Influence of Moisture Content on the Strength of Thermoplastic Reinforced Resins at Cryogenic Temperatures

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SUSMATx, Girona 06.09.2024

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NOVEL LOW-PRESSURE CRYOGENIC LIQUID HYDROGEN STORAGE FOR AVIATION

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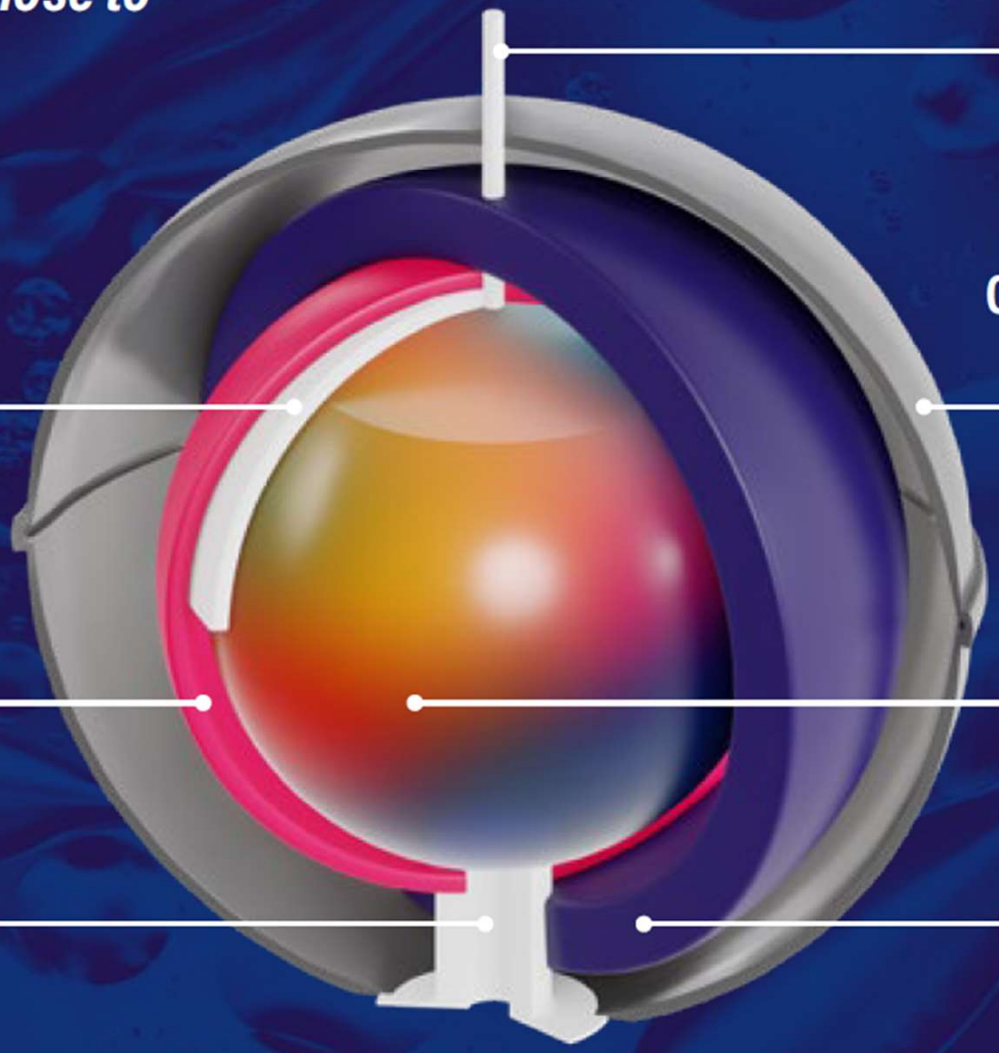
A 95 kg tank prototype will be tested in environmental conditions close to real operating ones



Inner thermoplastic composite tank

Specifications:
- LH2 storage at 20K
- Inner tank does not sustain pressure (external tank)
- There are only thermal stresses
- Additive manufacturing (3D Printing)
- Thermoplastic resin
- Avoid leakage

Motivation:
- During service, the material will absorb moisture
- How this moisture affects to the material strength?
- Does it depend on the porosity of the material?

Objective:
- Which is the influence of moisture content on the strength of the material?

Methodology

Material candidates

Porosity

Conditioning

Testing

Methodology

Material candidates

Porosity

Conditioning

Testing

PA11_BESNO_CNT4: PA11 Rilsan® BESNO 23 TL4 CC resin + 4%w CNT

PA11_BESNO_BF20: PA11 Rilsan® BESNO 23 TL4 CC resin + 20%w Basalt fibre

PA11_CLEARG820_BF20: PA11 Rilsan® CLEAR G820 + 20%w Basalt fibre

PEKK_BF20: PEKK KEPSAN® 6003 + 20%w Basalt fibre

ARKEMA — Resin provider

CANOE LE CENTRE TECHNOLOGIQUE NOUVELLE AQUITAINE COMPOSITES & MATÉRIAUX AVANCÉS — Filament manufacturing

