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PROOF-OF-CONCEPT EXPERIMENT REPORT

SURFACE ROUGHNESS AND WETTING PROPERTIES OF PLASTIC PIPES

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Proof-of-Concept experiment details

Received sample: the company provided three types of plastic pipes (length approximately 50 cm, diameter from 1.2 to 1.6 cm) with different compositions or processing parameters.

Sample number	Description	Material	Slippery, as reported by the company
1	Green pipe, perforated	HDPE	worst
2	Transparent pipe with blue lines, perforated	LDPE	excellent
3	Transparent pipe, not perforated	PP	good
4	Transparent pipe, not perforated	HDPE	very good

Main aim

The main aim of the POC experiment is to characterize the hydrophobicity of the samples, analyze the surface morphology and roughness and detect possible differences between three pipes with different compositions or processing parameters. The surface wetting properties are evaluated by contact angle measurements, surface morphology and roughness are evaluated using SEM and optical profilometry. In general, the aim is to see if surface analysis, from macroscale to nanometric level, can help in understanding the reasons for the different slippery properties of the samples.

Planned analysis:

* Contact angle measurements to determine hydrophobicity with water drops.

* SEM analysis of surface morphology and qualitative evaluation of surface roughness.

* Optical profilometry analysis of surface morphology and roughness.

Sample preparation:

For contact angle measurements: Pipes have been cut into 5 cm pieces, washed with soap and deionized water, and dried with nitrogen.

For SEM analysis: Pipes were cut to approximately 5x10 mm stripes, two samples were prepared from one type of pipe. Pieces were glued on glass support using conductive carbon tape. To make the surface of pipes conductive for SEM measurements 8 nm thick Au coating was applied by a sputter coating system (PECS, Gatan).

For optical profilometry: as for the contact angle measurements.

Measurement authors:

SEM @ UNG: the SEM analysis has been performed by Andraž Mavrič at the University of Nova Gorica.

Contact angle measurements @ IOM-CNR: contact angle measurements have been performed by Martina Conti, Simone Dal Zilio, and Andraž Mavrič at IOM-CNR.

Optical profilometry @ IOM-CNR: optical profilometry measurements have been performed by Erik Betz Gutner, Simone Dal Zilio, and Andraž Mavrič at IOM-CNR.

Observation/processing conditions:

Contact angle measurements @ IOM-CNR: Contact angle (CA) measurements were carried out on a DataPhysics OCA 15Pro optical instrument (DataPhysics Instruments GmbH, Germany) at ambient temperature by placing 20 µL of Milli-Q water onto the sensor surface.

Sample pretreatment for SEM measurements, Au sputtering @ UNG: Sputtering was performed by Precision etching coating system Gatan, Model 682.

SEM @ UNG: performed by JEOL JSM 7100f. Beam energy 15 KeV; secondary electron detector.

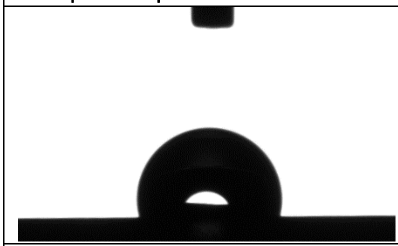
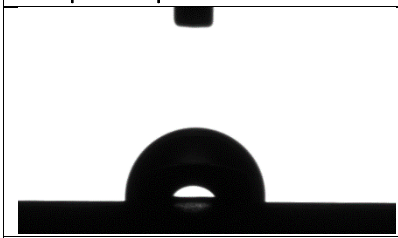
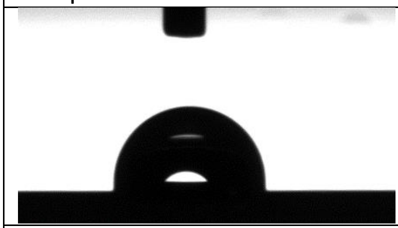
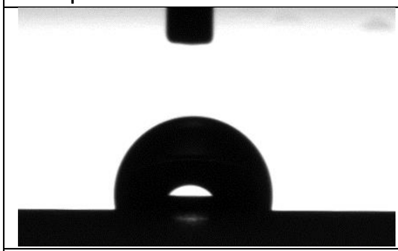
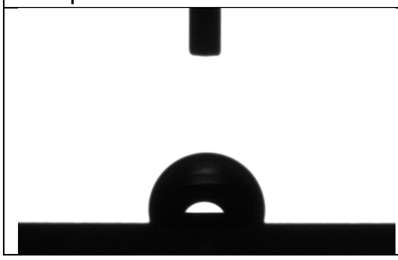
Optical profilometry @ IOM-CNR: All 3D optical profiles and maps were collected using a Profilm3D optical profilometer (Filmetrics Inc., USA).

