Impact Durability Assessment and Anti-Ice Adhesion Performance of Epoxy Coatings

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Purpose

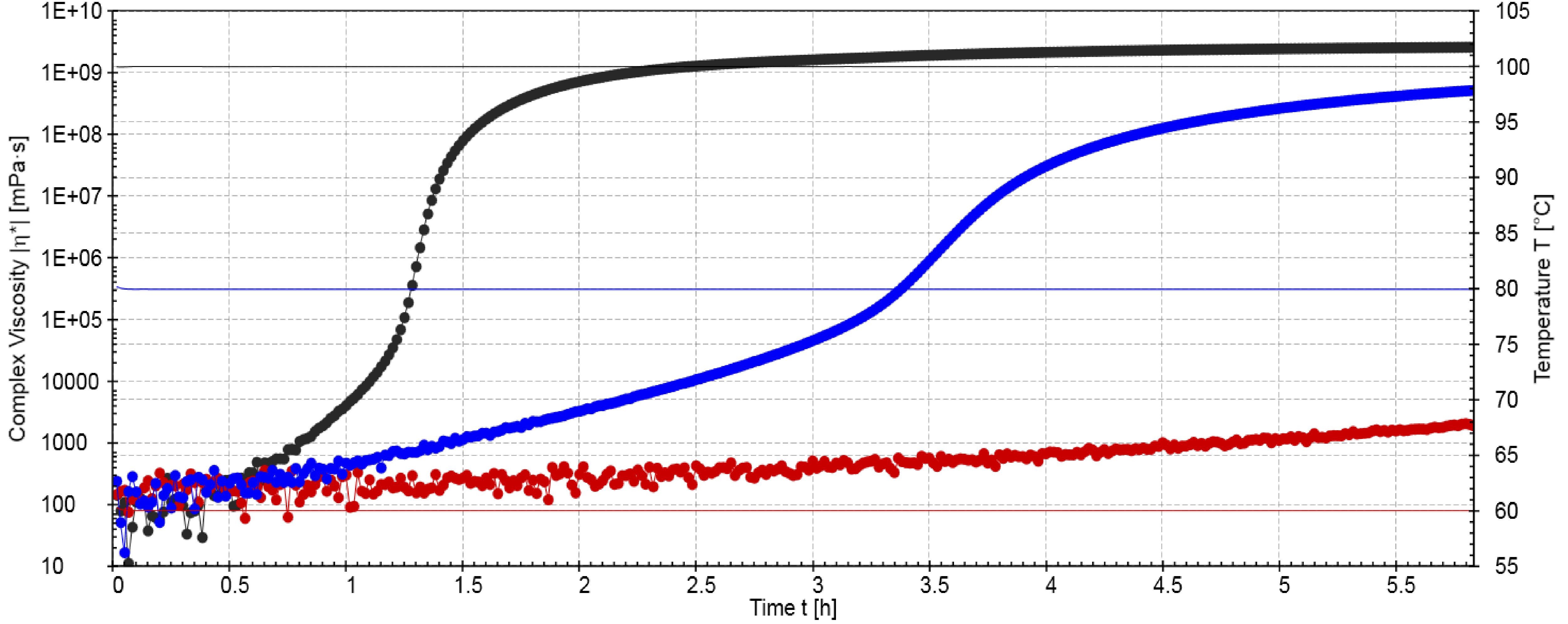
- This project is to establish a standard work practice for characterizing the durability of coating formulations in relation to aircraft weathering exposure.
- In order to assess coating durability, a panel of instruments have been arranged that will illustrate a broad understanding of their performance under environmental interference.

Coating Preparation

- In order to accurately assess a coating’s ability to withstand durability testing, an evenly coated surface has to be created.
- Upon curing a coating, a preliminary visual inspection is performed that determines whether it will advance to further characterization.
- The abilities and limitations of the coatings were assessed by experimenting with the epoxy’s reaction period.
- There was considerable challenge presented by the need to coat vertical surfaces that have an initial drop in viscosity during the high temperature curing process.

Manipulating Viscosity

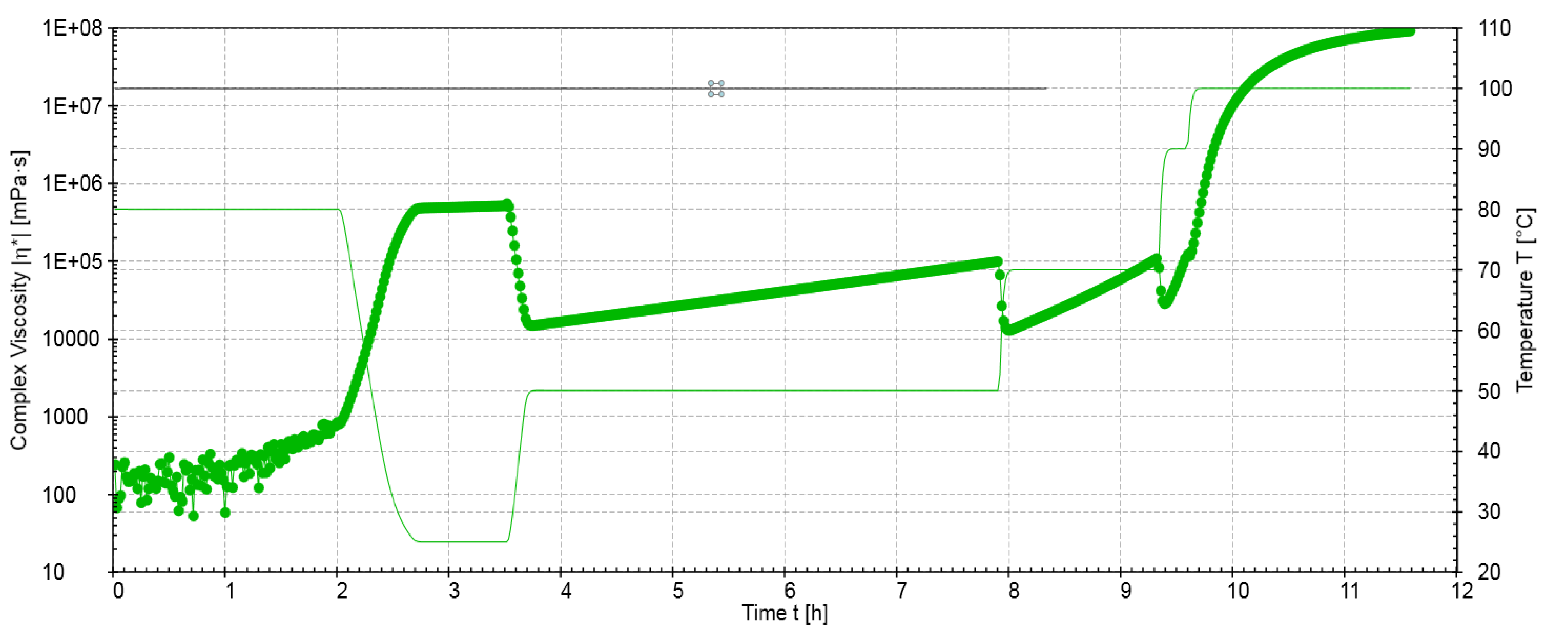
- It was understood that in order to evenly coat a metal substrate, an epoxy coating had to advance in reaction time (B-stage) until a sufficient viscosity was attained to prevent de-wetting the surface.
- Additionally, the flow of the epoxy couldn’t be so thin that it would “run” off the edges of the 7.62cm x 15.24cm surfaces. This became much more important when applying coatings to air foils that had vertical surfaces in need of curing at 177°C.
- The solution began with observing Rheological Data at programmed temperature cycles to create a customized cure schedule that enabled an even coated surface to cure in a period less than one week.



