

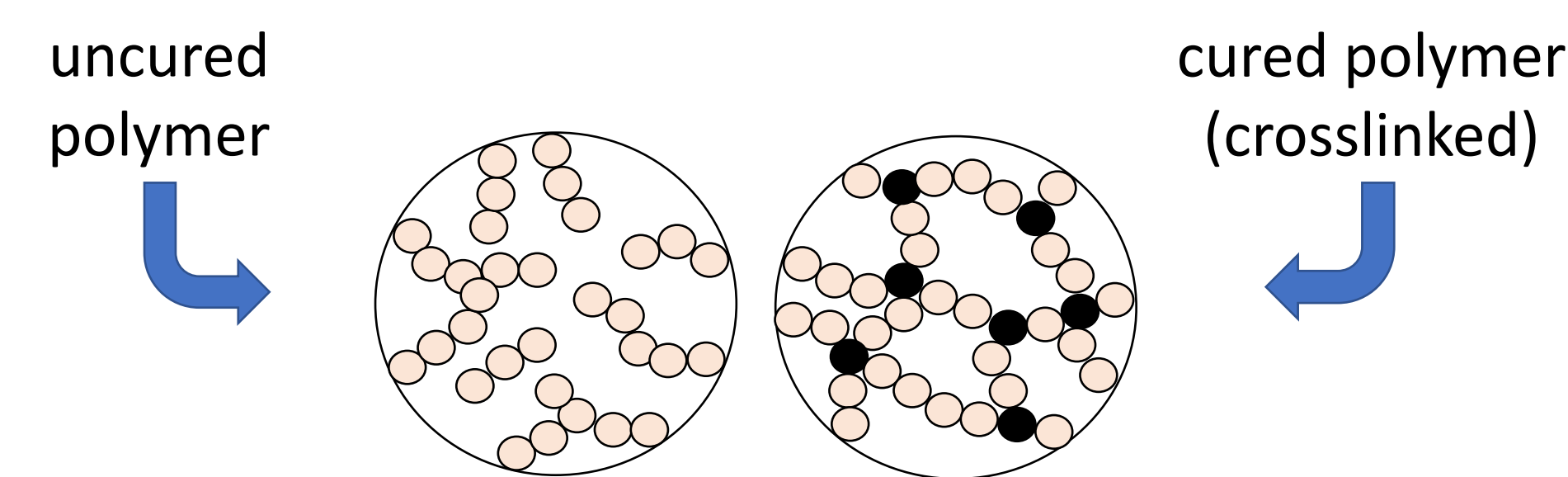
RESIN CURE KINETICS AND EXPLORING THE POSSIBILITIES OF MATERIAL DEGRADATION

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Background

Thermosets: Thermosets are polymers that are **irreversibly hardened** once cured.

- Curing process is heat induced
- Can be processed at high temperatures
- Tacky at room temperature



When a thermoset cures, the polymer **crosslinks**. Crosslinking is the connection of polymer chains. Crosslinking changes the properties of the polymer making it stronger

Cure Kinetics for a Thermoset Resin:

Thermosetting resins have a kinetics equation as follows:

$$\frac{d\alpha}{dt} = k(1 - \alpha)^n$$

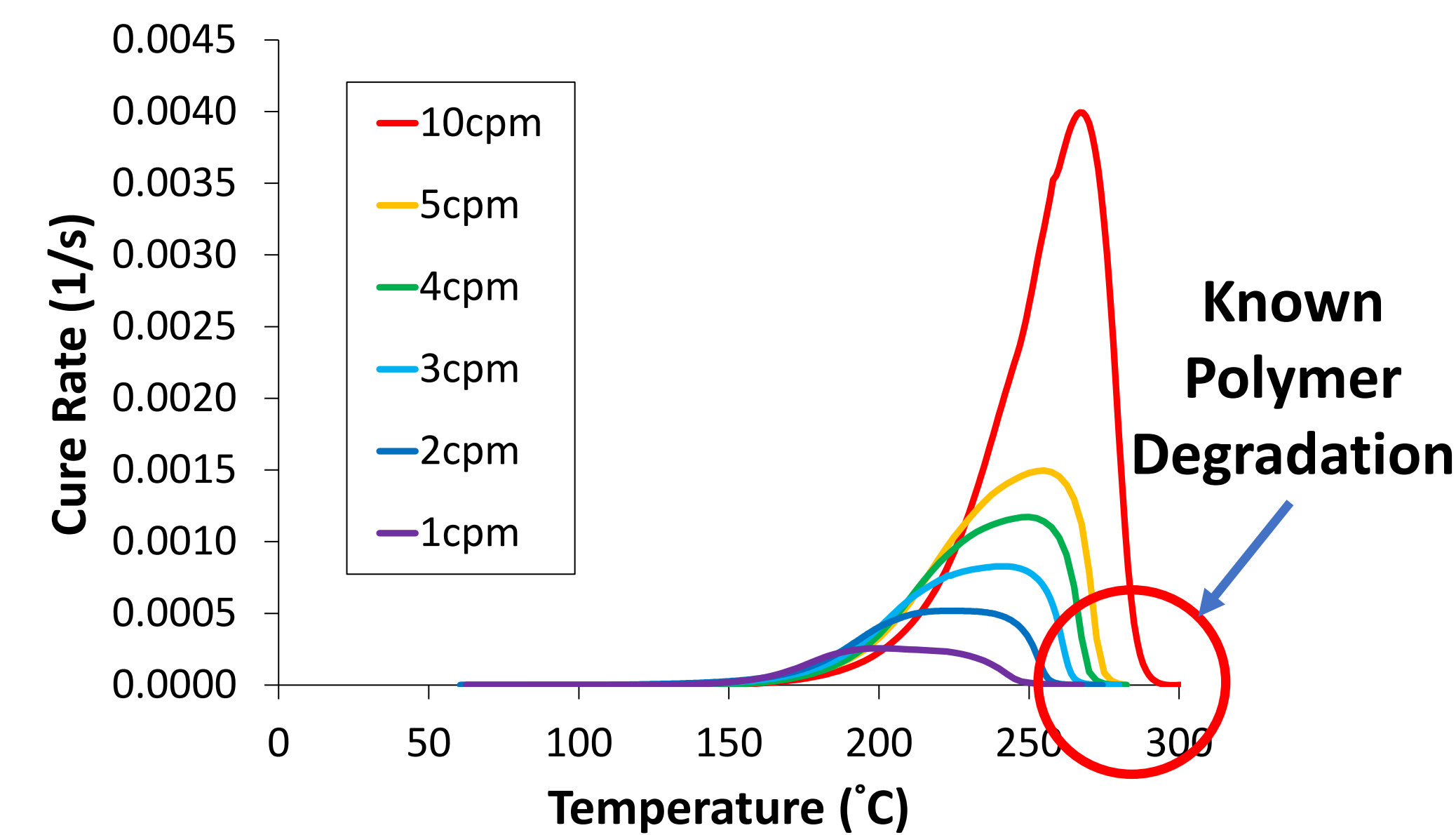
$$k = Ae^{\frac{-E_a}{RT}}$$

Variable	Description
α	degree of cure
k	reaction rate
n	reaction order
A	pre-exponential factor
E_a	activation energy
R	gas constant

Problem Statement

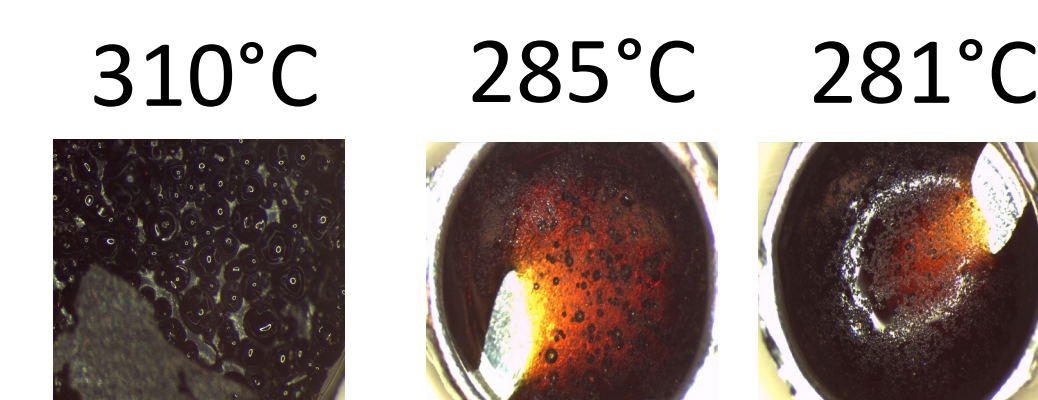
The goal of this research project is to develop a cure kinetics model for the thermoset resin at constant temperature ramp rates. The challenge with developing the model is the possibility of material degradation.

