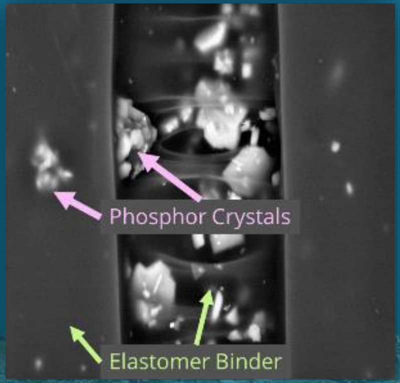
Flexible Polymer-based Luminescent Ink



Development and Characterization for Surface Temperature Measurements

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Overview

- This flexible polymer-based luminescent ink will allow for simultaneous measurements of temperature and strain in thermal–mechanical environments through combining phosphor thermometry and digital image correlation (TP + DIC).
- Phosphor crystals suspended in a Sylgard 184 binder produce visible and thermographic luminescence.
- Flexible polymer-based luminescent ink was found to *deform conformally with a metal substrate under tensile loading*, producing accurate strain measurements.[1]

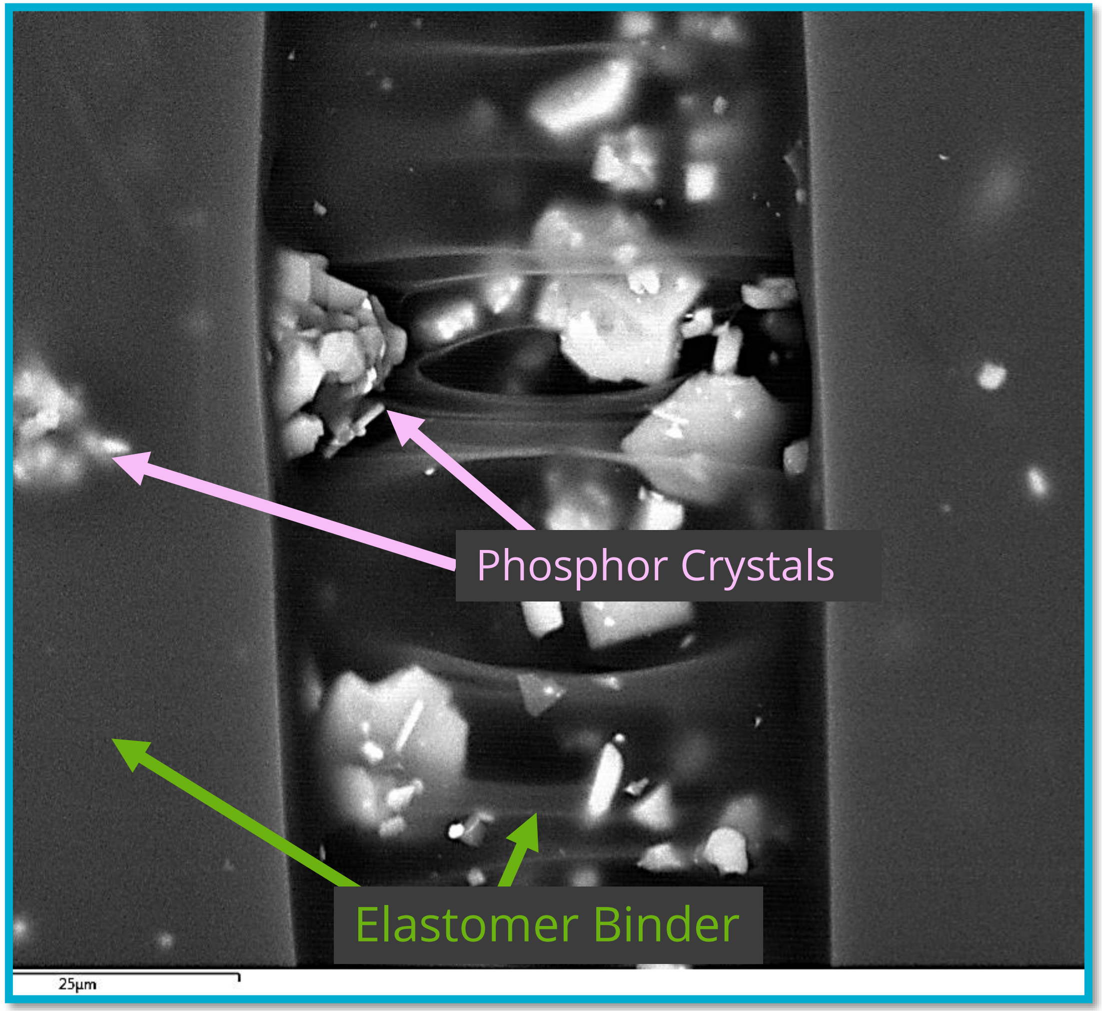


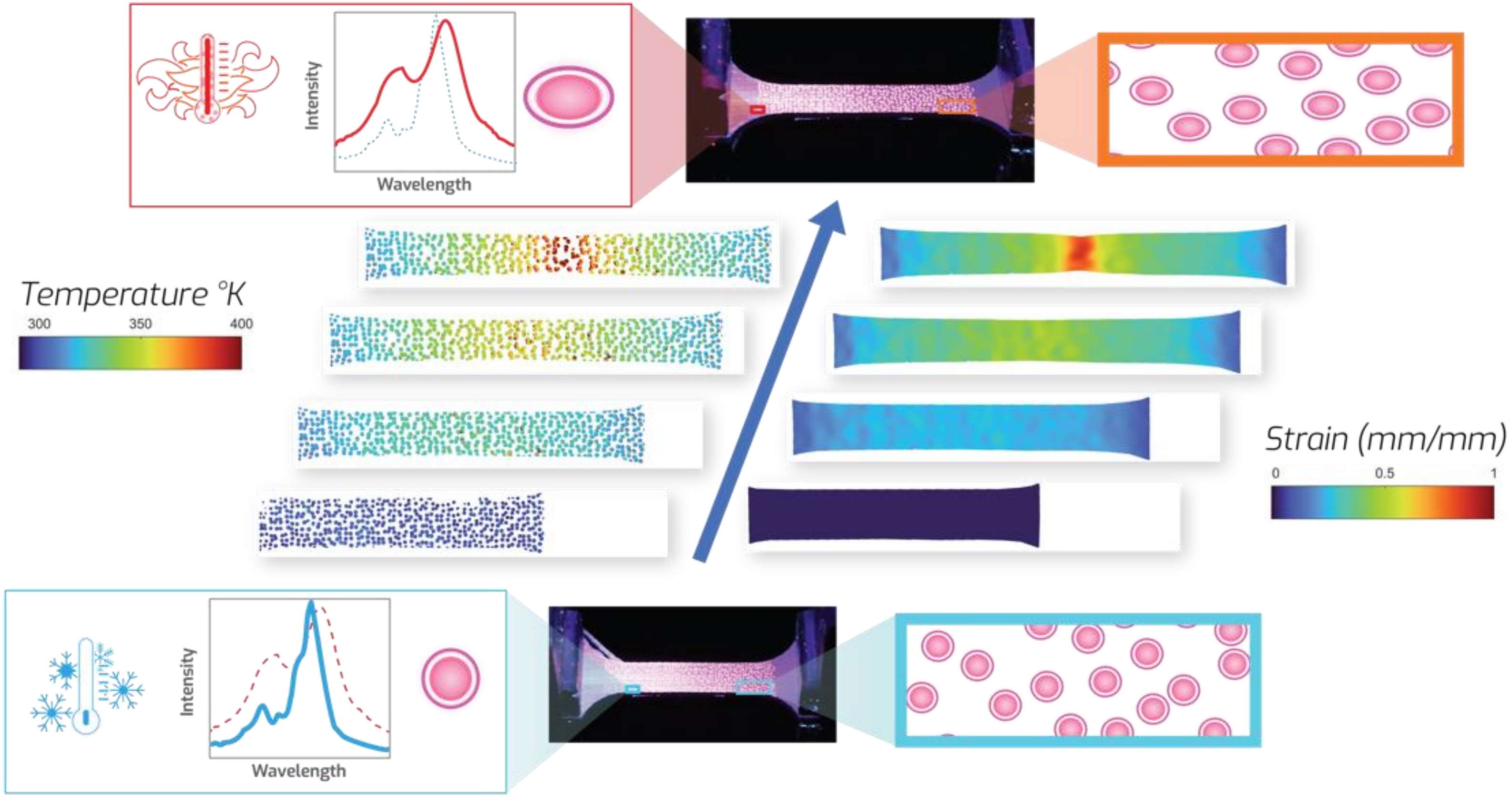
Figure 1. Secondary electron (SEM) images of mechanical deformation of the flexible ink over a steel substrate.

(1)Hansen, L. E.; Fitzgerald, K. M.; Huertas, N. A.; Jones, E. M. C.; Ruggles, T. J.; Gilliland, W. G.; Jauregui, L. J.; Murray, S. E.; Westphal, E. R.; Winters, C. A Flexible Polymer-Based Luminescent Ink for Combined Thermographic Phosphors and Digital Image Correlation (TP+DIC). *Optical Materials* **2023**, *141*, 113906. https://doi.org/10.1016/j.optmat.2023.113906.

Thermophosphor Digital Image Correlation (TP+DIC)

TP+DIC provides time-resolved thermometry & strain surface maps of hardware during accident testing. It is a synergistic combination & elevation of two optical diagnostics: phosphor two-color ratio thermography and DIC strain.

Figure 2. TP+DIC provides full-field visualizations of hardware response ('dogbone' specimen) during tension



- Phosphors are robust, chemically inert, inorganic ceramics that can sense temperature changes ranging from cryogenic (approximately -260°C) to reacting (approximately 1000°C), dependent on composition.

