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A Comprehensive Investigation of the Influence of Geometric Structure on the Shape Memory Performance of Nafion

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A Comprehensive Investigation of the Influence of Geometric Structure on the Shape Memory Performance of Nafion

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By

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ABSTRACT

While perfluorosulfonic acid (PFSA) membranes have primarily been used in fuel cells due to their chemical, thermal, and mechanical stability, one PFSA, Nafion, boasts two unique characteristics: a broad glass transition (~55 °C to 130 °C) and a temperature-persistent electrostatic network. The combination of these two characteristics endows Nafion with exceptional shape memory properties – the ability of a material to morph and transform into pre-programmed shapes when exposed to an external stimulus – with enhanced permanent shape memorization, and a potentially near-infinite number of temporary shape memorization. This study focused on expanding the base of knowledge surrounding Nafion's shape memory properties in different geometries and environments. Results have shown that as deformation/recovery temperature decreases below 100 °C, the time required to achieve an adequate recovery percentage (>95%) drastically increases, whereas at or above 100 °C, that time decreases below one minute. Surprisingly, varying the geometry of the material showed very little change in the recovery time and percentage. Furthermore, increasing the strain during testing also does not affect the shape memory behavior. Finally, processing samples with a blue laser at different power levels and in different directions did not affect the shape memory recovery percentage either. This study found that Nafion's shape memory property is extremely robust, and its enhanced abilities vastly improve upon existing shape memory alloy and polymer functionality and expand the range of applications for Nafion, as many movements can be achieved in one cycle – lending applicability to uses such as deployables, actuators, sensors, and smart adhesives, particularly for in-space uses.

INTRODUCTION

Background

Perfluorosulfonic acid (PFSA) membranes are a type of commercial thermoplastic polymer with a polytetrafluoroethylene backbone and perfluoroether sulphonic acid side chains.[1] Because of this composition, PFSA membranes have exceptional chemical, thermal, and mechanical stability, as well as enhanced proton conductivity – leading to its use as a cation exchange membrane, separator in chlor-alkali cells, as well as a polymer electrolyte membrane in polymer electrolyte fuel cells (PEFCs).[2,3]

The composition of Nafion, a specific PFSA membrane, also lends itself to another advantageous characteristic: a broad glass transition and a temperature-persistent electrostatic network.[1] A material's glass transition temperature is the temperature at which polymer chains begin to move, leading to amorphous regions of the material transforming from a rigid, glassy state to a flexible, rubbery state, and vice versa. The combination of these two characteristics endows Nafion with fantastic shape memory properties.[4]

Shape memory properties allow a material to morph and transform into pre-programmed shapes when exposed to an external stimulus, such as heat, electricity, magnetic fields, moisture, chemical compositions, and radiation.[1,5] This property has primarily been found in alloys, dubbed shape memory alloys (SMAs), and polymers, dubbed shape memory polymers (SMPs), with SMAs being the most widely used due to their intrinsically strong mechanical properties.

The typical shape memory process of a SMP involves relying on its glass transition temperature (Tg). The polymer is heated to a temperature above Tg, thus becoming flexible, and then deformed into the desired shape by an external stress. This stress is held fixed as the

polymer cools below Tg, until transforming back into a rigid state. Once the polymer is rigid, the stress can be removed, and the polymer will remain in the desired shape, as the polymer retains the fixed strain. Finally, to return the polymer to its original shape, it is heated back above Tg without any applied stress, whereupon the strain relaxes, allowing the polymer to transform back. This process is known as a stress-free strain recovery.[1,4,5] Since the shape memory effect relies on Tg, the number of shapes that can be memorized by the SMP is restricted by the number of glass transition temperatures the material has. For instance, there exist shape memory polymers that are able to memorize three shapes, known as triple-shape memory polymers, by programming shapes above and between two well-separated glass transition temperatures. However, tuning this effect would only be possible through changing the material composition.[1]

However, Nafion's composition enhances its shape memory property beyond the abilities of standard SMPs and SMAs. Nafion's temperature-persistent electrostatic network enables enhanced, permanent shape memorization and stability. This permanent shape is memorized when the sample is first annealed and will be the shape the sample returns to during the stress-free strain recovery process. While PFSAs typically show two thermally reversible transitions, below 150 °C and above 240 °C, Nafion's broad glass transition (~55 °C to 130 °C) is equivalent to a near infinite number of transitions, with precise and minutely differing transition temperatures.[1] In the context of the shape memory effect, every transition is an individual memory element, which can be triggered on or off during deformation, leading to a tunable shape memory effect, and potentially near-infinite number of memorized shapes.[1] To this end, Nafion has been successfully tested to four distinct shapes across its broad glass transition temperature range.[1]

These abilities vastly expand the range of applications for Nafion, as many movements can be achieved in one cycle – lending applicability to uses such as deployables, actuators, sensors, and smart adhesives, particularly for in-space uses.

Objectives

The primary objective of this study is to provide a comprehensive account of how Nafion changes under different constraints. To achieve this goal, the following constraints, or testing variations, were focused on:

- Variations in temperature at deformation set and recovery.
- Variations in geometry, including length and volume.
- Variations in strain, including during annealing.
- Variations in laser processing, including pre- and post-annealing.

Calculation

The equations below were used to calculate important metrics pertaining to a sample's shape memory properties.

$$R_f = 100\% * \frac{\varepsilon}{\varepsilon_{load}} \quad (1)$$

Equation 1 calculates the shape fixity, or percentage of shape fixation, R_f, using the maximum strain under load, ε_{load}, and the fixed strain after cooling and stress removal, ε.

$$R_r = 100\% * \frac{\varepsilon - \varepsilon_{rec}}{\varepsilon} \quad (2)$$

Equation 2 calculates the most important metric for this study, the shape recovery percentage, R_r. This equation compares the strain after recovery, ε_{rec}, to the fixed strain after cooling and stress removal, ε.

Finally, **Equations 1** and **2** can be expanded to calculate shape fixity and shape recovery for multiple-shape memory effects.

$$R_f(X \rightarrow Y) = 100\% * \frac{\varepsilon_y - \varepsilon_x}{\varepsilon_{y,load} - \varepsilon_x} \quad (3)$$

$$R_r(X \rightarrow Y) = 100\% * \frac{\varepsilon_y - \varepsilon_{x,rec}}{\varepsilon_y - \varepsilon_x} \quad (4)$$

In these equations, X and Y denote two different shapes.[1]

EXPERIMENTAL METHODS

Sample Preparation

Initial samples were created using an alcohol-based 1000 equivalent weight at 20% weight Nafion dispersion. A reverse mold was fabricated, first by a filament 3D printer, then by a resin 3D printer. This reverse mold was filled with silicone and allowed to cure. This silicone mold was then filled with the dispersion and cured with air, making strips of Nafion. While this method is useful for further work, such as reinforcing Nafion with carbon fiber, it produced non-uniform samples that were inadequate for the nature of testing conducted.

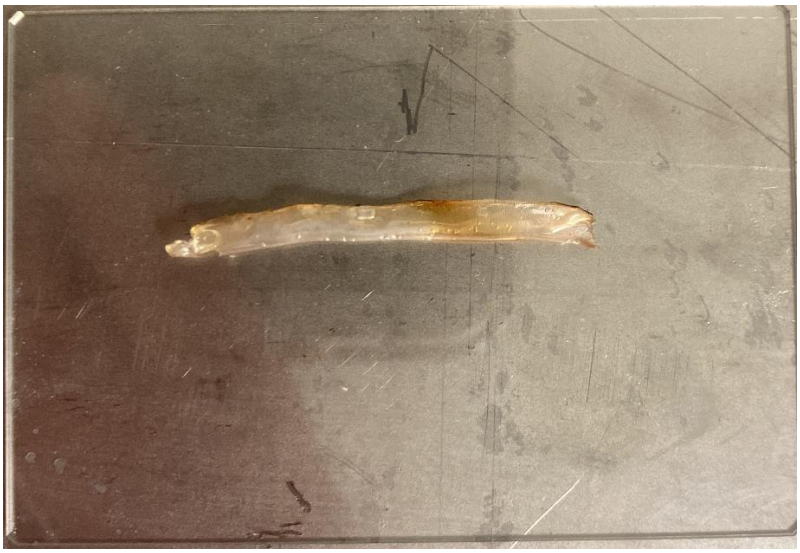

Figure 1: Non-uniform dispersion based Nafion sample.

Samples used during testing reported in this study are commercially bought, cured Nafion polymer sheets, with an equivalent weight of 1100 g/mol and a nominal thickness of 127 microns. All samples were annealed at 140 °C for 90 minutes before completing any stress-free strain recovery cycles.

Many tests were performed to find the best process of cutting uniform Nafion samples with minimal damage. Earlier tests utilized samples cut by hand.

Figure 2: Sheet based, hand cut Nafion sample.

After being unable to control uniformity and remain within dimension tolerance, samples were cut with a blue wavelength laser. However, the laser could not precisely cut the samples without either burning them or requiring a multitude of passes to cut through.

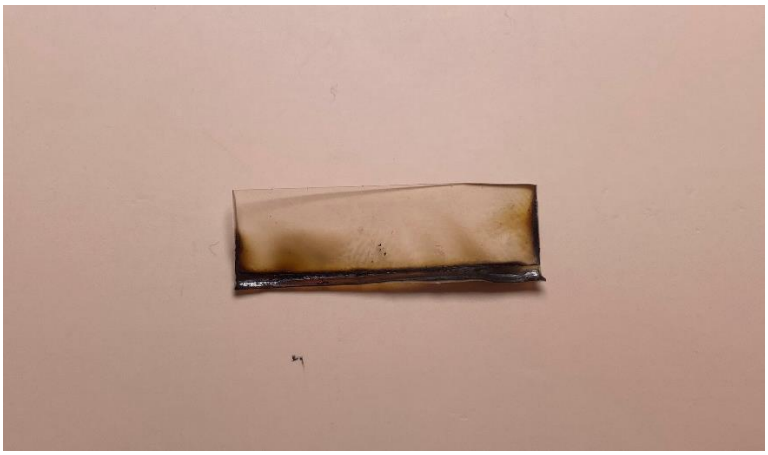

Figure 3: Sheet based, blue laser cut Nafion sample.

The best solution found was a CO2 laser. This method precisely cut each sample without causing any visible burn marks. This could be due to several different factors, including the larger wavelength of the CO2 laser (10.6 micron vs. 450-460 nm), which could allow better material absorption and penetration, or the more precise and focused beam produced by the CO2 laser, leading to more localized melting. Exact laser parameters are listed in the **Appendix**.

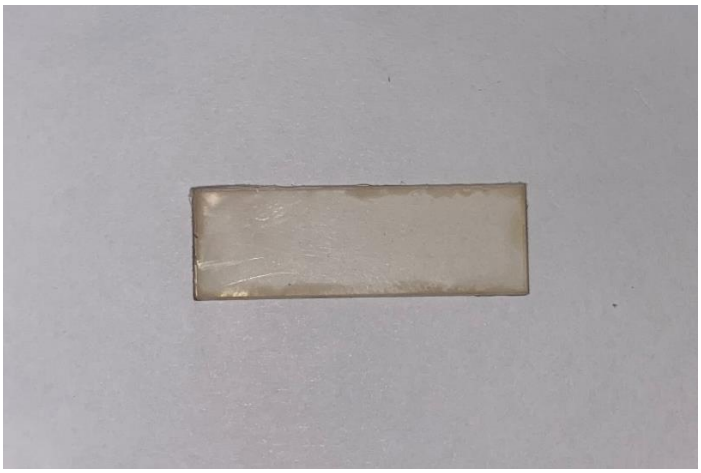

Figure 4: Sheet based, CO2 laser cut Nafion sample.

