MECHANICAL TESTING OF DEGRADED MAGNESIUM WIRES

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DECLARATION

This report entitled

Mechanical testing of degraded magnesium wires

Was composed by me and is based on my own work. Where the work of the others has been used, it is fully acknowledged in the text and in captions to table illustrations. This report has not been submitted for any other qualification.

Name Shalom Moise

Signed ………………..

Date ………………..

0. Abstract

This paper investigates the change in mechanical properties of a magnesium alloy (Mg AZ31) as it is degraded for a few months by Phosphate Buffered Saline (PBS). The purpose of this is to find out if it could be a suitable material for a tracheal stent to treat tracheal stenosis in neonates. The proposed stent would degrade as the patient no longer requires the stent. Mg is being tested as it is biodegradable, biocompatible. The degradation rate was determined by the mass loss and its yield strength and ultimate tensile strength were calculated by tensile tension. The data was averaged by Weibull's distribution to obtain more accurate results.

The results found that the degradation affected the samples the most at the beginning of the degradation, with the highest mass loss in the first few weeks and the only substantial difference in the yield strength and ultimate tensile strength in the first month (YS: 441.5±8.3 MPa to 426.6±11.1 MPa, UTS: 913.5±7.0 MPa to 900.8±21.0 MPa). The viability for Mg AZ31 in biodegradable tracheal stents remains inconclusive.

Contents

1. Introduction

Tracheal stenosis is a condition where the trachea (wind pipe) has not developed not developed properly. This results in narrow tracheas which severely restrict airflow into the lungs. The trachea of a healthy child is approximately 5 mm and should grow to 10-25 mm (depending on male or female). A child suffering from stenosis may have a trachea as small as 1-3 mm (Breatnach E, 1984). This can cause long term damage and potentially death, if it is not treated as soon as possible. It is particularly dangerous in neonates, as their growth will be greatly impeded if they do not receive enough air. Great Ormand Street Hospital in London is reported to treat around 70 newly born infants with tracheal stenosis every year. Operations like anastomosis and reconstructive surgery can be successful, they are best avoided at such a young age, as it is invasive and likely to cause infection (David Vondrys, 2011).

One solution that is being researched is to design bioabsorbable stent that could be inserted directly into the trachea and increase the airway. The proposed stent would keep the airway open until the trachea will naturally grow to the correct size and ideally, dissolve when it is no longer needed. Special care must be taken into designing a stent that meets the necessary requirements without being a potential danger to the infant. Leaving a stent in the trachea will be uncomfortable for the patient and may cause infections. Removing it surgically at young age would be very risky.

The proposed stent material must be strong to enough to keep the airway open constantly for a long duration of time (9-12 months) but also easy to deform for the stent production and enlargement once it enters the trachea. To asses a material's viability, its initial yield strength and fatigue strength and how these properties change as the material degrades would also need to be known. Additionally, the by-products of the material degrading must be identified as harmless to the human body. It is also important to investigate how the material fractures, as the stent is to be placed up to soft, delicate tissue.

Magnesium (Mg) is often seen a suitable candidate for the material of what the stent would be made density metal (1.74 gcm^{-3}) and is easily processed. Other common metals used in implants, like titanium and aluminium, have densities of 4.54 gcm^{-3} and 2.7 gcm^{-3}, respectively (Manuel Marya, 2006). Magnesium also has mechanical properties similar to that of human bone. The Young's modulus of magnesium is 45GPa and bone is 7-25GPa (Ellon & Tanner, 2012). This has made it a popular material in implants as it reduces stress shielding and speeds up bone resorption. Most importantly, (for the purposes of this report), it is a trace element found in the body and is biodegradable and bioabsorbable (Witte, 2008). It is even used in some metabolic processes in the body. Mg reacts with hydroxide ions in aqueous solutions to form magnesium hydroxide ($Mg(OH)_2$) and hydrogen gas, as seen in equation [1] below. There have been issues with built-up pressure from the formation of Hydrogen gas (Witte F. , 2010). However, this would not be an in a tracheal stent, where the gas would easily escape to the atmosphere.

$Mg(s) + 2H_2O(\text{l}) \leftrightharpoons Mg(OH)_2(\text{aq}) + H_2(g)$ [1] (Witte, 2008)

Pure Mg implants have been shown to degrade very quickly in the body and become very brittle. Many of the first treatments of magnesium implant caused severe pain to the patient (Witte, 2010). It has been shown that alloying magnesium with other metals can significantly alter it properties. Alloying magnesium with aluminium has been shown to increase its "yield strength, ultimate tensile strength, corrosion resistance and oxidation resistance" (Manuel Marya, 2006). It does this by forming an aluminium oxide 'cover', which protects the magnesium from degradation. Zinc has been also been shown to increase strength of magnesium, though it can make it more brittle too and may increase the degradation rate at too high a concertation.

Adekanmbi et al. have done in-vitro testing of Mg AZ31 alloy with the purpose of finding out if it can be a viable as a biomaterial to be used in the body. They degraded the alloy from periods ranging from one month to nine months, to see how degradation effects its mechanical properties (Adekanmbi, Mosher, Lu, Kubba, & Tanner, 2017). They showed that the size of the sample can affect the mass loss and observed significant losses in tensile strength as the samples were degraded for longer. The specimens that were used in their experiments were large dumbells shapes. As size affects the degradation rate, their results cannot be used to asses the use of magnesium in a stent, which be made from much smaller pieces. To show wether it may be used as a stent, we must degrade much smaller specimens and test its strength then.

2. Method

2.1. Material

The Mg alloy that was tested was Mg AZ31 (96% Mg, 3% aluminium, 1% Zinc). The specimens are thin wires (diameter- 0.1 mm).

Five groups of the Mg alloy were prepared. Each group contained 11 samples. The samples' lengths were 120mm and diameters 0.1mm. They were each immersed in Phosphate Buffered Saline (PBS) for extended periods of time, with exception of the first batch, which was not immersed at all. They kept in an incubator, which stayed at a constant 37°C The second batch was submerged for one month, the third for two months, the fourth for three months, and the fifth for four months.

2.2. Degradation Rate

All samples in that were to be immersed in PBS where weighed prior to immersion. Each sample was numbered and marked with a non-reacting dye. They were weighed both before and after being marked. Every 7-10 days, the batches were taken out of the PBS to be rinsed with sterile water and weighed. The PBS was also replaced. The mass loss recorded was converted to a percentage to represent the degradation rate through the time the samples were immersed. The degradation rate per hour was also calculated by dividing the average mass loss by surface area of the wire (75.46 mm^2) multiplied by the hours spent immersed in PBS.

2.3. Tensile Testing

Each sample underwent tensile testing with INSTRON **1000** until failure. The clamps were fitted with thin aluminium plates to prevent slipping. The middle 80mm of the samples were tested, with 20mm on each side were in the grips. The strain rate was set at $24\text{mm}/\text{min}$. From this, the yield strength, ultimate tensile strength, and elongation at failure were calculated. To accurately calculate the yield stress, the 0.2% proof stress was implemented.

2.3.1. Statistical Analysis

As the wires are very thin, a large variance in results was expected. To counter this, the averages were calculated using Weibull's distribution. The results where then compared using the Student's T-Test, to determine if any correlation was caused by chance or due to genuine causation.

2.4. Scanning Electron Microscopy

Samples were placed under a focused ion beam scanning electron microscope (FIB-SEM) to determine the type of fracture that took place in the tensile testing. In particular, the fracture surface and the outer degraded surface were examined.

3. Results

3.1. Degradation Rate

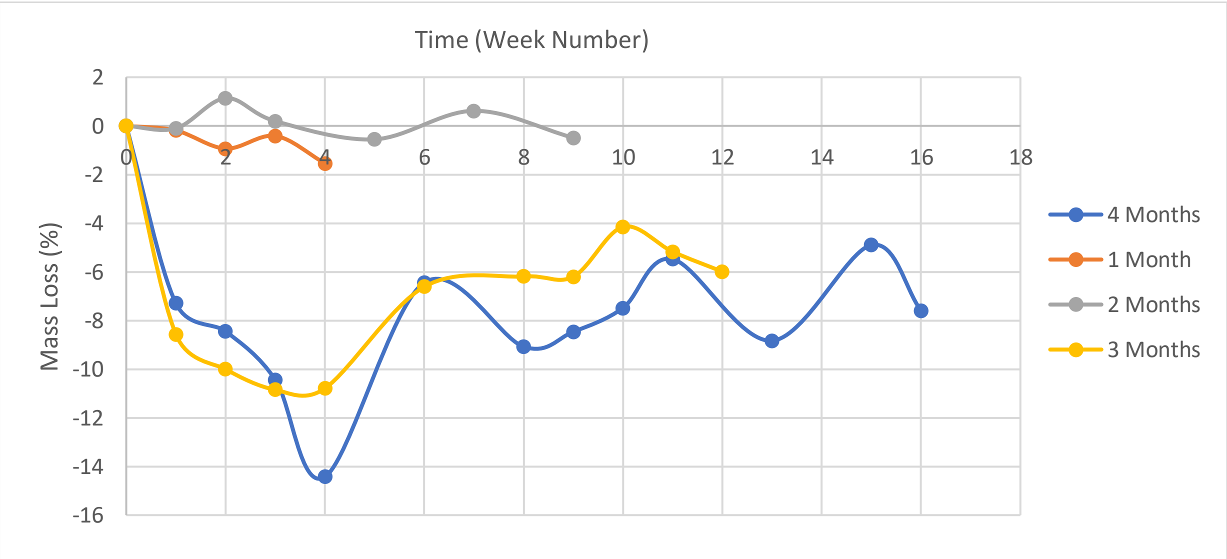

Figure 3-1 Average Mass Loss for Mg AZ31 Samples

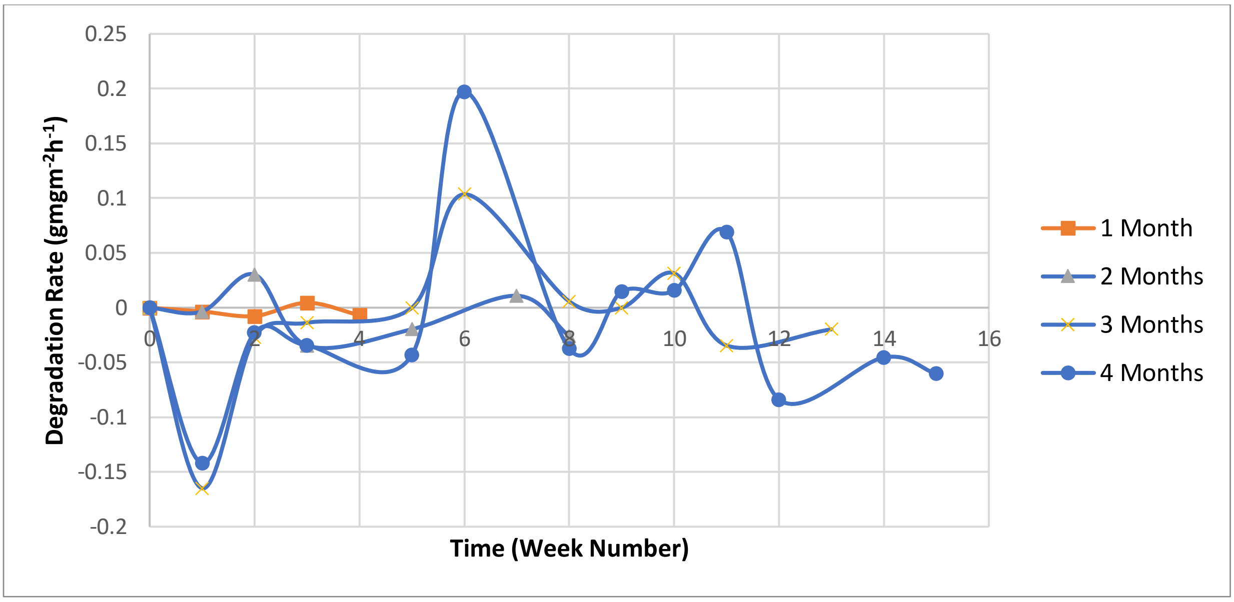

Figure 3-2 Degradation Rate in Time

Figure 3-1 represents the average mass loss in percentage by all the samples that immersed in PBS. Table 1 below shows the average initial weights before immersion average final weights before the tensile testing took place (± is the standard deviation). The mass loss varies substantively between each sample group. The initial degradation of the 3- and 4-months samples occurred at a much faster rate (0.165 $gm^{-2}h^{-1}$ and 0.142 $gm^{-2}h^{-1}$ respectively) than the 2 months and 1-month samples (both were 0.003 $gm^{-2}h^{-1}$). As the degradation progressed the samples' weights fluctuated. The 2- months sample even gained more weight than it originally had initially. The overall trend is that the mass decreases the longer the wires are immersed in PBS. However, it is difficult to determine an accurate degradation from these results. Figure 3-3 represents the different degradation rates and mass changes in each sample group. The trendlines for the degradation rates are shown on the graphs. The 1- month and 2-months graphs (a) and b)) both show an increasing degradation rate (trendline gradient is positive), whereas the 3- months and 4- months graphs show a decreasing degradation rate (trendline gradient is negative). This suggests that the longer the samples are degraded, the rate of degradation will decrease, despite the mass change being small (Table 1).

