Dymax 431 FTIR photocure analysis

Emile Verstegen
Material analysis
September 25th 2018

innovation + you

PHILIPS

Contents

- DoE
- Experimental
- Data processing
- Results
  - Current status (sample cured at Adimec, both Hg and 365 nm LED source0
  - Curing in air
  - Comparison air with oxygen shielded cure (only lowest exposure intensity condition)
  - Post curing at higher temperature (only for highest exposure intensity condition)
- Conclusions

“DoE”

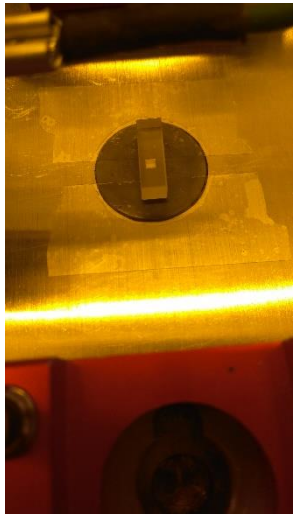
- Sensitivity to exposure intensity: 50, 100, 200 mW/cm^2 chosen (exposure in air)
- Sensitivity to exposure (UV) wavelength: Broadband Mercury source, 365 and 405 nm LED
- Sensitivity to environment, only for lowest intensities: Nitrogen shield applied
- Can the final cure be reached at room temperature: Only for the highest intensities post-cure step applied by heating the UV-cured glue to 140 °C*
  During the temperature step the sample is not exposed to UV

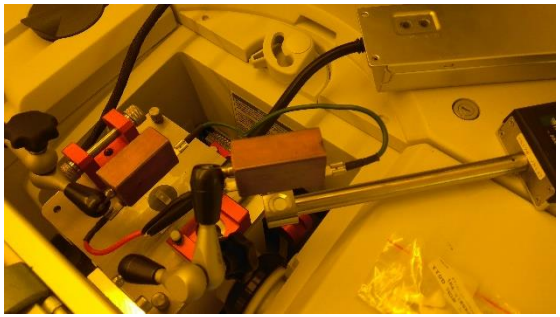
* T_g of cured Dymax 431 = 115 °C (from specification sheet)

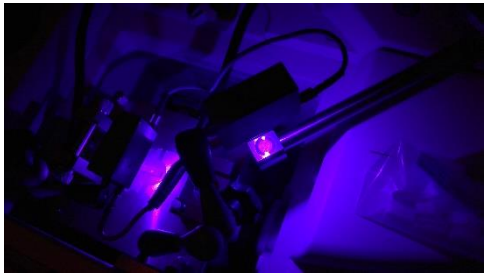
Experimental

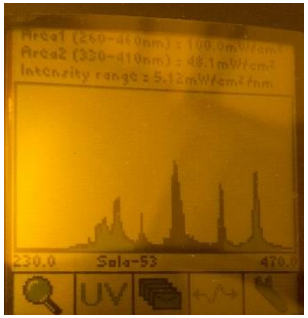
Tools

- Thermo iS50 FTIR with heatable Golden Gate ATR module
- Glue applied on module, with layer thickness of 50 µm (tape controlled)
- Exposure performed with supplied sources: Hg-source, 365nm + 405nm LEDs
- Exposure intensity monitored with spectroradiometer (Solatel Sola-scope 2000)
  The current was adjusted such as to allow for an intensity of 50, 100 and 200 mW/cm^2

