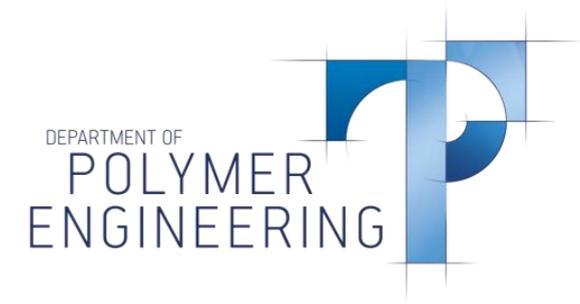


Budapest University of Technology  
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# PSEUDO-DUCTILITY IN LAYER-BY-LAYER HYBRID COMPOSITES THROUGH PRECISE CONTROL OF THE INTERLAYER THICKNESS

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ICCM23 – International Conference on Composite Materials,  
Queen’s University Belfast, UK, 30 July-4 August 2023

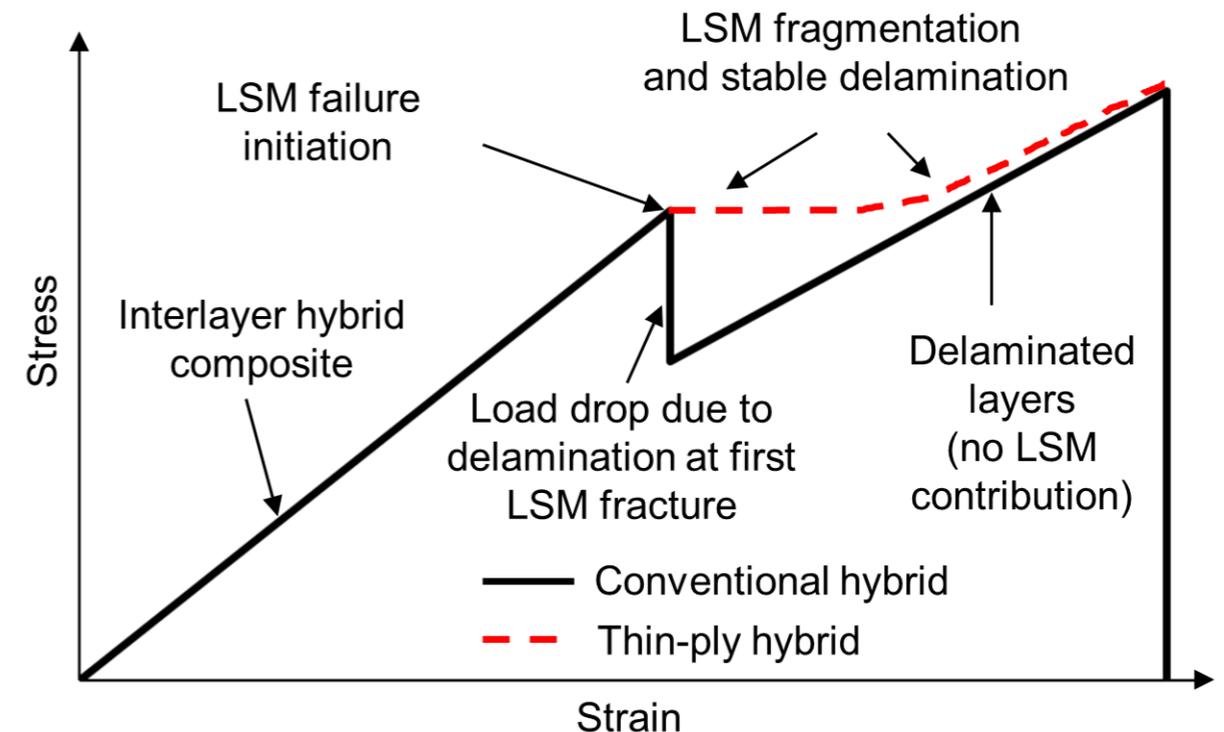
# Introduction

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- Composites are stiff, strong and lightweight
- But their failure is usually catastrophic
- Ductility is needed for safety
  - High deformation at break
  - Detectable damage accumulation
  - Warning before failure
  - Residual strength after severe damage

# State of the art

- **High performance composites: stiff and strong**, but **failure is sudden and brittle** with **little warning** and **poor residual strength**
- **Pseudo-ductility** with an **intrinsic safety margin** could **change design** approach and offer **major benefits**
- Excellent **pseudo-ductility demonstrated first with thin-ply UD carbon/glass interlayer hybrid laminates in tension**
  - Mechanisms: **fragmentation** and **stable delamination**



# State of the art

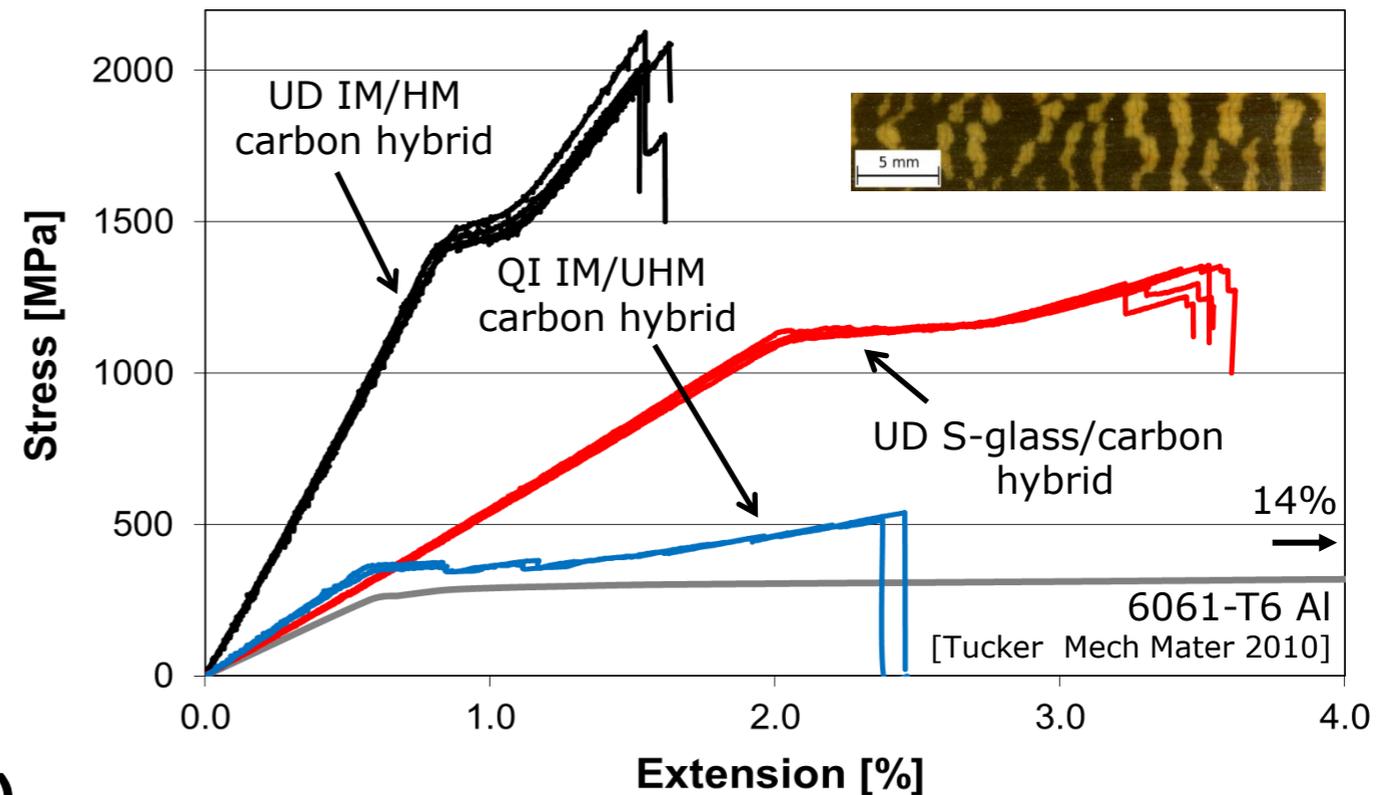
- **High performance composites: stiff and strong, but failure is sudden and brittle with little warning and poor residual strength**
- **Pseudo-ductility** with an **intrinsic safety margin** could **change design** approach and offer **major benefits**
- Excellent **pseudo-ductility demonstrated first with UD glass/carbon interlayer hybrid laminates in tension**

- Mechanisms: **fragmentation** and **stable delamination**

The concept is then extended to:

- **UD IM/HM carbon hybrids**
- **QI IM/UHM carbon hybrids**
- **Visible damage:**
  - **Overload indicator**

(UK patent pending- no. **GB2544792B**)



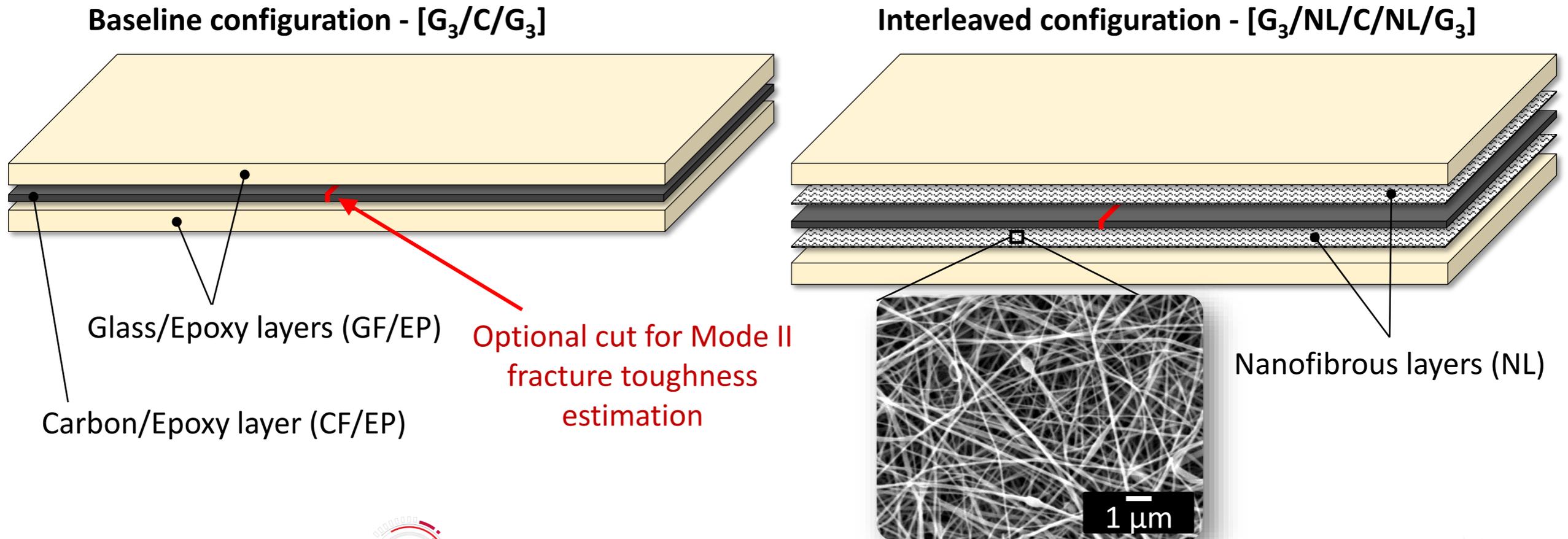
# Key challenges

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- To develop pseudo-ductile hybrid composites based on standard thickness plies
- Reduce cost by using cheaper normal CF/EP plies instead of thin-ply prepregs
- Improve performance by increasing the CF/EP to GF/EP ratio in the hybrid laminates while preserving pseudo-ductility

