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Fatigue Behavior of Ti-6-4 Alloy with Application of Calcium Phosphate

THESIS

submitted in partial satisfaction of the requirements
for the degree of

MASTER OF SCIENCE

in Materials Science & Engineering

by

Jaspal Singh Kamboj

Thesis Committee:
Professor James C. Earthman, Chair
Professor Farghalli A. Mohamed
Associate Professor Daniel Mumm

2014

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DEDICATION

To

my family and friends

in recognition of their worth

"…I believe there exists, and I feel within me, an instinct for the truth, or knowledge or discovery, of something of the same nature as the instinct of virtue, and that our having such an instinct is reason enough for scientific researches without any practical results ever ensuing from them"

- Charles Darwin

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ABSTRACT OF THE THESIS

Fatigue Behavior of Ti-6-4 Alloy with Application of Calcium Phosphate

By

Jaspal Singh Kamboj

Master of Science in Materials Science & Engineering

University of California, Irvine, 2014

Professor James C. Earthman, Chair

It is key to consider a material's mechanical properties when determining its use for any given application, including biomaterial implantations. One such characteristic includes the fatigue life, which is determined by repeatedly cycling specified amounts of load on any given material [2]. The fatigue life is then measured according to how many cycles of load the material can undergo before fracture [2]. When a material is introduced to the human body it is usually expected that the life of the material will not match that of the patient. It is crucial to understand the fatigue life of the material before implantation to pre-diagnose how often and when the implant will need to be replaced.

The purpose of this project is to help determine and compare the fatigue behaviors of a commonly used biocompatible coating on Titanium 6-4 metal alloy. The comparison will help identify how the material properties vary with the addition of calcium phosphate when compared to the bare alloy itself.

Multiple, small rectangular samples were cut by electrical discharge machining (EDM) of which half were covered with calcium phosphate coating. Fatigue crack initiations and

propagation would then be analyzed using scanning electron microscopy (SEM) to help determine the nature of the damage processes. It was found that the fatigue life of the coated samples varied at different stresses but was greater than the bare samples, and particularly high in the 900 MPa - 1000 MPa stress range.

Chapter 1

Introduction

It is a well-known fact that titanium based alloys are among the most biocompatible materials used for implants today. Although the biocompatibility varies from one individual to another, coatings help further reduce any immune reaction that the body may trigger in response to foreign materials [3]. Coatings such as Calcium Phosphate ceramics are among the many used today especially for orthopedic implantations. It is not only important for the material and coating to be biocompatible but in many instances this paring should also aid, for example, in tissue bone growth [1]. Knowing the lifespan of a material under given circumstances is vital information. Since materials are subjected to these repeated load cycles (fatigue) it is important to determine the total number of life cycles before material failure, which once again is defined as the materials fatigue life. Fatigue testing therefore gives a more accurate representation of how a material will behave in certain environments.

Titanium alloys are also known for having excellent corrosion resistance and overall material properties. They are commonly used as biomaterials since it has shown good bonding behavior with many types of coatings and does well in most vivo environments [1]. Therefore, the adherence of calcium phosphate coatings used in this experiment should not be an issue.

Chapter 2

Materials and Methods

2.1 Material Preparation

A large Ti-6-4 sheet was the original product purchased from TIMETAL, (batch number: AMS 4911_J; AMS-T9046_A AB-1; DMS 1592_F) from which several smaller shims would be made. The exact material composition can be seen in Table 1. These shims were cut via EDM (electron discharge machining) to the dimensions of 38mm x 0.5mm x 4mm. The shims were polished after the cutting process. Grit sizes of 400, 600, 800 and finally 1200 would be used to sand and polish the samples down to an average final thickness of 0.35mm.

Element	C	Fe	N	Al	V	O
Top	0.008	0.17	0.003	6.22	4.01	0.14
Bottom	0.010	0.14	0.003	6.25	3.99	0.14

Table 1.1: Chemical Analysis (weight percent) of Ti-6-4 alloy by TIMETAL

A custom-built three point bending machine was used for the fatigue testing. Each of the Ti 6-4 samples, both coated and bare, were tested at load amplitudes of roughly 10 - 12 N at a frequency of 30Hz. The fatigue machine is controlled through a desktop computer that runs a custom developed program in the LabView graphical programming environment (full setup is shown in Figure 1.1). After the fatigue testing, the samples were analyzed using SEM. Roughly 10 samples were machined via EDM

from the original Ti 6-4 sheet (each sample is roughly 38mm x 4mm with a thickness of 0.5mm) (Figure 1.2).

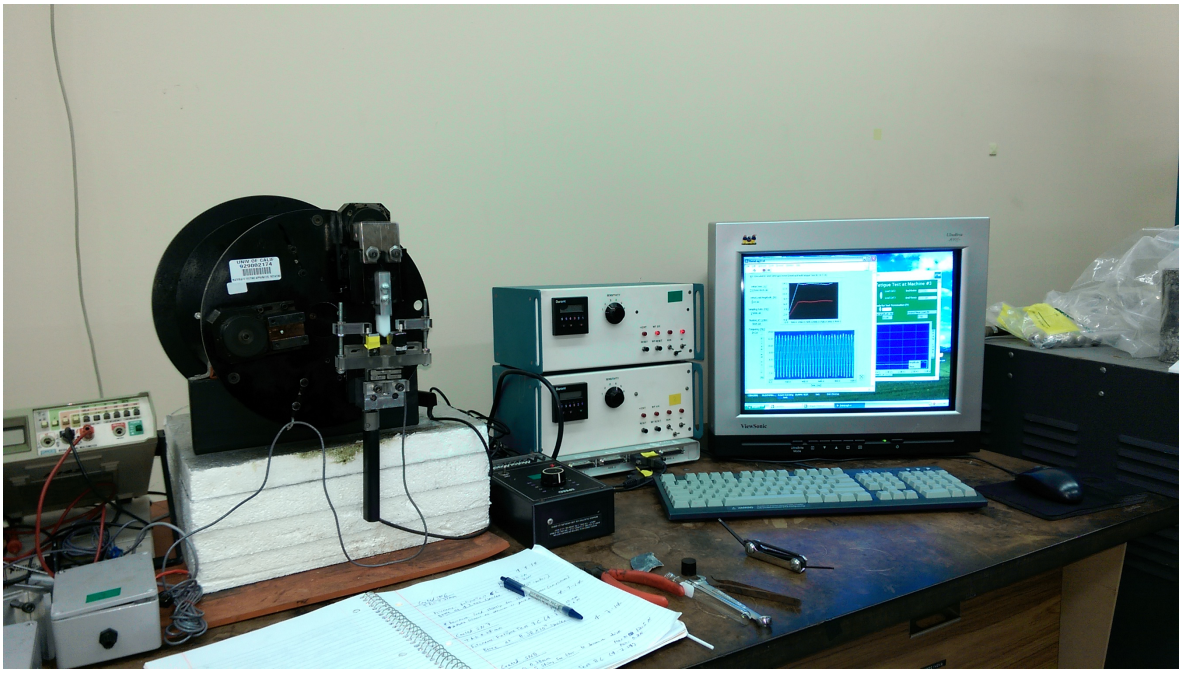

Figure 1.1: Testing equipment for three point-bend fatigue system

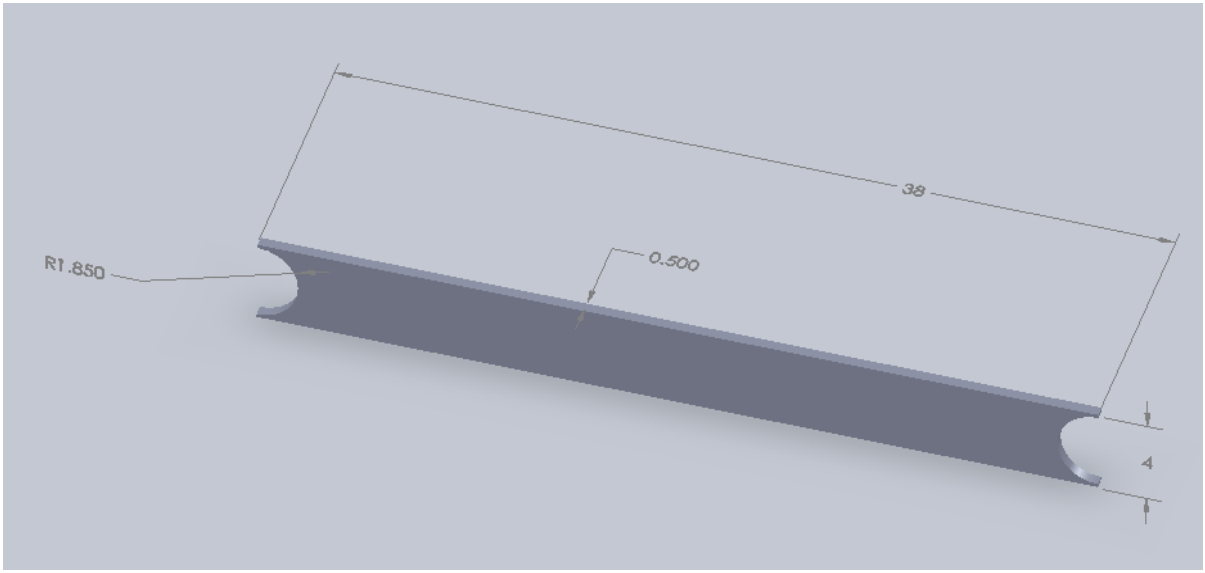

Figure 1.2: Drawing of the EDM samples. Both ends of the sample have a radii cut. This is where the columns of the fatigue machine are to hold the sample in place during testing.

After the EDM samples have been cut from the Titanium sheet they will require polishing. Each of the samples were be polished starting from a grit size of 400 to 1200. Since the EDM samples were small and thin, they required a custom machined aluminum block to hold them in place during the polishing procedure. The sanding/polishing brought the final thickness of the samples down to an average of 0.35 mm.

2.2 Three point bending test

All experiments were performed under three point bending fatigue. Illustration of this configuration is shown in Figure 2.1. This configuration follows the ASTM E855 standard. The span size used was roughly 28 mm with a sinusoidal deflection waveform of 30 Hz. The maximum stress in this configuration is thus at the outer fiber of the longitudinal midpoint for the samples. This is given by the equation

$$\sigma_{\max} = \frac{3PL}{2bh^2} \tag{1}$$

where P is defined as the applied load, L is the length of the span (or the distance between the two loading points on the tensile side of the sample), b is width of the sample and h is the thickness of the sample. The stress at the outer fiber at a given longitudinal distance is then shown by the equation

$$\sigma_D = \frac{\sigma \max (L-2D)}{L} \quad (2)$$

Throughout the experiments, the mean load was kept constant via the acquisition and control system part of the fatigue machine that included a Transducer Techniques MLP-50 load cell as well as a Newport Model CMA-12CCCL motor drive shown in Figure 2.1.

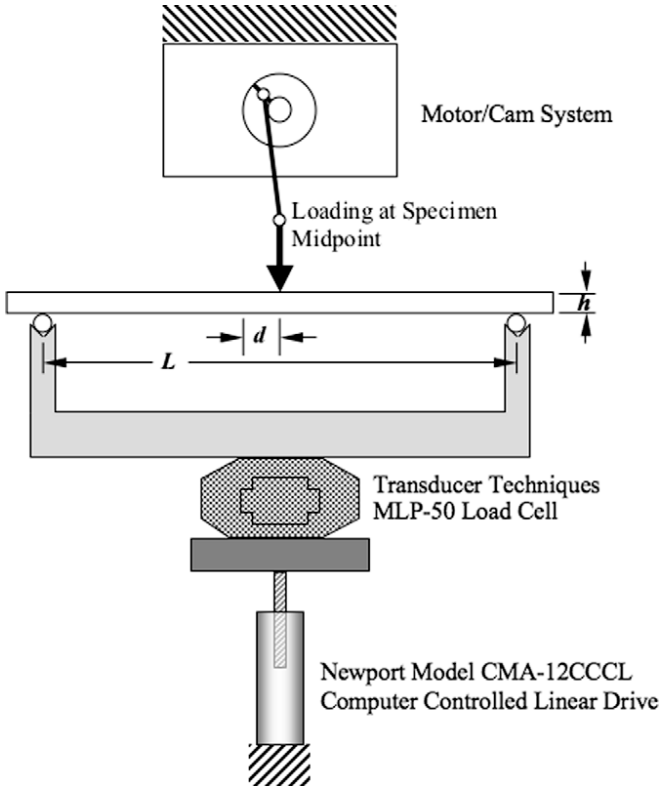

Figure 2.1: Representing the three-point bending fatigue set-up for the experiments

A mean stress ratio ($R = \frac{\sigma_{min}}{\sigma_{max}}$) of 0.2 was maintained throughout the testing. Cyclic displacement amplitude is placed on the samples using a rotating cam as illustrated in Figure 2.1. The LabView program records the number of cycles to failure (when a load

drop is detected), the minimum and maximum mean load, length of testing, load amplitude and frequency.

Chapter 3

Calcium Phosphate Coating

3.1 – Coating preparation and procedure

Before the samples could be coated with the calcium phosphate solution, a pre-treatment was required to assure the surface would be clean and optimal for the adherence of the calcium phosphate coating. The pre-treatment used was a procedure that required a two-day submersion in solution [4]. The samples were first ultrasonically cleaned in acetone for 10 minutes followed by ethanol, also for 10 minutes. After the samples were ultrasonically cleaned, they were rinsed in distilled water and dried.

Small vials were used for the pre-treatment into which a 5M sodium hydroxide solution was placed. The samples were then submerged into the solution and left to age. One sample per vial was used. The samples were left in the solution for approximately 48 hours after which they were removed from the vials and rinsed in distilled water and dried off. The coating procedure was to be followed after the pre-treatment.

The coating procedure followed required a supersaturated calcification solution technique [5]. The coating consisted of a mixed salt solution that contained calcium chloride, sodium phosphate and sodium bicarbonate. Table 1 shows the concentration of each solid used to make the calcium phosphate coating.

CaCl2	NaH2PO4	NaHCO3
555mg	600mg	126mg

Table 3.1: Mass of salts used for calcium phosphate solution

Small beakers were filled with the calcium phosphate solution. In these beakers the samples were hanging via a thin fishing line so that the coating would be uniformly applied throughout the entire sample. The small beakers were then placed inside a water bath which was digitally controlled to maintain a bath temperature of 37°C. The water bath contained individual compartments in which the beakers would sit and would allow to be shaken at 80 rpm. The beakers were left in the bath for a 48-hour period over which the bath was sealed from the top to minimize evaporation of the water in the bath as well as the calcium chloride solution. After the samples had been in the solution for 48 hours, they were taken out from the beaker and rinsed with distilled water. Immediately following the coating procedure, the samples would be placed in a small oven and left to bake for approximately two hours at a temperature of 60°C. After the baking process the samples were taken out and left to cool until reaching room temperature.

3.2 Coating Verification

To determine the surface coating presence, energy-dispersive X-ray spectroscopy (EDS/EDX) was used. Different areas of the coated samples were chosen to perform EDS and retrieve information via both spectrum and mapping. Figure 3.1 shows various peaks

of the elements that are picked up by EDS. These elements are representative of both the ones contained in the titanium alloy as well as the surface coating. The goal here is to show that the calcium phosphate does indeed exist at the surface of the substrate. Higher peaks of titanium (Ti) and aluminum (Al) are seen as expected whereas peaks of calcium (Ca) and phosphorus (P) are comparatively lower.

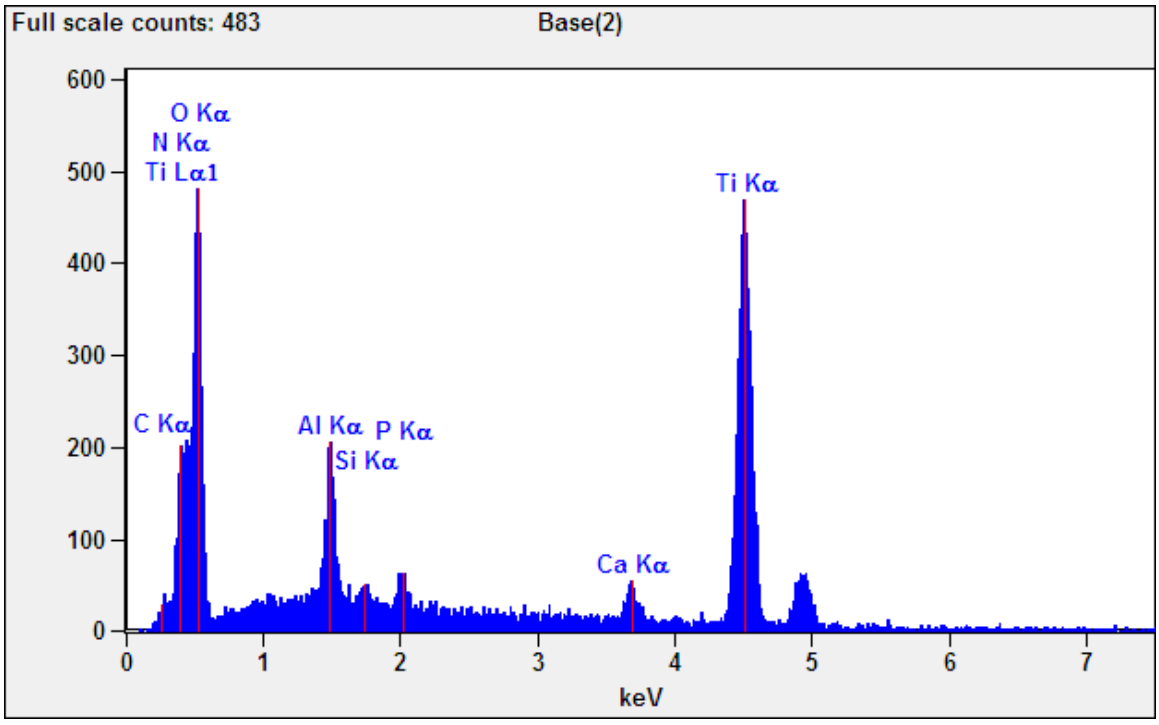

Figure 3.1: EDS mapping of substrate and coating

This shows that the calcium phosphate coating does exist at the substrate however, the EDS mapping images would further explain the uniform presence of the coating across the surface.

