

Refractory Porous Thin Film Targets for Medical Isotope Production

Nuclear Physics SBIR/STTR Exchange Meeting

August 7-8, 2018

Sponsor: Office of Nuclear Physics, DOE

Program Officer: Dr. Manouchehr Farkhondeh

Phase IIB Grant Number: DE-SC0007572

Small Business

InnoSense LLC

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Collaborator

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Presentation Overview

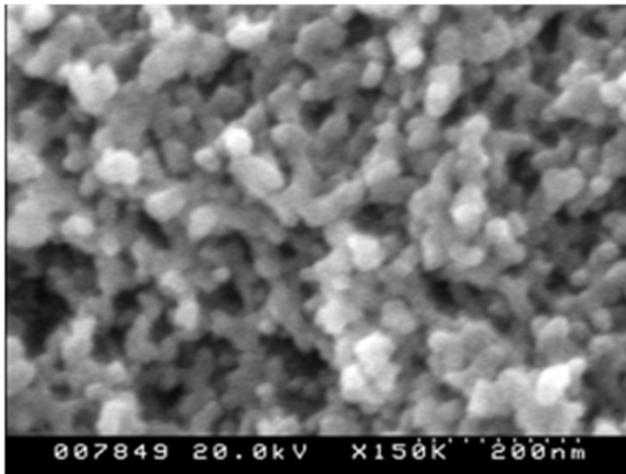
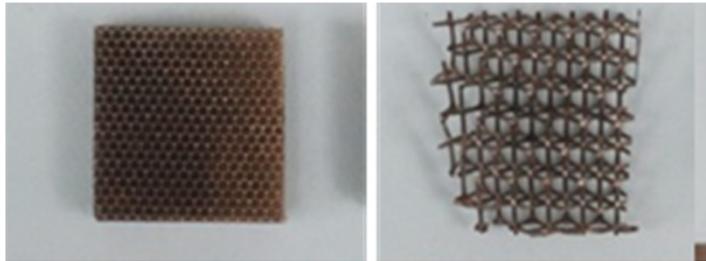
- About InnoSense LLC
- Commercialization Status
- Motivation
- Relevance to Nuclear Physics Programs
- Work in Progress
 - Refractory Oxide Porous Catchers
 - **Thin Film Porous Bismuth Oxide Targets**
- Summary
- Acknowledgments

About InnoSense LLC

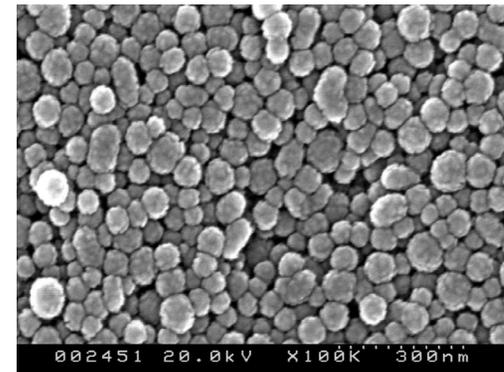
- **Established in 2002 by private investment, R&D operations in 2004, housed in a 9,000 square feet laboratory facility located in Torrance, California. Core Capabilities – Nanotechnology, Chemical and BioSensing**
 - Added 1400 sq. ft of space for testing and production capabilities – chemical sensors division
 - Planned expansion (~3000 sq. ft) for dedicated biotechnology/bioassay development
- **Seven “wet” chemical facilities equipped with fume hoods, a clean room, a spectroscopy facility, optics and chemical and biosensor testing laboratories, and two machine shops.**
- **Growth Phase - currently 28 employees, expanding to 30 soon**
 - 7 PhD, 7 MS and 2 MBA degree holders.
 - Dedicated business development team added in 2017.
 - Negotiating a large contract with MDA for production of 24/7 monitoring leak detectors
 - Gearing to spin-off divisions
 - Two Army funded efforts in preproduction/licensing activities

Commercialization – Building from ONP Funding

Silica aerogel coatings on metal lattices –
\$8,500 July 2015



Porous Scaffolds for Refractory Solar
Selective Coatings – SuNLAMP
\$200 K (2016-2017)



Prior DOE ONP funding enabled us to develop the technology for porous monoliths and expand the application base for these materials

