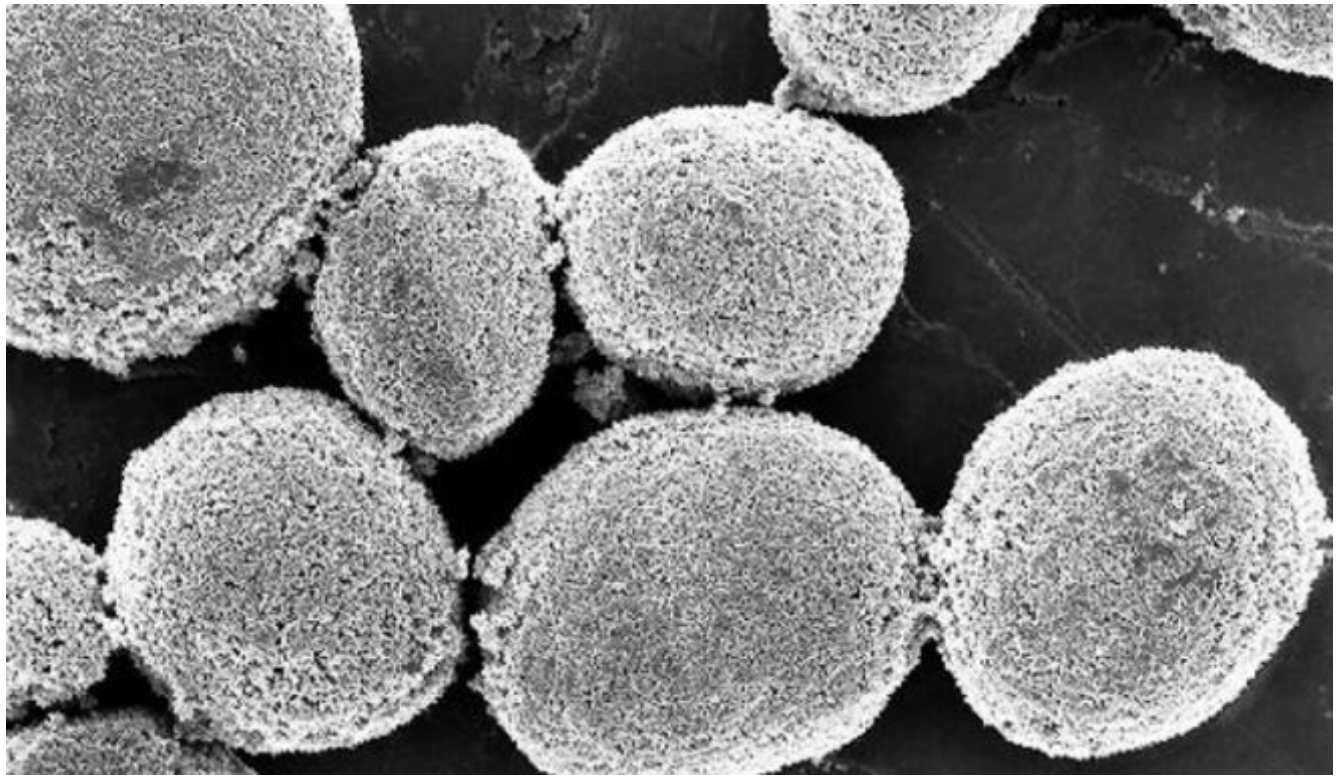
Novel Synthetic Silica for Matting Powder Coatings

Powder Coating Summit 2021



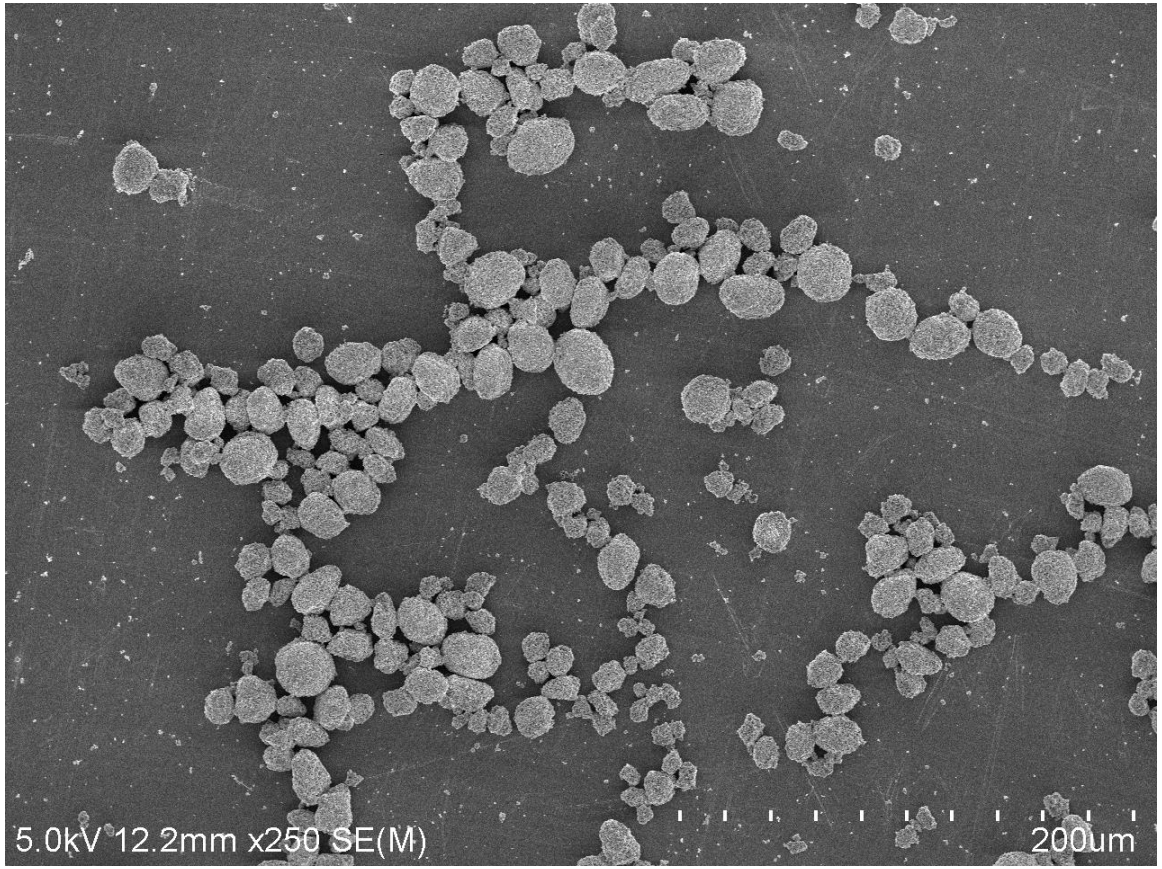
Bernhard Resch | Evonik Corporation
September 23, 2021

Current Technologies for Matting Powder Coatings

- Incompatibility:
  - 2-component dryblends (co-grind)
  - Chemical matting by introducing various binders or crosslinking reactions
- Surface Modifiers:
  - Micronized waxes as such as polyolefins, paraffins, and others
- Inorganic Fillers:
  - Coarse particle size barium sulfate, aluminum trihydroxide (ATH), calcium carbonate, nepheline syenite, Wollastonite, among others.
  - Commonly used due to their low oil absorption, but often have a broad particle size distributions with varying particle shapes as well as specific gravity increase.

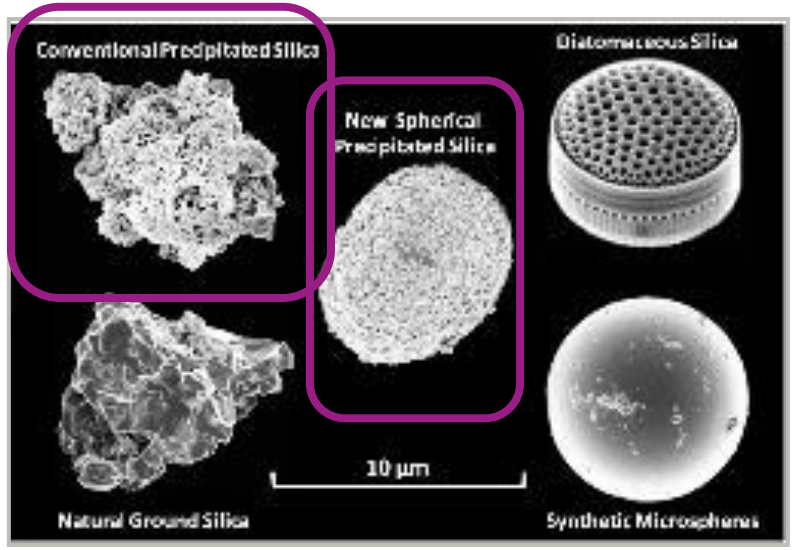
Spherical Silica Technology

- Spherical Precipitated Silica:
  - A unique precipitated amorphous silica that is made with a patented continuous loop reactor process
  - Very uniform with a narrow particle size distribution, for a defect free finish
  - Available in (3) different d(50) particle sizes, ranging from 5.5 to 15µm.



Spherical Silica Technology

- Spherical Precipitated Silica:
  - These particles have higher oil absorption listed than the conventional fillers used, however the particles are essentially nonporous.
  - Lower specific gravity
  - Properties unlike conventional precipitated Silica



Description	Particle Size D_{50} µm	Particle Size D_{95} µm	Oil Absorption ml/100g	Specific Gravity g/cm³	Specific Surface Area (BET) m²/g	Refractive Index
Barium Sulfate ($BaSO_4$)	3.0	<15	11	4.4		1.64
Aluminum Trihydroxide (ATH)	11.5	<36	20	2.4	2.7	1.57
Spherical Silica 0111	**5.5**	**<20**	**40**	**2.0**	**< 12**	**1.46**
Spherical Silica 0112	**10**	**<25**	**50**	**2.0**	**< 12**	**1.46**
Spherical Silica 0115	**15**	**<30**	**45**	**2.0**	**< 12**	**1.46**
Precipitated Silica 440	14.5		270	2.0	500	1.46

Evaluation of Spherical Silica Technology for Matting Powder Coatings

- Superdurable polyester HAA formulation
- Comparison to Barium Sulfate and Aluminum Trihydroxide (ATH)
- Evaluate
  - Matting efficiency
  - Coating performance
    - Pencil Hardness
    - Conical Bend Flexibility
    - Reverse Impact and Cupping
    - Corrosion
    - UV Weatherability
  - Ease of processing

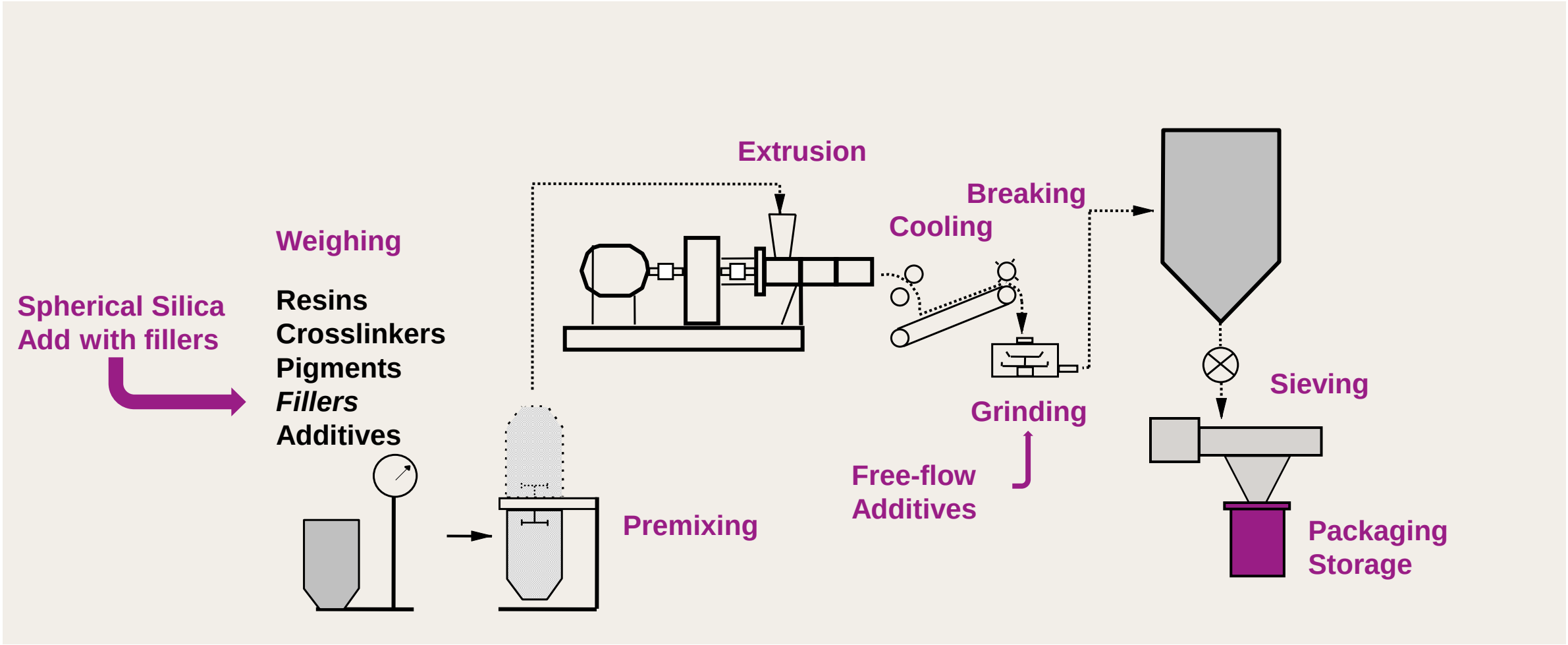
Formulation and Process Parameters – Polyester HAA Black Super Durable