# Concept, design

- Pseudo-ductility was demonstrated with thin, high strain CF/EP plies or normal thickness low strength (high modulus) CF/EP plies
- Increase the mode II fracture toughness by interleaving nanofibrous layers and use normal thickness high strain CF/EP



# Materials

## Composite plies

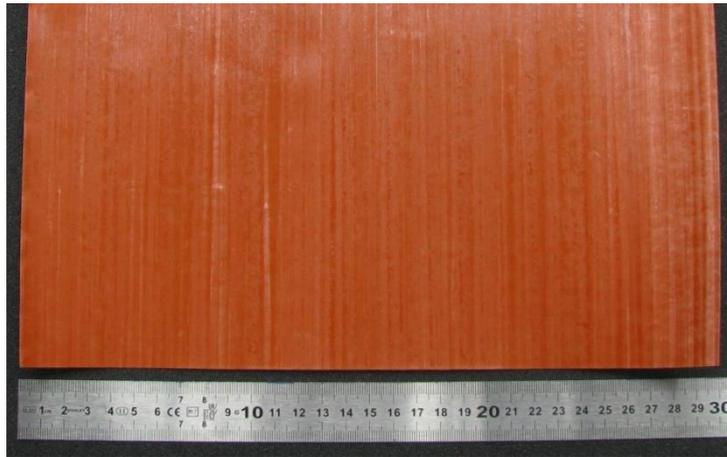
Prepregs	Nominal fibre areal density [g/m <sup>2</sup> ]	Fibre volume fraction [-]	Cured ply thickness [μm]	Tensile strain to failure [%]	Tensile modulus [GPa]	Coefficient of thermal expansion [1/K]
AGY Y-110 S-2 glass/913 epoxy	190	0.49	153.8	3.7	45.6	$3.20 \cdot 10^{-6}$
Hexcel IM7 carbon/913 epoxy	100	0.58	95.8	1.6%	163.2	$-0.103 \cdot 10^{-6}$

## Nanofibrous layers

Targeted areal weight [g/m <sup>2</sup> ]	<i>Electrospinning solution PA6 concentration: 8wt% (designation: 8PA6)</i>			<i>Electrospinning solution PA6 concentration: 15wt% (designation: 15PA6)</i>		
	Measured final areal weight [g/m <sup>2</sup> ]	Substrate speed [m/min]	Average fibre diameters [nm]	Measured final areal weight [g/m <sup>2</sup> ]	Substrate speed [m/min]	Average fibre diameters [nm]
<b>2</b>	2.3 (7.7)	0.20	108 (20.4)	2.44 (8.6)	0.80	267 (26.6)
<b>5</b>	5.26 (11.0)	0.12	103 (19.8)	5.68 (11.8)	0.36	243 (27.6)
<b>10</b>	10.92 (12.4)	0.05	121 (18.0)	10.66 (8.8)	0.22	280 (31.4)

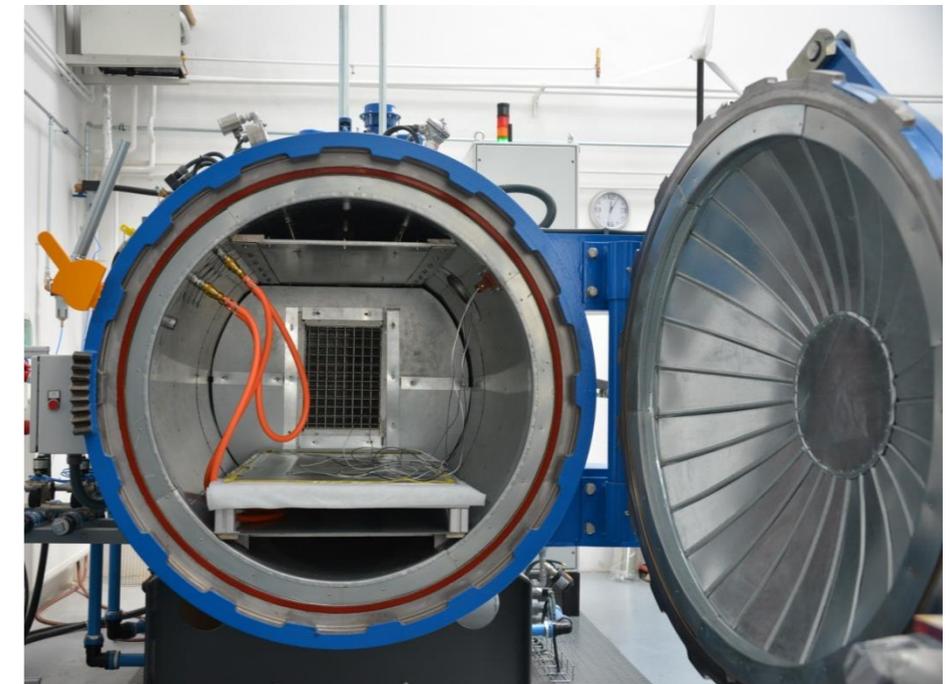
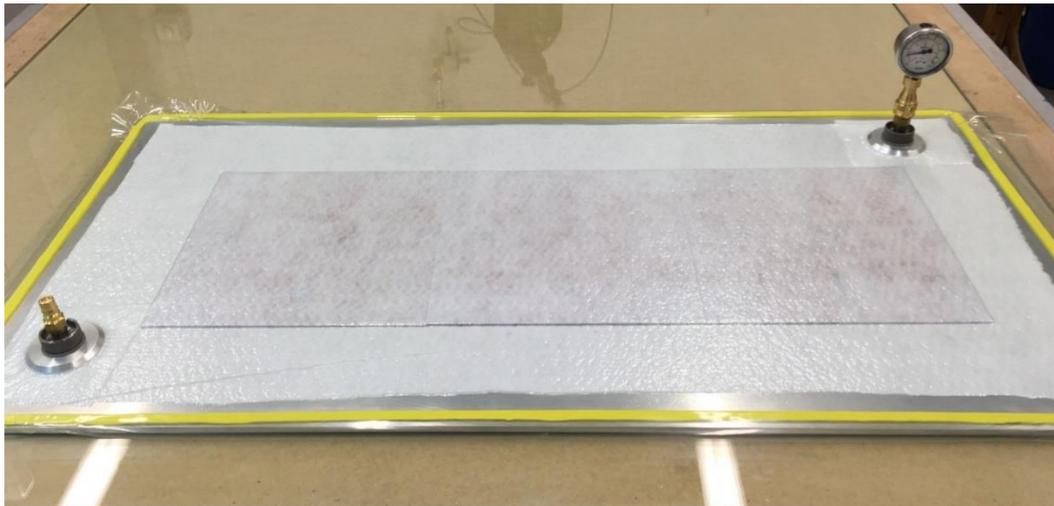
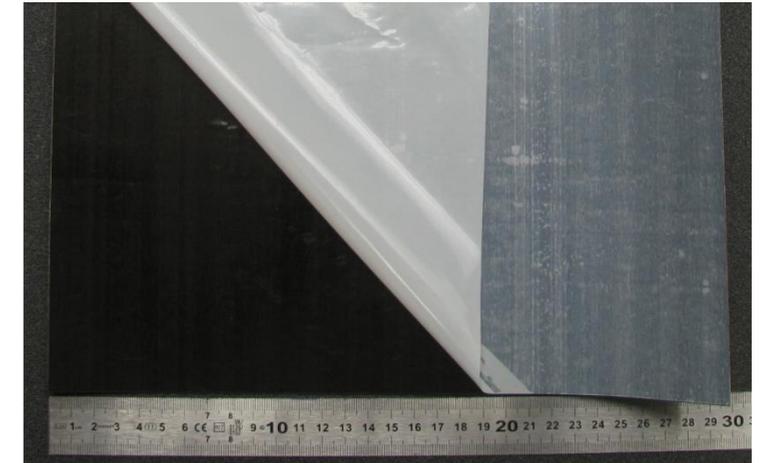
# Materials, manufacturing

UD glass/epoxy prepreg



- Manual lay-up of the composite prepreg plies
- Attaching dry nanofibrous layers to the prepregs
- Autoclave curing of the interleaved hybrid laminates at 125 °C and 7 bar

