Using Fringe Projection Phase-Shifting to Correct Contact Angles for Roughness Effects

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- Fringe Projection Phase-Shifting
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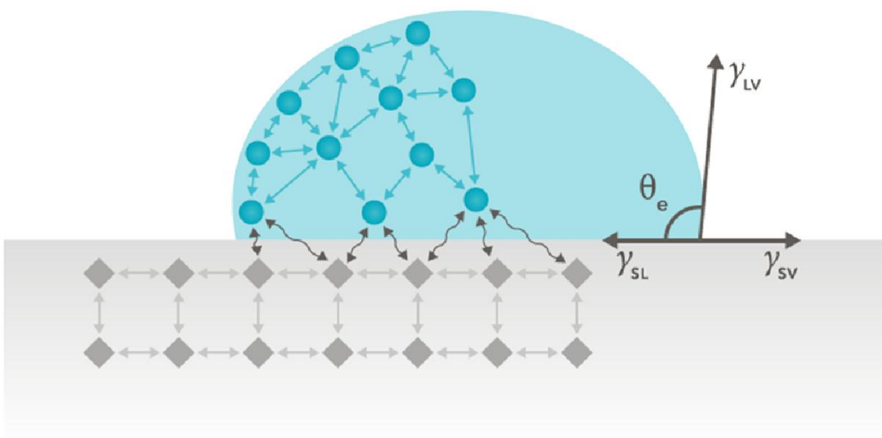
Contact Angle
A measure of wettability

- Defined by intermolecular interactions between three phases; solid, liquid and vapor/gas
- Young Equation (1805) on ideal substrates:



$$\cos\theta_Y = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}}$$

Θ_Y = *Young contact angle*
γ_{SV} = *solid-vapor interfacial tension*
γ_{SL} = *solid-liquid interfacial tension*
γ_{LV} = *liquid-vapor interfacial tension*

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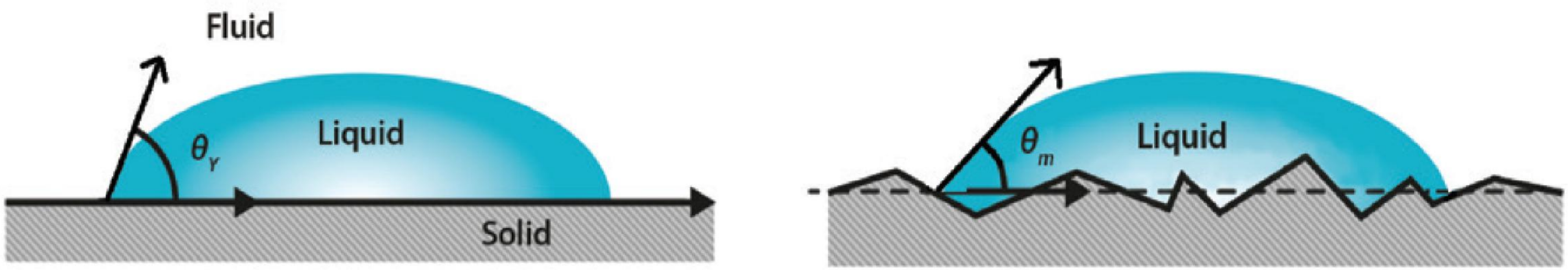
T. Young, In: Miscellaneous Works, Vol I, G. Peacock and J. Murray (Ed.), London, UK, 1855.

Young vs. Measured Contact Angle
Influence of roughness on contact angle



- Young contact angle assumes:
  - Surface is completely smooth
  - Surface is chemically homogeneous

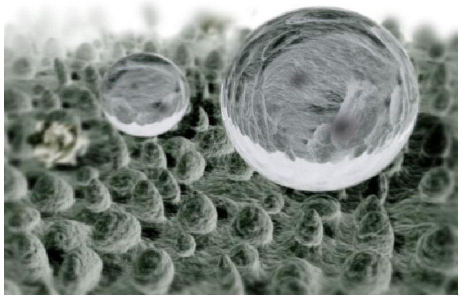
- Real surfaces are hardly ever compelety smooth

How Roughness Affects Contact Angles?

- Correction for surface roughness was extablished already in 1936 by Wenzel

$$\cos\theta_m = r \cos\theta_Y$$

θ_m = roughness dependent (measured) contact angle
θ_Y = Young's contact angle corresponding to an ideal surface



- Wenzel equation states that surface roughness enhances existing wetting behaviour
  - $\theta < 90^o$ → roughness lower the CA
  - $\theta > 90^o$ → roughness increase the CA

Wenzel, R.N. *Industrial and engineering chemistry* **1936**, *28* : 8, 988-994.