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Methodology

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Porosity

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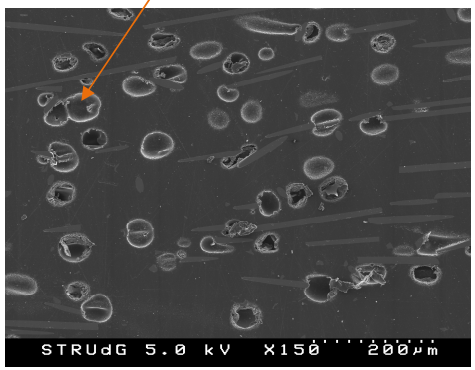
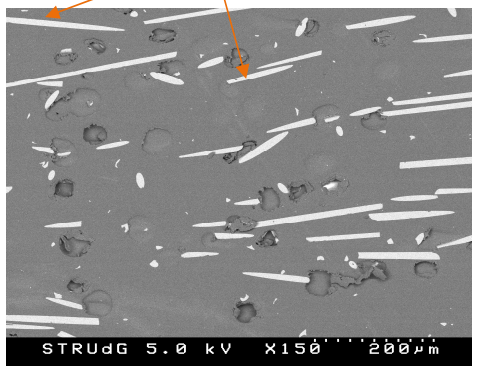
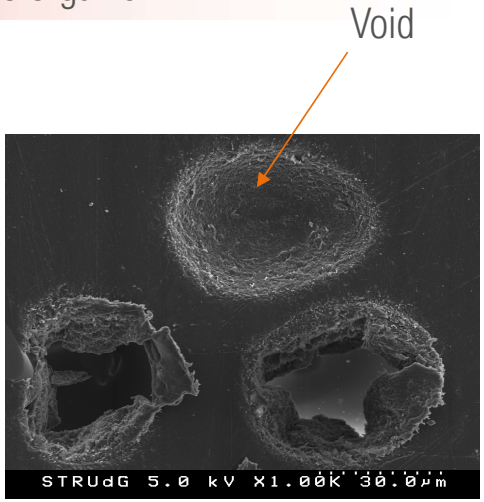
Acid digestion (EN 2564)
The resin eliminated by immersing the specimen in pure H2S04 at 160ºC. Then the fibres are cleaned, filtered and dehydrated with acetone 100%. Finally, the fibres are dried at 120ºC.

Calcination (ISO 1172)
The resin is eliminated by calcination at 625ºC during 6 hours

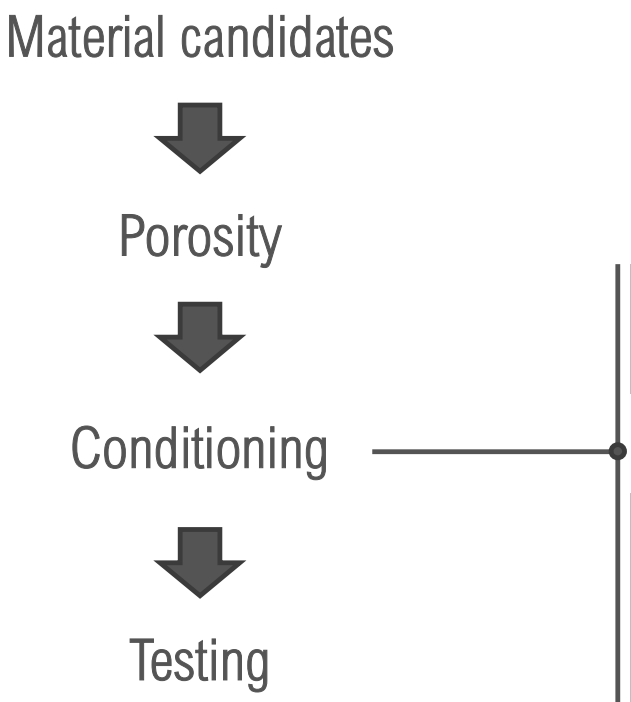
Scanning Electron Microscopy, SEM (no standard)
Only used for porosity content. Useful when reinforcements are organic.







Methodology



Baseline = Conditioning + Test at RT (23ºC):

RTA: Ambient (23ºC/50% RH) + Test

Conditioning + Immersion in LN2 (77K during 10min) + Test at RT (23ºC):

RTLN: Ambient (23ºC/50% RH) + Immersion in LN2 + Test (RTLN = RTA + Immersion)
RTDLN: Dry (80ºC during 8h) + Immersion in LN2 + Test
RTWLN: Wet (70ºC/85% RH) + Immersion in LN2 + Test

Methodology

Material candidates

Porosity

Conditioning

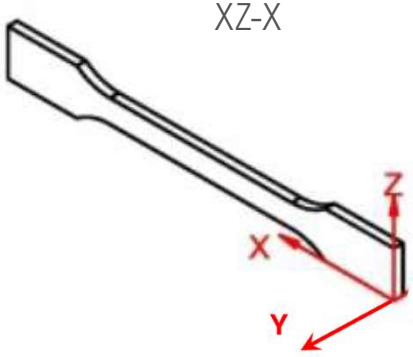
Testing

Test of dog bone specimens:

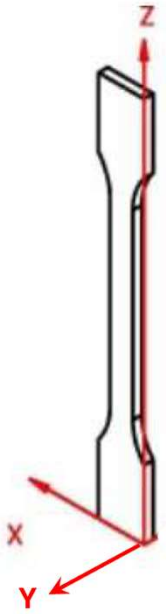
- Printing direction XZ-X
- Perpendicular to the printing direction XZ-Z

Printing of a vertical panel in the XZ plane. X is the printing direction.

