SMRC

SYSTEMS & MATERIALS RESEARCH CORPORATION

Nanosilicon Production and Formulation into Additive Manufacturing Feedstocks

David Irvin[1], Leslie Wood[1], Anjan Contractor[1], Malcolm Prouty[1], with Jennifer Irvin[2], and Michelle Pantoya[3]

[1] *Systems & Materials Research Corporation (SMRC)*

[2]*Texas State University* [3]*Texas Tech University*

Systems & Materials Research Corporation

SMRC specializes in research and development for the aerospace and defense sectors, with a focus on technology commercialization in…

Integrated Systems

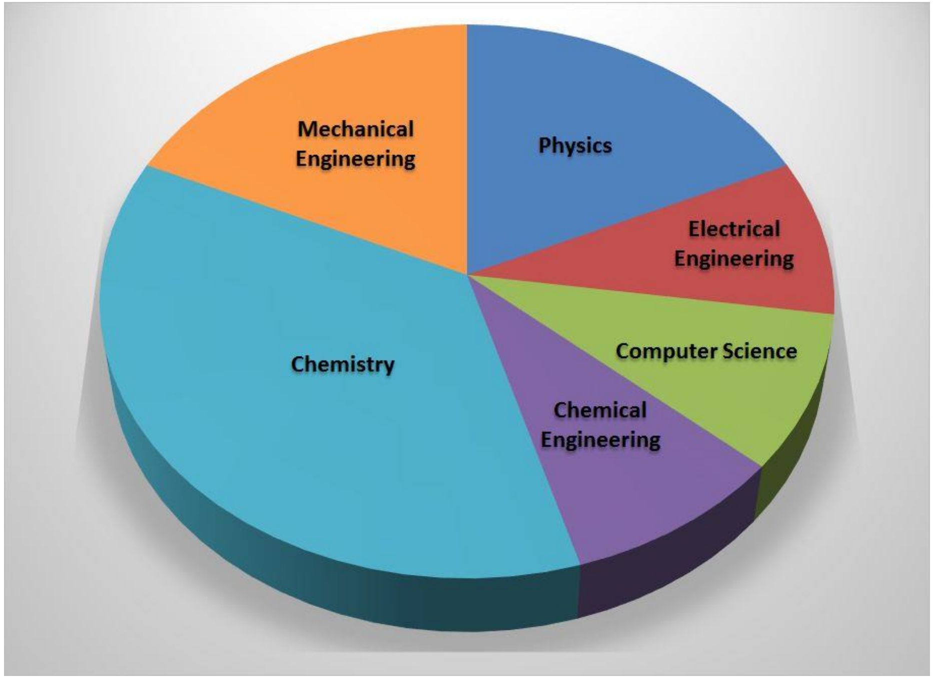
Manufacturing Technology

Nondestructive Evaluation

Advanced Materials

Team

- SMRC currently employs 9 full-time staff
- Advanced degrees in various areas of engineering and science
  - 3 Ph.D.
  - 3 M.S.
  - 3 B.S.



Facilities

Corporate Office

- 7,500 ft^2 facility
- 2,300 ft^2 laboratory space
- Fully equipped machine shop
- Electronics lab
- Chemistry lab

STAR Park Office

- 400 ft^2 facility
- 300 ft^2 wet laboratory space with 30 ft bench space and chemical hoods
- Synthesis and purification equipment

Corporate Timeline

MNDE Toolkit

*Qwik*Seal Production Machine

Gemini Kaps

1998 SMRC founded by Dr. Alan Bray

1999 First SBIR contract awarded: MCD development begins

Microwave Corrosion Detector (MCD)

2001 *Qwik*Seal and MNDE Toolkit development begins

*Qwik*Seal Pre-Sealed Fasteners

2005 First commercial sale: MCD to Bell Helicopter

2006 RoboChek and BladeChek development begins

RoboChek

BladeChek

2008 *Qwik*Seal production machine fabricated

2010 *Qwik*Seal qualification begins

2010 ESD-Safe Nano-thermite development begins

2011 Dr. Malcolm Prouty elected as SMRC President

2011 Gemini Kap development begins

2012 SmartMag and NanoSi development begins

2012 GripChek development begins

2013 3D Printed Food development begins

2014 Fasten E-Z & Environmental Chamber begins

2014 Commercial sale: ESD-Safe Nanothermites

ESD-Safe Nanothermites

2000 | 2005 | 2010 | 2015

Core Competencies

Integrated Systems

- **GripChek™** - Automated hole depth measurement system
- **SmartMag™** - Smart munitions sorting, selection, and deployment magazine
- **3D Printed Food** - Meals and nutrition for long duration space missions

Manufacturing Technology

- **Gemini Kaps™** - Intelligent Sealant Application System
- ***Qwik*Seal®** - Pre-sealed aerospace fasteners

Nondestructive Evaluation

- **MNDE Toolkit™** – Microwave nondestructive evaluation for coating thickness

Advanced Materials

- **NanoSi™** – Silicon powder for enhanced energetics applications
- **ESD-Safe Nanothermites** – Insensitive energetic materials

SMRC's goal

- 3D Printing of Energetics
  - Our goal is to position ourselves to be the systems integrator for 3D printing devices for energetics
  - These developing devices will work in concert with other known and unknown additive manufacturing techniques to produce application specific munitions and sub-munitions
  - To design and produce these printing devices and additional safety and processing data is needed
  - We are currently teaming with Texas Tech University and would like to expand the mutually beneficial relationship to the consortium

Metals as Fuel?