Complex Viscosity |η*| [mPa·s]
1E+10
1E+09
1E+08
1E+07
1E+06
1E+05
10000
1000
100
10
Temperature T [°C]
105
100
95
90
85
80
75
70
65
60
55
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
5.5
Time t [h]

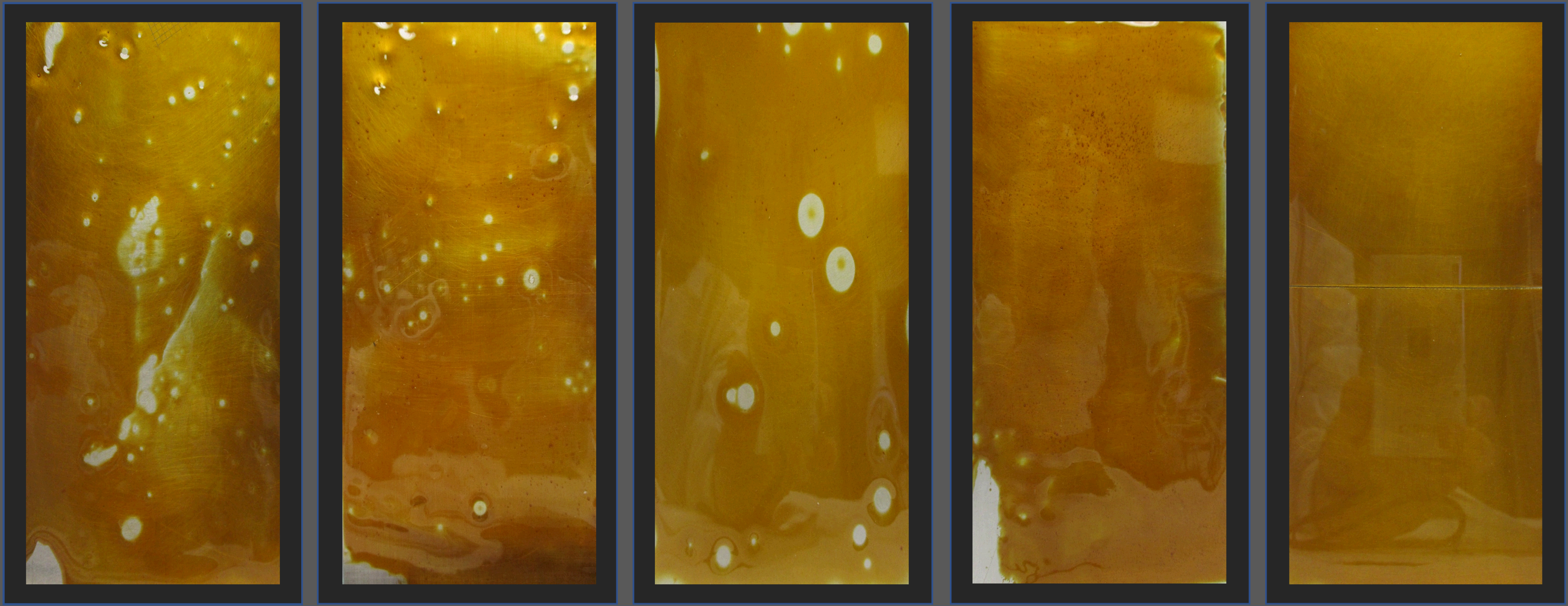
Rheological Data Success

- A Rheological study was conducted to monitor the epoxy’s viscosity changes throughout the cure process as temperature increases.
- A viscosity above ~10,000 cP is needed to be maintained in order to ensure that the coating remained adhered to a vertical surface.
- A series of low temperature ramp up cycles was designed to allow the coating samples to cool down and increase in viscosity before advancing to the next higher temperature. This allowed the epoxy to cure in increments before the final temperature of 177°C solidified the cure.



1E+08
1E+07
1E+06
1E+05
10000
1000
100
10
Complex Viscosity |η*| [mPa·s]
110
100
90
80
70
60
50
40
30
20
Temperature T [°C]
0
1
2
3
4
5
6
7
8
9
10
11
12
Time t [h]

