Applied Nanotech, Inc.

Graphene Backing for Radioisotope Targets

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Outline

- Who is Applied Nanotech (ANI)?
- Prior effort using graphene
- Problem need for radioisotope targets
- Approach using graphene material
- Phase II and future effort

About ANI



- Located in Austin, Texas
- Founded 1988, publicly traded
 - In 2014, merged with Nanofilm (Cleveland, OH), both are now subsidiaries of PEN Inc.
 - Nanotechnology R&D emphasis:
 - 1) Graphene foils and films
 - 2) Printed Electronics
 - 3) Sensors

- 4) Thermal Management
- PEN has staff of 20+ employees, ANI has 6 staff

Prior ANI Effort Using Graphene

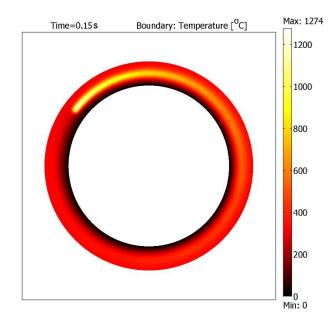
- Graphene based cold cathodes for flat panel displays
 - US Pat. 6,819,034 (2000, Pavlovsky) "Carbon flakes ... can be as thin as one or more layers of sp2-bonded carbon atoms (graphite layers)"
- Isotopic Carbon Graphene Foil Targets
 - DoE grant DE-SC0015140: The goal of this program is to develop ¹³C and ¹⁴C carbon targets for nuclear experiments
 - Fabricated ^{nat}C graphene targets, while commercially available ¹³C did not graphitize
 - Low risk approach for ¹⁴C that has high levels of impurities is to print 3-5 mg/cm² films using a binder (2% wt.)
 - Test run complete at ANL in June 2018

Prior ANI Effort Using Graphene

Graphene Stripper Foils for FRIB

- DoE grant DE-SC0000852: The goal of this program was to develop a high thermal conductivity, rigid, large area, uniform graphene foil for charge stripping in accelerators, capable of having long lifetime
- Fabricated by filtration of reduced GO. Size up to 5", typical thickness 0.1 to 1.0 mg/cm², can be cut per order
- Longer lifetime (up to 2x) in charge stripping applications compared to conventional carbon foils

Prior ANI Effort Using Graphene





- The foils are smooth and have a metallic luster
- The foils have a layered structure
- Foils are robust, easy to handle and survive express shipping

Problem

A variety of isotope targets are needed in NP research

- Need for robust backing made of a low Z material
- Targets of interest: refractory metals, targets with limited amounts of isotopes available (mg quantities), both stable and radioactive isotopes, B-11, etc.
- Effective methods of target fabrication are needed
- This program: Graphene Backing for Radioisotope Targets
- DoE Grant DE-SC0017208, currently Phase II (Topic 26(e) -Specialized Targets for Nuclear Physics Research)

Approach

- Use thin graphene backing films for target fabrication by magnetron sputtering: deposit enriched B-11 carbide
- Use graphene as a cathode for electroplating of isotopes: prepared Cr(III) bath to fabricate ^{nat}Cr plated target
- Use graphene as a matrix for composite targets where the target material is in the nanoparticle form. ^{nat}Ir nanoparticles were synthesized and used to fabricate ^{nat}Ir target with up to 50% target material mass loading.

Phase I Results

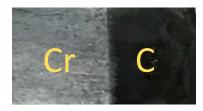
- 1. Graphene backing for target material deposition
 - Fabricated 3" enriched Boron-11 carbide sputtering target
 - Prepared graphene substrates
 - Deposited 200nm ¹¹B₄C on 0.1mg/cm² graphene: electrostatically attracted to substrate holder
 - Deposited 200nm ¹¹B₄C on 0.5mg/cm² graphene: substrate survived the film deposition in plasma



Phase I Results

- 2. Graphene backing for electroplating
 - Prepared 0.5mg/cm² graphene substrates (cathodes)
 - Formulated Cr(III) chloride electroplating bath and tuned a plating system
 - Plated 1mg/cm² natural Chromium targets on graphene substrates