- First experiments on combustion of metals were those of von Ingenn-Hausz in 1782
- In 1958, Grosse and Conway, produced a series on metal-oxygen torches

Tungsten

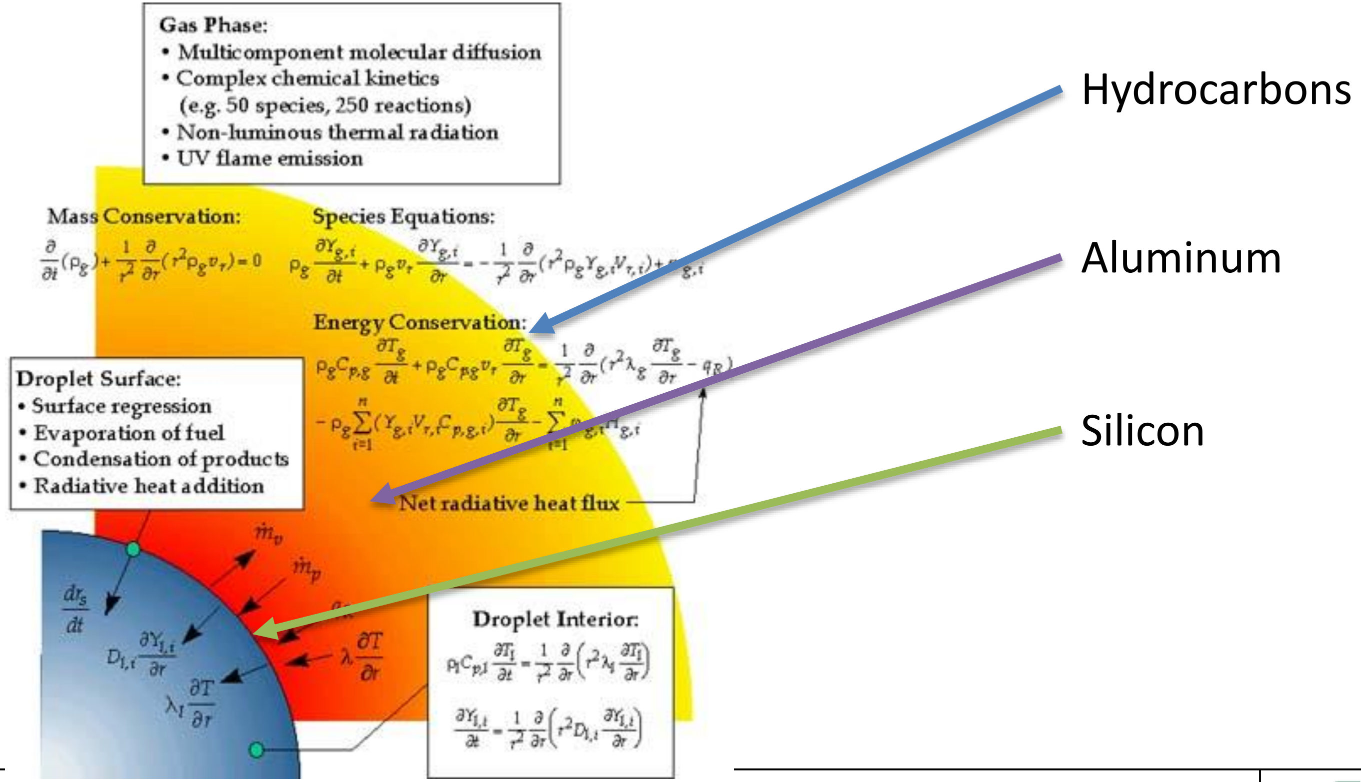
Iron - Steel wool

Magnesium

Modes of burning

- Metals with low boiling points (Al, Mg) burn in the vapor phase.
- High boiling points
  - Metals that melt readily, burn at the surface of a molten oxide + metal mixture, if the oxide also melts (Fe, Ti, Si)
  - Metals the melt but the oxide does not slows the reaction (Zr)
  - If the oxide sublimes at the burn temperature, combustion continues (Mo)
- Si melts at 1414°C SiO_2 melts at 1600 - 1725°C

Aluminum vs. Silicon Combustion



Al, Mg, and Si Combustion

Form	Ignition temp (°C)	Energy density Kcal/ g fuel	Melting point (°C)	Boiling point (°C)	Adiabatic Combustion Temperature (°C)
Silicon	780-1000	7.5	1415	3265	2800
Silicon dioxide	-	-	1600-1725	2230 dec	-
Aluminum	580	7.4	660	2470	3800
Aluminum oxide	-	-	2323	3800 dec	-
Magnesium	625	5.9	650	1100	3350
Magnesium oxide	-	-	3075	3350	-

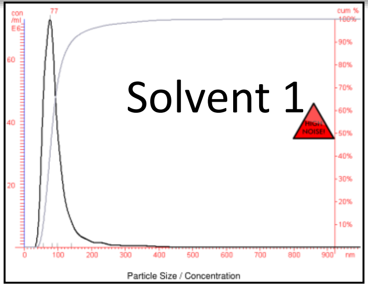
Energy Density

Fuel	Combustion products	Atomic or Mol. Weight	Kcal/mole Fuel	Kcal/gram Fuel
H_2	H_2O	2.0	68.3	33.9
C	CO_2	12.0	94.1	7.8
CH_4	CO_2 & H_2O	16.0	212.7	13.3
C_2H_2	CO_2 & H O	26.0	310.5	11.9
Li	Li_2O	6.9	71.3	10.25
Na	Na_2O	23.0	49.7	2.2
Be	BeO	9.0	147.0	16.3
Mg	MgO	24.3	143.7	5.9
B	B_2O_3	10.8	152.7	14.1
Al	Al_2O_3	27.0	200.1	7.4
Si	SiO_2	28.1	210.2	7.5

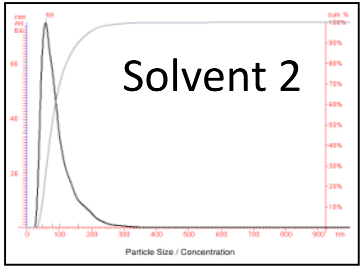
Nanosilicon

- Nanosilicon powders show great promise as **fuels**, electrode materials, inks for flexible IC, photovoltatics, and catalytic surfaces
- nSi has 1-2 nm oxide layer
- 85-95% active silicon
- Research grades Silicon is $234/kg
- SMRC Process <$100/kg

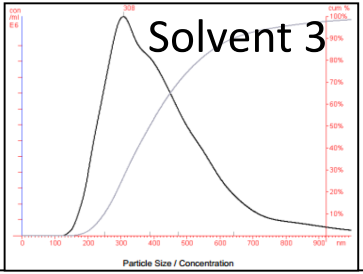
Size of Nanosilicon



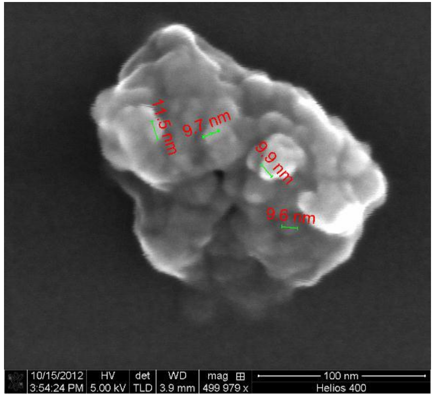
- **Mean** = 81 nm
- **Mode** = 61 nm
- **D90** = 120 nm