XZ-X



XZ-Z



Methodology

Material candidates

↓

Porosity

↓

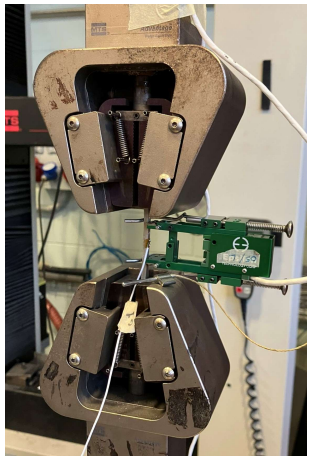
Conditioning

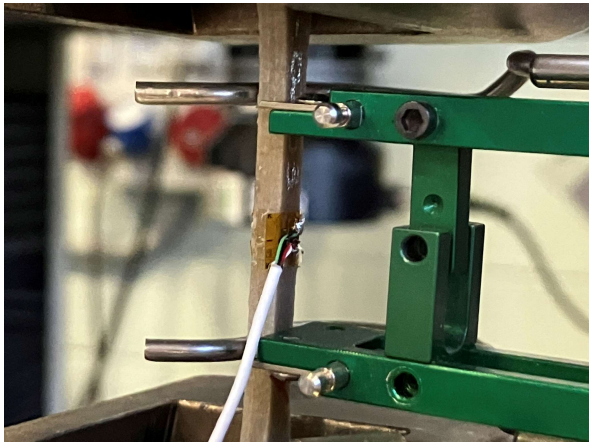
↓

Testing

Test of dog bone specimens:
- Printing direction XZ-X
- Perpendicular to the printing direction XZ-Z

Tensile tests according to ISO527-4 standard
- 1BA specimens:
- Tests at RT
- Before the tests the specimens were LN2 immersed during 10min





Results > Porosity

Material	Fibre weight content (%)	Resin weight content (%)	Porosity content (%)		
			Microscopy	Acid digestion	Calcination
PA11_BESNO_CNT4	-	-	0.2	-	-
PA11_BESNO_BF20	19.1	80.9	0.8	0.4	0.3
PA11_CLEARG820_BF20	19.1	80.9	6.9	5.8	6.1
PEKK_BF20	19.1	80.9	16.3	11	11.1

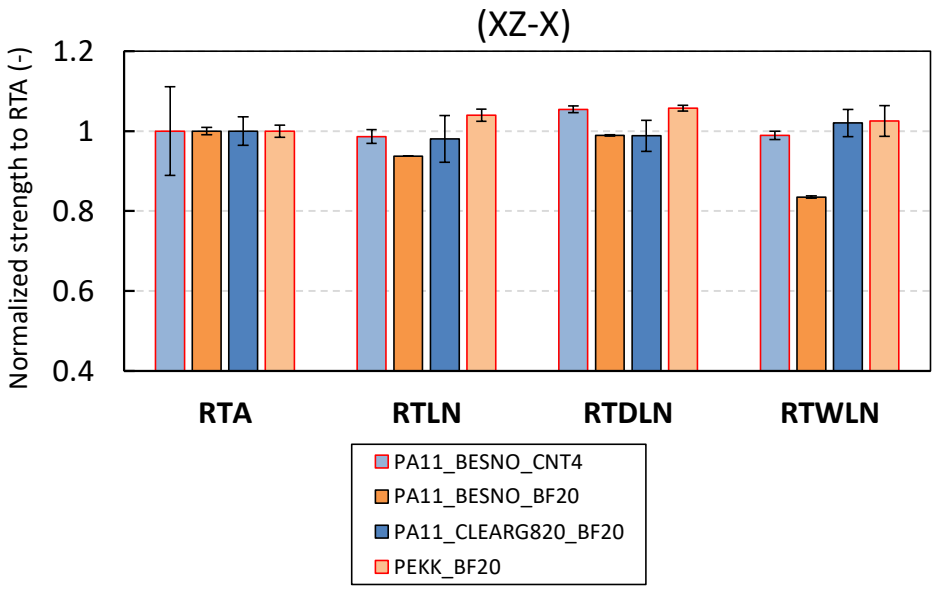
- PEKK: highest values of porosity
- PA11 Besno®: lowest values of porosity
- SEM allows to obtain values of porosity even in organic reinforcements or CNT

