Reval PET

Centre Català del Plàstic

UNIVERSITAT POLITÈCNICA DE CATALUNYA
BARCELONATECH

eplascom
Plásticos y Composites Ecológicos

Projet cofinancé par le Fonds Européen
de Développement Régional

Interreg POCTEFA

UNIÓN EUROPEA
UNION EUROPÉENNE

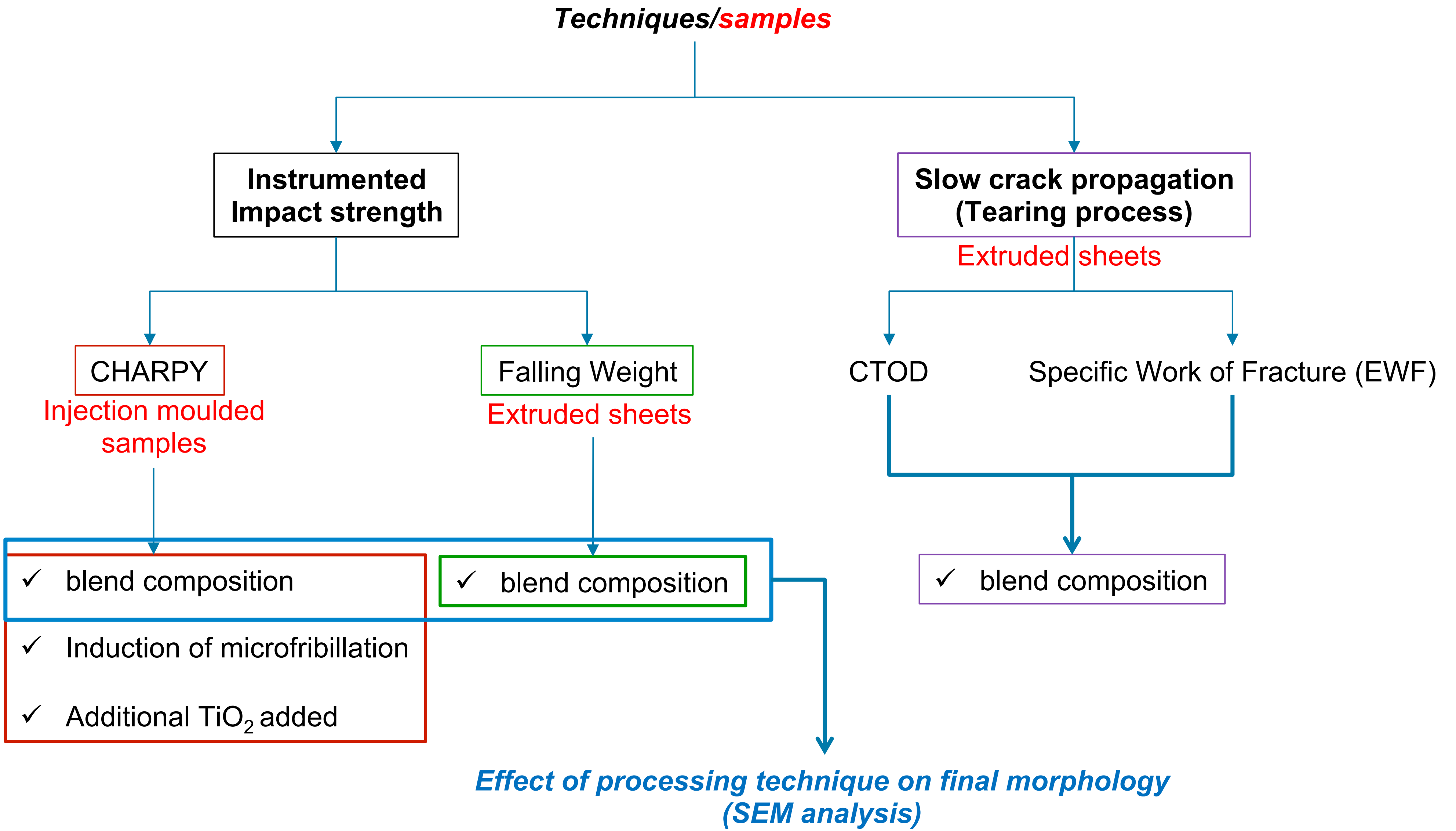
Action 3.
Development of high performance materials

3.4. Fracture characterization

Orlando SANTANA PÉREZ
CCP-UPC Characterization and fracture coordinator.

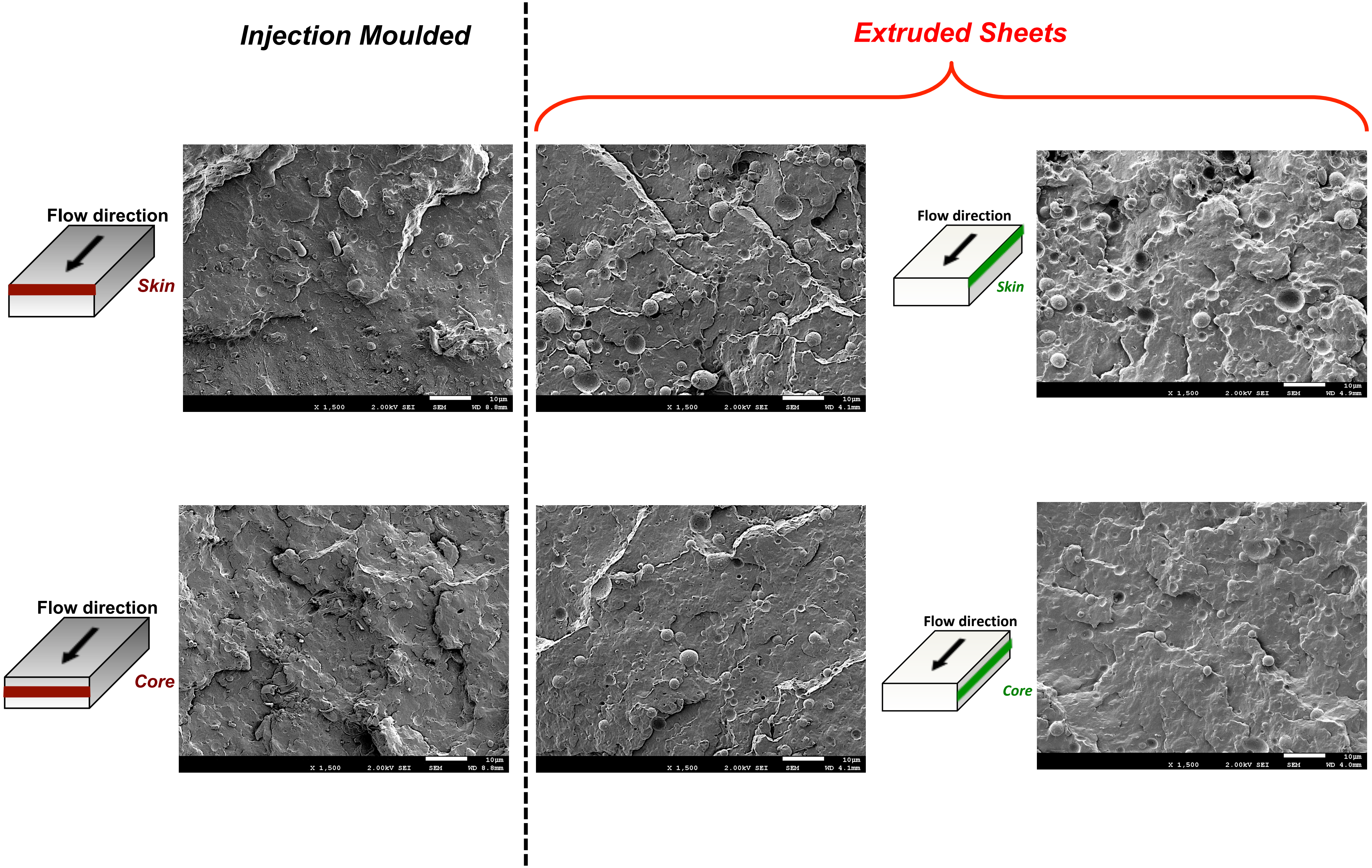
Magali KLOTZ
Hired Technician formaly contracted from November 15.
Ing. Msc. Material Science and Engineering form EEIGM – UPC.