Company Commercialization Status

Army: W15QKN-09-C-0153

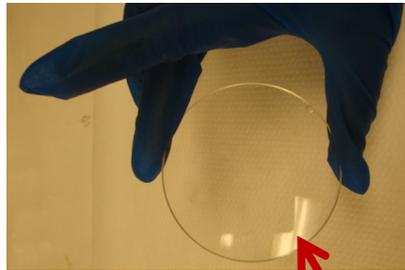
Passive Temperature Dosimeter



- Ongoing Phase III Funding ~\$1M
- Correlation Testing completed at Yuma Proving Grounds 2018
- Expanding customers in the Army to ramp up production

Army: W911NF-11-C-0056

Permanent anti-fog coating



PC lens and PU visor



- DOD DTRA RIF award – 2015
- Nanomaterials in coating
- Negotiations with DOD and Commercial vendor for licensing

MDA: HQ0147-14-C-7012

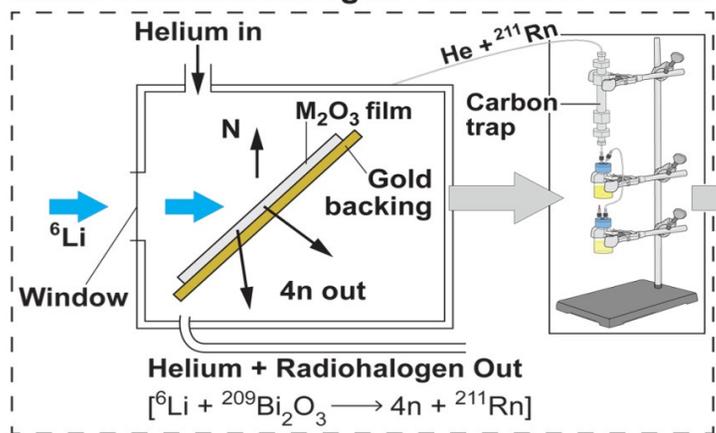
Hypergolic Leak Detector for THAAD



- Drop-in Replacement Leak Detector for MDA THAAD missiles
- \$200K Production order from Lockheed
- New contract under negotiation.

Medical Isotope Production Target Development (must be stable with beam power)

Noble Gas/Radiohalogen Production Scheme



Helium gas transports radioactive noble gas to carbon trap



where, $\text{M}_{(s)} = {}^{209}\text{Bi}$, ${}^{75}\text{As}$ or ${}^{121}\text{Sb}$

and $\text{N}_{(g)} = {}^{211}\text{Rn}$, ${}^{77}\text{Kr}$ or ${}^{123}\text{Xe}$ (Noble gases)

that decay to Radiohalogens

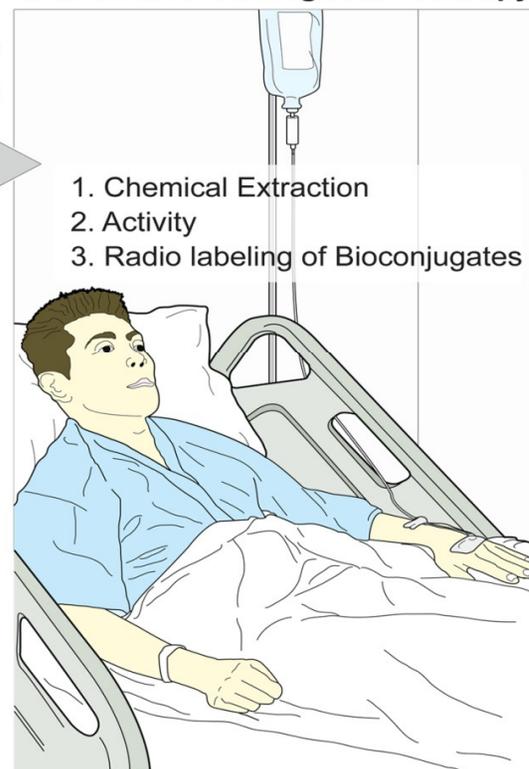
${}^{211}\text{At}$ ($t_{1/2} = 7.2$ h), ${}^{77}\text{Br}$ ($t_{1/2} = 2.78$ d), ${}^{123}\text{I}$ ($t_{1/2} = 13.4$ h)

