

# Development of $\text{Na}_2\text{SO}_4$ Application Technique for Hot Corrosion Testing

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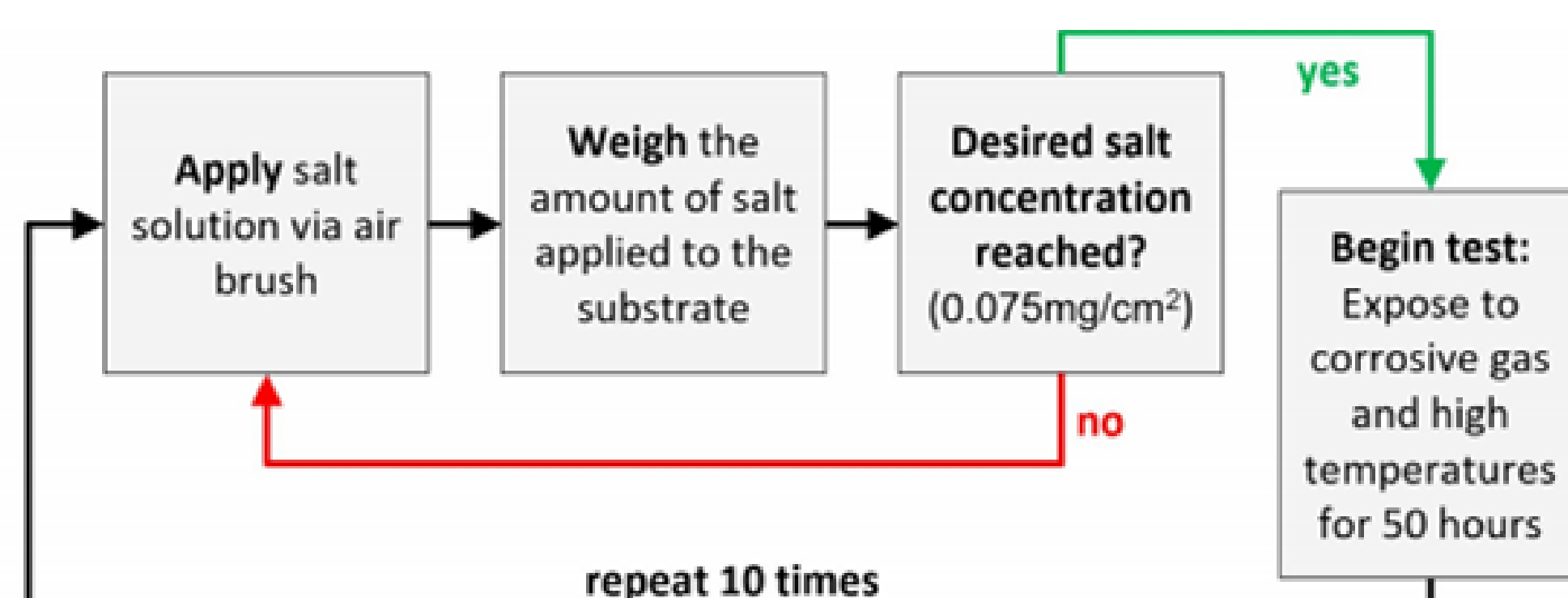
Rolls-Royce uses protective coatings for superalloy turbine blades to resist corrosion from  $\text{Na}_2\text{SO}_4$ . Hot corrosion testing is performed to analyze the effectiveness of such coatings. The current salt application method of  $\text{Na}_2\text{SO}_4$  for testing can take up to 8 hours. There is a need to reduce the application time of  $\text{Na}_2\text{SO}_4$ . The methods and results depicted are a continuation of previous work done for Rolls-Royce.  $\text{Na}_2\text{SO}_4$  was dissolved in water along with PVP and D-Xylose plasticizer, then cast into a PVPX (PVP/D-Xylose) film. This PVPX film was cut to sample size and placed on the substrate.  $\text{Na}_2\text{SO}_4$  concentrations of 0.086 and 0.090  $\text{mg}/\text{cm}^2$  were obtained with 14.8% and 20.4% error respectively, from the goal of 0.075  $\text{mg}/\text{cm}^2$ . The increased concentration was traced to Na in the release agent used in the production of the films. Hot corrosion testing was performed and SEM images indicated the film-applied samples experienced more hot corrosion than the salt-sprayed samples due to excess sodium sulfate forming during testing.

