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Major Goals: The major goal of this project is to acquire a state-of-the-art OXFORD PlasmaPro 100 Cobra inductively coupled plasma (ICP) etching system to enable researchers at the Alabama A&M University (AAMU), a leading HBCU, to fabricate devices at the micro- and nanoscales. The capability of fabricating materials and devices at the nanoscale is critical not only for the advancement of science and technology but also for the training of the future scientific workforce. Currently, 10 engineering and science faculty members and more than 30 graduate and undergraduate students rely on the class-1,000 cleanroom as a primary experimental resource for the design and fabrication of nanoscale materials and devices. Three of the key capabilities for nanoscale fabrication are the ability to create patterns using UV or e-beam lithography, the ability to grow nanoscale thin-film materials, and the ability to etch those patterns into a variety of substrates. Our lithographic capability is excellent: (1) a JEOL scanning electron microscope with a nanometer pattern generation system for e-beam lithography; (2) A Suss MABA 6 Gen3 mask aligner for UV lithography, which was installed with the DoD/ARO support in 2017. Our thin-film deposition capability is also excellent: (1) physical vapor deposition (PVD) including a sputtering deposition system and a thermal/e-beam evaporation system; (2) chemical vapor deposition (CVD) including a KJL ALD-150LX plasma-enhanced atomic layer deposition (PE-ALD) system, which was installed with the DoD/ARO support in 2018. However, we don’t have any high-density plasma etch system in our cleanroom, which is critical in the fabrication at the nanoscale. The requested plasma etch system is a necessary companion instrument to our clean-room fabrication tools. The acquisition of the etching system would propel nanoscale science and technology at AAMU serving northern Alabama to new frontiers.

Accomplishments: An advanced PlasmaPro 100 Cobra inductively coupled plasma etching system, which is manufactured by the Oxford Instruments Company, has been purchased with the DoD/ARO funding and installed in the AAMU-EE Clean Room in the Engineering building at the Alabama A&M University. The project has been completed successfully as proposed.

Training Opportunities: More than thirty students and faculty will be trained and use the etching system for their research and education projects each year.

Results Dissemination: Multiple ongoing research projects will be supported by the installed plasma etching system and the research results obtained from the research projects will be reported in the annual MRS Fall meeting and the annual AVS symposium and exhibition and will be published in scientific journals.

Honors and Awards: Nothing to Report

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Technology Transfer: Nothing to Report

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Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
National Academy Member: N

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Final Report for the DoD/ARO Project (W911NF2110195): Acquisition of an Advanced Plasma Etching System for Research at Nanoscale for Energy Harvesting and Nanoelectronics at Alabama A&M University

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The objective of this project is to acquire a state-of-the-art OXFORD PlasmaPro 100 Cobra inductively coupled plasma (ICP) etching system to enable researchers at Alabama A&M University (AAMU) to fabricate micro- and nanoscales devices for the application of energy harvesting and nanoelectronics. An advanced PlasmaPro 100 Cobra inductively coupled plasma etching system, which is manufactured by the Oxford Instruments Company, has been purchased with the DoD/ARO funding and installed in the AAMU-EE Clean Room in the Engineering building at the Alabama A&M University. The project has been completed successfully as proposed.

After the project was awarded by DOD/ARO in May 2021, the Purchasing Office at Alabama A&M University conducted the process for purchasing the ALD system. The Oxford Instruments Company was selected and awarded for manufacturing the system. The purchase order for purchasing the PlasmaPro 100 Cobra ICP etching system was issued to the Oxford Instruments Company by AAMU in July 2021. The system was then manufactured by the Oxford Instruments Company, and was received by AAMU in February 2022. The installation of the system in the AAMU-EE Clean Room was completed by the engineers from the Engineered Maintenance Services Company in Huntsville, AL together with the PI and Co-PIs under the guidance of the engineer in the Oxford Instruments Company in late August 2022. It took a longer time to obtain all the special etching gases and the parts for the installation of the system due to the delayed supplies in the Covid-19 spreading period. The system readiness form for the startup has been completed and returned to the Oxford Instruments Company, the system will be started by the engineers from the Oxford Instruments Company in October 2022 (the service engineer in the Oxford Instruments Company is extremely busy and tries to schedule the startup of system as early as possible), and system will then join our other cleanroom fabrication facility to serve our students and researchers for their research and education in the fabrication of micro and nanoscale devices in October 2022.

