SAND2008-8155C

Nanoparticle Synthesis Protocols For NINE LDRD/GoodYear CRADA

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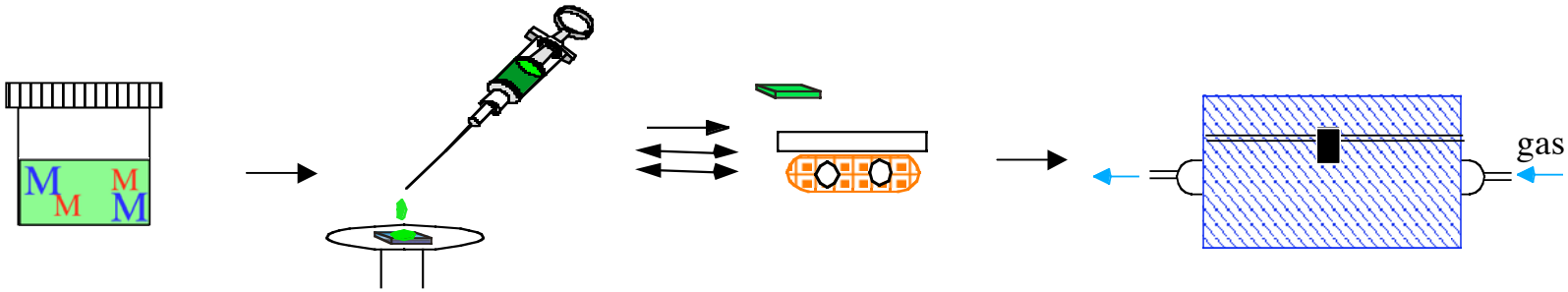
This work was partially supported by the Department of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

Synthetic techniques for the production of ceramic materials

Goal: Develop tailor-made precursors to generate optimized materials.