UD carbon/epoxy prepreg

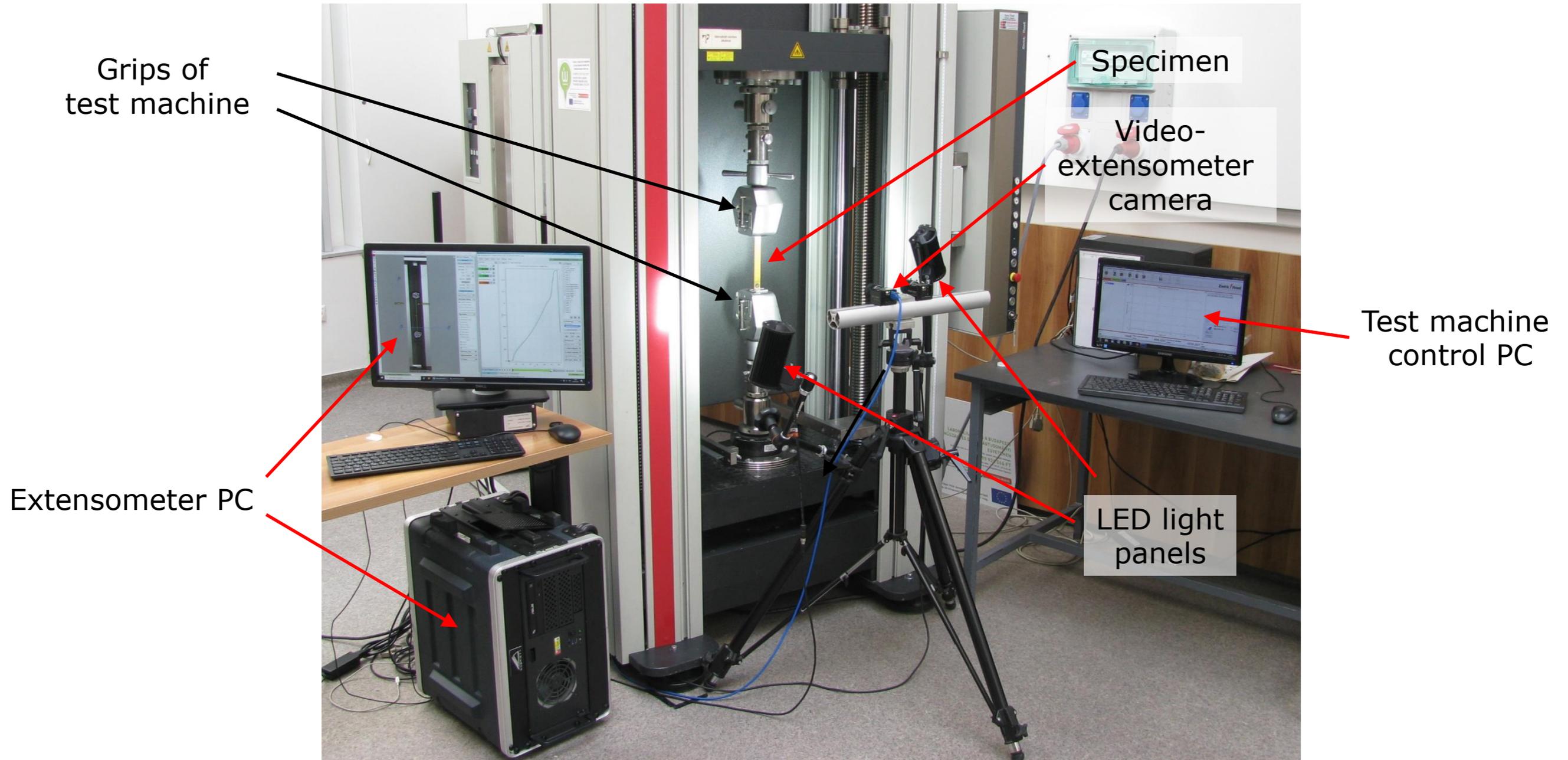


# Tested hybrid laminate configurations

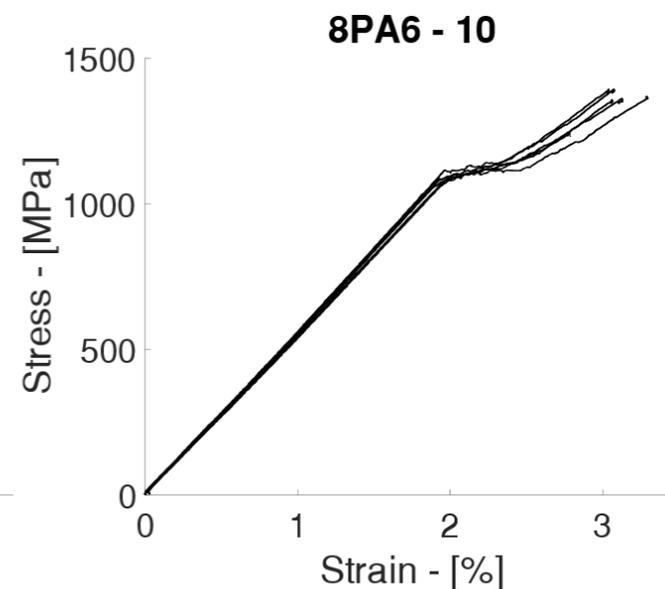
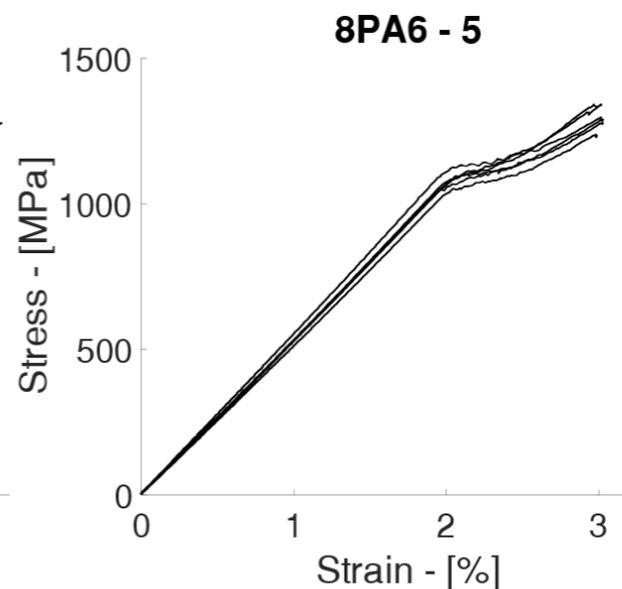
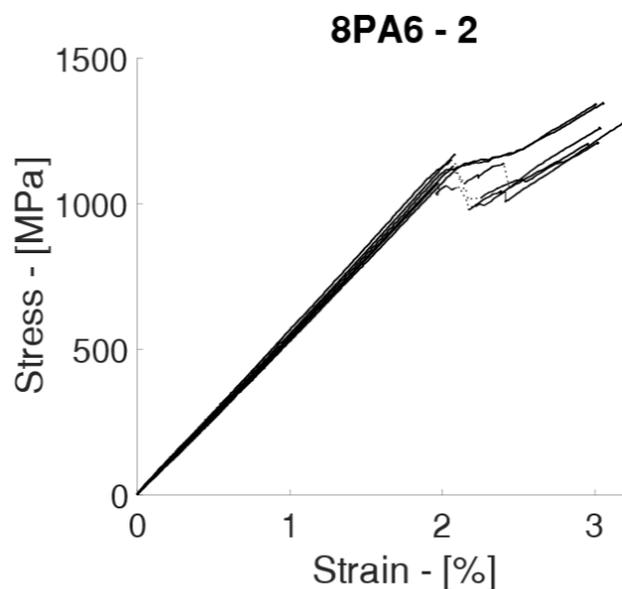
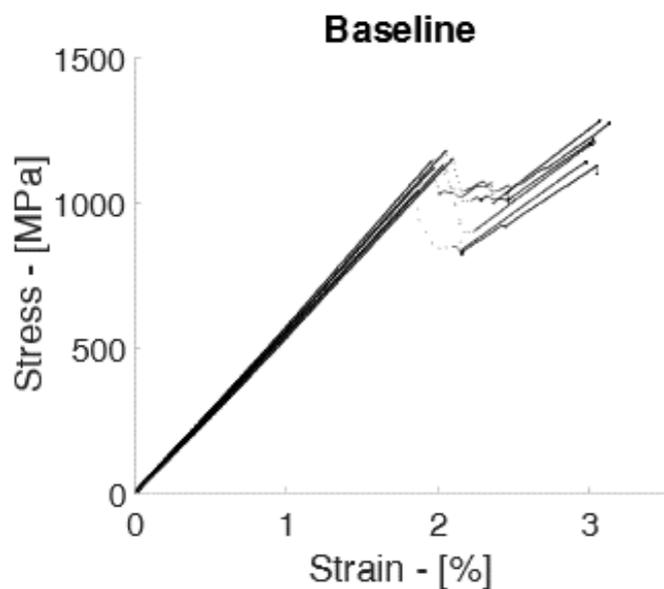
Configurations Lay-up sequence: [G <sub>3</sub> /NL/C/NL/G <sub>3</sub> ] Key: 8 PA6-2: 2 g/m <sup>2</sup> nanofibrous layer from a 8% PA6 solution	Fibre areal densities of the constituent plies	Measured thickness <i>h</i>
	[g/m <sup>2</sup> ]	[mm]
<b>Baseline</b>	[190 <sub>3</sub> /0/100/0/190 <sub>3</sub> ]	1.08 (1.9)
<b>8PA6-2</b>	[190 <sub>3</sub> /2/100/2/190 <sub>3</sub> ]	1.09 (2.1)
<b>8PA6-2+RF</b>	[190 <sub>3</sub> /2+34/100/2+34/190 <sub>3</sub> ]	1.14 (2.1)
<b>8PA6-5</b>	[190 <sub>3</sub> /5/100/5/190 <sub>3</sub> ]	1.11 (2.0)
<b>8PA6-10</b>	[190 <sub>3</sub> /10/100/10/190 <sub>3</sub> ]	1.11 (1.9)
<b>8PA6-5+5</b>	[190 <sub>3</sub> /5+5/100/5+5/190 <sub>3</sub> ]	1.07 (2.1)
<b>8PA6-10+10</b>	[190 <sub>3</sub> /10+10/100/10+10/190 <sub>3</sub> ]	1.10 (2.8)
<b>15PA6-2</b>	[190 <sub>3</sub> /2/100/2/190 <sub>3</sub> ]	1.09 (2.2)
<b>15PA6-5</b>	[190 <sub>3</sub> /5/100/5/190 <sub>3</sub> ]	1.10 (2.2)
<b>15PA6-10</b>	[190 <sub>3</sub> /10/100/10/190 <sub>3</sub> ]	1.10 (1.9)

NL- Nanofibrous layer  
C- Carbon/epoxy  
G- Glass/epoxy  
RF- 34 g/m<sup>2</sup> epoxy film

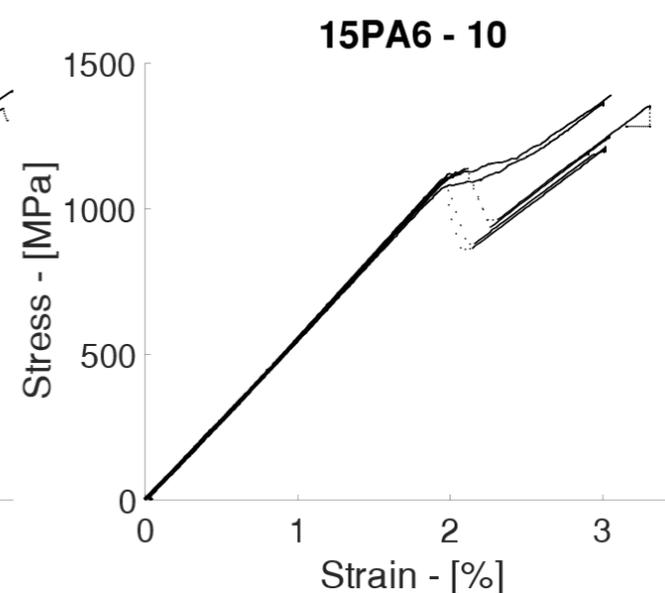
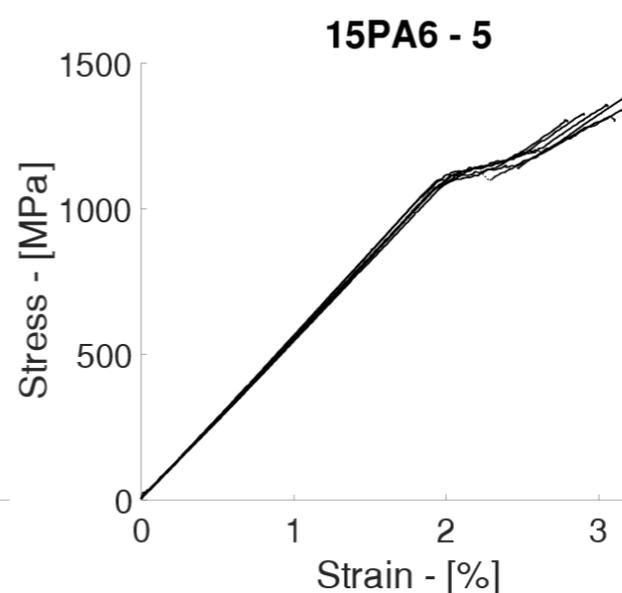
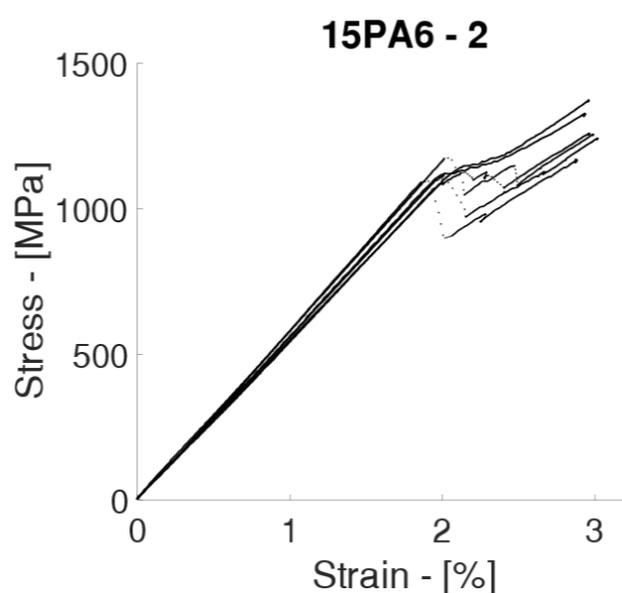
# Quasi-static uniaxial tensile test setup