Motivation

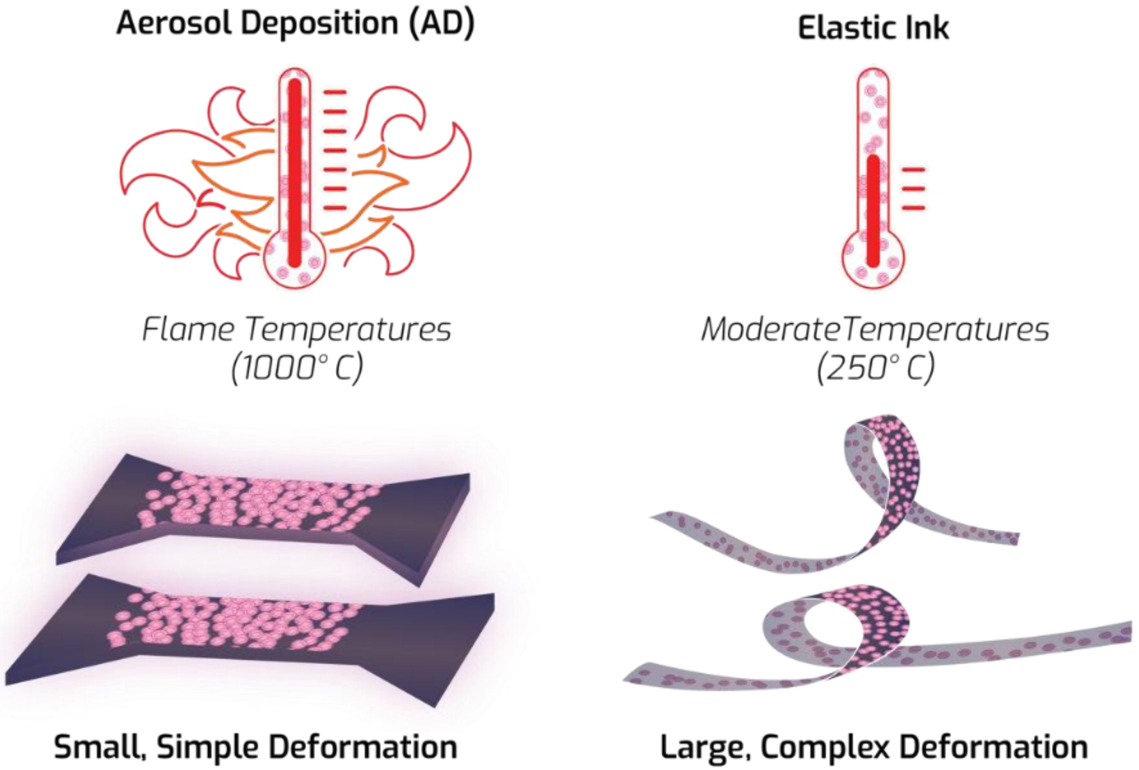


Figure 3. Multiple patterning techniques extend the usability of TP+DIC.

- Previous work has developed aerosol deposition (AD) for binderless coatings at high temperatures and small deformations.[2]
- Recent studies provide detailed information on stress–strain characteristics of Sylgard 184 binder.[3]
- However, its mechanical behavior changes significantly during polymer preparation, depending on curing time and temperature.[4,5,6]
- Small amounts of well-characterized $Mg_3F_2GeO_4$:Mn (MFG) powder can be added to Sylargard 184 binder to enhance material properties depending on intended application.
- ***The present study aims to further investigate the flexible polymer-based luminescent ink's capability for non-contact temperature measurements of an aluminum surface during mechanical and thermal testing.***

(2) Mahaffey, J., Murray, S. E., Winters, C., Jones, E. M., Jones, A., Flores-Brito, W., & Hoffmeister, K. (2021, September). Fabrication of phosphorescent oxide coatings using the aerosol deposition technique. In Advances in Optical Thin Films VII (Vol. 11872, pp. 86-95). SPIE.
(3)Hopf, R.; Bernardi, L.; Menze, J.; Zündel, M.; Mazza, E.; Ehret, A. E. Experimental and Theoretical Analyses of the Age-Dependent Large-Strain Behavior of Sylgard 184 (10:1) Silicone Elastomer. *Journal of the Mechanical Behavior of Biomedical Materials* **2016**, *60*, 425–437. https://doi.org/10.1016/j.jmbbm.2016.02.022.
(4)Johnston, I. D.; McCluskey, D. K.; Tan, C. K. L.; Tracey, M. C. Mechanical Characterization of Bulk Sylgard 184 for Microfluidics and Microengineering. *Journal of Micromechanics and Microengineering* **2014**, *24* (3), 035017. https://doi.org/10.1088/0960-1317/24/3/035017.
(5)Placet, V.; Delobelle, P. Mechanical Properties of Bulk Polydimethylsiloxane for Microfluidics over a Large Range of Frequencies and Aging Times. *Journal of Micromechanics and Microengineering* **2015**, *25* (3), 035009. https://doi.org/10.1088/0960-1317/25/3/035009.
(6)Brounstein, Z.; Zhao, J.; Geller, D.; Gupta, N.; Labouriau, A. Long-Term Thermal Aging of Modified Sylgard 184 Formulations. *Polymers* **2021**, *13* (18). https://doi.org/10.3390/polym13183125.

Mixing Methods

Table 1. Details of sample preparation. 0.05 grams of Phosphor powder in each sample. Kapton mask has 250 μm features.

Sample Name	Base:Curative Ratio (by mass)	Curing time (hrs) before mask removal	Result
A1, A2	10:1	2	Pass
A3, A4	10:1	4	Pass
A5, A6	10:1	24	Fail
B1, B2	1:1	2	Pass
B3, B4	1:1	4	Pass
B5, B6	1:1	24	Pass

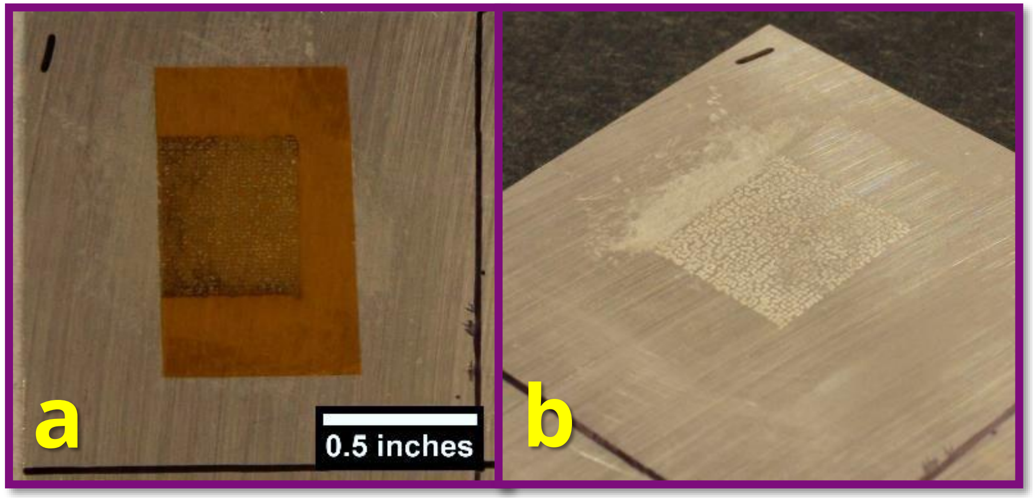


Figure 4. (a) Manual deposition over Aluminum substrate; (b) sample after Kapton mask removal, visible ink dot pattern.

