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<th>Technology &amp; Innovation</th>
<th>Drug Design and Delivery</th>
</tr>
</thead>
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<thead>
<tr>
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<th>Popular Chemistry</th>
<th>Business &amp; Entrepreneurship</th>
</tr>
</thead>
<tbody>
<tr>
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Younger Chemists Committee (YCC)

Early Career Development Workshops
Q&A
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Upcoming Broadcasts

**FACE MASKS**

**MATERIALS, DISINFECTION & REUSE DURING COVID-19**

**Friday Rebroadcast**

Friday, June 19, 2020 at 2-3pm ET
Speakers: Supratik Guha, University of Chicago, and Argonne National Laboratory; and Yi Cui, Stanford University
Moderator: Laura Cassiday, American Chemical Society

What You Will Learn
- What types of fabrics and household cloth are effective in particle filtration and why
- The basics of particle filtration and data on filtration efficiencies as a function of size for common fabrics that are used in cloth masks
- How to disinfect N95 masks and how many times you can do it without reducing filtration efficiency

Co-produced with ACS External Affairs & Communications and ACS Publications

**VIRTUAL INTERVIEWING**

**IN A FAST-CHANGING ECONOMY**

**Monday Rebroadcast**

Monday, June 22, 2020 at 2-3pm ET
Speakers: Bob Conerly and Allen Hadden of Staffing Advisors
Moderator: Brooke Lockhart, Staffing Advisors

What You Will Learn
- How virtual interviewing differs from in-person interviewing and how you can use the format to your advantage
- How to make an effective case for yourself (without bragging)
- How to apply timeless principles that are successful in any interview setting and at every career level

Co-produced with ACS Industry Member Programs

**Learning to LOVE BREVITY & CLARITY**

**Tuesday, June 23, 2020 at 2-3pm ET**
Speakers: Mark Jones, Dow Chemical
Moderator: Bryan Tweedy, American Chemical Society

What You Will Learn
- Accessible communication does not mean simplistic communication
- Brevity is both appreciated and improves clarity
- Self-editing is the surest way to improve clarity and attain brevity

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Diana Gerbi, 2018 POLY Chair
3M(retired)

“….the next generation of polymer scientists is where we put a lot of our focus and we’ve really established a tremendous network of scientists at all points in their career. …our more seasoned members are active in helping support and foster the growth of the next generation through mentoring and a very active awards program.”

Marc Hillmyer, 2017 POLY Chair
University of Minnesota

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Karl Haider, 2016 POLY Chair
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- Many Award Opportunities

SELF HEALING POLYMERS AND VITRIMERS
Self Healing Polymers and Vitrimers

Brent Sumerlin  
George B. Butler Professor, Department of Chemistry, University of Florida

Marek Urban  
J.E. Sirrine Foundation Endowed Chair and Professor, Department of Materials Science and Engineering, Clemson University

Christopher Bowman  
Distinguished Professor, Clinical Professor of Restorative Dentistry, Co-Director of the NSF I/UCRC for Fundamentals and Applications and Photopolymerizations, University of Colorado, Boulder

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SELF-HEALING POLYMERS

Marek W. Urban
Department of Materials Science and Engineering
Center for Optical Materials Science and Engineering (COMSET)
Clemson University, USA
SELF-HEALING APPROACHES IN POLYMERS

a Physical approaches

Interfacial regions
Inter-diffusion
Phase-separated morphologies

Damage
Shape-memory recovery
Melting inter-diffusion

γ-Fe₂O₃

b Chemical approaches

Reactive chain ends
Covalent rebonding
Free-radical rebonding

Supra-molecular chemistry
H-bonding
π–π stacking
Guest-host chemistry
Metal–ligand coordination
Ionic interactions

C Physico chemical approaches
Van der Waals interactions
Encapsulation
Cardiovascular network

www.nature.com/articles/s41578-020-0202-4
WHAT IS AN ESTIMATED LIFE-TIME OF
4- or 5-MEMBER HETERO CYCLIC RADICALS?

