Fatigue Crack Initiation and Propagation on Nitrided Ferritic Surfaces in Stainless Steel 439

by

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work obtained herein have not been undertaken or done by unspecified sources or persons.

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RICK RAYNOLD CLAUDIUS

ABSTRACT

Ferritic stainless steel grade 439 is not one of the most commonly used materials in the area of industrial application as compare to other stainless steel such as austenitic stainless steel grade 316 and 304 especially in the area of case hardening process of nitriding. Hence, a constructive study had been undertaken to study the process of nitriding on the ferritic stainless steel grade 439 and the effect of nitriding whether it has a significant improvement in crack initiation and propagation. The experimental procedures of the nitriding process and equipment involved were being determined through numbers of literature review. The effect of nitriding on fatigue crack initiation and propagation of various other grades of stainless steel plates was also being so that the expected results of the experiment could be understood prior to the experiment. The methodology of the project study had been arranged accordingly from initial literature review of the subject until the experimental procedures and the analysis method was determined.

AKNOWLEDGEMENT

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Nitriding process is considered as a highly specialized surface hardening treatment which gives a high hardness case on various types of steels. The main advantage of the nitriding process is that the case hardness is developed without the need of quenching. Finishing operations may be neglected or keep to a minimum.

The nitrided surfaces are highly wear resistant and most importantly, the fatigue life is greatly improved as well as the corrosion resistance. The surface hardness of the nitrided surface is also resistant to softening by temperatures up to the process temperature. The white layer which is a common by product of nitriding is a thin layer of extremely hard iron nitrided. This is the layer that is responsible in delaying the crack initiation and subsequently slowing down propagation so that the nitrided material will have a longer crack propagation life.

Nitriding process plays an important role in many industrial applications as it is one of the basic methods for increasing the fatigue strength of the stainless steel. Along with the derivative nitrocarburizing process, nitriding is commonly used in the manufacture of aircraft, bearings, automotive components, textile machinery, and turbine generation systems.

1.2 Problem Statement

Ferritic stainless steel grade 439 is not one of commonly used materials in industrial application as compared to austenitic stainless steel grade 316/304. Nitriding is one of the case hardening process producing a nitrided ferritic stainless steel with high hardness characteristics at the expense of other mechanical and thermal properties. Thus far, not many literatures can be found investigating the fatigue crack initiation and propagation on nitrided ferritic surfaces in stainless steel 439. Therefore, this study will investigate the effect of nitriding on the fatigue crack initiation and propagation on ferritic surfaces in stainless steel 439. The result of this study will help expand the application of the nitrided ferritic stainless steel 439.

1.3 Objectives

1) The objectives of this study are to investigate the effect of nitriding on the fatigue crack initiation and propagation of the ferritic stainless steel 439

2) The difference between high temperature nitriding and low temperature nitriding on the fatigue crack and propagation of the ferritic stainless steel 439

1.4 Scopes of Study

The scope of study will cover:

1) The understanding of the experimental procedures for nitriding process
2) The understanding of how nitriding will affect the fatigue crack initiation and propagation
3) The understanding of how the fatigue crack initiation and propagation occurs
4) The understanding of the experimental procedures for fatigue crack initiation and propagation testing

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Ferritic Stainless Steel 439

Stainless steel are known to be 'stainless' due to their chromium content which having a minimum of 10.5%. This gives them a great resistance against wet corrosion and high temperature oxidation [1]. Stainless steel 439 is ferritic steel that is designed to resist corrosion in various oxidizing environments [2]. Stainless steel 439 is attractive for numerous automotive exhaust applications and are normally used as tubular manifolds and exhaust system components [2-3].

The chemical composition for ferritic stainless steel grade 439 is shown on table below [2-3]:

TABLE 2.1 Chemical Compositions for Ferritic Stainless Steel Grade 439

Carbon	Manganese	Sulfur	Phosphorus	Silicon	Chromium	Nickel	Nitrogen
0.30 max.	1.00 max.	0.30 max.	0.40 max.	1.00 max	17.0-19.0	0.50 max.	0.15 max.

2.2 Cyclic Loading

Cyclic loading is the repeated or fluctuating stresses, strains, or stress intensities to locations on structural components such as rods and plates. The degradation that may occur on the structural components is referred to as fatigue degradation which started off in the form of cracks and slowly propagates till fracture.

In general, cyclic loadings has no repeated patterns or in situation where overloading occurs. However, a simple relation between stress and number of cycles to failure (time) can be expressed in a sinusoidal curve to investigate the fatigue behavior according to engineering purpose. [4]

2.3 Fatigue Crack Initiation and Propagation in Stainless Steel

Fatigue crack initiation and growth in stainless steel materials is well-understood in the industrial applications. In general, stainless steel components and structures contain notches and geometrical discontinuities [4]. These notches and geometrical discontinuities could be sites of crack initiation. During fatigue process, crack initiation will occurs as a consequence of micro structural changes in stainless steel [4-5]. Crack initiation and propagation will cause permanent damage to stainless steel [6]. Studies on the crack initiation show that plenty of cracks initiated suddenly and tended to be distributed perpendicular to the stress axis after first cracks were observed [7].

2.4 Nitriding

Nitriding is a thermochemical treatment to improve the fatigue endurance, wear as well as corrosion resistance of steel parts. The nitrogen may dissolve in the steel or form nitride precipitates depending on the nitriding technique used and range of temperature [8]. Typical nitriding process usually involves three major phases which are preheat nitriding and cooling [9].

FIGURE 2.1 Gas nitriding furnace

In gas nitriding technique, the steel is exposed in nitrogenous atmosphere within a furnace at elevated temperature [8-9]. It is considered as low temperature gas nitriding when gas nitriding occurs at 400 – 700 ° C and also known as conventional nitriding [10]. During the gas nitriding process, the steel surface will have a formation of white compound which consist of nitrogen solid solution or nitrides. The diffusion of nitrogen into steel takes place according to the following chemical reaction [11-12]:

$$^{1}/_{2}\, N_2\ (Gas) \leftrightarrow N \tag{2.1}$$

Gas nitriding at high temperature at around 1000 – 1200°C is known as solution nitriding or high temperature gas nitriding (HTGN) and quite different from conventional gas nitriding [13]. Nitrogen is added and diffused interstitially into the steel which leads to nitrogen dissolution in austenite [11-13].

2.5 Effect of Nitriding on Fatigue Crack Intiation and Propagation in Stainless Steel

Nitriding of the stainless steel will form a nitride layer which increase the surface toughness of the ferritic [10], austenitic and martensitic stainless steel. Apart from that, it will also increase wear and corrosion resistance [10-12]. Figure 2.2 below shows a typical nitrided case structure.

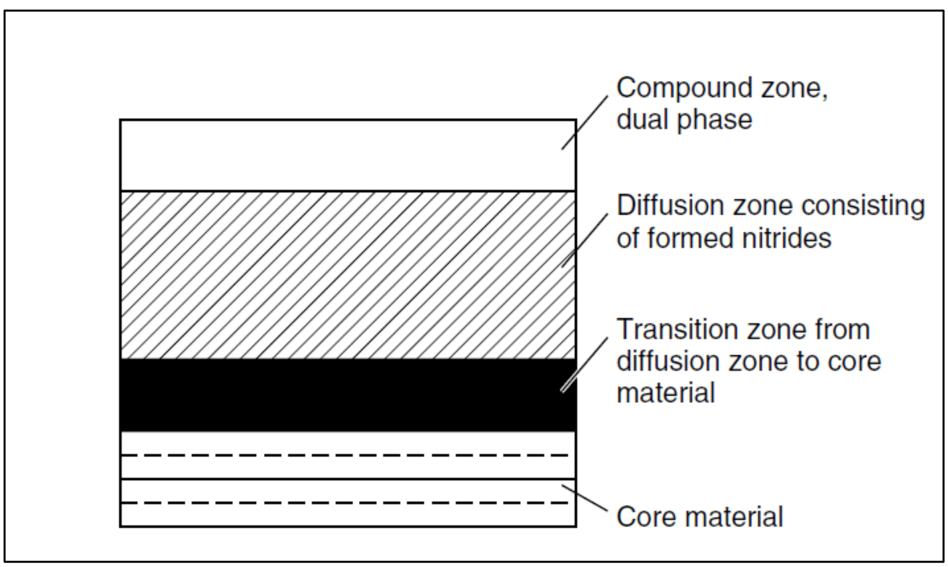


FIGURE 2.2 Typical nitrided case structures [18]

In terms of microstructure aspect of nitrided stainless steel, when nitrogen which is a strong austenite forming element, permeates from the surface into the stainless steel interior, the microstructure of the surface changes into austenite or martensite depending on the amount of nitrogen permeated as well as the process temperature [8].

Nitriding will improve the surface properties of the steel such as wear and fatigue strength. Fatigue strength is improved by a state of compressive residual stresses in the surface layer after nitriding [14-16]. This will in turn affect the behavior of the nitrided stainless steel towards fatigue crack initiation and propagation. The surface hardness of the nitrided steel was improved by three times at the compound layer according to a study [15]. A study also show that nitriding is found to affect fatigue crack behavior by delaying crack initiation and propagation of the austenitic steel due to its increased surface toughness [16].

Study on austenitic stainless steel show that high temperature gas nitriding not only improve fatigue life, but it can simultaneously improve creep resistance, tensile strength, localized corrosion resistance and wear resistance [17]. It was found that fatigue limit of nitrided steel were superior when compared to those of annealed. The crack growth rate was markedly decreased by nitriding [18].