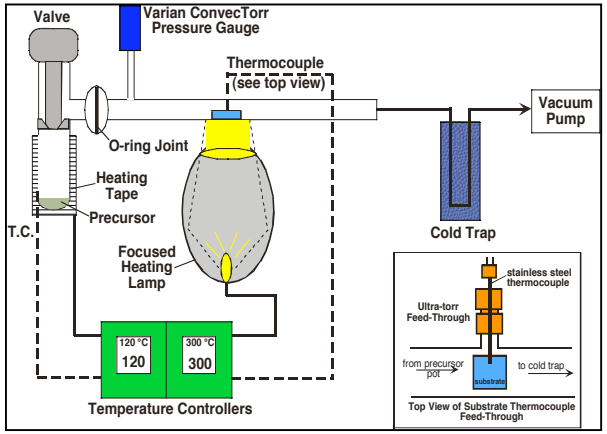


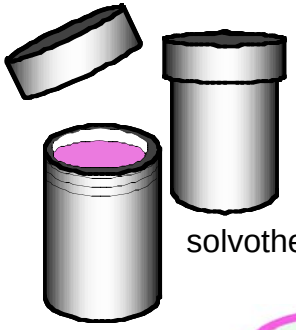
Sol-Gel

MOCVD

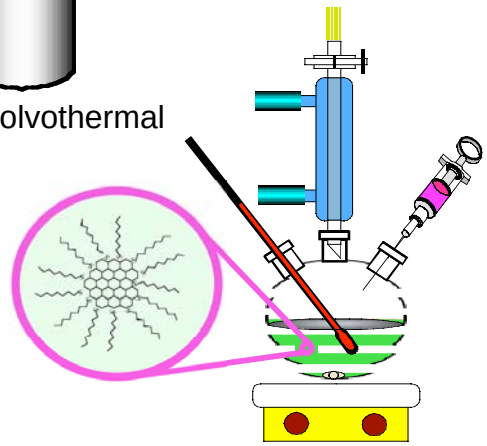
Nanoparticles

Electrospinning

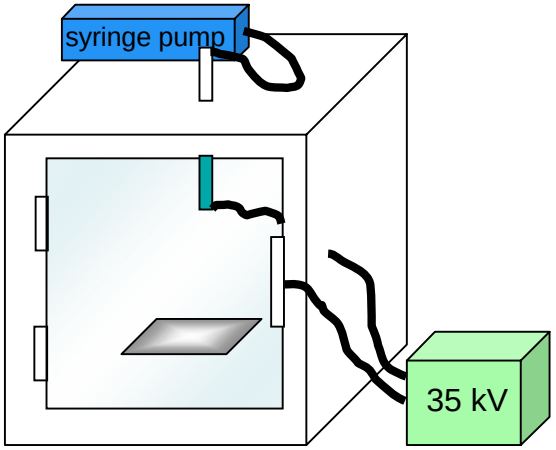




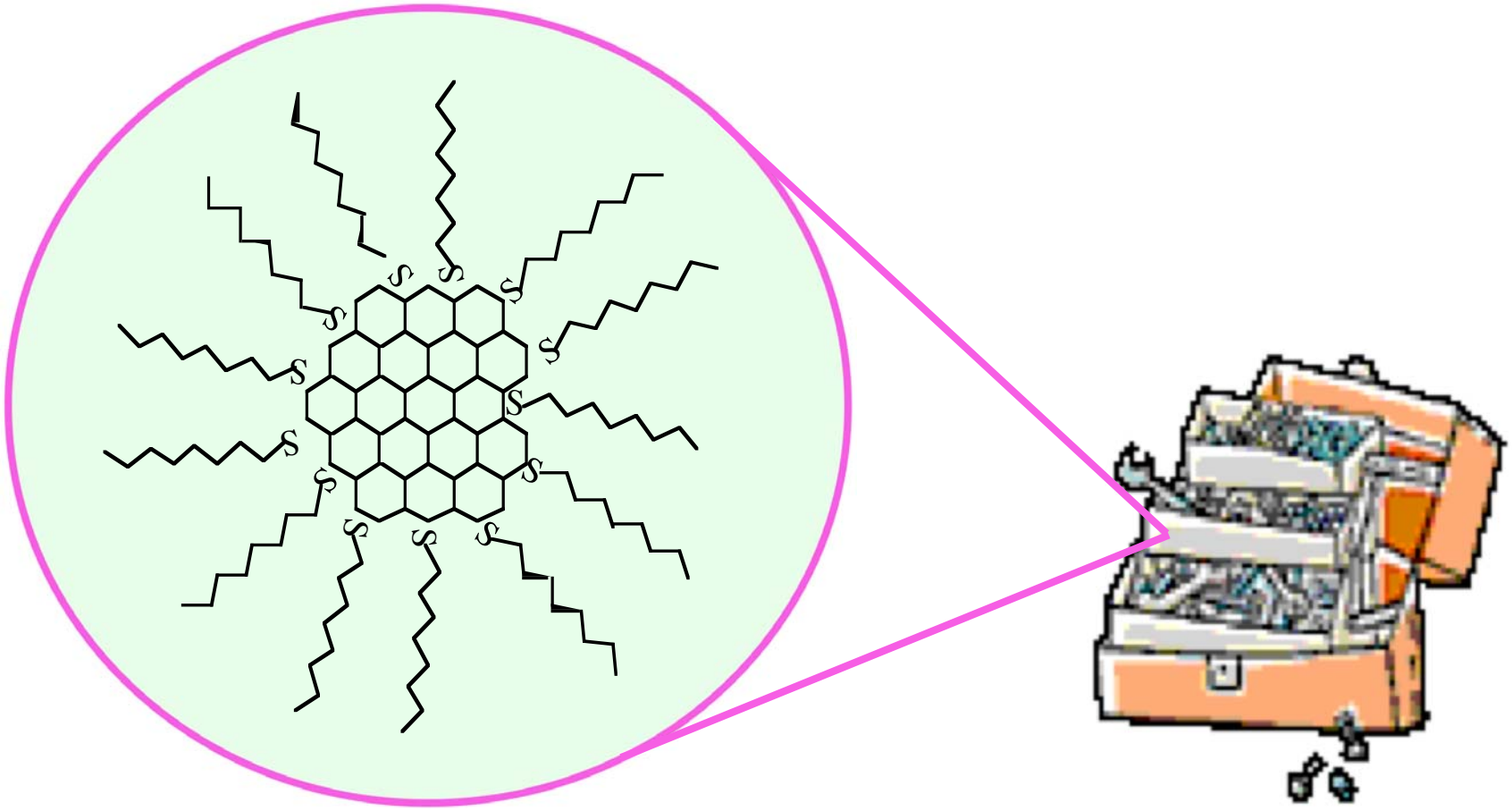
solvothermal



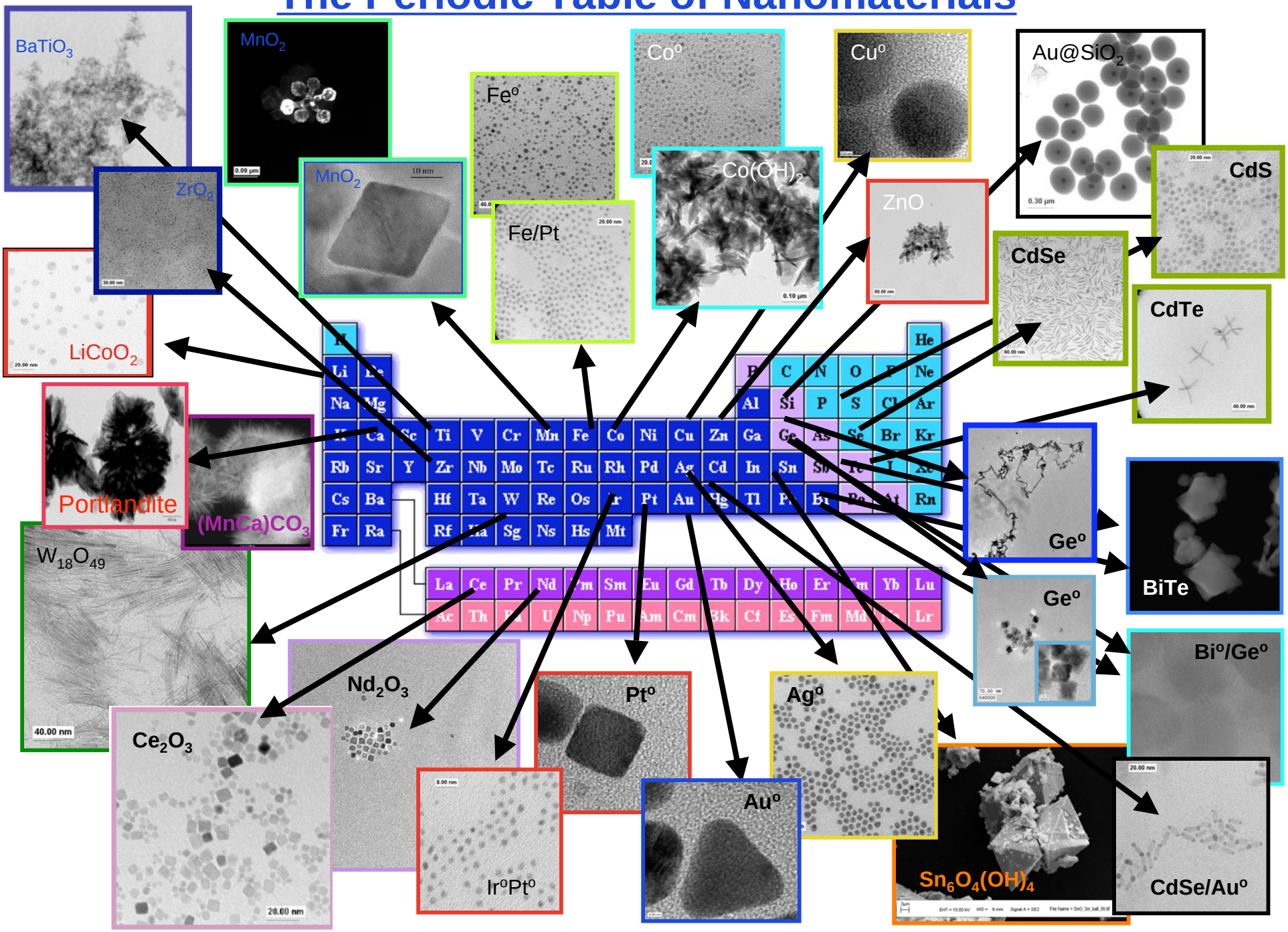
Solution precipitation



There is a great need to develop a ‘Nano tool-box’



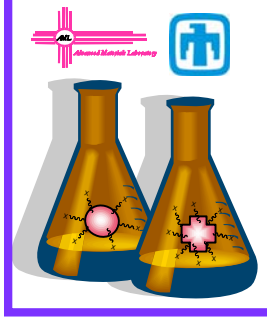
The Periodic Table of Nanomaterials

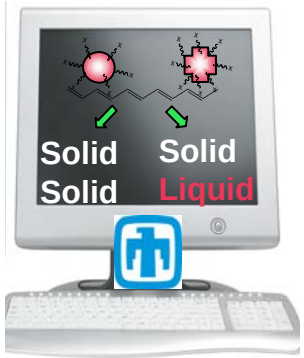


NINE-LDRD/CRADA

Goal: Develop a fundamental understanding of nanocomposite elastomers such that properties can be deduced *a priori*.