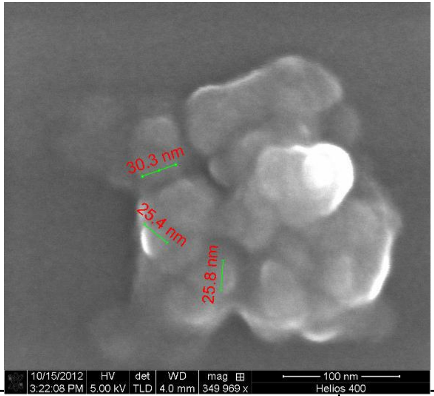


- **Mean:** 83 nm
- **Mode:** 53 nm
- **D90:** 140 nm

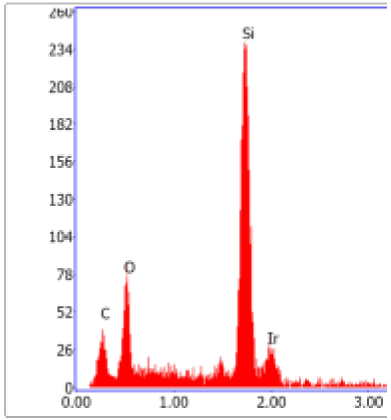


- **Mean:** 332 nm
- **Mode:** 235 nm
- **D90:** 516nm



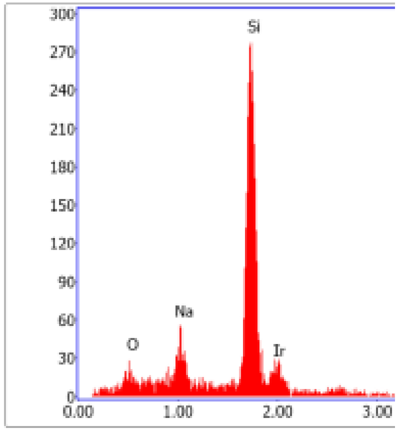


EDAX



methanol passivated nanosilicon

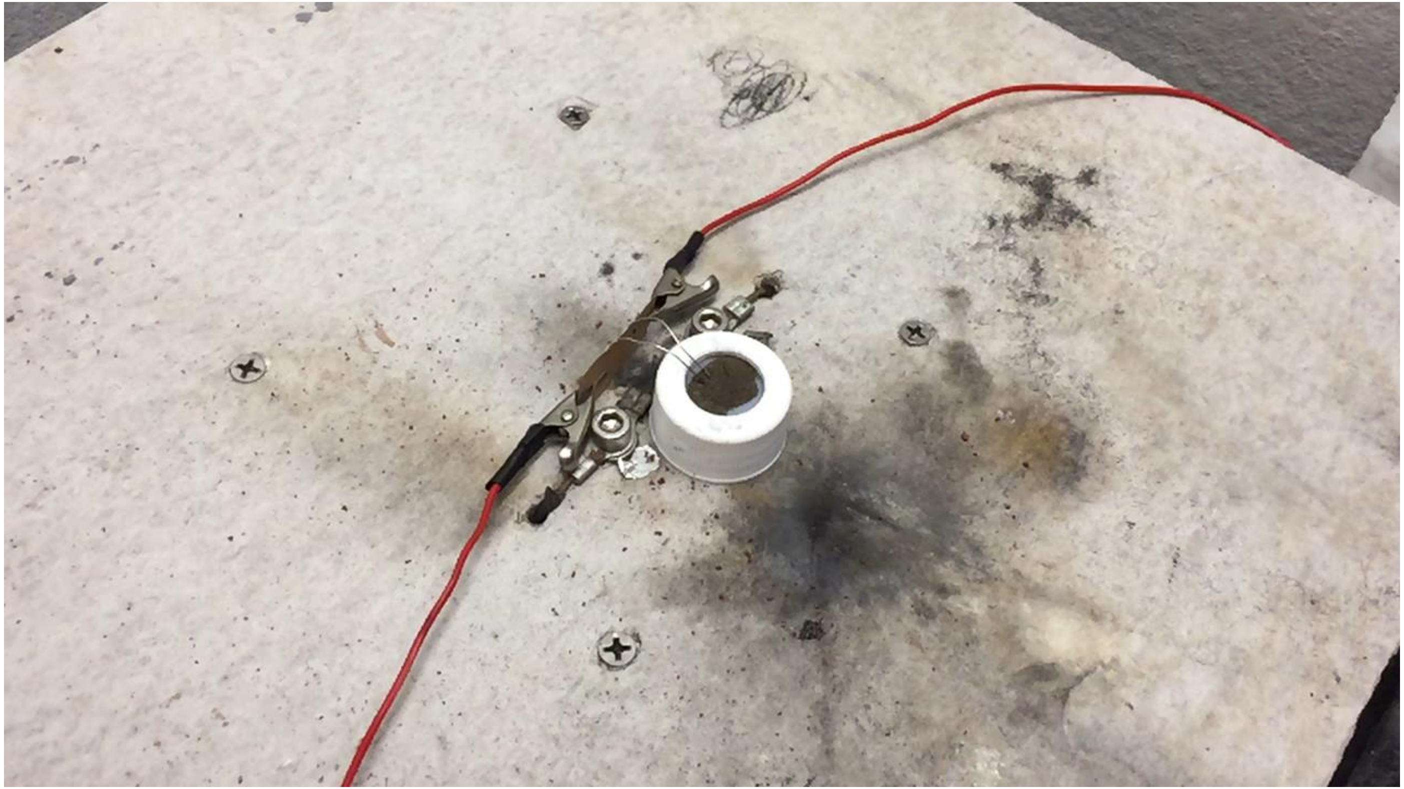
Element	Weight %	Atomic %	Net Int.
C K	12.05	26.09	11.63
O K	9.55	15.53	18.94
SiK	60.40	55.95	79.42
IrM	18.01	2.44	6.42



hydrogen passivated nanosilicon

Element	Weight %	Atomic %	Net Int.
O K	3.26	6.47	6.33
NaK	7.77	10.73	13.46
SiK	70.64	79.78	88.77
IrM	18.33	3.02	6.19

