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### Development of porous composite filament for additive manufacturing of lightweight components

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Xianbo Xu

*New York University, USA*

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TANDON SCHOOL  
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Composite Materials  
&  
Mechanics Laboratory

*Department of Micro and Nano Composites*

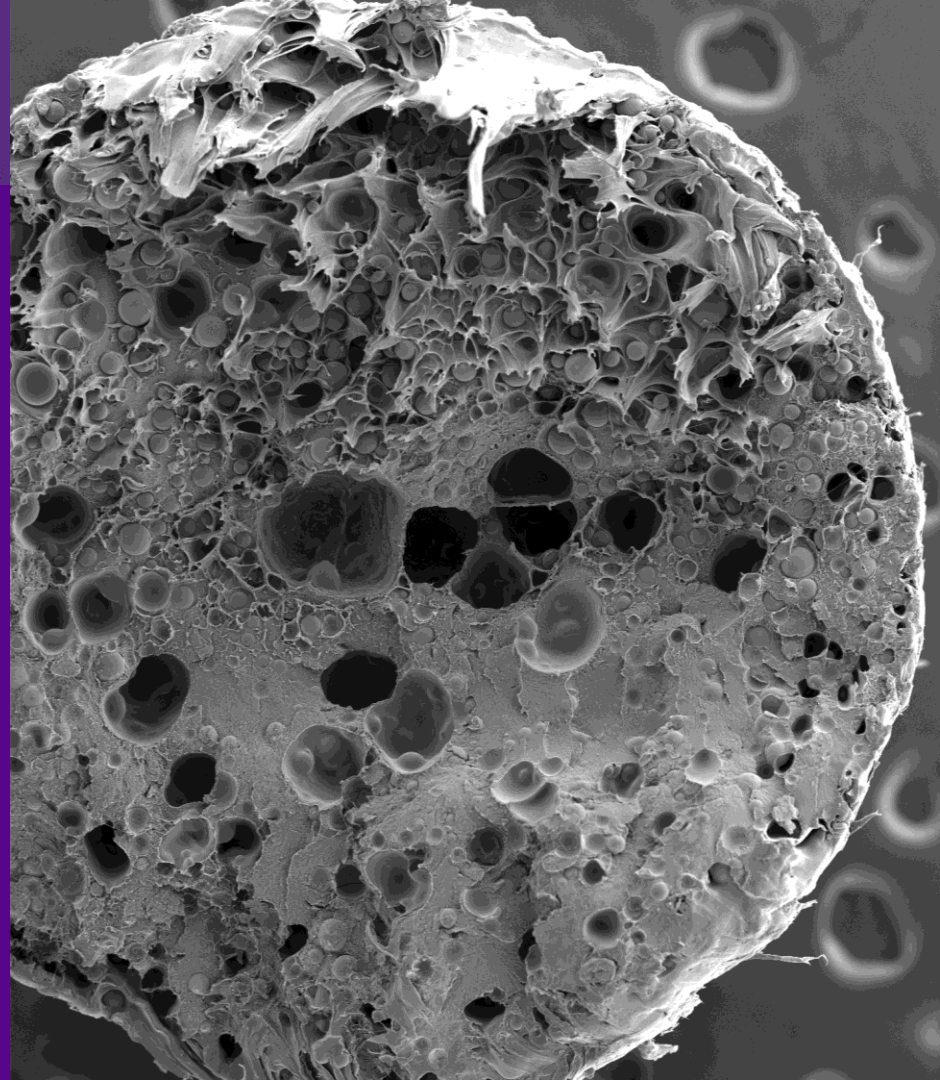
# Development of porous composite filament for additive manufacturing of lightweight components

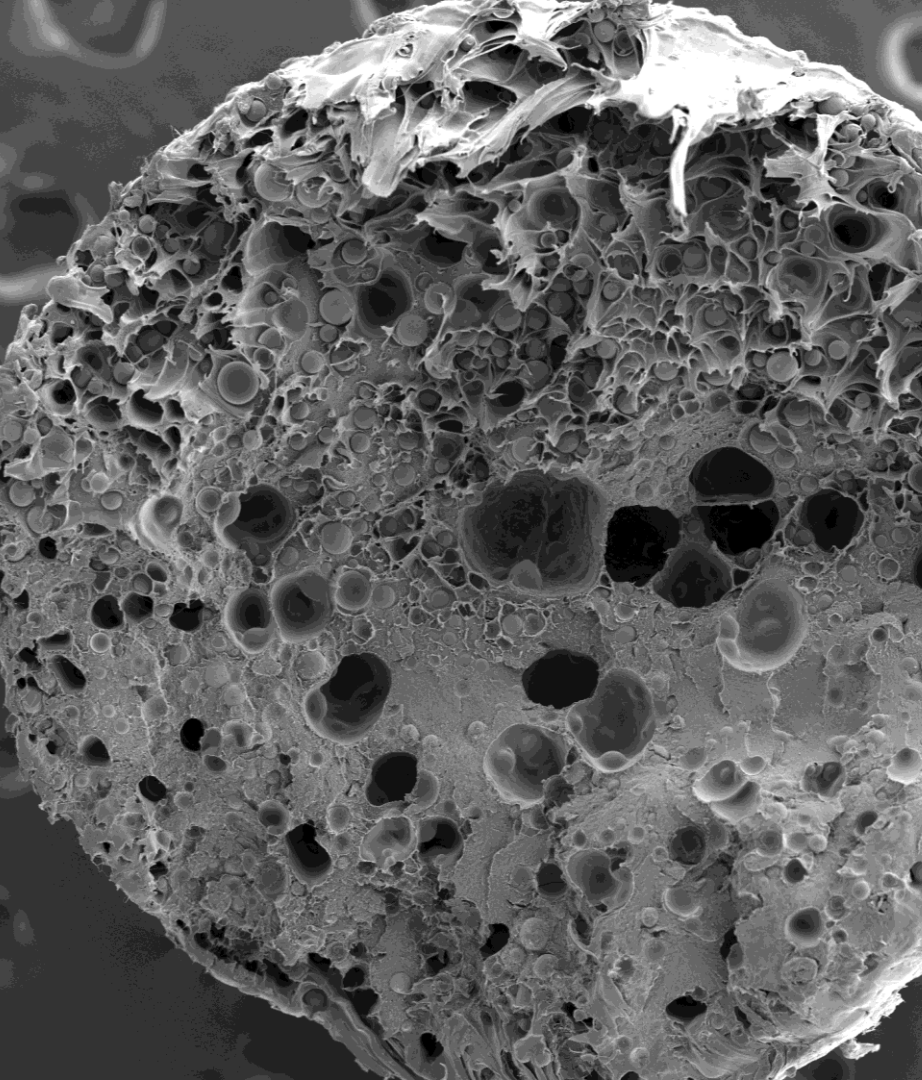
**Nikhil Gupta and Xianbo Xu**

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New York University  
Tandon School of Engineering  
Brooklyn, NY 11201 USA**

**Email: [ngupta@nyu.edu](mailto:ngupta@nyu.edu)**

03/10/2020





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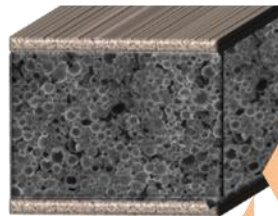
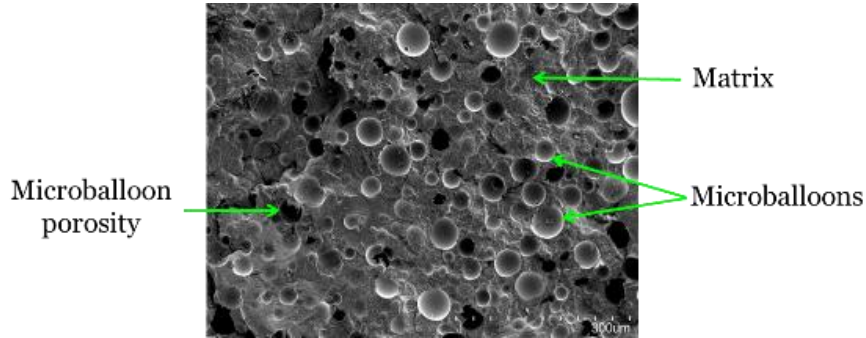
Composite Materials  
&  
Mechanics Laboratory  
Powered by Micro and Nano Computer

## Motivation

- **Pressure exerted during extrusion process is a major challenge in developing porous filaments**
- **The current practices of filament manufacture and 3D printing parameter optimization are mostly empirical**
- **DOE is used to test a number of combinations of processing parameters to find the optimum set**
- **Filament manufacture and 3D printing are extrusion processes that can be modeled**



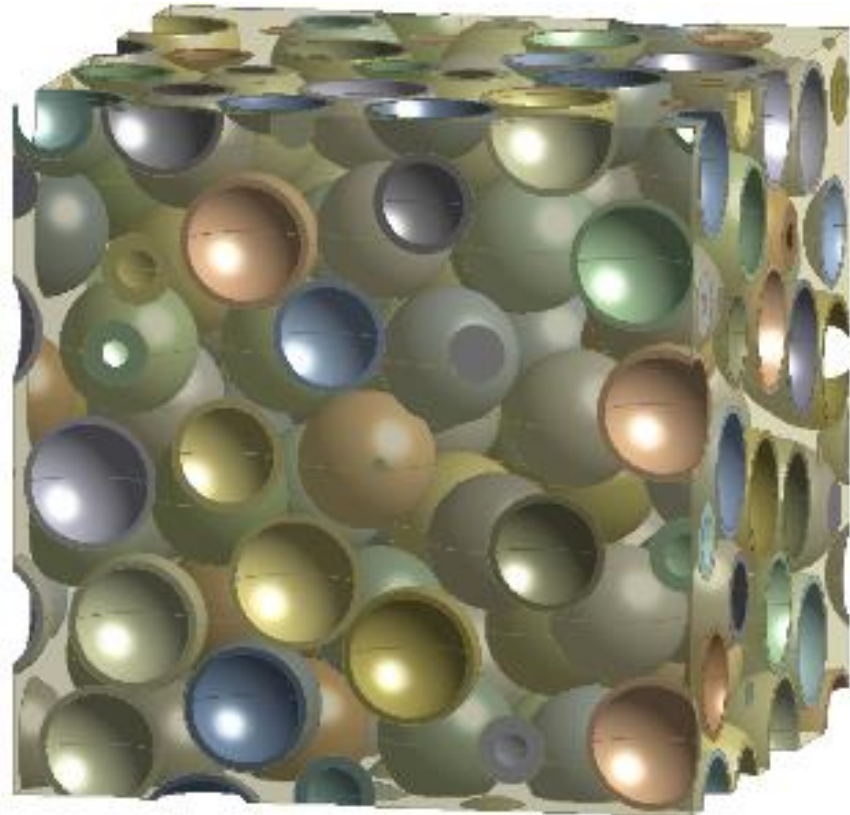
- Syntactic foams are composite materials synthesized by filling a metal, polymer, or ceramic matrix with hollow particles
- Core material in sandwich structures



