CHARACTERISATION AND MODELLING OF STATIC RECOVERY PROCESS OF PLAIN CARBON STEEL

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this project and in our opinion this project is satisfactory in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurently submitted for award of other degree.

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For my loving father, mother and family

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ABSTRACT

This thesis is an investigation of static recovery process due to different pre – strain, temperature and time on recovery process. In this project, plain carbon steel specimens were subjected to tensile test and heat treatment process. The specimens were the tensile test specimen shape according ASTM E8. Box furnace was used in this project to perform the heat treatment of the specimens. The specimens were heated to a temperature which is below the recrystallization temperature with certain range of time. In this research, the recovery temperature was varied to 100^{o}C, 200 ^{o}C, 300 ^{o}C and 400 ^{o}C. The pre – strain value was varied to 5%, 10%, 15% and 20% which is the value before the specimen break. The time was varied to 1 hour, 2 hours, 3 hours and 4 hours in order to analyze the static recovery process due to different time. The change of stress values were taken before and after static recovery for all variables to calculate the degree of recovery, X_{rec} by using Friedel's model equation. The graphs were plotted to analyze the relationship between degree of recovery with these variables. The linear equations that obtained from each graph were compared with Friedel's model equation in order to calculate the activation energy, Q. In this research, the activation energy for different temperature (1 hour fixed time, 10% pre - strain) was 118 kJ/mol and the value for different time (300^{o}C fixed temperature, 5% pre - strain) was 162 kJ/mol. Finally, the activation energies that obtained were compared with other journal in order to validate the static recovery process of plain carbon steel by using Friedel's method equation.

ABSTRAK

Tesis ini adalah kajian mengenai proses pemulihan statik untuk pelbagai ketegangan spesimen, suhu dan masa pemulihan. Dalam projek ini, spesimen besi keluli akan diuji dengan analisis ketegangan dan proses rawatan haba. Bentuk spesimen yang digunakan dalam projek ini adalah ASTM E8. Relau akan digunakan untuk proses rawatan haba dalam projek ini. Spesimen akan dipanaskan kepada suhu dan masa tertentu di bawah suhu penghabluran semula. Dalam projek ini suhu dibezakan kepada 100^{o}C, 200 ^{o}C, 300 ^{o}C dan 400 ^{o}C. Nilai pra – tegasan dibezakan kepada 5%, 10%, 15% dan 20% di mana nilai tegasan sebelum spesimen putus. Manakala, untuk perubahan masa, tempoh pemanasan dibezakan kepada 1 jam, 2 jam, 3 jam dan 4 jam. Perubahan nilai tegasan sebelum dan selepas pemulihan statik akan dianalisis untuk semua pembolehubah untuk pengiraan darjah pemulihan, X_{rec} mengunakan persamaan model Friedel. Hubungan di antara darjah pemulihan dan pembolehubah tersebut dianalisis melalui graf. Persamaan linear yang terhasil daripada graf akan dibandingkan dengan persamaan model Friedel untuk pengiraan tenaga pengaktifan, Q. Dalam projek ini, tenaga pengaktifan yang terhasil daripada perbezaan suhu (masa ditetapkan 1 jam, 10% pra - tegasan) ialah 118 kJ/mol, manakala untuk perbezaan masa (suhu ditetapkan 300^{o}C, 5% pra - tegasan) nilainya ialah 162 kj/mol. Akhir sekali, nilai ini dibandingkan dengan jurnal lain untuk sahkan proses pemulihan statik untuk besi keluli dengan menggunakan kaedah persamaan Friedel.

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LIST OF SYMBOLS

ε	Strain
σ	stress
σ_o	Yield stress of the undeformed specimen
σ_m	Maximum yield stress
σ_r	Yield stress of the static recovery
C	Constant
X	Degree of recovery
t	Time
Q	Activation Energy
T	Temperature
R	Gas constant (8.314 J/mol.K)
M	2 for BCC metal
α	Constant
G	Shear modulus
b	Burgers vector
ρ	Dislocation densities
σ_d	Stress due to dislocation

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
AISI	American Iron and Steel Institute
C	Carbon
Cu	Copper
Mn	Manganese
Si	Silicon
P	Phosphorus

CHAPTER 1

INTRODUCTION

1.1 PROJECT OVERVIEW

Static recovery is part of annealing process which is used for metal heat treatment. This process is done to change the properties of the material such as strength and hardness. When metal is heavily in cold- worked, much of strain energy expanded in the plastic deformation is stored in the metal in form of dislocations and other imperfections such as point defects. When cold – worked is reheat in the recovery temperature range just below recrystallization temperature range, sufficient thermal energy is supplied to rearrange the dislocation into lower energy configuration. During recovery, the strength of a cold – worked is reduced slightly but the ductility is significantly increased.

Basically, there are three types of plain carbon steel which are low carbon steel, medium carbon steel and high carbon steel. These types of carbon steel are differentiated with amount of carbon content. Low carbon steel or mild steel is approximately 0.05% – 0.15% carbon content. Example of this low carbon steel is AISI 1080 steel, AISI 1006, AISI 1009 and AISI 1020.

The second type of plain carbon steel is medium carbon steel. It has approximately 0.30% – 0.59% carbon content. The example of this type of steel is AISI 1040 steel, AISI 1023, AISI 1030 and AISI 1046. The third type of plain carbon steel is high carbon steel. In this steel, the carbon content is about 0.60% - 0.99%. The example of this steel is AISI 1055 and AISI 1070.

Plain carbon steel is used in this project. The heat treatment which is annealing is performed to change the mechanical properties of this material. The yield strength with different pre-strain (5%, 10%, 15% and 20%) can be determined using tensile test. In the static recovery process, the grain of material structure will be recovered from cold-worked imperfections. The change of stress value before and after static recovery is related to the amount of grain recovery. The behavior of this material can be characterized by using static recovery model.

1.2 PROBLEM STATEMENTS

The problem in this project is to find the change of stress value after static recovery for samples that has been pre - strain. The stress value before and after static recovery is different because of the grain structure is recovered. To find the stress value, the specimen of AISI 4140 steel is subjected to the tensile test with pre – strain values.

1.3 PROJECT OBJECTIVE

To validate static recovery process using Friedel's model for plain carbon steel.

1.4 PROJECT SCOPES

- Specimens are subjected to tensile test pre – strain.
- Using lathe machine to shape the specimens.
- Using material plain carbon steel.
- Using box furnace to perform heat treatment.
- Varying pre – strain (5%, 10%, 15%, 20%).
- Varying recovery temperature (100^{0}C, 200^{0}C, 300^{0}C and 400^{0}C)
- Varying recovery time (1 hour, 2 hours, 3 hours and 4 hours)
- Analyze static recovery using Friedel’s model.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The purpose of this literature review is to get information about the project from many sources such as books, journals, technical papers and web sites. In this chapter, information that contained is used to compare and to get the result of the project. The source that contain in this chapter is also used as a guild to conduct the overall project.