Formulation	Raw Material	20% Matting Agent
Resin	Super-durable polyester (AV=35)	73.6
Crosslinker	HAA	3.9
Flow aid	Acrylic polymer on silica carrier	1.0
Degassing	Benzoin	0.5
Carbon black	Carbon black pellets	1.0
Matting Agent*	***Various Fillers***	20.0
Total		100

Processing Parameters		
Extruder	APV 19mm Twin Screw	500 RPM
Set Temp °F	Zone 1/2	212/212
Actual Temp °F	Zone 1/2	214/212
Extrudate Temp °F	IR gun	225-234
Torque	Depending on formula	40-60%
Mill	Strand Benchtop Mill	
Screen	[mesh]	140
Powder cure	min / °F	15 / 400

**This powder coating formulation does not any matting waxes*

Manufacturing of Powder Coatings



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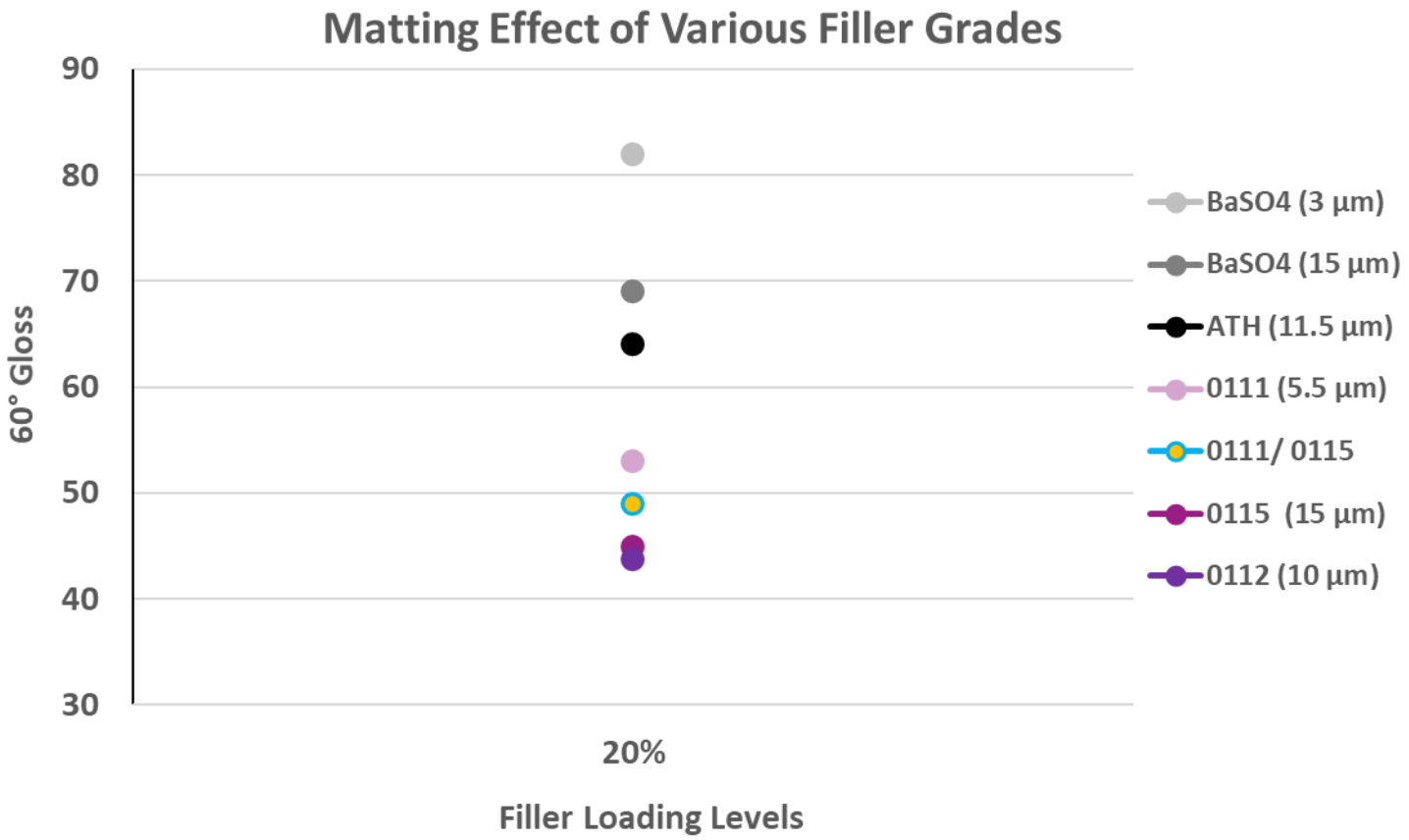
Matting Efficiency and Pill Flow – Polyester HAA Black Super Durable



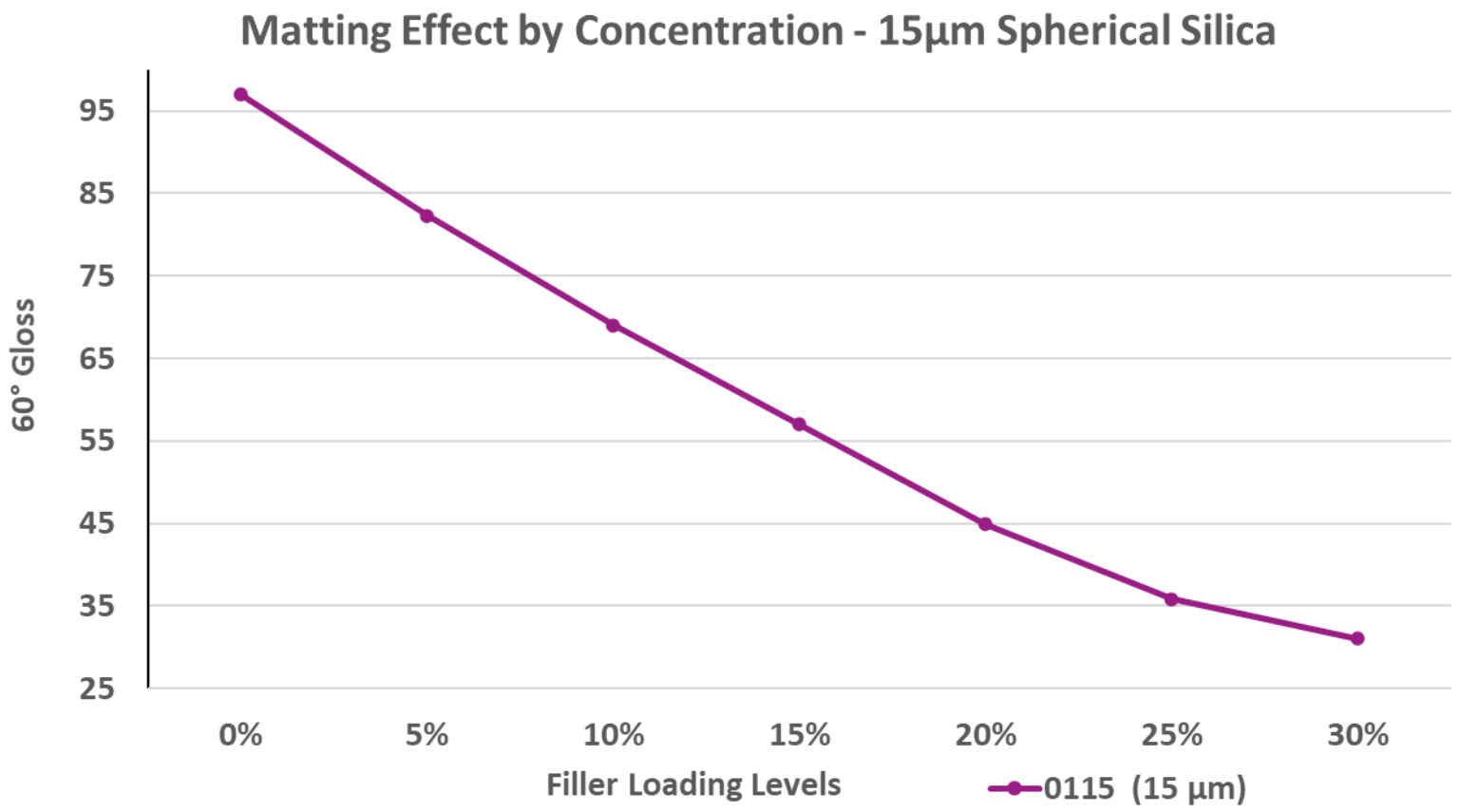
Modification	Gloss 60°	Pill flow* [mm]
Control	**97**	75
20% $BaSO_4$ (fine 3µm)	**82**	69
20% $BaSO_4$ (coarse 15µm)	**69**	76
20% ATH (11.5µm)	**64**	66
20% 0111	**53**	54
10% 0111/ 10% 0115	**49**	57
20% 0115	**45**	56
20% 0112	**44**	60

*Pill flow (inclined plate flow) uses 1g sample @400°F

This is an indirect measurement of melted powder coating viscosity.

Higher numbers in mm (distance) means, melt viscosity is lower. Lower numbers in RED means higher melt viscosity.

Matting Efficiency and Pill Flow – Polyester HAA Black Super Durable



Modification	Gloss* 60°	Pill flow [mm]
Control	**97**	75
5% 0115	**82**	70
10% 0115	**69**	61
15% 0115	**57**	64
20% 0115	**45**	56
25% 0115	**36**	55
30% 0115	**31**	58

*Average of multiple panels;