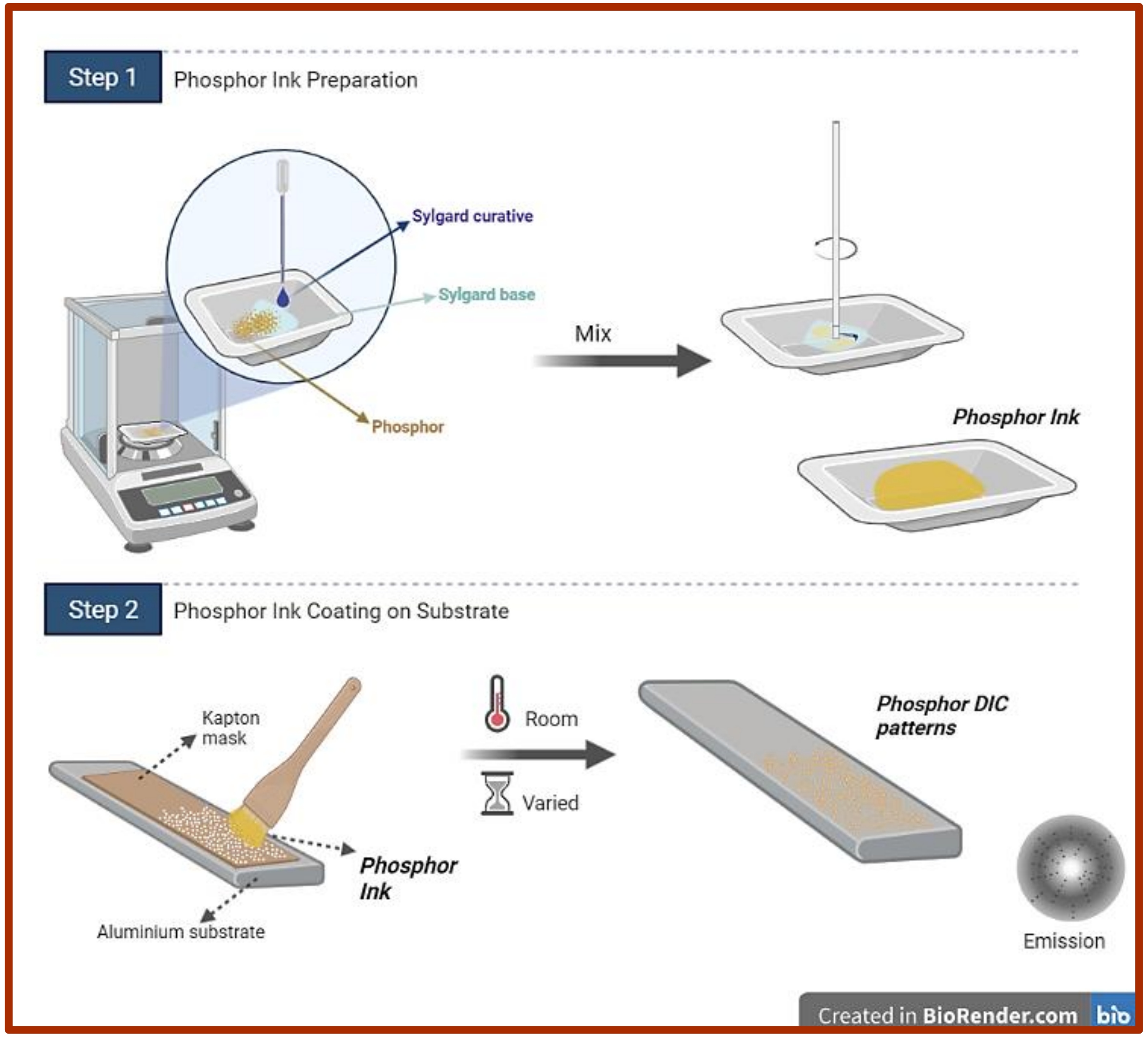


Figure 5. Sample preparation process and manual deposition over aluminum substrate.

