# Challenges and strategies for particle dispersion and process-induced microstructural design in functional particulate composites

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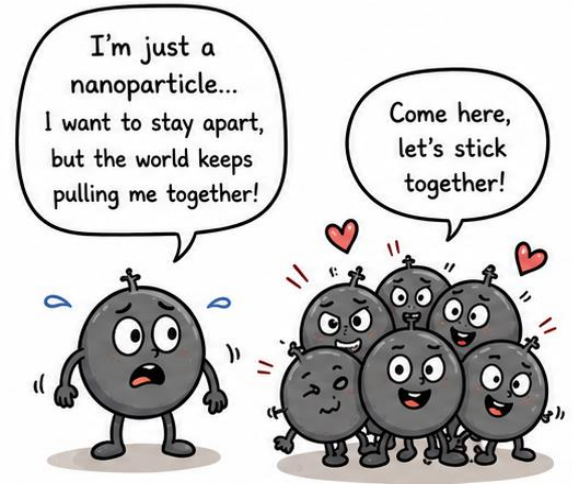
# The challenge of (nano)particle integration in particulate composites

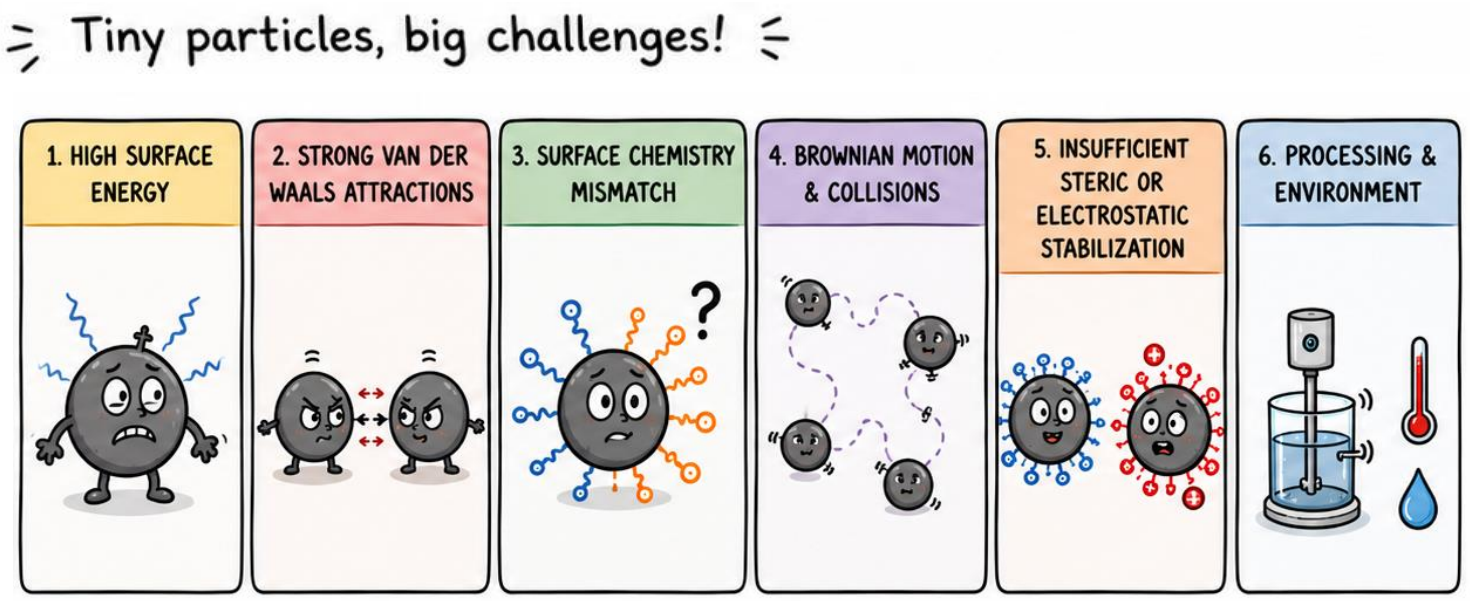
## Size and shape do matter

Why are smaller and 2D-like objects more challenging?

**Attractive particle surface interactions**





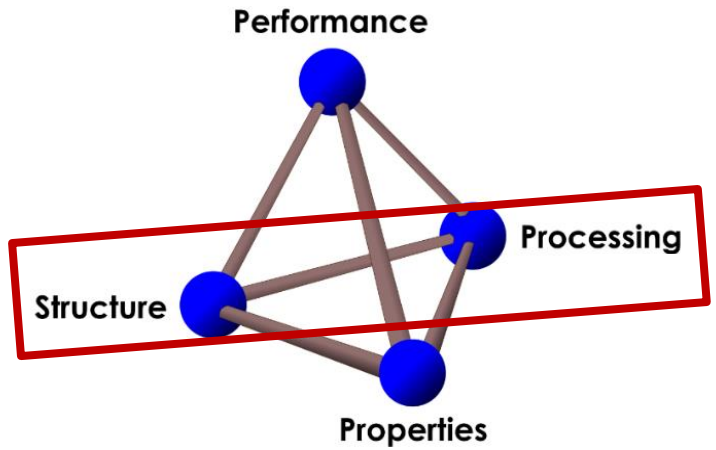

OpenAI, ChatGPT

# Promising potential of particulate composites

## Structural reinforcement and functional applications

### Importance of an integrated approach

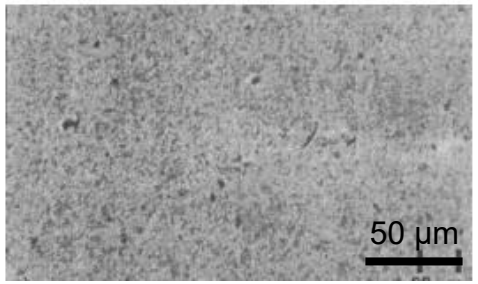
- Deagglomeration and dispersion crucial
- Interplay particle and matrix/solvent
- Link microstructure-processing?



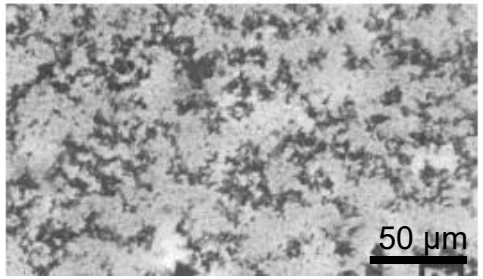




Deagglomerated
Dispersed




Reagglomerated
Non-dispersed




Schueler, J. Petermann, K. Schulte et al. *Journal of Applied Polymer Science* 1997, 63, 1741-1746.
Kaidarova, V. Naenen, R. Windey, et al. *Materials Science & Engineering R* 2024, 163, 100933. (Adapted with OpenAI, ChatGPT).



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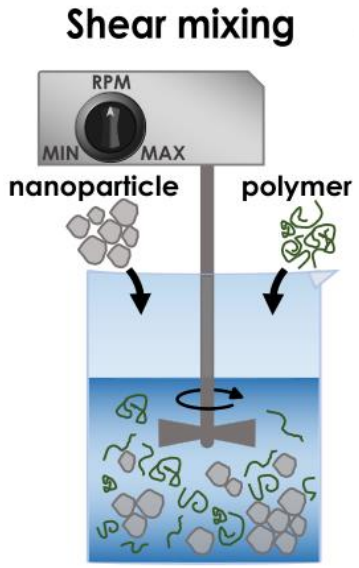
# Typical mixing approaches for nanoparticles in polymers

