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RPPR Final Report
as of 09-Jan-2019

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Major Goals: The major goal of this project is to acquire a state-of-the-art KJLC 150-LX atomic layer deposition (ALD) system to enable researchers at the Alabama A&M University (AAMU) to fabricate devices at the micro- and nanoscales. The capability of fabricating materials and devices at nanoscale is critical not only for the advancement of science and technology, but also for the training of the future scientific workforce. One of the key capabilities for micro and nanofabrication is the ability to grow nanoscale thin-film materials. Physical vapor deposition (PVD) and chemical vapor deposition (CVD) are two major methods to grow thin-film materials. Our PVD thin-film deposition capability is excellent: we have both sputtering deposition system and thermal/e-beam evaporation system for physical vapor deposition (PVD) of thin-film materials. However, we didn’t have any chemical vapor deposition (CVD) system in our clean room. CVD has advantage over PVD such as having higher film quality and better film conformity, which are critical in the fabrication at nanoscales. The atomic layer deposition (ALD) is chemical vapor deposition (CVD). The requested atomic layer deposition system is necessary companion instruments to our 2,500 sq. ft class 1,000 clean-room fabrication tools. The acquisition of the ALD system would propel nanoscale science and technology at AAMU serving northern Alabama to new frontiers.

Accomplishments: An advanced ALD-150LX atomic layer deposition system, which is manufactured by the Kurt J. Lesker Company, 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 project has been completed successfully as proposed.

After the project was awarded by DOD/ARO in September 2017, the Purchasing Office at Alabama A&M University conducted a bid process for purchasing the ALD system. The Kurt J. Lesker Company was selected and awarded for manufacturing the system. Because we received an education discount for the ALD system from the Kurt J. Lesker Company, we had a surplus funding of about $40,000 for purchasing a multi-pocket electron beam source from the Ferrotec Corporation and a Haskris R175 refrigerated water re-circulating system from the Haskris Company to upgrade the e-beam evaporation system, and purchasing an Elix-20 water purification system from the EMD Millipore Corporation to replace a 15-year-old water purification system, which are very important in the fabrication of micro and nanoscale devices and need to be upgraded in our Clean Room. The purchase order for purchasing the ALD-150LX system was issued to the Kurt J. Lesker Company by AAMU in January 2018. The system was then manufactured by the Kurt J. Lesker Company, and was received by AAMU in June 2018. The installation of the system in the AAMU-EE Clean Room was completed in July 2018, and the startup of the system and operation training were done by the engineer from the Kurt J. Lesker Company at the end of July 2018. The purchase order for purchasing the water purification system was issued to the EMD Millipore Company by AAMU in May 2018. The system was received by AAMU in June 2018. The installation and startup of the water purification system in the AAMU-EE Clean Room was completed by the engineer from the EMD Millipore Company in June 2018. The purchase order for purchasing the Haskris R175 refrigerated water re-circulating system was issued to the Haskris Company in May 2018. The system was manufactured by the Haskris Company, and was received by AAMU in July 2018. The installation of the water re-circulation system with the e-beam evaporation in the AAMU-EE Clean Room was completed by the PI in July 2018. The purchase order for purchasing the multi-pocket electron beam source was issued to the Ferrotec Corporation in August 2018. The e-beam source was manufactured by the Ferrotec Company, and was received by AAMU in September 2018. The installation of the e-beam source with the e-beam evaporation in the AAMU-EE Clean Room was completed by the PI in September 2018. Since installation and training of the ALD system, the system has been frequently used by the students and researchers at AAMU for research and education, and has become a very busy system in the AAMU cleanroom fabrication facility. The e-beam evaporation system and water purification systems are also very busy systems, and are used by our students and researchers for research and education every day. The instruments have run very well since being installed in the clean room, and have greatly contributed to our research and education in the fabrication of micro and nanoscale devices together with the other existing cleanroom fabrication facility.

Training Opportunities: 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 (totally 15 students) have been trained to operate the KJLC ALD-150LX system and been mentored to perform research in nanofabrication in the project. The ALD system significantly increases opportunities for AAMU students who will become tomorrow's researchers in government, academia, and industries to perform research and to be trained with nanofabrication in their pursuit of academic excellence.

Results Dissemination: The research results in the project have been reported in the AVS 65th international Symposium and Exhibition in Long Beach, CA in October 2018 and in the annual MRS meeting in Boston, MA in November 2018. Some of research results have been published in the Journal of Microelecctronic Engineering (Titled as: the fabrication of nanoscale Bi2Te3/Sb2Te3 multilayer thin film-based
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Authors: Z. Xiao, K. Kisslinger, E. Dimasi, J. Kimbrough
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Abstract: In this paper, we report our method of fabricating nanoscale multilayered Bi2Te3/Sb2Te3 thin film-based integrated thermoelectric devices, and detail the voltage and power produced by the device. The multilayered Bi2Te3/Sb2Te3 thin film was grown via e-beam evaporation; it had 20 alternating Bi2Te3- and Sb2Te3-layers, each layer being 1.5 nm thick. We characterized the film using high-resolution transmission electron microscopy (HRTEM), revealing its excellent cross-sectional structure without any obvious interface defects. The Bi2Te3/Sb2Te3 multilayer films were investigated by synchrotron X-ray scattering. An integrated device including 128×256 thermoelectric elements was fabricated from the multilayered film. An open-circuit voltage of 51 mV and a maximum power of 21 nW were produced from this 30 nm-thick Bi2Te3/Sb2Te3 multilayer TE device, and a temperature gradient of about 0.5 K/?m was established across the multilayered film.
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RPPR Final Report
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Final Report for the DoD/ARO Project (W911NF-17-1-0474): Acquisition of an Advanced Atomic Layer Deposition 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 atomic layer deposition (ALD) 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 KJLC ALD-150LX atomic layer deposition (ALD) system, which is manufactured by the Kurt J. Lesker 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 September 2017, the Purchasing Office at Alabama A&M University conducted a bid process for purchasing the ALD system. The Kurt J. Lesker Company was selected and awarded for manufacturing the system. Because we received an education discount for the ALD system from the Kurt J. Lesker Company, we had a surplus funding of about $40,000 for purchasing a multi-pocket electron beam source from the Ferrotec Corporation and a Haskris R175 refrigerated water re-circulating system from the Haskris Company to upgrade the e-beam evaporation system, and purchasing an Elix-20 water purification system from the EMD Millipore Corporation to replace a 15-year-old water purification system. Both the e-beam evaporation system and water purification system are important in the fabrication of micro and nanoscale devices and need to be upgraded in our Clean Room. The purchase order for purchasing the ALD-150LX system was issued to the Kurt J. Lesker Company by AAMU in January 2018. The system was then manufactured by the Kurt J. Lesker Company, and was received by AAMU in June 2018. The installation of the system in the AAMU-EE Clean Room was completed in July 2018, and the startup of the system and operation training were done by the engineer from the Kurt J. Lesker Company at the end of July 2018. The purchase order for purchasing the water purification system was issued to the EMD Millipore Company by AAMU in May 2018. The system was received by AAMU in June 2018. The installation and startup of the water purification system in the AAMU-EE Clean Room was completed by the engineer from the EMD Millipore Company in June 2018. The purchase order for purchasing the Haskris R175 refrigerated water re-circulating system was issued to the Haskris Company in May 2018. The system was manufactured by the Haskris Company, and was received by AAMU in July 2018. The installation of the water re-circulation system with the e-beam evaporation in the AAMU-EE Clean Room was completed by the PI in July 2018. The purchase order for purchasing the multi-pocket electron beam source was issued to the Ferrotec Corporation in August 2018. The e-beam source was manufactured by the Ferrotec Company, and was received by AAMU in September 2018. The installation of the e-beam source with the e-beam evaporation in the AAMU-EE Clean Room was completed by the PI in September 2018. Since installation and training of the ALD system, the system has been frequently used by the students and researchers at AAMU for research and education and has become a busy system in the AAMU cleanroom fabrication facility. The e-beam evaporation system and water purification systems are also very busy systems and are used by our students and researchers for research and education every day. The instruments have run very well since installation and have greatly contributed to our research and education in the fabrication of micro and nanoscale devices together with other existing cleanroom fabrication facility.