Testing Methods

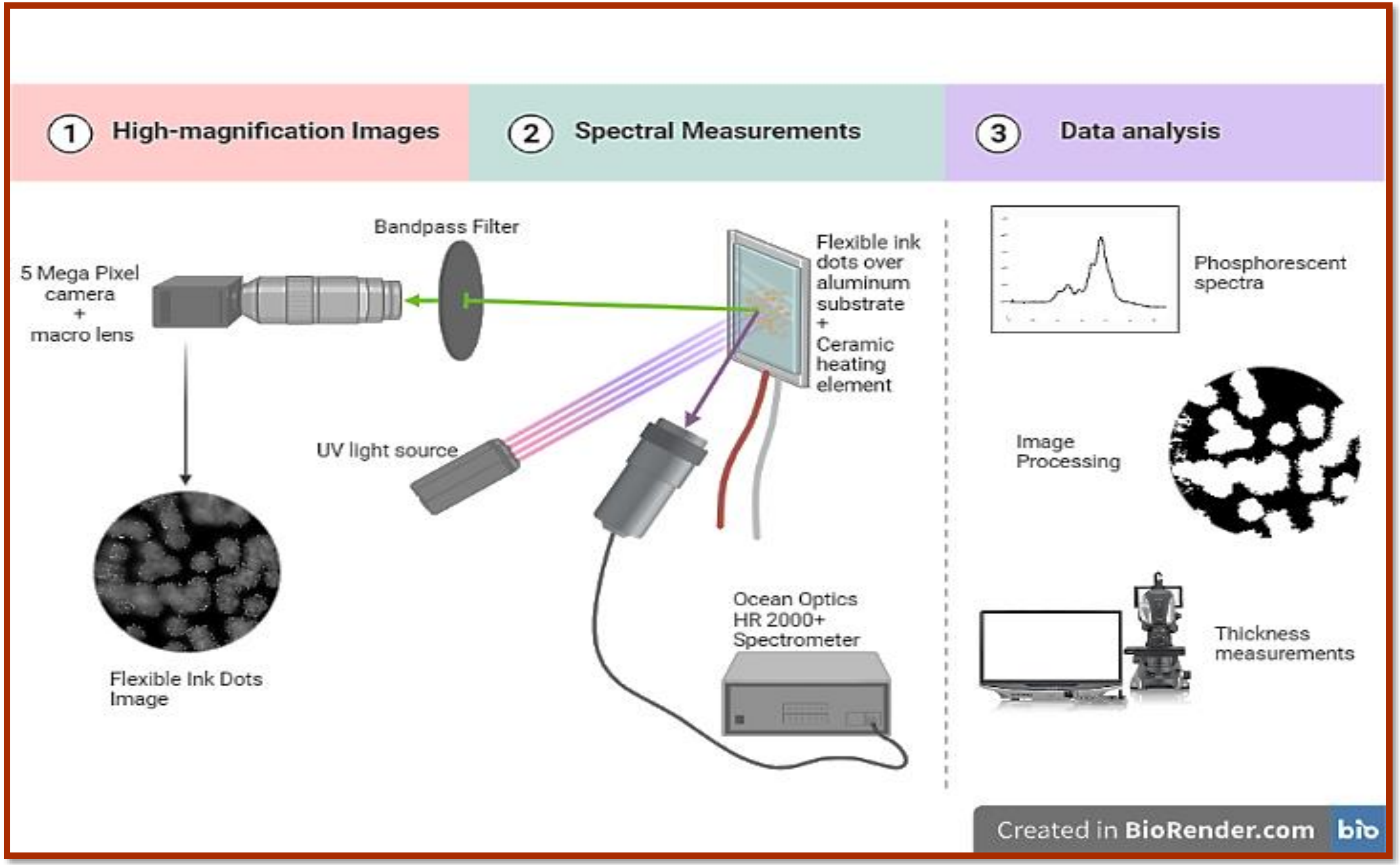


Figure 6. Testing set up for sample thermal testing, in situ spectral measurements and high-resolution imaging. Data analysis includes thickness measurements with a Keyence Digital Microscope (optical profilometer).

