Use of Thermal Black as filler in high performance HDPE films

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NRC at a glance

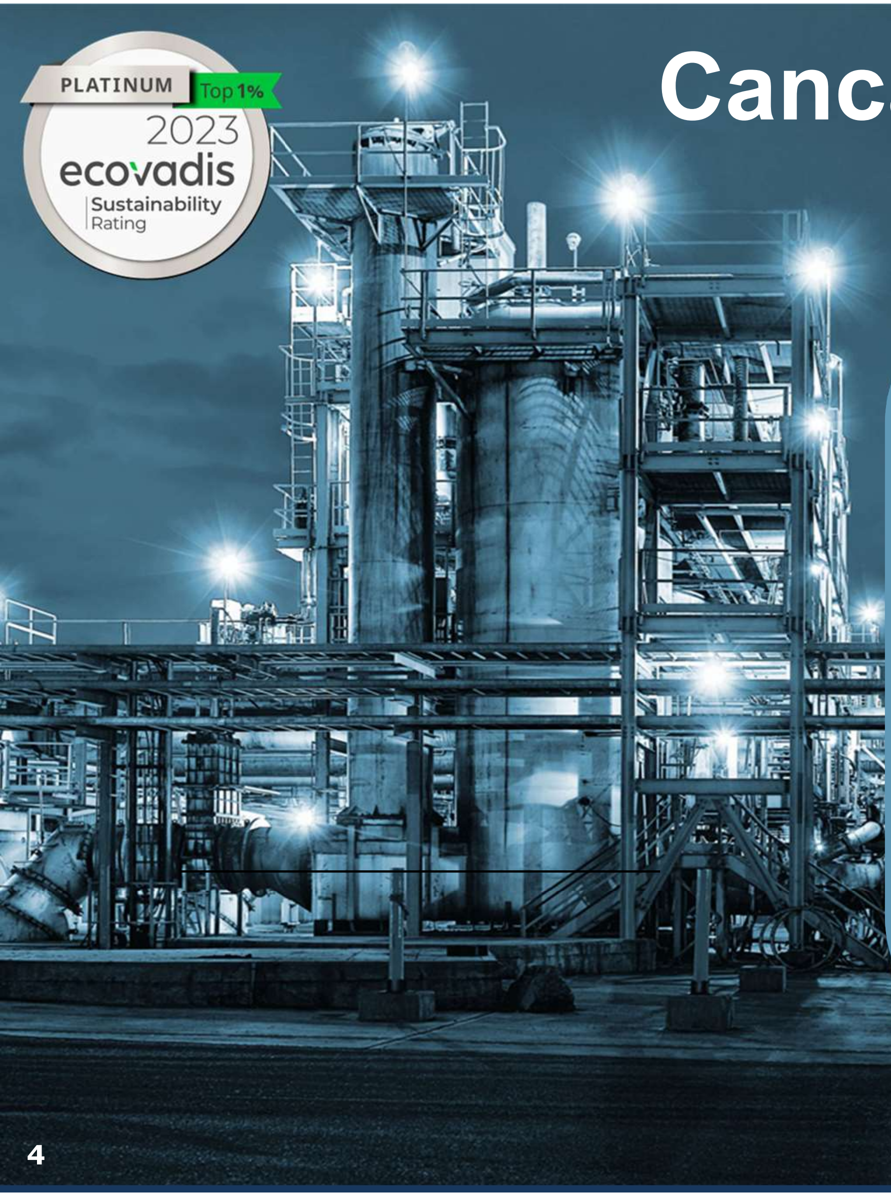
Canadian organization with regional presence and global reach

- **Government of Canada's premier research organization**
- **100-YEAR TRACK RECORD:** Supporting industrial innovation, advancement of knowledge & technology development. Fulfilling government mandates
- Serves thousands of industrial and government clients annually
- **≈ 3,700** Employees
- **≈ $1.2 B** Annual budget
  **Estimation including grants and contributions*

Advanced Manufacturing: Polymers and Composites Products

- High-volume, high-performance composites
- Industrial biomaterials and sustainable manufacturing
- Added-value polymer products using advanced materials and processing

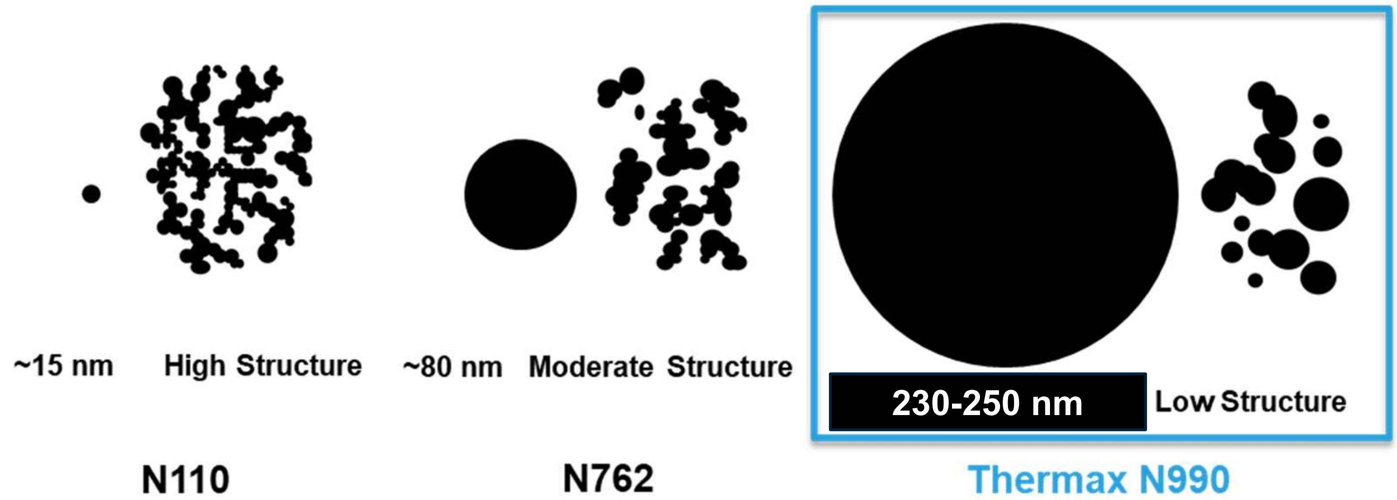
Cancarb overview



- The only company solely dedicated to the manufacturing of **Thermal Carbon Black**
- Thermax is one of the **purest and cleanest carbon blacks** available at the industrial scale
- Annual capacity of **120 million pounds**
- Thermax thermal black is produced by cracking natural gas into its constituent elements C and H
- **Production of zero-emission power** by capturing hot exhaust gases to produce stream that drives an electric generator
- Thermax can be used in rubbers, insulation, refractories, metallurgy, concrete, ceramics, in **thermoplastics, elastomers and their composites**

Thermal Black vs. Furnace Black

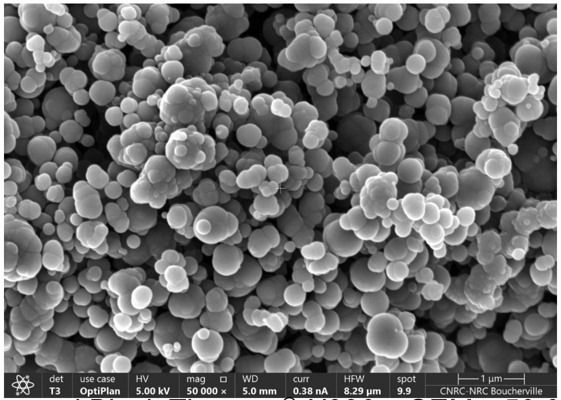
Particle Size Diameter and Structure



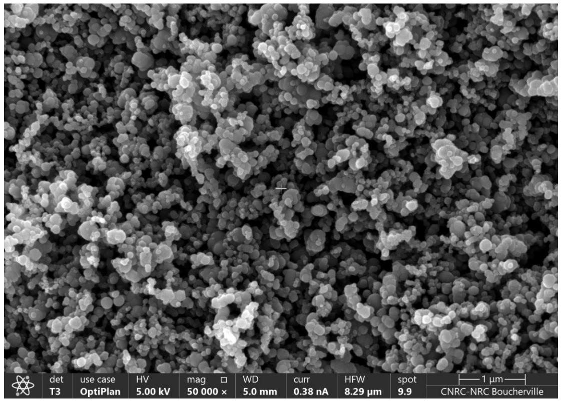
- Carbon black can be defined as very fine particle aggregates of carbon, possessing a paracrystalline molecular structure, while Thermal Black has an amorphous structure
- The main distinction between Thermal Black and Furnace black are particle size and structure
- Thermal Black, due to its higher particle size and lower structure, compared to even the most coarse furnace black, can be translated into excellent properties of the thermoplastic composites

Outline

- ➢ **State-of-the-art on HDPE films**
- ➢ **Compounds and cast-film extrusion**
- ➢ **Mechanical/Thermal/Morphology Characterizations**
  - **Cast films**
  - **Biaxially stretched films**
  - **Blown films**
- ➢ **Complementary characterization**
  - **Barrier properties**
  - **Optical properties**
- ➢ **Conclusions**