Results – Surface hydrophobicity by Contac angle measurements

Preparation: Pipes were cut to approximately 5 cm pieces, washed with soap and deionized water, dried with nitrogen, and attached to the sample stand. Two samples were prepared from one type of pipe. The pipes were positioned in a way that the perforated part of the pipe was avoided. Data reported with standard deviation for 5 measurements.

For sample 1 there was a high discrepancy between different positions, therefore we present data for two positions.

Results

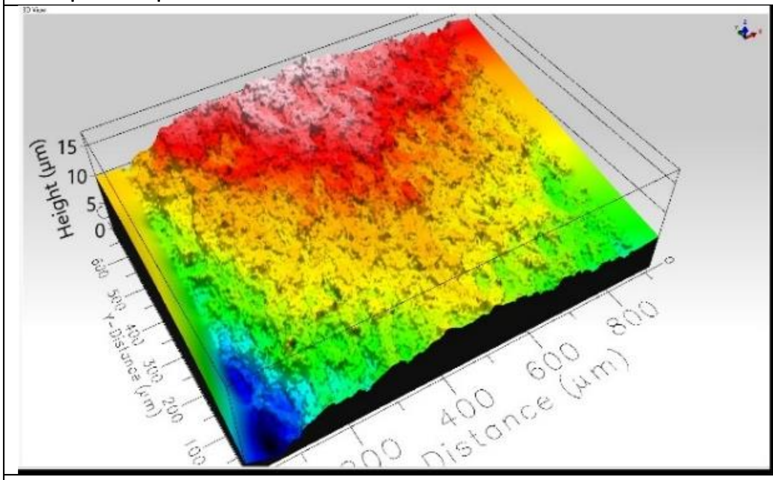
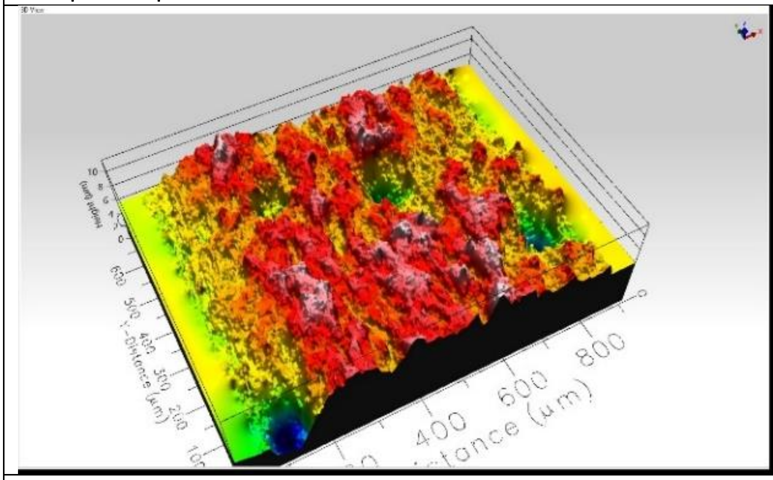
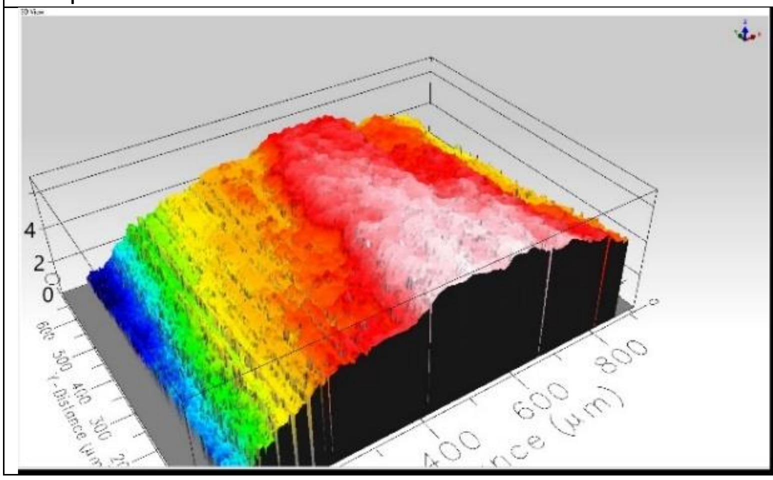
Sample 1 – position 1	
	Contact angle 103° ± 1°
Sample 1 – position 2	
	Contact angle 94° ± 4°
Sample 2	
	Contact angle 91° ± 3°
Sample 3	
	Contact angle 104° ± 4°
Sample 4	
	Contact angle 86° ± 6° (center of the pipe) Contact angle 89° ± 5° (edge of the pipe)