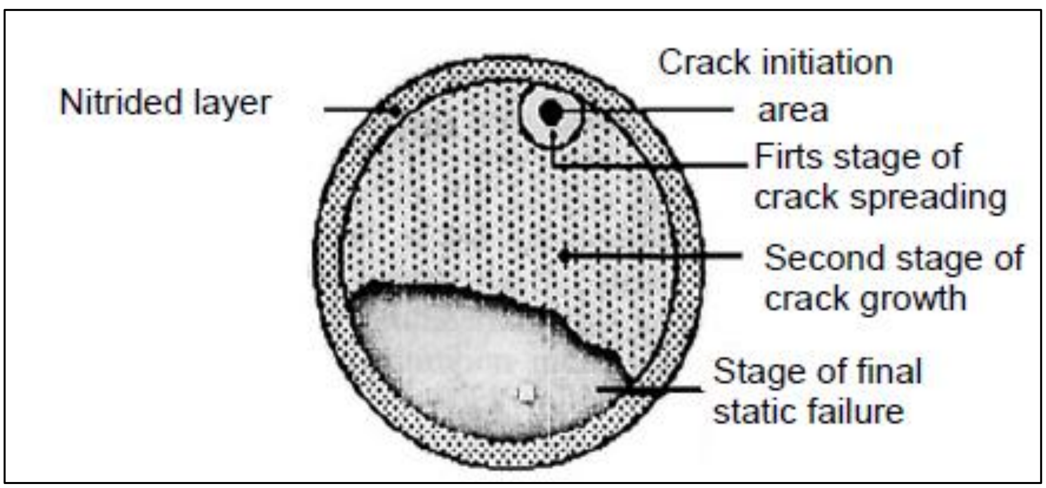


FIGURE 2.3 Scheme of fatigue fracture with near surface initiation of a crack [19]

A structure regarded as the nitride layer will block the crack which is shown clearly by the figure 2.2 above. The nitride layer on the steel will delay the growth of the crack compared to leading cracks in the base metal which normally happens on non nitrided stainless steel. It was concluded from a study that the fatigue limit of nitrided steel increased 30% as a result of nitriding [18-19]. The hardness of a nitrided layer will increase as the gas nitriding time increase [19]. The decrease of the fatigue limit crack growth rate in nitrided steel improved the fatigue limit. It was concluded from a study that the time for crack occurrence and the rupture life of the nitrided steel were longer compare with non nitrided steel [18].

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

The research methodology is summarized as follows:

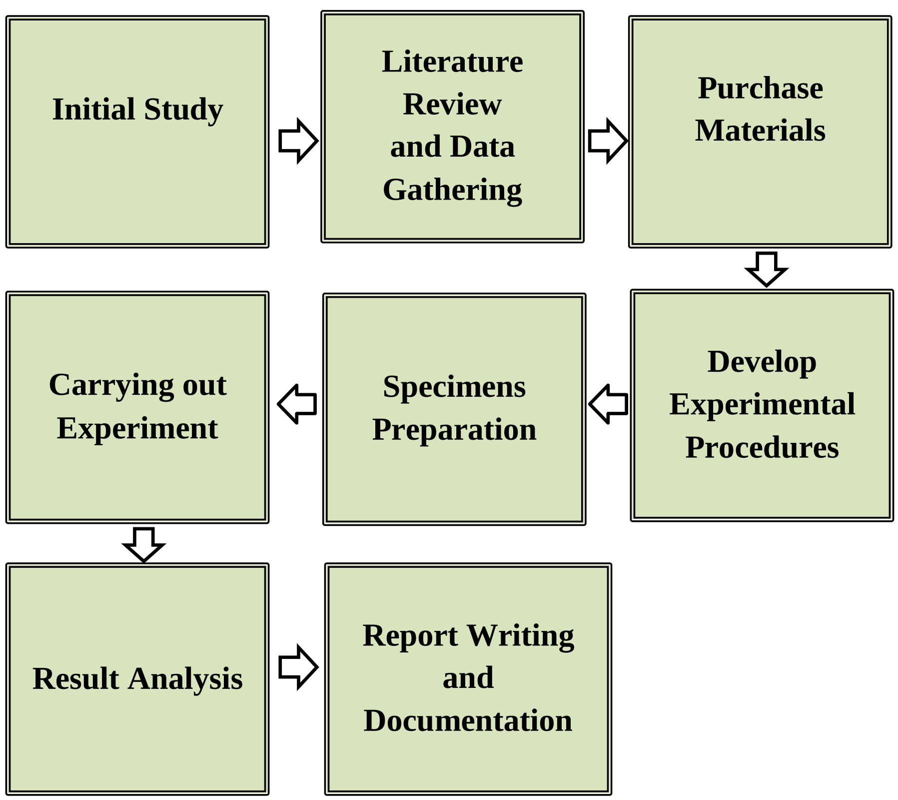


FIGURE 3.1 Final year project research methodology

The methodology for the final year project was initiated with an initial research on the final year project title. It is important to get the general ideas on what the project is all about before moving on for literature review and data gathering.

This is where enough sources of references must be gathered to assist the understanding of the final year project. The references will cover in depth study on the areas especially on the experimental procedures of the project undertaken.

With all the sufficient information gathered, the procedures for carrying out the experiment for the project are developed. This will then followed by the purchasing of the materials for the experiment.

After the experiment had been done, the results will be analyzed and further correlation studies will be carried out before documentation could start.

3.2 Experimental Procedures for the Study of Fatigue Crack Initiation and Propagation on Nitrided Ferritic Surfaces in Stainless Steel 439

Several important specimen preparations must be done before the fatigue testing can commence, the preparations are as follows:

a. The purchased material (stainless steel 439 rod) will be machined into 15 fatigue specimens that follow the standard dimension for fatigue testing. Figure 3.2 below shows the standard dimensions of a fatigue specimen that is used in rotating fatigue testing machine which is going to be use for this project.

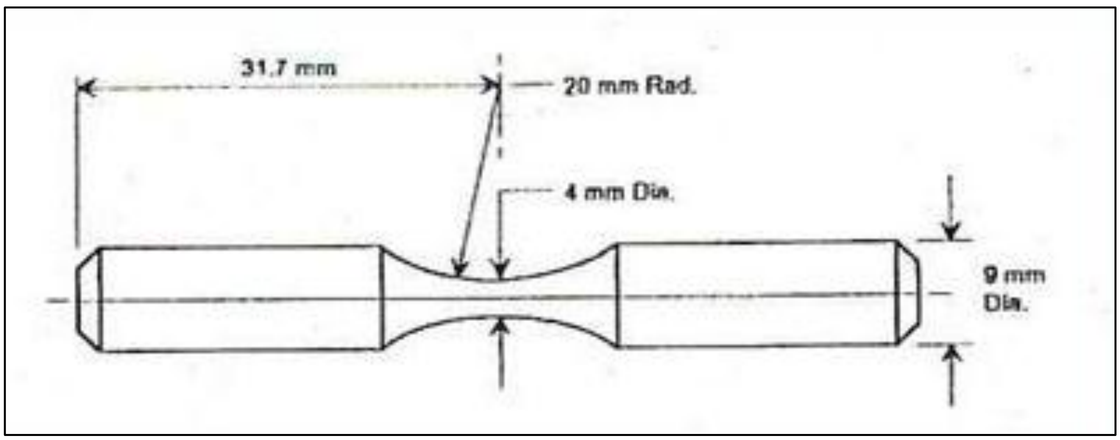


FIGURE 3.2 Fatigue specimen

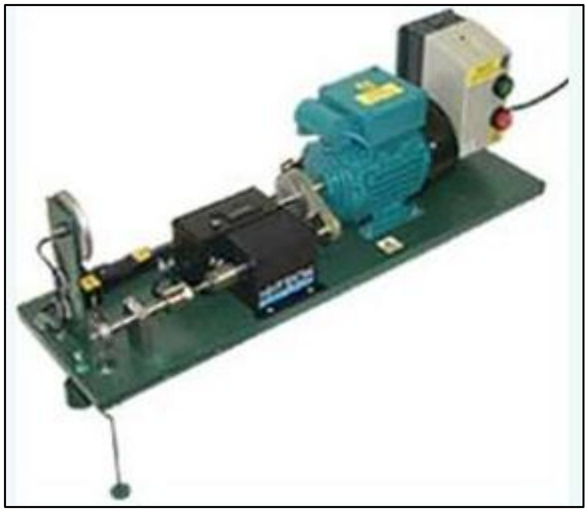


FIGURE 3.3 Rotating fatigue machine

Figure 3.3 above shows the rotating fatigue testing machine. During the fatigue testing, specimen rotating action will be driven by motor results in tensile stress in the lower fibrous and compressive stress in the upper fibrous of the specimen gauge length. The specimen therefore, will be subjected to alternating tensile and compressive stresses similar to the reversed cyclic loading.

b. The fatigue specimens that had been machined will be nitrided with two different temperatures. There will 10 specimens to be nitrided. 5 specimens will be nitrided with high temperature nitriding (1100°C) while the other 5 specimens will be nitrided with low temperature nitriding (500°C). The rest 5 specimens will be left without nitriding so that they could act as the control variable.

