#### **Graphene Stripper Foil**

#### SBIR Phase II Grant DE-SC0000852 Carbon Stripper Foil for the Next Generation Rare Isotope Beam Facility

This material is based upon work supported by the Department of Energy under Award Number DE-SC0000852

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# Outline

- Who is Applied Nanotech, Inc
- Scope of the Program
- Need for new stripper foils
- Graphene foil manufacturing
- Foil characterization
- Summary



# **ANI Introduction**



- Located in Austin, Texas USA
- Founded 1988
- Nanotechnology R&D with emphasis in:
  - 1) Thermal Management
  - 2) Nanocomposites
  - 3) Nanoelectronics
  - 4) Nanosensors
  - Three pronged business model:
    - 1) Commercialization by IP Licensing
    - 2) R&D Services
    - 3) Commercialization by Venture Formation



## **Scope of the Program**

- Develop and test robust carbon stripper foils for next generation Facility for Rare Isotope Beams
- SBIR Phase II: two year program Aug 2010 thru Aug 2012
- Deliverables: Progress and Final Reports
- Provide samples of the foils with area density 0.5 to 2.0 mg/cm<sup>2</sup> and diameter >15 cm



Diagram from http://www.phy.duke.edu

#### **Stripper Foils**

- Need to increase the charge state of the ions being accelerated and reduce the total voltage used in the accelerator thus reduce cost
- A thin media such as carbon film is inserted in the beam



Diagram from http://www.phy.duke.edu



# Challenges

#### High power density

- Uranium beam power at stripper energy (17.5 MeV/u) is ~ 50 kW
- Carbon foil equilibrium thickness is ~ 0.5 mg/cm<sup>2</sup> (2.2  $\mu$ m thick)
- Power deposited on the stripper foil  $\sim$  700 W  $\sim$  3.8 kW/mm^3
- Beam spot should be as small as possible (take 5 mm diameter beam)
- High temperature leads to foil <u>degradation</u>

#### Thermal and mechanical issues

- Foils tend to get thinner for heavy ions
- The typical failure modes are <u>foil thinning</u> (energy changes) and <u>foil tearing</u>

#### Formation of defects due to radiation damage

- Displaced atoms and vacancies are formed under irradiation. They have higher mobility at high temperatures affecting the foil lifetime.
- Lattice deformation results to the foil failure



# Approach

- New generation of stripper foils are needed to accommodate the ~700W beam power dissipated in the foil in 1 mm beam diameter
- One approach is to use large area carbon foils that are rotated to distribute the heat load from the beam to control foil temperature



Calculation of temperature increase of a rotating graphene disk at 1000W dissipated power



# **Technical Requirements**

#### We need to:

#### Maintain beam quality

- Small beam diameter minimize beam degradation from passing through stripper
- Small (<±5%) stripper thickness variation</li>

#### Have reliable operations

- Stripper system long lived and/or fast replacement
- High thermal conductivity
  - Needed to spread the dissipated heat over larger area
- Mechanically robust and thermally stable
  - High angular frequency of foil rotation requires high tensile strength of foil
- Large area fabrication
  - Currently 13 cm diameter --> 15 to 25+ cm diameter goal
- Low cost: target < \$1000 for large area</p>
  - Current materials cost: ~\$30 per 13-cm foil



#### Why Graphene?

- Very high thermal conductivity
- Excellent mechanical properties
- Can be made uniform over large area
- Low cost in production



# **Graphene Foil Preparation**



- Obtaining graphene oxide (GO) aqueous dispersion
  - Reduce GO to graphene dispersion (using hydrazine+ $NH_3$ )
- Create foils by vacuum filtration of dispersion
- Lift-off foil from the filter for free-standing foil
- Amenable to scale-up fabrication.

D. Li et al., Nature Nanotech, 2008, 3, 101-108



## **Images of Graphene foils**

- Foils of areal mass density 0.5 2.0 mg/cm<sup>2</sup> and diameter of 13 cm are made successfully
- The foils are quite smooth and have a metallic luster and shine.
- The foils have a layer structure. At the edge, the foil appears like a deck of cards.
- Foils are robust, easy to handle and survive express shipping.