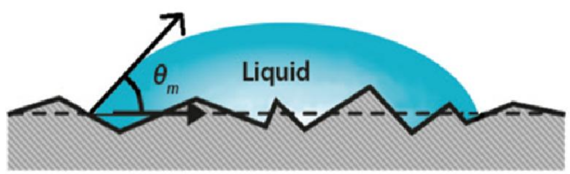
How to Correct for Roughness?

- 3D surface roughness parameter, Sdr, is needed for the Wenzel equation
  - ➢ r = 1 for smooth surface and >1 for rough surfaces
- Wenzel correction is valid when
  - ➢ Drop dimensions (1 mm) are larger than roughness by two (10 µm) to three (1 µm) orders of magnitude
  - ➢ Liquid wets the surface grooves

$$\cos\theta_m = r \cos\theta_Y$$

$$r = 1 + (S_{dr} / 100)$$

S_{dr} = ratio between interfacial and projected area



Marmur, A. *Soft Matter* **2006**, *2*, 12-17.

Why Measure Both Roughness and Contact Angle?

Industrial R&D and QC

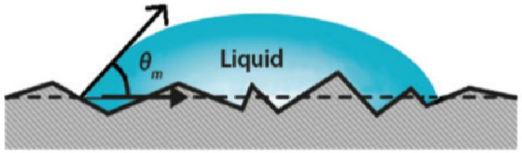
- Many surface modification and coating technologies influence both *surface chemistry* and *roughness*.

→ **Possibility to separate the impacts of surface chemistry and roughness of various coating formulation, surface modifications and QC problems.**

Academic

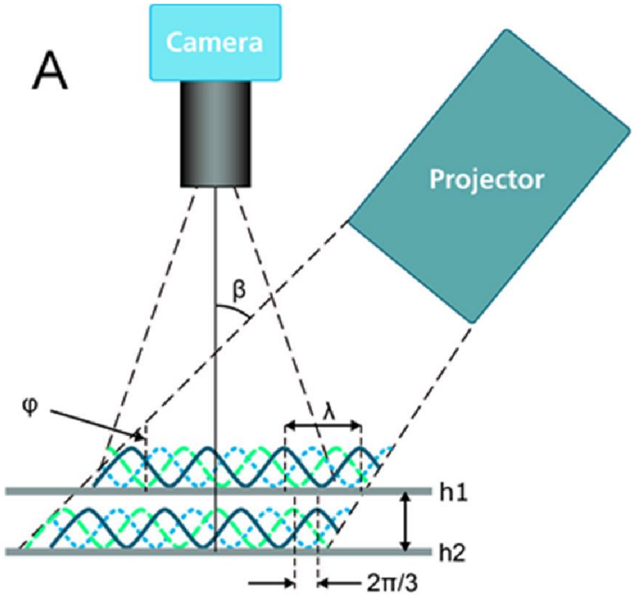
- Roughness correction enables defining the fundamental surface free energy values for rough surfaces

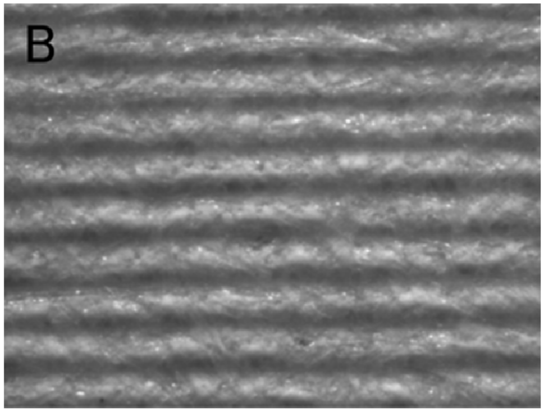
Development and quality control of coatings and surface modification technologies



Surface free energy on rough surfaces

Fringe Projection Phase-Shifting (FPPS)





- LED light source projects structured light onto the sample surface
- Here we use a sinusoidal fringe pattern slide – hence “fringe project phase-shifting”
- Digital camera captures the fringe patterns
- 3D shape of the object is reconstructed by phase-shift coding

- Simultaneously perform 2D and 3D characterization at pixel level resolution (1.1 μm x 1.1 μm) allowing for characterizing of **micron scale** surface features