2.2 ANNEALING

Annealing is a heat treatment process where metal is heating up to certain temperature in certain range of time [2]. The purpose of annealing process is to change the properties of the material. There are three parts of process occur in annealing as we increase the heat treatment temperature. The three regions are recovery, recrystallization and grain growth. This process that softens the cold – worked metal is called annealing and there are also term partial anneal and full anneal that refer to degree of softening [1]. An annealing operation that involves heating up to the austenite region is called a full anneal. There are also annealing that heating to a point just below the austenite transition temperature which is called partial anneal. It does not soften the steel as effectively as a full anneal. But since there is no crystal structure change, it usually produces less distortion than full anneal [2].

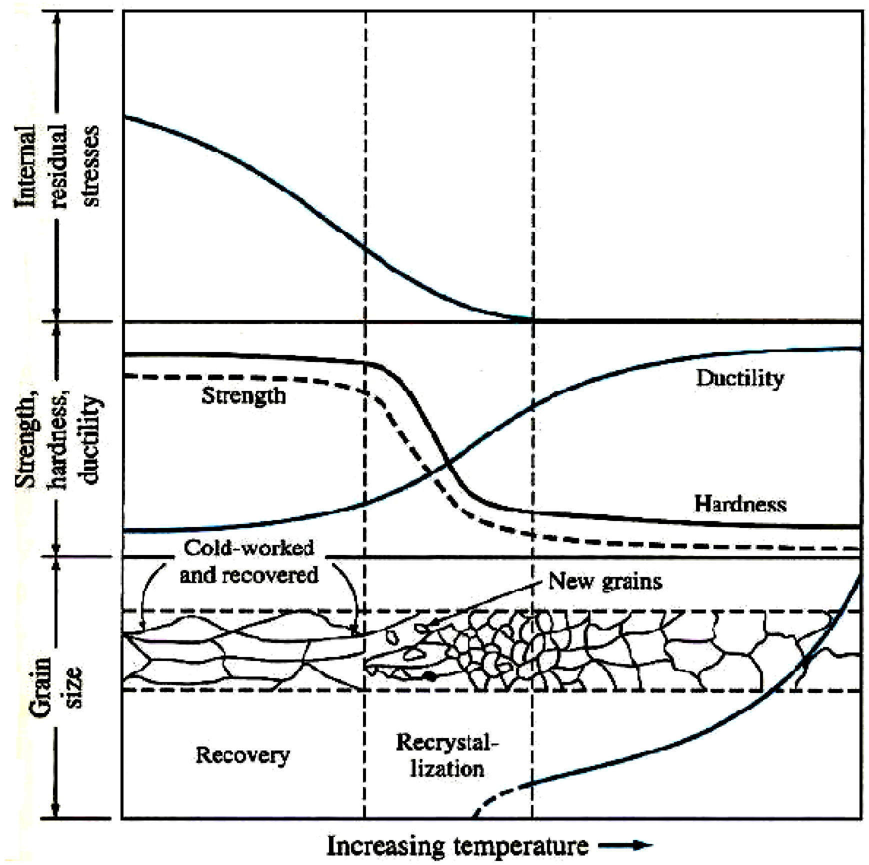

Figure 2.1: Structure and mechanical change during annealing [1]

2.2.1 Recovery

The term recovery comes from the observation that some of the physical properties of the material are recovered [3]. Recovery is the first stage in annealing process which is below the recrystallization temperature range. In recovery temperature range, internal stresses in the metal are relieved. When sufficient thermal energy is supplied, the dislocations of cold – work will rearrange themselves into lower energy configuration with low angle grain boundaries. Since many dislocations are rearrange to lower energy configuration, the internal energy of the recovered metal is lower than the cold – work metal. As the result, the strength of metal is reduced slightly and the ductility is increased significantly [1].

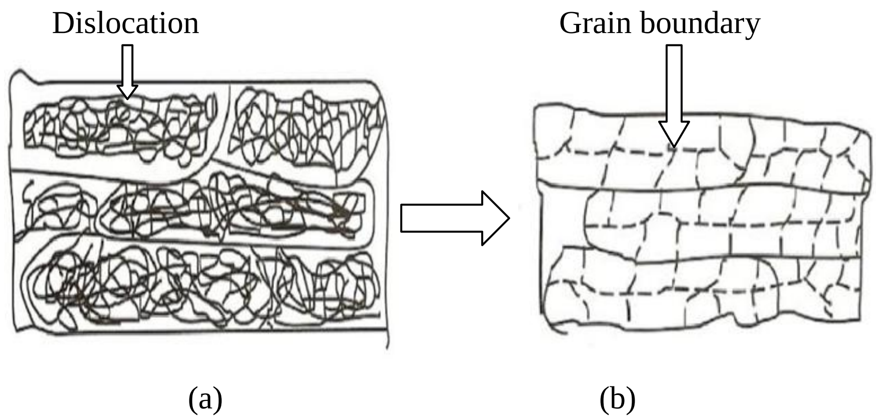

Figure 2.2: (a) Cold- work dislocation. (b) Recovered sub grains [3]

2.2.2 Recrystallization

The process of recrystallization is importance in the metal-forming process because it restores the ductility of metal which becomes hardened by plastic deformation, and controls the grain structure and the mechanical properties of the final product [5].

If we exert hammer force to the one of metal surface, the grains near the surface will become distorted and the hammered surface will plastically deform. If we wish to restore its microstructure to unstrained state, new grains must be formed and some of the metal surface must be plastically deform. As the metal is heated to its annealing temperature, it will become weaker. Then the microstructure such as dislocations and distortion part will adjust themselves to neutral and unstressed condition as explain in recovery process. By the time the temperature reach to certain point, the metal will be recrystallyzed [2]. This is the second stage of annealing process after recovery.

During the recrystallization stage, new grains are nucleated in the recovered metal and begin to grow forming recrystallized structure. After certain range of temperature, the cold – worked structure is completely replaced with recrystallization grain [1].

Recrystallization of a deformed metal is depending not only on the overall stored energy but also on microstructure of the material [5].