However, some samples showed fiber extraction during the laser cutting process. Samples showing defects like such were not used for further testing.

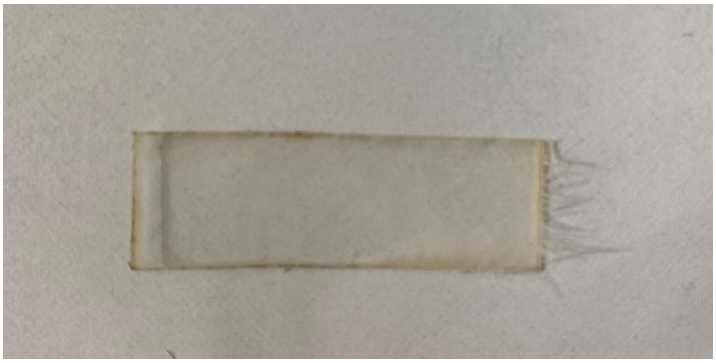

Figure 5: Sheet based, CO2 laser cut Nafion sample with fiber extraction.

While most samples did not show this level of fiber extraction, under a microscope, the internal fibers can be seen at the location of the laser cut.

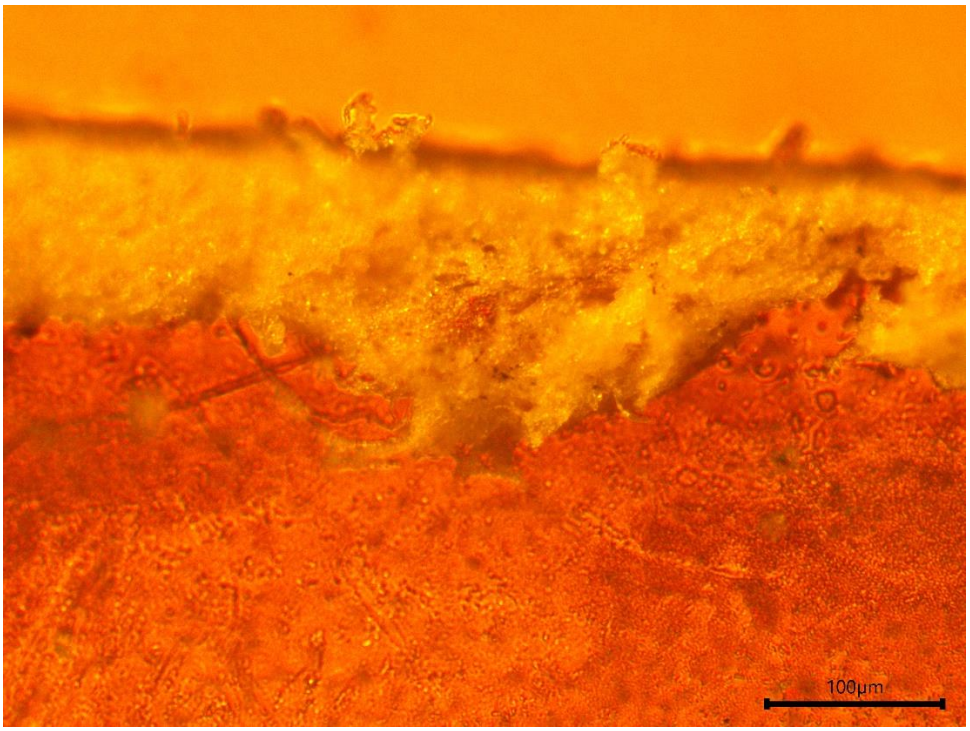

Figure 6: Fiber of sample at location of laser cut.

Heating Apparatus

All testing completed during this research involved applying heat to a sample. There was much work done in finding the best solution for a heating apparatus. Initially, a heating gun was used, modeling testing procedures after Herath.[5] However, this process resulted in a concentrated

heat source on the sample, instead of uniform heating, leading to the drastic temperature gradients across the sample.

Following this, a heating pad was utilized to provide a more uniform temperature across the sample. The issue found with this solution was that the direct heat applied to the sample tended to melt it to the surface. Furthermore, the heating pad did not provide a perfectly uniform temperature across the entire surface. Glass slides wrapped in Polyimide tape were used to mitigate the melting, but this caused a significant increase in the time needed to heat a sample.

For the next process, two translational plates were purchased in order to make the stretching process during testing more uniform. SolidWorks was used to model metal plates that could sit on the translational plates and hold a wire heating element. These plates were cut from aluminum blocks using a CNC machine. Finally, a thermocouple was embedded in the aluminum plates and a wire element was adhered to the surface, both of which were connected to an Arduino. Code was written in C++ to detect the temperature of the plate and control the wire heating element. The issue encountered with this method was the high thermal ramp caused the Arduino to cut power to the wire element for safety, and no solution could be found to modify the code to fix this issue.

However, there was not much time given to this issue because a thermal vacuum oven was purchased shortly after this issue arose. This oven proved to be the most uniform, easy-to-use method for the heating apparatus, and is what was used for all tests discussed in this paper.

Testing Procedure

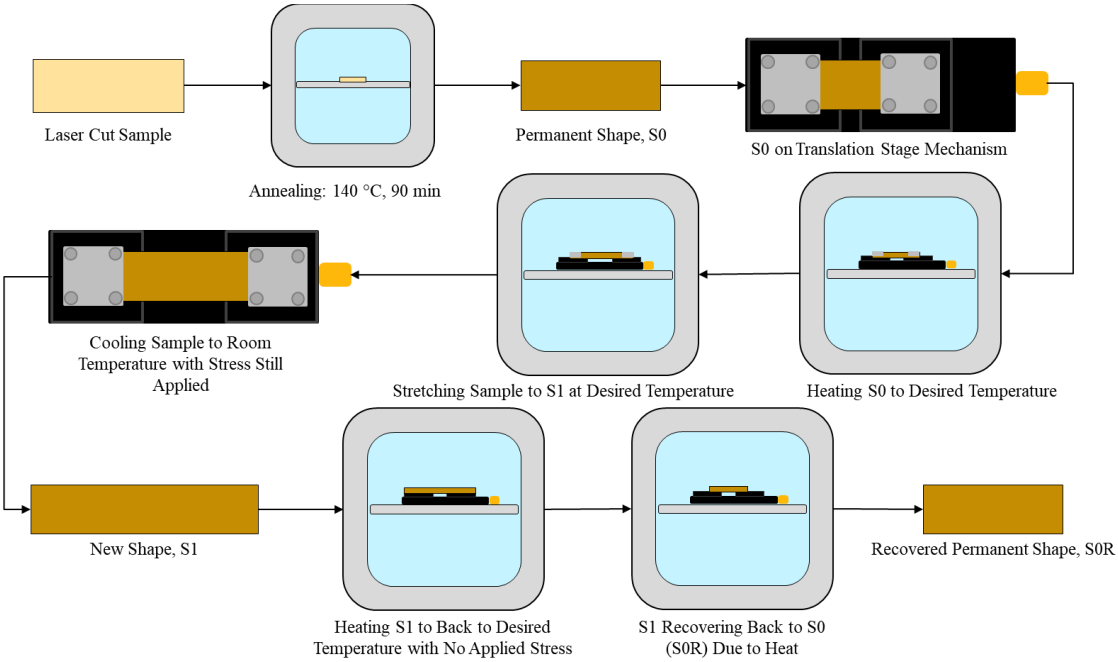

Figure 7: Schematic of standard testing procedure.

All tests conducted adhered to the following generalized procedure, known as the stress-free strain recovery procedure. First, the sample is placed in the oven at the starting temperature, typically 140 °C, under no stress for two minutes. Then, the sample is deformed, either stretched or bent, to the desired shape or strain, while remaining in the oven, for four minutes. After which, the sample is removed from the oven, with the deformation stress still applied, and allowed to sit in room temperature air for four minutes. Then, the deformation stress is removed. Finally, the sample is placed back in the oven at the same starting temperature under no stress, allowing it to recover to its original shape. Unless otherwise specified, samples were stretched to approximately 15% strain during the tests conducted in this paper. For any tests conducted that did not vary the geometry of the samples, the samples measured approximately 30 mm in length, 10 mm in width, and 0.15 mm in thickness before annealing.

For tests with multiple shape deformations, the same process is followed, however, instead of removing the sample from the oven after four minutes, allow the sample to cool in the oven to a lower deformation temperature, apply the new deformation stress for four minutes, and repeat for as many shapes as desired. Once all deformations have been completed, remove from the oven for four minutes and follow the previous procedure.

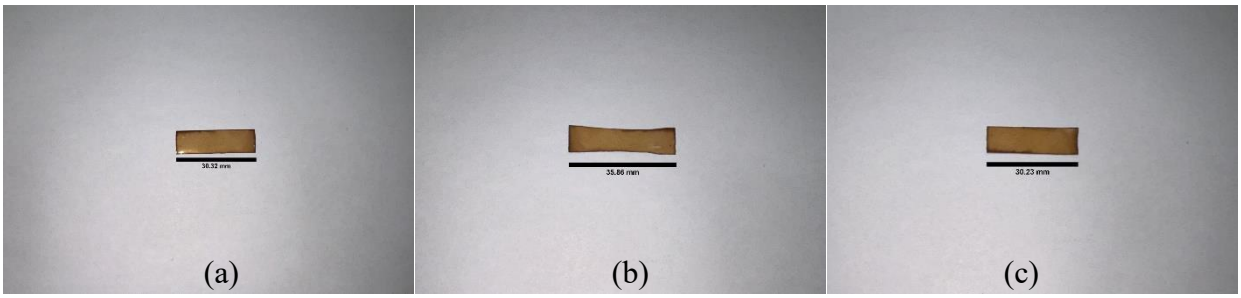

Figure 8: Example of shape memory behavior during a standard test cycle. (a) S0 – the permanent shape set during annealing, (b) S1 – the new shape set by application of stress and temperature, (c) S0R – the permanent shape recovered upon exposure to the same temperature S1 was set at.

Laser Processing

Extensive laser testing was also conducted on samples using a blue wavelength laser. Multiple factors were changed for these tests. Samples were scanned in pre- and post-annealing phases. Samples were also scanned in three different directions, horizontally, vertically, and diagonally. Finally, samples were scanned at a lower power, which created faint scan lines across the sample, and at a higher power, which burned in scan lines more severely. These processed samples were then tested under normal stress-free strain recovery procedures.

RESULTS & DISCUSSION

Annealing

While controlling the shape of the sample during annealing is difficult without applying stress to the sample, and the samples did not anneal in a uniform manner. However, despite the non-uniform annealing, the samples tended to remain within dimension tolerances after cooling.