FPPS Continued

- The sinusoidal fringes can be expressed by

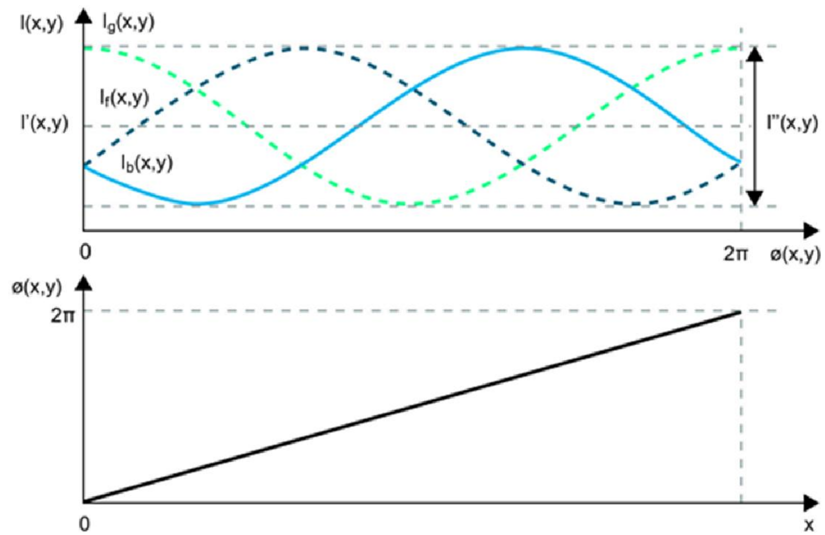
$$I_n(x, y) = a + b\cos(2\pi x / p + \varphi_0 + \delta_n)$$

(x, y) = the coordinate in the slide frame plane
a = background intensity
b = amplitude modulation
p = sinusoidal grating wavelength
φ_0 = the additional phase shift caused by the surface height
δ_n = the phase shift from the slide movement.

- the spatial phase shift can be expressed by

$$\varphi(x,y) = \arctan\left[\sqrt{3}(I_r - I_b)(2I_g - I_r - I_b)\right]$$

- Example of 3 Phase shifts



- The phase shift indicates the horizontal coordinate, i.e. the height difference in every pixel providing the sample topography.

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S. Zhang and P. Huang, "High-resolution, Real-time 3D Shape Acquisition", Computer Vision and Pattern Recognition Workshop, 2004.

Roughness Parameters

Symbol	Name	Equations	Description
R_a, S_a	Arithmetic average	$S_a = \frac{1}{MN}\sum_{j=1}^{N}\sum_{i=1}^{M}\left|\eta\left(x_i, y_j\right)\right|$	Average of |z|
R_q, S_q	Root mean square (RMS) roughness	$S_q = \sqrt{\frac{1}{MN}\sum_{j=1}^{N}\sum_{i=1}^{M}\eta^2\left(x_i, y_j\right)}$	Standard deviation of z
R_p, S_p	Maximum height of peaks	$S_p = MAX\ (\eta_p)$	Max z
R_v, S_v	Maximum depth of valleys	$S_v = MIN\ (\eta_v)$	Min z
R_z, S_z	Maximum height of the surface	$S_z = (\lvert S_p\rvert + \lvert S_v\rvert)$	Max z- Min z
R_{10z}, S_{10z}	Ten point height	$S_z = \frac{\sum_{i=1}^{5}\lvert\eta_{pi}\rvert + \sum_{i=1}^{5}\lvert\eta_{vi}\rvert}{5}$	Average of five highest local maxima and five deepest local minima.
S_{dr}	Area factor	$S_{dr} = \frac{(Textured\ surface\ area) - (Cross\ sectional\ area)}{Cross\ sectional\ area} * 100\% = \frac{\sum_{j=1}^{N-1}\sum_{i=1}^{M-1} A_{i,j} - (M-1)(N-1)\Delta x\Delta y}{(M-1)(N-1)\Delta x\Delta y} * 100\%$	Ratio between the interfacial and projected areas

ISO standard ISO25178: Geometrical product specifications (GPS)—Surface texture: Areal Part 2: Terms, definitions and surface texture parameters