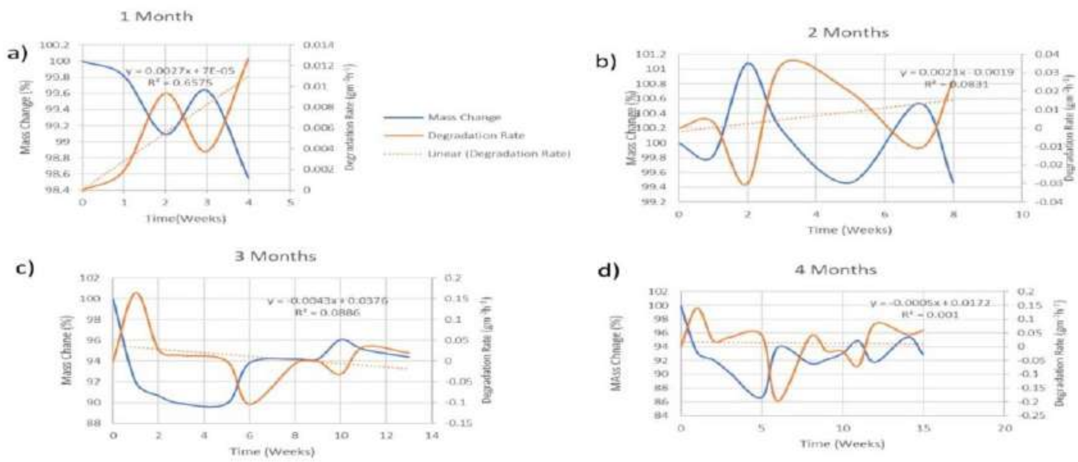

Figure 3-3 Graphs representing the degradation rates and mass changes during the immersion period.

Degradation Time (Months)	**Initial Painted Weight** (mg)	**Final Weight** (mg)	**Mass Loss** (%)
1	34.4±1.5	33.9±1.6	1.5±3.7
2	34.8±1.9	34.6±2.0	0.5±4.7
3	33.4±1.5	31.5±1.0	6.0±4.1
4	34.1±1.5	31.7±1.5	7.6±7.3

Table 1 Comparison of the average weight of samples before degrading and after degrading has been completed

However, due to the sharp difference in the initial degradation rates between the 1 and 2- months samples and between the 3 and 4- months samples (Figure 3-1), it is difficult to draw that conclusion.

3.2. Tensile Testing

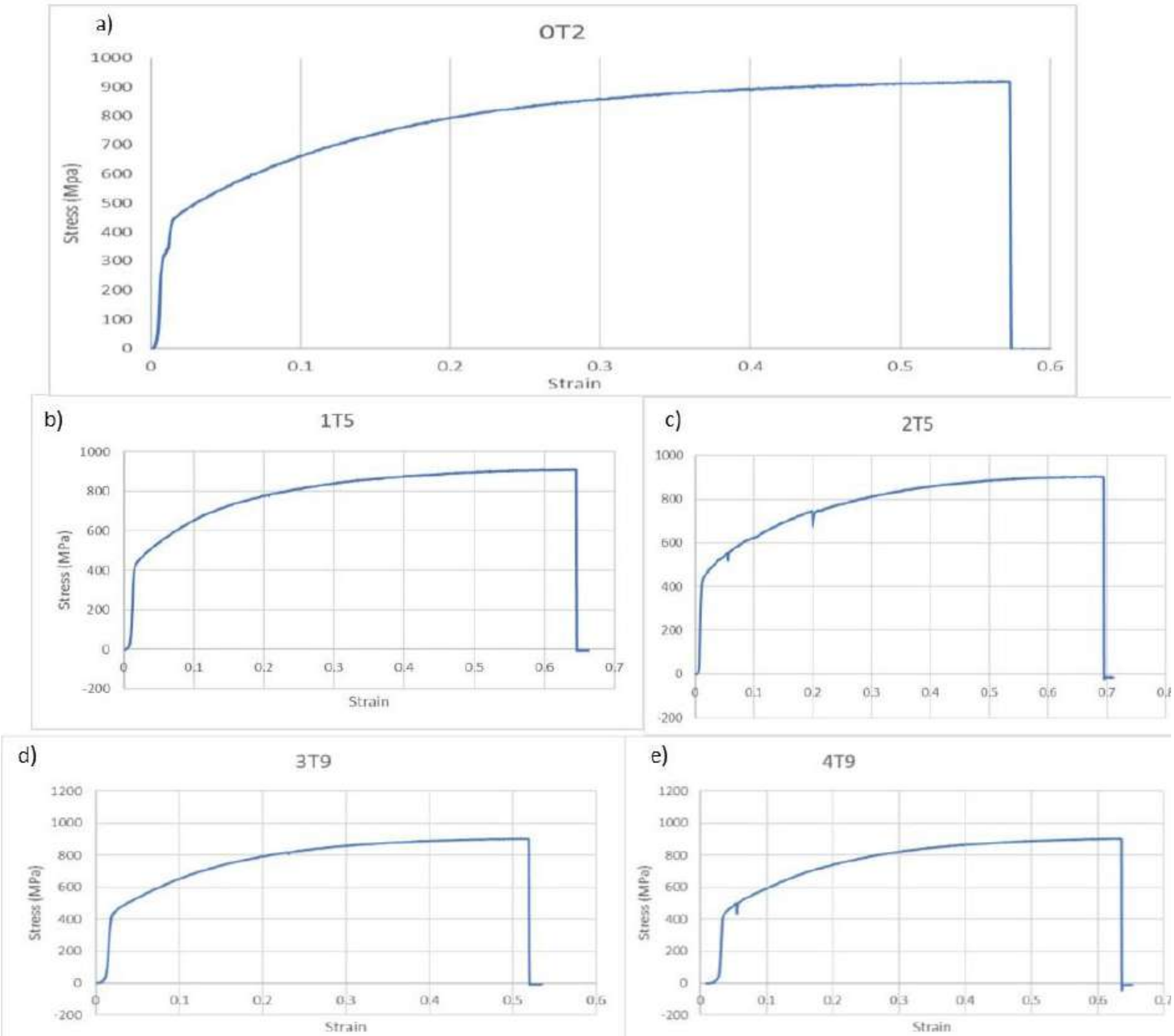

Figure 3-4 Stress-Strain graphs from tensile testing of Mg AZ31after degradation. a) n degradation, b) 1 month, c) 2 months, d) 3 months, e) 4 months degradation.

Figure 3-4 gives some examples of the behaviour of the wires under tension. Table 2 below shows the Yield stress, ultimate tensile strength, and the strain at fracture of all the sample groups with their respective standard deviations. The differences in all sets are relatively small and the standard deviations overlap. This may in fact show that the changes are insignificant. Moreover, the lowest yield stress is that of the 1-month sample group, which is unexpected. A possible trend to infer from table x is the ultimate yield strength (UTS), which decreases with time degraded. It seems that the first month is when the degradation makes the most significant effect on the mechanical properties of the wires. After that, the changes do not seem to be substantial.

Degradation Time (Months)	**Tensile Yield Stress** (MPa)	**Ultimate Tensile Strength** (MPa)	**Strain at Fracture** (%)
0	441.5±8.3	913.5±7.0	62.1±2.7
1	426.6±11.1	900.8±21.0	57.6±13.0
2	433.1±3.1	895.6±39.2	71.9±7.2
3	429.7±4.3	894.2±36.7	63.7±10.7
4	433.9±7.4	894.6±12.3	65.5±6.3

Table 2 Comparison of the mechanical properties of the specimens of Mg AZ31 degrading and after degrading has been completed

Table 3 below shows the results of applying the Student's T-test to the results from the tensile testing. For the yield stress, the 1- month, 2- months and 3- months sample groups all had a value below 1%, which means that there is less than a 1 in 100 chance that the differences are due to chance and so are reliable. The 4- month sample obtained a value between 1 and 5%, which means there is more than a 1 in 100 chance that the differences are random but still less than 1 in 20. Most of the values for the UTS and strain at fracture are above 5%, which means that they are likely due to chance. It is important to note that some samples did not give reliable results for the UTS and strain at fracture due to much slipping in the grips.

Degradation Time (weeks)	**Tensile Yield Stress** (%)	**Ultimate Tensile Strength** (%)	**Strain at Fracture** (%)
1	0.1<**0.15**<1	**5.9**>5	**28.9**>5
2	**1.0**>0.1	**9.6**>5	0.1<**0.23**<1
3	0.1<**0.11**<1	**7.8**>5	**73.0**>5
4	1<**3.4**<5	5<**1.3**<1	**18.1**>5

Table 3 Student's T-Test comparing the degraded sample groups with the non- degraded sample group

3.3. FIB-SEM

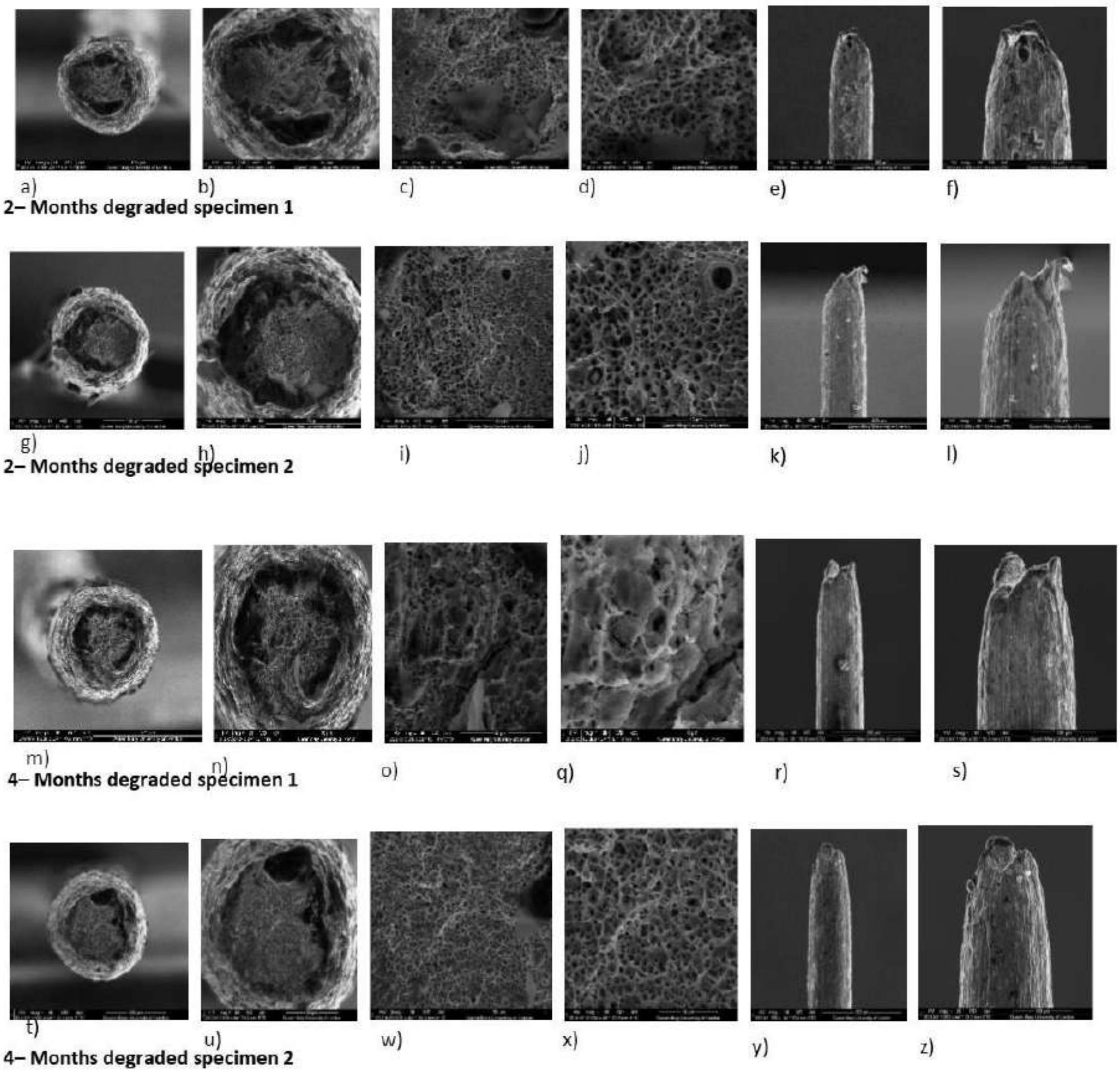

Figure 3-5 FIB-SEM of two 2-month samples and tow 4- months samples

The images in figure 3-5 display the difference between the fracture surface and the outer degraded surface between the 2- months degraded samples and the 4- month degraded samples. Figures 0-5 a)-b) and g)-h) show the fracture surface of 2- months samples. The dark portions on the outer circle of the crack may be oxides and magnesium hydroxide formed from the magnesium reacting with the PBS. The oxides in the 4- months samples seem less prevalent. Zooming into the crack surfaces (c)-d), i)-j), o)-q), and w)-x)), the difference in the microstructure of the alloy can be observed. In the 2- months samples, the grain refinement is clear. In the 4- months samples, q) in particular, the breakdown of the sample can be seen.