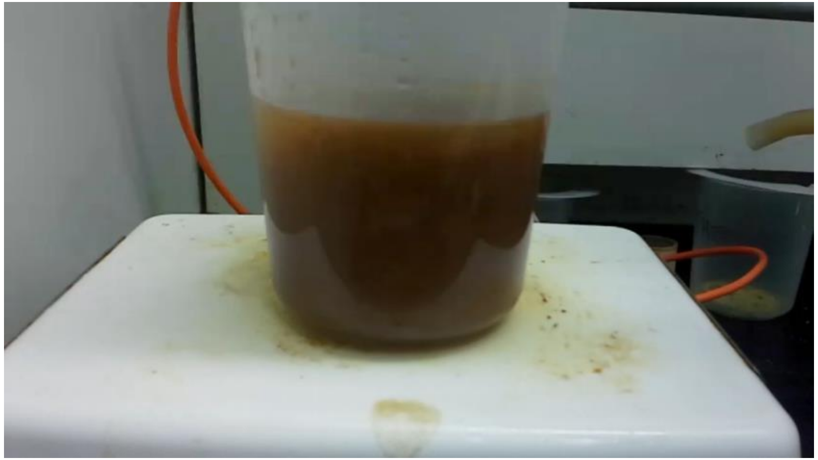
Silicon/CuO nanothermites



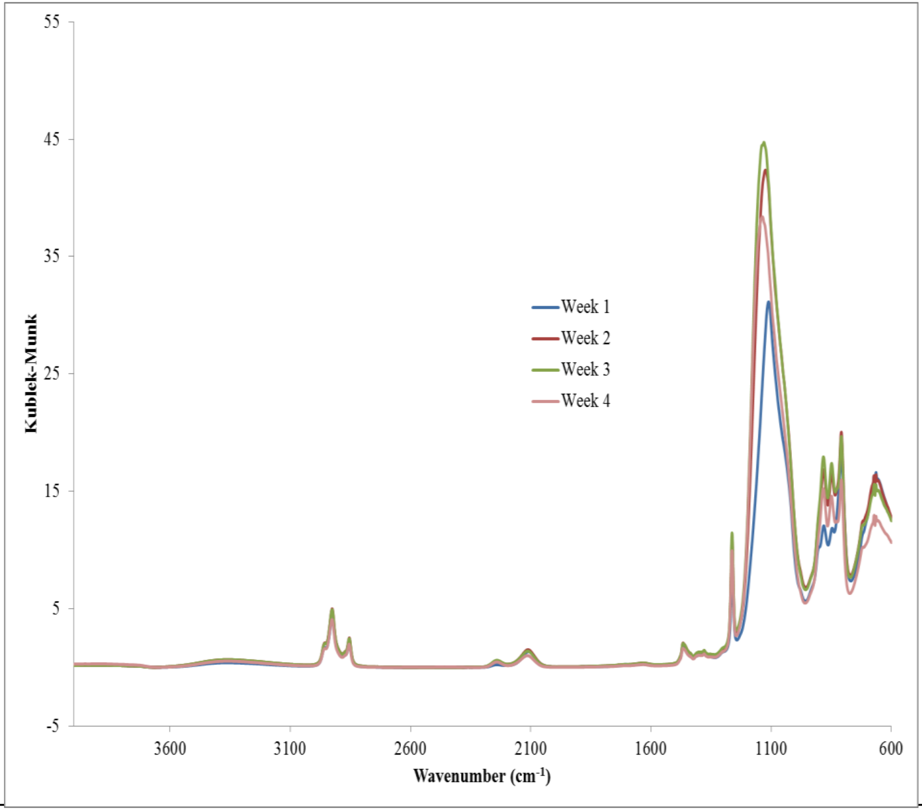
SMRC

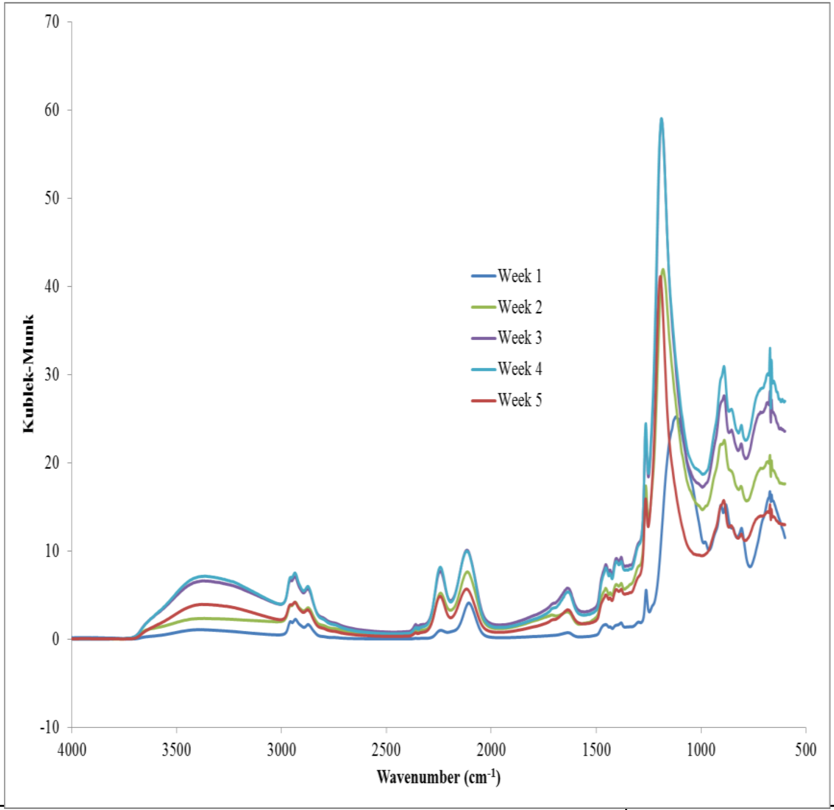
Aluminized propellants?

