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THIS ACS WEBINAR® WILL BEGIN SHORTLY…
👋 Say hello in the questions window!

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How Polymeric Materials Protect Our Armed Forces

JOSEPH L. LENHART, PhD
Chief, Polymers Branch, Weapons and Materials Research Directorate, DEVCOM Army Research Laboratory

DAVIDE SIMONE, PhD
Senior Research Chemist, Air Force Research Laboratory

Peter Zarras, PhD
Research Chemist, Naval Air Warfare Center Weapons Division

This ACS Webinar® is co-produced with the ACS Division of Polymer Chemistry.

U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMY RESEARCH LABORATORY

Polymeric Materials for Army Applications

Joseph L. Lenhart
Chief, Polymers Branch
Weapons and Materials Research Army Research Laboratory
Aberdeen Proving Ground, MD
ARL / ABERDEEN PROVING GROUND (APG)

Fun life stuff
• Centrally located
• On the Chesapeake Bay
• Cultural, sports, outdoor activities

Fun work stuff
• Chemistry to processing
• Proto-type development
• Full-scale assessment

POLYMERS BRANCH OVERVIEW

Overarching Polymer Expertise:
- Physics – structure / property relations (mechanics and functional)
- Synthesis – new materials through chemistry and additives
- Processing – new materials through chemistry and structural control
- Modeling – insight and guidance (quantum, molecular, meso-scales)

Current State:
- ~20 staff
- Skilled workforce (PhD)

Underpinning Research Thrusts:
1. Polymer Processing: semi-crystalline, highly particle loaded, multi-material, new feedstock
2. Resins / Adhesives: fiber and particle loaded composites, complex laminate structures, dynamically responsive feedstock

Targeted Applications:
- Soldier Protection: Body armor, signature management
- Vehicle Protection: Complex laminates, novel mechanisms
- Long Range Weapons: Small and large caliber munitions, propellants, survivability, range, lethality

Approved for Public Release
Have you considered a defense laboratory as a career option?

- Yes, I already work for a defense laboratory
- Yes, I am very interested
- I knew it was an option, but did not consider it
- I never knew it was an option

* If your answer differs greatly from the choices above tell us in the chat!

POLYMERS FOR THE DISMOUNTED SOLDIER

**Protective Equipment**
- Body armor
- Helmet
- Fabrics for extremity / groin protection
- Eye protection

**Lethality**
- Small caliber weapons
- Ammunition
- Grenades

**Electronics**
- Night vision
- Optics
- Radio
- Batteries

**Uniform**
- Camouflage – visibility
- Thermal management
- Boots / pads

Soldier carries 70 to 130 lbs depending on their job, mission type, and mission duration
POLYMERS FOR GROUND VEHICLES

**Lethality systems**
- Large, medium, and small caliber munitions
- Missiles
- Lightweight composites
- Propellants

**Protection systems**
- Passive protection
- Active protection
- Laminate composite armors packages
- Transparent armors
- Coatings – chemical protection and visibility control

**Structural systems**
- Historically metallic
- Opportunities for structural composites laminates
- Composite track pads

**Electronics systems**
- Ruggedization
- Sensor and electronics protection
- Packaging

1. **Many types of ground vehicles:** tracked vehicles (Abrams Bradley); Wheeled (Stryker, Humvee, transport vehicles)
2. **Opportunities:** Unmanned systems for fighting and resupply

---

**CHALLENGE OF EXTREME ENVIRONMENTS**

1. **Environmental Durability**
   - Temperature
   - Humidity
   - Life-time
   - Fuel, solvent, dirt, mud
   - Chem / Bio / Radiological
   - Electrical, optical, …
   - Cycling

2. **Mechanical extremes**
   - Quasi-static, impact, ballistic, blast strain rates
   - High pressure
   - Launch conditions

