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# RPPR Final Report
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**INVESTIGATOR(S):**


**Name:** Kaveh Heidary Ph.D.
**Email:** kaveh.heidary@aamu.edu
**Phone Number:** 2563725587
**Principal:** N

**Name:** Satilmis Budak
**Email:** satilmis.budak@aamu.edu
**Phone Number:** 2563725894
**Principal:** N

**Name:** Zhigang Xiao
**Email:** zhigang.xiao@aamu.edu
**Phone Number:** 2563725679
**Principal:** Y

Organization: **Alabama A&M University**
Address: 4900 Meridian Street NorthWest, Normal, AL 357620411
Country: USA


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**Title:** Acquisition of an Advanced Mask Aligner and a Spin Coater Unit for Research at Micro and Nanoscale for Energy Harvesting and Nanoelectronics at Alabama A&M University
**Begin Performance Period:** 29-Aug-2016    **End Performance Period:** 28-Aug-2017
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Submitted By: Zhigang Xiao    Email: zhigang.xiao@aamu.edu
Phone: (256) 372-5679
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**STEM Degrees:**    **STEM Participants:** 15

**Major Goals:** The major goal of this project is to acquire a state-of-the-art mask aligner system and a spin coater unit to enable researchers at the Alabama A&M University (AAMU) to fabricate the micro- and nanoscales devices. The capability of fabricating materials and devices at micro and 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 create micro and nanoscale patterns in the device fabrication. UV lithography is normally used for the patterning in the device fabrication, where a spin coater is used to spin photo resist, and then a mask aligner system is required to align the patterns and make the UV light exposure for the device wafer fabrication. The requested mask aligner system and spin coater are necessary companion instruments to our 2,500 sq. ft class 1,000 clean-room fabrication tools. The acquisition of the mask aligner system and spin coater would propel the nanoscale science and technology at AAMU to new frontiers.

**Accomplishments:** An advanced SUSS MA6 Gen4 Pro manual mask aligner, a CEE® Model 200CBX spin/bake unit, and a Venus50XL-HF bench-top plasma cleaning/etching system have 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 ARO in August 2016, we immediately contacted the Suss MicroTec Company and the Brewer Science Company which were proposed as the manufacturers of the two systems in the original proposal, and ordered the SUSS MA6 Gen4 Pro manual mask aligner, the newest model of Suss MA6, and the CEE® Model 200CBX spin/bake unit as proposed. Because we received an education discount for the mask aligner from the Suss MicroTec Company, we had a surplus funding of about $20,000 for purchasing a bench-top plasma cleaning/etching system, which is an important instrument in the fabrication of micro and nanoscale devices and was lacking in our Clean Room. After the approval of the ARO Program Managers, a Venus50XL-HF bench-top plasma cleaning/etching system was ordered from the Plasma

Etch Company. The purchase order for purchasing the mask aligner was issued to the Suss MicroTec Company by AAMU in October 2016. The system was then manufactured by the Suss MicroTec Company, and was received by AAMU in February 2017. The installation of the system in the AAMU-EE Clean Room was completed in March 2017, and the start up of the system and operation training were done by the engineer from the Suss MicroTec Company in March 2017. The purchase order for purchasing the CEE® Model 200CBX spin/bake unit was issued to the Brewer Science Company by AAMU in October 2016. The spin/bake system was manufactured by the Brewer Science Company, and was received by AAMU in December 2016. The installation of the spin/bake system in the AAMU-EE Clean Room was completed in January 2017. The start up of the system and operation training were done by the PI in February 2017. The purchase order for purchasing the Venus50XL-HF bench-top plasma cleaning/etching system was issued to the Plasma Etch Company by AAMU in June 2017. The system was manufactured by the Plasma Etch Company, and was received by AAMU in August 2017. The installation of the plasma cleaning/etching system in the AAMU-EE Clean Room was completed in September 2017. The start up of the system and operation training were done by the PI in September 2017. Since installation and training of the systems, the three systems have been frequently used by the students and researchers at AAMU for research and education, and have become very busy systems in the AAMU cleanroom fabrication facility. The three instruments have run very well in a very stable condition since installation, and have greatly contributed to our research and education in the fabrication of micro and nanoscale devices together with our other cleanroom fabrication facility.

**Training Opportunities:** Graduate and undergraduate students and researchers at AAMU have been trained to operate the SUSS MA6 Gen4 Pro manual mask aligner, the CEE® Model 200CBX spin/bake unit, and the Venus50XL-HF bench-top plasma cleaning/etching system by the engineer from the manufacturers or by the PI.

**Results Dissemination:** One manuscript, which is titled as "The fabrication of nanoscale Bi2Te3/Sb2Te3 multilayer thin film-based thermoelectric power chips", has been submitted to the Journal of Microelectronic Engineering for publication, and is under review.