Std. Dev. 1 GU

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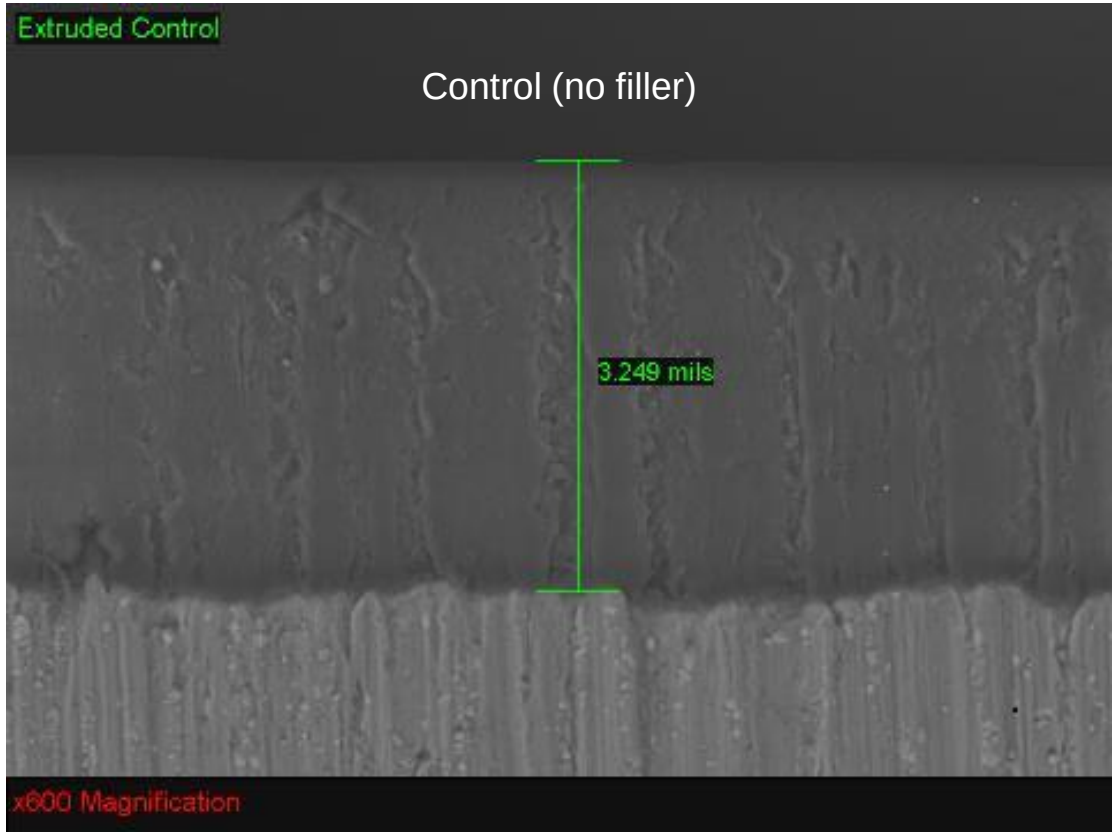
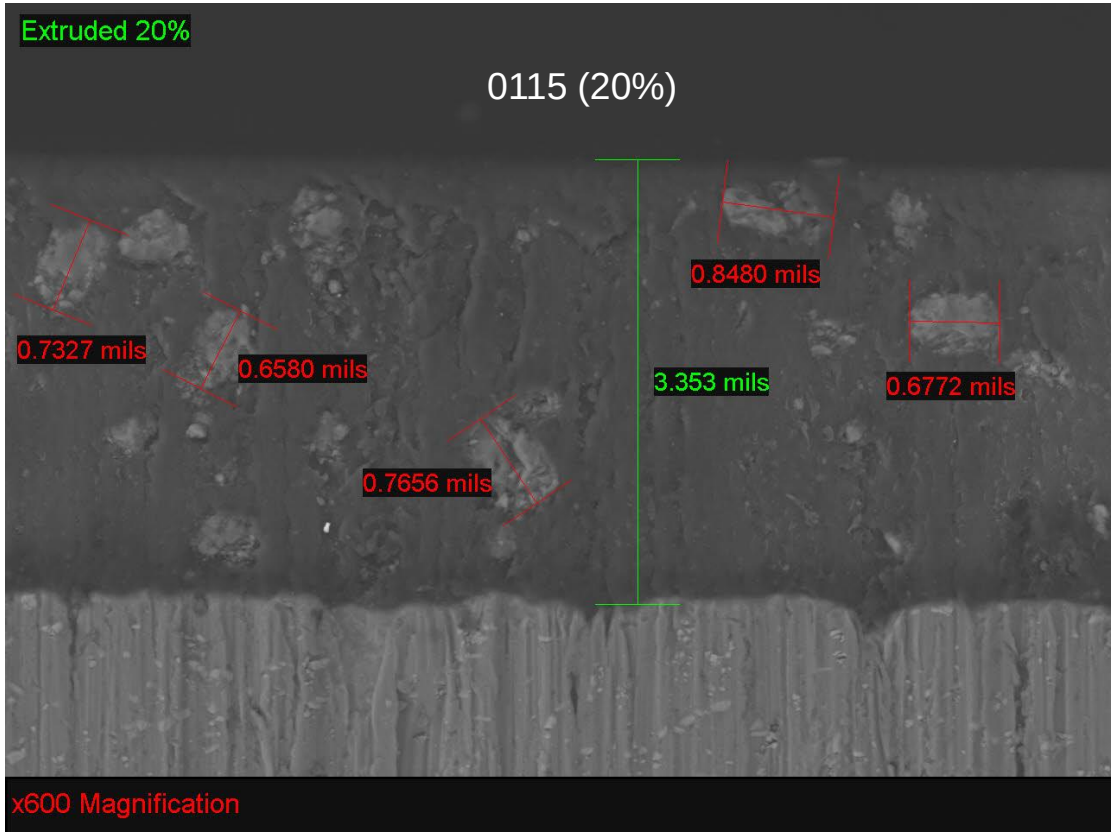
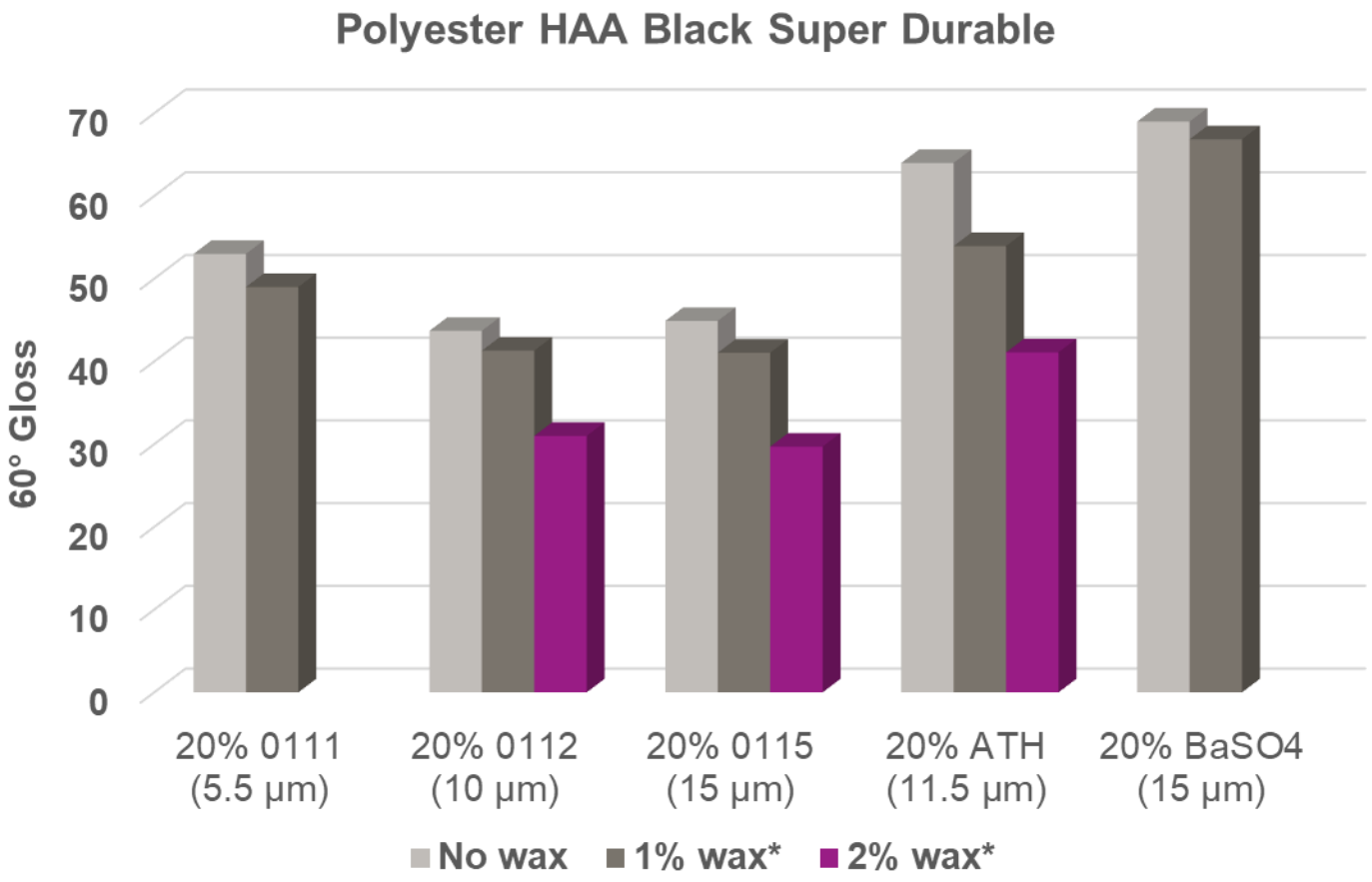
Cross-Sectional Analysis – Polyester HAA Black Super Durable





* 1mil = 25.4µm

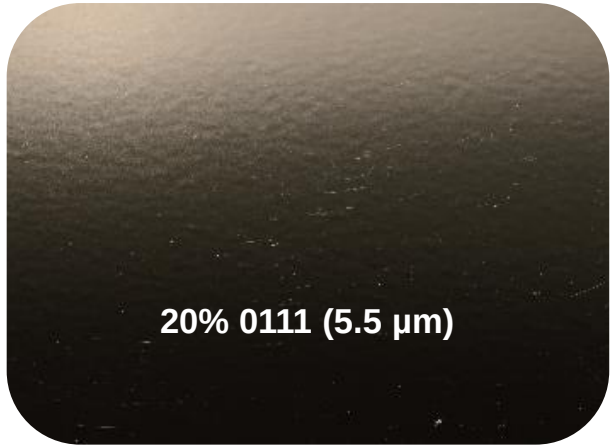
Matting Efficiency in Combination with Matting Waxes

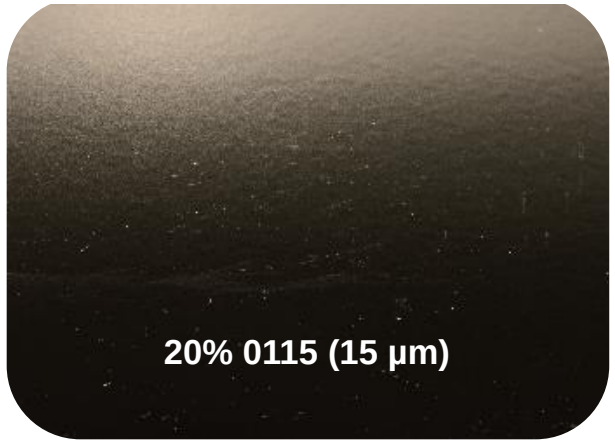


Modification	Gloss (60°)		
	No wax	1% wax*	2% wax*
20% 0111 (5.5 µm)	53	49	
20% 0112 (10 µm)	44	41	31
20% 0115 (15 µm)	45	41	30
20% ATH (11.5 µm)	64	54	41
20% $BaSO_4$ (15 µm)	69	67	

* Polyolefin matting wax

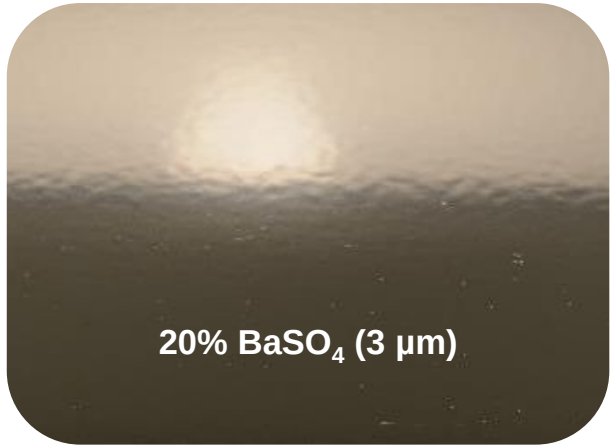
Appearance vs. Conventional Fillers – Polyester HAA Black Super Durable

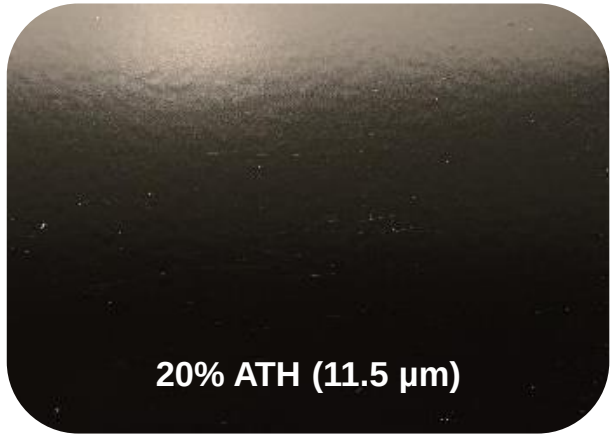




Substrate: CRS B1000

DFT: 3 mils





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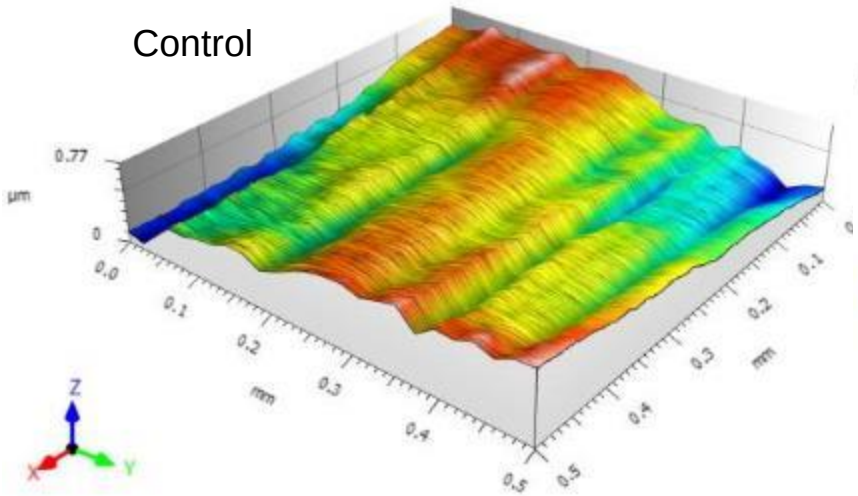
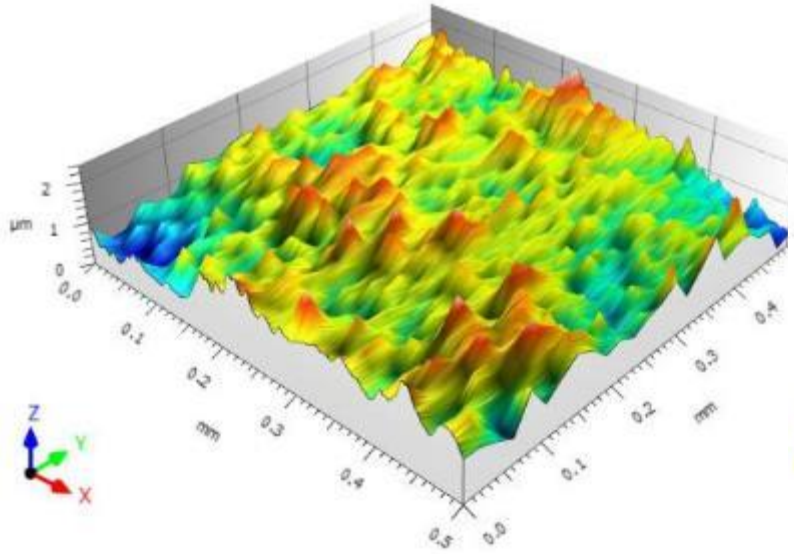
Matting Mechanism and Surface Profilometry* – Polyester HAA Black SD