- Core in sandwich composites

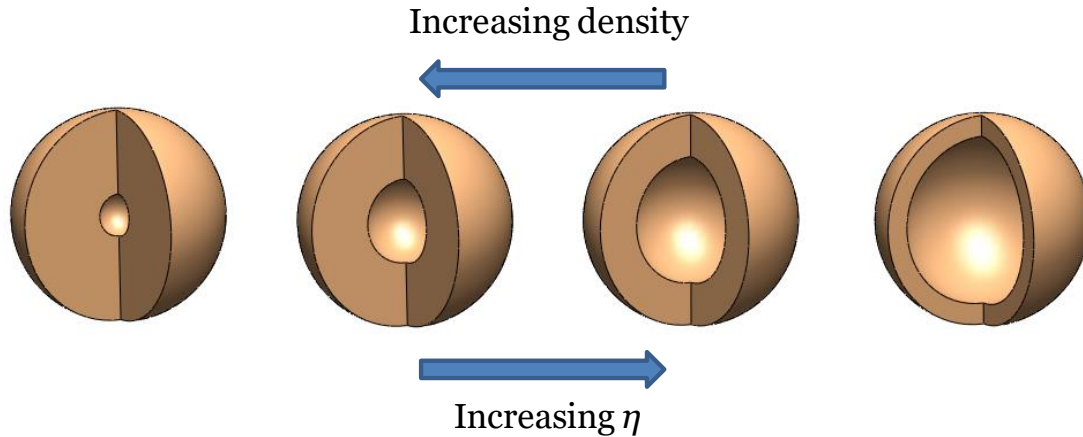
Syntactic foam core

Fiber reinforced face sheets

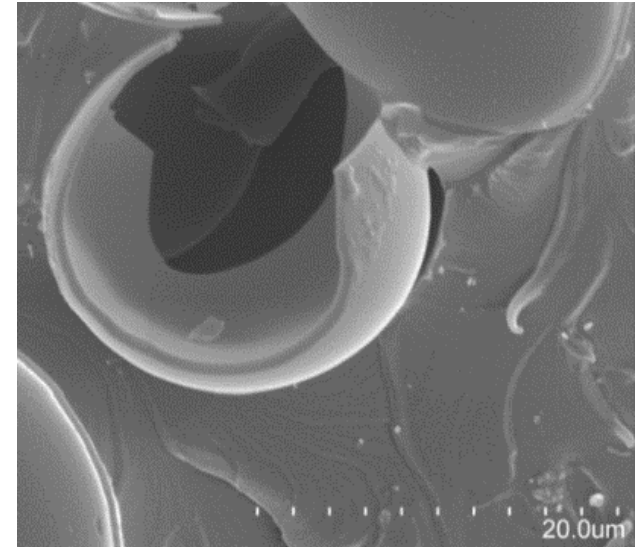


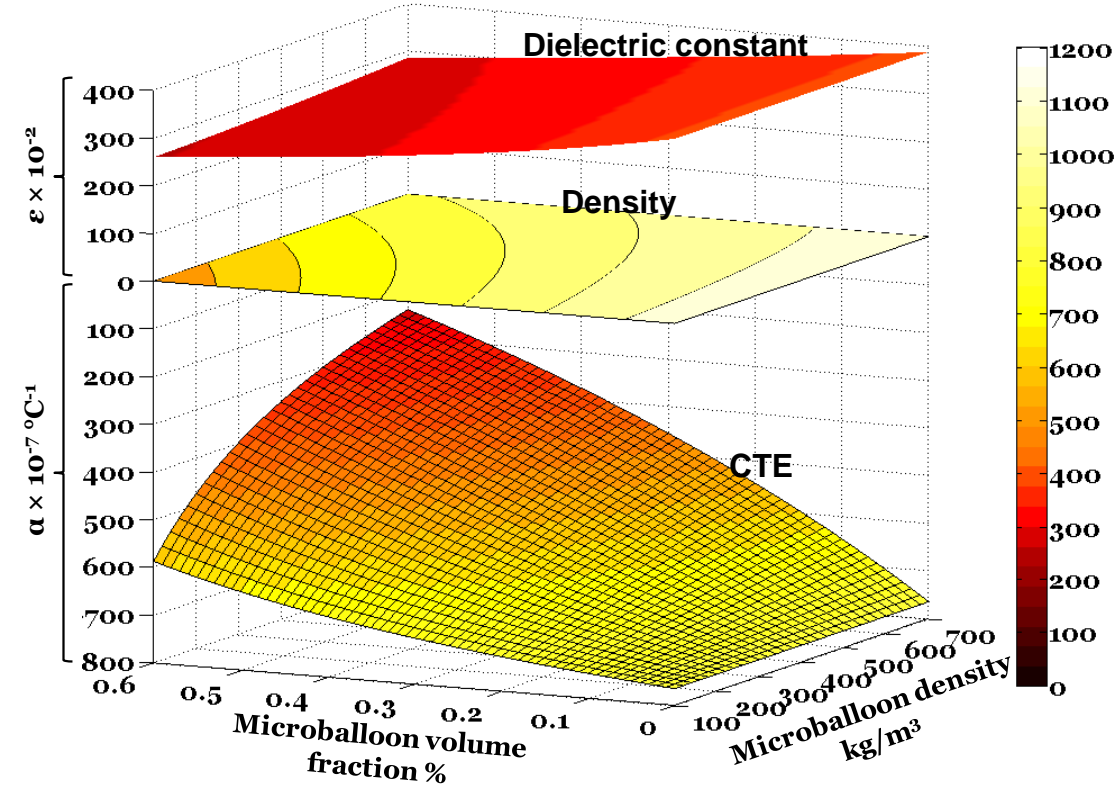
- **Advantages of syntactic foam:**

- Wall thickness and volume fraction of hollow particles can be independently tailored to conduct multi-criteria optimization
- This ability also allows tailoring their properties over a wide range

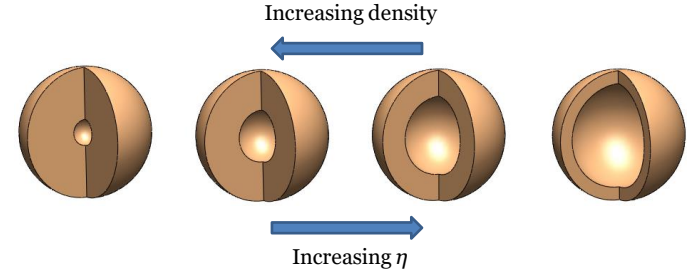


$$\text{Radius ratio } \eta = \frac{r}{r_0}$$



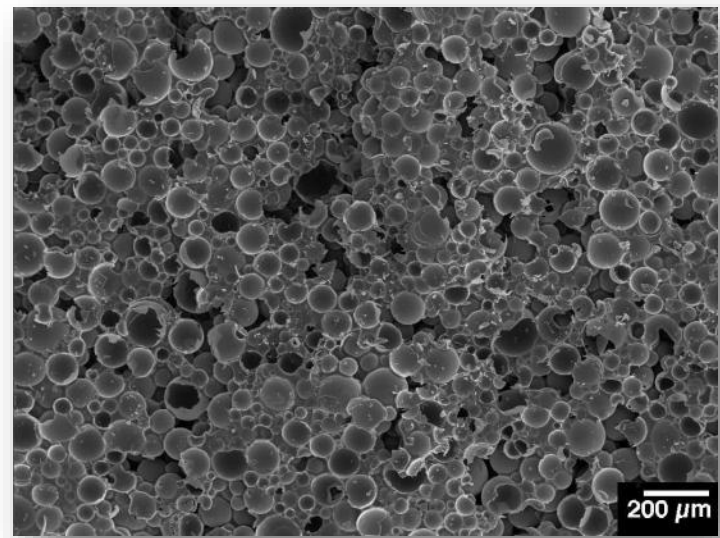


- Multi-criteria optimization of syntactic foams by selecting hollow particle
  - Wall thickness
  - Volume fraction
  - Material



- Reinforcing polymer matrix by nanoparticles

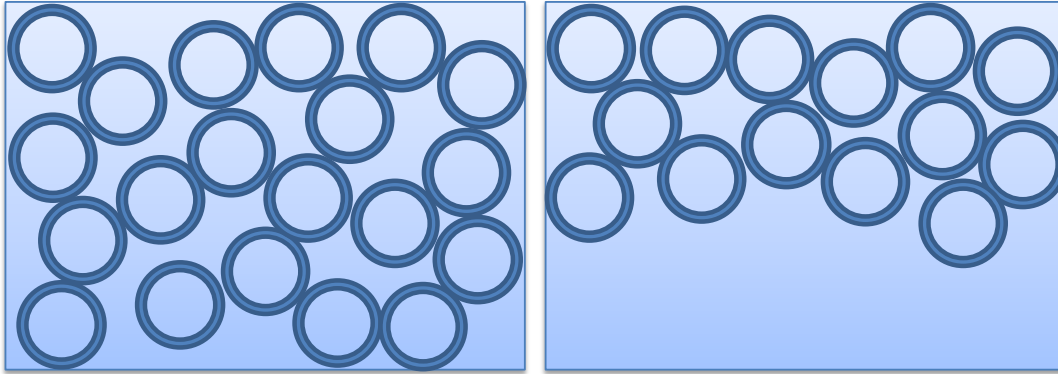




- Mixing and casting, In-situ molding, Injection molding, Compression molding



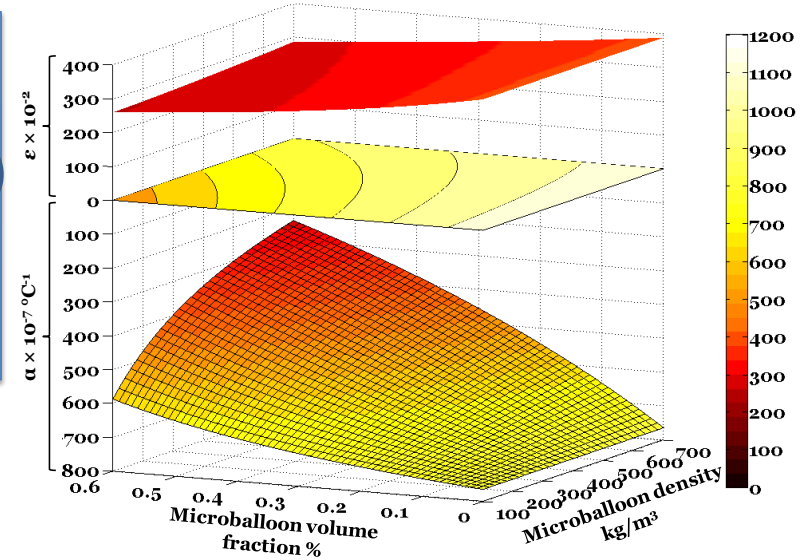
## Motivation 1



Segregation of lightweight hollow particles if the volume fraction is less than 35%