Extraction and Chemical Preparation of Radioactive Halogen for Therapy



Overnight Transport within Continental US

1. Chemical Extraction
2. Activity
3. Radio labeling of Bioconjugates



- Efficient production and release of radioactive noble gas precursors at low and room temperature – **Higher production rates of ${}^{211}\text{At}$, ${}^{77}\text{Br}$ and ${}^{123}\text{I}$**
- ${}^6\text{Li}$ induced reaction for parent/daughter production system, concept for a dedicated linac or cyclotron for radio-halogen production – **Overnight delivery to users from single national facility.**

Catchers/Targets Being Studied at ISL and ANL

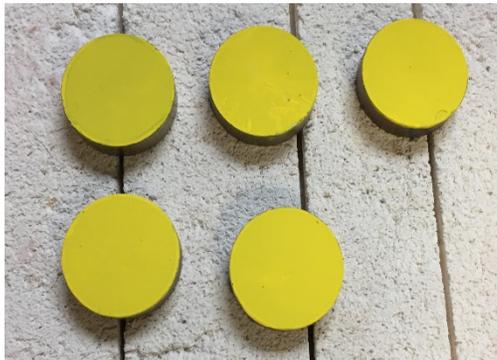
Refractory Catcher/Target	Production beam	Collected Isotopes
Tungsten-coated SiO ₂ Aerogel	¹⁸ O (typical)	⁸⁻¹¹ Li ^{6,8} He
Carbon Aerogel	¹⁶ O, ⁴⁸ Ca, etc.	¹² C ¹⁴ O– ¹² C ²⁴ O ¹² C ¹⁴ O ₂ – ¹² C ²⁴ O ₂
Ytria-Stabilized Zirconia (YSZ) and Hafnia (HfO ₂) Porous Monolith	¹² C, ⁴⁸ Ca, etc.	⁹ C ¹⁶ O– ²² C ¹⁶ O ⁹ C ¹⁶ O ₂ – ²² C ¹⁶ O ₂
Sintering-inhibited Disks of Tungsten, Tungsten + ALD-Hafnia and Tungsten Carbide	¹⁸ O, ⁴⁸ Ca, etc.	“All of the above”
Nanoporous CaO Monolith	⁴⁰ Ca	³¹⁻³⁵ Ar
Nanoporous Metal Oxide (M ₂ O ₃) Thin Films* (M = ²⁰⁹ Bi, ⁷⁵ As, ¹²¹ Ab)	⁴ He, ^{6,7} Li	²¹¹ Rn/ ²¹¹ At, ⁷⁷ Kr/ ⁷⁷ Br, ¹²³ Xe/ ¹²³ I t _{1/2} [14 h/7.4 h]; [1.24 h/2.78 d]; [2.08 h/13.4 h]