Progression of Coating Procedure Improvement



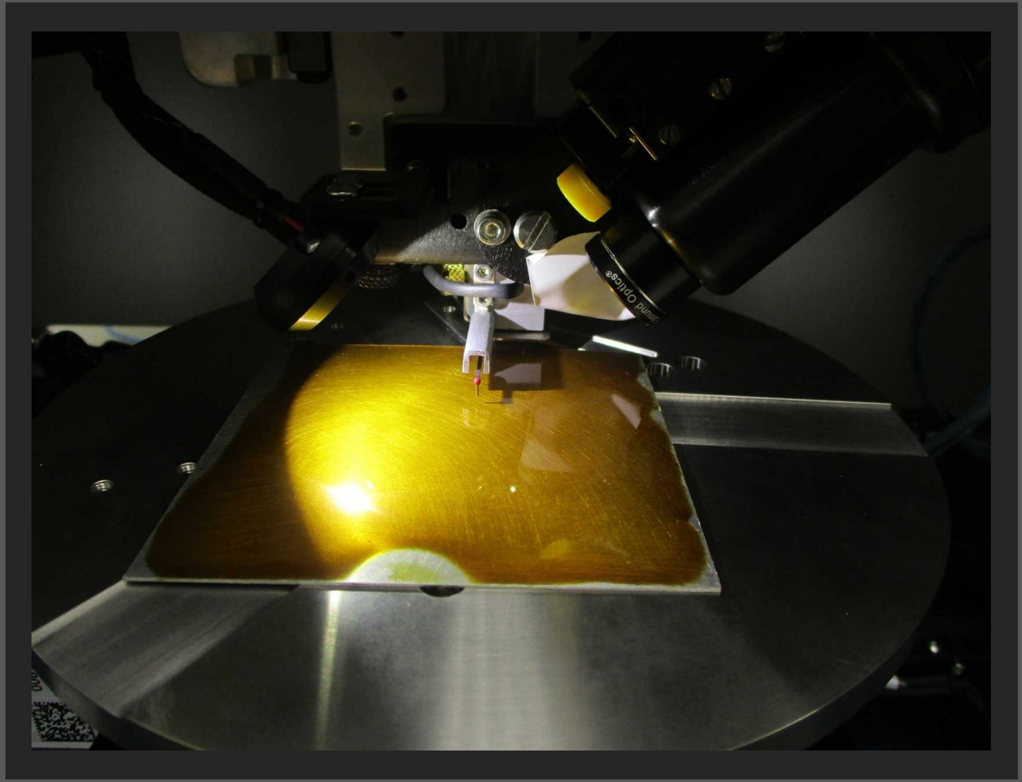
Durability Assessment

- Durability assessment began after a scaled-up procedure yielded multiple panels on which to test.
- Each panel was assigned a group of tests that assessed its durability in a sequence, testing non-invasive data prior to mechanical testing.

Panel I.D.		ü/û		ü/û		ü/û		ü/û		ü/û		ü/û		ü/û	B&A	Test Abreviations	
P(A)	Impact	✓	Prof	✓												Pencil Hardness Test	PHT
P(B)	CA	✓	PHT	✓	Scler	✓	Flex		Gloss	✓	FTIR					Sclerometer	Scler
P(C)	~~Mandrel Bend Training Sample~~															Cross Hatch	CH
P(D)	~~Sub-par coating~~															Impact	Imp
P(E)	~~Sub-par coating~~															Flexibility (Mandrel)	Flex
P(F)	Impact	✓														Profilometry	Prof
P(G)	CA	✓	PHT	✓	Scler	✓	Flex		Gloss	✓	FTIR					Gloss	Gloss
P(H)	UV		PHT		Scler				Gloss		Imp		CA			Contact Angle	CA
P(I)	TC		PHT		Scler		CH		Gloss				CA			FTIR	FTIR
P(J)	UV		PHT		Scler		CH		Gloss				CA			Thermal Cycle	TC
P(K)			Prof	✓			CH	✓								UV Weathering	UV
P(L)	TC		PHT		Scler				Gloss		Imp		CA			Ethylene Glycol Soak	EGS
FP(A)	EGS 1 Day		PHT		Scler		CH		Gloss		FTIR					Skydrol Soak	SDS
FP(B)	EGS 7 Day		PHT		Scler		CH		Gloss		FTIR					Jet Fuel Soak	JFS
FP(C)	SDS 1 Day		PHT		Scler		CH		Gloss		FTIR					Insect Impact	Bugs
FP(D)	SDS 7 Day		PHT		Scler		CH		Gloss		FTIR						
FP(E)	JFS 1 Day		PHT		Scler		CH		Gloss		FTIR						
FP(F)	JFS 7 Day		PHT		Scler		CH		Gloss		FTIR						

Profilometry

- This test is conducted prior to the coating surface being damaged by the more physical mechanical tests.



Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Flat Panel (A) - Coated 7/3/2019 Profilometry Tested: 7/10/2019				
	Ra	**Rq**	**Rsk**	**Rku**
P (A) 1	0.025µm	0.033µm	0.342	3.41
P (A) 2	0.033µm	0.041µm	-0.061	2.361
P (A) 3	0.032µm	0.043µm	-0.139	2.878
P (A) 4	0.029µm	0.037µm	-0.3	2.211
P (A) 5	0.002µm	0.025µm	-0.166	2.559

Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Flat Panel P(K) - Coated 7/3/2019 Profilometry Tested 07/15/2019				
	Ra	Rq	Rsk	Rku
P (K) 6	0.013µm	0.016µm	-0.76	3.86
P (K) 7	0.016µm	0.023µm	-0.087	3.4
P (K) 8	0.018µm	0.024µm	-0.1	2.793
P (K) 9	0.014µm	0.018µm	-0.198	2.459
P (K) 10	0.012µm	0.016µm	-0.216	3.566

Contact Angle Goniometry

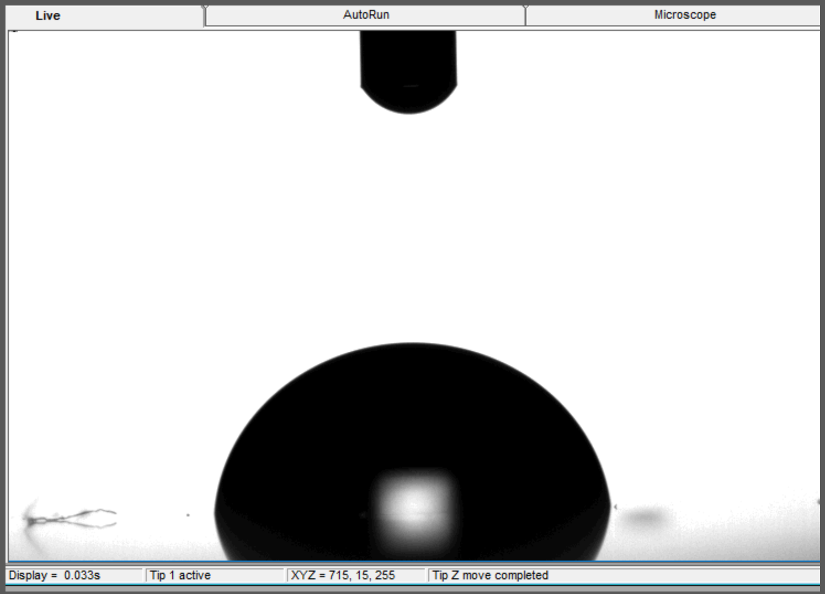
- This test uses pure water and ethylene glycol to characterize the epoxy coating surface chemistry.
- By using a sessile droplet method observing the surface energy of the coating, an assessment of the coating's wettability, can be understood in comparison to other epoxy formulations. [1]

Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Panel P(B) **H_2O** 7/15/2019			
Sample ID	Sessile	Advancing	Receding
P(B) 1	72.93	74.62	56.15
P(B) 2	75.36	76.02	59.84
P(B) 3	75.06	76.04	58.95

Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Panel P(B) **$C_2H_6O_2$** 7/16/2019				
Sample ID	Sessile	Advancing	Receding	Roll Angle
P(B) 1	54.43	58.43	30.42	38°
P(B) 2	58.31	64.58	28.93	56°
P(B) 3	55.94	64.51	25.59	52°

Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Panel P(G) **H_2O** 7/15/2019			
Sample ID	Sessile	Advancing	Receding
P(G) 1	84.29	84.04	66.71
P(G) 2	85.20	86.50	67.53
P(G) 3	81.68	86.84	70.14

Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% 7.62cm x 15.24cm Panel P(G) **$C_2H_6O_2$** 7/16/2019				
Sample ID	Sessile	Advancing	Receding	Roll Angle
P(G) 1	63.33	66.41	35.89	48°
P(G) 2	59.38	64.75	33.27	48°
P(G) 3	64.72	68.83	36.49	52°

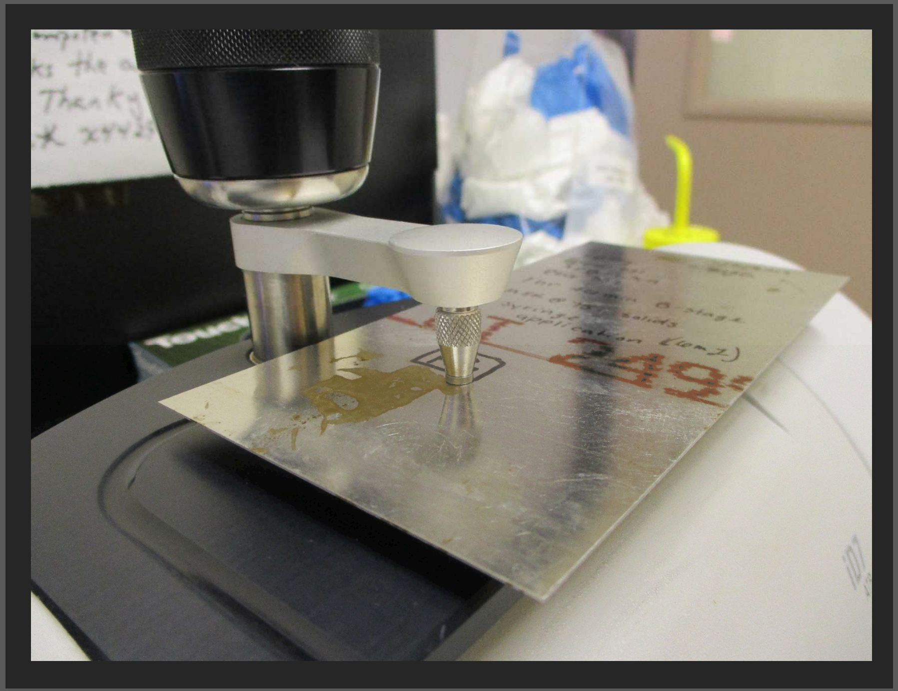


Specular Gloss Meter

- This test measures the way light is reflected off a coating surface to assess its shininess. [2]
- This helps to understand its visibility factors like surface haze or texture that will be compared before and after solvent soak and weathering exposure. [2]