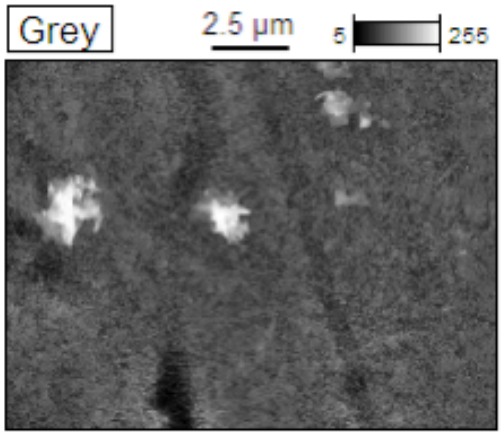

Figure 3.2(a): Area sampled in EDS

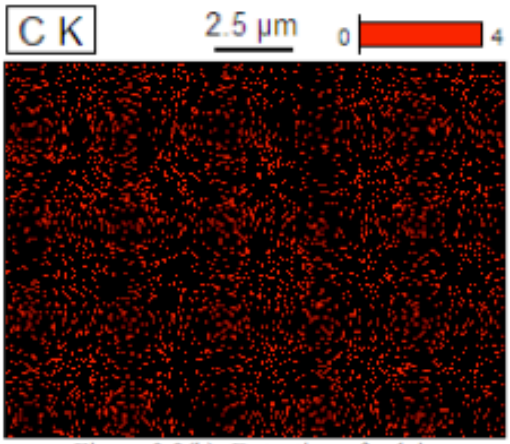

Figure 3.2(b): Detection of calcium

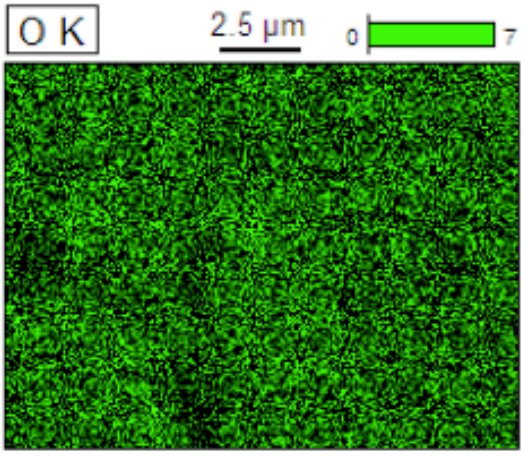

Figure 3.2(c): Detection of oxygen

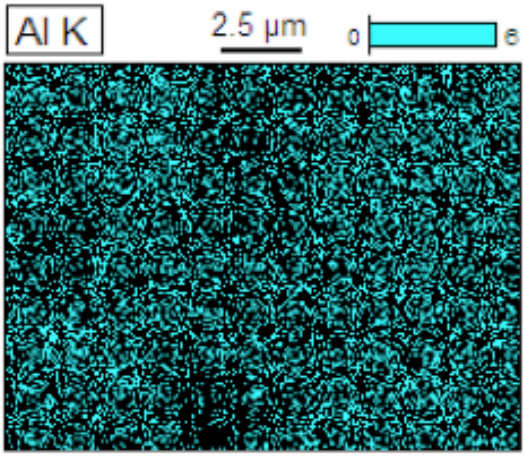

Figure 3.2(d): Detection of Aluminum

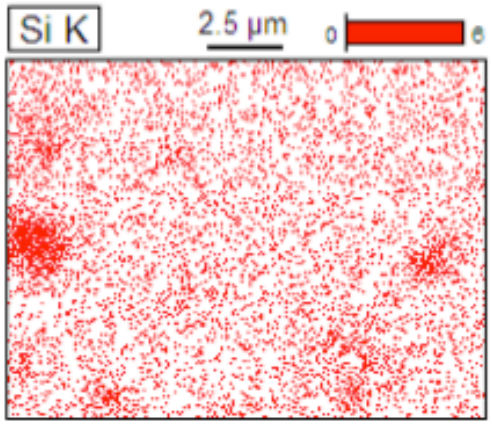

Figure 3.2(e): Detection of Silicon

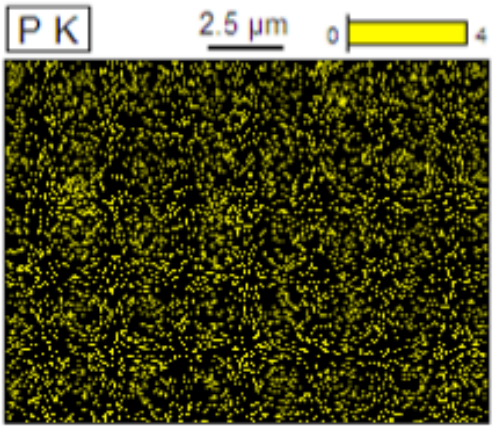

Figure 3.2(e): Detection of Phosphorus

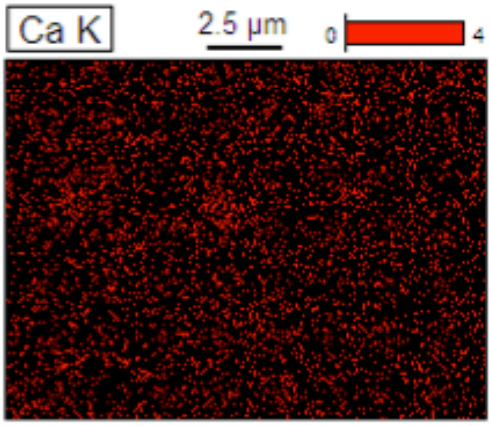

Figure 3.2(g): Detection of Calcium

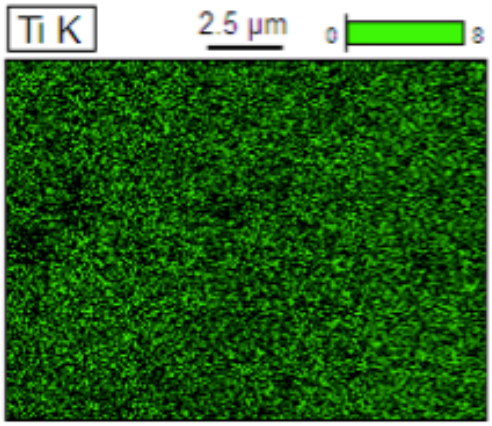

Figure 3.2(h): Detection of Titanium

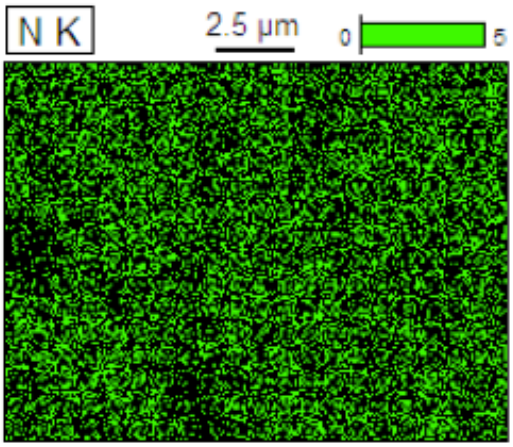

Figure 3.2(i): Detection of Nitrogen

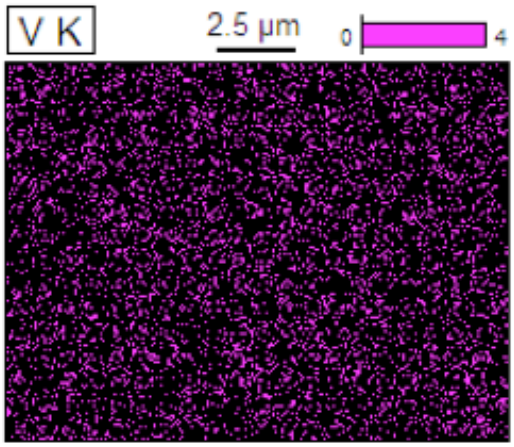

Figure 3.2(j): Detection of Vanadium

Figure 3.2(a) is the area that is being mapped to show which part of the sample was chosen for the mapping. From these images we can confirm that the calcium phosphate coating is uniformly spread throughout the substrate. A small count of silicon is apparent on the material as well but this is most likely due to dust particles that the material may have picked up. Also notice that the titanium substrate is significantly higher in terms of counts, which is expected since the coating is relatively thin.

Throughout the surface of the material, precipitates can be found scattered in a random fashion. First thought is that these are part of the calcium phosphate coating and are rather clumps of slat built up together as seen in Figure 3.3a. To confirm that these

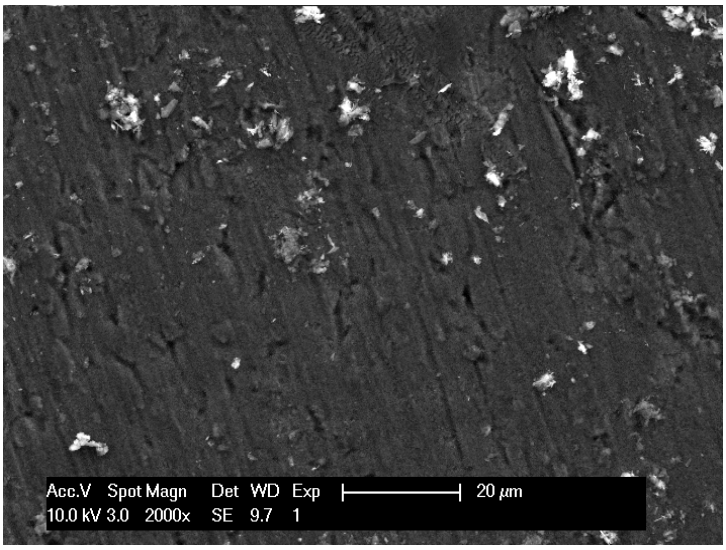

Figure 3.3(a): Precipitates found at surface of coated samples

Figure 3.3(b): Higher magnification of area seen in Figure 3.4(a)

are indeed precipitates formed by from the coating procedure and not dust or other particles, EDX mapping was performed on various (Figures 3.6) precipitates on the substrate. The above SEM images (Figure 3.3a,b) show the randomly scattered precipitates on the substrate. A larger precipitate found shows a common salt-like

crystalline structure. Many jagged edges oriented outward are observed, further indicating that these are indeed the salts that are contained in the coating. EDS was then performed on the precipitates to further confirm that these are indeed part of the calcium phosphate coating (Figure 3.4).

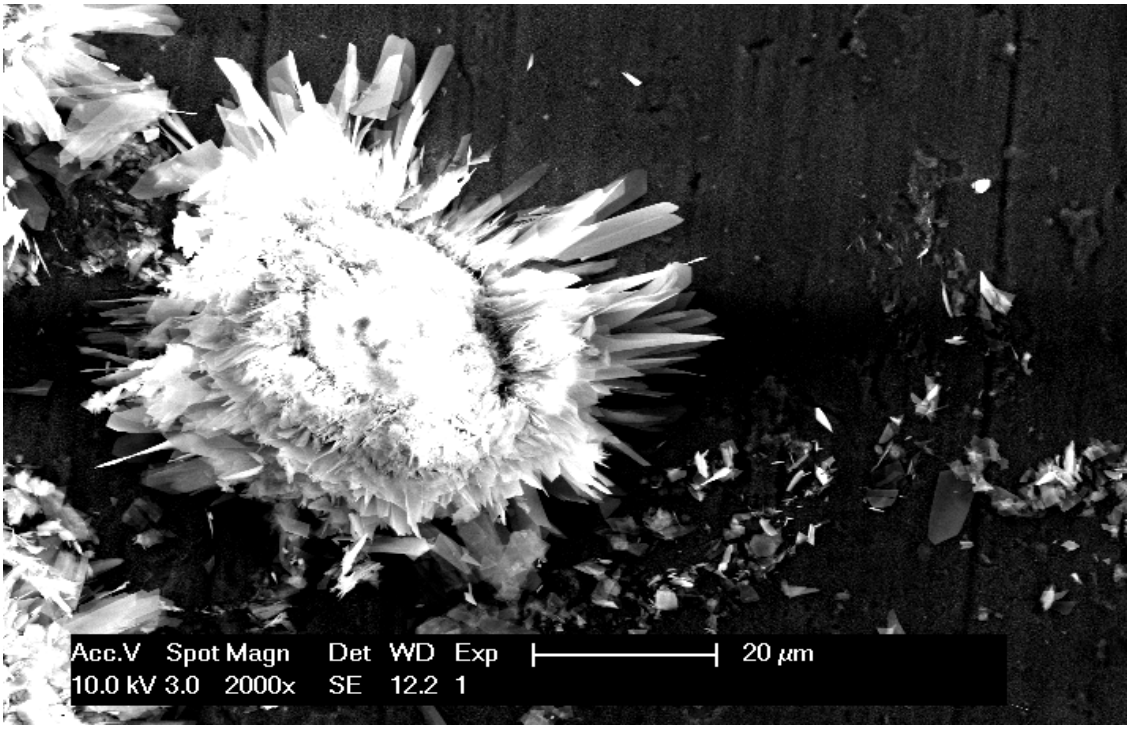

Figure 3.4: Crystalline structure is apparent in the calcium phosphate precipitates