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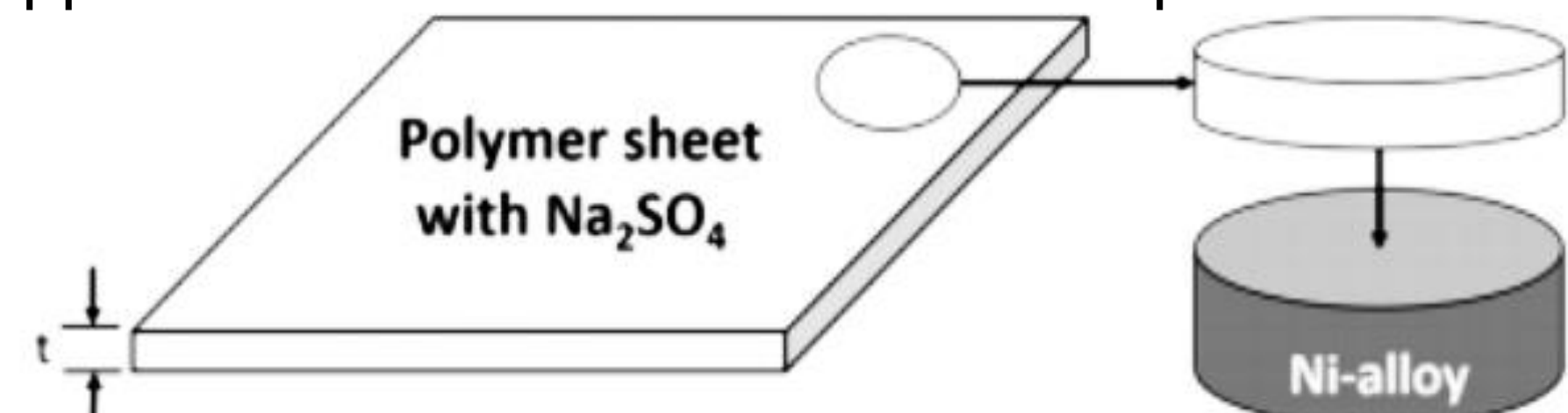
## Project Background

Rolls-Royce is a leading manufacturer of turbine engines for aircraft. These engines experience hot corrosion in the presence of  $\text{Na}_2\text{SO}_4$ , which forms from Na in marine environments and S from jet fuel. Low temperature hot corrosion is the focus of our study and occurs from 650 – 850 °C when high partial pressures of  $\text{SO}_3$  are present. Hot corrosion degrades the alloy and can cause catastrophic failure. Rolls-Royce utilizes hot corrosion tests to determine the performance of their alloys when these conditions are present. During this test,  $\text{Na}_2\text{SO}_4$  is applied to the surface of a small sample and exposed to  $\text{SO}_2$  gas and high temperatures to simulate hot corrosion. Currently, the manufacturer uses a salt-spray to apply  $\text{Na}_2\text{SO}_4$  to samples for corrosion testing. The application process is illustrated below.



Kim, A. et. al. (2017). Development of Hot Corrosion Technique for Hot Corrosion Testing.

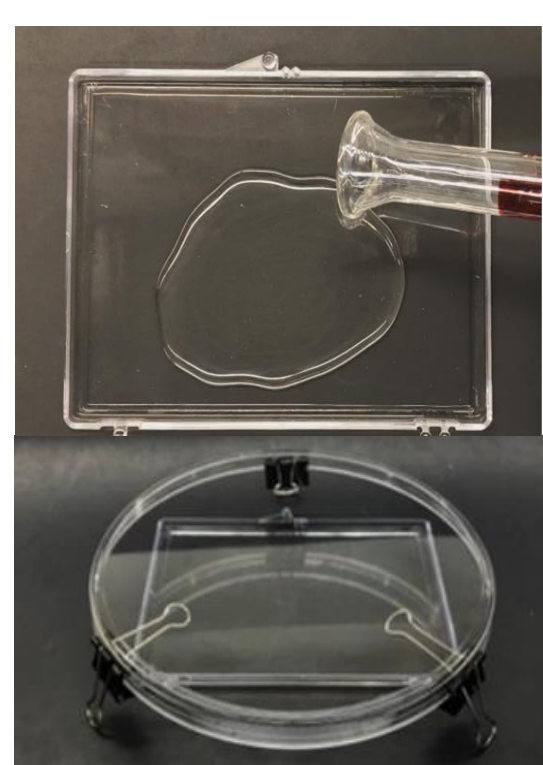
This salt application process is inefficient. To reduce preparation time, a polymer film has been proposed to replace the salt spray technique. The film has a known salt concentration per unit area and is cut to the shape of the alloy sample, then placed directly on the sample surface. This application process reduces salt application time from 8 hours to a couple minutes.