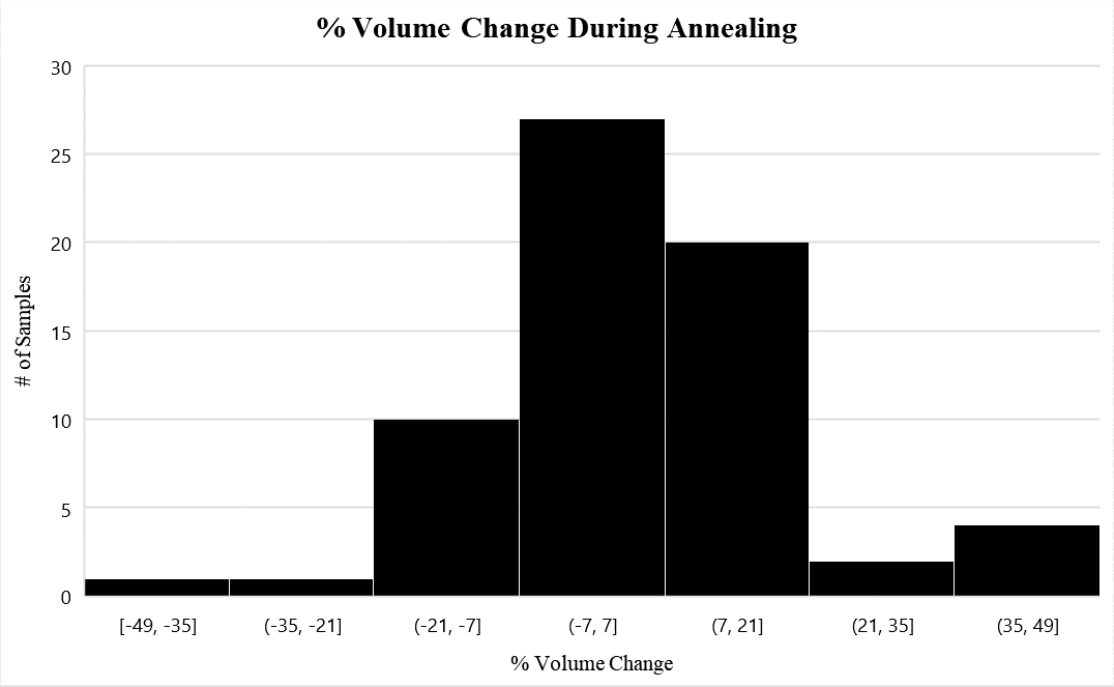

Figure 9: Percentage of volume change of samples during annealing.

Temperature Variation

Initial testing focused on finding the ideal temperature for shape memorization and recovery in the samples. From previously published data, it was known that Nafion can accurately memorize shapes at any temperature within its glass temperature range.[1] Tests were conducted within the glass temperature range at a 10 ˚C variation.

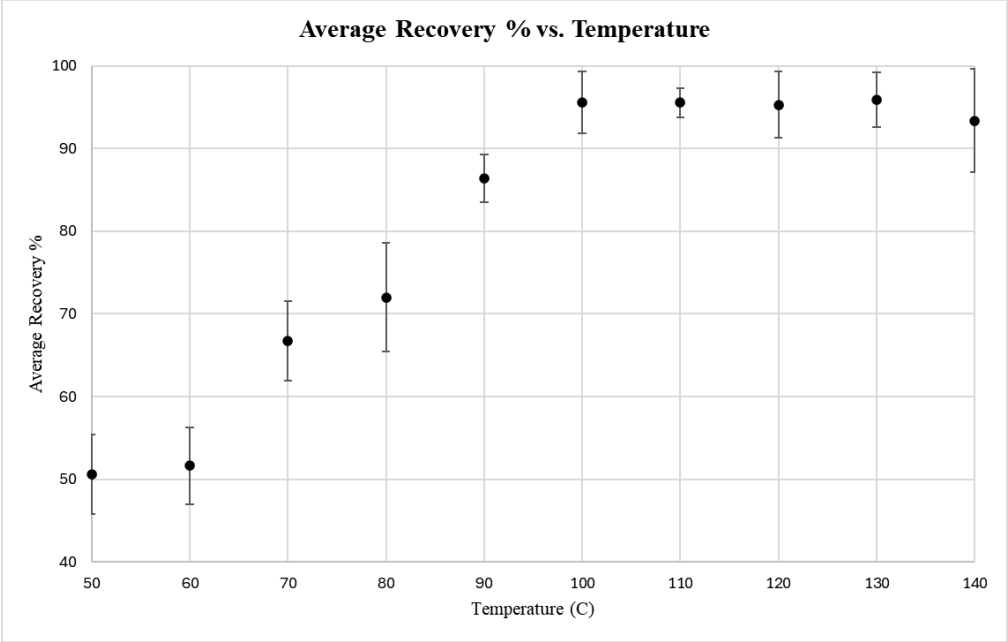

Figure 10: Average recovery percentage across the glass transition temperature range, with standard deviation.

From this data, it was determined that Nafion recovers best in the range between 100 ˚C and 140 ˚C. In this temperature range, Nafion can recover to a high percentage (>95%) in less than 1 minute. Below this temperature range, between 50 ˚C and 90 ˚C, Nafion can still recover to a high degree, however, the recovery process takes longer as temperature decreases.

Geometry Variation

Extensive testing was conducted with a variety of geometric variations in the samples. The first variation tested was a variation in the length of the samples, while maintaining width and thickness dimensions.

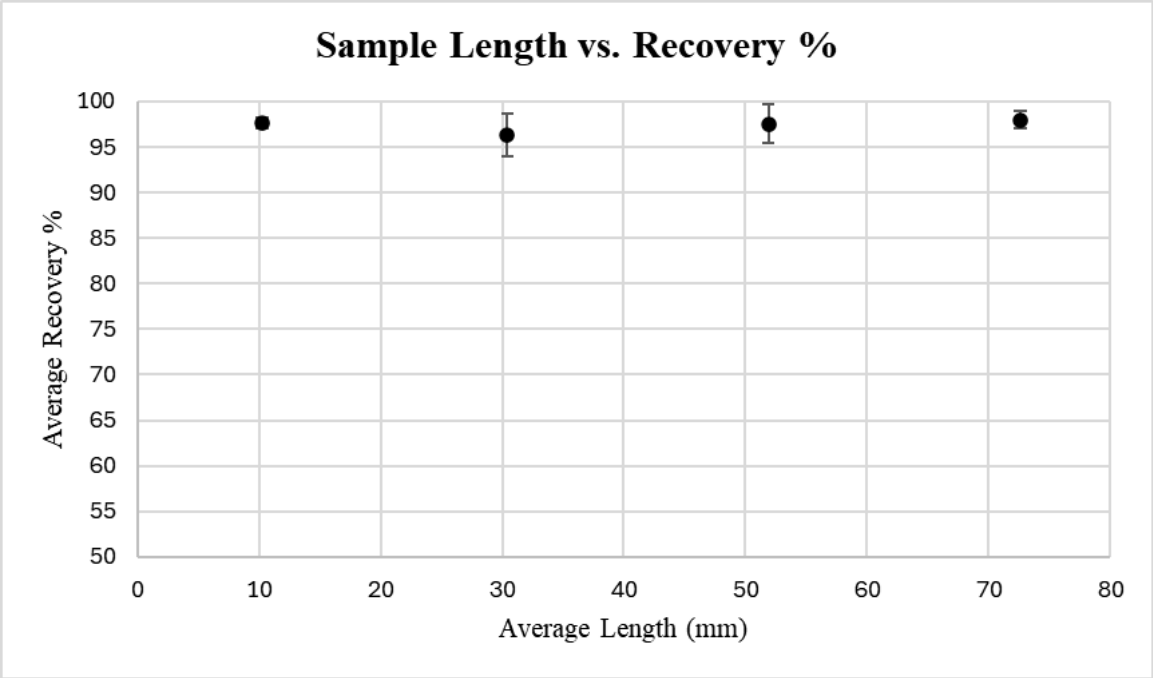

Figure 11: Average sample length vs. average recovery percentage for samples of uniform width and thickness, with standard deviation.

The data from these experiments indicates that recovery time and percentage is not affected by the length of a sample.

Similarly, changing the volume of a sample does not affect its ability to recover, as can be seen in **Figure 12**. For these tests, several sets of samples with unique geometry variations were prepared and tested using the standard procedure.

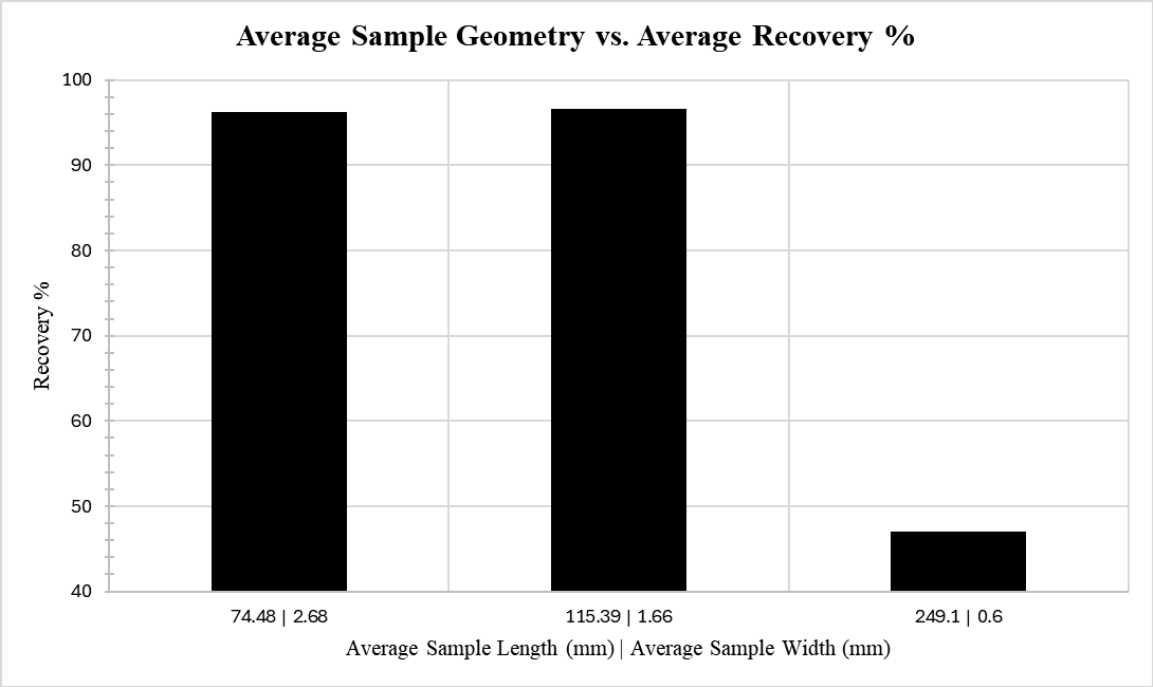

Figure 12: Average geometry of samples vs. average recovery percentage.

There were several samples that tore during elongation and one during measurement, which can be seen in the full data in the **Appendix**. This was due to the fragility of the thinner, longer samples. For the first two variations in geometry, samples still recovered to a high degree. The last set, the thinner, fiber-like samples, were susceptible to tearing during elongation, and, due to their length and small width, stretched too thin, significantly reducing the shape memory recovery percentage. They also had a unique tendency during recovery that was not apparent in other samples tested. The fiber-like samples tended to significantly curl into a spiral-like shape during the recovery process. Only one of these fiber-like samples recovered to a high degree.