The grain size of new recrystallized structure is depend on the amount of cold – worked, temperature of anneal, time range and composition of material [3]. Some of the characteristics are:

I. The smaller the amount of cold – worked, the higher the temperature to cause recrystallization [3].
II. When the temperature for recrystallization is increase, the time necessary to complete it is decrease [1].
III. The greater the degree of deformation, the lower the annealing temperature for recrystallization and smaller the recrystallizaton grain size [1].
IV. The recrystallization temperature decrease with increasing purity of the metal. Solid solution of alloying addition always increases the recrystallization temperature [1].

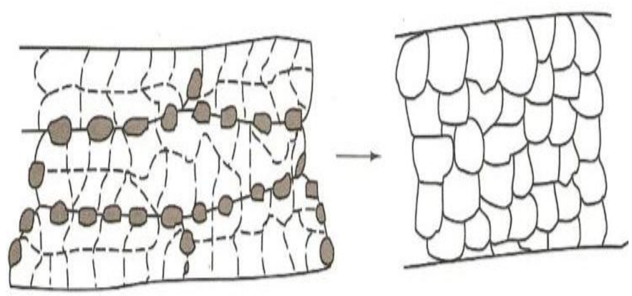

Figure 2.3: Start of recrystallization to the fully recrystallized [3]

2.2.3 Grain Growth

Grain growth starts when the recrystallization is complete. It is characterized by a gradual decrease in strength of the material to increase its grain size. When the growth occurs uniformly among all of the grains which is the average of the grains size are about the same, it is called normal grain growth. When the size some of the grain much larger than others, it called abnormal grain growth or secondary recrystallization [3]. It will result in a new grain structure with a low dislocation density. The loss by these processes of the dislocations produces mechanical softening of the material [6].

2.3 TENSILE TEST

Tensile test is used to evaluate the strength of metal and alloys. In this test, the sample is pulled with axial load until fracture occurs in a relatively short time at a constant rate.

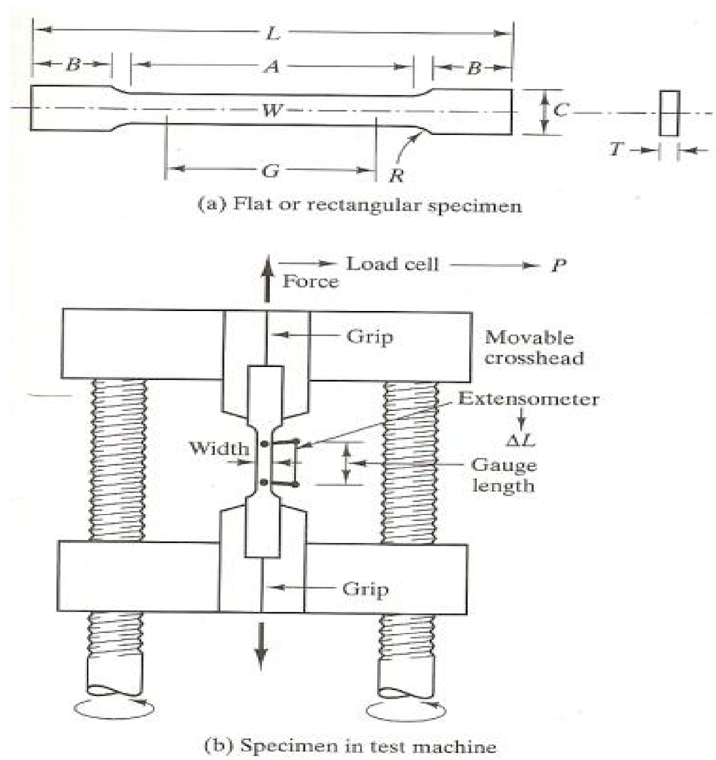

Figure 2.4: Performing tensile test[1]

Figure 2.4 shows that the process of tensile test.This test tend to pull a member apart. The tensile test machine apply a tensile load when one end of the test sample is attached to a movable crosshead with the other end fixed to stationary member. The crosshead is then pull the sample apart. The result of cross section and elongation of the sample is recorded to obtain stress and strain.

2.3.1 Stress Versus Strain

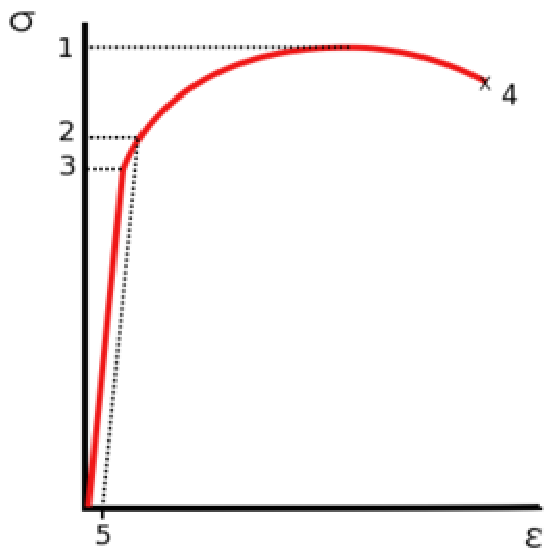

Figure 2.5: Stress versus strain diagram [8]

Figure 2.4 shows the result stresses versus strain after performing tensile test where:

1. Ultimate Strength
2. Yield strength
3. Proportional Limit Stress
4. Tensile strength
5. Offset Strain (typically 0.002).

This graph is very important in this project where the value of stress before and after recovery process can be determined. In addition, the maximum tensile strength is also can be determined which is used to analyze the degree of recovery.

2.4 PLAIN CARBON STEEL

2.4.1 Composition

Plain carbon steel do not exceed following limit:

Table 2.1: Plain carbon steel composition[3]

Element	C	Cu	Mn	P	Si
Composition (%)	<1.0	0.60	1.65	0.4	0.6

2.4.2 Mechanical Properties

Steel can be used for heat treatment which allows the material to easily forming and shaping. If the carbon content present is enough, the material can be hardened to increase its strength, wear resistance and impact resistance. The application of plain carbon steel is widely use in the industries. The common usage of this material is for building construction where the core structure of the building is made from plain carbon steel. This can be seen at the beam of the building and also the for the road structure.

Table 2.2: Plain carbon steel mechanical properties[3]

Properties	Value
Density (×1000 kg/m^3)	7.861
Elastic Modulus (GPa)	210
Tensile Strength (Mpa)	500
Melting point (^{0}C)	1426-1538
Recrystallization temperature (^{0}C)	725

2.4.3 Heat Treatment of Carbon Steel

Heat treatment of steel such as fully annealed will result in soft and ductile with no internal stresses. Carbon steel is usually heat up to 550^0C – 650^0C for 1 hour. Sometimes, this temperature is increased to 700^0C.