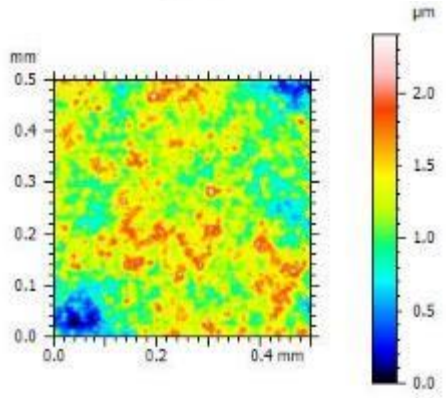
* Measurements taken with Hommel Tester T8000

Control

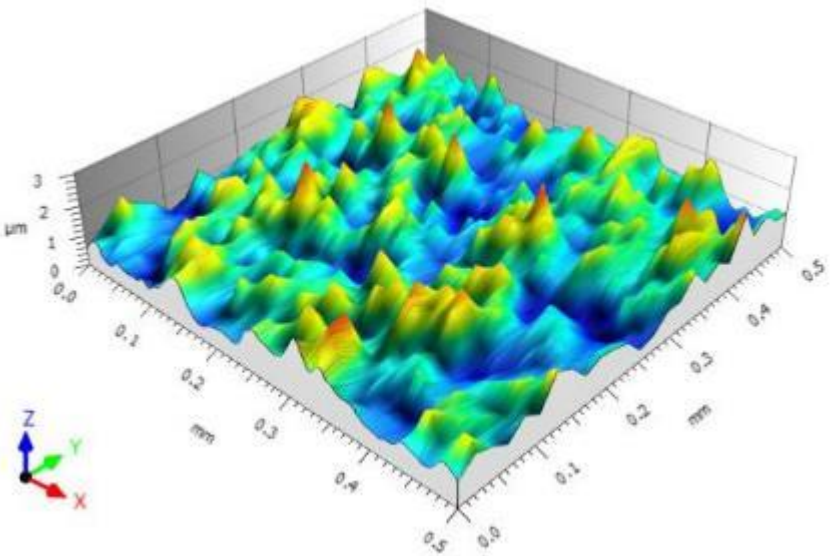


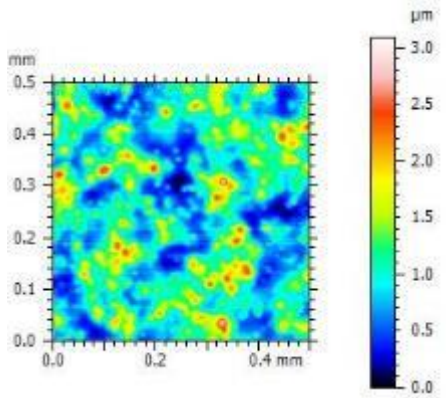
20% 0111 (5.5 µm)





20% 0115 (15 µm)





The even color distribution in the profile shows excellent uniformity of the microroughness throughout the coating

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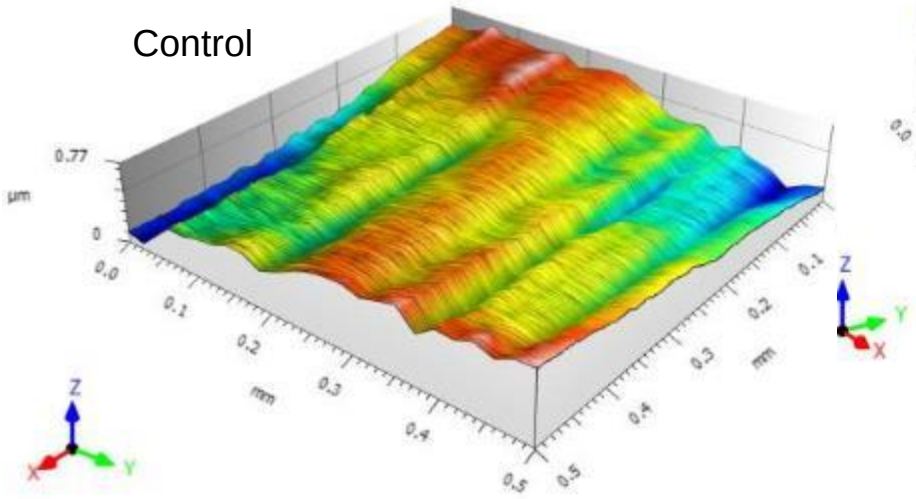
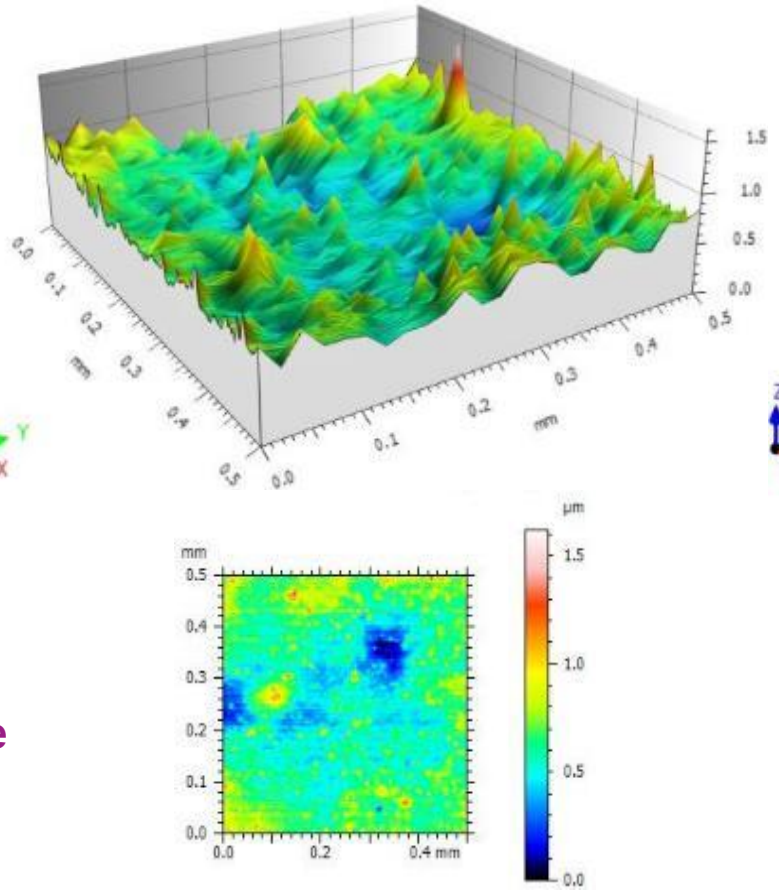
Matting Mechanism and Surface Profilometry – Polyester HAA Black SD

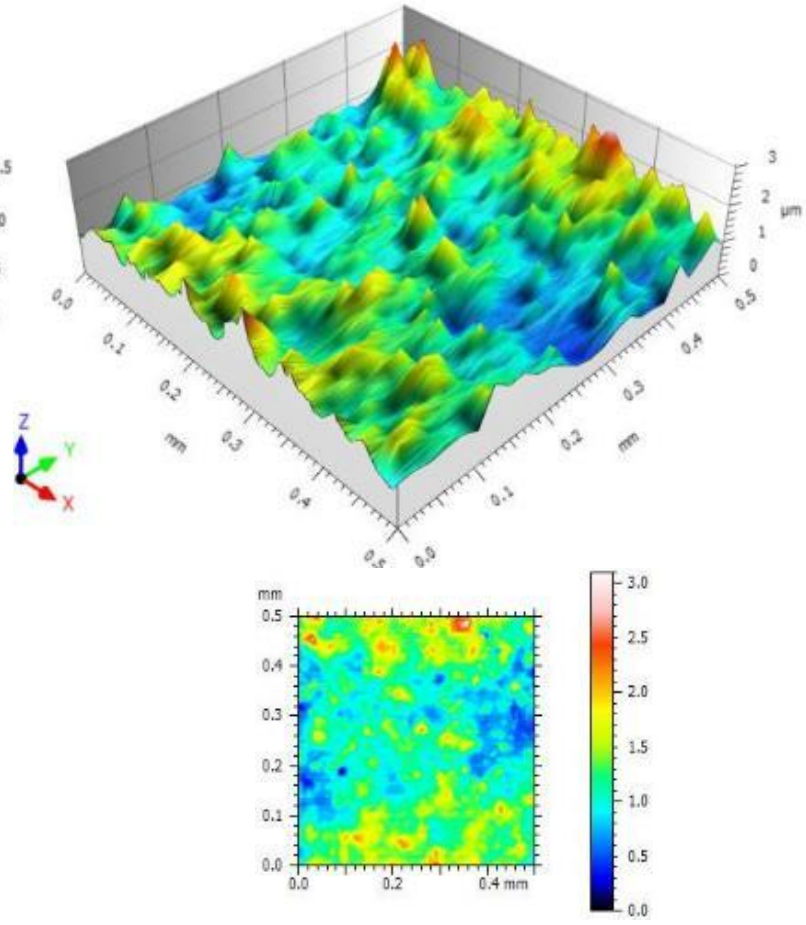
Control



20% $BaSO_4$ (3 µm)



20% ATH (11.5 µm)



The concentrated colors in the profile shows poor uniformity of the coating microroughness