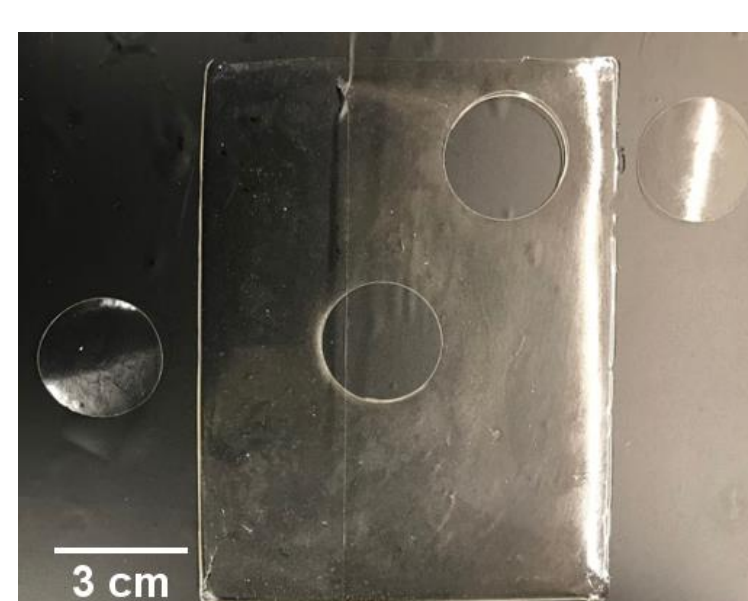
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## Film Preparation

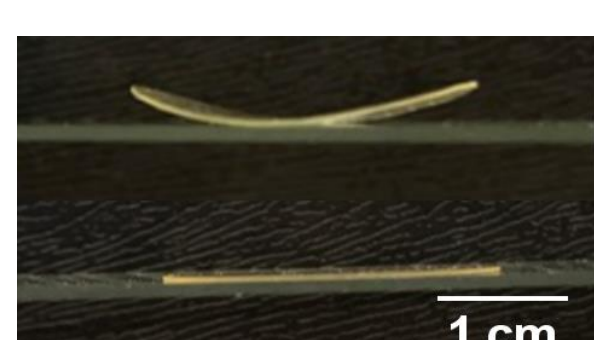
Using water as a solvent, a solution was prepared of 10 vol% polyvinylpyrrolidone (PVP), 3.5 vol% D-Xylose, and the mass of  $\text{Na}_2\text{SO}_4$  such that when cast it will have an area concentration of 0.075  $\text{mg}/\text{cm}^2$ . After the polymer completely dissolved, the solution (right) was speed mixed to ensure homogeneity.



The polymer solution was poured into a polystyrene container coated with an organic mold release. The solution dried for 24 hours at room temperature on a flat surface with a cover to block dust (left).



The dried polymer film was removed from the mold, cleaned with acetone, and cut into the desired shapes (right).



To resolve warping, the samples were annealed at 160°C until completely flat (left).

## Mold Release Methods

Polyvinylpyrrolidone (PVP), D-Xylose plasticizer, and  $\text{Na}_2\text{SO}_4$  were dissolved in water, then the solution was cast in a mold. During the drying process, if the PVPX film adheres strongly to the mold, then it cannot be removed or used. A mold release is necessary to prevent adhesion while still allowing the solution to spread evenly throughout the mold.