- 10 msec
- 25 sec
- 2 days
- ~1 month
- 10 psec

Prog. Polym. Sci., 2020, 102, 101208
REFORMATION OF COVALENT BONDS

Exposure to UV Light

<table>
<thead>
<tr>
<th></th>
<th>0</th>
<th>15</th>
<th>30 MIN</th>
</tr>
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<tr>
<td><strong>Science</strong>, 2009, <strong>323</strong>, 1458.</td>
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<td><strong>J. Mater. Chem.</strong>, 2011, <strong>21</strong>, 14473</td>
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</table>

**Heterocyclic Compounds**


Angew. Chemie, 2014, **53**, 12142–12147
REVERSIBLE REACTIONS ENABLING REFORMATION OF COVALENT BONDS

DYNAMIC REFORMATION OF COVALENT BONDS

a Schiffs base bond regeneration

\[
\begin{align*}
\text{C} & + \text{H}_2\text{N} & \xrightarrow{\text{---}} & \text{C} \\
& & & \text{N}\\
\end{align*}
\]


b Acylhydrazone rebonding

\[
\begin{align*}
\text{\text{--CH}} & + \text{H}_2\text{N} & \xrightarrow{\text{---}} & \text{\text{--CH}} \\
& & & \text{N}\\
\end{align*}
\]


c Oxime rebonding

\[
\begin{align*}
\text{R}_1\text{C} & + \text{H}_2\text{N} & \text{O} & \xrightarrow{\text{---}} & \text{R}_1\text{N} & \text{O} \\
& & & & & \text{R}_2
\end{align*}
\]


d Boronic ester reformation

\[
\begin{align*}
\text{\text{---B}} & + \text{H}_2\text{O} & \xrightarrow{\text{---}} & \text{\text{---B}} \\
& & & \text{OH} \\
\end{align*}
\]


e Boronic ester reformation in rotaxane-based polymers

Chem 1, 766–775 (2016).
SUPRAMOLECULAR DYNAMIC CHEMISTRY

a Triple H-bonding


b Combining strong and weak H-bonds

SUPRAMOLECULAR DYNAMIC CHEMISTRY

Excessive H-bonding

\[
\text{Amidoethyl imidazolidone}
\]

\[
\text{Di(amideethyl) urea}
\]

\[
\text{Diamido tetraethyltrisutra}
\]

\[
\text{Oligomeric mixture}
\]

SELF-HEALING USING METAL–LIGAND COORDINATION CHEMISTRY

**a** Photoresponsive system

![Chemical structure](image)


**b** Self-healing dielectric elastomer

![Chemical structure](image)

SELF-HEALING USING METAL–LIGAND COORDINATION CHEMISTRY


IONIC INTERACTIONS APPLIED IN SELF-HEALING

a. Self-healing ionomer
   - pEMMA

b. Polyelectrolyte complexes with phosphate anions
   - PAH/tripolyphosphate


f. Self-healing in a commodity polymer
   - Heat

SELF-HEALING “Key-and-Lock” ACRYLIC COPOLYMERS

Poly(methyl methacrylate/n-butyl acrylate) (pMMA/nBA)

Self-healing occurs within a narrow compositional range

Science, 2018, 362, 220.

van der Waals (vdW) INTERACTIONS IN SELF-HEALING

Science, 2018, 362, 220.

Poly(methyl methacrylate/n-butyl acrylate)

Crystalline nanodomain of E–E segments

Self-Healing of Plants – Delosperma Cooperi

Delosperma cooperi plants (Pink Carpet or Iceplant) in the Freiburg Botanical Garden. © Plant Biomechanics Group Freiburg and 2018 Speck et al.; licensee Beilstein-Institut

How does the elastic component contribute to damage closure of polymer?

SELF-HEALING vs. PHASE MORPHOLOGY

**IR IMAGES**
- PCL-PUR (P)
  - Polymerization induced phase separation
- PCL-PUR (M)
  - Thermally induced phase separation

**OPTICAL IMAGES**
- Tensile properties
  - Micro-phases separated PCL-PUR (P)
  - Nano-phase separated PCL-PUR (M)

**TENSILE PROPERTIES**
- PCL-PUR (P)
  - 87% RECOVERY
  - 2 hrs Healing 65°C
- PCL-PUR (M)
  - 51% RECOVERY
  - 2 hrs Healing 65°C

**Tm** = 51 and 168°C (DSC)

**Tm** = 51 and 162°C (DSC)

Self-Healing Polymers Inspired by Leaves

Fibers are drawn during polymerization

PCL - PUR (P) FIBER

CHEM, 2018, 4, 1928
This slide – show some biological self-healing events

Nano- to Macro-Scale Self-Healing

Shape Memory Effect vs Rebonding

Polyurethane Fibers

WHAT IS ANTICIPATED GROWTH OF SELF-HEALING POLYMER TECHNOLOGIES IN USA BY 2025?