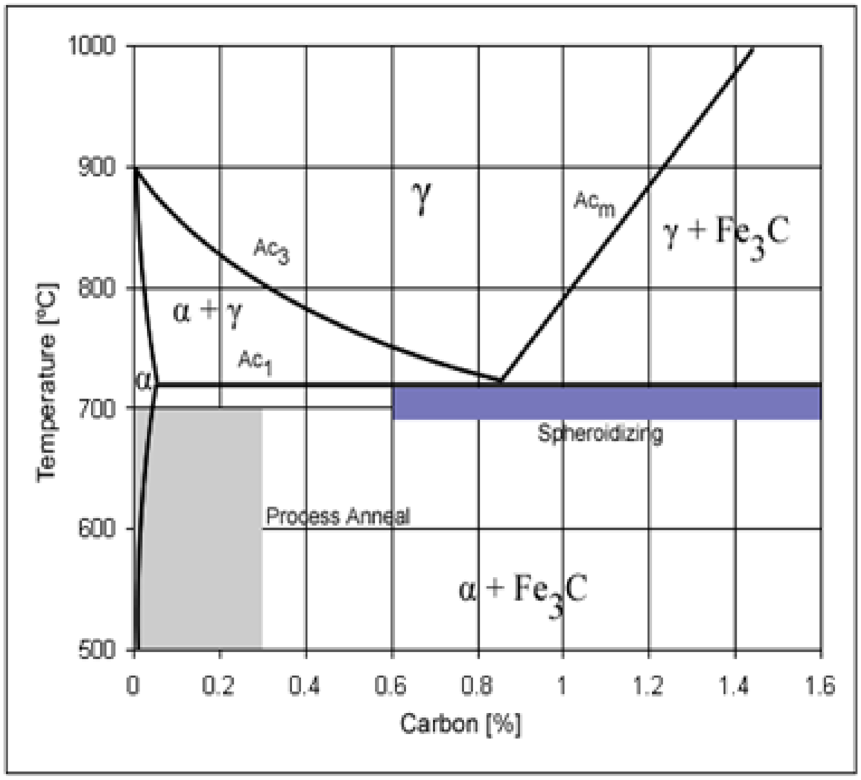

Figure 2.6: Heat treatment temperature with increasing carbon content[2]

2.5 FRIEDEL'S MODEL

Friedel's model can be used in modeling equation for static recovery process where the characteristic of the material before and after static recovery can be known.

$$X = \frac{\sigma_m - \sigma_r}{\sigma_m - \sigma_o} \tag{2.1}$$

Equation (2.1) shows that the fraction of static recovery (X) is define in terms of the yield stress of the static recovery (σ_r), the maximum yield stress of deformed specimen (σ_m) and the yield stress of the undeformed specimen (σ_0) [4].

$$X = c\ln t - \frac{Q}{RT} \qquad (2.2)$$

The relationship between the amount of recovery, time and temperature can be calculated using equation (2.4) where (Q) is activation energy, (T) is temperature, (R) is gas constant, (t) is time and (c) is constant [4]. This equation is based on the figure 2.7 as shown below. The stress values before and after static recovery were taken. From these values, there were change occurred to the stress of the specimen after recovery. This mean that the dislocations in the structure was were reduced.

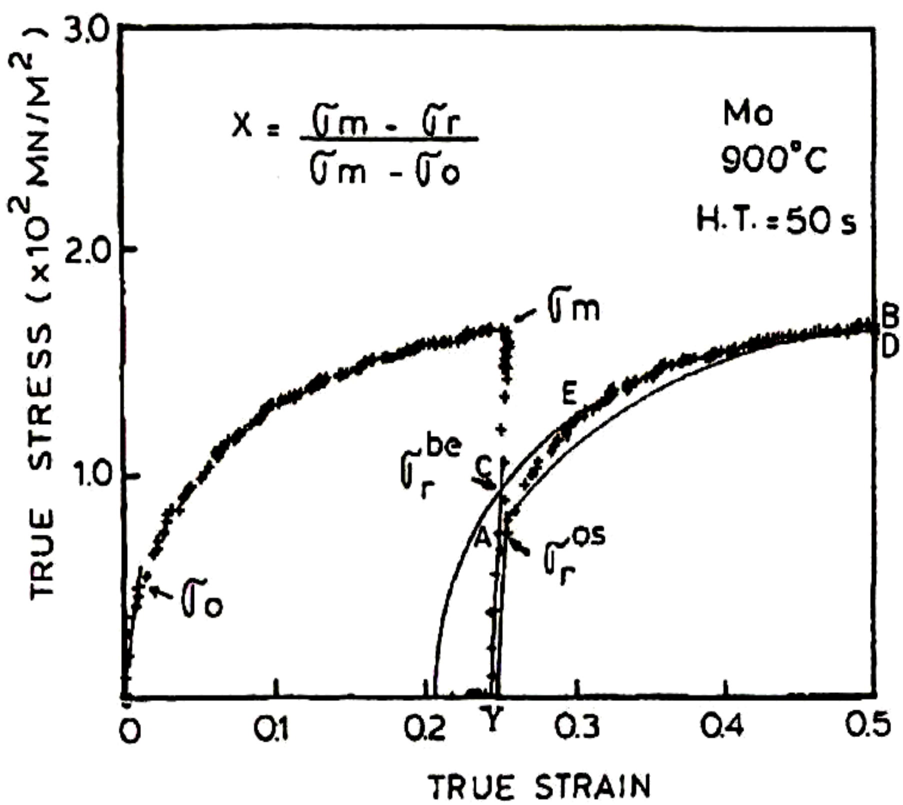

Figure 2.7: Determination of reloading flow stress and degree of softening by the back extrapolation (be) and offset (os) [7].

2.6 EFFECT OF TEMPERATURE ON RECOVERY KINETICS

For every recovery temperature, the stress value decreases with increasing time. The effect of increasing temperature on the recovery kinetic has two stages. Firstly the initial stress will decrease and secondly the stress relaxation rate will increase. This effect is due to a larger degree of recovery during deformation. The other effect is because of the motion of dislocation in the structure that occurs by thermally activated mechanism. This is due only to recovery stage, since there is no distinctive three stages curve observed which is the recrystallization occurs [8].