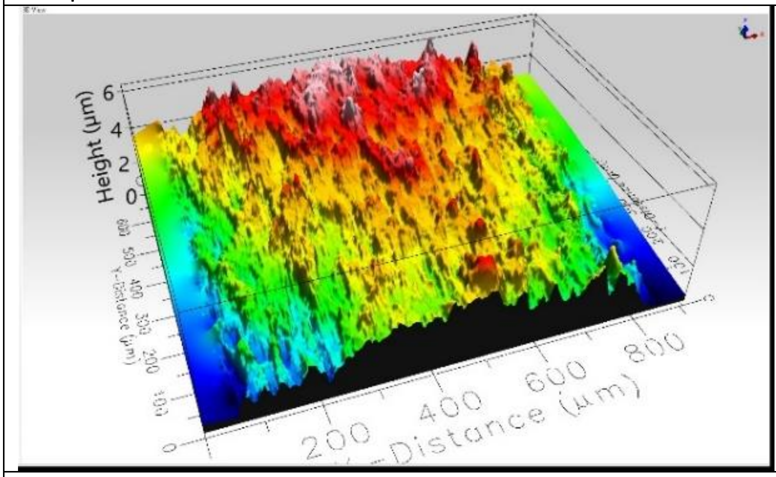
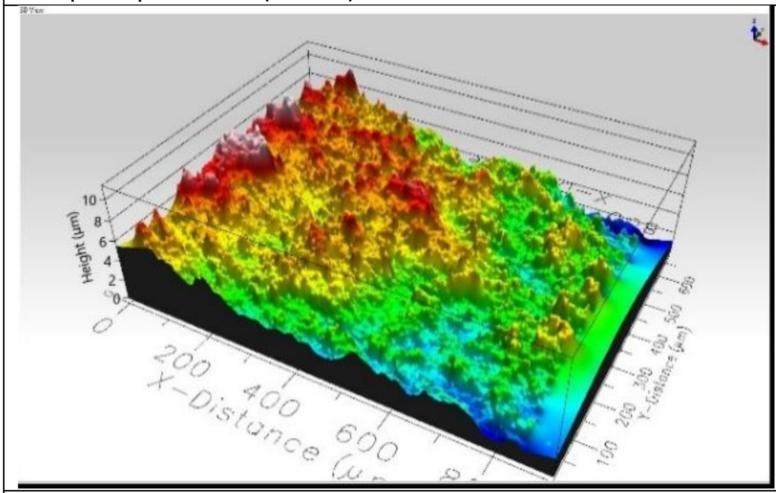
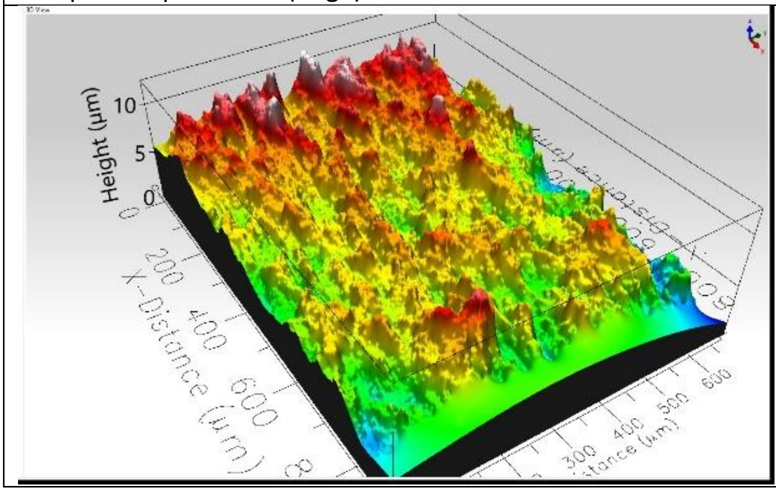
Results – Surface roughness analysis by Optical profilometry

Preparation: Pipes were cut to approximately 5 cm pieces, washed with soap and deionized water, dried with nitrogen, and attached to the sample stand. Two samples were prepared from one type of pipe.

Surface roughens were evaluated via ISO 25178 standard using Profilm software and reported as the arithmetic mean height of a surface (*S*a).

Results

Sample 1 – position 1		
		*S*a = 0.43 ± 0.02 µm
Sample 1 – position 2		
		*S*a = 0.53 ± 0.13 µm
Sample 2		
		*S*a = 0.28 ± 0.05 µm