Nanoparticle Synthesis and Functionalization





Computer Modeling

In situ Mechanical Experiments

ABQ
CA

PURDUE UNIVERSITY

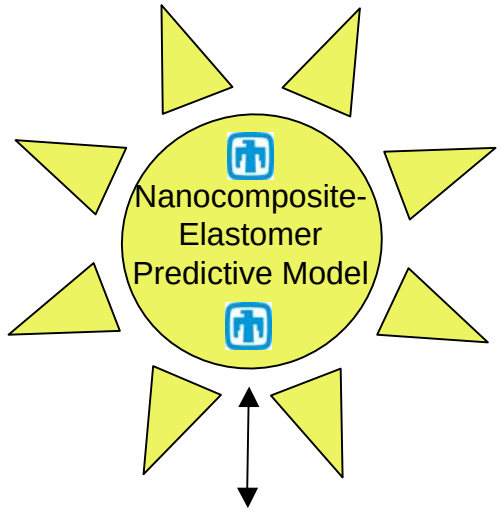
Spray Pyrolysis Large Scale Synthesis

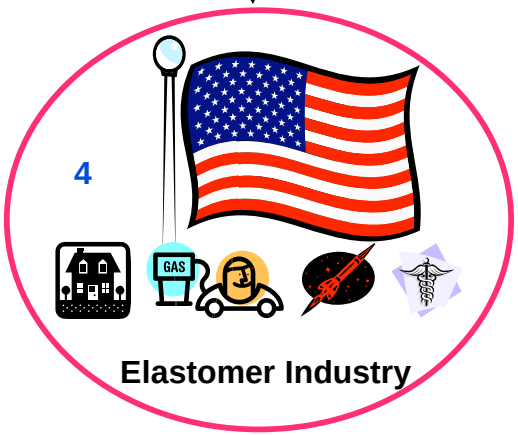
THE UNIVERSITY of NEW MEXICO

GOODYEAR

Functionalization
Nano-interface Characterization

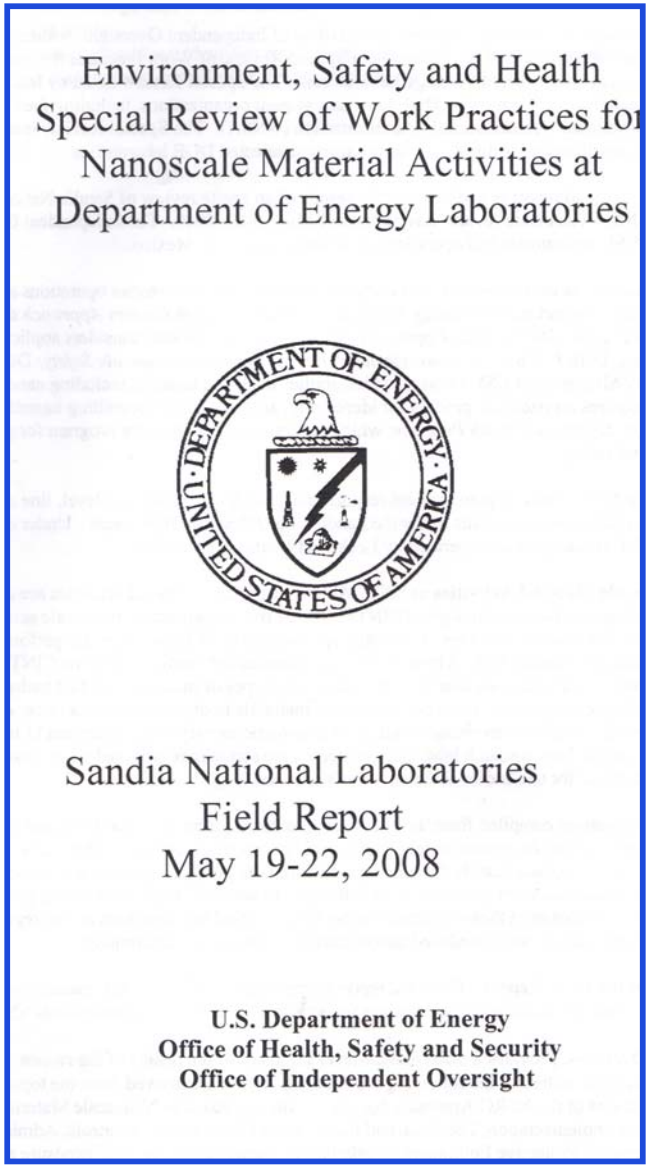
PURDUE UNIVERSITY





Elastomer Industry

DOE Guidance for Nanomaterials Safety at Sandia


Environment, Safety and Health
Special Review of Work Practices for
Nanoscale Material Activities at
Department of Energy Laboratories

Sandia National Laboratories
Field Report
May 19-22, 2008

U.S. Department of Energy
Office of Health, Safety and Security
Office of Independent Oversight

ESH Chapter 6Q defines SNL requirements for nanomaterial safety

ISMS star: - nanomaterials issues need to be specifically addressed **(Nano-training on-line).**
Oversight: 6Q did not contain include all recommended nanomaterial controls.
Working health monitoring: medical monitoring procedures for workers with nanomaterials.

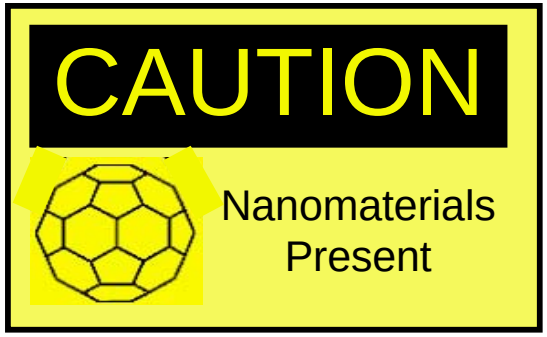
Marking, Labeling, Signage: standard labels/postings for nanomaterial use.
Chem. Inventory and MSDS: need to address nanomaterials for both documents.
Management of nanomaterial waste streams: waste containing nanomaterials identified/labeled.

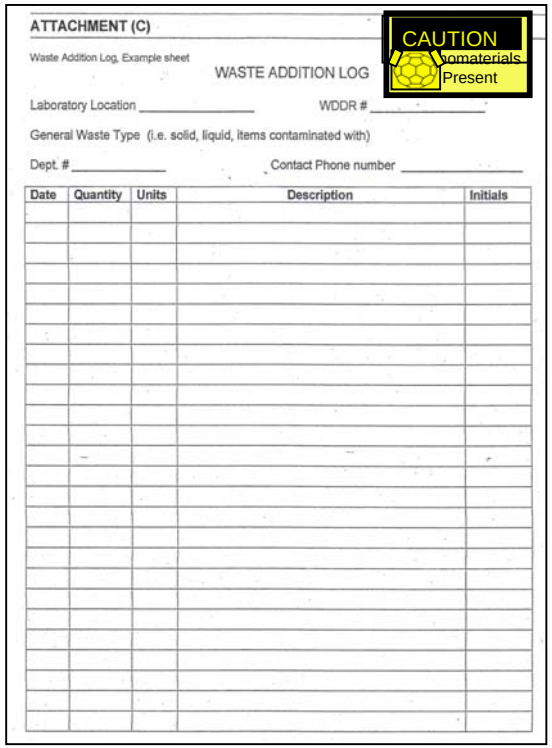
Engineering controls: HEPA filtration.
PPE: emphasis on glovebox use.