4. Discussion

4.1 Interpretation of Results

The results from the testing seem to show that the degradation mainly affected the samples in the first month, as evidenced from the initial average mass loss in all the samples (figure 3-1) and the decrease of yield stress and UTS from when comparing the sample of no degradation with the 1-month sample group (Table 2). The initial average yield stress was 441.5±8.3MPa and decreased to 426.6±11.1 MPa, a difference of 14.9 MPa. Additionally, the initial UTS of the Mg wire decreased in the first month from in the non-degradation sample group (913.5±7.0 MPa) than 1- month sample group (900.8±21.0), with a similar decrease of 12.7 MPa. Though the strain at failure does not seem to show any trend and the T-tests would suggest that you cannot conclusively prove the results in UTS and yield stress were not up to chance, the fact they coincide with a mass drop/ increasing degradation rate would imply that the results are valid and do show a substantial difference between 0 months degraded and 1 month degraded.

However, due to the small difference in the results of the yield stresses and UTS in the 1-4- months sample groups, the fluctuations in the degradation rates (figure 3-1) and inconsistencies in mass loss (figure 3-2), it is difficult to determine whether or not degradation rate is vastly reduced or if it is due to error.

4.2 Implications

As these results are vague, it is inconclusive weather Mg AZ31 alloy would be suitable for a tracheal stent. Though, the samples did retain most of their strength, it is doubtful the degradation process by PBS is similar to the degradation the proposed stent would go through in the trachea. The body would aim to keep the pH of the fluids in the trachea (e.g. mucus) at a constant due to homeostasis. Simulating that process in-vitro would require a constant replenishing of PBS as the magnesium dissolves.

4.3 Improvements

Adekanmbi et al. successfully and clearly showed the long-term degradation of Mg AZ 31 alloy over a period of nine months (Adekanmbi, Mosher, Lu, Kubba, & Tanner, 2017). The results produced from this report are less clear. This is due in large part to the size of the Mg AZ31 alloy. Testing with such thin wires proved to be a difficult challenge in the tensile testing. The Instron 1000 was not sensitive enough to perform the tensile testing with the wires. The lowest available control load was 30 kN, far more than what is necessary to fracture the wire. Although the aluminium plates did help prevent the wires from slipping whilst under tension, there still where many times where the wire did slip (figure 4-1), compromising the results. Some samples slipped so much the UTS could not be determined from them, reducing the reliability in calculating the average UTS. Repeating the experiment with more samples would help to circumvent this issue in the future. There were a few attempts to high-cycle fatigue testing, but the wires proved to react too difficult to perform the test accurately. More sensitive equipment would be required to test the wires more accurately.

Unfortunately, do to time restrictions, microscopy was only available for the 2- and 4- month samples. Having microscopic images of the other stages of degradation would have helped determine if the samples had degraded.

The mass loss of the wires was expected to decrease steadily and gradually as the time immersed in PBS increased. It turned out that the mass loss fluctuated from week to week. In the second week of the 2- month cycle it weighed even more than it did initially. The trends in the different samples were incoherent. The 3- and 4 months sample groups initial mass loss was substantially greater than that of the 1- and 2 months sample groups. This may have been a due to being unable to replenish the PBS as often as desired. Replacing the PBS every few days rather than once a week may have prevented the reactions between the alloy and the PBS from reaching an equilibrium and so would increase the mass loss. It would also give more chances to weigh the samples, giving more data points and make the results more accurate. Alternatively, rather than replenishing the PBS on a set schedule, it may be more beneficial to measure the pH of the PBS and wires regularly, and only replenish the PBS when the pH has reached a certain level. Magnesium hydroxide would be formed which would raise the pH level. This would give more detail as to how wires react to the PBS. Additionally, it would be closer to replicating the conditions in the human body.

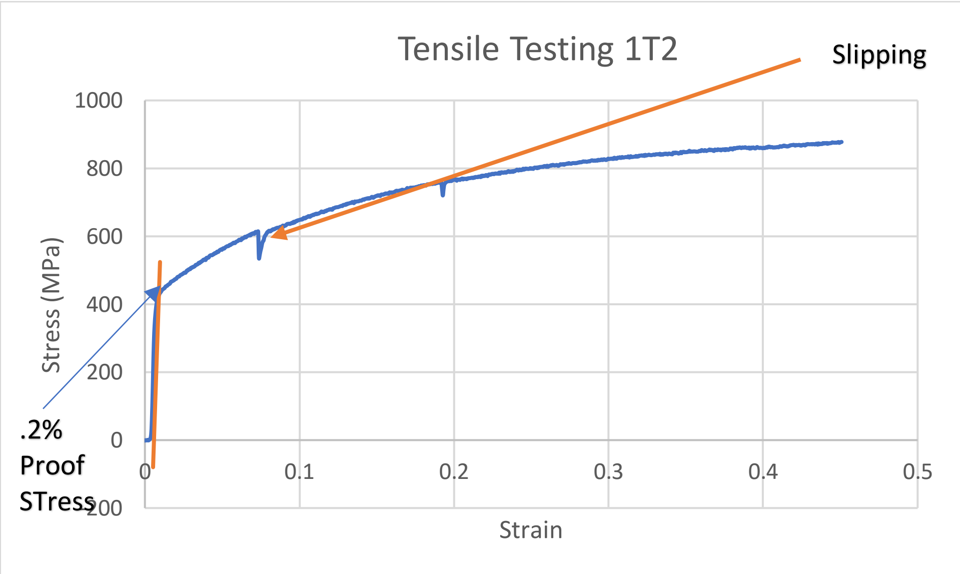

Figure 4-1 Stress-Strain graph from tensile testing. The bumps on the curve our as a result of slipping

5. Conclusions

The degradation rate of Mg AZ31 wires in PBS is initially high but fluctuates as time continues. The degradation rate past one month cannot be reliably determined from this report. The initial degradation does reduce the yield stress by ≈15 MPa and the UTS by ≈13 MPa. This results if this study are inconclusive of weather this magnesium alloy would be a suitable material for a tracheal stent, though it should offer other avenues of in-vitro research if it can.

6. Acknowledgments

I would like to express my deep gratitude to my project supervisor, Professor E. Tanner, who provided the idea for this project, as well as advice on how to interpret the results and of course, for providing the magnesium alloy wires.

Thank you for all the lab technicians who aided me in the various labs: Dr Di Federico, who taught me how to use the Instron 1000 for the tensile testing and for providing the aluminium plating for the clamps; Mr Iqbal who provided me with PBS solution and a place in the incubator to keep the samples for extended periods of time; and Dr Bailey who did the microscopy of the samples.

7. References

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8. Appendices

8.1. Calculating Yield Stress

- Tensile testing gives the force applied and the extension of the wire.

$$Stress\ (MPa) = \frac{Force\ (N)}{Area\ (mm^2)}\ [2]$$

$$Strain = \frac{Extension\ (mm)}{Original\ Length\ (mm)}\ [3]$$

- Use [2] to convert the force to stress and [3] to convert extension to strain. Plot Stress-Strain graph:

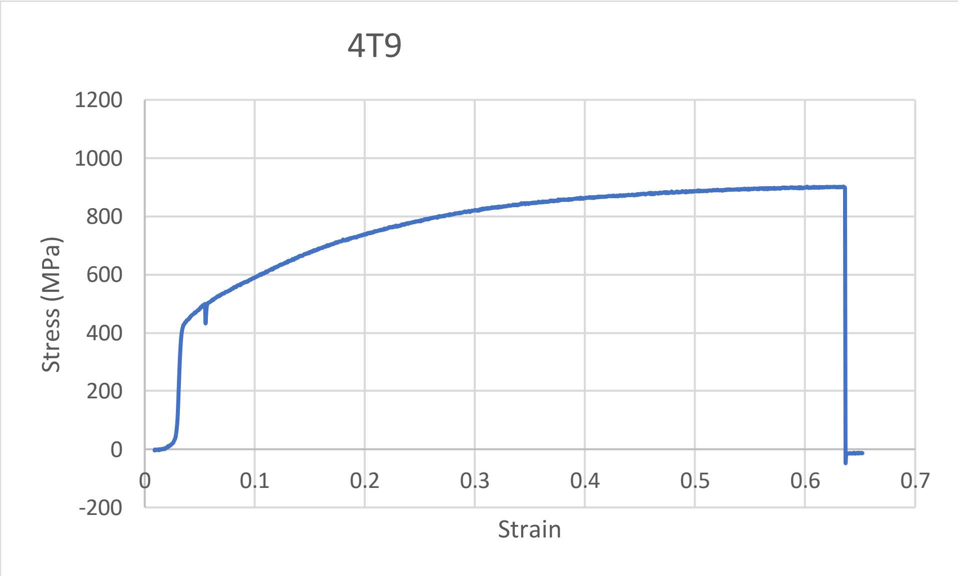
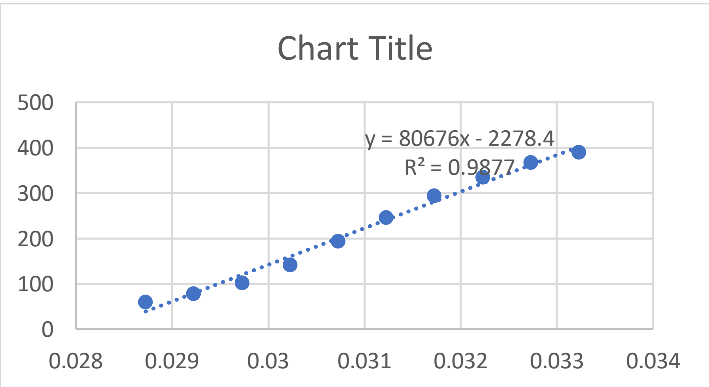


Figure 8-1

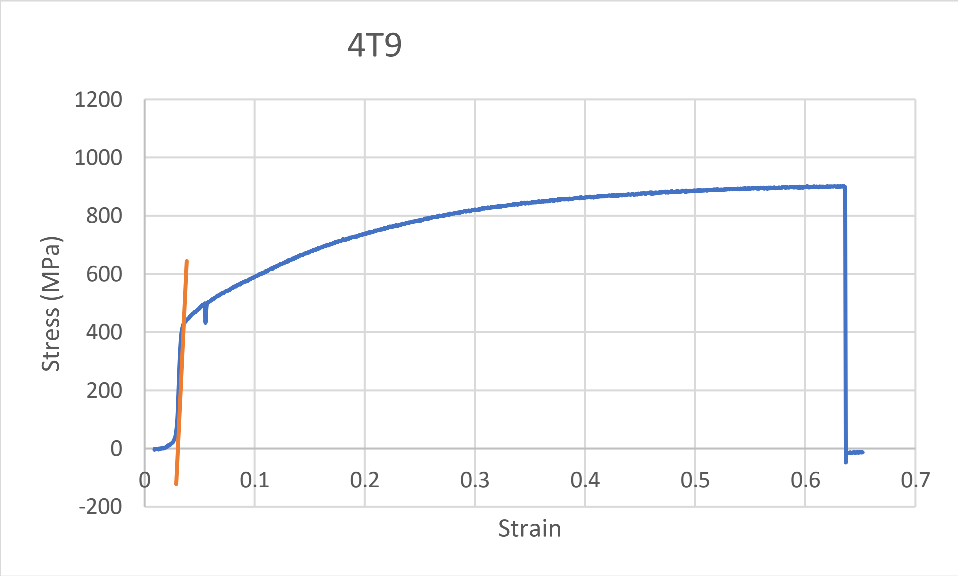
0.2% Proof Stress

- Plot stress strain graph of elastic deformation only (R^2 of trendline should be equal or close to 1)
- Find equation of trendline y=mx+c=y=80676x-2278.4



- .2%: c-0.002*m=-2278.4-.002*80676=-2439.752

- Plot equation y=80676x-2439.752 (x= Strain) on original stress strain graph.



- Point at which the lines intercept will be the yield stress.
- Use (x,y) values from graphs and interpolate.
- $x_1, y_1, y_{.2\%1} : y_{.2\%1} < y_1$
- $x_2, y_2, y_{.2\%2} : y_{.2\%2} > y_2$
- e.g.

	x	y	$y_{.2\%}$
1	0.035224	427.5768	402.0097
2	0.035725	428.6994	442.3779

- $m_1 = \frac{428.6994-427.5768}{0.35725-0.035224}$ $m_2 = \frac{442.3779-402.0097}{0.35725-0.035224}$
- $c_1 = \text{intercept}(\frac{428.6994-427.5768}{0.35725-0.035224})$ $c_2 = \text{intercept}(\frac{442.3779-402.0097}{0.35725-0.035224})$
- $y_1 = y_2 = m_1x_1 + c_1 = m_2x_2 + c_2$
- Y will be yield stress= 428.308135884801= 428.3 MPa

8.2. Using Weibull's Distribution

- Take a range of results, e.g. the calculated yield stresses when there is no degradation.