Figure 2.8 shows the dislocation density evolution versus time increment. The stress relaxation curve that shown in the figure is the series of temperature which are 450^{o}C, 500^{o}C, 550^{o}C and 600^{o}C. In this curve, the strain is 0.15 and the strain rate is 0.1 s^{-1}. The best fit for each temperature data was marked with the solid lines as shown in the figure. It can be seen that the dislocation density generally decreases with increasing temperature. The internal stress was found to decrease as recovery proceeds where the overall dislocation density was reduced. Stress due to dislocation, (σ_d) were calculated by equation (2.3). During the early stages of recovery, the dislocation density is higher than the lower temperature. This can cause the error in determining $\sigma_{d.}$ Although this error occurs, the value of G in the equation does not as the effect of composition.

$$\sigma_d = M\alpha G\sqrt{\rho} \qquad (2.3)$$

Where:

M = 2 for BCC metal

α= Constant

G= Shear modulus

b= Burgers vector

ρ= Dislocation densities

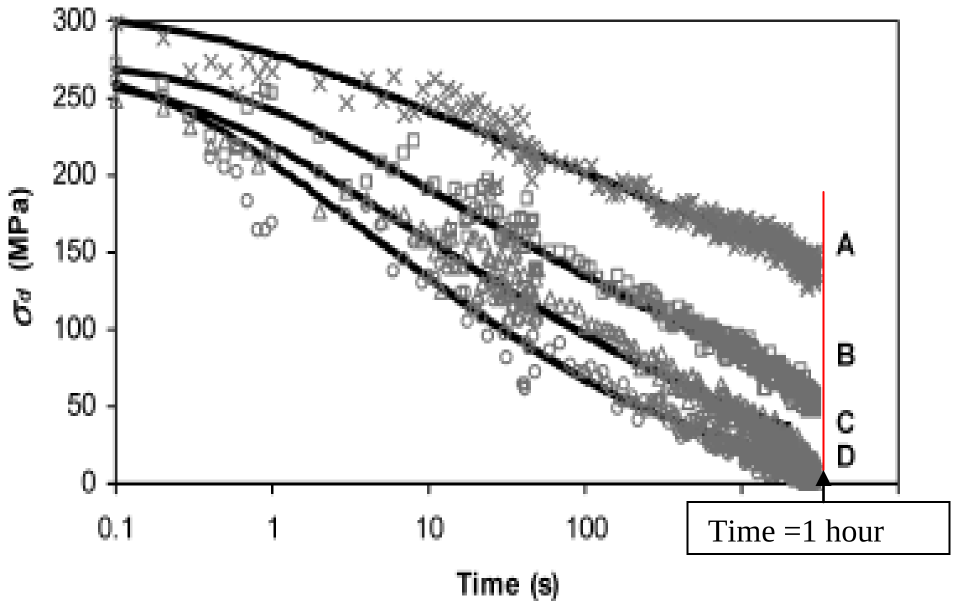

Figure 2.8: Stress relaxation curve. A= 450°C, B= 500°C, C= 550°C, D= 600°C [8]

From figure 2.8, we can see that the stress was decrease with increasing recovery time. When the stress is decrease, that mean the structure is softening. This will result the recovery that occur in the structure is increase.

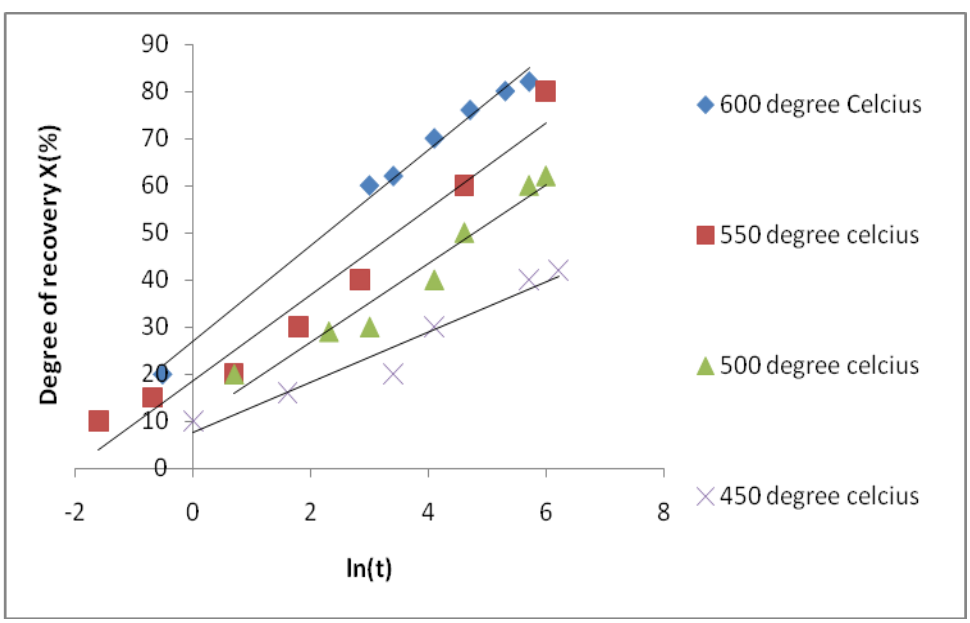

Figure 2.9: Graph degree of recovery versus ln(t) for various temperature

The graph in figure 2.9 shows the degree of recovery versus ln(t) for various temperature. This is the replotted graph which is the data taken from graph in figure 2.8. The degree of recovery is calculated by equation (2.1) where σ_m is value σ_d when time approximately zero and σ_o is zero.

To make the comparison of activation energy for 1 hour in the experiment result, reverse calculation from curve in figure 2.8 is done. This calculation is made by using Friedel's model equation (2.1) and (2.2). The data was taken in the graph from figure 2.8 for each temperature within 1 hour. Then the data was plotted into graph degree of recovery versus 1/temperature as shown below.

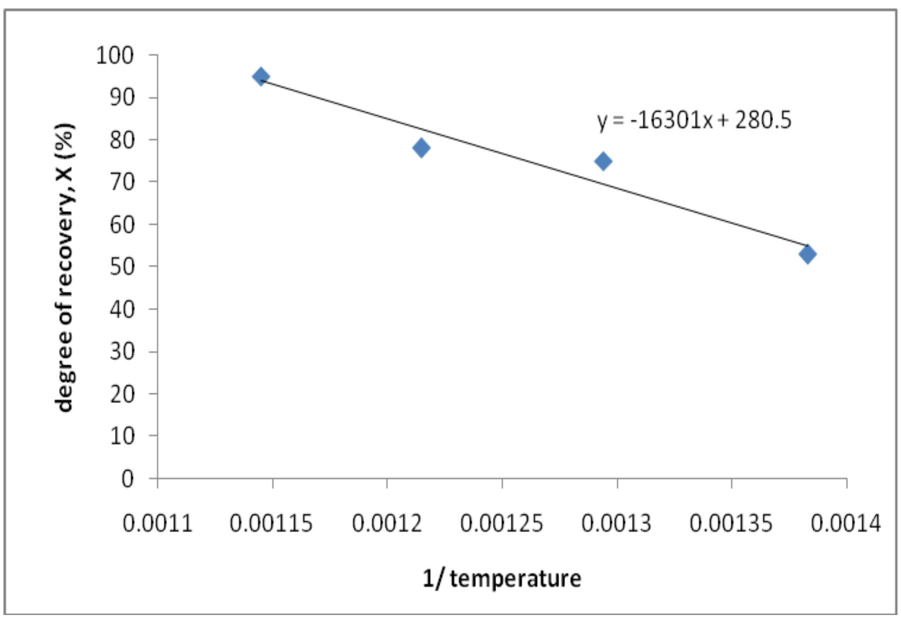

Figure 2.10: Graph degree of recovery versus 1/temperature with 1 hour recovery time

After the graph was plotted as in figure 2.10, the linear equation was obtained which is:

$$y = -16301x + 280.5 \qquad (2.4)$$

This equation was compared to Friedel's model equation and the activation energy was calculated and the value is 136 kJ/mol.