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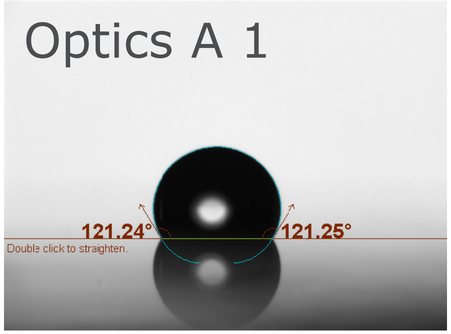
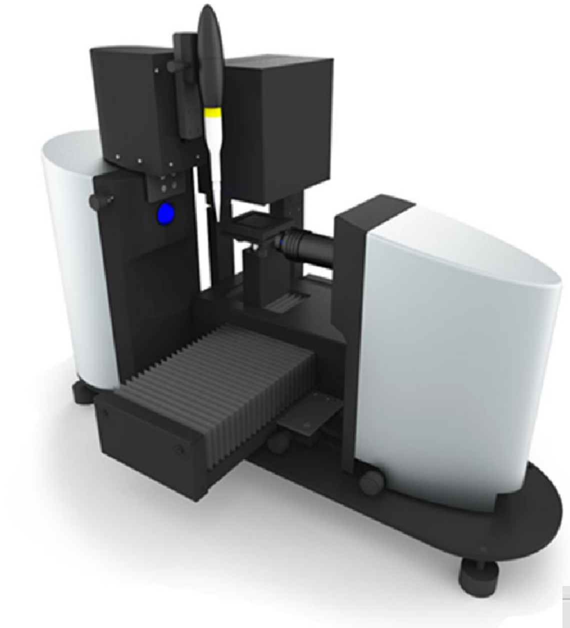
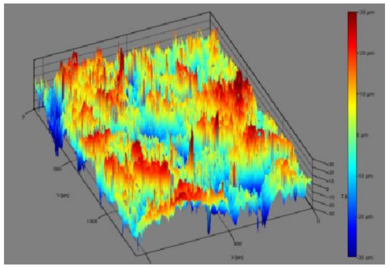
Experimental Details





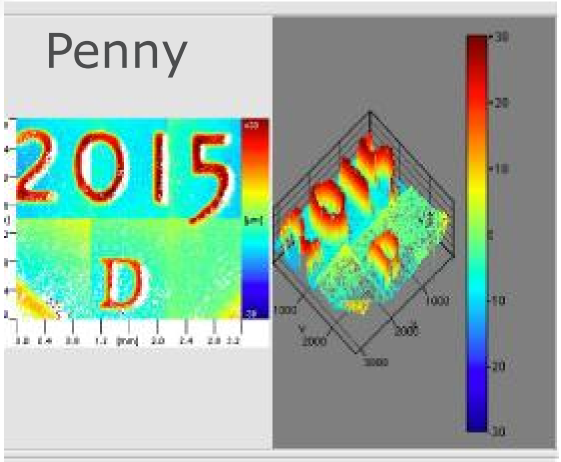
- Optimize back lighting
- Calibrate camera
- Calibrate XYZ sample stage position
- Calibrate height of 3D topography module
- Measure topography of 1.4 μm x 1.1 μm area
- Measure sessile drop Contact Angles of ~3 uL drops

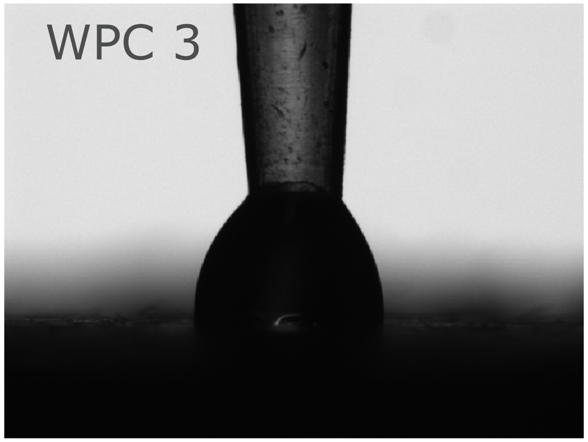
Tile, Gloss



Calculated results

Quantity	Magnitude
Noise estimate [μm]	3.513
r	2.700
Sa [μm]	9.540
Sdr [%]	169.979
Sq [μm]	12.206
Horizontal Ra [μm]	9.834
Horizontal Rq [μm]	11.915
Horizontal Rp [μm]	29.137
Horizontal Rv [μm]	-28.180
Horizontal Rz [μm]	57.317
Horizontal R10z [μm]	55.027
Vertical Ra [μm]	10.110
Vertical Rq [μm]	13.059
Vertical Rp [μm]	49.407
Vertical Rv [μm]	-49.719
Vertical Rz [μm]	99.126
Vertical R10z [μm]	92.815