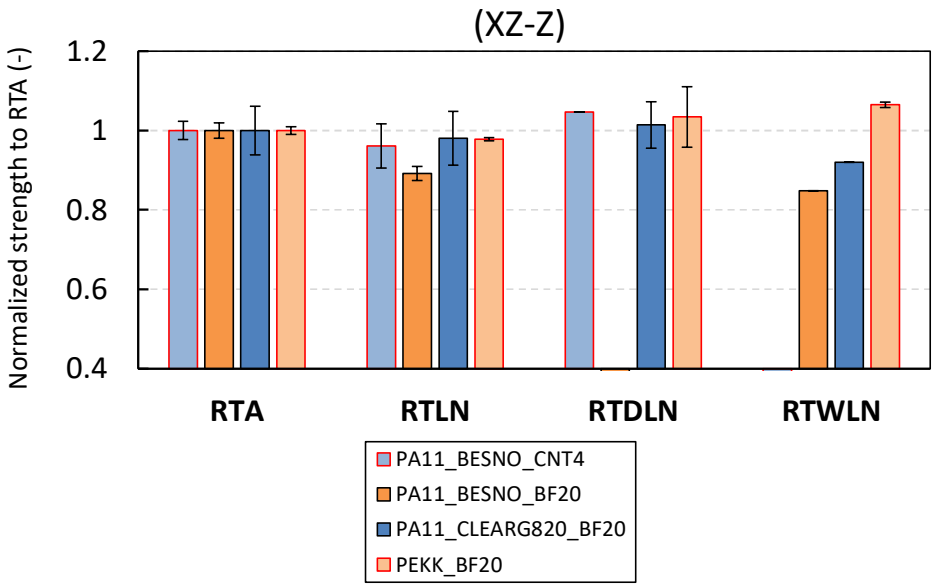
Results > Moisture Content

Material	Moisture content (%w)	
	RTLN / RTA (Ambient)	RTWLN (Wet)
PA11_BESNO_CNT4	0.98	1.56
PA11_BESNO_BF20	0.63	1.36
PA11_CLEARG820_BF20	0.80	1.78
PEKK_BF20	0.33	0.64

RTDLN Dry specimens: 0.00% of moisture

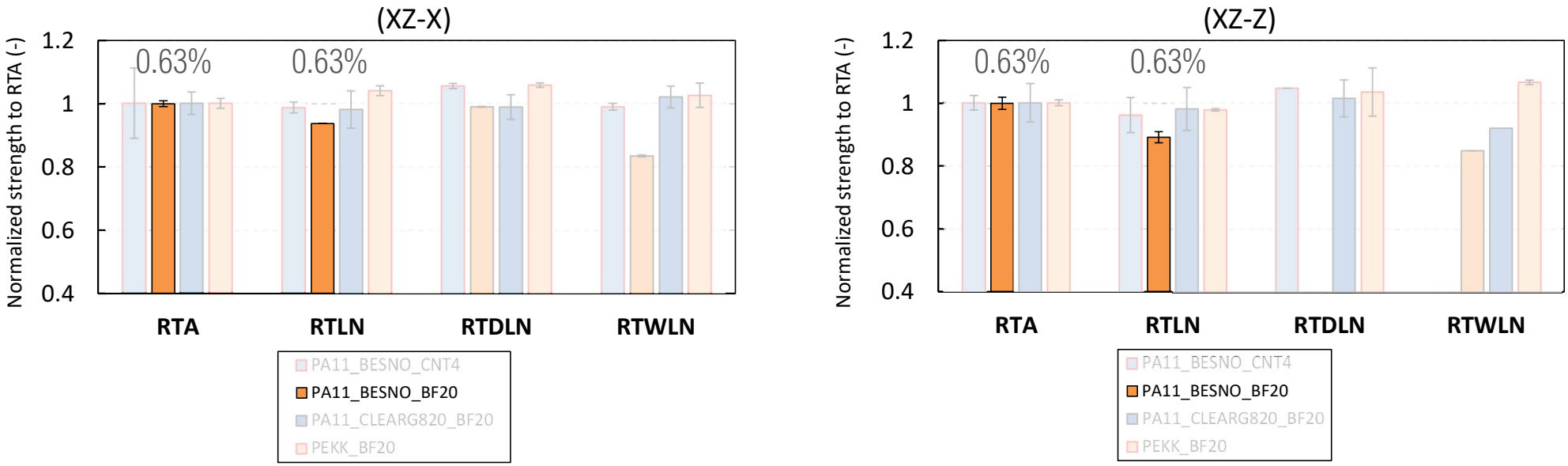
Results > Tensile tests > Conditioning + Immersion + Test