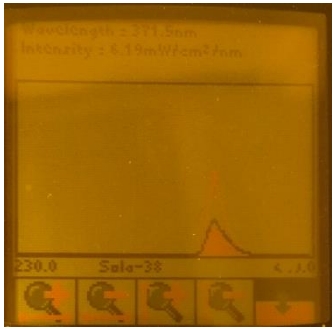




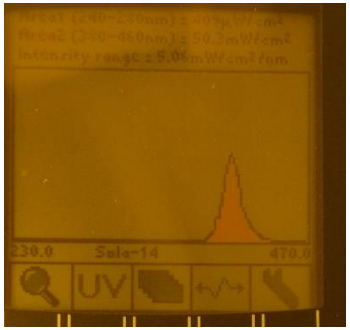




Hg-source



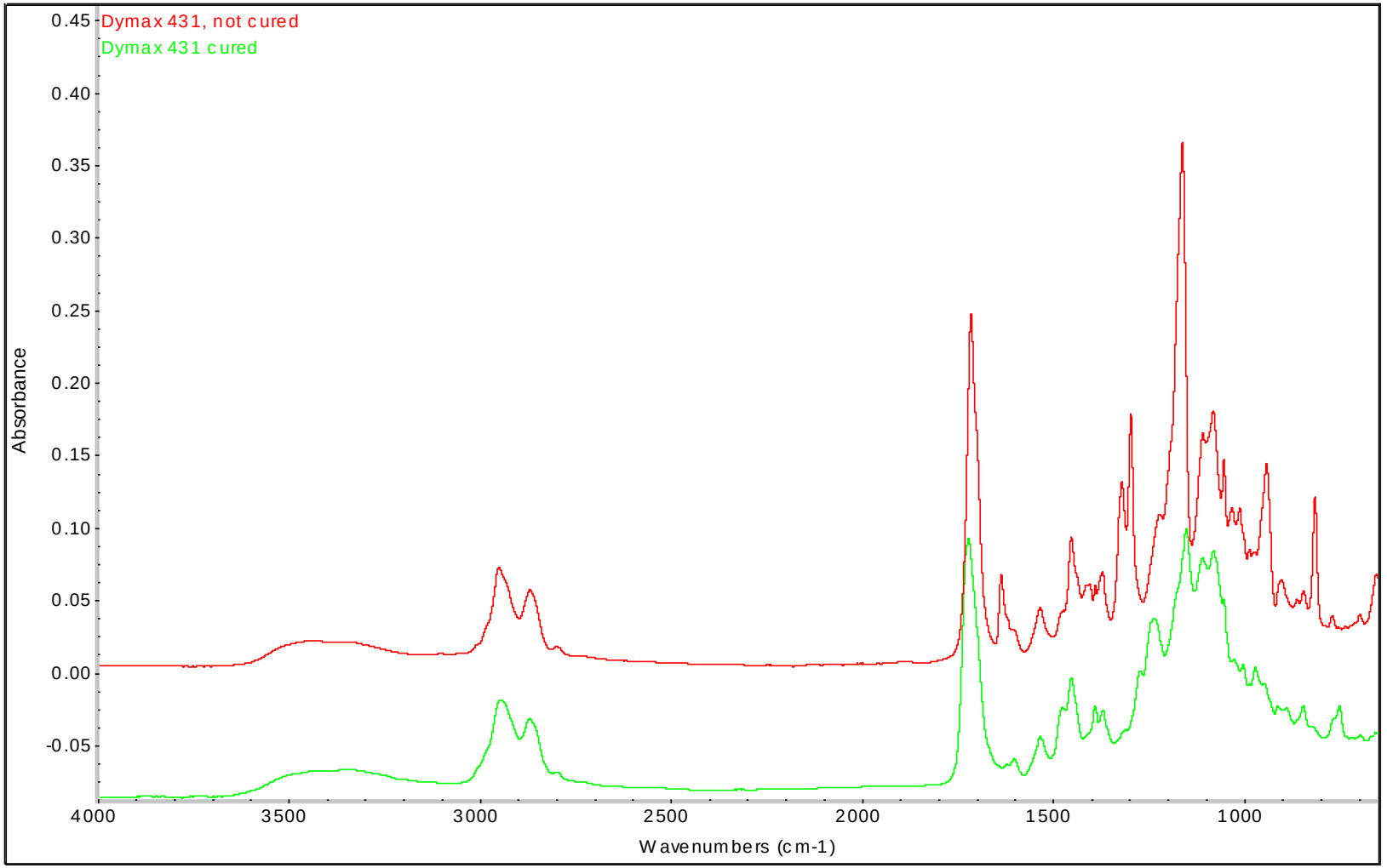
365 nm LED



404 nm LED

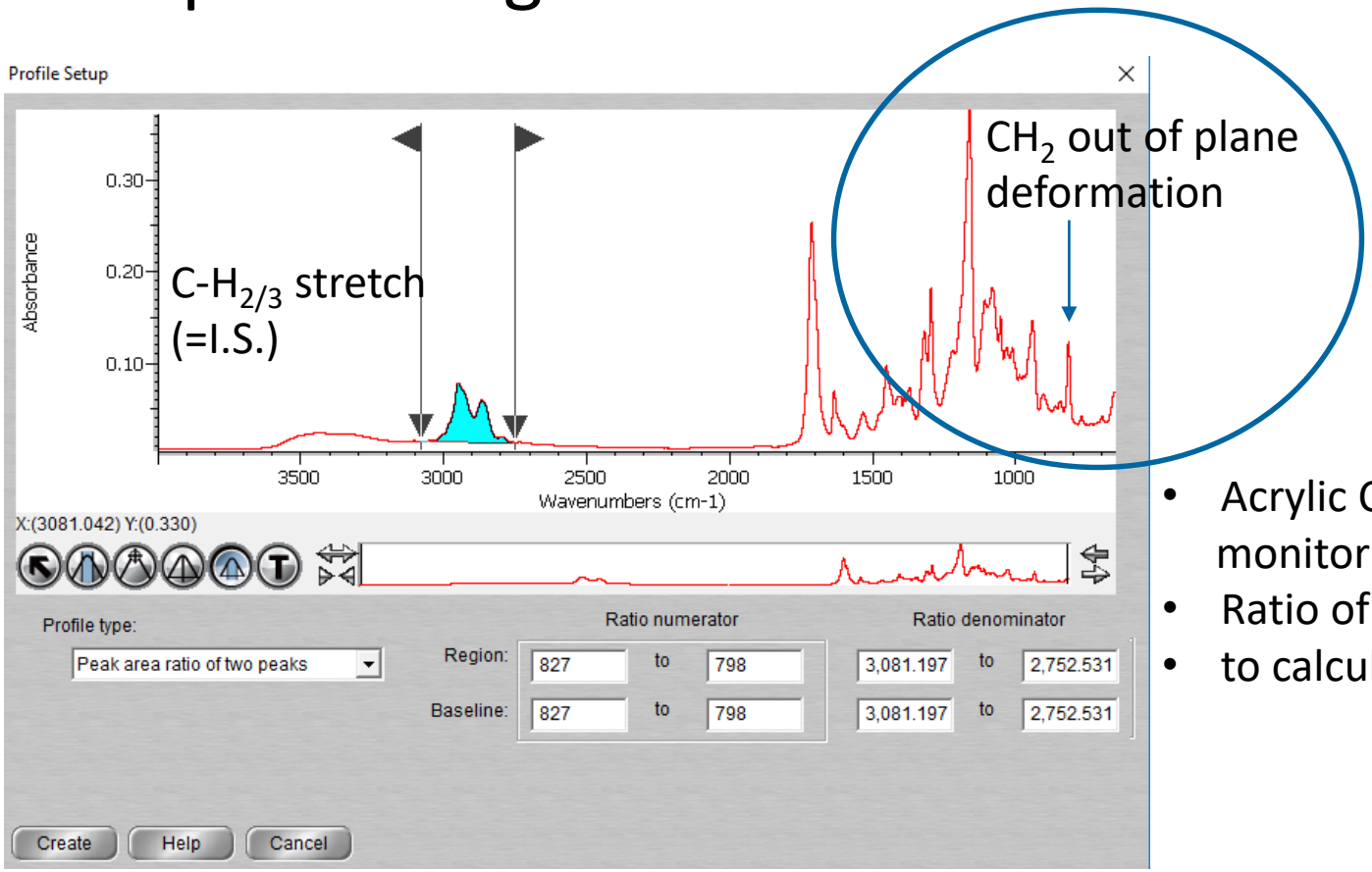
Spectral example, before and after curing

- FTIR spectra recorded of Dymax 431 in liquid state vs cured



PHILIPS

Data processing