- 0%
- 11.1%
- 25.1%
- 46.1%
- 63.6%
Molecular chemical events are responsible for macroscopic responses without intervention.

Morphology control may facilitate energy storage and recovery during damage-repair cycles.

Narrow monomer molar ratios in simple alternating/random copolymers offers repeatable self-healing properties of thermoplastic polymers.

A key characteristic feature are enhanced van der Waals (vdW) interactions rather than the reformation of hydrogen or covalent bonds.

Self-healing driven by entropic energy recovery stored during damage.

Self-healing driven by interfacial flow and diffusion.
ACKNOWLEDGEMENTS

URBAN RESEARCH GROUP
www.clemson.edu/cecas/urbanresearch

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- Industrial Sponsors
**Vitrimer**s

<table>
<thead>
<tr>
<th>Concept</th>
<th>Transesterification exchange reactions</th>
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<td><img src="image1" alt="Diagram" /></td>
<td><img src="image2" alt="Diagram" /></td>
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</tbody>
</table>

- **Transamidation exchange reactions**
  - ![Diagram](image3)

- **Transcarbamoylation exchange reactions**
  - ![Diagram](image4)

- **Transamination of vinylogue amides or urethanes**
  - ![Diagram](image5)

- **Transcarbonation exchange reactions**
  - ![Diagram](image6)

- **Dioxaborolanes metathesis**
  - ![Diagram](image7)

References:

Covalent Adaptable Networks as Stimuli Responsive Materials

Or...

How Can We Make a Square Peg Fit Into a Round Hole?

Christopher N. Bowman
Department of Chemical and Biological Engineering
Materials Science and Engineering
Department of Restorative Dentistry
University of Colorado

Conflict of Interest Disclosure
CNB has a financial interest in patents and royalties from those patents that have been licensed related to addition fragmentation in dental and other materials
Covalently Crosslinked Networks (i.e., Thermosets)

Covalently crosslinked polymeric materials are ubiquitous but suffer from significant shortcomings.

Hydrogels for Tissue Engineering

Orthopedic Devices and Shape Memory Polymers

The Trabant – made largely of Duroplast

Epoxy encapsulated hybrid circuit

Composite Structural Materials

Methacrylate-based dental restorative

Flat Panel Display Coatings

Cell Phone and Other Optics

Contact Lenses

Image source: wikipedia
What opportunities and alterations in material properties would be possible if the bond structure wasn’t “permanent”? 
Question…

What opportunities and alterations in material properties would be possible if the bond structure wasn’t “permanent”?

- Recycling and reprocessing
- Stress relaxation
- Actuation
- Adhesion
- Self-healing
- Etc.
Dynamic Covalent Chemistry

Broadly there are two categories of reversible reactions that we can consider:

Reversible Addition Reactions

\[ \text{Reversible Exchange Reactions} \]

So, why would we care about these reactions and what would they enable in crosslinked networks?
Non-Equilibrium State $\rightarrow$ Relaxed State

Stress, Molecular Alignment, Birefringence, Interfaces, Phase Separation, Healing, Shape, Etc.

**Covalent Adaptable Network Paradigm**

Thermosets

Thermoplastics

stimulus

DCC

Non-Equilibrium State

Relaxed State

Stress, Molecular Alignment, Birefringence, Interfaces, Phase Separation, Healing, Shape, Etc.

Covalent Adaptable Network Paradigm: Inducing a Solid-to-Fluid Transition

During Light Exposure the Dynamic Covalent Chemistry is Activated, Leading to the Formation of a Fluidic Material

Once the Light is Extinguished, the Material Returns to its Solid State
Covalent Adaptable Networks: Permanent Network Rearrangement

Permanent Shape Change Achieved When Bond Exchange Is Activated During Strain

Altering Shape: The Most Important Property
Nature of the Dynamic Covalent Crosslinks Constitutes the Type of Covalent Adaptable Network

**Reversible Addition Reactions**

- Diels-Alder Reactions


**Reversible Exchange Reactions**

- Addition—Fragmentation Chain Transfer


- Transesterification — i.e., vitrimers

Covalent Adaptable Networks: An old concept with new applications

Tobolsky 1956: “In actual fact, we discovered that all rubbers show $E_r(t)$ curves that decay to zero stress at sufficiently high temperatures, and we attributed this stress decay to chemical reactions such as chain scission by oxidative cleavage or reorganization of the network structure by ionic interchanges” – This includes polyesters, polyurethanes, vulcanized rubbers

Chemorheology: Stress relaxation due to chemical reactions

Proposed DCC in vulcanized rubbers

Fig. 1. Chemical stress relaxation for various vulcanized rubbers at 130° (reference 25).

