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				Jon-Paul Maria				
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Report Title

Final Report: Instrumentation up-fit for reactive nanolaminate PVD

ABSTRACT

PI Maria conducted an instrument upfit for physical vapor deposition (PVD) systems that combines electron beam (e-beam) evaporation with magnetron sputtering and pulsed laser deposition. The original instrumentation was supported by a 2014 DURIP award to enable clean, uniform, and rapid deposition of a wide variety of metallic, semiconducting, and ceramic thin films that serve multiple current and pending DoD programs at NCSU. While this instrumentation benefits several programs, its primary role is to support nanoenergetic materials research and the new NCSU-led MURI: Multi-modal Energy Flow at Atomically Engineered Interfaces.

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)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

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

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

	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 Man	uscripts:
	Books
Received	Book
TOTAL:	

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME

PERCENT_SUPPORTED

FTE Equivalent: Total Number:

Names of Post Doctorates

<u>NAME</u>

PERCENT_SUPPORTED

FTE Equivalent: Total Number:

Names of Faculty Supported

NAME

PERCENT_SUPPORTED

FTE Equivalent: Total Number:

Names of Under Graduate students supported

NAME

PERCENT_SUPPORTED

FTE Equivalent: Total Number:

Student Metrics This section only applies to graduating undergraduates supported by this agreement in this reporting period
The number of undergraduates funded by this agreement who graduated during this period: 0.00 The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields: 0.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields: 0.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale): 0.00 Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering: 0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

<u>NAME</u>

Total Number:

Names of personnel receiving PHDs

<u>NAME</u>

Total Number:

Names of other research staff

NAME

PERCENT_SUPPORTED

FTE Equivalent: Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

please see attached document

Technology Transfer

PROJECT DESCRIPTION

PI Maria used the RI support to enhance and expand physical vapor deposition (PVD) systems that combines electron beam (e-beam) evaporation with magnetron sputtering and pulsed laser deposition. The instrumentation was originally supported by a 2014 DURIP award to enable clean, uniform, and rapid deposition of a wide variety of metallic, semiconducting, and ceramic thin films that serve multiple current and pending DoD programs at NCSU. While this instrumentation benefits several programs, its primary role is to support nanoenergetic materials research under the current following programs:

W911NF-13-1-0493: Rational Engineering of Reactive Nanolaminates for Tunable Ignition and Power

W911NF-16-1-0406: "Multi-modal Energy Flow and Atomically Engineered Interfaces"

DESCRIPTION OF EXISTING AND ENHANGED EQUIPMENT

The 2014 DURIP program supported fabrication of two physical vapor deposition systems that accommodate sputtering, e-beam evaporation, and, pulsed laser deposition. While the configurations are optimized for nanolaminate deposition, they include design elements and capabilities that complement existing infrastructure at NCSU to ensure maximum impact on existing and future research programs.

The original proposal was designed to fund one system that could accommodate all modes of deposition. However, doing so would have put restrictions on the composite set of capabilities. For example; changing between modes would have required source exchanges that would compromise vacuum cleanliness, it would be difficult to have one sample manipulator position that offered ideal source-to-substrate distances for evaporation, laser deposition, and sputtering, and the composite geometry that accommodated all sources would impart limits on film thickness uniformity.

To eliminate these compromises, the Maria Group incorporated a series of existing components from the Maria labs into the construction plan. In so doing, the same amount of DURIP support was able to create two companion systems that could accomplish, with ideal deposition conditions/configurations, all of the deposition modes desired. Pressure ranges, temperature ranges, and source-to-substrate distances that are suited ideally for each individual technique could be achieved by separating the chambers. In addition, two chambers increase substantially the total throughput.

The original RI grant request was to support modest additions to the current system that could impart additional deposition capabilities well beyond those proposed initially. Specifically:

- 1. A load lock chamber that serves both chambers and allows sample hand-off between systems in vacuo; and,
- 2. A substrate manipulator for the evaporator chamber that allows deposition at elevated temperatures.

While the RI was pending review, the Maria Group and collaborators competed for and ultimately were awarded an ARO MURI titled: Multi-modal Energy Flow at Atomically Engineered Interfaces. This MURI supports a companion effort to W911NF-13-1-0493 focusing on energetic nanomaterials and a partnering effort on energy relaxation in interfacial plasmonic structures. With this large new program pending, the PI requested (and was granted permission) to redirect some of the funding to best serve the entire set of instrumentation that serves ARO programs at NCSU.

To this effect, approximately 40% of the funds were redirected from the originally proposed intent. The substrate manipulator that would be fitted into the existing evaporator was purchased from NBM designs as originally planned. The manipulator was installed and operates as intended. Substrates can now be heated to temperatures as high as 850 °C which will enable reactive depositions, improved microstructures, epitaxy if needed, and smoother surfaces. An image of the system with the new manipulator is shown below:



New high temperature substrate manipulator assembly for existing ebeam evaporation system

Figure 1: E-beam evaporator unit built with 2014 ARO DURIP grant with the addition of a high temperature substrate manipulator

Instead of a load-lock that would link both existing systems, we redirected funding to assist construction of a new PVD system that would be dedicated to the plasmonic thrust of the new NCSU MURI. The funds left over after manipulator purchase were applied to the purchase of a new sputtering chamber, a pulsed DC power supply, and several custom source flanges. These components, combined with existing equipment at NCSU enabled our team to construct the system shown below that is dedicated to CdO-based plasmonic heterostructures.



Figure 2: Photograph of new multi-magnetron sputtering system at Maria Labs for preparing plasmonic multilayers for the NCSU MURI: Multi-modal Energy Flow at Atomically Engineered Interfaces. The three items supported by this RI are highlighted in the image.

All up-fits were completed by November 2016 and both systems are fully tested and qualified, and deposition optimization research is underway.