5.0kV 7.3mm x3.00k SE(U.LA1) 9/14/2011



# **Tensile Strength of Graphene Foil**

Tensile strength was measured for as-produced and treated foils. Treatment conditions: 600°C for 1 hr in 4%  $H_2$  in  $N_2$  gas mixture

Sample#	thk, um	width, cm	force, N	TS, MPa
10U1	3.8	1.9	12	166.2
10U2	3.8	2.25	13	152.0
10U3	3.8	2.23	15.5	183.3
Average untreated				167.2
Standard deviation				15.7
10T4	3.8	2.15	2.5	30.6
10T5	3.8	2.1	9.5	119.0
10T6	3.8	2.15	3	30.6
10T7	3.8	2.15	5	61.2
10T8	3.8	2.1	12.75	36.7
10T9	3.8	2.1	3	37.6
10T10	3.8	2.1	6	159.8
Average treated				67.9
Standard deviation				51.3

Tensile strength of **150 MPa** is sufficient to keep the integrity of a foil rotating at angular frequencies up to **60,000** rpm for a 13-cm circle foil and over **400,000** rpm for 1 cm wide ring foil



## **Thermal Conductivity of Graphene Foil**

# A 20-micron thick foil was fabricated for a thermal conductivity test.

Measured\* in-plane thermal conductivity - 1480 W/m·K

	thickness	bulk density	temperature	specific heat	diffusivity	conductivity
	@ 25°C	ρ@ 25℃		Cp	α	λ
Sample	(mm)	<b>(g/cm<sup>3</sup>)</b>	(°C)	(J/g-K)	(mm²/s)	(W/m-K)
graphene foil	0.020	1.55	25	0.730	1308	1480
(in-plane)						

ASTM E1461 Flash Method Thermal Conductivity Results

\*Measured by NETZSCH Instruments North America LLC



# **Temperature Stability of Graphene Foil**



Stripper foil will operate in vacuum. After temperature conditioning, good stability is expected (tested in  $N_2$ , peaks due to water, -OH groups, and possibly oxidation in residual air; TGA Q50).



## **Alpha Probe Characterization**



Spectrum of Cf source through graphene foil in the center of the foil and 1-cm off center (foil obtained from U. Wollongong).

Spectra lie on top of each other with calculated mass density 0.421 +/- 0.01 mg/cm<sup>2</sup>, achieving <5% uniformity.



#### **Tests at Michigan State U.**





- Over 300% foil lifetime vs. predicted value was observed using graphene foil with <sup>87</sup>Kr 13 MeV/u beam
- <u>E-beam</u> tests performed to mimic ion beam exposure.
- E-beams were positioned a fixed spots for many hours. No difference seen on sample between fixed spot or fast beam sweep line across the sample.
- Current of electron gun adjusted to maintain temperature below 1800K
- Slight mass loss measured may be degassing of residual oxygen from production process
- No pinholes created by e-beam.
- \*Graphene foil obtained from U. Wollongong

#### H-Ion Beam tests at Texas A&M University

- Foils of 1.0 2.0 mg/cm<sup>2</sup> tested in 1-60 MeV Hions to protons and 10 – 60 MeV D- ions to deuterons on K150 cyclotron.
- No reduction of stripped beam current after testing at 30 MeV H- ions at 20  $\mu\text{A}$  for over 3 days

tests courtesy of Dr. Henry Clark of TAMU



# **Windows for Gas Targets**

- Potential application of graphene foils to replace metal foils as windows for gas targets (ATLAS, Argonne National Laboratory).
- Metal windows used now create secondary radioactive beams.
- Desire low-Z windows strong enough to hold gas pressure in target chambers and also must be vacuum tight.
- Preliminary tests on 0.4 mg/cm<sup>2</sup> windows showed that 8 mm diameter window held up 1 atm gas pressure. Tests are continuing.



# **Summary**

- Graphene stripper foils 0.5 2.0 mg/cm<sup>2</sup> with up to 13 cm diameter prepared.
- Graphene foils are smooth, flat and robust.
- Thickness uniformity of > 5% achieved.
- Can scale to larger area for FRIB applications.
- High temperature e-beam tests demonstrate thermal robustness – no pinholes created at fixed beam spots.
- New application as windows for gas targets identified; preliminary tests show promise.



## Acknowledgements

We acknowledge and thank the Dept. of Energy for support of this program through SBIR Contract DE-SC0000852.

ANI thanks J.A. Nolen, F. Marti and S. Hitchcock for foil characterization and advice, and S. Gambhir, D.L. Officer and G.G. Wallace for providing graphene foils.

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