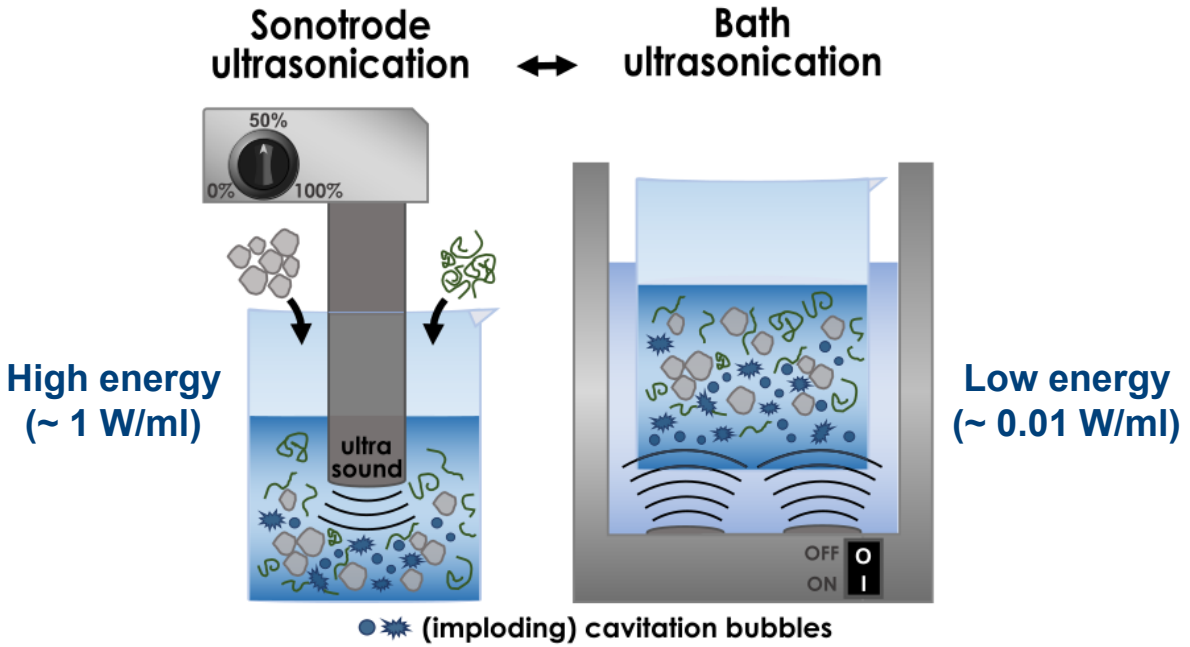
## The need for high-energy dispersion methods

### Classical low-shear methods for particles




- Mechanical mixing
- Macroscopic mixing & limited deagglomeration

### Tailored high-shear methods for nanoparticles




- Sonofragmentation in liquid
- Microscopic mixing & effective deagglomeration

# The material system-specific challenge of optimising ultrasonication

## The power of ultrasound

### Framework for optimisation

- Few studies adequately report parameters
- Material system-specific behaviour
- Universal scaling parameters: energy and power density

$$E_\rho = \frac{\text{power x active time}}{\text{volume}} \left[\frac{W.s}{ml} \text{ or } \frac{J}{ml}\right] \qquad P_\rho = \frac{\text{power}}{\text{volume}} \left[\frac{W}{ml}\right]$$

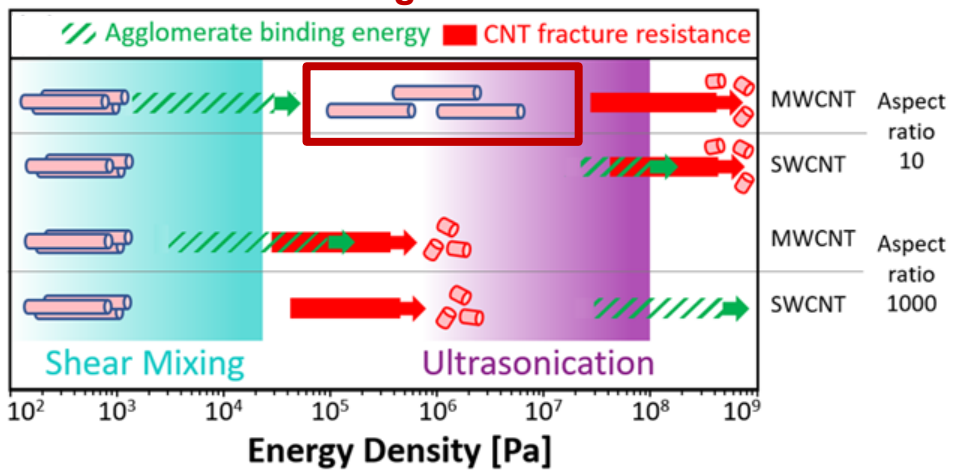
### Challenge: intraparticle before interparticle breakage




Particle/polymer breakage



Processing window



# Development of strategies for optimising an ultrasonication procedure

## A case-study-based approach for functional particulate composites

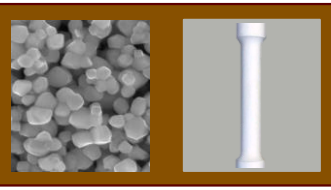
**Integrated evaluation: microstructure-performance**

Morphology & Microstructure  Functional performance

**3 case studies of functional particulate composites**

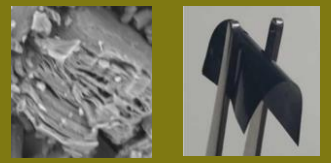
**Case study 1**

3D-like $BaTiO_3$ particles in epoxy matrix for Digital Volume Correlation (DVC)



**Case study 2**

2D-like MXene particles in PVDF matrix for EMI shielding



**Case study 3**

1D-like short carbon fibre (CF) particles in PU matrix for acoustic shielding





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# Case study 1: 3D-like $BaTiO_3$-epoxy for Digital Volume Correlation

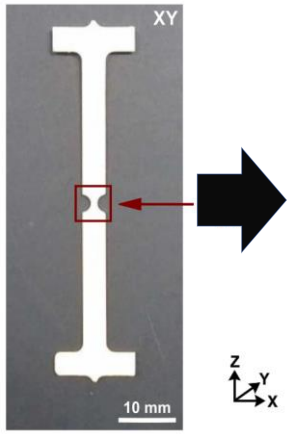
## Microstructure and morphology

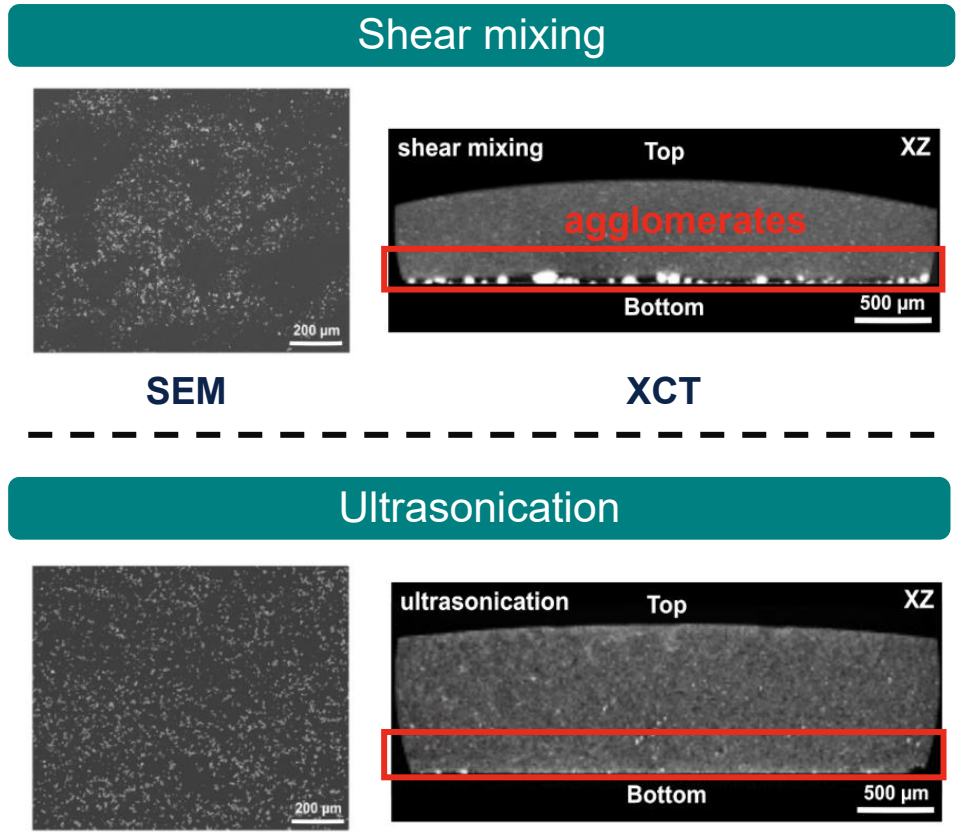
### Dispersion procedure

- 3 vol% $BaTiO_3$ (400 nm) in epoxy resin
- Mechanical shear mixing (7 min)
- Sonotrode ultrasonication (50 J/g – 720 J/g)