Characterization scheme followed



Effect of the processing technique on the final morphology

10PET/90PP

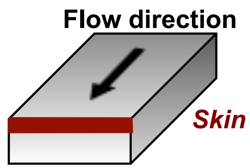


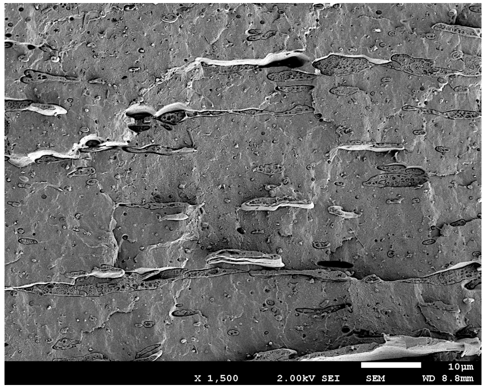
Effect of the processing technique on the final morphology

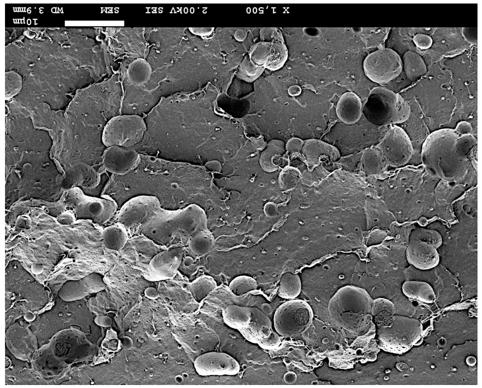
20PET/80PP

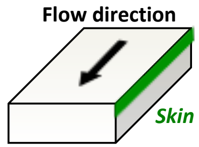
Injection Moulded

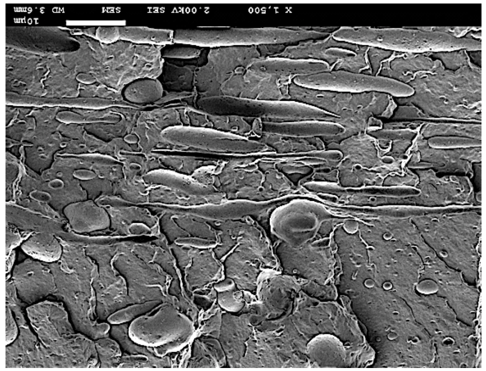
Extruded Sheets

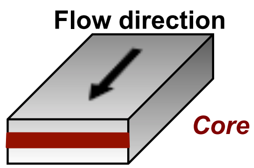


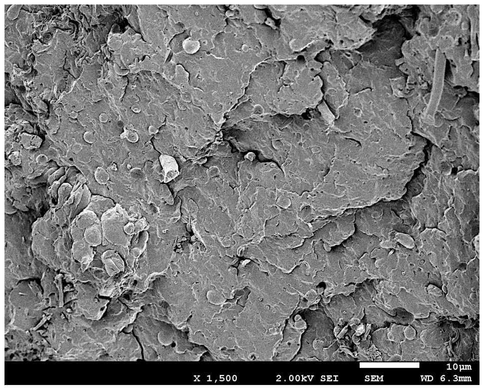


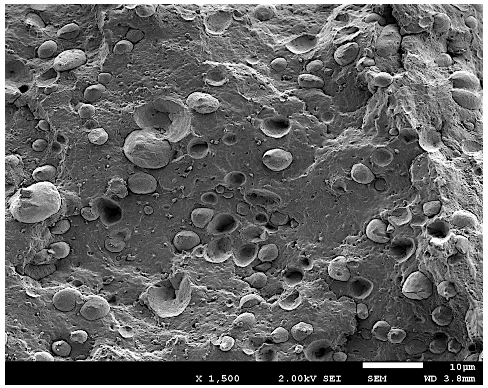


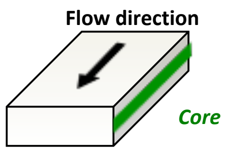


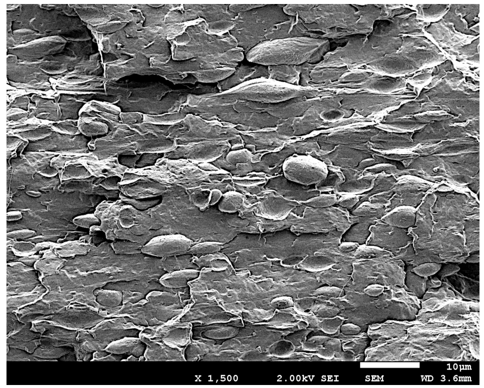












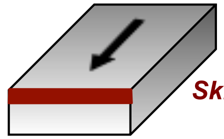
Effect of the processing technique on the final morphology