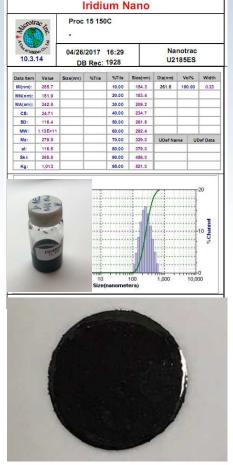






Phase I Results

- 3. Graphene matrix with nanoparticle target materials
 - Prepared ^{nat}Ir nanoparticles by reduction of H₂IrCl₆ in aqueous solution
 - Pressure filtration of graphene and target material aqueous dispersions
 - Prepared 2mg/cm² free-standing natural Ir targets in graphene matrix at 50%wt. loading



Phase II Goals

- Objective 1: Demonstrate refractory metal targets (Nb, Re*) in graphene matrix with metal mass loading of 50% or greater
- Objective 2: Demonstrate sputtering deposition of ¹¹B₄C films on graphene backing with mass area density not exceeding 0.1 mg/cm²
- Objective 3: Demonstrate W and Re* targets with area density ranging from 0.5 to 5 mg/cm² fabricated by spin coating over graphene backing
- Objective 4: Demonstrate electroplated ⁵²Cr isotopic targets with area density up to 1 mg/cm².
- Objective 5: Demonstrate ^{nat}Cu targets with area density up to 1 mg/cm² electroplated over graphene foils

*Excluded the initially proposed Mo target development per DoE request

Phase II Schedule

Task or Milestone	M1	M2	M4	M6	M8	M10	M12	M14	M16	M18	M20	M22	M24
MS1 - Program kickoff meeting	Х												
Task 1: Obtain target materials, chemicals, prepare graphene films													
Subtask 1a. Obtain materials, chemicals, components													
Subtask 1b. Fabricate graphene films for the following tasks													
MS2 - All materials needed for the program are available				Х									
Task 2: Fabricate W targets by spin coating over graphene													
Subtask 2a. Fabricate spin-coated W target on graphene													
Subtask 2b. Fabricate spin-coated Mo target on graphene													
MS3 - Spin coated targets are fabricated					Х								
Task 3: Electroplating Cu and Cr-52 targets													
Subtask 3a. Electroplate Cu target on graphene film													
Subtask 3b. Optimize natCr electroplating conditions													
Subtask 3c. Electroplate Cr-52 target on graphene film													
MS4 - Cr-52 target electroplating complete							Х						
MS5 - Year I Technical Report Complete							Х						
Task 4: Sputtering of B-11 carbide on 0.1 mg/cm2 graphene													
MS6 - B-11 carbide deposited on thin graphene film									Х				
Task 5: Refractory metal targets fabrication													
Subtask 5a. Fabricate W target in graphene matrix													
Subtask 5b. Fabricate Nb target in graphene matrix													
Subtask 5c. Fabricate Mo target in graphene matrix													
MS7 - Refractory metals targets fabricated													X
Task 6: Target fabrication per ANL request													
MS8 - Order fulfilled													X
Task 7: Program Management													
MS9 - Final Report complete													Х

Phase II Deliverables

- Deliverables include:
 - Progress Reports (RPPRs) and Final Report
 - Deliverables to be evaluated at ANL:
 - ¹¹B₄C films on graphene backing
 - spin coated W and Re targets
 - Nb, Re nanoparticle targets in graphene matrix
 - Electroplated ⁵²Cr isotopic targets and ^{nat}Cu targets
- Delivered to date:
 - Ir, Re, WO₃, HfO₂ natural isotopic targets in graphene matrix with ~50% mass loading
 - Re nanoparticles fabricated in house, other materials obtained commercially

Future Plans

- Test Ir and Re targets in graphene matrix at ATLAS
- Fabricate specialty isotopic targets for NP community
- Develop methods and targets for production of radiopharmaceuticals
- Possible application: graphene windows for X-ray sources and low energy particle beams

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