Figures 1, 2, 3, and 4 show the PlasmaPro 100 Cobra ICP etching system which has been installed in the cleanroom fabrication facility in the Engineering building at the Alabama A&M University.

The PlasmaPro 100 Cobra ICP etching system is built on a 200mm wafer platform with the capability of atomic layer etching (ALE), and has 3 kW radio frequency plasma generation; high capacity turbomolecular pumping and mechanical pump; vacuum load lock for substrate introduction; 200 mm substrate handling with substrate chuck cooling and heating; radio frequency substrate bias power supply; seven process gas lines; and automatic load matching for coil and chuck.

The etching system has the capability of etching semiconductors, dielectrics, and metals, with high selectivity, vertical sidewalls, dimensional linewidth control, and atomic layer etching (ALE) while the system was designed specifically for etching silicon (Si), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), hafnium oxide (HfO_2), zirconium oxide (ZrO_2), aluminum (Al), copper (Cu), two-dimensional (2D) semiconductors such as 2D molybdenum disulfide (MoS_2) and molybdenum selenium ($MoSe_2$) nanomaterials materials to support our current on-going education and research projects in nanofabrication. The process modules offer excellent uniformity and high throughput processes on a range of applications. The ICP reactive-ion etching (RIE) is a versatile and highly efficient plasma etching technique used in microfabrication. Independent control of plasma density through the ICP source means that high density plasmas can be realized without increasing DC bias on the lower electrode. High etch rates, increased selectivity to mask, improved ion directionality and low device damage are all realized using this technique. Robust auto matching across a wide parameter range means that processes are repeatable and reliable ensuring high performance every run, every day. The central system features are summarized below.

Low Operating Gas Pressure. This system has a large-capacity turbomolecular pump (Adixen ATH 1600MT Turbo Pump) to handle substantial gas loads, backed by a mechanical pump (Pfeiffer Duo 65 C Pump). Operation at low gas pressures is desirable for extremely fine-scale etching; at higher pressures ion-neutral collisions will scatter and deflect corrosive ions into the vertical etched sidewalls, resulting in unwanted lateral or isotropic etching.

Controllable Ion Energies. The proposed system has an up to 600 W substrate bias power supply for independent control of impinging ion energy at the substrate surface. Excessive impinging ion energies can damage or destroy very fine scale features.

Substrate Handling, Size, and Cooling. For a research environment it is sufficient to load substrates manually and to dispense with cassette-to-cassette operation; however, the installed plasma etching system does have a separately pumped vacuum load-lock, which is essential to provide sufficient throughput for the large number of faculty and students who will use this instrument. The system also has a low temperature substrate cooling option to minimize thermally stimulated isotropic etching and to avoid degradation of temperature-sensitive films. The substrate can also be heated if it is needed.

Process Gases. The plasma etching system is intended to facilitate a variety of research projects, including high-efficiency thermoelectric materials and integrated devices for the application of power generation and solid-state cooling, development of wafer-scale fabrication of carbon-based integrated electronic devices, and bio-sensor and device fabrication, and fabrication of 2D nanomaterials and nanoelectronic devices. Seven process gases have been connected with the system, including argon (Ar), oxygen (O_2), sulfur hexafluoride (SF_6), octafluorocyclobutane (C_4F_8), Trifluoromethane (CHF_3), chlorine (Cl_2), and boron trichloride (BCl_3). The seven process gases can meet the requirement of etching for our current on-going education and research projects in nanofabrication.

The PlasmaPro 100 Cobra ICP etching system has the following detailed features and functions:

Plasma Generation

- RF low power bias kit: Power damping for lower electrode power with auto switching between low and high power ranges (<30 W and >30 W) to allow accurate control in the 0.2 - 30 Watt range.
- ALE gas dose kit: Gas dose kit for atomic layer etch. The ALE gas dose hardware enables short (down to 10msec pulses), controlled, reproducible gas doses required for the ALE process.
- RF 300W generator and amu match for ETCH: 300W 13.56MHz RF generator for etch processes. Connected to lower electrode via a vacuum capacitor auto match unit.
- Cobra300 3kW ICP plasma etch source: 3kW 2MHz RF generator connected to 300mm diameter ICP source via a vacuum capacitor auto match unit. Includes laser endpoint detector port and mounting.
- Electrostatic Shield for ICP plasma source: Electrostatic shield to suppress the capacitive coupling of the RF energy to the plasma. The plasma current circulates within the plasma and does not intersect the ICP tube, reducing process particles and providing improved control of plasma ion energies at substrate surface.