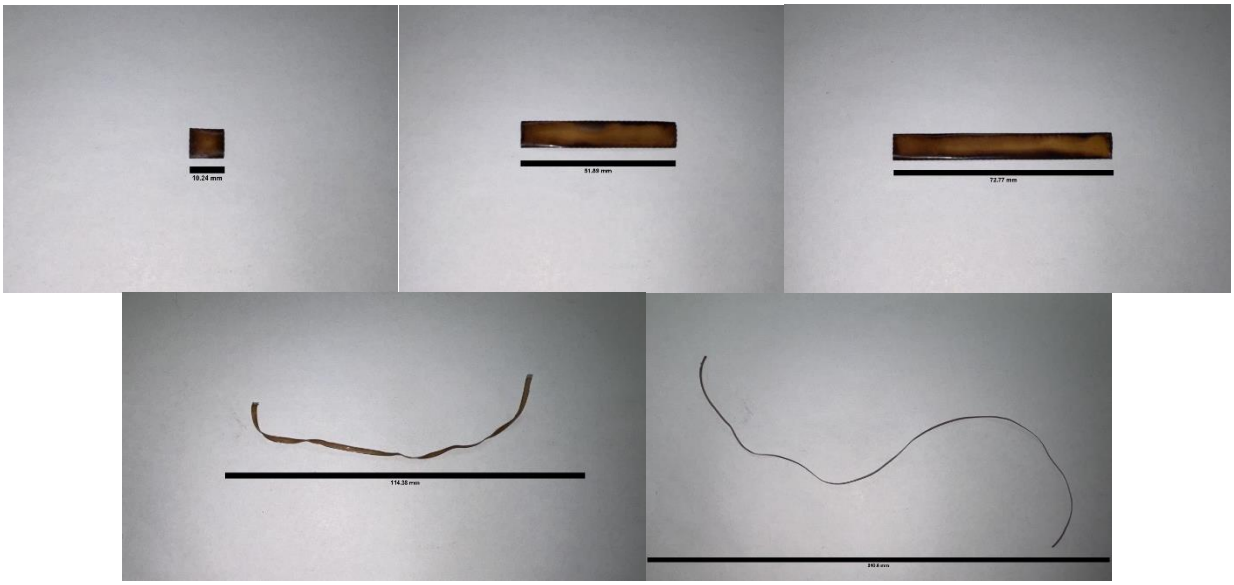

Figure 13: Collection of samples of various geometries.

Strain Variation

Another parameter tested was variation in the strain of a sample. While standard tests were conducted at 15% strain, these experiments tested 50%, 100%, and 120% strain.

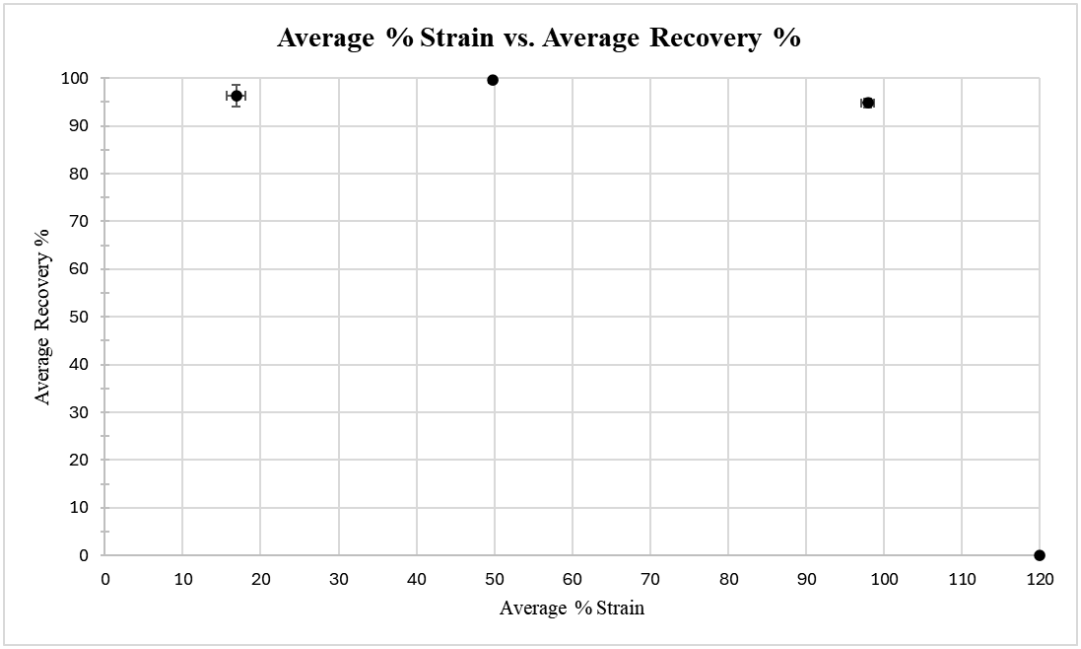

Figure 14: Average percent strain vs. average recovery percentage for samples of uniform geometry, with standard deviation.

From this data, the samples recovered to a high degree in under one minute regardless of the amount the sample was stretched, aside from the samples stretched to 120%. For this level of strain, the samples tore between the range of 115% - 120%, thus no results could be quantified.

Finally, one sample was stretched during its entire annealing time, and subsequently tested using normal procedures to quantify any changes applied stress during annealing would cause. Once initially put in the oven for testing, this sample shrunk to a size slightly longer than halfway between its original length and its post-annealing/stretching length. After completing a full testing cycle at this new length, it recovered to 99.35%. The data for this sample can be found in the **Appendix**.

Laser Processing

There were a range of laser tests conducted with the hypothesis that lightly processing a sample with a laser could realign the monomer chains, thus "tuning", or affecting, the shape memory abilities. The laser used in these tests was the blue wavelength laser originally utilized during sample preparation. During these tests, variable parameters included laser power [high (3 W)/low (0.5 W)], laser scanning direction (vertical/horizontal/diagonal), and when the sample was processed (pre-/post-anneal).

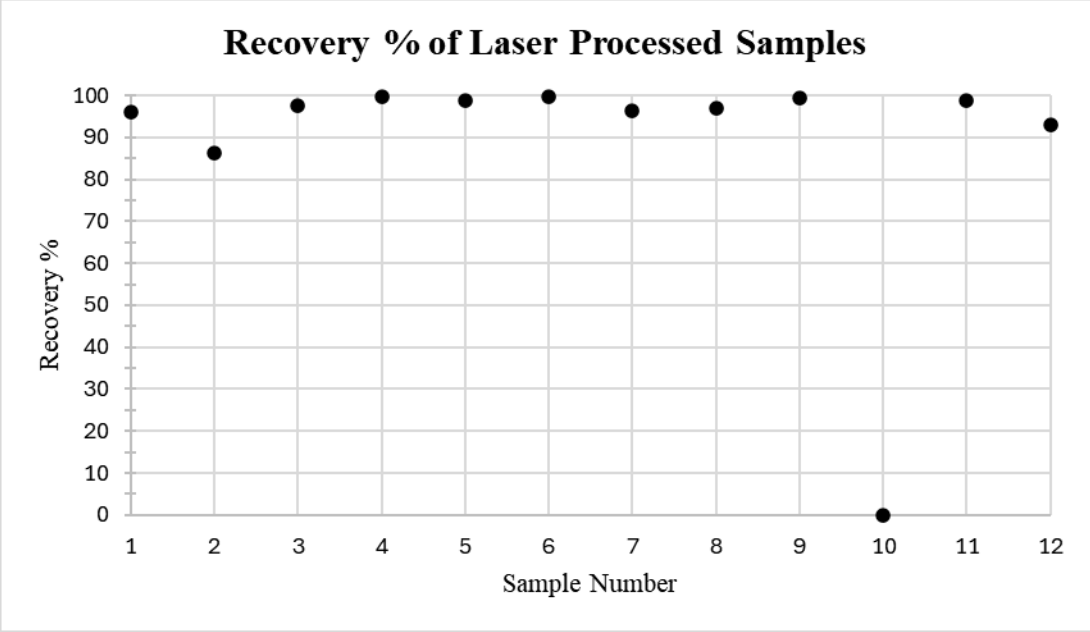

Figure 15: Recovery percentage of samples processed by the laser.

From **Figure 15**, laser processing, regardless of power, direction, or pre-/post-anneal, had no apparent effect on the shape memory behavior of the samples, as they all recovered normally. The sole diversion to this assessment is one sample which tore during elongation due to the higher laser power burning through and compromising the center of the sample. While all samples processed with the higher power laser setting had more prevalent burn marks, it appeared that samples processed before annealing had greater burn marks than those processed after annealing. Furthermore, it was more difficult to control the level of strain of laser processed samples compared to unprocessed samples.

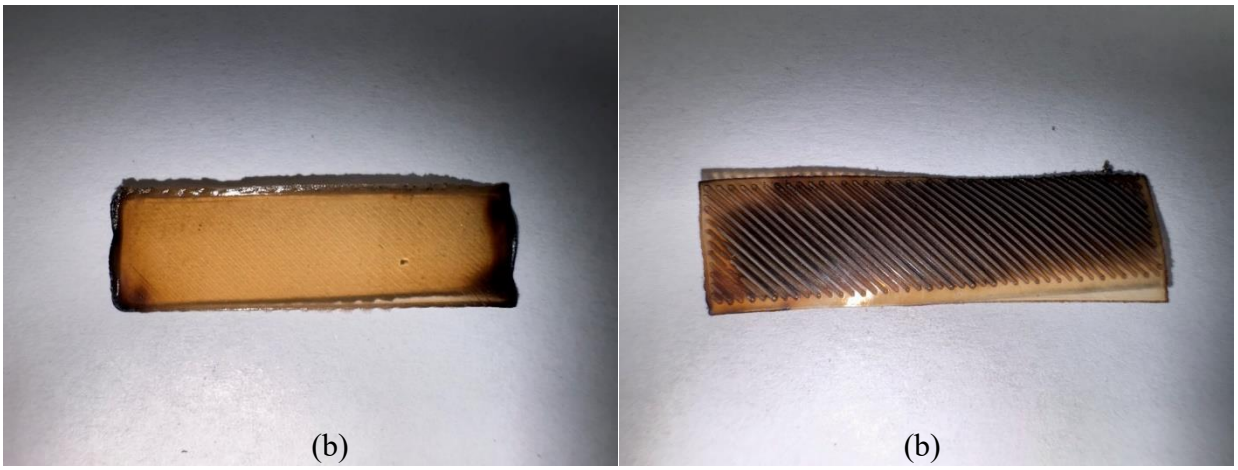

Figure 16: Example of diagonally laser scanned samples. (a) low power (0.5 W), (b) high power (3 W).

Samples that had been processed by the laser were analysed under a microscope, providing the following figures.