Figures 1, 2, and 3 show the KJLC ALD-150LX atomic layer deposition (ALD) system which has been installed in the cleanroom fabrication facility in the Engineering building at the Alabama A&M University. Figures 4 and 5 shows the Elix-20 water purification system and the Haskris R175 refrigerated water re-circulating system in the clean room. Figure 6 shows the e-beam evaporation system upgraded with a four-pocket electron-beam source in the clean room.

The KJLC ALD-150LX system is a single wafer ALD system incorporating automated control and can be used with up to 150mm substrates. Key features of the ALD system include remote plasma capability, standard analytical ports for in-situ real-time analysis, fast cycle times, as well as the ability to integrate the ALD tool into a multi-tool configuration. The ALD system incorporates a vertical flow process utilizing an inert gas barrier to encapsulate and focus reactants onto substrate surface thereby minimizing unwanted side-wall reactions. Benefits include shorter cycle times and efficient reactant utilization. The significant system features for the ALD-150LX system, EMD Elix-20 water purification system, Ferrotec multi-pocket e-beam source, and Haskris R175 refrigerated water re-circulation system are summarized below.

The KJLC ALD-150LX system has the following features and functions:

Process chamber

- Heated stainless steel, perpendicular flow reactor chamber for efficient vapor and gas delivery.
- Five independent precursor lines – four independently temperature-controlled vapor inputs and one plasma with reactant manifold.
- Front-side wafer loading port.
- Top mounted vapor delivery system.
- Temperature controlled reactant inputs.
- Stainless steel substrate heater stage with heater element capable of operation to 500 °C.
- Temperature uniformity across 150mm Si substrate <+/- 3%.
- Pin lift mechanism effects substrate transfer.
- Chamber heating to 150°C prevents unwanted film deposition on chamber walls.

Vacuum pumping

- Dry pump with 70 cfm chemical series water-cooled rotary screw pump.
- Stainless steel discharge silencer and inlet flange adapter.
- Nitrogen regulating valve, pressure gauge and flow meter.
- Typical process pressure around 1 Torr.

Precursor inlet

- Heated high vapor pressure (HVP) precursor delivery module (for liquid or solids).

- Carrier gas mass flow controller (MFC), high-speed ALD valve, and shut off valve.
- Reactant delivery lines are heated to 200 °C.
- Input is introduced to chamber through a top mounted dispersion plate for uniform distribution.

Remote plasma source

- Remote Plasma Source with 1kW RF power supply.
- Top mounted 13.56 MHz inductively coupled remote plasma source.
- Cylindrical quartz plasma tube.
- Helical inductive coil geometry.
- Includes reactant gas input for plasma gas delivery.

In-situ ellipsometer

- FS-1 Multi-Wavelength Ellipsometer System.
- 4-Wavelength LED Light Source Unit.
- 465, 525, 580, and 635 nm Wavelengths.
- High Reliability Ellipsometric Detector Unit – No Moving Parts.
- Compact Light Source & Detector Designed for In-Situ Integration.

Residual gas analyzer (RGA)

- Differentially pumped.
- 300 AMU stand alone system with operation and analysis software.

Load lock

- Load lock connects directly to deposition chamber via a slit valve. Substrates are transferred to the load lock from ALD chamber using a linear rack and pinion transfer mechanism which is mounted directly to the load lock. The load lock is turbo pumped and has a base pressure capability of 5 x 10-7 Torr (clean and dry). Substrates are manually loaded into the load lock and transferred to the ALD chamber.
- High vacuum slit valve between ALD chamber.
- Dedicated 210 l/s turbo pump, isolation gate valve with appropriate dry roughing pump (vacuum level 5 x10-7 Torr or better – clean and dry).
- Wide range pressure gauging.
- A pin-lift mechanism effects substrate transfer.
- Motor assisted, operator supervised and controlled substrate transfer using a linear rack and pinion transfer probe mounted directly to the load lock.

KJLC eKLipse advanced control software package

- User Interface via .NET application run on Windows 10 PC platform.
- Standalone Real Time Controller (RTC) executes equipment automation.
- Fully customizable recipe control and process automation.
- Programming/control via a keyboard/touch pad or pop-up window on touch screen.
- Four standard user security levels with user access assignable to controls via user security Level.

The EMD Elix-20 water purification system has the following features and functions:

- Water Resistivity: > 5 MegOhm-cm
- Flow Rate: 20 L/h.
- Can be integrated into a centralized system, providing total control of all parameters within the system itself as well as within the external pure water distribution loop.

The multi-pocket electron beam source has the following features and functions:

- KL-6 four-pocket e-beam source with optional straight clam.
- EV M-t top cover.

The Haskris R175 refrigerated water re-circulating system has the following features and functions:

- Supply water temperature range: 55 to 90 °F.
- Thermostat temperature control.
- Cycling refrigeration design.
- Hermetically sealed compressor.
- Horizontal air discharge.
- Regenerative turbine pump with robust seal and bypass relief valve.
- Integrated fluid tank (non-pressurized) with fluid level switch.
- Alternate pump to provide 8 GPM at 60 psi.

