



# High-Performance Fiber-Reinforced Vitrimer Composites through Compression Molding

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**2020 DOE Vehicle Technologies Office Annual Merit Review**

# Overview

## **Timeline**

- Project start date: July, 2019
- Project end date: June, 2020
- Percent complete: 80%

## **Budget**

- Total project funding
- Funding for FY 2019: \$200 K
- Future Funds anticipated (\$0)

## **Partners**

- University of Colorado Boulder
- NCOTechnologies, LLC (Project lead)

## **Barriers and Technical Targets**

- The use of carbon fiber composites with higher potential for vehicle weight reduction
- Develop lower cost carbon fiber composites with net-shape manufacturing cycle time <3 min
- Improve recycling of carbon fiber composites.

*USDRIE Materials Technical Team Roadmap, October 2017, sections 4 and 5*

# Relevance

## **Impact**

- Carbon-fiber-reinforced-composites (CFRCs) have excellent strength-to-weight ratio and great potential as light weight materials in auto-industry. However, they are expensive to produce (long cycle time) and difficult to recycle.
- The use of vitrimers as polymer binders in CFRCs is a “killing several birds with one stone” strategy, enabling rapid processing by compression molding, easy repair of structure defects, and nearly 100% recycle of all the chemical components and carbon fibers in their virgin state.