### Characterisation

- 2D SEM (local evaluation)
- 3D XCT (global evaluation)



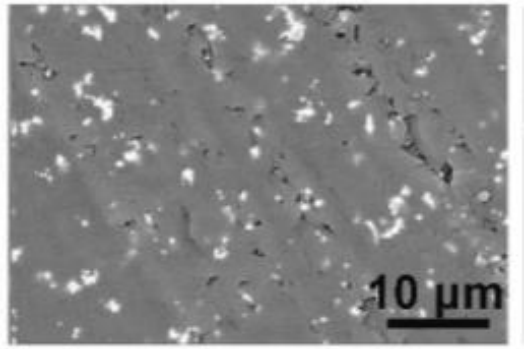
# Case study 1: 3D-like $BaTiO_3$-epoxy for Digital Volume Correlation

## Functional performance

**Optimised ultrasonication**

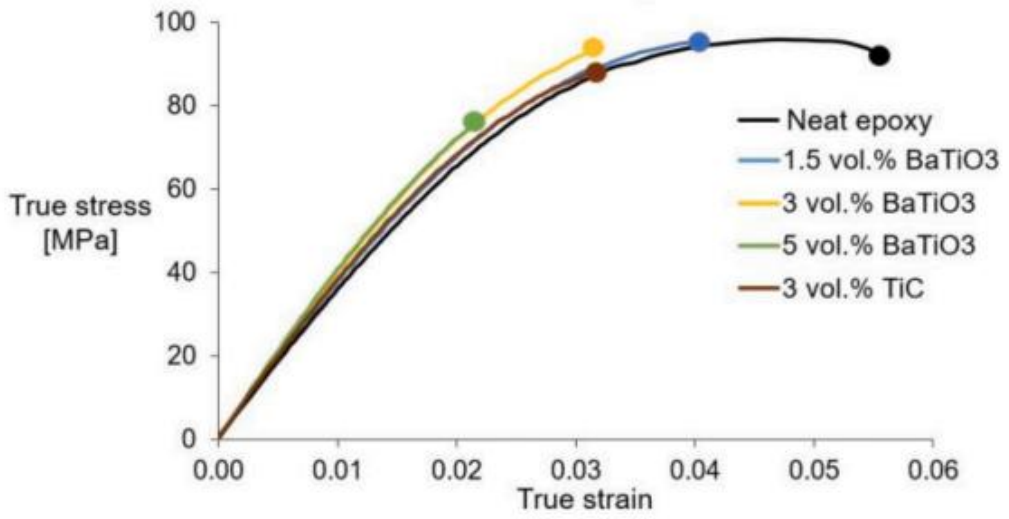
- Nanoparticle size < XCT voxel size (0.5 µm)
- Mild agglomeration good for speckle pattern
- Sonotrode ultrasonication (200 J/g)




**Structural performance**

- Rule of thumb: < 3 vol% particles
- Observation: no significant influence for 1.5 vol% $BaTiO_3$




*Epoxy-$BaTiO_3$ and epoxy-TiC (T. Chatziathanasiou)*

# Case study 2: 2D-like MXene-PVDF for EMI shielding

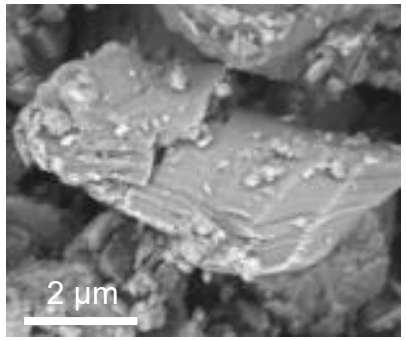
## Microstructure and morphology

### Dispersion procedure

- $Ti_3C_2T_z$ MXene (~ µm) & thermoplastic PVDF in DMF solvent
- Bath ultrasonication (15 min - 10 J/ml)