**Honors and Awards:** Nothing to Report

**Protocol Activity Status:**

**Technology Transfer:** Nothing to Report

**PARTICIPANTS:**

**Participant Type:** PD/PI
**Participant:** Zhigang Xiao
**Person Months Worked:** 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

**Participant Type:** Co PD/PI
**Participant:** Satilmis Budak
**Person Months Worked:** 1.00 **Funding Support:**
Project Contribution:
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International Travel:
National Academy Member: N
Other Collaborators:

**Participant Type:** Co PD/PI
**Participant:** Kaveh Heidary
**Person Months Worked:** 1.00 **Funding Support:**
Project Contribution:

International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

**Final Report for the DoD/ARO Project: Acquisition of an Advanced Mask Aligner and a Spin Coater Unit for Research at Micro and Nanoscale for Energy Harvesting and Nanoelectronics at Alabama A&M University (W911NF-16-1-0554)**

Dr. Zhigang Xiao (PI), Dr. Satilmis Budak (Co-PI), and Dr. Kaveh Heidary (Co-PI)
Department of Electrical Engineering and Computer Science, Alabama A&M University, Normal, AL 35762

The objective of this project is to acquire a state-of-the-art mask aligner system and a spin coater unit to enable researchers at the Alabama A&M University (AAMU) to fabricate the micro- and nanoscales devices. An advanced SUSS MA6 Gen4 Pro manual mask aligner, a CEE® Model 200CBX spin/bake unit, and a Venus50XL-HF bench-top plasma cleaning/etching system have 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 ARO in August 2016, we immediately contacted the Suss MicroTec Company and the Brewer Science Company which were proposed as the manufacturers of the two systems in the original proposal, and ordered the SUSS MA6 Gen4 Pro manual mask aligner, the newest model of Suss MA6, and the CEE® Model 200CBX spin/bake unit as proposed. Because we received an education discount for the mask aligner from the Suss MicroTec Company, we had a surplus funding of about $20,000 for purchasing a bench-top plasma cleaning/etching system, which is an important instrument in the fabrication of micro and nanoscale devices and was lacking in our Clean Room. After the approval of the ARO Program Managers, a Venus50XL-HF bench-top plasma cleaning/etching system was ordered from the Plasma Etch Company. The purchase order for purchasing the mask aligner was issued to the Suss MicroTec Company by AAMU in October 2016. The system was then manufactured by the Suss MicroTec Company, and was received by AAMU in February 2017. The installation of the system in the AAMU-EE Clean Room was completed in March 2017, and the start up of the system and operation training were done by the engineer from the Suss MicroTec Company in March 2017. The purchase order for purchasing the CEE® Model 200CBX spin/bake unit was issued to the Brewer Science Company by AAMU in October 2016. The spin/bake system was manufactured by the Brewer Science Company, and was received by AAMU in December 2016. The installation of the spin/bake system in the AAMU-EE Clean Room was completed in January 2017. The start up of the system and operation training were done by the PI in February 2017. The purchase order for purchasing the Venus50XL-HF bench-top plasma cleaning/etching system was issued to the Plasma Etch Company by AAMU in June 2017. The system was manufactured by the Plasma Etch Company, and was received by AAMU in August 2017. The installation of the plasma cleaning/etching system in the AAMU-EE Clean Room was completed in September 2017. The start up of the system and operation training were done by the PI in September 2017. Since installation and training of the systems, the three systems have been frequently used by the students and researchers at AAMU for research and education, and have become very busy systems in the AAMU cleanroom fabrication facility. The three instruments have run very well in a very stable condition since installation, and have greatly contributed to our research and education in the fabrication of micro and nanoscale devices together with our other cleanroom fabrication facility. Figures 1, 2, 3, 4, and 5 show the SUSS MA6 Gen4 Pro manual mask aligner, the CEE® Model 200CBX spin/bake unit, and the Venus50XL-HF bench-top plasma cleaning/etching system systems, which have been installed in the cleanroom fabrication facility in the Engineering building at the Alabama A&M University.

The SUSS MA6 Gen4 system has mask aligner, windows graphical user interface and recipe management, and can provide an alignment accuracy of 0.25μm and enhanced uniform UV exposure (less than 3% non-uniformity). The CEE Model 200CBX Spin/Bake unit has a spin coater with a spin speed of up to 12,000 RPM and a hot plate with heating temperature of up to 300 $^{o}$C, and is controlled by an onboard windows based PC control. The Venus50XL-HF bench-top plasma cleaning/etching system has 125W and 13.56 MHz high frequency RF power supply and automatic RF matching network, and has two mass flow controllers for precise process gas flow control and gas mixing. The plasma system has PC based control system with Plasma Etch proprietary software for fully automatic system operation. The significant system features for the three systems are summarized below.