30PET/70PP

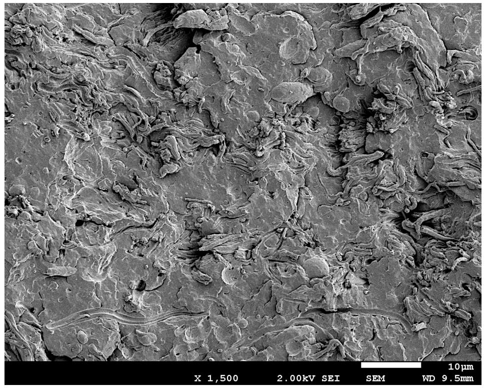
Injection Moulded

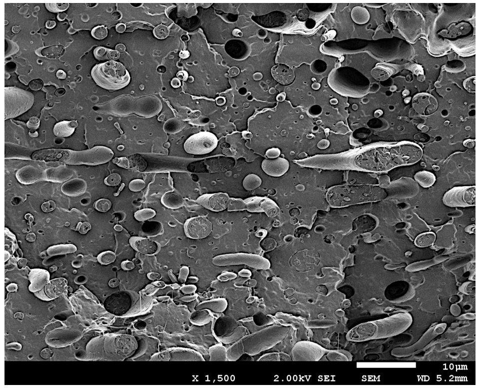
Extruded Sheets

Flow direction

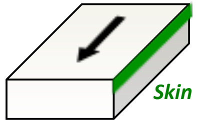


Skin

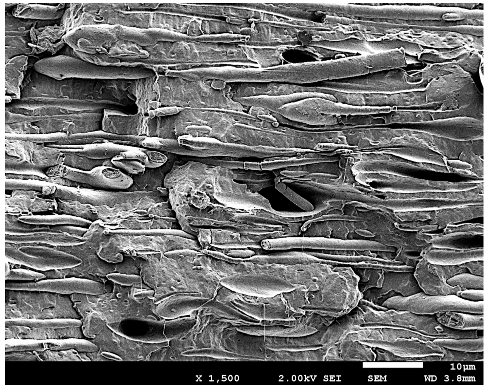




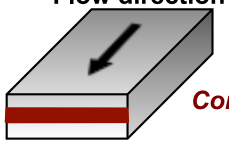
Flow direction



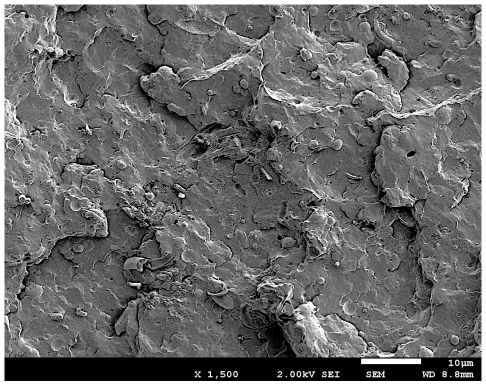
Skin

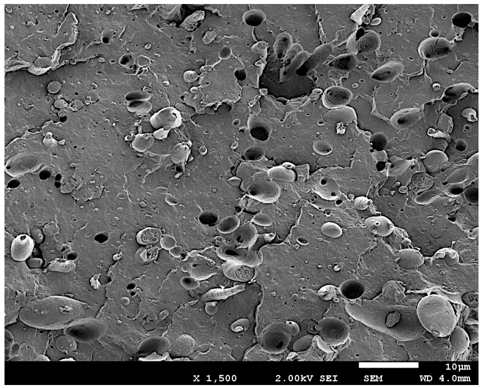


Flow direction

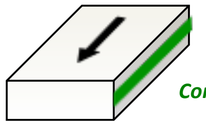


Core

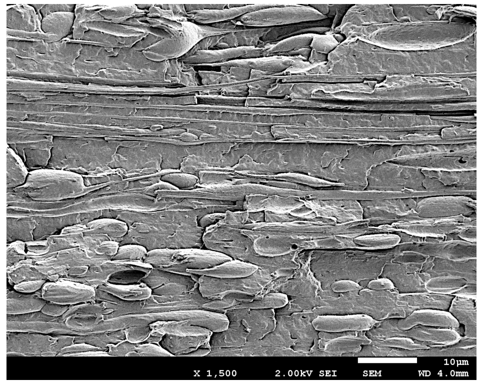




Flow direction



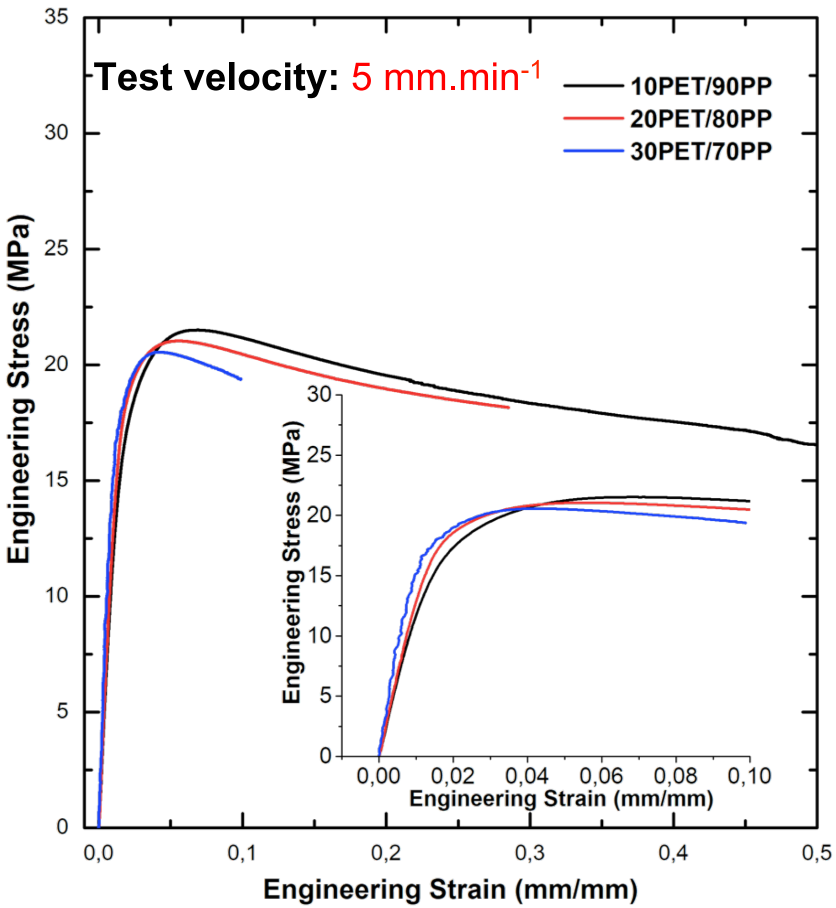
Core

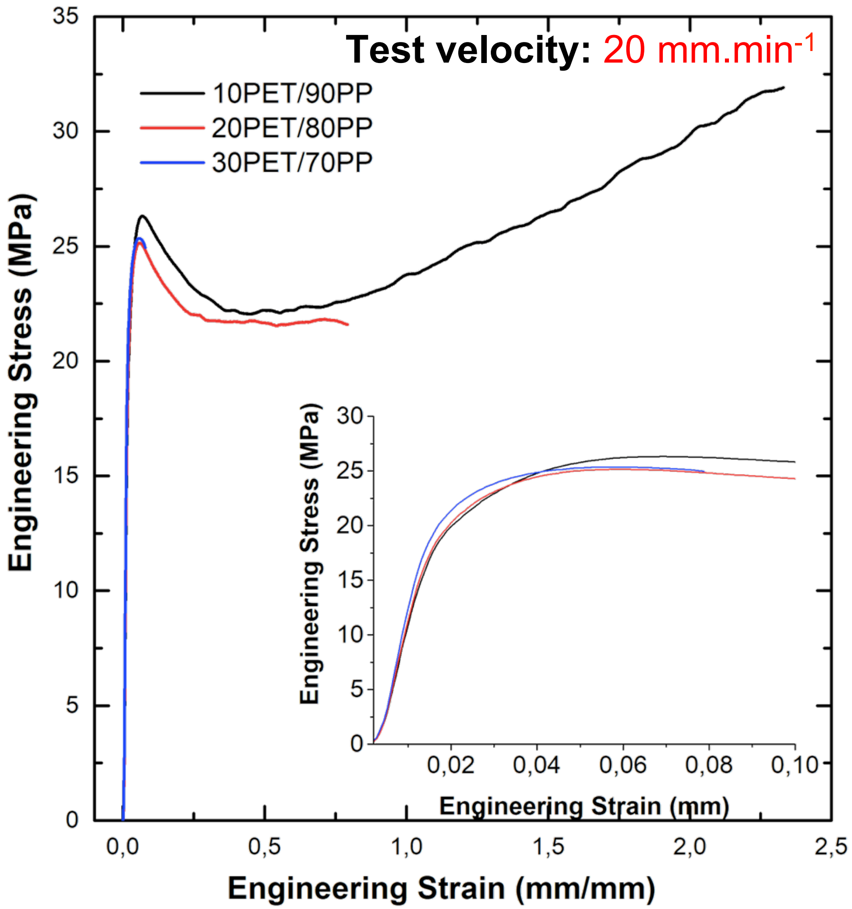


Effect of the processing technique on the final morphology

Tensile tests:

Equipment: Universal testing machine GALDABINI 2500 + Videoextensometer MITRON OS-65D
Temperature: 23ºC ± 1ºC





Effect of the processing technique on the final morphology

Equipment: Universal testing machine GALDABINI 2500 + Videoextensometer MITRON OS-65D
Temperature: 23ºC ± 1ºC

Injection moulded

Sample	Elastic Modulus E [GPa]	Yielding Stress σ_y [MPa]	Yielding Strain ε_y [%]	Strain at braeak ε_b [%]		
10PET/90PP	1,5 ± 0,1	21,8 ± 0,4	6,7 ± 0,2	56,2 ± 5,3	9,5 %	
20PET/80PP	1,6 ± 0,1	20,8 ± 0,2	5,6 ± 0,1	18,1 ± 4,6	25 %	- 68 %
30PET/70PP	1,7 ± 0,1	20,7 ± 0,3	4,3 ± 0,1	7,4 ± 0,6	81%	- 87%

Extruded sheets

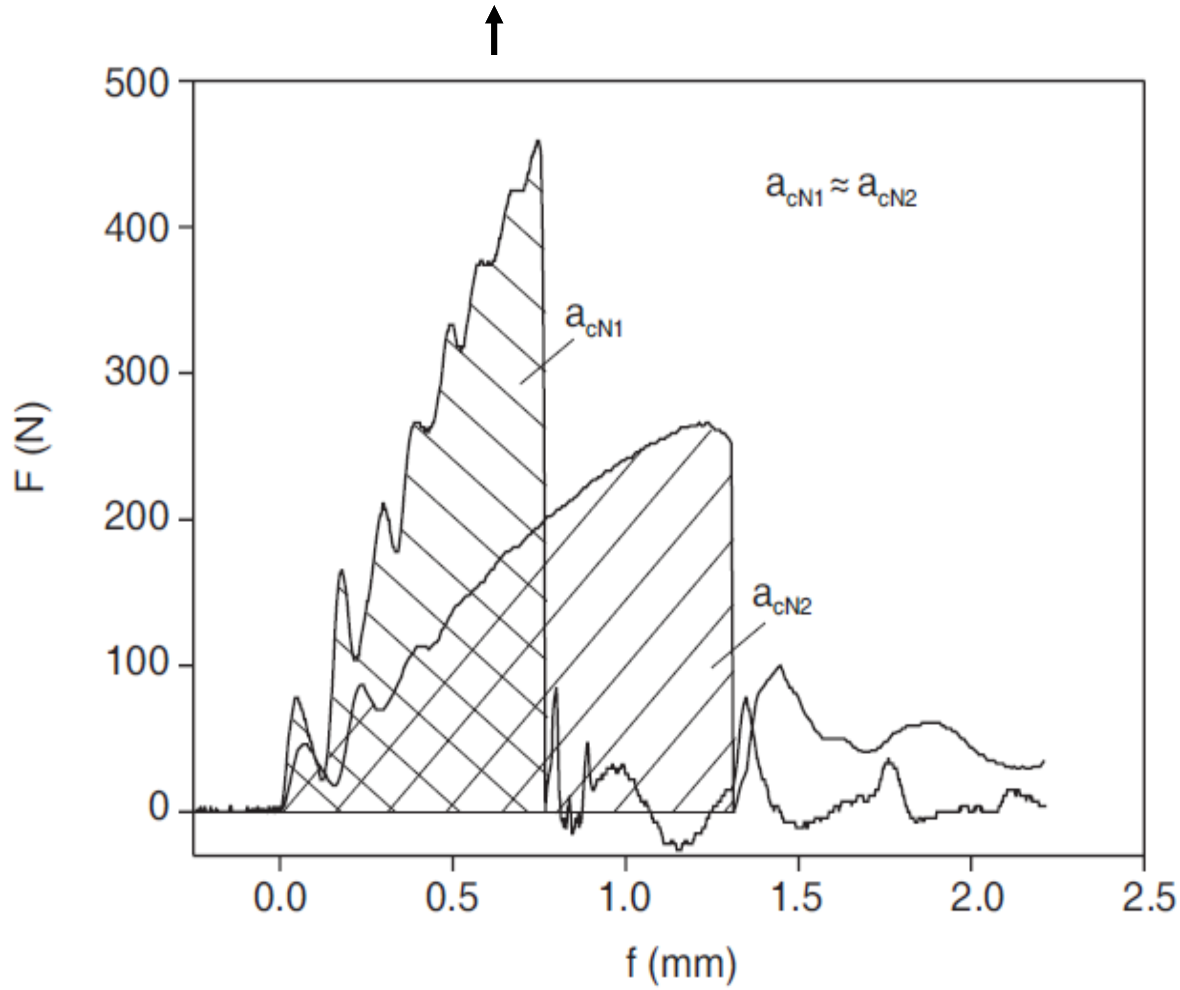
Sample	Elastic Modulus E [GPa]	Yielding Stress σy [MPa]	Yielding Strain εy [%]	Strain at braeak εb [%]		
10PET/90PP	1,17 ± 0,02	26,1 ± 0,2	12,1 ± 0,4	233 ± 6	2,5 %	
20PET/80PP	1,16 ± 0,01	24,0 ± 0,7	11,1 ± 0,7	77 ± 34	4 %	- 67 %
30PET/70PP	1,21 ± 0,02	25,4 ± 1,3	8,7 ± 1,4	10 ± 1	10%	- 96%