Results > Tensile tests > Conditioning + Immersion + Test

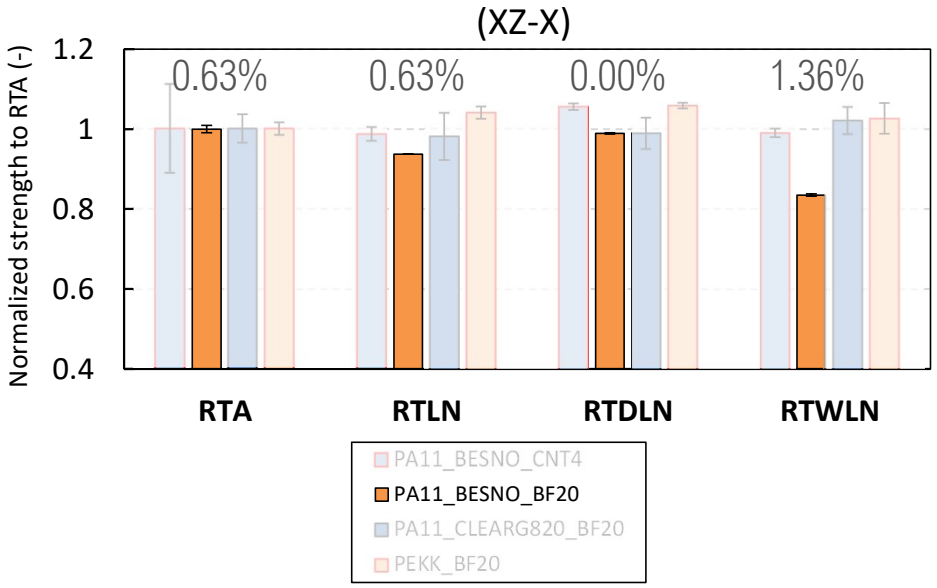
PA11_BESNO_BF20: RTA and RTLN

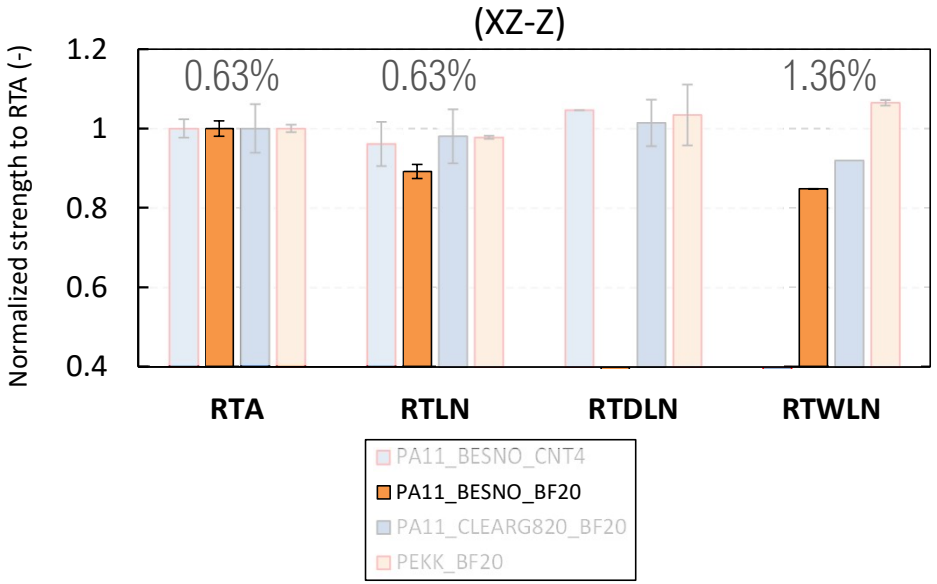


- RTA and RTLN have the same moisture content (0.63%), the immersion in LN2 slightly reduces the strength value in XZ-X and XZ-Z