**The SUSS MA6 Gen4 Pro manual mask aligner system has the following features and functions:**

- SUSS MA6 Gen4 for precision Top Side Lithography applications
- Equipped for up to 6" diameter semi-spec wafers and wafer pieces
- WINDOWS User Interface
- Alignment Stage with Precision Micrometers
- SUSS M608 Video Split Field Microscope w/ viewing screen and Olympus 5X and 10X Objectives
- Proximity/Contact Mask Holder and Contact Chuck for printing in proximity, hard contact and soft contact modes on 6" diameter wafers
- Contact Mask Holder and Contact Chuck for printing in hard contact and soft contact on wafer pieces 10mm x 10mm, 20mm x 20mm and 2" wafers
- High Resolution Diffraction Reducing Optics for the UV400 wavelength with a 1000W exposure lamp
- SUSS CIC1200 Exposure Lamp Power Supply
- SUSS UV Optometer with 365nm/405nm Probe
- Integrated Vibration Isolation Table
- Manufactured to CE Safety Guidelines
- Manual Video Alignment
- Constant Dose Function

**The CEE Model 200CBX spin/bake unit has the following features and functions:**

- Spin Coater with spin speed of up to 12,000 RPM
- Hot Plate with 300ºC Temperature Capability
- Onboard Windows Based PC Control w/ Full Color 7" Touchscreen Display
- 2.25" Interchangeable Spin Chuck with Base
- Polyethylene Spin Bowl
- 2.25" Interchangeable Spin Chuck with Base

- 3" Handheld Centering Device
- 100MM Handheld Centering Device
- Multi-substrate Configuration - 2", 3", 4", 5", 6" & 8" Round Wafers

**The Venus50XL-HF bench-top plasma cleaning/etching system has the following features and functions:**

- Bench-top design.
- Powder coated durable control console.
- Welded 6061 T6 rectangular Aluminum chamber measuring 8” x 9” x 4” with 2” diameter viewport.
- 7” x 8” horizontal RF powered electrode with approximately 2” substrate height clearance.
- 125W x 13.56 MHz high frequency RF power supply, continuously variable, with automatic RF matching network.
- Two Mass Flow Controllers for precise process gas flow control and gas mixing. Controlled with PC.
- CFM Krytox charged vacuum pump.
- Pirani vacuum gauge, 1-2000 mTorr. Integrated into PC control system.
- PC Based Control system with Plasma Etch proprietary software for fully automatic system operation.

**System Installation:**

The three systems (mask aligner, spin/bake unit, and plasma cleaning/etch system) have 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 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 three systems were shipped to the Clean Room, the systems were connected with the compressed air, water, nitrogen gas, and electricity by our engineers with consultation to the manufacturing companies.

**System Start Up and Operation Training:**

The mask aligner was started up by the engineer from the Suss MicroTec Company while the other two systems were started by the PI. After the SUSS MA6 Gen4 Pro manual mask aligner system was appropriately installed in the Clean Room and meets the installation requirements, an engineer from the Suss MicroTec 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 CEE® Model 200CBX spin/bake unit and the Venus50XL-HF bench-top plasma cleaning/etching system are relatively simpler, and were started up by the PI. The PI is very familiar with the two systems and the operation, and tested all the functions of the two systems after the installation. Both the spin/bake unit and the plasma cleaning/etching system function very well, and meet all technical specifications and the pre-designed requirements. The PI has trained students and researchers to use the two systems for their research. The three instruments function as a system for the UV lithography for research and education in the fabrication of micro and nanoscale devices.

**Research and Education**:

After the SUSS MA6 Gen4 Pro manual mask aligner, CEE® Model 200CBX spin/bake unit, and the Venus50XL-HF bench-top plasma cleaning/etching system were successfully installed and started up in the Clean Room, they have been used for our research and training students immediately, together with other cleanroom facility. The three systems are currently used for UV lithography 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.

**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 6(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 6(b) shows the high-resolution TEM (HR-TEM) image of a graphene film grown with the setup in Figure 6(a); Figure 6(c) shows the schematic of a graphene field-effect transistor (GFET) with semiconductor as the source/drain contact; Figure 6(d) shows the SEM image of a fabricated GFET device.

**Fabrication of Nanoscale Multilayered Thin Film-Based Integrated Thermoelectric Devices for the Application of Highly-Efficient Thermal-to-Electrical Energy Conversion 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 150 to 300 layers with a total thickness of about 150 nm to 1500 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.

**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 systems to perform UV lithography for patterning in the fabrication of CMOS integrated circuits. They used the e-beam evaporation 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 and achieved 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.

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 9-15 show the students' research activities in the clean room. The three installed systems 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 three systems together with the other clean room facility, starting from the summer of 2019.