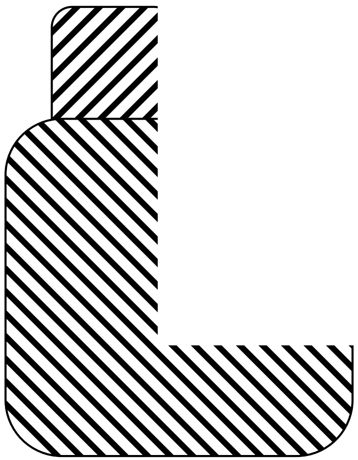
Instrumented impact tests

acement

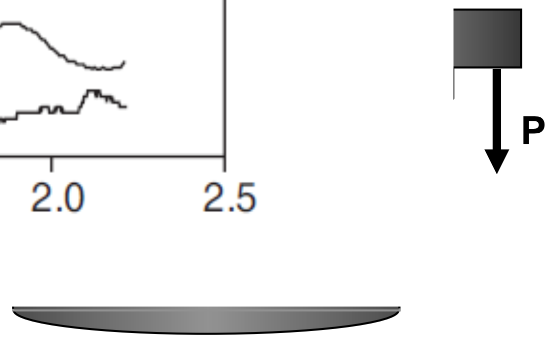
:he event

Piez





Charpy configuration (side view)



“campled” Falling Weight configuration
(close to multiaxial loading)

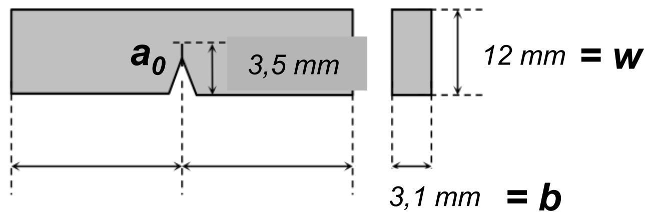
Effect of blend composition (rPET-O added: 10, 20 and 30%)

Instrumented impact tests:

Device: Dartvis (CEAST)
Hammer: 25 J (instrumented)
Impact velocity: 1 m/s (quasi-static loading conditions)
Notch: machined (0,25 mm)

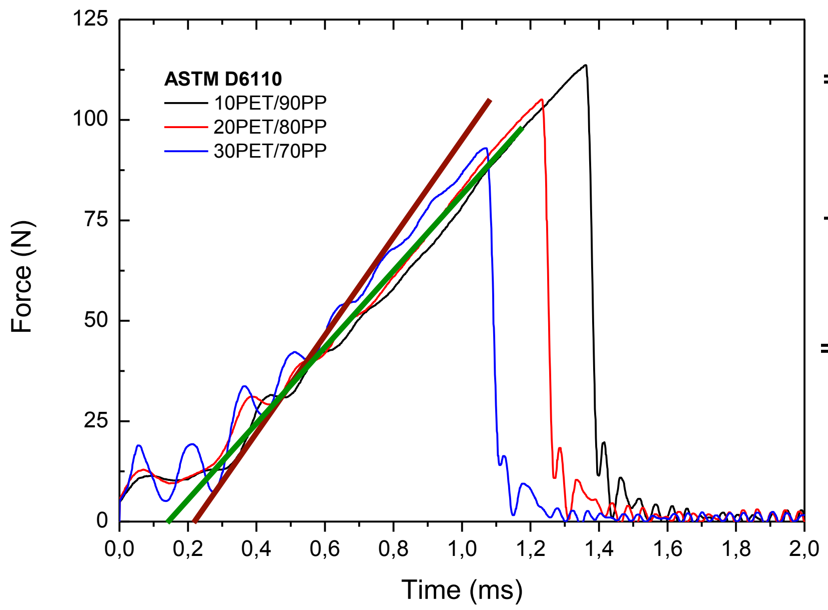
ASTM prismatic bars dimensions (taken from calibrated zone)

Geometry: Charpy configuration (SENB geometry)



S *(span) = 52,8 mm (4,4**w**)*

Typical traces hammer contact force vs. contact time



Charpy Impact strength parameters

Sample	Impact Strength RI [kJ.mm^{-2}]	Maximum Load F_{max} [N]	Maximum residual stress* σ_{max} [MPa]	Slope N/ms
10PET/90PP	3,4 ± 0,2	114 ± 5	20,2 ± 0,8	88,6 ± 0,2
20PET/80PP	2,6 ± 0,2	106 ± 5	18,8 ± 0,9	96,2 ± 0,7
30PET/70PP	1,7 ± 0,2	92 ± 6	16 ± 1	103,5 ± 0,8

* Calculated according to elastic beam loaded in 3 point bending

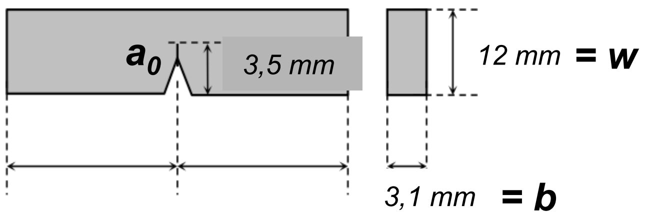
Effect of induction of microfibrillated morphology (20% w/w of rPET-O)

Instrumented impact tests:

Device: Dartvis (CEAST)
Hammer: 25 J (instrumented)
Impact velocity: 1 m/s (quasi-static loading conditions)
Notch: machined (0,25 mm)
Nº of samples: 5

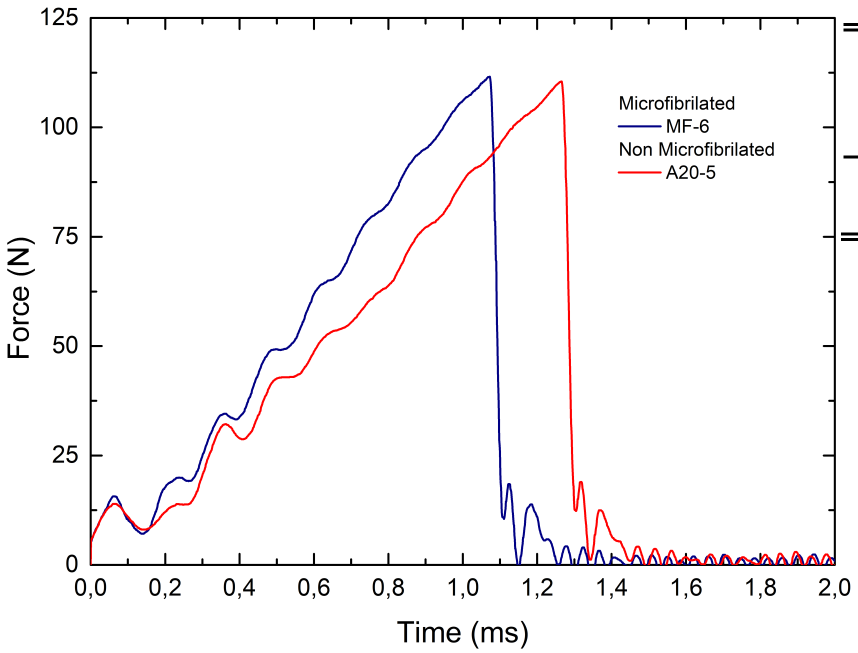
ASTM prismatic bars dimensions (taken from calibrated zone)

Geometry: Charpy configuration (SENB geometry)



S *(span) = 52,8 mm (4,4**w**)*

Typical traces hammer contact force vs. contact time



Charpy Impact strength parameters

Sample	Impact Strength RI [kJ.mm^{-2}]	Maximum Load F_{max} [N]	Maximum residual stress* σ_{max} [MPa]	Slope N/ms
20PET/80PP	2,6 ± 0,2	106 ± 5	18,8 ± 0,9	96,2 ± 0,7
20PET/80PP-MF	2,0 ± 0,1	103 ± 5	18,9 ± 0,3	118,1 ± 0,4

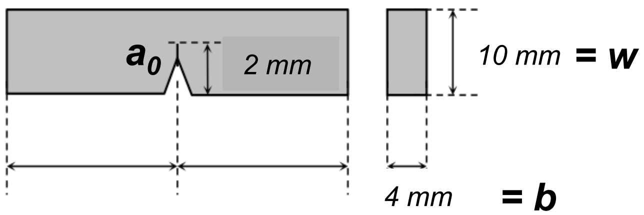
* Calculated according to elastic beam loaded in 3 point bending

Effect of adding additional TiO_2 (20% w/w of rPET-O + 1, 3 and 5% w/w of TiO_2)

Instrumented impact tests:

Device: Dartvis (CEAST)
Hammer: 25 J (instrumented)
Impact velocity: 1 m/s (quasi-static loading conditions)
Notch: machined (0,25 mm)
Nº of samples: 5