System Installation:

The ALD-150LX system, the EMD Elix-20 water purification system, and the Haskris R175 refrigerated water re-circulating system have been installed in the Clean Room fabrication facility in the Engineering building at the Alabama A&M University. The PVD 75 e-beam evaporation system upgraded with the multi-pocket e-beam source is also in the clean room. 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 systems, 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 ALD-150LX system was shipped to the Clean Room, it was installed and connected with the compressed air, water, gases, and electricity by the engineers from a local company (Engineered Maintenance Services Company) with consultation to the manufacturing company.

The EMD Elix-20 water purification system was installed by the engineer from the EMD Millipore Company. The Haskris R175 refrigerated water re-circulation system and the Ferrotec multi-pocket e-beam source were installed by the PI.

System Start Up and Operation Training:

The ALD was started up by the engineer from the Kurt J. Lesker Company. After the ALD system was appropriately installed in the Clean Room and meets the installation requirements, an engineer from the Kurt J. Lesker Company came to AAMU for the system start up and operation training. During the start up and training, the system was started up successfully and all the functions were tested by the engineer. The specifications for the system meet the pre-designed requirements. The PI and students attended the training. The PI has also trained students and researchers to use the upgraded e-beam evaporation systems for their research. The installed ALD system functions very well together with other instruments in the clean room as a system for research and education in the fabrication of micro and nanoscale devices.

Research and Education:

After the ALD system was successfully installed and started up in the Clean Room, it has been used for our research and training students immediately, together with other cleanroom facility. The system is currently used for growth of various oxide thin film materials such as aluminum oxide (Al_2O_3), hafnium oxide (HfO_2), and zirconium oxide (ZrO_2) in the fabrication of carbon-based nanoelectronic circuits, high-efficiency thermoelectric devices, and CMOS integrated circuits (ICs). More than twenty students and faculty use the systems for their research and education projects each year.

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 fabricated nanoscale multilayered supperlattice thin films and integrated TE devices are further modified with the innovative rapid cooling and high-energy (MeV) ion beam bombardment for achieving higher thermoelectric figure of merit. The fabrication of integrated TE devices with an extremely high density of TE elements are specifically explored using the UV and e-beam lithography in this project. The dependence of efficiency on the density of TE elements at nanoscale will be investigated and found, and high-efficiency integrated TE devices are fabricated and achieved for thermal-to-electrical energy conversion and solid-state cooling. Figure 7(a) shows the arrangement of the multilayered film with alternating Bi_2Te_3 and Sb_2Te_3 layers; Figure 7(b) shows the high-resolution TEM (HRTEM) image of a multilayer film grown by e-beam evaporation; Figure 7(c) shows the selected electron diffraction pattern (SAED) of the grow multilayer film; Figure 7(d) shows the working principle for an integrated TE device; Figure 7(e) shows the SEM image of a fabricated integrated TE device consisting of large number of elements, where Bi_2Te_3/Sb_2Te_3 (p-type) and Bi_2Te_3/Bi_2Te_2Se (n-type) multilayer thin films are fabricated as the two elements.

Development of Nanostructured Metal Plasmonic Electrodes as Enhanced Solar Cell Transparent Conductors: The objective of this project is to develop nanostructured metal plasmonic electrodes as enhanced solar cell transparent conductors for the application of high-efficiency light-to-electrical energy conversion. Photovoltaic (PV) cells have the potential to harness the nearly limitless source of clean energy provided by the sun. However, their widespread adoption in replacing conventional dirty energy sources has to date been limited by high costs and low energy conversion efficiencies. Consequently, there are presently considerable worldwide research and development efforts targeting improved PV device efficiency using low-cost materials and fabrication methods. In this project, our research focuses on using surface plasmons in solar cells in a unique new way, combining their ability to improve light coupling to the active semiconductor material and at the same time provide an improved transparent conducting device electrode. Solar device electrode materials must strike a balance between transparency (to allow for light coupling into the device) and electrical conductivity (to provide for efficient charge collection). Most device architectures use materials such as indium tin oxide (ITO) for this purpose. Our approach will improve the electrical properties of the transparent contact (by reducing ohmic losses) while simultaneously enhancing light coupling over a spectral range optimized for the device active material. We are currently performing fabrication of more traditional planar silicon-based PV devices. Ultimately, we will incorporate the nanostructured plasmonic electrode concepts into the fabrication of these technologically relevant solar cells in order to enhance their power conversion efficiency.

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. Figure 8(a) shows the schematic of a CMOS-based operational transconductance amplifier (OTA) circuit; Figure 8(b) shows the SEM micrograph of a fabricated CMOS OTA circuit.

Wafer-Scale Fabrication of Carbon-Based Nanoelectronic Circuits: The research objective of this project is to develop wafer-scale fabrication of carbon-based integrated electronic devices. A major problem in the realization of carbon nanotube devices is the difficulty to position and assemble carbon nanotubes in a controlled way. In this project, an unconventional approach, based on the electric field directed dielectrophoresis method is used to deposit and align ultra-dense carbon nanotubes. The poor yield of functional devices is another major problem, because currently there is no effective way to separate the metallic carbon nanotubes from the semiconducting tubes, and the metallic tubes unavoidably exist in fabricated transistors, resulting in poor electrical properties. In the project, semiconductor materials are used to replace metals as the source/drain contacts for solving the problem of poor yield. Another major research effort in this project is to use electrical fields together with nanoscale electrodes to grow nanostructured carbon thin films and graphene using the e-beam/thermal evaporation. The research will make the wafer-scale fabrication of graphene devices. Figure 9 (a) shows the schematic of a setup for growth of graphene between a pair of nanscale Cu-Ni alloy electrodes with applying electric fields; Figure 9 (b) shows the high-resolution TEM (HR-TEM) image of a graphene film grown with the setup in Figure 9 (a); Figure 9 (c) shows the schematic of a graphene field-effect transistor (GFET) with semiconductor as the source/drain contact; Figure 9 (d) shows the SEM image of a fabricated GFET device.

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 have been trained to operate the systems and been mentored to perform research in nanofabrication in the project. Figures 10 and 11 show the students' research activities using the ALD system and the upgraded e-beam evaporation system in the clean room. Figures 12, 13, and 14 show the students' presentations for their research in the AVS 65th international Symposium and Exhibition in Long Beach, CA in October 2018 and in the annual MRS meeting in Boston, MA in November 2018. The newly-installed ALD system and upgraded e-beam evaporation system significantly increase opportunities for AAMU students who will become tomorrow's researchers in government, academia, and industries to perform research and to be trained with nanofabrication in their pursuit of academic excellence. High school students will also be trained and mentored to conduct summer research in the Clean Room using the installed ALD system together with the other existing clean room facility, starting from the summer of 2019.

The ALD systems 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 carbon-based nanoelectronic 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 KJLC ALD-150LX atomic layer deposition (ALD) 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 ALD system has been tested and found to meet all the technical specifications and pre-designed requirements. In addition, the e-beam evaporation has been upgraded with a new four-pocket e-beam source; a new Elix-20 water purification system and a Haskris R175 refrigerated water re-circulating system have been installed in the clean room through the project. The installed ALD system has been used for our research in the fabrication of micro and nanoscale devices. The addition of the instruments 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 KJLC ALD-150LX atomic layer deposition (ALD) system.