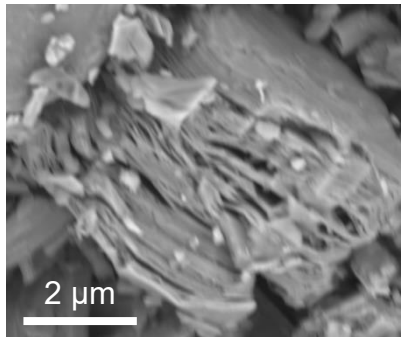

**$Ti_3AlC_2$ MAX**




Etching Al

Intercalation

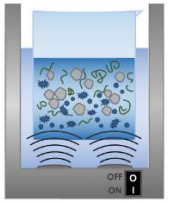
**$Ti_3C_2T_z$ multi-layered MXene**



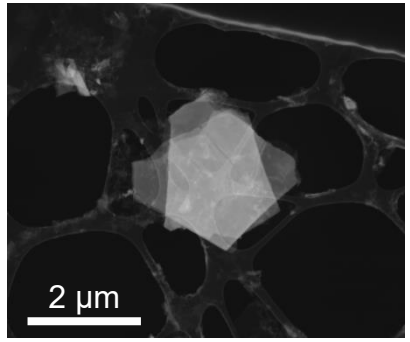



**Clay suspension**

Delamination



**$Ti_3C_2T_z$ mono-layered MXene**






**Supernatant suspension**

# Case study 2: 2D-like MXene-PVDF for EMI shielding

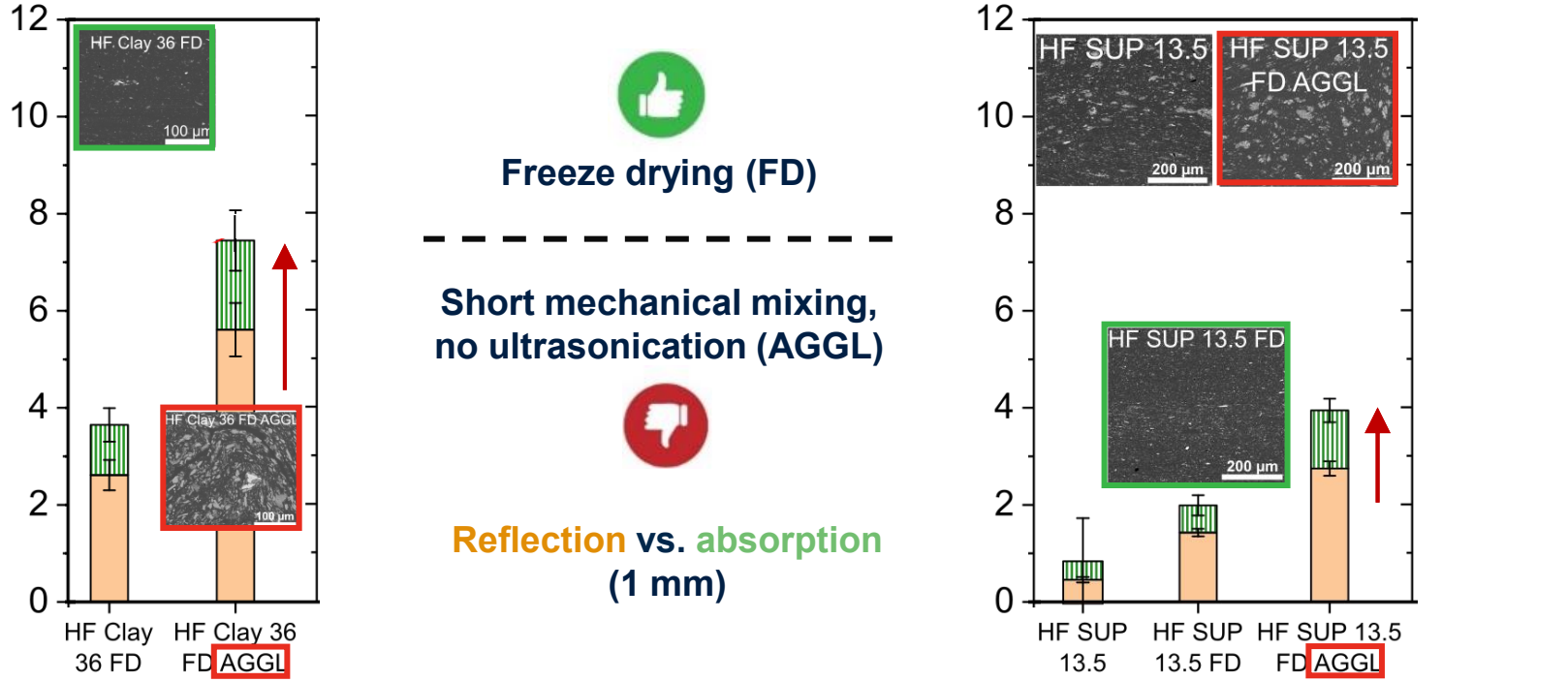
## Functional performance



**Multi-layered clay (CLAY - 36 wt%)**

**Few-layered supernatant (SUP - 13.5 wt%)**

Shielding effectiveness [dB]




**Freeze drying (FD)**

**Short mechanical mixing, no ultrasonication (AGGL)**

**Reflection vs. absorption (1 mm)**

Hypothesis: reduced dispersion and deagglomeration introduce ‘local defects’ beneficial for EMI shielding



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# Case study 3: 1D-like short carbon fibre-PU for acoustic shielding

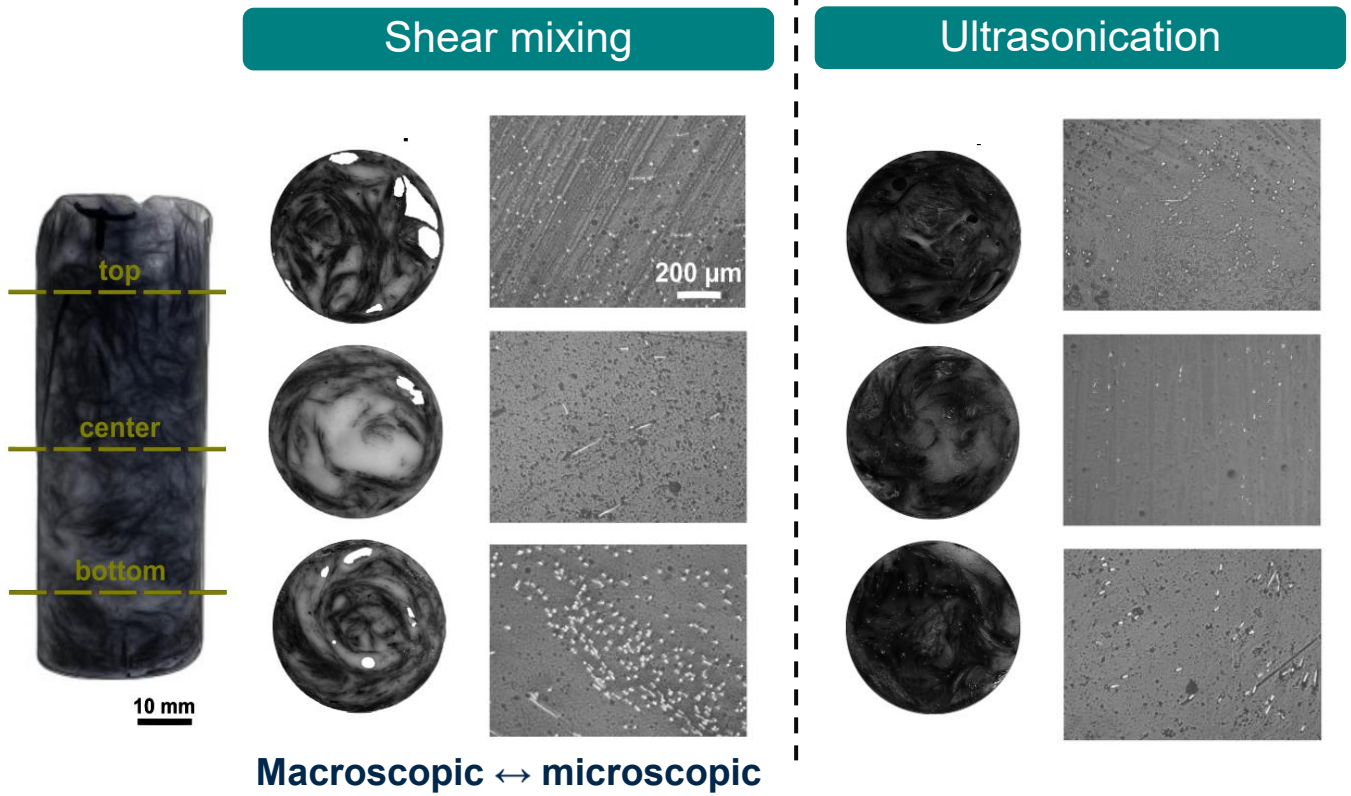
## Microstructure and morphology

### Dispersion procedure

- 1 vol% short CF (4 mm – Ø 5 µm) in PU resin
- Mechanical shear mixing (10 min)
- Sonotrode ultrasonication (100 J/ml)

### Characterisation

- 2D photographic images (global evaluation)
- 2D optical microscopy (local evaluation)

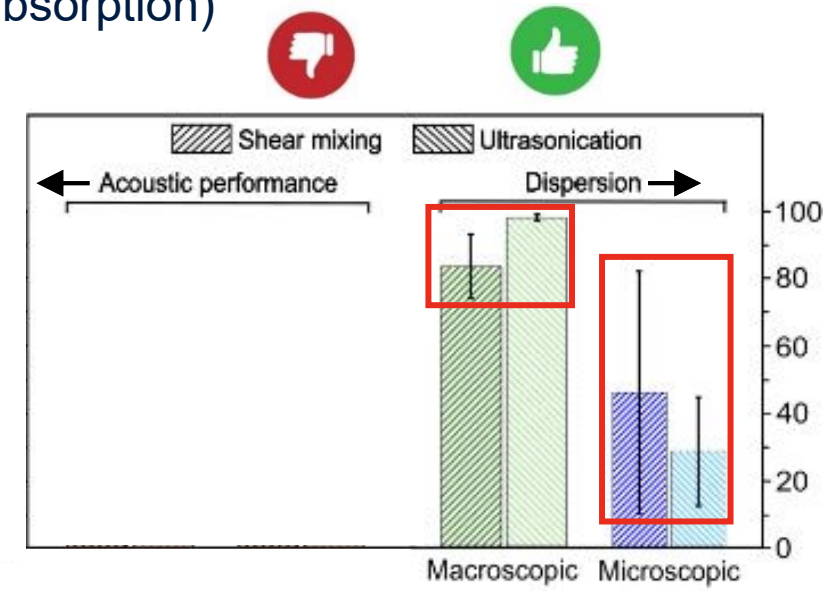


# Case study 3: 1D-like short carbon fibre-PU for acoustic shielding

## Functional performance

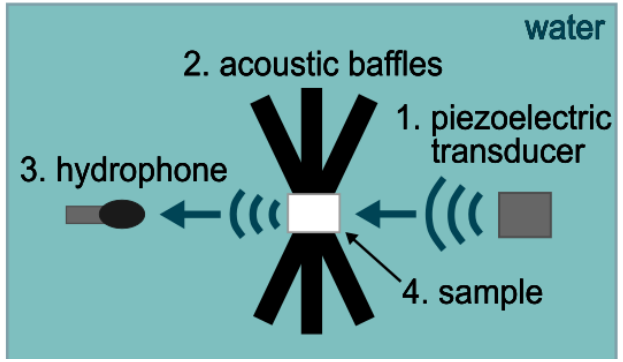
**CF-PU specimen (20 mm thickness)**

- Room temperature curing: heating issues
- Sound transmission loss (~ absorption)