- Hydrogen- passivated nanosilicon stable to acid
- As aluminize propellants are heated, they liberate perchloric acid
- This reacts with aluminum generating flammable hydrogen gas



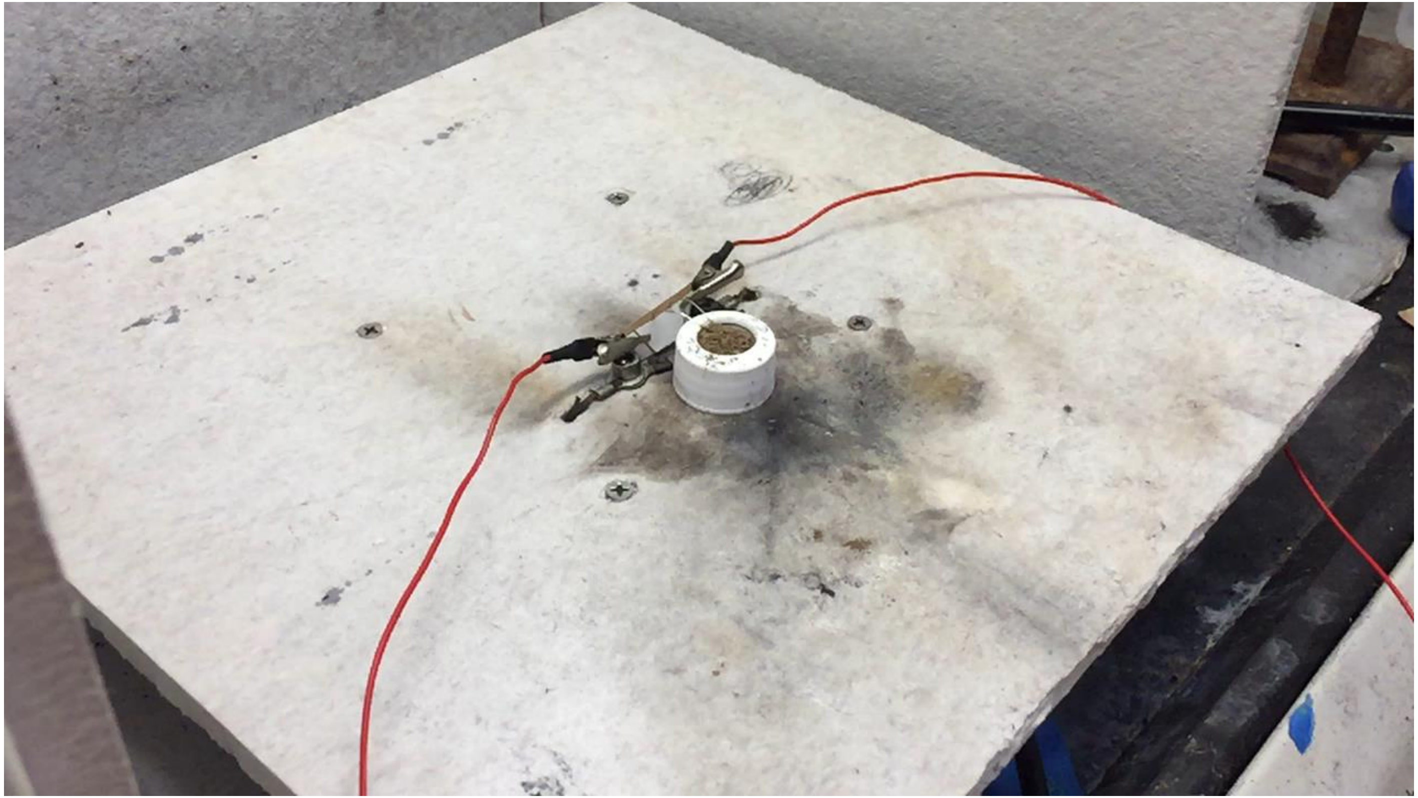
Hydrolytic Stability via IR





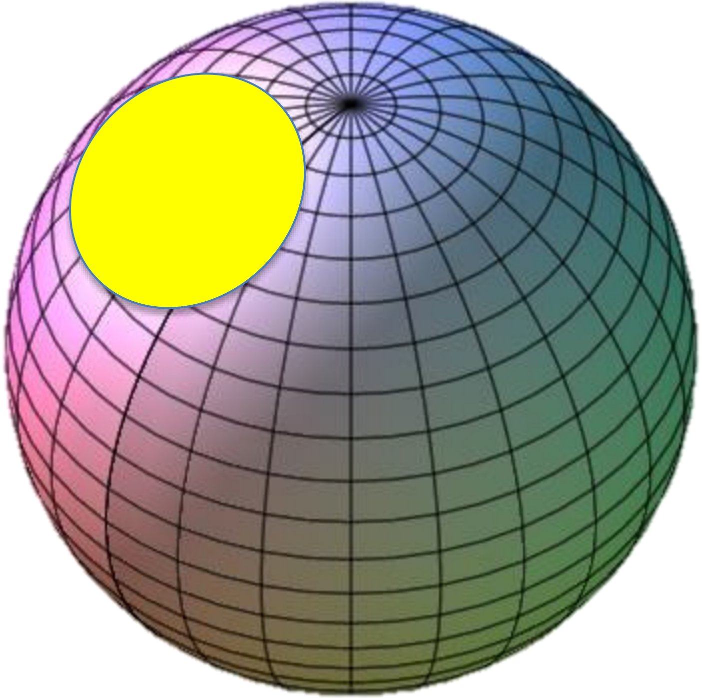
Approved for Release – 2015 National Energetic Materials Consortium

Silicon/Teflon/Viton



Increased range of liquid rocket motors

- LOX/RP-2 base rocket motor has a designed range
- To increase range, the rocket must lose weight, increase thrust, or be redesigned
- Unless….



Increased range of liquid rocket motors

- Nanosilicon suspended in RP-2 increases combustion rate by 5x
- This should equate to higher thrust and range with no modifications to the motor or payload

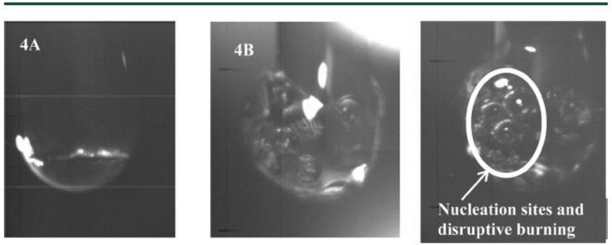


Figure 4. Still frame images of RP-2 with (A) no additives (stage 2), (B) 0.25 wt % MgO (stage 2), and (C) 0.25 wt % MgO showing nucleation sites circled and disruptive burning with an arrow and text.