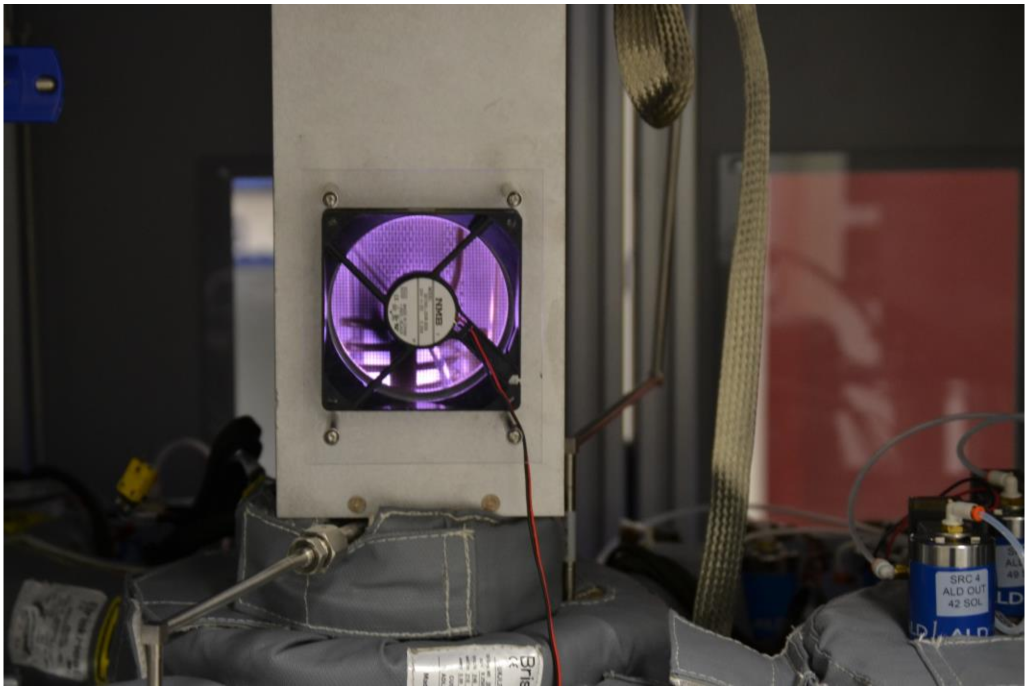


Figure 2. The ICP plasma in the ALD system.

Figure 3. One of the control and operation software interfaces for the ALD system.

Figure 4. The EMD Millipore Elix-20 water purification system.

Figure 5. The Haskris R175 refrigerated water re-circulation system.

Figure 6. The upgraded e-beam evaporation system with a new four-pocket e-beam source.

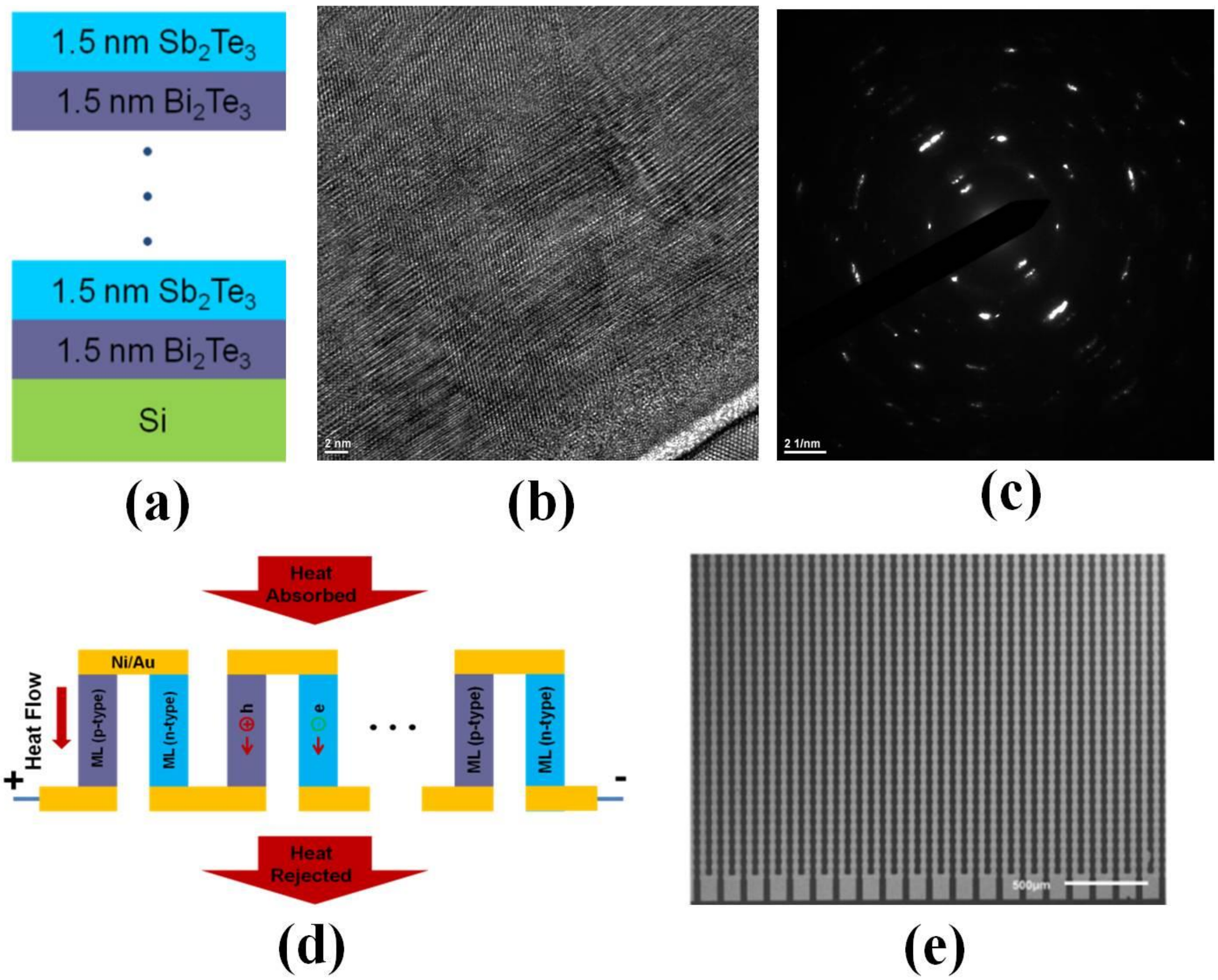
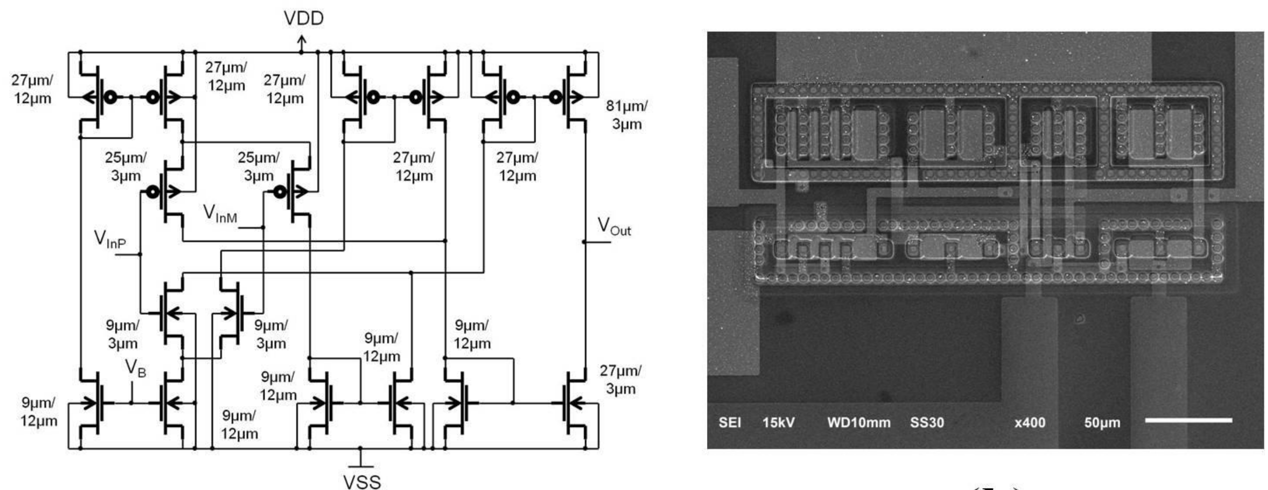