Substrate Electrode

- Cryo-cooled / electrically-heated etch lower electrode kit: 240 mm diameter aluminum lower electrode LN2 cryo cooled and electrically heated with a temperature range of -150 °C to +400 °C, automatically switching between LN2 cooling and electrically heating modes. Features an axial lifting mechanism for wafers and platens up to 200 mm diameter. The electrode is fixed height with an Integral dark space shield. Suitable for use with RF bias powers up to 600W maximum.
- Wafer clamping and helium backing kit: Wafer clamping and helium backing for lower electrode.
- Julabo FP51 chiller: Remotely controlled air cooled recirculating heater/chiller unit suitable for ultra-low electrode temperature control. Temperature stability of ±0.5 °C, and 1500 watts of cooling at 0 °C.
- Quartz clamp with continuous contact.

Gas Supply

- Standard Gas Pod externally mounted: The gas pod is ventilated and fitted with an extraction port for maximum safety.
- Split gas manifold: Split gas manifold to provide two separate gas outlets from the external gas pod into the same process chamber.
- Gas pod wall mounted: Gas pod provided ready for wall mounting remote from the system console.
- 5 standard gas lines and MFCs for non-toxic gases (argon (Ar), oxygen (O_2), sulfur hexafluoride (SF_6), octafluorocyclobutane (C_4F_8), and Trifluoromethane (CHF_3)): Non-toxic gas line with digital, programmable mass flow controller.

- 2 by-passed gas lines and MFCs for toxic gases (chlorine (Cl_2) and boron trichloride (BCl_3)): Toxic gas line with metal sealed digital, programmable mass flow controller.
- 1 gas line heating kit suitable for non-toxic gas line: used for low vapor pressure gas for C_4F_8.

Vacuum Measurement

- 100mT CM gauge: 100 mTorr capacitance manometer gauge used for process pressure measurement.

Process Pumping

- Adixen ATH1600MT turbo, ISO200 pipework, apc and heated backing valve kit: Corrosive compatible, magnetic bearing turbomolecular pump including integration kit for use on a process chamber.
- Pfeiffer A124H ELT pump kit: 110 m^3/hr dry pump for process turbo backing.
- PlasmaPro 100 Chamber and pump down pipe Heating Kit: Electrical heating kit to warm the chamber and pump down pipework, minimizing process deposition. Maximum temperature 60 °C.
- PlasmaPro 100 Pumping Foreline Heating Kit.

End-Point Detector

- Variable wavelength Ocean Optics optical EPD Kit: 200-850nm spectrometer integrated into the tool software for endpoint purposes. Process endpointing is achieved by specifying the wavelength to be monitored.

Wafer Handling

- PlasmaPro 100 advanced single wafer loadlock kit: Load lock for up to 200 mm diameter single wafer. Substrates are loaded directly onto the wafer transfer arm via a hinged lid. Transfer isolation valve fitted between the load lock and the process chamber. Substrates may be transferred under vacuum into the single process chamber.

Loadlock Pumping

- Roughing only to Dry Pump - Isolation Valve included.
- Loadlock soft pump kit: Allows for gradual chamber pumping in order to prevent wafer movement and minimize particle generation.
- Loadlock soft vent kit: Allows for gradual chamber venting in order to prevent wafer movement and minimize particle generation.
- Pfeiffer ACP15G dry pump kit: 14 m^3/hr dry pump with hardware to integrate the pump to the loadlock.

Software

- PTIQ: The latest intelligent software solution for the Plasma Pro processing system. It provides an exceptional level of system control.

System Installation:

The PlasmaPro 100 Cobra ICP etching system has been installed in the Clean Room fabrication facility in the Engineering building at the Alabama A&M University. The Clean Room facility has over 2500 square feet of class 1000 processing space, with deionized water installations. The facility has the electrical capacity and ventilation required for the system, as well as the necessary compressed air, water, and nitrogen lines. The facility maintains equipments for lithography, thin-film deposition, etching, oxidation and diffusion, and a variety of characterization equipment.

After the PlasmaPro 100 Cobra ICP etching system was shipped to the Clean Room, it was installed and connected with the compressed air, water, gases, and electricity by the engineers in the Engineered Maintenance Services Company together with PI and Co-PIs under the guidance of the engineer in the Oxford Instruments Company.