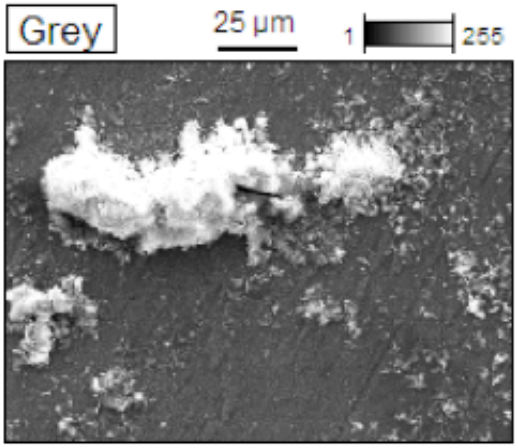

Figure 3.5(a): Precipitate on substrate

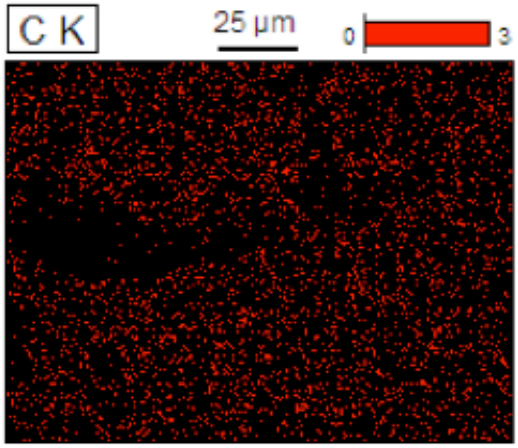

Figure 3.5(b): Calcium presence

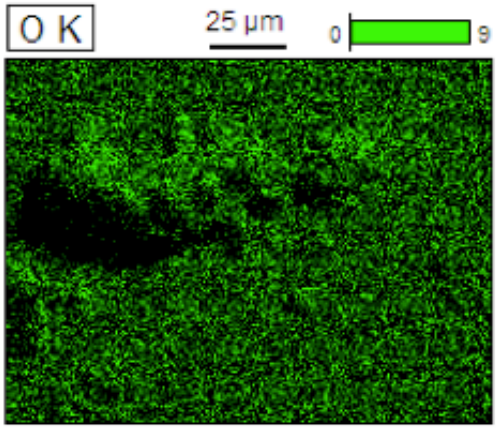

Figure 3.5(c): Oxygen presence

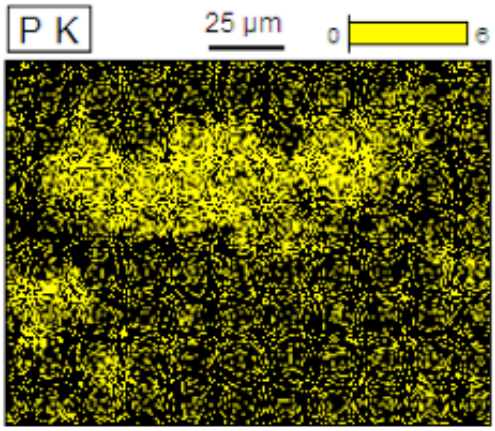

Figure 3.5(d): Phosphorus presence

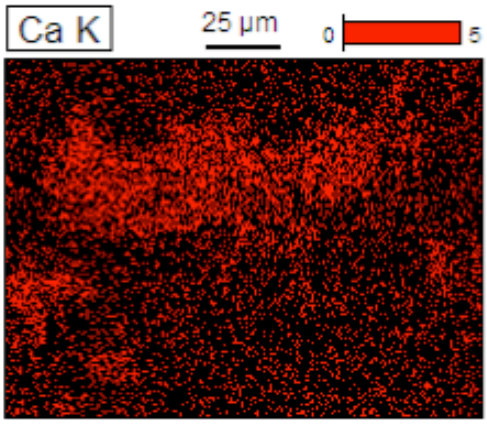

Figure 3.5(d): Calcium presence

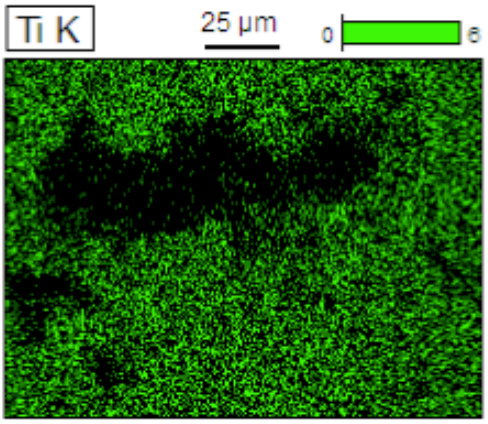

Figure 3.5(e): Titanium presence

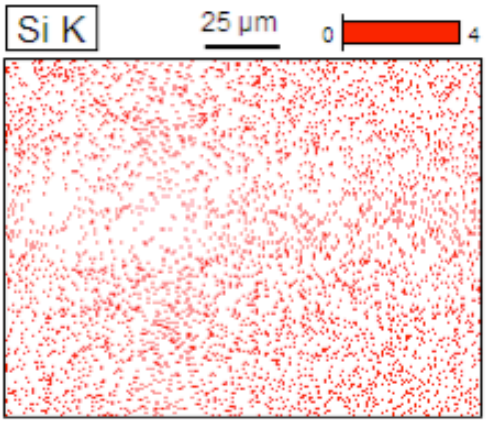

Figure 3.5(f): Silicon presence

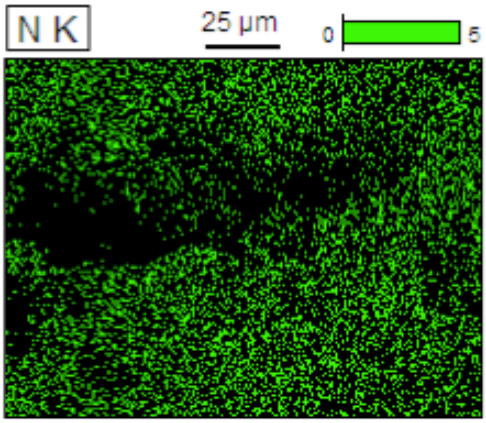

Figure 3.5(g): Nitrogen presence

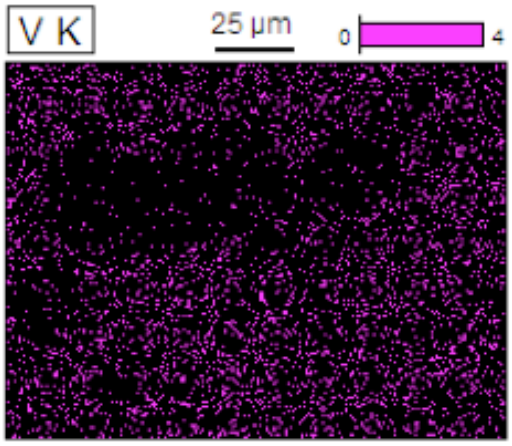

Figure 3.5(h): Vanadium presence

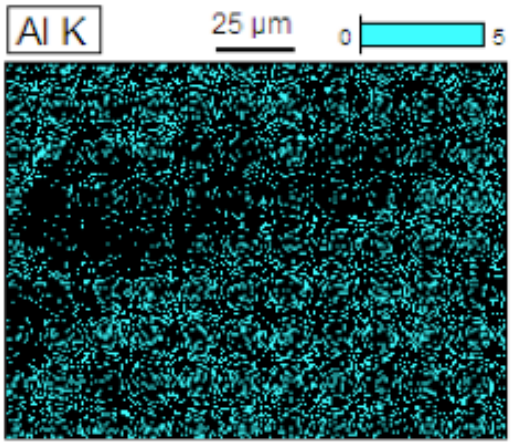

Figure 3.5(i): Aluminum presence

From these images we see that the area and precipitate that was chosen for EDS mapping gives a much higher count of calcium and phosphate than does silicon or the substrate elements, namely titanium. Therefore this confirms our speculation of the precipitates indeed being due to the calcium phosphate coating.

3.3 Coating to Substrate Adherence

Scanning electron microscopy was used for all imaging of the samples. SEM analysis was necessary to determine how well the calcium phosphate coating adhered to the surface of the titanium alloy. Images were taken at several different magnitudes to compare the coating adherence before testing. Figure 3.6(a) shows a fairly consistent and uniform layer of coating on the surface of the material. Figure 3.6(b) is at a higher magnitude of the same area. This uniform coating shows a pattern similar to that found in

the bone matrix. Therefore the coating profile is not only important for the fatigue properties of the material but also further help cells in vivo conditions bind to the surface substrate when dealing with in bone growth [1]. At the top and bottom surfaces of the substrate the coating covers the material throughout as seen in Figure 3.6(a, b). On the edges of the material the coating is found to be partially smeared over the material. As seen in figure 3.8, an SEM image taken from the side of a coated sample, the smearing of the coating slightly covers one side of the edge and only a small portion of it. This may be due to the longitudinal polishing that was achieved before the samples were submerged in the calcium phosphate solution for the coating procedure. A ridge under the coating is visible which may have caused some slight, improper uniform adherence to the side of the samples. This adherence of the coating at the edges of the sample may perhaps also be due to insufficient polishing and etching.

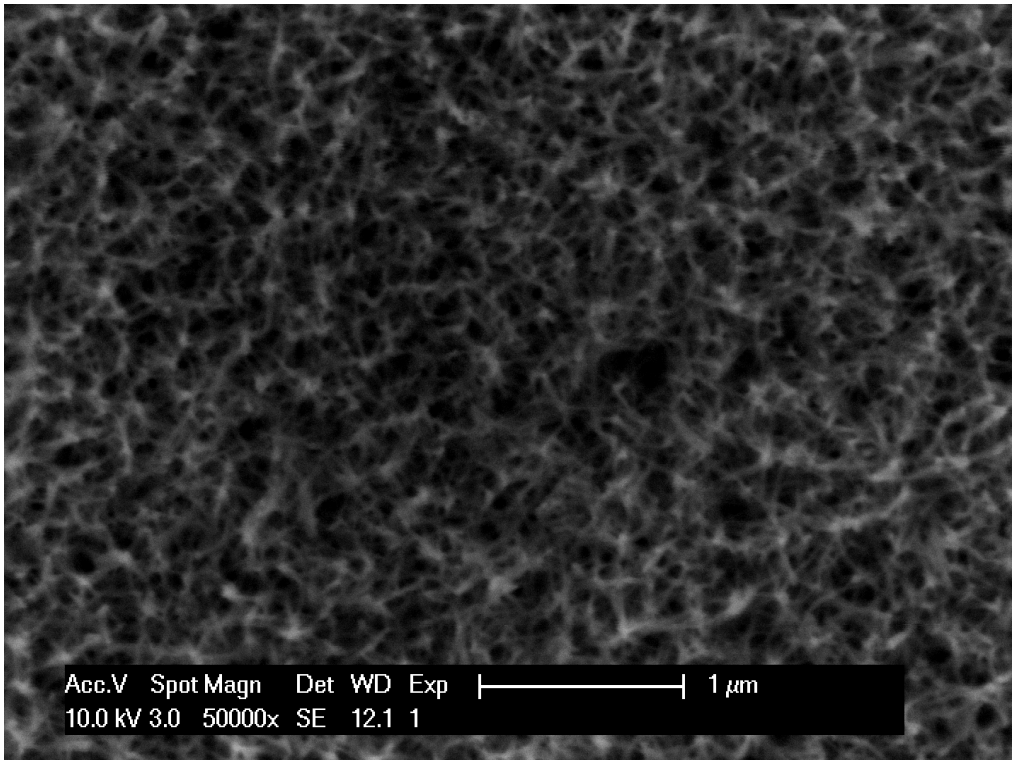

Figure 3.6(a): Coating can be seen uniformly distributed on substrate

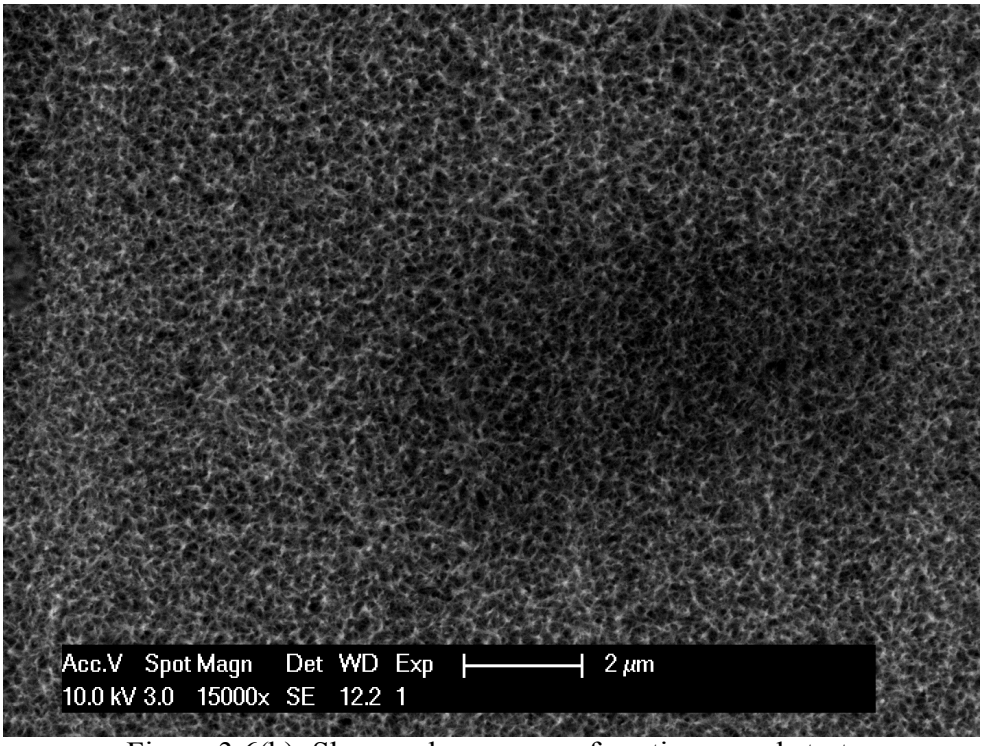

Figure 3.6(b): Shows a larger area of coating on substrate

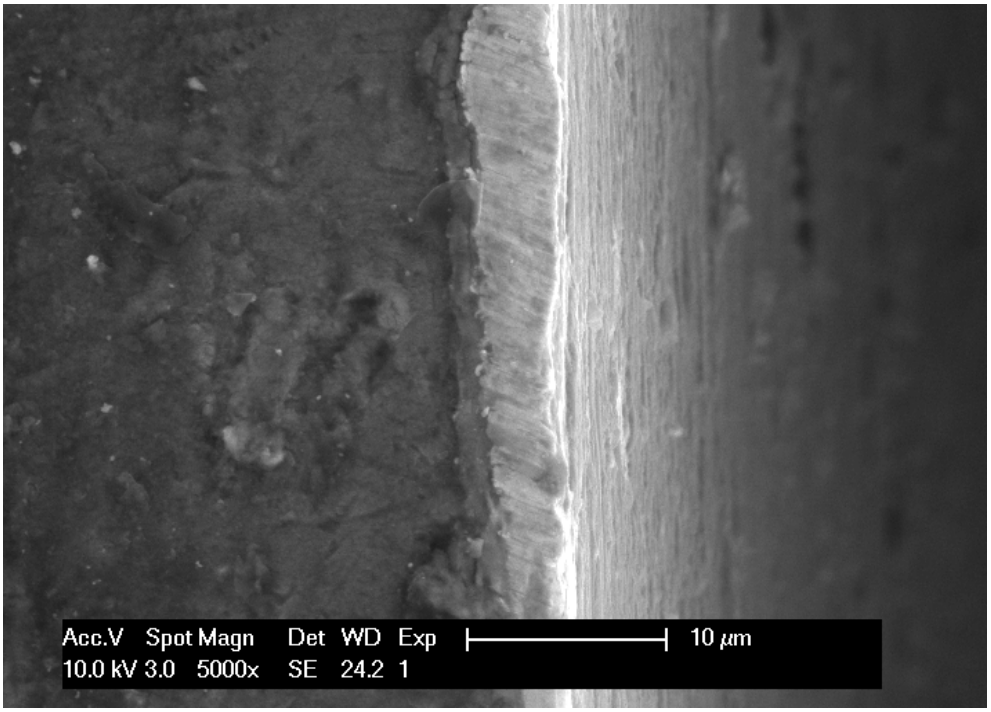

Figure 3.7: Edge of coated-sample sample.

3.4 Coating Thickness Determination

Hard mounts were prepared with the coated samples in order to determine the thickness of the coating. These mounts used phenolic powder, which mainly consisted of carbon material. They were prepared using a mounting press at a temperature of 150C and a pressure of 4200 psi. Once the coated samples were mounted, they were immediately followed by polishing and etching. The polishing began with a 400 grit sized paper and went as fine as 1200 grit in order to obtain a smooth and uniform surface to further help in the imaging process later used. Scanning electron microscopy was used for imaging as well as EDS to determine coating on the surface. Figure 3.8 shows the side of a coated sample in a mount. The material seen on the left is the substrate and on the right is the mount. A BSE (back scatter electron) image of the same area is shown in Figure 3.9.

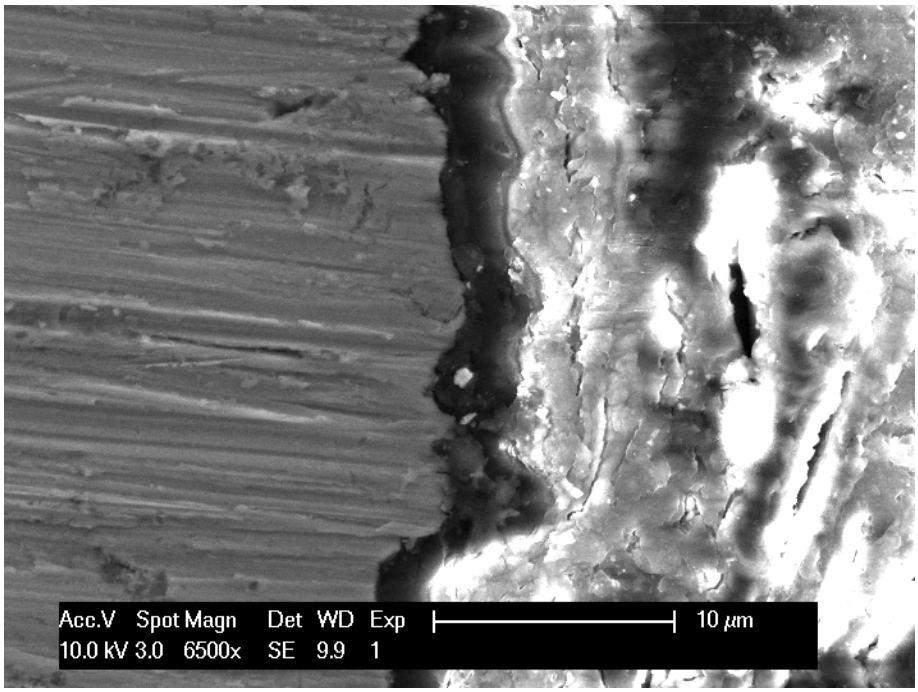

Figure 3.8: Sample in mount. Left side is substrate.