Figure 7. (a) Multilayered film with alternating Bi_2Te_3 and Sb_2Te_3 layers: (b) High-resolution TEM (HRTEM) image of a multilayer film grown by e-beam evaporation; (c) The selected electron diffraction pattern (SAED) of the grow multilayer film; (d) Working principle for an integrated TE device; (e) SEM image of a fabricated integrated TE device consisting of large number of elements.



(a) (b)

Figure 8. (a) Schematic of a CMOS-based operational transconductance amplifier (OTA) circuit; (b) SEM micrograph of a fabricated CMOS OTA circuit.

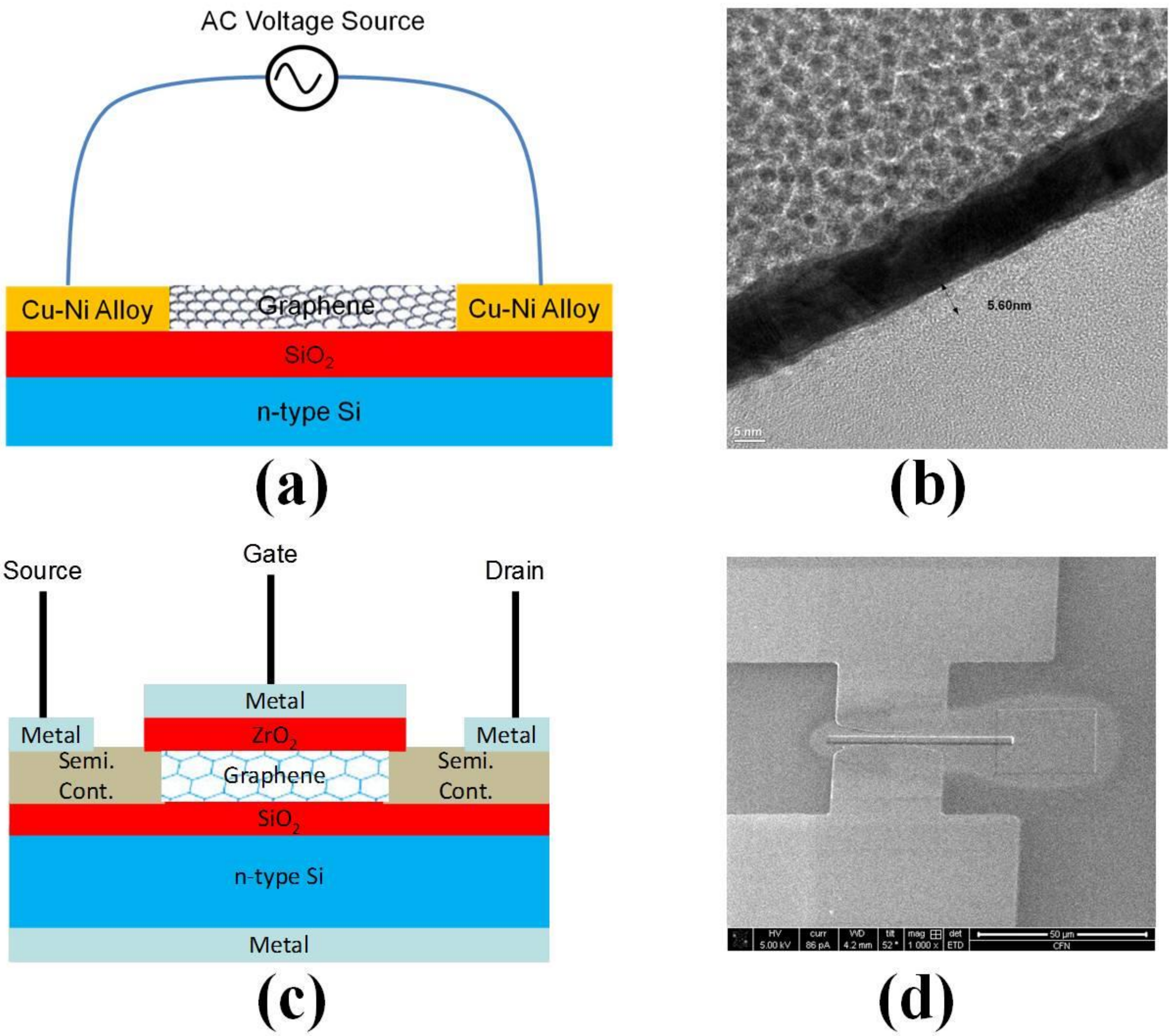


Figure 9. (a) Schematic of a setup for growth of graphene between a pair of nanscale Cu-Ni alloy electrodes with applying electric fields; (b) High-resolution TEM (HR-TEM) image of a graphene film grown with the setup in (a); (c) Schematic of a graphene field-effect transistor (GFET) with semiconductor as the source/drain contact; (d) SEM image of a fabricated GFET device.