	1	456.535801
	2	451.3703469
	3	447.4272093
	4	441.1846895
	5	433.2508262
	6	435.1371975
	7	434.9677522
	8	439.171254
	9	439.6496999
	10	432.7712876
	11	431.2143848
Mean		440.2436772
SD		8.266824227

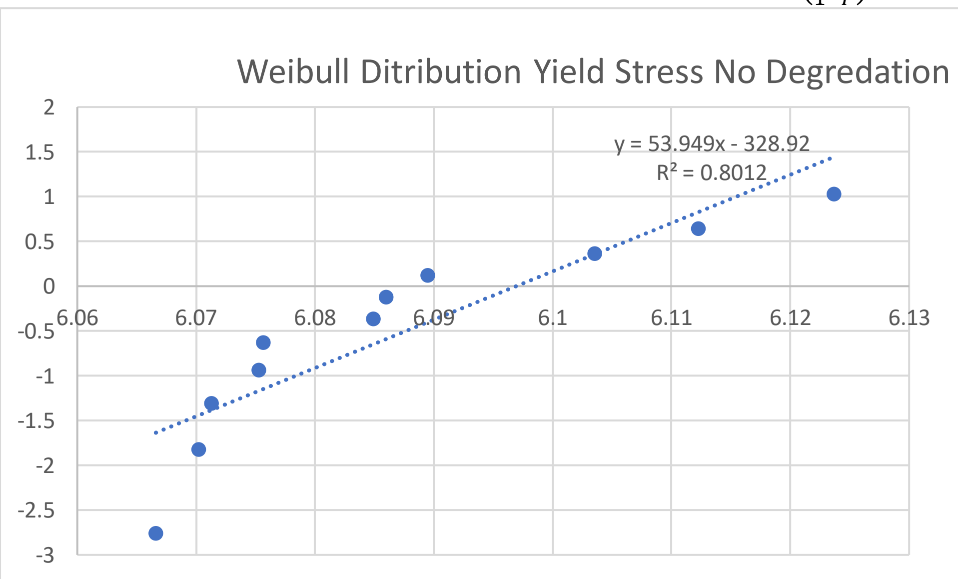
- Re-order the list from smallest to largest and re number them.

1	431.2143848
2	432.7712876
3	433.2508262
4	434.9677522
5	435.1371975
6	439.171254
7	439.6496999
8	441.1846895
9	447.4272093
10	451.3703469
11	456.535801

- Then calculate P= (i-0.3)/(n+4), i is the number on the list and n is 11 (11 samples)

1	0.061404
2	0.149123
3	0.236842
4	0.324561
5	0.412281
6	0.5
7	0.587719
8	0.675439
9	0.763158
10	0.850877
11	0.938596

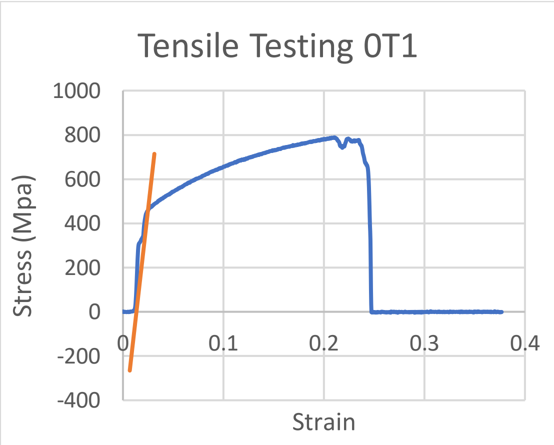
- Plot natural logs of yield stresses on the y- axis, and $Ln(Ln\left(\frac{1}{1-P}\right))$ on the x axis.

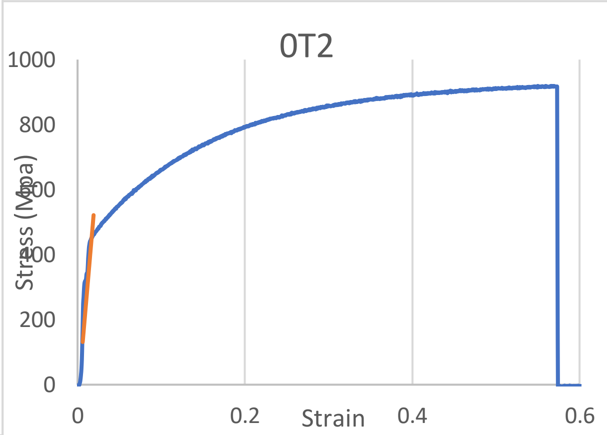


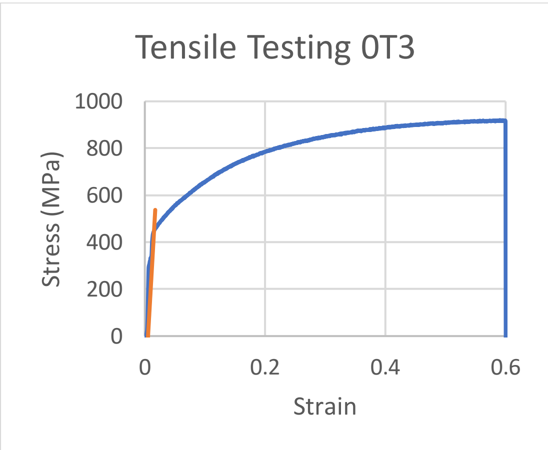
Use equation of trendline to calculate yield stress.

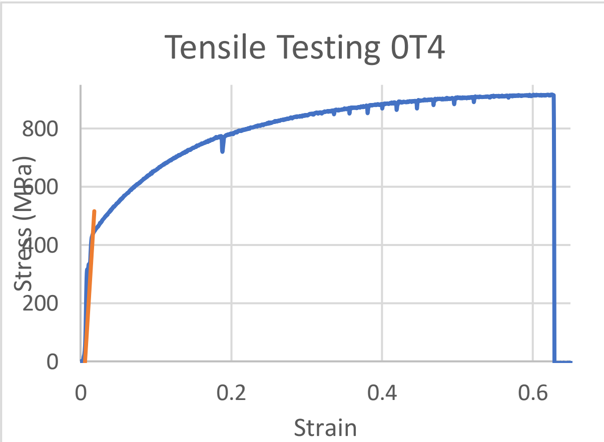
$= \exp(\frac{-0.3665-c}{53.9})$*=441.4549= 441.5 MPa

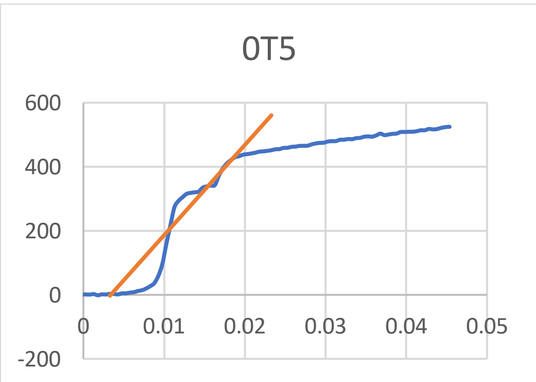
8.3. Tensile Testing Results

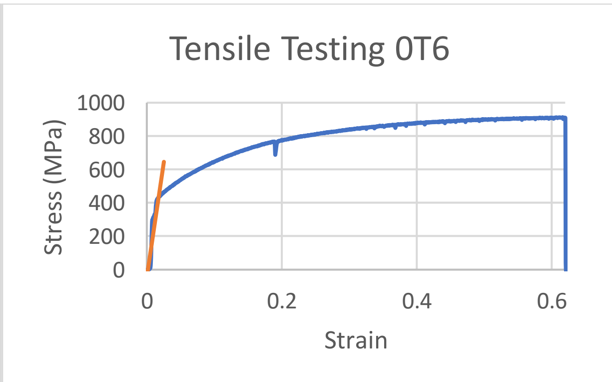


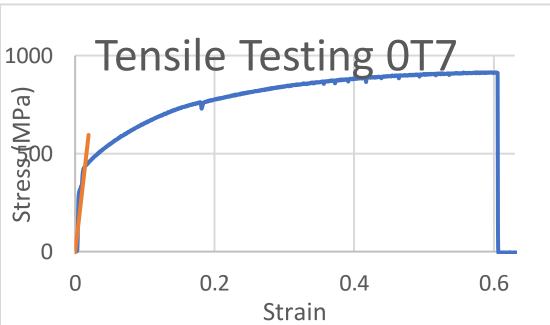


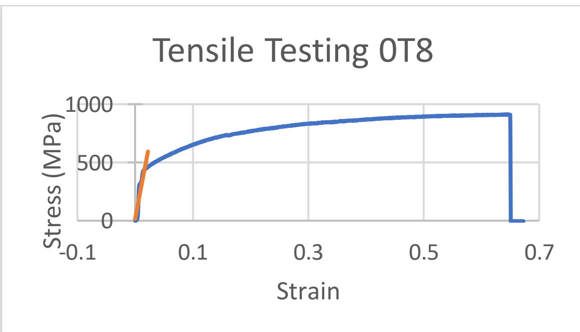


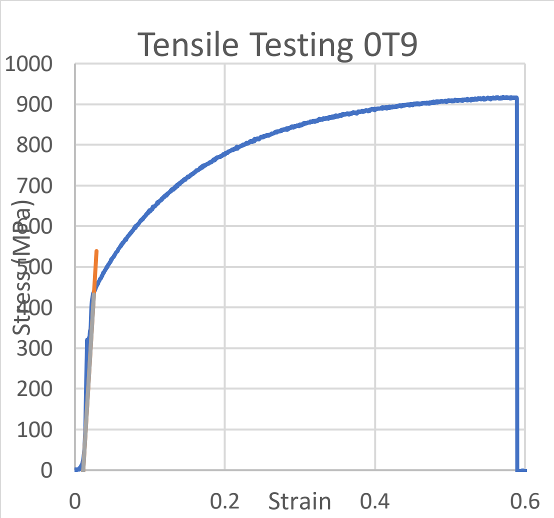


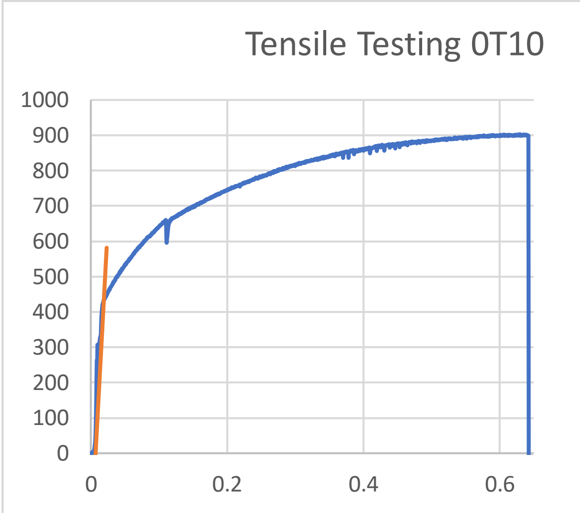


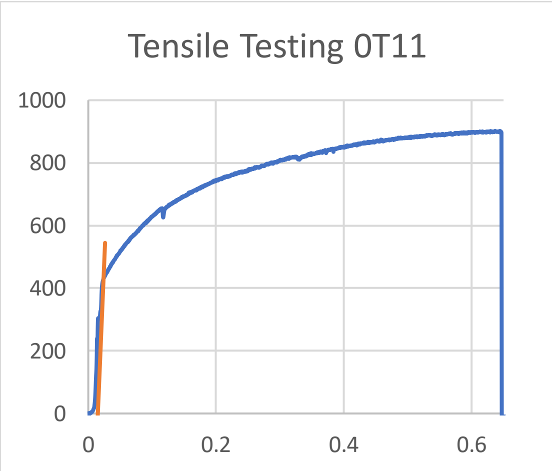


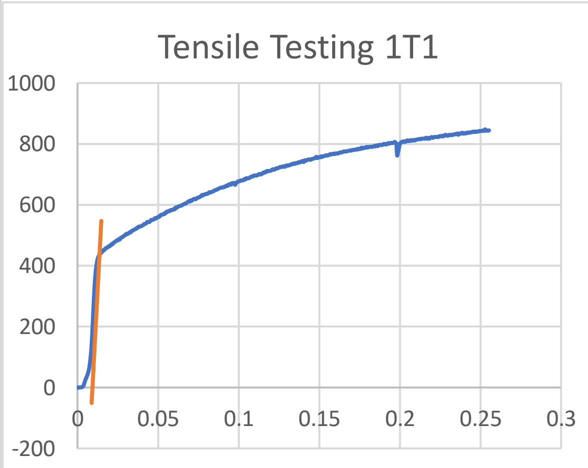


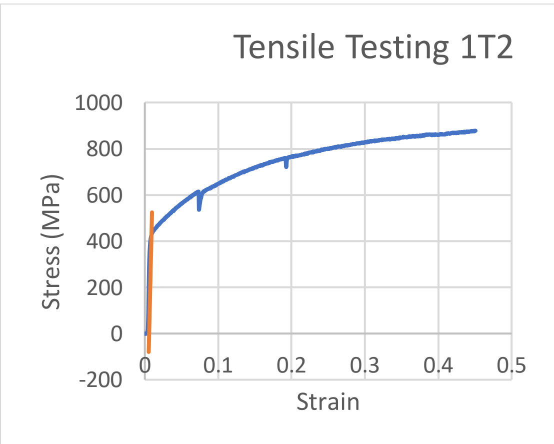


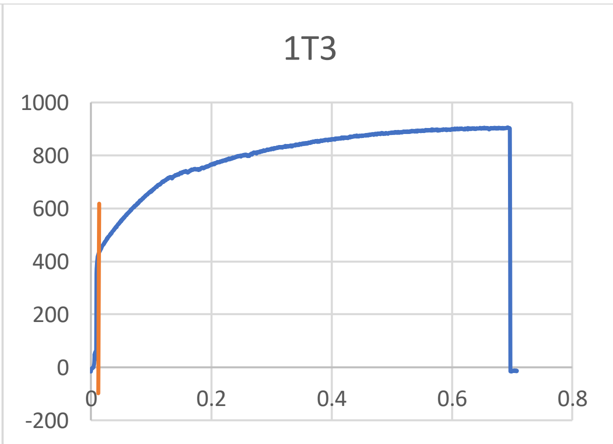

Tensile Testing 0T9
Stress (MPa)
1000
900
800
700
600
500
400
300
200
100
0
0
0.2
Strain
0.4
0.6