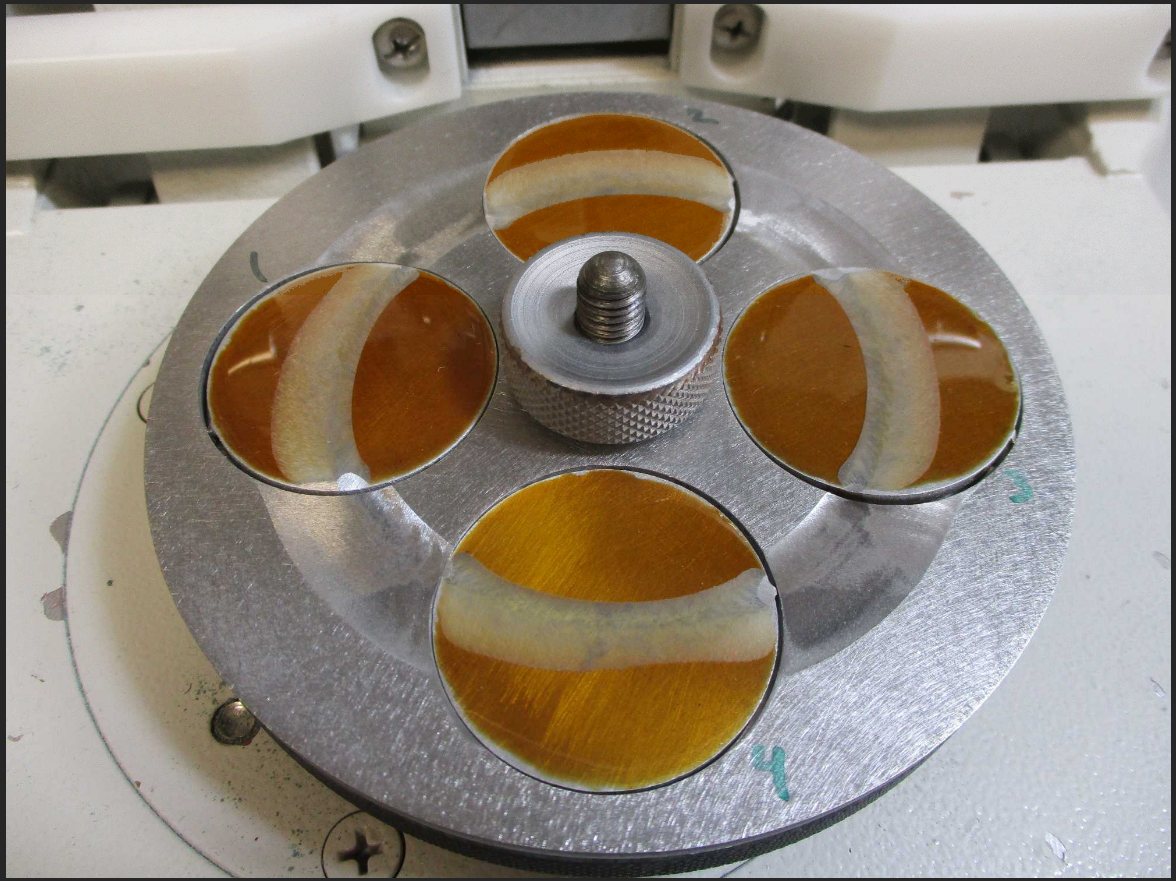
ATR-FTIR Spectroscopy

- This test examines the functional groups of the cured epoxy's chemical composition.



Taber Abrasion

- This instrument measures the rate which the coating can be abraded with friction over series of 1200 rotations. [3]
- By calculating a wear index per the ASTM Standard, different coating formulas can be compared in terms of abrasion durability. [3]



Disc #	Rotations	Wear Index
#1B	400-1200	**17.7500**
#2B	400-1200	**18.5833**
#3B	400-1200	**17.1667**
#4B	400-1200	**19.4167**

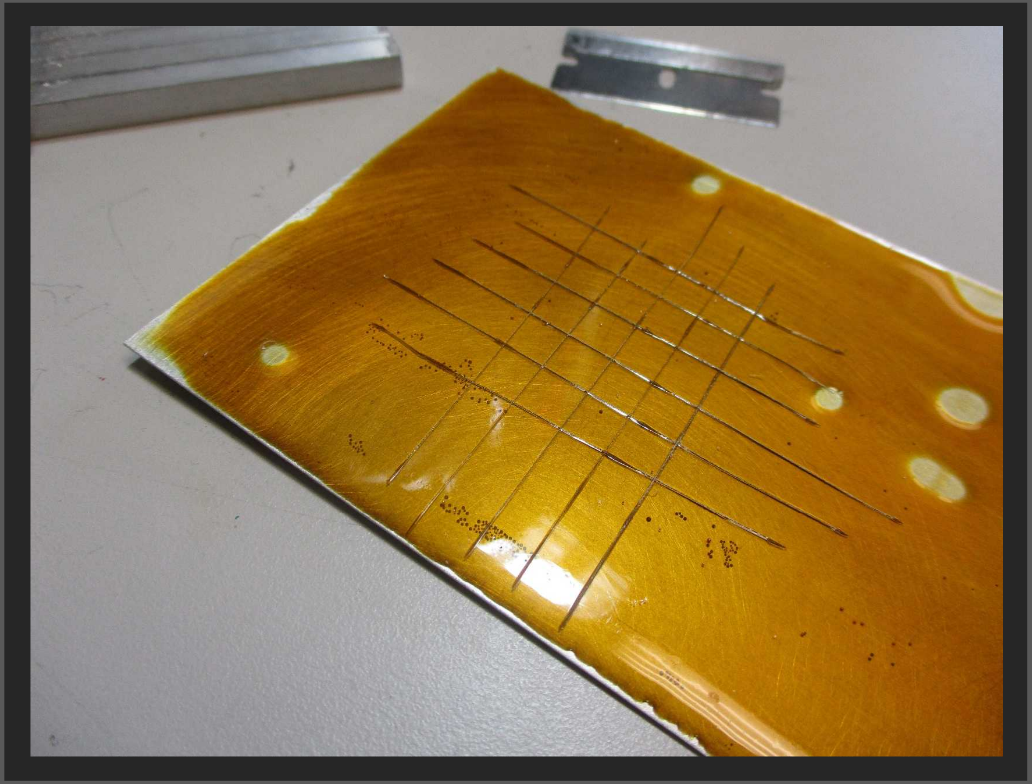
Epoxy APB 1,3,4 - D.E.R. 331 - P.E.G.: 35% - Coated 7/8/2019
Taber Abrasion Tested on: 7/12/2019

Disc #	Initial Thickness	After 400 Rnds	Final Thickness		Initial Mass		After 400 Rnds		Final Mass	g
#1B	626.4 ± 46.1µm	577.3 ± 48.6µm	518.1 ± 75.4µm		7.7423	g	7.7236	g	7.7023	g
#2B	615.8 ± 42.0µm	561.2 ± 32.7µm	484.2 ± 83.2µm		7.7244	g	7.7054	g	7.6831	g
#3B	624.6 ± 21.4µm	604.4 ± 88.8µm	556.6± 73.7µm		7.7746	g	7.7541	g	7.7335	g
#4B	592.2 ± 31.1µm	554.6 ± 58.4µm	493.1 ± 63.0µm		7.7237	g	7.7039	g	7.6806	g

SJG(2

Cross Hatch

- This mechanical surface test characterizes the coating's adhesion to the metal surface after being scratched or damaged.[4]
- By placing perpendicular slices in the surface of the coating and applying upward force, the adhesion between the coating and substrate can be assessed.[4]



