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14. ABSTRACT
The research objective of this proposal is to discover new phases of topological matter by experimentally exploring magnetism and correlation effects in f-electron systems. This project aims at the discovery of emergent topological phases "beyond band theory." In order to address these goals, we have developed a unique instrument that couples a top loading cryogen free dilution refrigerator with both traditional solenoid and gradient magnets to our molecular beam epitaxy (MBE) growth chamber. The coupling is accomplished via an air free interface with a glove box and

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Report Title

Final Report: Ultracold Field Gradient Magnetometry and Transport to Study Correlated Topological Phases

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The research objective of this proposal is to discover new phases of topological matter by experimentally exploring magnetism and correlation effects in f-electron systems. This project aims at the discovery of emergent topological phases "beyond band theory." In order to address these goals, we have developed a unique instrument that couples a top loading cryogen free dilution refrigerator with both traditional solenoid and gradient magnets to our molecular beam epitaxy (MBE) growth chamber. The coupling is accomplished via an air free interface with a glove box and top loading probe which can additionally couple to variable temperature environments. We can therefore synthesize volatile f-electron based films and transfer them to measurement systems without degradation caused by exposure to air. The milliKelvin temperatures of the dilution unit allow exploration of low temperature dynamics of the f-electron systems and higher temperature environments allow material characterization. The instrument may be seen as the transport and magnetic measurement equivalent to combined MBE-photoemission or MBE-scanning tunneling microscopy systems that have recently made significant impact in the field of topological materials. The additional requirements for device fabrication necessary for transport devices are met by including processing tools in the inert gas glove box environment.

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)

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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)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

N/A

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

N/A

Patents Awarded

N/A

Awards

National Science Foundation CAREER Award

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	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:	

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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

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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

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The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

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Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment
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Scientific Progress and Accomplishments

Foreword
The goal of this DURIP award was to create a combined MBE – Dilution refrigerator measurement system. As we describe herein, we have realized this combination of instrumentation via an inert gas glovebox as proposed. This is a unique instrument that will allow our research group to enter a new parameter space of thin films previously unstudied due to degradation effects. This is an exciting new instrument for both the research group of the PI and the subfield of new correlated topological phases.

List of Illustrations

Statement of Problems Studied
To discover new correlated topological phases of matter, it is highly desirable to be able to synthesize and study materials in extreme conditions without exposure to ambient conditions. An example of this is the discovery of the quantum anomalous Hall phase [1-2], where early efforts to probe these materials suffered from the difficulty in growing epitaxial thin films and measuring their properties at dilution refrigerator temperatures without degradation. This difficulty stems from the lack of direct coupling of instruments and the corresponding exposure of samples to ambient conditions. The problem studied here was how to overcome this difficulty in the specific context of studying f-electron based thin films which are expected to both suffer from degradation due to reaction with oxygen and water as well as require low temperature characterization due to the dynamics of the f-electron moments. If such a problem could be overcome, it would open the doors to exploring the physical properties of these new materials, evaluations of how to control their emergent properties, and expand the search for topological phases beyond single particle band theory.

Summary of the most important Results
We have constructed a combined MBE – Dilution refrigerator system coupled via an inert gas glove box. This allows growth of epitaxial thin films by MBE that can be transported in vacuum to the glove box. In the glove box the materials can be processed. Probes for a dilution refrigerator and other cryogenic systems in the lab can then be connected to the glovebox for an air free transfer. The dilution unit is capable of this air free transfer via a top loading design. The refrigerator has capabilities for both constant magnetic fields and gradient magnetic fields to allow for measurements of Faraday and torque magnetization in addition to electrical transport.

Fig. 1(a) shows the MBE unit before connection to the glove box system. The manipulator arms for shuttling substrates in to the growth chamber and to the glove box connector are labeled. The MBE has an additional preparation chamber to allow for deposition of contact metals and etching for device patterning. Fig. 1(b) shows a rotated view of the MBE system after connection to the glove box. The connection is through the back wall of the glove box. The load lock chamber can be vented with Argon gas to allow passage in to the glove box. Note that in Fig. 1(b) baking blankets are attached to the MBE, but are removed during normal operation of the system. The manipulator arms are also labeled.

Figure 1 – (a) Molecular Beam Epitaxy (MBE) chamber before glove box attachment. Manipulator arms to connect to the growth chamber and glove box are labeled. (b) MBE chamber after glove box attachment (rotated view). The same manipulator arms are labeled. In this image heating blankets are attached to the MBE.