- Acrylic C=C group is used to monitor the degree of curing
- Ratio of C=C to I.S. is used
- to calculate the cure degree

PHILIPS

Results Current cure state

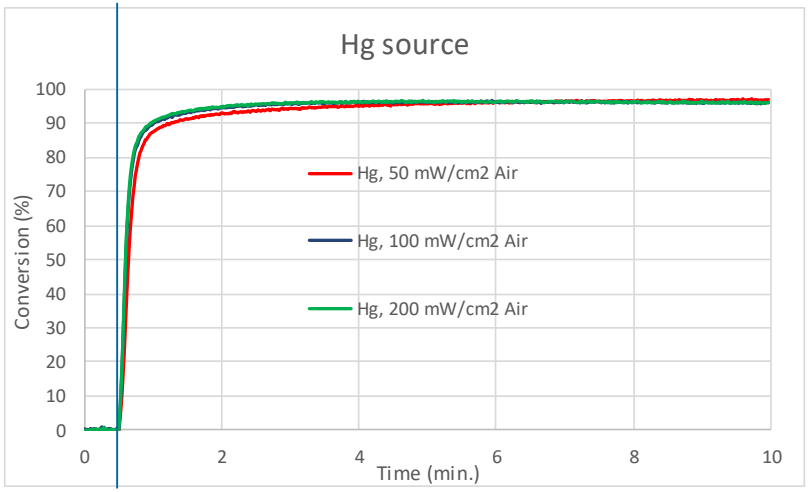
- Glass block was removed (with force) from metal part
- In all cases glue released from metal interface
- FTIR spectra were recorded both in the middle of the sample and close to the edge