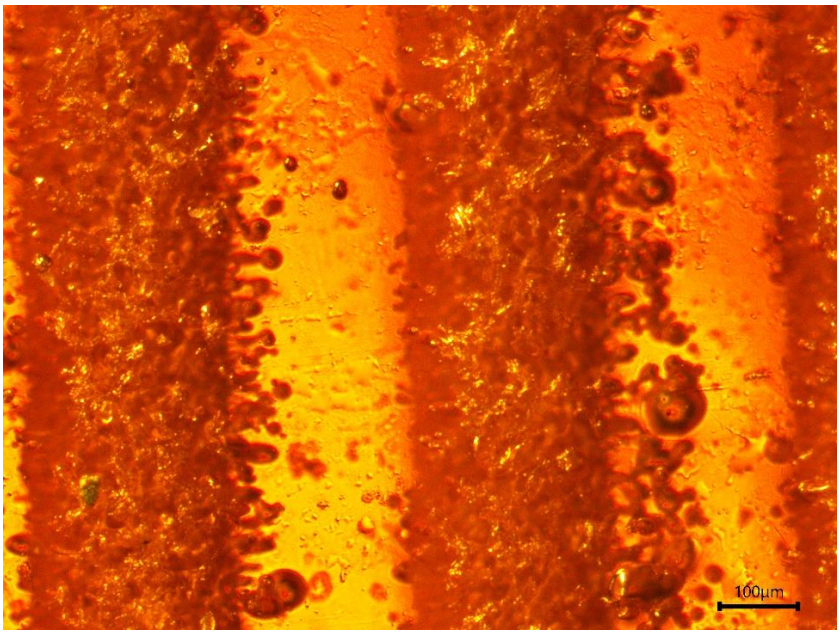

Figure 17: Sample processed by 3W laser scan. Amber-colored sections are untouched by the laser, while darker, charcoal-colored sections are the paths of the laser across the sample.

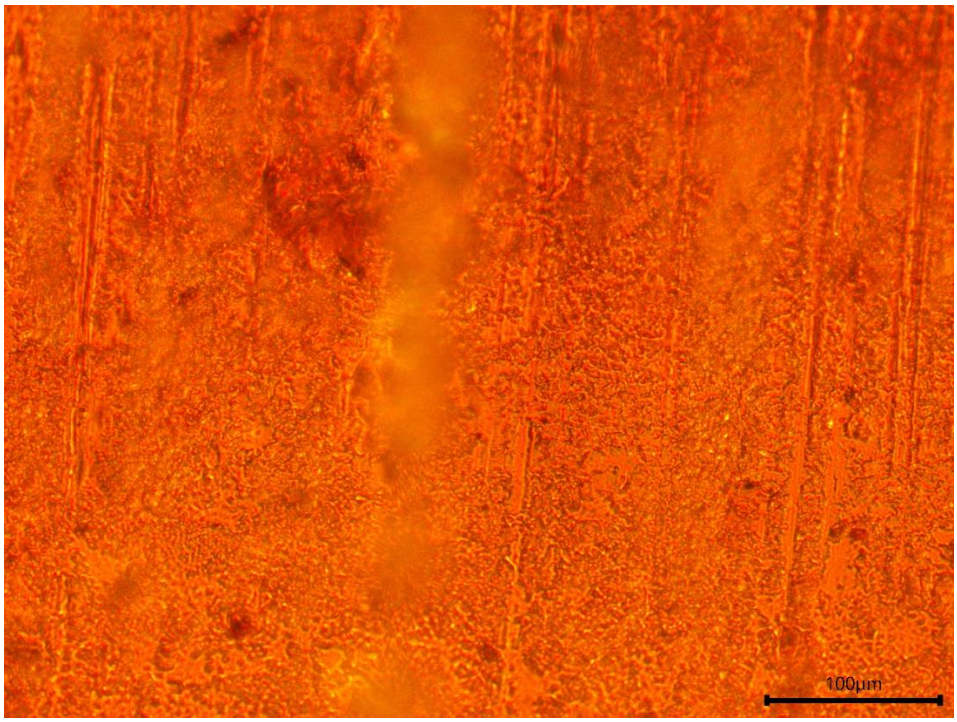

Figure 18: Sample processed by 0.5W laser scan. Laser scan lines are much fainter.

Figures 17 & **18** show the difference between the high and low-power laser scans. Despite the more significant burning on the high-power laser scans, both types of laser processed samples recovered to a high degree.

The volume of each sample before and after laser processing was also measured. Similarly to the volume changes during annealing, the samples did not change in a uniform manner. Rather, the volume changed by laser processing varied from approximately -12 to 12%, as can be seen in **Figure 19**.

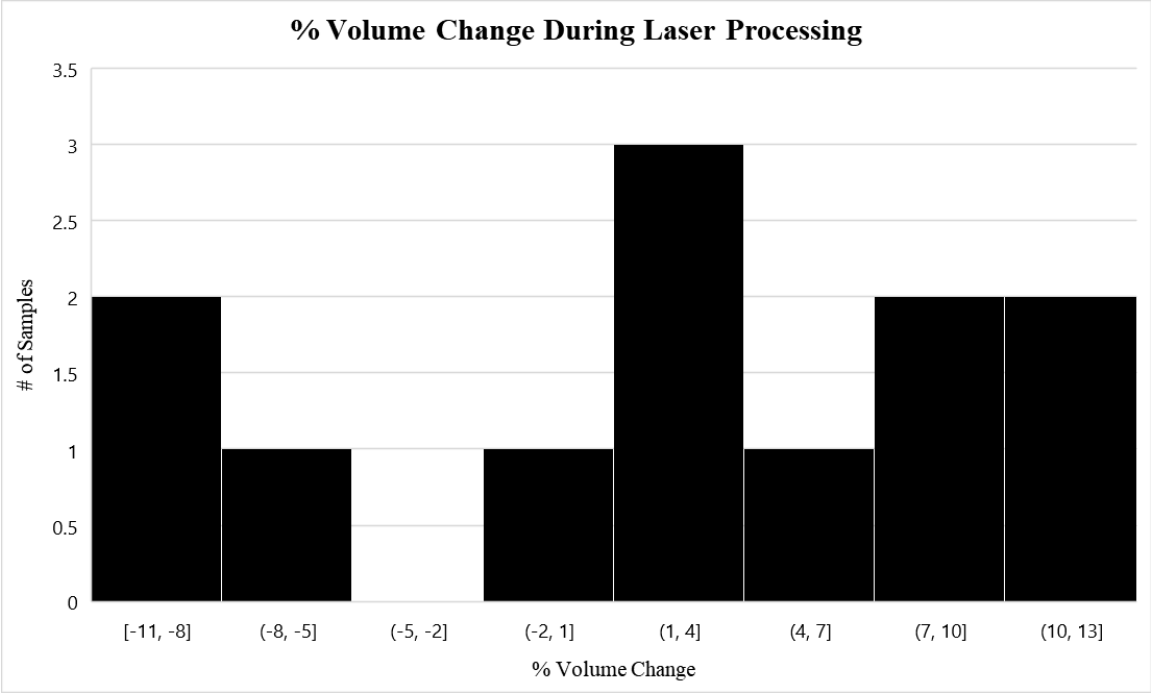

Figure 19: Percentage of volume change of samples during laser processing.

However, there was some unique variation in volume change based on what direction the laser scanned. This variation is plotted in **Figure 19**.

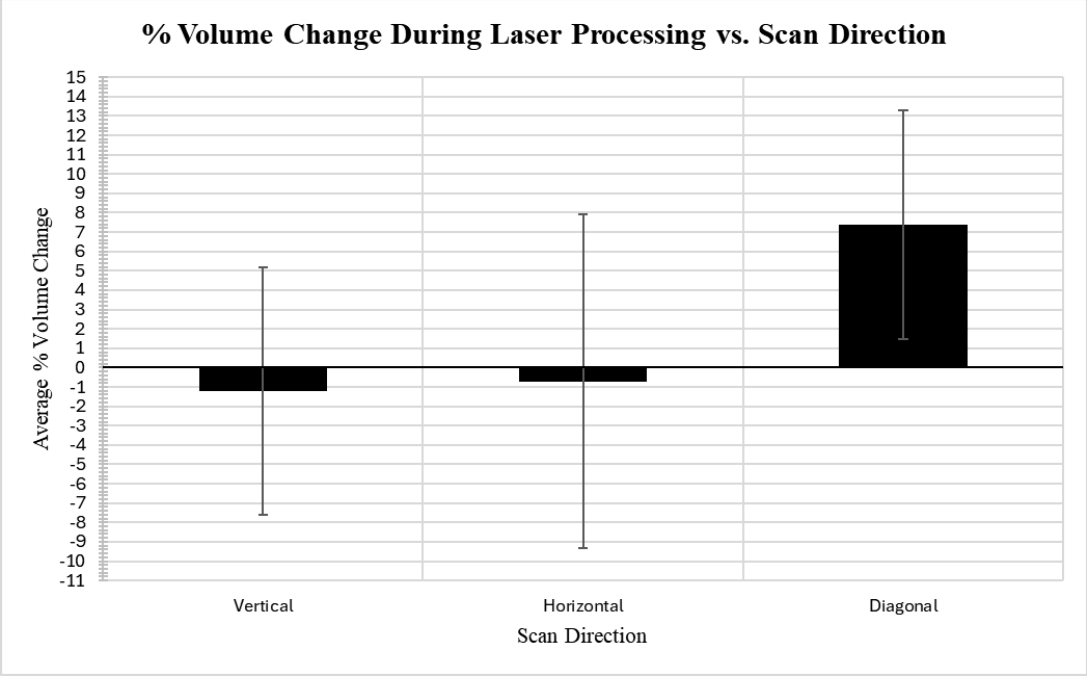

Figure 20: Average percentage of volume change during laser processing by scan direction, with standard deviation.

Every sample scanned diagonally by the laser gained volume, while those scanned vertically or horizontally were much more varied in their volume change. However, more significance cannot be drawn from this data without more extensive measurement, as there were several dynamic variables in the laser tests, including minute differences in the geometries of the samples. For instance, **Figure 21** displays how laser power could also influence whether samples gained or lost volume during processing.

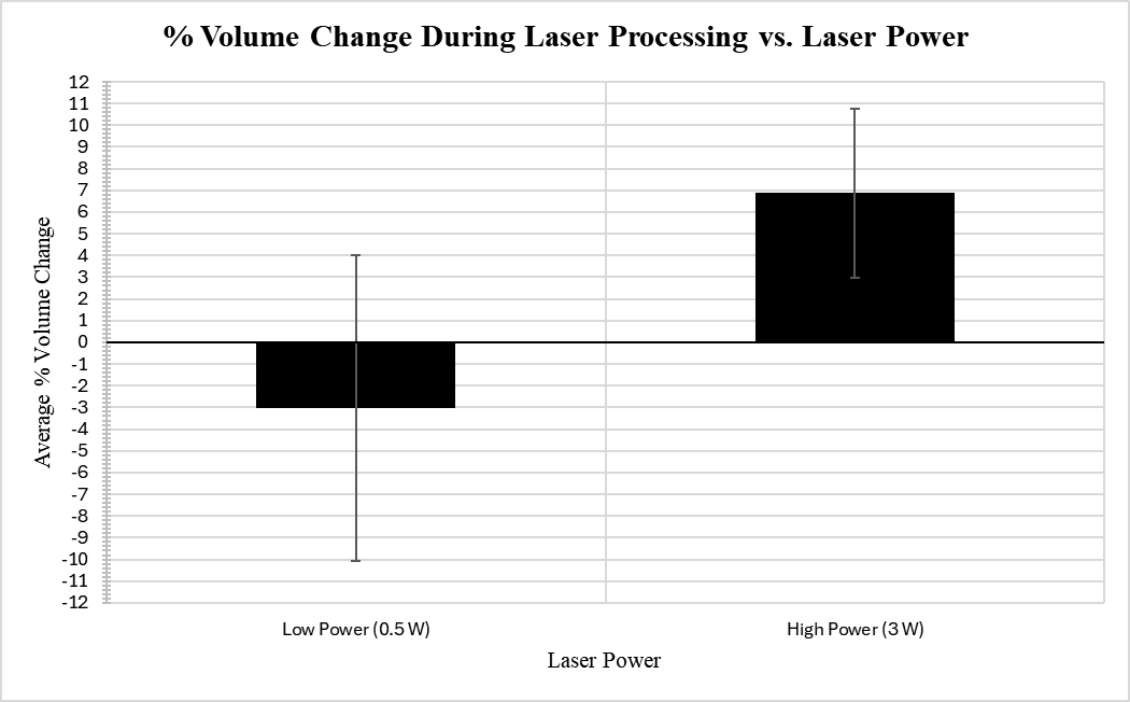

Figure 21: Average percentage of volume change during laser processing by laser power, with standard deviation.