System Start Up and Operation Training:
The system readiness form for the startup has been completed and returned to the Oxford Instruments Company. The system will be started by the engineers from the Oxford Instruments Company in October 2022, and the operation training for our faculty and students will be completed by the engineers from Oxford during the startup. The system will then be used for our research and education immediately after the startup in October 2022 and join other instruments in the clean room as a system for our research and education in the fabrication of micro and nanoscale devices.

Research and Education:
More than thirty students and faculty will use the etching system for their research and education projects each year. The following on-going research projects will first benefit from the installed PlasmaPro 100 Cobra ICP etching system while more new research projects will be completed with using the etching system later.

Development of High-Efficiency Thermoelectric Materials and Integrated Devices for the Application of Power Generation and Solid-State Cooling: The research objective of this project is to use nanofabrication to develop highly-efficient integrated thermoelectric thin film power generators and cooling devices with an extremely high density of thermoelectric elements at nanoscale for high-efficiency thermal-to-electrical energy conversion and solid-state cooling. Nanoscale multilayered thin films such as Bi_2Te_3/Sb_2Te_3, $Bi_2Te_3/Bi_2Te_{3-x}Se_x$, $Si_{1-x}Ge_x/Ge$, $Si_{1-x}Ge_x/Bi_2Te_3$, and $Si_{1-x}Ge_x/Sb_2Te_3$ are used as the thermoelectric (TE) material systems for the fabrication of high-efficiency integrated power generators and cooling devices. Ultra-high-vacuum E-beam/thermal evaporations are used to grow the nanoscale multilayered thin films. The multilayered thin films are prepared to have a periodic structure consisting of alternating layers, where each layer is about 1 to 5 nm thick, and have over 100 layers with a total thickness of about 100 nm to 500 nm. Integrated TE power generators and cooling devices are fabricated with the multilayered thin films using the clean room-based nanofabrication techniques such as UV and e-beam lithography. The integrated TE devices will consist of thousands to millions of TE elements, where each TE element is fabricated with the multilayered thin film as the active layer, and has 20 to 1000 nm by 20 to 1000 nm in dimensions. The plasma etching system is used to etch the dielectric oxide thin films, semiconducting thermoelectric thin films, and metal contact thin films in the fabrication of high-efficiency integrated thermoelectric devices for this project.

Fabrication of Silicon-Based CMOS Devices and Integrated Circuits (ICs): The objective of this project is to design and fabrication silicon-based complementary metal-oxide semiconductor (CMOS) devices and integrated circuits (ICs) such as CMOS ring oscillator and CMOS-based operational amplifier electronic circuits, Four or five senior students design, fabricate, and characterize CMOS devices to perform their one-year senior project each year. They use the ALD system to grow zirconium dioxide (ZrO_2) and hafnium oxide (HfO_2) thin films as the high-κ gate oxide in the fabrication of CMOS devices for achieving excellent electrical property. The plasma etching system is used to etch the dielectric oxide and contact metal thin films in the fabrication of silicon-based CMOS devices for this project.

Development of Wafer-Scale Fabrication of Carbon-Based Integrated Electronic Devices: The goal of this project is to achieve the wafer-scale fabrication of carbon-based integrated electronic devices, including carbon nanotube- and graphene-based integrated electronic devices. The electric field directed dielectrophoresis method is used to deposit and align ultra-dense carbon nanotubes, and semiconductor materials are used to replace metals as the source/drain contacts for achieving the wafer-scale fabrication of carbon nanotube-based integrated electronic devices. Nanoscale electrodes with electrical fields applied between the electrodes are used to grow nanostructured carbon thin films and graphene with the ultra-high-vacuum e-beam/thermal evaporation for achieving the wafer-scale fabrication of graphene integrated electronic devices. The plasma etching system is used to etch the dielectric oxide, carbon, and metal contact thin films in the fabrication of carbon-based nanoelectronic circuits for the project.

