CURING TRADITIONAL FORMULATIONS WITH MULTI-WAVELENGTH LEDS

RadTech 2018 (Chicago, IL) | 9 May 2018, B. Skinner

AGENDA

- **Overview**
  - Status of UV LED technology
  - Basis of current research
- **Experimental setup**
  - Clear coat chemistries
  - Irradiators- Irradiance profiles and spectra
  - Drawdown and physical tests to judge "cure"
- **Results**
  - Marginal success point (MSP) for each formulation- LED vs. µwave
  - Order of wavelengths, gloss or matte finish
- **Conclusions**
- **Questions**

OVERVIEW

- **UV Curing has been used for many diverse applications**
- **Until recently, Hg based UV lamps have primarily been utilized**
- **Key Advantages of Hg-based lamps:**
  - Mature technology
  - Fast process speed (higher UV output)
  - Low cost of ownership
- **Drawbacks**
  - High electrical power consumption
  - Requires large blowers
  - Ozone formation
  - Presence of Hg

OVERVIEW- STATUS OF UV LED TECHNOLOGY

- **White light LED manufacturing technology has made steady progress over the past decade**
- **Associated manufacturing technology for UV LEDs have benefited**
- **Some key benefits of LED technology are:**
  - Instant on-off
  - Hg free
  - Longer life
  - Possible lower cost of ownership
- **UV LEDs have shown significant potential for UV-curing applications**
- **Until now, mostly UVA LEDs have been used for some curing applications**
- **Recent emergence of UVC LEDs are expected to bring further innovation**

OVERVIEW- BASIS OF CURRENT RESEARCH

- Most existing formulations are optimized for broadband illumination (for Hg-based lamps)
- Reluctance by some formulators to reformulate for LED and by end-users to qualify a new potentially more expensive formulation
- **We will look at the feasibility of curing traditional formulations with multi-wavelength LED lamps**

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EXPERIMENTAL SETUP- CLEAR COAT CHEMISTRIES

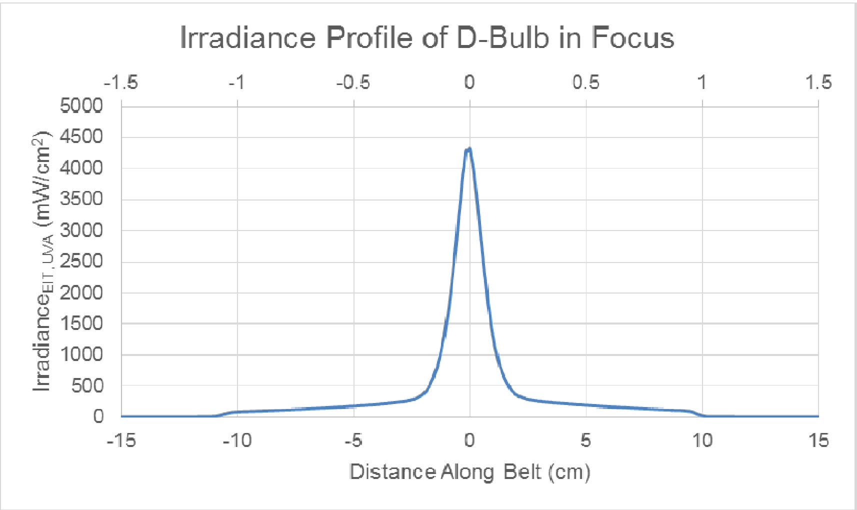
- **4 custom formulations with common commercial monomers, oligomers, and photoinitiators**

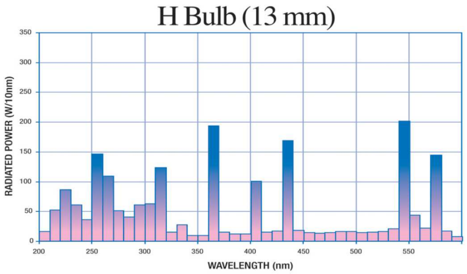
- **White Leneta paper and 14 microns DFT for all tests**

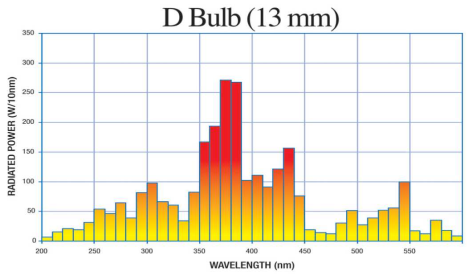
Formulations, Weight (%)					
Material		**1**	**2**	**3**	**3B**
EBECRYL 3700		40			
EBECRYL 5781				75	75
EBECRYL 221			45		
EBECRYL 85		45	40		
EBECRYL P115		10	10		
EBECRYL LED 02				20	20
PI:	TPO	2.5	2.5	2.5	
PI:	Irg 184	2.5	2.5	2.5	5
		100	100	100	100