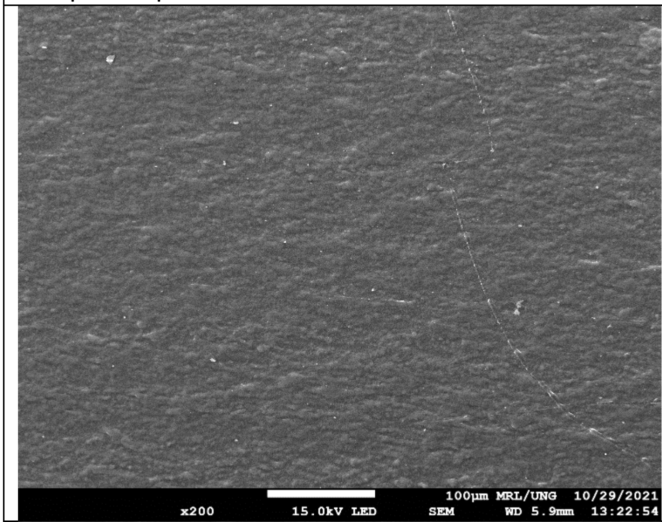
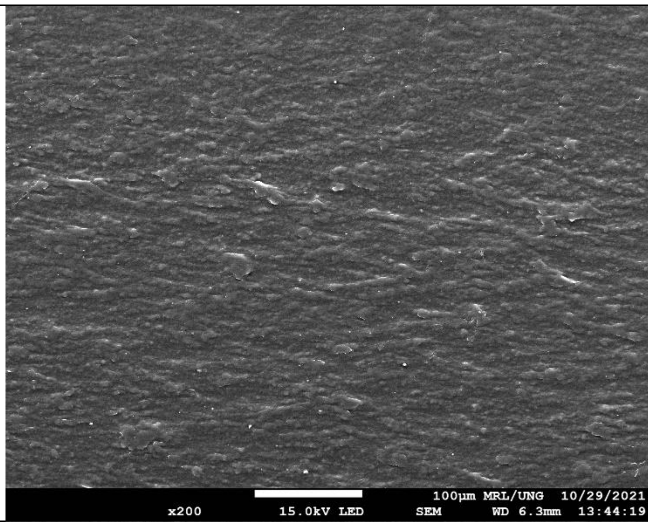
Sample 3	
	*S*a = 0.33 ± 0.02 µm
Sample 4-position 1 (center)	
	*S*a = 0.8 ± 0.2 µm
Sample 4 – position 2 (edge)	
	*S*a = 1.0 ± 0.4 µm

Results – Surface analysis by SEM

Preparation: Pipes were cut to approximately 5x10 mm stripes, two samples were prepared from one type of pipe. Pieces were glued on glass support using conductive carbon tape. To make the surface of pipes conductive for SEM measurements 8 nm thick Au coating was applied.

Sputtering process: performed by Precision etching coating system Gatan, Model 682, Ar+ ion energy 4.8 kV; deposition rate: 0.6 Å/s; deposition time 1-2 min. The stage is rotating and rocking during deposition.