The installed 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 SUSS MA6 Gen4 Pro manual mask aligner, a CEE® Model 200CBX spin/bake unit, and a Venus50XL-HF bench-top plasma cleaning/etching system have been purchased with the DoD/ARO funding and installed in the Clean Room in the Engineering building at the Alabama A&M University. The three systems have been tested and found to meet all the technical specifications and pre-designed requirements. One of the key capabilities for micro and nanofabrication is the ability to create micro and nanoscale patterns in the device fabrication. The three installed instruments form a system for the patterning in the device fabrication, where the spin coater is used to spin photo resist, and then the mask aligner system is used to align the patterns and make the UV light exposure for the device wafer fabrication, and the plasma etcher is finally used to strip and clean the photo resist on the device wafer. The three instruments have been used for our current research in the fabrication of micro and nanoscale devices. The addition of the three 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 SUSS MA6 Gen4 Pro manual mask aligner system.


**Figure 2.** The SUSS MA6 Gen4 Pro manual mask aligner system.

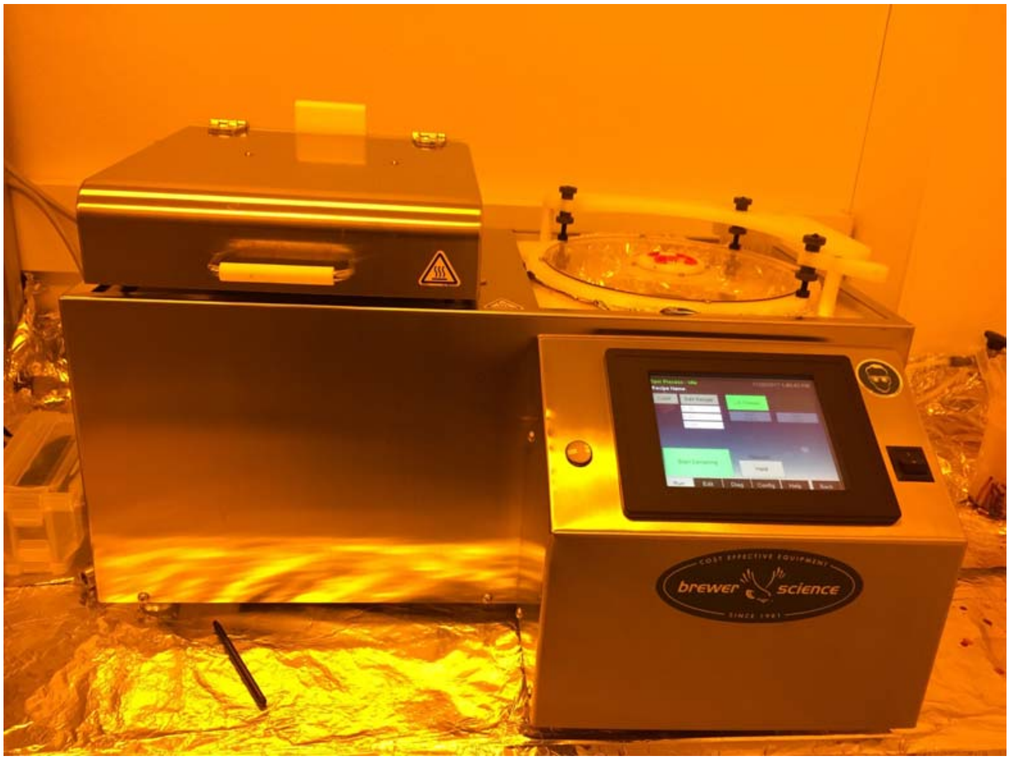



**Figure 3.** The CEE® Model 200CBX spin/bake unit.


**Figure 4.** The Venus50XL-HF bench-top plasma cleaning/etching system.

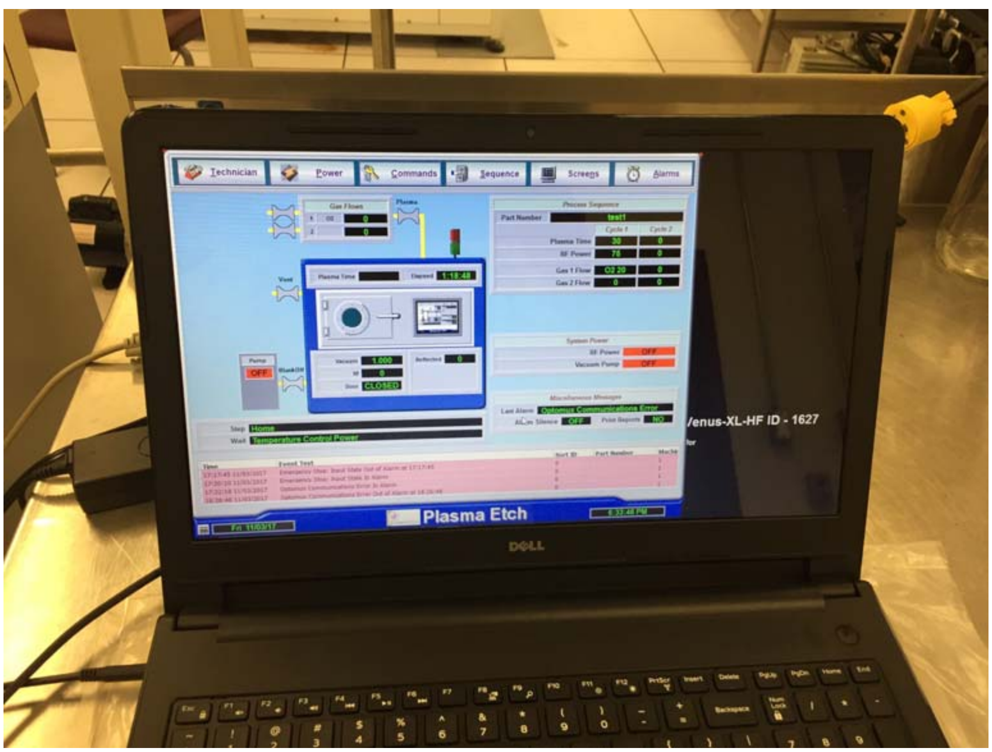



**Figure 5.** The computer and control software for the Venus50XL-HF bench-top plasma cleaning/etching system.

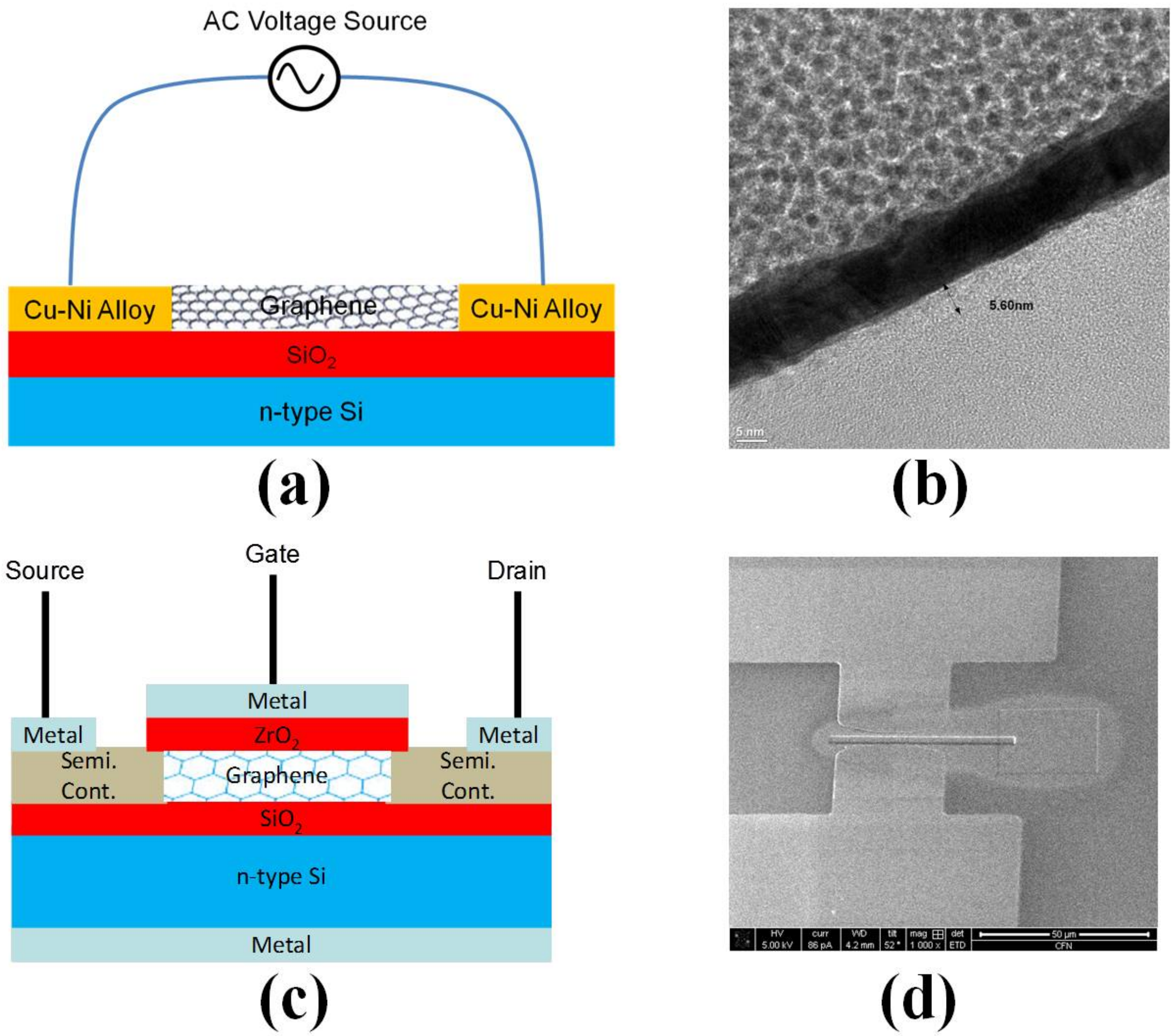