**Strain Rate:**
- Humans: $10^{-4} \text{ s}^{-1}$
- Cars: $10^{0} \text{ s}^{-1}$
- Airplanes: $10^{2} \text{ s}^{-1}$
- Bullets: $10^{4} \text{ s}^{-1}$

- Quasistatic Testing
- Standard Impact Tests
- Ballistic Testing

• **Complex composites and structures are critical**
• Understanding deformation / failure processes for both the individual materials and the composites is critical
Introduction to Polymers Research for Vehicle and Soldier Protection Applications:

1) Resins and adhesives

Polymer Networks: Fundamental and Applied Research

Some Key Factors:
- $M_c$
- Interactions
- Stiffness
- Packing

Armor (Vehicles, Rotorcraft, Body Armor):
- 30% weight savings for glass based transparent armor
- Transparent and non-transparent

Armor Durability:
- Processing protocols
- Materials solutions

Weapons:
- Propellant coatings for temperature compensation
- Insensitive munitions
- Structured propellants

Vehicles:
- Fuel hose and storage
- Fuel tank ballistic / fire protection

Protection Assessment:
- ARL Room Temperature Insensitive Clay (ARTIC)
- Environmentally stable gel for assessment of Pelvic Protection System
- Anthropomorphic forms
IMPACT TESTING WITH MODEL SPHERICAL BB:
IMPORTANCE OF GLASS TRANSITION

Shear plug failure (rubber)

Extensive spall zone
Small radial cracking (low Tg glass)

Small spall zone
Extensive radial cracking (high Tg glass)

SEGMENTAL RELAXATIONS NEAR Tg ARE CRITICAL FOR IMPACT PERFORMANCE

- Impact temperature relative to Tg is dominant factor in epoxy network ballistic response
- Temperature dependent response correlates with Vogel Temperature
ROMP RESINS FOR STRUCTURAL PERFORMANCE AND TOUGHNESS

Ring Opening Metathesis Polymerization (ROMP)

a) Extension of “rigid” and non-polar chain

b) Bulky pendant chain on backbone


1. Weak chain-chain interactions and high Mc provides ductility
2. Stiff junction points and chains enable higher Tg

UNIQUE BALLISTIC FAILURE FOR P-DCPD COMPARED TO GLASSY EPOXIES


pDCPD exhibits a ductile like failure during ballistic impact with no large scale radial fracture
CONTROLLED CROSSLINK DENSITY

Control effective crosslink density and $T_g$ with co-monomer content

$$M_{c,a} = \frac{3\rho RT}{E'_{r}}$$

CRITICAL $M_c$ FOR BRITTLE-TO-DUCTILE TRANSITION

- Critical effective $M_c$ (400-600 g/mole, 6 units)
- As $M_c$ effective approaches persistence length resin becomes brittle (lose orientational correlation around 6 monomeric units)

Series of ROMP resins with: a) no radial fracture; b) $T_g$ 120-145°C; c) 3.5-4X impact resistance relative to structural epoxy with similar $T_g$
MOLECULAR DYNAMICS INSIGHT INTO 

pDCPD TOUGHNESS


1) Polar epoxy void formation is energetically unfavorable. Non-polar ROMP void formation is energetically neutral
2) Atomic fluctuations (relaxation processes) are faster around voids than in bulk
3) Atomic fluctuations are suppressed with increasing crosslink density and polarity

- ROMP resins are more tolerant to formation of molecular voids during deformation
- Increasing crosslink density and polarity suppresses local relaxations around the voids

Introduction to Polymers Research for Vehicle and Soldier Protection Applications:
2) Polymer gels
- **Need**: Tissue "simulants" to evaluate blast / ballistic effects
- Tunable modulus, toughness, and ballistic penetration depth

### POLYMER GELS FOR TISSUE SIMULANTS

Use gel for unique testing protocols that require long times in outdoor ranges

- Material has better time, temperature, humidity stability than ordinance gelatin
- Enabling testing that is difficult with ballistic gelatin