Results

<table>
<tr><td colspan="2">Sample 1 – position 1</td></tr>
<tr><td>
</td><td>
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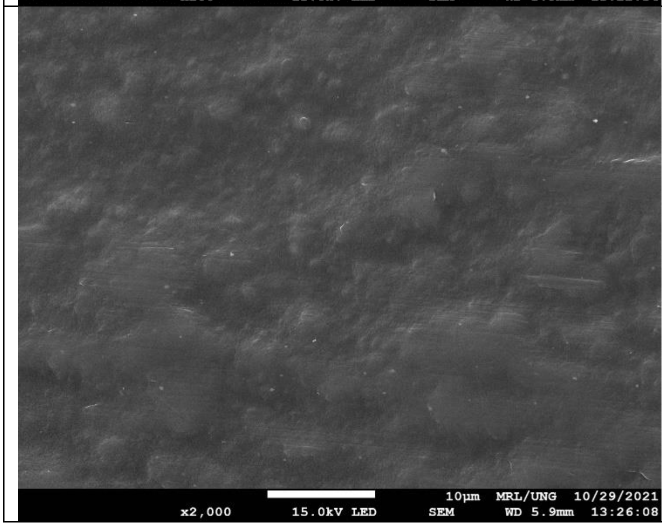
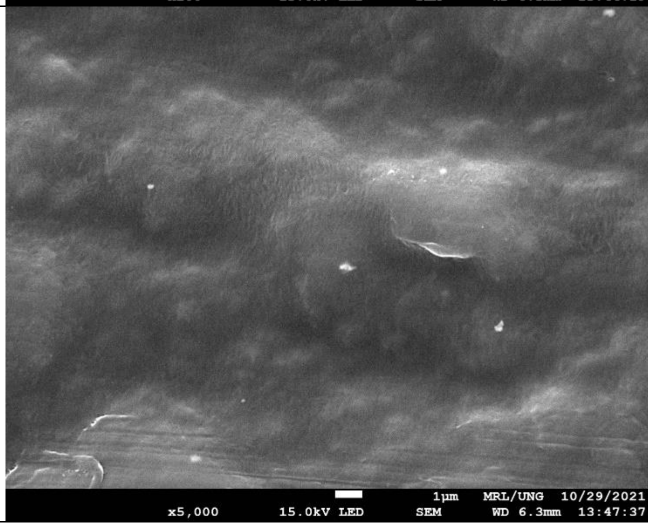
<tr><td>
</td><td>
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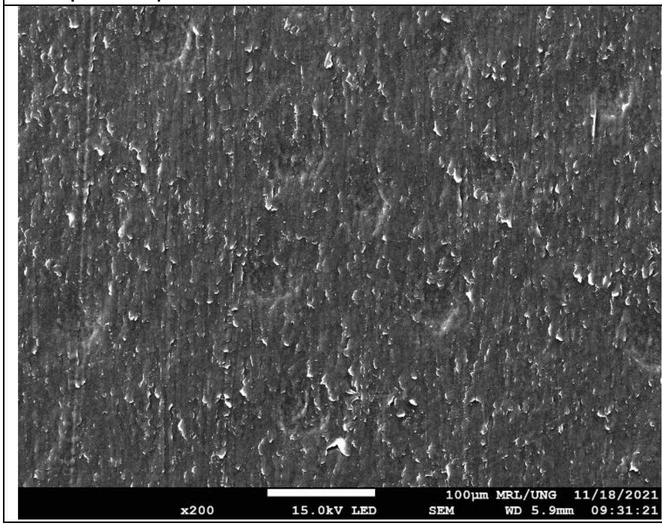
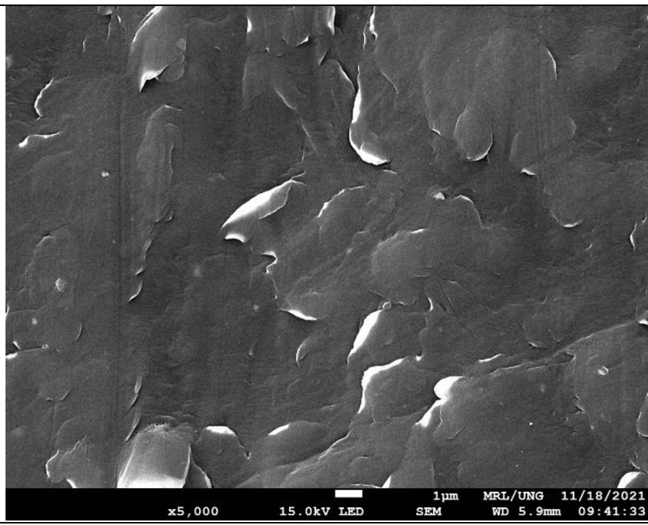
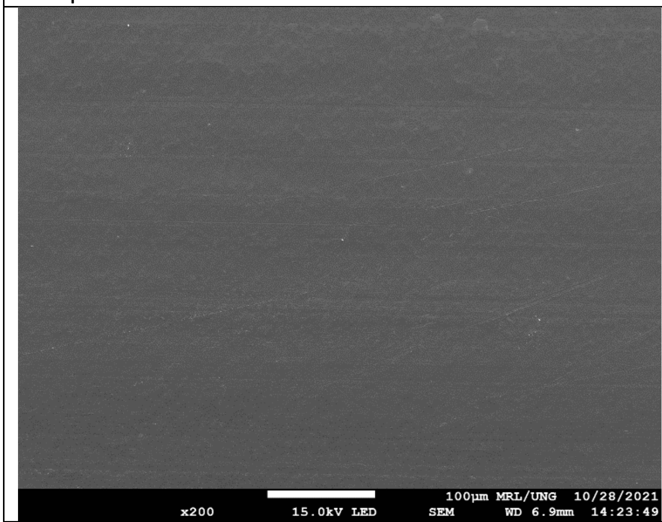
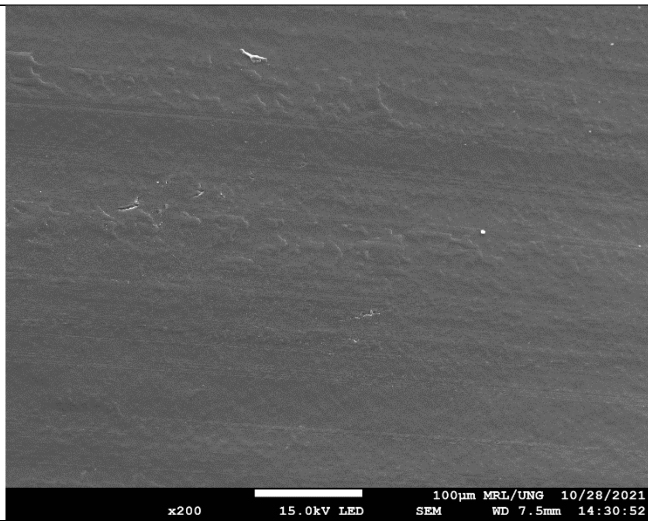
Sample 1 – position 2