**Figure 6.** (a) Schematic of a setup for growth of graphene between a pair of nanscale Cu-Ni alloy electrodes with applying electric fields; (b) High-resolution TEM (HR-TEM) image of a graphene film grown with the setup in (a); (c) Schematic of a graphene field-effect transistor (GFET) with semiconductor as the source/drain contact; (d) SEM image of a fabricated GFET device.

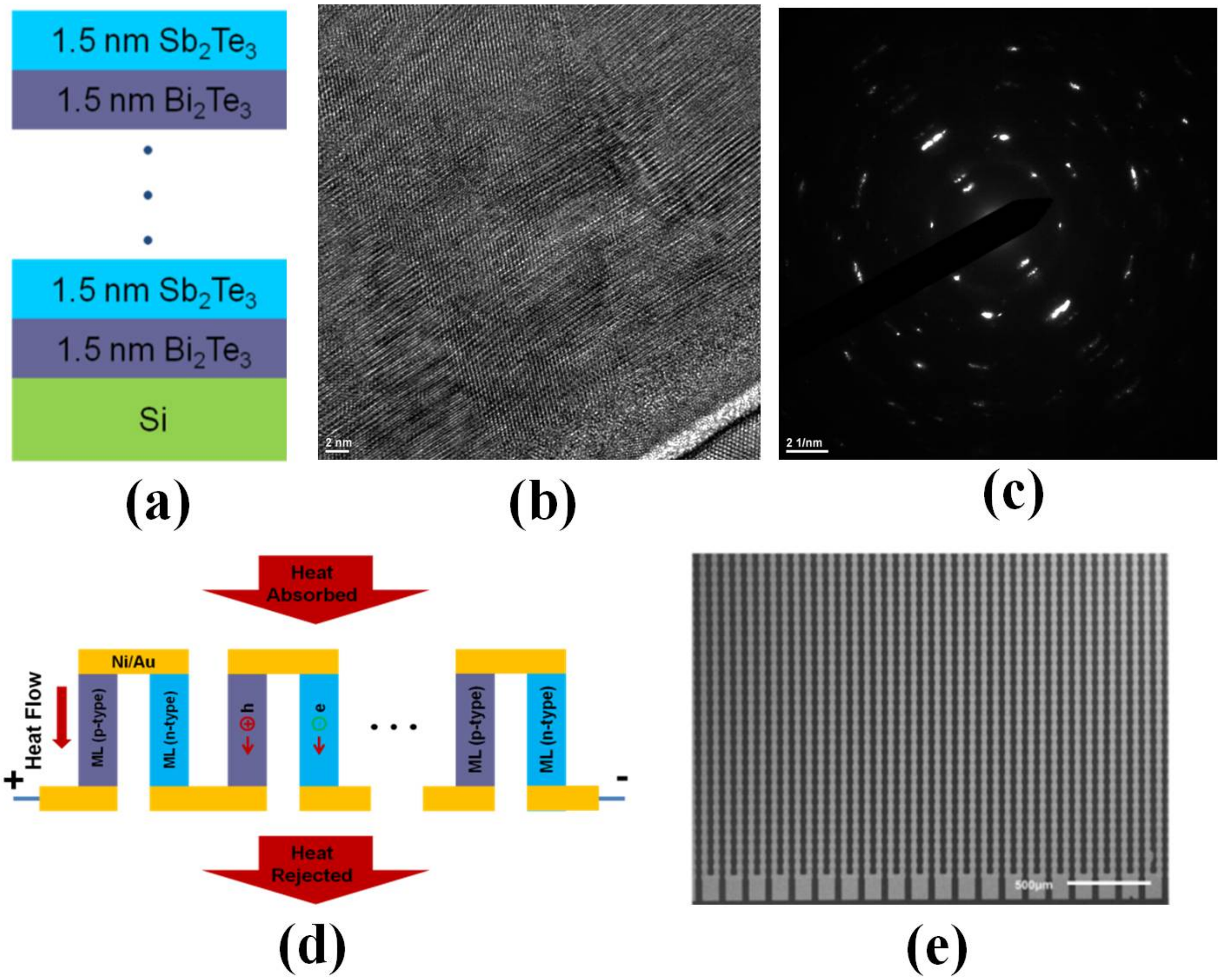



**Figure** 7. (a) Multilayered film with alternating $Bi_2Te_3$ and $Sb_2Te_3$ layers: (b) High-resolution TEM (HRTEM) image of a multilayer film grown by e-beam evaporation; (c) The selected electron diffraction pattern (SAED) of the grow multilayer film; (d) Working principle for an integrated TE device;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