* Thin film targetry for medical isotope production

Refining Bismuth Oxide Thin Film Processing



As-Deposited

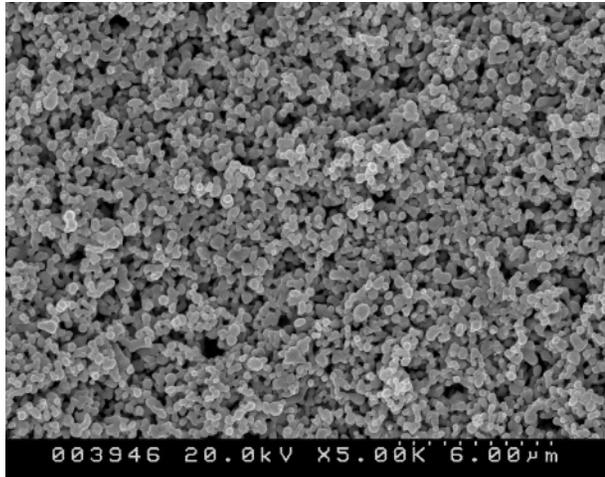


Fired @600 °C; 3x

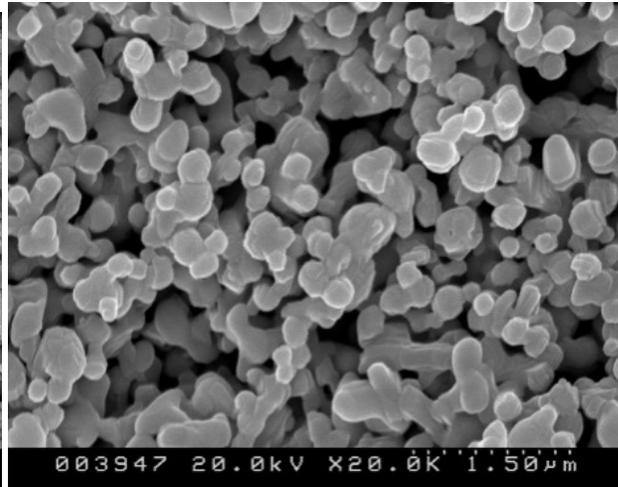


Nanoporous Bismuth Oxide Thin Films