A DSC (Differential Scanning Calorimeter) was used to measure the heat flux as a function of time and temperature. To formulate a model, data from dynamic runs was collected at different heating rates.



- The dynamic runs show 3 observations that could be degradation:

- **Exotherm** at the end of reaction
- **Double peaks**
- **Weight Loss: 2.18%**
- **Discoloration**



Material Degradation: reduction of its physical properties such as strength and weight.

Materials and Methods

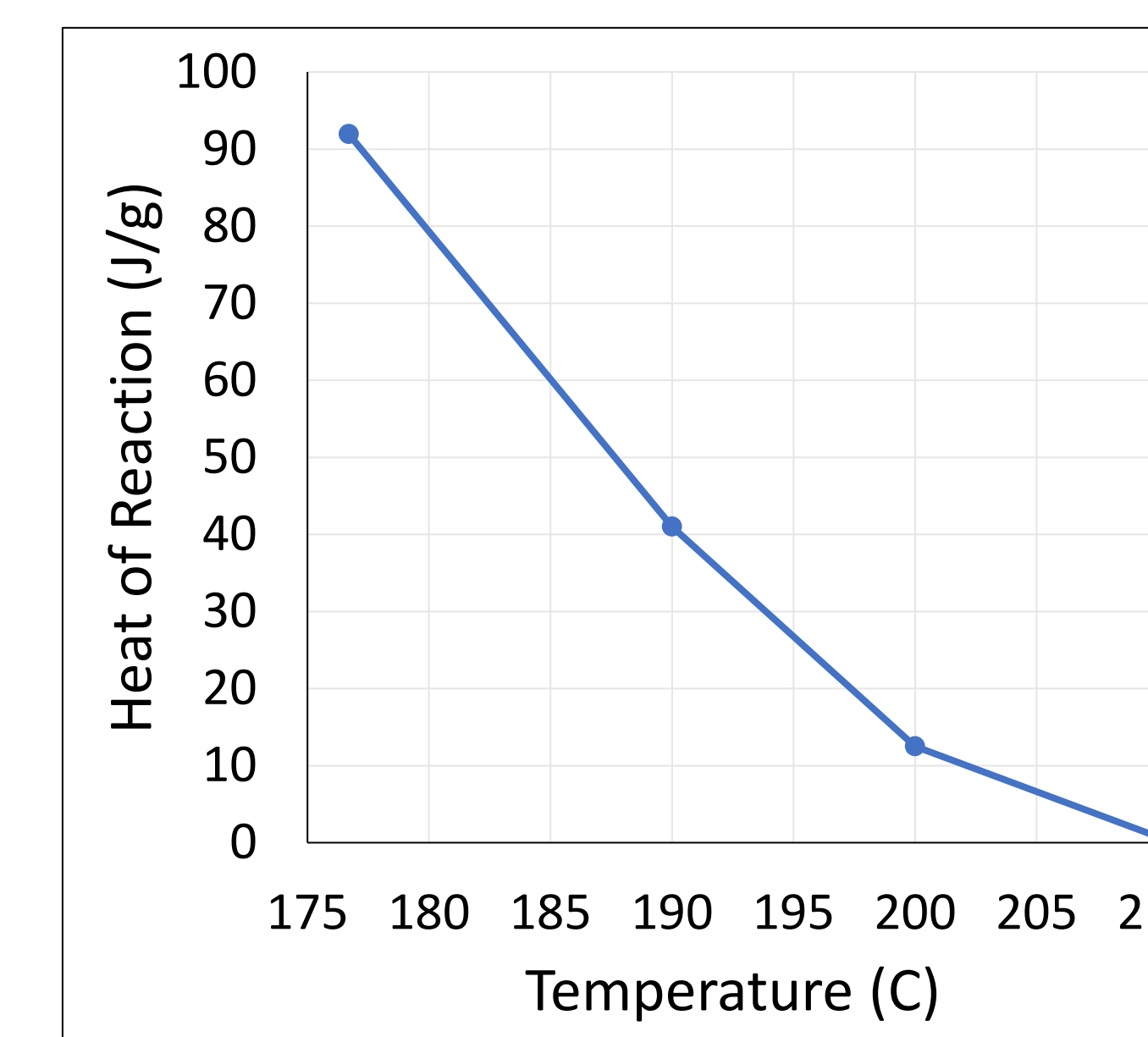
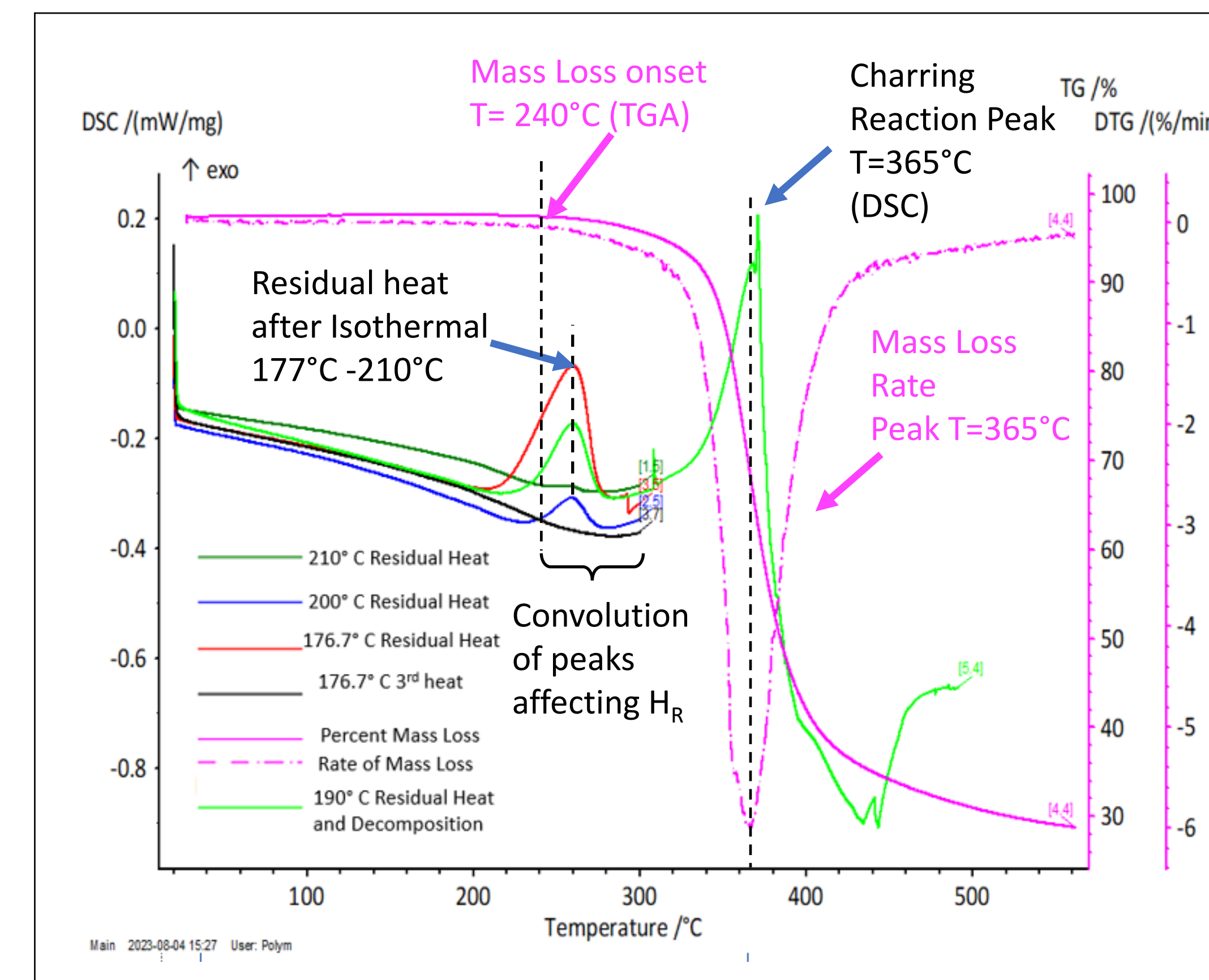
DSC: The recommended cure cycle at different isothermal hold temperatures for 6 hours was followed by 2 additional reheats to evaluate the residual heat of reaction.

TGA (Thermogravimetric Analysis): A TGA measures the mass loss as a function of temperature to analyze where mass loss begins.