CONCLUSIONS

While Nafion is primarily used for other purposes, it is a robust shape memory polymer. Nafion's shape memory property is affected by very little outside influence or geometry variations within its ideal recovery conditions. Nafion can recover at any temperature within its

glass temperature range (50 ˚C – 140 ˚C), but it recovers to the highest degree in the shortest amount of time at temperatures between 100 ˚C and 140 ˚C.

At 140 ˚C, Nafion's shape memory recovery percentage was not affected by length, unique geometries, strain, or laser processing. All sets of samples, aside from those tested at a low temperature, had an average recovery percentage above 90%.

One unique variation between samples was the volume changes during annealing and laser processing. For both scenarios, the percentage of volume change was different for each sample, but this may be due to minute, nonuniform difference between the geometries of the samples after annealing or laser processing.

FUTURE WORK

While this paper details the strength of Nafion's shape memory property, more extensive testing can be conducted to build a larger dataset from the data in this study. Further testing on Nafion in different environments and compositions, such as further laser testing and reinforcement with other materials to fabricate composites, would also improve the base of knowledge. Time constraints prevented testing the limits of Nafion's shape memorization, an important metric that sets Nafion apart from other shape memory polymers; current published research stops at 4 shapes, but Nafion can certainly memorize more. Finally, extensive testing of Nafion as a deployable or actuating structure or device would greatly benefit multiple industries, further establishing Nafion's veracity as a commercial deployable mechanism.

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APPENDIX

% Volume Change During Annealing:

	Pre-Anneal				Post-Anneal							
Sample Name	Length	Width	Thickness	Volume	Length	Width	Thickness	Volume	% Length Change	% Width Change	% Thickness Change	% Volume Change
S001	29.41	9.88	0.1524	44.28298992	29.94	8.95	0.127	34.031301	1.802108126	-9.412955466	-16.66666667	-23.15039915
S002	29.41	9.83	0.1397	40.38731191	30.12	8.92	0.152	40.8379008	2.414144849	-9.257375381	8.804581246	1.115669424
S003	29.51	9.8	0.127	36.728146	30.07	8.89	0.152	40.6329896	1.89766181	-9.285714286	19.68503937	10.63174711
S004	29.49	9.86	0.139	40.4172246	29.97	8.92	0.127	33.9512148	1.627670397	-9.53346856	-8.633093525	-15.99815392
S005	29.46	9.79	0.127	36.6285018	30.17	9.04	0.152	41.4559936	2.410047522	-7.660878447	19.68503937	13.17960485
S006	29.49	9.83	0.127	36.8156109	30.07	8.94	0.152	40.8615216	1.966768396	-9.053916582	19.68503937	10.98966064
S007	29.57	9.93	0.1397	41.02012497	30.49	8.89	0.12	32.526732	3.111261414	-10.47331319	-14.10164639	-20.70542929
S008	29.86	9.78	0.127	37.0879116	30.56	8.97	0.13	35.636016	2.344273275	-8.282208589	2.362204724	-3.91474078
S009	29.72	9.86	0.127	37.2159784	30.58	8.86	0.14	37.931432	2.893674293	-10.14198783	10.23622047	1.922436627
S010	29.79	9.88	0.127	37.3793004	30.49	8.91	0.12	32.599908	2.349781806	-9.817813765	-5.511811024	-12.78620078
S011	29.54	9.8	0.14	40.52888	30.35	8.86	0.13	34.95713	2.742044685	-9.591836735	-7.142857143	-13.74760418
S012	29.51	9.86	0.127	36.9530122	30.47	8.99	0.14	38.349542	3.253134531	-8.823529412	10.23622047	3.77920423
S013	29.79	9.8	0.15	43.7913	30.48	8.88	0.15	40.59936	2.316213494	-9.387755102	0	-7.288982058
S014	29.62	9.91	0.127	37.2788434	30.42	8.84	0.15	40.33692	2.700877785	-10.79717457	18.11023622	8.203249675
S015	29.57	9.87	0.14	40.859826	30.17	8.7	0.14	36.74706	2.029083531	-11.85410334	0	-10.06554947
S016	29.79	9.75	0.127	36.8874675	30.46	8.89	0.14	37.910516	2.249076871	-8.820512821	10.23622047	2.773431112
S017	29.69	9.91	0.127	37.3669433	30.65	8.95	0.15	41.147625	3.233411923	-9.687184662	18.11023622	10.11771734
S018	29.74	9.88	0.127	37.3165624	30.49	8.91	0.14	38.033226	2.521856086	-9.817813765	10.23622047	1.920497371
S019	30.05	9.91	0.127	37.8200285	30.58	8.9	0.13	35.38106	1.763727121	-10.19172553	2.362204724	-6.448880651
S020	29.51	9.93	0.15	43.955145	30.48	8.94	0.16	43.598592	3.287021349	-9.96978852	6.666666667	-0.811174665
S021	29.85	9.88	0.125	36.86475	30.78	9.1	0.15	42.0147	3.115577889	-7.894736842	20	13.96984925
S022	29.49	9.86	0.11	31.984854	30.55	8.92	0.14	38.15084	3.594438793	-9.53346856	27.27272727	19.27783069
S023	29.67	9.93	0.11	32.408541	30.75	9.05	0.16	44.526	3.640040445	-8.86203424	45.45454545	37.38970847
S024	29.54	9.75	0.125	36.001875	30.4	9.1	0.14	38.7296	2.911306703	-6.666666667	12	7.576619273
S025	29.36	9.68	0.125	35.5256	30.76	9.18	0.14	39.532752	4.768392371	-5.165289256	12	11.27961808
S026	29.64	9.88	0.1	29.28432	29	9.3	0.13	35.061	-2.159244265	-5.870445344	30	19.72618794
S027	29.62	9.94	0.11	32.386508	30.45	8.8	0.15	40.194	2.802160702	-11.46881288	36.36363636	24.10723626
S028	29.6	9.75	0.125	36.075	30.47	8.76	0.14	37.368408	2.939189189	-10.15384615	12	3.585330561
S029	29.74	9.88	0.125	36.7289	30.56	8.82	0.15	40.43088	2.757229321	-10.72874494	20	10.07920194
S030	29.62	9.74	0.1	28.84988	30.34	8.73	0.16	42.378912	2.430790007	-10.36960986	60	46.89458674
S031	29.82	9.75	0.125	36.343125	30.5	8.93	0.14	38.1311	2.280348759	-8.41025641	12	4.919706272
S032	29.82	9.83	0.11	32.244366	30.69	8.73	0.15	40.188555	2.91750503	-11.19023398	36.36363636	24.63744829
S033	29.57	9.7	0.125	35.853625	30.43	8.62	0.15	39.34599	2.908353061	-11.13402062	20	9.740618975
S034	29.59	9.86	0.14	40.846036	30.61	8.72	0.14	37.368688	3.44711051	-11.56186613	0	-8.513305918
S035	29.59	9.73	0.125	35.9888375	30.58	9.05	0.14	38.74486	3.345724907	-6.988694758	12	7.657992565
S036	29.87	9.9	0.14	41.39982	30.48	8.66	0.08	21.116544	2.042182792	-12.52525253	-42.85714286	-48.99363331
S037	29.34	9.34	0.12	32.884272	29.95	8.22	0.14	34.46646	2.079072938	-11.99143469	16.66666667	4.811382171
S038	29.72	9.85	0.11	32.20162	30.32	8.88	0.14	37.693824	2.01884253	-9.847715736	27.27272727	17.05567608
S039	29.9	9.83	0.13	38.20921	30.8	8.85	0.14	38.1612	3.010033445	-9.96948118	7.692307692	-0.125650334
S040	29.78	9.81	0.11	32.135598	30.6	8.81	0.14	37.74204	2.753525856	-10.19367992	27.27272727	17.44620405
S041	29.68	9.75	0.11	31.8318	30.5	8.91	0.14	38.0457	2.762803235	-8.615384615	27.27272727	19.52104499
S042	29.7	9.78	0.1	29.0466	30.79	8.9	0.15	41.10465	3.67003367	-8.99795501	50	41.51277602
S043	29.2	9.36	0.12	32.79744	29.94	8.44	0.14	35.377104	2.534246575	-9.829059829	16.66666667	7.865443157
S044	28.61	9.1	0.12	31.24212	29.41	8.46	0.14	34.833204	2.796225096	-7.032967033	16.66666667	11.49436722
S045	29.82	9.49	0.1	28.29918	30.75	8.61	0.15	39.713625	3.118712274	-9.272918862	50	40.33489663
S046	28.4	9.13	0.12	31.11504	29.3	8.18	0.15	35.9511	3.169014085	-10.40525739	25	15.54251577
S047	28.56	9.63	0.13	35.754264	29.52	8.95	0.14	36.98856	3.361344538	-7.061266874	7.692307692	3.452164475
S048	30.08	9.83	0.14	41.396096	30.92	8.74	0.13	35.131304	2.792553191	-11.08850458	-7.142857143	-15.13377493
S049	28.89	9.66	0.12	33.489288	29.49	8.68	0.13	33.276516	2.076843198	-10.14492754	8.333333333	-0.635343457
S050	9.74	9.76	0.14	13.308736	10.06	8.91	0.14	12.548844	3.285420945	-8.709016393	0	-5.709723298
S051	9.75	9.7	0.14	13.2405	9.99	8.71	0.15	13.051935	2.461538462	-10.20618557	7.142857143	-1.424153166
S052	9.73	9.74	0.14	13.267828	10.08	9	0.14	12.7008	3.597122302	-7.597535934	0	-4.273706292
S053	49.48	9.68	0.14	67.055296	51.29	8.82	0.14	63.332892	3.658043654	-8.884297521	0	-5.551245348
S054	49.42	9.94	0.14	68.772872	51.3	8.68	0.15	66.7926	3.804127883	-12.67605634	7.142857143	-2.879437695
S055	49.62	9.78	0.15	72.79254	51.68	8.99	0.15	69.69048	4.151551794	-8.077709611	0	-4.261508116
S056	69.42	9.75	0.13	87.98985	71.97	8.71	0.14	87.760218	3.673292999	-10.66666667	7.692307692	-0.260975556
S057	69.32	9.68	0.13	87.232288	71.96	9.93	0.14	100.038792	3.808424697	2.582644628	7.692307692	14.68092182
S058	69.53	9.62	0.14	93.643004	72.25	8.75	0.14	88.50625	3.91198044	-9.043659044	0	-5.485464776
S059	29.33	9.89	0.12	34.808844	30.13	9.05	0.13	35.447945	2.72758268	-8.493427705	8.333333333	1.836030521
S060	29.77	9.85	0.12	35.18814	30.14	8.91	0.12	32.225688	1.242861942	-9.543147208	0	-8.418893411
S061	29.8	9.83	0.13	38.08142	30.22	8.88	0.12	32.202432	1.409395973	-9.664292981	-7.692307692	-15.43794323
S062	29.76	9.36	0.13	36.211968	29.5	9.05	0.13	34.70675	-0.873655914	-3.311965812	0	-4.156686541
S063	29.39	8.37	0.13	31.979259	29.15	8.18	0.13	30.99811	-0.816604287	-2.270011947	0	-3.06807922
S064	29.94	8.58	0.14	35.963928	29.49	8.42	0.14	34.762812	-1.503006012	-1.864801865	0	-3.339779793
S066	29.95	9.92	0.12	35.65248	30.44	9.05	0.13	35.81266	1.6360601	-8.77016129	8.333333333	0.449281509
								Averages	**2.538035904**	**-8.954336619**	**11.21531415**	**3.829099058**