Thermal Black Thermax® N990 – SEM, x50,000



Carbon Black N762 – SEM, x50,000

State-of-the-art on HDPE films

Data from manufacturers of HDPE recommended for film applications

TS at break MD/TD (MPa)	Elongation at break MD/TD (%)	Film thickness (µm)	Grade/Manufacturer	Recommended Applications
62 / 52	350 / 400	Not specified	STAR HDPE F8952, Global Plastics, LLC	Bags, Film Industrial Applications, Liners
62 / 28	300 / 410	Not specified	STAR HDPE F5949, Global Plastics, LLC	Bags, Film Industrial Applications, Liners
36 / 31	680 / 690	20	Terralene® LL 1101, HDPE + LLDPE, FKuR Kunststoff GmbH	Blown films for different applications
60 / 56	400 / 550	15	SABIC® HDPE F00952, High Density Polyethylene Copolymer, SABIC	Films for high strength grocery sacks, shopping bags and high quality thin films for multi wall sack liners and replacement for thin paper products.
61 / 31	240 / 450	12	SCG HDPE H5604F, High Density Polyethylene, SCG Chemicals Co., Ltd	For Bags, General Purpose & Heavy-duty Bags (from blown process)
50 / 55	250 / 300	15	ALCUDIA® HDPE TR-156, High Density Polyethylene film, REPSOL	Carrier bags, Industrial liners
57 / 41	300 / 540	13	Formolene® E922, High Density (MMW) Polyethylene, Formosa Plastics Corporation, U.S.A.	Shopping Bags, Trash Can Liners, Thin Film for Laminate Applications
32 / 17	500 / 700	15	Jam 486H2, High Density Polyethylene, Jam Petrochemical Company	Blown films for different applications

State-of-the-art on HDPE films

Data from TDS of commercial HDPE films

Commercial HDPE films

TS at break MD/TD (MPa)	Elongation at break (%)	Film thickness (µm)	Reference	Recommended Applications
24 / 25.5	500 to 1000	Not specified	https://www.cheeverspecialty.com/specialty-paper-industries/building-and-construction	Films for building and construction Industry House wrapping for wind and moisture protection
46 / 37	810 / 930	15 - 60	https://www.molgroupchemicals.com/userfiles/products/48/48_tds_en.pdf	Films for shopping and garbage bags
50 / 45	400 / 450	20	http://www.b2bpolymers.com/TDS/SABIC_FI0750.pdf	Films from blown film extrusion for uses in: heavy duty bags, grocery sacks, shopping bags, carrier- and T-shirt bags, refuse bags, liners for multi-wall sacks and liners for frozen food meat
27 to 29	Not Specified	100 - 127	https://www.mcmaster.com/hdpe-film/	Moisture-Resistant Polyethylene (HDPE) Film For construction interior/exterior application
27.5 27.5	> 600 > 600	381 760	https://catalog.cshyde.com/viewitems/films/hdpe-film-high-strength	HDPE is moisture resistant and offers good heat resistance
85 / 45	590 / 780	12.5	https://fkur.com/en/brands/im-green/green-hdpe/green-hdpe-sgm-9450-f/	Green HDPE SGM 9450 F for: Blown films, Film rolls, Geodesic film

Compounding and cast-film processing

Extrusion:
HDPE 8800S - grade recommended for films for industrial, shopping and trash bags, and for agriculture.
Thermal Black (TB) Thermax ® N990 - from Cancarb Ltd.
Furnace Carbon Black (CB) N762 was used as reference.

Matrix	TB N990 content wt.%						CB N762 content wt.%
HDPE	0	3	10	20	**15**	40	20

These compounds were used further in cast-film extrusion, biaxial stretching and film blown processes.

Film casting:
Cast films were prepared with a thickness of ca. 100 µm from each formulation and from pristine HDPE as well.

The cast film containing 40 wt.% TB proved to be brittle, therefore a dilution was done down to HDPE / 15 wt.% TB and extruded to obtain again cast films.

Compounding

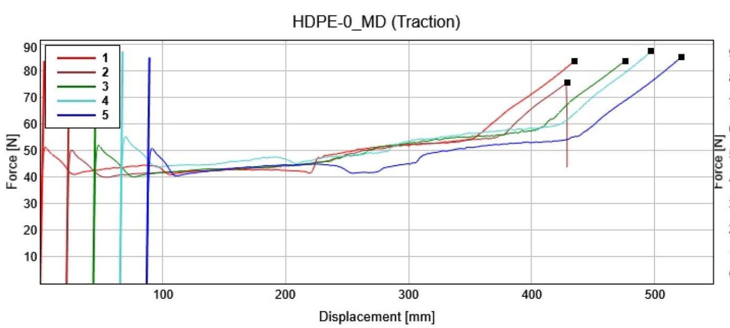
Extrusion casting

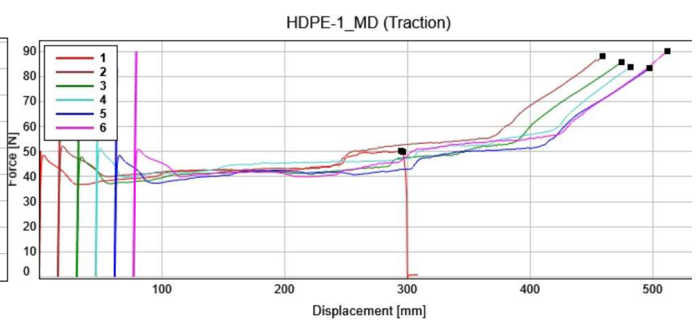
Characterization of HDPE / TB cast films

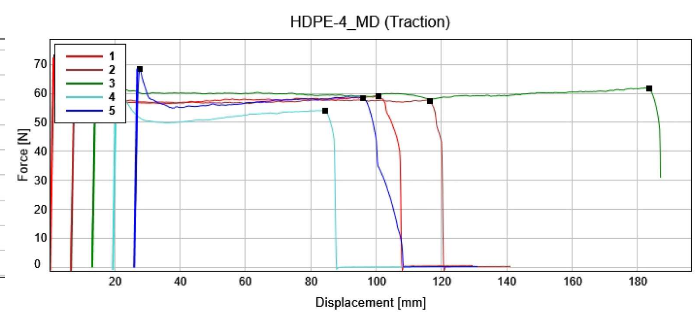
<u>Mechanical testing</u>:

- Tensile properties were evaluated according to ASTM D882 - 18: *Standard Test Method for Tensile Properties of Thin Plastic Sheeting;* Testing parameters: 5 kN cell, 100 mm grips distance, 500 mm/min speed
- All samples were conditioned at 23 °C, 50% RH and 40 hours and tested in Machine Direction (MD)
- At TB contents from 3 up to 20 wt.%, TS was 26 to 38 MPa and Elongation was 690 to 880 % - similar performance as for heavy duty bags, grocery sacks, shopping bags, carrier- and T-shirt bags, construction interior/exterior films..

Thermal Black content wt.%	**Thickness µm**	SD Thickness mm	**Tensile Modulus MPa**	SD TM MPa	**Tensile Strength MPa**	SD TS MPa	**Elongation %**	SD %
0	112	0.00	823.2	151.3	37.2	1.8	856.4	23.8
3	112	0.00	958.4	34.4	38.5	2.4	877.1	9.2
10	112	0.00	1043.4	87.4	26.9	3.9	702.7	54.2
20	122	0.00	1055.6	136.9	26.5	3.6	688.2	68.0
20 (CB)	135	0.01	886.8	208.6	25.0	4.6	398.8	311.1
40	144	0.01	1224.3	120.9	24.5	1.1	3.9	0.2

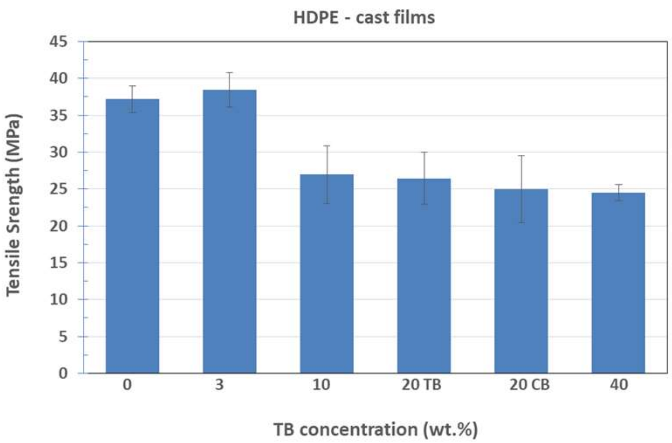


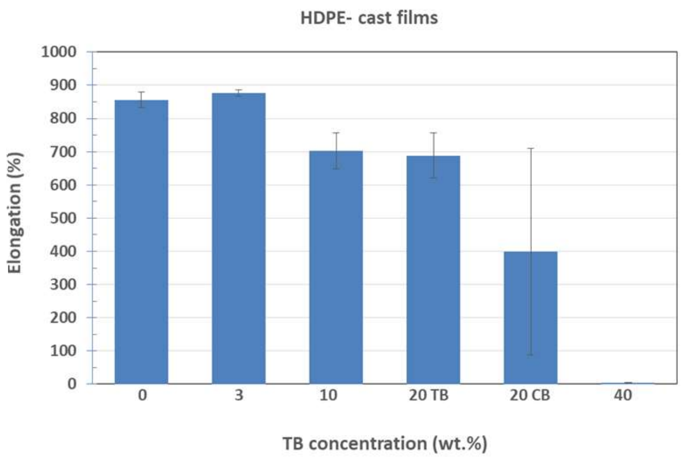


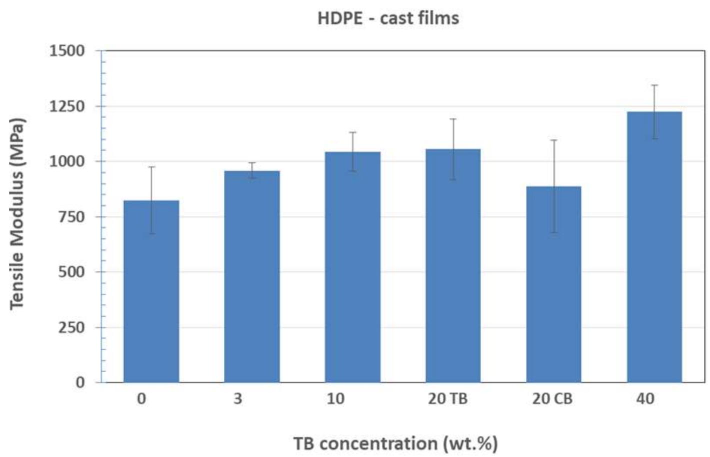


Characterization of HDPE / TB cast films

<u>Tensile results</u>: TS values for HDPE-TB cast films are in the same range to commercial HDPE films recommended for moisture-resistant films for construction interior/exterior application: i.e. at similar film thickness TS = 25-29 MPa (commercial, as per slide 7) vs. TS = 25-37 MPa (HDPE-TB cast films)







Due to its very low elongation %, HDPE/40 wt.% TB was diluted down to HDPE/15 wt.% TB. Cast film of HDPE/15 wt.% TB was also extruded and it was further characterized.