# Test results- Stress-strain response

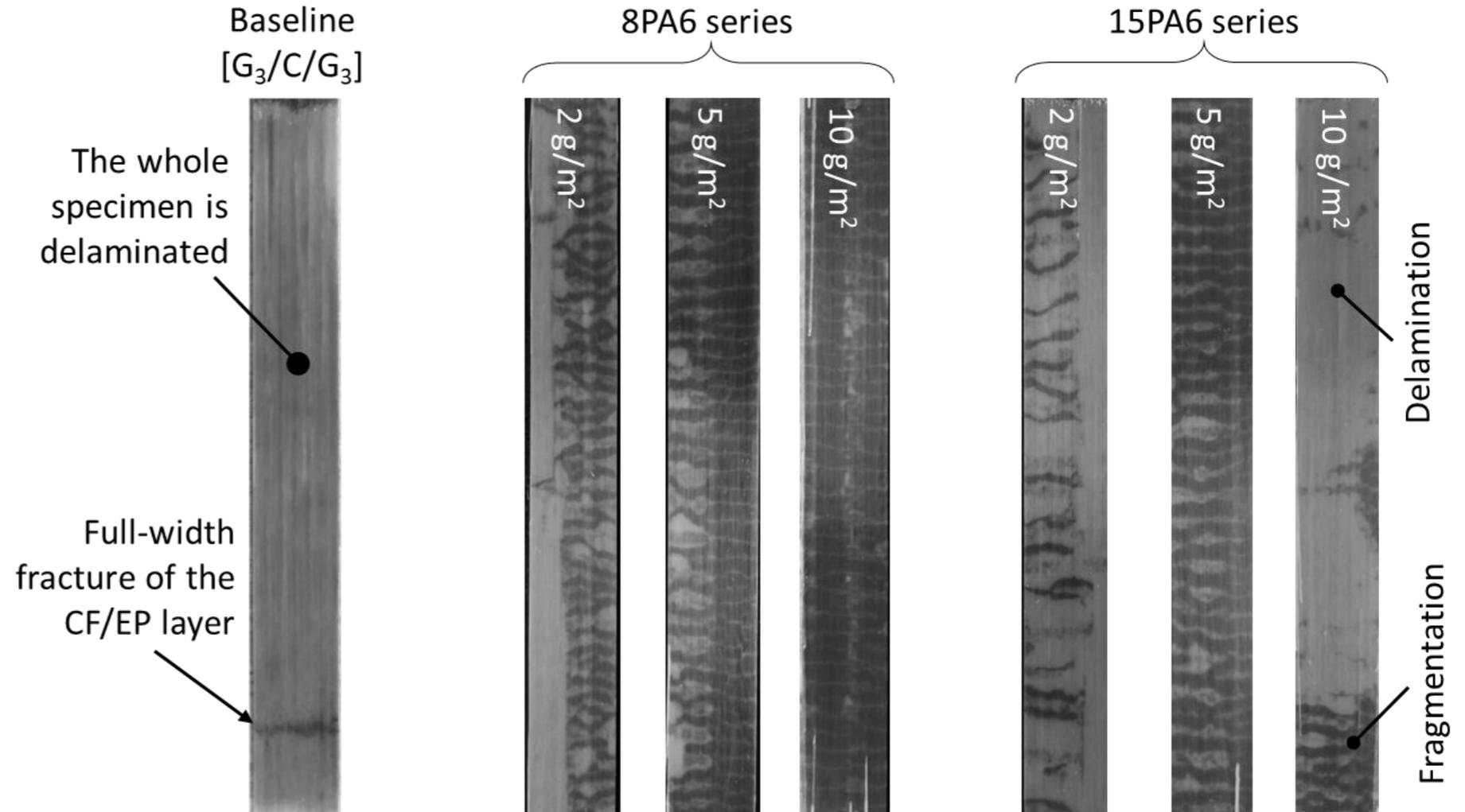


- Pseudo ductility correlates with NL areal weight for the 8 PA6 series
- Different response for different NLs
- Probably due to different structure



# Test results- Damage modes

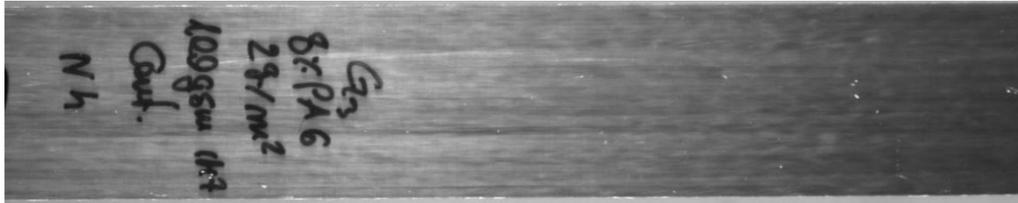
- All series performed much better than baseline
- Mixed delamination and fragmentation for the non-pseudo-ductile series
- Close to borderline



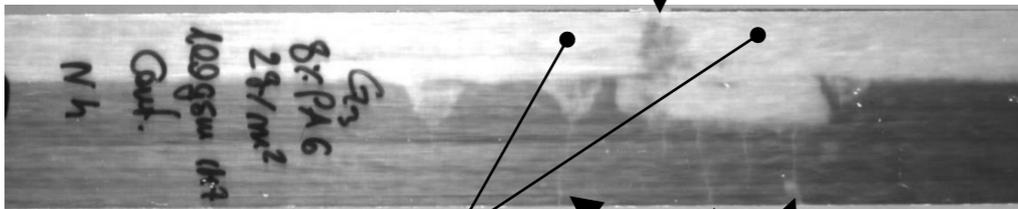
# Test results- Damage sequence

## Delamination + fragmentation (8PA6-2)

1.92% strain (no damage)