CF area fraction [%]

**Transducer-sample-hydrophone setup**



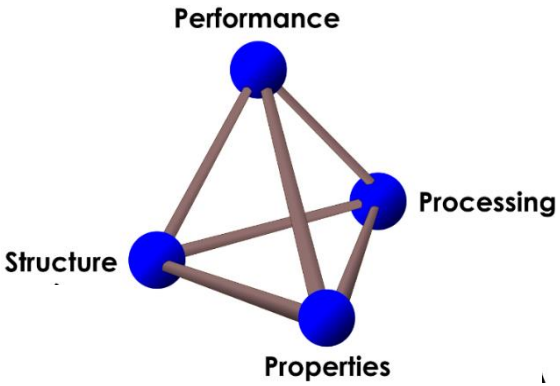

Hypothesis: reduced dispersion and deagglomeration introduce ‘local defects’ beneficial for acoustic shielding

# Conclusions and challenges

## Ultrasonication as an effective method for particle dispersion in (nano)composites

### Main takeaways

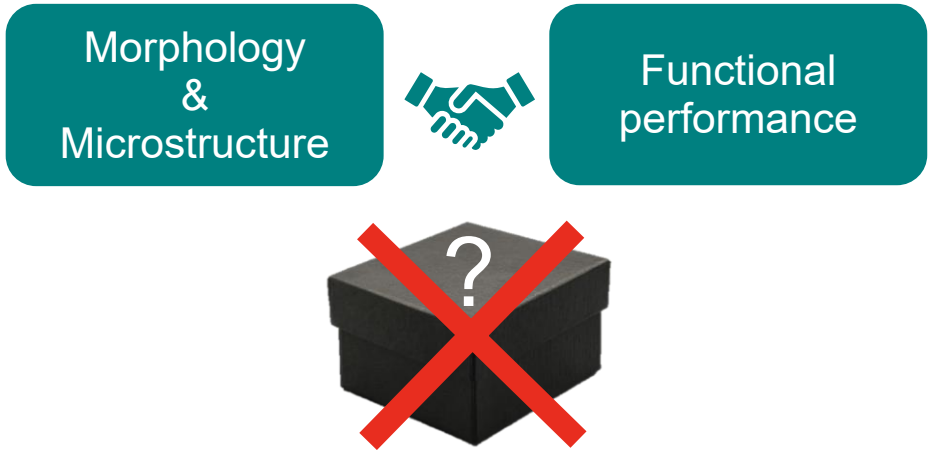
- Particle surface interactions: high-energy dispersion methods
- Ultrasonication: effective, but system-specific optimisation
- Optimisation framework: integrated evaluation processing-performance




### Challenges

- Understanding link microstructure-performance
- Quantitative reporting ultrasonication parameters

  → Design guidelines



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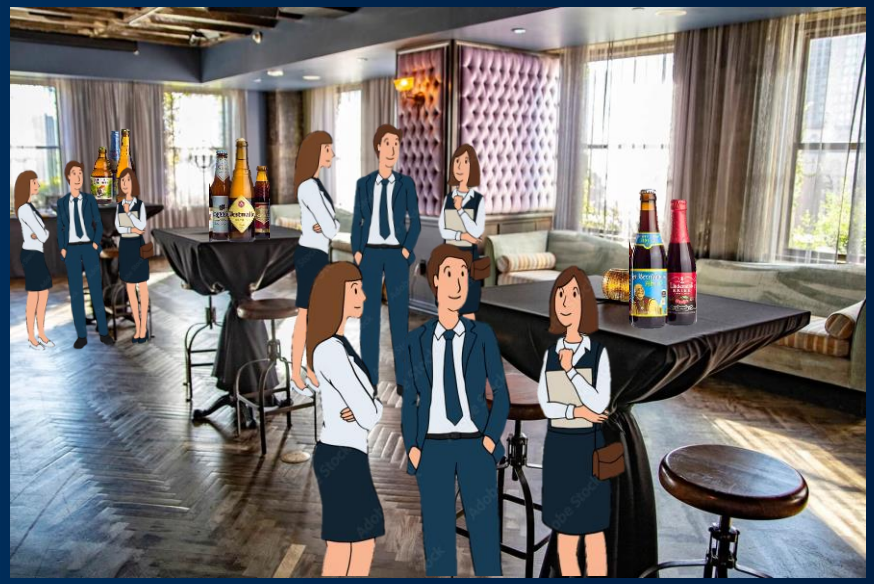
# The Belgian beer reception metaphor

Selecting the right dispersing approach to maximally exploit the inherent particle properties





Energetic dispersion approach

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Materials Performance Nondestructive Testing — Roxanne Baeten, dr. Jeroen Soete, dr. Helge Pfeiffer, prof. Martine Wevers

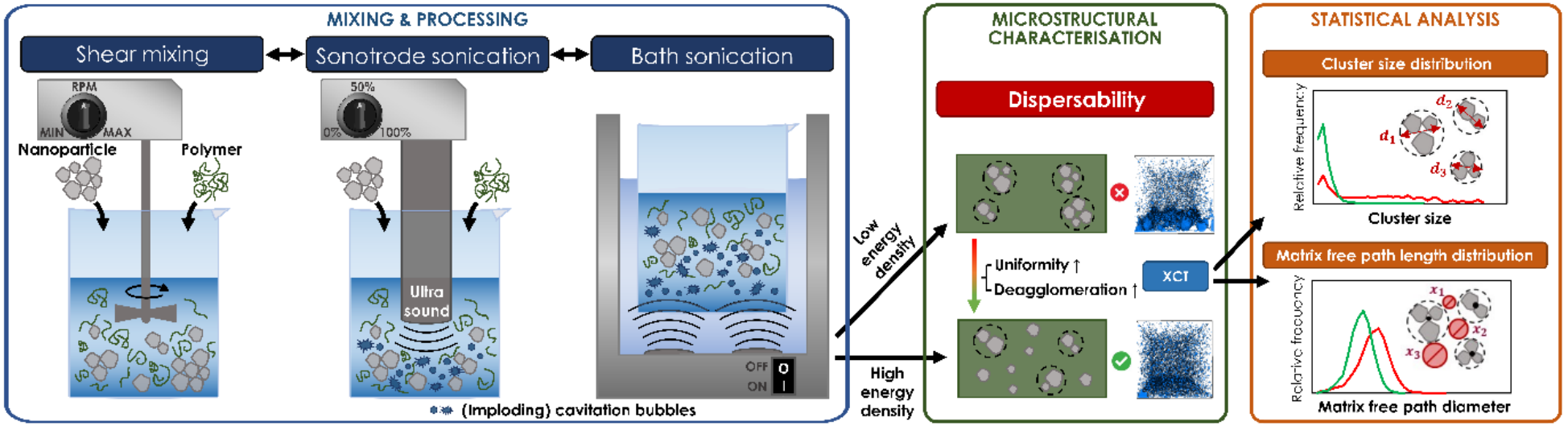
Composite Materials Group — Marion Cardous, dr. Sina AhmadvashAghbash, prof. Yentl Swolfs, prof. Frederik Desplentere, prof. David Seveno

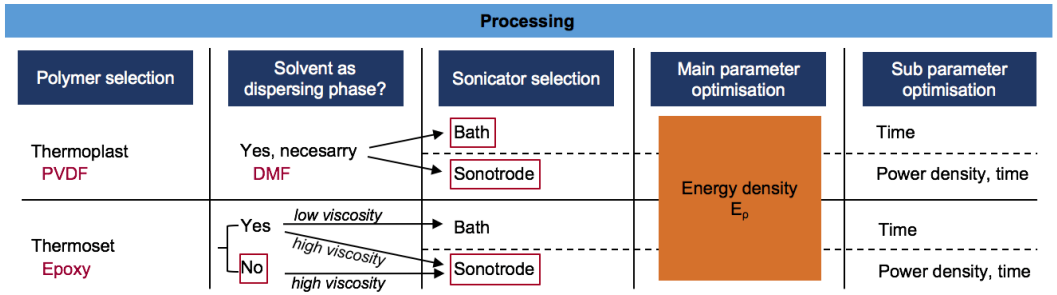
KU LEUVEN — Prof. Jef Vleugels, prof. Ruth Cardinaels