EXPERIMENTAL SETUP- IRRADIATORS I

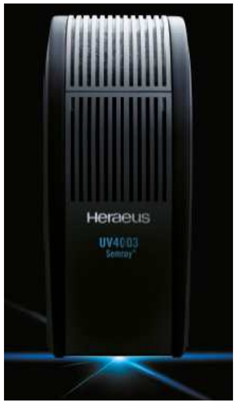
- **Heraeus F300, Microwave-Powered Irradiator, 300W/in, 6-inch bulb**
  - Broadband, H and D-bulb
  - In focus (2.1in on 53mm), standard bulb position, R500 reflector, perpendicular





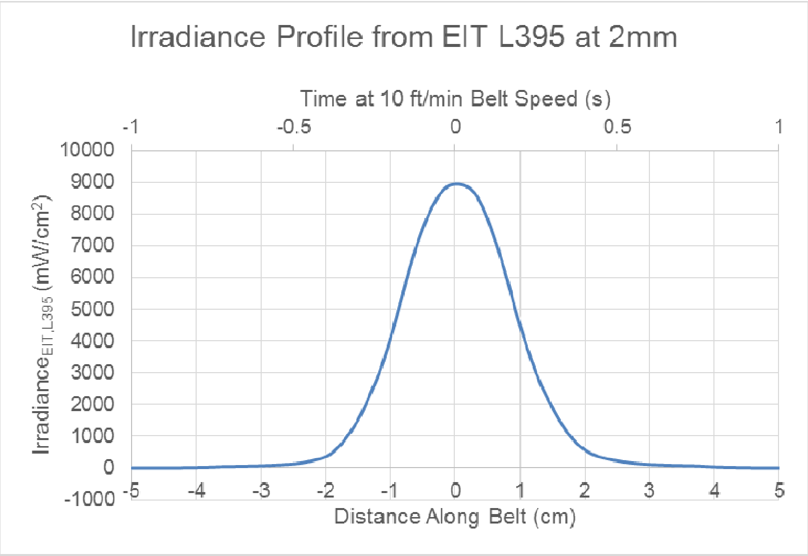


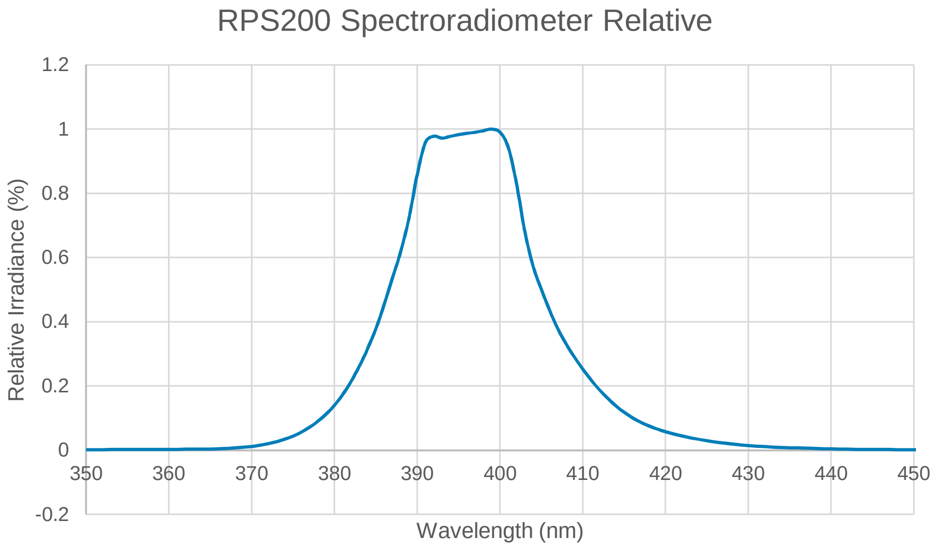
EXPERIMENTAL SETUP- IRRADIATORS II



Semray 395nm Lamp System

- 14 W/cm^2 at the glass with NobleProbe
- 8900 mW/cm^2 at 2mm with EIT LEDCURE L395
- 1830 mJ/cm^2 at 20 ft/min