Phosphor-ink in Mechanical Environments

A DIC speckle pattern on a stainless steel specimen was produced with either a polymer-based phosphor ink or a binderless coating produced via Aerosol Deposition.

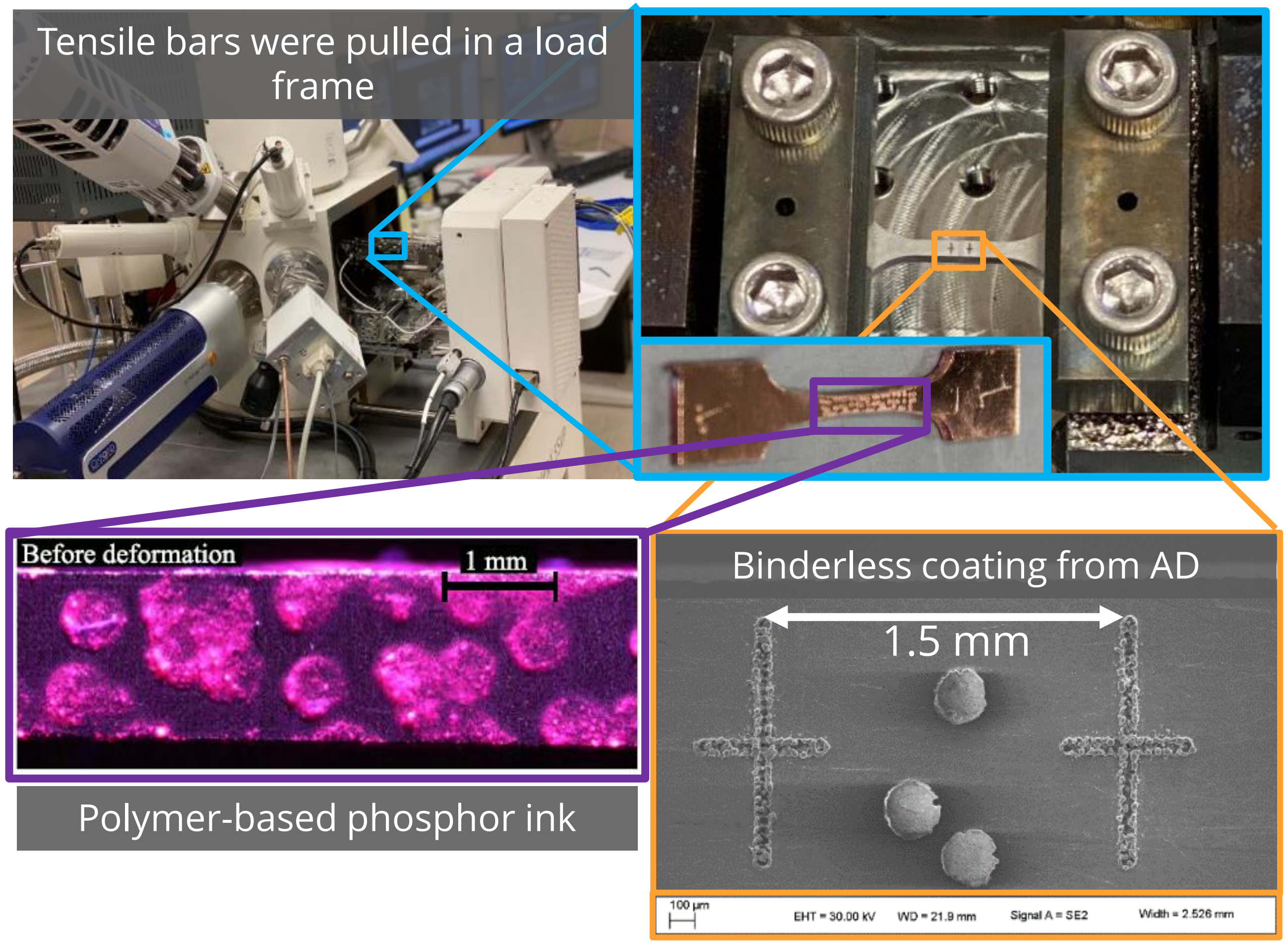
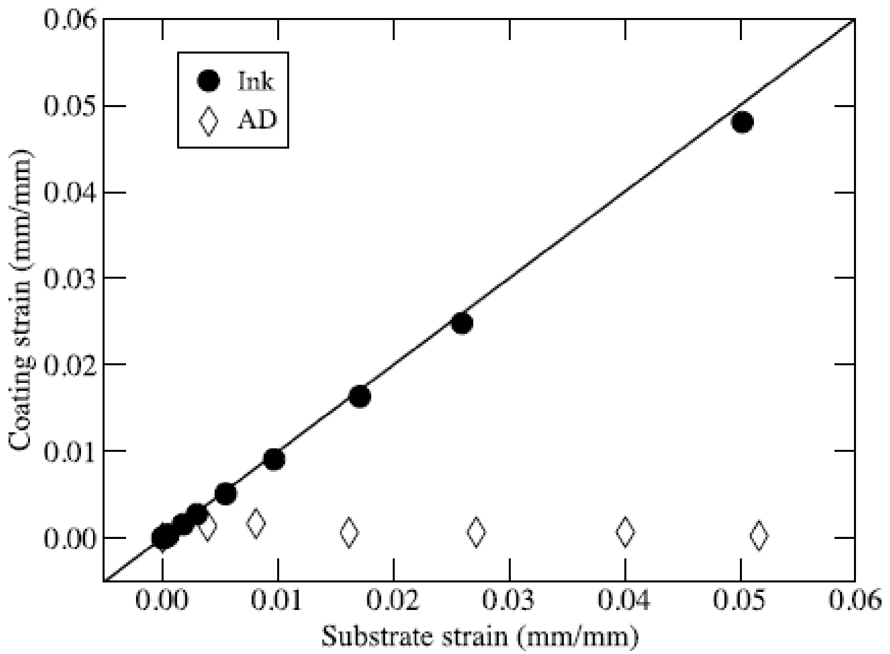