Figure 10. Students using the ALD system for their research in the AAMU-EE Clean Room.

Figure 11. Student operating the e-beam evaporation system for his research in the clean room.

Figure 12. Joevonte Kimbrough (Graduate student) and Sam Chance III (Undergraduate student) making presentations for their research in the AVS 65th international Symposium and Exhibition in Long Beach, CA in October 2018.

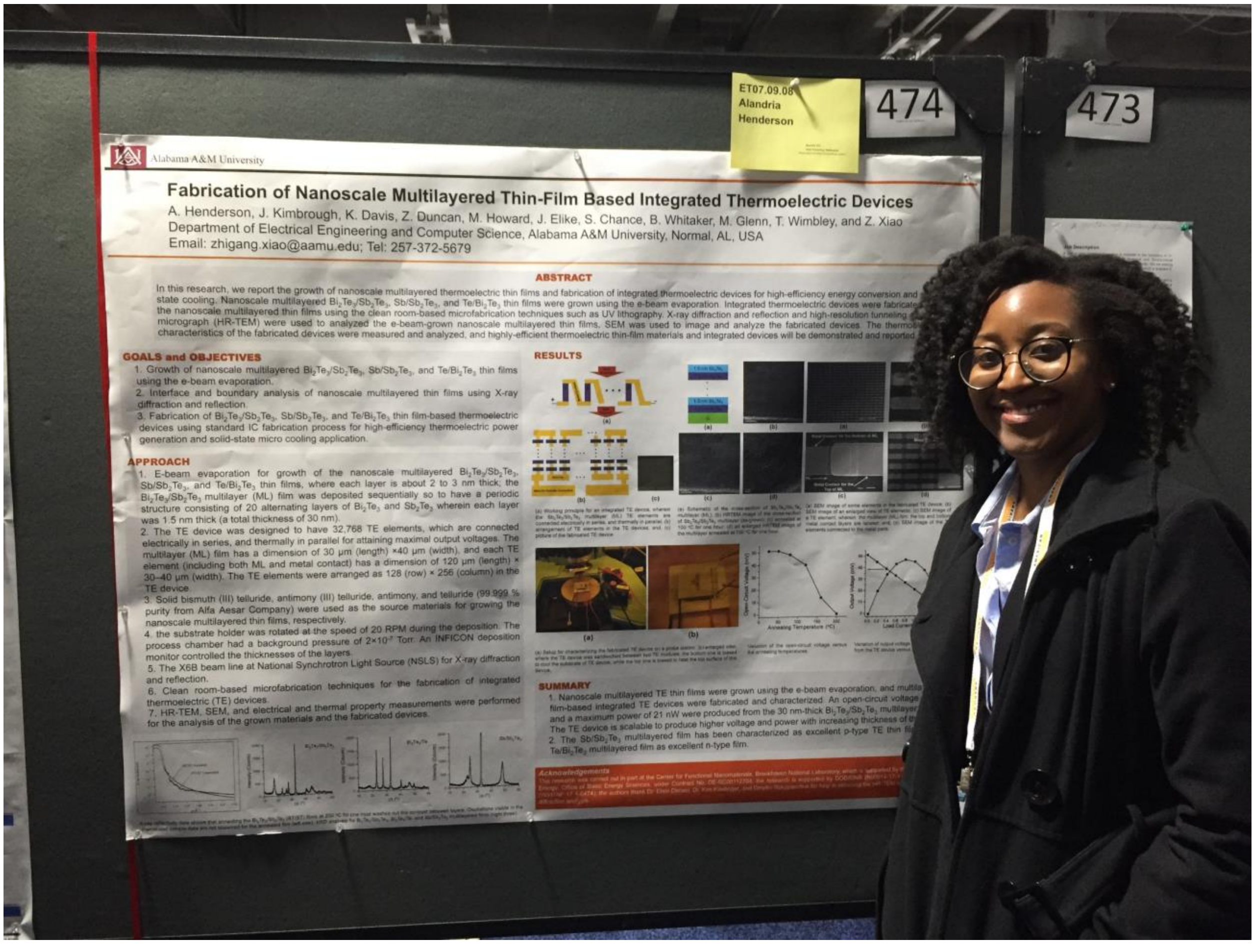


Figure 13. Alandria Henderson (Undergraduate student) making presentations for her research in the annual MRS meeting in Boston, MA in November 2018.

