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14. ABSTRACT The aim of this project was to acquire instrumentation to augment a molecular beam epitaxy (MBE) system used in the synthesis of complex oxide heterostructures. A RF oxygen plasma source was acquired to increase the oxidizing ability of the growth environment, an improvement that will prove critical in stabilizing materials with high oxidation states. The plasma source and accompanying electronics were purchased from Mantis Deposition, Inc and successfully installed on the MBE system. Additionally, a low noise electron source was acquired to improve in situ reflection high energy electron diffraction (RHEED) capabilities of monitoring this film growth. The RHEED					
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Report Title

Final Report: Instrumentation for Epitaxial Growth of Complex Oxides

ABSTRACT

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Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

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Graduate Students

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PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Names of Post Doctorates

NAME

PERCENT SUPPORTED

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Total Number:

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Steven May	0.00	
FTE Equivalent:	0.00	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
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The number of undergraduates funded by this agreement who graduated during this period: 0.00

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Names of Personnel receiving masters degrees

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Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
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Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

See Attachment

Instrumentation for Epitaxial Growth of Complex Oxides
W911NF-14-1-0493
PI: Steven May (Drexel University)

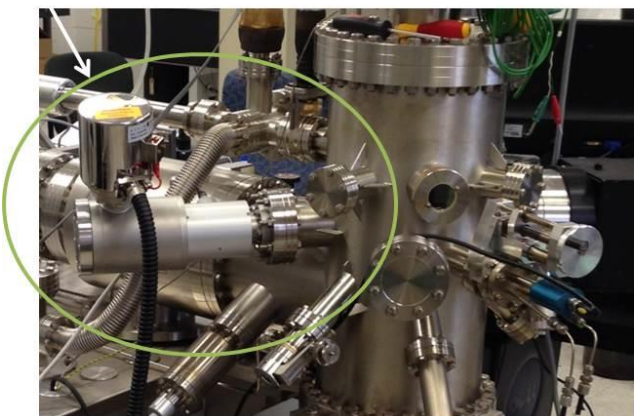
Period of Performance: 08/15/2014 – 08/14/2015

Project Accomplishments

The aim of this project was to acquire instrumentation to augment a molecular beam epitaxy (MBE) system used in the synthesis of complex oxide heterostructures. A RF oxygen plasma source was acquired to increase the oxidizing ability of the growth environment, an improvement that will prove critical in stabilizing materials with high oxidation states. The plasma source and accompanying electronics were purchased from Mantis Deposition, Inc and successfully installed on the MBE system. Additionally, a low noise electron source was acquired to improve *in situ* reflection high energy electron diffraction (RHEED) capabilities of monitoring thin film growth. The RHEED source was purchased from Staib Instruments and successfully installed on the MBE system. The equipment is and will be used extensively to synthesize complex oxide heterostructures for ongoing and future investigations of correlated electron behavior, electronic phase transitions, and novel electronic devices.

Both the plasma source and the RHEED source were installed on the MBE system in the PI's lab by the end of this project (August 2015). Photographs of the two pieces of equipment, adjoined to the MBE, are shown in Figure 1. Due to initial noise issues with the RHEED source that are believed to originate with the local Philadelphia subway system, inner shielding was also added to the MBE chamber to shield the electron beam on the flight path prior to the sample. The installation of this shielding was founded to dramatically increase the beam stability and mitigate intensity fluctuations. The RHEED source has been in continuous use with no downtime since it was installed. The plasma source is also operating as intended, after initially having to replace the discharge tube (this was supplied free of charge from the purchasing company).

Installed RHEED source



Installed plasma source

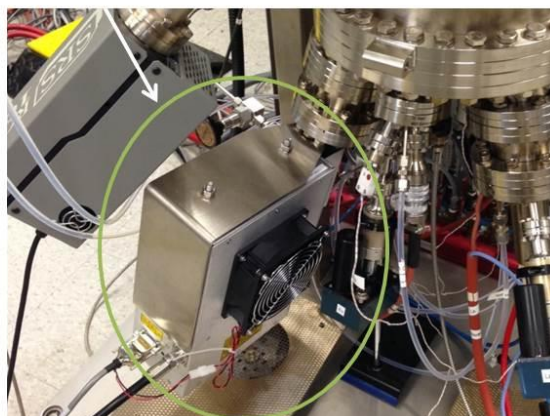


Figure 1. Photographs of the installed equipment on the MBE chamber. The RHEED source is shown on the left; the plasma source is shown on the right. The instrumentation is highlighted by the green circle and white arrow.

The RHEED source enables *in situ* monitoring of the surface electron diffraction pattern during thin film growth. Since the installation of the RHEED source, this technique has been used to monitor the deposition of LaFeO_3 , SrFeO_3 , $(\text{La,Sr})\text{MnO}_3$, and $(\text{La,Sr})\text{MnO}_3/(\text{La,Ca})\text{MnO}_3$ films. The beam is very stable, allowing for tracking of the specular and off-specular spot intensities during growth. This information can then be used to provide insight into the number of monolayers deposited, surface reconstructions, and any degradation in film quality during deposition. An example of RHEED data collected using the new source is shown in Figure 2. Images of the electron diffraction pattern before and during the growth of a LaFeO_3 film on SrTiO_3 are shown. Intensity oscillations are observed throughout the deposition, the period of which corresponds to the growth of a single unit cell. The *in situ* information provided by RHEED will be of increasing importance as the PI's group moves to the synthesis of more complex materials, such as $\text{Ca}_{1-x}\text{Sr}_x\text{Mn}_7\text{O}_{12}$, and complex layered heterostructures, such as three component superlattices.