Casting methods are difficult to use for such compositions

Infiltration methods require at least 50 vol.% particles



The desired compositions may contain less than 30% microballoons based on the models



## Motivation 2



Cracking of syntactic foams at joints in deep sea environment

4000+ m depth UUVs would benefit from elimination of joints

4/4/2020



### Nose:

- Vehicle Sensors
  - Pressure, Temp, Altitude, IMU
- Mission Processing
- Power Distribution
- OLED Display

### Tail:

- Propulsion
  - 350W External Motor (3000m rated)
- Vehicle Control
- Communications (WiFi Standard)
- Emergency Systems
  - Dropweight / LED Strobes
- GPS



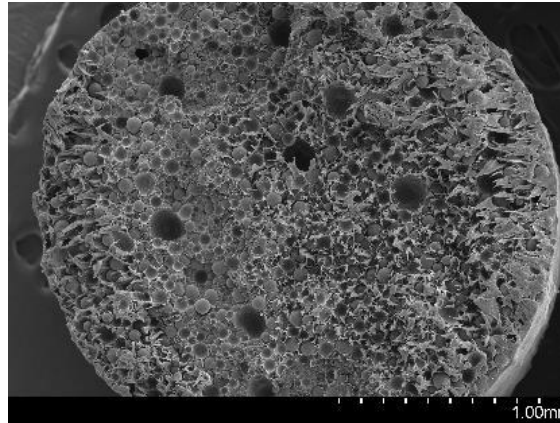
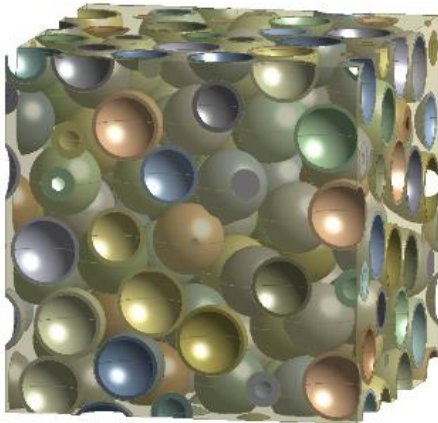
### Midbody:

- Power
- Payload
  - Dry or Flooded Volumes

Marine Technology News website

## • Challenges

- Use of industrial thermoplastics –HDPE, PP
- Manufacture filaments for use in commercial 3D printers without any hardware modification
- Close control over porosity in the printed part – control over particle breakage



## Hollow particles

### 1. Fly-ash cenospheres

- By-product of coal fired power plants
- Very inexpensive
- True particle density: 0.85 g/cc
- Defective and irregularly shaped

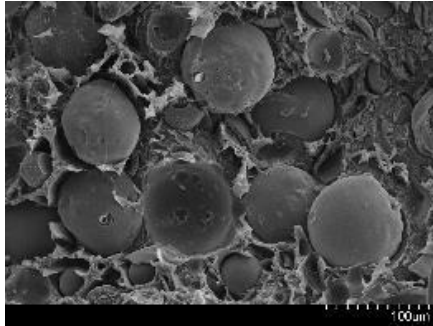


Table I. Chemical analysis details of cenospheres

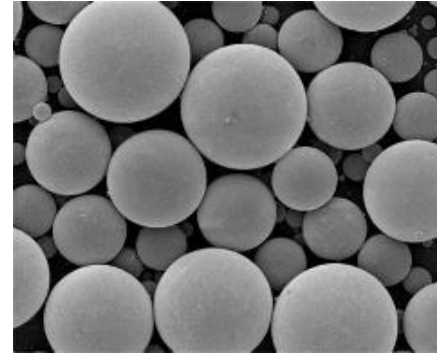
Chemical analysis	
SiO <sub>2</sub>	52–62%
Al <sub>2</sub> O <sub>3</sub>	32–36%
CaO	0.1–0.5%
Fe <sub>2</sub> O <sub>3</sub>	1–3%
TiO <sub>2</sub>	0.8–1.3%
MgO	1–2.5%
Na <sub>2</sub> O	0.2–0.6%
K <sub>2</sub> O	1.2–3.2%

### Matrix material: HDPE

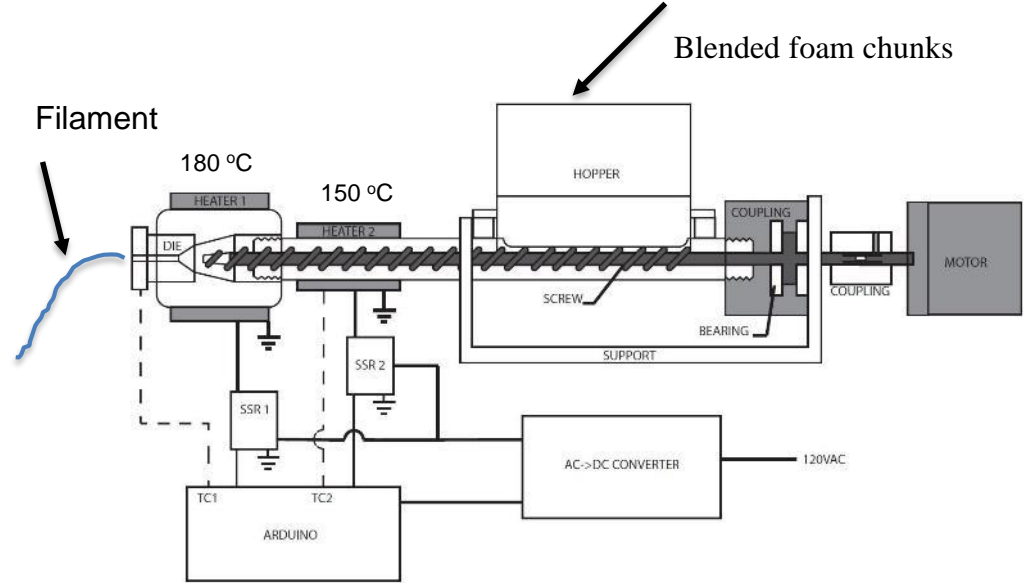
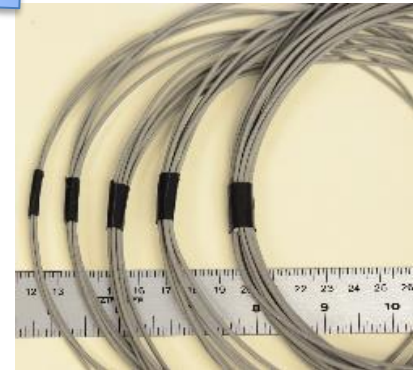
- Reliance polymers, Mumbai, India
- Density: 1.02 g/cc
- Molecular weight 97,500 g/mol

### 2. Glass microballoon

- Trelleborg Offshore Ltd
- Borosilicate glass
- Tighter particle size distribution
- Spherical shape



GMB type	Average GMB diameter (μm)	True particle density (kg/m <sup>3</sup> )
SID200	53	200
SID270	50	270
SID350	45	350

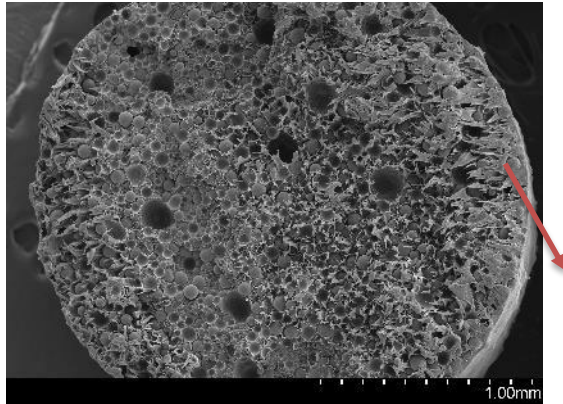


- Parameters that influence the filament quality
  - Extrusion temperature
  - Screw RPM
  - **Filament tension**

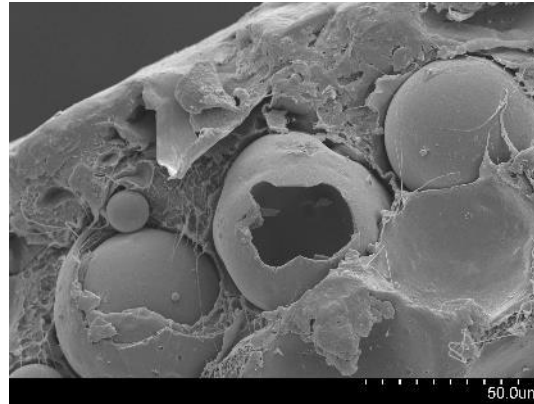


## Material recycling

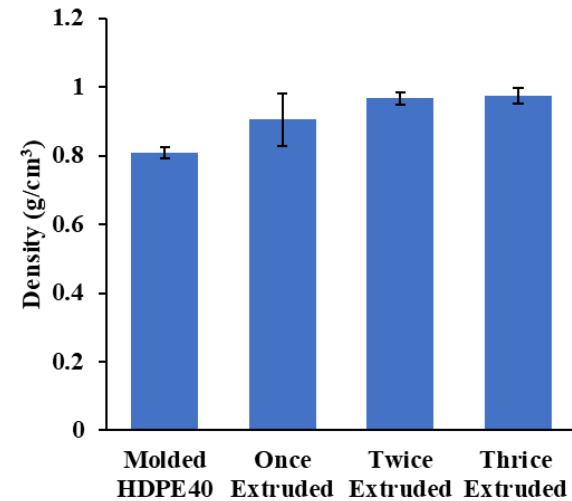
- As cenosphere break during extrusion process and fill the porosity, density of the filaments change with number of extrusion process
- The density stabilizes after extrusion and recycling for 3 times