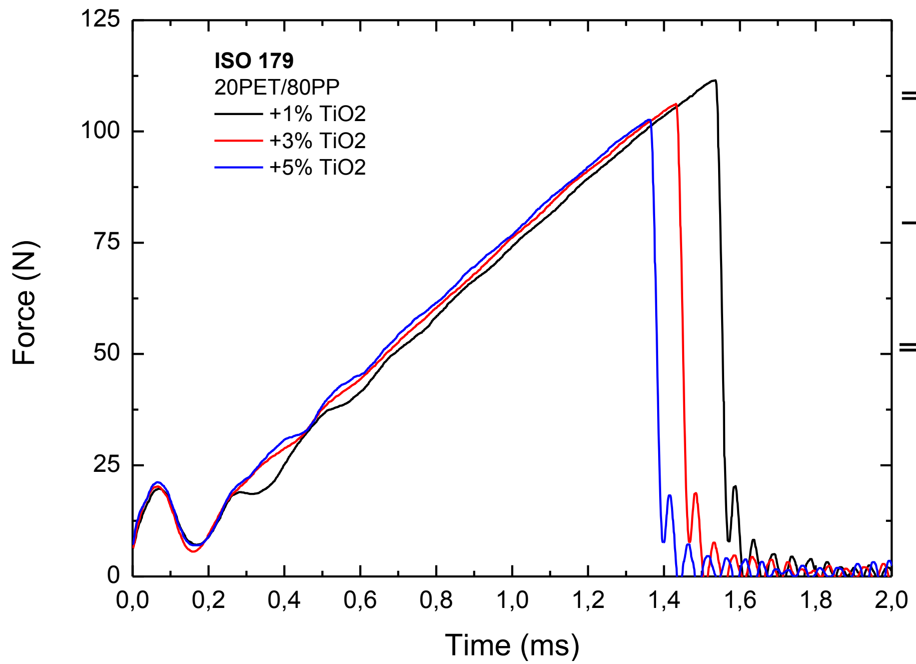
Geometry: Charpy configuration (ISO)



S (span) = 62 mm

ISO multipurpose test specimen (taken from calibrated zone)

Typical traces hammer contact force vs. contact time



Charpy Impact strength parameters

Sample	Impact Strength RI [$kJ.mm^{-2}$]	Maximum Load F_{max} [N]	Maximum residual stress* σ_{max} [MPa]	Slope N/ms
+1 %	3,0 ± 0,2	109 ± 3	26 ± 1	80,7 ± 0,3
+ 3%	2,69 ± 0,08	105 ± 3	24,8 ± 0,5	80,8 ± 0,2
+ 5%	2,7 ± 0,1	106 ± 6	25 ± 1	82,8 ± 0,3

* Calculated according to elastic beam loaded3 point bending

Extruded sheets samples of rPP/rPET-O: 90/10, 80/20 and 30/70

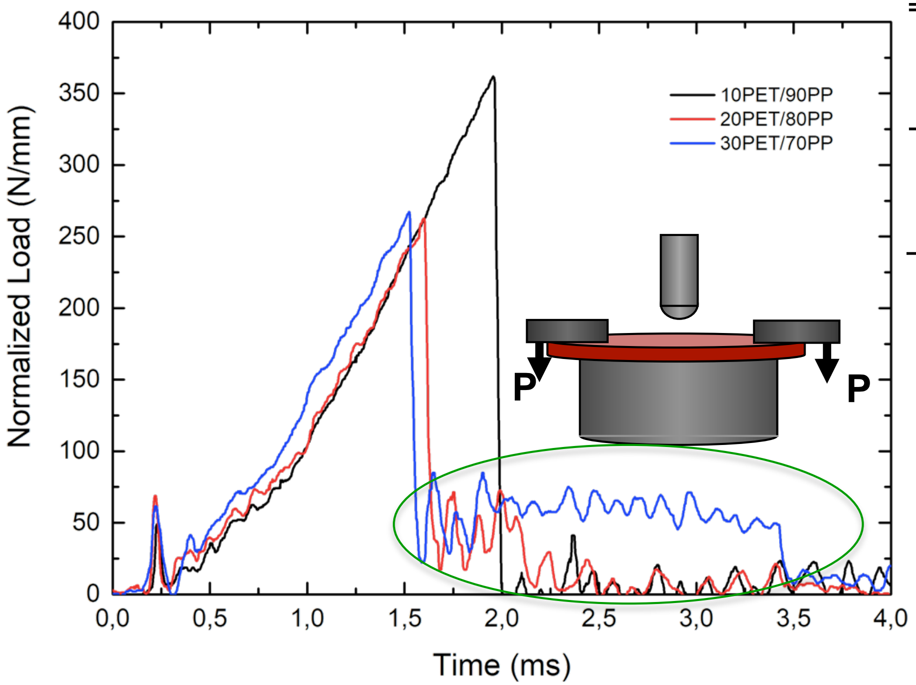
Instrumented Falling weigth impact tests:

Geometry: Square coupons 80 x 80 mm.
Suport: cilindrical (60 mm of diameter)

Device: Dartvis (CEAST)
Dart weight: 3,243 kg
Falling height: 0,4 m
Impact velocity: 2,8 m/s (quasi-static loading conditions)
Clamped configuration

Test performed with lubrication

Typical traces of dart contact force vs. contact time



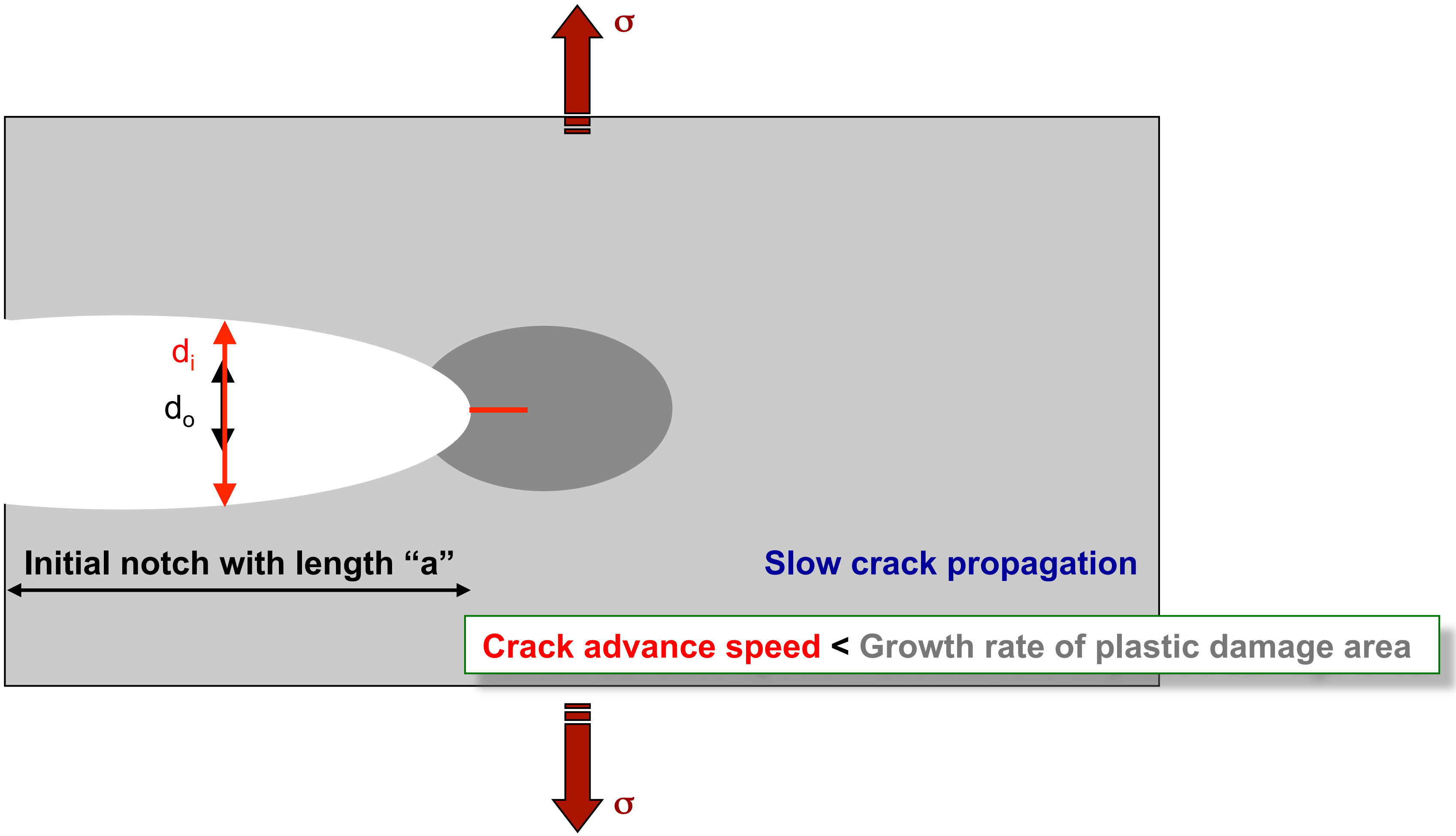
Falling weight impact parameters

Sample	Normalized Maximum Load F_{max-N} [N/mm]	Normalized Peak Energy E_{p-N} [J/mm]	Normalized Total Energy E_{T-N} [J/mm]	Normalized Slope N/mm.ms
10PET/90PP	368 ± 44	0,79 ± 0,09	0,8 ± 0,1	138,4 ± 0,4
20PET/80PP	271 ± 22	0,48 ± 0,06	0,8 ± 0,2	170 ± 1
30PET/70PP	213 ± 22	0,64 ± 0,09	0,9 ± 0,3	208 ± 1

CTOD: Crack Tip opening displacement

Relative displacement of a notch flank at the onset of crack propagation

$d_i - d_o = \textbf{\textit{CTOD}}$



CTOD: Extruded sheets samples of rPP/rPET-O: 90/10, 80/20 and 30/70

CTOD:

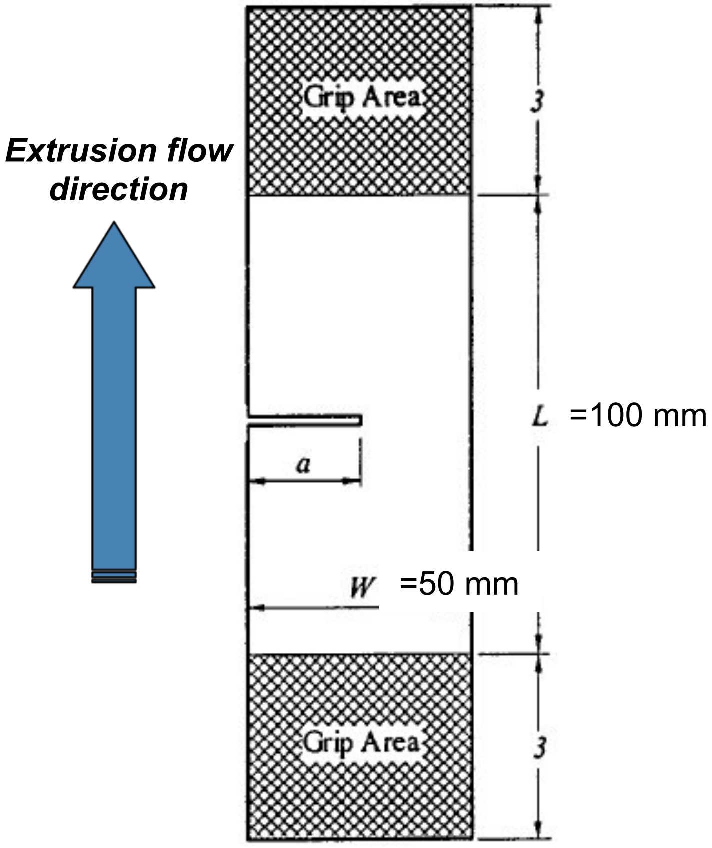
Equipment: Servohydraulic Universal testing machine Zwick Amsler HC25 + 3D viedoextensometer GOM - ARAMIS

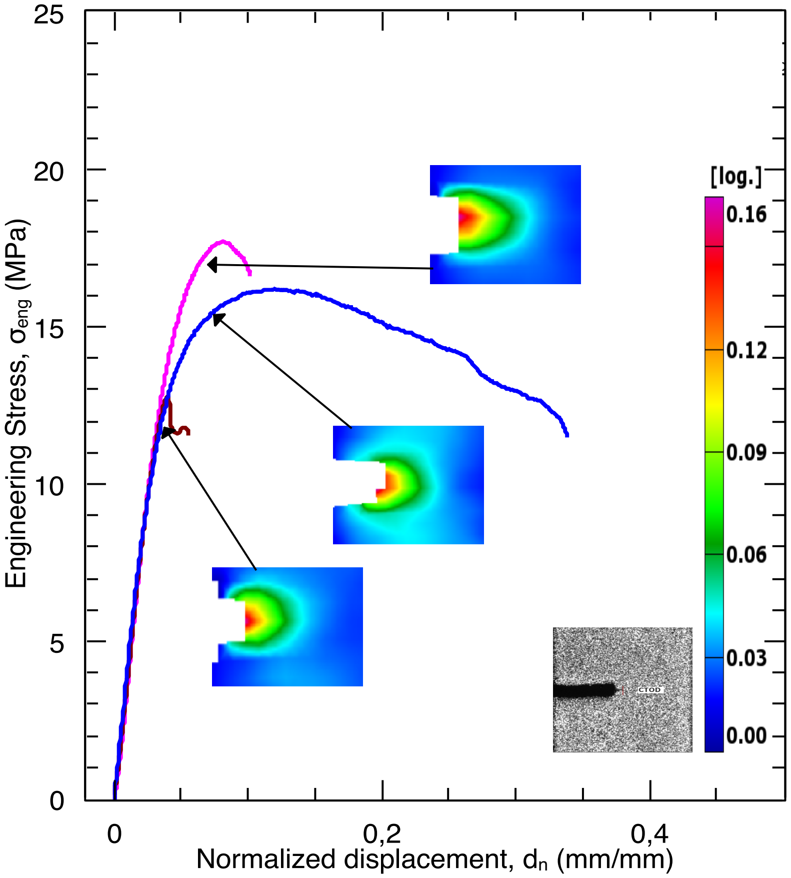
Test speed: 10 mm.min^{-1}

Temperature: 18ºC ± 2ºC

Sample geometry: SENT (ligament length = 30 mm)

Nº of tested samples: 5 per material





CTOD: Extruded sheets samples of rPP/rPET-O: 90/10, 80/20 and 30/70

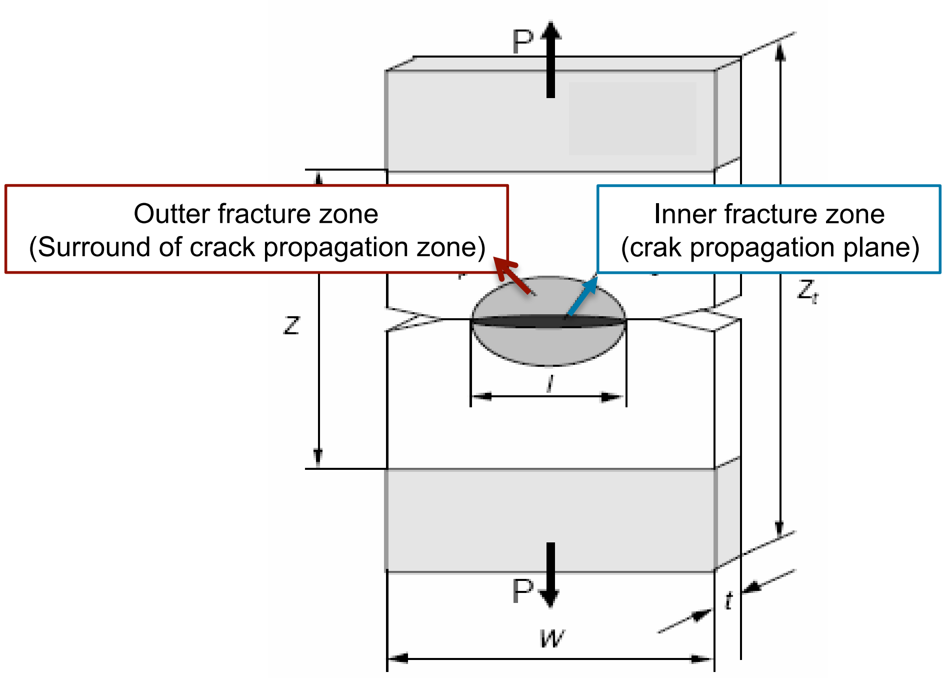
CTOD:

Equipment: Servohydraulic Universal testing machine Zwick Amsler HC25 +
3D viedoextensometer GOM - ARAMIS
Test speed: 10 mm.min^{-1}
Temperature: 18ºC ± 2ºC
Sample geometry: SENT (ligament length = 30 mm)
Nº of tested samples: 5 per material

CTOD parameters

Sample	Load at CTOD [N]	CTOD [mm]	Specific work at CTOD [kJ.m^2]
10PET/90PP	282 ± 6	0,7 ± 0,2	8 ± 1
20PET/80PP	241 ± 10	0,4 ± 0,1	3 ± 1
30PET/70PP	242 ± 6	0,5 ± 0,1	5,0 ± 0,9

Extruded sheets: Tearing process characterization: EWF



Specific Work of fracture:

$$w_f = w_e + \beta w_p l$$

Essential term (essential work of fracture) (w_e):

Free energy (by unit surface) required for ***new free surfaces generation during tearing process*** (***crack initiation***)

Non-essential work of fracture (βw_p):

Density of "plastic" energy outside the process zone, related to ***resistance to crack propagation***.

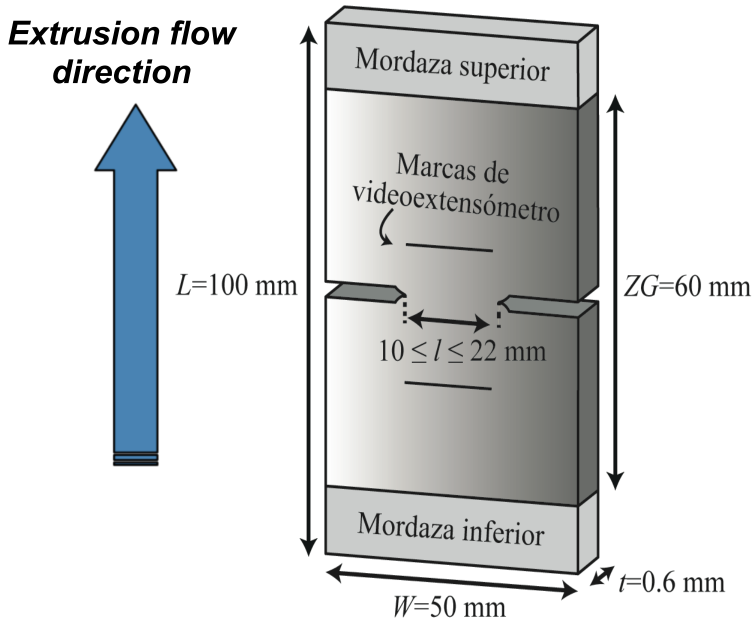
β:

Adimensional parameter related with the size of the "Outter fracture zone", where dissipative process during tearing are observerde.