Nanosilicon suspensions in RP-2 from 1.0-0.1 wt% (left to right) after 24 hours

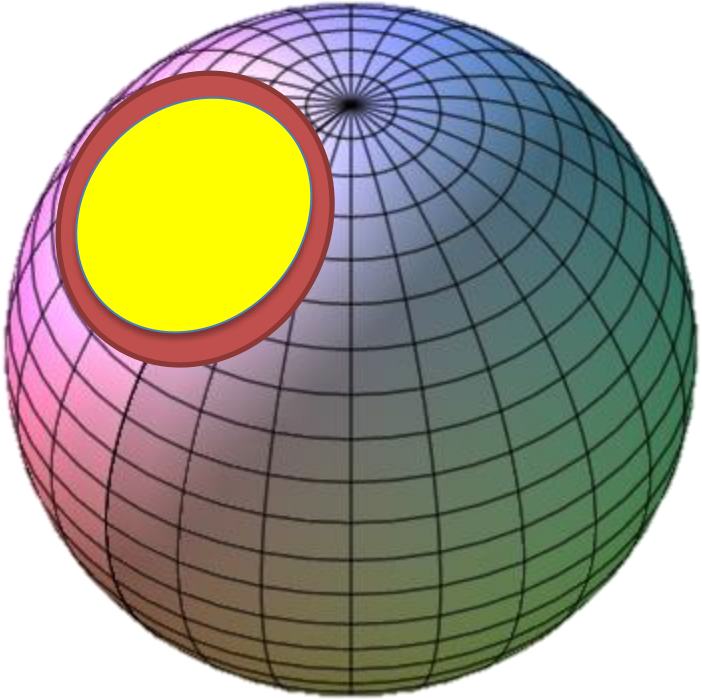
Percent weight	H_c[1] nSi	Regression rate[2] nSi
0	11076*	4.45*
0.1	11048	13.4
1.0	10801	24.5

[1]Hc is heat of combustion by bomb calorimetry in Kcal/g
[2]Regression rate is measured mm/s
**Unmodified RP-2*

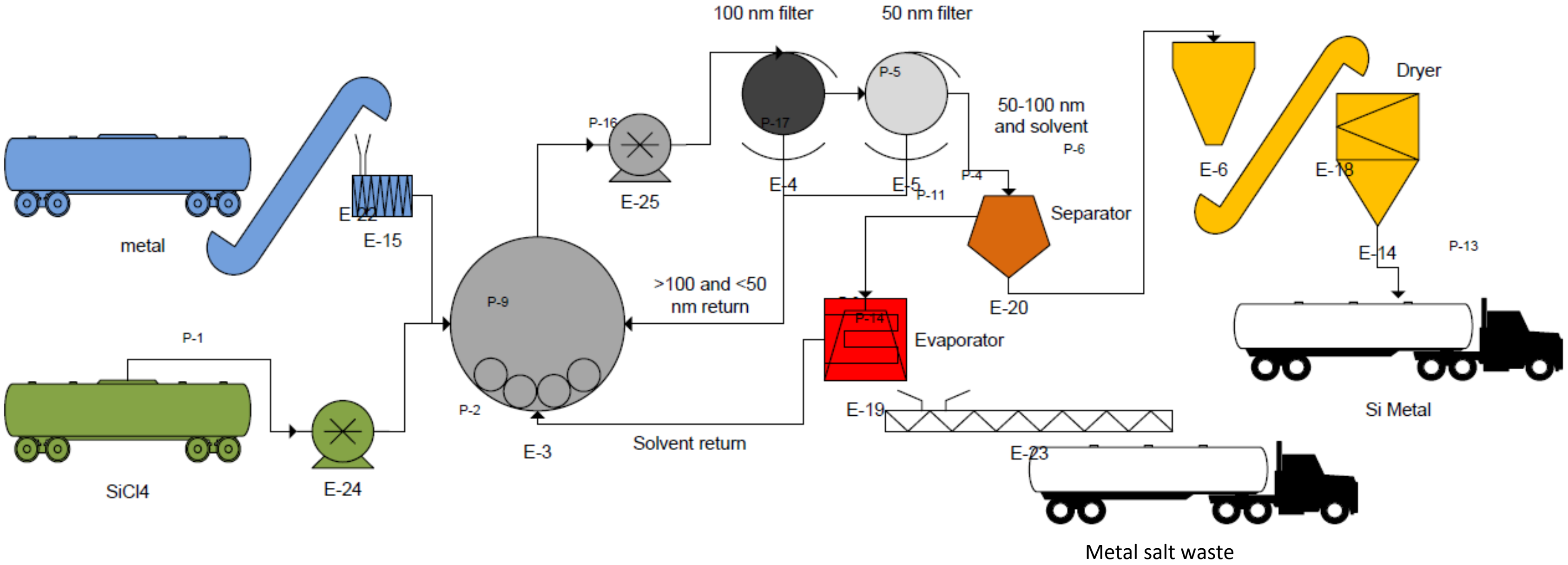
Bello, Michael N., Michelle L. Pantoya, Keerti Kappagantula, William S. Wang, Siva A. Vanapalli, David J. Irvin, and Leslie M. Wood. "Reaction Dynamics of Rocket Propellant with Magnesium Oxide Nanoparticles." *Energy & Fuels*(2015).