1.93% strain



First Fracture of the CF/EP layer

Sudden spread of delamination

Fragmentation

2.8% strain



Damage saturation

Delamination around fragments

## Fragmentation (8PA6-10)

1.97% strain



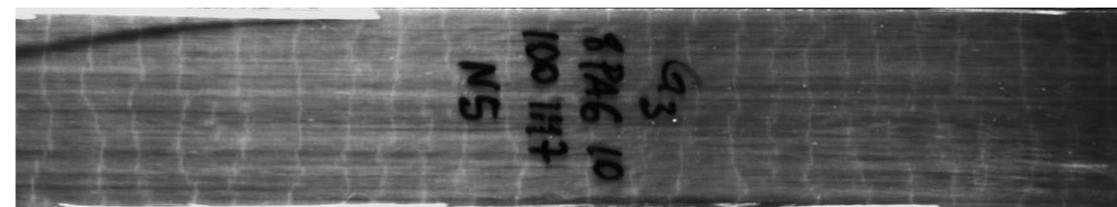
First fracture of the CF/EP layer

2.1% strain



Progression of fragmentation

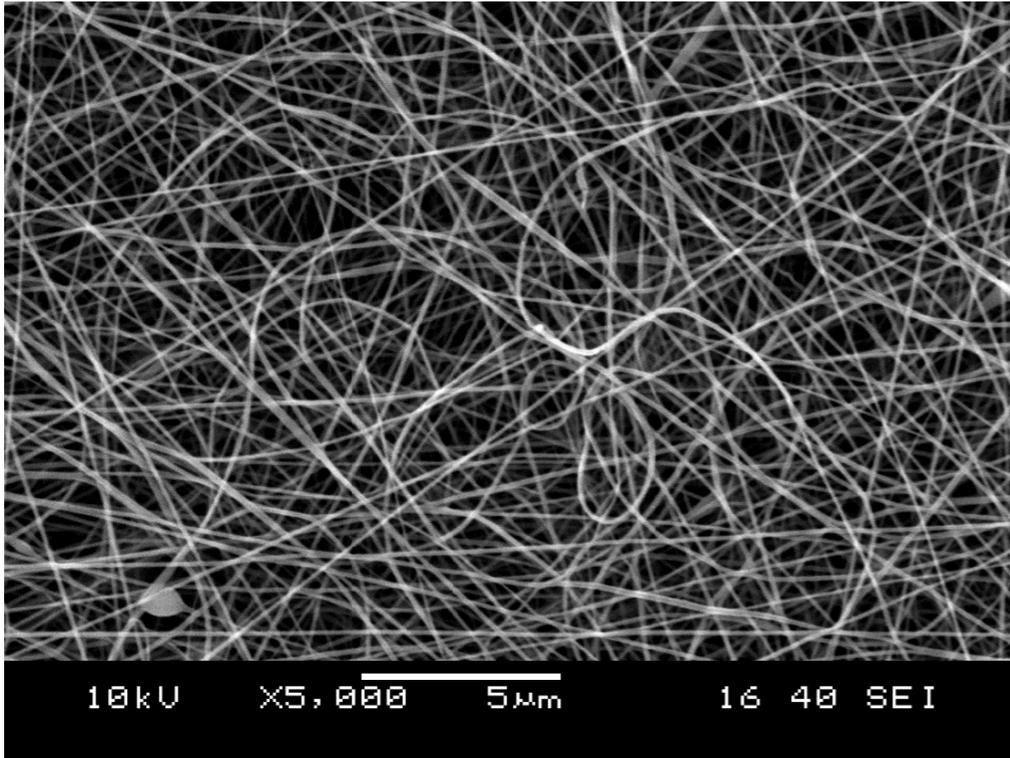
2.8% strain



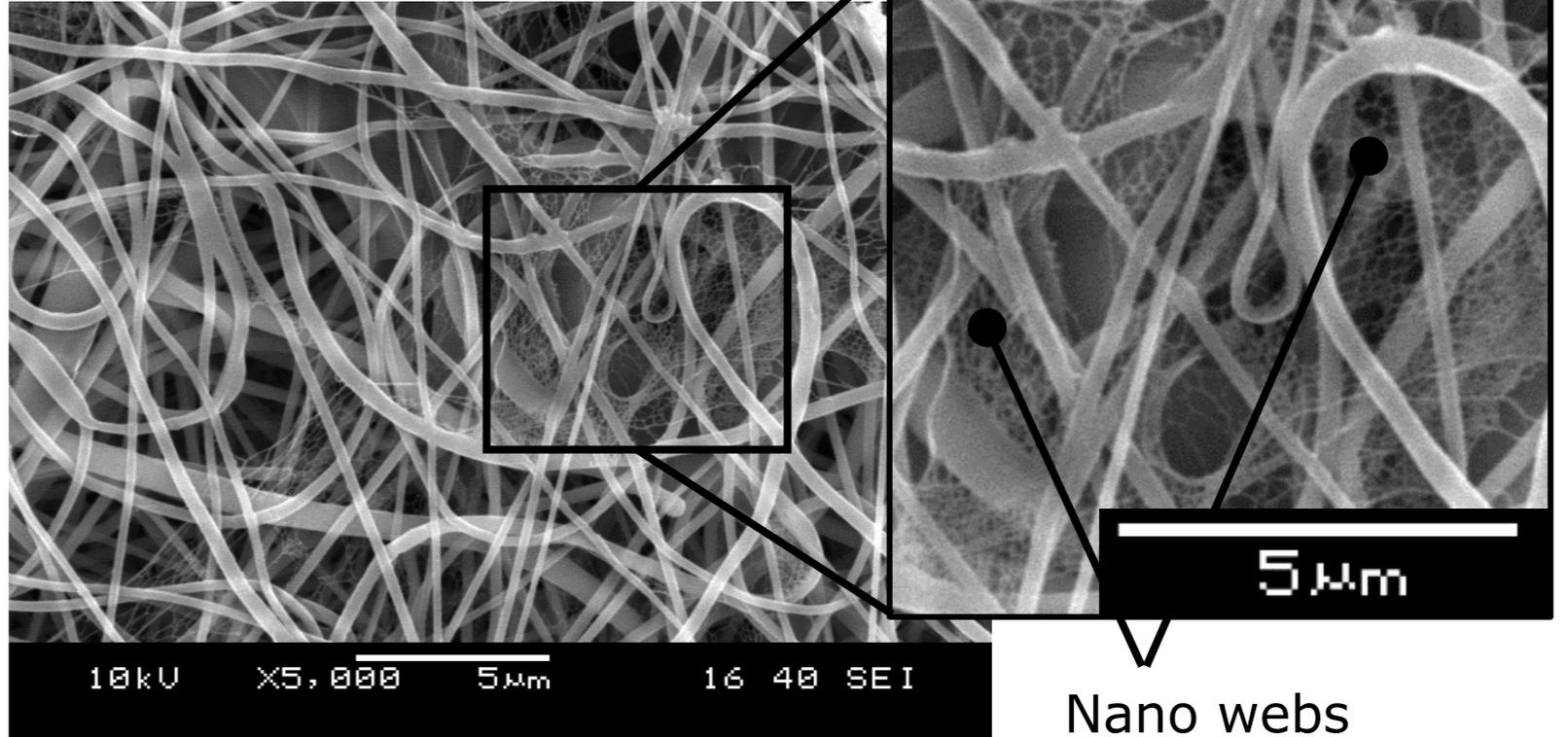
Saturation of fragmentation

# Test results- NL structure

**8PA6**

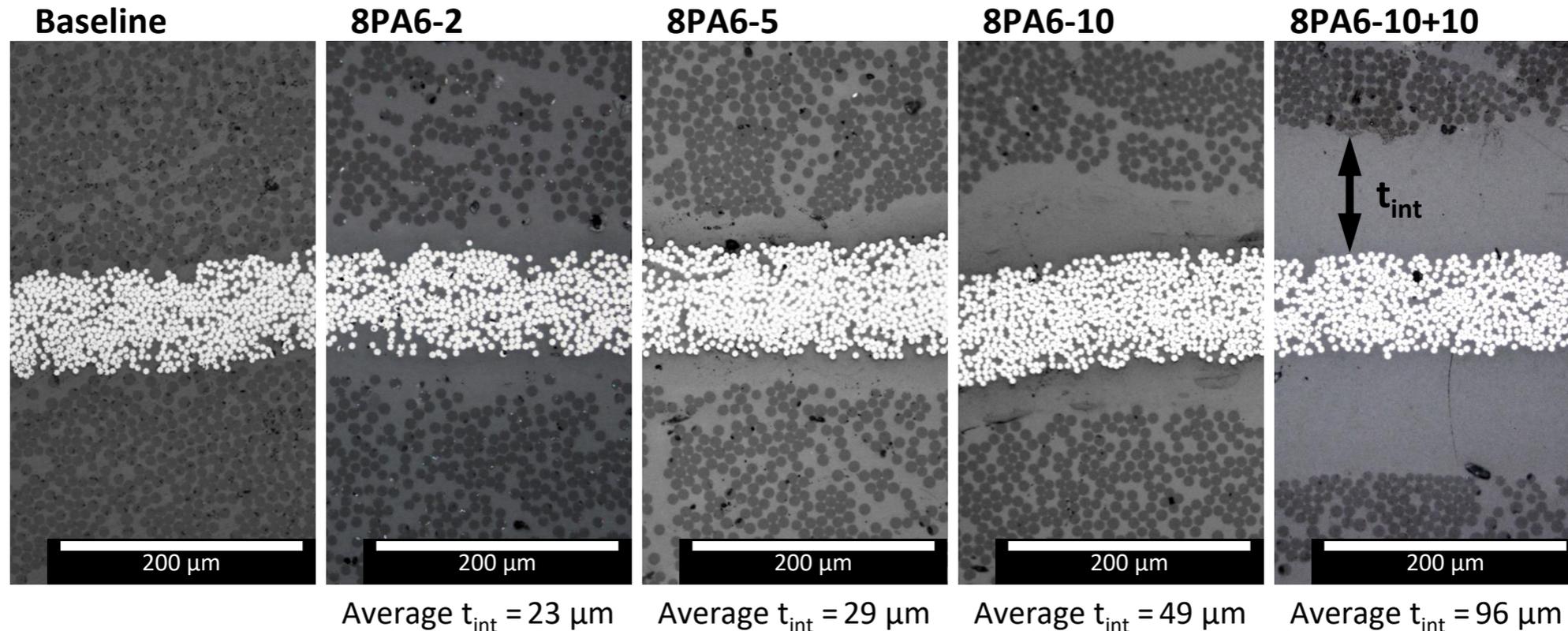


**15PA6**

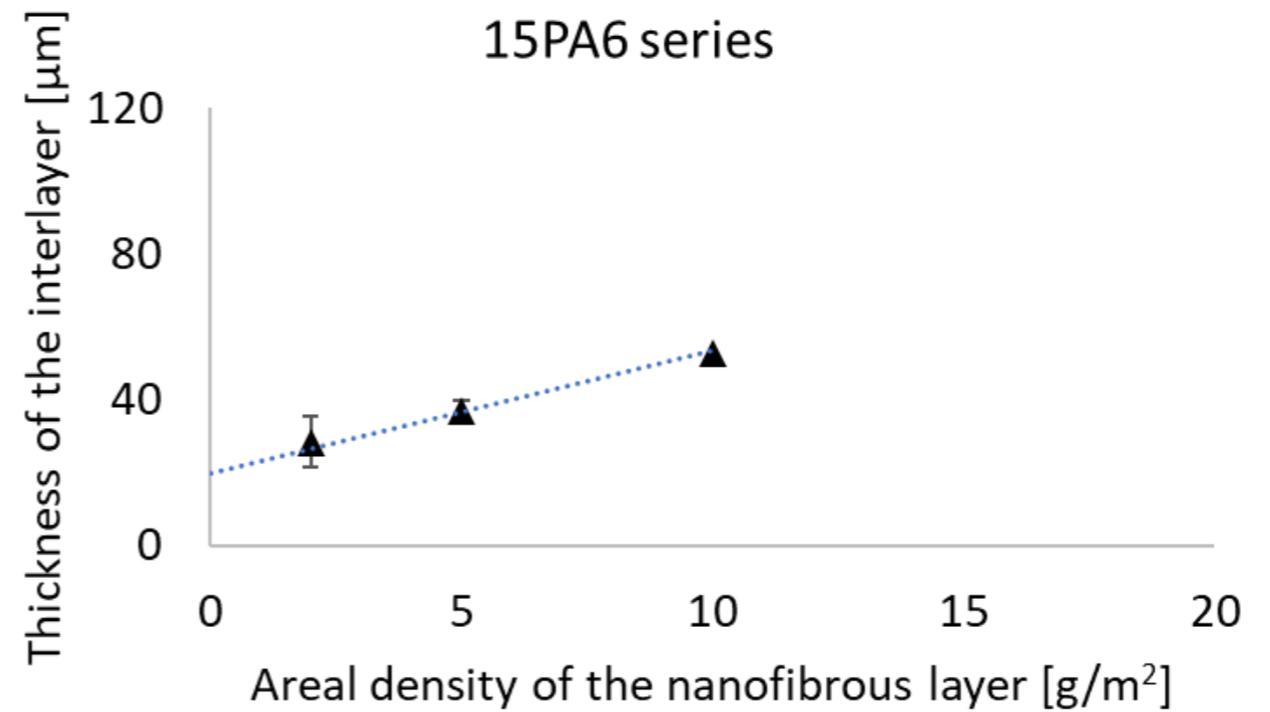
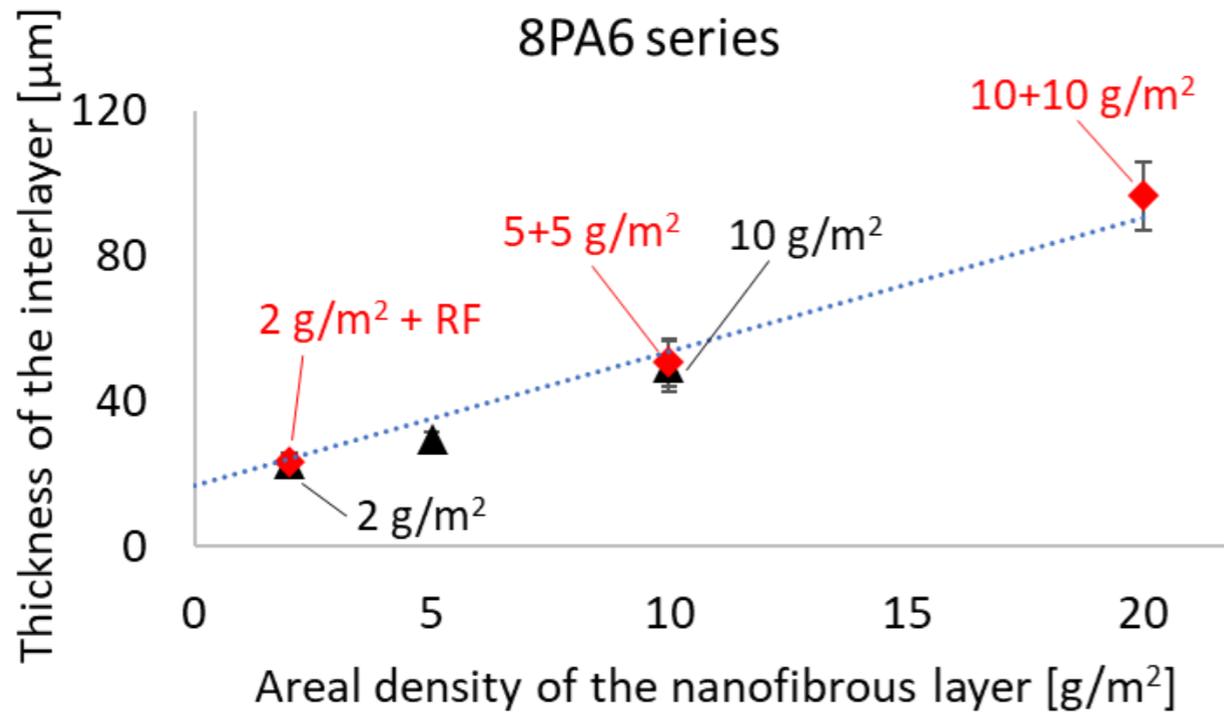


- Different diameters for different PA6 concentrations
- Nano webs for 15PA6
- Possibly different impregnation properties