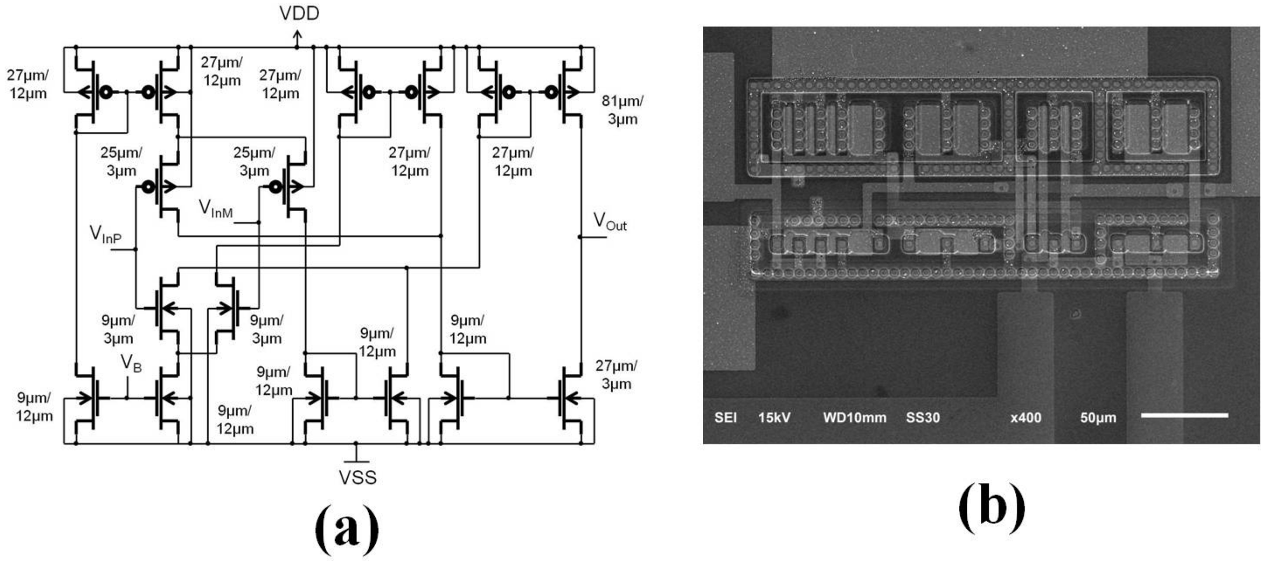
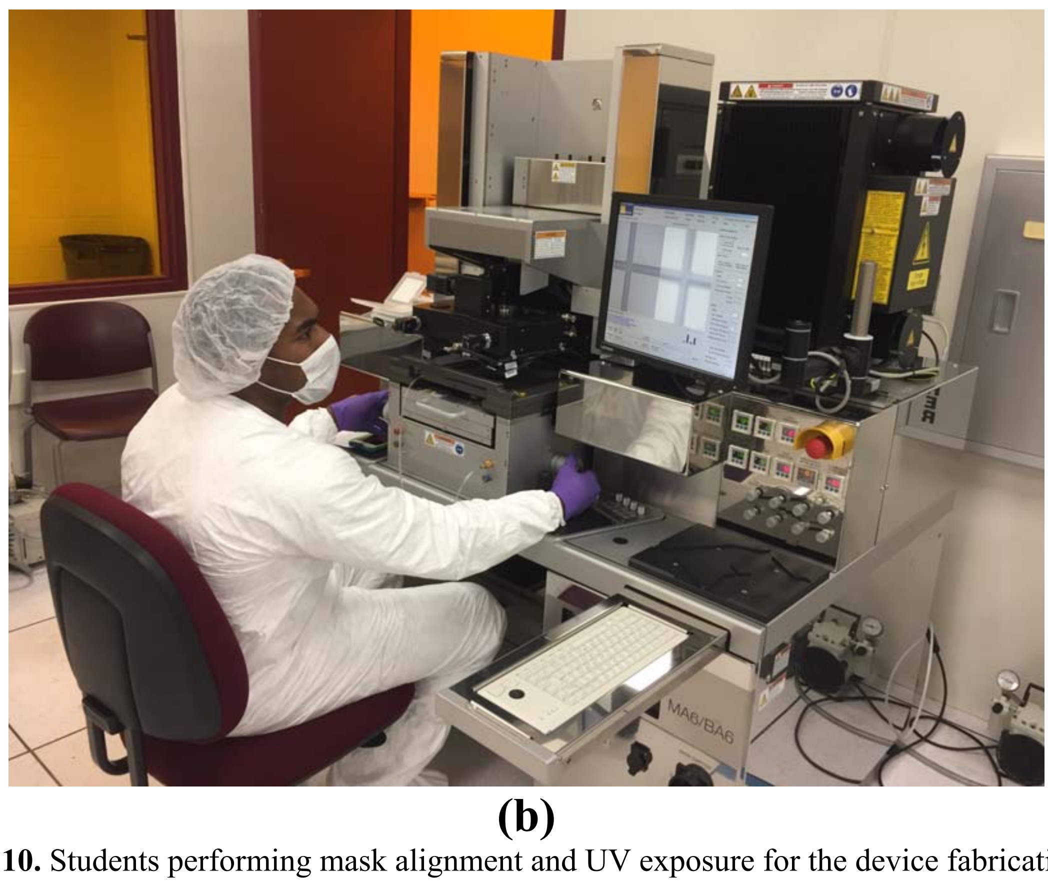