Increased range of liquid rocket motor fuels

- With no hardware modifications
- 10% increase in range with less than 1% addition of a stabilized nanosilicon particles



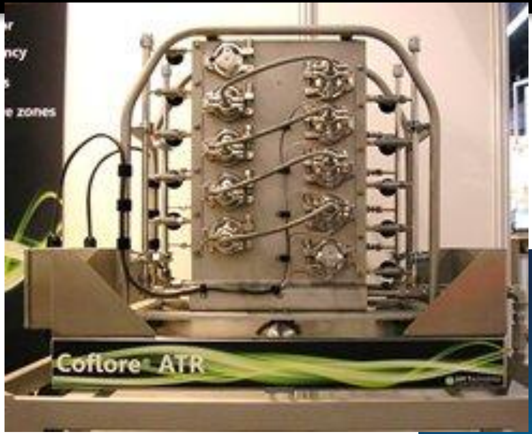
Full scale production process



50 kg/day production reactors

- 100 L flow through reactor
- 1 hour residence time
- $350K one time capital expense
- Lower operating cost

- 20 gal Parr reactor
- 1.5 batch time
- $120K one time capital expense
- Higher operating cost



Culmination of 6 SBIR's

Contract Number: *NNX13CJ18P*
Contractor Name: *Systems and Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: *23 November 2020*

Contract Number: *N68335-15-C-0280*
Contractor Name: *Systems and Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: *15 December 2020*

Contract Number: *FA9300-13-M-1006*
Contractor Name: *Systems &Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: *10 April 2019*

Contract Number: *W15QKN-12-C-0083*
Contractor Name: *Systems and Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: *28 February 2020*

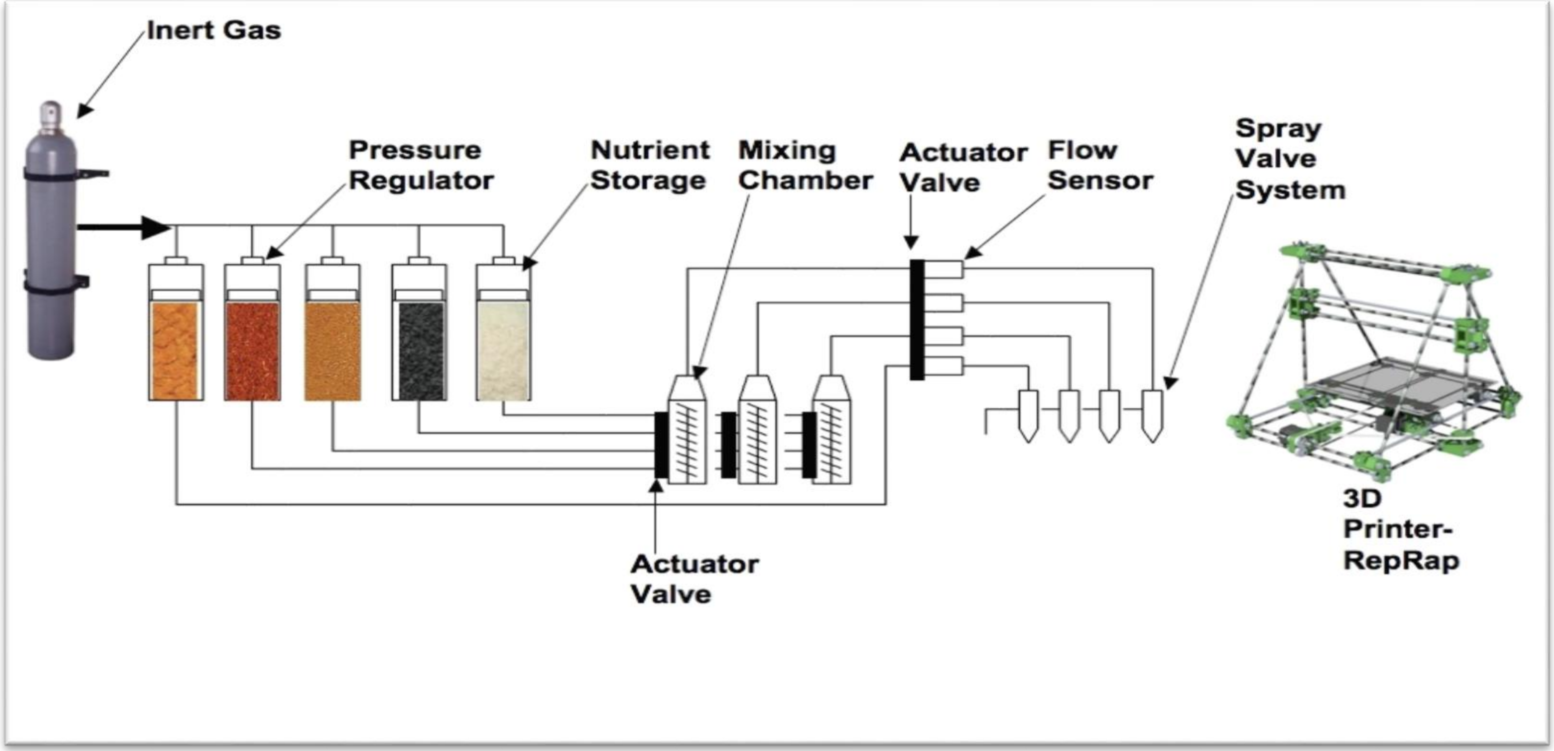
Contract Number: *W15QKN-14-C-0011*
Contractor Name: *Systems &Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: 20 December 2022

Contract Number: *W15QKN-11-C-0011*
Contractor Name: *Systems &Materials Research Corporation*
Contractor Address: *11525 Stonehollow Drive, Suite A120*
Austin, TX 78758
Exp of Data Rights: 17 June 2016