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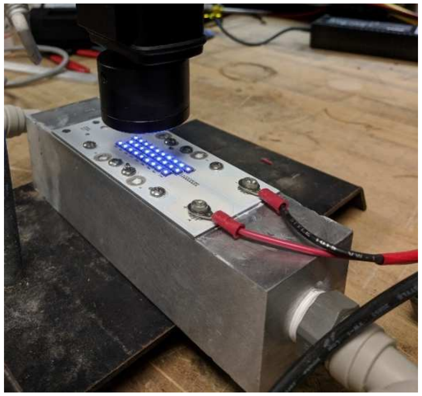
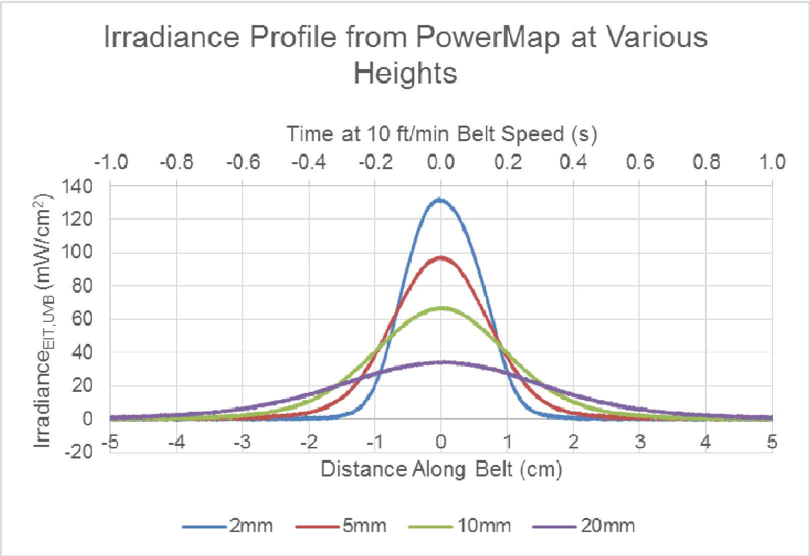
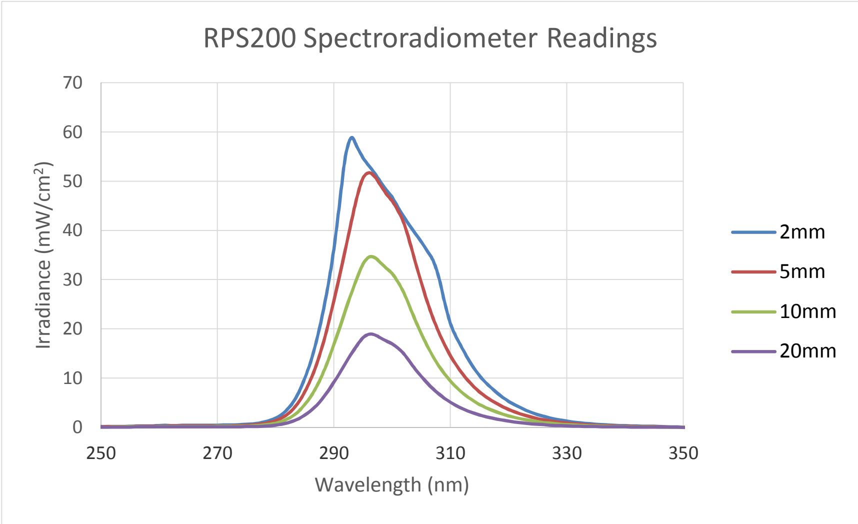
EXPERIMENTAL SETUP- IRRADIATORS III



Custom-built LED array

- **305nm, 30mW chips**
- **130 mW/cm^2 Peak Irradiance UVBEIT at 2mm**
- **36 mJ/cm^2 at 10 ft/min, 2mm**
- **6V, 9.6A**