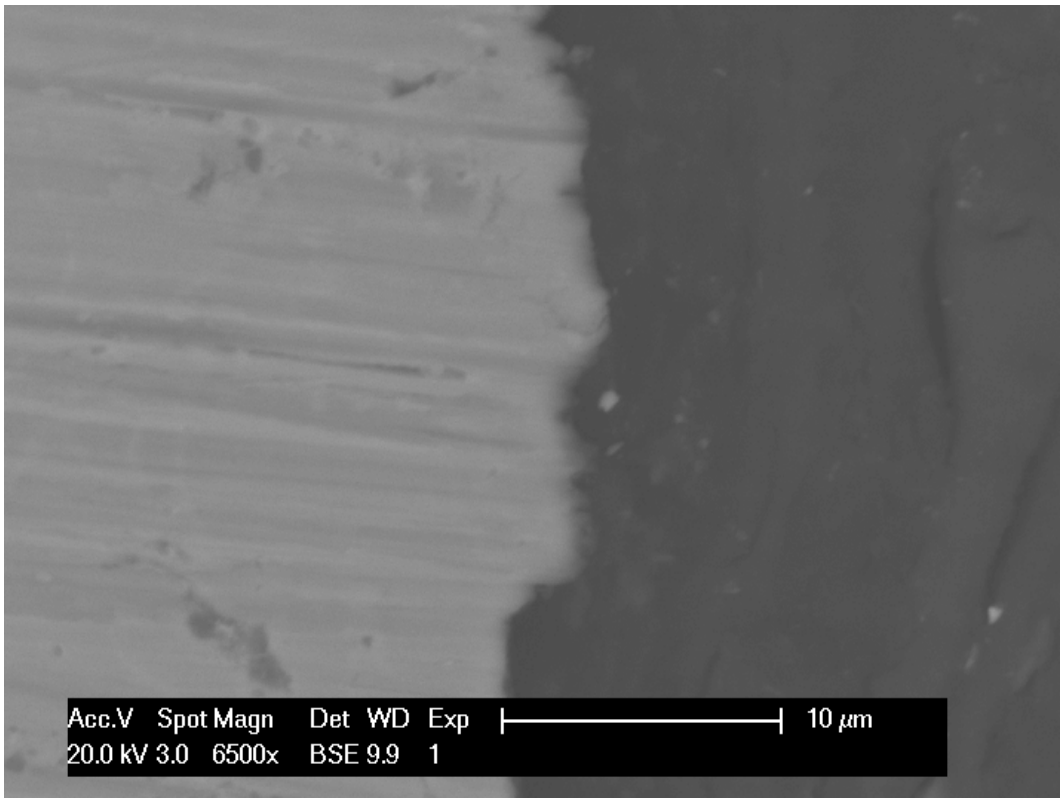

Figure 3.9: BSE image of Figure 3.8

In the BSE image, we can see a thin layer running vertically across the image between the substrate and mount material. This thin layer is suspected to be the coating. To confirm, EDS would be used on this thin layer. Figure 3.10 is the first area that was selected to perform EDS on. On this figure, two points were selected, one at the suspected coated layer and one away from the layer. The peaks that are identified as seen in Figure 3.11(a, b), demonstrate that the point selected on the far left is only the substrate and the point selected at the thin layer is indeed part of the coating. We see a relatively small peak for the calcium when compared to the elements found at the substrate and no phosphorus peak could be detected. Another area was chosen to perform

EDS on. For this selected area (Figure 3.12), three different points were selected along the coating. This confirms that the thin layer is indeed the coating. However, still only a calcium peak could be identified. Since the calcium peak is already relatively small, and the calcium to phosphorus ratio is one to two, it is probable that one of the much smaller peaks that were not identified is phosphorus. From this layer, the coating was thus measured and found to be about 2 µm in thickness.

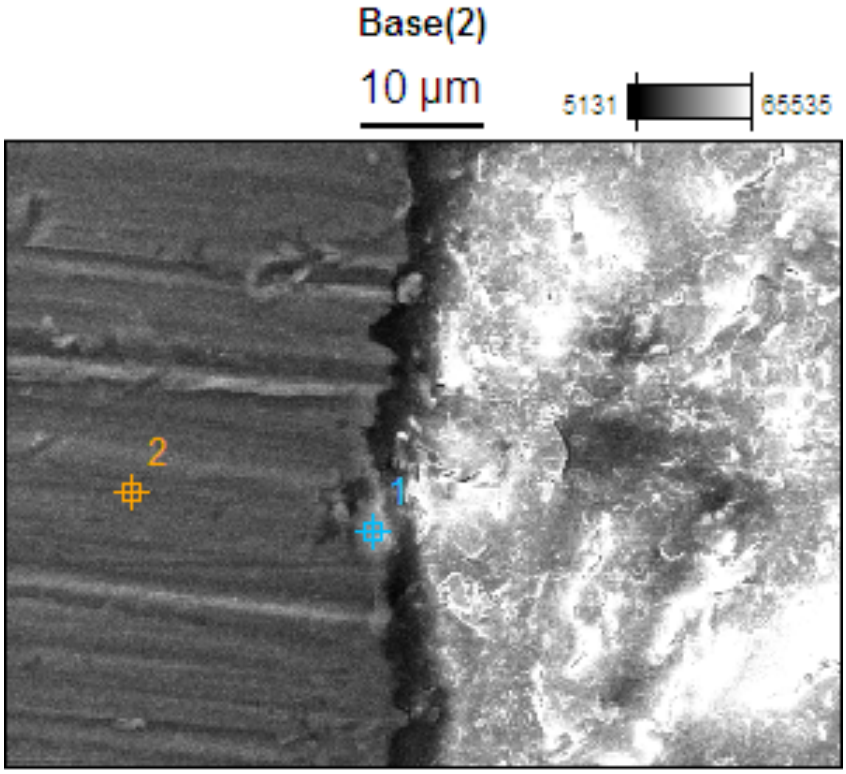

Figure 3.10: EDS image from mounted sample. Point 1 is selected at the coating, point 2 on the substrate.

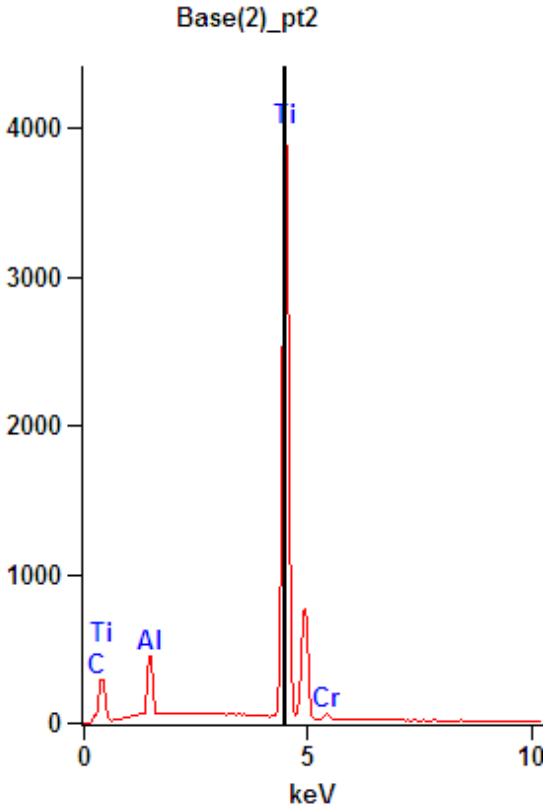

Figure 3.11(a): Spectrum for point 2

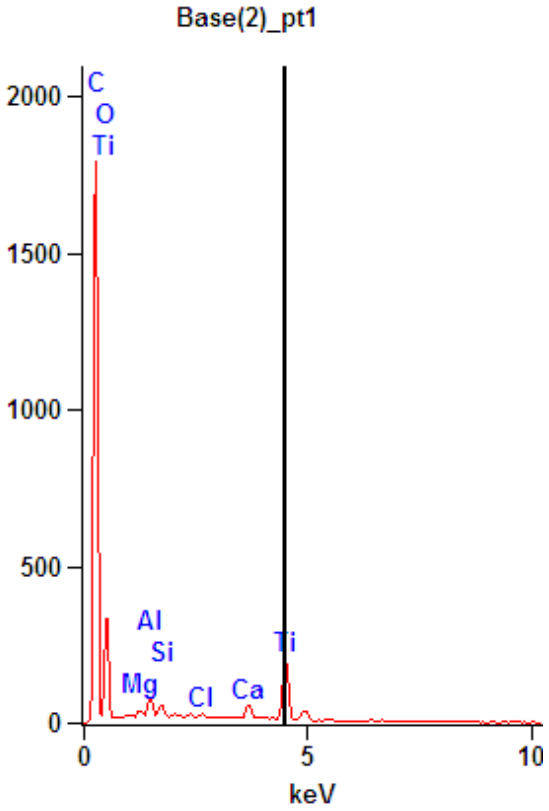

Figure 3.11(b): Spectrum for point 1

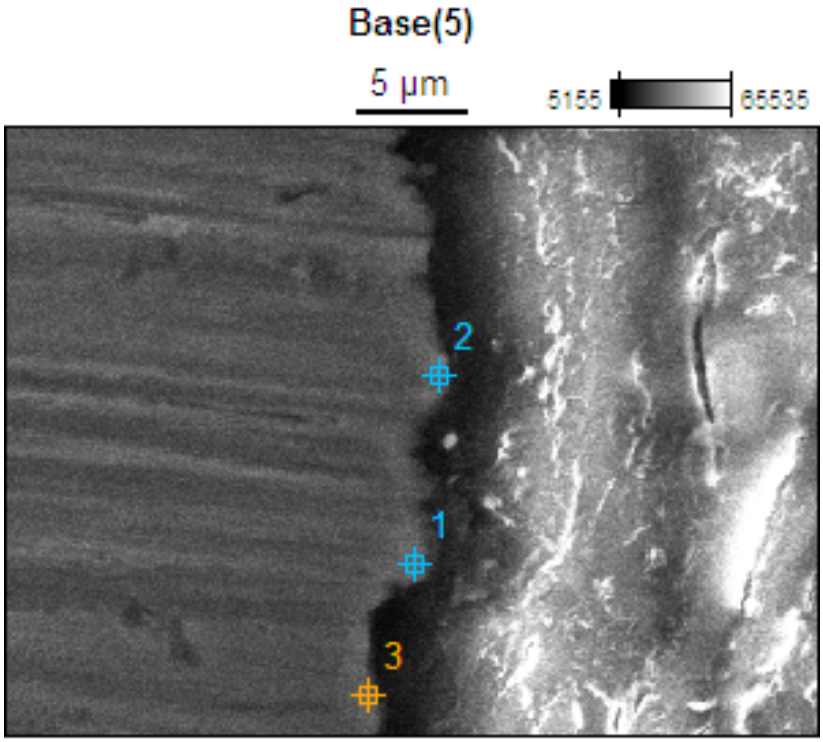

Figure 3.12: Three different points selected along the coating in EDS.

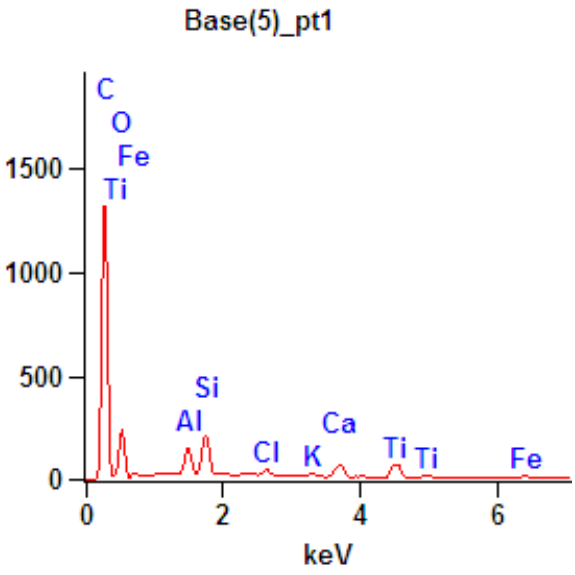

Figure 3.13(a): Spectrum for point 1

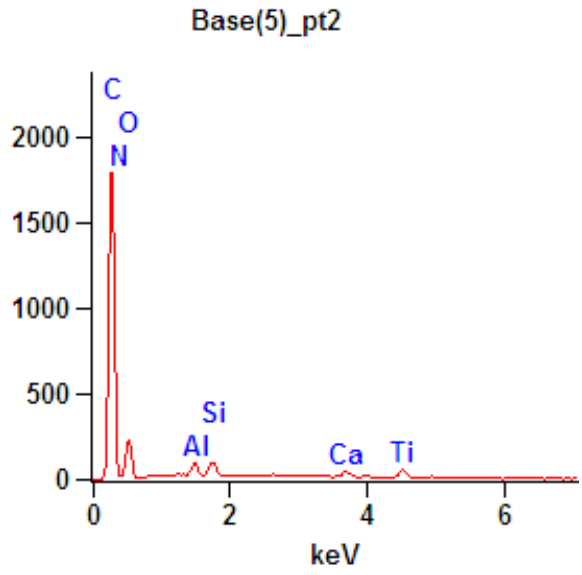

Figure 3.13(b): Spectrum for point 2

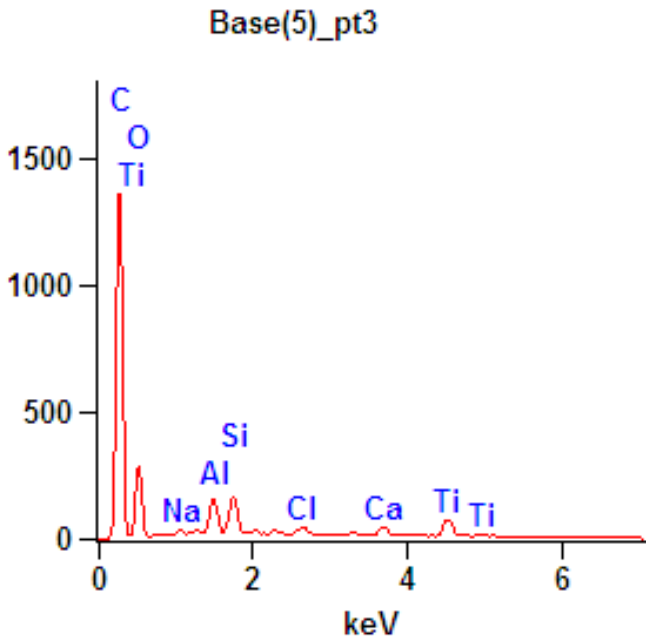

Figure 3.13(c): Spectrum for point 3

Chapter 4

Fatigue Testing of Samples

4.1 Bare sample testing

Non-coated samples of Ti-6-4 alloy would be tested first to later compare their results to the coated samples. The average max load achieved in the cyclic motion was on average 12 N with average load amplitude of 10 N. After the samples broke under bending, they would be analyzed under SEM to further help investigate the fatigue properties of the material.

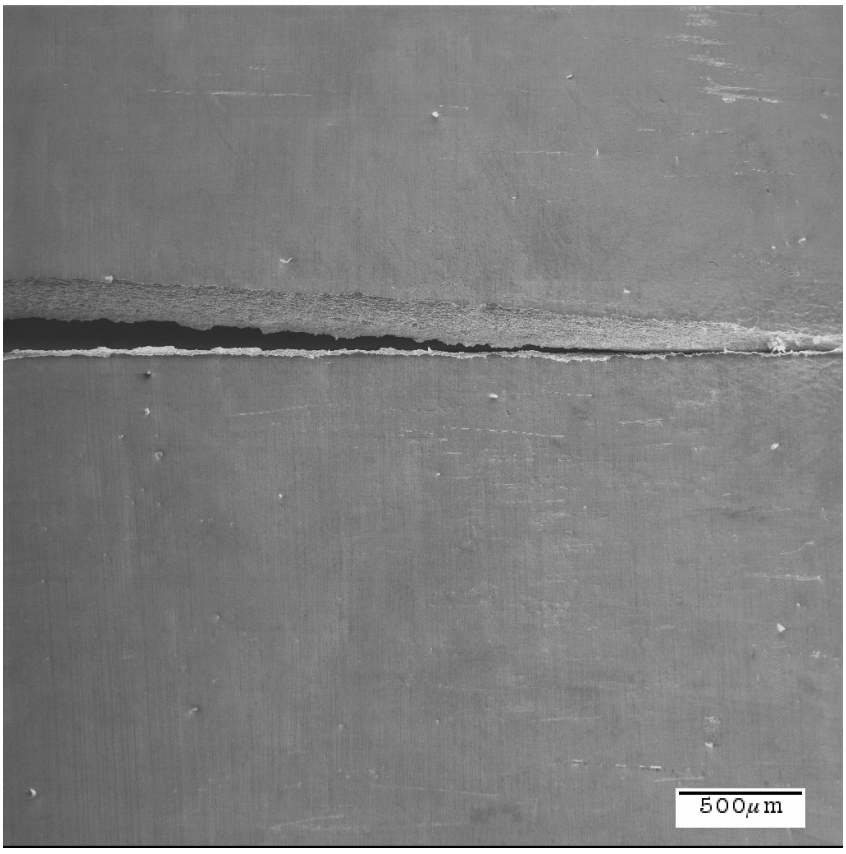

Figure 4.1: Bare sample failed under fatigue

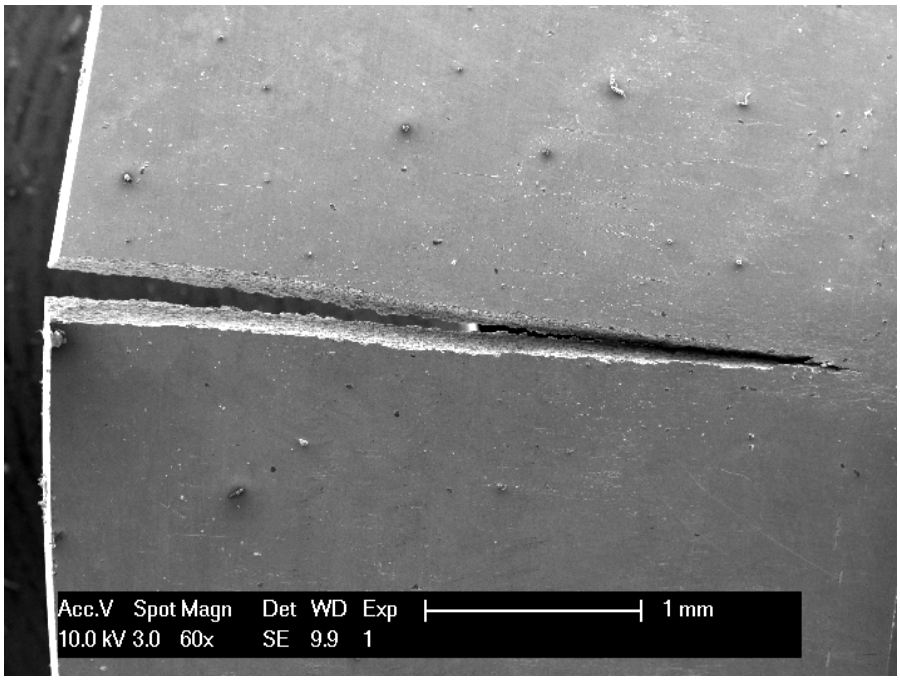

Figure 4.2: Bare sample broken under fatigue

The above two images show a bare Ti-6-4 sample after fatigue testing. These images show the tensile side of the sample. From these images we can deduce that the crack initiated from one side of the sample and propagated across the material. We can also notice the ridges on the edges of where the sample failed, further indicating that this is indeed the tensile side of the sample.