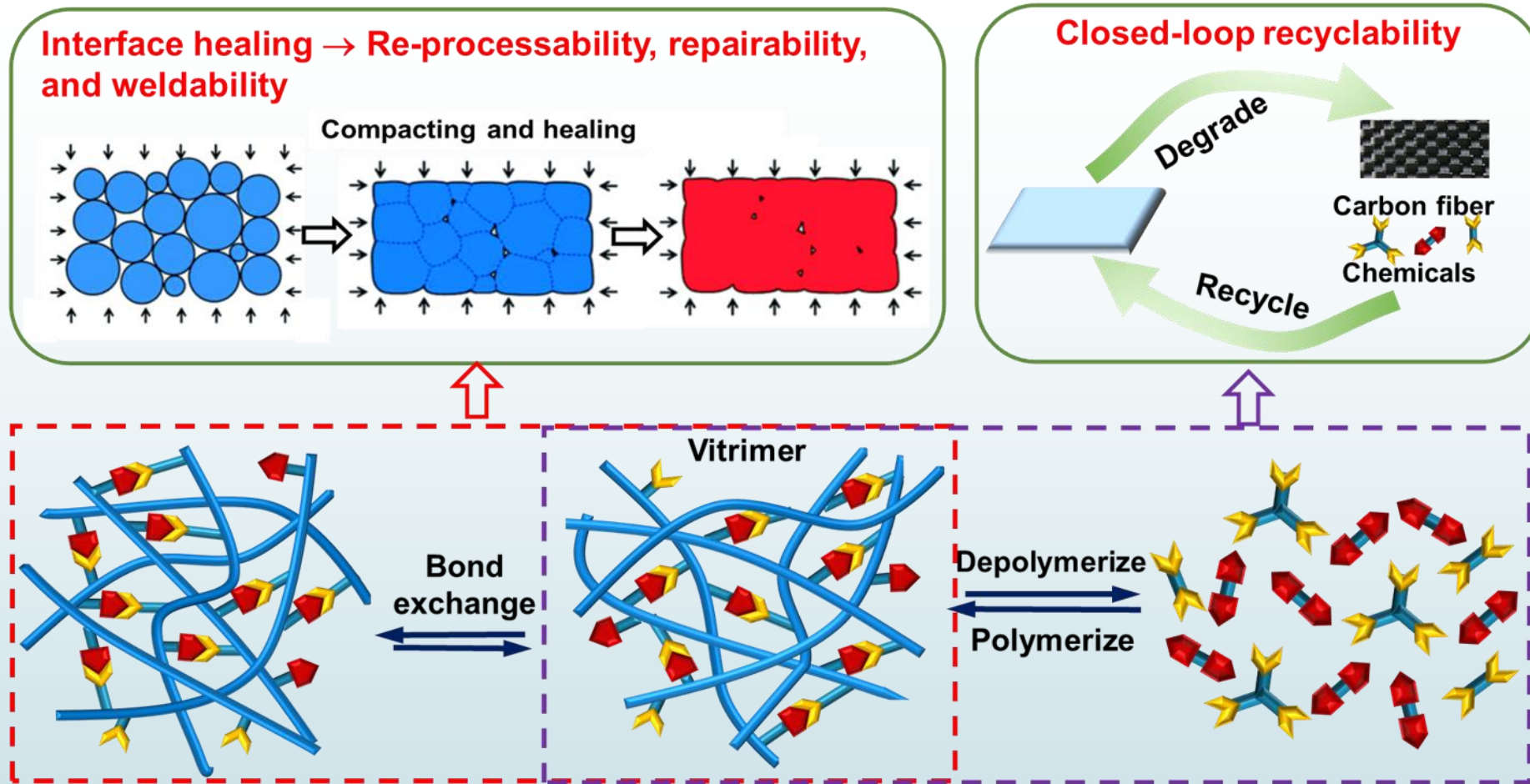
## **Objectives**

- Develop fast-moldable vitrimers with good mechanical and thermal properties
- Develop fast compression molding technique to form CFRCs in a short processing time 2-3 min
- Investigate reprocessability and repairability of CFRCs through simple heat-pressing
- Develop closed-loop recycling of all the chemicals and carbon fibers in their original full length and woven state

# Milestones

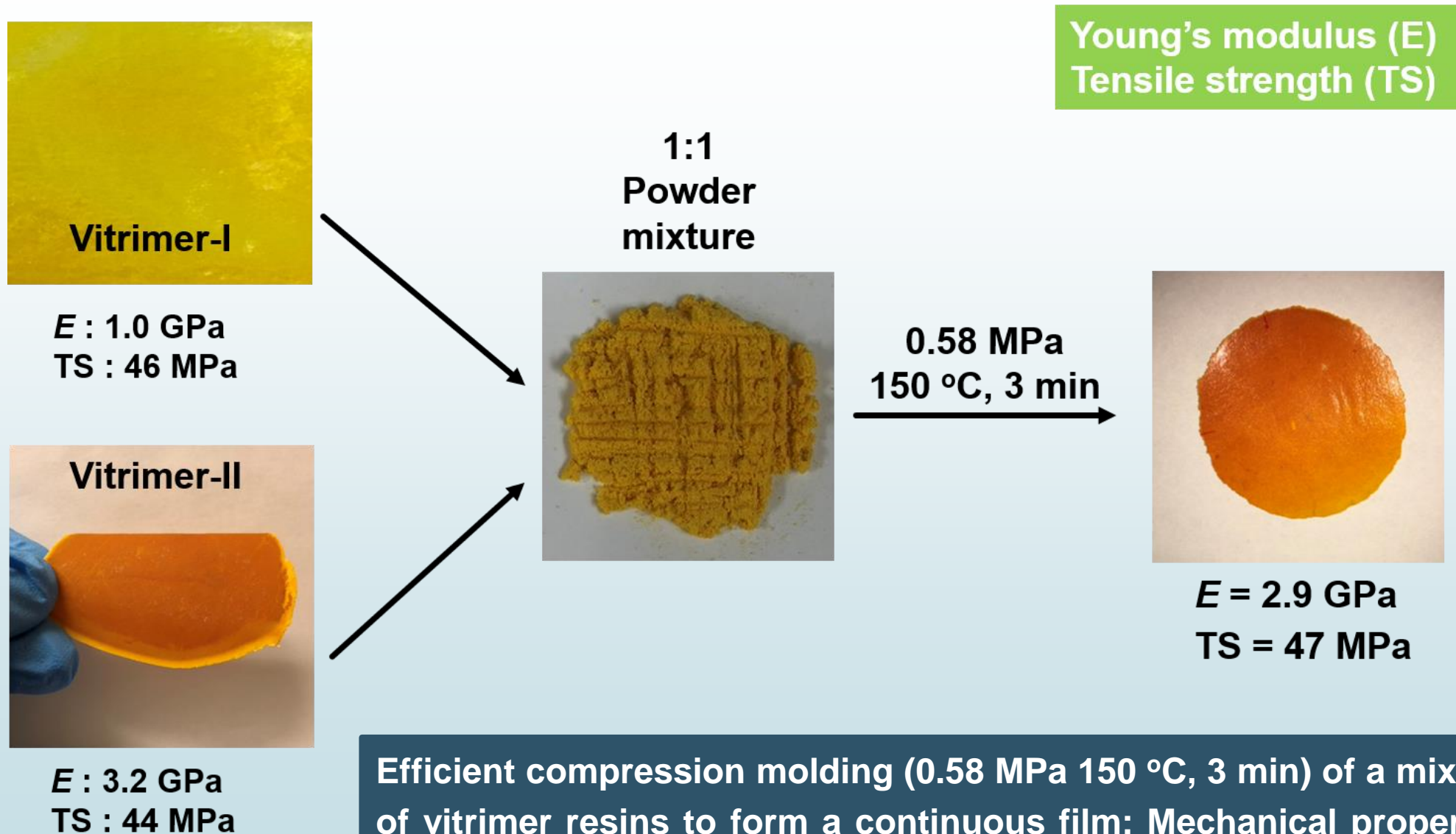
| Month/Year   | Description of milestone  | Status      |
|--------------|---|-------------|
| January 2020 | Develop vitrimers with high mechanical properties that can be efficiently compression molded                                  | Complete    |
| March 2020   | Develop efficient compression molding technique to prepare vitrimer films with tunable properties from a mixture of vitrimers | Complete    |
| April 2020   | Develop efficient compression molding (2-3 min) protocol to form CFRCs using vitrimer resin                                   | Complete    |
| June 2020    | Investigate repairability, reprocessability and depolymerization-based closed-loop recycling of chemicals and fibers          | On schedule |
| June 2020    | Conduct computational modeling study on the particle fusion process during compression molding                                | On schedule |