**Sand Cannon Test**
- Detailed Damage Zone
- Individual Sand Grains

**Summertime testing**
- Instrumented gels

**Pelvic Protection System (PPS) evaluation**
Introduction to Polymers Research for Vehicle and Soldier Protection Applications:

3) Thermoplastic processing (~7000 ft\(^2\) of processing laboratories)
   a) Processing as tool to make new materials (chemistry and structure in-situ)
   b) Facilitate transition by enabling scale-up for engineering evaluations (coupon to pilot scale)
   c) Engineering controls for safe and reproducible processing (clean rooms)

COMPounding AND COUpon PREPARATION LABORATORY (CLEANROOM)

Compounding / Mixing
- Capability from grams to 100 Kg/hr (11, 16, 24, 35 mm twin screws)
- Up to 52 L/D
- Heated, cooled, high shear / torque

Fiber, filament, tape processing
- Single screw extruders
- Coupon scale continuous (1-6 inch width)
- Uni- and bi-axial stretching

Safety and room design
- Powder handling laminar flow hoods
- Ionizers / stainless steel - static control
- Temperature and humidity control
- Emergency exhaust
FIBER AND FILM PROCESSING LAB (CLEANROOM)

Tape processing
• 5 layer cast film line
• Multi-layer forced assembly co-extrusion processing (MLFACE)

Tape rolling, drawing, and high pressure lamination
• Machine direction orientation on cast film line
• High pressure roll mill

Fiber and filament processing
• Water or air cooling
• Single or bi-component
• Flexible dies and drawing

We can use COTS or in-house developed formulations from twin screw extruders

HOW DO WE USE FIBERS AND FILMS IN BALLISTIC COMPOSITES

SOA: UHMWPE and Kevlar-based composites arranged in a 0 – 90 configuration

Limitations with current composites:
• Fibers are macro-scale
• Resin starved composite with voids
• Poor mechanical binder (~ 15% parasitic)
• Poorly designed binder-fiber interface
• Solvent heavy fiber processing is high cost

What we would like
• Fine scale fibers – near theoretical
• Intimate binder-reinforcement contact
• Tough binder with lower content
• Designed interface
• Low or solvent-less process

Gap: Manufacturing strategy to enable all of these improvements
WHAT ARE WE TRYING TO DO?

Concept: 1) Fine scale (sub-micron) fibers and films are stiffer and stronger than macro-scale; 2) quality binder and interface will enhance performance

Gap: Cost effective continuous manufacturing

MULTILAYER FORCED ASSEMBLY CO-EXTRUSION (MLFACE) COLLABORATION WITH CWRU – PEAK NANO

✓ Tapes with fine scale reinforcement structure (sub 100 nm dimensions)
✓ Intimate binder content due to co-extrusion process
✓ Suitable for melt or gel casting operations (cost and performance)

Courtesy of Dr. Ponting at PeakNano

Layered or fibrous
WHAT DO WE NEED TO DO TO MAKE MLFACE WORK FOR ARMY?

**TASK 1:** Feedstock for high performance MLFACE tapes
- Reinforcement phase: HDPE and HDPE/UHMWPE blends
- Binder phase: PE copolymers (co-crystallize at interface)
- Temperature and shear dependent viscosity

Identified highly linear PE feedstock

- HDPE – High crystallinity
  Mw ~ 80K – 500K (Entanglement)
  Melt processable

- LLDPE – low crystallinity
- LDPE – low crystallinity

- UHMWPE – High crystallinity
  Mw ~ 500K – 12,000K (highly entangled)
  Gel spinning

---

**TASK 1: HDPE PROCESSING DEVELOPMENT AND PERFORMANCE ASSESSMENT**

- Extrude 6 inch wide tapes
- Cut out dog-bones
- Draw with controlled rate and temperature

✓ Draw coupons 20-40X
✓ 40-60 GPa modulus
✓ 600-900 MPa strength (lower than we expected)
✓ Feedstock and properties are OK but not great
IMPROVING FEEDSTOCK PERFORMANCE: HIGH MW TAIL