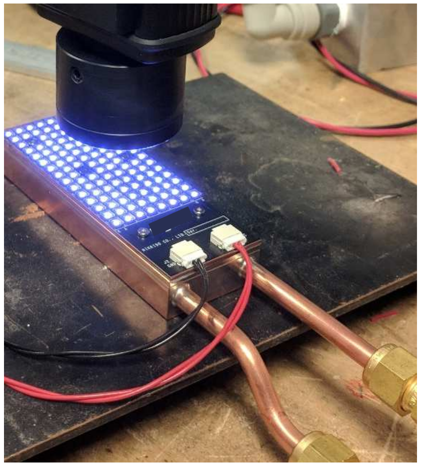


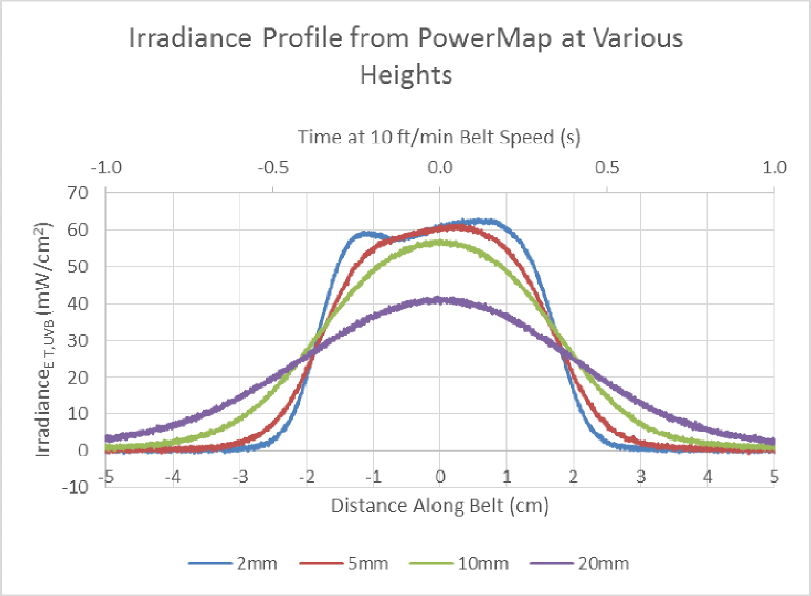


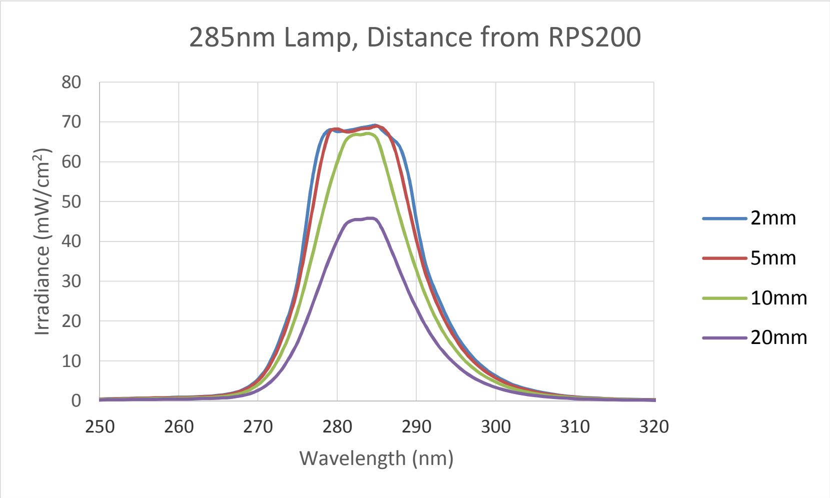
EXPERIMENTAL SETUP- IRRADIATORS IV

285nm Lamp specified at 80mW/cm²

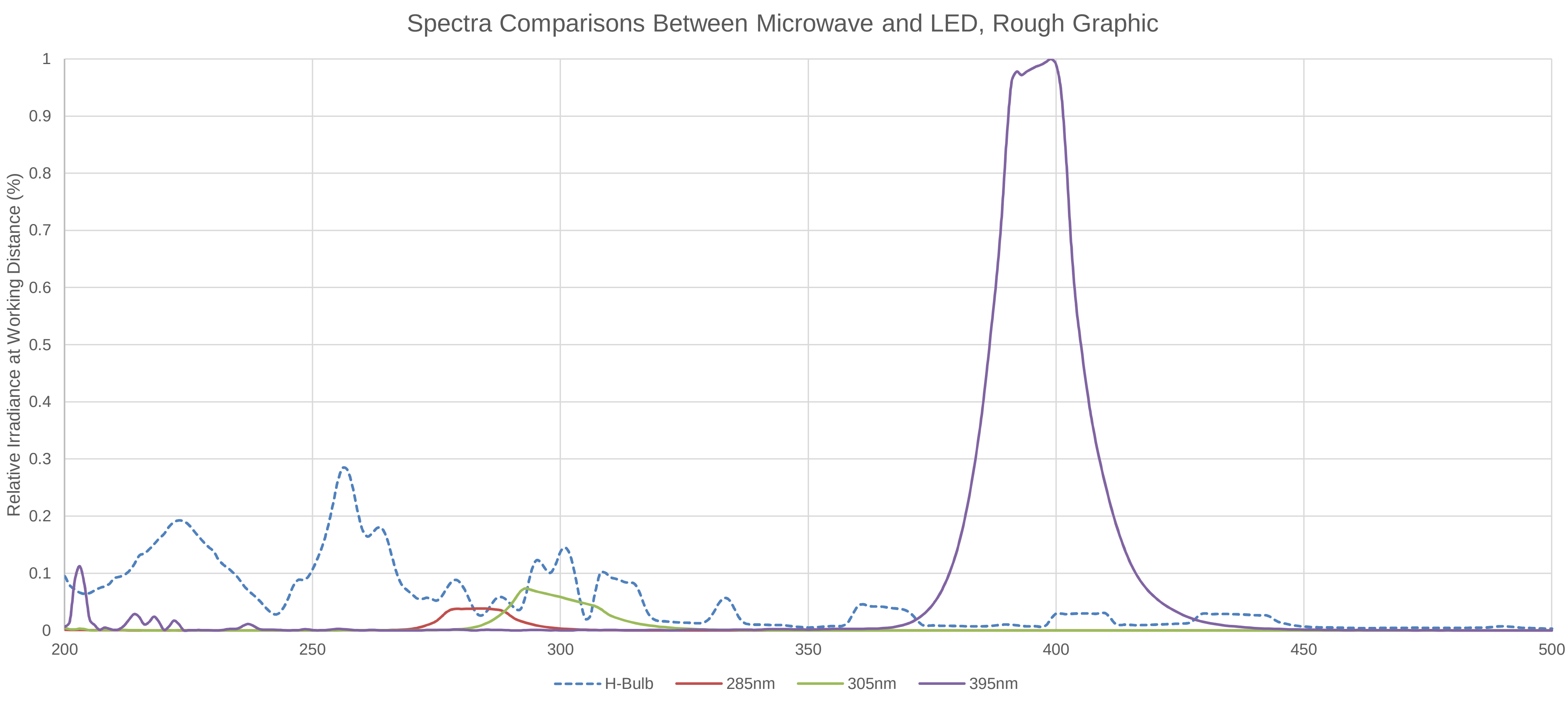
- **285nm**
- **70 mW/cm² at 2mm (bumped up 10% from EIT measurement)**
- **45 mJ/cm² at 10ft/min, 2mm**
- **48V, 3A**