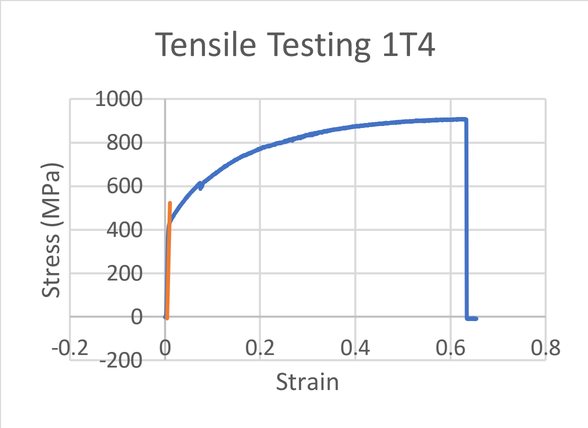

Tensile Testing 0T10
1000
900
800
700
600
500
400
300
200
100
0
0
0.2
0.4
0.6

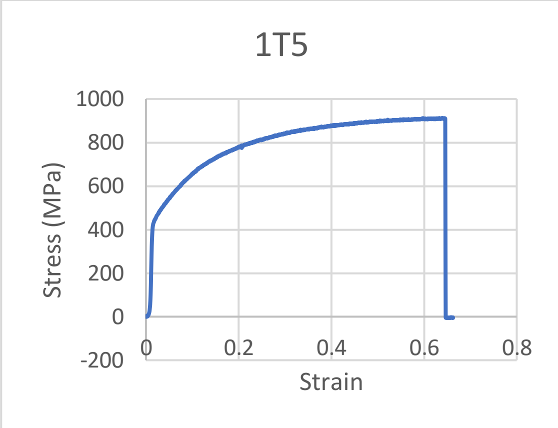

Tensile Testing 0T11
1000
800
600
400
200
0
0
0.2
0.4
0.6

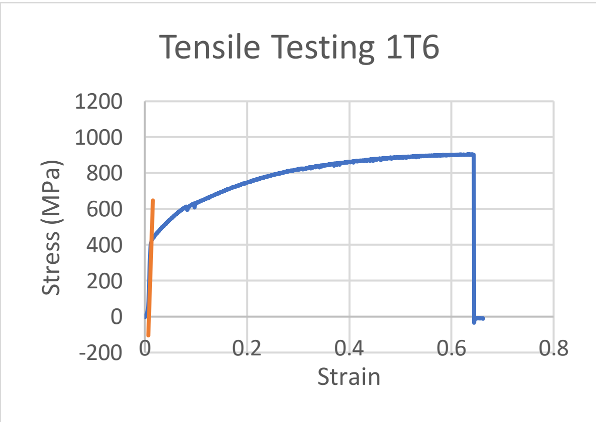

Tensile Testing 1T1
1000
800
600
400
200
0
-200
0
0.05
0.1
0.15
0.2
0.25
0.3

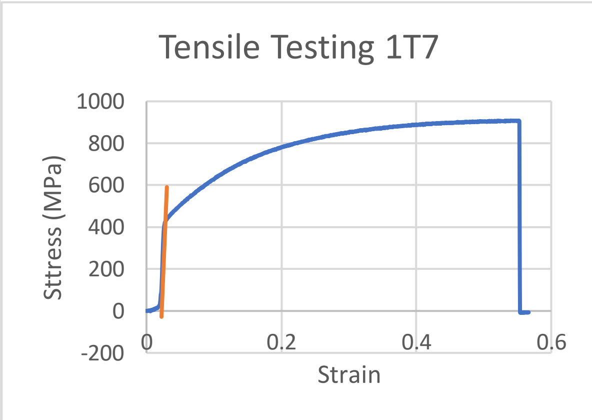

Tensile Testing 1T2
Stress (MPa)
1000
800
600
400
200
0
-200
0
0.1
0.2
0.3
0.4
0.5
Strain

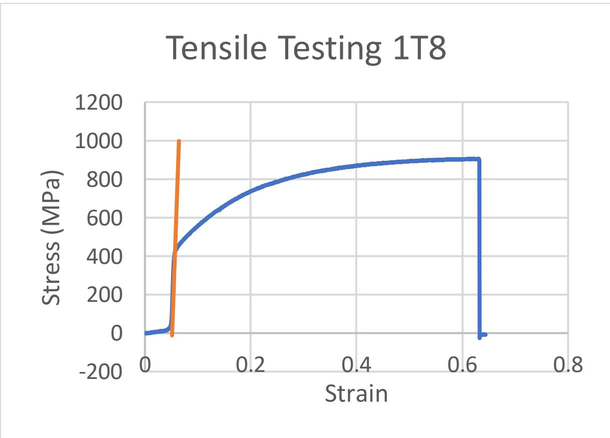

1T3
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

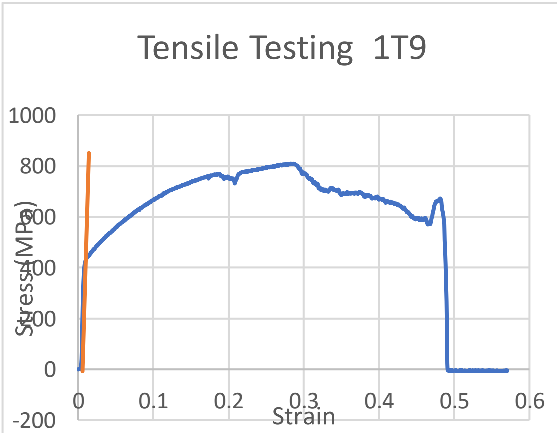

Tensile Testing 1T4
Stress (MPa)
1000
800
600
400
200
0
-200
-0.2
0
0.2
0.4
0.6
0.8
Strain

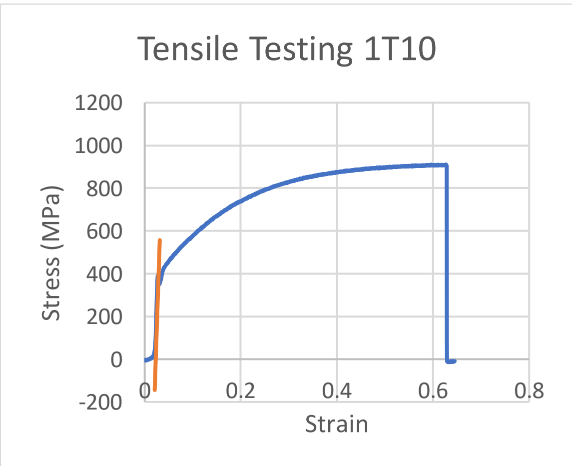

1T5
Stress (MPa)
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
Strain

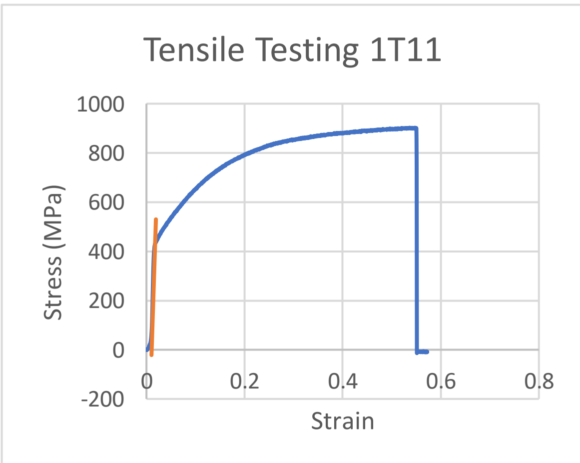

Tensile Testing 1T6
Stress (MPa)
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
Strain

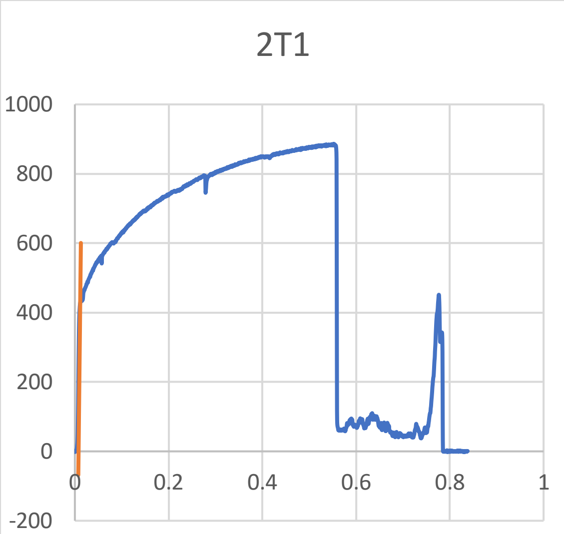

Tensile Testing 1T7
Sttress (MPa)
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
Strain

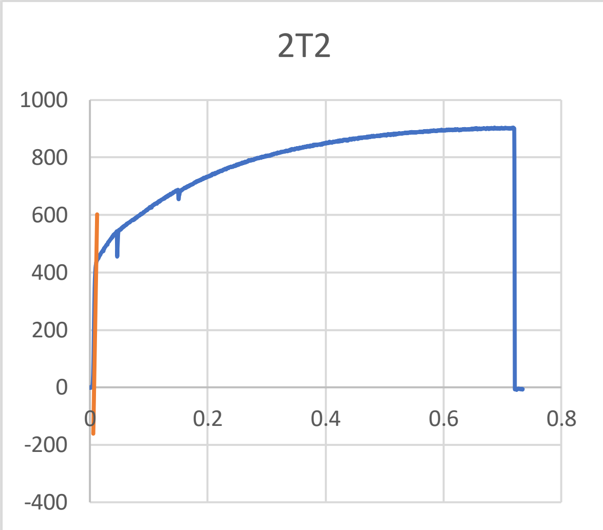

Tensile Testing 1T8
Stress (MPa)
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
Strain

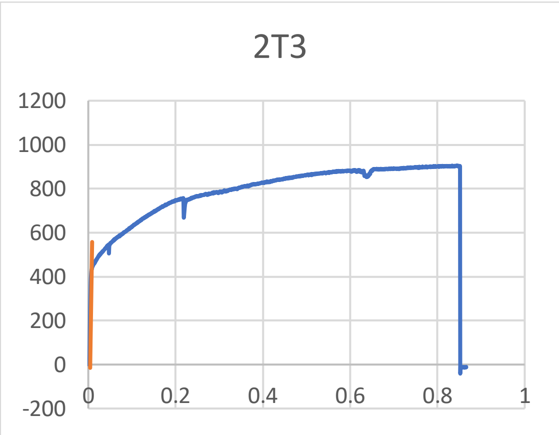

Tensile Testing 1T9
Stress (MPa)
1000
800
0
-200
0
0.1
0.2
0.3
0.4
0.5
0.6
Strain

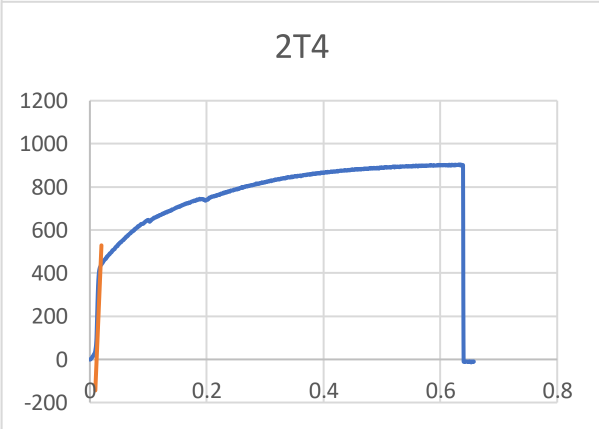

Tensile Testing 1T10
Stress (MPa)
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
Strain