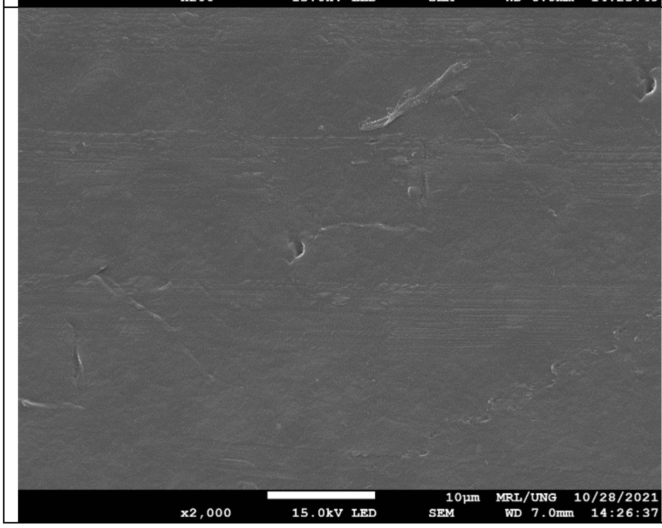


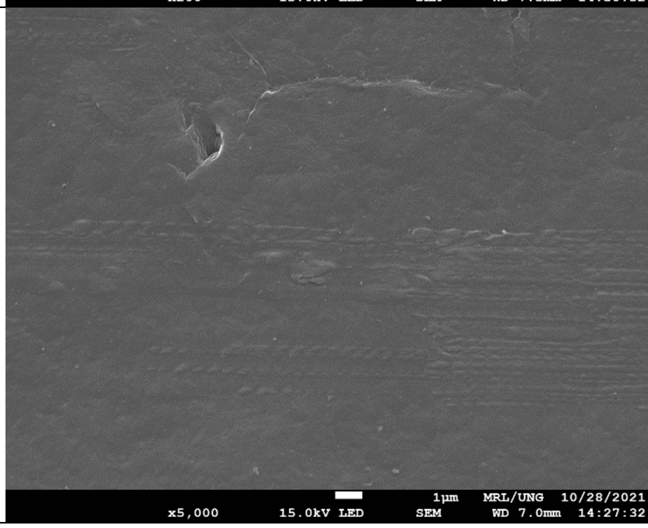


Sample 2

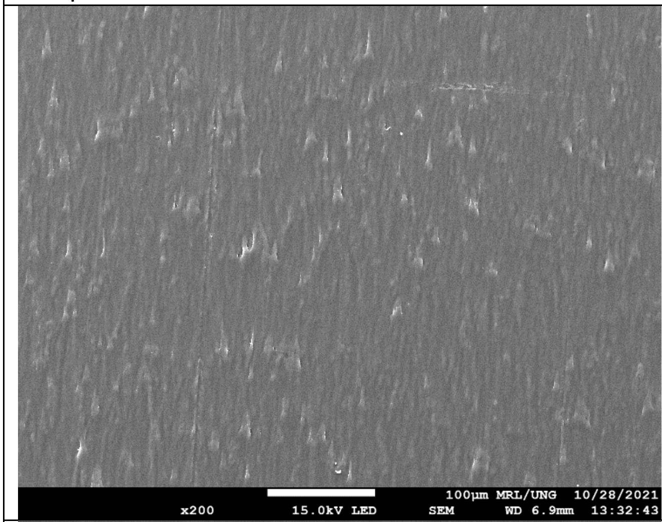


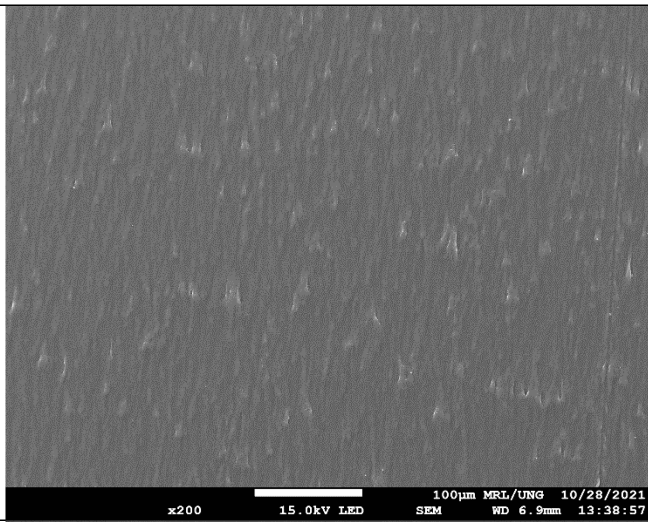






Sample 3


100µm MRL/UNG 10/28/2021