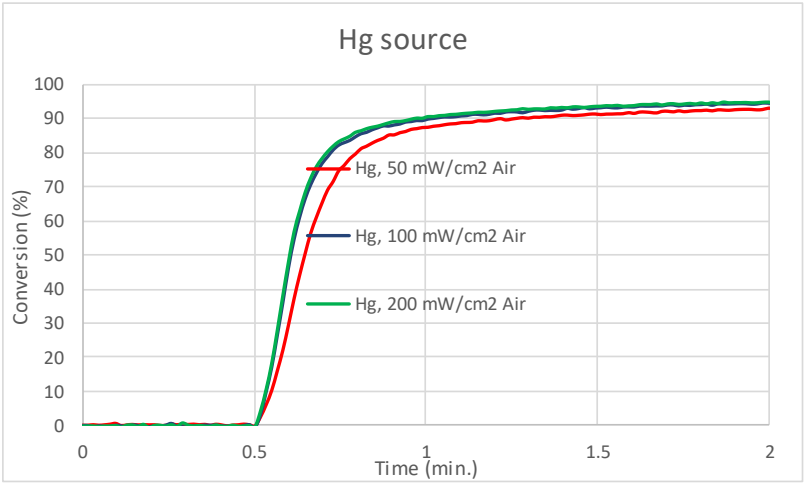
	Conversion (%)
2 min. Hg, center	97.1
2 min. Hg, edge	96.9
1 min. 365 LED, center	96.6
1 min. 365 LED, edge	95.7