We are here (increasing lamellae organization with draw)

Solution: Blend UHMWPE to create “shish” to get here

Drawing

Fiber axis

• Scattering shows that we are orienting lamella and increasing crystallinity
• Not forming extended chain crystals

Hsaio Polymer, 2005 46(10), 8587

TASK 1: IMPROVING FEEDSTOCK PERFORMANCE: HDPE / UHMWPE BLENDS

Initial Sheets:
• HDPE and UHMWPE poorly mixed
• Low draw ratio and poor performance

Recent Films:
• Higher quality and drawable blends
• Measuring performance / structure

Mixing temperature, residence time, screw design on twin screw extruders
TASK 3: INITIAL DRAWING AND MECHANICS OF MLFACE FILMS

- Successfully MLFACE processing with our feedstock materials
- Numerous multipliers to get pre-drawn fiber and tapes with ~ micron dimensions
- Initial tape drawing with 20% binder
- Good mechanics but still need to improve
- Semi-continuous structure

1. Challenges with tape uniformity
2. Refining feedstock
3. Moving to larger processing systems for compounding (52 L/D) and tape casting (better processing controls)
4. Good progress – long way to go

LONG TERM RESEARCH OPPORTUNITIES?

Long term research opportunities for ARL – academic – industry partnership in polymers

- Higher risk, longer term, more labor than ARL can invest in by itself
  1) Dynamically responsive rigid systems
  2) Complex multi-material processing
WHY DYNAMICALLY RESPONSIVE POLYMERS


Response Times

microsecond millisecond second

1’s 10’s 100’s 1000’s

Magnitude of Change

Challenge: Broad range of potential performance parameters that are dependent on the applications

Objective: Map out this space. What is possible under what conditions?

Major Gap: Dynamic response in rigid systems

Particle loaded multi-layers
- Near jamming limit (50+ vol%)
- Continuous processing
- Controlled porosity
- Complex form factors

Multi-material processing
- Adapted to other materials
- Co-processing of metals ceramics, polymers
- In-situ interfacial control
- Down-stream drawing / shear deformation to refine and align microstructures

Thick multi-materials composites
- Interfacial control
- Curved and complex shapes
- High pressure consolidation

Automated design
- Robotic assisted materials design
- Automated composite assembly
- Complex multi-material structures
- AI/ML integrated manufacturing

Processing directed cellular assembly
- Synthetic Biology and Cellular synthesis
- Polymer processing of cellular structures

TAKE HOME MESSAGES / AUDIENCE QUESTIONS

1. ARL is great place to work and live
2. Work on fundamental and applied programs
3. Excellent facilities for chemistry, processing, analysis, and engineering evaluation
4. Polymeric materials are important for Army systems: soldier, vehicles, weapons

- Are you interested in working or collaborating with ARL?
- Needs: Generically in polymer science
- Specifically: processing, mechanics
- BS-PhD

Contact me at: joseph.l.lenhart.civ@army.mil

RESPONSIVE MATERIALS APPROACH

1) Temperature / Segmental relaxations
2) Molecular weight between crosslinks (Mc)
3) Non-covalent interactions (polarity)

If we can change temperature, Mc, or polarity dynamically then we can make dynamically responsive polymer network glasses

Questions
- How many responsive groups?
- How should we organize them?
- How fast is local response?
- How does local response perturb surrounding polymer and how fast?
- How does the response depend on the polymer and environment?