Below are the parameters that will be used in the nitriding process stainless steel 439 fatigue specimens:

TABLE 3.1 Parameters for the nitriding process

Parameters	**Value**
Temperature	1100^{o}C (high temperature nitriding) 500°C (low temperature nitriding)
Time	10 hours
Pressure	50 – 100 mmHg
Gas	Purified Nitrogen
Temperature rate	3^{o}C/min
Flow rate	1000 cm^{3}/min

Nitrided case depth generally increases with temperature and nitriding time. Figure 3.4 below shows a typical image of nitrided case structure of a specimen under the stereoscopic microscope. The thin white layer is a typical by-product of nitriding. It is an extremely hard thin layer of iron nitrided.

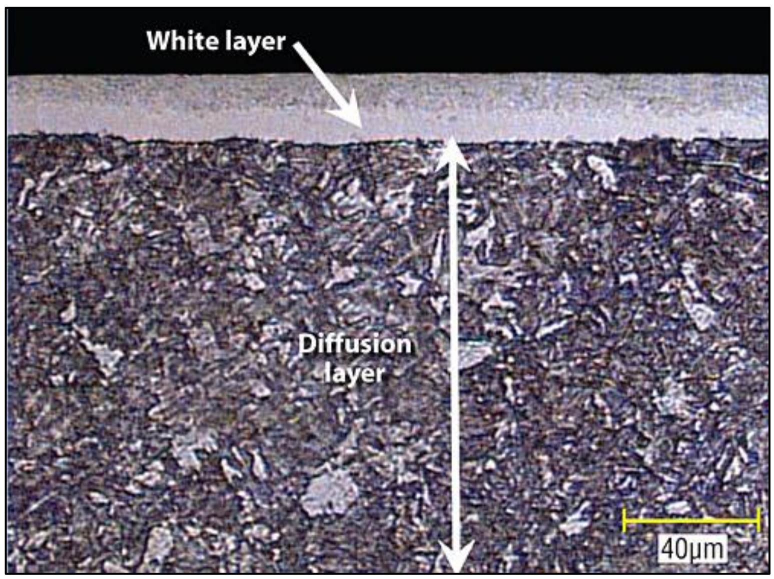


FIGURE 3.4 Typical image of nitrided case structure of a specimen under stereoscopic microscope [14]

Once all the sample preparations had been done, the project could proceed to study the fatigue life and fatigue crack initiation and propagation of the specimens.

The rotating fatigue testing to determine the fatigue life of the specimens follows these procedures:

1. Set up the non nitrided specimen into the rotating fatigue testing machine (refer FIGURE 3.3) with a load of 40 N.

2. Run the rotating fatigue testing machine till the specimen fail and break.

3. Record the number of cycles.

4. Repeat step 1 to 3 with low temperature nitrided specimen and high temperature nitrided specimen.

5. Repeat step 1 to 4 with a load of 30 N, 20 N and finally 10 N.

Following the rotating fatigue testing done to determine the fatigue life, another rotating fatigue testing will be done to study on the fatigue crack initiation and propagation of the specimens. The experiment will follow these procedures:

1. Set up the non nitrided specimen into the rotating fatigue testing machine (refer FIGURE 3.3) with a load of 40 N.

2. Interrupt the fatigue testing at two different appropriate numbers of cycles based on the results for the previous rotating fatigue testing. Observe any cracks initiated and its propagation with optical microscope by using appropriate magnification.

3. Save the captured images.

4. Repeat step 1 to 3 with low temperature nitrided specimen and high temperature nitrided specimen.

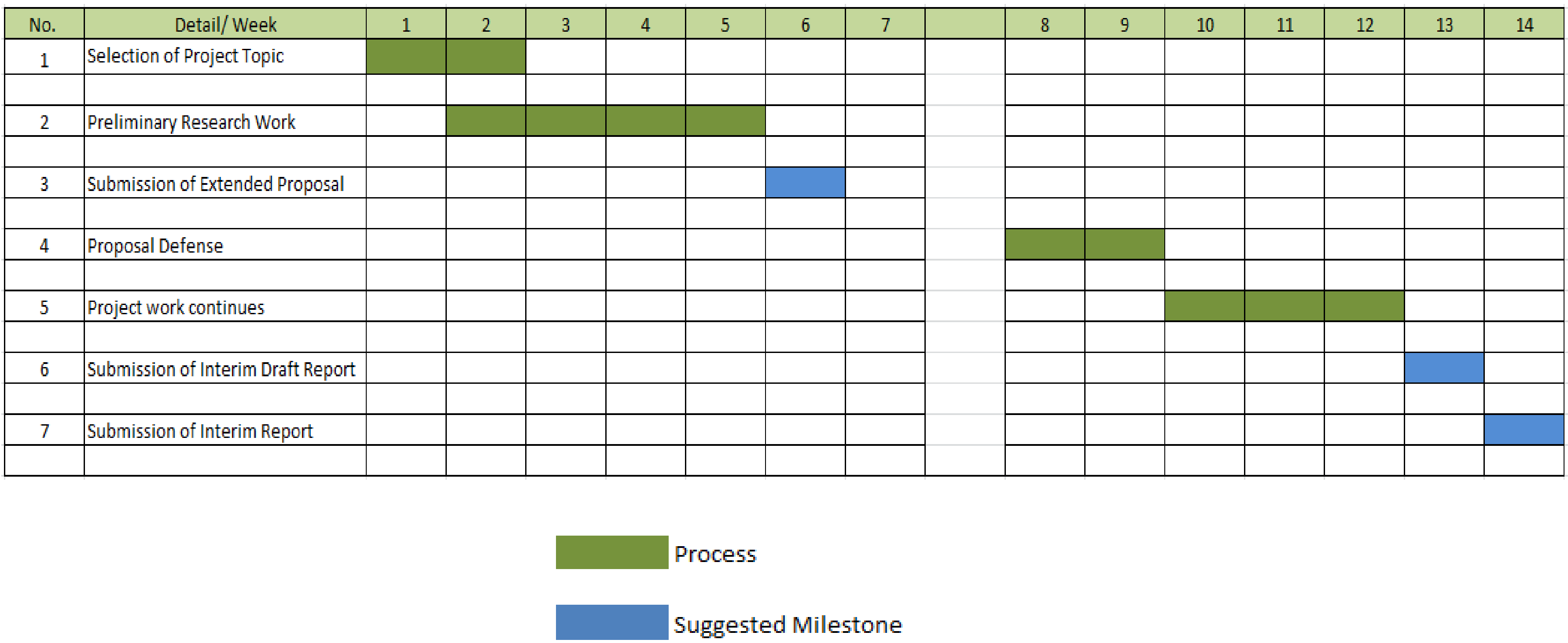
Lastly, the study of fracture surface will be done. The experiment follows these simple procedures:

1. Study the fracture surface of non nitrided specimen due to cyclic loading of 40 N by capturing the image of the fracture surface.

2. Save the image.

3. Repeat step 1 and 2 with low temperature nitrided specimen and high temperature nitrided specimen.

3.3 Gantt Chart



CHAPTER 4

PROJECT ATIVITIES

4.1 Machining of the Stainless Steel 439 Fatigue Testing Specimens

Stainless steel 439 was purchased in the form of a long rod. This long rod was machined into fatigue testing specimens which follow the standard dimensions for rotational fatigue testing machine. Figure 4.1 below shows the stainless steel 439 fatigue testing specimens after machining processes.

FIGURE 4.1 Stainless steel 439 fatigue specimen

4.2 Nitriding of the fatigue testing specimens

The specimens were nitrided with high temperature nitriding (1100^{o}C) and low temperature nitriding (500°C). Figure below shows the tube nitriding furnace that was used during the nitriding process.

FIGURE 4.2 Tube nitriding furnace

Figure below shows the pure nitrogen gas tank that was used during the nitriding process.

FIGURE 4.3 Pure nitrogen gas tank

CHAPTER 5

RESULTS AND DISCUSSION

5.1 The Surface Properties

The three types of specimen:

- **Non nitrided**
- **Low temperature nitrided**
- **High Temperature nitrided**

was then observe and compare to see any significant differences between their surface properties (color, roughness) and to note any changes after the specimens were nitrided with high and low temperature. Figure below shows a low temperature nitrided specimen.

Figure 5.1 Low temperature nitrided specimen

Figure below shows a high temperature nitrided specimen.

Figure 5.2 High temperature nitrided specimen

Non nitrided specimens basically have a silver color for its surface. It also has a smooth surface finish after the machining process.

After it was nitrided, there are significant changes in terms of its color and surface roughness. The low temperature nitrided specimens have a gold color for its surface while maintaining a smooth surface similar to the non nitrided specimens.

The high temperature nitride specimens have a dark yellow color for its surface. The surface shows a burnt characteristic where there are some dark areas on some part of the specimen. These specimens posses a rough surface compared with the non nitrided specimens and low nitride specimens.

5.2 Rotating Fatigue Testing Results

Figure below shows the specimen is being loaded into the machine before the rotating fatigue testing was carried out.