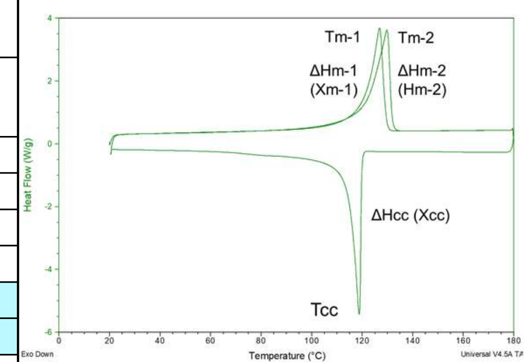
Characterization of HDPE / TB cast films

Differential Scanning Calorimetry DSC:

The DSC analysis was done between 20 °C and 180 °C, at a rate of 10 °C/min.

TB Content wt.%	Heat 1 Behavior after casting			Cooling			Heat 2 Innate behavior		
	ΔHm-1 J/g	**XH1 %**	Tm-1 °C	ΔHcc J/g	Xcc %	Tcc °C	ΔHm-2 J/g	XH2 %	Tm-2 °C
0	147.4	**50.3**	125.9	143.7	49.0	118.9	162.8	55.6	129.8
3	141.0	**49.6**	126.8	135.7	47.7	120.2	155.6	54.7	129.7
10	129.9	**49.3**	126.6	125.6	47.6	118.9	144.2	54.7	129.6
15	127.4	**51.2**	126.7	126.5	50.8	119.1	139.4	56.0	129.5
20	112.7	**48.1**	126.6	108.9	46.5	118.7	124.3	53.0	129.6
20 (CB)	127.8	**54.5**	126.6	123.2	52.6	119.2	141.2	60.2	129.2
40	91.6	**52.1**	126.4	88.3	50.2	119.3	101.6	57.8	129.2

The enthalpy of 100% perfectly crystalline form of HDPE ***is 293 J/g***



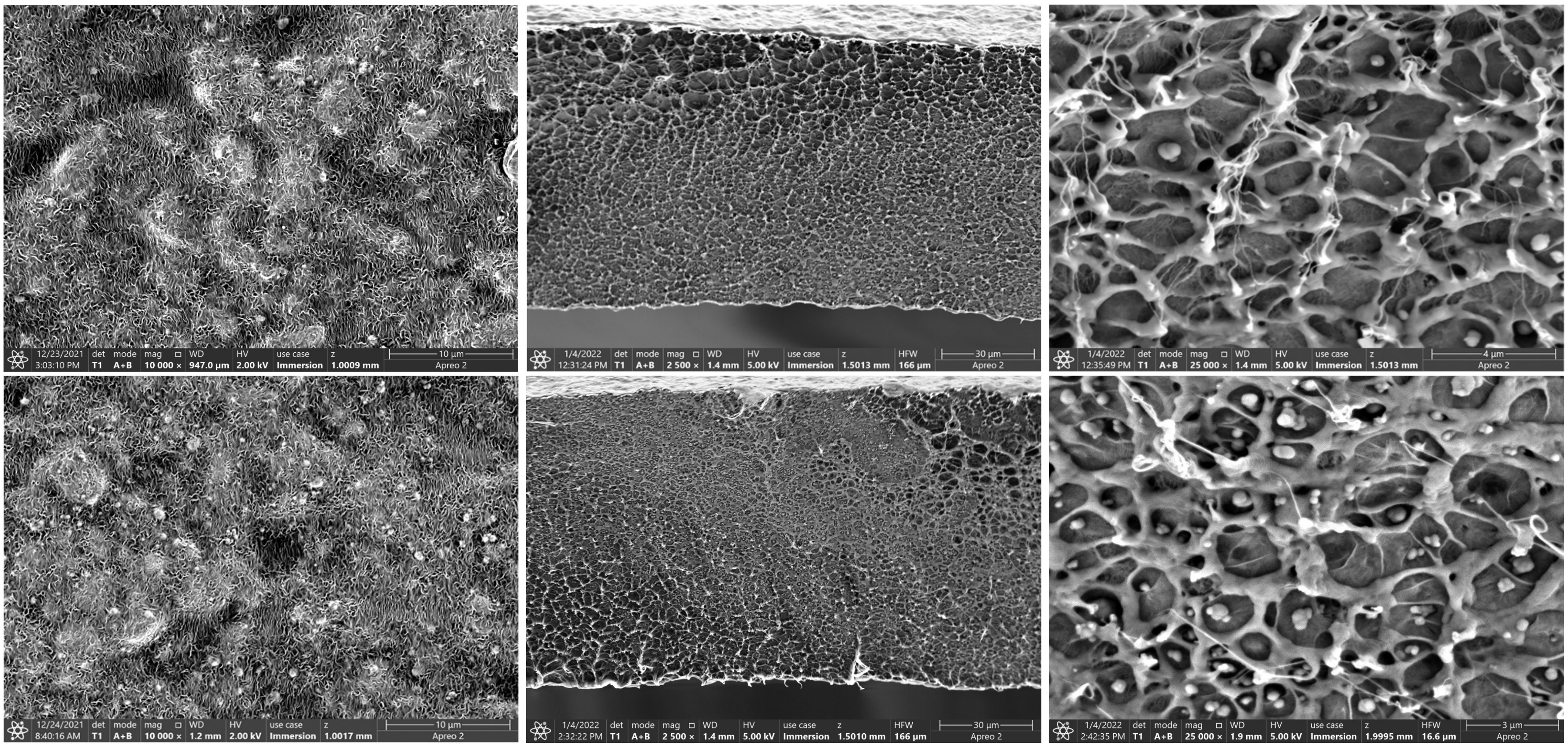
- The melting temperatures, Tm, for both heating cycles and all composites, remained unchanged with increasing TB content compared to pure HDPE, i.e. around 126 ºC for the Heat 1 cycle and around 129 ºC for Heat 2 cycle
- Crystallization temperature, Tc, for the cooling cycles, remained also unchanged with increasing TB content, around 119 ºC
- High crystallinity contents were observed, near 50% and beyond!
- The composites containing 20 wt.% CB present slightly higher crystallinities than 20 wt.% TB because of their smaller diameter