2.7 ACTIVATION ENERGY FOR PLAIN CARBON STEEL

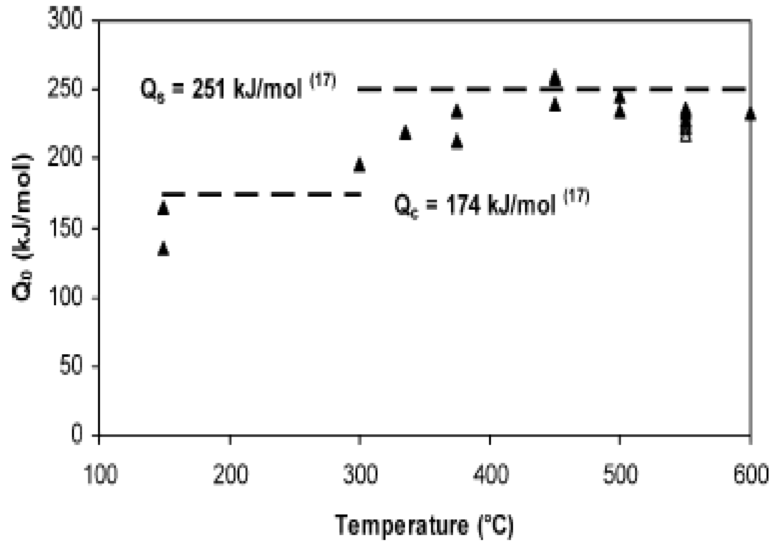

Figure 2.11: Effect of temperature energy on activation energy for recovery[8]

From figure 2.11, the activation energy is increase until the temperature is 450°C. After 450°C, the value of activation energy is approximately constant. When the temperature range is between 150 °C and 300 °C, the activation energy value is close to dislocation core diffusion which is 174 kJ/mol. For temperature range between 300 °C and 500 °C, the activation energy value is in lattice self diffusion which is 251kJ/mol. In this graph, the strain value was fixed to 0.15 and the strain rate is 0.1^{-1}. The previous study suggested that there was no activation energy for lattice self-diffusion. However, the analysis showed that for temperature 300°C, 335 °C and 375 °C, there were contribution of dislocation reduction that observed due to activation energy. [10]

2.7.1 Activation Volume for Recovery

Figure 2.11 below shows the effect of temperature on activation volume which is plotted by activation volume versus temperature. . In this graph, the strain value was fixed to 0.15 and the strain rate is 0.1^{-1}

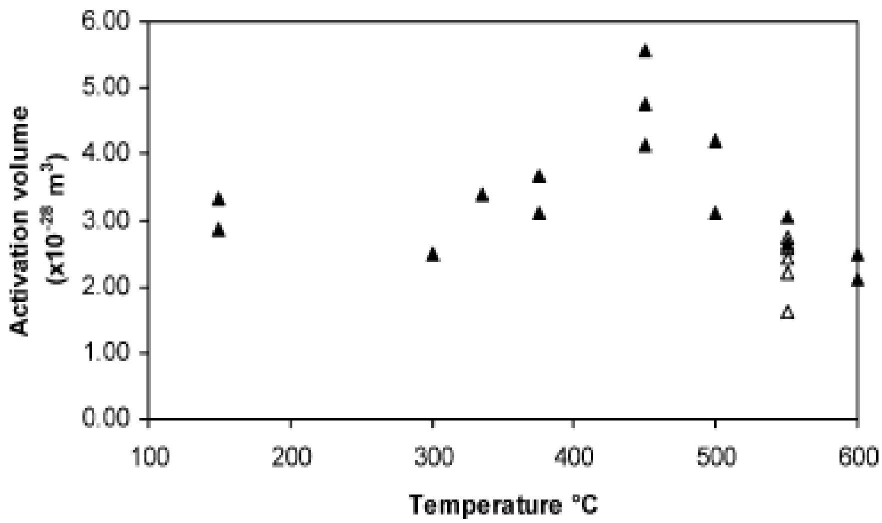

Figure 2.12: Effect of temperature on activation volume[8]

From figure 2.12, the value of activation volume is approximately constant at $3x10^{28}m^3$ between temperature 150 ^oC and 375 ^oC. The activation volume is increase above 375 ^oC until 450 ^oC. Above 450 ^oC, the activation volume value is decreasing with increasing temperature.[8]

2.7.2 Yield Stress Kinetic Evolution

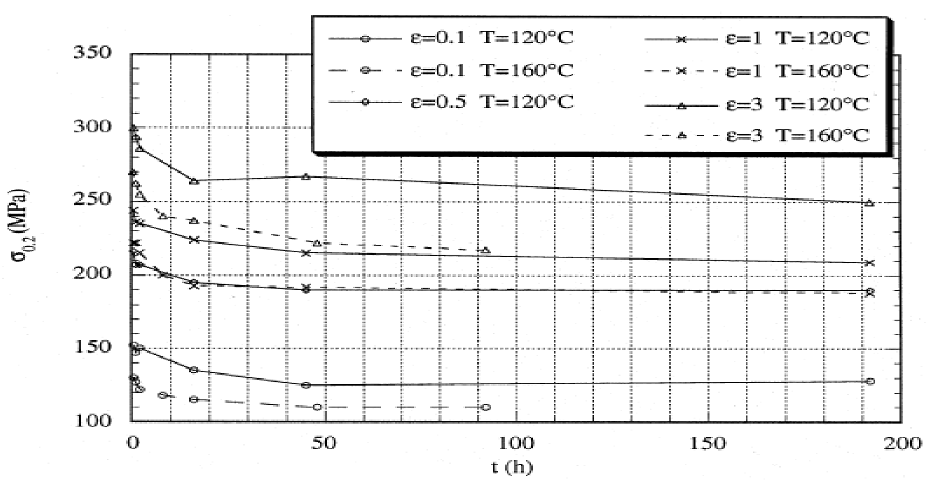

Figure 2.13: Yield stress kinetic evolution for the (1070+ 2.5Mg) alloy recovered at 120^oC and 160 ^oC.[9]