Results > Tensile tests > Conditioning + Immersion + Test

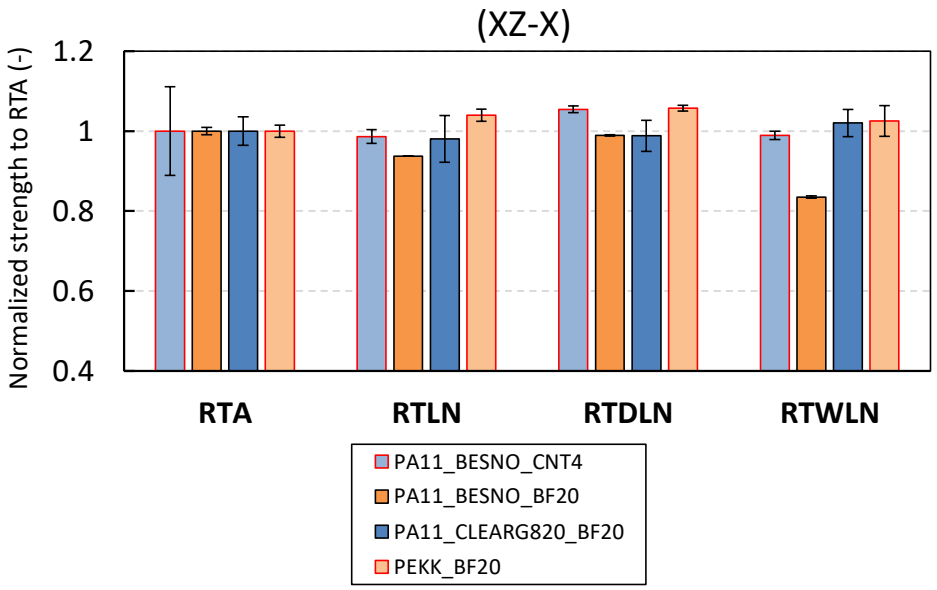
PA11_BESNO_BF20

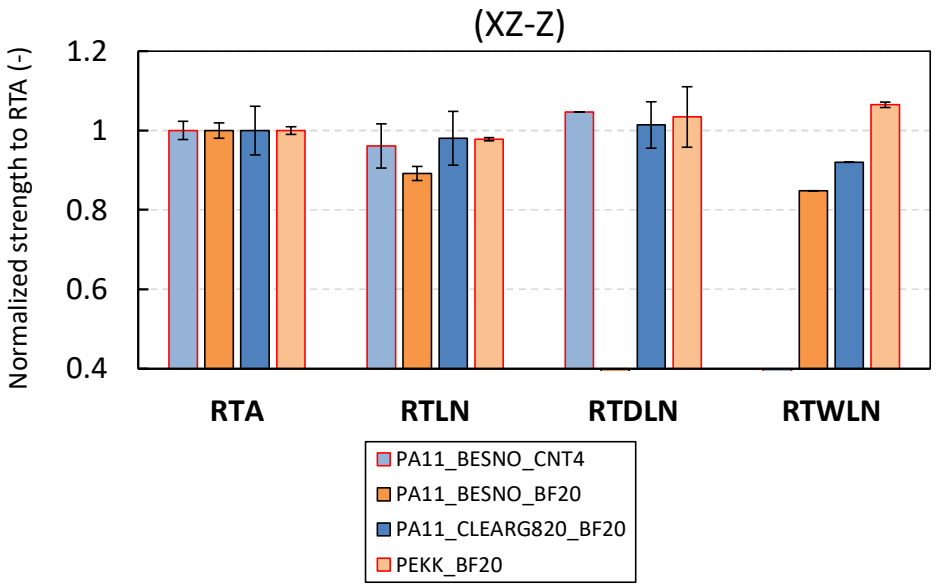




- PA11_BESNO_BF20 strength is affected by moisture content + LN2 immersion

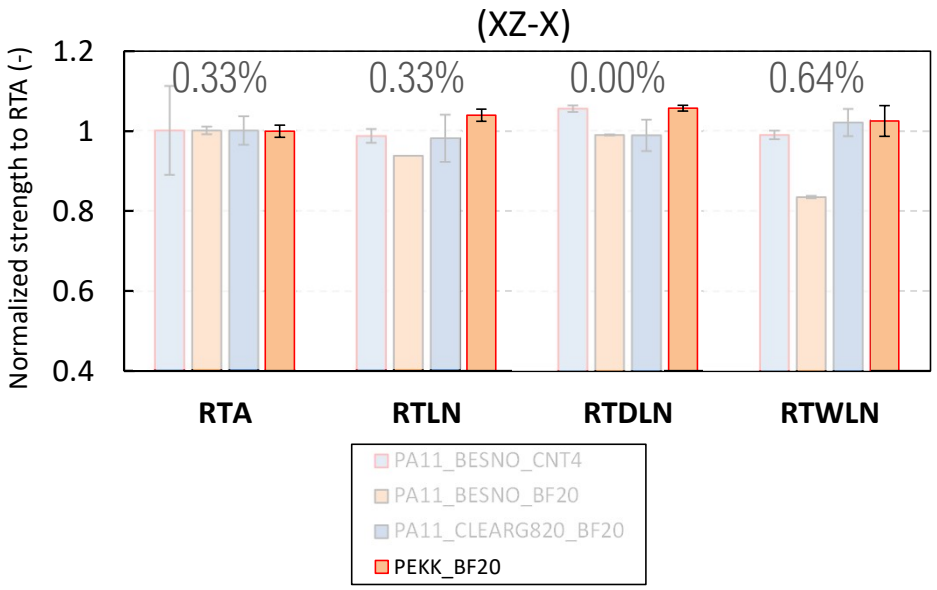
Results > Tensile tests > Conditioning + Immersion + Test