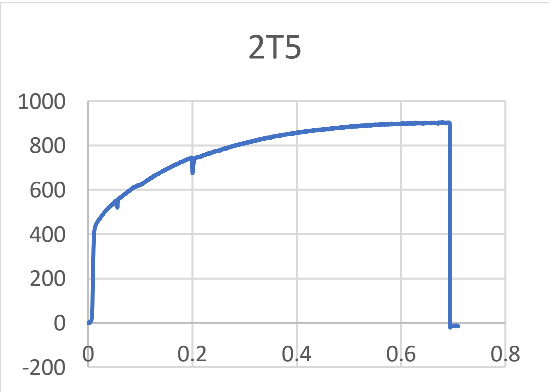

Tensile Testing 1T11
Stress (MPa)
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
Strain

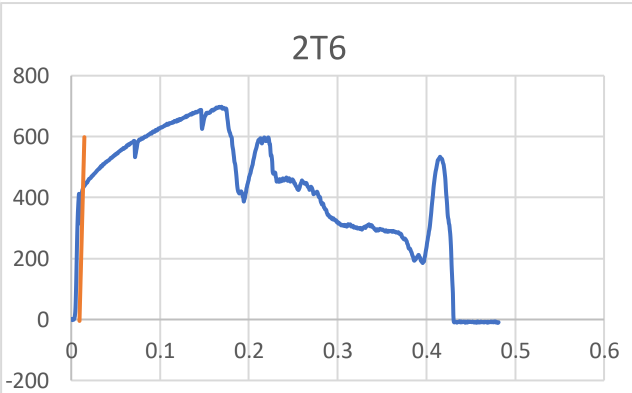

2T1
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

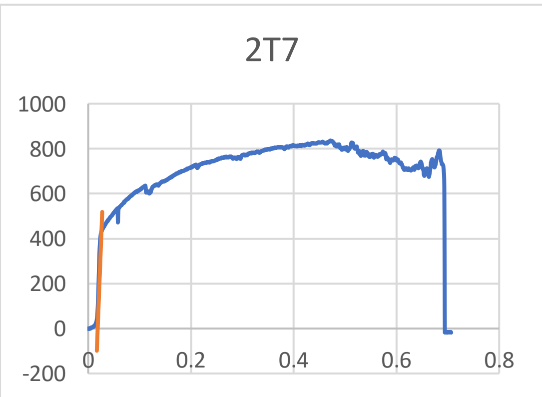

2T2
1000
800
600
400
200
0
-200
-400
0
0.2
0.4
0.6
0.8

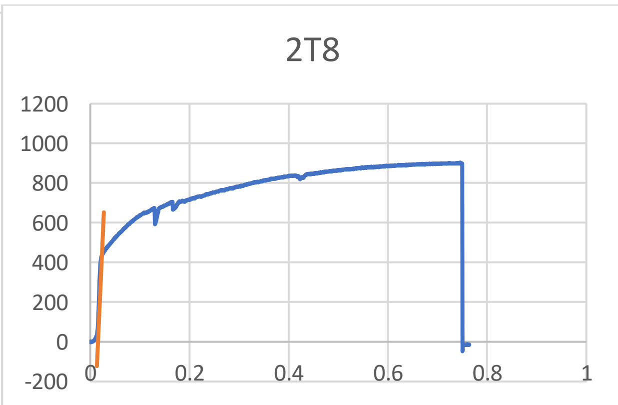

2T3
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

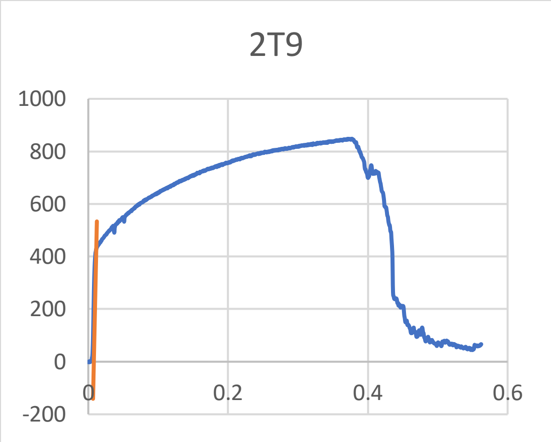

2T4
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

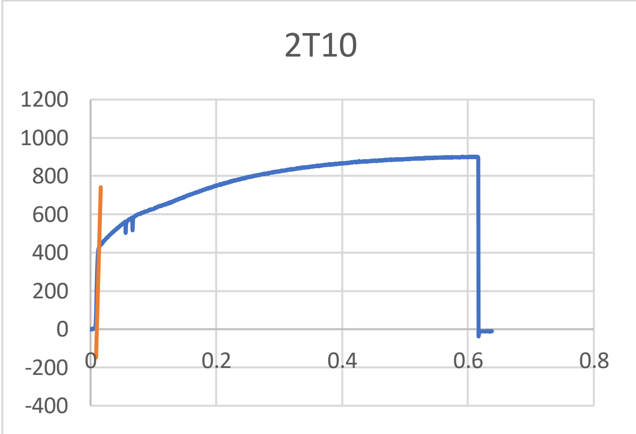

2T5
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

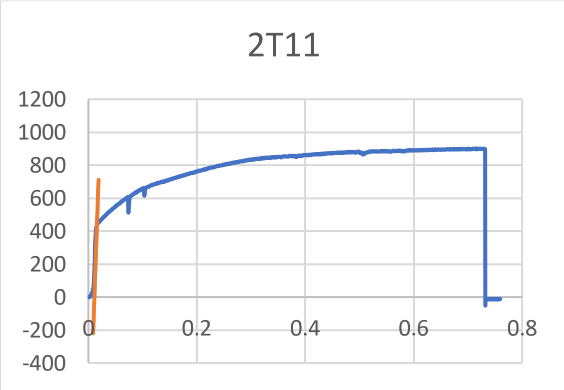

2T6
800
600
400
200
0
-200
0
0.1
0.2
0.3
0.4
0.5
0.6

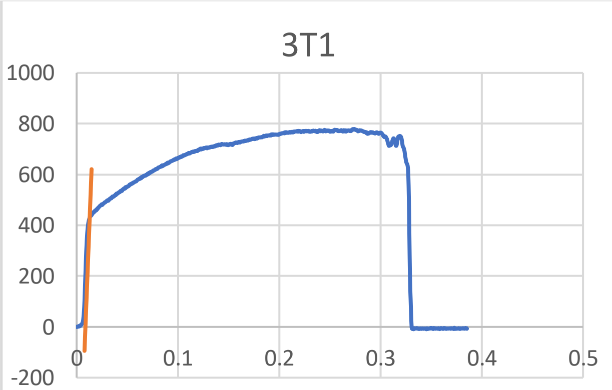

2T7
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

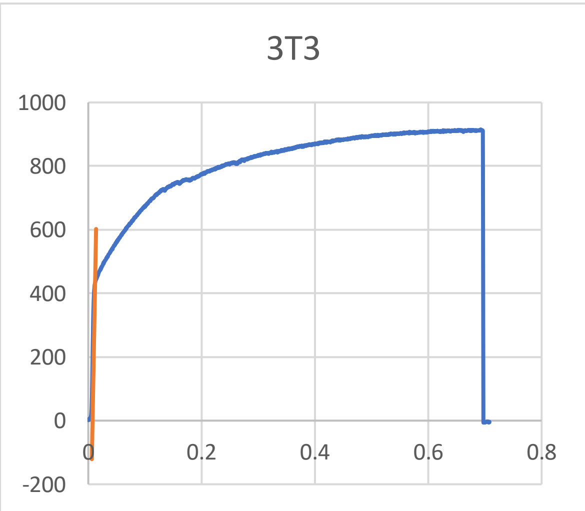

2T8
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

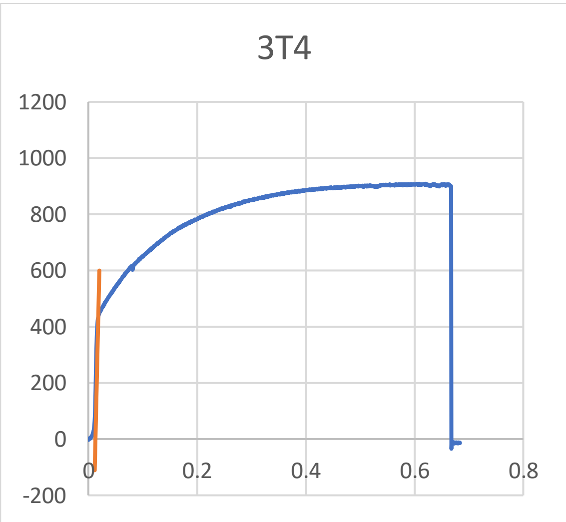

2T9
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6

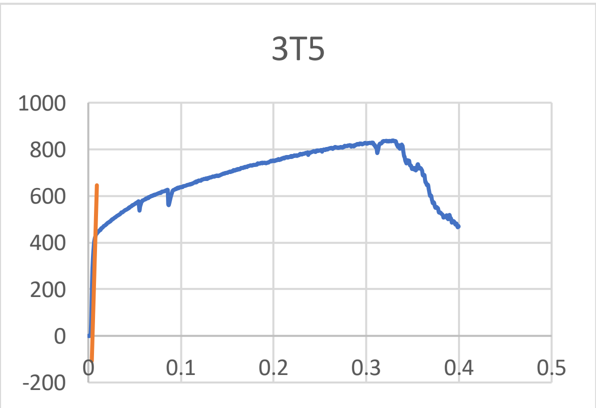

2T10
1200
1000
800
600
400
200
0
-200
-400
0
0.2
0.4
0.6
0.8

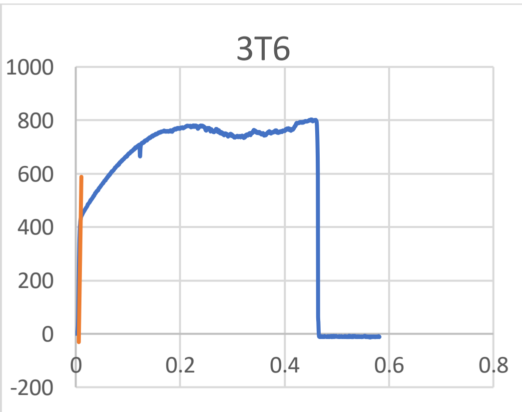

2T11
1200
1000
800
600
400
200
0
-200
-400
0
0.2
0.4
0.6
0.8

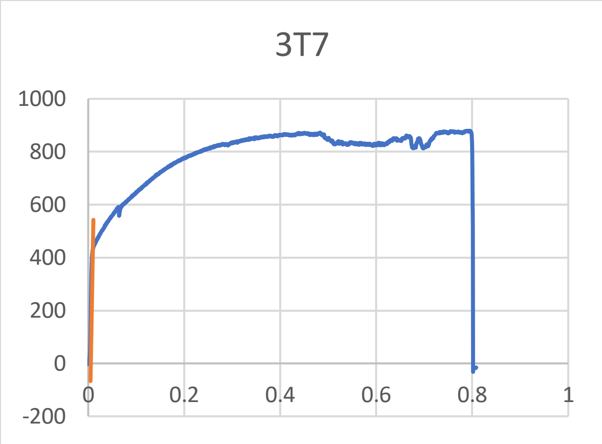

3T1
1000
800
600
400
200
0
-200
0
0.1
0.2
0.3
0.4
0.5

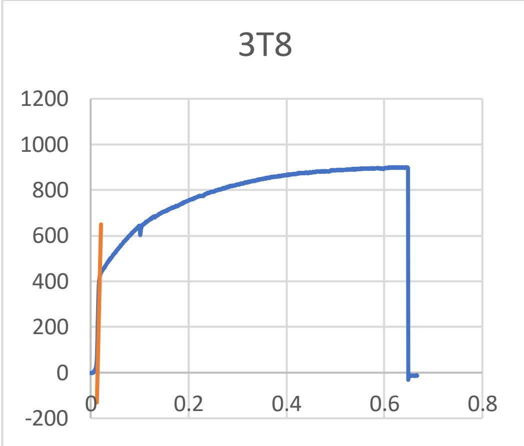

3T3
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

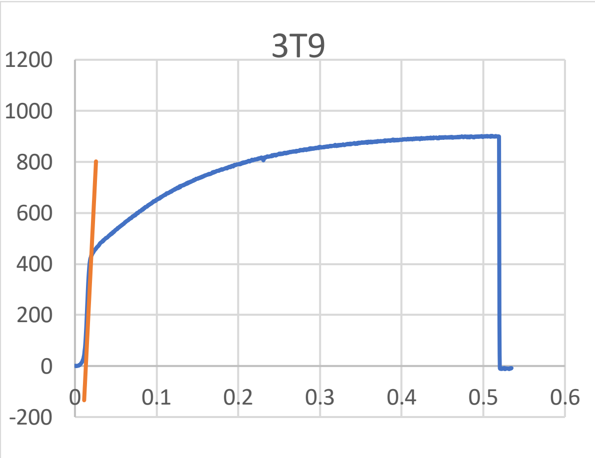

3T4
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

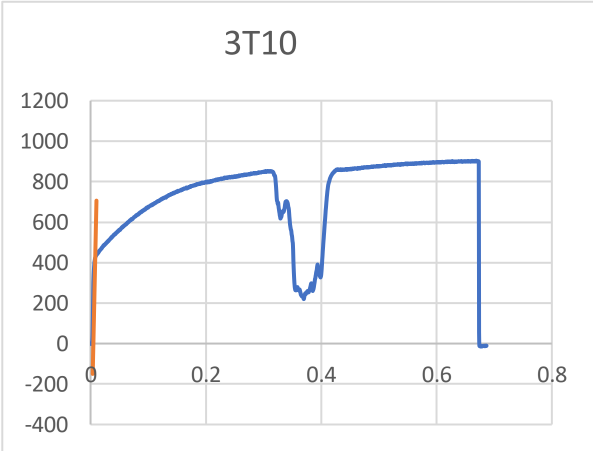

3T5
1000
800
600
400
200
0
-200
0
0.1
0.2
0.3
0.4
0.5

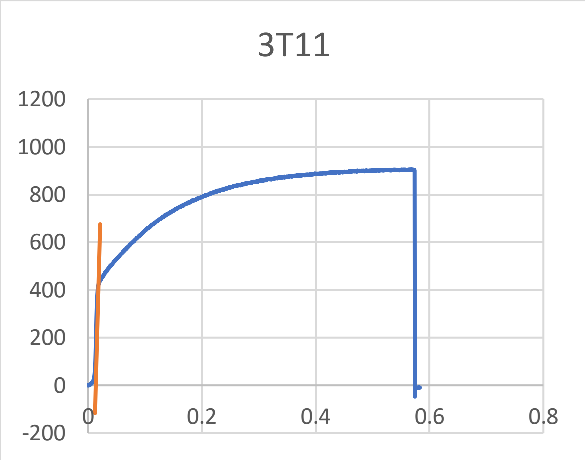

3T6
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

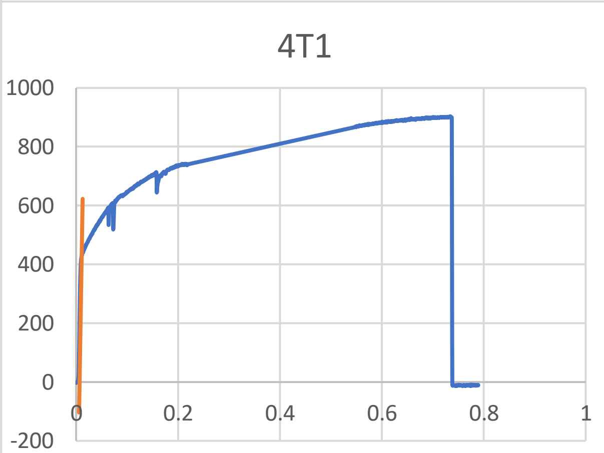

3T7
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