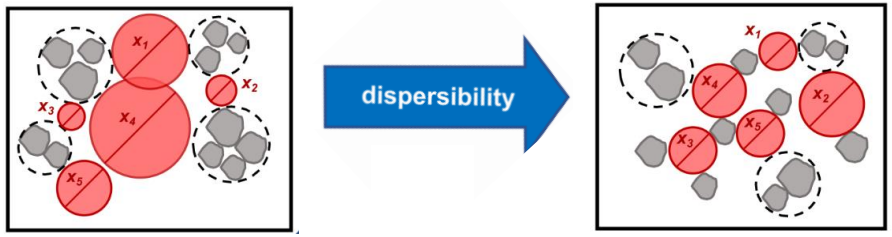
# Proposed framework for optimising an ultrasonication procedure

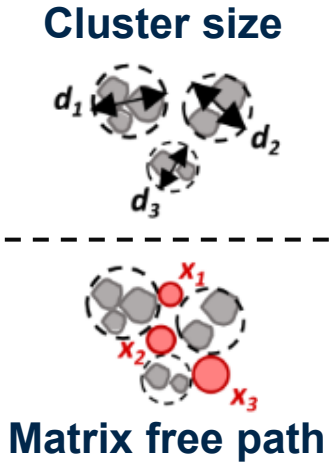
## Optimisation particle-polymer dispersion