x200 15.0kV LED SEM WD 6.9mm 13:32:43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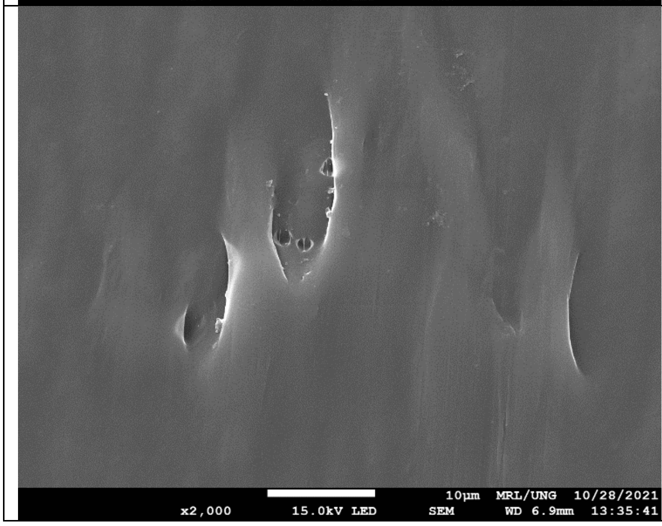
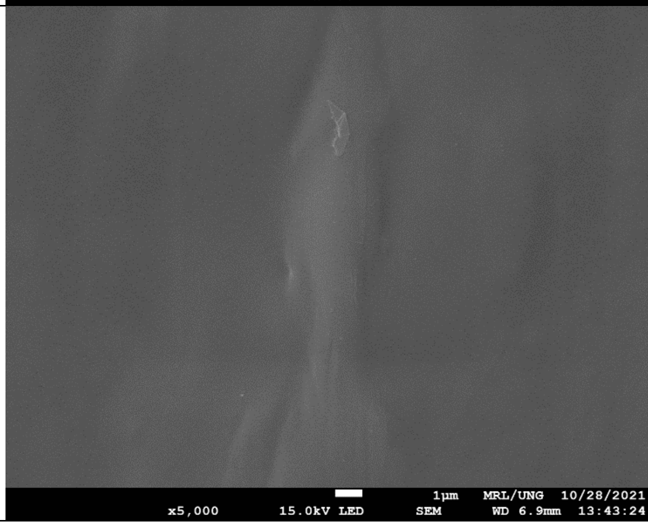
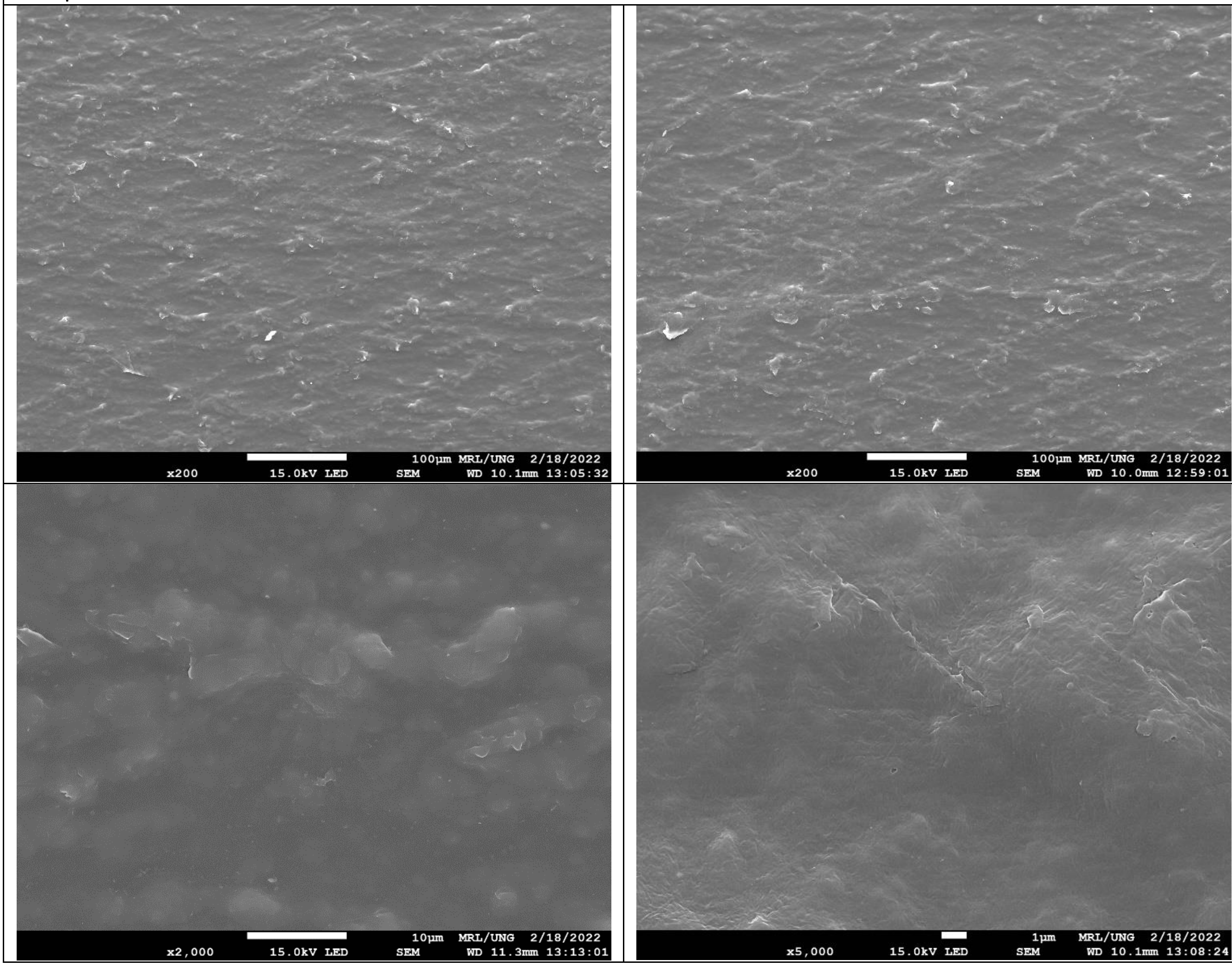
x200 15.0kV LED SEM WD 6.9mm 13:38:57