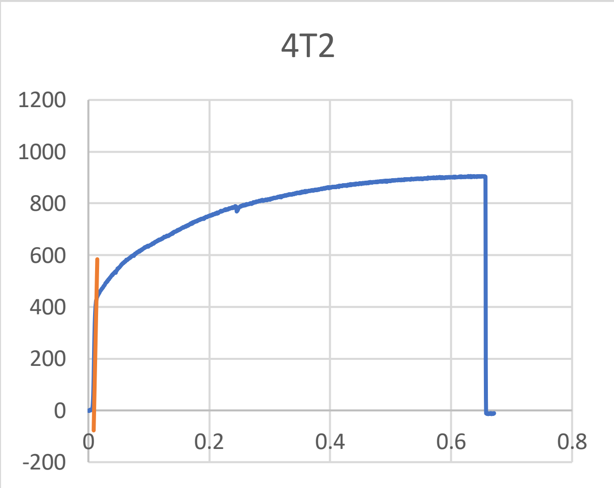

3T8
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

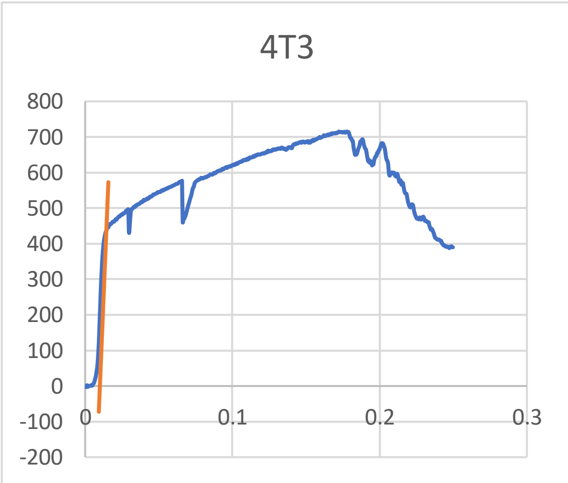

3T9
1200
1000
800
600
400
200
0
-200
0
0.1
0.2
0.3
0.4
0.5
0.6

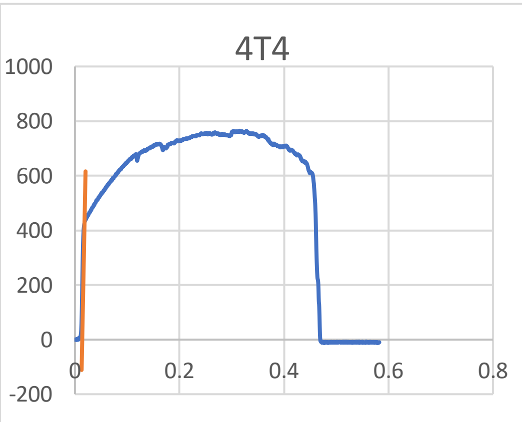

3T10
1200
1000
800
600
400
200
0
-200
-400
0
0.2
0.4
0.6
0.8

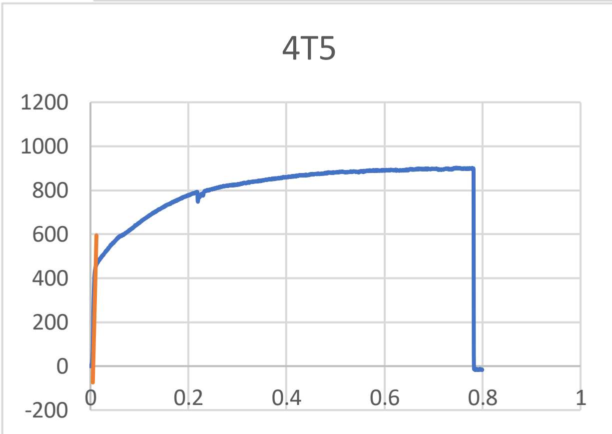

3T11
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

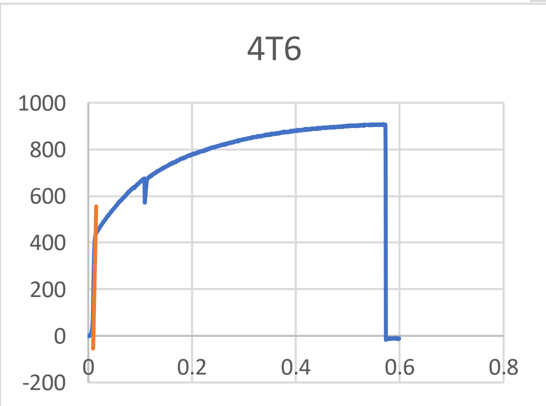

4T1
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

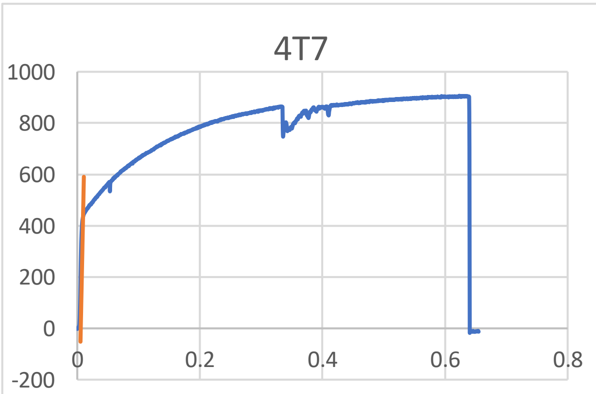

4T2
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

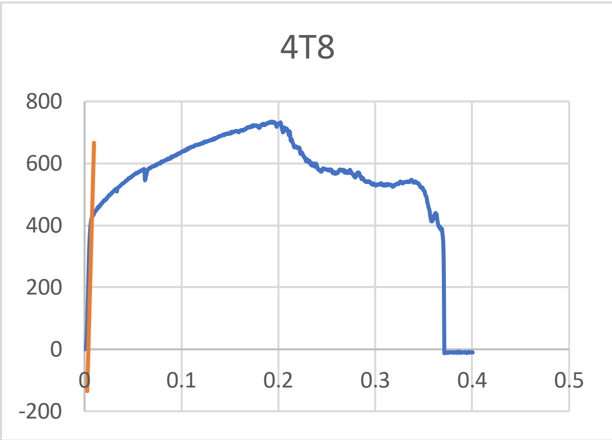

4T3
800
700
600
500
400
300
200
100
0
-100
-200
0
0.1
0.2
0.3

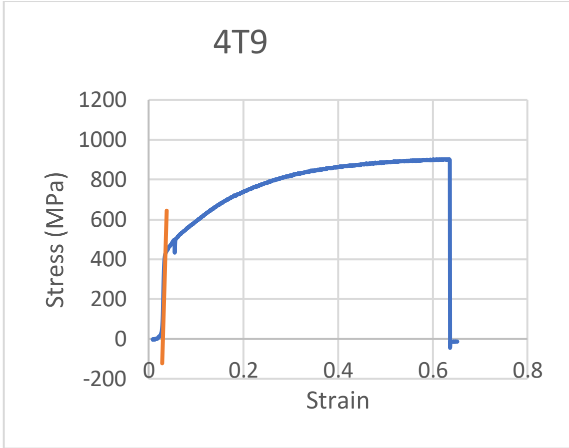

4T4
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

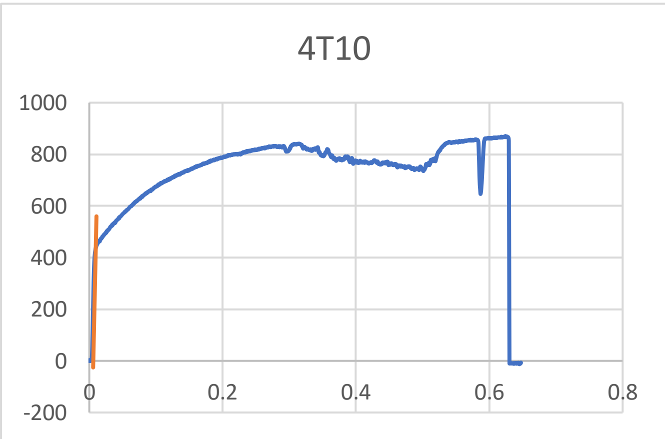

4T5
1200
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8
1

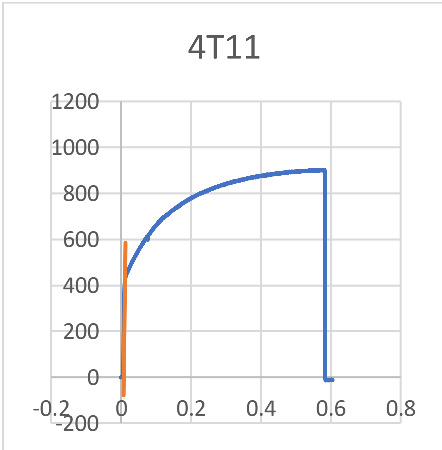

4T6
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8


4T7
1000
800
600
400
200
0
-200
0
0.2
0.4
0.6
0.8

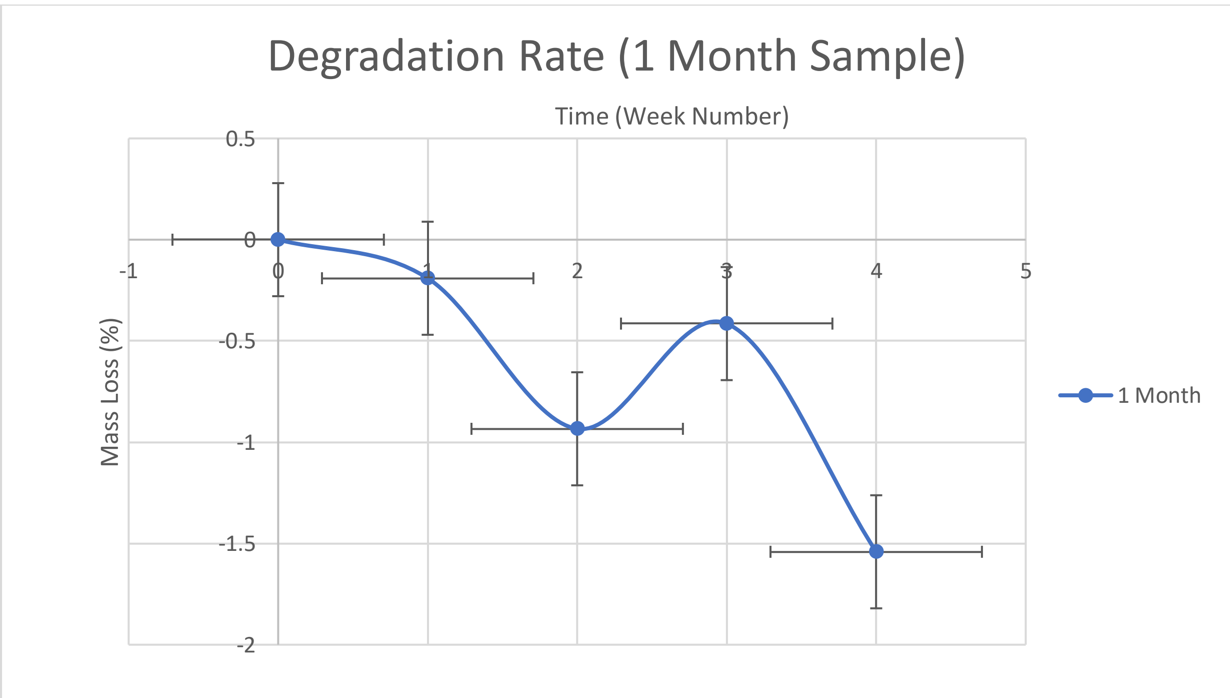


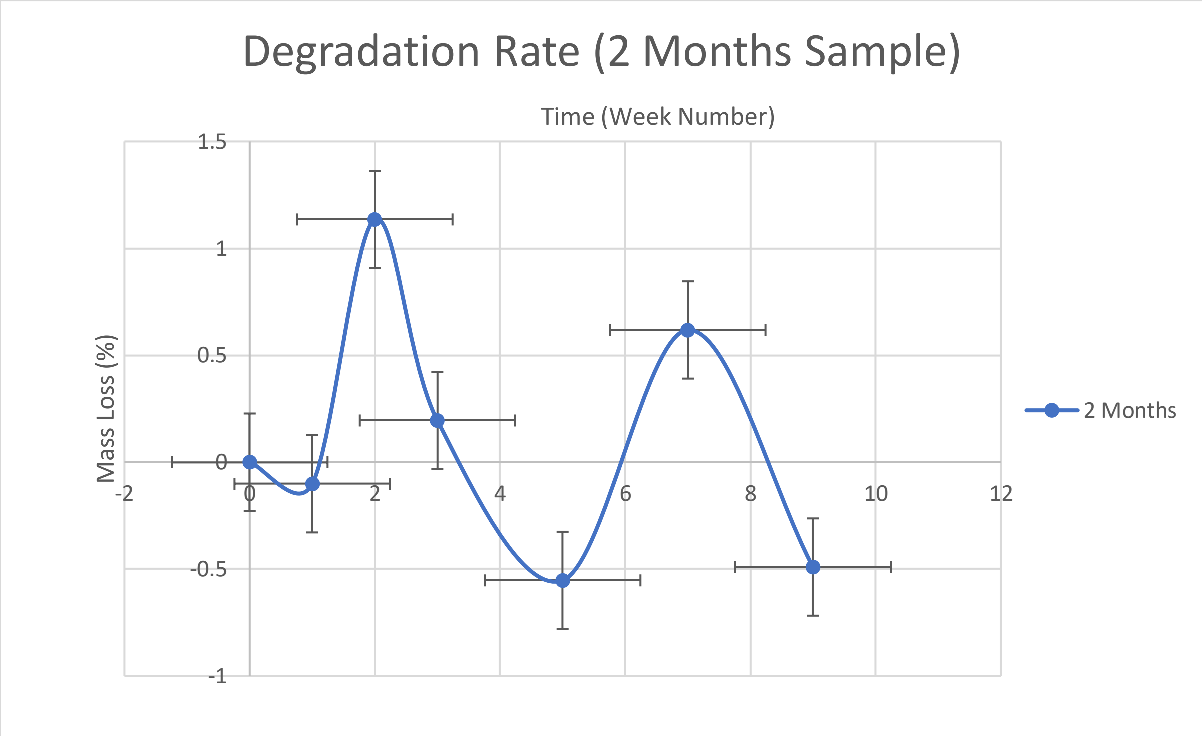


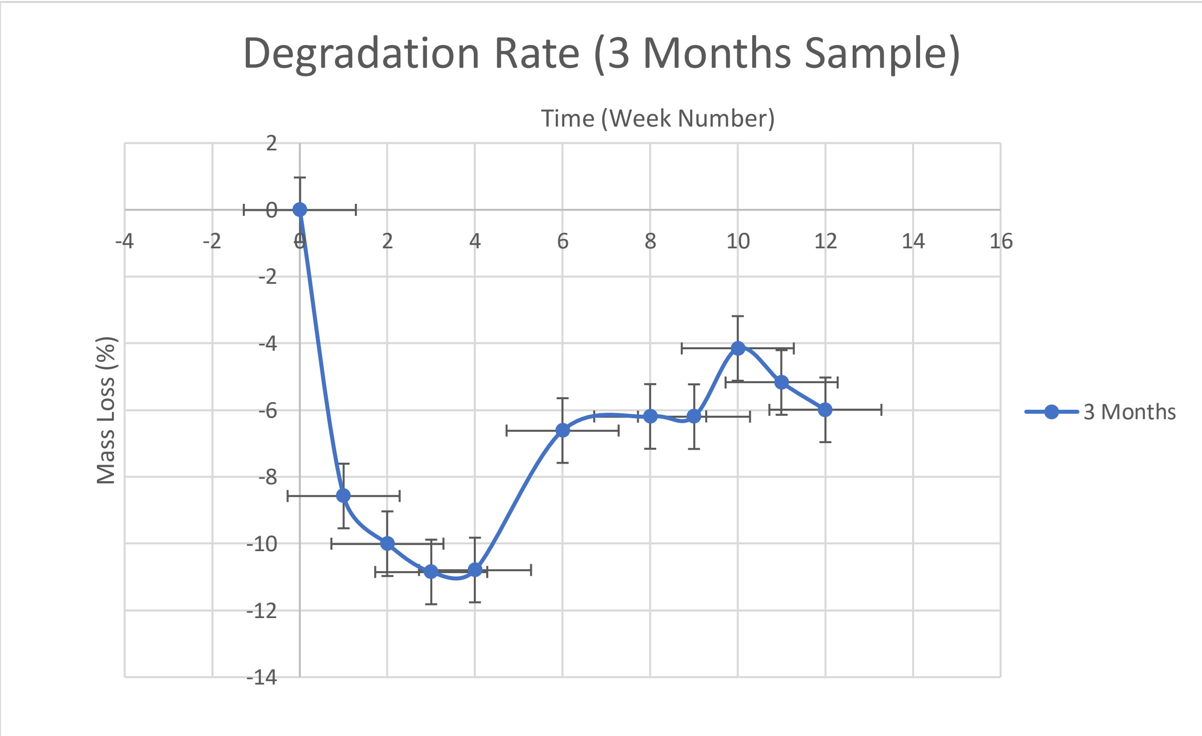


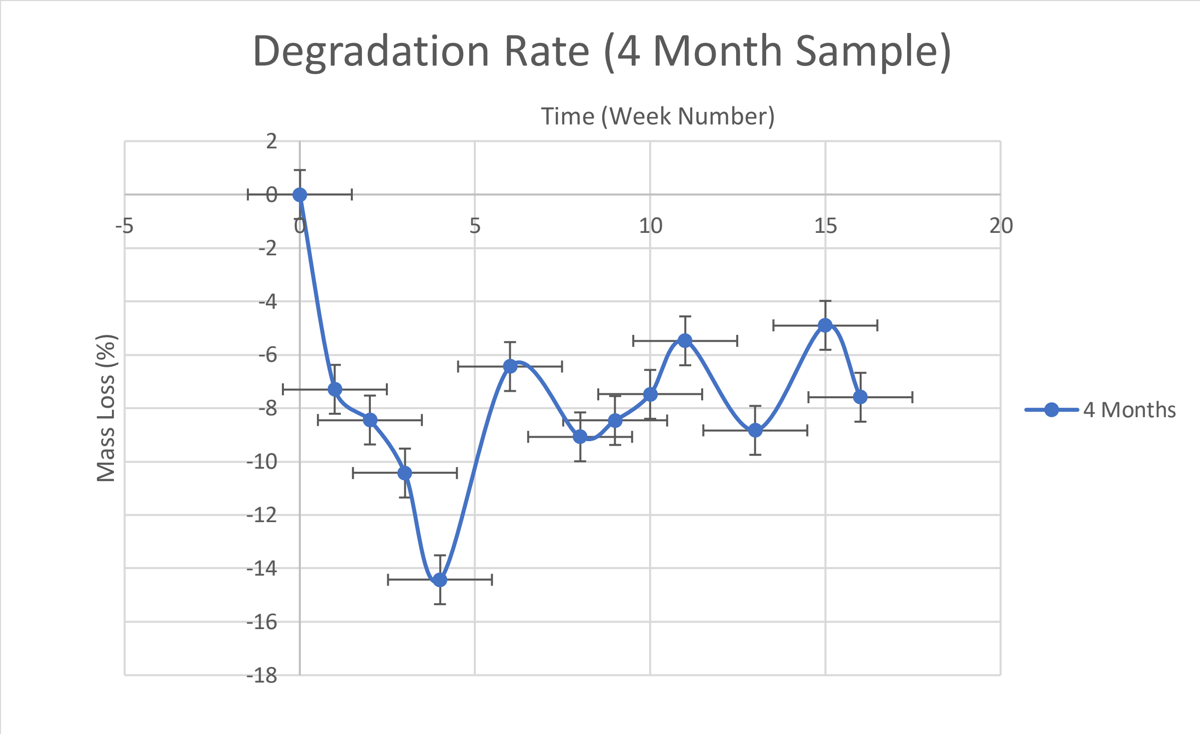


8.4. Mass Loss Results




Degradation Rate (2 Months Sample)
Time (Week Number)
Mass Loss (%)
1.5
1
0.5
0
-0.5
-1
-2
0
2
4
6
8
10
12
2 Months


Degradation Rate (3 Months Sample)
Time (Week Number)
Mass Loss (%)
2
0
-2
-4
-6
-8
-10
-12
-14
-4
-2
0
2
4
6
8
10
12
14
16
3 Months



8.5. Yield Stress Results

0	0	1	2	3	4
1	456.535801	437.3902975	428.4138071	430.2903	429.246
2	451.3703469	426.2099813	435.2208298		428.0901
3	447.4272093	433.3580952	432.9449661	432.8076	436.783
4	441.1846895	394.9267857	434.3633522	433.2775	427.4778
5	433.2508262	428.6447296	436.4052106	425.574	451.2947