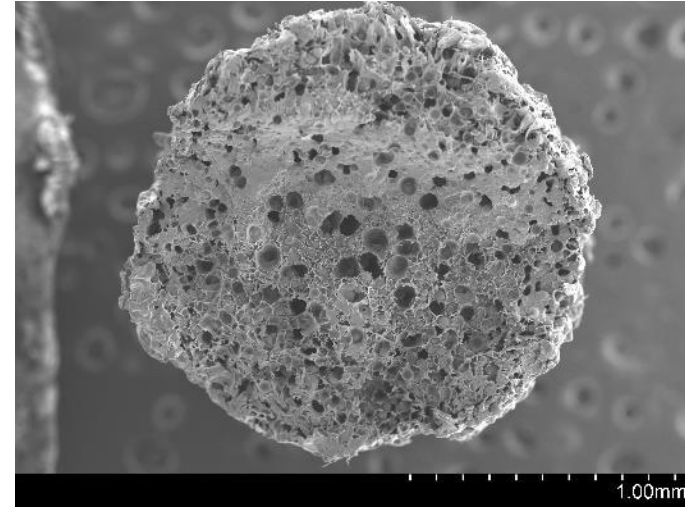
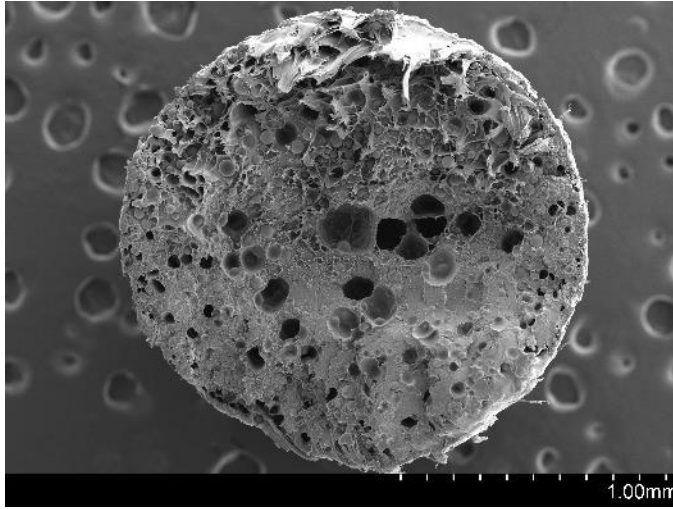
HDPE40-2X Filament



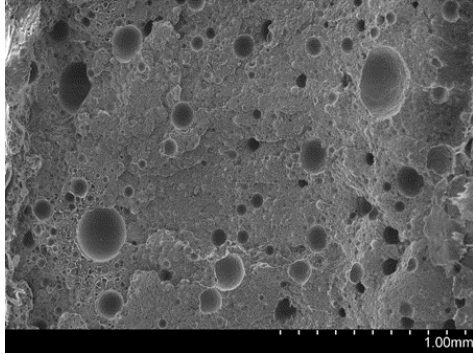
Broken particle filled with matrix



# HDPE/GMB filament



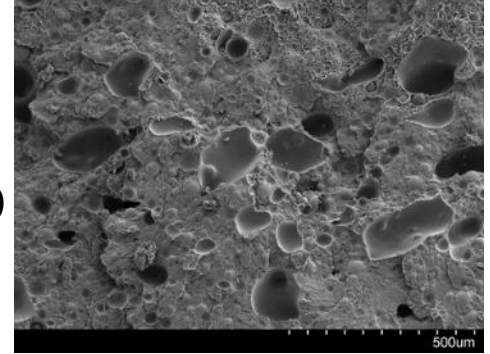
Material	Density (g/cc)	Air voids	
		Based on theoretical density	Based on cenosphere breakage
H350-20	0.778	11.6	8.7
H350-40	0.709	8.5	4.4



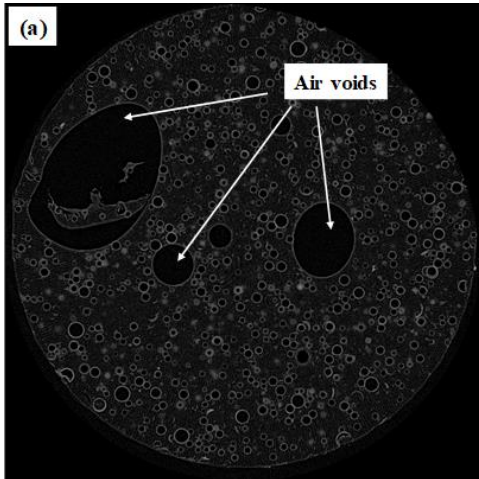
3DP H350-20

CT scan reveals:

- The porosity changes its shape and size when the volume fraction changes
- More exposure is achieved for H350-40 with same parameters were used

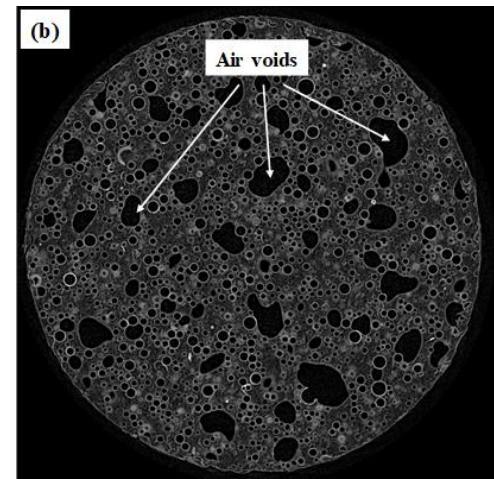


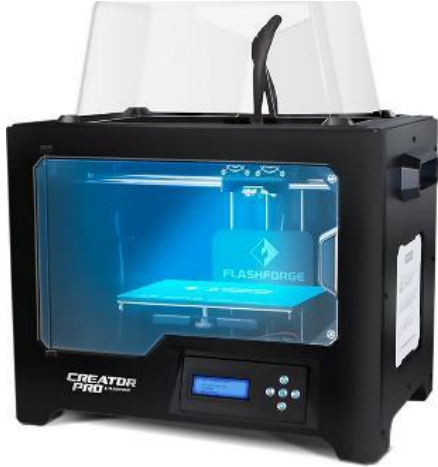
3DP H350-40



Limitation and challenges:

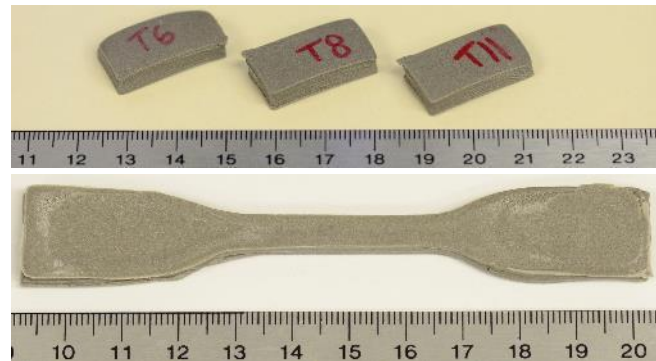
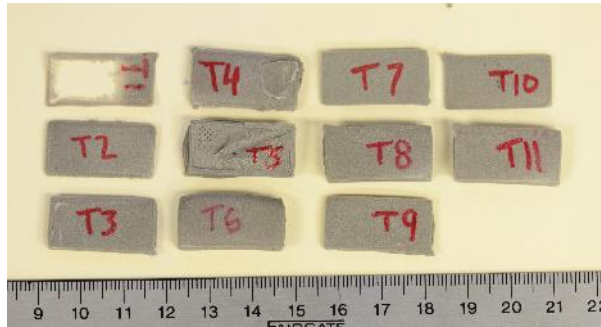
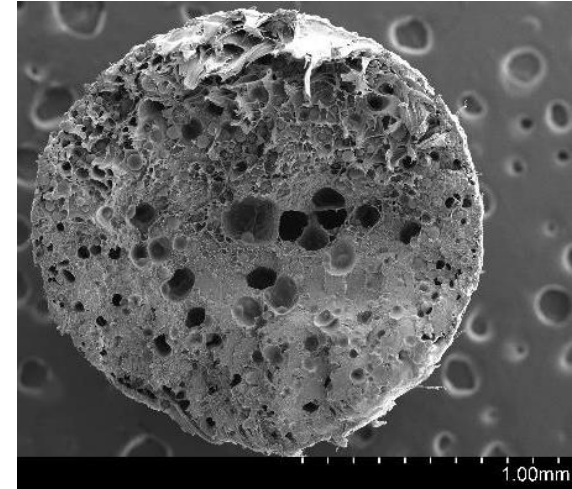
- HDPE has very low X-ray attenuation co-efficient
- Hard to distinguish from air and voids
- The thickness of microballoon walls are very thin





Flashforge Creator Pro

- Specimen geometry ASTM D638 Type IV
- Flashforge CreatorPro 3D printer
- The filament porosity does not directly translate into syntactic foam porosity