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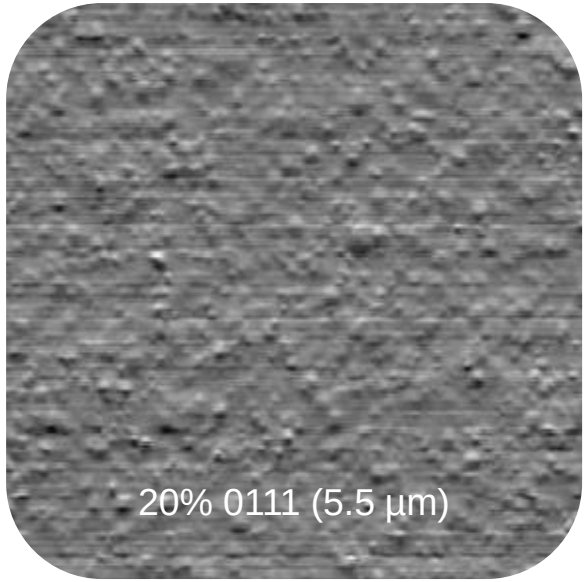
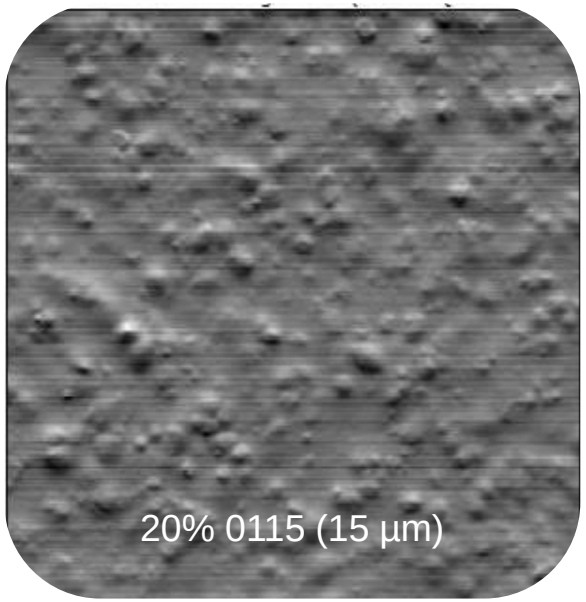
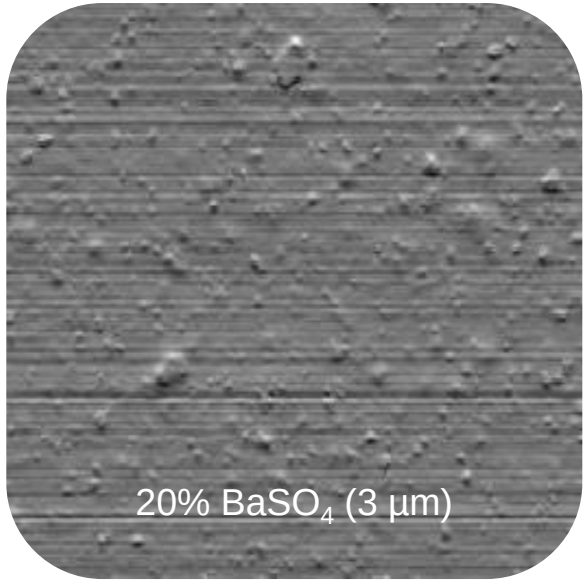
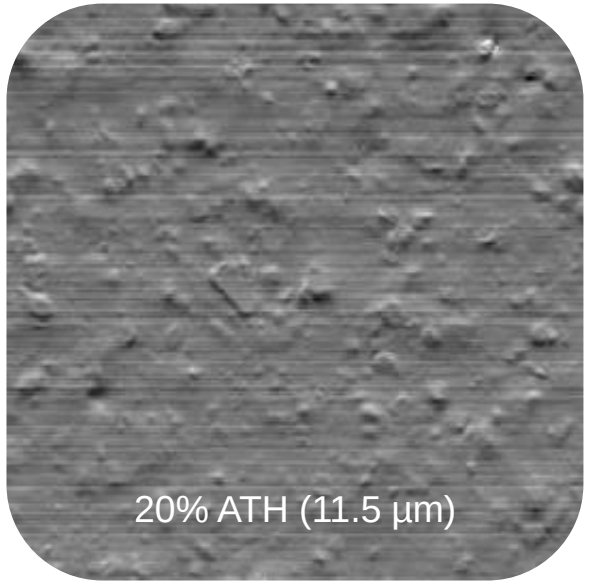
Matting Mechanism and Surface Profilometry – Polyester HAA Black SD









* Images created with Hommel Tester T8000

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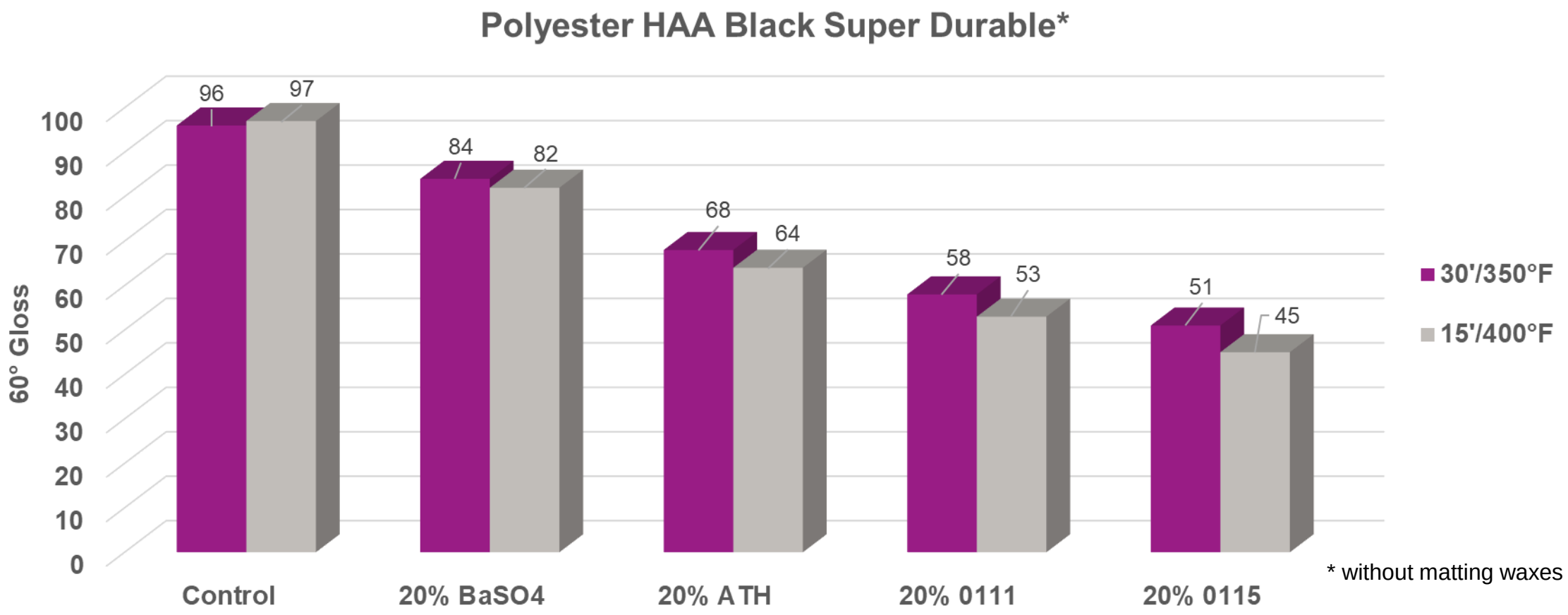
Temperature Dependence Matting Effect



* without matting waxes

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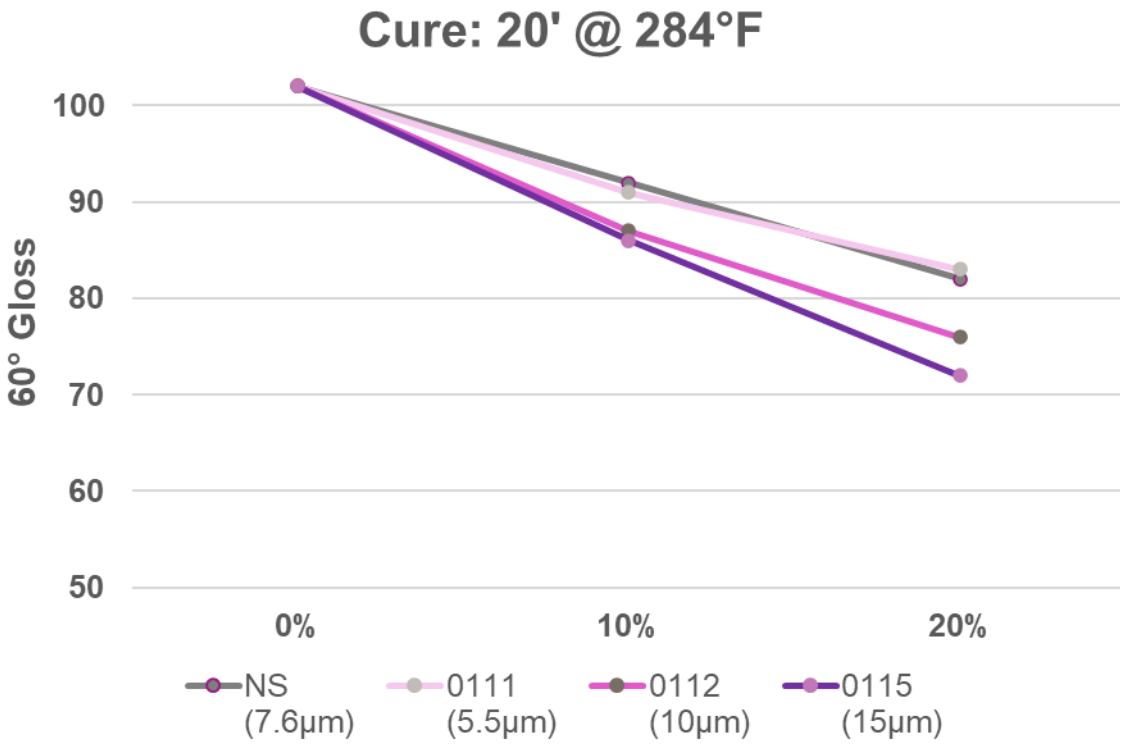
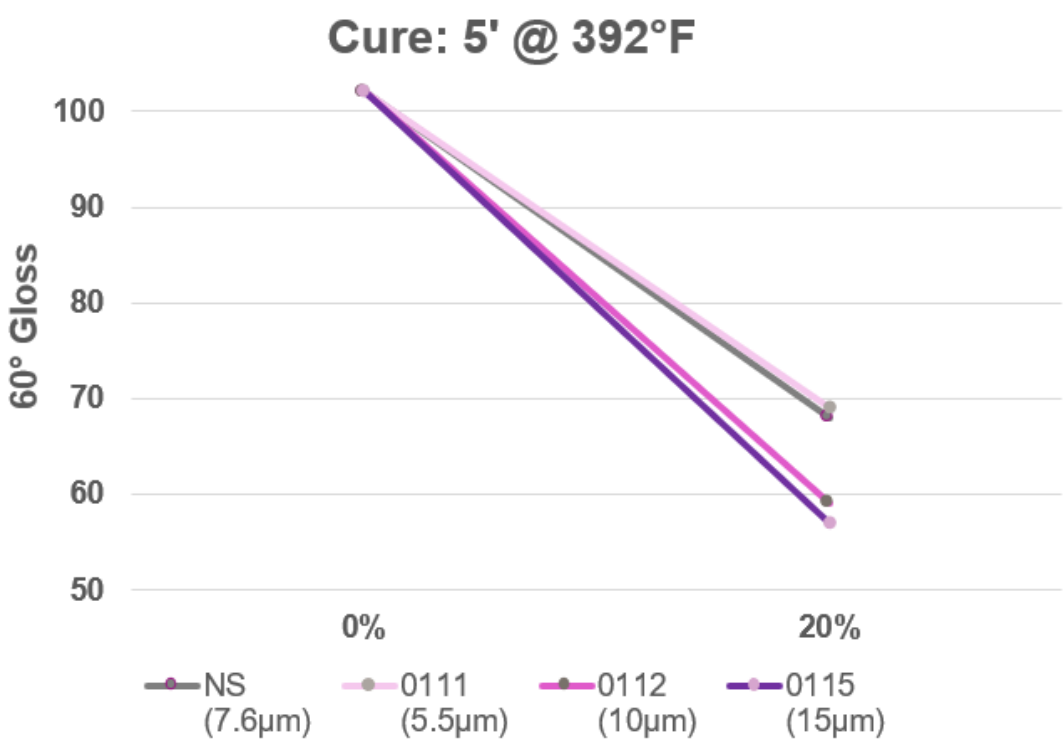
Temperature Dependence Matting Effect – MDF Coating

- Low cure gray polyester / epoxy hybrid (50:50) with nepheline syenite as reference filler; no matting waxes.