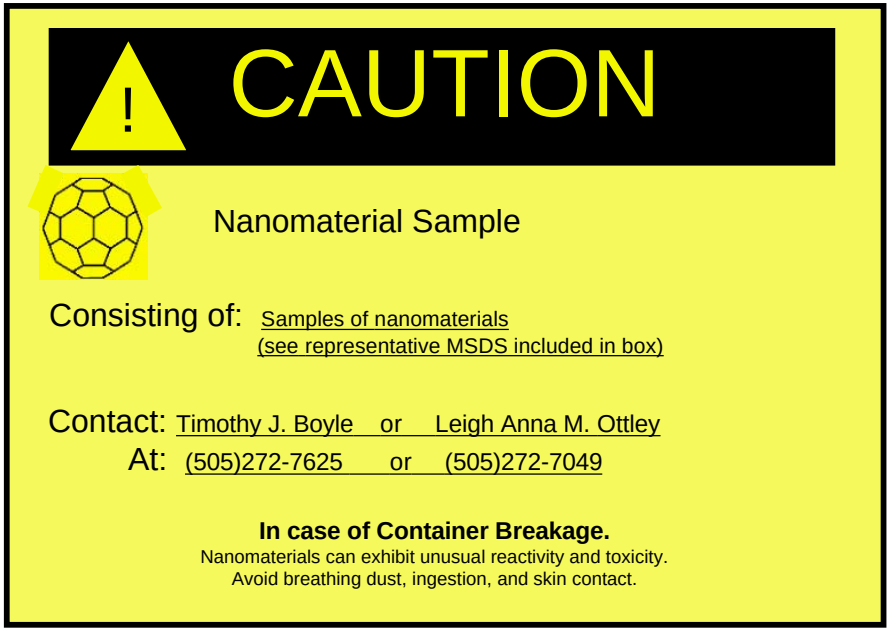
Transport: ?????

Nanomaterials Safety at Sandia

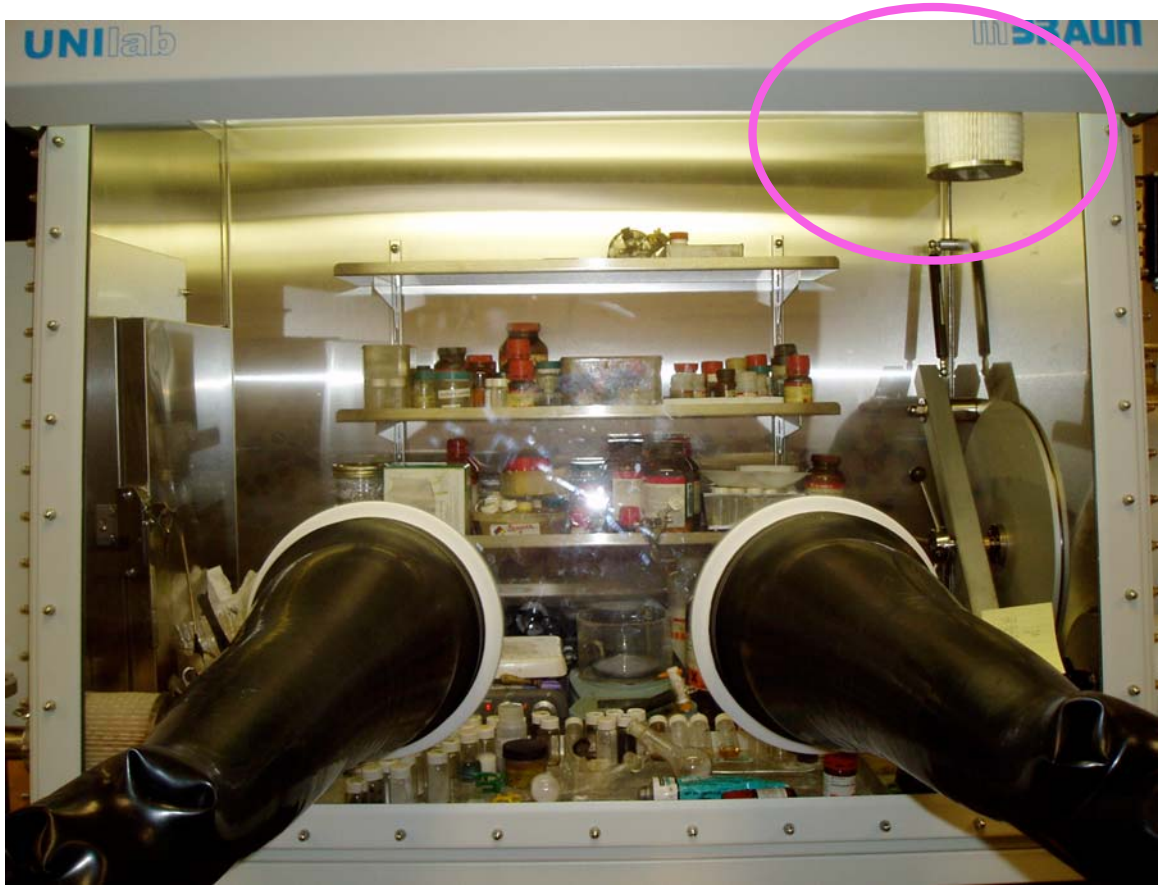
Signage for Dry dispersible nanomaterial's (DDNM) handling developed and implemented