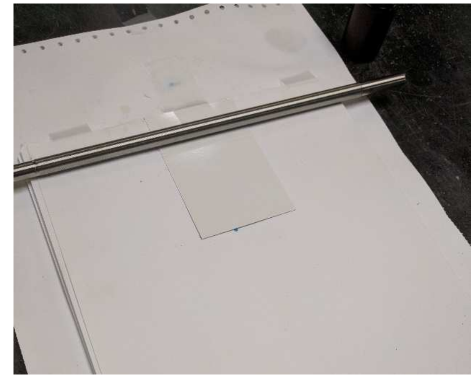




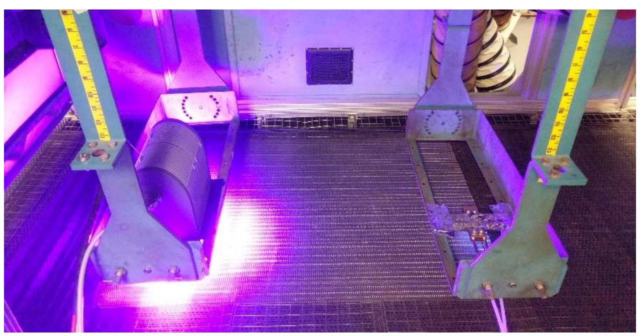
EXPERIMENTAL SETUP- IRRADIATORS V



DRAWDOWN AND PHYSICAL TESTS TO JUDGE “CURE”



- **Drawdown rod to apply 14 micron film to white Leneta cards**
- **Find Marginal Success Point (MSP)- lowest exposure where all physical tests pass**
  - Thumb twist to test “bulk cure” or “through cure”
  - Scratch test for scratch resistance with tongue depressor
  - Planned to use tack but all coatings felt tacky even when cured
- **No FTIR**



RESULTS- FORMULATION 1 MSP

- **395/285 was able to cure the coating with significantly more UVA energy but comparable UVB to µwave**
- **Under 395/305, coating failed ‘through cure’ and scratch test at slowest speeds allowable**
  - Slower speeds under 395nm Semray burned and damaged the coating, and 3 ft/min was the conveyor’s lower limit
- **395/285 could cure the coating even at lower UVB peak irradiance and exposure than 395/305**

MSP, Formulation 1

Exposure	Belt Speed (ft/min)	Exposure (mJ/cm^2)				Peak Irradiance (mW/cm^2)			
		UVC	UVB	UVA	UVV	UVC	UVB	UVA	UVV
F300-H	260	7	36	32	45	438	2103	1901	2542
F300-D	160	8	48	127	144	293	1665	4325	4817
395, 305	10, 3		120	3660			132	8940	
395, 285	10, 10		46	3660			69	8940	