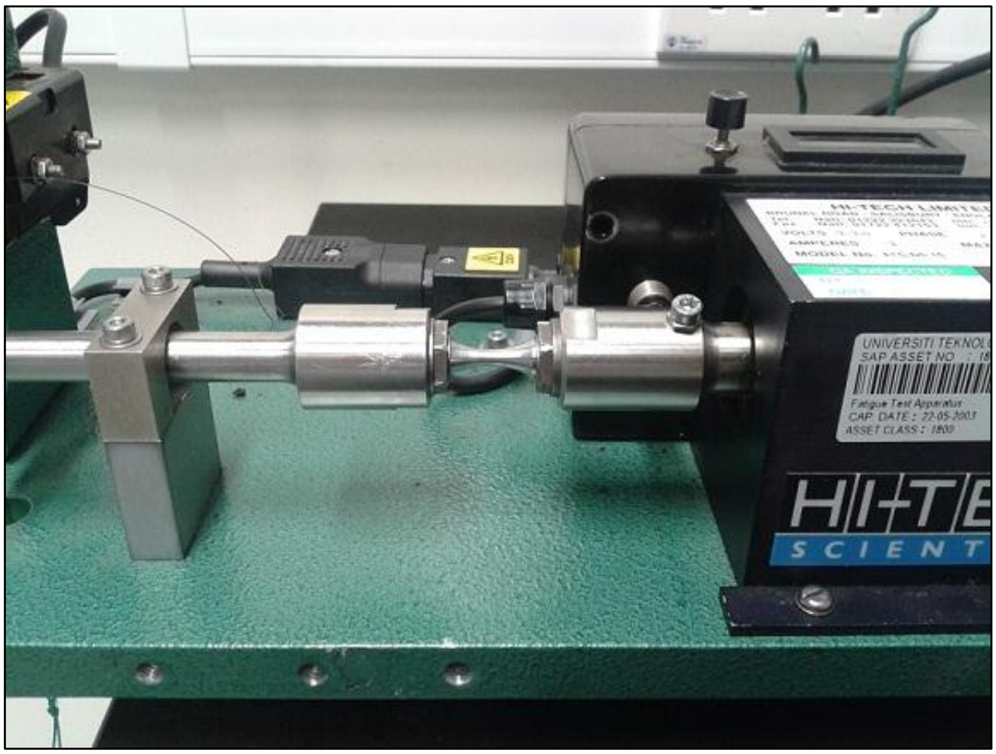


Figure 5.3 Specimen being loaded into the rotating fatigue machine

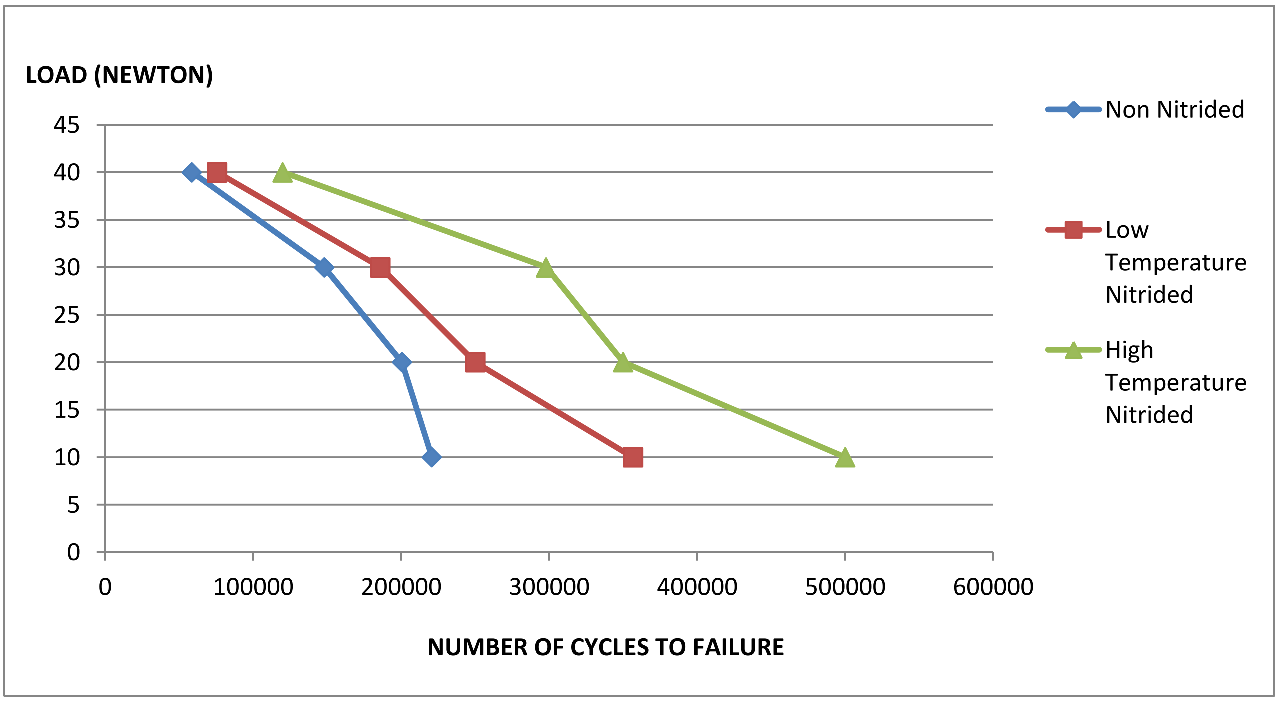
Table below shows the rotating fatigue testing for non nitrided, low temperature nitride and high temperature nitride specimens.

TABLE 5.1 Rotating fatigue testing data

Load (Newton)	Type of specimens	Number of cycles to failure
40	Non nitrided	58705
	Low temperature nitrided	75619
	High temperature nitrided	120056
30	Non nitrided	148086
	Low temperature nitrided	186075
	High temperature nitrided	298067
20	Non nitrided	200671
	Low temperature nitrided	250106
	High temperature nitrided	350106
10	Non nitrided	220798
	Low temperature nitrided	356718
	High temperature nitrided	500148

Table below shows the result from TABLE 5.1 that is represented in the form of Load (N) versus Number of Cycles curves.

TABLE 5.2 Load versus number of cycles to failure



Based on the results for all the three types of specimens, the higher the cyclic loading being applied the lower the number of cycles to failure.

The results from the rotating fatigue testing shows a significant increase in fatigue strength of the nitrided specimens compared with the non nitrided can be noted. This is proven from the acquired data where more number of cycles to failure needed to cause the nitrided specimens to be in a fatigue state and eventually fracture and failed. This applies to the load of 40 N, 30 N, 20 N and 10 N respectively.

For the 40 N loads, there is 104.5% increase in fatigue strength for the high temperature nitrided specimen compared with low temperature nitrided specimen which only 28.8%.

For the 30 N loads, there is 101.3% increase in fatigue strength for the high temperature nitrided specimen compared with low nitrided temperature which only 25.6%.

For the 20 N loads, there is 74.4% increase in fatigue strength for the high temperature nitrided specimen compared with low temperature nitrided specimen which only 24.6%.

For the 10 N loads, there is 126.5% increase in fatigue strength for the high temperature nitrided specimen compared with low temperature nitrided specimen which only 61.5%.

In average, there is 101% increase in fatigue strength for the high temperature nitrided specimen compared with low temperature nitrided specimen which only 35.1%.

5.3 Fatigue Crack Initiation and Propagation Patterns

Figure below shows the study of fatigue crack initiation and propagation patterns of the specimens with optical microscope.

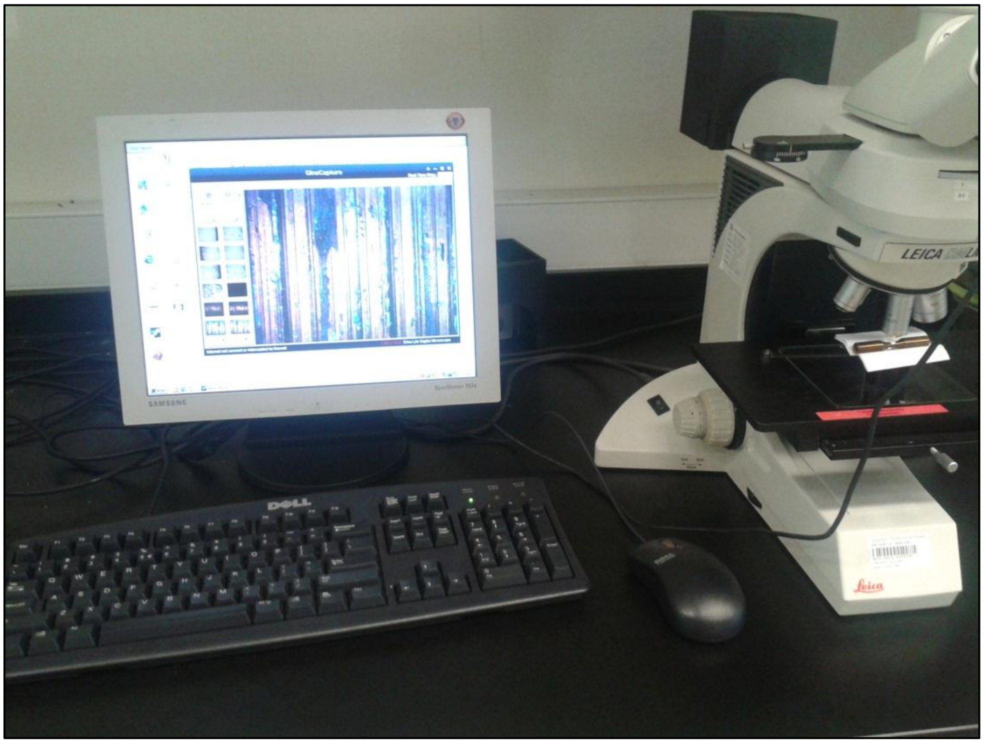


Figure 5.4 Study of fatigue crack initiation and propagation patterns with optical microscope

Figure below shows the surface of non nitrided specimen before cyclic loading being applied.