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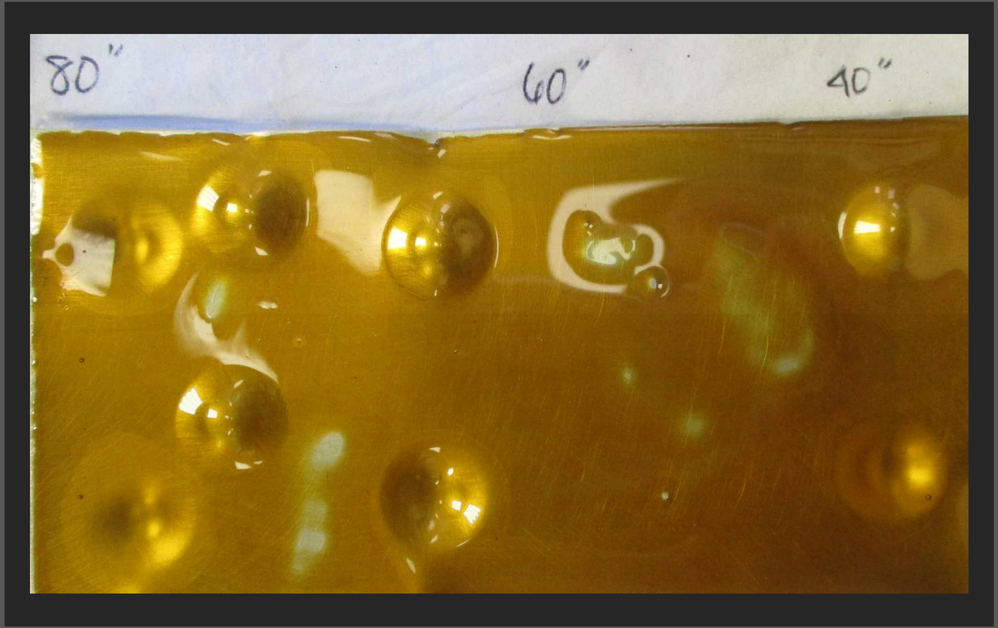
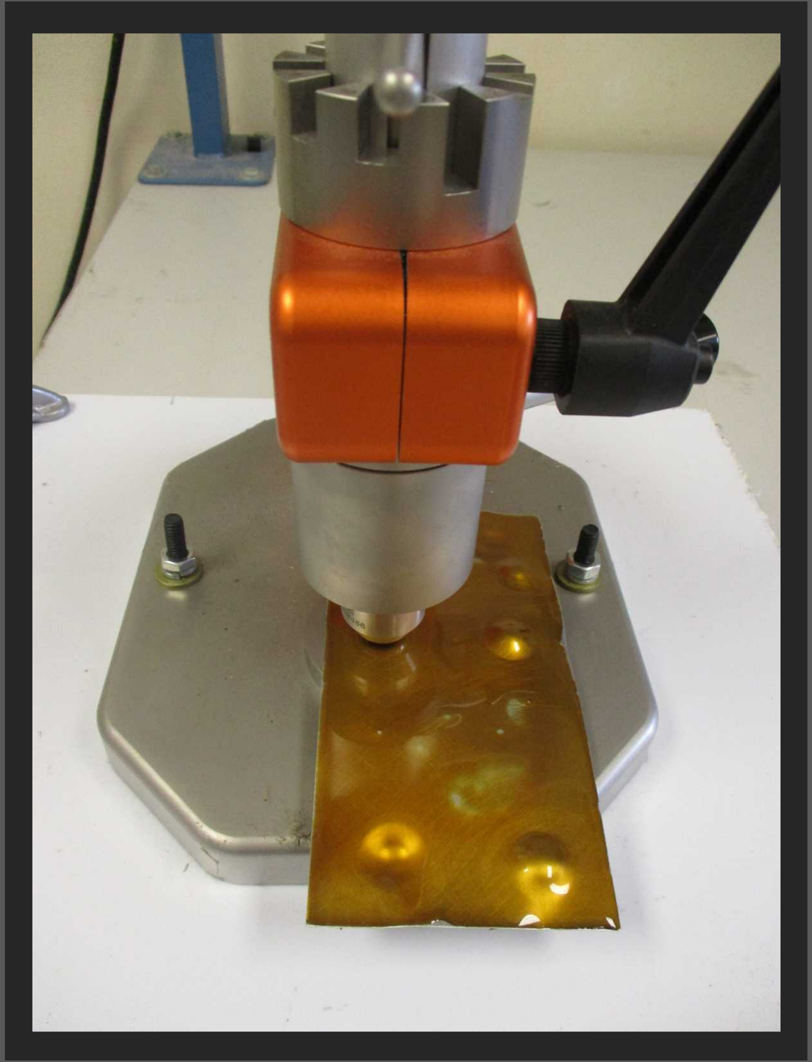
SJG(2 bulleted items are redundant in what they state
Smith, Joseph G. (LARC-D307), 7/30/2019

Impact Testing

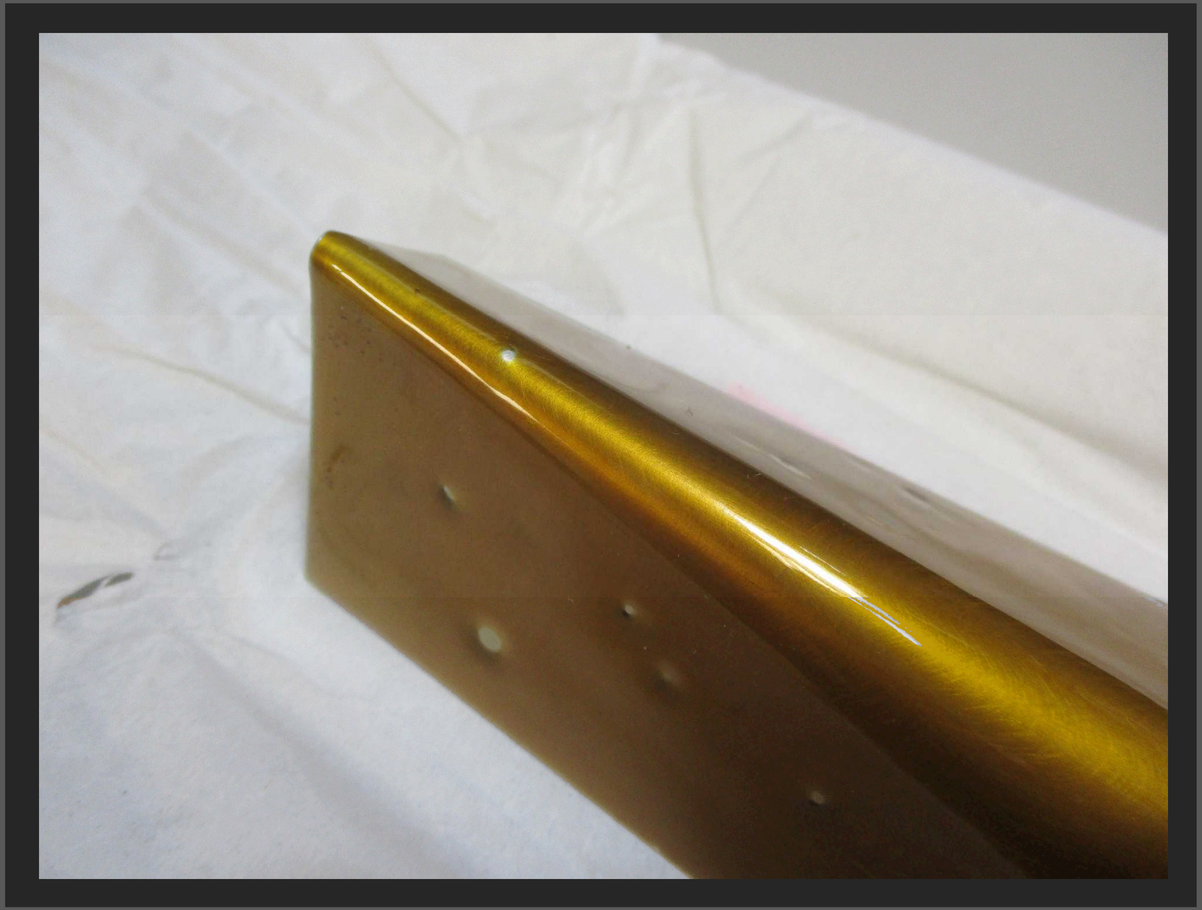
- This mechanical test uses rapid deformation to mimic an in-flight or falling object debris impact.[5]
- This test characterizes a coating's ability to flex with impact and resist cracking.[5]