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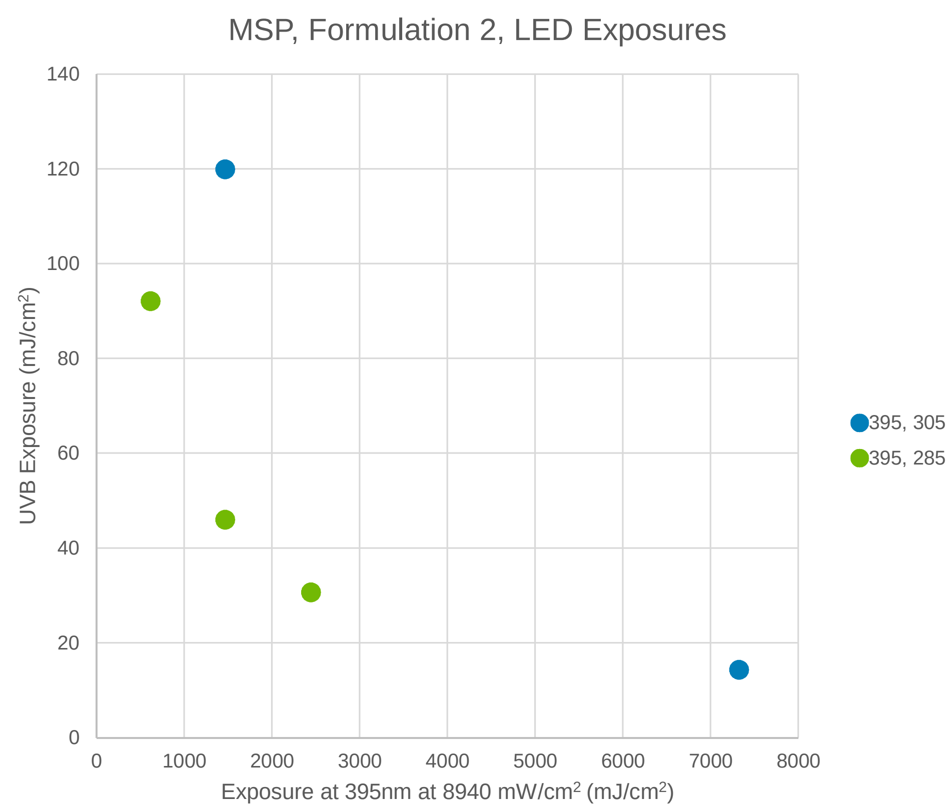
RESULTS, FORMULATION 2 MSP

- **Both LED exposures achieved cure at different levels of UVB and UVA exposure**
- **Lower exposure possible with 305nm, but for this much more 395nm energy is needed**
- **The coating is less sensitive to changes in exposure with 285nm compared to 305nm**
- **Flexibility in equipment selection**

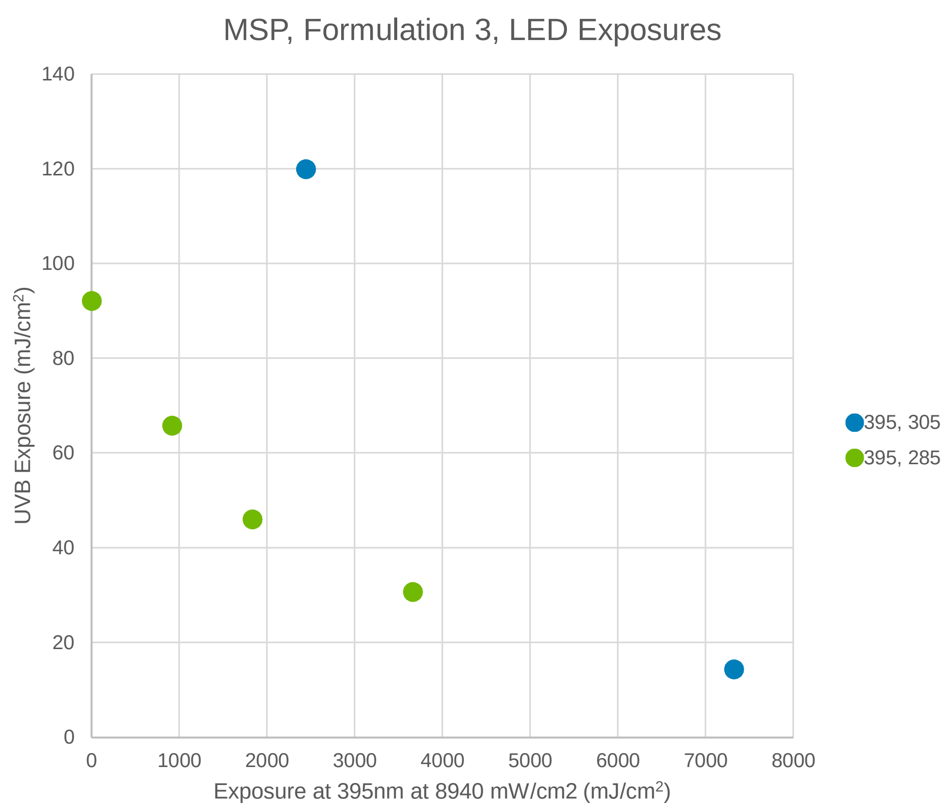
MSP, Formulation 2, Microwave-Powered Lamp (300W/in)									
Exposure	Belt Speed (ft/min)	Exposure (mJ/cm^2)				Peak Irradiance (mW/cm^2)			
		UVC	UVB	UVA	UVV	UVC	UVB	UVA	UVV
F300-H	260	7	36	32	45	438	2103	1901	2542
F300-D	220	6	35	92	105	293	1665	4325	4817