# Technical Approach



- Achieve fast bond exchange reactions at an elevated temperature
- Enable efficient vitrimer particle fusion and interface healing through bond exchange reactions during compression molding
- Depolymerize the polymer matrix by upsetting the stoichiometry of the end groups at the end of product life

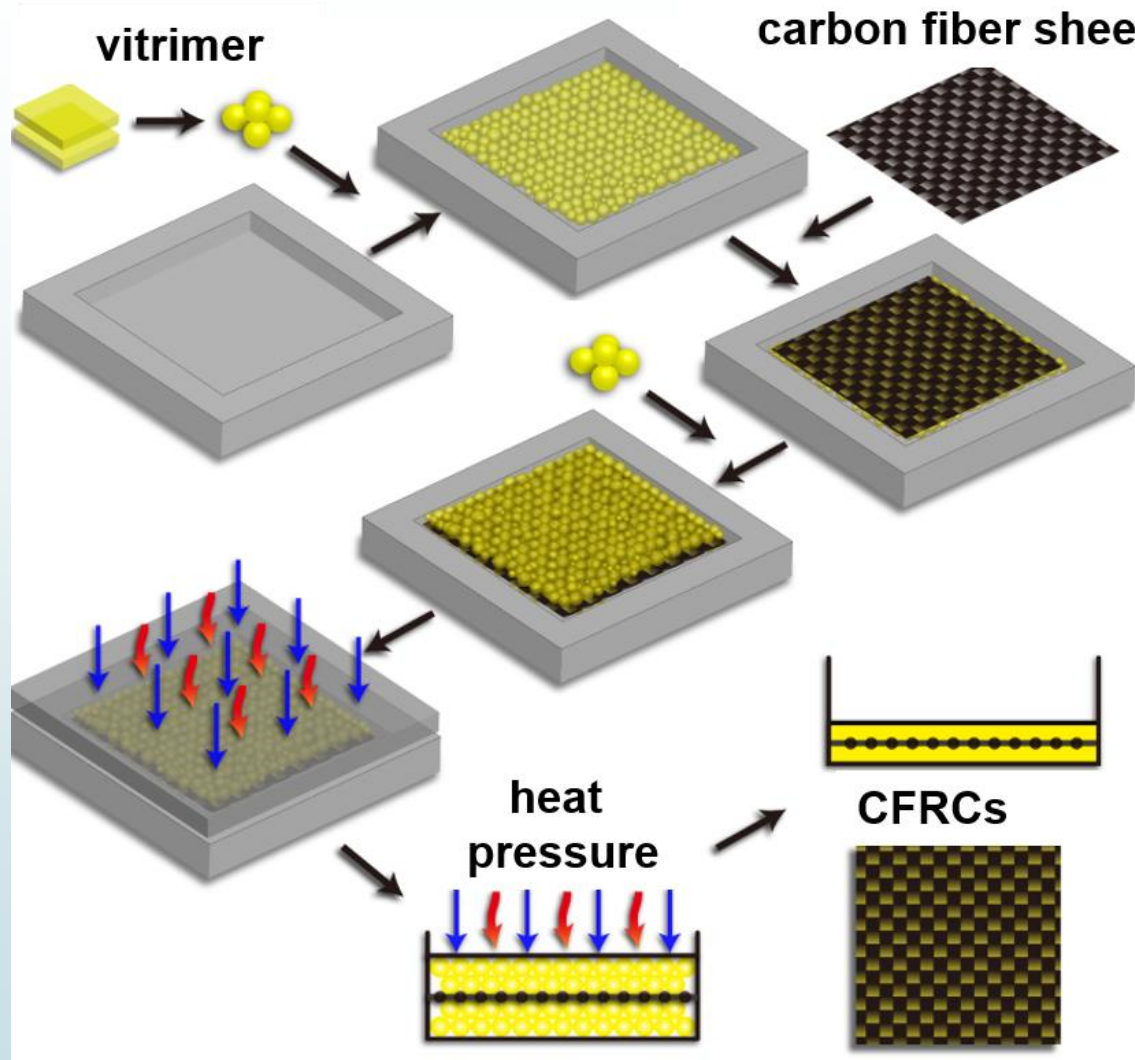
# Accomplishments: *Vitrimer development*