Note: Corresponding FTIR spectra are shown at the end of the presentation

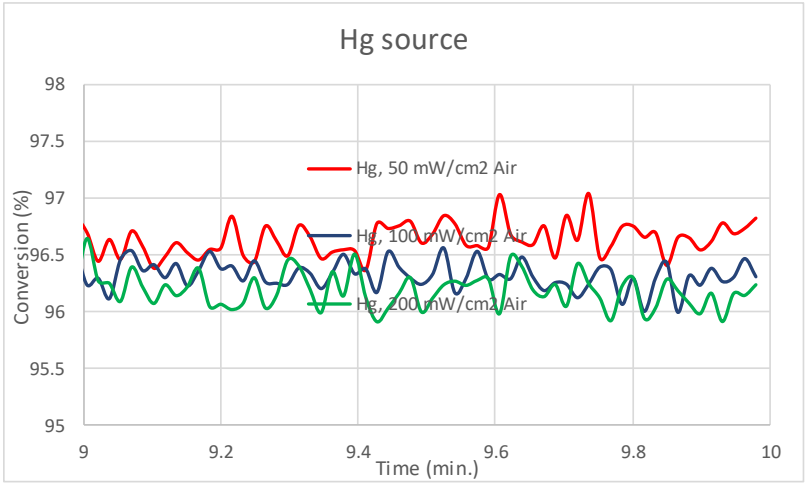
Cure monitoring Curing in air

Hg-source





Exposure was started at t=0.5 minutes

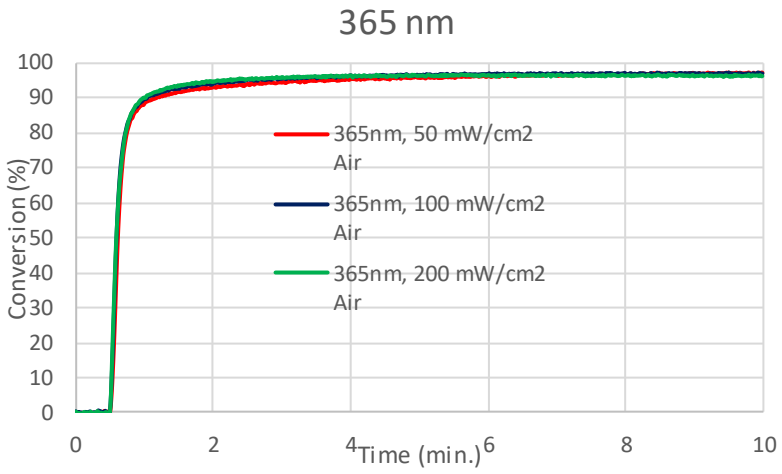


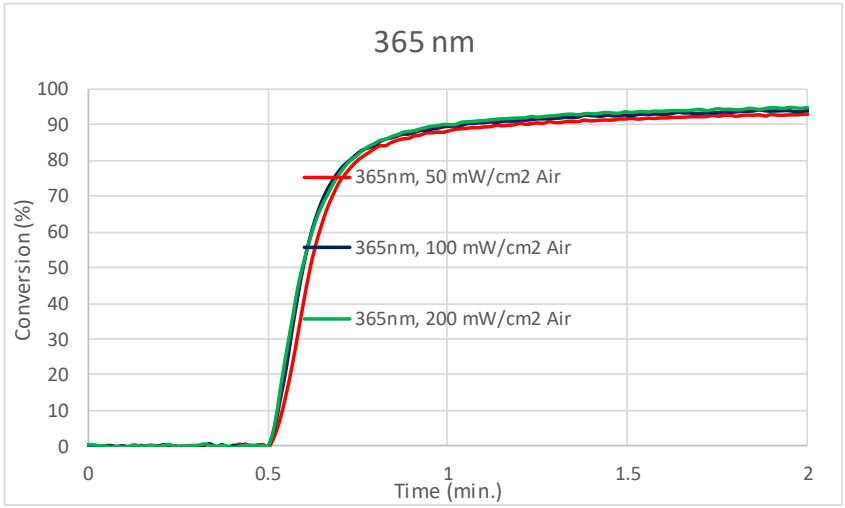
- From 50 to 100mW /cm^2 a small difference is observed in cure rate
- After 1 minute of exposure the cure extent exceeds 90%
- Final conversion is over 95%

PHILIPS

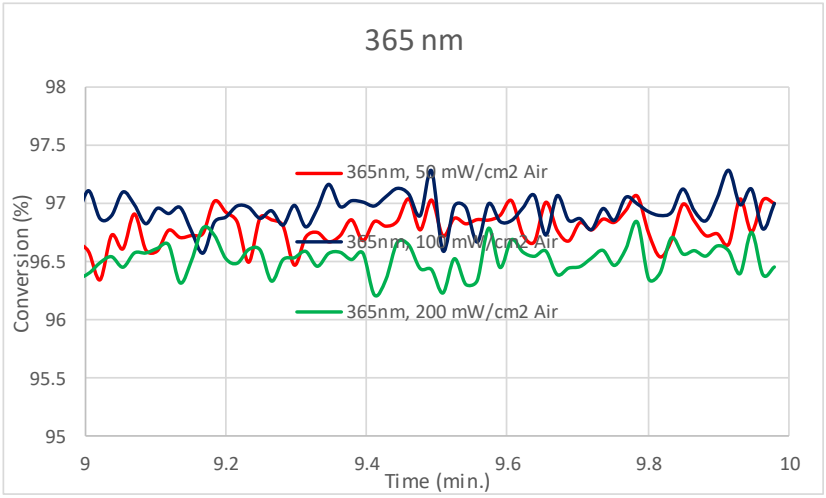
Curing in air

365 nm LED source





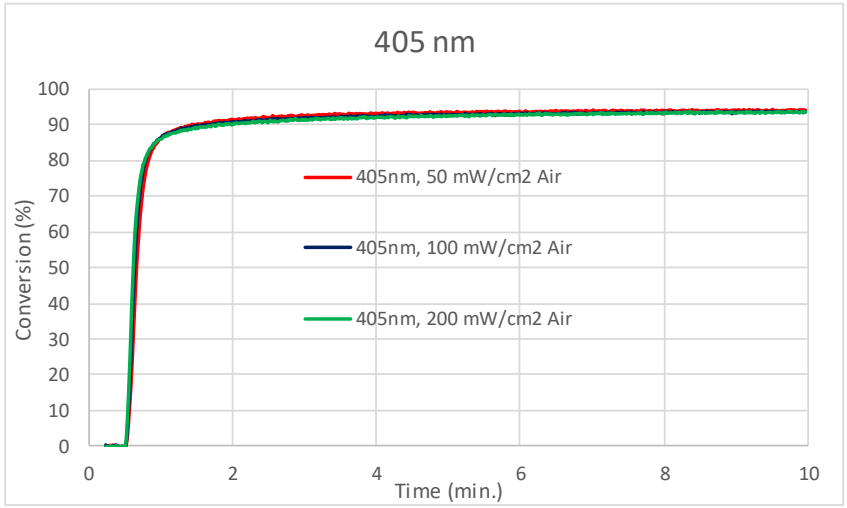
- From 50 to 100mW /cm^2 a small difference is observed in cure rate
- After 1 minute of exposure the cure extent exceeds 90%
- Final conversion is over 95%