Characterization of HDPE / TB cast films
SEM morphology

x 10,000 surface | x 2,500 cross section | x 25,000 cross section

3 wt.% TB

10 wt.% TB

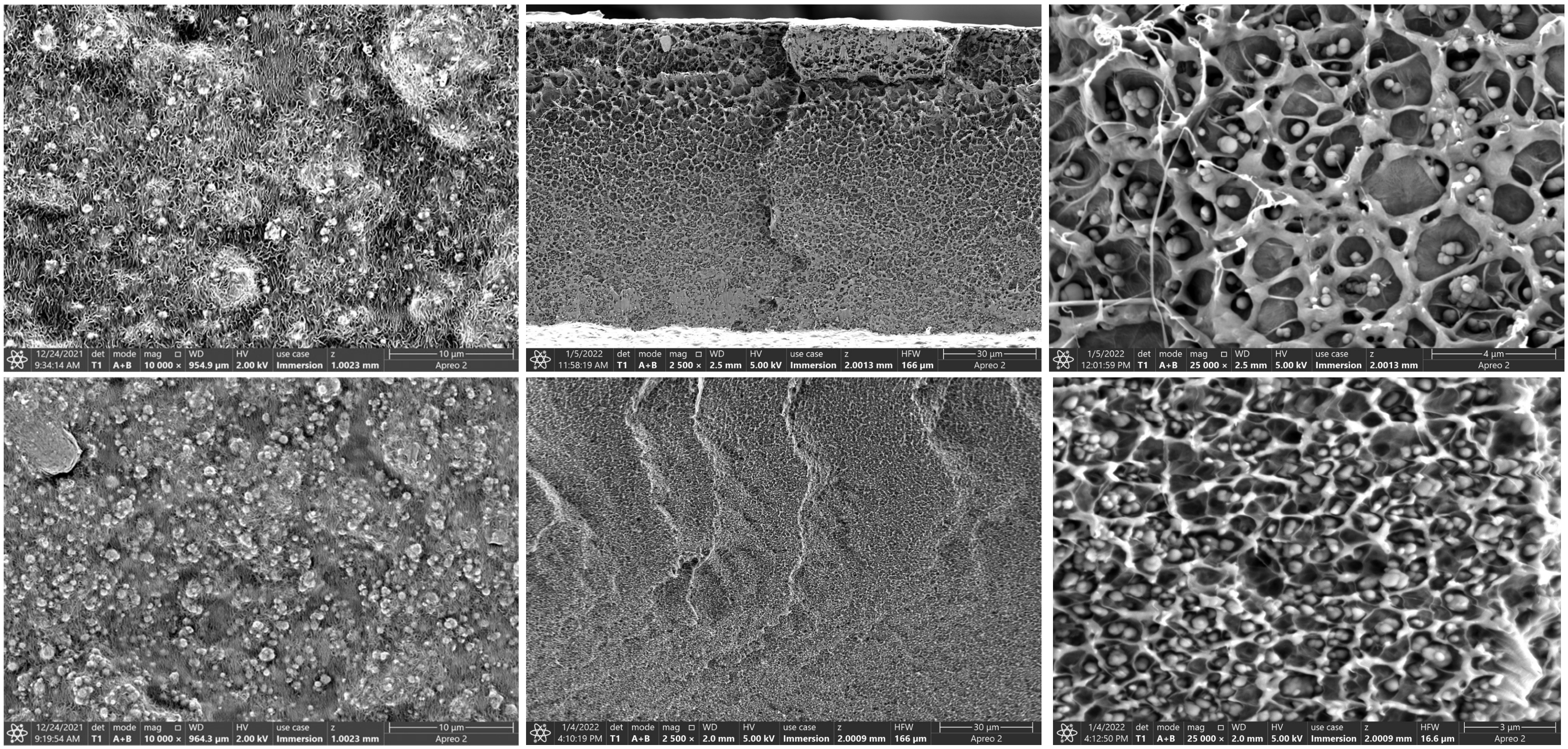


Characterization of HDPE / TB cast films
SEM morphology

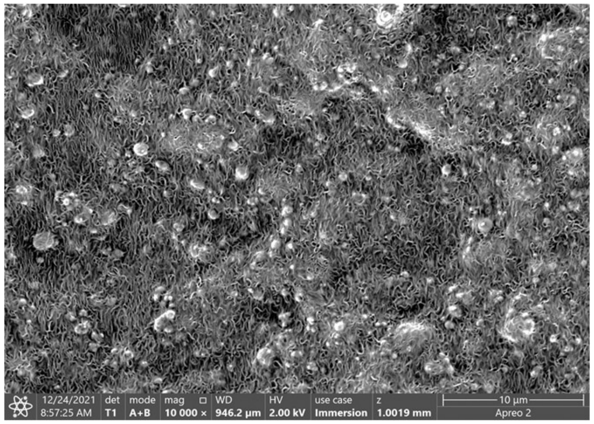
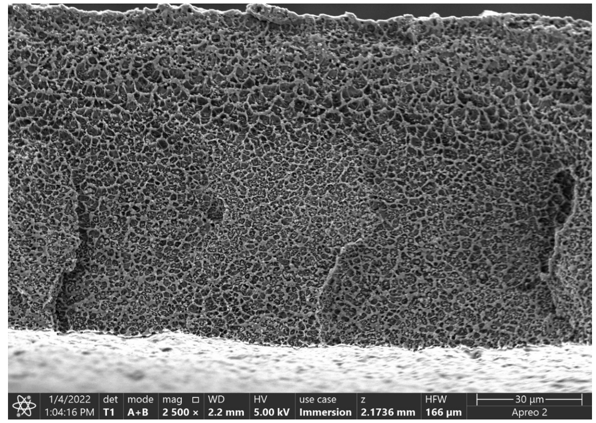
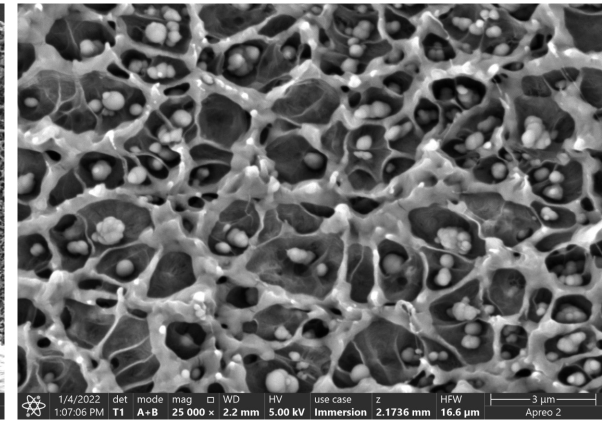
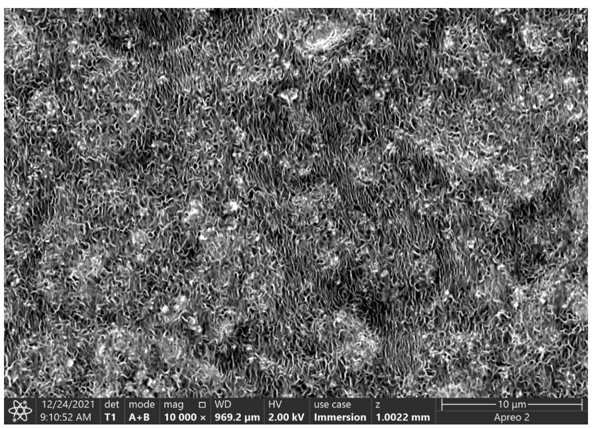
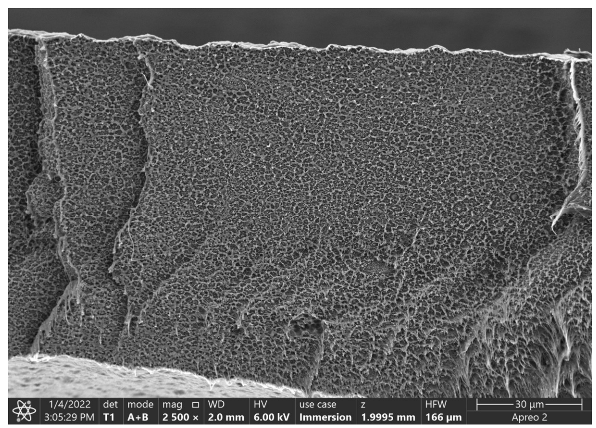
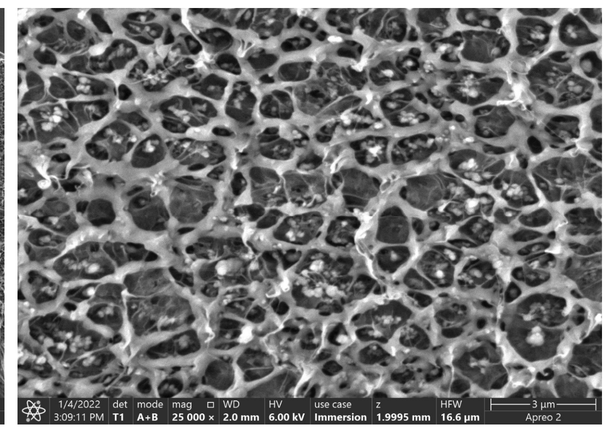
x 10,000 surface | **x 2,500 cross section** | **x 25,000 cross section**

15 wt.% TB

40 wt.% TB



Characterization of HDPE / TB cast films
SEM morphology

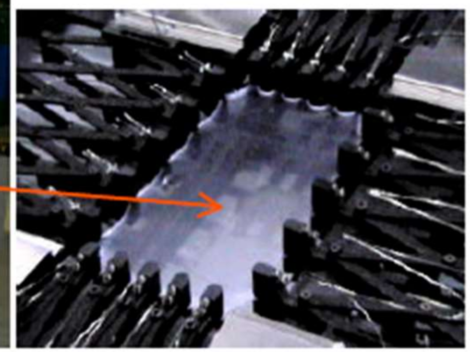
	x 10,000 surface	x 2,500 cross section	x 25,000 cross section
20 wt.% TB			
20 wt.% CB			

Bi-axial stretching of HDPE / TB cast films

Objective:

To test the processability of HDPE-TB cast films in bi-axial stretching process and to evaluate the performance of obtained stretched films using similar characterization methods as for cast films.

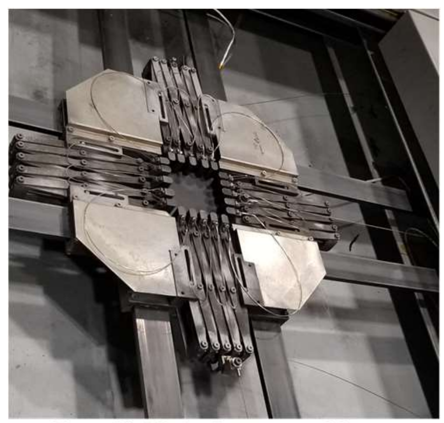
Biaxial stretcher apparatus (Karo IV from Bruckner)



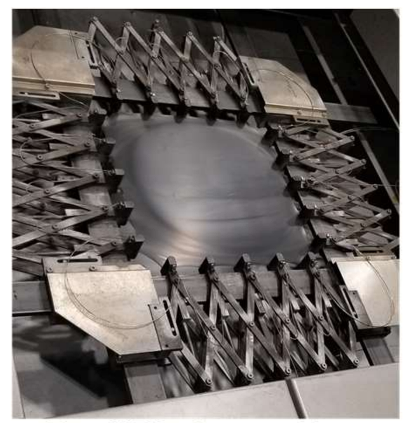
Clamping section of the stretcher

Stretching conditions:

Pre-heating time: 5 sec
Pre-heating temperature: 130 ^{o}C



20 wt.% TB, before stretching



20 wt.% TB, after stretching

Bi-axial stretching
Characterization of bi-ax HDPE / TB films

Mechanical testing:

- Tensile properties according to ASTM D882 - 18: *Standard Test Method for Tensile Properties of Thin Plastic Sheeting*
  - *5 kN cell, 50 mm grips distance, 500 mm/min speed*
- All samples were conditioned at 23 °C, 50% RH and 40 hours and tested in Machine Direction (MD)
- Bi-axial stretched films of HDPE-TB (TB from 3 to 40 wt.%) demonstrated similar performance in tensile deformation as pristine HDPE; There is a fit in the performance for bags and liners applications (even higher performance)

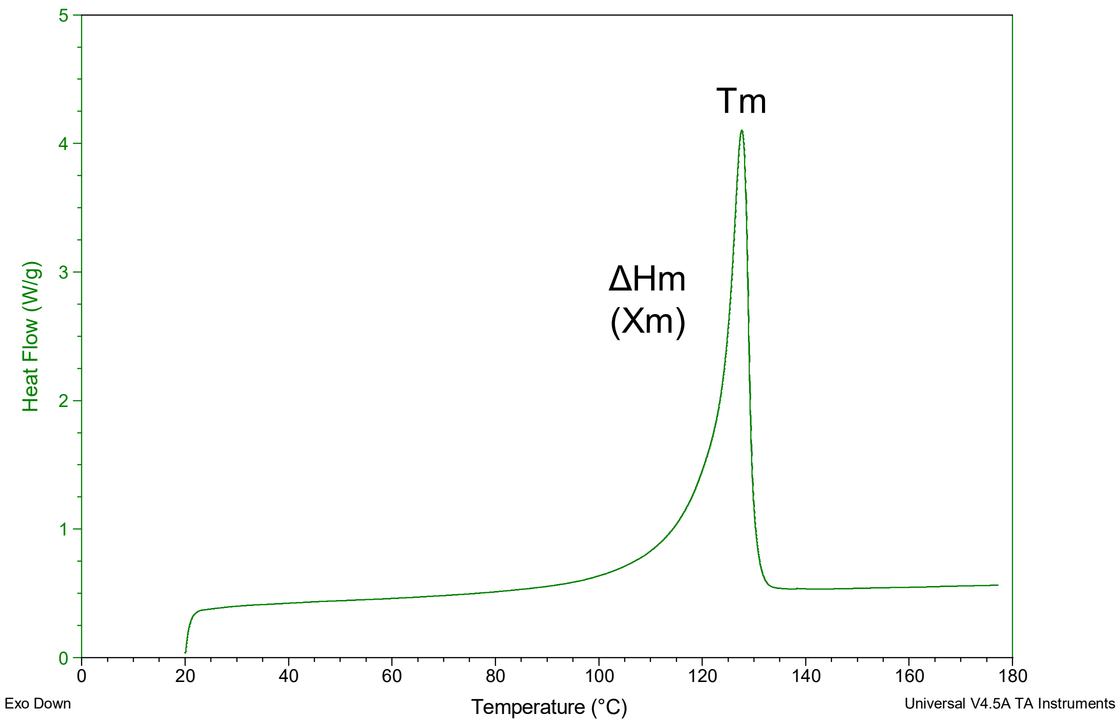
Stretch ratio	Thermal Black content wt.%	Thickness µm	SD Thickness mm	Tensile Modulus MPa	SD TM MPa	Tensile Strength MPa	SD TS MPa	Elongation %	SD %
2x2	0	15	0.01	1694.4	269.5	81.9	18.3	212.2	40.6
4x4	0	8	0.00	1008.1	339.9	29.7	11.3	11.9	6.3
2x2	3	12	0.01	1424.1	373.3	43.6	14.5	80.3	39.3
4x4	3	7	0.00	1718.3	1232.0	30.8	16.9	7.5	3.2
2x2	10	-	-	-	-	-	-	-	-
4x4	10	7	0.00	1828.0	1132.2	15.9	4.7	6.5	1.2
2x2	15	15	0.01	1823.3	195.1	86.2	22.8	126.5	43.5
4x4	15	7	0.00	1557.3	290.9	24.4	10.3	5.4	1.6
2x2	20	20	0.01	1985.4	426.1	76.5	9.5	127.7	57.4
4x4	20	7	0.00	1381.7	151.9	40.8	9.7	9.9	2.7
2x2	20 (CB)	40	0.01	1034.2	413.1	45.9	14.2	101.7	19.9
4x4	20 (CB)	18	0.00	684.0	65.6	29.5	12.0	16.2	12.1
2x2	40	32	0.01	1758.7	456.9	65.9	23.2	109.1	27.4
4x4	40	7	0.00	1836.2	554.7	41.2	12.9	5.7	3.4

Bi-axial stretching
Characterization of bi-ax HDPE / TB films

Differential Scanning Calorimetry analysis:

Only a heat cycle (ramp 10.00 °C/min up to 180.00°C) was applied with the purpose to evaluate if the bi-axial stretching developed more crystallinity starting from the one of cast films. The supplementary crystallinity resulted from bi-axial stretching was calculated.

No crystallization took place during the heating cycles for each bi-axial stretched sample.



Bi-axial stretching
Characterization of bi-ax HDPE / TB films

	TB Content wt.%	Heat 1 Behavior after casting			Heat 1 Behavior after stretching			Crystallinity Difference
		ΔHm-1 J/g	XH1 %	Tm-1 °C	ΔHm-1 J/g	XH1 %	Tm-1 °C	XH1 bi-ax - XH1 cast %
cast	0	147.4	50.3	125.9	NA	NA	NA	NA
bi-axe					170.2	58.1	127.6	7.8
bi-axe					170.1	58.1	128.0	7.7
bi-axe					171.4	58.5	127.9	7.7
cast	3	141	49.6	126.8	NA	NA	NA	NA
bi-axe					161.8	56.9	127.9	7.3
bi-axe					161.7	56.9	128.6	7.3
bi-axe					166.3	58.5	127.9	7.7
cast	10	129.9	49.3	126.6	NA	NA	NA	NA
bi-axe					156.5	59.3	127.5	10.1
bi-axe					152.6	57.9	128.0	8.6
bi-axe					171.5	65.0	132.1	7.7
cast	15	127.4	51.2	126.7	NA	NA	NA	NA
bi-axe					158.8	63.8	127.1	12.6
bi-axe					155.0	62.2	127.8	11.1
bi-axe					159.1	63.9	127.2	7.7
cast	20	112.7	48.1	126.6	NA	NA	NA	NA
bi-axe					146.5	62.9	127.3	14.8
bi-axe					145.2	61.9	126.9	13.9
bi-axe					151.7	64.7	127.1	7.7
cast	20 (CB)	127.8	54.5	126.6	NA	NA	NA	NA
bi-axe					150.4	64.2	127.4	9.6
bi-axe					145.5	62.1	127.7	7.6
bi-axe					151.7	64.7	127.2	7.7
cast	40	91.6	52.1	126.4	NA	NA	NA	NA
bi-axe					114.3	65.0	129.3	12.9
bi-axe					110.7	63.0	127.0	10.9
bi-axe					112.3	63.9	127.0	7.7

DSC analysis:

More crystallinity is developed during bi-axial stretching for all HDPE / TB films (compared at cast films).

The crystallinity can increase from 50 %, for pure HDPE cast film, up to 65 % for HDPE / 40 wt.% TB stretched at 4x4

The crystallinity increased with TB content

Crystallinity presence and new-born crystals during bi-axial stretching might mean formation of pores, therefore a possibly increase in O_2 and water vapor permeabilities…

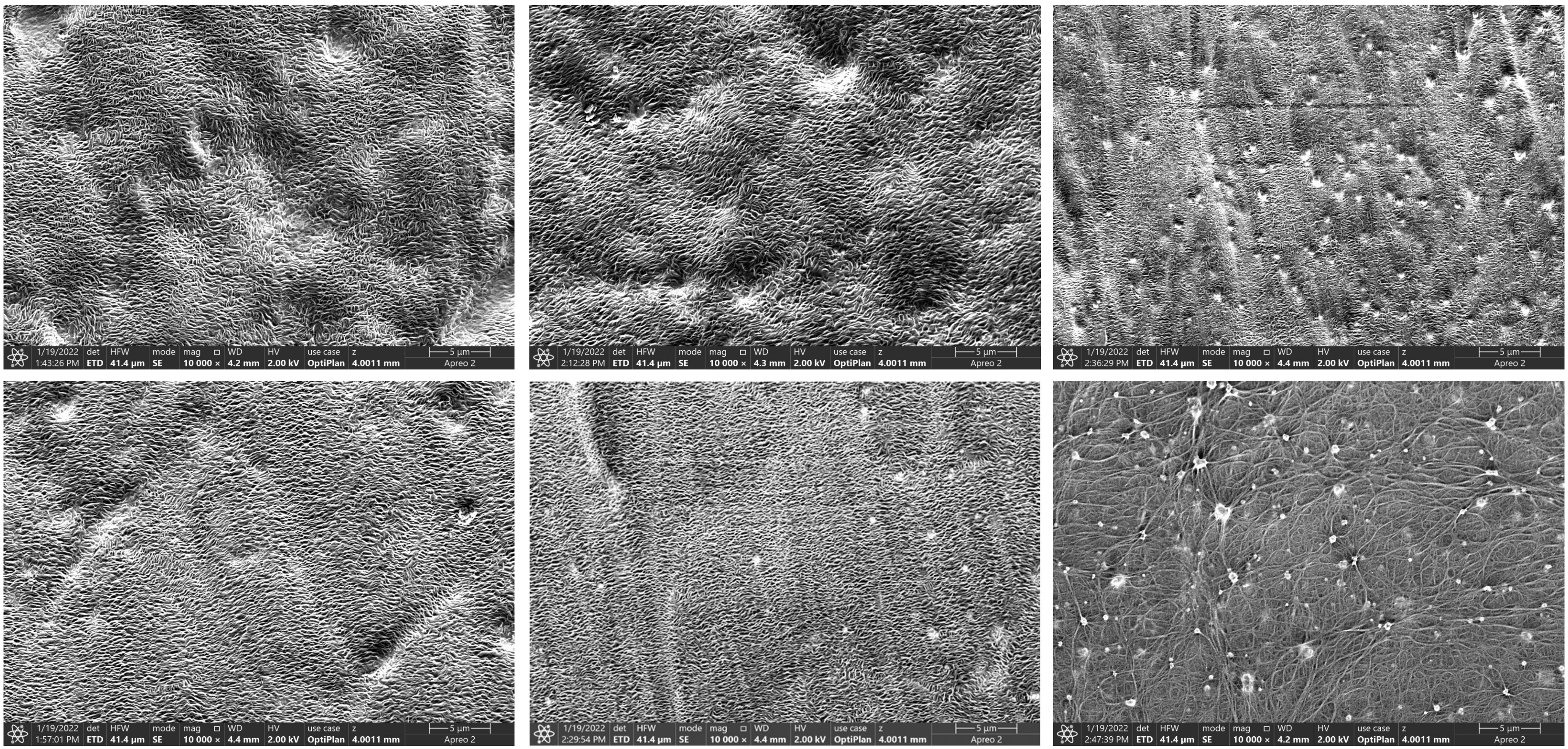
Bi-axial stretching
SEM of bi-ax HDPE / TB films