Efficient compression molding (0.58 MPa 150 °C, 3 min) of a mixture of vitrimer resins to form a continuous film: Mechanical properties can be tuned by varying the mass ratio of two polymers.



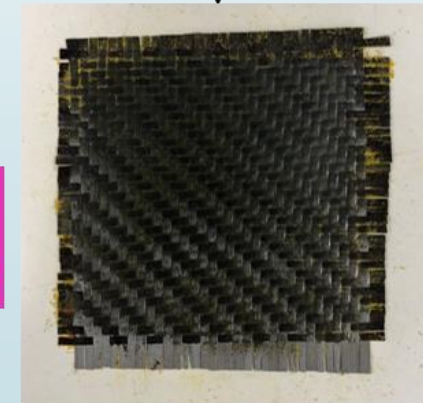
# Accomplishments: *Compression molding of CFRCs*



Vitrimer mixture



0.58 MPa  
150 °C, 3 min

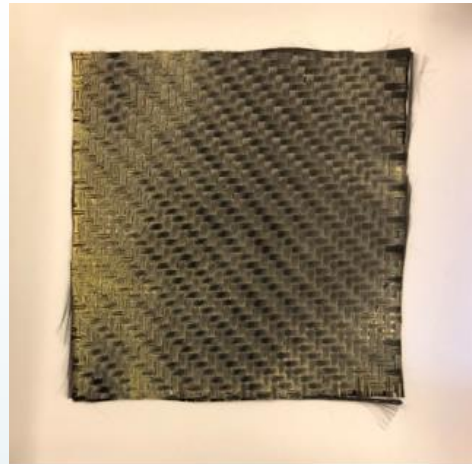


$E = 30 \text{ GPa}$   
 $TS = 225 \text{ MPa}$

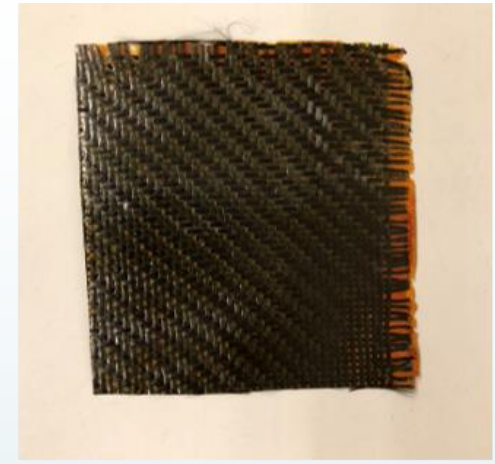
Efficient compression molding (0.58 MPa 150 °C, 3 min) of CFRCs with high mechanical properties (tensile strength 225 MPa, modulus 30 GPa)