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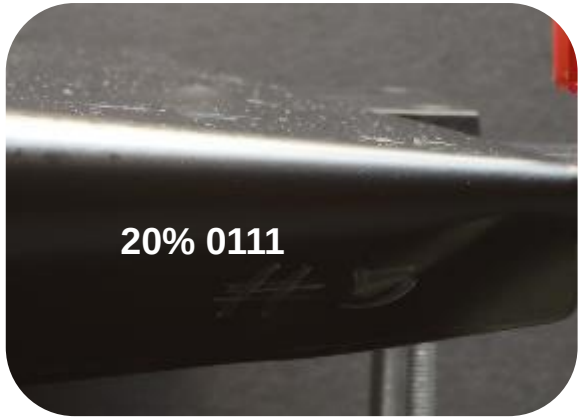
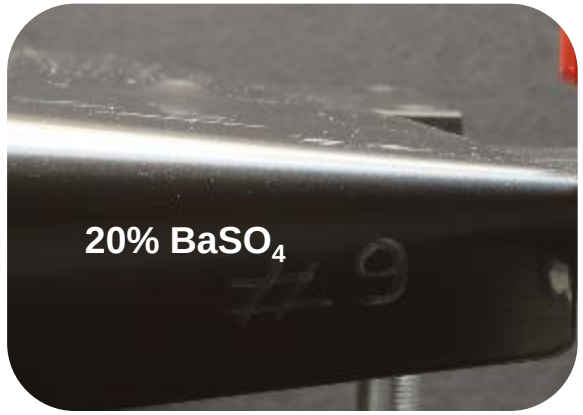
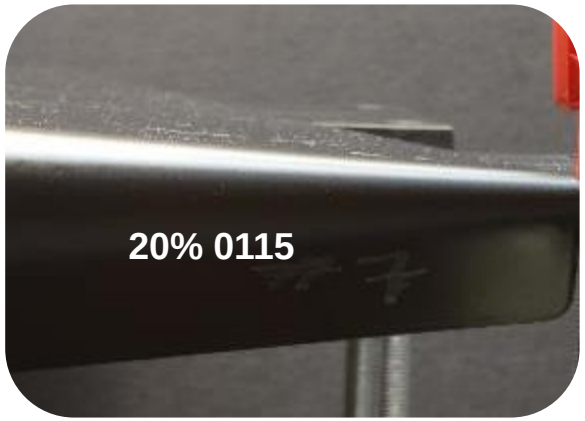
Physical Properties - Conical Mandrel Test – Polyester HAA Black SD

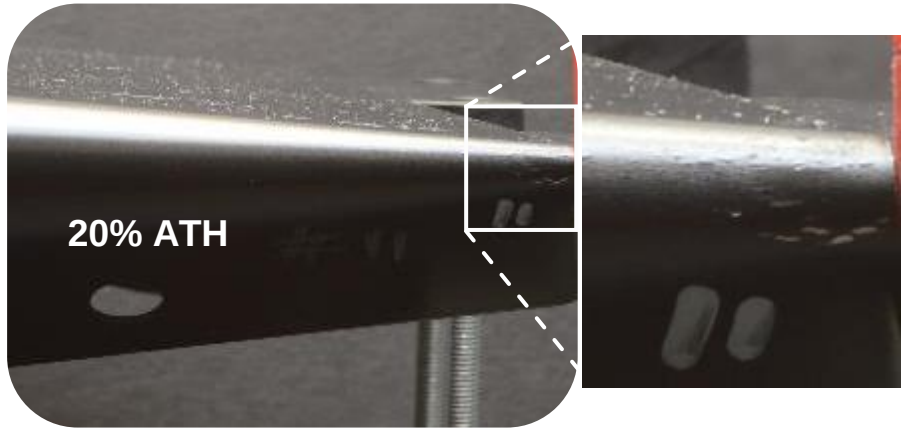
Modification	Conical Mandrel
Control	Pass
5% 0111	Pass
10% 0111	Pass
15% 0111	Pass
20% 0111	Pass
5% 0115	Pass
10% 0115	Pass
15% 0115	Pass
20% 0115	Pass
20% $BaSO_4$	Pass
5% 0115 / 15% $BaSO_4$	Pass
20% ATH	Fail
5% 0115 / 15% ATH	Fail





Substrate:
CRS B1000





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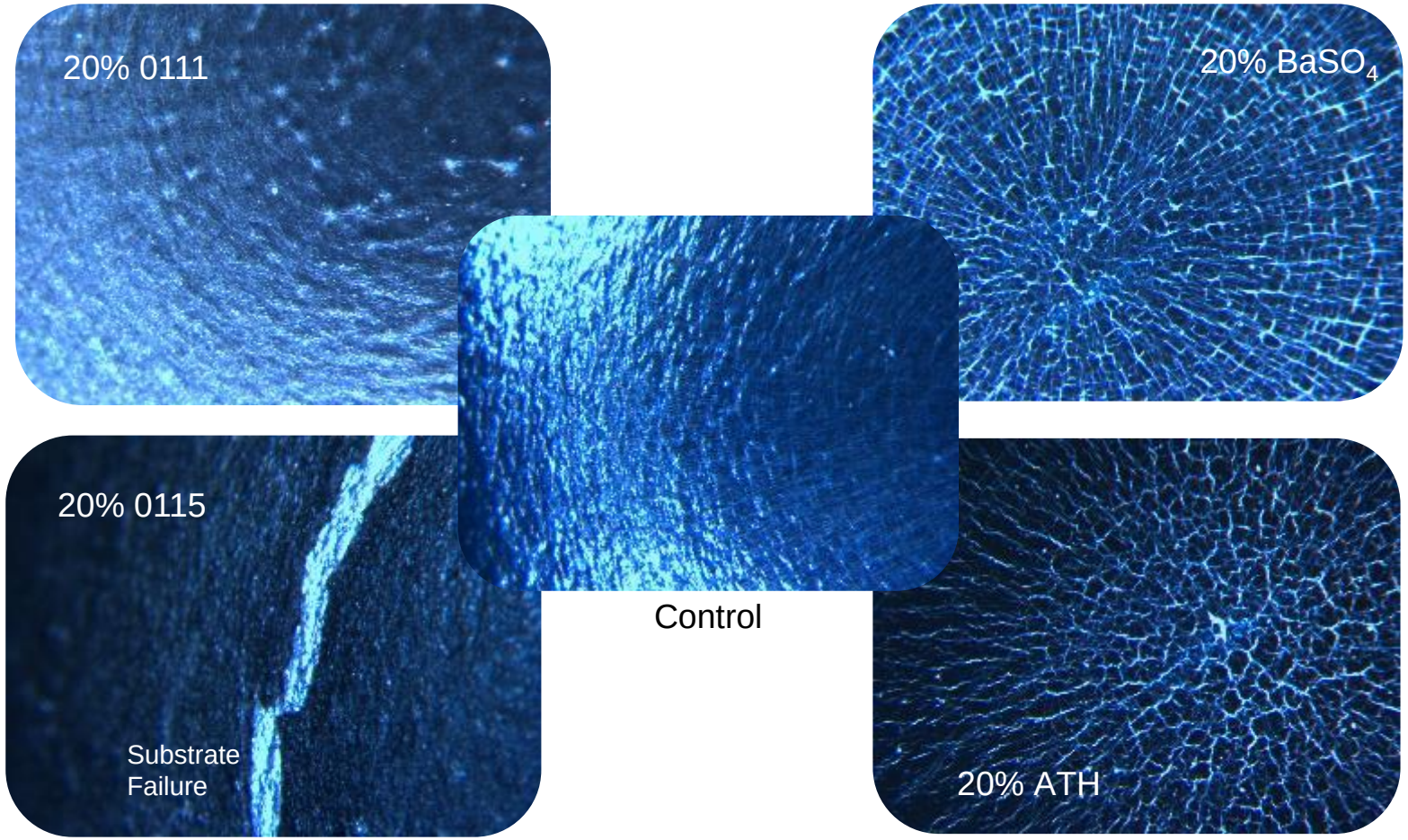
Physical Properties – Cupping Test – Polyester HAA Black SD

Modification	Cupping 6mm/s failure at [mm]
Control	2.0
5% 0111	4.4
10% 0111	8.6
15% 0111	7.8
20% 0111	4.0
5% 0115	5.6
10% 0115	6.9
15% 0115	9.7
20% 0115	8.9
20% $BaSO_4$	1.5
5% 0115/15% $BaSO_4$	2.1
20% ATH	1.4
5% 0115/15% ATH	1.9



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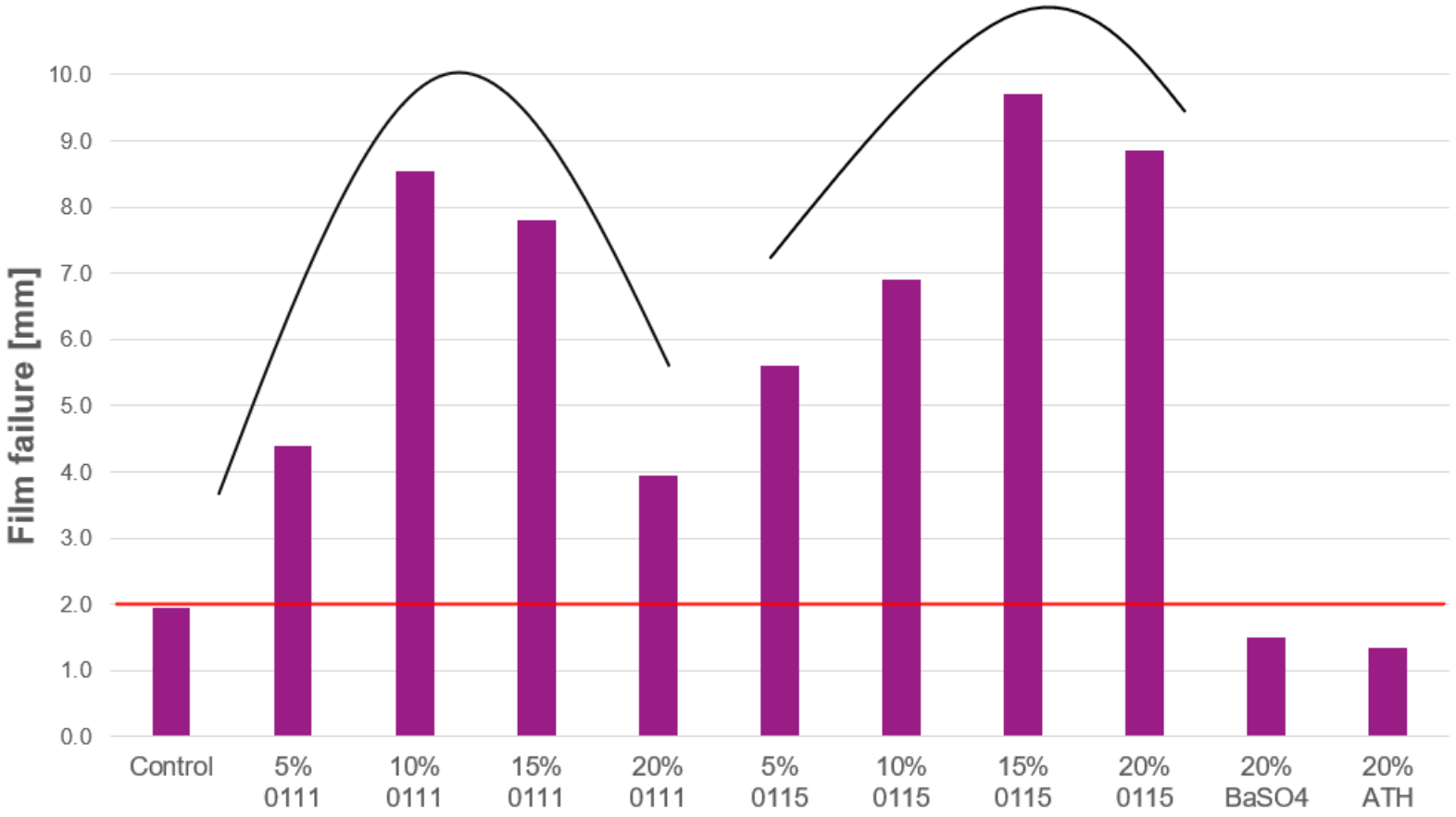
Physical Properties - Cupping Test – Polyester HAA Black SD

Modification	Cupping 6mm/s failure at [mm]
Control	2.0
5% 0111	4.4
10% 0111	8.6
15% 0111	7.8
20% 0111	4.0
5% 0115	5.6
10% 0115	6.9
15% 0115	9.7
20% 0115	8.9
20% $BaSO_4$	1.5
5% 0115/15% $BaSO_4$	2.1
20% ATH	1.4
5% 0115/15% ATH	1.9