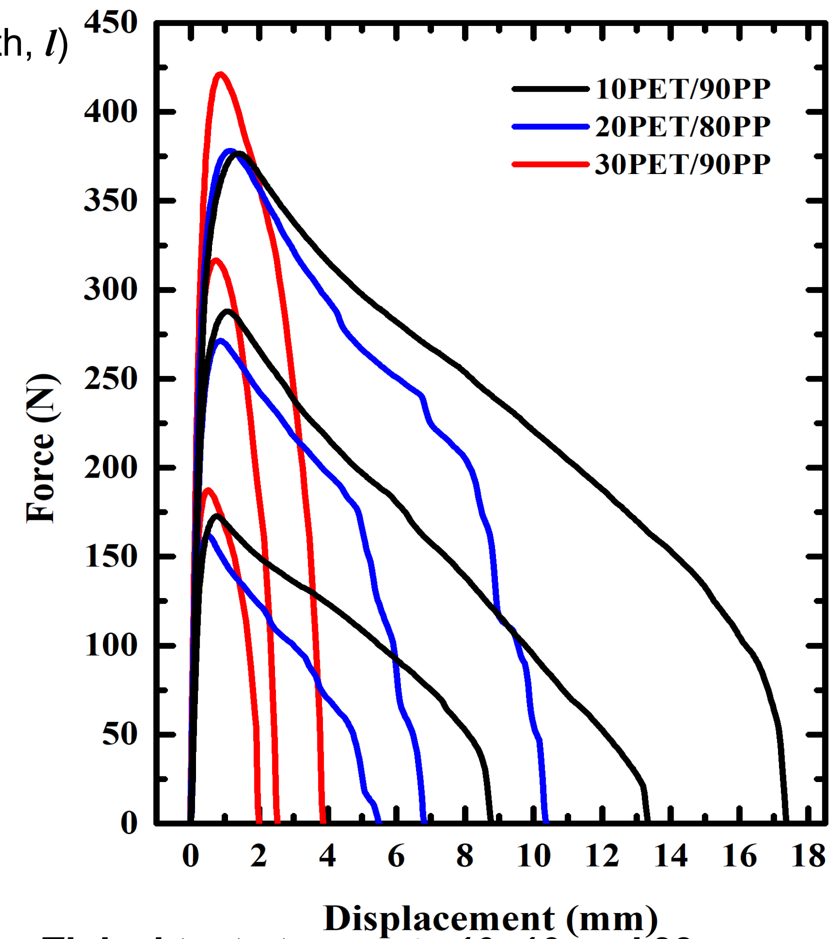
Extruded sheets: Tearing process characterization: EWF

Specific work of fracture:

Methodology: ESIS-TC4
Equipment: Universal testing machine GALDABINI + viedoextensometer MITRON OS 65D
Velocity: 10 mm.min^{-1}
Temperature: 18ºC ± 2ºC
Sample geometry: DDENT
Nº of tested samples: 15 (3 replicates at 5 ligament length, *l*)



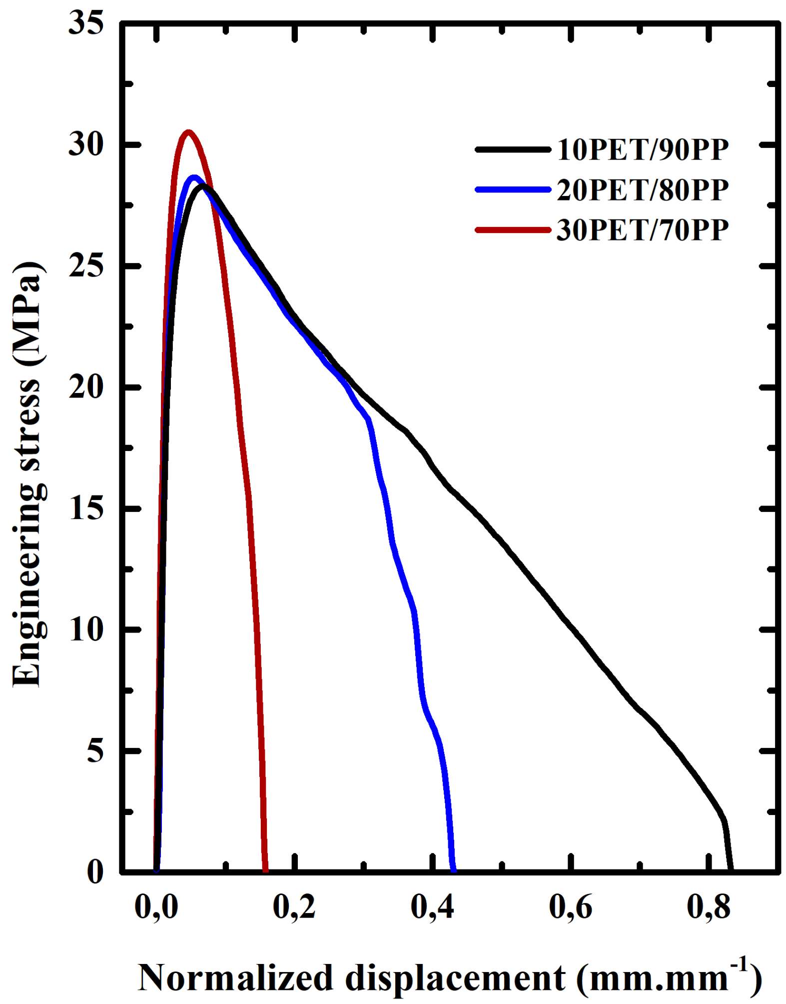
DDENT geometry used.
Crack propagation transverse to flow direction

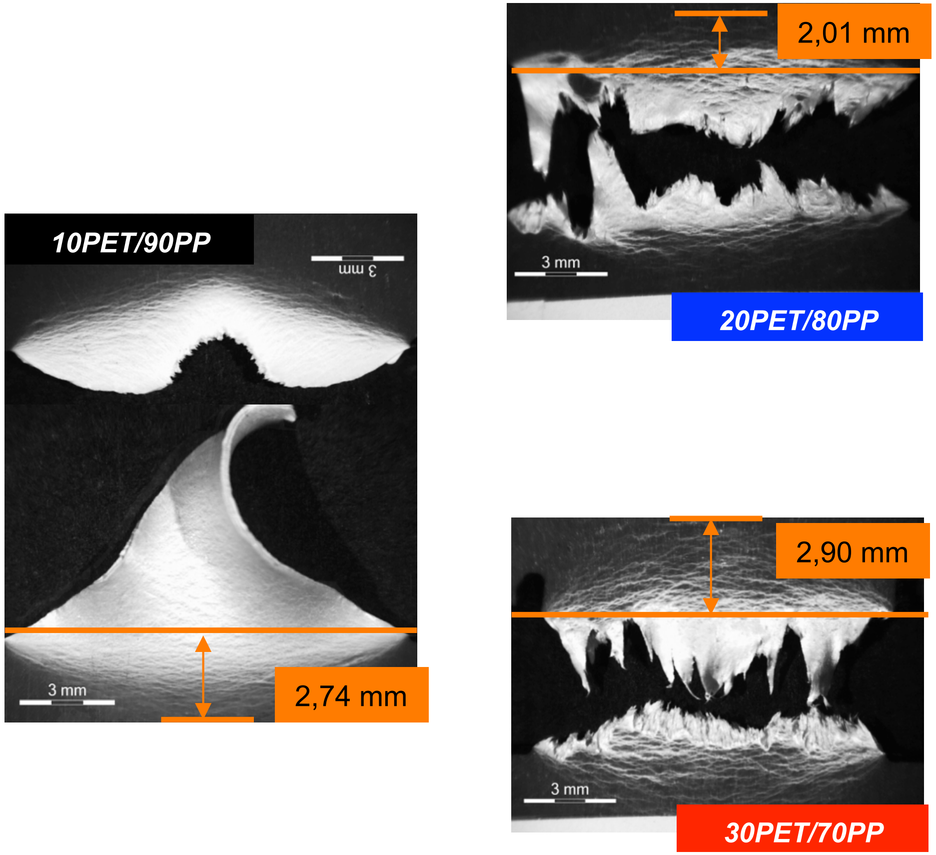


Tipical tests traces. l =10, 16 and 22 mm

Extruded sheets: Tearing process characterization: EWF

Outter process zone (Optical Microscopy)