# Accomplishments: *Recycling of CFRCs*

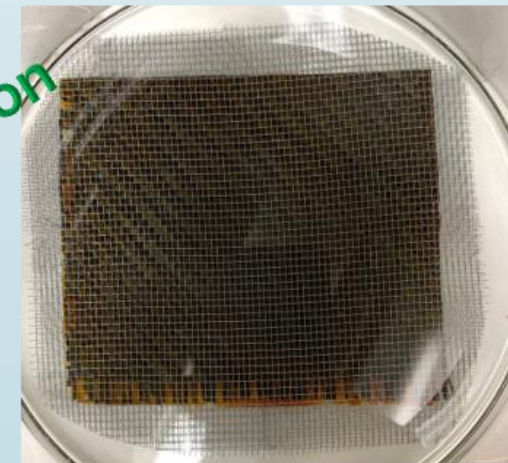
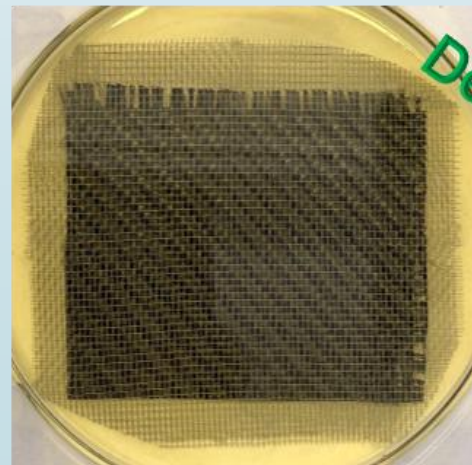
- Chemical components can be reused after evaporating all the volatiles to reform the vitrimer with similar properties.
- Carbon fibers in their original length and state (woven or non-woven) can be recovered and directly reused in the next production cycle after simple drying
- 95-100% recovery of mechanical properties