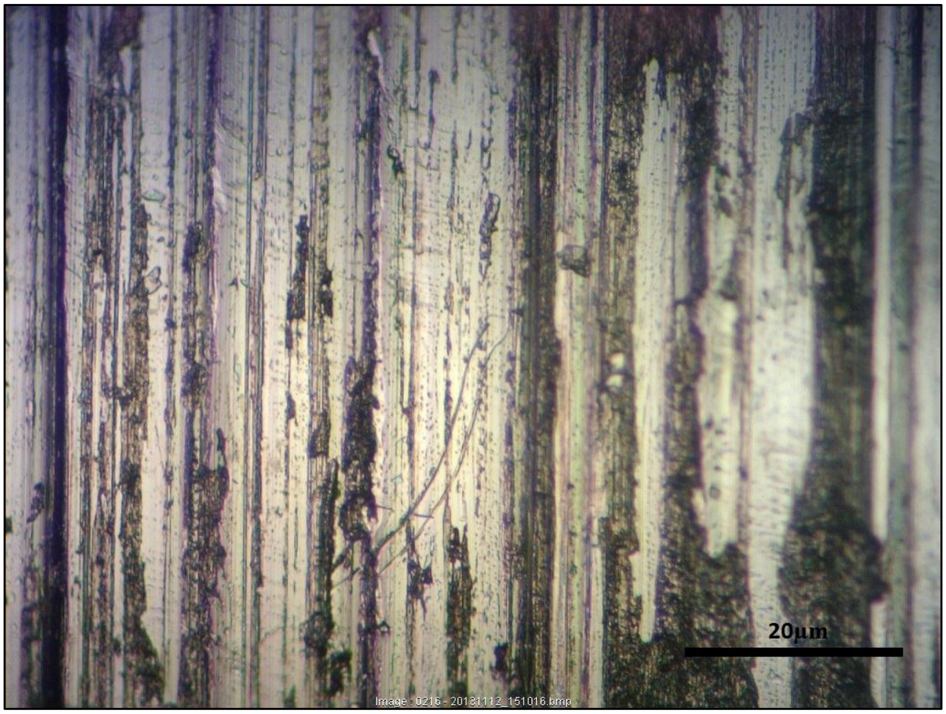


Figure 5.5 Non nitrided specimen surface before cyclic loading being applied

Figure below shows the non nitrided surface after cyclic loading being applied for approximately 35000 cycles under 40 N loads.

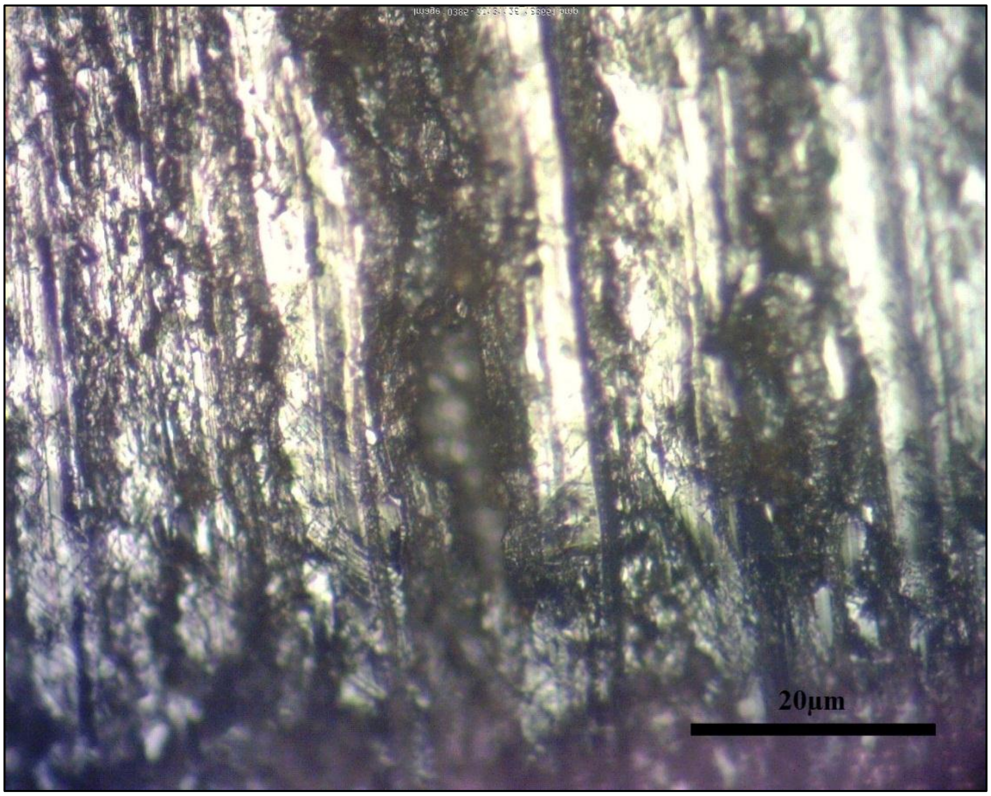


Figure 5.6 Non nitrided specimen surface after cyclic loading being applied for approximately 35000 numbers of cycles

Figure below shows the non nitrided surface after cyclic loading being applied for approximately 50000 cycles under 40 N loads.

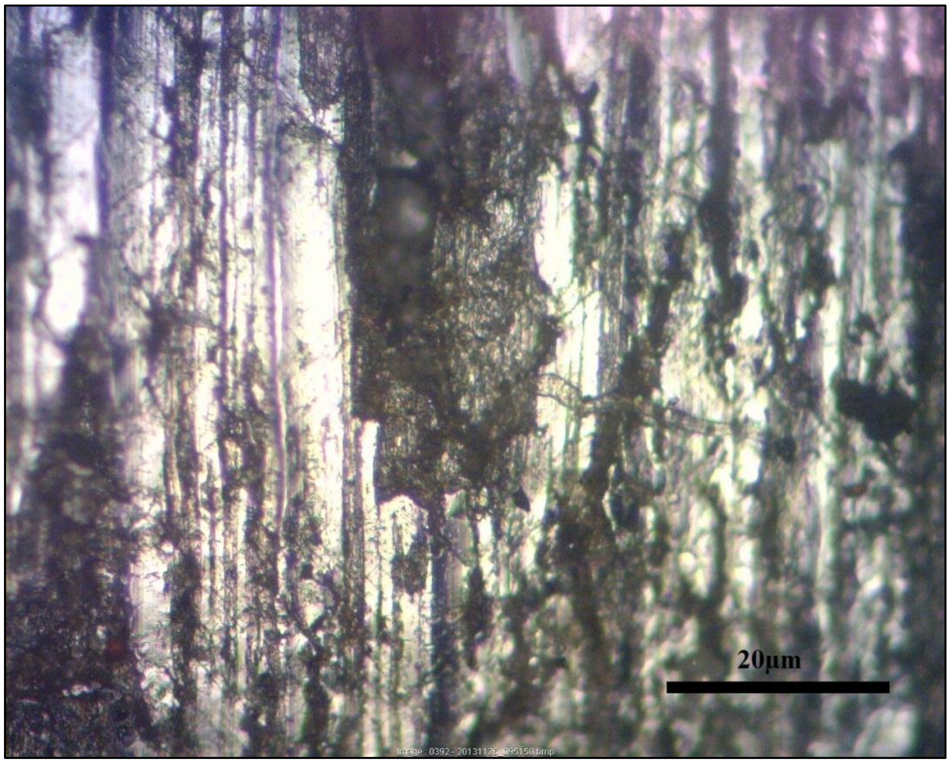


Figure 5.7 Non nitrided specimen surface after cyclic loading being applied for approximately 50000 cycles under 40 N loads

Figure below shows low temperature nitrided specimen surface before cyclic loading being applied.

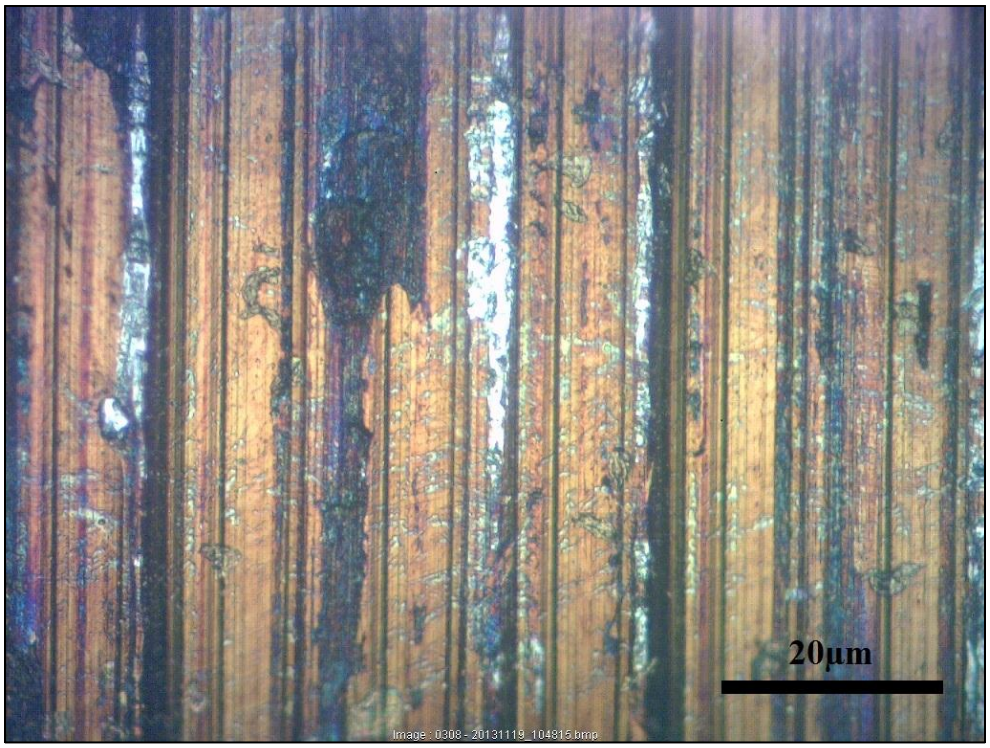


Figure 5.8 Low temperature nitrided specimen before cyclic loading being applied

Figure below shows low temperature nitrided specimen after cyclic loading being applied for approximately 35000 cycles under 40 N loads.