Average Recovery % vs. Temperature:

		Sample Parameters (mm)			Test Parameters					Averages	
Test Number	Sample Number	Length	Width	Thickness	Temperature (C)	Recovery Time (min:sec)	% Elongation	Recovery %	Temperature (C)	Average Recovery %	StDev
T001	S001	30.48	9.14	0.13	130	1:00	15	46.17	140	14.75428571	6.222424118
T002	S002	30.53	9.04	0.15	130	1:00	15.23	92.88	130	14.762	3.306738574
T003	S003	30.75	9.07	0.14	130	1:00	14.82	94.07	120	14.35	4.023975232
T004	S004	30.56	9.02	0.14	140	1:00	16.38	97.47	110	14.18833333	1.786975098
T005	S005	31.04	9.35	0.11	140	1:00	14.28	83.63	100	14.21666667	3.755036174
T006	S006	30.91	9.42	0.13	140	1:00	14.02	87.69	90	13.69333333	2.876655465
T007	S007	30.49	8.89	0.12	140	1:00	13.55	95.88	80	16.85333333	6.602079723
T008	S008	30.56	8.97	0.13	120	1:00	13.55	89.13	70	16.12	4.831277264
T009	S009	30.58	8.86	0.14	120	1:00	15.6	96.02	60	14.87333333	4.659405184
T010	S010	30.49	8.91	0.12	120	1:00	15.12	99.57	50	15.90666667	4.786357348
T011	S011	30.5	8.98	0.13	110	1:00	13.21	99.26			
T012	S012	30.44	9.06	0.14	110	1:00	13.8	97.62			
T013	S013	30.54	8.96	0.14	110	1:00	16.08	97.76			
T014	S014	30.49	8.96	0.12	100	1:00	12.66	99.48			
T015	S015	30.45	8.88	0.13	100	1:00	16.26	91.31			
T016	S016	30.78	8.99	0.13	100	1:00	14.07	96.07			
T017	S017	30.63	8.85	0.14	90	1:00	14.33	89.76			
T018	S018	30.46	8.89	0.13	90	1:00	14.97	84.87			
T019	S019	30.84	9.04	0.13	90	1:00	13.81	84.51			
T020	S020	30.4	8.9	0.13	80	1:00	14.08	72.66			
T021	S021	30.82	9.03	0.13	80	1:00	16	69.37			
T022	S022	30.59	8.97	0.13	80	1:00	13.21	74.01			
T023	S023	30.65	8.91	0.13	70	1:00	14.29	71			
T024	S024	30.45	8.89	0.13	70	1:00	13.33	61.98			
T025	S025	30.73	9.17	0.14	70	1:00	14.22	67.28			
T026	S026	29.02	9.35	0.14	60	1:00	15.58	51.33			
T027	S027	30.55	9.04	0.14	60	1:00	13.69	54.78			
T028	S028	30.5	8.94	0.13	60	1:00	13.38	48.77			
T029	S029	30.6	8.89	0.13	50	1:00	12.97	54.16			
T030	S030	30.54	8.81	0.14	50	1:00	14.05	49.65			
T031	S031	30.66	8.85	0.13	50	1:00	14.06	48.03			
T032	S032	30.38	8.78	0.13	140	1:00	15.27	97.63			
T033	S033	30.32	8.81	0.13	140	1:00	18.27	98.38			
T034	S034	30.55	9.04	0.14	140	1:00	17.02	93.08			
T035	S035	30.57	9.1	0.13	130	1:00	15.08	95.88			
T036	S036	30.79	8.81	0.14	130	1:00	15.62	99.79			
T037	S038	31.08	8.81	0.13	130	1:00	17.66	97.09			
T038	S039	31.03	9.03	0.13	120	1:00	15.53	95.44			
T039	S040	30.82	9.02	0.13	120	1:00	15.02	96.98			
T040	S041	31.05	9.11	0.14	120	1:00	14.07	94.74			
T041	S042	30.91	9.08	0.14	110	1:00	16.63	91.25			
T042	S043	30.33	8.65	0.14	110	1:00	16.68	94.47			
T043	S044	29.78	8.76	0.14	110	1:00	14.41	93.01			

Sample Length vs. Recovery %:

		Sample Parameters (mm)			Test Parameters				Averages			
Test Number	Sample Number	Length	Width	Thickness	Temperature (C)	Recovery Time (min:sec)	% Elongation	Recovery %	Average Length	Average Recovery	StDev - L	StDev - R
T044	S050	10.2	9.1	0.14	140	1:00	22.52	98.42				
T045	S051	10.2616	8.9916	0.13	140	1:00	17.08	97.1	10.23386667	97.63	0.025519	0.569386
T046	S052	10.24	9.09	0.15	140	1:00	18.86	97.37				
T032	S032	30.38	8.78	0.13	140	1:00	15.27	97.63				
T033	S033	30.32	8.81	0.13	140	1:00	18.27	98.38	30.41666667	96.36333333	0.097411	2.34177
T034	S034	30.55	9.04	0.14	140	1:00	17.02	93.08				
T047	S053	51.92	8.97	0.146	140	1:00	15.41	94.6				
T048	S054	51.89	9.17	0.146	140	1:00	15.64	98.75	51.89333333	97.56666667	0.020548	2.112003
T049	S055	51.87	9.18	0.15	140	1:00	15.08	99.35				
T050	S056	72.48	9.19	0.146	140	1:00	15.37	96.69				
T051	S057	72.64	8.95	0.146	140	1:00	15.4	98.18	72.63	97.95666667	0.118603	0.956185
T052	S058	72.77	8.99	0.14	140	1:00	18.32	99				

Average Sample Geometry vs. Average Recovery %:

		Sample Parameters (mm)			Test Parameters				Averages		
Test Number	Sample Number	Length	Width	Thickness	Temperature (C)	Recovery Time (min:sec)	% Elongation	Recovery %	Average Length	Average Thickness	Avg Recovery %
T075	S087	74.73	2.64	0.1	140	1:00	15.66	96.07			
T076	S088	73.86	2.59	0.09	140	1:00	17.36	99.22	74.48	2.68	96.26
T077	S089	74.84	2.82	0.09	140	1:00	14.98	93.49			
T078	S090	115.13	1.69	0.1	140	1:00	14.63	95.49			
T079	S091	114.38	1.44	0.08	140	1:00	14.98	97.66	115.39	1.66	96.575
T080	S092	116.65	1.84	0.11	--	--	--	--			
T081	S093	240.6	0.58	0.08	140	1:00	14.21	98.25			
T082	S094	241.5	0.43	0.06	--	--	--	--			
T083	S095	254.5	0.82	0.1	140	1:00	14.34	21.92	241.05	0.51	47.035
T084	S096	251.5	0.69	0.11	140	1:00	11.13	35.71			
T085	S097	257.5	0.48	0.09	140	1:00	12.04	32.26			

Average % Strain vs. Average Recovery %:

Sample Parameters (mm)					Test Parameters				Averages			
Test Number	Sample Number	Length	Width	Thickness	Temperature (C)	Recovery Time (min:sec)	% Elongation	Recovery %	Average Elongation %	Average Recovery %	StDev - E	StDev - R
T032	S032	30.38	8.78	0.13	140	1:00	15.27	97.63				
T033	S033	30.32	8.81	0.13	140	1:00	18.27	98.38	16.85333333	96.36333333	1.230401922	2.34177046
T034	S034	30.55	9.04	0.14	140	1:00	17.02	93.08				
T066E	S070	30.86	9.15	0.14	140	1:00	50.32	99.48				
T067E	S071	30.65	9.21	0.14	140	1:00	49.3	99.8	49.75	99.71666667	0.424970587	0.169771088
T068E	S072	30.71	9.27	0.14	140	1:00	49.63	99.87				
T069E	S073	30.81	9.37	0.15	140	1:00	97.14	93.52				
T070E	S074	30.77	9.13	0.15	140	1:00	99.06	95.24	97.90333333	94.81333333	0.831718435	0.931999046
T071E	S075	30.89	9.31	0.15	140	1:00	97.51	95.68				
T072E	S076	31.12	9.28	0.14	140	1:00	~120	---	120	0	0	0
T073E	S077	30.85	9.25	0.15	140	1:00	~120	---				

Recovery % of Laser Processed Samples:

Sample Parameters (mm)					Test Parameters				Laser Parameters				
Test Number	Sample Number	Length	Width	Thickness	Temperature (C)	Recovery Time (min:sec)	% Elongation	Recovery %	Direction	Power %	Stage Scanned	Speed (mm/min)	Spacing (mm)
T053L	S045	31.33	8.85	0.13	140	1:00	16.18	96.06	Vertical	5	Post-Anneal	1500	0.5
T054L	S046	29.73	8.25	0.13	140	1:00	14.73	86.3	Horizontal	5	Post-Anneal	1500	0.5
T055L	S047	29.76	9.1	0.14	140	1:00	19.09	97.71	Diagonal	5	Post-Anneal	1500	0.5
T056L	S048	30.77	8.64	0.14	140	1:00	17.94	99.82	Vertical	30	Post-Anneal	1500	0.5
T057L	S049	30.18	8.81	0.14	140	1:00	22.13	98.95	Horizontal	30	Post-Anneal	1500	0.5
T058L	S059	30.13	9.05	0.13	140	1:00	19.08	99.83	Vertical	5	Pre-Anneal	1500	0.5
T059L	S060	30.14	8.91	0.12	140	1:00	18.61	96.43	Horizontal	5	Pre-Anneal	1500	0.5
T060L	S061	30.22	8.88	0.12	140	1:00	19.39	96.93	Diagonal	5	Pre-Anneal	1500	0.5
T061L	S062	29.5	9.05	0.13	140	1:00	18.54	99.45	Vertical	30	Pre-Anneal	1500	0.5
T062L	S063	29.15	8.18	0.13	140	--	--	--	Horizontal	30	Pre-Anneal	1500	0.5
T063L	S064	29.49	8.42	0.14	140	1:00	19.67	98.79	Diagonal	30	Pre-Anneal	1500	0.5
T065L	S066	31.19	8.82	0.14	140	1:00	15.84	93.12	Diagonal	30	Post-Anneal	1500	0.5