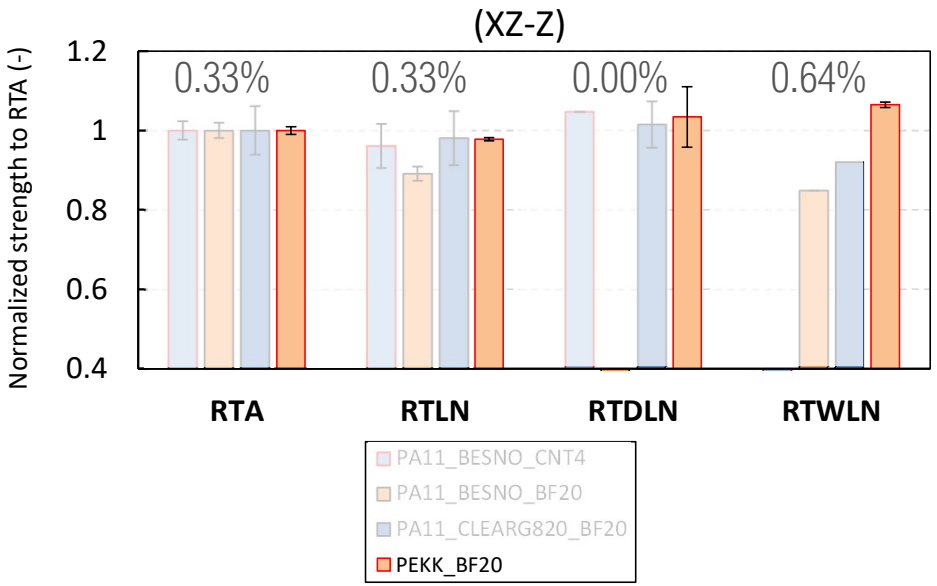




Results > Tensile tests > Conditioning + Immersion + Test

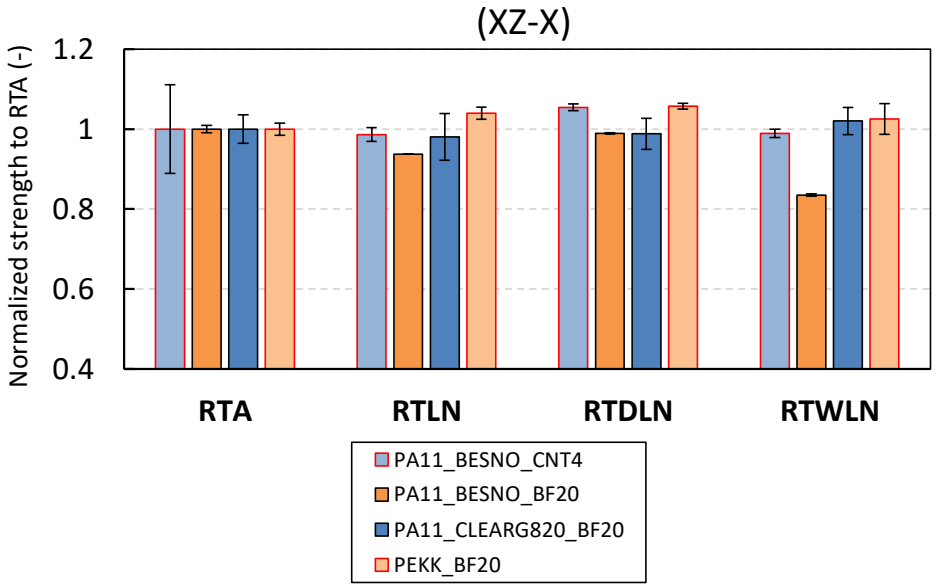
PEKK_BF20

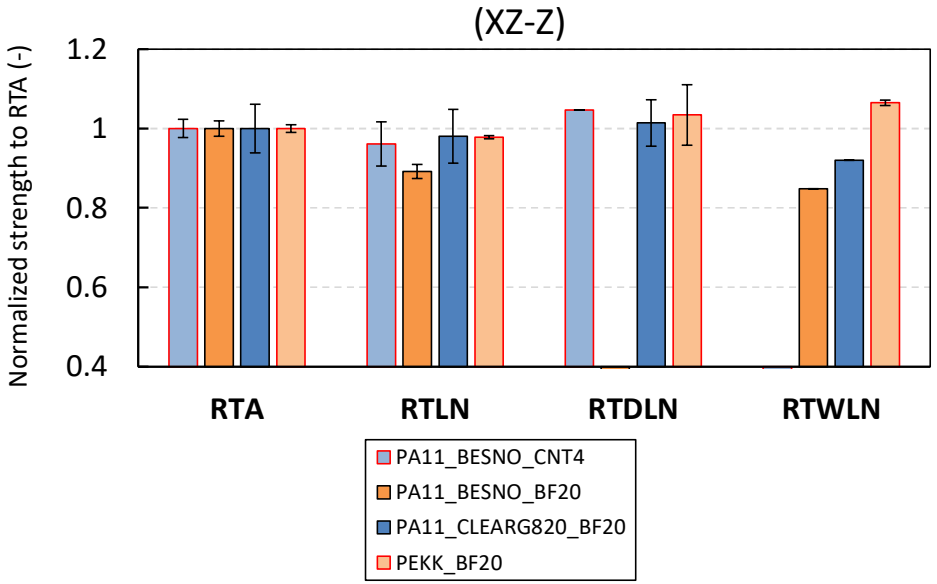




- PEKK_BF strength is not affected by moisture content + LN2 immersion, is the material with the highest porosity (11%).
- PEEK_BF20 has lowest moisture content (0.33% and 0.64%) compared to the other candidates.

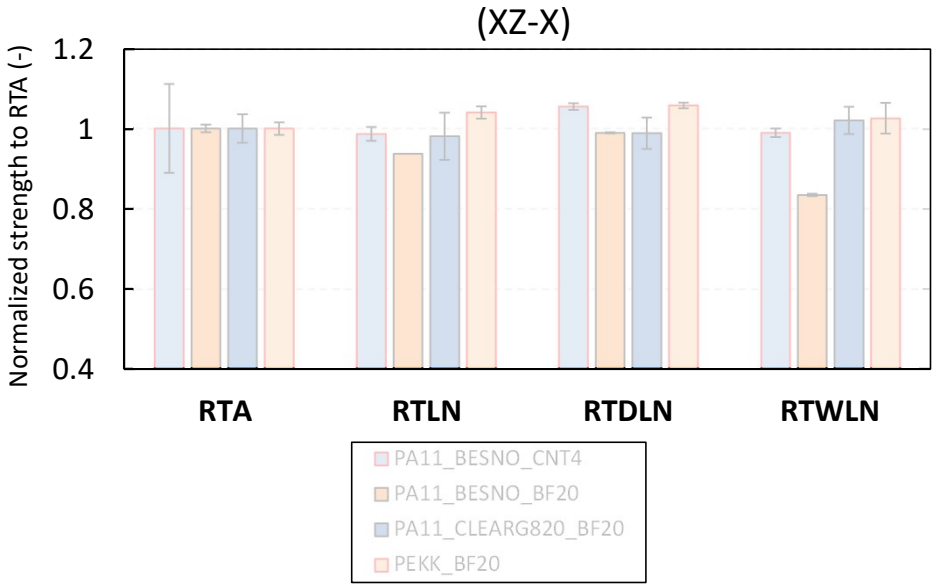
Results > Tensile tests > Conditioning + Immersion + Test