Figure 7. Coating strain versus substrate strain



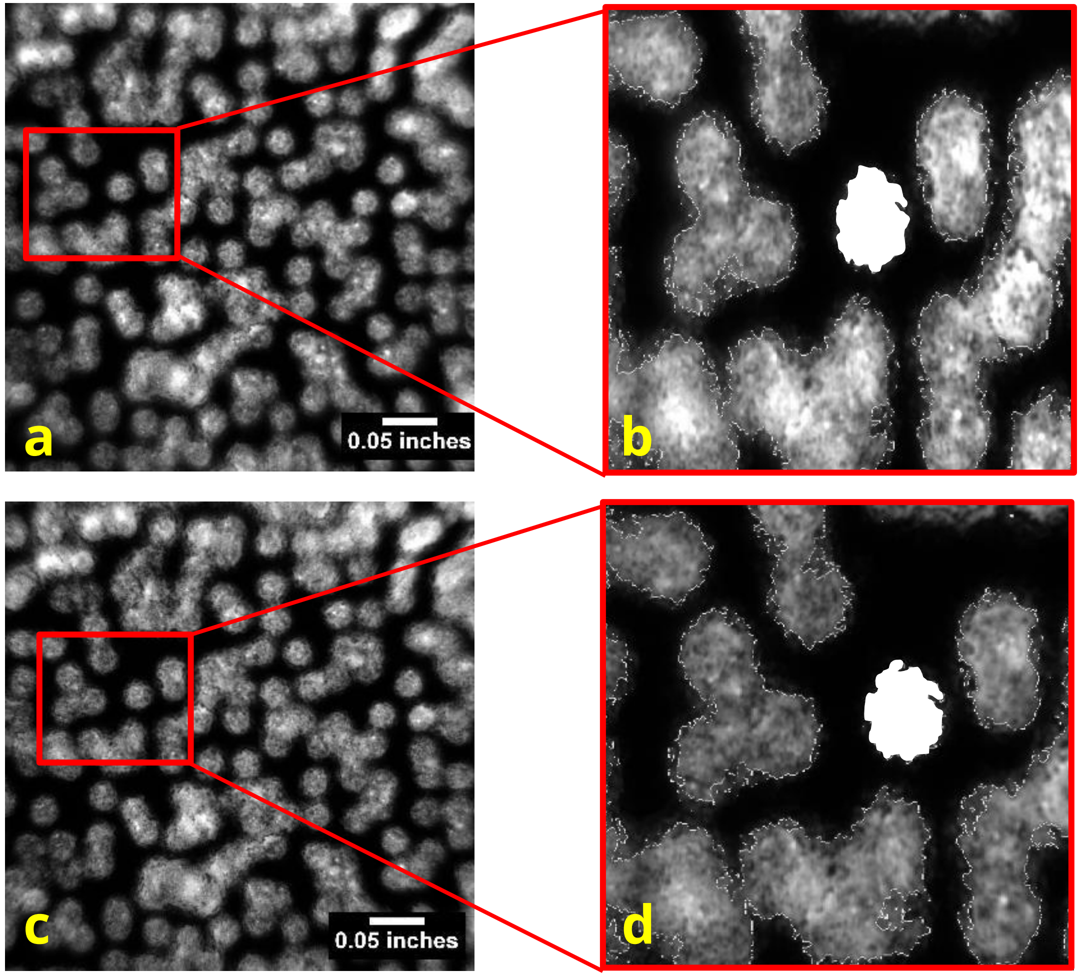
At the highest substrate deformation, the ink-coating strain deviates from the substrate strain by 0.002 mm/mm (a 4% difference). Because the AD coating partially debonded from the surface when stress was applied, the coating strain remains nearly zero as the substrate deforms.