% Volume Change During Laser Processing, with Scan Direction & Laser Power:

Pre-Laser					Post-Laser												
Sample Name	Length	Width	Thickness	Volume	Length	Width	Thickness	Volume	% Length Change	% Width Change	% Thickness Change	% Volume Change	Direction	Power %	Stage Scanned		Average	StDev
S045	30.75	8.61	0.15	39.713625	31.33	8.85	0.13	36.045165	1.886178862	2.787456446	-13.33333333	-9.237283174	Vertical	5	Post-Anneal	**Vertical**	-0.52902011	5.543853065
S046	29.3	8.18	0.15	35.9511	29.73	8.25	0.13	31.885425	1.467576792	0.855745721	-13.33333333	-11.30890293	Horizontal	5	Post-Anneal	**Horizontal**	-1.2360651	9.175750265
S047	29.52	8.95	0.14	36.98856	29.76	9.1	0.14	37.91424	0.81300813	1.675977654	0	2.502611618	Diagonal	5	Post-Anneal	**Diagonal**	7.393259176	3.205037947
S048	30.92	8.74	0.13	35.131304	30.77	8.64	0.14	37.219392	-0.485122898	-1.14416476	7.692307692	5.94366779	Vertical	30	Post-Anneal		Avg	StDev
S049	29.49	8.68	0.13	33.276516	30.18	8.81	0.14	37.224012	2.339776195	1.497695853	7.692307692	11.86270822	Horizontal	30	Post-Anneal	**Low Power (5%)**	-3.03078991	7.025604704
S059	29.33	9.89	0.12	34.808844	29.33	9.89	0.12	34.808844	0	0	0	0	Vertical	5	Pre-Anneal	**High Power (30%)**	6.871075902	3.908263824
S060	29.87	9.87	0.13	38.326197	29.77	9.85	0.12	35.18814	-0.334784064	-0.202634245	-7.692307692	-8.187759928	Horizontal	5	Pre-Anneal			
S061	29.94	9.81	0.12	35.245368	29.8	9.83	0.13	38.08142	-0.46760187	0.203873598	8.333333333	8.046594945	Diagonal	5	Pre-Anneal			
S062	29.7	9.99	0.12	35.60436	29.76	9.36	0.13	36.211968	0.202020202	-6.306306306	8.333333333	1.70655504	Vertical	30	Pre-Anneal			
S063	29.39	8.83	0.12	31.141644	29.39	8.37	0.13	31.979259	0	-5.209513024	8.333333333	2.689694224	Horizontal	30	Pre-Anneal			
S064	29.97	8.97	0.12	32.259708	29.94	8.58	0.14	35.963928	-0.1001001	-4.347826087	16.66666667	11.48249699	Diagonal	30	Pre-Anneal			
S065	30.44	9.05	0.13	35.81266	31.19	8.82	0.14	38.513412	2.463863338	-2.541436464	7.692307692	7.541333149	Diagonal	30	Post-Anneal			
								Averages	**0.251913929**	**-2.629120361**	**5.952380952**	**3.325559203**						

Sample Preparation by Laser Cutting Parameters:

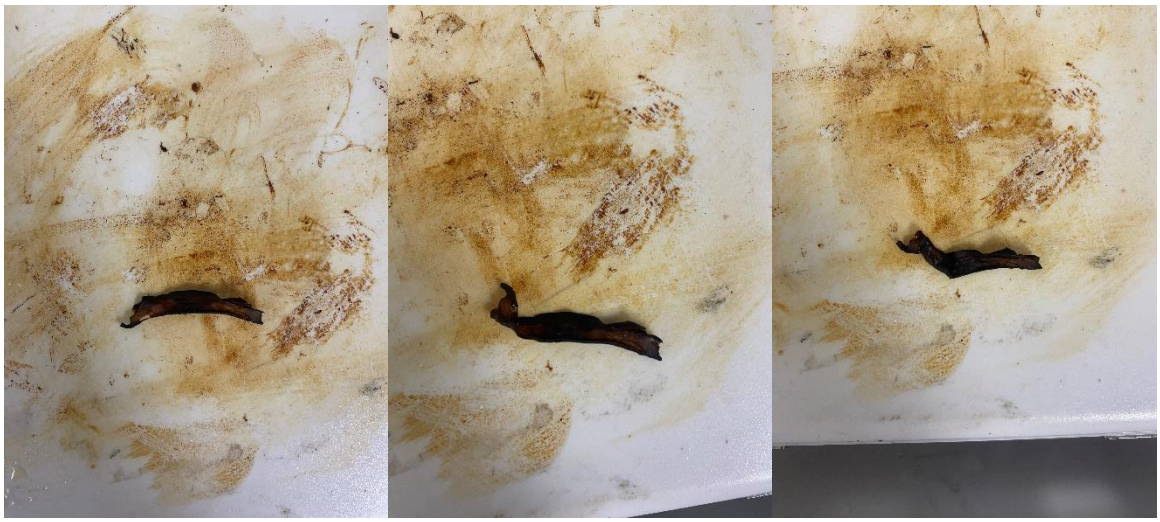
- 7% Power (4.2 W)
- 7% Speed
- 1% Frequency

Full Equipment and Material List:

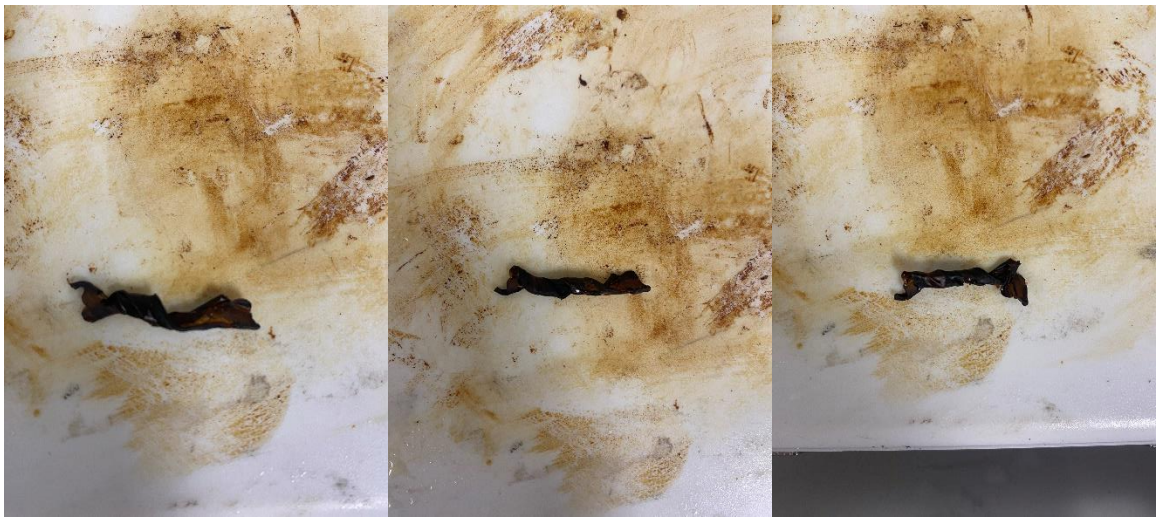
- Nafion Dispersion
  - Ionpower
  - D2020CS
- Nafion Film
  - Ionpower
  - N115
- Filament 3D Printer
  - Raise3D
  - Pro2 Plus

- Resin 3D Printer
    - Formlabs
    - Form 3
- Blue Laser
    - Snapmaker 2.0 10W Laser
- CO2 Laser
    - Epilog
    - Laser Fusion M2 60W Laser
- Heat Gun
    - X-Tronic
    - 5000 Series
    - Model #5040-XR3
- Heat Plate
    - Ohaus
    - Guardian 5000
- Vacuum Oven
    - Across International
    - 316L 250C 0.9 CF Vacuum Oven

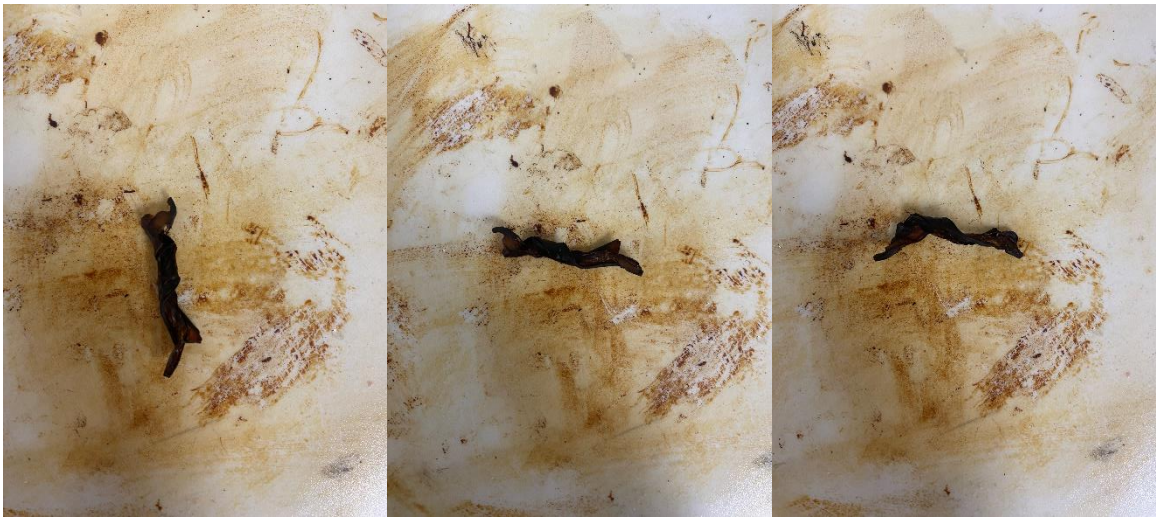
5 Shape Transition Test:



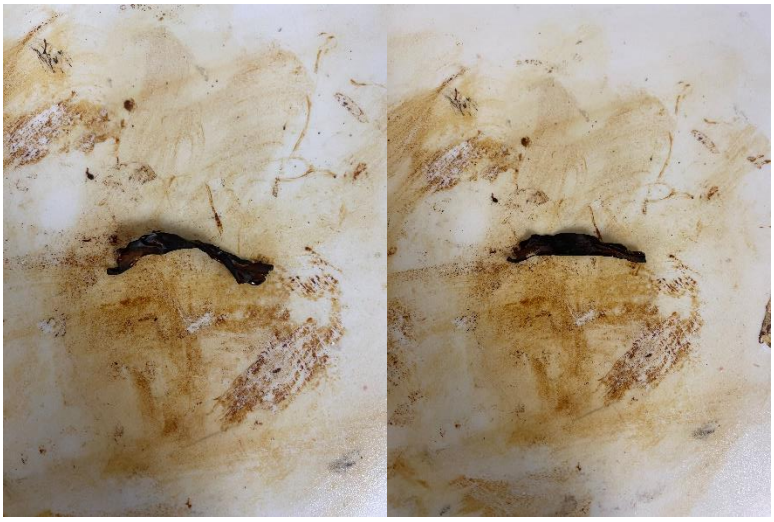
S0 S1 S2



S3 S4 S5



S4R S3R S2R



S1R S0R