x 10,000 surface

0 wt.% TB | **3 wt.% TB** | **10 wt.% TB**

2x2

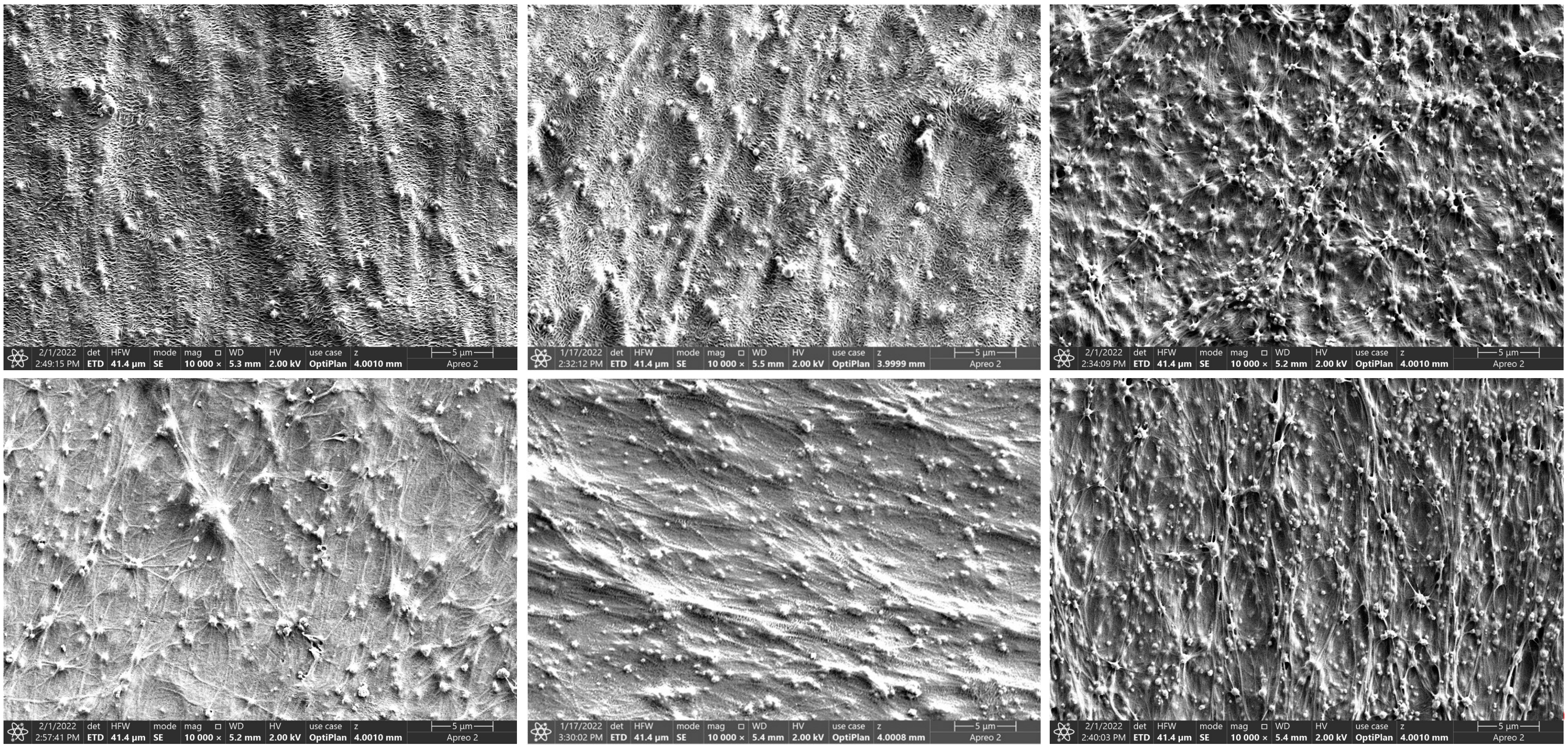
4x4



Bi-axial stretching
SEM of stretched HDPE / TB films

x 10,000 surface

	15 wt.% TB	20 wt.% TB	40 wt.% TB
2x2			
4x4			



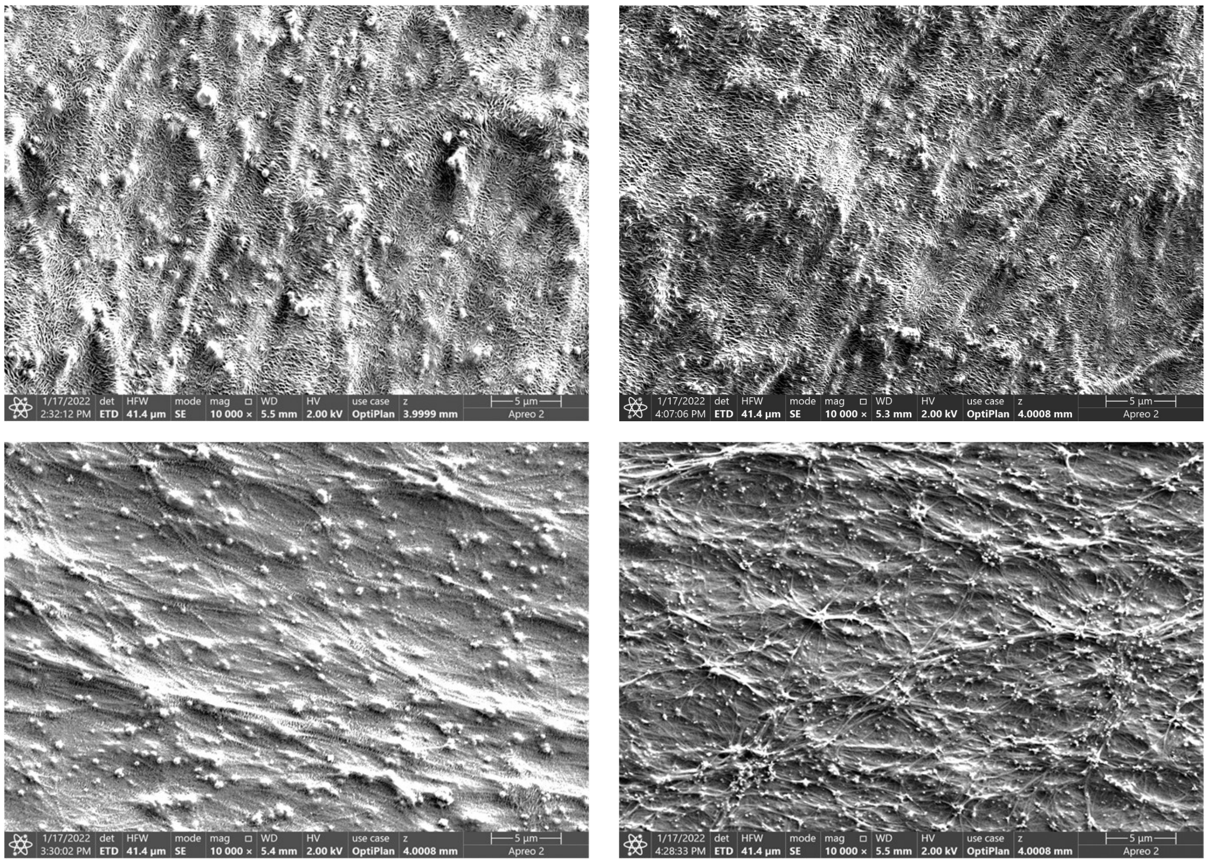
Bi-axial stretching
SEM of bi-ax HDPE / TB films