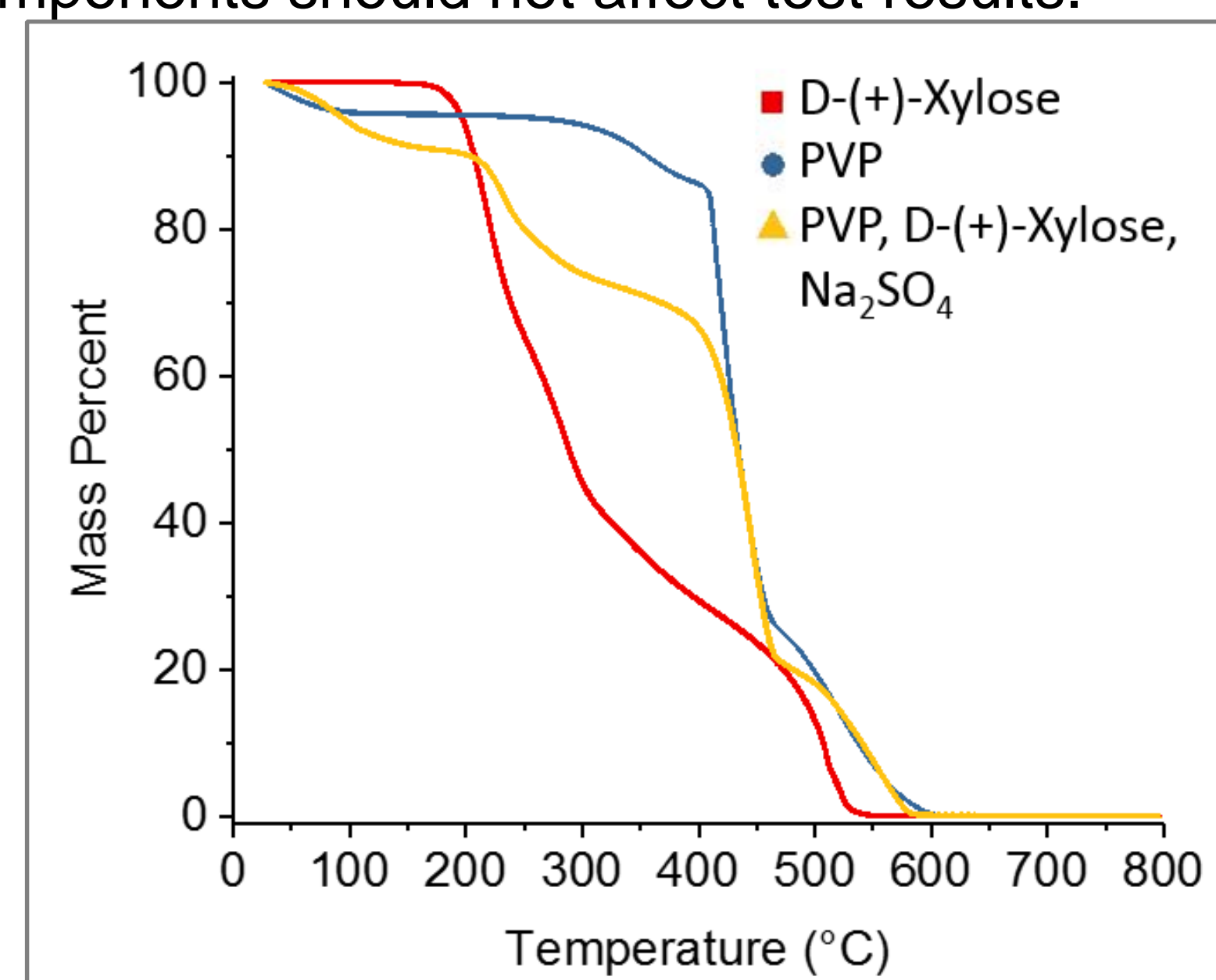
PTFE tape worked well as a mold release liner but ethanol had to be added to the solution due to the high surface energy of PTFE. It also resulted in uneven edges due to the tape being tucked into the crevices and corners.