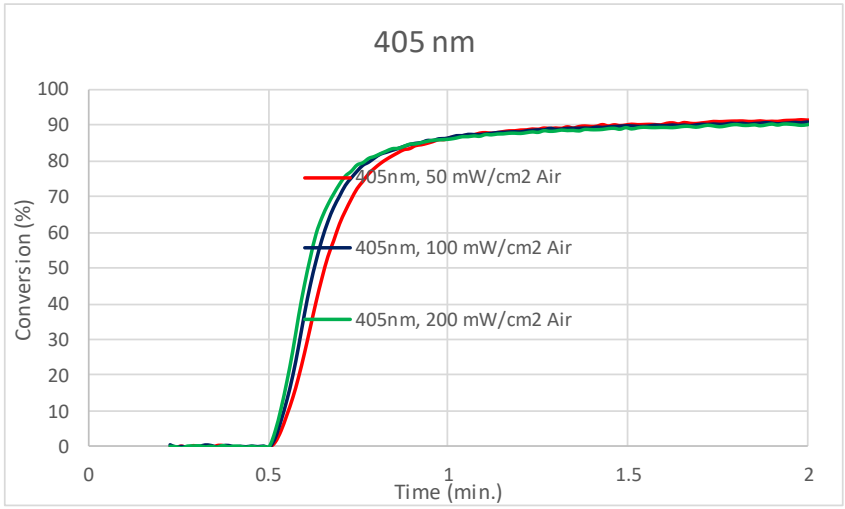


PHILIPS

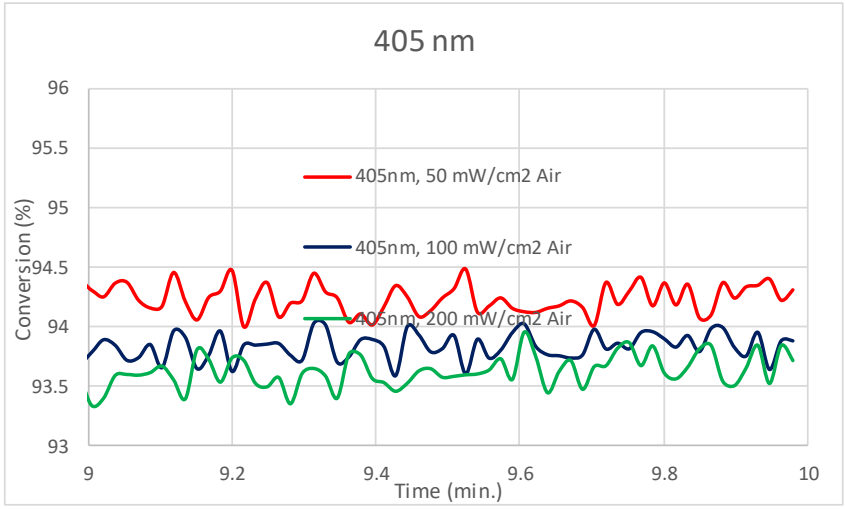
Curing in air

405 nm LED source



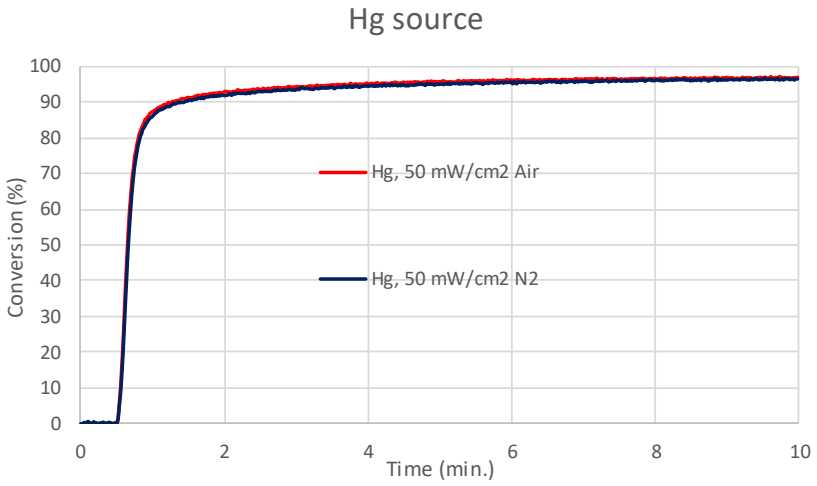


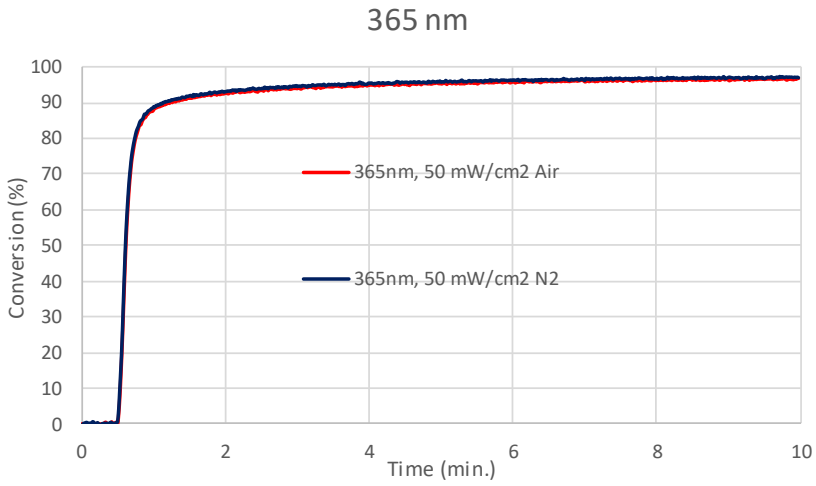
- From 50 to 200mW /cm^2 small differences are observed in cure rate
- Cure rate is lower than for Hg-source and 365nm LED
