### **APPARENT ELASTIC MODULUS OF** POLYETHYLENE AND ITS NANOCOMPOSITES MEASURED AT DIFFERENT SCALES

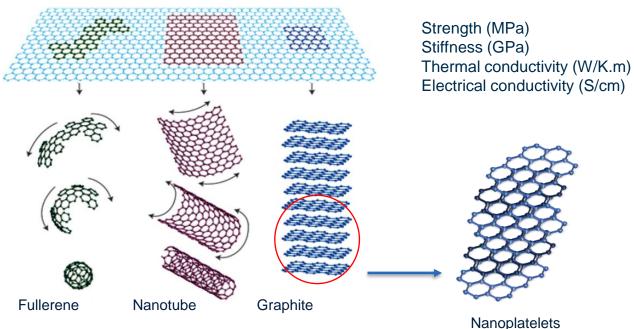
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# INTRODUCTION



# **Graphene/Graphite Nanoplatelets (GNPs)**



Graphene Ref. 130000 350 (steel) 1000 200 (steel) 4000 400 (silver) 6000 70% > silver

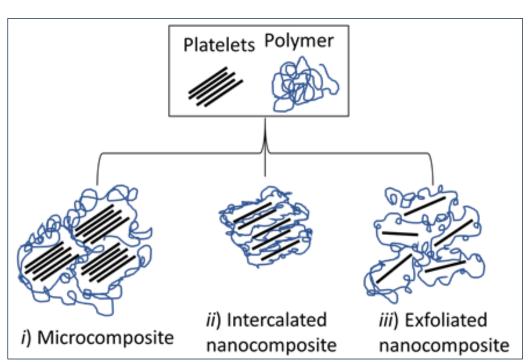


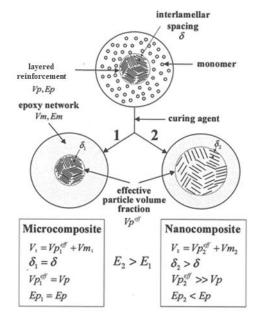
Nanoplatelets

Kumar, A., Sharma, K. & Dixit, A.R. A review on the mechanical properties of polymer composites reinforced by carbon nanotubes and graphene. Carbon Lett. 31, 149–165 (2021).



# **Agglomeration Challenges**





Kornmann, X. (2001). Synthesis and characterisation of thermoset-layered silicate nanocomposites (Doctoral dissertation, Luleå tekniska universitet).

Real composite is a mixture of all of the above! Challenging to predict the properties at this scale

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# **OBJECTIVES**



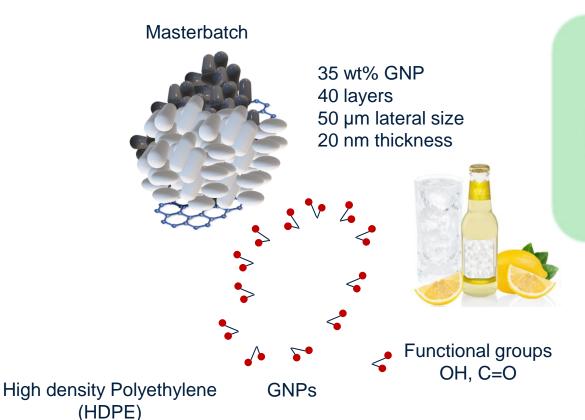
- How can we best characterize nanocomposites?
- How valid is the different techniques at different scales? How do they compare? Which is the most representative?
- What are advantages and disadvantages of each of those techniques?



# **METHODOLOGY**



#### **GNP Masterbatch**



Commercial product

Recyclable

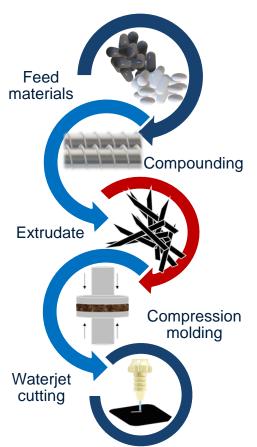
Stable production

Suitable form for industry

Less hazardous



# **Melt Compounding**



Advantages

Upscaling possibility

Use of masterbatch

No process modification

Environmentally friendly

Solvent free

Limitations

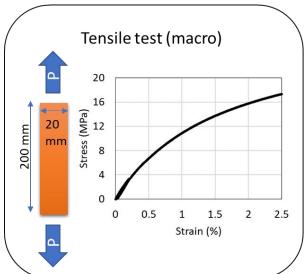
High viscosity of the melt

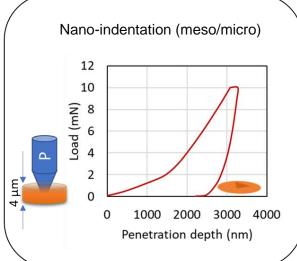
High percolation threshold

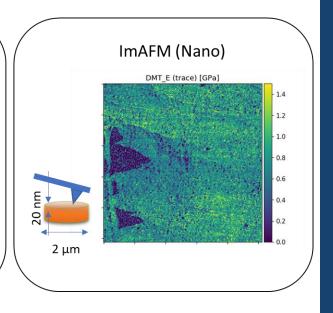
Limited to short fibers



#### **Tests at Different Scales**



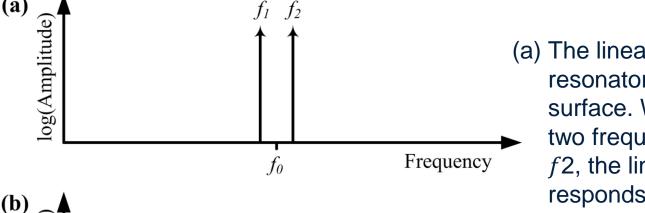


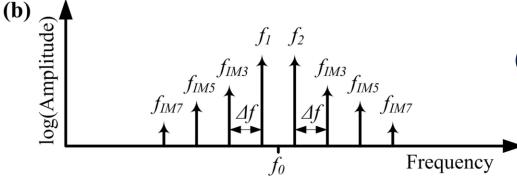


Instron 3366
Displacement control test
2mm/min

NanoTest Vantage 10 mN max load 15 sec loading time Berkovitch indenter A Bruker Dimension Icon AFM ImAFM mode
Frequencies near resonance
TAP300 DLC Probe