Further investigation indicates a possible stage one cracking beginning at the edge of the tensile side of the material as shown on the fracture face of Figure 4.3 and a slightly angled view in Figure 4.4. The features shown on the fracture face are most likely due to the crack changing orientations between or at the grain boundaries, which are on the

order of roughly 10 μm. These features can be seen moving in a wave like pattern in the direction of the crack. Higher magnification of this feature is shown in Figure 4.5.

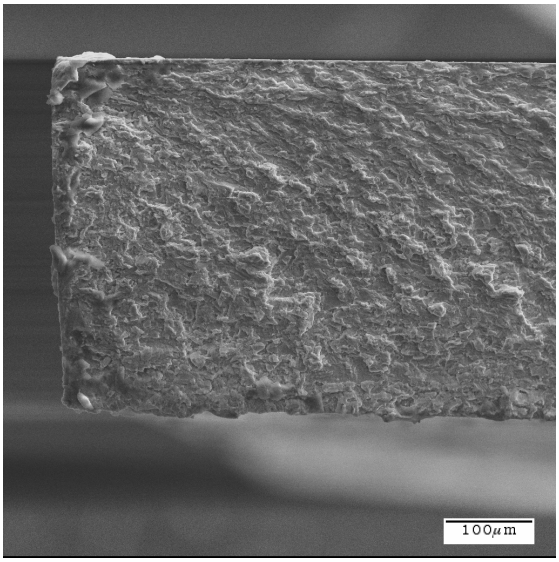

Figure 4.3: Fracture face of bare sample

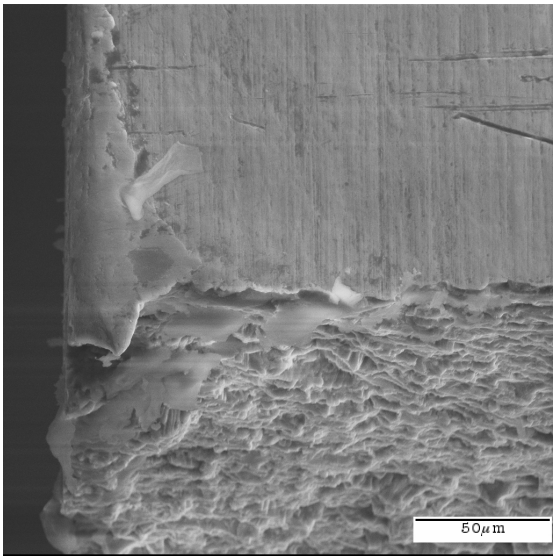

Figure 4.4: Alternate view of Figure 4.3

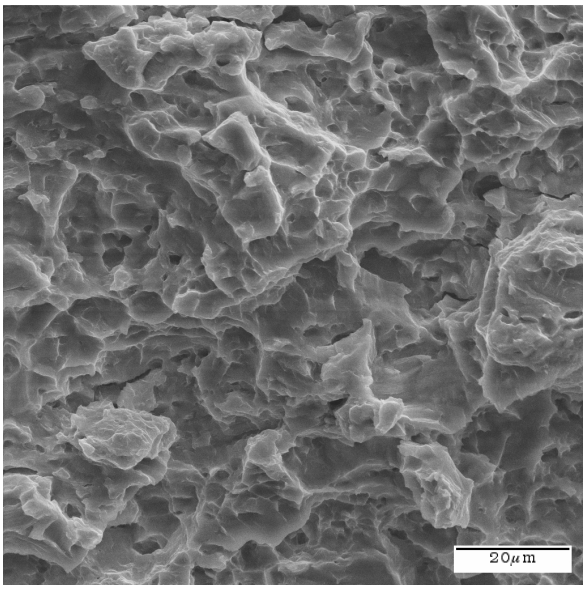

Figure 4.5: Higher magnification of fracture face

4.2 Coated Sample Testing

Testing of the coated samples would follow after the bare samples. Similar max load and load amplitude conditions were used for the calcium phosphate coated samples. Crack propagation is shown in Figure 4.6.

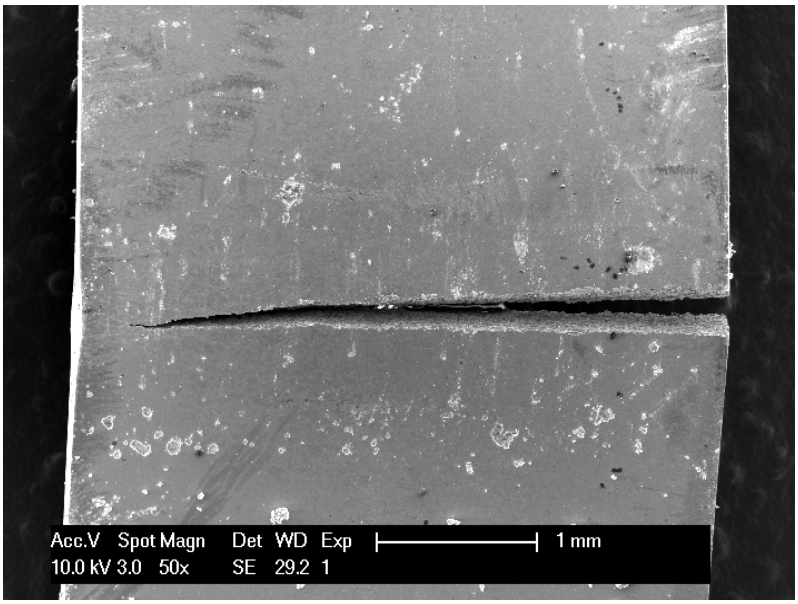

Figure 4.6: Coated sample broken under fatigue

Again, the calcium phosphate precipitates can be seen scattered at the surface of the sample. At higher magnifications the crack tip can be spotted near the opposite edge of the crack initiation.

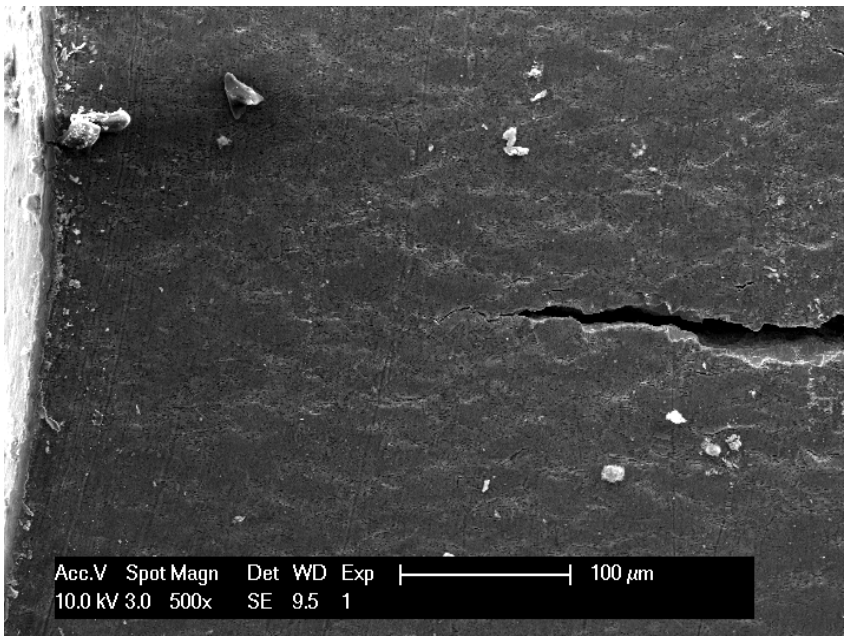

Figure 4.7: Cracking seen along coated sample

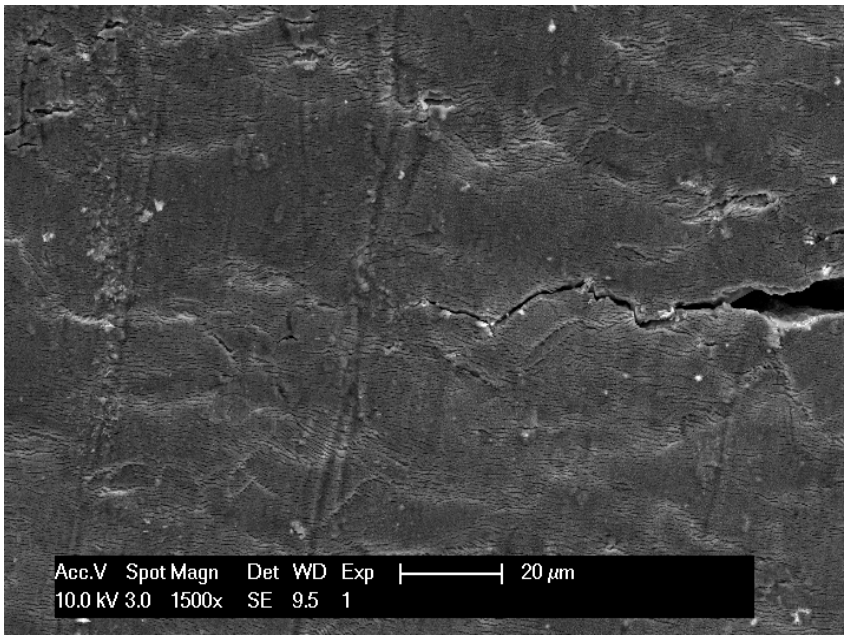

Figure 4.8: At higher magnification, crack tip is seen on sample

Imaging of the crack initiation site was also investigated, similar to that of the non-coated sample. Comparatively then, stage one cracking likely exists at the same sites (tensile side and at the crack initiation site) as seen at a slightly angled view of the samples side in Figure 4.9 and Figure 4.10.

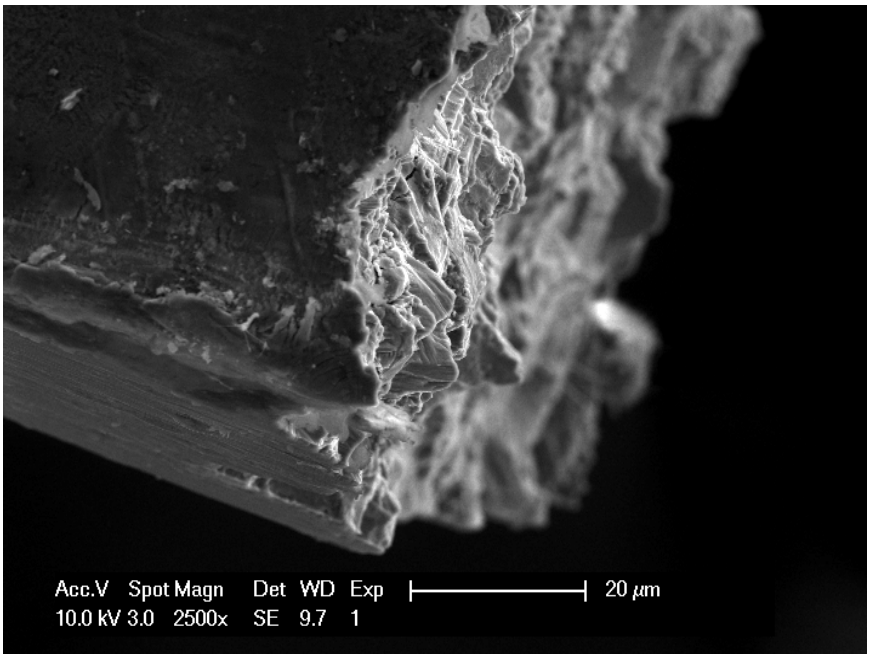

Figure 4.9: Stage one cracking seen at edge

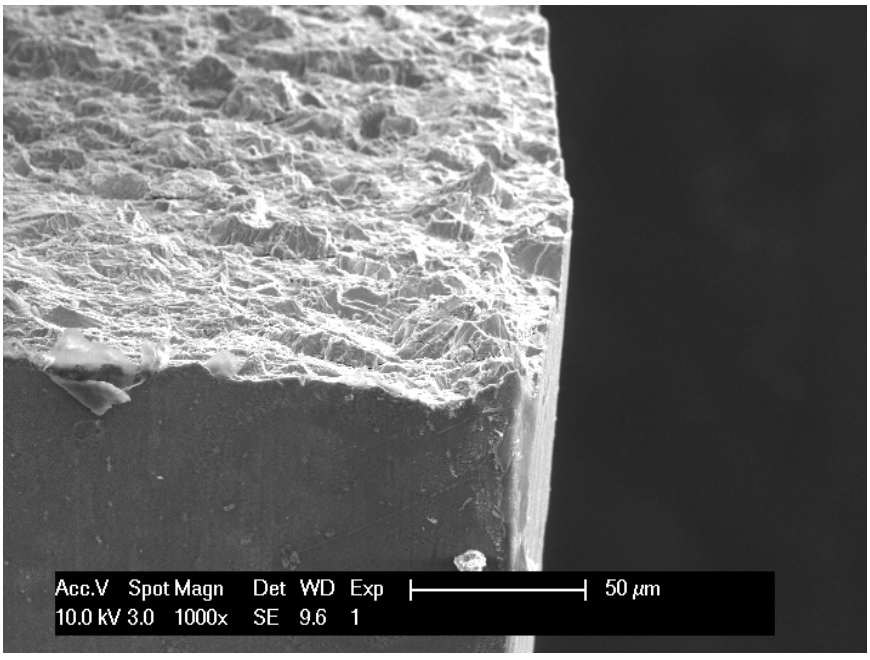

Figure 4.10: Stage one cracking at lower edge of sample

Another notable feature on these samples is that even after the samples fractured, most of the coating was still clearly apparent at the surface. Compared to the SEM images taken before the fatigue testing, majority of the coating still shows a similar profile. This being said, there is coating separation due to the cyclic loading that can be identified, as shown in the images below.