Mandrel Bend

- This mechanical test determines coating flexibility by folding it over a steel conical roller at 135°C. The stress lines or cracks are then measured in length to determine the coating's elongation. [6]



Pencil Hardness Test

- This mechanical test inexpensively assesses the coating’s sensitivity to scratching.[7]
- The known hardness of pencil lead values will be used to scratch the surface before and after weathering and solvent soak to assess the break down of the surface’s integrity.[7]

Sclerometer

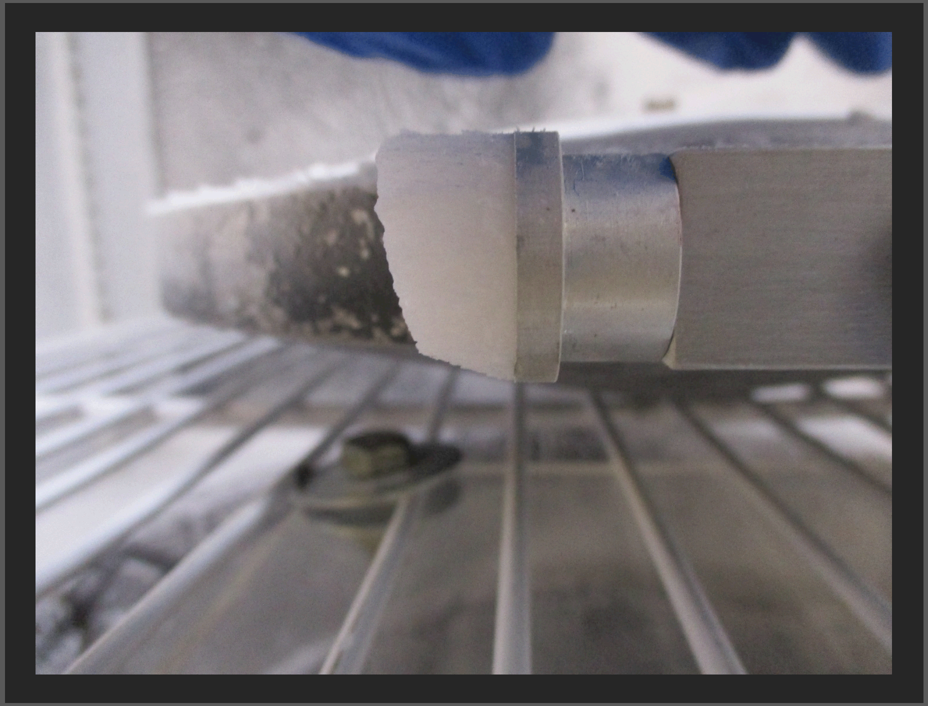
- This mechanical test performs a similar action as the Pencil Hardness test, but the results are expressed in terms of Newtons required to scratch a coating surface.

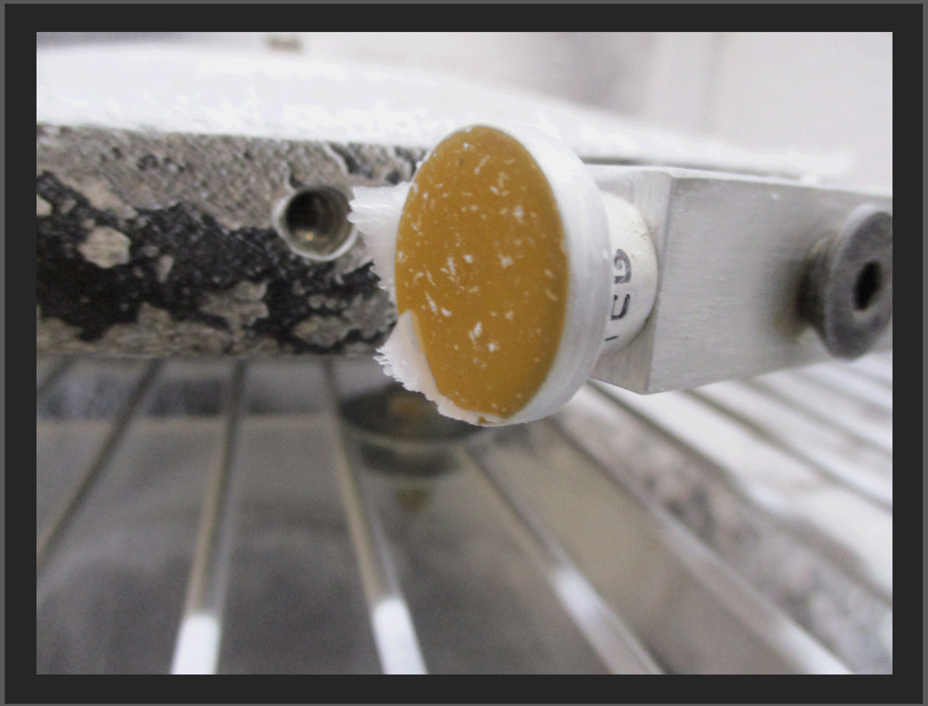
AERTS Jr.