NASA: Micronutrition control

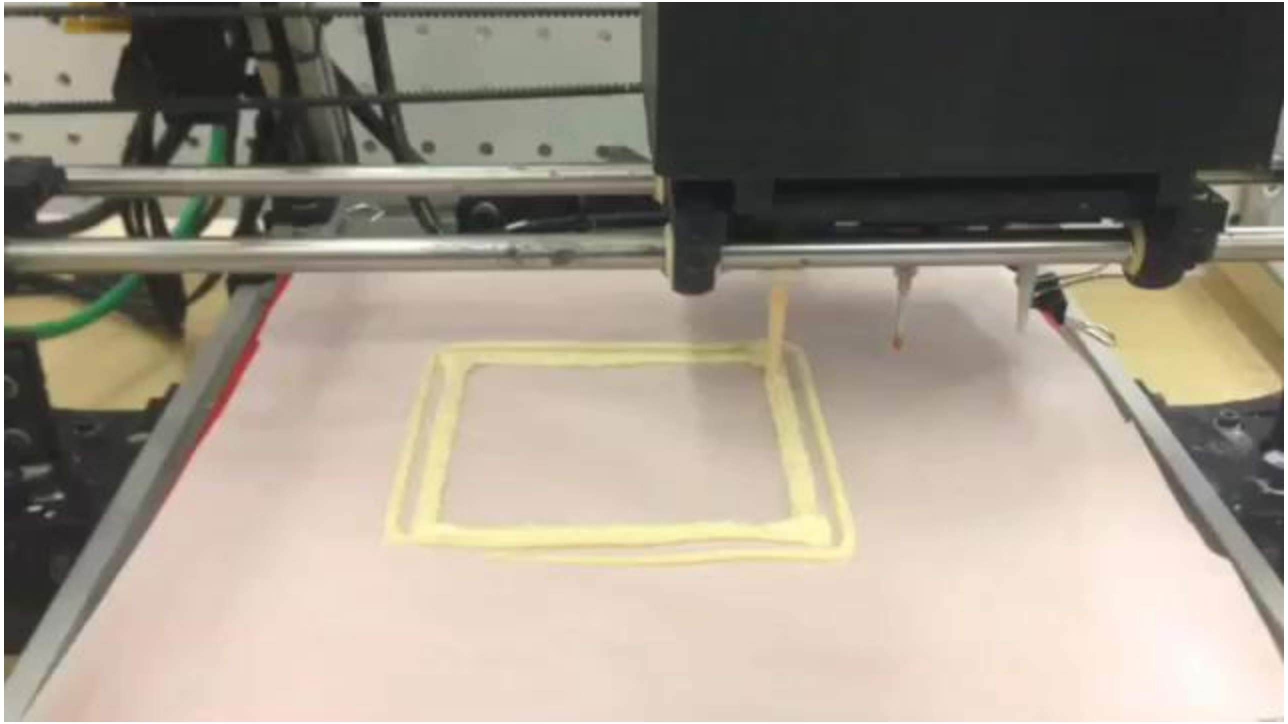
- Astronaut food is prepackage meals ready to eat
- Micronutrients begin to decompose the moment the food is sealed
- 3D printing can reintroduce the lost nutrients as a function of time in space

Systems design and constrants



Design constraints

- No off gassing
- FDA safe food contact surfaces
- Powder and liquid transport at 1g, 1/3g and 0g
- Three phase mixing at 1g, 1/3g and 0g
- Printing at 1g, 1/3g and 0g
- Printing different viscosities
- Surface tension/adhesion
- Light weight
- Low power
- Low water use
- Sanitation before each print
- Cook with out astronants contacting hot surfaces
- …….

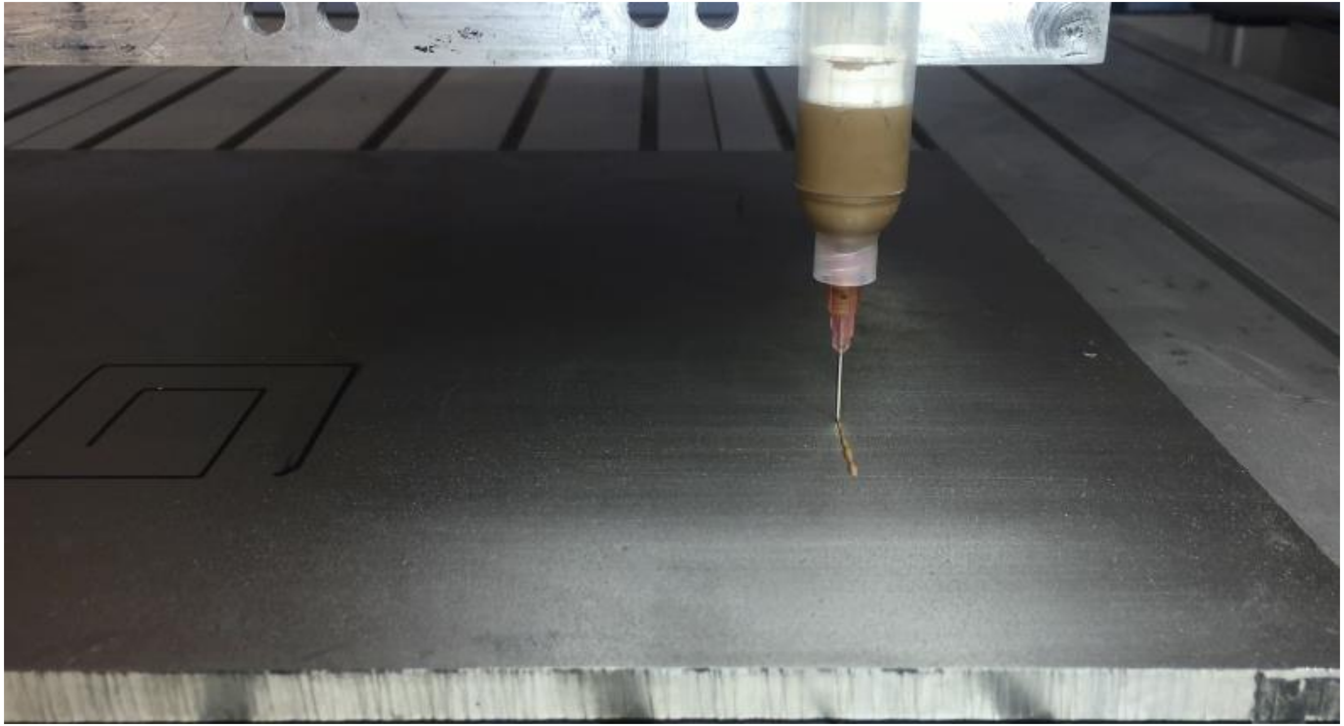


Finished Product

Energetics Design constraints

- "Inherently safe"
- Not hot spots
- No pinch points
- Sheer limits
- If the is an incident, minimize fragmentation
- Static dissipation
- Speed
- Accurate location
- Accurate mass
- Feedback controls
- Quality controls
- ……

Initial printing of silicon based energetics



General Discussion

Questions?

Contact Information:

Systems & Materials Research Corporation
11525 Stonehollow Drive, Suite A120, Austin, TX 78758
Office: (512) 535-7791 • Fax: (512) 206-4966
www.systemsandmaterials.com

Malcolm D. Prouty, Ph.D.
President
Cell: (512) 968-4750
malcolmprouty@systemsandmaterials.com