**Figure 8.** (a) Schematic of a CMOS-based operational transconductance amplifier (OTA) circuit; (b) SEM micrograph of a fabricated CMOS OTA circuit.

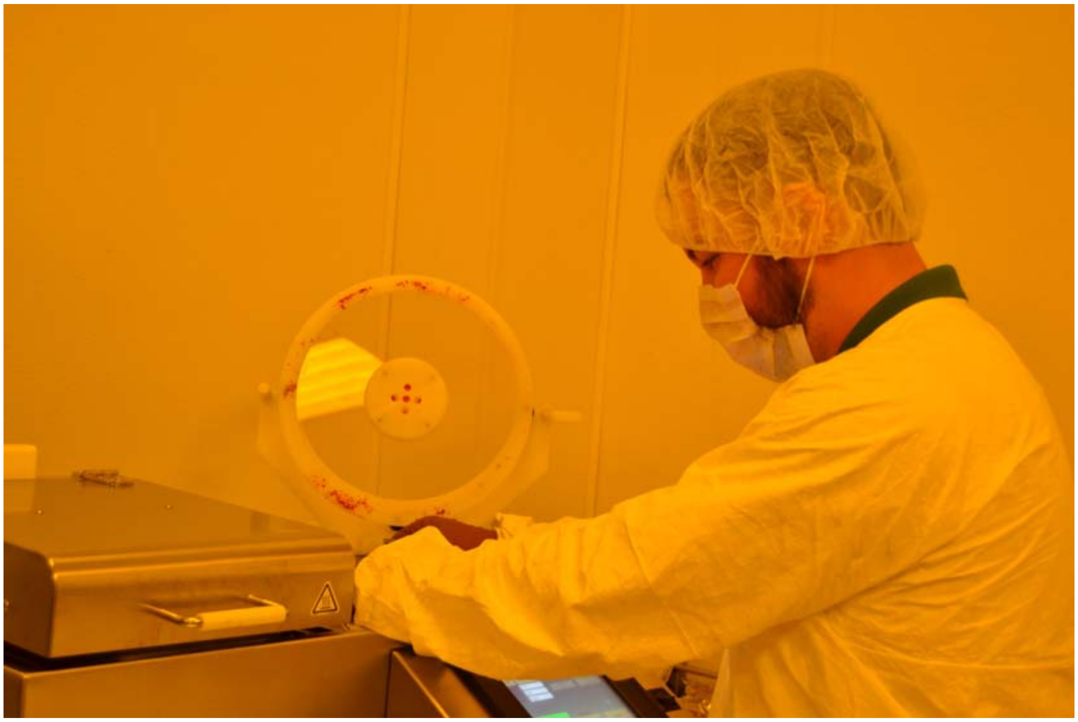
**Figure 9.** Students, who are supported by NSF and DoD through the research project, working in the AAMU-EE Clean Room.

**(a)**




**(b)**

**Figure 10.** Students performing mask alignment and UV exposure for the device fabrication with the SUSS MA6 Gen4 Pro manual mask aligner system.



**(a)**

**(b)**

**Figure 11.** Student performing spin coating of photo resist and soft baking for the device fabrication with the CEE® Model 200CBX spin/bake unit.

**(a)**

**(b)**

**Figure 12.** Students performing spin coating of photo resist and soft baking for the device fabrication with the CEE® Model 200CBX spin/bake unit.

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**(a)**

**(b)**

**Figure 13.** Students developing photo resist and processing device wafer fabrication.

**Figure 14.** Students operating the Venus50XL-HF plasma etching system for cleaning photo resist.


**Figure 15.** Students operating the E-beam/thermal evaporation system for the deposition of thin films in their device fabrication.