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Final Report: Cryofree Toploading Rotator System for High-throughput Study of Novel Low-Dimensional Electron Systems.

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The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

We report on the DURIP-enabled design, construction, and operation of a low-temperature system capable of high-throughput electronics measurements of novel materials. The system can achieve millikelvin temperatures without consuming liquid helium and can apply large magnetic fields applied in arbitrary directions via a two-axis sample rotator.

15. SUBJECT TERMS

cryogenics, electronics, cryogen-free, piezoelectrics, rotator, magnetic field, high-throughput, quantum transport, materials research

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a. REPORT	b. ABSTRACT	c. THIS PAGE			David Goldhaber-Gordon
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Report Title

Final Report: Cryofree Toploading Rotator System for High-throughput Study of Novel Low-Dimensional Electron Systems.

ABSTRACT

We report on the DURIP-enabled design, construction, and operation of a low-temperature system capable of high-throughput electronics measurements of novel materials. The system can achieve millikelvin temperatures without consuming liquid helium and can apply large magnetic fields applied in arbitrary directions via a two-axis sample rotator.

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
01/30/2015	1.00	F. Amet, A. J. Bestwick, J. R. Williams, L. Balicas, K. Watanabe, T. Taniguchi, D. Goldhaber-Gordon. Composite fermions and broken symmetries in graphene, Nature Communications, (02 2014): 5838. doi: 10.1038/ncomms6838
TOTAL:	**1**	

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

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
01/30/2015	3.00	A. J. Bestwick, E. J. Fox, Xufeng Kou, Lei Pan, Kang L. Wang, D. Goldhaber-Gordon. Precise quantization of anomalous Hall effect near zero magnetic field, arXiv:1412.3189 (12 2014)
01/30/2015	2.00	Patrick Gallagher, Menyoung Lee, Trevor A. Petach, Sam W. Stanwyck, James R. Williams, Kenji Watanabe, Takashi Taniguchi, David Goldhaber-Gordon. A high-mobility electronic system at an electrolyte-gated oxide surface, arXiv:1410.3034 (10 2014)
TOTAL:	**2**	

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Andrew Bestwick	0.00	
Eli Fox	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
David Goldhaber-Gordon	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**1**	

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

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

Please see PDF attachment for statement of scientific progress.

Technology Transfer

We codesigned the two-axis rotator with a nanopositioning company, Attocube Systems AG. Key design constraints specific to top-loading cryogenic systems required close collaboration on matters such as size, electrical wiring, positioner precision, and electrical noise shielding. The result of this work will be a commercial model available to other scientists for sample rotation in similar conditions.

Final Report: Cryofree Toploading Rotator System for High-Throughput Study of Novel Low-Dimensional Electron Systems

In the study of electronic transport in quantum materials, the following capabilities can enable new and better research: ability to set sample temperature over a wide range as low as tens of millikelvin, high-throughput measurement of samples (including rapid temperature control) to quickly improve materials and fabrication techniques, the ability to apply large magnetic fields in arbitrary directions relative to the sample, and refrigeration technology that does not require the consumption of expensive, non-renewable liquid helium. This DURIP grant helped enable the design, purchase, and construction of a measurement system that achieves all four objectives.

The starting platform on which this DURIP built is a Leiden Cryogenics CF-900 cryogen free-dilution refrigerator, a system with a pulse-tube cryocooler (utilizing closed-circuit compression and expansion of high-pressure helium gas) to reach temperatures as low as 3.5 kelvin, as well as a conventional 3He-4He dilution circuit to further cool interior regions of the fridge to the millikelvin scale. The system was designed to plan for top-loading use: the fridge is kept cold, and samples are inserted into it from the outside world with probes that then make thermal contact to its interior metal surfaces. In this way, samples can be cooled from room temperature to 4 kelvin in 4 hours, and to base temperature, around 20 millikelvin, in an additional 2 hours. This allows for high sample throughput: a full cool-measure-warm cycle to be performed in as little as a single workday. Also, the ability to tune the fridge-probe thermal connection allows for control of the sample temperature between base and 300 kelvin. However, the actual top loading system and probes were purchased on the DURIP.

Two special apsects make the system especially powerful for characterizing quantum materials. The fridge was built with a single-axis 14-tesla magnet, allowing for measurements of magnetotransport from which detailed information about electronic behavior can be extracted. Secondly, we are in the process of integrating onto one of the probes a cryogenic two-axis rotator (obtained under other funding), which will allow the sample to be oriented in any angular position relative to the applied field. This is especially useful for low-dimensional systems, allowing us to separate the effect of the field on electrons' orbital motion (typically confined to a 2D plane or along a 1D line) from the effect on their spins.