Hotstage Microscope: Creates a video of the sample during the DSC run to see where discoloration begins

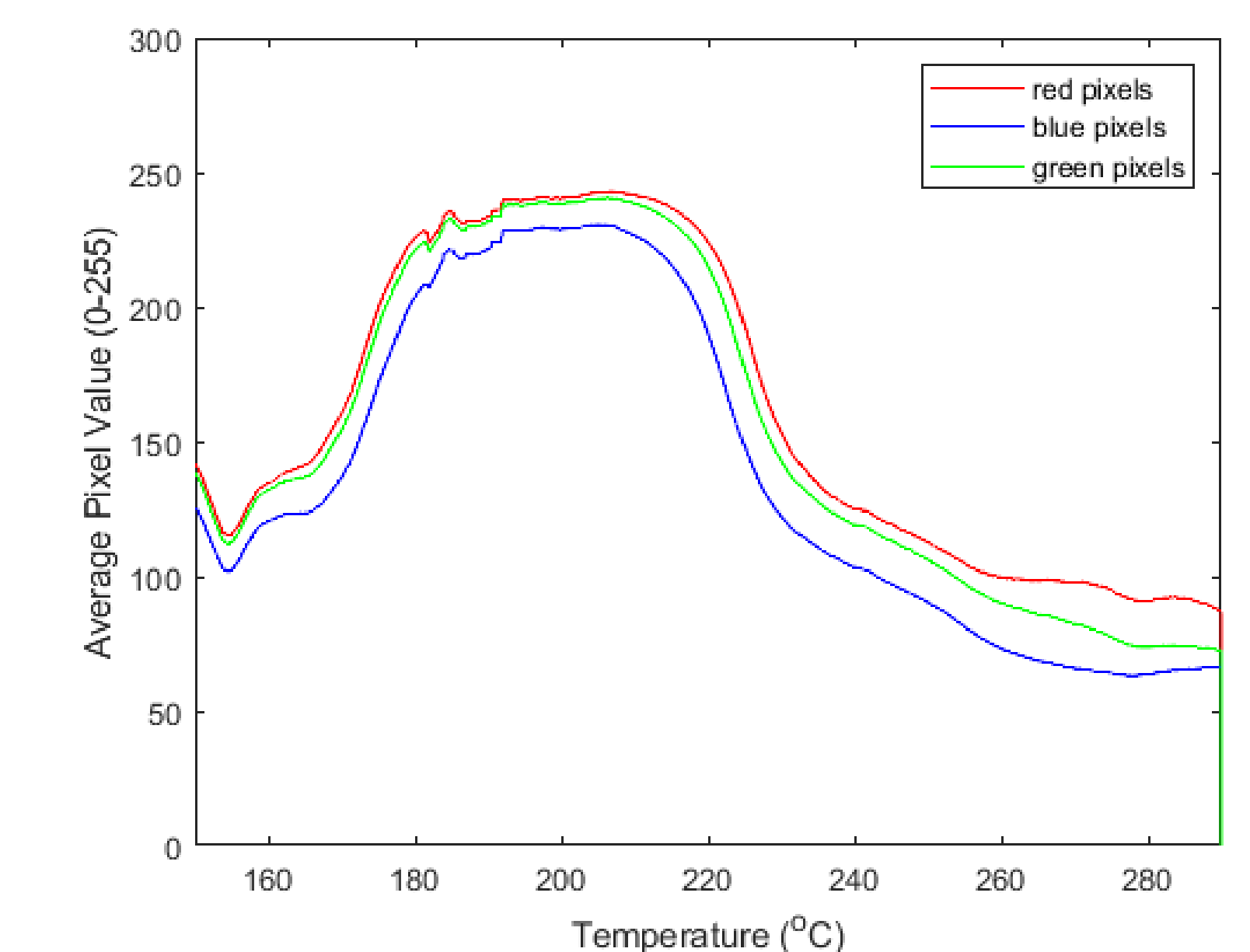
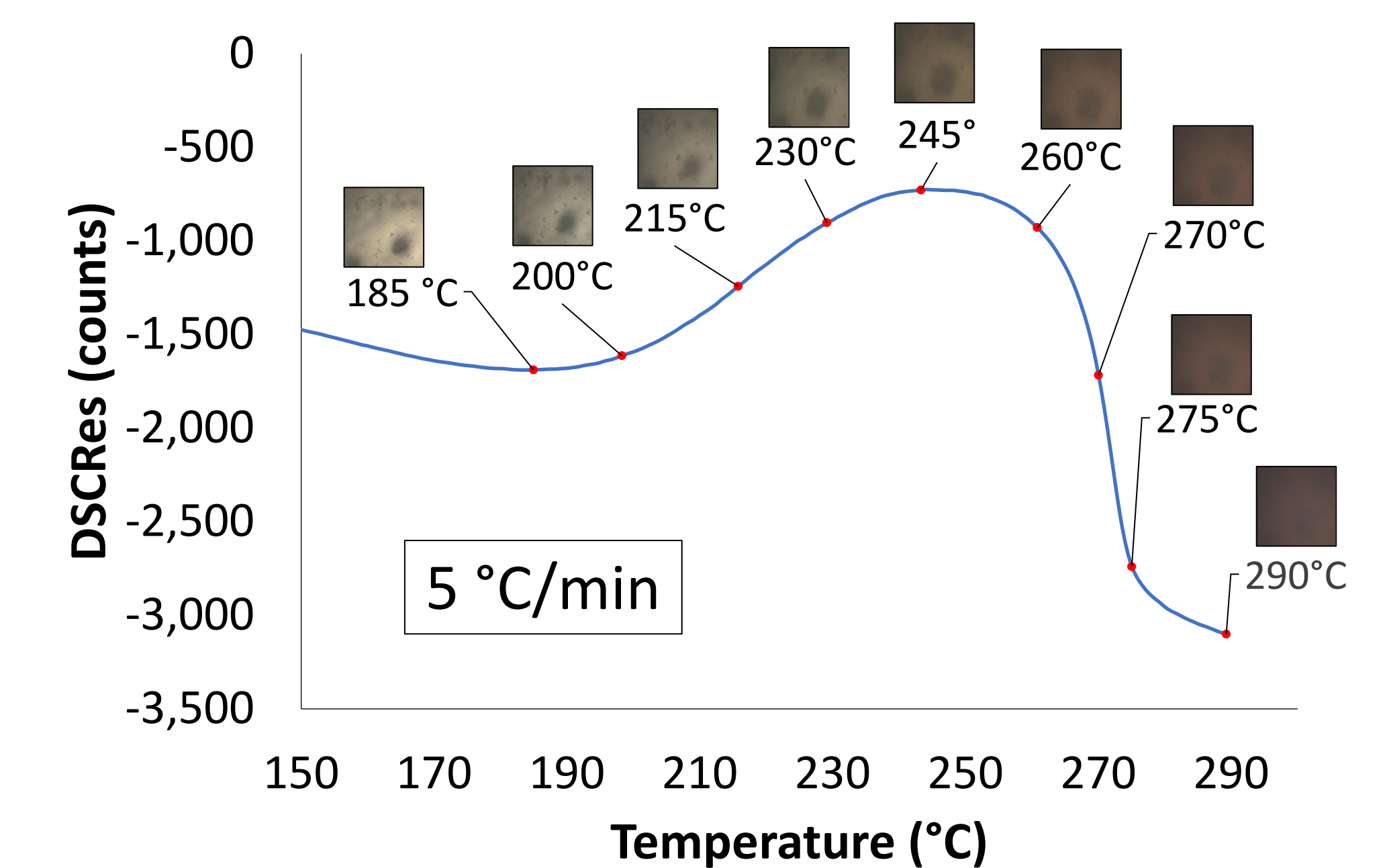
Results

DSC and TGA Analysis:



The first graph shows the peak of the degradation from the TGA overlaps the DSC data. From the first graph, the convolution of peaks affecting the residual heat of reaction is shown. The residual heat was found after running cure cycles with different isothermal hold temperatures for 6 hours. The relationship of residual heat of reaction against temperature is shown in the second graph. As the temperature increases for the isothermal hold temperature, the residual heat of reaction decreases.

Hotstage Microscope Analysis:



The first graph shows the images captured from the hotstage microscope at different temperatures along the peak of a dynamic run at 5°C/min. The second graphs shows the average pixel value for the video against temperature. From the 2 graphs, the onset of discolorations occurs around 230°C.

Conclusions and Future Work

The overlapping data from the DSC and TGA analysis helped to find out where the sample decomposes. The DSC also gave insight on the decreasing residual heat of reaction. The results suggest further evaluation of a new analysis method.

Acknowledgements

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