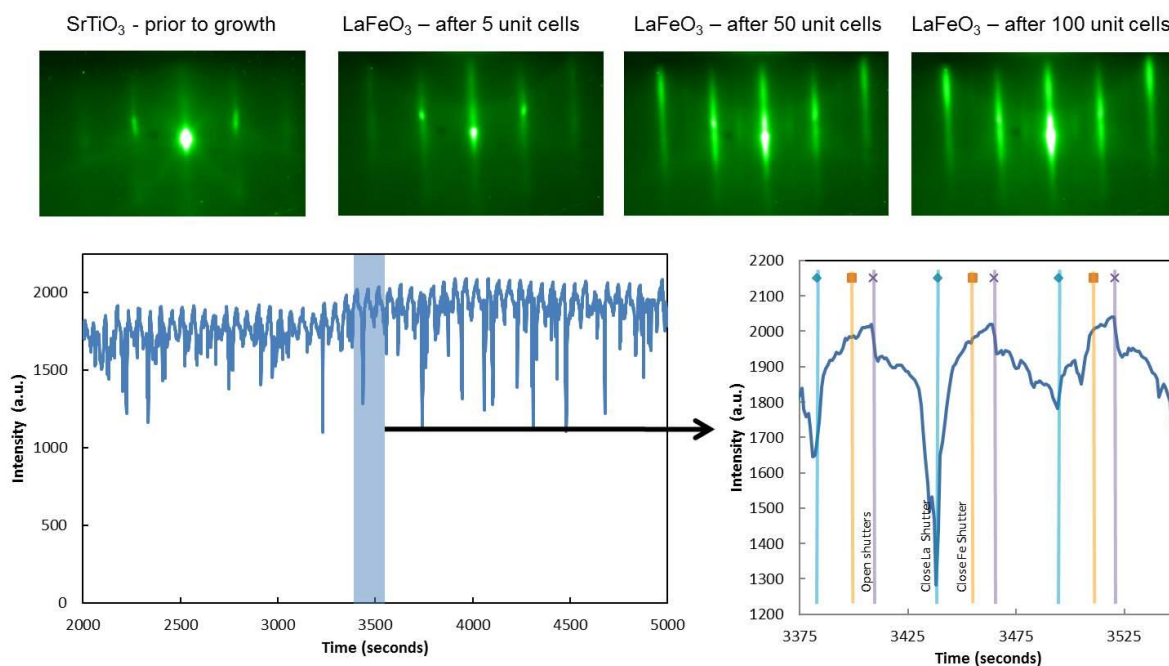


Figure 2. The RHEED patterns (top) obtained during various stages of the deposition of LaFeO_3 on SrTiO_3 . The bottom panels show the RHEED intensity oscillations (raw data) over ~50 minutes of a deposition (left) and a higher resolution plot of the oscillations over the course of three unit cells (right).

The plasma source, which produces monoatomic oxygen (O), enables the synthesis of oxide materials that require a stronger oxidizing environment than what can be achieved in either O_2 or dilute O_3 in O_2 . The plasma source has been installed and the conditions needed to ignite and maintain the O plasma have been established, as shown in Figure 3. Efforts are now ongoing to optimize the growth conditions for films in an O plasma environment. The plasma source will play a critical role in stabilizing stoichiometric materials that have a strong propensity for oxygen vacancies such as SrFeO_3 and SrMnO_3 .

The acquired instrumentation has made a direct impact on the education and scientific training of students and postdoctoral researchers at Drexel University. In the 3 months since its installation, the RHEED and plasma sources have been used by 4 graduate students and one

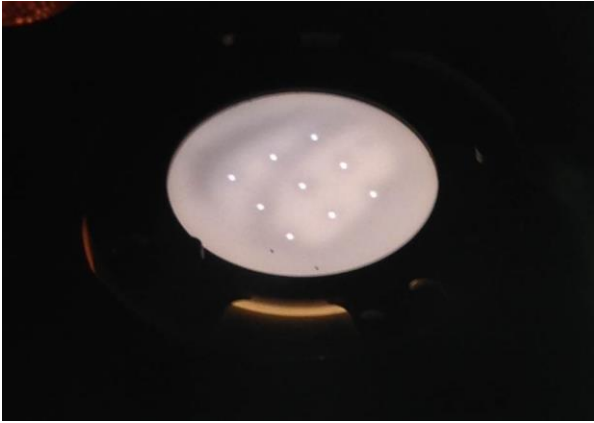


Figure 3. Photograph of the plasma source with an oxygen plasma ignited. The photo was taken through a viewport on the MBE chamber.

postdoctoral researcher in the PI's lab. The RHEED source is the only such system at Drexel. The instrumentation will be used for current and future ARO-funded projects focused on oxide heterostructures on which May is the PI or a co-PI. To enhance the educational opportunities of materials science students at Drexel, the PI plans to develop an activity utilizing the RHEED source within MATE 355 ("Structure and Characterization of Crystalline Materials"), a course he teaches annually. The activity would involve bringing the undergraduate students (juniors) to the PI's lab to measure surface electron diffraction patterns as a function of varying voltage (electron wavelength). This activity will

provide a direct demonstration of the role of the Ewald sphere in diffraction.

Summary of Accomplishments

- Acquired and installed a low noise, intensity stabilized RHEED source on the PI's oxide MBE chamber.
- Acquired and installed an RF oxygen plasma source on the PI's oxide chamber.
- Both the RHEED and plasma sources are operating as intended.
- Use of the RHEED source is now a routine part of film growth within the PI's lab. Growth conditions utilizing the oxygen plasma are now being optimized to yield high quality, oxygen stoichiometric films.