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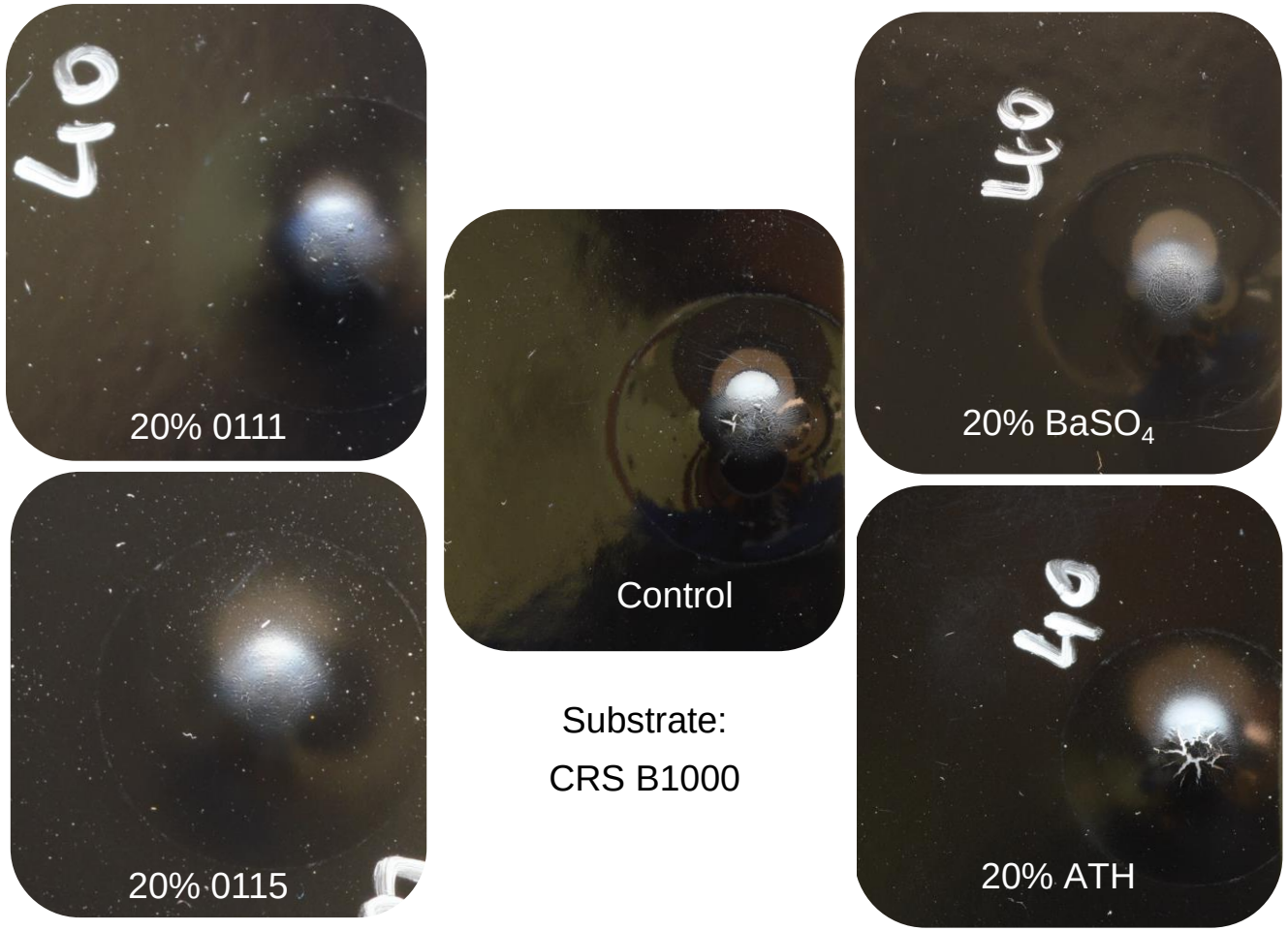
Physical Properties - Reverse Impact Test – Polyester HAA Black SD

Modification	Reverse Impact 40 in-lb @ 3.0 mils
Control	some cracking
5% 0111	some cracking
10% 0111	no cracking
15% 0111	no cracking
20% 0111	minor cracking
5% 0115	minor cracking
10% 0115	no cracking
15% 0115	no cracking
20% 0115	minor cracking
20% 0111/0115	minor cracking
20% $BaSO_4$	some cracking
5% 0115/ 15% $BaSO_4$	some cracking
20% ATH	severe cracking
5% 0115/ 15% ATH	severe cracking



Physical Properties - Pencil Hardness Test – Polyester HAA Black SD

	Mitsubishi Pencil Set
Modification	Pass Rating
Control	H
5% 0111	H
10% 0111	2H
15% 0111	3H
20% 0111	3H
5% 0115	2H
10% 0115	3H
15% 0115	4H
20% 0115	5H
25% 0115	7H*
30% 0115	9H*
10% 0111/ 10% 0115	4H
20% $BaSO_4$	H
5% 0115/ 15% $BaSO_4$	3H
20% ATH	3H
5% 0115/ 15% ATH	3H

Test Protocol

- Pencil hardness testing according to ASTM D3363

Results

- In addition to improved flexibility, coating systems containing Spherical Silica particles also improve in surface hardness.
- These properties often stand in contradiction to each other.

* difficult to measure / test

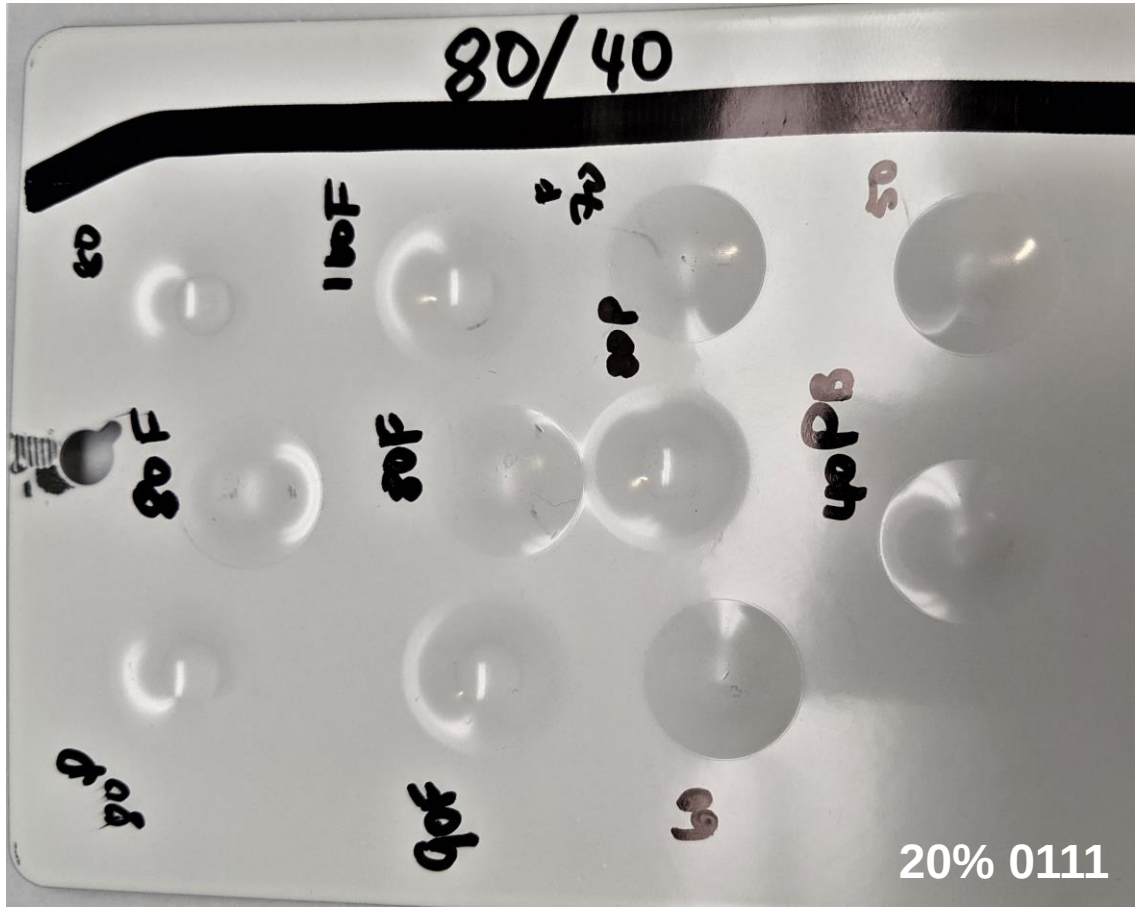
Spherical Silica Technology in Matting UV Cure Powder Coatings*

- Tested Formula:
  - White UV powder coating (2.0 - 2.5mils)
  - Standard durable polyester backbone
  - Compare to / blend with $BaSO_4$ filler
- Test Parameters and Results:
  - Gloss reduction – larger Spherical Silica particles have stronger matting effect
  - Impact resistance – incremental improvement with increased loading levels of Spherical Silica
  - Solvent resistance – no change
  - Conical mandrel – all no cracking
  - Adhesion – no change

Modification	Gloss	Impact [in-lb]	
	60°	Direct	Reverse
Control (20% $BaSO_4$)	90		
5% 0111 (15% $BaSO_4$)	86	50	10
10% 0111 (10% $BaSO_4$)	85	60	20
20% 0111 (no $BaSO_4$)	81	80	40
5% 0115 (15% $BaSO_4$)	83	40	10
10% 0115 (10% $BaSO_4$)	82	60	20
20% 0115 (no $BaSO_4$)	74	60	40

* Results provided by Kevin Otto at Keyland Polymer UV Powder, LLC

Spherical Silica Technology in Matting UV Cure Powder Coatings*



Modification	Gloss	Impact [in-lb]	
	60°	Direct	Reverse
Control (20% $BaSO_4$)	90		
5% 0111 (15% $BaSO_4$)	86	50	10
10% 0111 (10% $BaSO_4$)	85	60	20
20% 0111 (no $BaSO_4$)	81	80	40
5% 0115 (15% $BaSO_4$)	83	40	10
10% 0115 (10% $BaSO_4$)	82	60	20
20% 0115 (no $BaSO_4$)	74	60	40

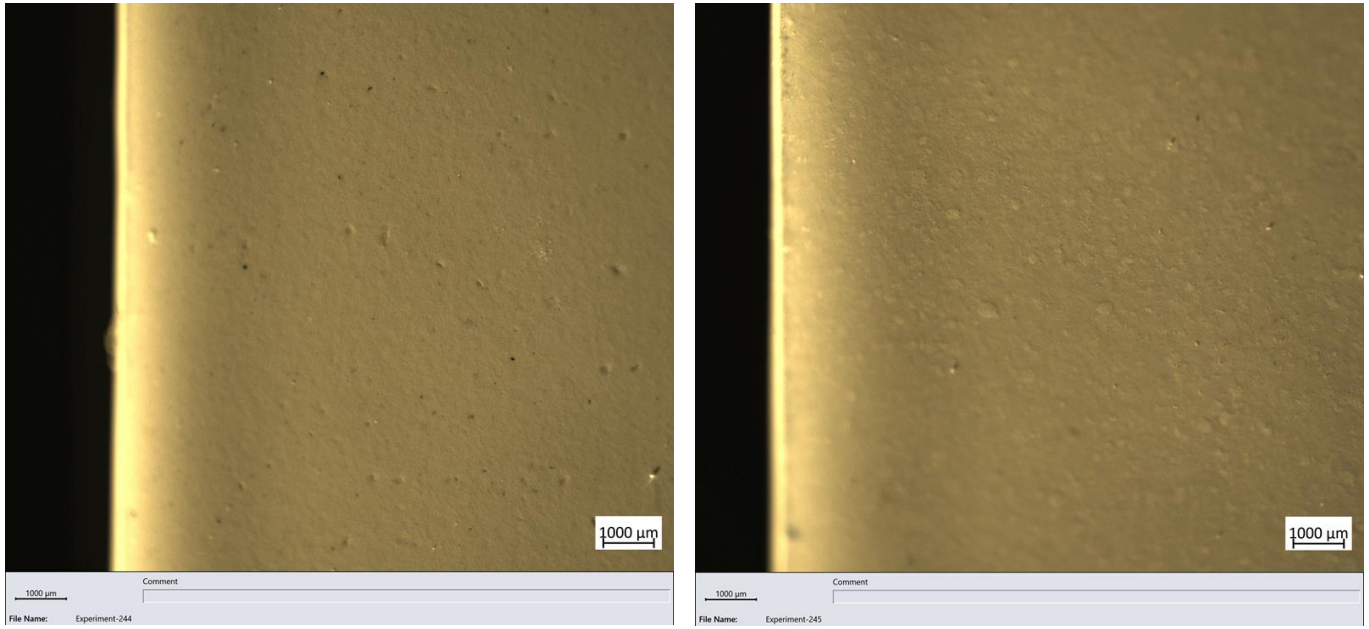
* Results provided by Kevin Otto at Keyland Polymer UV Powder, LLC

Spherical Silica Technology in Matted FEVE Powder Coatings*

- Little to no effect on processing conditions
- Passed FGIA (AAMA 2605) specifications (adhesion, flexibility)
- Gloss reduction from high 70s to low 50s at 60° angle (20% use level)
- Smoother surface appearance than other commercial matting agents

➢ Spherical Silica technology eliminated voids seen in commercial filler technology

➢ Spherical Silica technology produced raised textured appearance under microscope



Left image: 100% FEVE white with 20% commercial filler/ matting agent

Right image: 100% FEVE white with 20% Spherical Silica

* Results provided by Connie Przeslawski at AGC Chemicals America, Inc.