+ Functional performance

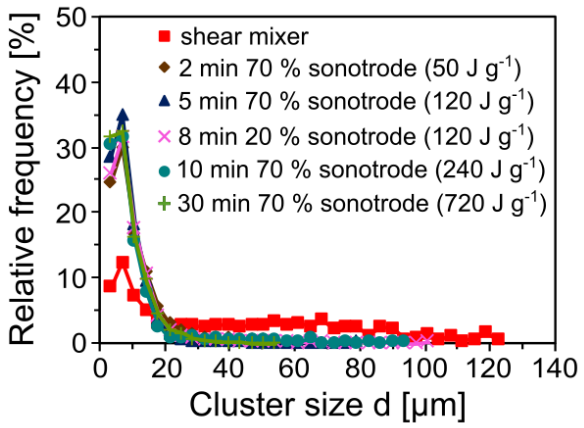


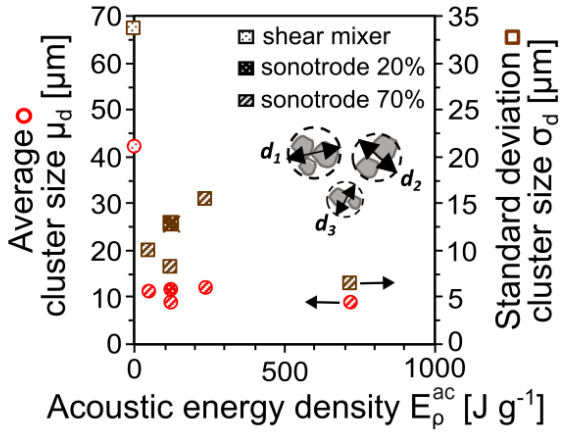



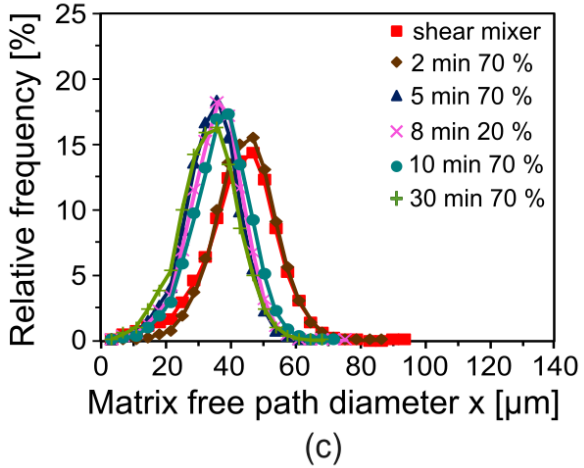



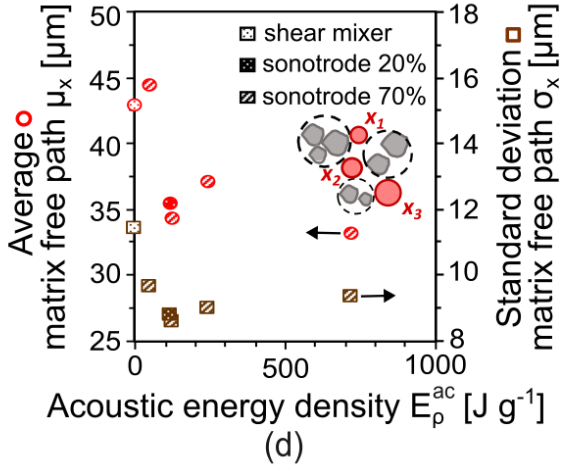



# Deagglomeration and dispersion of $BaTiO_3$ in epoxy

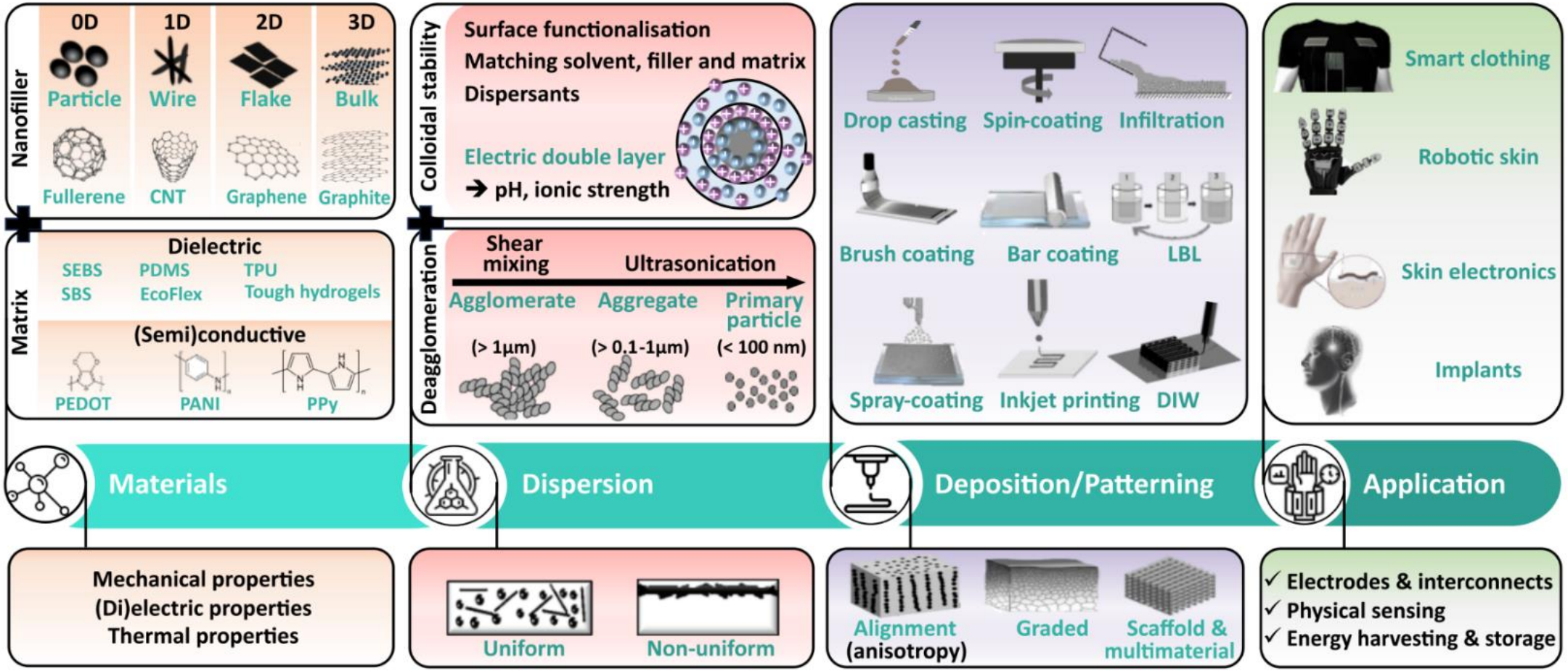


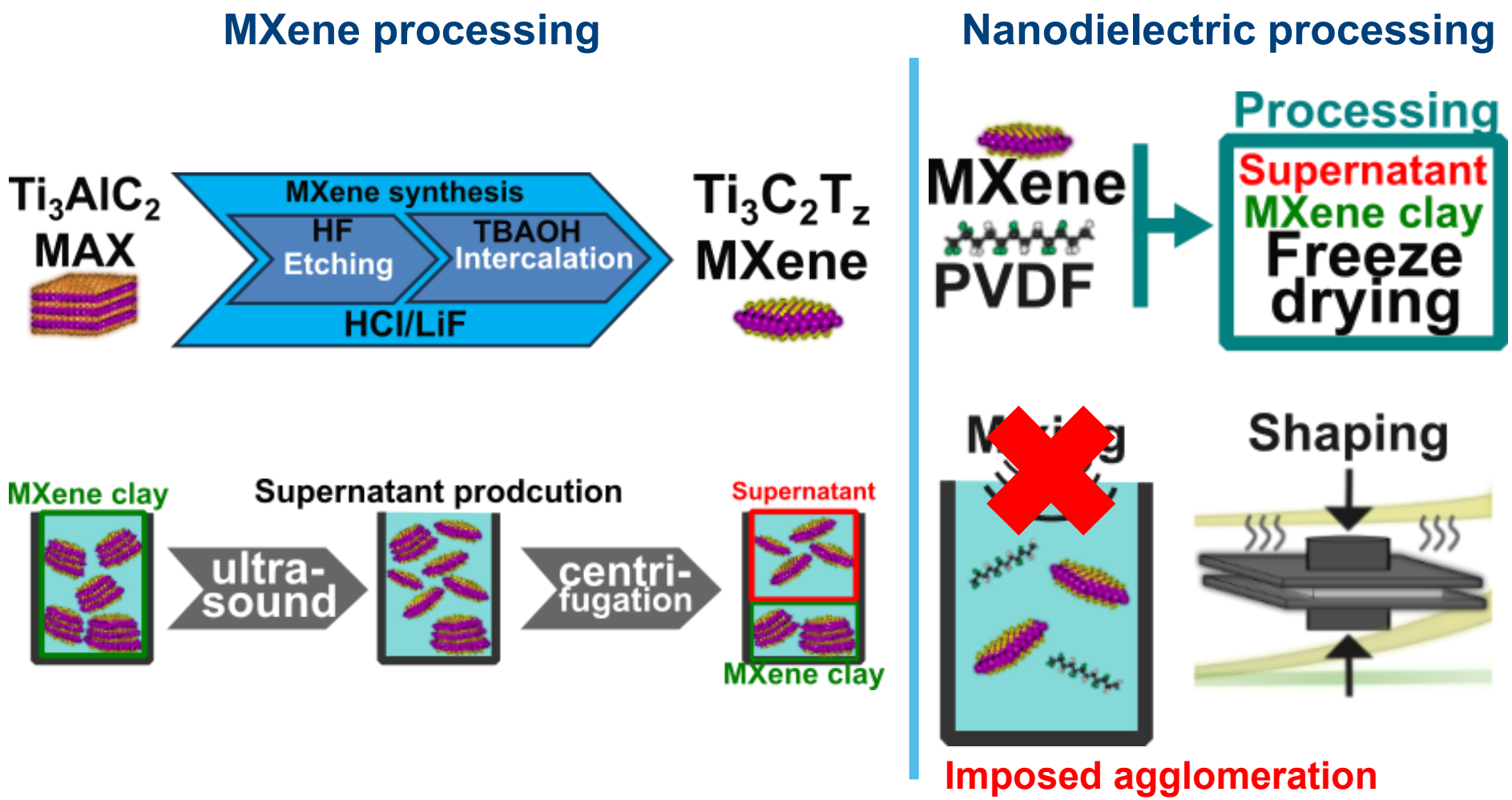
# Integrated approach for functional particulate composites





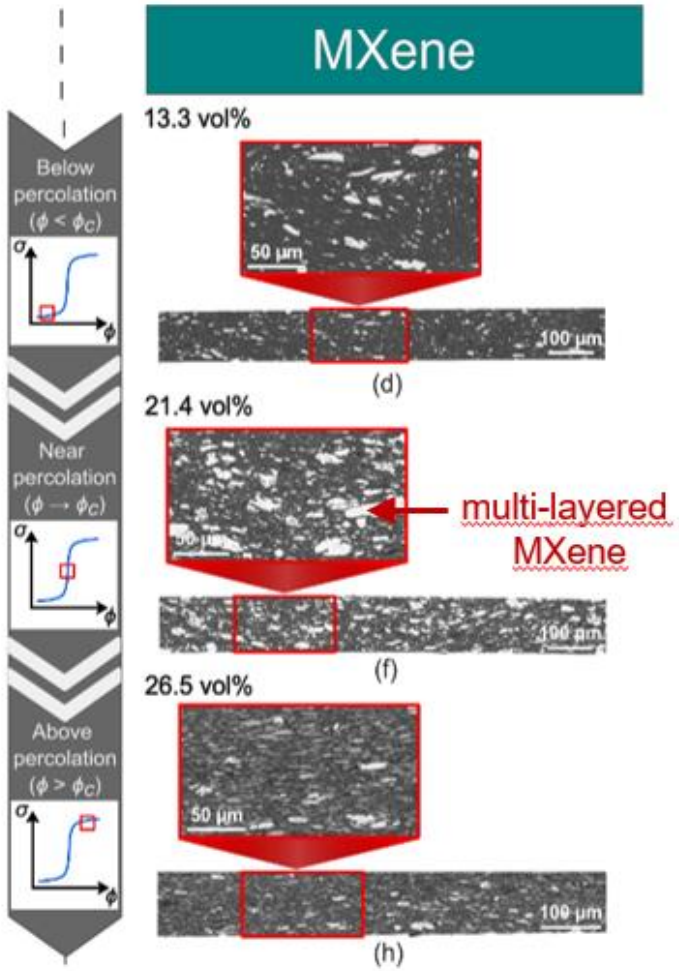

Kaidarova, V. Naenen, R. Windey, et al. *Materials Science & Engineering R* 2024, 163, 100933. (Adapted with OpenAI, ChatGPT).

# PVDF-MXene dielectrics



# MXenes composition and filler loading

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | M | | A | | X | | $T_z$ | | B | C | N | O | F | Ne |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | Ar |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| Cs | Ba | Lu | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hf | Tl | Pb | Bi | Po | At | Rn |
| Fr | Ra | Lr | Rf | Db | Sg | Bh | Hs | Mt | Uun | Uuu | Uub | | Uuq | | | | |





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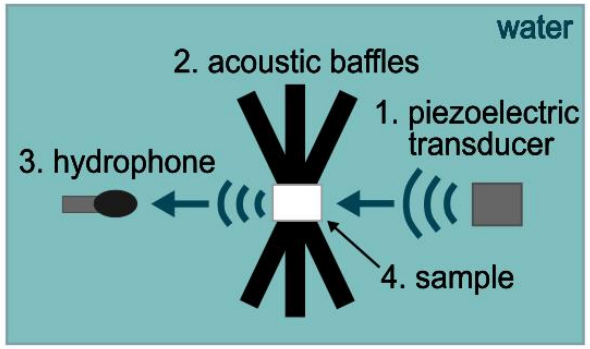
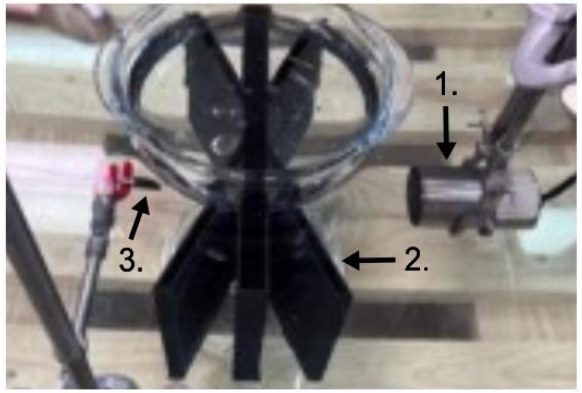
# Acoustic shielding setup PU-CF composites