#### **ImAFM**



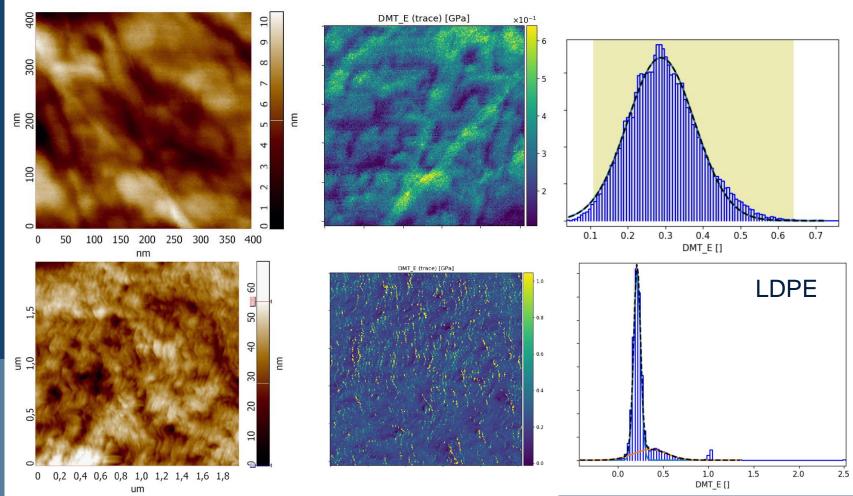


- (a) The linear response of the resonator away from the surface. When driven with two frequencies, f1 and f2, the linear system responds with oscillation at f1 and f2.
- (b) When close to the surface, nonlinear tip-surface interactions generate IMPsof many orders.

PLATZ, Daniel, et al. Intermodulation atomic force microscopy. Applied Physics Letters, 2008, 92.15: 153106.

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# **RESULTS**



# Comparison of results at the different scales

#### **Apparent Modulus (GPa)**

	Tension	NI	ImAFM
LDPE	0.42	0.71	0.42-1.1
LDPE4	0.79	0.71	0.9
HDPE	1.89	1.84	2.7
HDPE2	2.11	3.46	3.3
GNPs	4.79*	22.6*	~4

HDPE2

1 2 3 4 5 6 7 8

Modulus (GPa)



<sup>\*</sup> Calculated using micromechanics

#### **Tensile test**

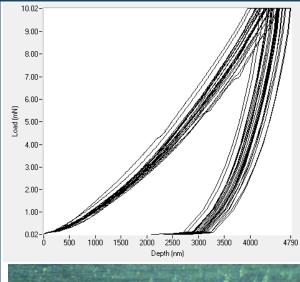
- Can obtain more than one property (modulus, strength, Poisson's ratio, etc.)
- More averaged/homogenized value useful for material selection for application (on the final stage of materials development)
- Limitation require large amount of material to produce statistically representative results

#### **Nano-indentation**

- Relatively small samples good for materials under development
- Could do large number of indents in relatively short time for statistics
- Limitations more complicated sample preparation routines,
   expensive equipment, viscoelastic effect (VE-effect)
- Needing to know Poisson's ratio to evaluate the modulus (in

$$rac{1}{E_{
m r}} = rac{1- arphi_m^2}{E_m} + rac{1- arphi_i^2}{E_i}$$

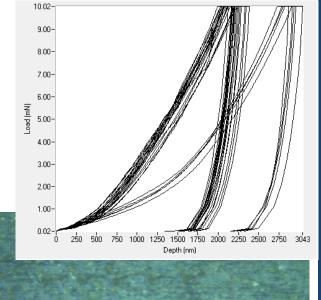


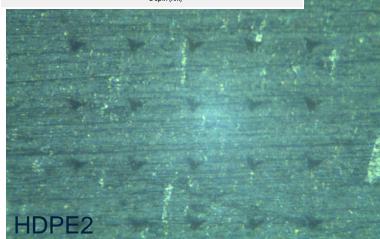


#### VE-effect in NI

Indents disappear with time for materials exhibiting higher VE

Evaluating stiffness based on other missing properties of particle





LDPE 4

### **ImAFM**

- Can resolve microstructure of polymer as well
- Affected by surface freshness (presence of oligomers)
- Working in the scale of reinforcement
- Quantitative output together with image of mapped property
- Relatively less demanding surface preparation (compared to NI)



### **Conclusions**

- Characterization has been performed at the different scales and ImAFM seems to be good complementary technique in the development stage of material.
- ImAFM gives reasonable values of the effective modulus of particles that could be used for modelling
- Nano-indentation gives values depending on the indented area and requires wider pre-knowledge of the material
- All techniques fail to predict or solve the problem at higher concentrations due to interaction between the particles



# THANK YOU FOR LISTENING

For details related to ImAFM contact

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