The front view of the glovebox and all other components of the combined system are shown in Fig. 2. Starting with Fig. 2(a), the probe connection with pump/purge capability is shown. Probes from the dilution refrigerator and other cryogenic systems in the lab (see Fig. 2(b)) can be rigidly attached to this port, which extends with a cap in to the glove box. Upon loading in to the system the entire port is pumped and purged repeatedly with the pump and Ar gas of the glove box. The cap can then be removed inside the glove box to expose the probe to the inert environment. A front view of the glovebox is shown in Fig. 2(c). The digital display on the left hand side of the glovebox connects through a sealed connector to an optical microscope inside the glove box with a large working distance to allow for manipulations for device processing, contacting making, and connections to the probe. The connection to the MBE chamber is on the back wall of the glove box, as shown in Fig. 2(d). This has transparent cover to allow for alignment checks and visual confirmation during loading and unloading. The dilution refrigerator is can be seen in the background of Fig. 2(c) and also in Fig. 2(e). The unit is situated far enough away from the MBE system to avoid interference with the RHEED and related components, but is in the same laboratory space so that transfer via the top loading probes is straightforward.

Figure 2 - (a) Probe connection on glove box. This allows measurement probes to be interfaced with the glove box. (b) Probe with vacuum seal and vacuum flange that mates with probe connection. (c) Overall view of glove box in foreground with MBE in background. (d) Port on rear wall of glovebox that allows passage of sample in inert atmosphere conditions to MBE chamber. (e) Dilution refrigerator unit for low temperature measurements of thin films grown in MBE.

All components in Fig. 2 are coupled together with the MBE, allowing a direct transfer of films from growth to processing to measurement without exposure to air. An important design consideration was to allow for the probe connection to accommodate not only the dilution refrigerator but also higher temperature cryostats in the laboratory. We are thus able to not only study the ultra-low temperature behavior in these systems but characterize samples over a broad range of temperature where structural and electronic changes may occur. For X-ray characterization we have also developed an air-free sample holder that can be inserted in to the glove box so that materials can be processed for X-ray characterization without exposure to air.

This "assembly line" approach will allow for a large number of growth campaigns that have not been previously possible without sample degradation. Based on our recent results of f-electron systems containing Gd in bulk single crystals we have the knowledge that such materials are of high interest but also degrade in air [3]. Thin films of monolayer thickness will be significantly more sensitive to such degradation but also be of high interest in the 2D limit where new behavior may be observed and also allowed for manipulation of materials by electrostatic gates. This is therefore an ideal tool for this study.

Bibliography
1. C.-Z. Chang, J. Zhang, X. Feng, J. Shen, Z. Zhang, M. Guo, K. Li, Y. Ou, P. Wei, L.-L. Wang, Z.-Q. Ji, Y. Feng, S. Ji, X. Chen, J. Jia, X. Dai, Z. Fang, S.-C. Zhang, K. He, Y. Wang, L. Lu, X.-C. Ma, Q.-K. Xue. Experimental Observation of the Quantum Anomalous Hall Effect in a Magnetic Topological Insulator, Science 340, 167-170 (2013).

2. J. G. Checkelsky, R. Yoshimi, A. Tsukazaki, K. S. Takahashi, Y. Kozuka, J. Falson, M. Kawasaki, Y. Tokura. Trajectory of Anomalous Hall Effect toward the Quantized State in a Ferromagnetic Topological Insulator, Nature Physics 10, 731–736 (2014).

3. T. Suzuki, R. Chisnell, A. Devarakonda, Y.-T. Liu, W. Feng, D. Xiao, J. W. Lynn, J. G. Checkelsky, Nature Physics (2016) doi:10.1038/nphys3831 .

Technology Transfer

N/A

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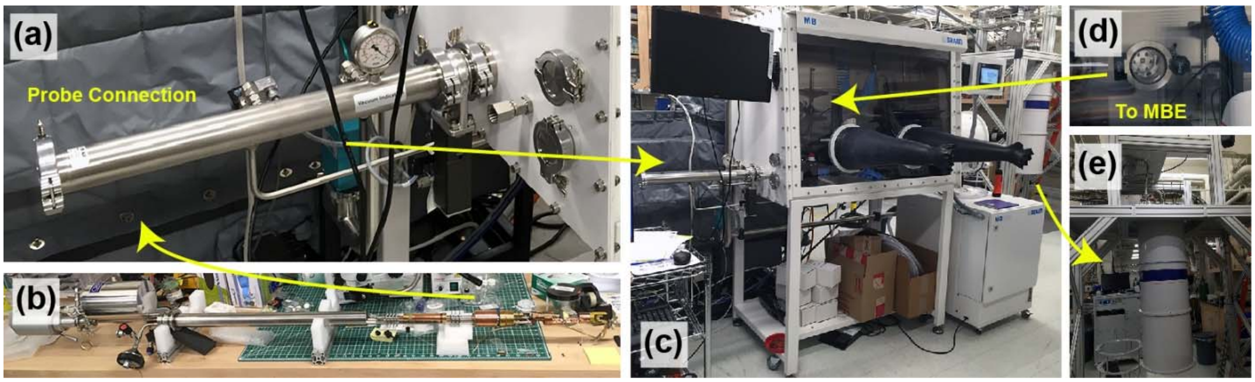

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