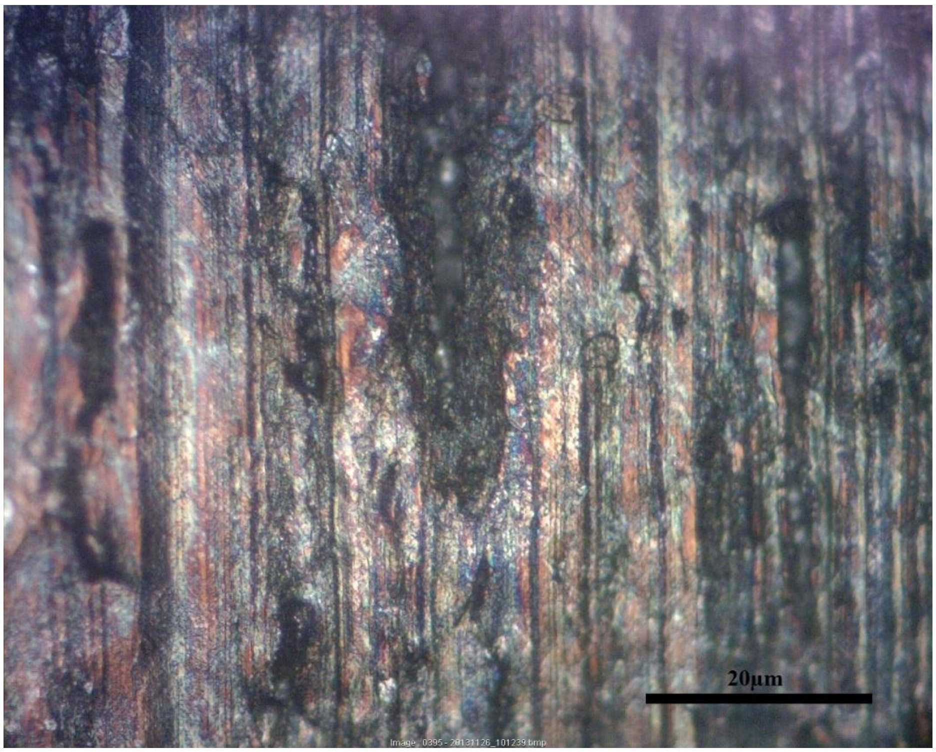


Figure 5.9 Low temperature nitrided specimen after cyclic loading being applied for approximately 35000 cycles under 40 N loads

Figure below shows low temperature nitrided specimen after cyclic loading being applied for approximately 50000 cycles under 40 N loads.

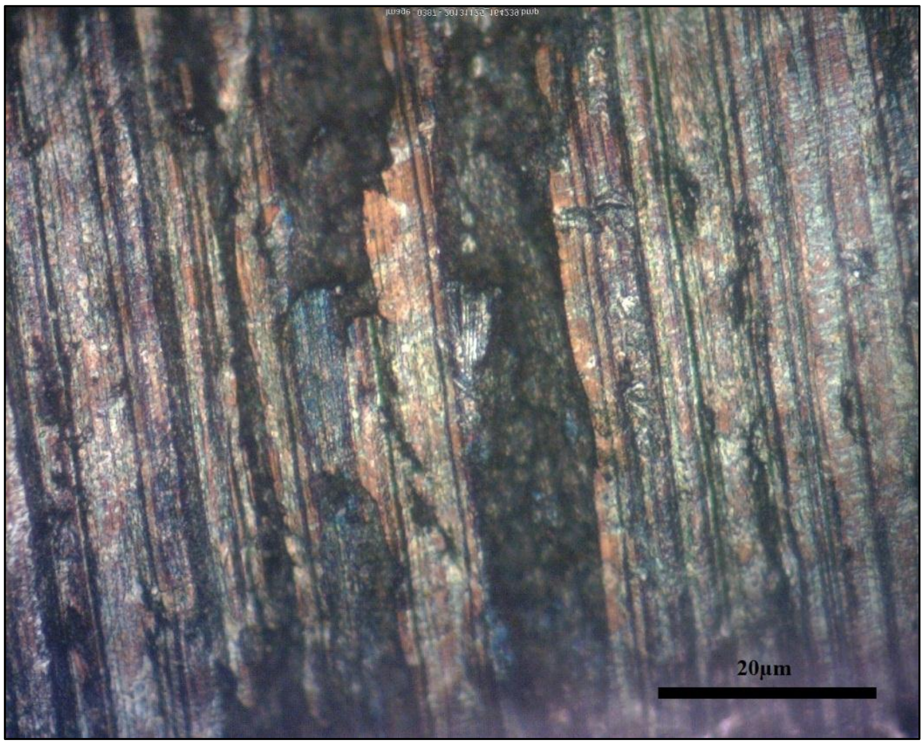


Figure 5.10 Low temperature nitrided specimen after cyclic loading being applied for approximately 50000 cycles under 40 N loads

Figure below shows high temperature nitrided specimen surface before cyclic loading being applied.

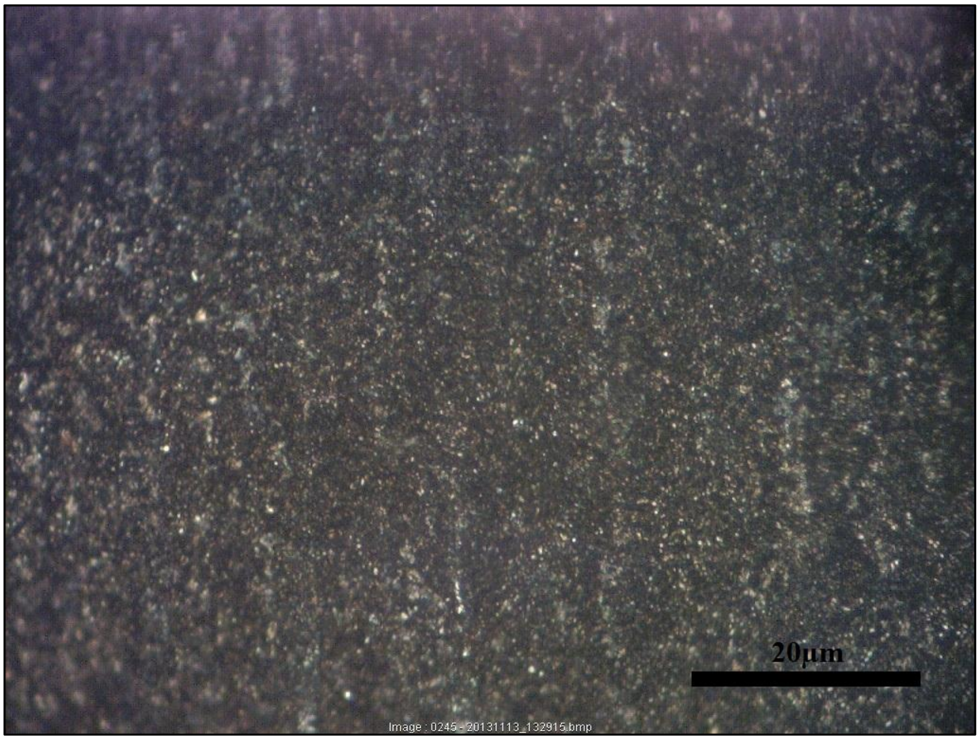


Figure 5.11 High temperature nitrided specimen before cyclic loading being applied

Figure below shows high temperature nitrided specimen after cyclic loading being applied for approximately 35000 cycles under 40 N loads.

