Thermo-Mechanically Stable Tungsten Powders as Solid Catchers for the Fast Release of Stopped Rare Isotopes

Nuclear Physics SBIR/STTR Exchange Meeting

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Small Business

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Collaborator

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Presented on Aug 8, 2018

Presentation Overview

- About InnoSense LLC
- Commercialization Status
- Motivation
- Relevance to Nuclear Physics Programs
- Work Completed
- 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 strengths – 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, BSL 2 hood, optical, 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; 4 issued patents, several pending
 - 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





PC lens and PU visor



- DOD DTRA RIF award 2015
- Nanomaterials in coating
- Negotiations with Company who supplies DOD and other markets 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 in 2017-2018
- New contract under negotiation



Refractory Hot Catchers for Rare Isotopes (no primary beam power)



- Porous solid catchers with thicknesses in the range of ~20 g/cm² will complement gas catchers which are the FRIB base-line concept for stopping energetic rare isotopes and delivering them for stopped beam research or for reacceleration.
- Tungsten catcher to stop and release ¹¹Li and ^{6,8}He isotopes

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.	$^{12}C^{14}O_{-}^{12}C^{24}O_{-}^{12}C^{24}O_{2}$
Yttria-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_2O_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 _½ [14 h/7.4 h]; [1.24 h/2.78 d]; [2.08 h/13.4 h]

* Thin film targetry for medical isotope production

Catcher Thickness Considerations



- Desired areal density (η) or thickness for efficient isotope capture can range from 3–20 g/cm² depending on the material used.
- Areal density can be related to the apparent volumetric density as:
 - η =ρL
- This value is used to screen catcher disks after the 1000–1500 °C vacuum heat treatment

Must be thick to stop high energy radioactive beams at FRIB

Refine Processing of Candidate Powders



- 0.6–1 µm Tungsten
- 150–300 nm Tungsten Carbide
 - Porogens open pores in disks
 - Diameter ~12.6 mm
 - Thickness ~1.5 mm
 - Stacked for x g/cm² (catcher thickness)

Before Firing

600 °C/4h; 1400 ° C/2h in Ar 1000 °C/1 h in 10⁻⁵ HPa



- Change in disk dimensions <3% post processing
- Mass change ~ 3-4%



Open Porosity Retained Post Heating in Argon and Vacuum

Tungsten (W)



Tungsten Carbide (WC)







004783 20.0kV X20.0K 1.50 m

Avg. Pore Dia.: ~1.25 μm



04791 20.0kV X20.0k 1.50 m

~500 nm

- Minimal grain growth and sintering achieved
- Open porosity retained
- Apparent density (g/cm³)
 - W = 7.71±0.22 (n=15)
 - WC = 4.51±0.16 (n=20)
- Apparent Porosity
 - W = 60%
 - WC = 71%
- Intrusion Porosity
 - W~60%
 - WC ~71%



Setup for solid stopper tests at NSCL

- First on-line test of the porous solid tungsten WC catchers at NSCL using a very short-lived isotope (⁸He, 119 msec)
 - Collaboration of Argonne and NSCL scientists in May, 2018



Setup for solid stopper tests at NSCL







Sample Chamber and Count Rate in Nal Detector







"Effective" half-life of ⁸He decreased with heater power up to present limit of 400 W, ~500 °C. Release estimate ~20% - very good for a noble gas at this low temperature





Summary

- Processing methods developed for refractory nanoporous tungsten and tungsten carbide powders for use as solid catchers
 - 12.5 mm diameter, 1.5 mm thick disks stacked for 1 cm thick catcher
 - Apparent density
 - Tungsten ~7.7 g/cm³
 - Tungsten carbide ~4.5 g/cm³
 - - Tungsten ~60%
 - Tungsten carbide ~71% (tested on-line at MSU/NSCL)
- Successful on-line test conducted at NSCL in May 2018
 - ~20% release of very short-lived ⁸He (119-msec half-life) already at relatively low temperature, ~500 °C

The participants in this test at the NSCL were: Jeongseog Song, Ravi Gampa, Jim Specht, John Greene, Matt Gott, and Jerry Nolen from Argonne, and Mauricio Portillo, Antonio Villari, Mathias Steiner, and Tom Ginter from MSU/NSCL.



Future Plans

- Initial results are promising
- Further testing is dependent on FRIB interest and resources being made available
 - Variety of materials have been processed into disks and waiting to be tested (backlog from multiple projects)
 - Tungsten
 - Tungsten Carbide
 - ALD-Hafnia coated Tungsten
 - Carbon
 - ALD-Tungsten Coated Silica Aerogels
 - Carbon Aerogels
 - Feedback, required to refine the materials processing
 - Potential users RIBF at RIKEN; RISP in South Korea; GANIL in France



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