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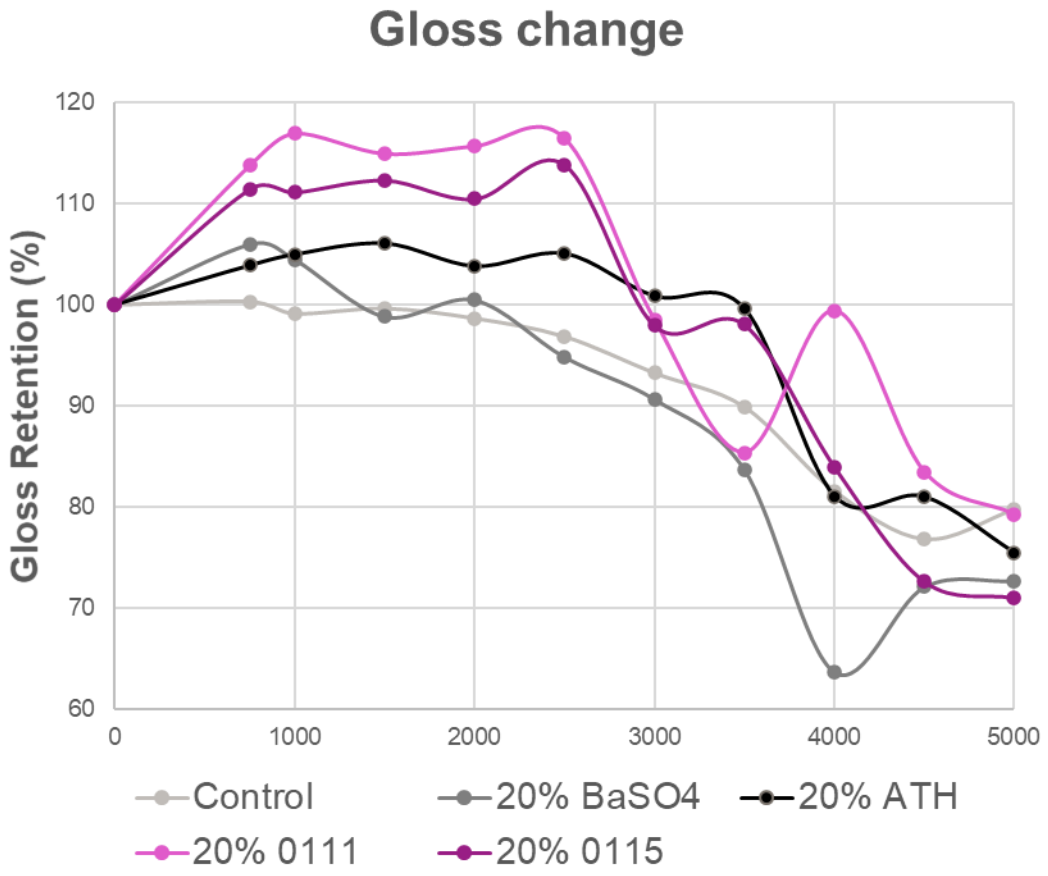
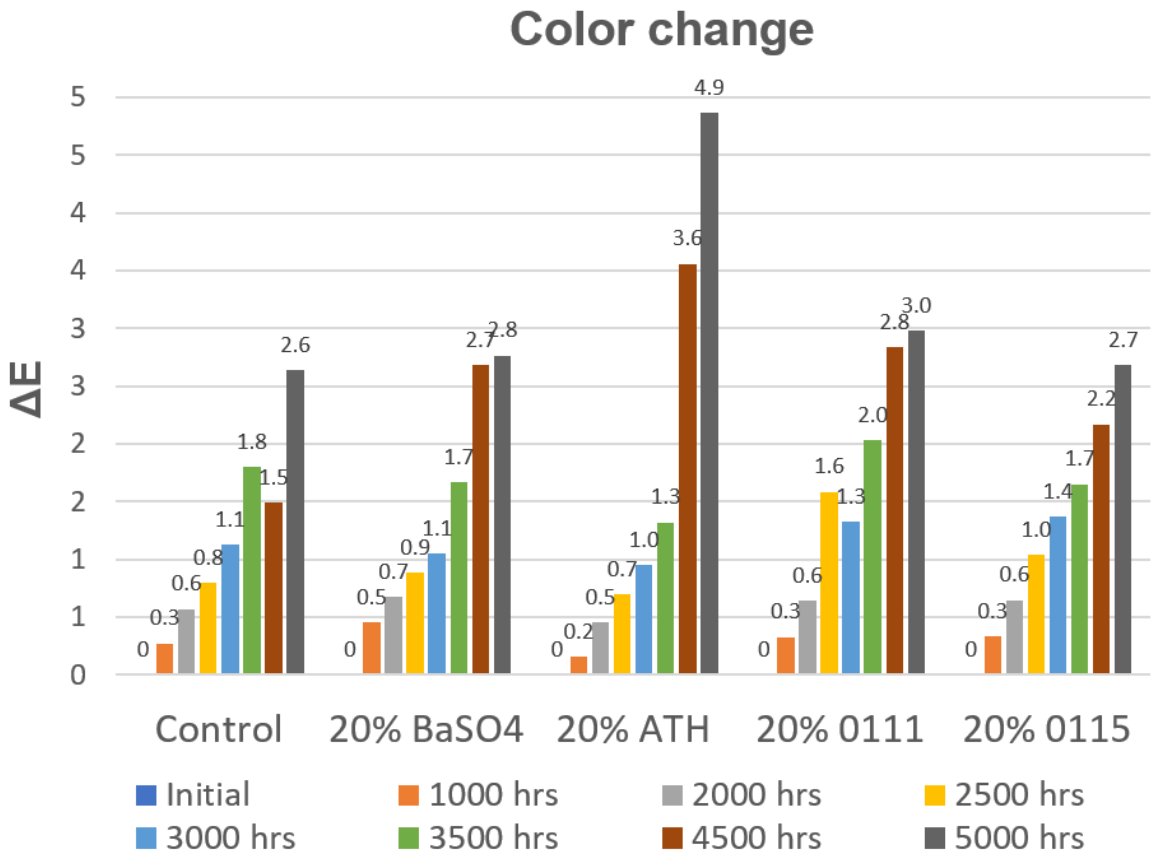
QUV-A Exposure – Polyester HAA Black Super Durable





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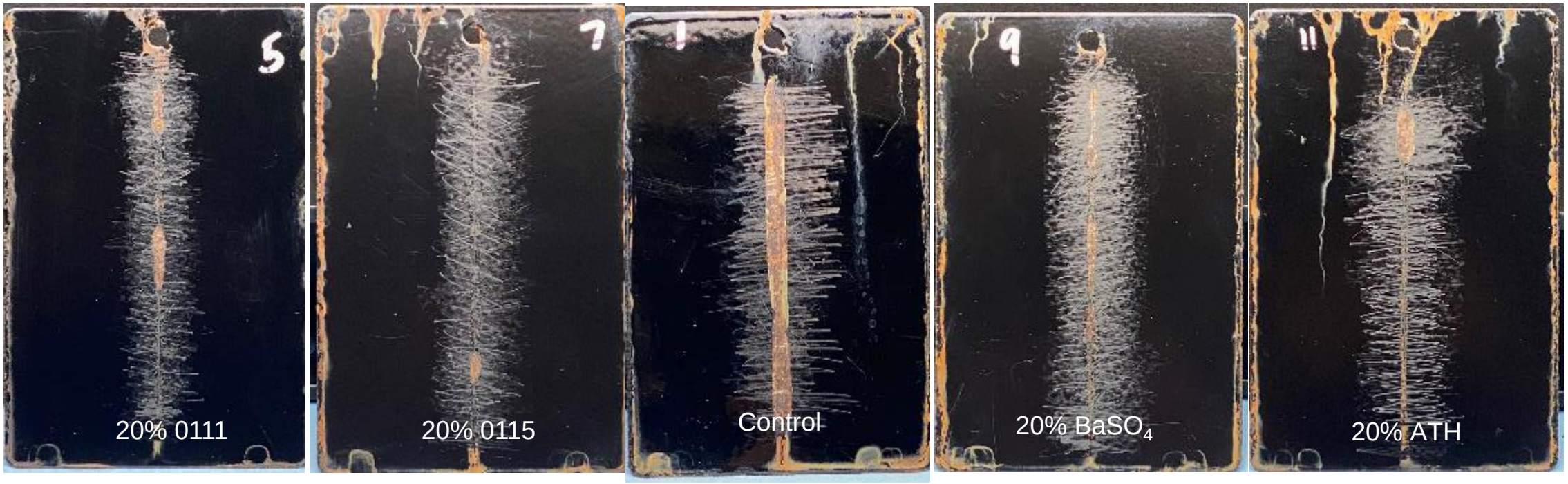
Corrosion Test – Polyester HAA Black SD – 1000 hrs B117 Salt Fog Cabinet



Substrate: CRS B1000

DFT: 75µm / 3 mils

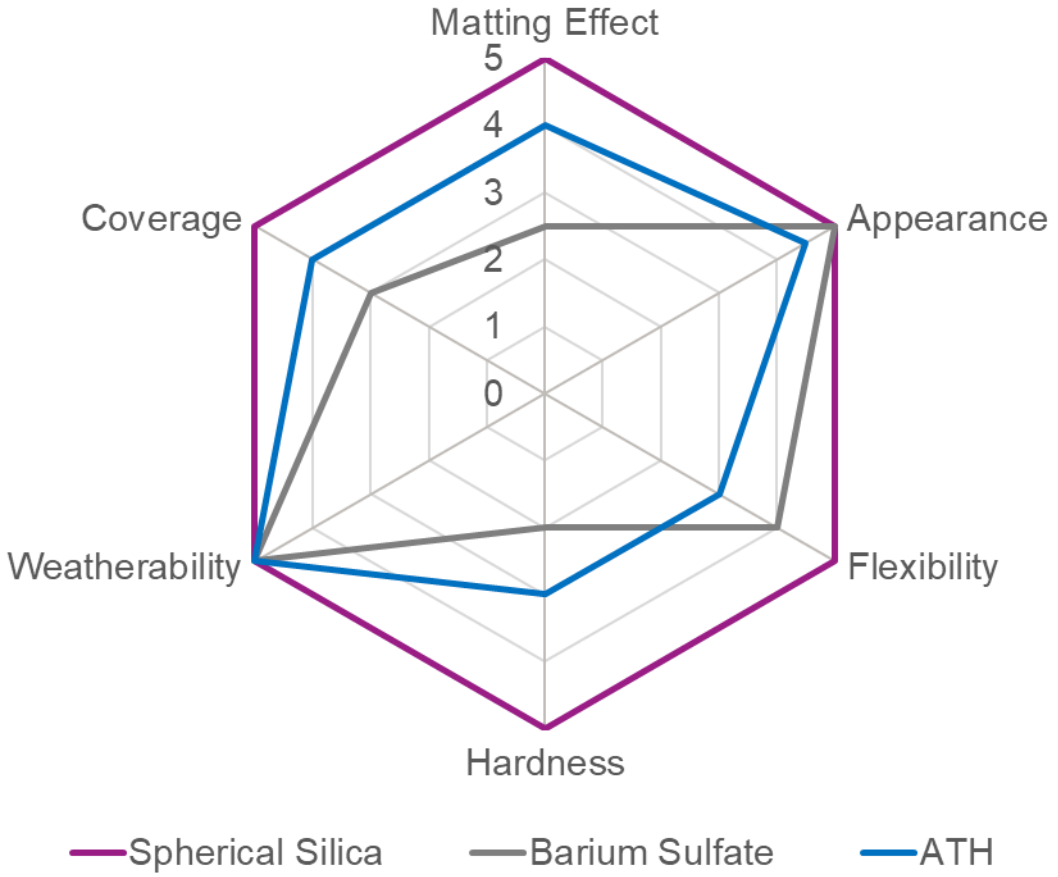
Calculated Powder Specific Gravity and Coverage – Polyester HAA Black SD

Modification	Gloss	Specific Gravity	Theoretical coverage [sq.ft./lb] @ 2.5mils	Coverage
Control	97	1.25	61	111%
15% 0111	63	1.33	58	104%
15% 0115	57	1.33	58	104%
20% Aluminium Trihyroxide	64	1.39	55	100%
5% 0115 / 15% Portafill A40	61	1.38	56	101%
12.5% 0115	63*	1.32	58	106%
10% 0115 / 10% BaSO4	62*	1.41	55	99%
20% Barium Sulfate	82	1.46	53	95%

* Estimated gloss level based on linear matting performance

Summary of Performance Characteristics – Spherical Silica Technology for Matting Powder Coatings

- Improved matting efficiency versus ATH or $BaSO_4$
- Matting efficiency is linear and predictable
- Excellent uniformity and consistent appearance across substrate
- Does not affect adhesion but improves hardness and flexibility
- Lower specific gravity allows for greater coverage
- Choose between the smaller 0111 or larger 0112 and 0115 particles, depending on desired coating appearance and performance.



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