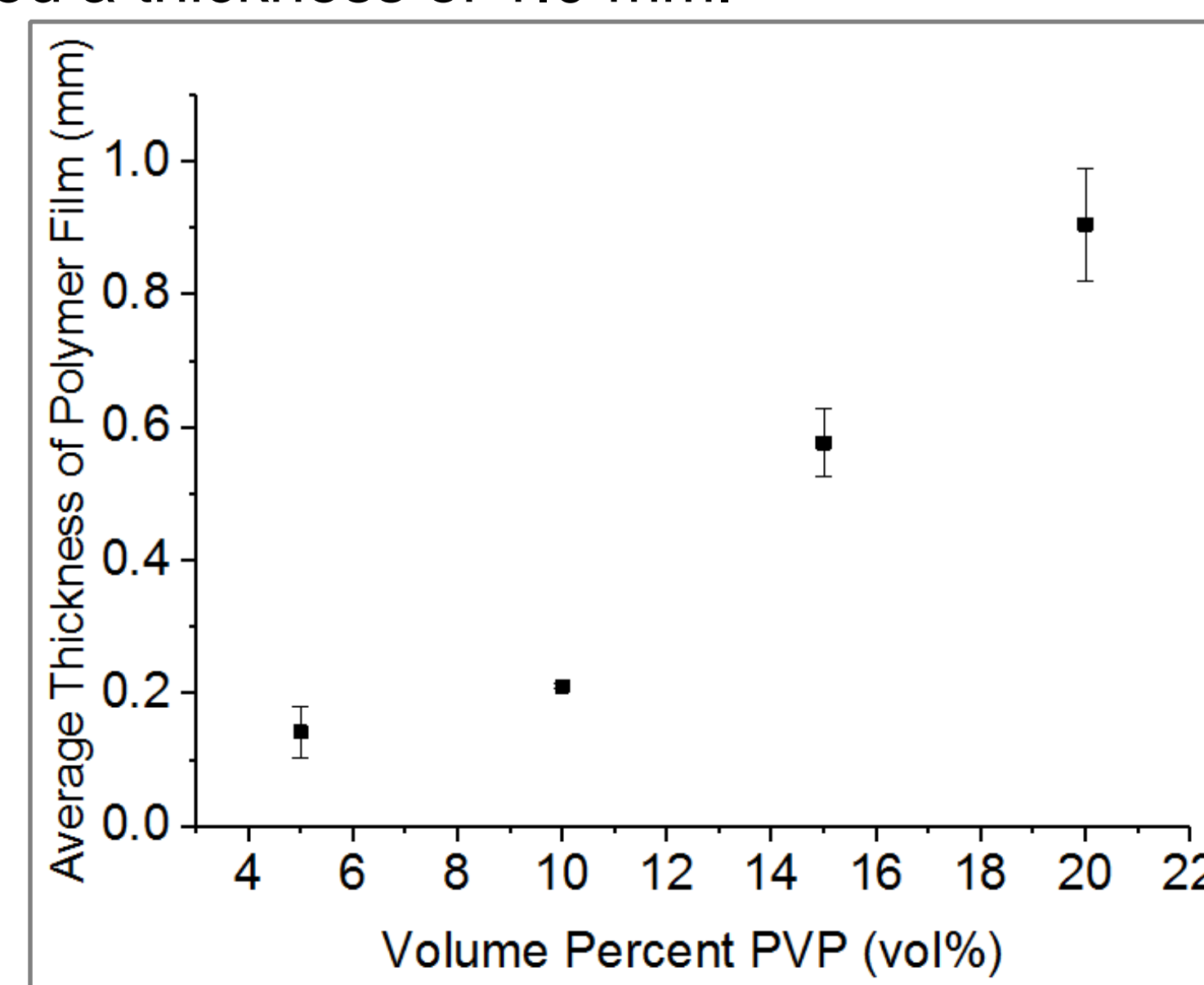
PAM™ cooking spray is an organic mold release that dispersed evenly and allowed the product to easily pop out of the mold, maintain flexibility, and be cleaned with acetone. However, PAM™ contains 0.4 mg of Na per gram which may be adding extra sodium to the film.

## Polymer Film Characterization

To characterize the burn-off of the PVPX film, thermal gravimetric analysis (TGA) was performed. A sample of a PVPX film was tested which comprised of PVP, D-Xylose, and  $\text{Na}_2\text{SO}_4$ . Pure D-Xylose and PVP were also tested to examine the influence of the individual components on the film's degradation. The results indicate that the polymer film had completely burned off at 600°C, leaving only  $\text{Na}_2\text{SO}_4$ . This is below the hot corrosion test temperature of 700°C, indicating the organic components should not affect test results.



The thickness of the film was measured with respect to the volume percent of PVP in the casting solution. The most uniform film created reached a thickness of 0.2 mm, obtained from a 10 volume percent PVP solution. Previous sheets made last year were measured to have 22 volume percent PVP and yielded a thickness of 1.0 mm.