- After 1 minute of exposure the cure extent has just reached 90%
- Final conversion is just below 95%

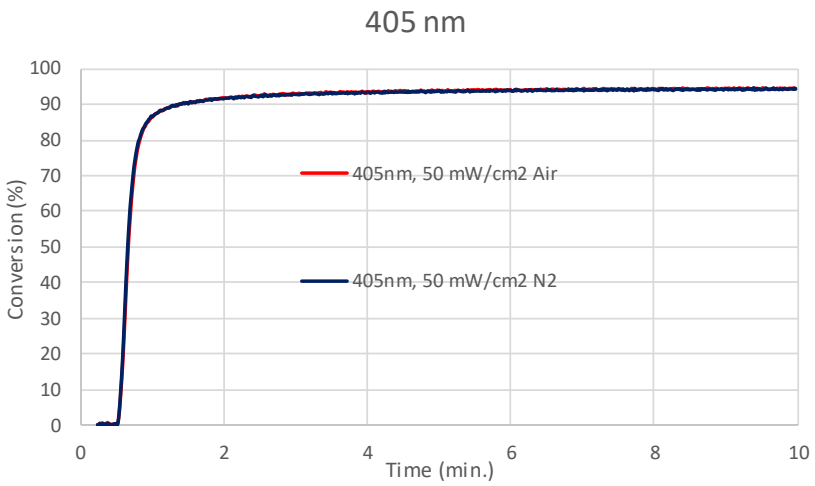


PHILIPS

Comparison without/with oxygen shield



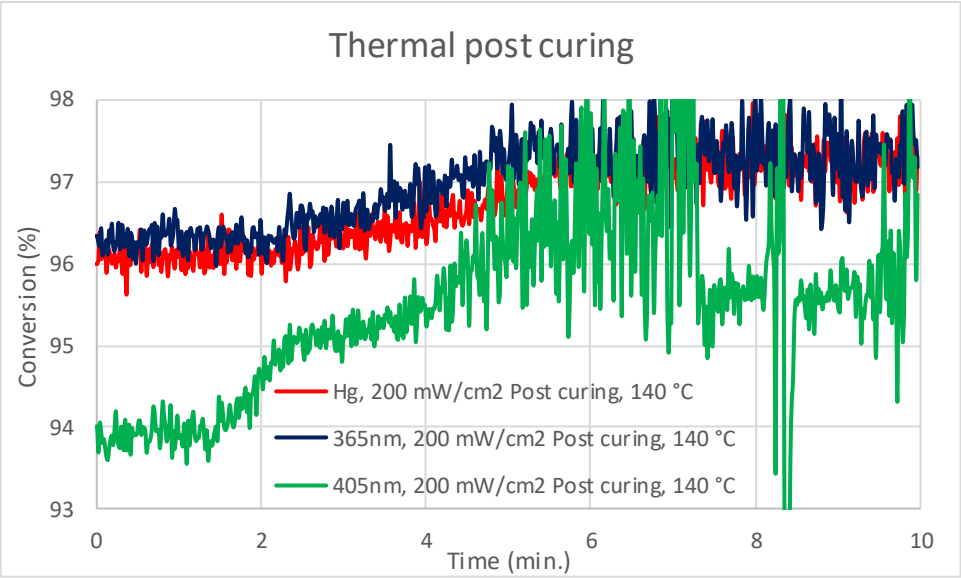




- No clear differences observed
  Glass block provides sufficient shielding to prevent oxygen inhibition