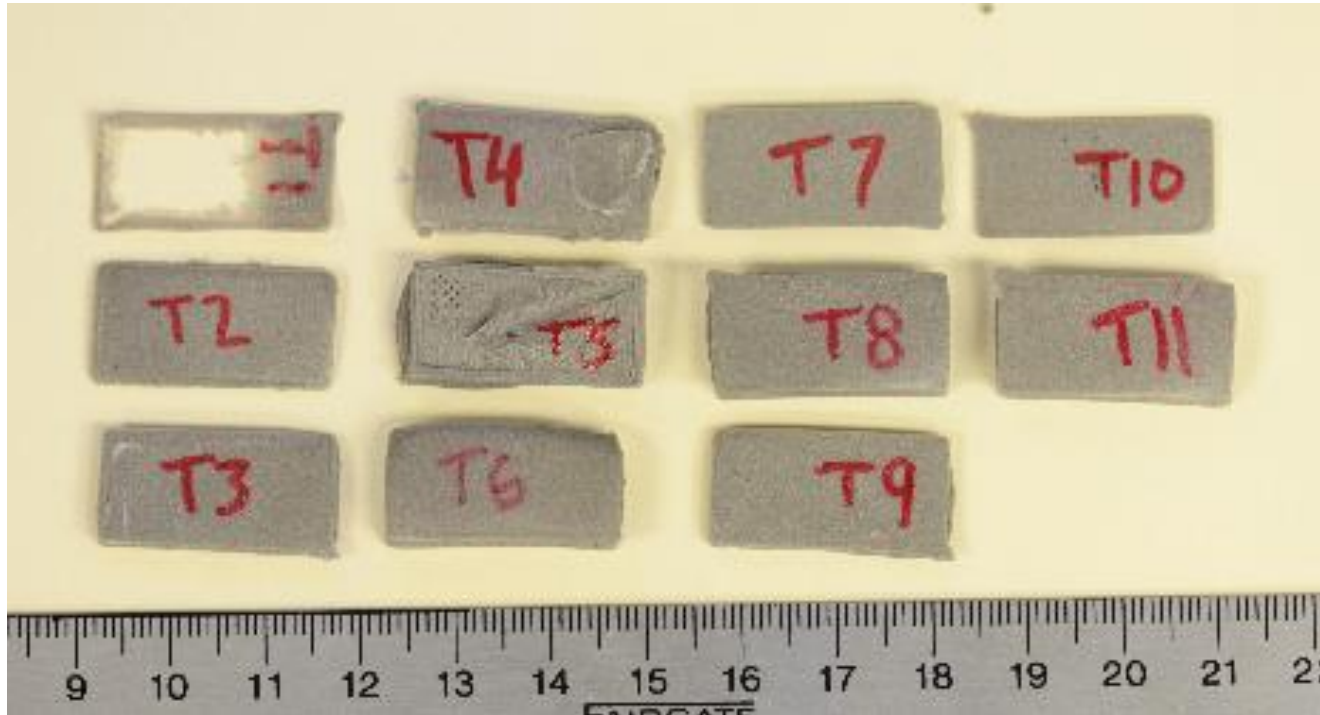




# AM using syntactic foams

Technique	Material	Elastic modulus (MPa)	Density (g/cm <sup>3</sup> )	Yield strength (MPa)	Ultimate tensile strength (MPa)
3D printed	3DP HDPE40-2X	1337±109	0.950	7.0±0.4	10.1±0.1
3DP-Recycled	3DP HDPE40-3X	1569±143	0.959	7.4±0.5	10.7±0.2
Injection molded	PIM HDPE40	723±27	1.0078	-	12.1±0.44

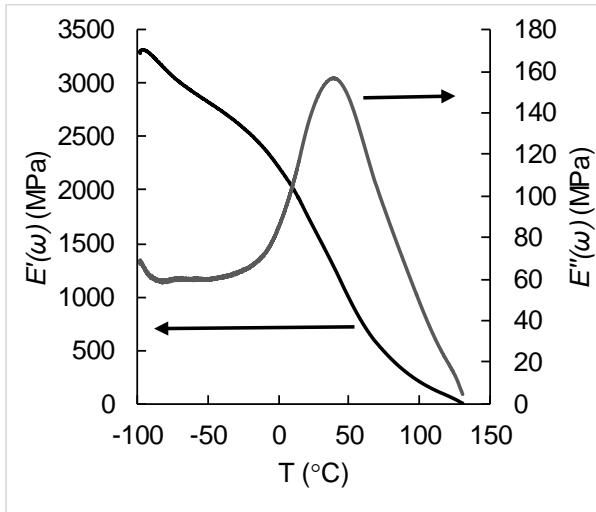




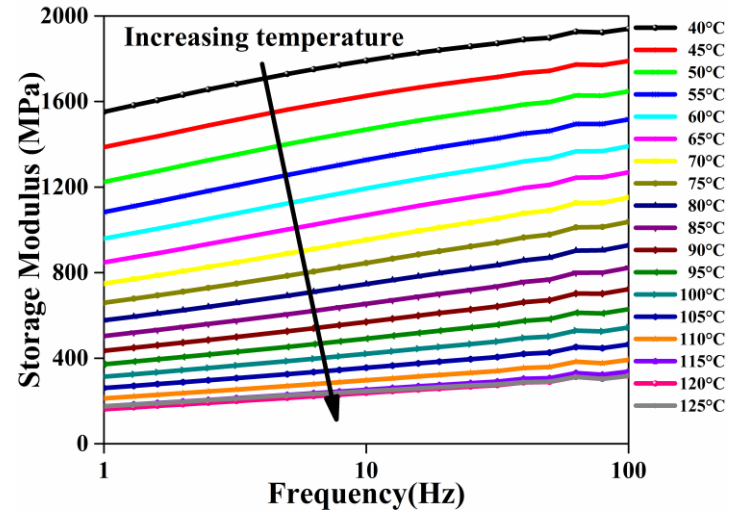
Sinusoidal load cycle is applied to obtain the frequency domain viscoelastic properties

Storage modulus ( $E'$ ), loss modulus ( $E''$ ), damping parameter ( $\tan \delta$ )

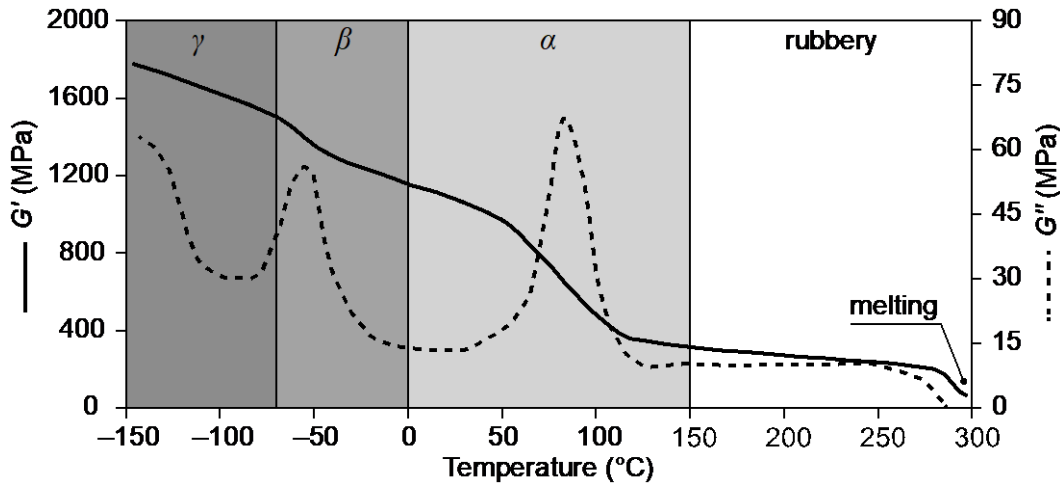
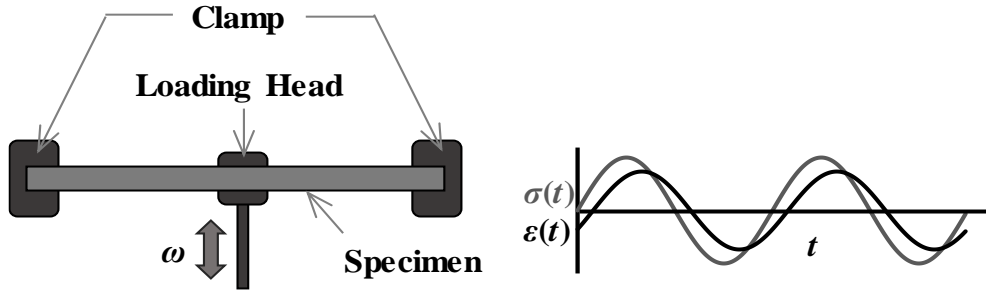
$$E^* = E' + iE''$$



A representative DMA temperature sweep.



A representative set of DMA frequency sweeps for 5.0 wt.% CNF/HDPE composite





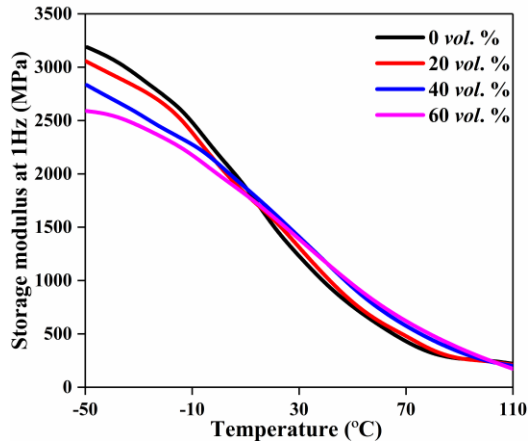
Sinusoidal load cycle is applied to obtain the frequency domain viscoelastic properties

Storage modulus ( $E'$ ), loss modulus ( $E''$ ), damping parameter ( $\tan \delta$ )

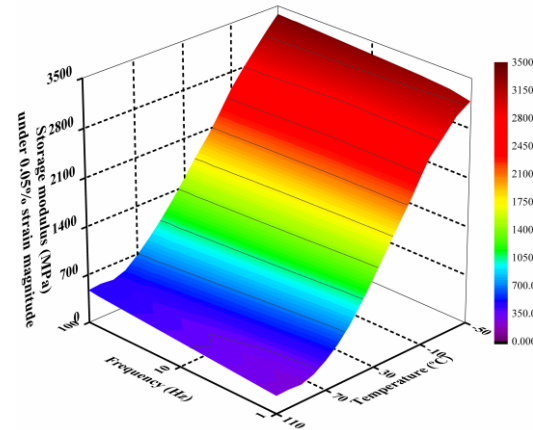
$$E^* = E' + iE''$$

Storage compliance ( $C'$ ), loss compliance ( $C''$ )

$$C^* = C' + i C'', \quad E^* \cdot C^* = 1$$



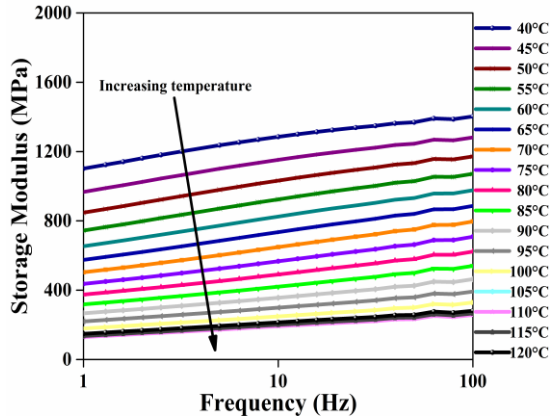
Storage modulus of various volume fraction GMB/HDPE syntactic foam at 1 Hz



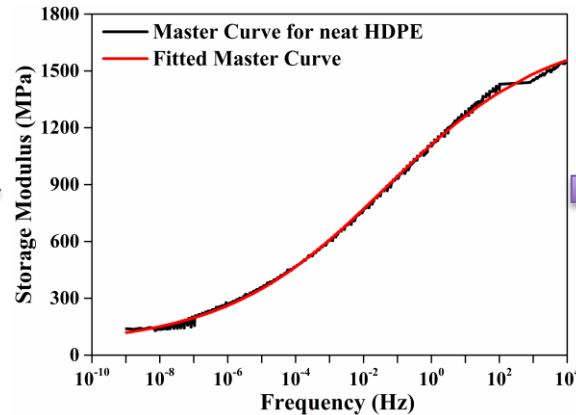
Response surface of storage modulus for 20 vol.% GMB/HDPE syntactic foam

## Transform $E'$ to $E(t)$

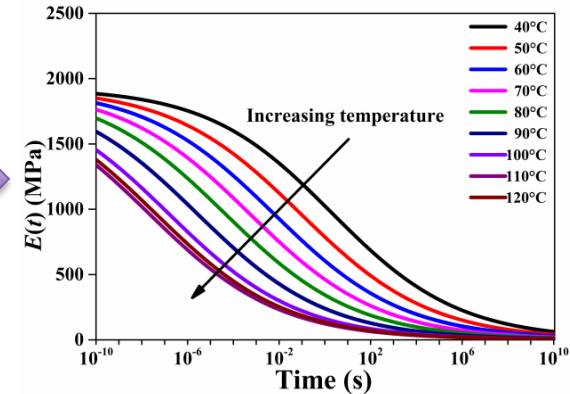
Step 1: The master relation can be transformed to time domain relaxation function  $E(t)$  using integral relation of viscoelasticity and time temperature superposition principle.