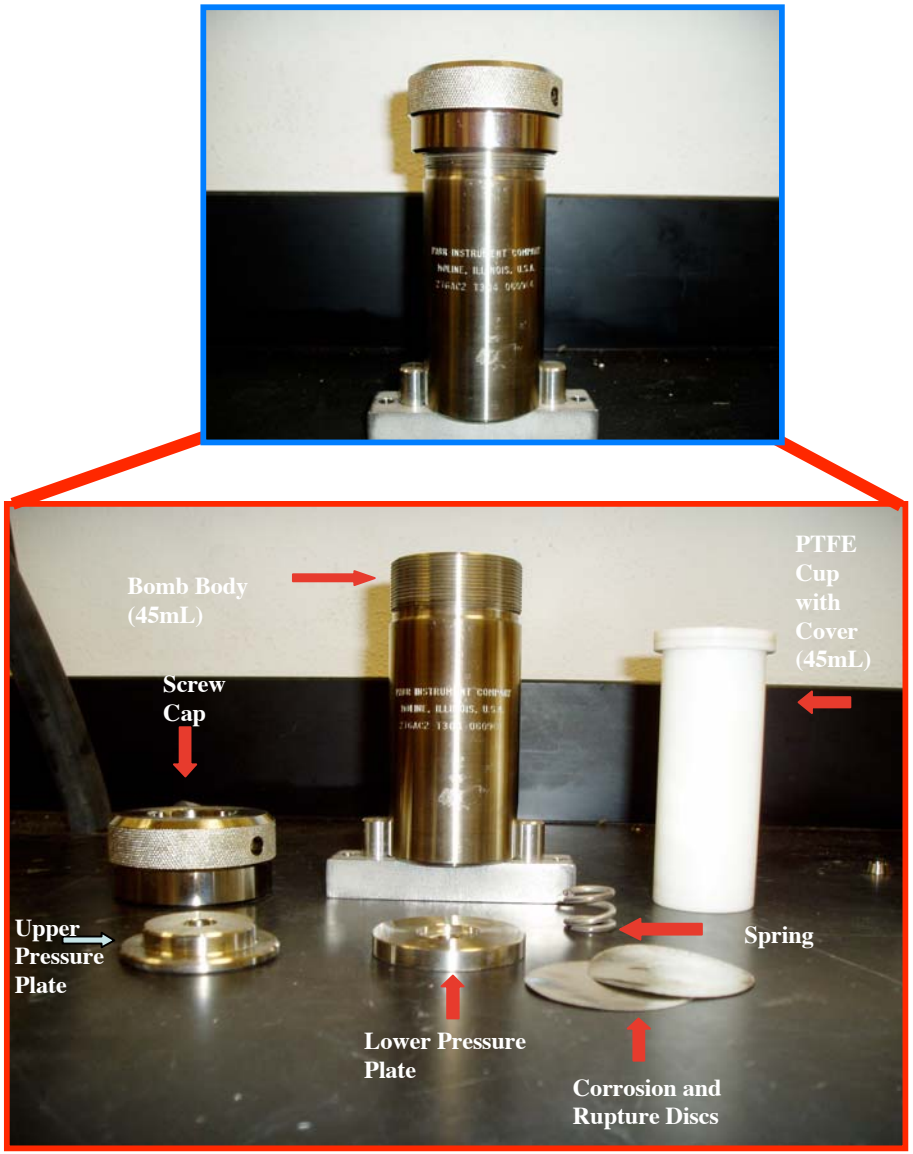
Glovebox that use nanomaterials have all purge ports HEPA filter protected



HEPA filtered outlets installed allows for air-sensitive handling of DDNM

Nanoparticles Synthesis Routes

Solution Precipitation Route

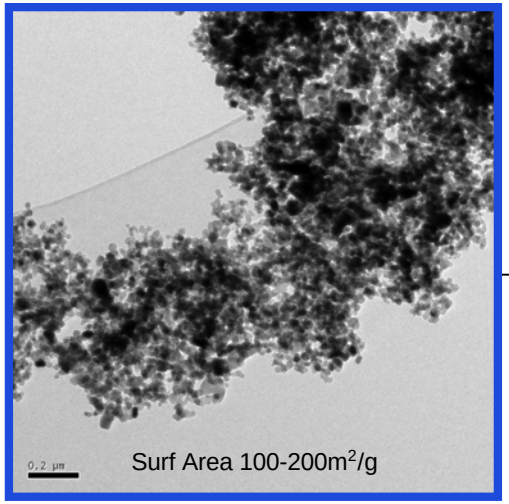


Parr Digestion Bomb

Bulk DDNM Handled in a newly installed negative HEPA filtered purge box

General purpose negative box available for handling DDNM

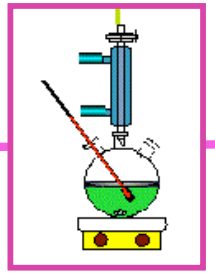
TiO_2 nanowires of various thicknesses synthesized under a variety of conditions



TiO_2 Nanopowder
+
(xs) KOH(aq)

Hydrothermal

7 days @
160 °C

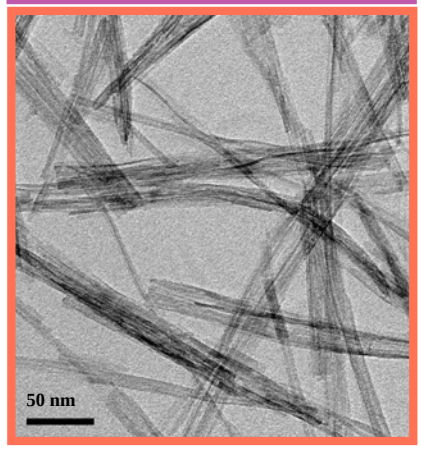


Solution Precipitation

3 days @
100 °C

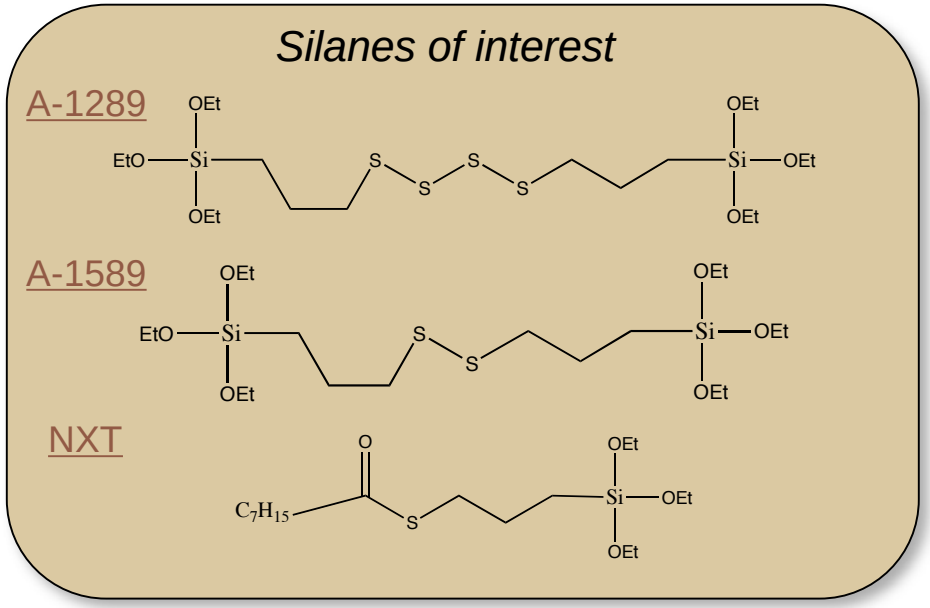
Hybrid

3 days @
100 °C

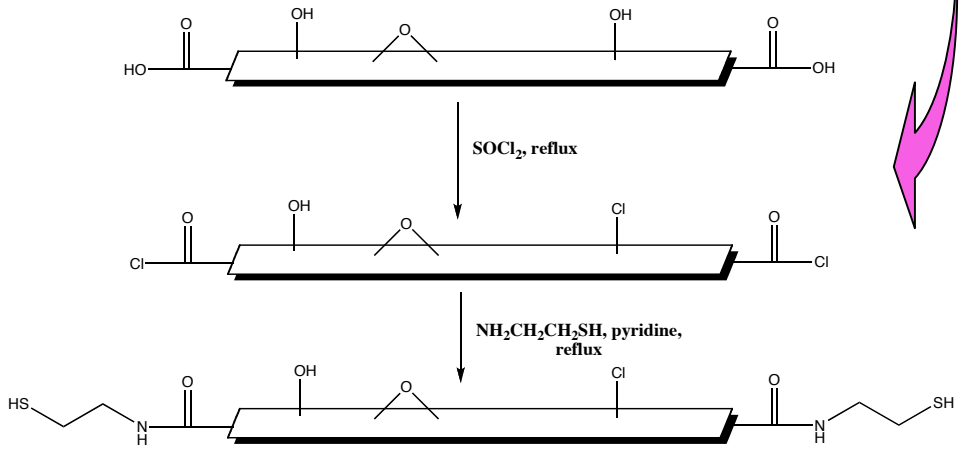


All DDNM synthesized and handled in a hood, <u>wearing appropriate PPE and capped when dried.</u>