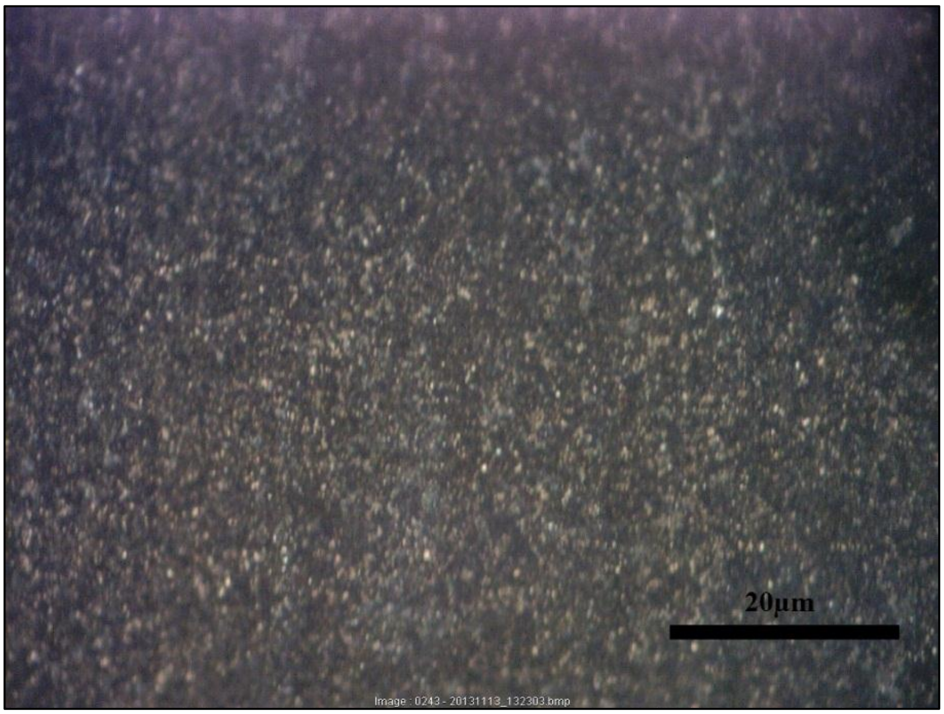


Figure 5.12 High temperature nitrided specimen after cyclic loading being applied for approximately 35000 number of cycles

Figure below shows high temperature nitrided specimen after cyclic loading being applied for approximately 50000 cycles under 40 N loads.

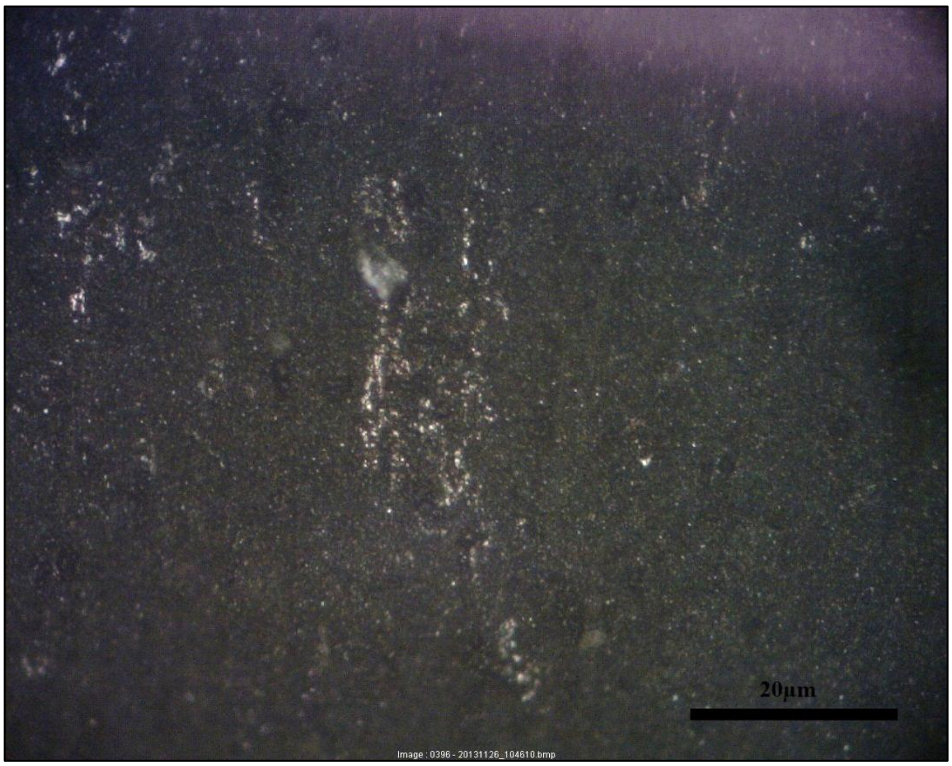


Figure 5.13 High temperature nitrided specimen after cyclic loading being applied for approximately 50000 cycles under 40 N loads

The results, based on the optical microscope images presented in figure 5.5 to figure 5.13 show how nitriding improves the fatigue resistance. This further illustrated when comparing the crack growth patterns between the three different specimens.

Based on the observation of the fatigue crack initiation and propagation patterns with optical microscope, results shows a significant difference in terms of the severity of the fatigue crack initiated when comparison being between the three different specimens at the same approximate number of cycles and under the same cyclic loading.

When compared, the non nitrided specimen shows the most severe fatigue crack initiation and propagation among all the specimens while the low temperature nitrided specimen comes second. The fatigue crack initiate and propagate fast for the non nitrided specimen while the fatigue crack initiation and propagation for the high temperature nitrided specimen is the least severe.

The results also give the idea of how deep the fatigue crack had initiated especially for the non nitrided specimen and low temperature nitrided specimen. The crack initiated for the high temperature nitrided specimen seems to propagate near surface and slowly.

In general, the results show that:

1. Nitriding delays crack initiation
2. Nitriding slows propagation (longer crack propagation life)
3. High temperature nitriding delays crack initiation better
4. High temperature nitriding slows propagation better (longer crack propagation life)

5.4 Fracture Surface Observations

Figure below shows the fracture surface of non nitrided specimen under 40 N cyclic loading.

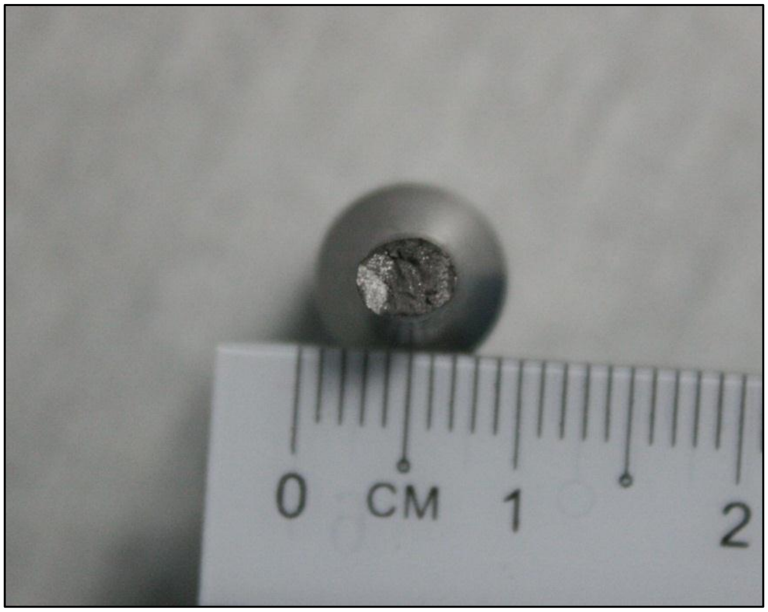


Figure 5.14 Fracture surface of non nitrided specimen under 40 N cyclic loading

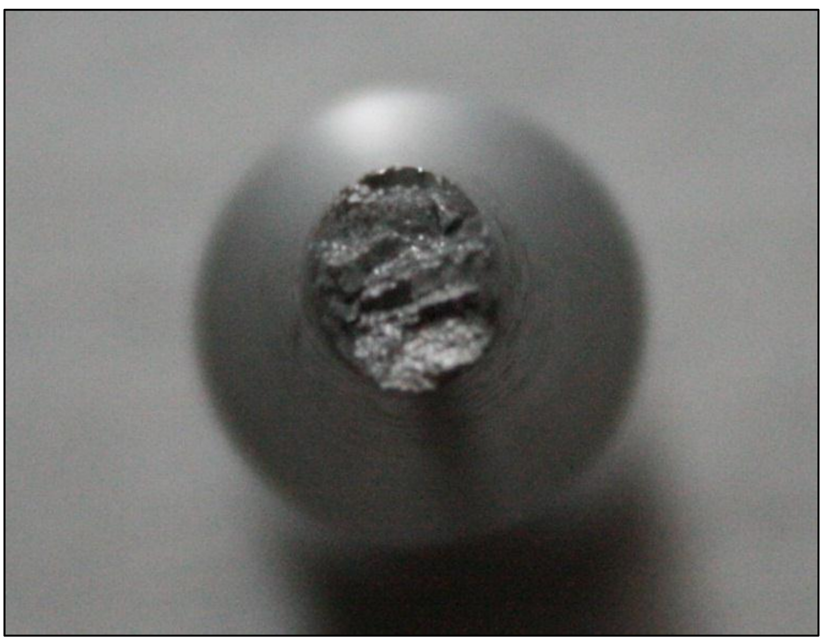


Figure 5.15 Fracture surface of non nitrided specimen under 40 N cyclic loading

Figure below shows the fracture surface of low temperature nitrided specimen under 40 N cyclic loading.