A representative set of DMA frequency sweeps for neat HDPE.

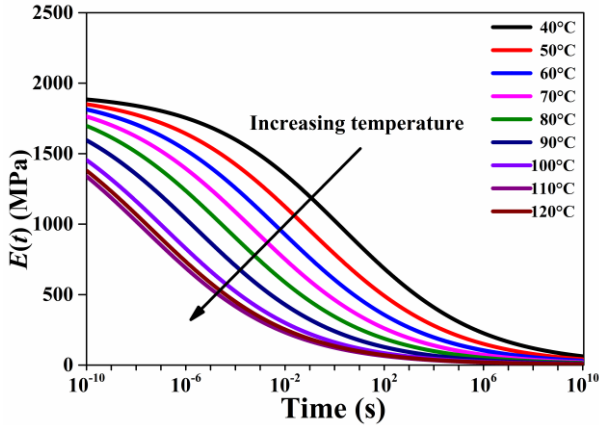


A representative fitting result at 40 °C of neat HDPE

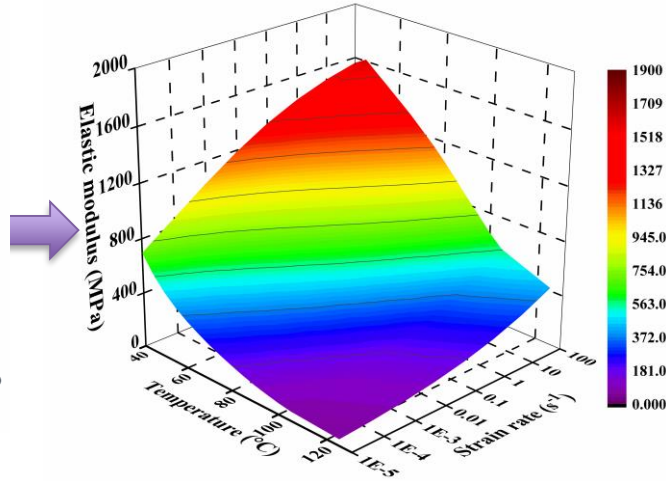


Relaxation function for neat HDPE resin

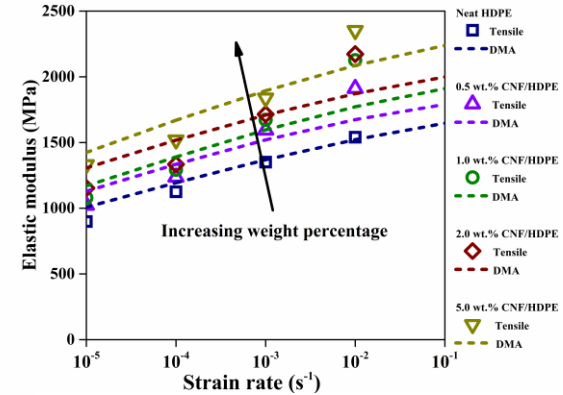
- Step 2:  $E(t)$  can be used to predict stress response with certain strain history and extract elastic modulus.



Relaxation function for neat HDPE resin

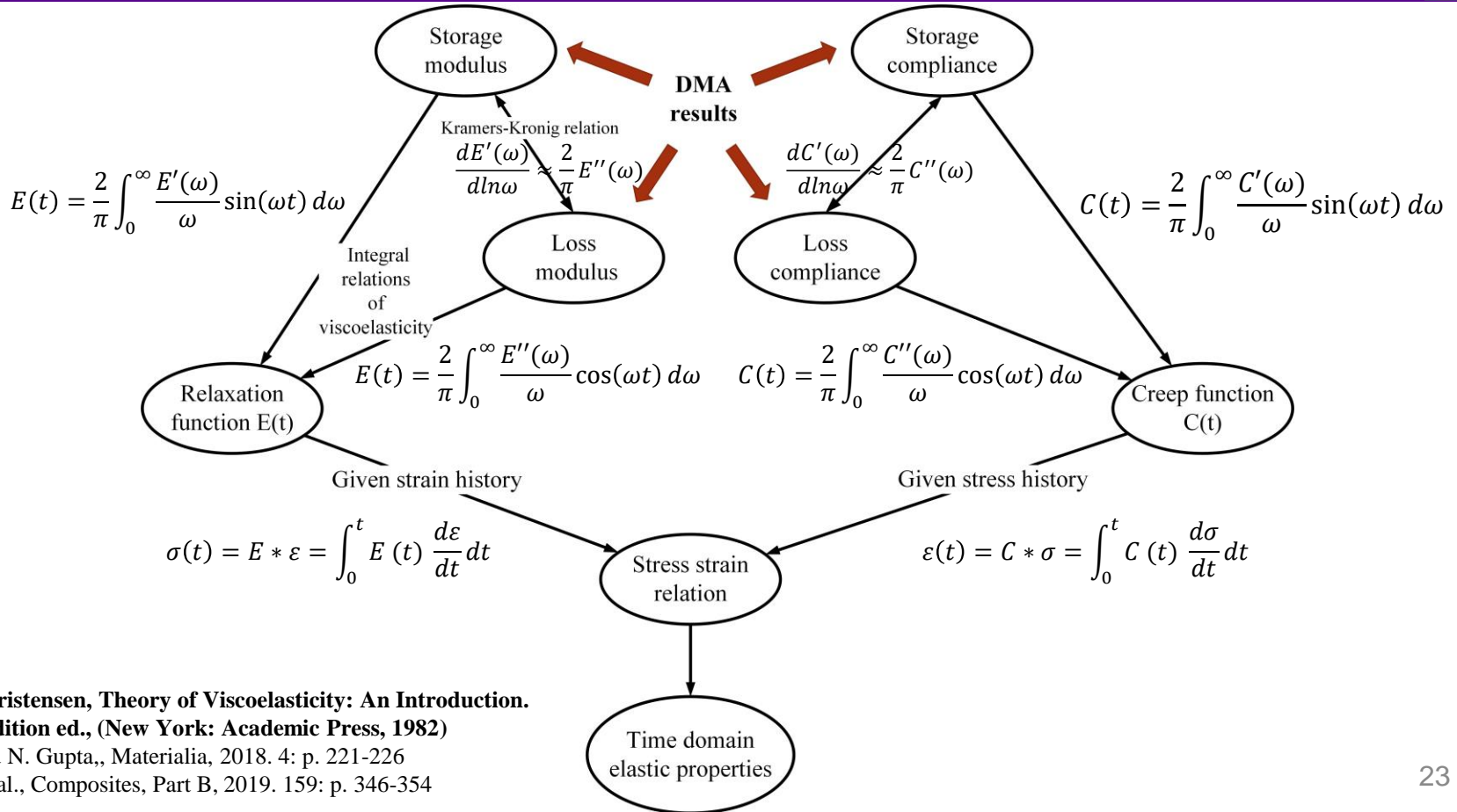


3d plot of Elastic modulus predicted by DMA with respect to temperature and strain rate for neat HDPE resin



Comparison of elastic modulus for CNF/HDPE composite material with different weight percentage at room temperature

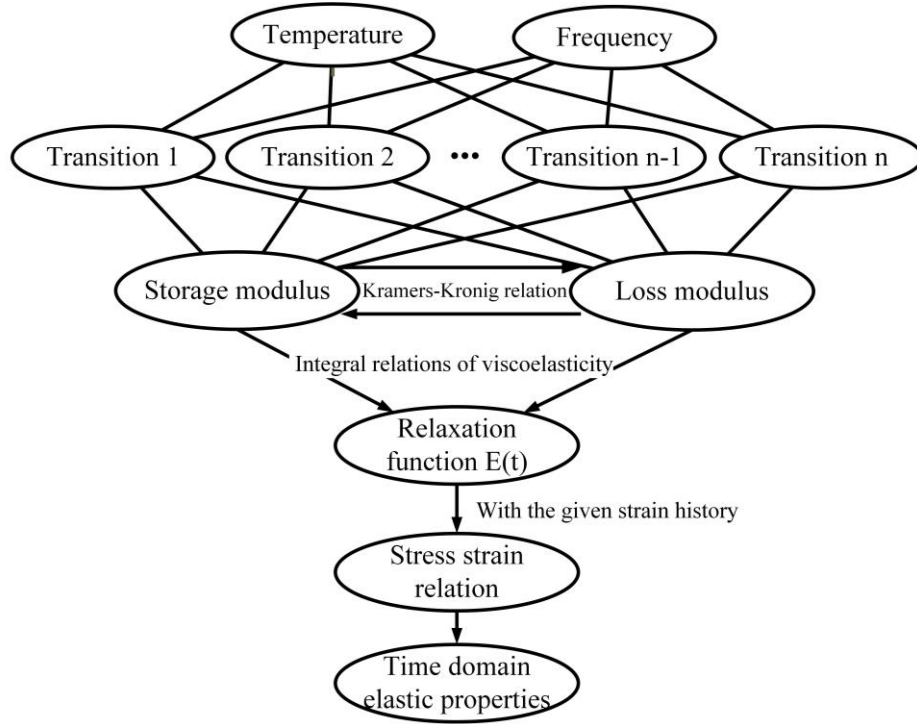
# Viscoelastic mechanics system



R. M. Christensen, **Theory of Viscoelasticity: An Introduction**.  
 Second edition ed., (New York: Academic Press, 1982)  
 X. Xu and N. Gupta., *Materialia*, 2018. 4: p. 221-226  
 X. Xu, et al., *Composites, Part B*, 2019. 159: p. 346-354



# Artificial neural network approach



Flow chat of artificial neural network modeling scheme

Artificial neural network is used to build the master relation for complex material systems.