Post curing at higher temperature

- Curing was performed with 200 mW / cm^2
- Next the temperature was raised to 140 °C



- Initial conversion was already quite high, though still a small influence is observed due to further curing

Conclusions

Samples cured at Adimec

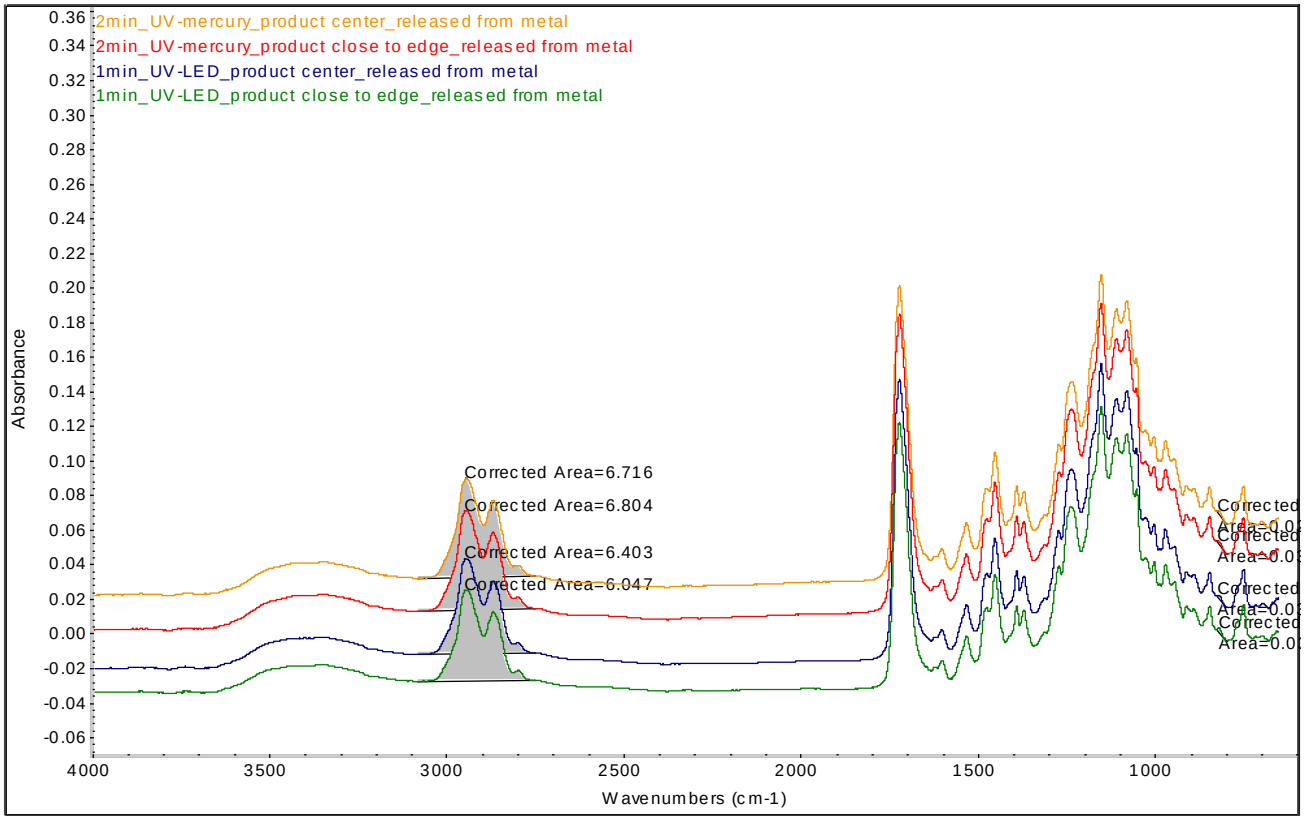
- Dymax 431 glue sample shows conversion over 95%
- 2 minute Hg source cured sample shows ~1% higher degree of curing
- Difference between center and edge <1% , indicative of very homogeneous curing, and limited oxygen inhibition

Cure monitoring

- All sources show fast curing, with a high curing degree. The 405nm diode shows the lowest cure degree (close to 95%). Hg and 365 nm LED sources show cure degrees > 95%
- The transition from 50 to 100 mW/cm^2 shows the largest increase of the cure rate
- The use of oxygen masking does not contribute much to the cure extent, probably because most of the oxygen is already masked by the glass block
- As room temperature curing already offers a large cure extent, thermal post curing does not offer a large increase (~1 - 1.5%)


PHILIPS

Spectral overview individual spectra



PHILIPS