The phosphor-ink deformed conformally with the substrate. The AD coating partially debonded.

Phosphor-ink in Thermal Environments

Start Temperature: 24.6°C

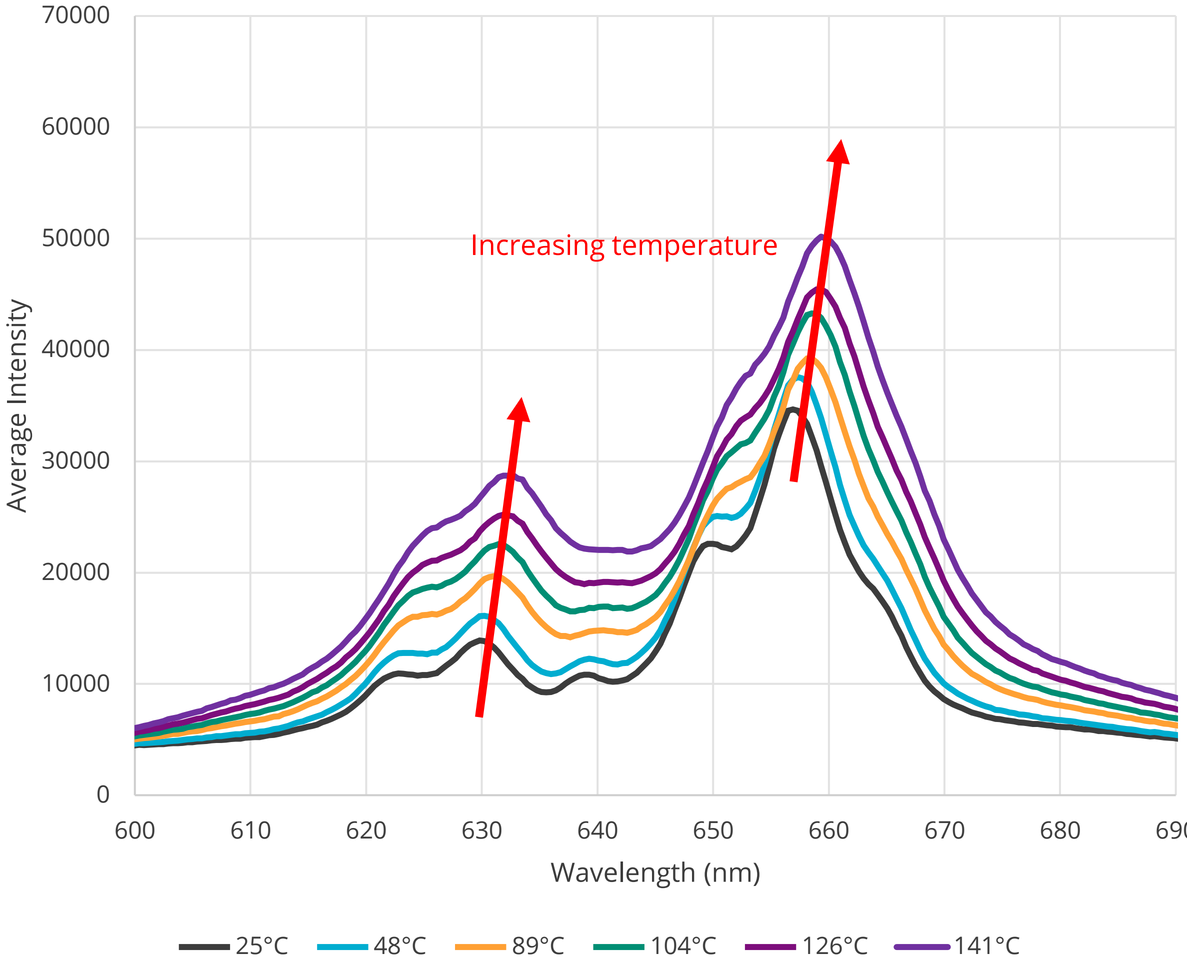
Areas: 0.206 $mm^2 \pm 0.003$



End Temperature: 141.1°C

Areas: 0.203 $mm^2 \pm 0.003$

Figure 8. Thermal Test MFG 10:1 (2 h curing time)



The phosphor-ink produced thermographic behavior, AD sample behaved similarly.