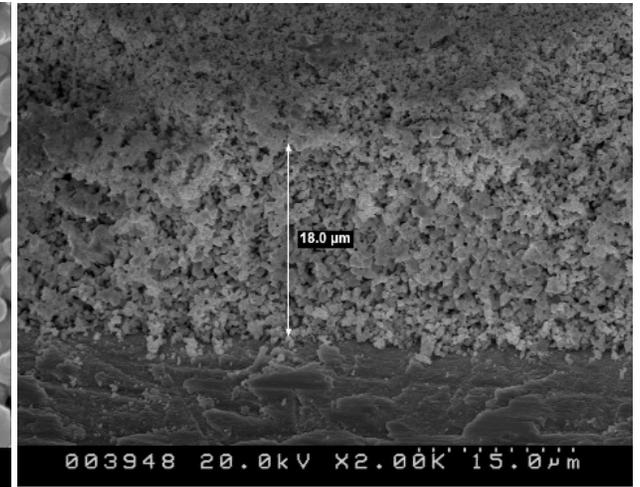
x5K



x20K



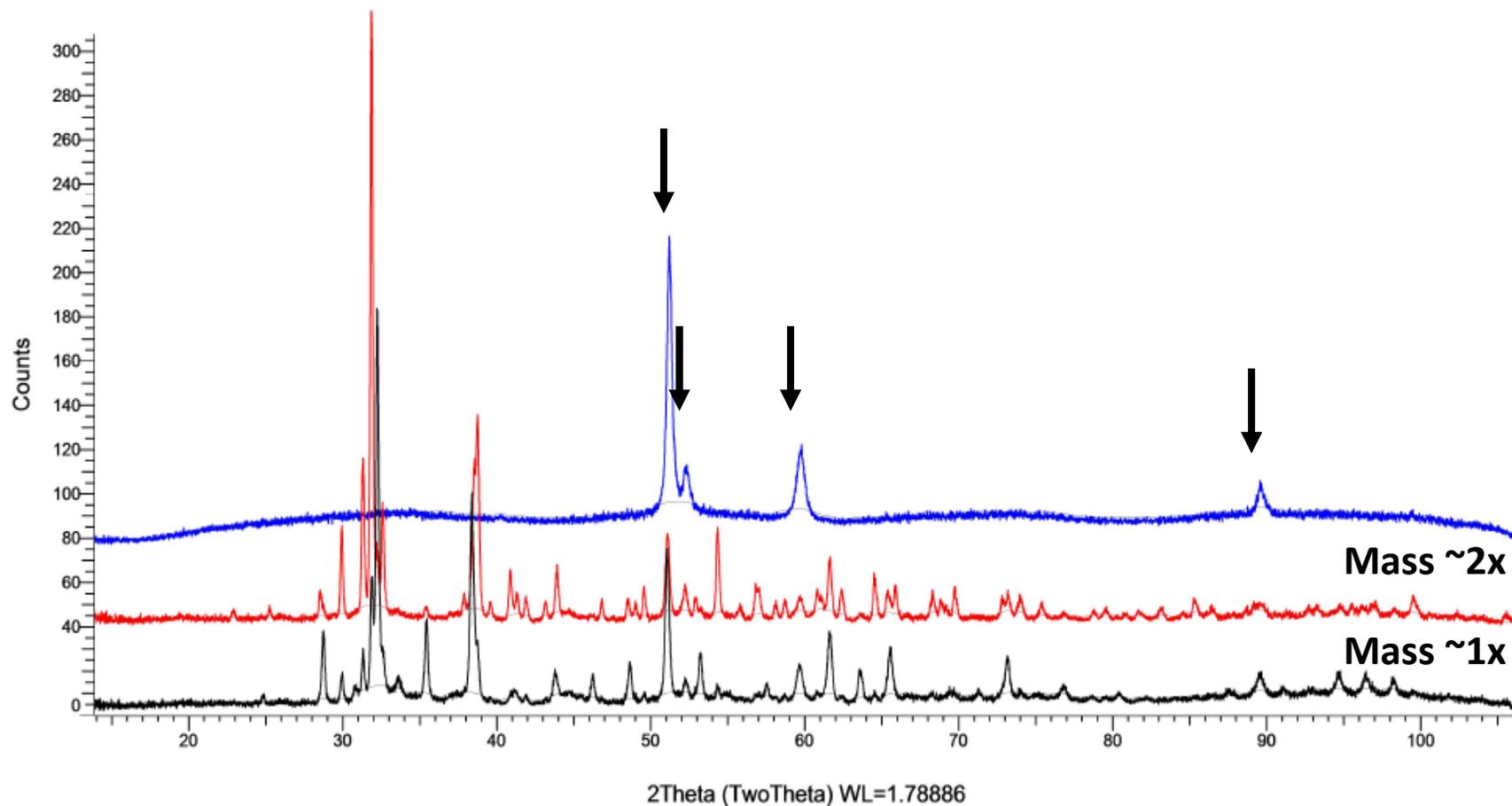
Imaged @ 44.6 degree



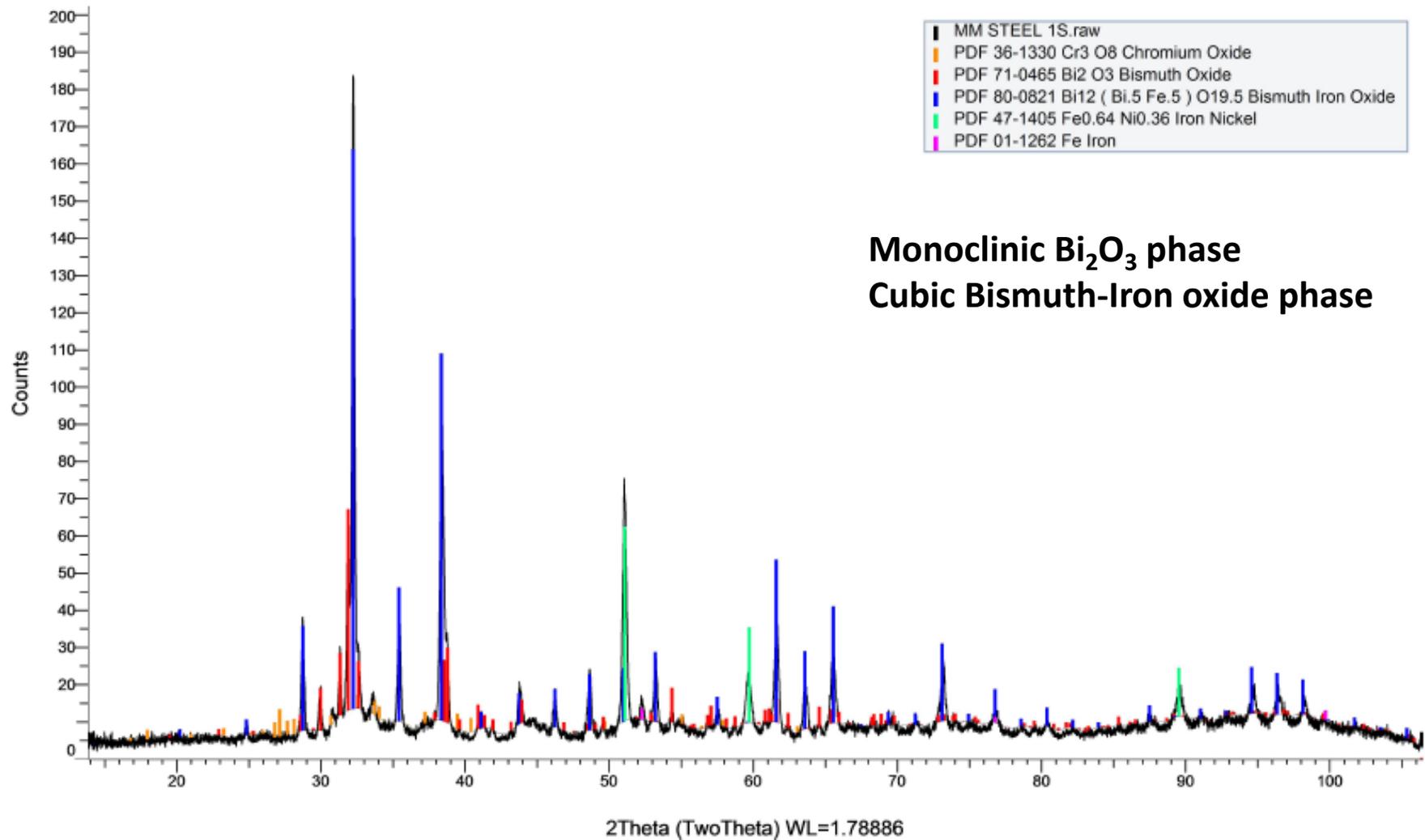
- Processing developed for contiguous nanoporous bismuth oxide films on stainless steel 303 coupons (0.5" thick, 7/8" diameter)
- Film thickness ranged from 9-15 mg/cm²
- Films remain adhered after 3x in vacuum heating to 600 °C
 - Some mass loss noted - investigating this
- Tested at ATLAS in May 2018 with energetic ⁶Li beams for formation and release of radiohalogen Radon-211 to decay to Astatine-211.