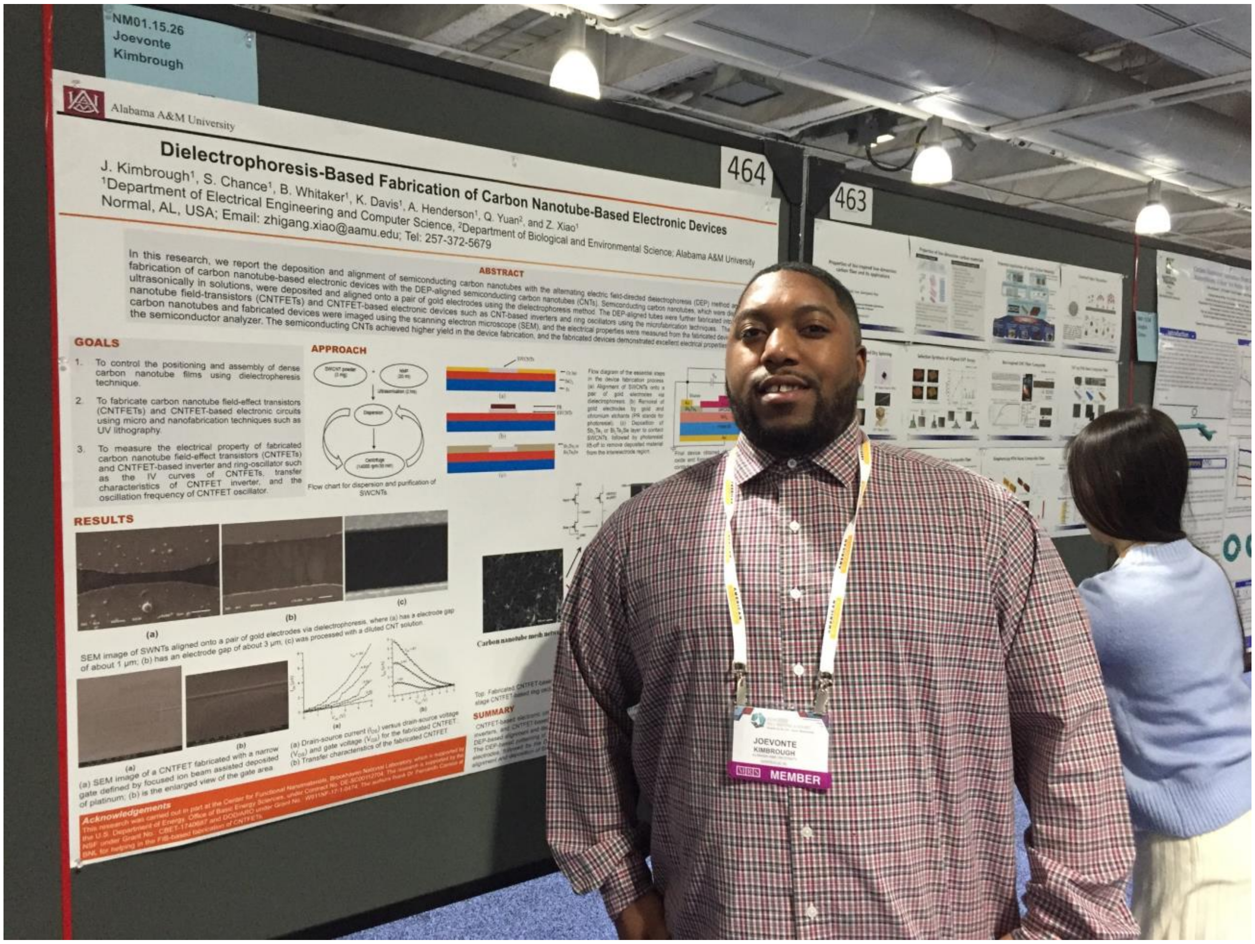


Figure 14. Joevonte Kimbrough (Graduate student) making presentation for his research in the annual MRS meeting in Boston, MA in November 2018.

Poster presented in the AVS 65th international Symposium and Exhibition in Long Beach, CA in October 2018

Alabama A&M University

Comparison of Hafnium Oxide and Zirconium Oxide for Fabricating Electronic Devices

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ABSTRACT

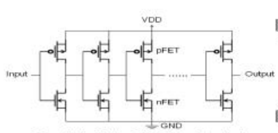
Thin films of hafnium dioxide (HfO_2) and zirconium oxide (ZrO_2) are used widely as the gate oxide in fabricating integrated circuits (ICs) because of their high dielectric constants. In this research, we report the growth of hafnium dioxide (HfO_2) and zirconium oxide (ZrO_2) thin film using atomic layer deposition (ALD), and the fabrication of complementary metal-oxide semiconductor (CMOS) integrated circuits using the HfO_2 and ZrO_2 thin films as the gate oxide. 15-stage CMOS ring oscillators and operational transcoductance amplifiers (OTA) were fabricated to test the quality of the HfO_2 thin film as the gate oxide. Excellent rail-to-rail oscillation waveforms and DC and AC electrical property were obtained from the fabricated devices, denoting that the HfO_2 and ZrO_2 thin films functioned very well as the gate oxide. It was also found that the ring oscillator with ZrO_2 as the gate oxide had a higher oscillation frequency (about 20% higher) than those with the HfO_2 as the gate oxide.

GOALS

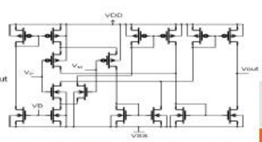
1. To grow hafnium dioxide (HfO_2) and zirconium dioxide (ZrO_2) using atomic layer deposition (ALD) method.
2. To fabricate CMOS-based silicon ring oscillator and OTA integrated circuits.
3. To measure the electrical property of fabricated integrated devices.

APPROACH

1. Plasma-enhanced ALD system (Kurt J. Lesker) was used to grow the HfO2 and ZrO2 thin films.
2. Tetrakis(dimethylamino)hafnium ($C_{16}H_{48}Hf_2N_8$) and Tetrakis(dimethylamino)zirconium ($Zr[N(CH_3)_2]_4$) from Strem were used as the precursors for the growth of HfO2 and ZrO2 thin films, respectively.
3. Argon/oxygen plasma was used to enhance the chemical reaction; the substrate was heated at 300 °C; the films were prepared as about 30 nm thick (500 cycles).
4. 3-inch-diameter n-type (001) silicon wafer was used in making the *complementary metal-oxide semiconductor* (CMOS) devices.
5. The fabrication processes included boron doping for the fabricating the p-well and the source/drain of pFETs; phosphorous doping for the making the source/drain of nFETs; depositing the HfO_2 thin film for the gate oxide; and, depositing the aluminum (Al)/chromium (Cr)/gold (Au) thin films for the metal contact. Thermal diffusion was used for doping both with boron and phosphorous. Ultra-violet (UV) lithography was adopted for patterning in fabricating the device.
6. The electrical property of the fabricated devices was measured using Agilent precision source/measurement unit and Agilent mixed-signal oscilloscope.

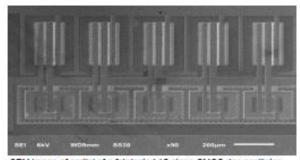


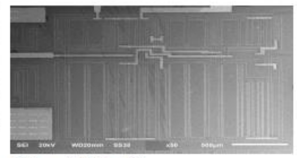
Schematic of a 15-stage CMOS ring oscillator circuit.

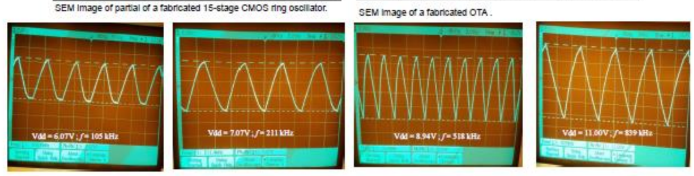


Schematic of a rail-to-rail operational transconductor amplifier (OTA) circuit.

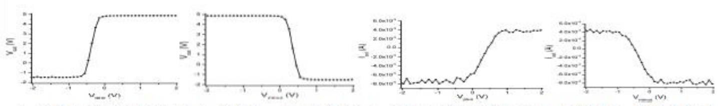
RESULTS


SEM image of partial of a fabricated 15-stage CMOS ring oscillator.


SEM image of a fabricated OTA.



Output frequency versus various applied power voltages (Vdd), which was measured from a 15-stage CMOS ring oscillator fabricated with HfO2 as the gate oxide with a oscilloscope.


V_{out} versus V_{plus} measured from the OTA circuit. V_{out} versus V_{minus} measured fro the OTA circuit. I_{out} versus V_{plus} measured from the OTA circuit. I_{out} versus V_{minus} measured from the OTA circuit.

Summary The CMOS ring oscillator and OTA fabricated with the HfO_2 and ZrO_2 thin film as the gate oxide demonstrated satisfactory electrical properties, indicating that the HfO_2 and ZrO_2 thin films can be an excellent gate oxide for applications in CMOS integrated circuits (ICs).