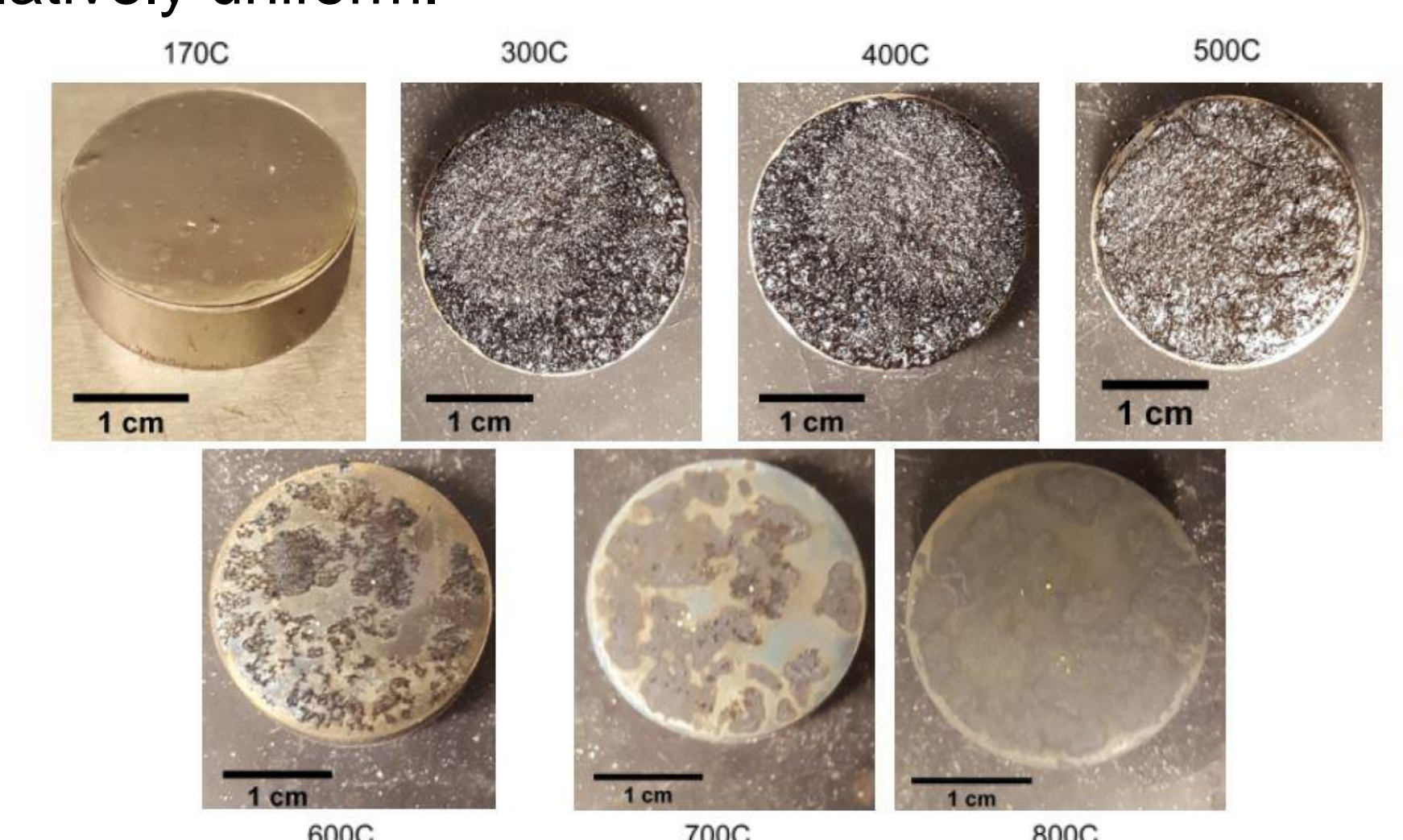


Film samples were sent to NSL Analytical to test for the concentration of Na using ICP-MS, and for the concentration of S by Parr Bomb testing followed by ion chromatography. Compared to the calculated theoretical values, the collected data seen below displayed a higher content of Na, and lower content of S than expected. This is due to the excess Na present in the organic release agent.