Figure 2.13 shows that yield stress kinetic evolution for the (1070+ 2.5Mg) alloy versus time. This material is recovered at 120^{o}C and 160 ^{o}C with difference pre-strain. The rate of yield stress is decrease continuously at both temperatures. The recrystallization starts when the temperature is higher than 200 and pre-strain larger than 10%. From this figure, the dislocation structure is occurs at low pre-strain.[9]

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, a structure of overall methodology has been planned to achieve the goal of this project. The methodology of this project is important to make sure that the processes of the project are done completely and according to schedule. Several experiments had been made to complete this project. There are main steps had been done which are:

1. Literature review
2. Machining specimens
3. Annealing
4. Pre – strain
5. Static recovery
6. Modeling equation

These steps can be referred to the figure 3.1 which is the flowchart for overall project. In this project, 36 experiments had been conducted to test every specimen for all variables. Each variable was subjected to 12 experiments. Three specimens had been test for the same variable value. This is done to find the major spot when graphs were plotted. This is because the stress value for a specimen was not the exact value due to the error occurred during the experiment proceeding.

3.2 FLOW CHART

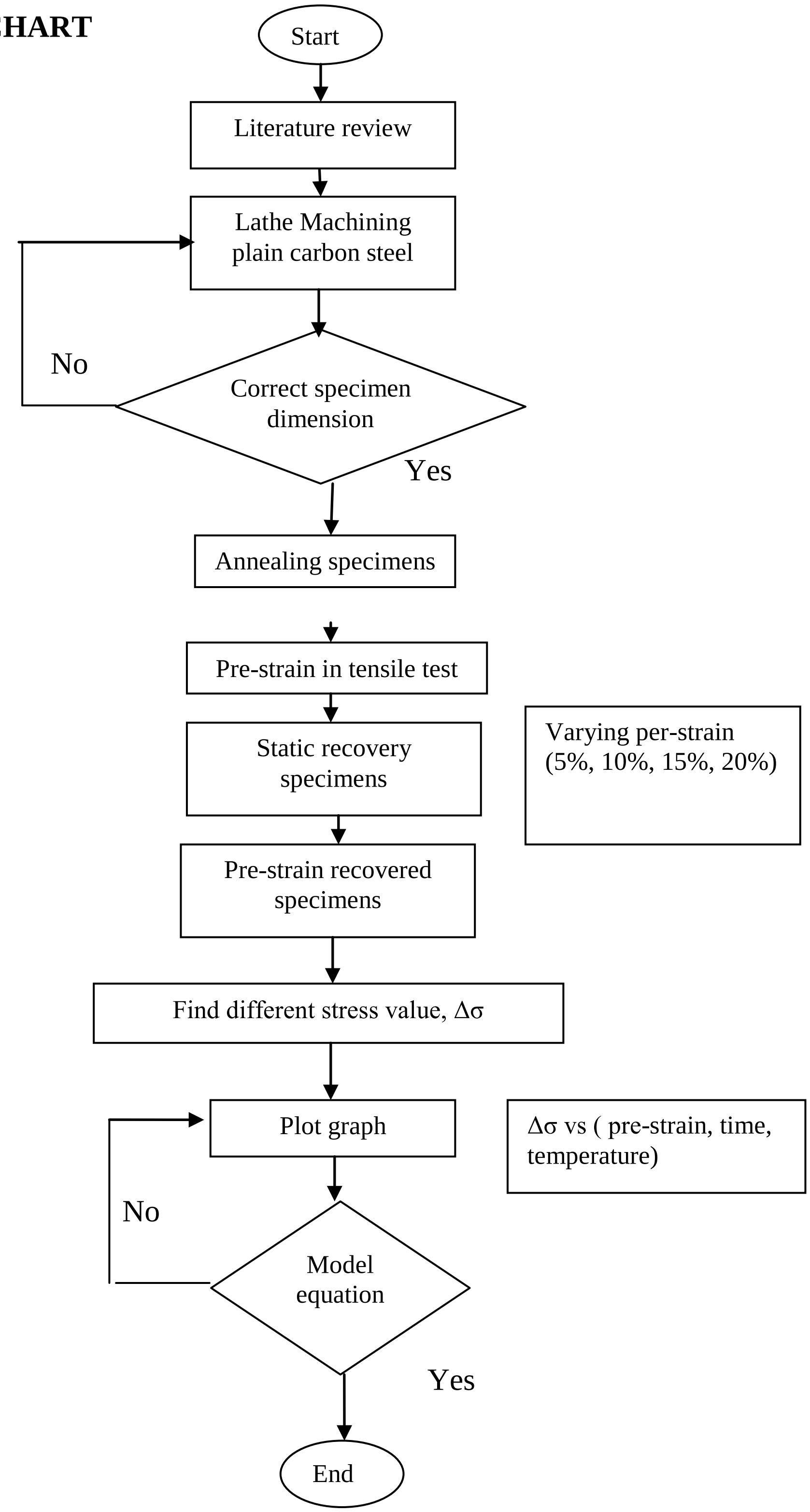

Figure 3.1: Flow chart of overall FYP

3.3 FINDING LITERATURE REVIEW

The main purpose of this literature review is to study and research about the project. The sources of the research were referred to the books, journals, technical paper and web sites. First of all, the title of this project must be understood very well so that related information can be found. Most of the sources are from journal. This is because the result from this project is compared to the journals to validate the data and result. The other reviews are studied to get the information about the material properties such as annealing temperature, recrystallization temperature and recovery temperature. The most research in this project was about Friedel's model equation. This is because this method that purposed by Friedel was about relationship between degree of recovery with the temperature, time and activation energy.

3.4 MACHINING SPECIMENS

After all the informations about the project are studied, the specimens are started to be made. The specimens that needed in this project are tensile test specimen which is according to ASTM E8. There are 50 specimens are made for this project. The overall length of this specimen is 160mm. The dimension of the specimen is shows in figure 3.2 below.