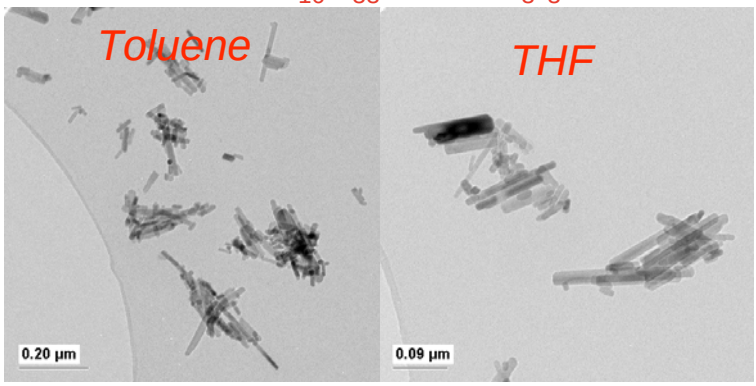
Once synthesized, DDNM must be chemically manipulated to possess desired properties



TiO_2 NW + 0.5 A-1289 $\xrightarrow[120\ ^{o}C,\ 24\ h.]{Tol\ 0.25\ NEt_3}$

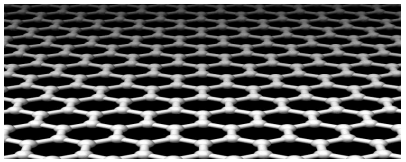


1.0 g ZnO Rods, 0.2 eq. silane in THF, $C_{16}H_{33}$-$Si(OCH_3)_3$



T. Furuzono *et al. Journal of Materials Science Letters*, 22,**2003**, 1737-1740

Graphene oxide

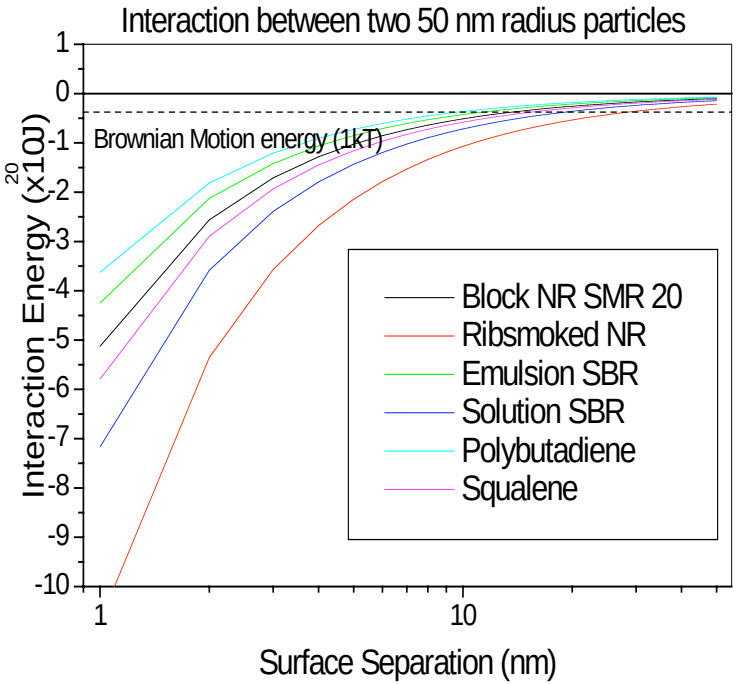


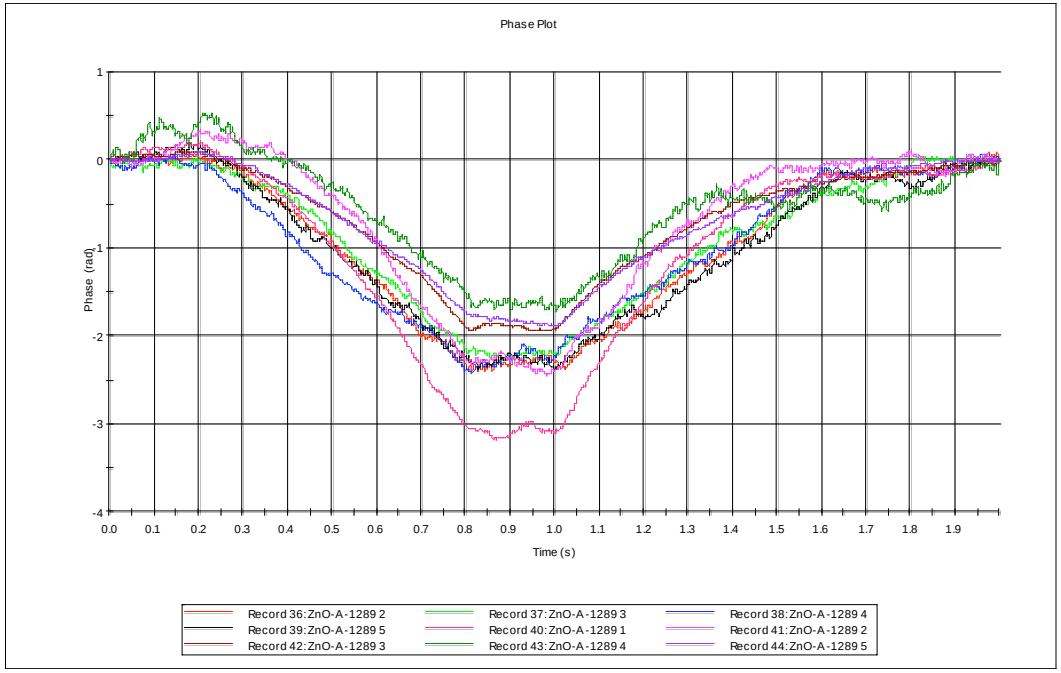
T. Lambert

Transport from the AML now involves <u>triple packaging for all DDNM.</u>

Appropriate labeled triple packaged used to transport TEM grids of Nanomaterials

Transport from the AML for NM: elastomer interfacial measurements.





Phase plot for ZnO A-1289 materials during zeta potential measurement.

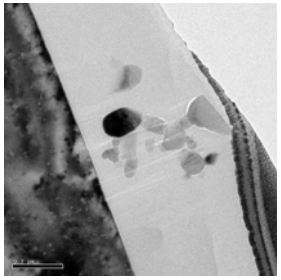
- Wetting Interactions show that all surface modifications favor mixing in rubber, but dispersion is not guaranteed.