Diels-Alder (thermal Mc change)
Azobenzene (optical shape change)
ACKNOWLEDGEMENTS

Resins and Adhesives:
ARL: Joseph Dennis, Tim Sirk, Randy Mrozek, Ngon Tran, Alice Savage, Kevin Masser, Dan Knorr
University of Chicago: Juan de Pablo, Stuart Rowan, Heinrich Jaeger
Northwestern University: Ken Shull

Polymer Processing:
ARL: Randy Mrozek, Dayne Plemmons, Brian Morgan, Chris Gold, Gene Napadensky, Rick Beyer
Case Western Reserve University: Gary Wnek, Eric Baer, Eric Davis
New Jersey Innovation Institute: Mike Jaffe
Peak Nano, LLC: Mike Ponting

Materials Discovery and Development of Aerospace Composites

Dr. Davide Simone
Air Force Research Laboratory
RXCCM
6/15/22
AFRL at a Glance

Employees
- 1,100 Military (USAF/USSF)
- 5,400 Civilians
- 4,700 Contracted Personnel

3 in 5 Gov. Civilians are Scientists and Engineers (S&E)
79% of CIV S&Es hold an MS or Higher
33% Hold a PhD

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Technology Applications for our Airmen

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Munitions
Nuclear Deterrence
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Survivability
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RX Research Teams

PMC Materials & Processing (RXCCM)
Dr. Hilmar Koerner
hilmar.koerner.1@us.af.mil
- Composite Processing Science
- Multifunctional Composite Structures

Ceramics Materials & Processing (RXCCM)
Dr. Mike Cinibulk
michael.cinibulk.2@us.af.mil
- Fiber Reinforced Composites
- Environmental Effects

Composites Performance (RXCCP)
Dr. Craig Przybyla
craig.przybyla@us.af.mil
- Continuous Fiber Reinforced Composite Behavior & Life Prediction
- Ceramic Matrix Composite Durability in Extreme Environments

Metals Materials & Processing (RXCM)
Dr. Eric Peyton
eric.peyton@us.af.mil
- Metals Additive Manufacturing and Processing Science
- Discovery and Design of New Alloys for Extreme Environments

Metals Probabilistic Performance Prediction (RXCM)
Dr. Todd (TJ) Turner
todd.turner.5@us.af.mil
- High temperature durability assessment
- Location-specific (microstruct-property) probabilistic property prediction

Characterization Sensing and Analytics (RXCA)
Dr. Mike Uchic
michael.uchic@us.af.mil
- Materials characterization, with focus on nondestructive methods
- Analytics & uncertainty quantification

Digital Manufacturing (RXMS)
Dr. Sean Donegan
sean.donegan@us.af.mil
- Material Process Monitoring & Automation
- Data Analytics & Visualization for Manufacturing

Biological Materials & Processing (RXAS)
Dr. Nancy Kelley
nancy.kelley.loughnane.1@us.af.mil
- Biomacromolecular-Material Interactions
- Synthetic Biology for Materials

Polymers and Responsive Materials and Processing (RXAS)
Dr. Christopher Crouse
christopher.crouse.1@us.af.mil
- Conformal & compliant materials
- Novel responsive devices and architectures

Integrated Opto-Electronic Materials & Processing (RXAN)
Dr. Robert Bedford
robert.bedford@us.af.mil
- Infrared Detectors & Quantum Sources
- Integrated Photonics

Agile Electronic Materials & Processing (RXAN)
Dr. Mike McConney
michael.mcconney.1@us.af.mil
- High Power & Frequency Electronic Materials
- Reconfigurable RF Materials

Structured Optical Materials & Processing (RXAP)
Dr. Jonathan Vernon
jonathan.verbong.2@us.af.mil
- Optical Thin Films and Coatings

Non-Linear EM Materials & Processing (RXAP)
Dr. Joy Haley
joy.haley.1@us.af.mil
- Source Materials
- Broadband Nonlinear Materials

"New concepts are too high risk and evolve too slow for the next aerospace revolution ..." (RX CS)

Complexity

Limited Life

Present

Now/Future

Enabling Capabilities Faster

Exquisite

Biosynthesis

ML

Aut
The Pathways Program offers financial tuition and employment opportunities for students pursuing undergraduate, graduate, and doctorate degrees and those with an advanced degree. There are three different paths available: The Research Training Program, The Research/Teaching Program, and The Research/Teaching/Academic Program. The Pathways Program is designed to help students achieve their academic and career goals.