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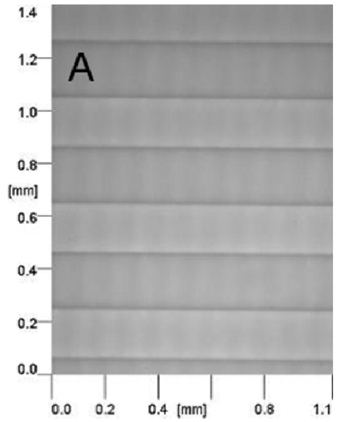
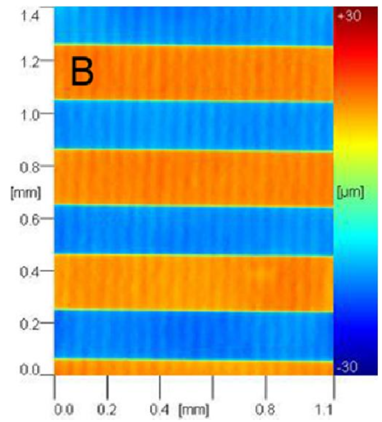
Topography Validation Sample

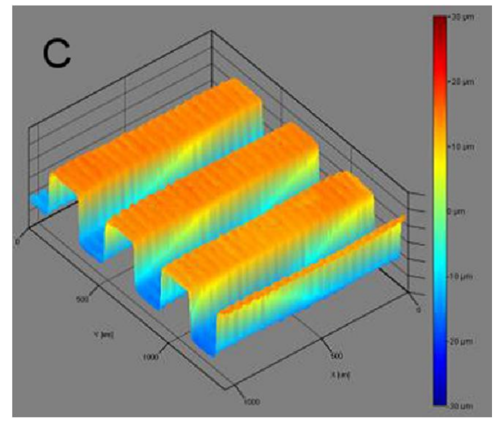
- optical image



- 2D image



- 3D image

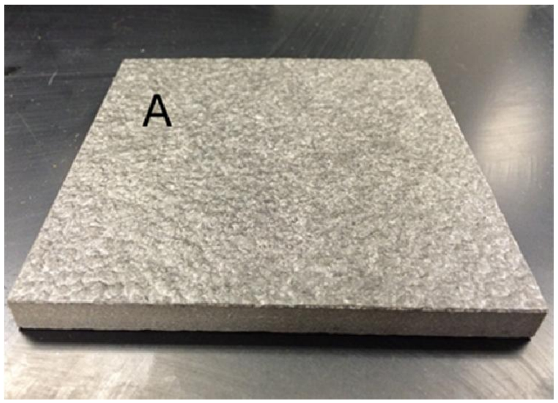


- Structured pattern of lines 200 µm wide with 200 µm trenches 30 µm deep
- The images were each 1.4 µm long by 1.1 µm wide
- Images B) and C) both have height scales from +30 µm (red) to -30 µm (blue).