x 10,000 surface

20 wt.% TB | **20 wt.% CB**

2x2

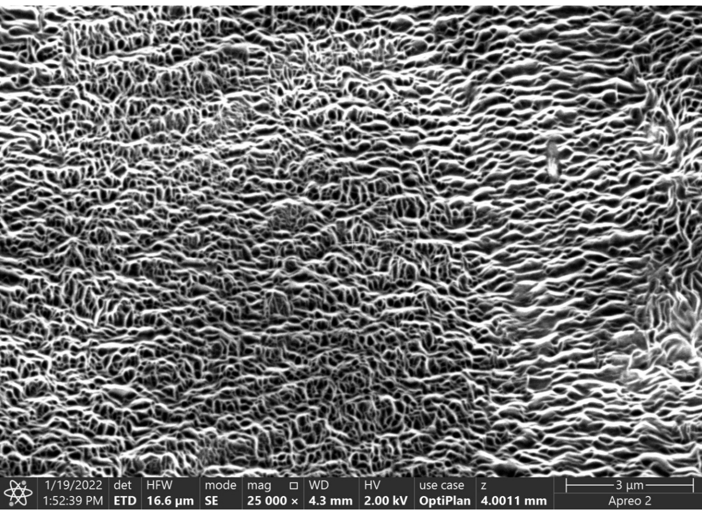
4x4



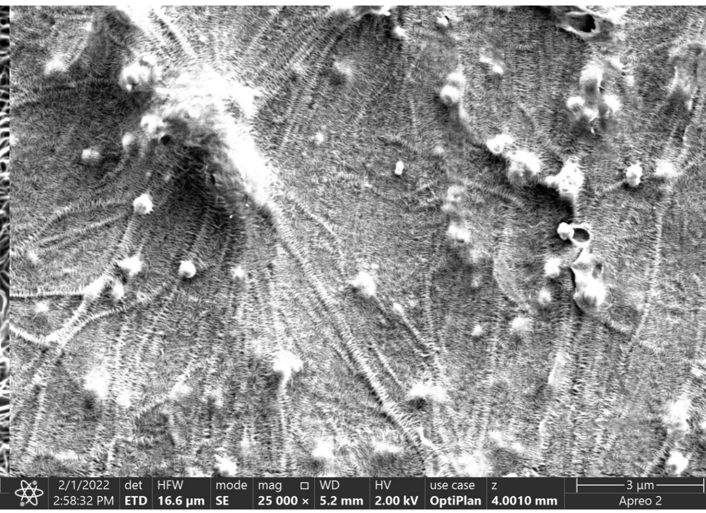
Bi-axial stretching
SEM of bi-ax HDPE / TB films

x 25,000 surface

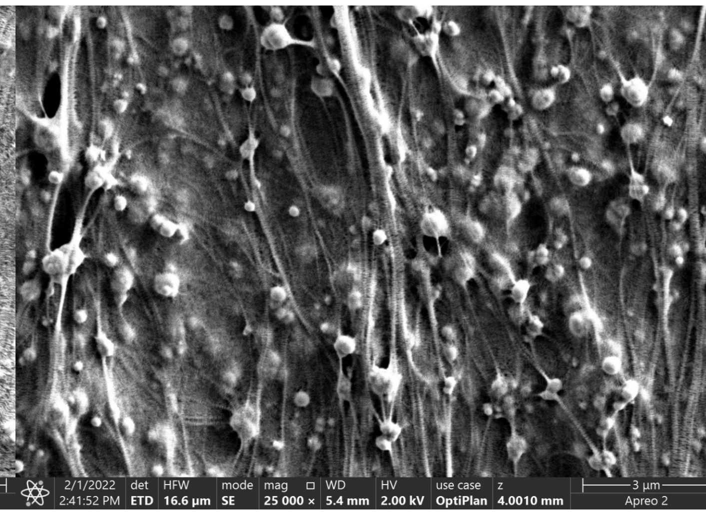
2x2
0 wt.% TB



4x4
15 wt.% TB



4x4
40 wt.% TB



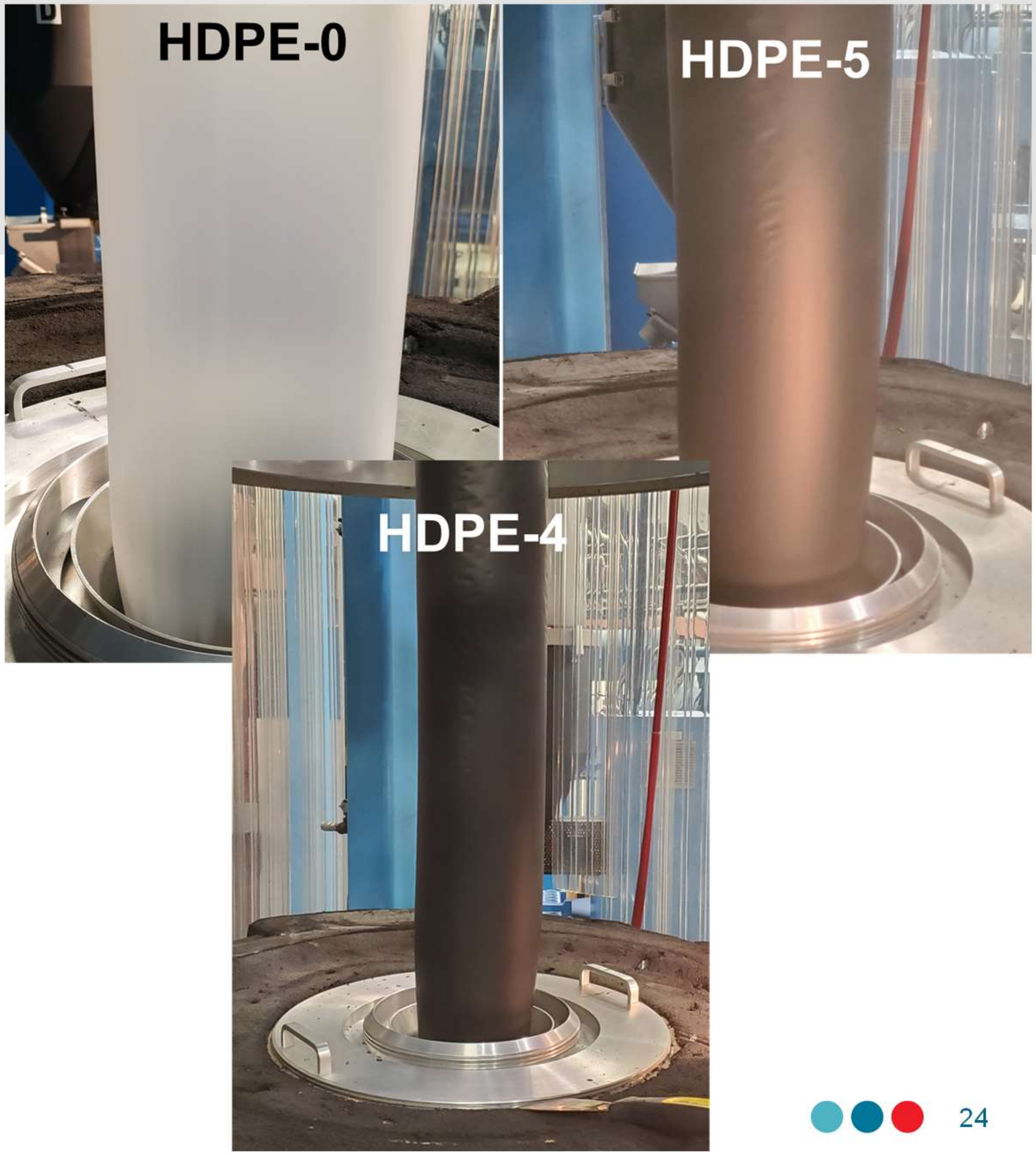
Film blowing

Objective:

To test in film blowing process two (2) optimal HDPE / TB formulations using the NRC's semi-industrial scale film blowing line, Brampton Engineering with a 100 mm die.

The obtained films were tested using the same methods as for cast and bi-axial stretched films.

Name of blown film	Thickness
HDPE-0 reference	around 50 microns
HDPE-5 (15 wt.% TB)	around 90 microns
HDPE-4 (40 wt.% TB)	around 170 microns



Film blowing
Characterization of blown HDPE / TB films

Tensile testing:

- Tensile properties according to ASTM D882 - 18: *Standard Test Method for Tensile Properties of Thin Plastic Sheeting*
  - *5 kN cell, 50 mm grips distance, 500 mm/min speed*
- All samples were conditioned at 23 ^{o}C, 50% RH and 40 hours and tested in Machine Direction (MD)
- Samples were tested in MD direction
- Blown film based on HDPE-5 (15 wt.% TB) demonstrate adequate properties for uses in shopping and garbage bags

Blown film	Thermal Black content wt.%	Thickness µm	SD mm	Tensile Modulus MPa	SD TM MPa	Tensile Strength MPa	SD TS MPa	Elongation %	SD %
HDPE-0 from TDS	0	10	-	-	-	61.8	-	260	-
HDPE-5	15	90	10	834.7	113.2	24.5	4	669	138.4
HDPE-4 (!)	40	170	10	1084.1	148.2	24.5	2.7	5	0.3