X-Ray Powder Diffraction of Bismuth Oxide Films on 303SS Substrates

MM STEEL 1S.raw
MM STEEL 2S.raw
STEEL BLANK 303.raw



Indexed to JCPDS Files



X-ray Photoelectron Spectroscopy of Bi_2O_3

S1-surface0001_1.SPE:

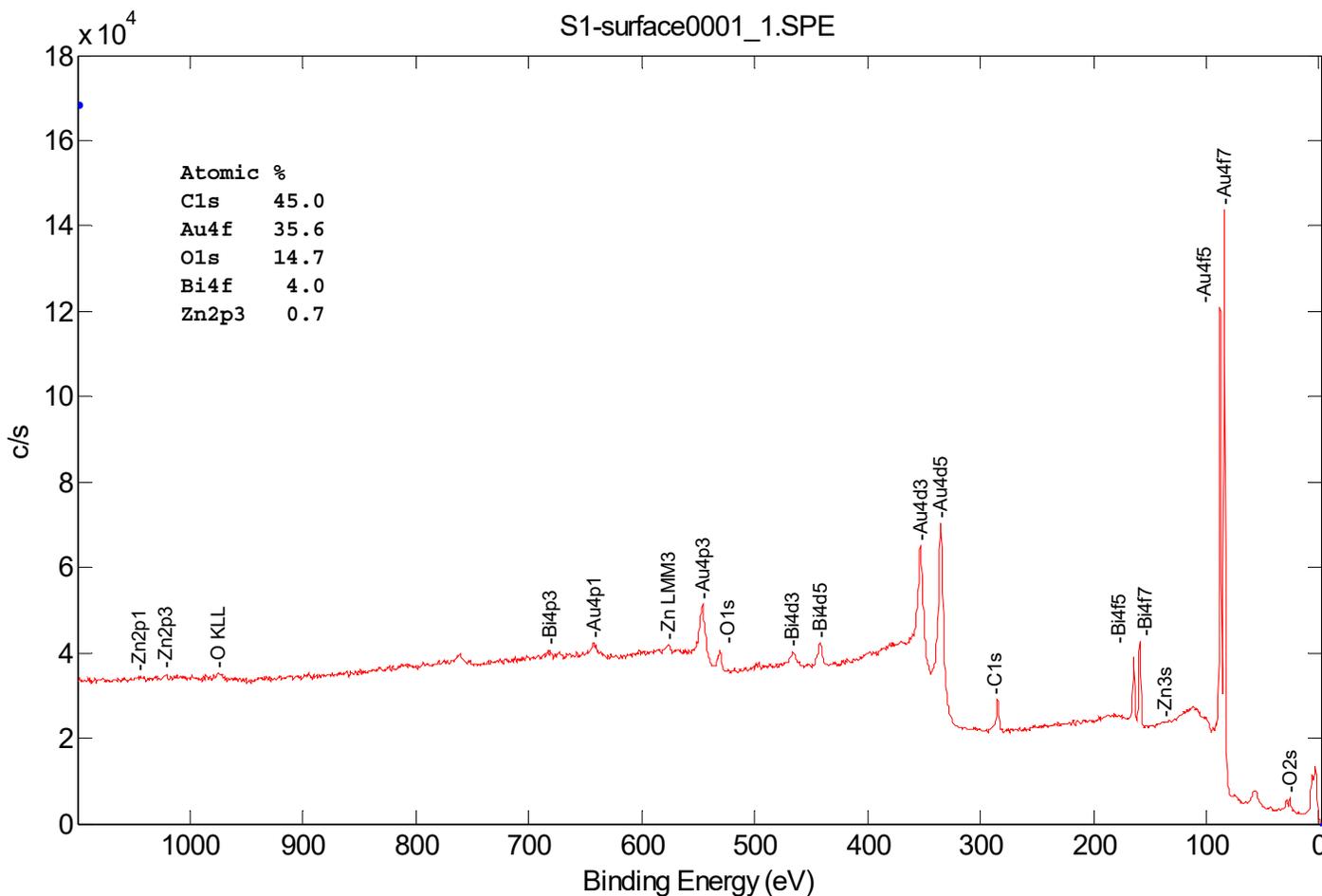
2018 May9 Al mono 0.0 W 200.0 μ 44.7° 93.90 eV

SUR/Area1/1 (SG5 Shift)

1.4385e+005 max

Company Name

9.17 min

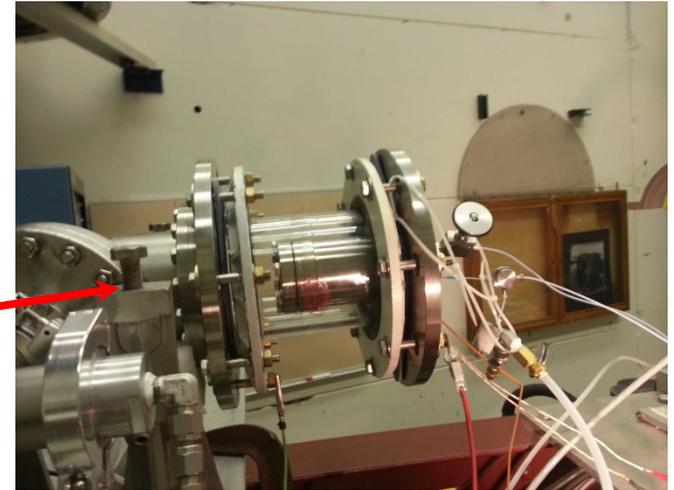


Initial Tests at ATLAS with a Bismuth Metal Target



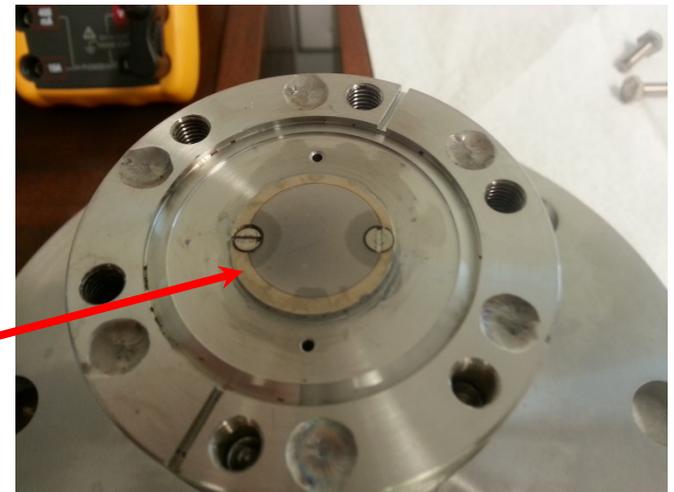
Health physicist,
Post-doc,
Undergraduate

