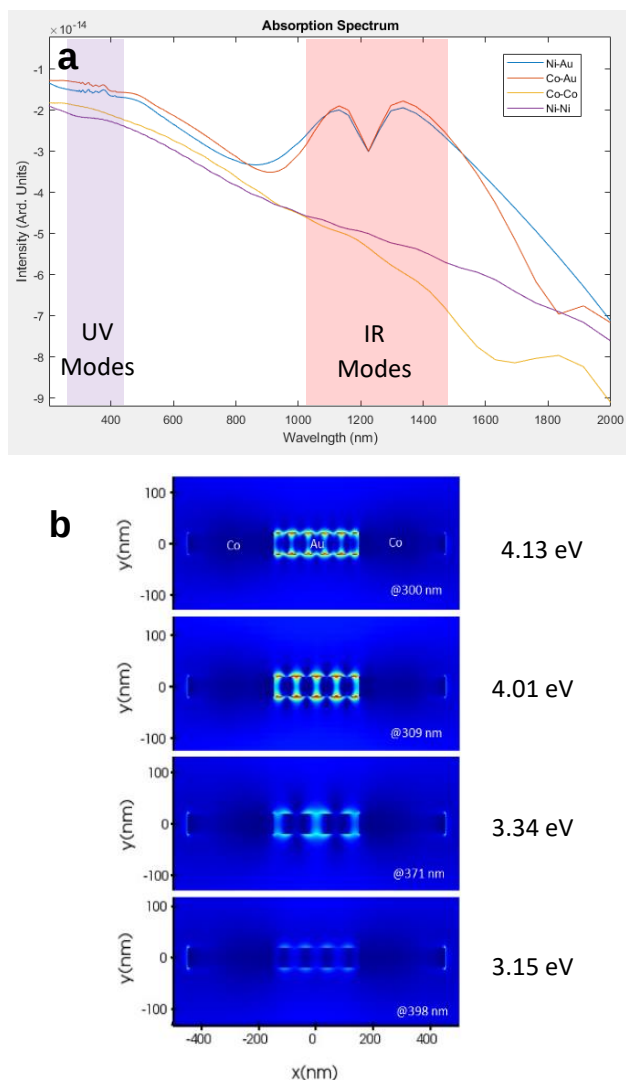


Figure 2. Simulated (a) optical absorption spectra and (b) electric field distributions of hybrid plasmonic/magnetic nanowires.

spectra of HSA-gold nanoparticle bioconjugates, in which gold nanospheres are decorated with HSA adsorbed to the metal surface.

Please see the “Dissemination” section for information on the publications and presentations relating to the project.

Student Education and Training

In addition to the research progress described here, please see the “Training” section for further information on the work accomplished under our goals related to student research training, including graduate student training as well as undergraduate and high school apprenticeship programs.

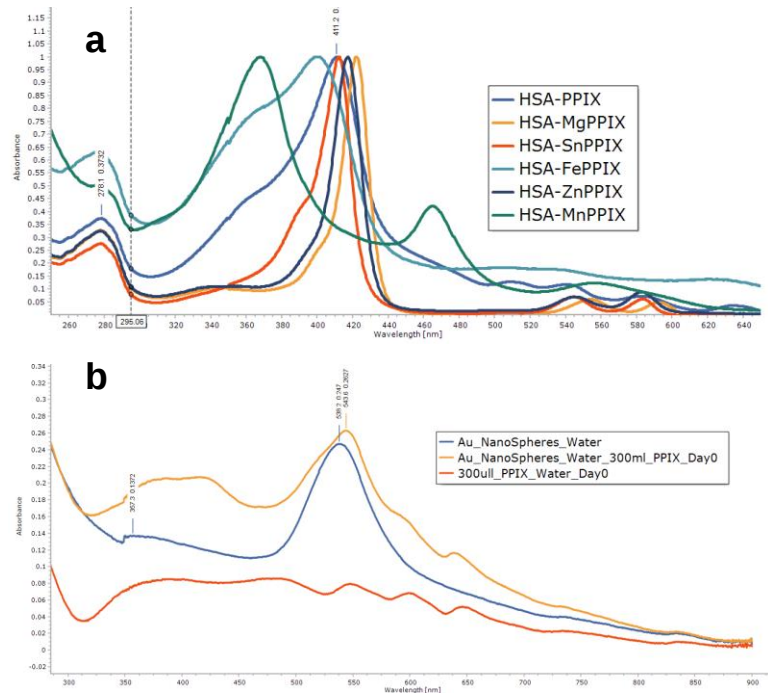


Figure 3. Optical absorbance spectra of (a) human serum albumin (HSA) modified with metalloporphyrins and (b) HSA-gold nanoparticle bioconjugates.