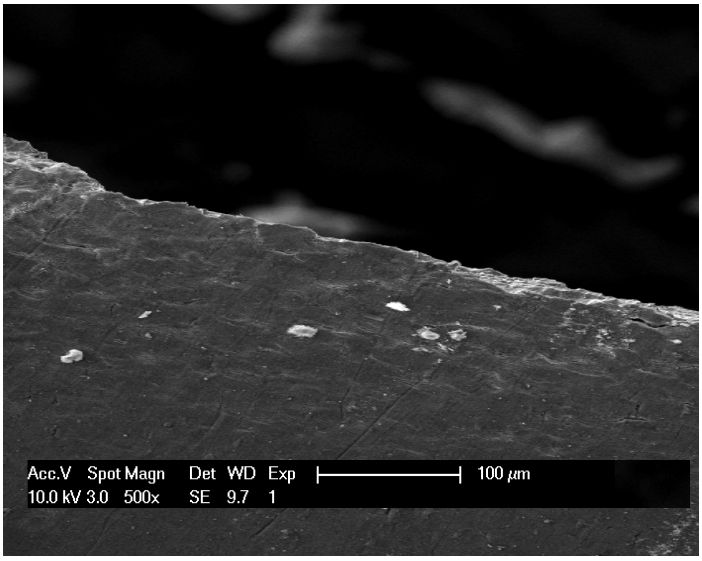

Figure 4.11(a): Coating separation on tensile side

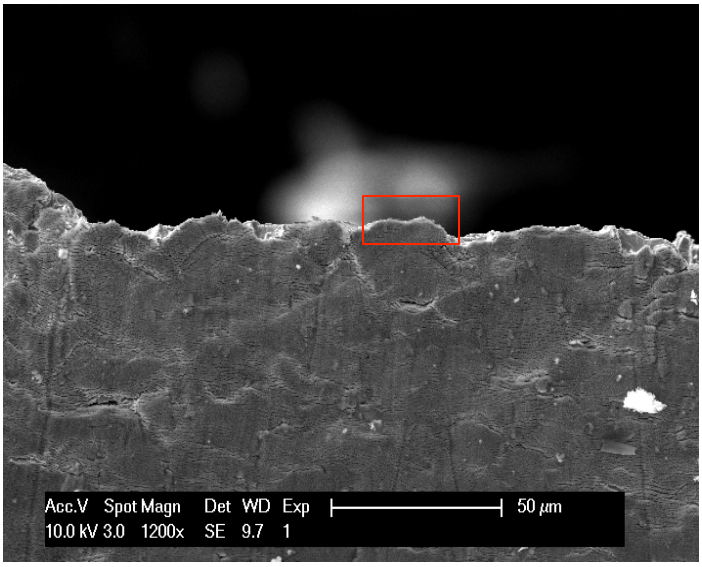

Figure 4.11(b): Higher magnification of figure 4.11(a)

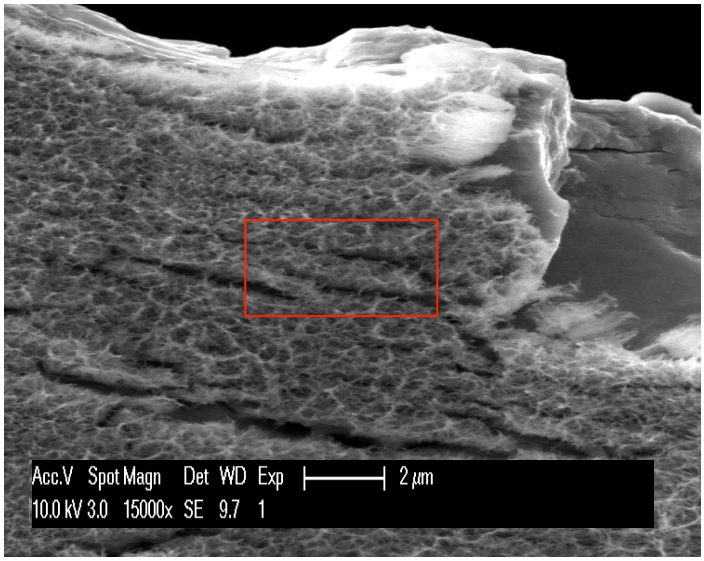

Figure 4.11(c): Higher magnification of area highlighted on Figure 4.11(b)

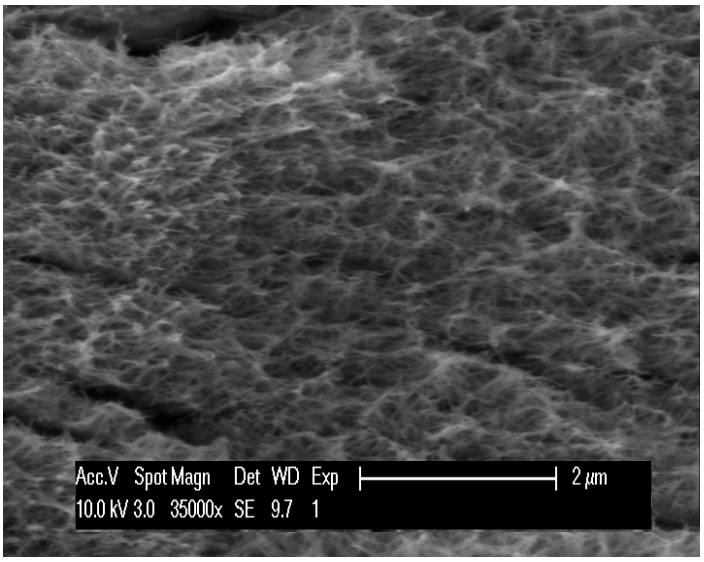

Figure 4.11(d): Higher magnification of area highlighted on Figure 4.11(c)

4.3 Results & Discussion

Although the fracture analysis of the two groups looked similar, their fatigue behavior was not. Figure 5.1 compares the life cycles of the coated and the non-coated samples. The graphs data is analyzed in cycles to failure versus maximum stress range. We see from the data generated that the fatigue life of the coated samples is significantly higher at the 900 – 1000 MPa range. This may be due to the calcium phosphate coating on the samples impeding or slowing down both the initiation of cracking as well as the crack growth propagation across the material.

The data shown on the graph represents the samples tested which lead to failure. Two samples tested at a significantly lower stress range, both for coated and non-coated, were found to have experienced run-off (indicated by arrows), where their cycles to failure exceeded that of ten million cycles. The solid line curve on the graph represents the typical Ti-6-4 alloy fatigue behavior.

We can conclude that the fatigue behavior of the coated samples was better than that of the bare samples, particularly in the 900 - 1000 MPa range (as shown in Figure 4.12). The difference between the coated samples themselves however (in number of life cycles to failure) is most likely due to slight variation in sample thickness uniformity throughout. During the polish and etching process the samples were measured after each new grit sized paper was used. Multiple measurements were made on each sample and the average of the readings was taken as the final thickness of the sample. Thus, although the average thickness was maintained throughout the samples, the point of the cyclic loading may have cycled on a point of the sample that was slightly thinner or thicker

compared to the average, resulting in varied fatigue results. Although the variation itself is minimal, it is however significant in this study since the samples themselves have such a thin width. The data available for the Ti-6-4 alloy material (solid line curve) shows a slightly similar profile as the non-coated samples, at least for the lower life cycle range.

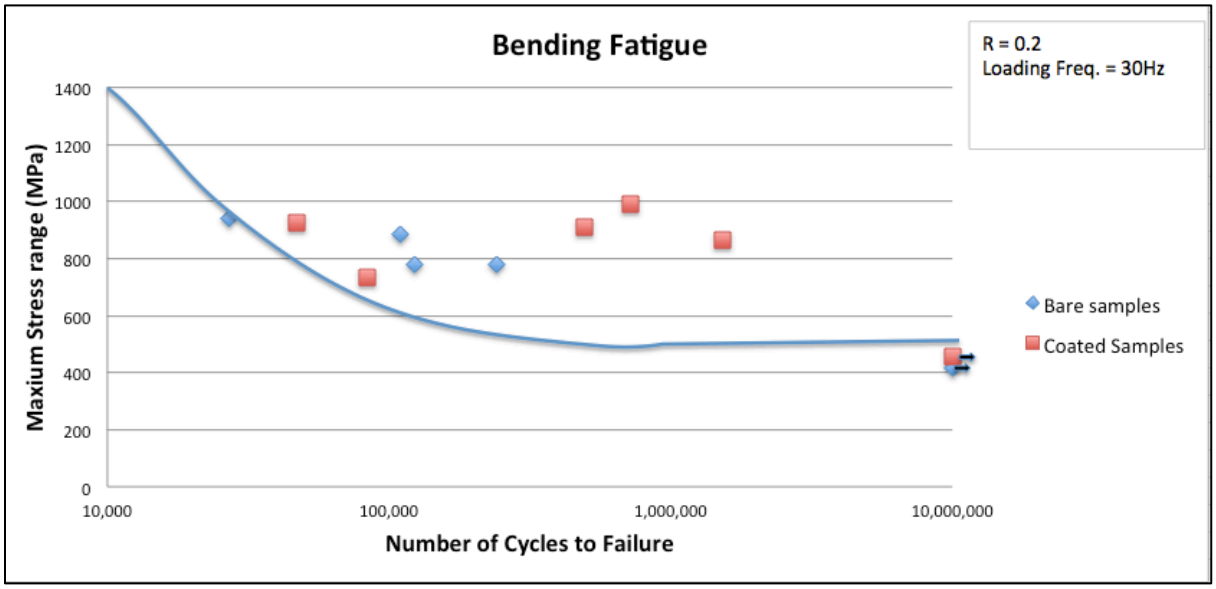

Figure 4.12: Representing the data generated from fatigue testing of both coated and bare samples. The generated fatigue data is compared to Ti-6-4 alloy data (solid curve). The arrows on data points represent samples that had reached their fatigue limit.

Overall, the data suggests at or better fatigue life for the coated samples compared to the bare samples throughout the different stress ranges. This is consistent with various other types of coatings that have been tested for fatigue life. Fatigue testing performed on a stainless steel material with TiN based coating was shown to increase the fatigue life of the steel between 400 – 2119% when compared to the bare metal itself [6]. Hydroxylapatite (HA) is also an important biocompatible coating used on various titanium alloys. It too has been tested for its fatigue properties and has been shown that it

does not negatively impact the fatigue life of the substrate. Rather, it maintains the materials (Ti-6-4) fatigue life when the coating is 50 μm or thinner [7].

Chapter 5

Conclusions

The present study finds the fatigue behavior of the calcium phosphate coated Ti-6-4 alloy samples to have a higher fatigue life when compared to its bare (non-coated) counterpart. Testing of all samples was maintained at a constant loading frequency of 30Hz and an r ratio maintained of 0.2. The coating method used for this study was effective and a relatively fast technique for adherence of calcium phosphate since it was a mere 24-hour submersion versus other methods used for calcium phosphate adherence, which can take several days to weeks [4]. Surface pre-treatments and preparations were then a possible result of the fast adherence of the calcium phosphate to the Ti-6-4 substrate.

From the three point bend testing we found a similar fracture surface in both the coated and bare samples. A possible stage-one cracking was observed in both groups at the crack initiation site from which the crack would propagate across the material and thus lead to the sample failing under fatigue. The fatigue life however of the coated samples varied across the stress range, which may be due to a slight coating thickness variation across the samples as well as the sample thickness itself that would cause this

behavior. Delay in the initial cracks in the coated samples may be the reason they experienced a better fatigue behavior when compared to the bare samples.

Chapter 6

Future Work

One of the main concerns in the present study was to try and get an accurate measurement of the coatings thickness on the samples. Although it is estimated based on the SEM and EDS analysis that the coating thickness is 2 μm, this could not be confirmed throughout the uniformity of all of the samples. Hard mounts were made with the samples, however coating thickness was difficult to measure and hard to identify while in the mounts. The longitudinal polishing of the samples prior to the coating procedure may also have led to the hard mounts covering the edge of the samples and thus making it more difficult to see the coating while in the mounts.

Environmental factors to fatigue behavior are another interesting phenomena that should be tested on this paring of coating and material. Testing of similar samples in saline solution at 37°C to help mimic an in-vitro environment would also be interesting to see how the coating behaves in such an environment and if the fatigue properties change.

References

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<http://www.sciencedirect.com/science/article/pii/S0257897203008867>

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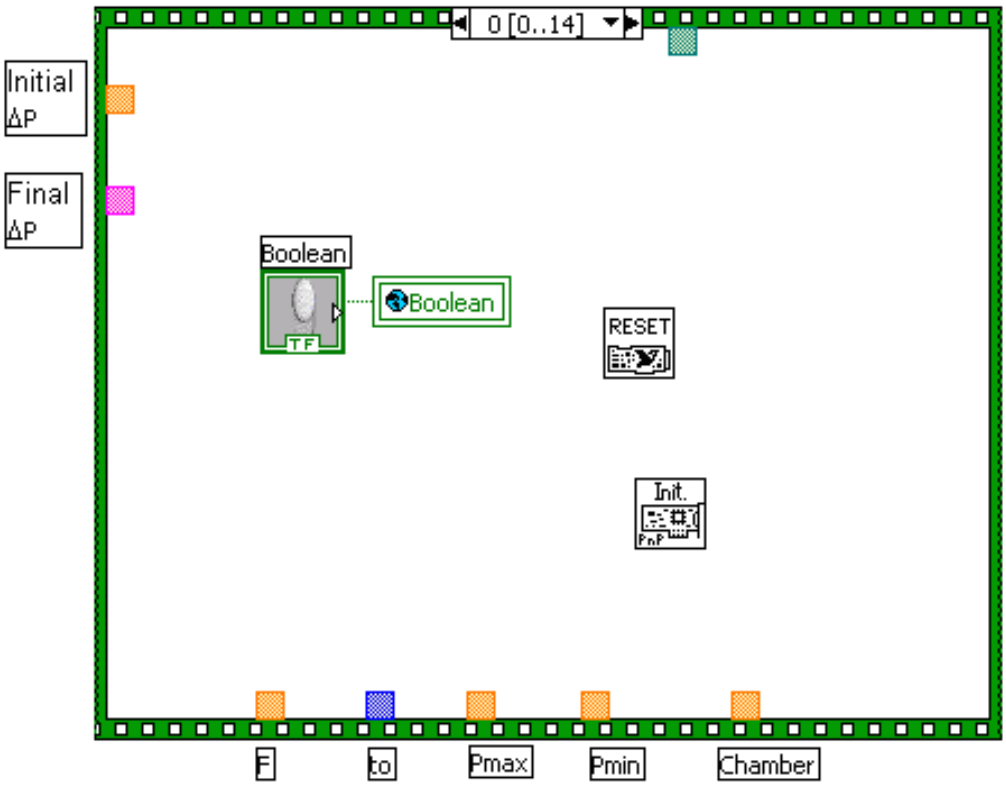
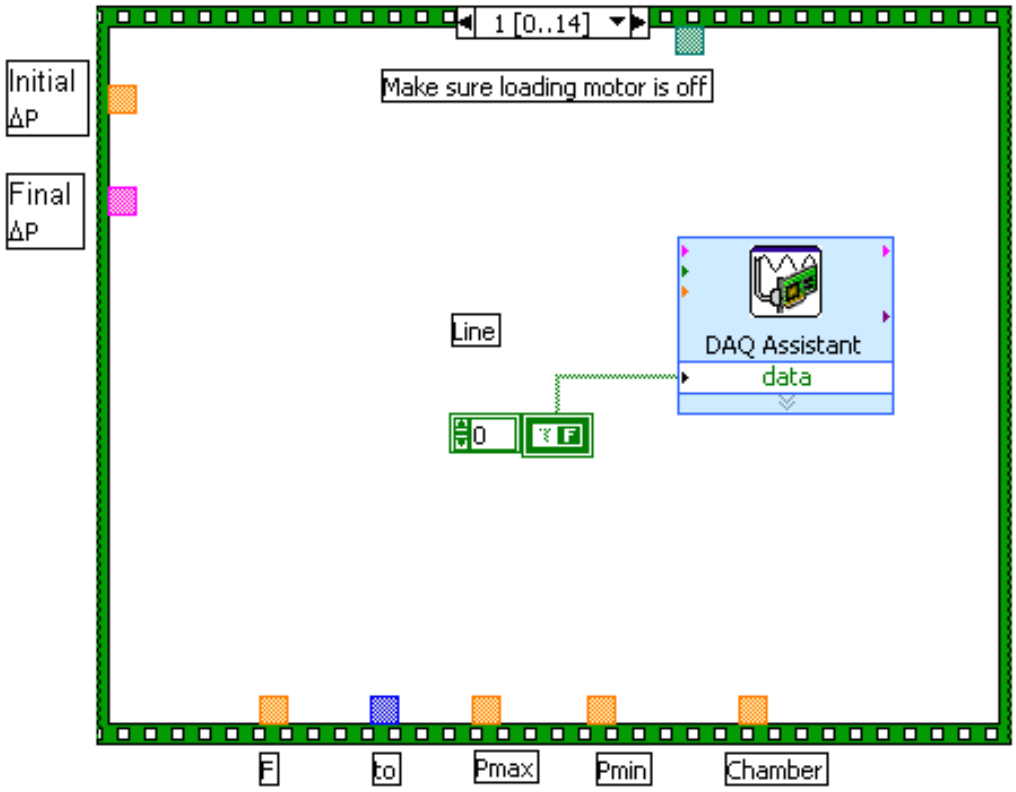
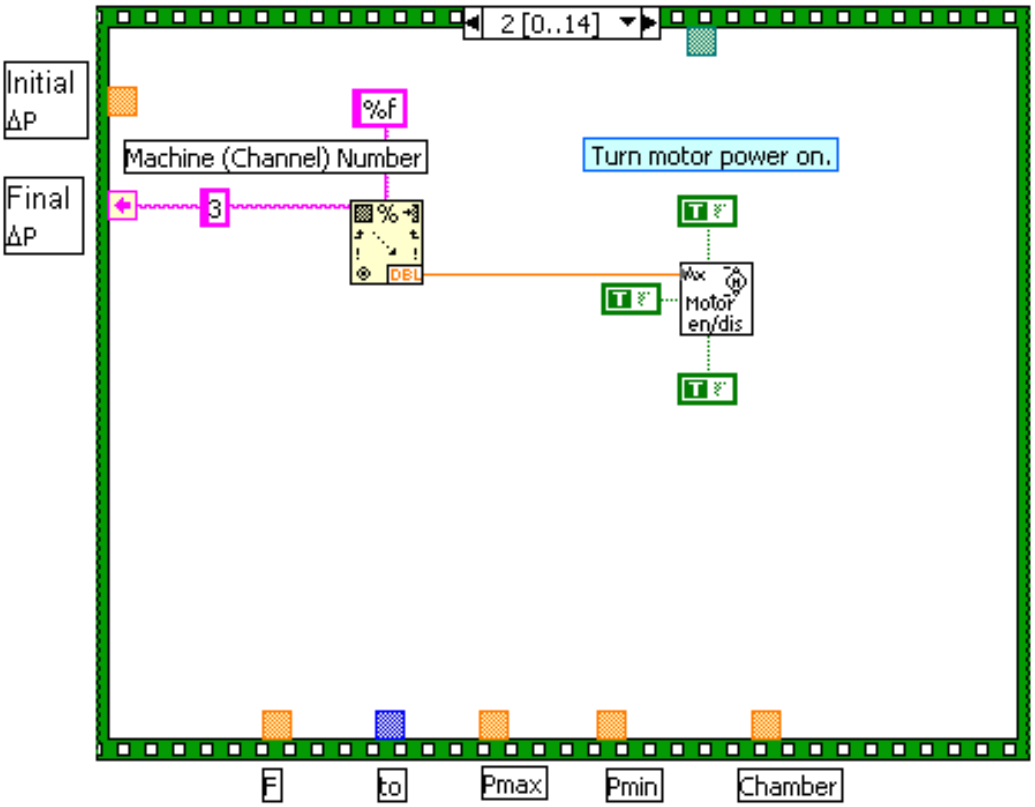
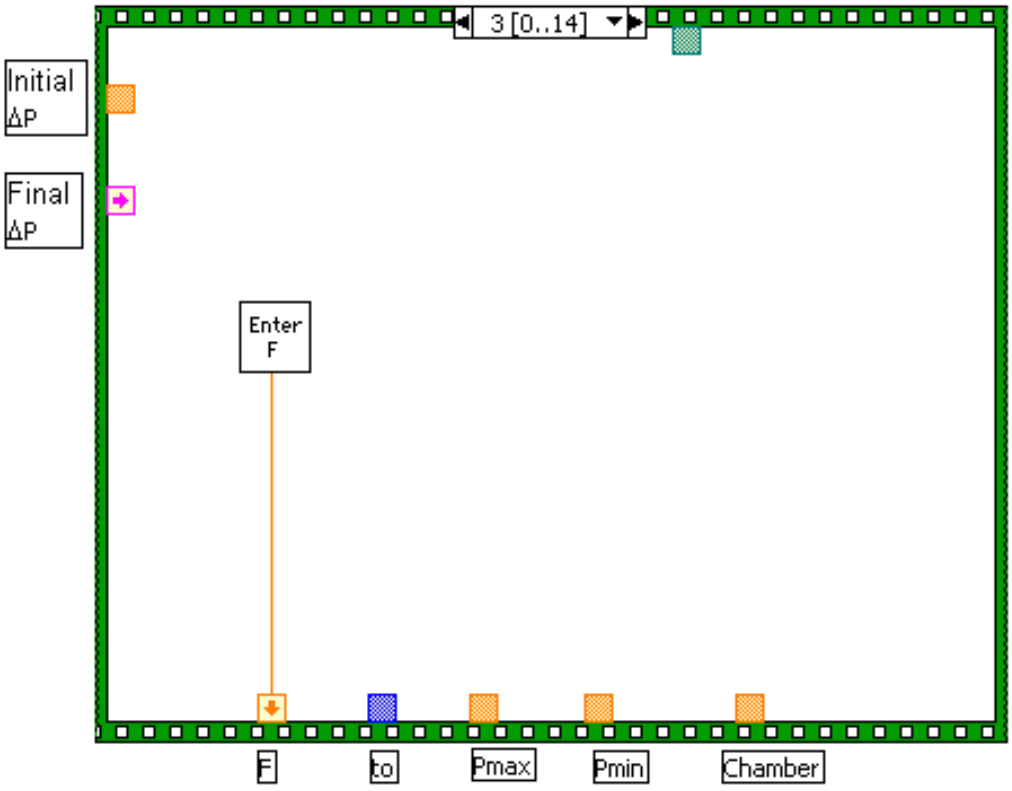
< http://onlinelibrary.wiley.com/doi/10.1002/jbm.820211203/full>