RESULTS- FORMULATION 3 MSP

- **Both LED exposures achieved cure at different levels of UVB and UVA exposure**
- **Lower exposure possible with 305nm, but for this much more 395nm energy is needed**
- **The coating is less sensitive to changes in exposure with 285nm compared to 305nm**
- **Flexibility in equipment selection**
- **Cured with only 285nm but not with 395nm only**

MSP, Formulation 3, Microwave-Powered Lamp (300W/in)									
Exposure	Belt Speed (ft/min)	Exposure (mJ/cm^2)				Peak Irradiance (mW/cm^2)			
		UVC	UVB	UVA	UVV	UVC	UVB	UVA	UVV
F300-H	240	8	39	35	49	438	2103	1901	2542
F300-D	200	6	38	101	115	293	1665	4325	4817



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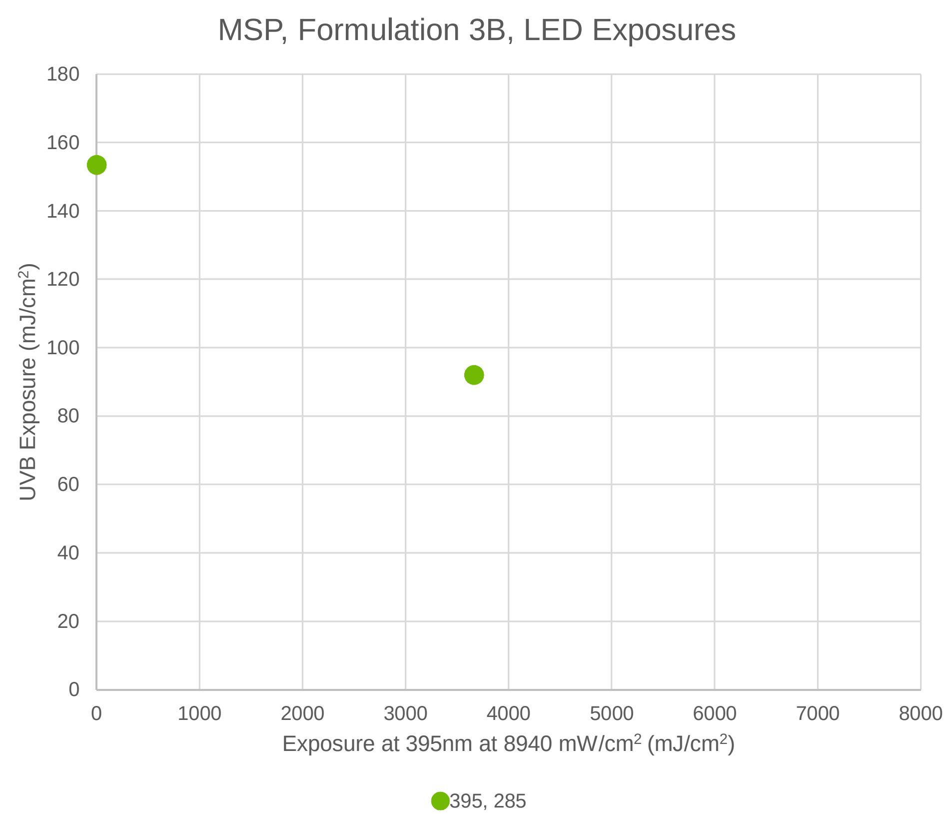
RESULTS, FORMULATION 3B (WITHOUT TPO)

- **Under 395/305, coating failed 'through cure' and scratch test at slowest speeds allowable**

- **Coating could cure with just 285nm with enough energy**
  - Adding UVA reduced the required UVB energy by 40%