To improve generalization, the  $L^2$  regularization, or so called ridge regression, is used as the regularization term as

$$\tilde{F}(E'; \omega, T) = F(E'; \omega, T) + \alpha \Omega(\theta)$$

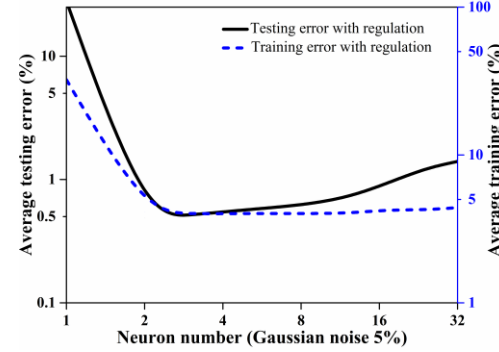
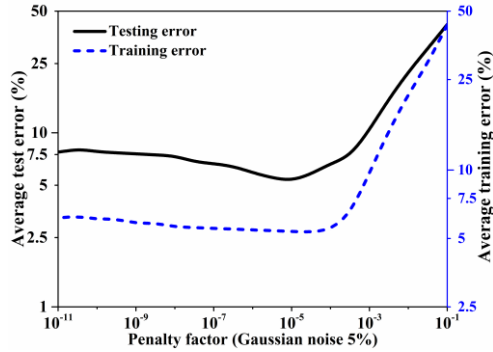
where

$$F(E'; \omega, T) = \frac{1}{N} \sum_{i=1}^N [(\ln \tilde{E}'(\omega, T) - \ln E'(\omega, T))]^2$$

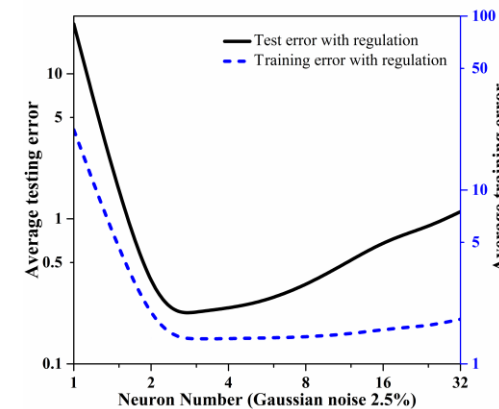
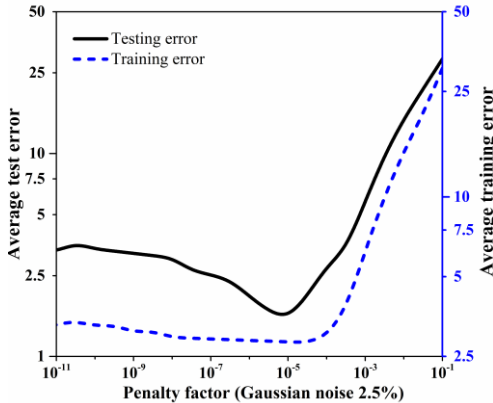
$$\text{or} = \frac{1}{N} \sum_{i=1}^N [E''(x_i) - \tilde{E}''(x_i)]^2$$

$$\Omega(\theta) = \frac{1}{n} \sum_{j=1}^n \theta_j^2$$

# Artificial neural network approach



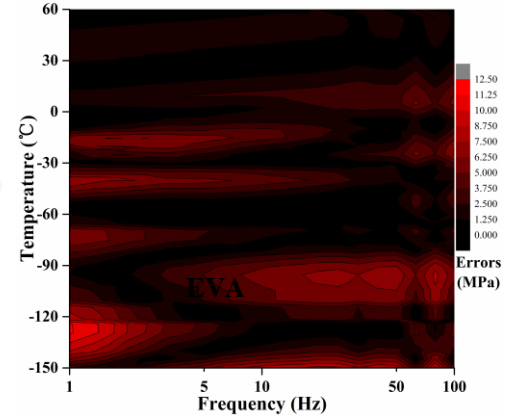
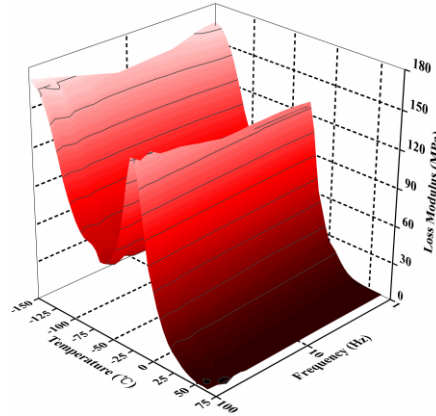
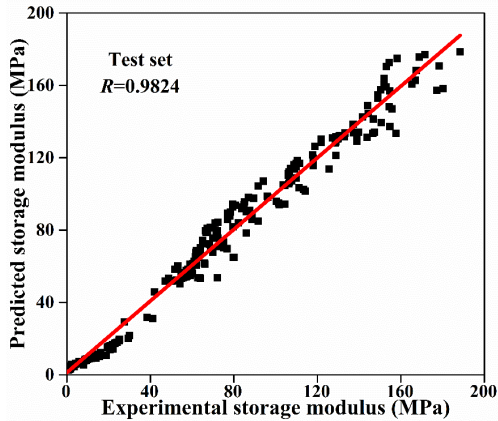
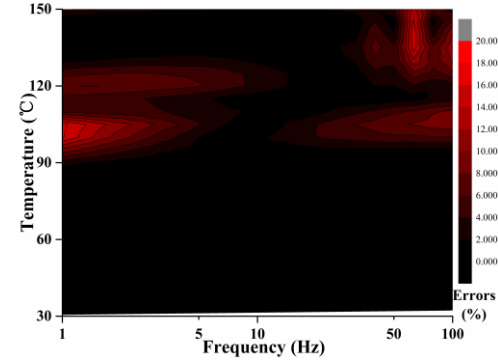
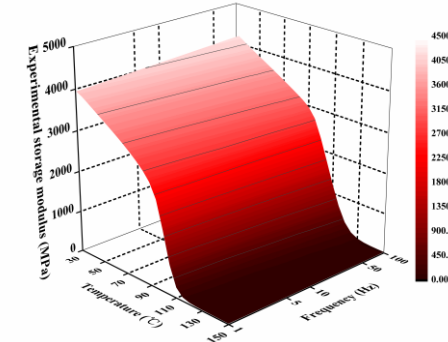
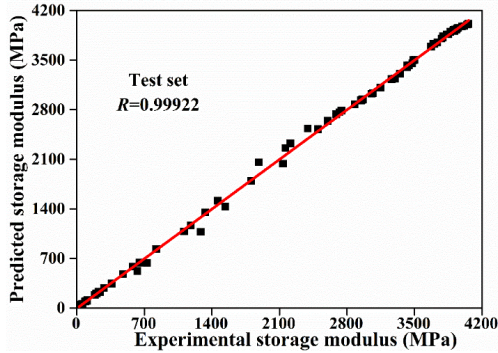
Training and test accuracy with respect to  $L^2$  regulation factor and neuron number for storage modulus using feed forward neural network



Training and test accuracy with respect to  $L^2$  regulation factor and neuron number for loss modulus using radial basis neural network

Model tuning

# Artificial neural network approach



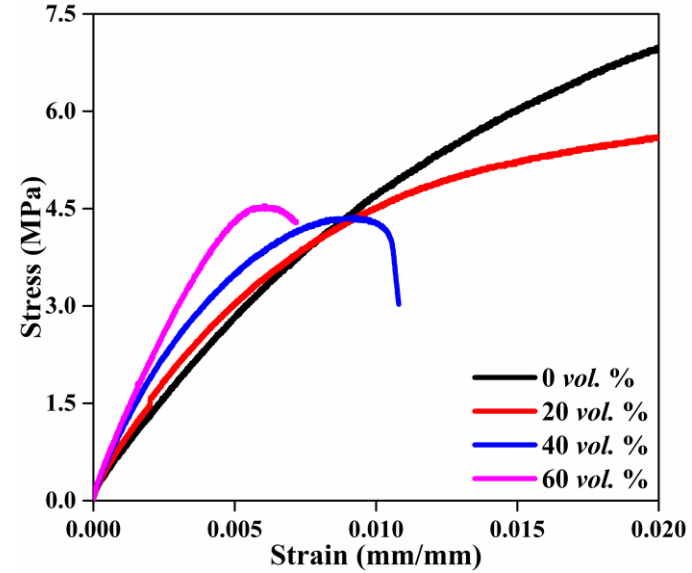
The Pearson's correlation coefficient for test set using feed forward and Radial basis neural network from storage and loss modulus

3D response surfaces for (a) storage modulus and (b) loss modulus of graphene reinforced composites

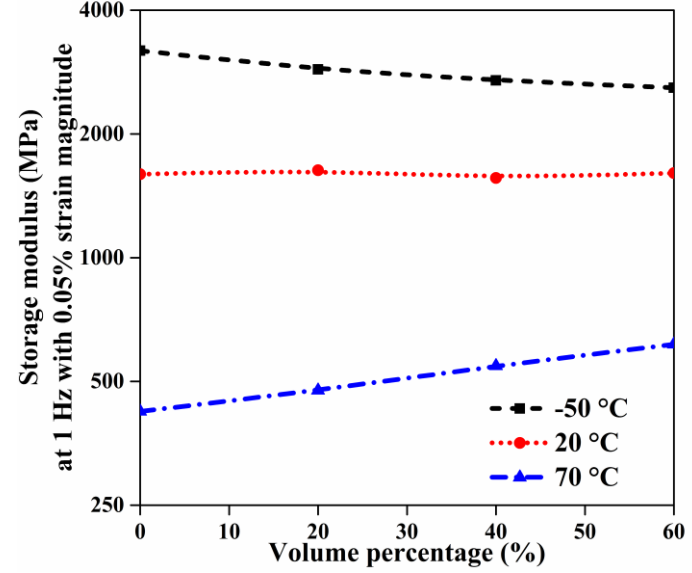
Map of the fitting error with respect to temperature and frequency.

- Many materials transform from ductile to brittle material as the composition changes

- The materials shows strong nonlinear behavior due to the coupling effects of strain, temperature, volume % and strain.