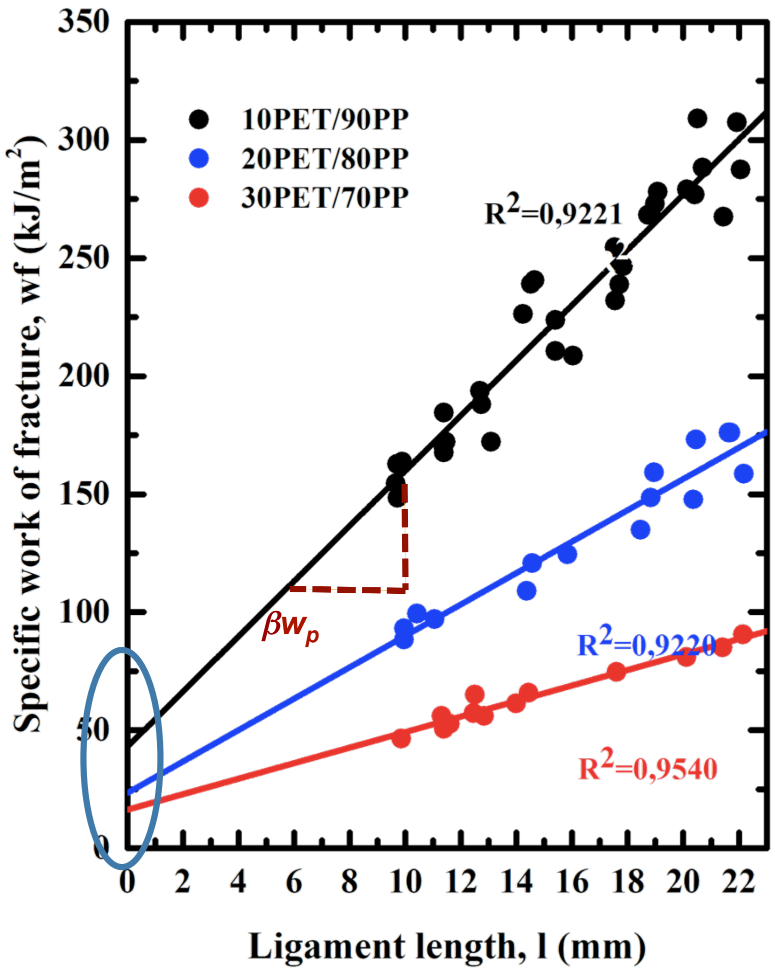




Extruded sheets: Tearing process characterization: EWF

Essential term (essential work of fracture) (w_e):
Free energy (by unit surface) required for ***new free surfaces generation during tearing process*** (***crack initiation***)

Non-essential work of fracture (βw_p):
Density of "plastic" energy outside the process zone, related to ***resistance to crack propagation***.

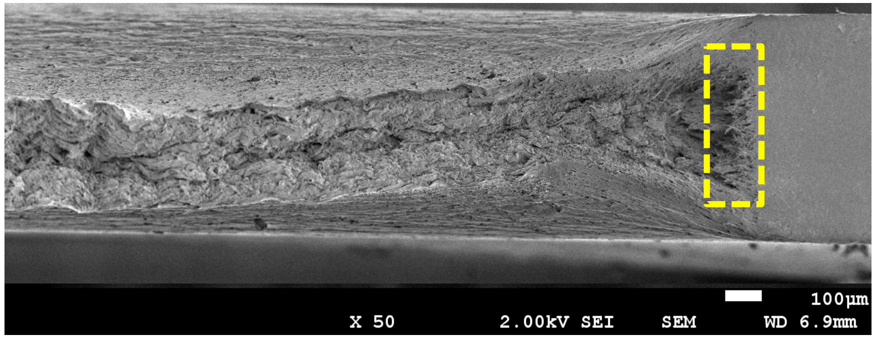


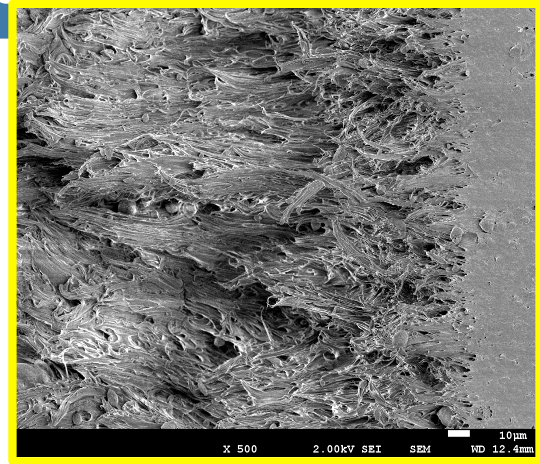
Material	w_e (kJ.m^{-2})	βw_p (MJ.m^{-3})	β x 10^2
10PET/90PP	44 ± 4	11,6 ± 0,6	25 ± 2
20PET/80PP	24 ± 3	6,6 ± 0,5	26 ± 2
30PET/70PP	16 ± 3	3,3 ± 0,2	33 ± 2

It does not seem to reflect the real situation. (considering the highest levels of effective engineering tension at the moment of the beginning of the plastic collapse of the ligament that were recorded in the tests)

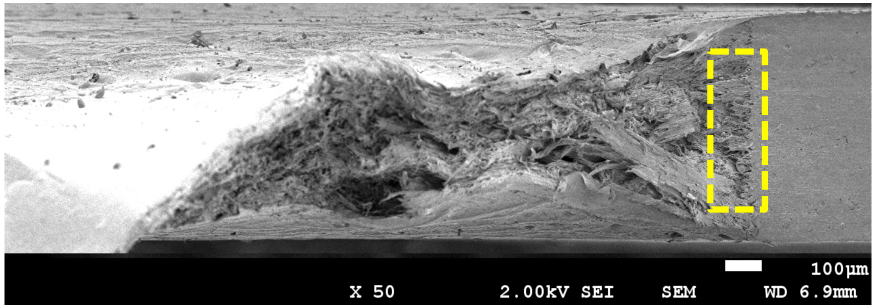
Extruded sheets: Tearing process characterization: EWF

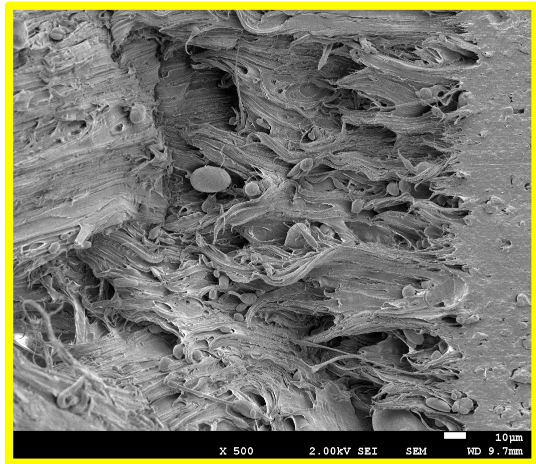
10PET/90PP



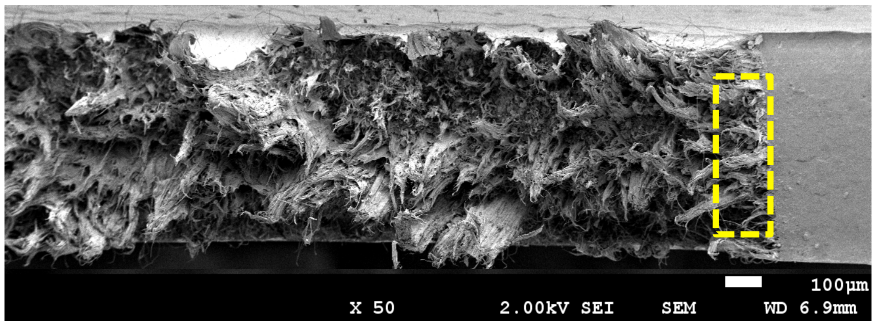


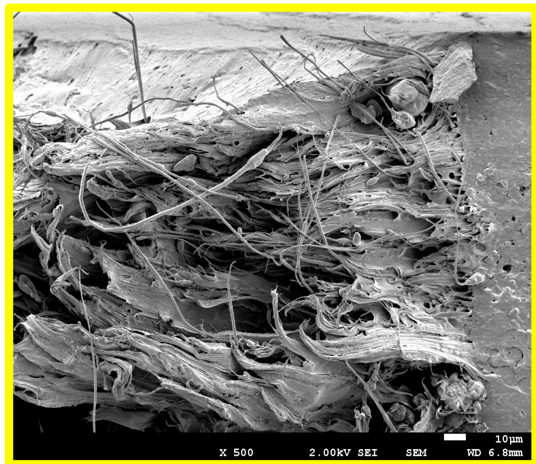
20PET/80PP





30PET/70PP





Conclusions

- ✓ Depending on the processing conditions and their composition, the ***rPP/rPET-O*** blends can generate a microfibrillar morphology of ***rPET-O*** that act as reinforcement.

% PET added	Injection Moulding	Extruded sheets
10	Droplet	Droplet (higher size)
20	Fibrillation	Gradient of morphology: Skin: fibrillar Core: droplets
30	Droplets (finer)	Fibrillar

- ✓ Impact Strength, and crack propagation resistance decrease with the ***rPET-O*** content..

- ✓ Addtion of about 5%ww of TiO_2 does not affect the impact strength, but increase the stiffness of the system.

Thank you

Centre Català del Plàstic-UPC

Campus Diagonal-Besos
Edifici C
Av. Eduard Maristany,14-16
Barcelona

Tel. 937.837.022

http://www.upc.edu/ccp

orlando.santana@upc.edu

maria.lluisa.maspoch@upc.edu

Reval PET

Projet cofinancé par le Fonds Européen
de Développement Régional

Interreg POCTEFA

UNIÓN EUROPEA
UNION EUROPÉENNE