Target/ helium
plumbing/ heater
assembly



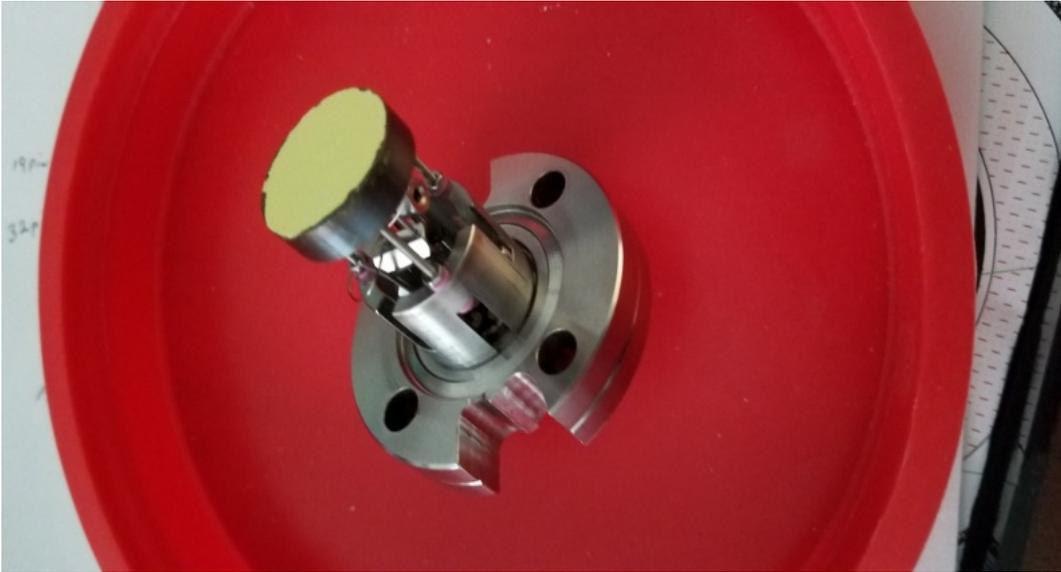
Havar window

32 mg/cm² Bi on
Ni



Release and capture efficiency ~1% at ~200 °C

First Test at ATLAS with Bismuth Oxide Target



Sample 3 = 15.08 mg/cm²
area density



Sample chamber at
ATLAS beam line

Test Setup at ATLAS

Helium
circulation
pump and
cold trap



Cold trap
and Ge
gamma
detector



Counts
gammas
from ^{211}Rn
transferred
to charcoal

Off-line counting of long lived ^{207}Bi

50% of ^{211}Rn (14 hour) decays to ^{207}Bi (32 year), so counting the ^{207}Bi is useful for tracking the final location of the $^{211}\text{Rn}/^{211}\text{At}$



Counting the ^{207}Bi gammas from the cold charcoal trap (1% transferred at 65 °C)



Counting the ^{207}Bi gammas from the Bi_2O_3 target. Results scale to 64 μCi of ^{211}Rn produced @ 30 μA current, 10 h.

Summary

- Refractory nanoporous bismuth oxide thin films developed on 303 stainless steel substrates
- Retain open porosity and remain adhered through thermal cycling – robust, ceramic-like coating on backing
- Tested 15 mg/cm² target on-line at ATLAS to produce ²¹¹Rn
- Next steps (at Argonne)
 - Improve sample heater to reach 600 °C to increase release
 - Add sample chamber neutron shielding to permit higher beam currents (ANL funded) and on-line counting of yields
 - Quantitatively determine ions/cm² limit of target lifetime
- Ultimate goal is to use large area Bi₂O₃ targets for production and distribution of ²¹¹Rn/²¹¹At radioisotope generator for cancer therapy.

Acknowledgments

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Dr. Michelle Shinn**