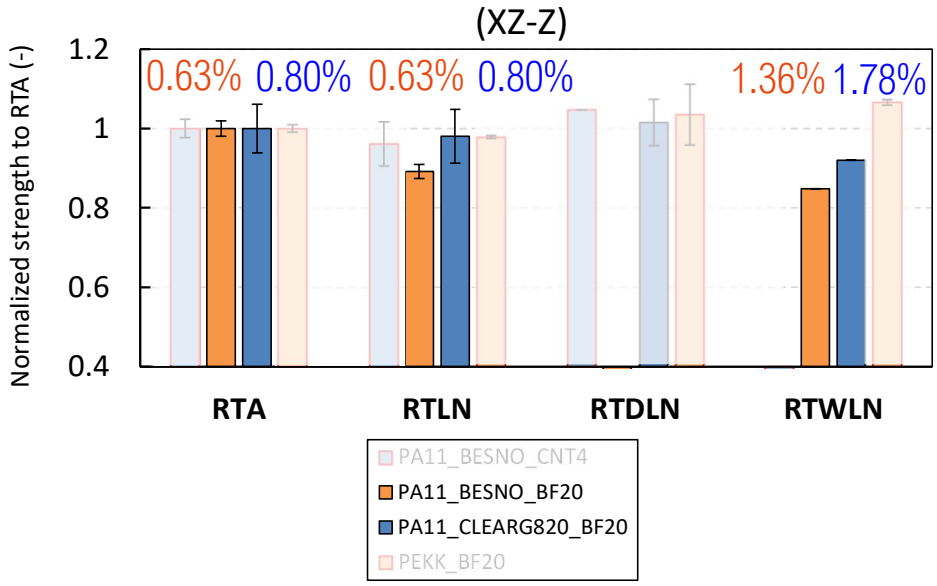




Results > Tensile tests > Conditioning + Immersion + Test

PA11 candidates in XZ-Z direction:





- The strength of both PA11 candidates is affected by Moisture + LN2 immersion
- Despite having a higher moisture content, PA11_CLEARG820_BF20 is less affected than PA11_BESNO_BF20.
- PA11_CLEARG820_BF20 has higher porosity (6%) than PA11_BESNO_BF20 (0.3%).

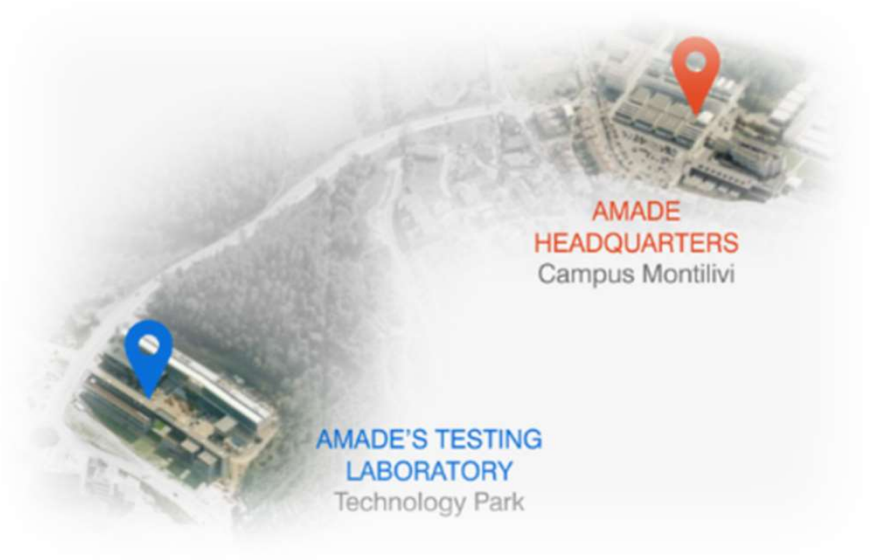
Preliminary observations

From the material candidates analysed and after the condintioning + immersion in LN2 we observe that:

- **Candidates with the highest moisture content** (PA11 based resin candidates) experience a **reduction in strength as humidity increases** and after the immersion in LN2.

- For Basalt Fiber reinforced candidates **with a higher porosity exhibit less reduction in strength** as humidity increases.

- This strength reduction depends on the **humidity content** but also on **the fibre/resin,** and the **printing direction**.



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