- Van der Waals interactions show that 10-20 nm separation distance needed for non-interacting reinforcement.

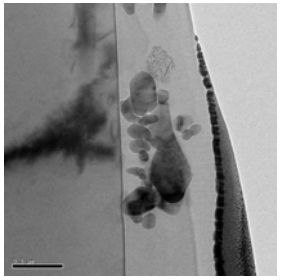
- Light scattering in squalene and zeta potential characterization of the particles attempted to quantify dispersion in rubber matrices.

Aggregate of ZnO in transverse sections of Polyisoprene cast by electrospinning from Toluene-DMF solutions.

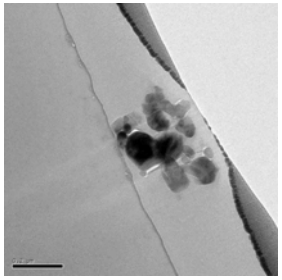
A-1289



A-1589

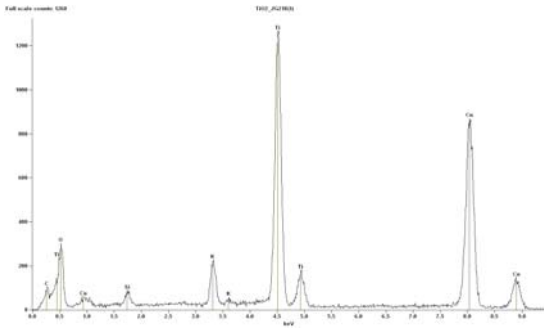


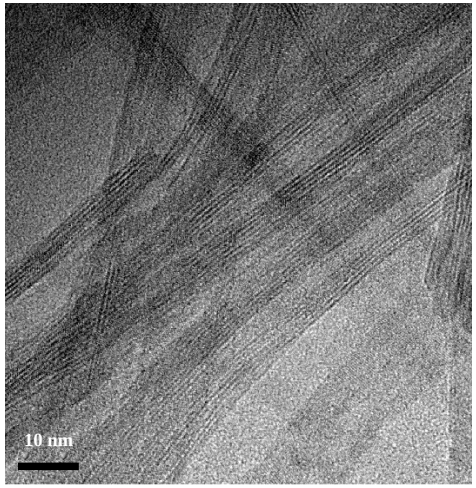
NXT

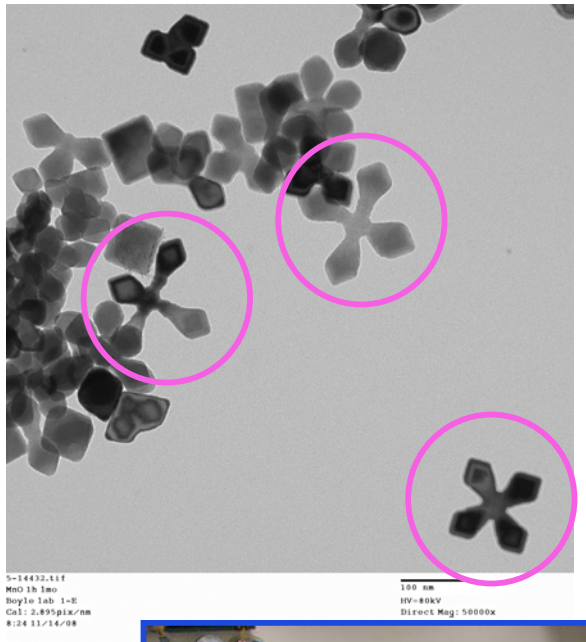


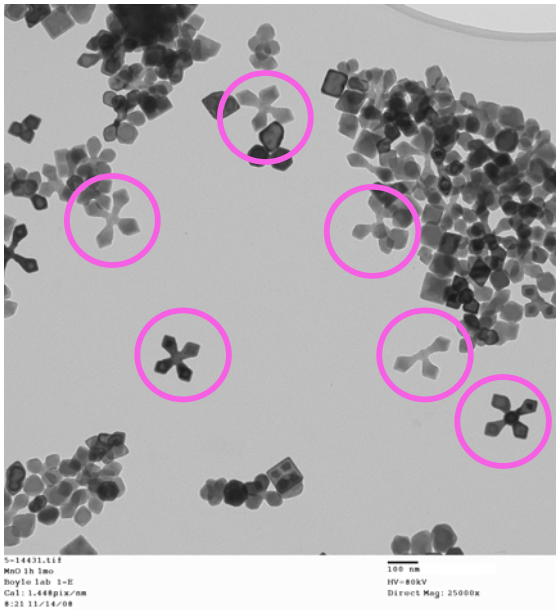
N. Bell

Transport from the AML also to TEM for <u>imaging and measurements.</u>



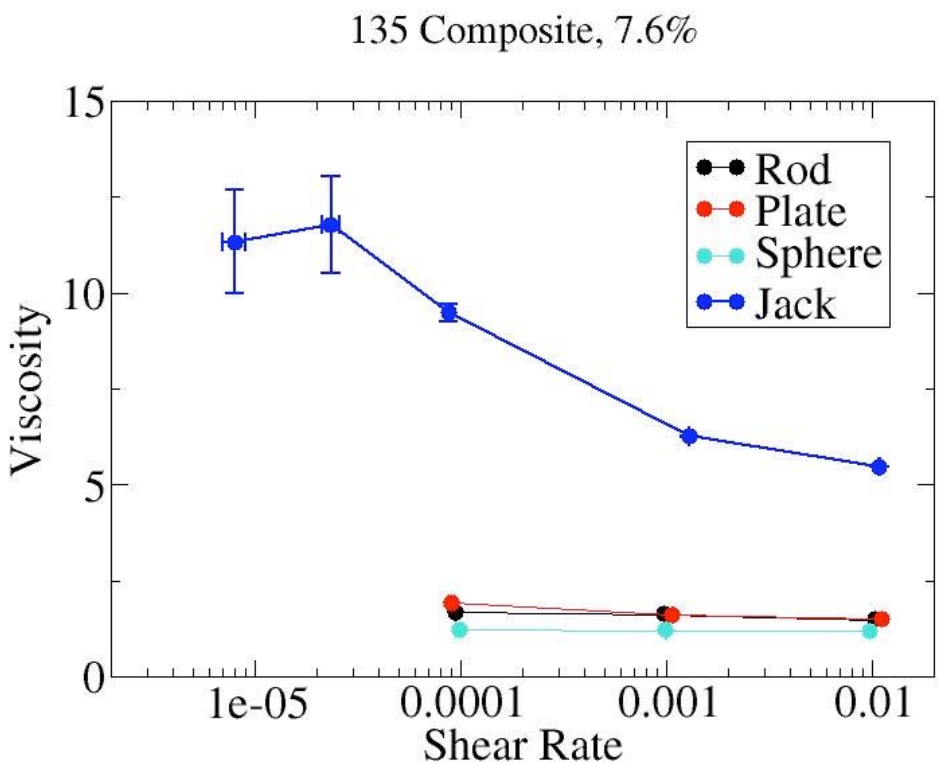


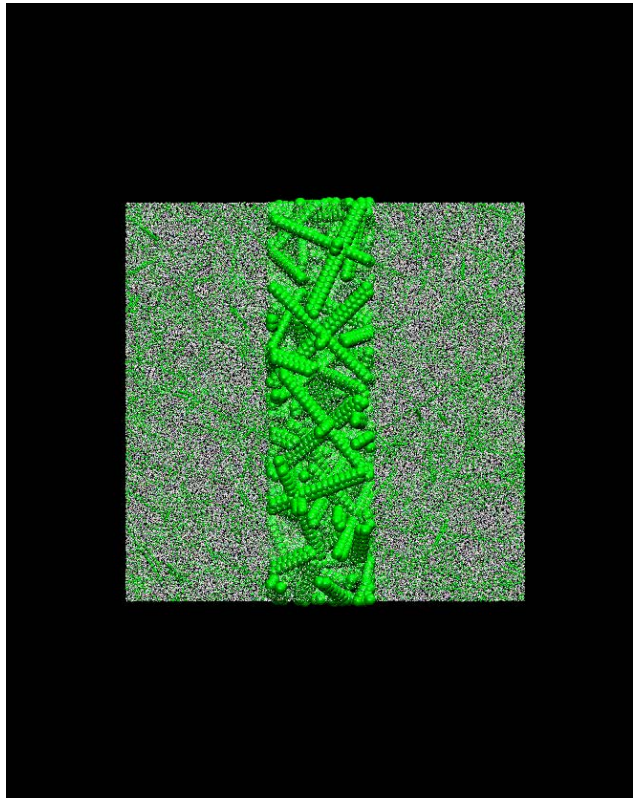


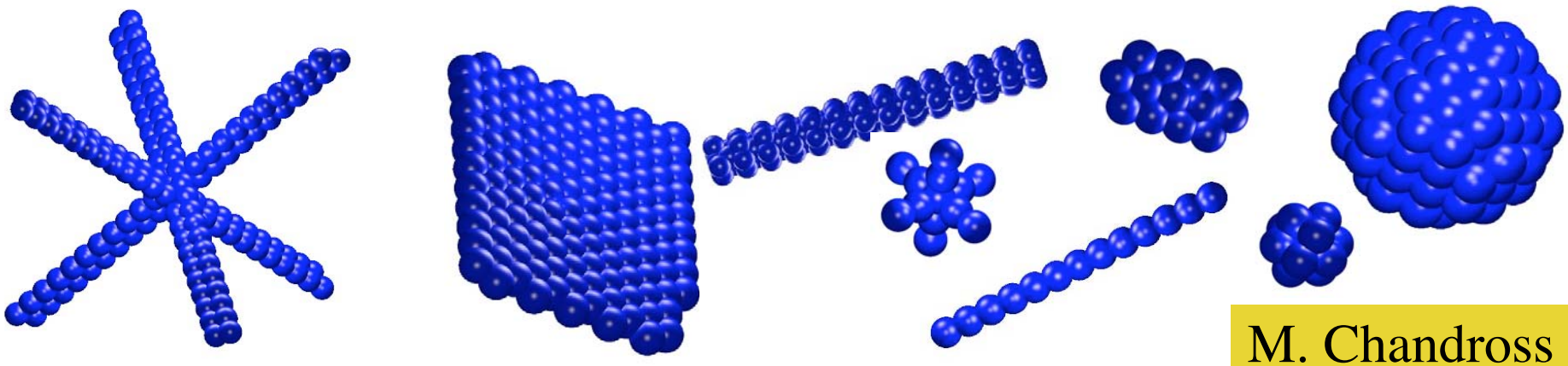


P. Lu
L. Brewer

Nanoparticle Modeling indicates that JACKS influence <u>the viscosity the most.</u>