Film blowing
Characterization of blown HDPE / TB films

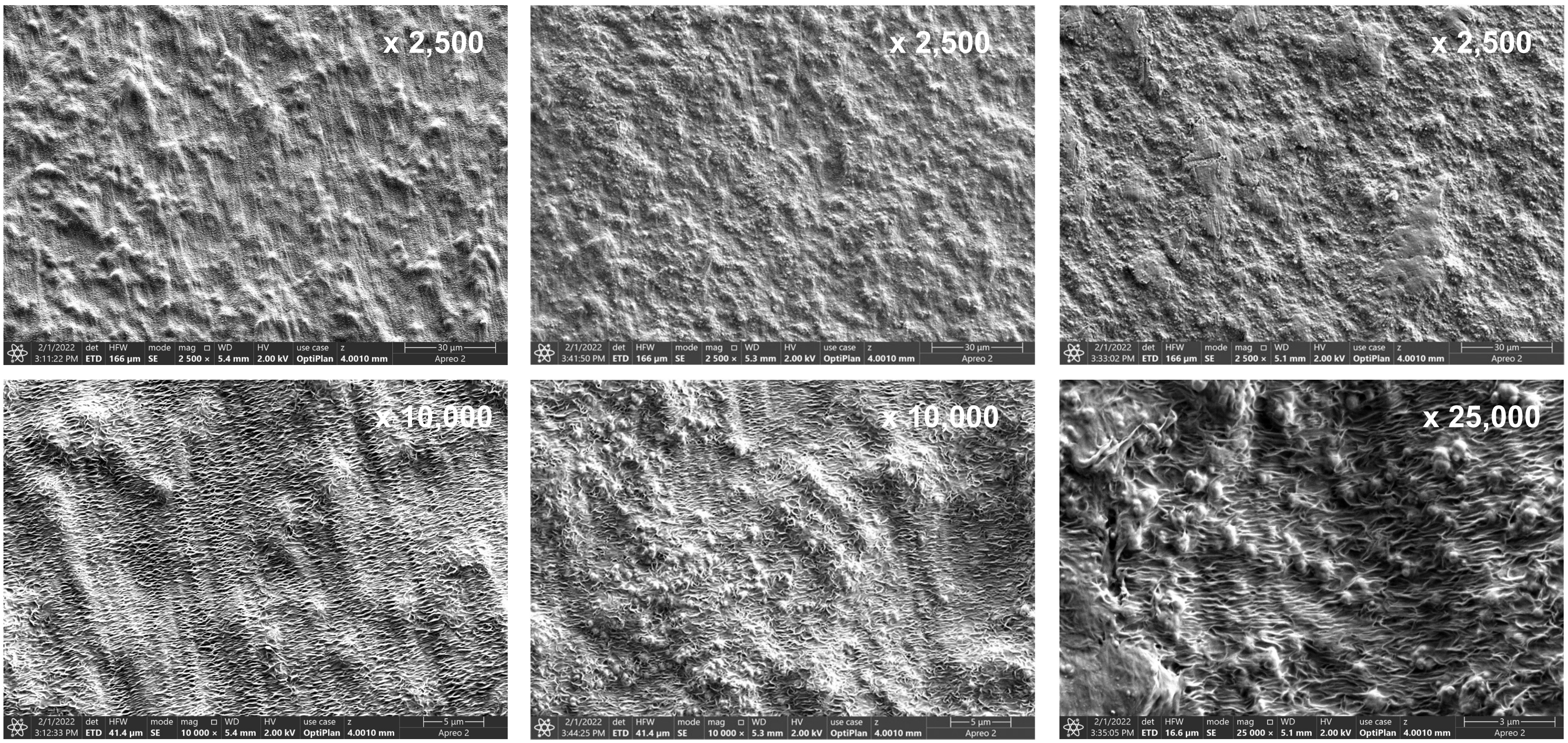
Differential Scanning Calorimetry analysis:

- A heat cycle (ramp of 10°C/min up to 180°C) was applied with the purpose to evaluate the crystallinities formed during the film blowing process.
- No crystallization took place during the heating cycles for blown film samples (as for the bi-ax stretched ones).
- Almost full crystallization are observed for HDPE-TB blown films during their processing (blowing is a kind of bi-axial stretching, similar to the one applied in Brűckner bi-ax stretcher).

		TB Content Wt.%	Heat 1 Behavior after processing		
			ΔHm-1 J/g	XH1 %	Tm-1 °C
cast	**HDPE-0**	0	147.4	50.3	125.9
blown			165.7	56.6	128.7
cast	**HDPE-5**	15	127.4	51.2	126.7
blown			143.5	57.6	128.2
cast	**HDPE-4**	40	91.6	52.1	126.4
blown			110.1	62.6	128.1

Film blowing
SEM of blown HDPE / TB films

0 wt.% TB | **15 wt.% TB** | **40 wt.% TB**



Cast, bi-axial stretched and blown HDPE / TB films - Barrier properties

Sample	TB wt.%	Thickness µm	Crystallinity %	ASTM D-3985 OTR cc/m^2.day	SD	ASTM F2714-08 2013 OTR cc/m^2.day	ASTM F-1249-06 WVTR g/m^2.day	SD
HDPE* reference	**Pure**	**25**	**50 to 58**	**3000**	**NA**	**NA**	**9**	**NA**
HDPE-1, cast	3	112	49.3	878	386	NA	0.6	0.0
HDPE-2, stretched 2 x 2	10	15	59.3	2836	568	NA	3.5	0.5
HDPE-3a, stretched 3 x 3	20	14	61.9	5196	1639	NA	5.1	1.3
HDPE-4, bi-axe 4 x 4	40	40	63.9	NA	NA	754, 19 069, 216	108.0	19.6

*http://usa.dupontteijinfilms.com/wp-content/uploads/2017/01/Oxygen_And_Walter_Vapour_Barrier_Properties_of_Flex_Pack_Films.pdf

Conclusions OTR:
HDPE film has, in general, **a low to fair oxygen barrier properties**.
OTR values increased with TB content and the level of stretching, i.e. proportionally to the crystallinity content of tested films
HDPE films containing 3 and 10 wt.% TB has an OTR to HDPE (fair OTR). HDPE / 20 wt.% and 40 wt.% TB were highly permeable
*An industry rule-of-thumb is that a film material is considered a "high oxygen barrier" if its OTR is **less than 15.5 cc/m^2/day***

Conclusions WVTR:
HDPE film has, in general, good water vapor barrier properties.
WVTR values increased with TB content and the level of stretching, i.e. proportionally to the crystallinity content of tested films.
HDPE films containing 3 to 20 wt.% TB have an WVTR under the one of HDPE. HDPE with 40 wt.% TB is highly permeable to water vapors.

OTR & WVTR increased with crystallinity content in films. Att: these crystallinity development in films depend on the parameters used for cast and stretching processes. The HDPE-TB crystallinities can be further fine-tuned to achieve other values for OTR and WVTR for specific applications.

Cast, bi-axial stretched and blown HDPE / TB films - Optical properties

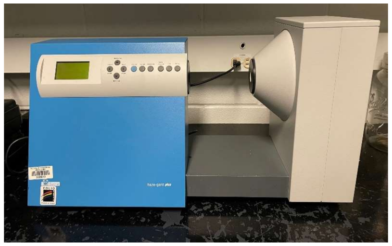
Transparency (transmittance):
- Measurement according to ASTM D1746-09
- Haze-guard plus from BYK Gardner was used
  - No sample→ 100% transparency
  - Blocked light path → 0% transparency

Conclusions:
- All cast and blown films with fillers have 0 transparency
- For bi-axial stretched films with filler:
  - 2x2 stretch ratio is not transparent except if filler is minimum quantity
  - Increased stretch ratio (3x3 and 4x4 increases film transparency)
- High potential to block the UV

Biax film	Average T Value	SD
HDPE_0_2x2	93.3	0.3
HDPE_0_3x3	93.4	0.4
HDPE_0_4x4	93.5	0.2
HDPE_1_2x2	27.5	8.6
HDPE_1_3x3	63.3	7.2
HDPE_1_4x4	75.6	2.5
HDPE_2_2x2	0.4	0.2
HDPE_2_3x3	28.3	5.1
HDPE_2_4x4	37.8	1.7
HDPE_3a_2x2	0.1	0.1
HDPE_3a_3x3	7.1	3.4
HDPE_3a_4x4	24.2	3.1
HDPE_3b_2x2	0.0	0.0
HDPE_3b_3x3	0.0	0.0
HDPE_3b_4x4	0.2	0.2
HDPE_4_2x2	0.0	0.0
HDPE_4_3x3	0.0	0.0
HDPE_4_4x4	0.9	0.4
HDPE_5_2x2	0.5	0.4
HDPE_5_3x3	8.9	10.2
HDPE_5_4x4	23.9	6.5

Cast film	Average T Value	SD
HDPE_0	91.6	0.1
HDPE_1	0	0
HDPE_2	0	0
HDPE_3a	0	0
HDPE_3b	0	0
HDPE_4	0	0
HDPE_5	0	0

Blown film	Average T Value	SD
HDPE_0	92.6	0.2
HDPE_4	0	0
HDPE_5	0	0



Conclusions

HDPE / TB films seem to be appropriate to replace HDPE in many applications;
They would be a lower-cost and a more eco-responsible choice due to TB eco-filler content

Blown HDPE / TB films
Thickness = up to 170 µm
TS MD = 25 MPa
ε MD = up to 670 %

HDPE / TB films from Cast extrusion
Thickness = 100 - 120 µm
TS MD = 25 - 38 MPa
ε MD = 690 - 880%
More ductil in puncture

HDPE / TB films from Biaxial stretching
Thickness = 7 - 30 µm
TS MD = 30 - 86 MPa
ε MD = up to 130%
More ductil in puncture

Increase mechanical performance (TS-MD; ε%)

Decrease film thickness (µm)

750 - 130
TS MD ≤ 27 MPa
ε MD ca. 600 - 800%

Commercial **PURE** HDPE film: Moisture resistant, good heat resistance

130 - 85
TS MD = 27 - 46 MPa
ε MD ca. 600%

Commercial **PURE** HDPE films: moisture-resistant film for construction interior/exterior application

85 - 12
TS MD = 32 - 85 MPa
ε MD = 240 - 680 %

Commercial **PURE** HDPE films: Carrier bags, industrial liners, shopping bags, trash can liners, thin film for laminate applications

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THANK YOU!

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