Conclusions

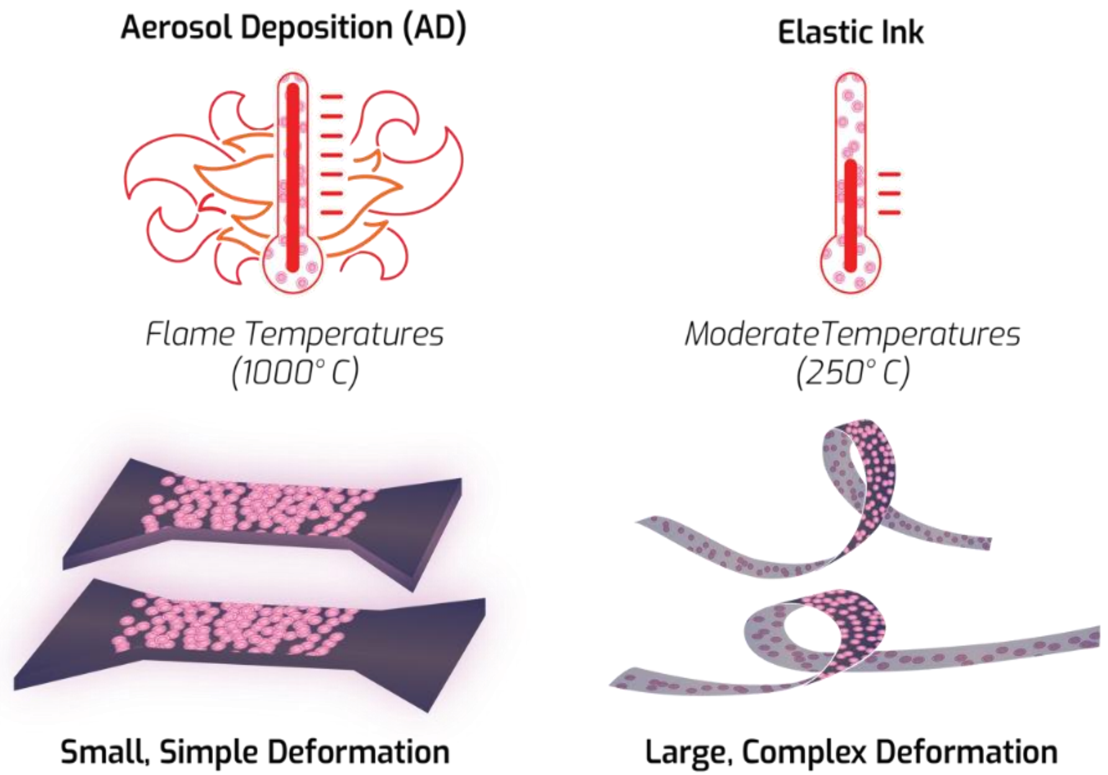


Figure 3. Multiple patterning techniques extend the usability of TP+DIC.

- Aerosol deposition – a binderless, impact consolidation coating – can resolve flame temperatures in reacting environments but only small deformations (left).
- Elastic ink with embedded thermo-phosphors can resolve and large deformations but only at moderate temperatures (right).
- The phosphor-ink produced thermographic behavior, similarly to the AD sample.
- For phosphor-ink, the phosphorescence intensity was directly related to temperature.
- Phosphor-ink adheres to substrate and no significant smearing or blurring occurs while temperature increases.
- Future work includes: optical profilometer data analysis, contrast manufacturing 10:1 ratio recommendation with 1:1 ratio.

Acknowledgments

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New things I learned this summer!

Matlab: image processing

ImageJ: image processing

Optical profilometer

THANK YOU

Background to Discovery: Strain-induced bias error in phosphor thermometry

*Phosphor emission should only change with **temperature.** However, the prior coating caused an unexpected change with **strain***

Figure 4. Image of a tensile specimen with a DIC pattern coated using an impact consolidation process called Aerosol Deposition (AD)

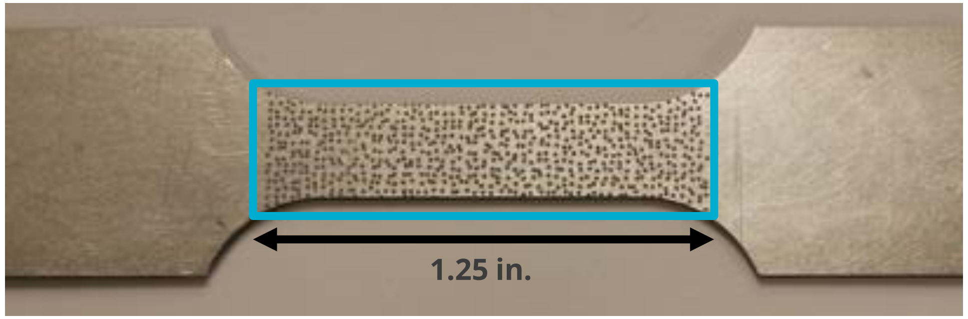
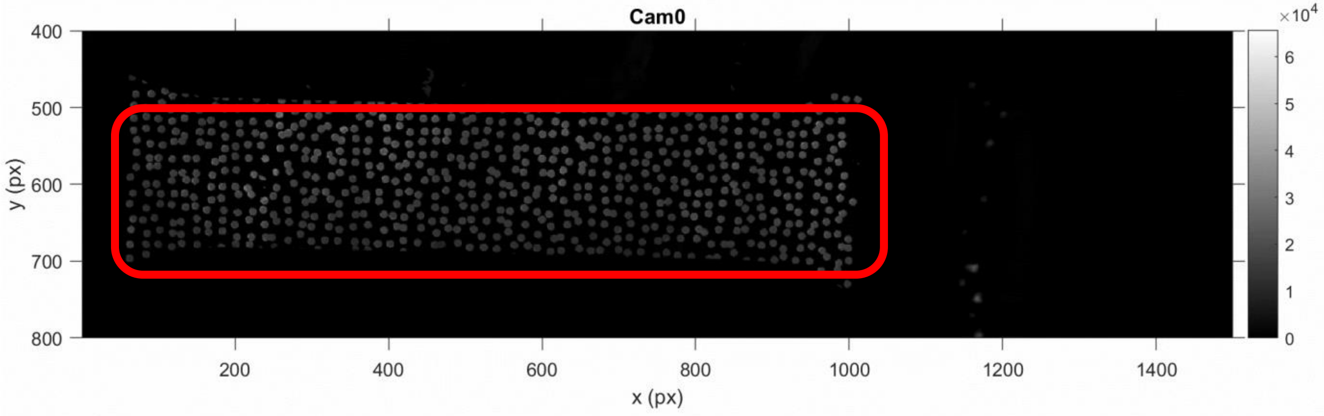


Figure 5. Raw data of a tensile specimen coated with AD under tensile loading



- The phosphor for this work is Manganese doped Magnesium fluorogermanate (MFG)

- Aerosol Deposition originally coated MFG onto a 304L stainless steel tensile bar in a random dot-pattern for DIC
- Under tension the strain of the sample affected both the **brightness & the spectral response** of the phosphor
- The change led to a **5-10°C error** in inferred temperature for the thermophosphor digital image correlation (TP+DIC) diagnostic.

We hypothesize that an elastomer-based ink can hold phosphors onto the surface, without imparting substrate strain and temperature biasing.