Question…

What stimuli would be desirable to use as activators for turning the dynamic covalent chemistry on/off?

(a) Temperature Change
(b) Irradiation
(c) Molecular Detection
(d) Solvent Condition Change
(e) None of the above
What stimuli would be desirable to use as activators for turning the dynamic covalent chemistry on/off?

(a) Temperature Change – easily activated and controlled
(b) Irradiation – spatiotemporal control
(c) Molecular Detection – useful as a sensor
(d) Solvent Condition Change – biological applications
(e) None of the above
Photoplasticity: Covalent Adaptable Networks (CANs)

Addition-fragmentation in the network alleviates stress as bonds are broken and reformed.

Addition-Fragmentation of Allyl Sulfides

Science, 308, 1615 (2005); Advanced Materials, 18, 2128 (2006)
Stress Relaxation via Photoinduced Plasticity

Light on (405 nm, 20 mW/cm²)

50% Strain

Stress (MPa)

Time (min)

Commercialized by 3M for Reduction of Polymerization Stress

Networks formed by Thiol Click Michael Addition are Capable of Complete Relaxation of Stress

C.J. Kloxin et al., Advanced Materials, 2011
Phase Transitions in LCNs:
Reversible Phase and Shape Changes

Liquid Crystal Phase
“Order”

Isotropic Phase
“Disorder”
Programming Monodomain to and from Polydomain

Polydomain

Stretch

Highly sheared regions in domain walls

Bond Exchange

Stress relaxed in domain wall

Monodomain

Stress relaxation occurs through allyl sulfide exchange in the polymer and stress-rich regions at the wall
**Fully Reversible Surface and Bulk Shape Control**

**Cube**
- Strained area
- Note: Only strained regions will program

**Flower**

**Miura Ori**

**Nanoimprint**
- Actual time
- 25°C
- 100°C

- Strain sample by folding
- Irradiate with light
- Heat to 120°C
- Allow to cool to 25°C

McBride et al., *Science Advances*, 2018
Does a Square Peg Fit into A Round Hole?

McBride et al., Science Advances, 2018
Covalent Adaptable Networks (CANs): Dynamic Thiol-Thioester Exchange
Inducing a Solid-to-Fluid Transition: Switching from One State to Another

During Light Exposure the Dynamic Covalent Chemistry is Either Activated or Deactivated By Generating or Eliminating a Catalyst

Once the Light is Extinguished, the Material *Remains* in its New State – It is Thus Bistable
Thioester Based Networks are Reconfigurable Under Ambient Conditions

- No birefringence
- Stretch at RT
- Birefringent
- Hold at RT for 30 min
- No birefringence

No birefringence

- Stretch at RT
- Birefringent
- Hold at RT for 30 min
- Birefringent
Polymerizing ~10 grams of material into an optically clear puck, it was cut, healed at room temperature by loading into a syringe and applying mild pressure.
The “ON” Switch:
Demonstration of Spatial Control via Photobase Generation

[Diagram showing the process of stretching, holding, releasing, and the corresponding sideview of the film with specified steps: 400 nm Light, Release Photobase, Stretch and Thinning]
The “ON” Switch: Demonstration of Spatial Control via Photobase Generation
Question:
In Conventional Composite Systems Where the Filler Is Much Higher Modulus, Where Do the Stresses Generally Concentrate?

a) The resin phase
b) The filler phase
c) The interface between the filler and the composite
d) All of the above
**Question:**

In Conventional Composite Systems Where the Filler Is Much Higher Modulus, Where Do the Stresses Generally Concentrate?

a) The resin phase  
b) The filler phase  
c) The interface between the filler and the composite  
d) All of the above

While all regions in a composite bear stresses, the *interface* between the filler and the polymeric matrix is often a region of concentrated stresses that have a significant influence on the mechanical performance and lifetime of composite materials.
While all regions in a composite bear stresses, the interface between the filler and the polymeric matrix is often a region of concentrated stresses that have a significant influence on the mechanical performance and lifetime of composite materials.