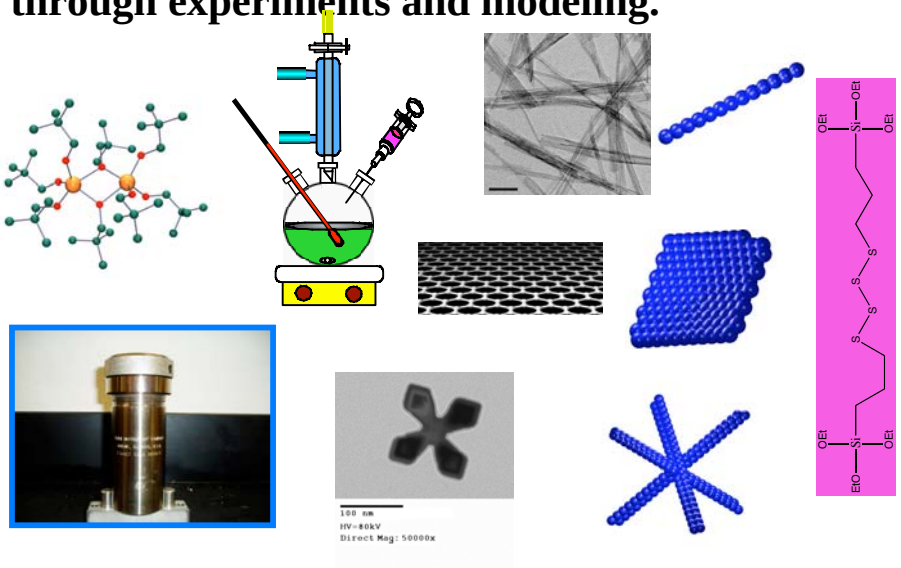
M. Chandross

IMPLEMENTED NANOSAFETY

RESEARCH PROBLEM

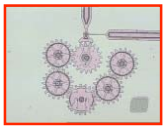
To develop and synthesize functional nanomaterials that will become part of a scientific tool box. Nanomaterials are being used as probes, sensors, and building blocks for architectures to help address a variety of research questions.

APPROACH: **determining how the <u>nanomaterials</u> effect composite materials through experiments and modeling.**

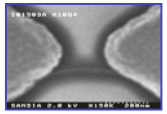


RESULTS: **Our precursor library (>1000) has led to synthesis of functional nanomaterials for:**

Cancer/Apoptosis Detection

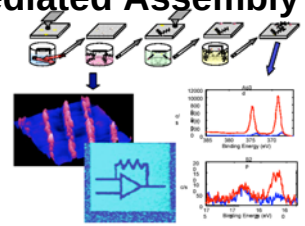


Nanolubricants

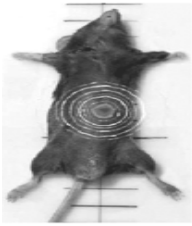


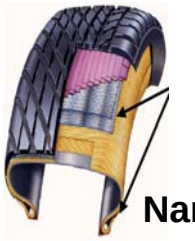
Sensors

Mediated Assembly



Non-Toxic Probes
Multi-Modal Sensors





Nanofillers