AFRL Scholarship Program

The AFRL STEM Workforce Development Program (AWDP) is administered by the National Science Foundation (NSF) and the Department of Defense (DoD). The program is designed to increase the number of scientists and engineers in the workforce by providing financial support to students pursuing degrees in science, technology, engineering, and mathematics (STEM). The program is open to undergraduate and graduate students.

SMART Infrastructure Program

The SMART Infrastructure Program (SIP) is a joint program between the Air Force Research Laboratory (AFRL) and the Department of Energy (DOE). The program is designed to develop and deploy advanced materials for infrastructure systems, including components for bridges, buildings, and transportation systems. The program focuses on the development and testing of new materials and technologies for infrastructure systems.

AFRL/AFSC Scholarships

The AFRL/AFSC Scholarships are awarded to undergraduate and graduate students pursuing degrees in science, technology, engineering, and mathematics (STEM). The scholarships are open to students attending accredited universities in the United States and are awarded based on academic merit.

Direct Hire Authority (DHA)

The Direct Hire Authority (DHA) is a program that allows the Department of Defense (DoD) to hire qualified candidates for positions in the military without requiring a formal recruitment process. The program is designed to provide opportunities for qualified candidates who may not have been able to compete through the traditional recruitment process.

Center for Excellence in Education

The Center for Excellence in Education is a program that provides support and resources to educators and students in science, technology, engineering, and mathematics (STEM). The program is designed to improve the quality of STEM education and to increase the number of STEM professionals in the workforce.

Student STEM programs (K-12)

The Student STEM programs (K-12) are designed to provide opportunities for students in grades K-12 to explore STEM fields and to develop an interest in pursuing STEM careers. The programs are open to students from all over the United States and are designed to provide hands-on learning experiences in STEM fields.

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In summary, the Pathways Program, AFRL Scholarship Program, SMART Infrastructure Program, AFRL/AFSC Scholarships, Direct Hire Authority (DHA), and Center for Excellence in Education are all programs designed to support students pursuing STEM careers. The Student STEM programs (K-12) are programs designed to provide opportunities for students in grades K-12 to explore STEM fields and to develop an interest in pursuing STEM careers.

Which Armed Service was to first to test air vehicles and in which year did they do so?

- US Air Force, 1947
- US Army Air Service, 1918
- US Army Signal Corp, 1908
- US Army Air Corp, 1927

* If your answer differs greatly from the choices above tell us in the chat!
Hand Lay-Up of PMC's

Time, Cost

Rapid Prototyping Complexity

3D Printed Composites

Resin Transfer Molding (RTM)

Solvent-Free/Imidized Resin
Unitized Components
Tolerances
$\downarrow$Cycle Time
3D Preforms

Key Metrics for Aerospace Resins

**Toughness**- Resins tend to be highly crosslinked “oligomers”.

**Temperature Capability**- Extending usage time and temperature would be advantageous to the physical limit of bond strengths.

Balance requisite viscosities/TOS for RTM and AM applications.

**Water uptake**- One of the main root causes of failure in PMC’s. Dramatic loss in moisture during use initiates micro-cracks that lead to further ingress of moisture and oxygen.

**No truly non-invasive in-situ monitoring tools** during cure and post-cure.
Hypothesis

• Thermally Robust monomers for high temperature applications.

• Large Dichroism along the helical axis, has implications for sensing.

Synthetic Hurdles

• Syntheses tend to be lengthy and low yielding.

• Enantioselective synthesis is key for some applications.

Highly dichroic, non-planar, oxidatively robust fused aromatic rings.