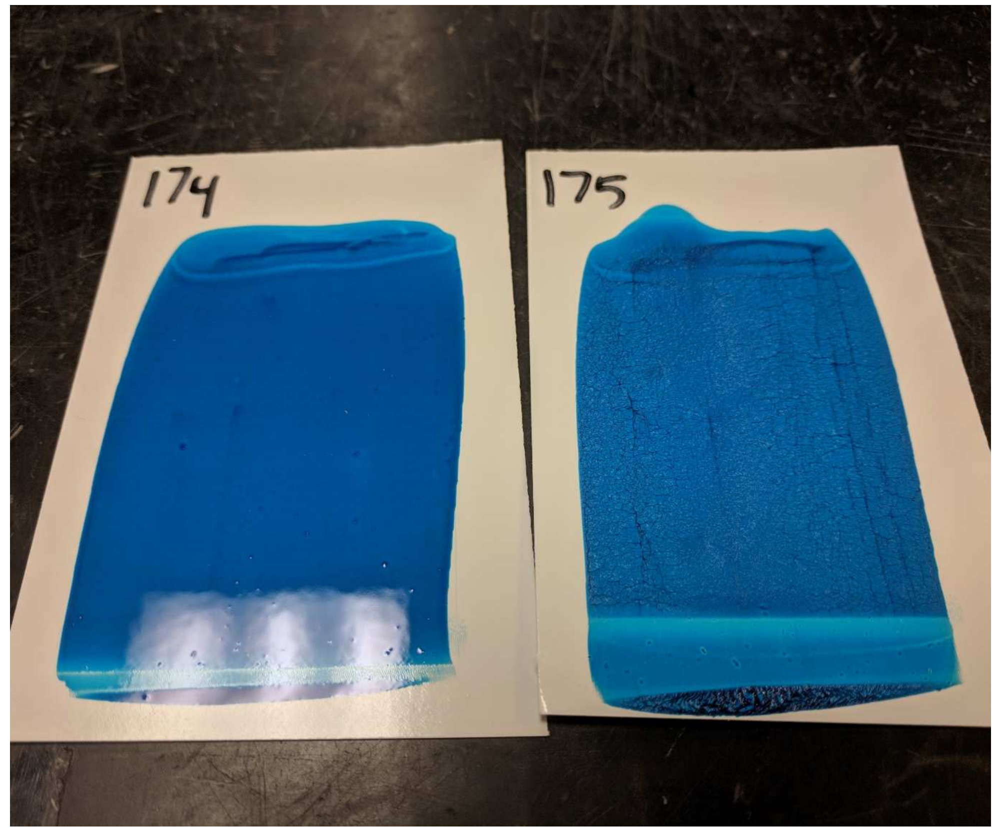
- **50-60% more energy required under microwave lamp to cure vs. coating with TPO**

MSP, Formulation 3B									
Exposure	**Belt Speed (ft/min)**	**Exposure (mJ/cm^2)**				**Peak Irradiance (mW/cm^2)**			
		UVC	**UVB**	**UVA**	**UVV**	**UVC**	**UVB**	**UVA**	**UVV**
F300-H	160	12	59	53	73	438	2103	1901	2542
F300-D	125	10	61	162	184	293	1665	4325	4817
395, 305	5, 3		120	7320			132	8940	
395, 285	10, 5		92	3660			69	8940	



RESULTS- ORDER OF LED WAVELENGTHS

- **Did not affect clear coats**
  - 60° gloss under both conditions was 94.6 GU
- **Affected cyan inkjet ink**
  - Short wavelength first for matte (13.4 GU).
    - Physically rough surface
  - Long wavelength first for glossy (94.0 GU)
- **285nm and 395nm used here at 15 ft/min**



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CONCLUSIONS I

- **In some situations, lower UVB wavelengths, even at lower peak irradiances and exposures, are more effective**
- **For LED, when decreasing exposure, scratch resistance fails before bulk cure fails; coating is still cured but not scratch resistant.**
- **For formulations 2 and 3, cure achieved at multiple levels of UVB and UVA exposure**
  - Lower exposure possible with 395/305, but much more 395nm energy was needed
  - Less sensitive to changes in exposure with 285nm compared to 305nm

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CONCLUSIONS II

- **Formulation 3B (without TPO)- 285nm alone could cure it**
  - Adding UVA reduced required UVB energy by 40%
  - 50-60% more energy required for microwave lamp exposure compared to Formulation 3 (with TPO)
- **Short wavelength first had no effect on gloss for all clear coats, but did impact inkjet ink**
  - Long wavelength first reflected 7x more light at 60° angle than short wavelength first
- **Time between exposures is critical**
  - Sample at MSP with 10s between exposures would fail bulk cure and scratch test when time extended to 60s
  - Increasing both LED exposures by 20% enough to regain passing physical tests

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CONCLUSIONS III

- **305nm and 285nm exposures had roughly same hardness (qualitative)**
- **A little UVC/UVB goes a long way (20-50 mJ/cm^2) at 70-130 mW/cm^2 is effective)**
- **Much more UVA is required with multi-wavelength LED approach**
  - 4-100x depending on UVB exposure
- **Inadequate UVA resulted in loss of adhesion and coating to flake off during scratch test**

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