- This is a miniaturized instrument derived from Pennsylvania State University's Adverse Environment Rotor Test Stand (AERTS) that allows a coating to be tested in a simulated in-flight icing environment where a traditional freezer cannot accurately mimic supercooled ice adhesion.[8]
- The coatings travel through an air circulated drum at approximately 5000 RPM at temperatures of -8, -12, and -16°C.[8]

AERTS Jr. Images

- Ice accumulates on the coating surface and after a period of time in the icing environment, the ice will fracture or “shed” from the coated surface. Once a shedding event occurs, the data (rpm, mass, area) is collected to determine ice adhesion strength .[8]

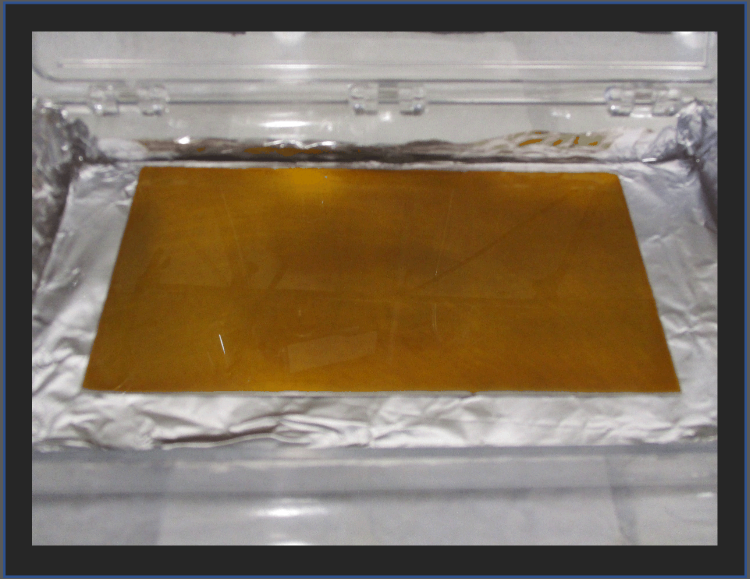




Solvent Soaking

- Coatings experience a period of both 1 Day and 7 Days soaking in 3 different aircraft fluids that are likely to come in contact with the coating once installed on an aircraft.

Ethylene Glycol

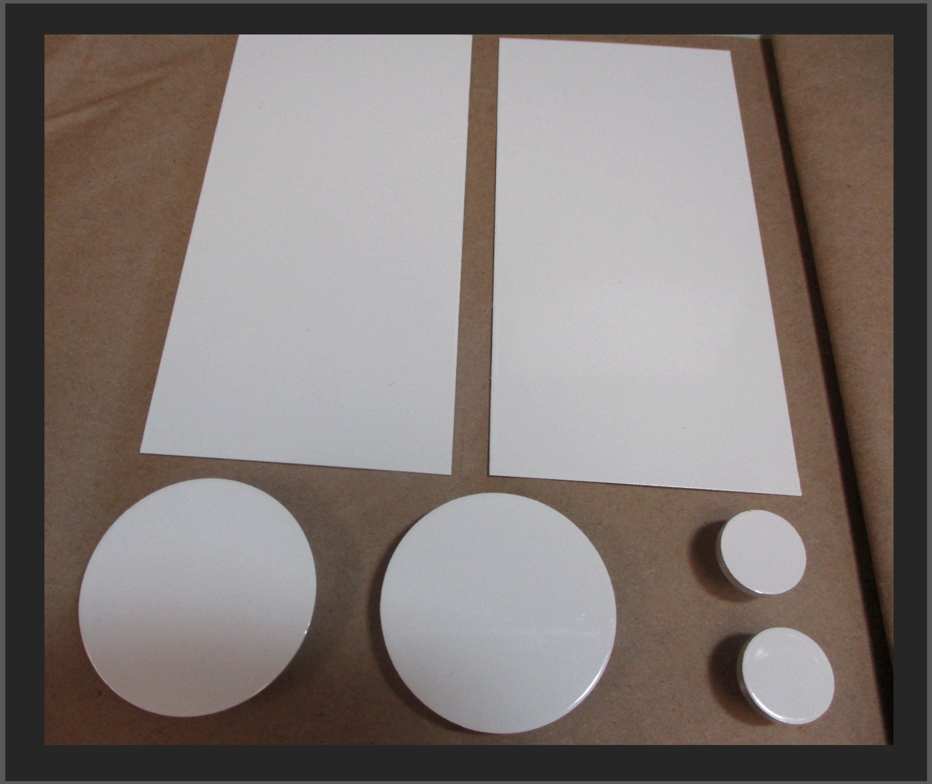


Skydrol™ Hydraulic Fluid

JP-5 Jet Fuel

Moving Forward

- The action plan for these testing procedures is to create Process Map Visuals (PMVs) for each testing instrument so that any coating can be tested in a standardized way that enables comparison accuracy.
- Through the screening procedures performed by this array of durability tests, coating formulas will be down-selected and advanced through testing simulations to include, insect accretion studies and ice-adhesion testing in Pennsylvania State's full scale AERTS chamber.



References

[1] ASTM D7334-08. (2013). Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement. ASTM International. doi: 10.1520/D7334-08R13

[2] ASTM D523-14. (2018). Standard Test Method for Specular Gloss. ASTM International. doi: 10.1520/D0523-14R18

[3] ASTM D4060-10. Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser. ASTM International. doi: 10.1520/D4060-10

[4] ASTM D3359-17. Standard Test Methods for Rating Adhesion by Tape Test. ASTM International. doi: 10.1520/D3359-17

[5] ASTM D2794-93. (2019). Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact). ASTM International. doi: 10.1520/D2794-93R19

[6] ASTM Standard D522-93a. Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings. ASTM International. doi: 10.1520/D0522-93A

[7] ASTM Standard D3363-05 e2. (2011). Standard Test Method for Film Hardness by Pencil Test. ASTM International. doi: 10.1520/D3363-05R11E02

[8] Smith, J. G., Jr., Wohl, C. J., Palacios, J. & Connelly, B. D. (2018). Design and Development of a Laboratory-scale Ice Adhesion Testing Device. NASA Langley Research Center, doi: 20180006287