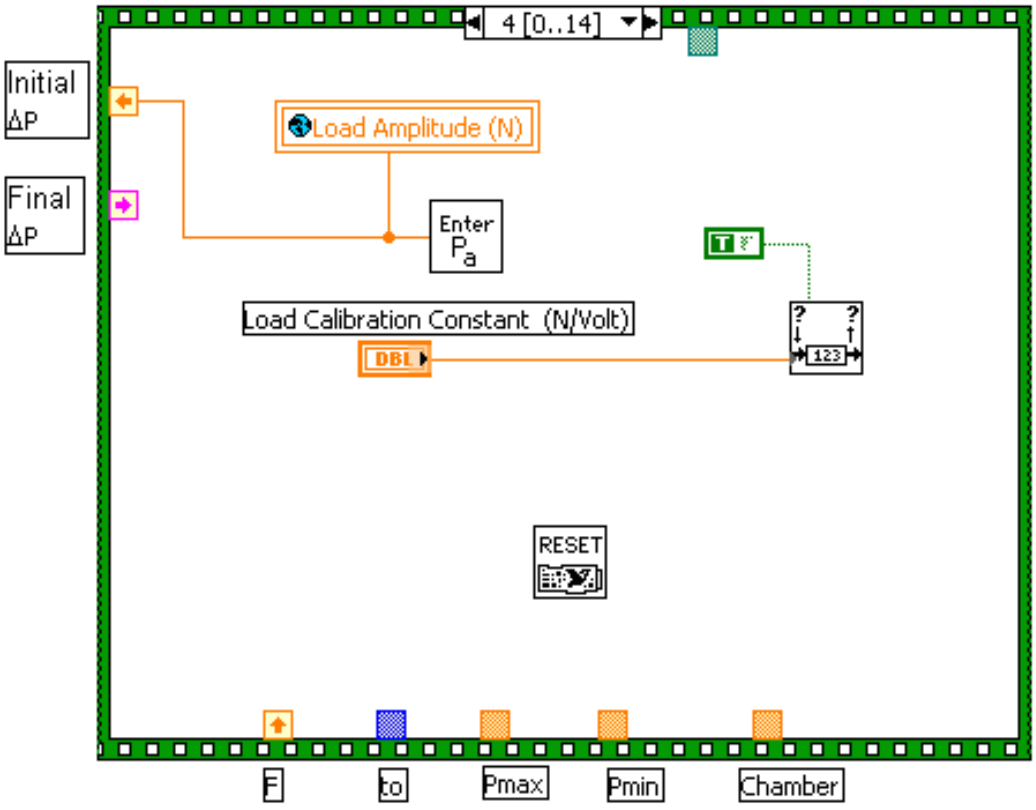
APPENDIX: Custom Labview Program

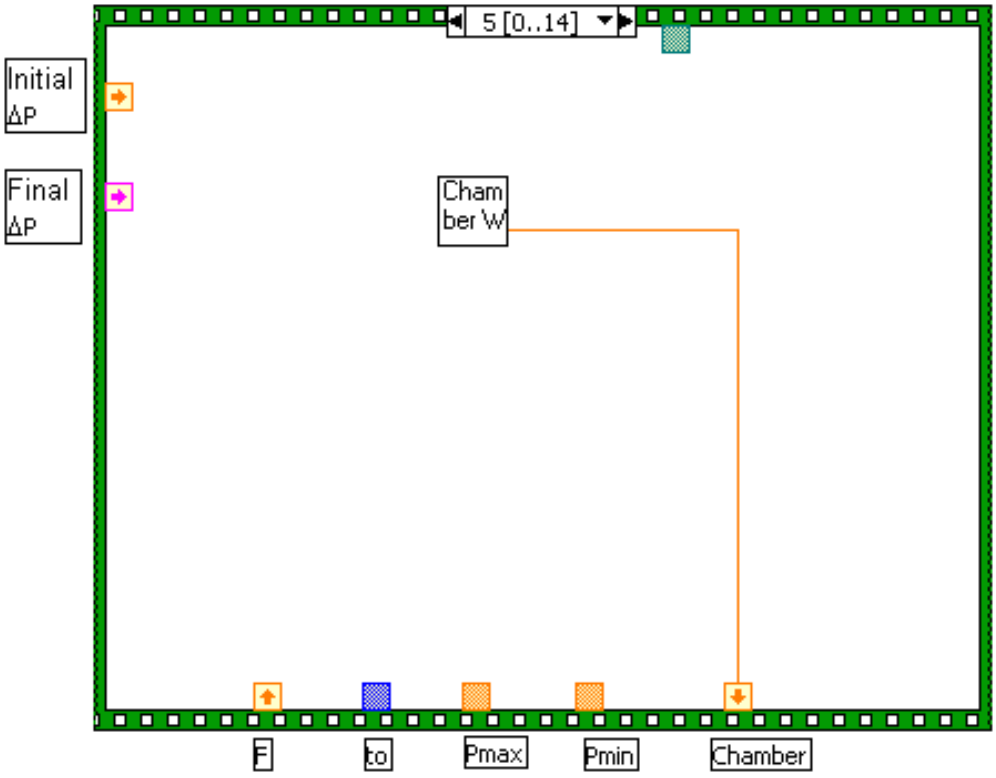


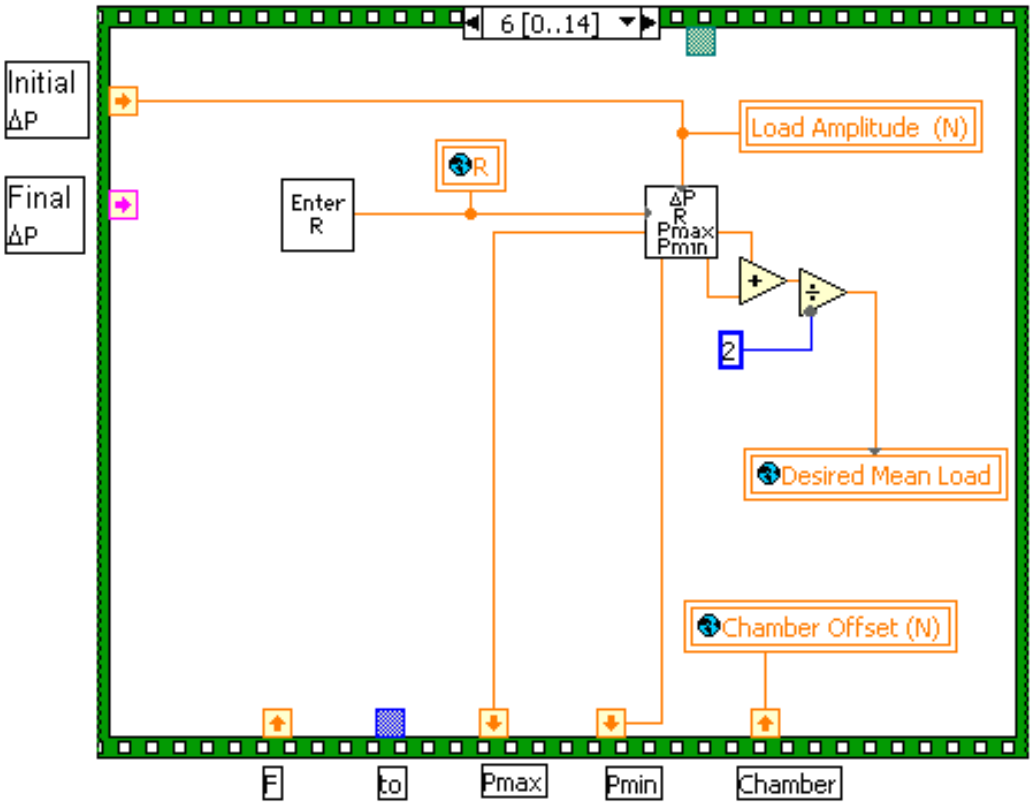

1 [0..14]
Make sure loading motor is off
Initial ΔP
Final ΔP
Line
DAQ Assistant
data
0
F
to
Pmax
Pmin
Chamber

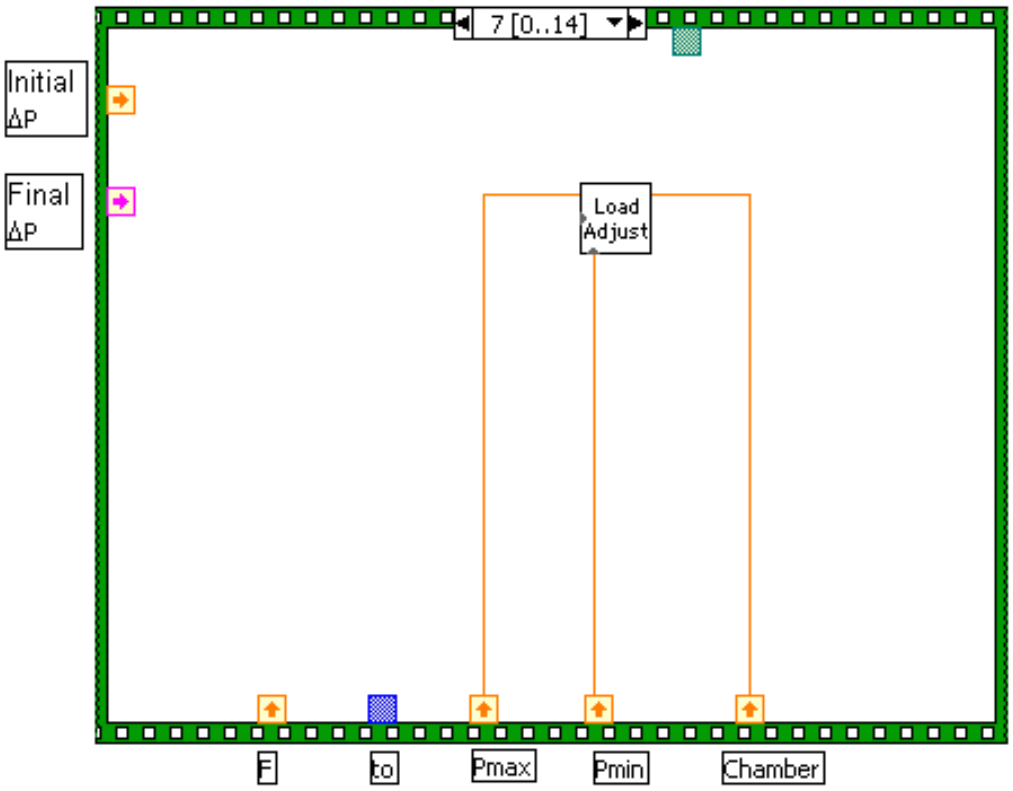

2 [0..14]
Initial ΔP
Final ΔP
%f
Machine (Channel) Number
3
DBL
Turn motor power on.
Motor en/dis
F
to
Pmax
Pmin
Chamber

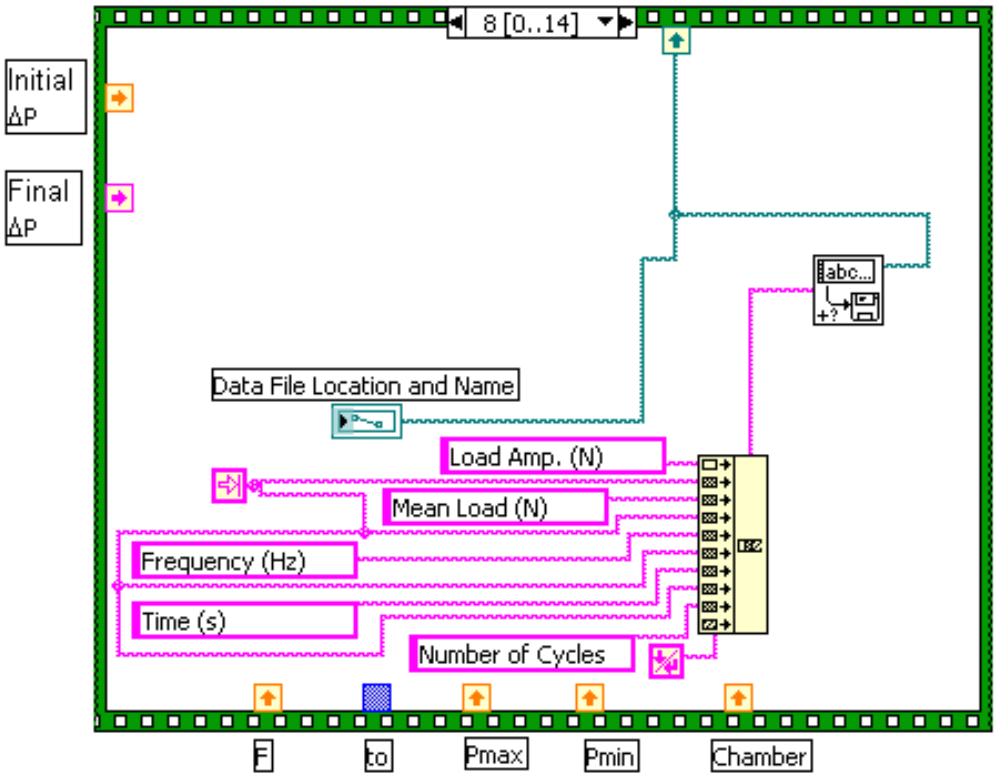

3 [0..14]
Initial ΔP
Final ΔP
Enter F
F
to
Pmax
Pmin
Chamber

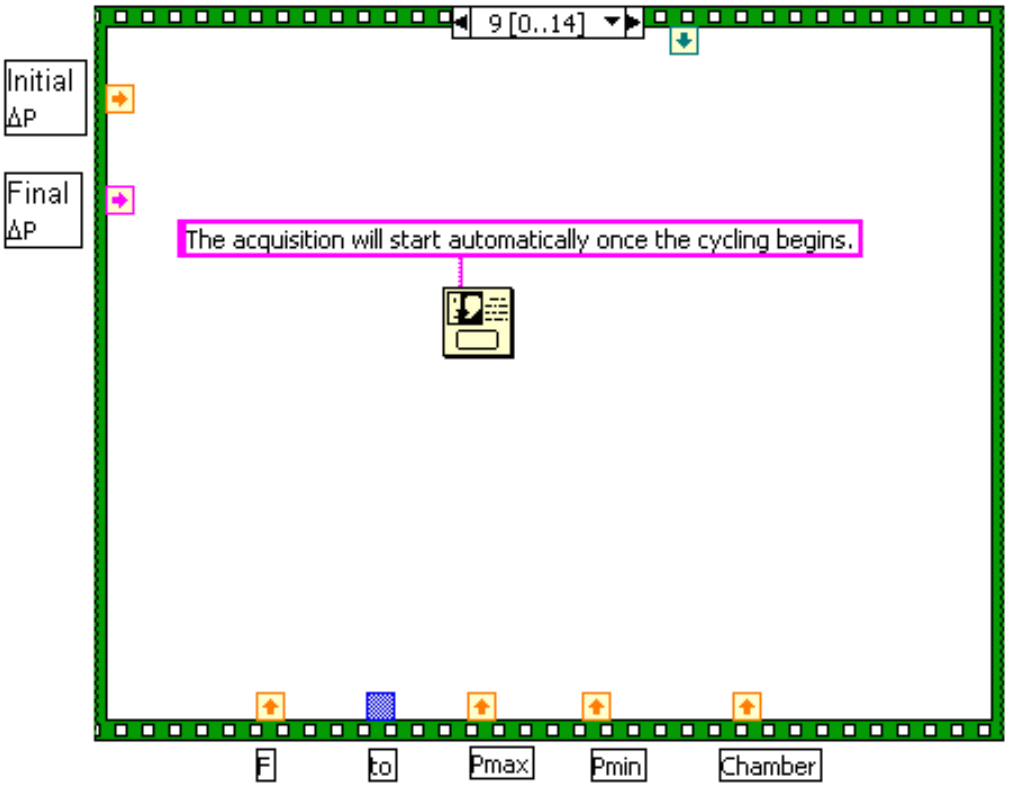

4 [0..14]
Initial ΔP
Final ΔP
Load Amplitude (N)
Enter Pa
Load Calibration Constant (N/Volt)
DBL
RESET
F
to
Pmax
Pmin
Chamber