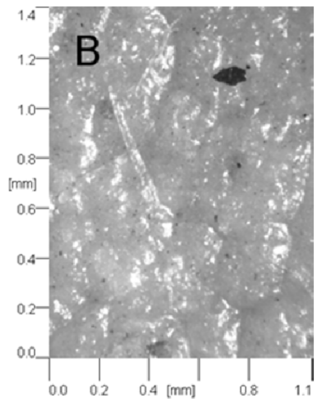
Representative Sample Images

Ceramic Tile with Gloss Finish

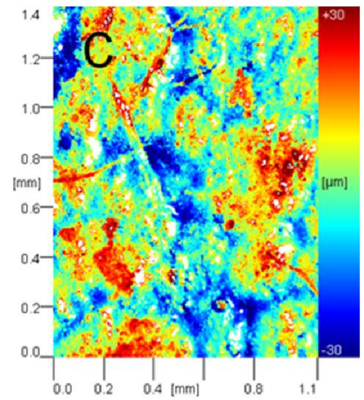
- Photograph of Tile



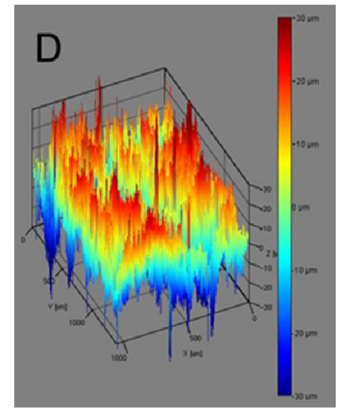
- optical Image



- 2D Image



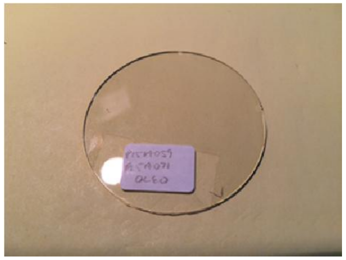
- 3D Image

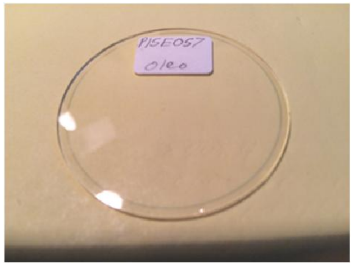


- Tile was ~ 4 inches square
- The images show a topographic map of the surface with the peaks being white or red and the valleys being black or blue.
- B, C, D images were each 1.4 µm long by 1.1 µm wide
- Images C) and D) both have height scales from +30 µm (red) to -30 µm (blue).

Results: Optics

Sample	CA, °	CA (corrected), °	CA - CAc, °	Sdr, %
Optics A 1	121	96	25	371
Optics A 2	108	99	9	99
Optics A, Avg	115	98	17	235
Optics A, STD	9	2	11	192
Optics E 1	119	94	25	662
Optics E 2	118	94	24	568
Optics E, Avg	119	94	25	615
Optics E, STD	1	0	1	66

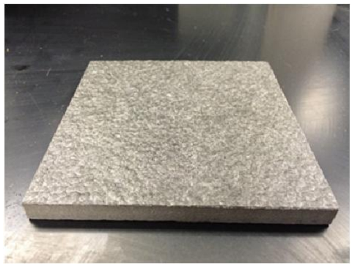


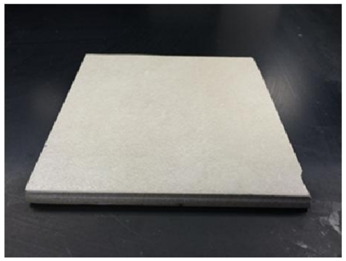


- Coated with *anti-reflective coatings* by the manufacturer
- Surfaces appeared hydrophobic based on measured CA
- Correcting for roughness shifts angles closer to 90 °
- Roughness **amplifying** the angles
- It was impossible to tell if the roughness was occurring from the coating, the glass or a combination of the two
- Optics E appeared more uniform

Results: Ceramic Tiles

Sample	CA, °	CA (corrected), °	CA - CAc, °	Sdr, %
Tile, Gloss	35	72	-37	170
Tile, Matte	46	83	-36	434





- The ceramic tile's glaze and texture was prepared by the manufacturer
- Measured contact angles < 90 °
  - meaning the surface displays hydrophilic characteristics
  - the most hydrophilic of the samples tested
- Accounting for the roughness shifts the angle closer to 90 °
  - roughness is amplifying the angle and making it appear smaller
  - this is evident in the large negative difference
- The "matte" tile gave ~2x larger roughness

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Results: Wood Plastic Composite

Sample	CA, °	CA (corrected), °	CA - CAc, °	Sdr, %
WPC 1	78	80	-2	25
WPC 2	80	82	-2	25
WPC 3	88	89	-1	25
WPC, Avg	82	84	-2	25
WPC, STD	5	5	1	0

- WPCs contain recycled thermoplastics (PE, PP, PVC), wood filler, additives
- Since the angles were ~ 90 ° and roughness was low the correction did not affect the measured value much
- The WPC surface was very homogeneous
- Since the magnitude of the contact angle was ~90 ° there likely is poor adhesion to this particular material (better adhesion ~0 °)

Results: Titanium Screws

Sample	CA, °	CA (corrected), °	CA - CAc, °	Sdr, %
Ti 1	96	95	1	22
Ti 2	107	102	5	41
Ti 3	103	98	5	65
Ti 4	110	101	9	78

- Titanium screws were prepared by the manufacturer
- The samples increased in roughness
- The contact angles do not follow the same trend
- Separating the chemical influence on the wettability from the roughness gives scientists more control over design variables

Conclusion

- We developed and applied the FPPS method to measure roughness and contact angles on the same spot on the sample
  - This allows correcting the CA for the underlying roughness
- Accounting for roughness on hydrophobic surfaces lowered the CAc
- Accounting for roughness on hydrophilic surfaces raised the CAc
- When the measured CA was close to 90 º and the sample had small roughness the correction did not change the angle much
- This method gives the researcher more flexibility to separate out the effect of roughness from surface chemistry

Acknowledgements




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