10µm MRL/UNG 10/28/2021
x2,000 15.0kV LED SEM WD 6.9mm 13:35:41


1µm MRL/UNG 10/28/2021
x5,000 15.0kV LED SEM WD 6.9mm 13:43:24

Sample 4


100µm MRL/UNG 2/18/2022
x200 15.0kV LED SEM WD 10.1mm 13:05:32
100µm MRL/UNG 2/18/2022
x200 15.0kV LED SEM WD 10.0mm 12:59:01
10µm MRL/UNG 2/18/2022
x2,000 15.0kV LED SEM WD 11.3mm 13:13:01
1µm MRL/UNG 2/18/2022
x5,000 15.0kV LED SEM WD 10.1mm 13:08:24

Discussion

The surface of sample pipes 1, 2, and 3 show slightly hydrophobic properties (contact angle >90°). The most hydrophobic is sample 3 (104° ± 4°), while the lowest hydrophobicity is found in sample 4 is slightly hydrophobic (86° ± 6°). Contact angle across the surface of sample 1 was not homogenous, with big discrepancies in hydrophobicity depending on the position. The contact angle for sample 1 was measured in a range from 89° to 104°. We identified positions with lower contact angles (93° ± 4°) and higher contact angles (103° ± 1°). Namely, the contact angle for sample 1 was homogenous within 5-10 cm of the pipe length. This behavior indicates inconsistency during the molding process. Similarly, sample 4 shows a position-dependent discrepancy of surface properties. Regions with a contact angle of 86° ± 6° and 89° ± 5° were found at the center and the edge of the pipe, respectively.

SEM imaging was used to study surface morphology. On a macro scale, we observe that the surface of sample 2 and sample 4 is crack-free, while up to 10 µm cracks are observed for sample 3 and sample 1. For sample 1, a higher number of cracks is observed at the position where the contact angle is low and the cracks are randomly oriented. For sample 1 the cracks are oriented in the same direction, more precisely along the pipe length. At higher magnification, surface morphology shows closely packed polymer particles. Ignoring the presence of cracks, we can qualitatively describe the surface of samples 1 and 4 as rough, while the surface of samples 2 and 3 is flat.

The roughness of the surface was quantitatively studied by optical profilometry. We provide the roughness map and evaluate the roughness as the arithmetic mean height of a surface. Sample 2 is the flattest (Sa = 0.28 ± 0.05 µm) followed by sample 3 with relatively similar roughness value (Sa = 0.33 ± 0.02 µm). For sample 1 we determined roughness at 2 locations, in particular, at the location that showed a higher contact angle and at a location that showed a lower contact angle. Both locations have much higher roughness than sample 2 and sample 3, Sa = 0.434 ± 0.02 µm for higher contact angles and Sa = 0.53 ± 0.13 µm for lower contact angles. The highest roughness was found for sample 4 with Sa = 0.8 ± 0.2 µm and Sa = 1.0 ± 0.4 µm for the center and edge of the pipe, respectively.

Concluding remarks on the capabilities of the demonstrated techniques

Contact angle measurements allow to quantitatively determine the surface wetting properties and showed that pipes are on the border of hydrophobicity and hydrophilicity. Due to the small deviation in contact angle, hydrophobic, the wettability cannot be correlated with slippery properties. However, it can be used to indicate the inconsistency in the surface morphology along the same pipe.

SEM allows fast evaluation of surface morphology with minimum pretreatment requirements (Au sputtering). It allows us to identify the presence of cracks and qualitative determination of the roughness.

Optical profilometry allows the estimation of surface roughness over large areas, despite sample curvature and without the need for specific sample treatment.

According to the slippery evaluation provided by the company, we conclude that slippery properties are related to the homogeneity of the surface morphology (i.e. presence of cracks) rather than surface wettability and local roughness. The presence of cracks results in the worse slippery performance of the pipe (Samples 1 and 3), and it seems to be the first parameter to address when optimizing the slippery properties. Hence, for analysis of similar samples for slippery optimization, SEM inspection is probably more effective than profilometry or contact angle at a first stage.

This report has been written by Andraž Mavrič, Mattia Fanetti, and Simone Dal Zilio (02/03/2022).