**95-100%  
recovery of  
*E* and TS**

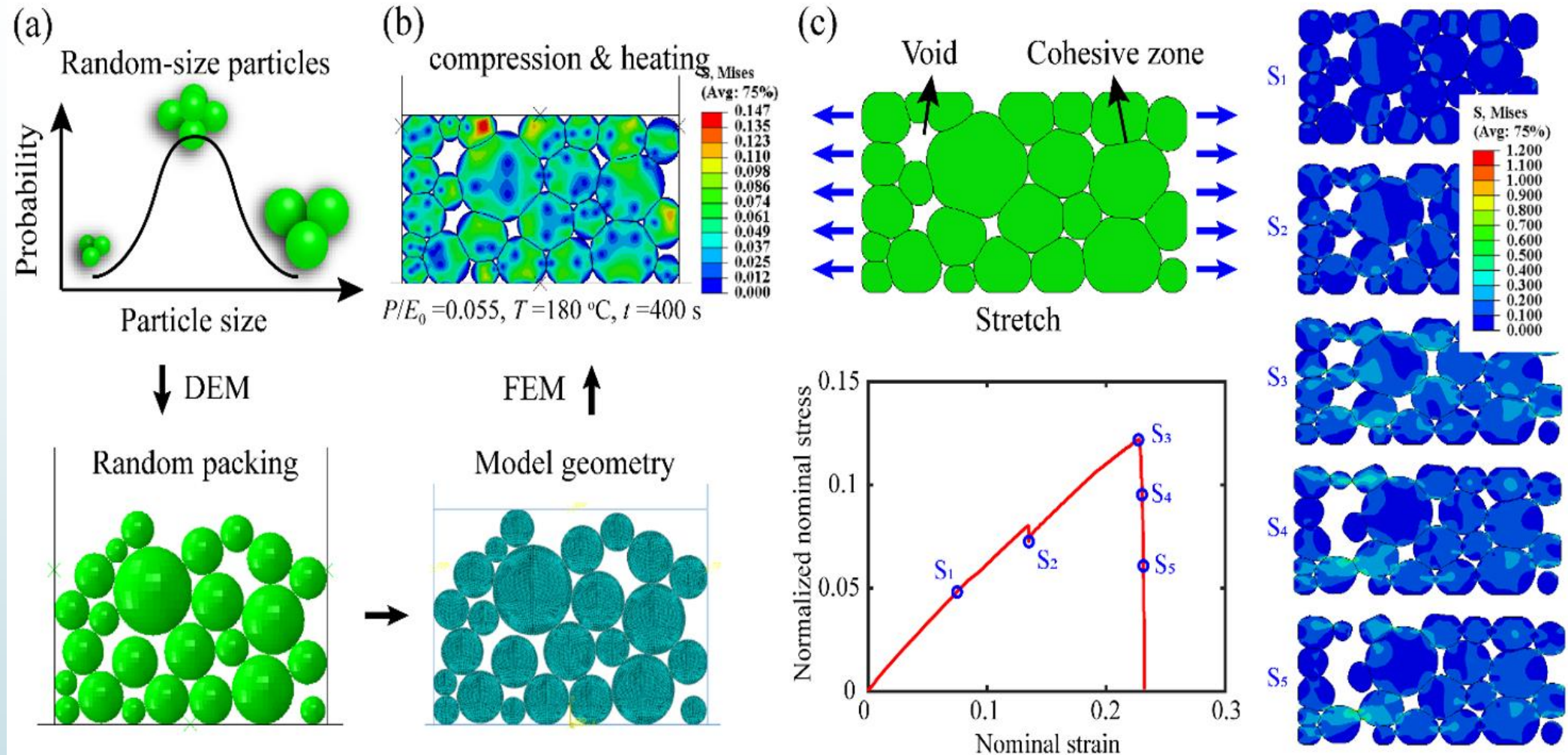


**Depolymerization**





# Accomplishments: *Modeling study of vitrimer fusion*



The uniaxial tensile behavior of the compressed materials can be simulated based on the particle size and distribution



## **Responses to Previous Year Reviewers' Comments**

This project was not reviewed last year.

# Remaining Challenges and Barriers

Vitrimeres are novel malleable thermosets with many distinct advantages that would offer significant environmental and economic benefits. However, the lack of fundamental and systematic understanding of these novel materials holds back further advance of the technology.

- Structure-property relationships of vitrimers are poorly understood, thus rational design of monomers and the selection of suitable dynamic bonds are nearly impossible. The combination of computational prediction and experimental data is desired.
- Dynamic bond exchange reactions, whose reversibility can be turned on-off as needed, are limited. In this regard, the development of a catalyst system that can function on-demand under mild conditions is highly desired.
- Better understanding of interfacial interactions between fiber/vitrimeres and vitrimer/vitrimer is needed to further improve the vitrimer-fiber adhesion and mechanical properties of CFRCs.
- Even distribution of vitrimers in CFRCs is challenging. This leads to the formation of defect sites and inhomogeneity of the materials.



# Collaborations

- **NCO technologies**

Design and synthesis of vitrimers, vitrimer structure formulation and hybridization, compression molding techniques, repairing and recycling of CFRCs

- **University of Colorado Boulder**

- Department of Chemistry (Sub)**

- polymer structure characterization, surface imaging, morphology characterization, thermal property characterization