6	435.1371975	421.5248323	429.577057	430.8237	434.2828
7	434.9677522	427.5026291	433.7402572	433.175	434.1759
8	439.171254	421.4670301	434.5157253	423.7074	427.3074
9	439.6496999	429.7893877	428.8309959	433.0452	428.3081
10	432.7712876	422.2176124	429.3229458	424.4848	436.8212
11	431.2143848	430.392013	436.5446206	423.2141	425.173
Average	440.2436772	424.8566722	432.7163425	429.04	432.6327
Standard Deviation	8.266824227	11.103482	3.107490818	4.284055	7.413013
Wieball's Ditribution	441.4548776	426.5607651	433.1463152	429.7134	433.8509

8.6 Ultimate Tensile Strength

0	0	1	2	3	4
1		844.4026			901.9673
2	919.1332	877.8547	904.1984		905.2946
3	919.0972	905.9809	904.8229	913.9281	
4	916.6405	906.778	904.1261	906.3982	
5		912.2083	904.8745		901.1308
6	913.0442	904.7366		801.6641	907.0094
7	914.6033	907.7058	792.2297	879.1869	904.8748
8	911.9375	904.2996	900.6763	901.1967	
9	916.3947			900.1043	901.0824
10	900.2396	911.0548	901.3931	903.789	869.05
11	901.7484	901.4969	900.1612	905.6909	902.4235
Average	912.5376	897.6518	889.0603	888.9948	899.1041
Standard Deviation	6.989049	21.03397	39.17199	36.6788	12.33234
Wieball's	913.4827	900.8488	895.5818	894.1828	894.6253

8.7 Strain at Fracture

0	0	1	2	3	4
1		0.255288			0.726797
2	0.573159	0.450799	0.720273		0.65627
3	0.599786	0.695776	0.851785	0.696275	
4	0.628784	0.632776	0.638776	0.667272	
5		0.645769	0.693268		0.751286
6	0.620285	0.643785		0.463785	0.572268
7	0.605285	0.552777	0.693283	0.800272	0.639272
8	0.649784	0.632271	0.749783	0.648273	
9	0.589285			0.518772	0.636225
10	0.642269	0.628285	0.616285	0.673283	0.629281
11	0.646773	0.550279	0.73177	0.573783	0.583543
Average	0.617268	0.56878	0.711903	0.630214	0.649368
Standard Deviation	0.027093	0.130019	0.072359	0.106998	0.062548
Wieball's Ditribution	0.620563	0.57595	0.718587	0.636657	0.655399