Acknowledgements
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Poster presented in the annual MRS meeting in Boston, MA in November 2018

Alabama A&M University

Fabrication of Nanoscale Multilayered Thin-Film Based Integrated Thermoelectric Devices

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ABSTRACT

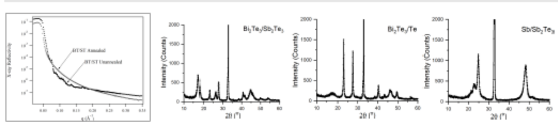
In this research, we report the growth of nanoscale multilayered thermoelectric thin films and fabrication of integrated thermoelectric devices for high-efficiency energy conversion and solid-state cooling. Nanoscale multilayered Bi_2Te_3/Sb_2Te_3, Sb/Sb_2Te_3, and Te/Bi_2Te_3 thin films were grown using the e-beam evaporation. Integrated thermoelectric devices were fabricated with the nanoscale multilayered thin films using the clean room-based microfabrication techniques such as UV lithography. X-ray diffraction and reflection and high-resolution tunneling electron micrograph (HR-TEM) were used to analyzed the e-beam-grown nanoscale multilayered thin films. SEM was used to image and analyze the fabricated devices. The thermoelectric characteristics of the fabricated devices were measured and analyzed, and highly-efficient thermoelectric thin-film materials and integrated devices will be demonstrated and reported.

GOALS and OBJECTIVES

1. Growth of nanoscale multilayered Bi_2Te_3/Sb_2Te_3, Sb/Sb_2Te_3, and Te/Bi_2Te_3 thin films using the e-beam evaporation.
2. Interface and boundary analysis of nanoscale multilayered thin films using X-ray diffraction and reflection.
3. Fabrication of Bi_2Te_3/Sb_2Te_3, Sb/Sb_2Te_3, and Te/Bi_2Te_3 thin film-based thermoelectric devices using standard IC fabrication process for high-efficiency thermoelectric power generation and solid-state micro cooling application.

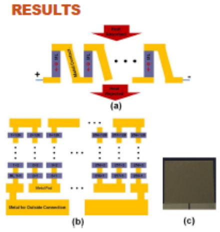
APPROACH

1. E-beam evaporation for growth of the nanoscale multilayered Bi_2Te_3/Sb_2Te_3, Sb/Sb_2Te_3, and Te/Bi_2Te_3 thin films, where each layer is about 2 to 3 nm thick; the Bi_2Te_3/Sb_2Te_3 multilayer (ML) film was deposited sequentially so to have a periodic structure consisting of 20 alternating layers of Bi_2Te_3 and Sb_2Te_3 wherein each layer was 1.5 nm thick (a total thickness of 30 nm).
2. The TE device was designed to have 32,768 TE elements, which are connected electrically in series, and thermally in parallel for attaining maximal output voltages. The multilayer (ML) film has a dimension of 30 μm (length) ×40 μm (width), and each TE element (including both ML and metal contact) has a dimension of 120 μm (length) × 30–40 μm (width). The TE elements were arranged as 128 (row) × 256 (column) in the TE device.
3. Solid bismuth (III) telluride, antimony (III) telluride, antimony, and telluride (99.999 % purity from Alfa Aesar Company) were used as the source materials for growing the nanoscale multilayered thin films, respectively.
4. the substrate holder was rotated at the speed of 20 RPM during the deposition. The process chamber had a background pressure of 2×10^{-7} Torr. An INFICON deposition monitor controlled the thicknesses of the layers.
5. The X6B beam line at National Synchrotron Light Source (NSLS) for X-ray diffraction and reflection.
6. Clean room-based microfabrication techniques for the fabrication of integrated thermoelectric (TE) devices.
7. HR-TEM, SEM, and electrical and thermal property measurements were performed for the analysis of the grown materials and the fabricated devices.

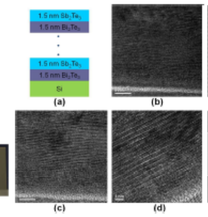


X-ray reflectivity data shows that annealing the Bi_2Te_3/Sb_2Te_3 (BT/ST) films at 200 °C for one hour washes out the contrast between layers. Oscillations visible in the unannealed sample data are not observed for the annealed film (left one); XRD analysis for Bi_2Te_3/Sb_2Te_3, Bi_2Te_3/Te, and Sb/Sb_2Te_3 multilayered films (right three).

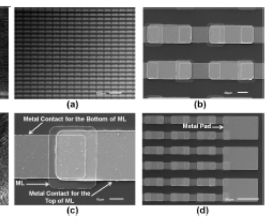
RESULTS



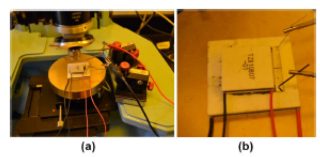
(a) Working principle for an integrated TE device, wherein the Sb_2Te_3/Sb_2Te_3 multilayer (ML) TE elements are connected electrically in series, and thermally in parallel; (b) arrangement of TE elements in the TE devices; and, (c) picture of the fabricated TE device.



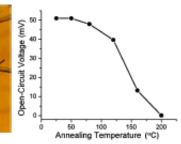
(a) Schematic of the cross-section of Sb_2Te_3/Sb_2Te_3 multilayer (ML); (b) HRTEM image of the cross-section of Sb_2Te_3/Sb_2Te_3 multilayer (as-grown); (c) annealed at 100 °C for one hour; (d) an enlarged HRTEM image of the multilayer annealed at 100 °C for one hour.



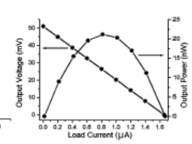
(a) SEM image of some elements in the fabricated TE device; (b) SEM image of an enlarged view of TE elements; (c) SEM image of a TE element, wherein the multilayer (ML) film, the top and bottom metal contact layers are labeled; and, (d) SEM image of the TE elements connected to the metal pads.



(a) Setup for characterizing the fabricated TE device on a probe station; (b) enlarged view, where the TE device was sandwiched between two TE modules; the bottom one is biased to cool the substrate of TE device, while the top one is biased to heat the top surface of this device.



Variation of the open-circuit voltage versus the annealing temperatures.



Variation of output voltage and power produced from the TE device versus the load current.

SUMMARY

1. Nanoscale multilayered TE thin films were grown using the e-beam evaporation, and multilayered thin film-based integrated TE devices were fabricated and characterized. An open-circuit voltage of 51 mV, and a maximum power of 21 nW were produced from the 30 nm-thick Bi_2Te_3/Sb_2Te_3 multilayer TE device. The TE device is scalable to produce higher voltage and power with increasing thickness of the ML.
2. The Sb/Sb_2Te_3 multilayered film has been characterized as excellent p-type TE thin film, while the Te/Bi_2Te_3 multilayered film as excellent n-type film.

Acknowledgements

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Poster presented in the annual MRS meeting in Boston, MA in November 2018