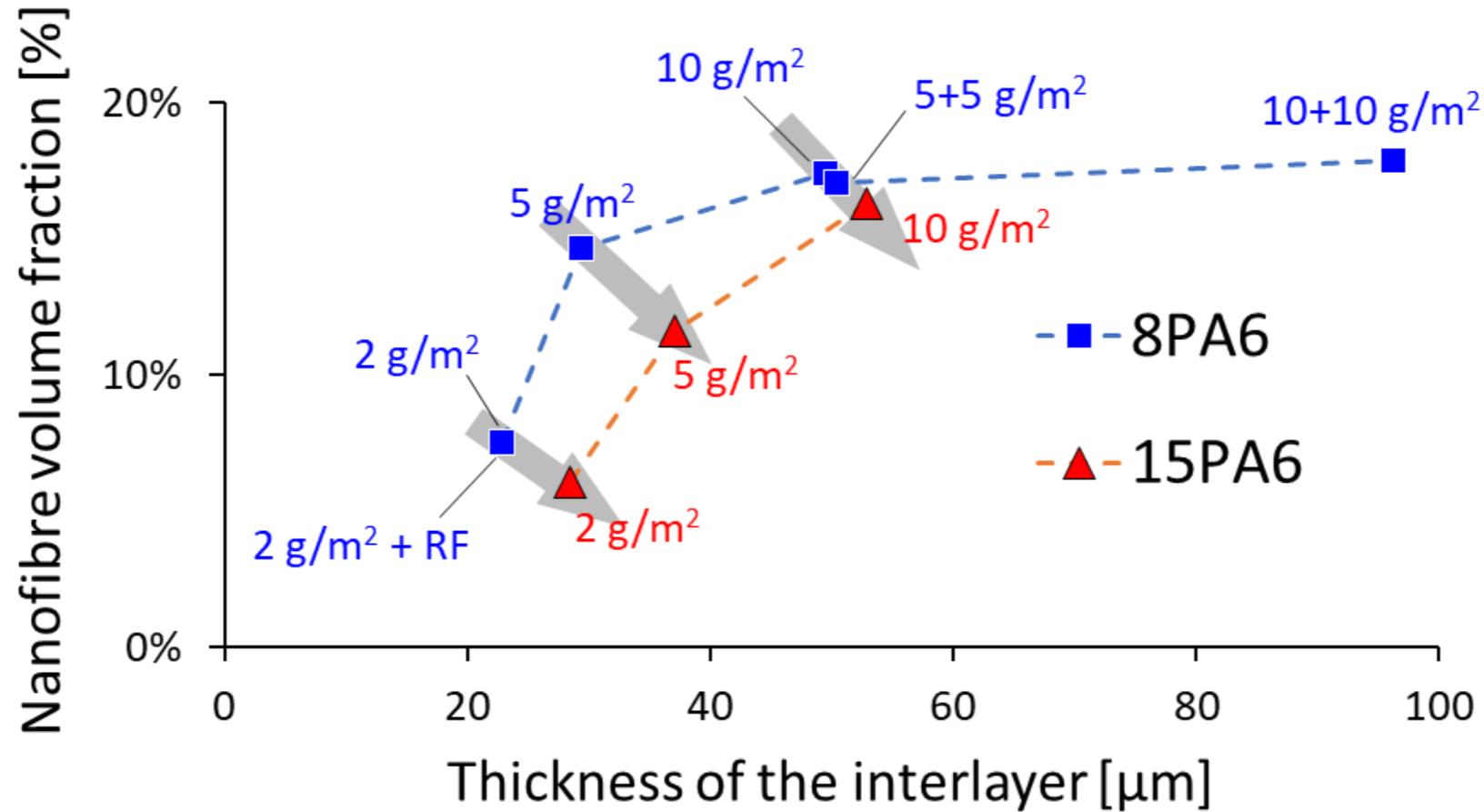
# Test results- Thickness of the interlayer



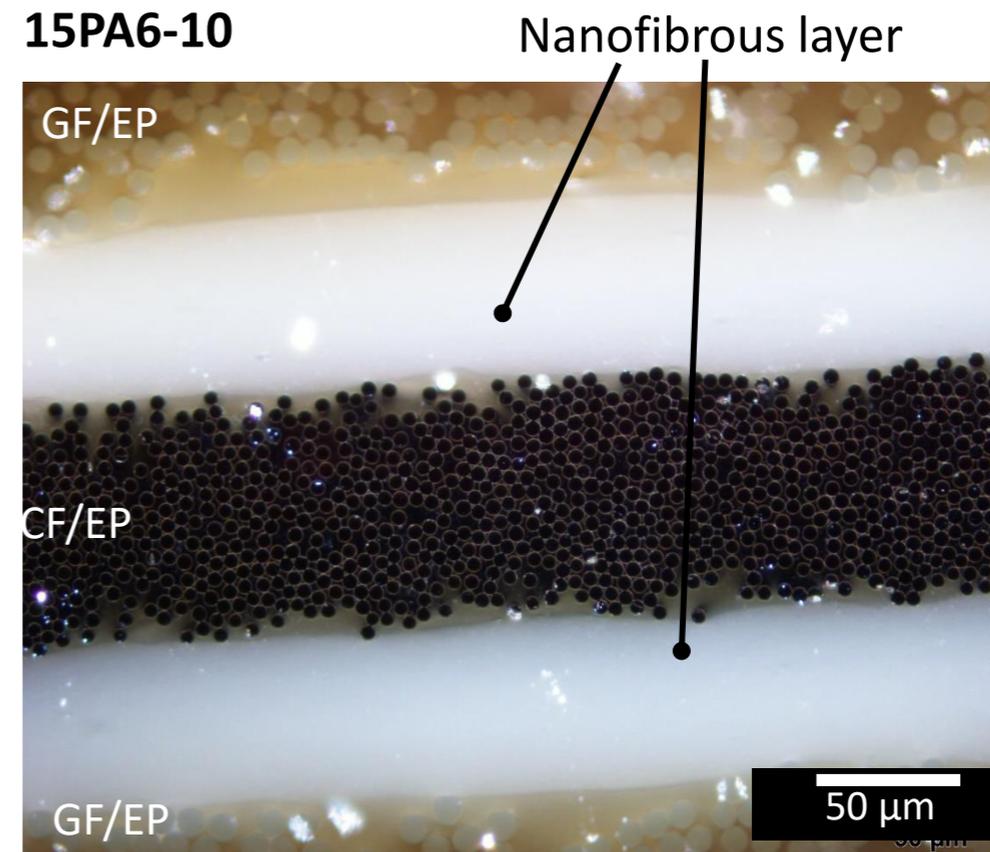
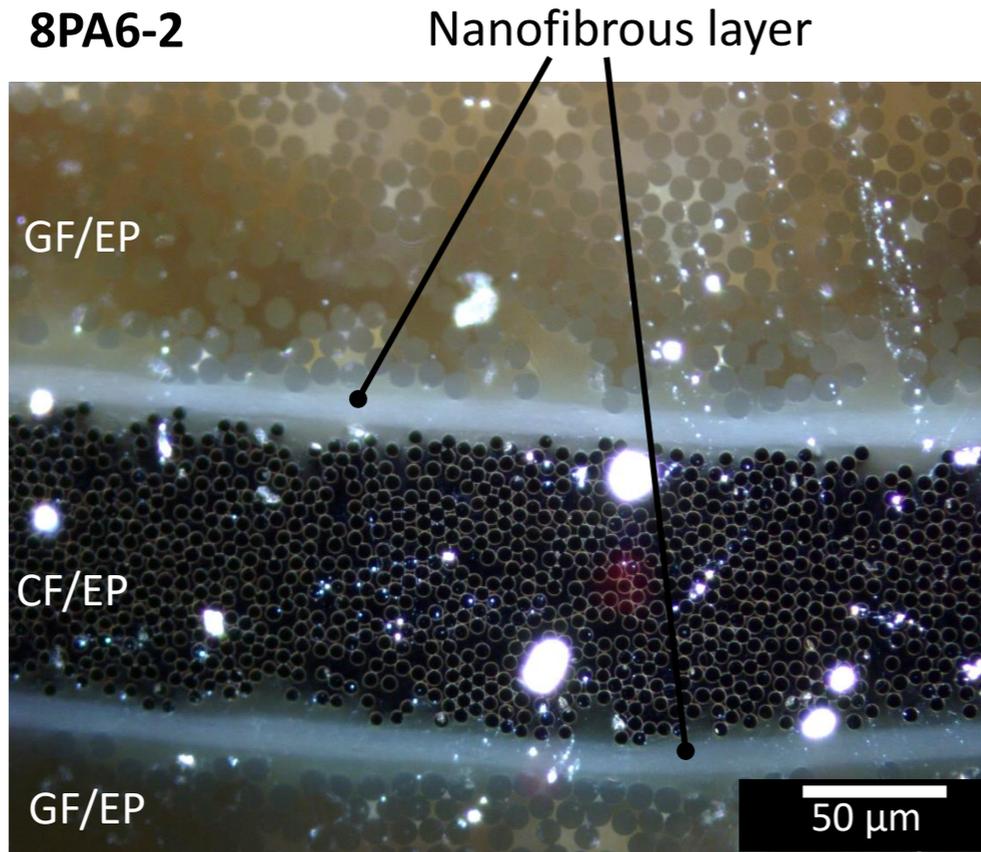
# Test results- Thickness of the interlayer



# Test results- Nanofibre volume fraction in the interlayer



# Results- Cross section microscopy

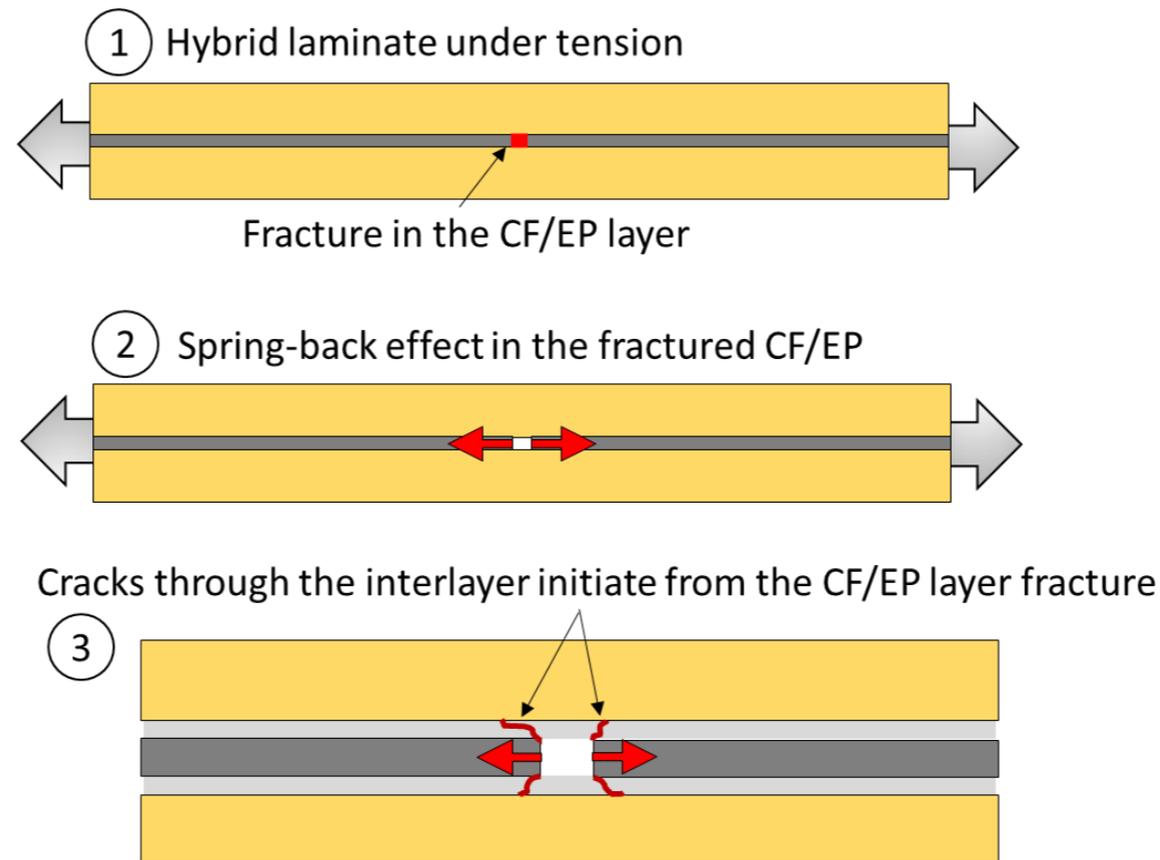


- NLs are well infiltrated with epoxy and form interlayers between the composite layers
- Interlayer thickness is well controlled by the NL areal weight