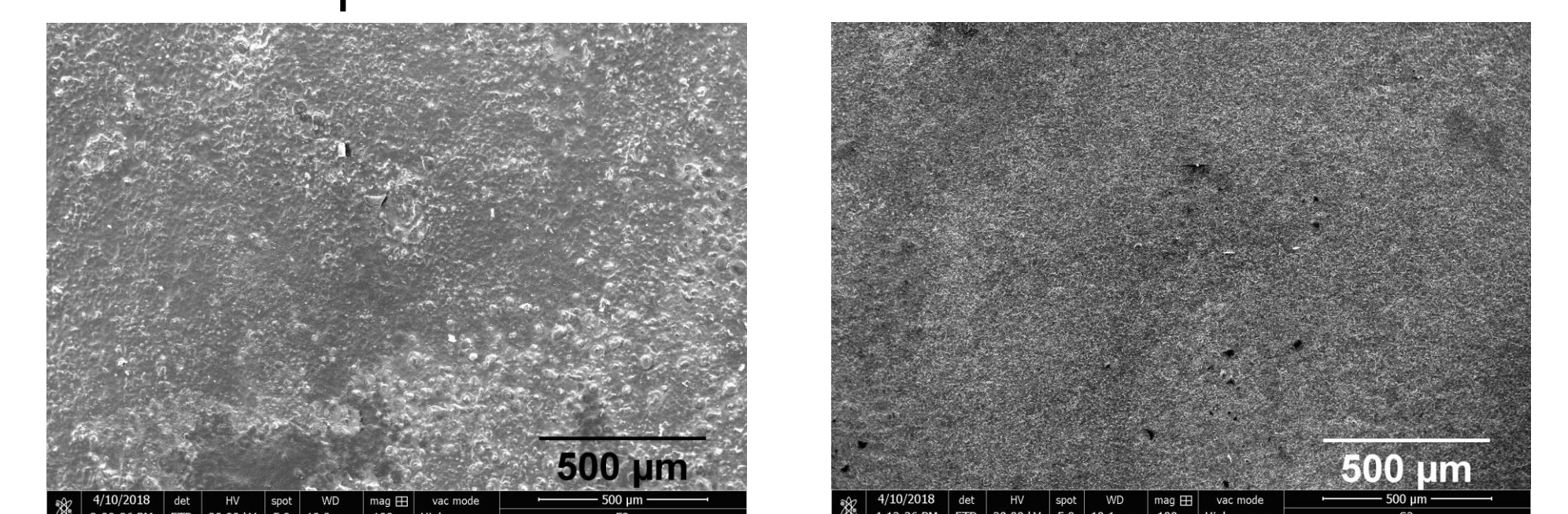
|        | Na (wt%) | S (wt%) |
|--------|----------|---------|
| 1      | 0.083    | 0.032   |
| 2      | 0.087    | 0.024   |
| Theor. | 0.071    | 0.050   |

## Hot Corrosion Analysis

A polymer burn off test was conducted in an  $\text{O}_2$  environment to understand the salt distribution once the polymer has been burned off. The furnace heating rate was approximately 12.5°C/min. A single, large bubble formed during film burn-off. However, after reaching 800°C the sample surface appeared relatively uniform.



Hot corrosion testing was performed on 3 samples:  $\text{Na}_2\text{SO}_4$  was applied via the salt-spray method for 1 sample, and applied via the polymer film for 2 samples. The test was performed at 700°C in an  $\text{SO}_2$  -  $\text{O}_2$  balanced environment for 100 hrs. The salt was reapplied at the 50 hour mark. SEM images of these samples are shown below.



The surface of the film-applied sample was more textured than the salt-sprayed sample. This indicates that the film-applied sample experienced more corrosion than the salt-sprayed sample due to the higher sodium content in the film.

## Conclusions and Future Work

The mold casting process was successful in creating a homogeneous polymer film with a uniform thickness throughout. The area concentration of the Na detected in the films was higher than desired, and the polymer film burned off completely and induced more hot corrosion than the salt-sprayed sample. Some nonuniformity can be seen in the SEM images of the film-applied sample. The increased amount of Na is due to the release agent used during the molding process. The amount of hot corrosion was increased due to the excess Na reacting with  $\text{SO}_2$  gas to create extra  $\text{Na}_2\text{SO}_4$  during testing.

Future work includes refining the  $\text{Na}_2\text{SO}_4$  concentration in order to obtain the desired 0.075  $\text{mg}/\text{cm}^2$ . While it could be because of higher amounts of  $\text{Na}_2\text{SO}_4$  on the surface of the sample, the polymer films could also be influencing the corrosion rate during degradation.