## High frequency

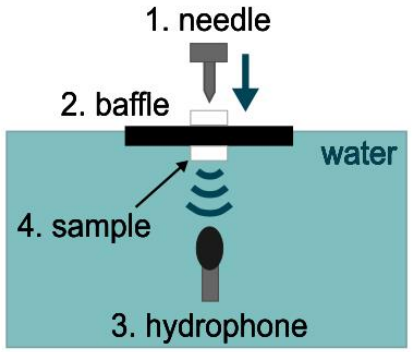



(a)

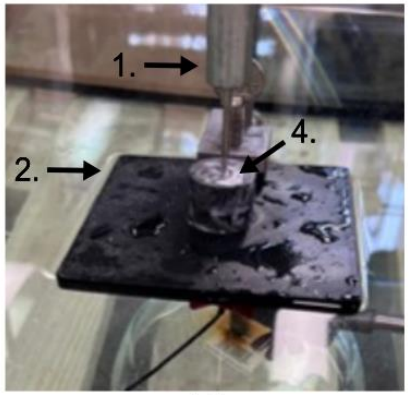



(c)

## Low frequency




(b)

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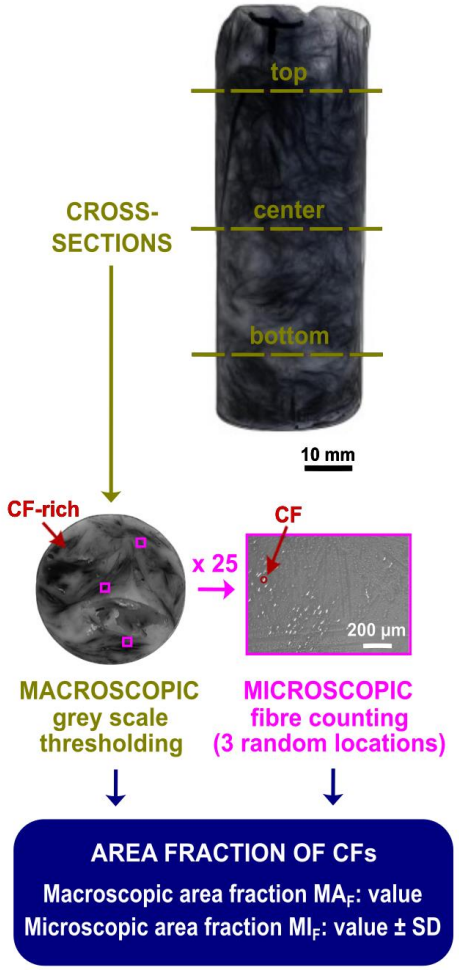




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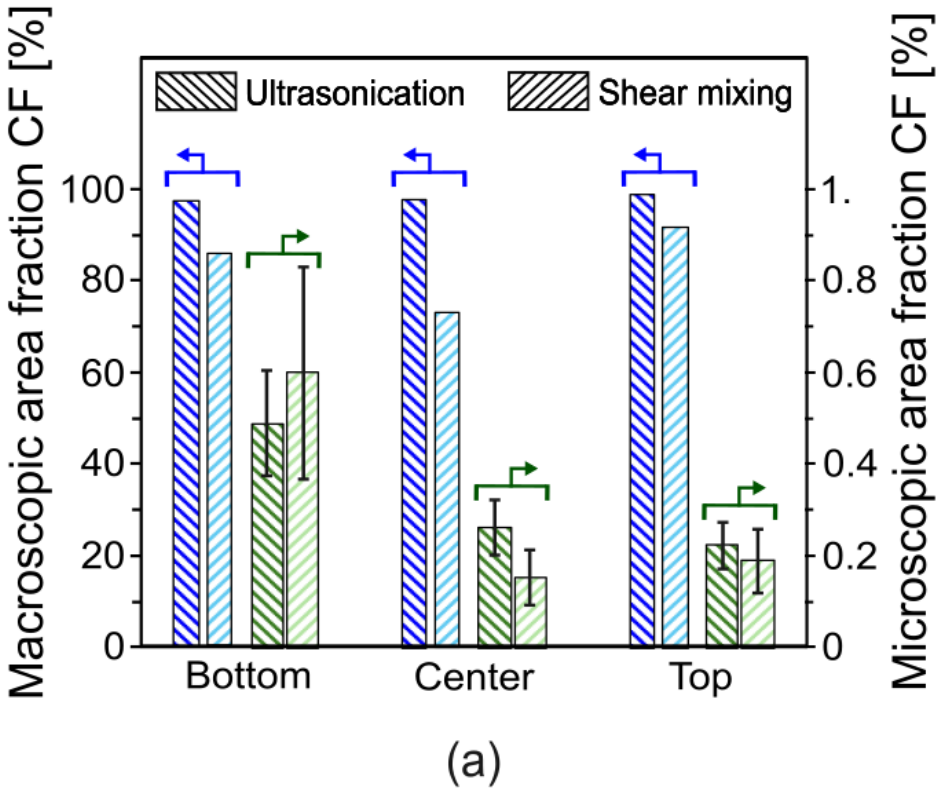
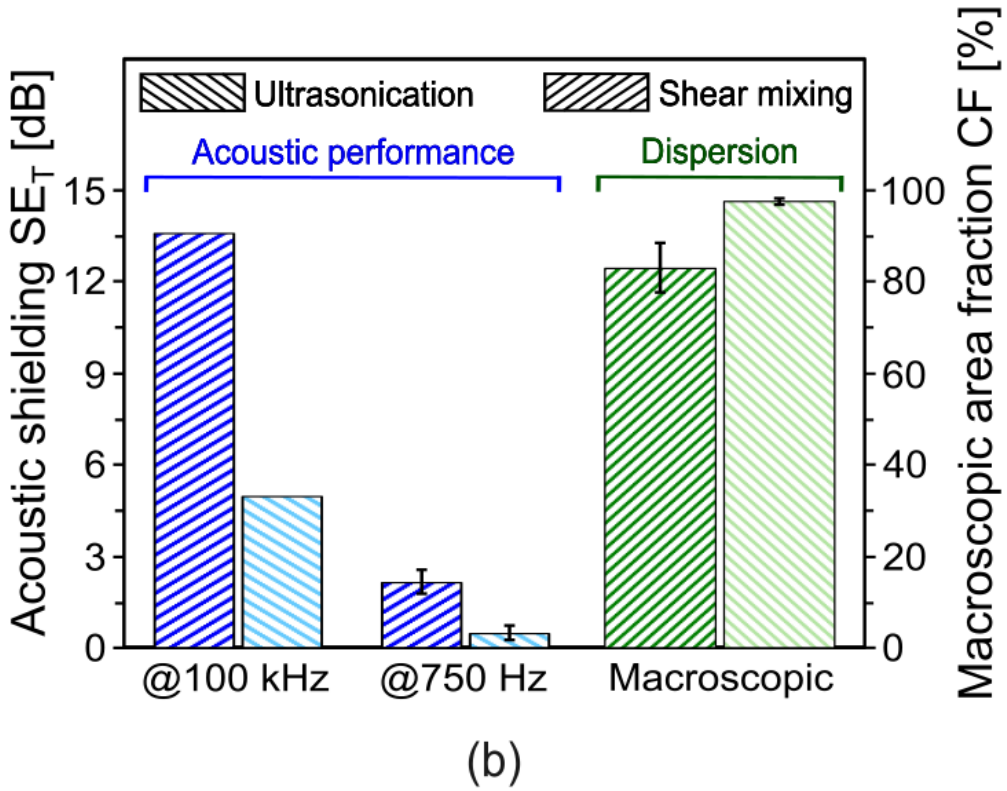
# Acoustic shielding and microstructure of PU-CF composites (2)



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