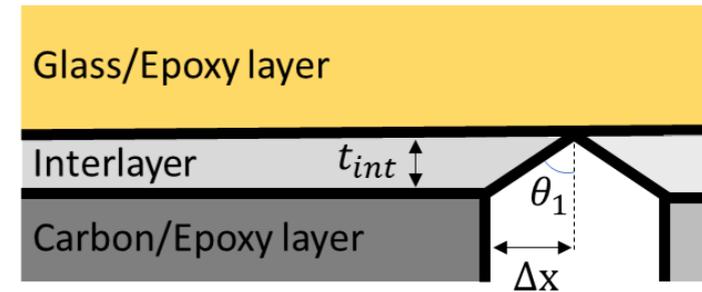
# Anticipated toughening mechanism

- Thick interlayer goes under proportionally lower shear strain
- Can accommodate higher displacements across the interface
- Can extend the damage process zone
- Expected to knock down singular shear stress at the delamination crack tip

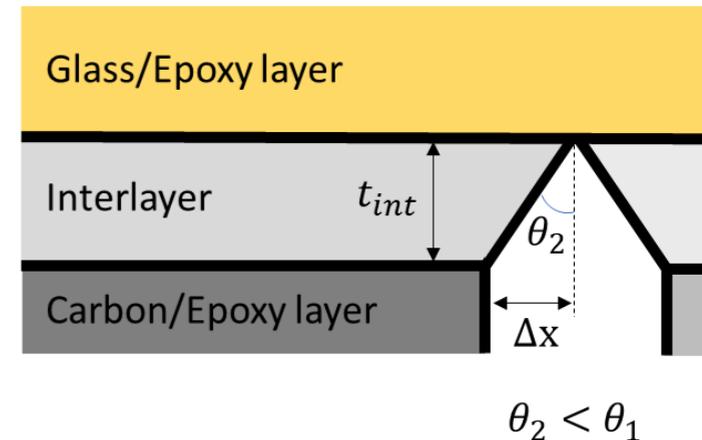
## Initiation of delamination in hybrid samples