**Hypothesis:** Triggered DCC activated in the resin during formation combined with perpetual DCC at the interface is optimal.
Implementation of TTE at the Resin-Filler Interface

Activated TTE Interface composite failure

Thioester filler with DABCO catalyst

TTE sample fails at the small notch, requiring more than twice the total energy to fail the material– unlike any other conventional material.

(1.1:1) Thiol:ene, 10 wt% SNPs, 6 mol% DABCO, 1 wt% I 819. Crosshead speed = 1 mm/min

Sowan, Polymer Chemistry, In Press
**Implementation of TTE at the Resin-Filler Interface: Cyclic Loading and Fatigue**

Dynamic composites and improvement in cyclic behavior

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>TTE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stress (MPa)</td>
<td>12 ± 3</td>
<td>28 ± 6</td>
</tr>
<tr>
<td>Strain (%)</td>
<td>20 ± 8</td>
<td>30 ± 10</td>
</tr>
<tr>
<td>Toughness (MJ/m³)</td>
<td>1.9 ± 0.5</td>
<td>6.1 ± 0.9</td>
</tr>
</tbody>
</table>

The **thioester-modified composite** was found to survive more than **20 cycles** while the **control** composites were only able to survive **3-4 such cycles**.

(1.1:1) Thiol:ene, 10 wt% SNPs, 1 wt% DABCO, 1 wt% I 819. Curing conditions: (400-500) nm, with 50 mW/cm² intensity.

Sowan, Polymer Chemistry, In Press
Summary

• Implementation of Dynamic Covalent Chemistry in Crosslinked Networks Represents a Powerful Approach to Identify and Control Polymer Properties

• Breadth of Chemistries and Triggers Possible

• Control is possible of
  – Type of reaction
  – On/Off trigger
  – Timescale of reaction
  – Implementation relative to the lifecycle of the material
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“Being a member of POLY has helped me identify a network of colleagues and establish myself in the polymer chemistry community. For the small cost of a POLY membership, you can join a strong and passionate group of scientists that can assist you throughout your career, through discussions, networking, and guidance.”

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Brent Sumerlin
George B. Butler Professor, Department of Chemistry, University of Florida

Marek Urban
J.E. Sirrine Foundation Endowed Chair and Professor, Department of Materials Science and Engineering, Clemson University

Christopher Bowman
Distinguished Professor, Clinical Professor of Restorative Dentistry, Co-Director of the NSF I/UCRC for Fundamentals and Applications and Photopolymerizations, University of Colorado, Boulder

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**FACE MASKS**
**MATERIALS, DISINFECTION & REUSE DURING COVID-19**

*Friday Rebroadcast*

Friday, June 19, 2020 at 2-3pm ET
Speakers: Supratik Guha, University of Chicago, and Argonne National Laboratory and Yi Cui, Stanford University
Moderator: Laura Cassiday, American Chemical Society

What You Will Learn:
- What types of fabrics and household cloth are effective in particle filtration and why.
- The basics of particle filtration and data on filtration efficiencies as a function of face for common fabrics that are used in cloth masks.
- How to disinfect N95 masks and how many times you can do it without reducing filtration efficiency.

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**VIRTUAL INTERVIEWING**
**IN A FAST-CHANGING ECONOMY**

*Monday Rebroadcast*

Monday, June 22, 2020 at 2-3pm ET
Speakers: Bob Conell and Aileen Hedin of Staffing Advisors
Moderator: Brooke Lockhart, Staffing Advisors

What You Will Learn:
- How virtual interviewing differs from in-person interviewing and how you can use the format to your advantage.
- How to make an effective case for yourself (without bragging).
- How to apply timeless principles that are successful in any interview setting and at every career level.

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**Learning to LOVE BREVITY & CLARITY**

*Tuesday, June 23, 2020 at 2-3pm ET*
Speakers: Mark Jones, Dow Chemical
Moderator: Bryan Tweedy, American Chemical Society

What You Will Learn:
- Accessible communication does not mean simplistic communication.
- Brevity is both appreciated and improves clarity.
- Self-editing is the surest way to improve clarity and attain brevity.

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