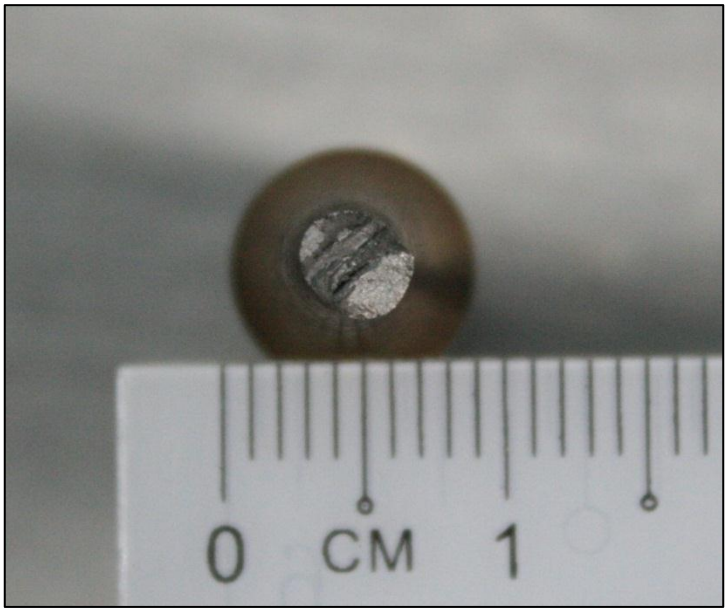


Figure 5.16 Fracture surface of low temperature nitrided specimen under 40 N cyclic loading

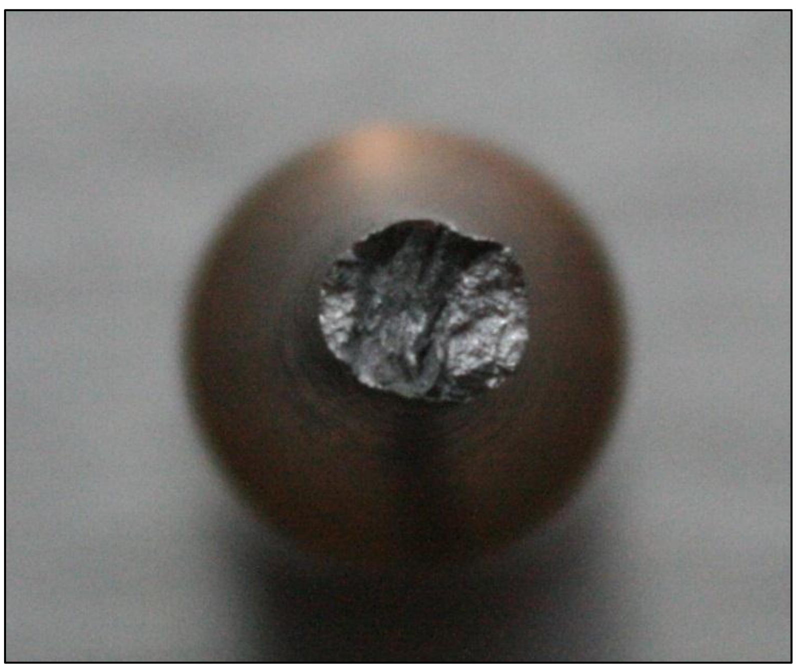


Figure 5.17 Fracture surface of low temperature nitrided specimen under 40 N cyclic loading

Figure below shows the fracture surface of high temperature nitrided specimen under 40 N cyclic loading.

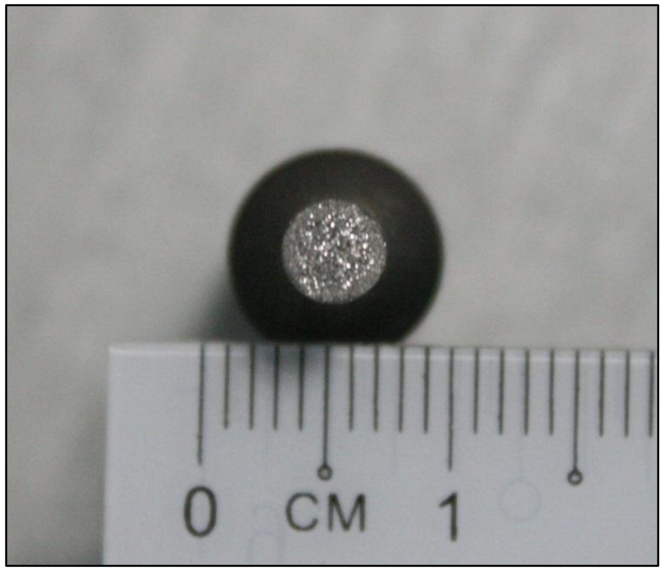


Figure 5.18 Fracture surface of high temperature nitrided specimen under 40 N cyclic loading

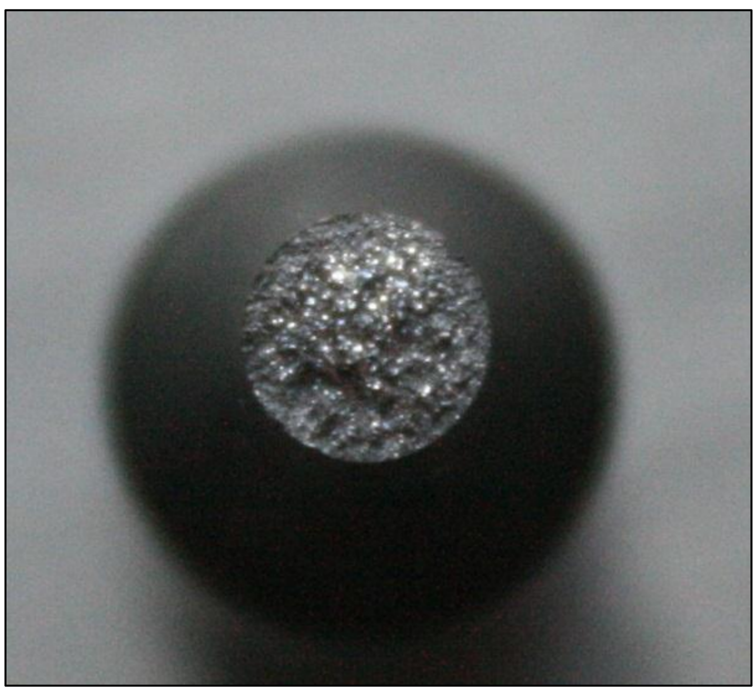


Figure 5.19 Fracture surface of high temperature nitrided specimen under 40 N cyclic loading

The results show that the non nitrided specimens have the most severe fracture surface and the high temperature nitrided specimen is the least severe.

The non nitrided specimen fracture surface has a significant uneven fracture surface. There are clear crack propagations that can be observe on the fracture surface until the point of final fracture.

The high temperature nitrided specimen has a quite even fracture surface which proves that it has a slow and steady propagation of fatigue crack until the point of final fracture.

5.5 Discussion

This study confirms many basic engineering rules in nitriding and also the effect of nitriding based on the initial studies done before. For example, fatigue life of the specimens is increased substantially by nitriding, especially with high temperature nitriding. The plotting of cyclic load and the number of cycles to failure for rotating fatigue testing gives a more thorough understanding on how improvement on fatigue life are achieved as well as the crack initiation patterns and observations of the fracture surfaces. This enables comparison to be made of how severe the crack initiation and propagation between non nitrided specimen, low temperature nitrided specimen and high temperature nitrided specimen.

Based on the rotating fatigue testing results, fatigue crack initiation and propagation patterns and also the fracture surfaces observations, nitriding is found to delay initiation and to slow propagation of cracks.

Also, the cracks initiated seem to propagate deeper within the non nitrided specimen at a faster rate under cyclic loading when compared to high temperature nitrided specimen. This is because for nitrided specimens, there are strong protective nitride layer on the surface of the steel. Thus, this strong protective nitride layer resists and delays the crack initiation and slows down the propagation, making the fatigue life of the nitrided specimens longer than the normal non nitrided specimen.

The surface of the non nitrided specimen is exposed completely without any protection to the risk of crack initiation and faster propagation of the cracks due to fatigue cyclic loading which makes their fatigue life to be lower.

The low temperature nitrided specimens have a lower fatigue life and weaker resistance against fatigue crack initiation and propagation when compared with the high temperature nitrided specimen because it has a weaker nitride layer.

This is due to the lower temperature used during the nitriding process where the nitrogen ions does not diffuse deeper and more efficiently within the stainless steel and form a stronger bond of nitride layer. Thus, it has a less efficiency in fatigue resistance as well as fatigue life when compared with the high temperature nitrided specimens.

CHAPTER 6

CONCLUSIONS

Nitriding is found to affect the fatigue crack behavior by delaying both crack initiation and crack propagation. Nitriding significantly increases the fatigue life of the nitrided specimens especially the high nitriding specimens in comparison with non nitrided specimens.

High temperature nitriding was found to delay both crack initiation and crack propagation compared with the low temperature nitriding based on the results from rotating fatigue testing. High temperature nitriding specimens has a better and stronger nitride layer and have a better fatigue resistance against crack initiation and propagation as well as the overall fatigue life.

Fatigue crack initiation and propagation patterns were shown to change between the non nitrided specimen, low temperature nitrided specimen and high temperature nitrided specimen. The high temperature nitrided specimen has the highest fatigue resistance in delaying both crack initiation and crack propagation.

The objectives for this project had been achieved.

Nitriding have a significant effect on the fatigue crack initiation and propagation in stainless steel 439 by increasing its fatigue resistance against crack initiation and crack propagation as well as fatigue life.

High temperature nitriding produce a better fatigue resistance in delaying crack initiation and crack propagation as well as fatigue life.

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