The Application: Strain Sensing

Silicone Elastomers

- and

Pt Catalyst

Epoxy Thermosets

- Polyfunctional Epoxy or Epoxy Silicone
- Amine or Anhydride Catalyst
Optimization of Helicene Products

![Chemical structures and reactions](image)

**Precursor Design**

<table>
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<th>Carbohelicene</th>
<th>Precursor*</th>
<th>% Yield</th>
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**Thermodynamics vs Kinetics**

Refs:


---

**Inducing Enantioselectivity: Chiral Auxiliary Groups**

![Reactions and structures](image)

<table>
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<tr>
<th>Temperature (°C)</th>
<th>Ratio of Diastereomers (+/−)</th>
<th>Diastereomeric excess</th>
</tr>
</thead>
<tbody>
<tr>
<td>+80</td>
<td>20:80</td>
<td>60%</td>
</tr>
<tr>
<td>+25</td>
<td>24:76</td>
<td>52%</td>
</tr>
<tr>
<td>0</td>
<td>38:62</td>
<td>24%</td>
</tr>
<tr>
<td>−78</td>
<td>98:2</td>
<td>96%</td>
</tr>
</tbody>
</table>

Refs:

Synthetic Futures: Multifunctional Helicene Precursors

Dihydroxy[11]-Helicene

Versatile Building Block


DARPA-AMD Program Definition

Increase the Pace of Discovery and Optimization

Develop Closed Loop Systems that Exploit, Build and Integrate Tools for:

1) Extracting Existing Data from Databases and Text;

2) Execute Autonomous Experimental Measurement and Optimization;

3) Incorporate Computational Approaches to Develop Physics-Based Representations and Predictive Tools.

https://www.darpa.mil/program/accelerated-molecular-discovery

UIUC Molecule Maker Research: Modular Suzuki Couplings

Suzuki Reaction: $R - B(OH)_2 + R' - X \xrightarrow{\text{Pd, Base}} R - R' \text{ (or Ester)}$

MIDA Borane

Modular Synthesis and Purification Using LC-MS as a DOR Probe


Molecule Maker ARES OS Dashboard

Autonomous Command Lines

On Board Planner to Integrate All Autonomous Commands, Reaction Campaigns and Data Analysis Tools
AFRL's Version of the Molecule Maker

- Reagents, Solvent, Waste
- Inert Gas Bubblers
- Hot Plate/Stirrer
  16 vials x 20 mL
- Workup and Silica Columns
- Uv-vis Spectrometer
- Fluorescence Detector
- 4/8 Teflon Valve Array (192 valves)

First Molecule Maker with Closed Loop (Autonomous) NMR Analytical Tool:
Direct Structural Evidence of Reaction Outcome(s)

**Power of Closed-Loop Iteration**

- **Autonomy**: System designs its own experiments using AI & Machine Learning
- **Automation**: Execute experiments automatically
- **Modeling**: Integrate modeling & simulation directly in the loop
- **Analysis**: Knowledge Representation
- **Science**: through in-line hypothesis generation and testing
- **More iterations >> More experiments**

Semi-autonomous, closed loop NMR acquisition

Base: Cs2CO3, excess
Solvent: Toluene

Continuous Variables
- Catalyst Loading
- Temperature (°C-180°C)
- Time (0-6hrs)

Discrete Variables
- Precatalyst Source
- Legend

Reaction is complete within 3-4hrs.

Autonomous NMR Data Workup and Analytics

Phasing
Baseline Correction
Reaction Progression
Noise Reduction
Data Fitting & Integration
From Biotic to Extraterrestrial (Abiotic?) Organic Synthesis


~10^5 kg/yr of organic carbon delivered to Earth

Extraterrestrial Sourcing of Organic Matter

Aromatic Hydrocarbons

L-Amino Acids

Non-Proteogenic and Rare

Sugar Acids

RNA and DNA Nucleobases

Terestrially Rare

Can the Space Force harvest, extract, modify and utilize?
Acknowledgements

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NEXT WEEK!

Thurs., June 16, 2022 | 2:00pm–3:15pm ET

Starting a Company: How to Set Up Equity and Securities Structures
Co-produced with ACS Division of Small Chemical Businesses and ACS Division of Business Development & Management

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NEXT WEEK!

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