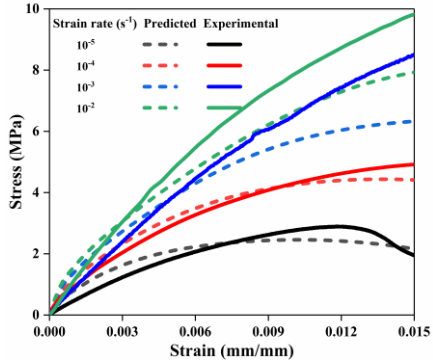


Stress strain curve of HDPE syntactic foam at sample strain rate ( $10^{-5}/s$ ) and temperature ( $30^{\circ}C$ ) with four different volume percentage.

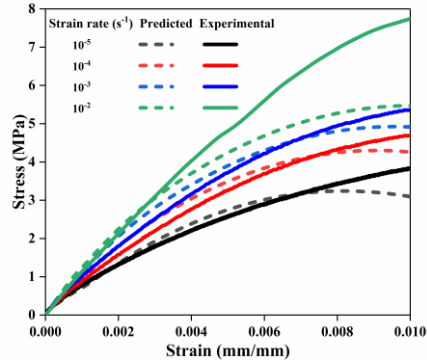


Storage modulus of HDPE syntactic foam at same frequency, strain magnitude, but under different temperatures

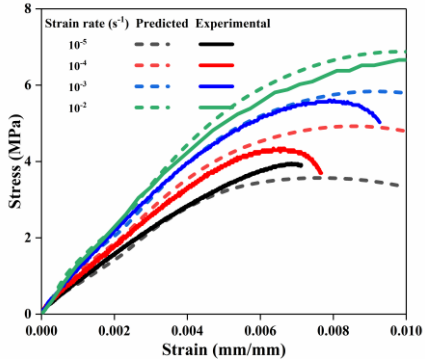




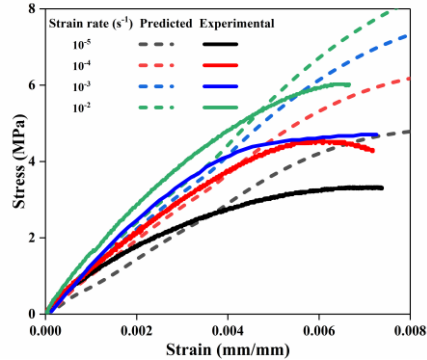
(a)



(b)

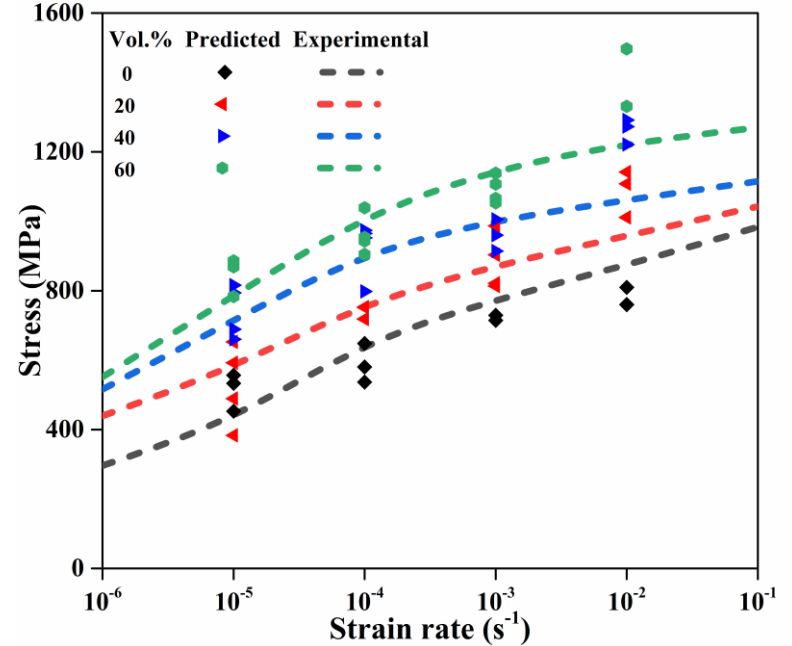


(c)

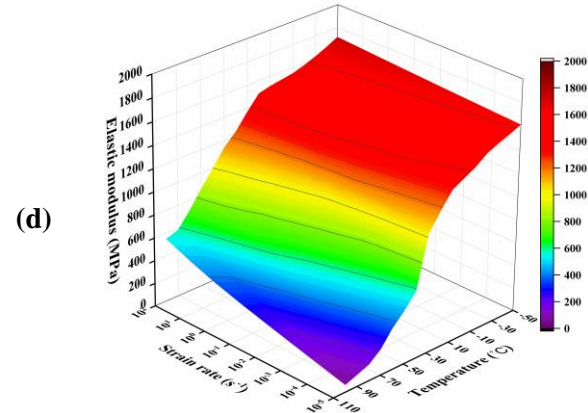
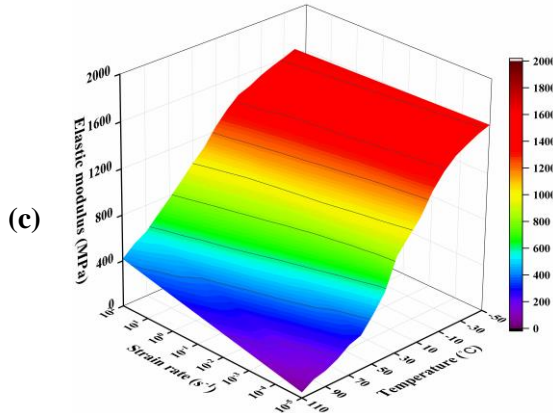
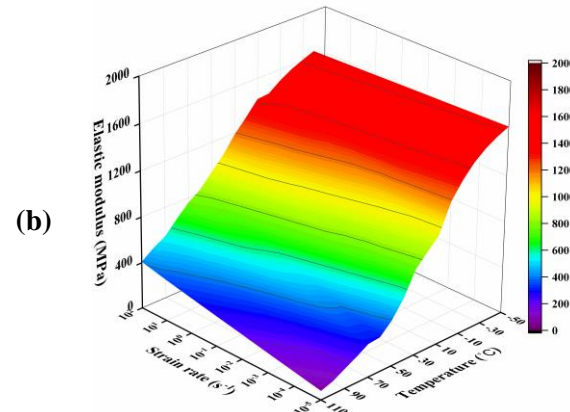
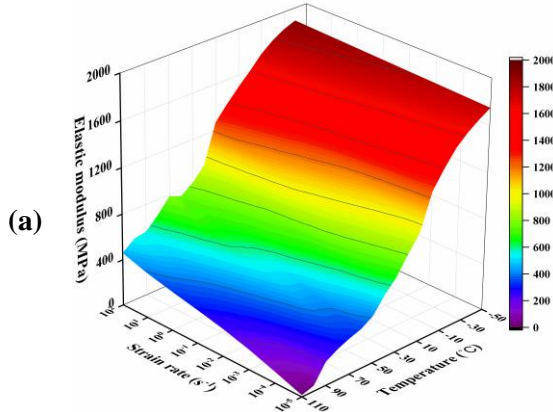


(d)

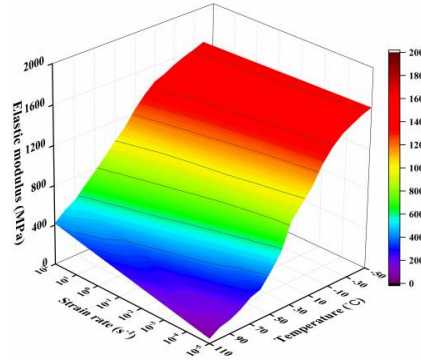
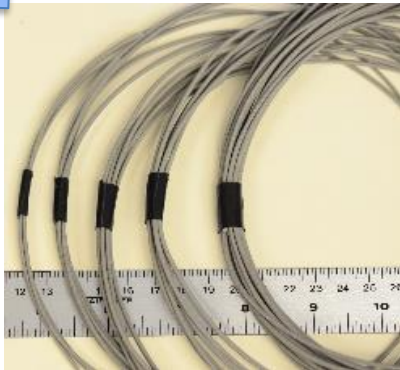
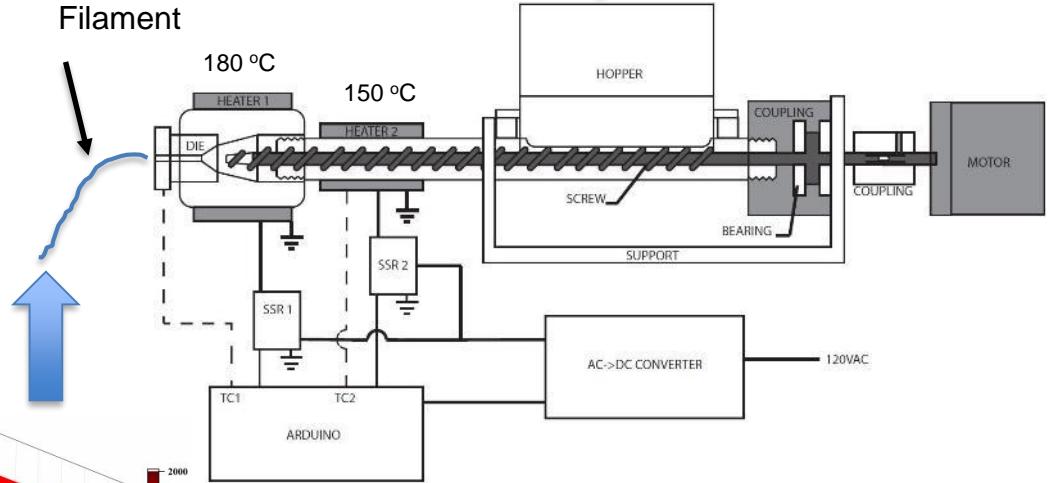
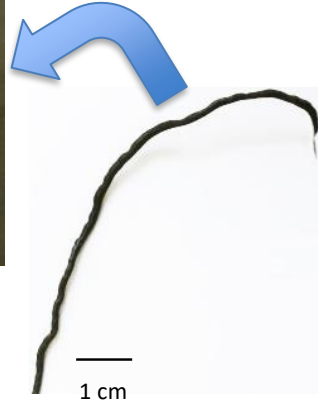
Verification of stress strain prediction for (a) pure HDPE (b) 20 vol.% (c) 40 vol.% (d) 60 vol.% HDPE syntactic foam



Verification of elastic modulus prediction for (a) pure HDPE (b) 20 vol.% (c) 40 vol.% (d) 60 vol.% HDPE syntactic foam



Elastic modulus prediction for (a) pure HDPE (b) 20 vol.% (c) 40 vol.% (d) 60 vol.% HDPE syntactic foam under wide range of temperature and strain rate





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*Research in Micro and Nano Composites*

# Thanks!