The following studies were performed using this system:

F. Amet *et al.*, Composite fermions and broken symmetries in graphene, *Nature Communications* **6**, 5838 (2014).
This study of the fractional quantum Hall effect revealed intricate details of the interactions and symmetry-breaking effects that lead to novel electronic order in graphene. It required the application of the full 14 tesla magnetic field for measurements in this system (we also applied even higher fields only available at the National High Magnetic Field Laboratory). To extract the size of the zero-field gap at charge neutrality, the fridge-probe system's wide temperature control was employed. Also, the paper demonstrates the power of using sample rotation in magnetic fields to separate orbital and spin behavior, albeit performed at the NHMFL before we had rotation working in our own system.

P. Gallagher *et al.*, A high-mobility electronic system at an electrolyte-gated oxide surface, publication in *Nature Communications* forthcoming, preprint available at arXiv:1410.3034.

Synthesizing methods originating from research in graphene, complex oxides, and extreme electrostatic gating, this study demonstrates a method for dramatically improving the quality of novel 2D electron systems obtained by ionic liquid gating of strontium titanate surfaces. Because ionic liquids freeze at ~200 kelvin while measurements were performed at base temperature, the experimental method requires rapid thermal cycling to change parameters. With multiple samples each cooled repeatedly, the total number of cooldowns exceeded 20, which would have been impractical without the top-loading probes.

A. J. Bestwick *et al.*, Precise quantization of anomalous Hall effect near zero magnetic field, preprint available at arXiv:1412.3189.
This paper reports nearly-ideal edge transport in a topological insulator film with ferromagnetic doping. It required quickly testing several dozen rounds of devices to provide quick feedback to the research groups optimizing their material synthesis process. Each test required the lowest possible temperatures and, in many cases, the full 14 tesla of applied field.

Studies in progress:

Transport signatures of spin polarization in quantum spin Hall effect edge modes
In the quantum spin Hall effect, all electron transport takes place along sample edges, with electron spin determined by direction of motion. However, many questions remain about along which direction the spins are polarized and the precise effect of applied magnetic fields. Here, the sample rotator will provide detailed information about the field direction relative to the sample surface along which the quantum spin Hall effect is most sensitive.

Comparison between optical and transport properties of magnetic topological insulator films
Using the top-loading probes to their fullest high-throughput extent, we measured a series of thin films at varying thicknesses (between 2 and 20 nanometers) to compare with parallel measurements of optical spectroscopy performed by collaborators. This, as well as other possible surveys of material parameter space, may illuminate the conditions necessary to achieve the quantum anomalous Hall effect.

List of equipment purchased:

Quant.	Item	Vendor	Price per item
2	Probe for CF-900	Leiden Cryogenics BV	$17,624
1	Custom wiring for fridge	Leiden Cryogenics BV	$42,601
2	Custom wiring for probe	Leiden Cryogenics BV	$11,934
2	Edwards XDS46i pump	Leiden Cryogenics BV	$7,427
2	CMN thermometer	Leiden Cryogenics BV	$2,392
2	Platinum thermometer	Leiden Cryogenics BV	$252
1	AVS47 resistance bridge	Leiden Cryogenics BV	$6,987
1	Fridge installation	Leiden Cryogenics BV	$7,553
1	Exchange rate increase cost of above*	Leiden Cryogenics BV	$23,500
1	Impedance bridge	Leiden Cryogenics BV	$1,550
1	AVS47-IB IEEE-488 interface	RV Elektroniikka Oy Picowatt	$1,770
2	Exchange 1x 40-ft for 2x 20-ft helium lines	Cryomech Inc.	$2,120
1	1000 ft CuNi flexible coaxial cable	Calmont Wire & Cable Inc.	$10,580

1	Additional 1000 ft CuNi flexible coax	Calmont Wire & Cable Inc.	$6,700
3	LI-75A low noise preamplifier	Global Test Solutions	$2,180
1	PS-70A DC power supply	Global Test Solutions	$1,295
3	2400 digital sourcemeter	Tektronix Inc.	$3,672
1	Single rack mounting kit	Tektronix Inc.	$74
1	Dual rack mounting kit	Tektronix Inc.	$103

The basic probe models were discussed, and all other items were necessary for making the top loading measurement system functional.

*Items were quoted and approved for purchase in Euros. Because of how the university purchasing system is configured, the line items are listed in dollars, converted at the exchange rate in place at the time the items were ordered. At time of payment (several months later), the exchange rate had changed, so the dollar price was higher than originally expected. This line item reflects that adjustment.