## a) Thin interlayer

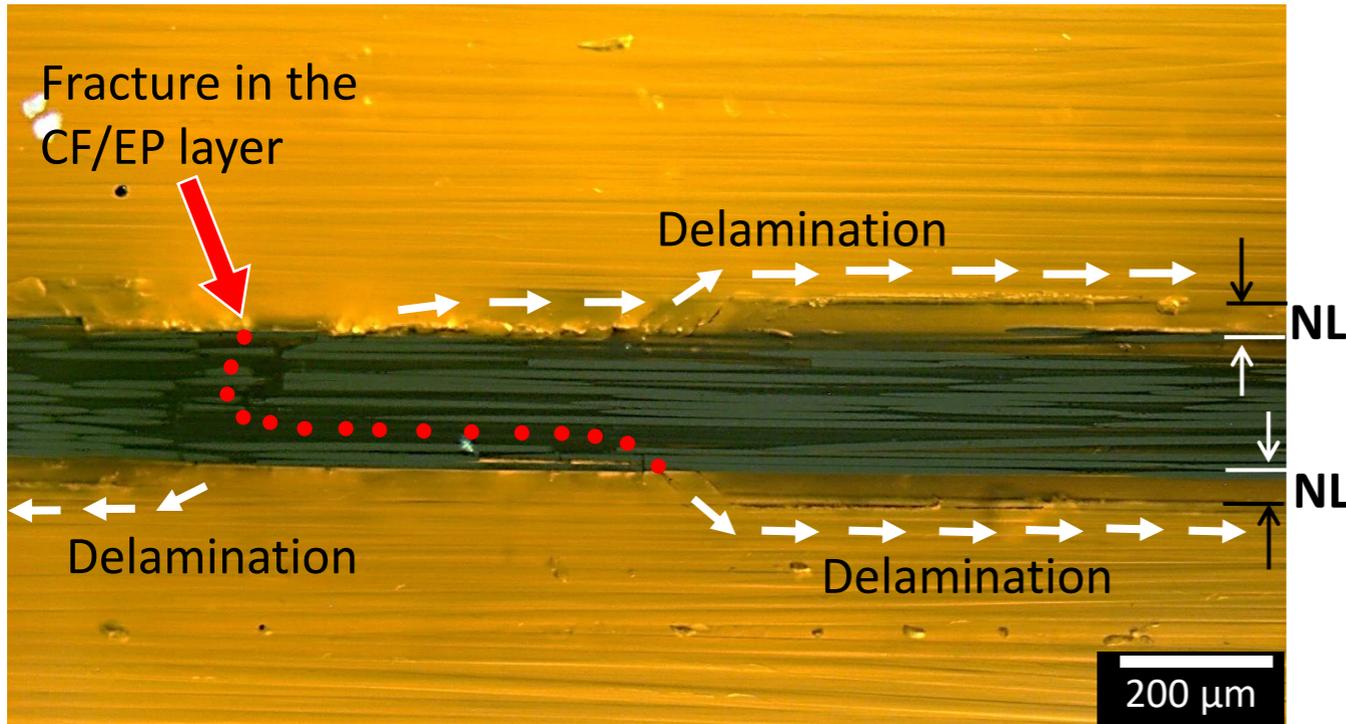


## b) Thick interlayer

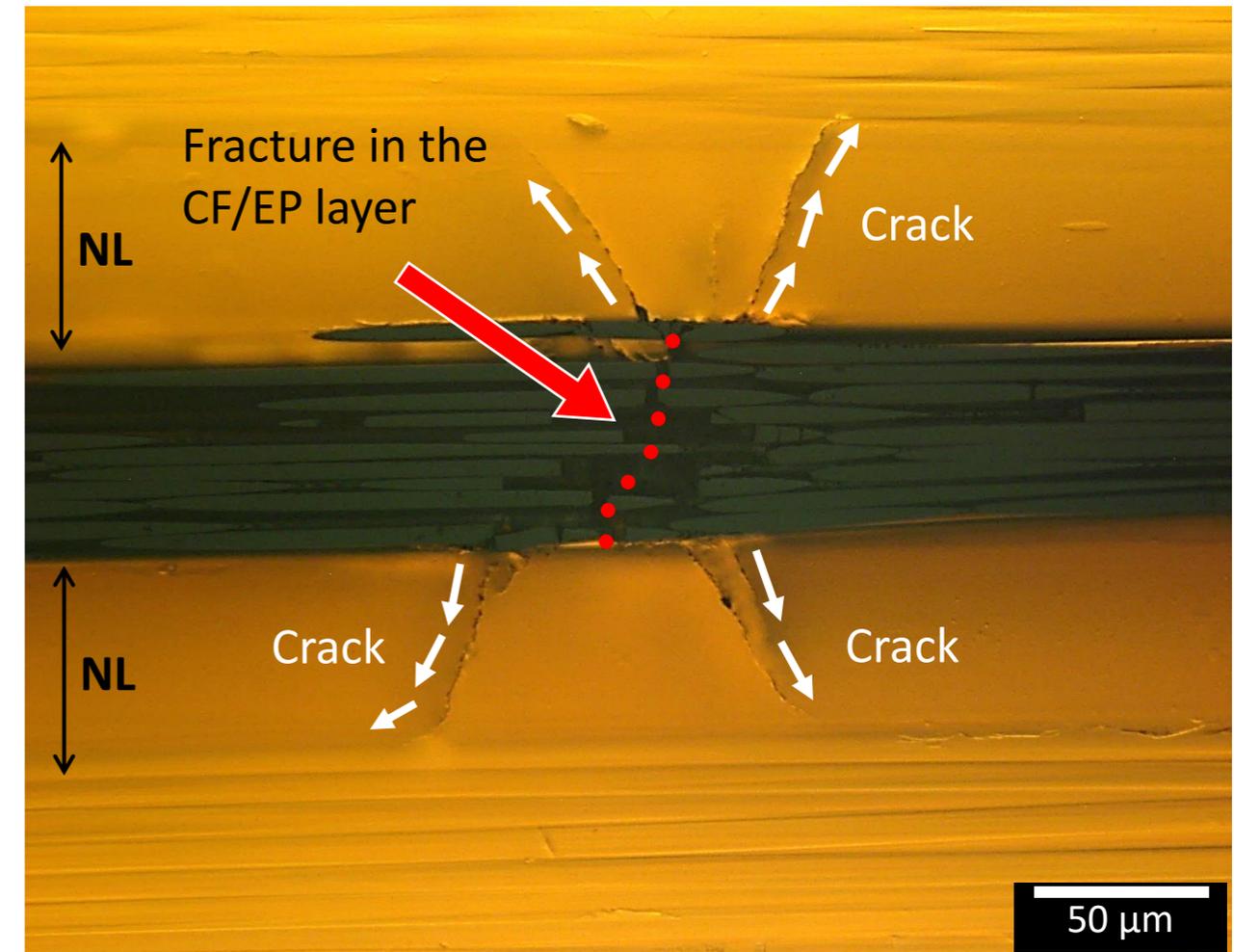


# Results- Longitudinal section microscopy

8PA6-2

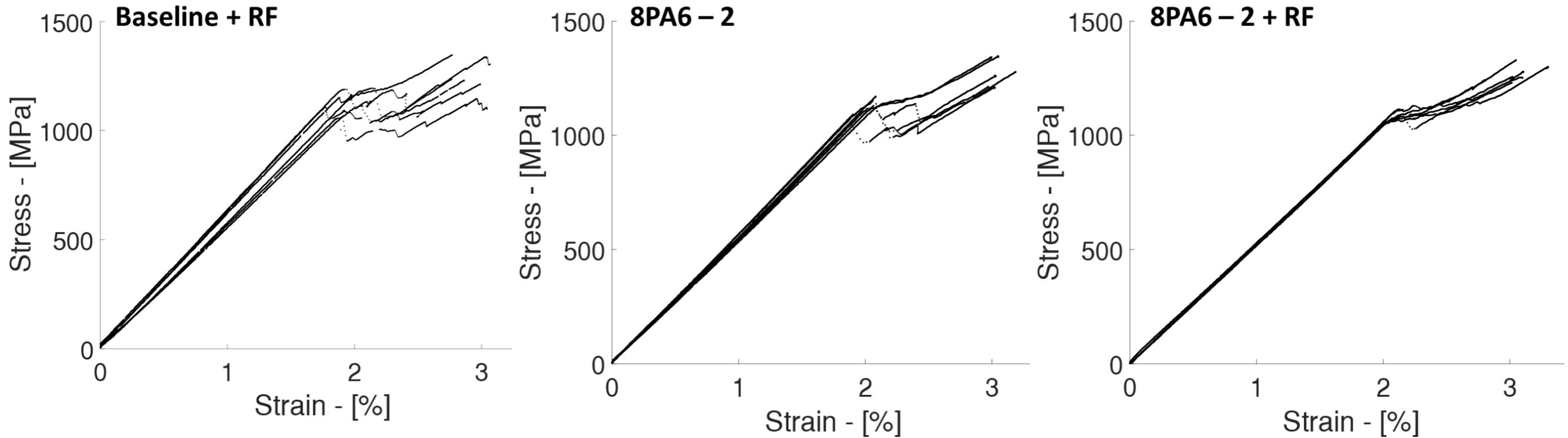


8PA6-10



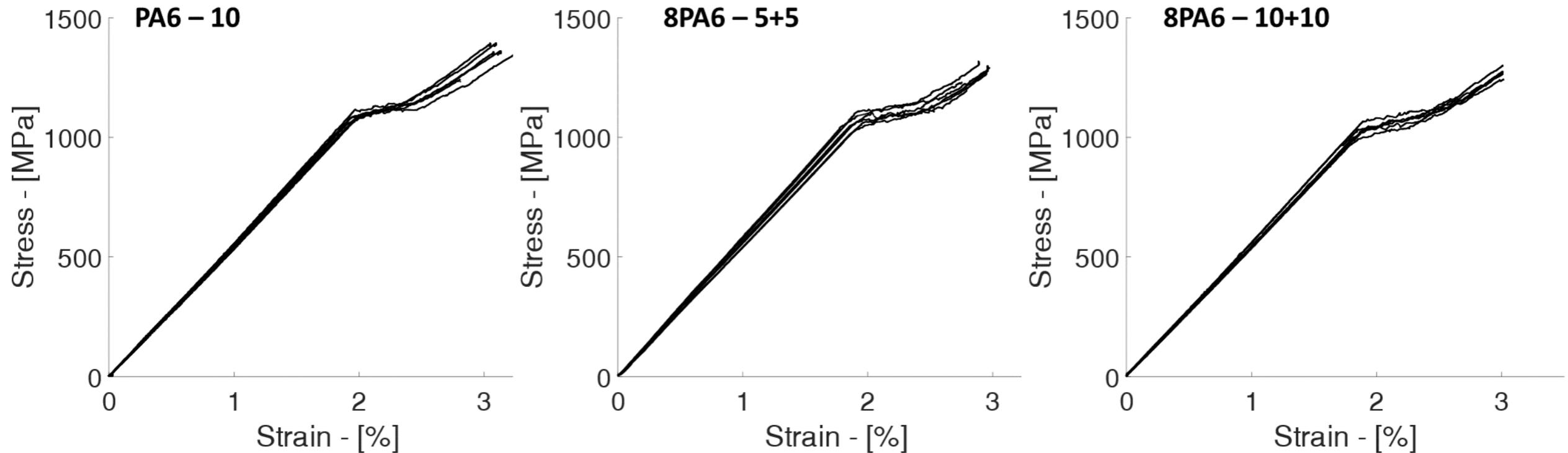
- Thin interlayer cannot suppress delamination completely
- Thick interlayer arrests delamination cracks

# Test results- Effect of extra epoxy film



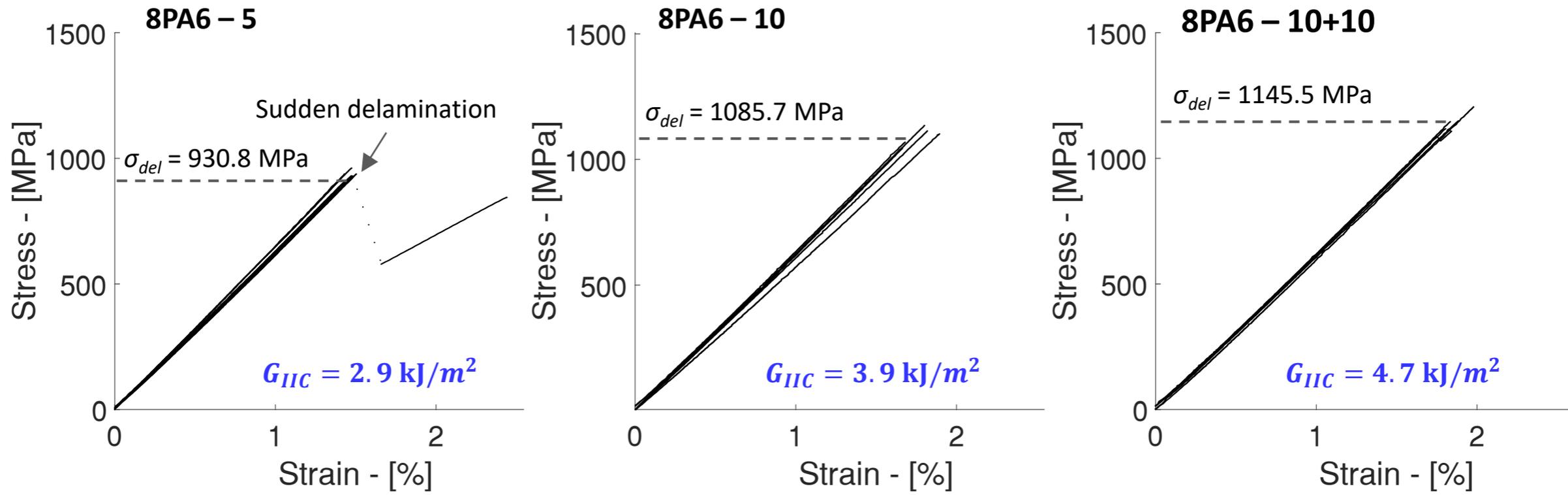
- Possible synergy between NL and epoxy film stabilising borderline response

# Test results- Effect of stacking multiple NLs



- Stacked NLs with the same areal weight perform similarly
- Thicker NL stack improves pseudo-ductility and mode II fracture toughness

# Test results- Mode II fracture toughness improvement



- Significant improvement of  $G_{IIC}$  from 1.8 kJ/m<sup>2</sup> (baseline) up to 4.7 kJ/m<sup>2</sup>
- Saturation is expected above 10+10 g/m<sup>2</sup> NL areal weight

# Results summary- 8PA6 series

Configuration	Interleaved configurations [G <sub>3</sub> /NL/C/NL/G <sub>3</sub> ] (NL refers to the Nanofibrous Layer)						
	Baseline	8PA6-2	8PA6-2+RF	8PA6-5	8PA6-10	8PA6-5+5	8PA6-10+10
Measured thickness [mm]	1.08 (1.9)	1.09 (2.1)	1.14 (2.1)	1.11 (2.0)	1.11 (1.9)	1.07 (2.1)	1.10 (2.8)
Elastic modulus [GPa]	58.0 (2.7)	56.6 (2.6)	54.5 (2.0)	55.7 (3.8)	56.8 (1.5)	56.5 (4.1)	54.8 (4.2)
Knee-point stress [MPa]	1134 <sup>(a)</sup> (3.7)	1124 (3.0)	1070 (2.9)	1099 (3.9)	1096 (2.1)	1074 (3.9)	1014 (4.1)
Knee-point strain [%]	1.99 <sup>(a)</sup> (3.4)	2.02 (3.3)	2.02 (3.4)	2.02 (2.7)	1.97 (1.3)	1.93 (1.9)	1.87 (2.2)
Strain energy release rate @knee-point [kJ/m <sup>2</sup> ]	2.4 (9.3)	2.5 (8.3)	2.4 (6.9)	2.4 (10.2)	2.4 (6.4)	2.1 (11.1)	2.0 (11.9)
Mode II interlaminar fracture toughness <sup>(b)</sup> [kJ/m <sup>2</sup> ]	1.8 (7.4)	- -	- -	2.9 (8.1)	3.9 (6.6)	- -	4.7 (7.8)

(a) Evaluated from the load drop in the stress-strain curves of this configuration.

(b) Measured on [G<sub>3</sub>/NL/C<sub>2</sub>/NL/G<sub>3</sub>] laminates with a cut in the middle of the CF/EP layer.

# Results summary- 15PA6 series

Configuration	Interleaved configurations [G <sub>3</sub> /NL/C/NL/G <sub>3</sub> ] (NL refers to the Nanofibrous Layer)			
	Baseline	15PA6-2	15PA6-5	15PA6-10
Measured thickness [mm]	1.08 (1.9)	1.09 (2.2)	1.10 (2.2)	1.10 (1.9)
Elastic modulus [GPa]	58.0 (2.7)	56.8 (3.1)	57.0 (2.7)	57.3 (1.5)
Knee-point stress [MPa]	1134 (3.7)	1122 (2.8)	1113 (2.3)	1108 (2.2)
Knee-point strain [%]	1.99 <sup>(a)</sup> (3.4)	1.97 (4.0)	1.96 (2.2)	1.96 (1.6)
Strain energy release rate @knee-point [kJ/m <sup>2</sup> ]	2.4 (9.3)	2.4 (8.0)	2.4 (6.0)	2.4 (6.9)

<sup>(a)</sup> Evaluated from the load drop in the stress-strain curves of this configuration.

# Conclusions

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- Pseudo-ductility was achieved in a range of interlayer hybrid configurations by inserting nanofibrous layers (NL) between the GF/EP and CF/EP composite layers.
- Nanofibres electrospun from 8% PA6 concentration solutions performed better than the 15% version.
- The thickness of the interlayers between the composite layers were precisely controlled by the areal weight of the NLs.
- Thick interlayers between the composite plies suppressed delamination because of lower shear strains experienced at given displacements across the interface. Interlaminar cracks were arrested in case of NLs with 5 g/m<sup>2</sup> areal weight and above. Saturation is expected at NL areal weights higher than 20 g/m<sup>2</sup>.
- The  $G_{IIC}$  was significantly increasing with the areal density of the nanofibrous interleaves in the case of 8PA6 NLs, i.e. from 1.8 kJ/m<sup>2</sup> (baseline) up to 4.7 kJ/m<sup>2</sup> for the 8PA6-10+10 configuration containing NLs with a total areal density of 20 g/m<sup>2</sup>.

Further details:

Marino S. G., Kuželová Košťáková E. , Czél G. : Development of pseudo-ductile interlayer hybrid composites of standard thickness plies by interleaving polyamide 6 nanofibrous layers. Composites Science and Technology, **234**, 109924/1-109924/14 (2023)

[10.1016/j.compscitech.2023.109924](https://doi.org/10.1016/j.compscitech.2023.109924)

# Acknowledgement



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Sciences

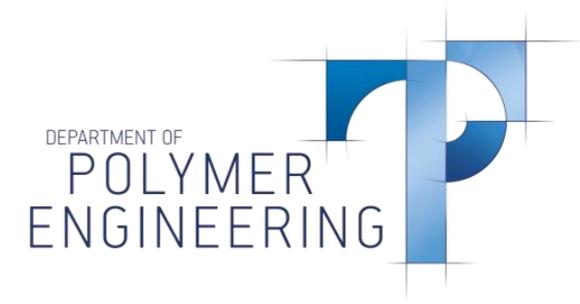
1825

Special thanks to **Dr. Eva Kuželová Košťáková** for providing the nanofibrous layers for our study. The research leading to the presented results has been performed within the framework of the HyFiSyn project and has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 765881. The research was also supported by the National Research, Development and Innovation Office (NRDI, Hungary) through grant OTKA FK 131882. Gergely Czél is grateful for funding through the János Bolyai Research Fellowship Programme of the Hungarian Academy of Sciences. The work was supported by the ÚNKP-22-5-BME-323 New National Excellence Program of the Ministry for Culture and Innovation from the source of the National Research, Development and Innovation Fund.

## Thank you for your attention!



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Queen’s University Belfast, UK, 30 July-4 August 2023