- **University of Colorado Boulder**

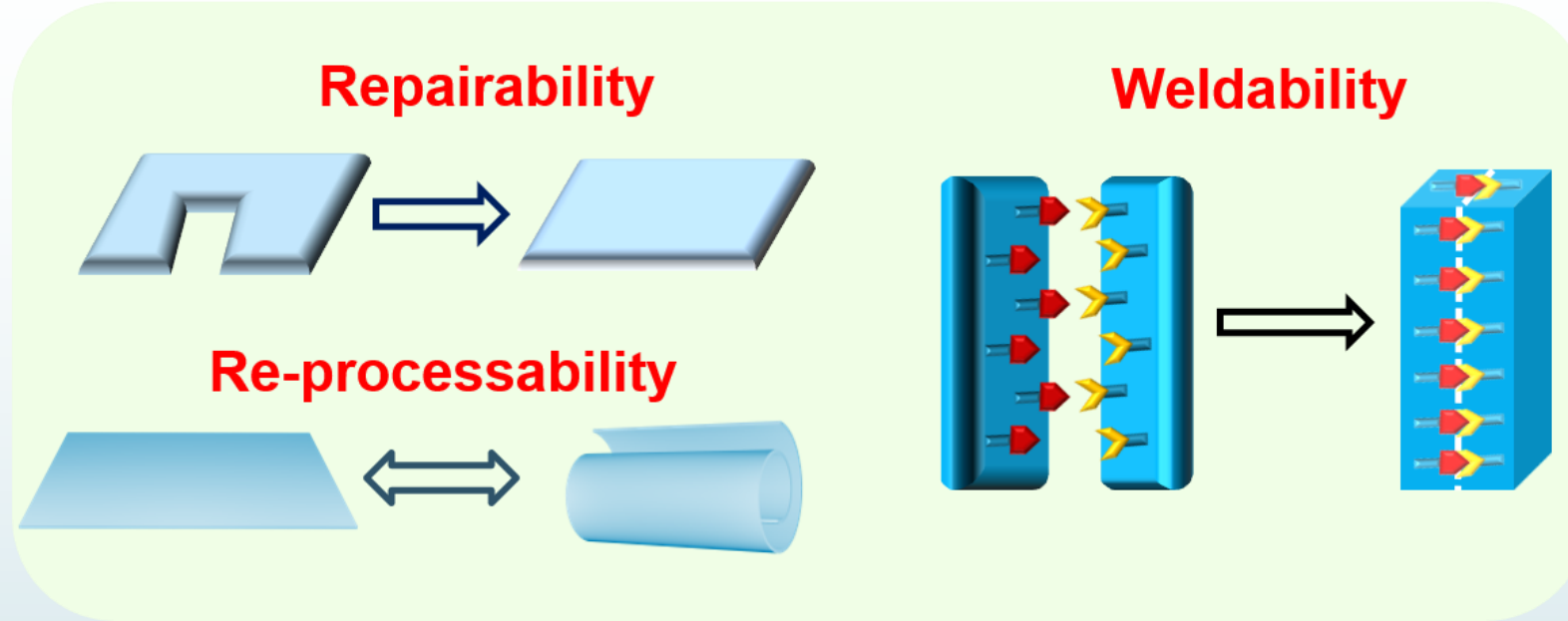
- Department of Mechanic Engineering (Sub)**

- Mechanical property characterization (tensile and flexural), computational modeling, access to high performance computing clusters



**Boulder**

# Future and On-going work



- Continue working on the development of high-performance vitrimer and CFRCs
- Reduce CFRC processing time to 90 s
- Investigate heat-driven reprocessability and weldability of CFRCs under mild conditions ( $<150\text{ }^{\circ}\text{C}$ ,  $< 3\text{ min}$ )
- Repair of CFRCs damage (breaking of covalent bonds, intralaminar matrix cracks, delamination) through vitrimer particle fusion under heat and pressure

Any proposed future work is subject to change based on funding levels



# Summary

## Technical highlights

- Developed moldable vitrimers with high mechanical properties ( $E \sim 3\text{-}4$  GPa)
- Developed fast compression molding technique of high performance CFRCs ( $E = 30$  GPa, TS = 225 MPa) with processing time of 3 min

## Advantages

- Low cost and time-efficient
- Reprocessible, repairable, and recyclable
- Significant commercial potential

## Impacts toward VTO objectives:

By replacing currently used steel with CFRCs, the weight of automobile body system can be reduced over 20 %, equivalent to 12-16% increase in fuel economy (DOE). The reduction of the processing time of CFRCs from hours to minutes could lower their price more than half. The use of vitrimers as the matrix of CFRCs could extend their shelf life to infinite. Finally, 100% closed-loop recycling of end-of-life CFRCs can be achieved to produce similar value products, further reducing the overall cost of CFRCs.