Biological Synthesis of Nanoparticles for the Application of Bioengineering and Biotechnology in Army: The research objective of this project is to genetically engineer E. coli cells to simplify the processes of nanoparticle biosynthesis in bacteria and help scale up to large scale nanoparticle production for the biomedical and bioengineering application in Army. Specific goals include: 1) Clone or synthesize genes that have been implicated in nanoparticle synthesis in various bacteria and transform the genes into E. coli host cells; 2) Construct fosmid libraries that contain large fragment of genomic DNA from bacteria known to be able to synthesize nanoparticles and transform these libraries into E. coli cells; 3) Examine the ability of these engineered E. coli cells to resist heavy metal ions and synthesize various nanoparticles; 4) Characterize the properties of nanoparticles using different methods; 5) Optimize the conditions that allow the engineered E. coli cells to produce monodisperse, morphologically uniform nanoparticles at large scale; 6) analyze and measure the spectroscopic and optical properties of the biosynthesized nanoparticles and determine size/shape-property relationships; and 7) Investigate the interaction of biosynthesized nanoparticles with other materials and explore the fabrication of nanoparticle-based nanostructures and devices. The plasma etching system is used to fabricate the nanoscale electrodes and the devices in the fabrication of nanoparticle-based sensors and devices for this project.

Development of two-dimensional (2D) molybdenum disulfide (MoS_2) and molybdenum selenium ($MoSe_2$) thin-film nanomaterials and nanoelectronic devices: The research objective of this project is to grow two-dimensional (2D) molybdenum disulfide (MoS_2) and molybdenum selenium ($MoSe_2$) nanomaterials using plasma-enhanced atomic layer deposition (PE-ALD) and fabricate 2D nanomaterial-based nanoelectronic devices. Two-dimensional (2D) molybdenum disulfide (MoS_2) and molybdenum selenium ($MoSe_2$) nanolayered thin-film materials will be grown using the plasma-enhanced atomic layer deposition method in this project. The 2D molybdenum disulfide (MoS_2) and molybdenum selenium ($MoSe_2$) nanomaterials will then be used as the active channel material to fabricate field-effect transistors (FETs) and integrated electronic circuits such as inverters and oscillators using the cleanroom-based micro and nanofabrication techniques. The plasma etching system is used to etch the 2D nanomaterials and the dielectric thin films in the fabrication of 2D MoS_2 and $MoSe_2$ nanoelectronic devices for this project.

The primary educational goal of this project is to integrate the research objectives to enhance the educational experiences of students. Both graduate and undergraduate students will be trained to operate the systems and be mentored to perform research in nanofabrication in the project.

The PlasmaPro 100 Cobra ICP etching system will greatly advance research of interest to DoD. The research which is being conducted using the installed systems resonates with the mission of the DoD, where research programs focus on the development and understanding of nanoscale materials that address the Nation Defense's challenges in energy and electronics. The nanostructured TE materials and devices could be excellent candidates for the application of high-efficiency power generation with lighter weight and smaller size in Defense, while the 2D nanoelectronic devices and circuits would be excellent candidate for the application of future nanoelectronics with higher speed and lower power in Defense. DOD/ARO has greatly supported us to develop the AAMU fabrication clean room in the past ten years, which has now become one of the best micro and nanofabrication facilities in Alabama. We will use the facility to do collaboration research with the scientists, researchers and engineers at ARO and ARL, and do our best to support the DOD research and to appreciate the DOD/ARO support.

In summary, the project has been completed successfully as proposed. An advanced PlasmaPro 100 Cobra ICP etching system has been purchased with the DoD/ARO funding and installed in the Clean Room in the Engineering building at the Alabama A&M University. The installed plasma etching system will support more than thirty students and faculty for their education and research projects each year. The addition of the PlasmaPro 100 Cobra ICP etching system to our 2,500 sq. ft class 1,000 clean-room fabrication tools greatly propels the nanoscale science and technology at AAMU to new frontiers.

Figure 1. The PlasmaPro 100 Cobra ICP etching system installed in the clean room in the Engineering building at the Alabama A&M University.

Figure 2. The backside of the installed PlasmaPro 100 Cobra ICP etching system which are connected with power, water, compressed air, process gas, nitrogen gas, helium gas, chiller, and vacuum.

Figure 3. The installed process gas box of the PlasmaPro 100 Cobra ICP etching system in the clean room, which are connected with seven etching process gases including argon (Ar), oxygen (O_2), chlorine (Cl_2), sulfur hexafluoride (SF_6), octafluorocyclobutane (C_4F_8), boron trichloride (BCl_3), and Trifluoromethane (CHF_3).

Figure 4. The chiller of the PlasmaPro 100 Cobra ICP etching system in the clean room.