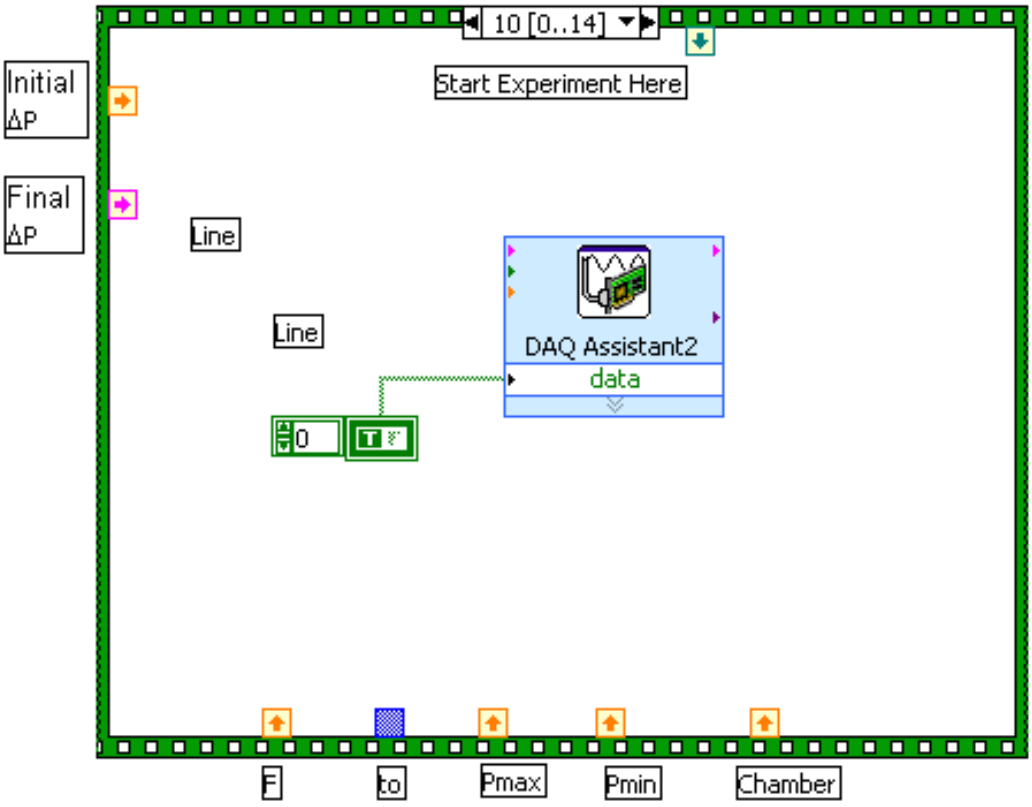

5 [0..14]
Initial ΔP
Final ΔP
Cham ber W
F
to
Pmax
Pmin
Chamber

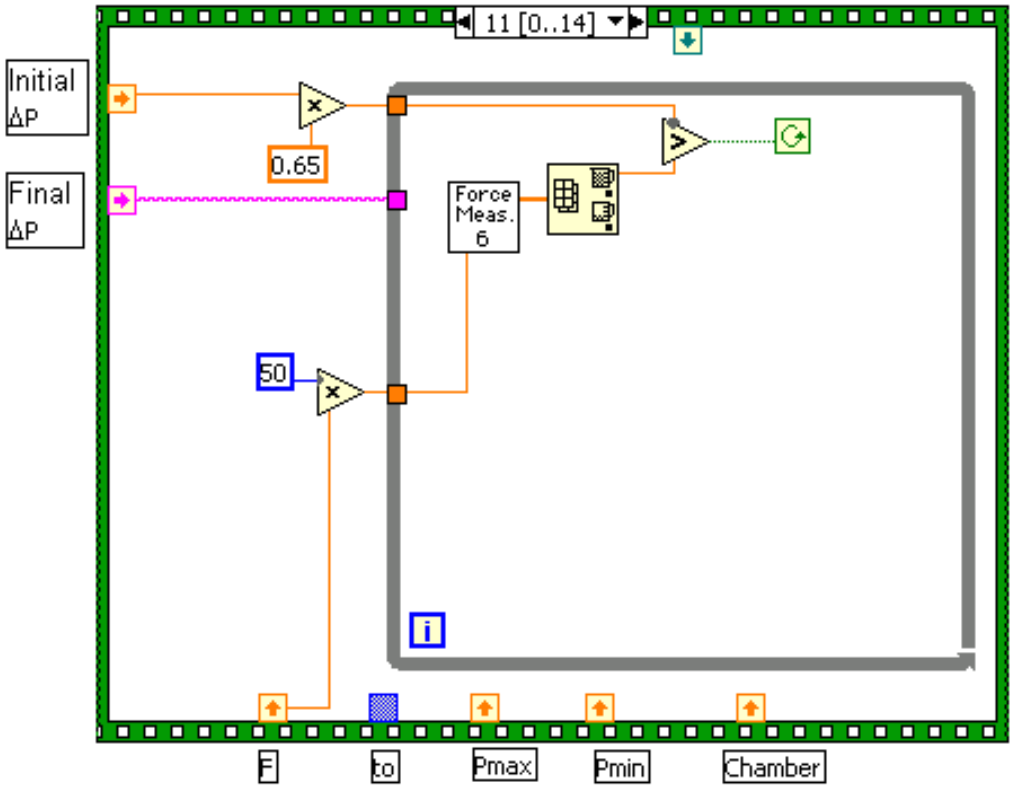

6 [0..14]
Initial ΔP
Final ΔP
Enter R
R
ΔP
R
Pmax
Pmin
2
Load Amplitude (N)
Desired Mean Load
Chamber Offset (N)
F
to
Pmax
Pmin
Chamber

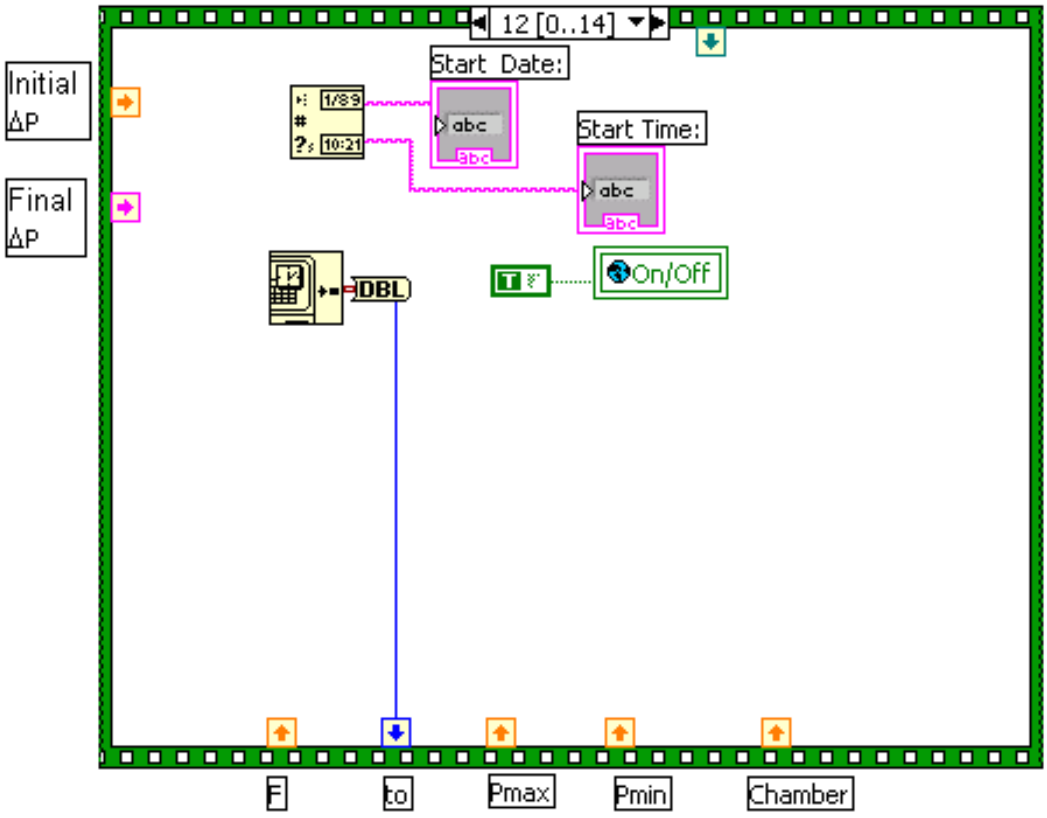

7 [0..14]
Initial ΔP
Final ΔP
Load Adjust
F
to
Pmax
Pmin
Chamber

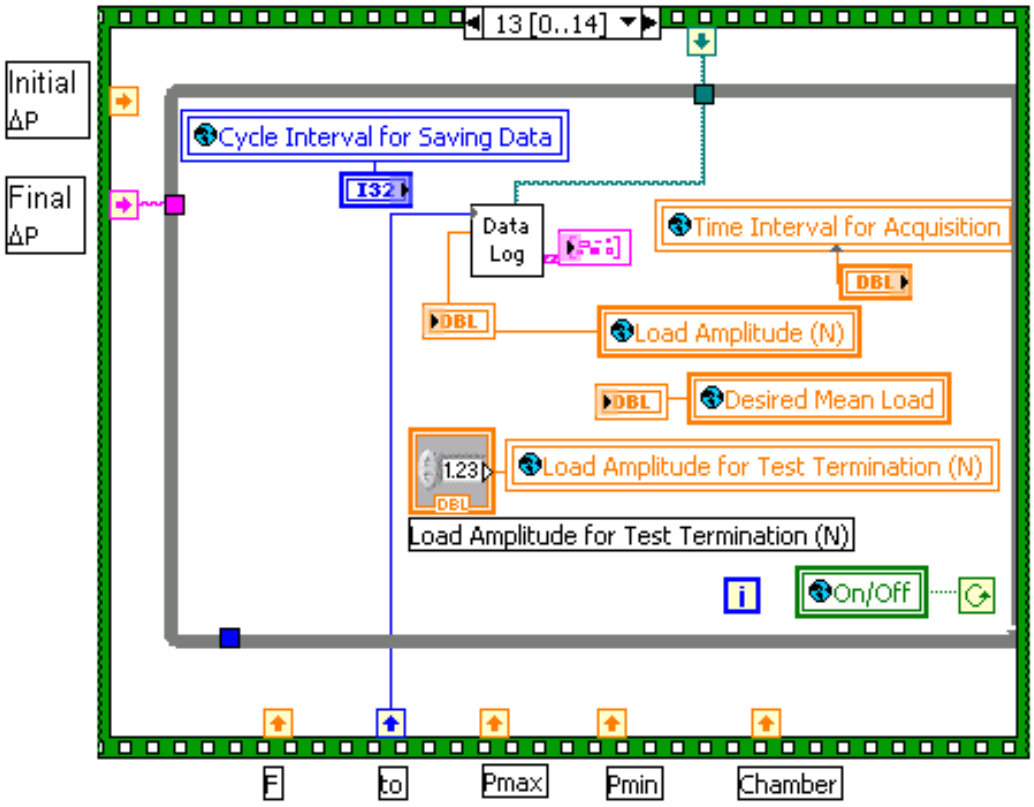

8 [0..14]
Initial ΔP
Final ΔP
abc...
Data File Location and Name
Load Amp. (N)
Mean Load (N)
Frequency (Hz)
Time (s)
Number of Cycles
F
to
Pmax
Pmin
Chamber

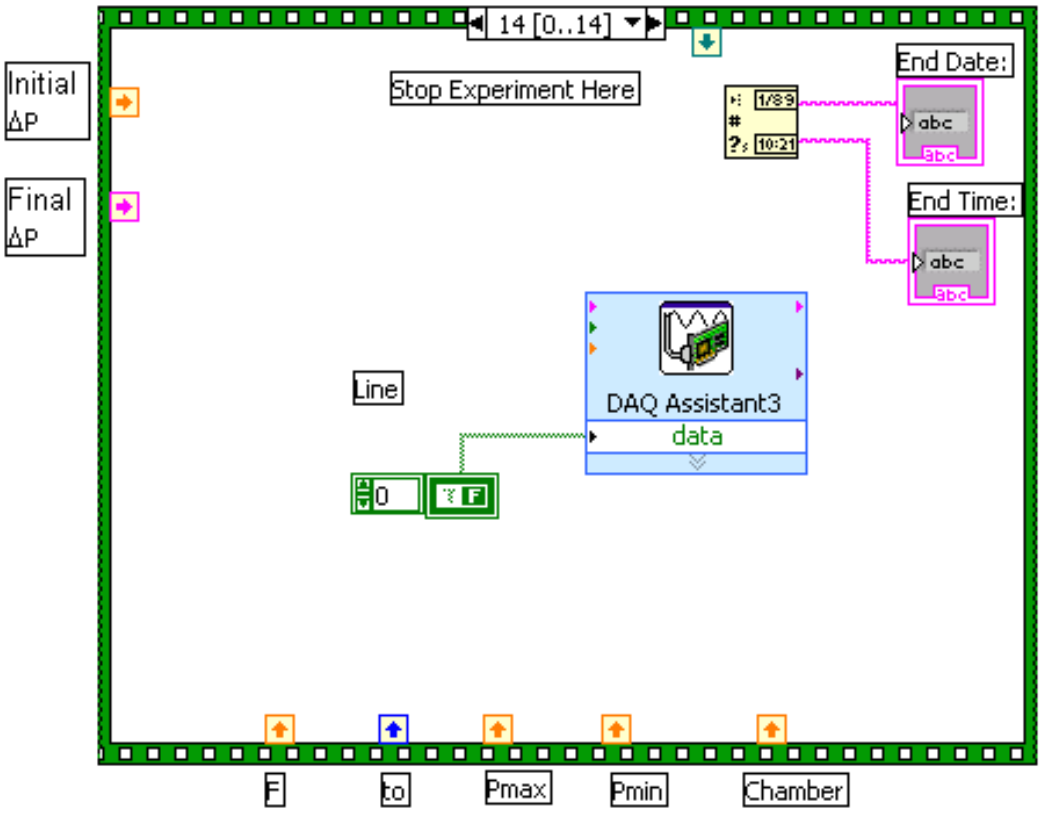

9 [0..14]
Initial ΔP
Final ΔP
The acquisition will start automatically once the cycling begins.
F
to
Pmax
Pmin
Chamber


10 [0..14]
Start Experiment Here
Initial ΔP
Final ΔP
Line
Line
DAQ Assistant2
data
0
F
to
Pmax
Pmin
Chamber


11 [0..14]
Initial ΔP
Final ΔP
0.65
Force Meas. 6
50
i
F
to
Pmax
Pmin
Chamber


12 [0..14]
Initial ΔP
Final ΔP
Start Date:
Start Time:
abc
DBL
On/Off
F
to
Pmax
Pmin
Chamber


13 [0..14]
Initial ΔP
Final ΔP
Cycle Interval for Saving Data
I32
Data Log
Time Interval for Acquisition
DBL
DBL
Load Amplitude (N)
DBL
Desired Mean Load
1.23
DBL
Load Amplitude for Test Termination (N)
Load Amplitude for Test Termination (N)
On/Off
F
to
Pmax
Pmin
Chamber


14 [0..14]
Initial ΔP
Final ΔP
Stop Experiment Here
End Date:
End Time:
Line
DAQ Assistant3
data
0
F
to
Pmax
Pmin
Chamber