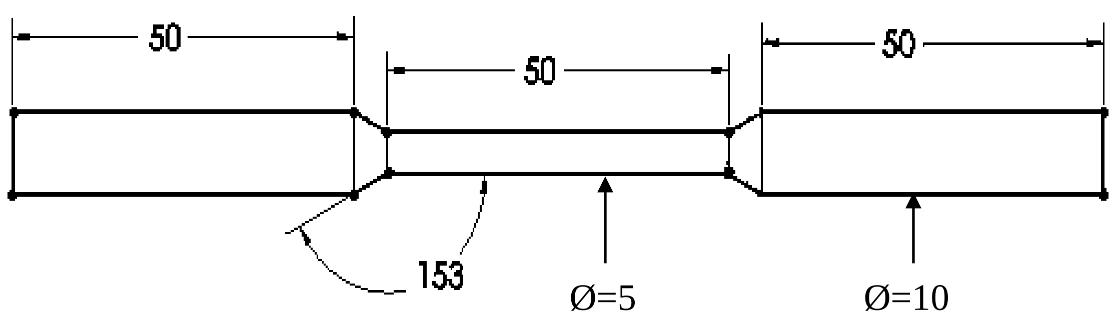

Figure 3.2: Specimen dimension in (mm)

To make the specimen as showed in figure 3.2, the material (plain carbon steel) is undergo several machining processes.

3.4.1 Bandsaw

The plain carbon steel material is come in cylinder shape with long dimension (length) from the manufacturer. In order to make it shorter with desire dimension, bendsaw machine is used to cut the material. The material is cut slight longer than 160mm length (+ 3mm tolerance) because the end surface needs to be facing with lathe machine.

3.4.2 Lathe

This machine is used in machining processes with the capability to producing part that needed in this project basically in round shape. These processes are performed by turning the work piece in the lathe jaw. Turning means that the work piece is rotating while it is machined. The following processes are used to produce specimen in this project using lathe machine:

1. Centre drill
   - Centre drill was applied at both end surface of the work piece. It is done because the work piece is needed to be hold at the end so it did not bend when making the turning process.

2. Turning
   - Turning process is used to produce straight, conical, curved or grooved work piece. In this project, it was applied to make the diameter of the specimen 10mm and 5mm (the middle section).

   - This process is also used to make the angle part as showed in figure 3.2. The tool bit angle was set to 30^{o} to make this specimen.

3. Facing
    - This process is used to produce a flat surface at the end of the work piece.
    - In this project, it was used to flatten both end of the specimen so the length is 160mm.

Figure 3.3: Lathe machine

Figure 3.4: Tensile test specimen

3.5 ANNEALING PROCESS

After the specimens are made, the next step is to do the annealing process. All of 50 specimens are subjected to this process. Annealing process is used to relieve the residual stress in the specimen structure because of the machining process. In this project, the annealing process was conducted in the box furnace with the data below:

- Annealing temperature = 725 ^oC
- Soaking time = 1 hour
- Heating rate = 5 ^oC per minute
- Cooling rate = 1.28^oC per minute
- Heating time (min) $= \frac{725 - ambient\ temperature}{5}$ (3.1)

Figure 3.5: Box furnace used to perform annealing and recovery process

3.5.1 Precaution Step of Box Furnace

Precaution step while conduct the box furnace is very important because it can produce temperature up to 3000 ^{o}C. To avoid accident happen because of the danger, several precaution steps are taken such as:

- Do not assume the furnace is cold
- Do not open the furnace while the temperature inside is high
- Measure the temperature first before using the furnace

3.6 PRE – STRAIN THE SPECIMENS

After all of the specimens were undergo annealing process, the next step is to pre – strain the specimens. This process is done by using the tensile test machine. The purpose of this process is to get the yield strength and maximum strength of the specimen. In this project, the pre – strain was made by varying its value to 5%, 10%, 15% and 20%. The maximum load that can apply by tensile test machine is 50kN. Each of the pre – strain value is using 3 specimens. This is because the error will occur, so the average value will be considered.

Figure 3.6: Tensile test machine

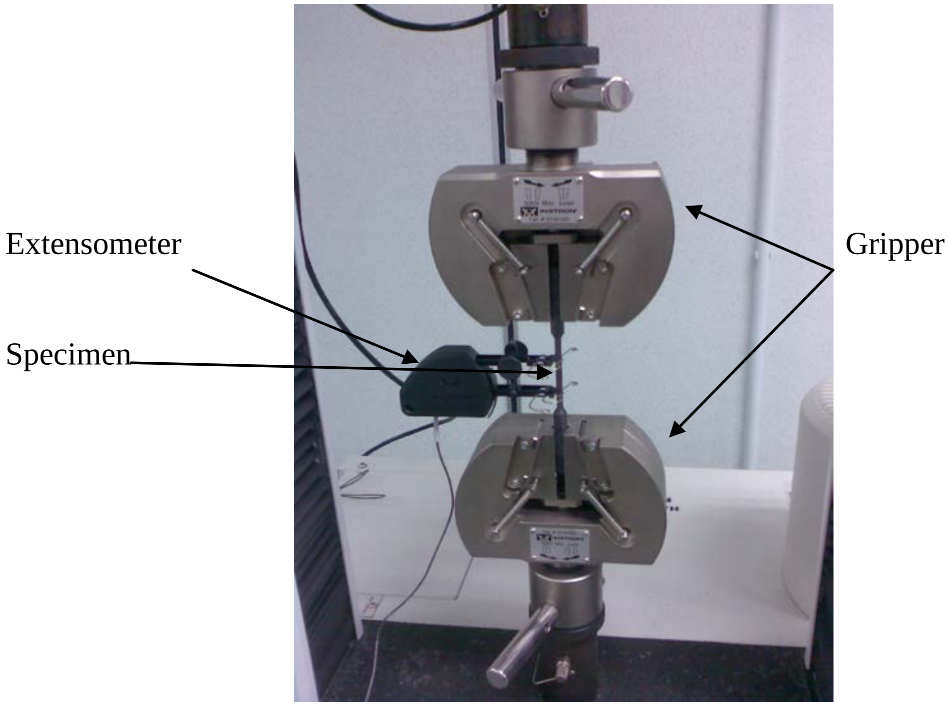

Figure 3.7: Performing tensile test machine

3.7 RECOVERY PROCESS

Recovery process is the important process in this project. This is because the process is needed to find the degree of recovery, *X*. In this project, the recovery process which is varying with pre – strain, temperature and time. In this project, box furnace was used to performed static recovery process. The static recovery temperature was selected before the recyistallization temperature. This is because, the research was only in the recovery region. For every variable, there are 12 specimens subjected to recovery process. The properties for each variable are conducted as follow:

1. Varying pre – strain:
    - Varying pre – strain to 5%, 10%, 15% and 20%
    - Fixed recovery temperature to 300^{o}C
    - Fixed recovery time to 1 hour

- Heating rate =5^{o}C per minute

2. Different temperature:
   - Varying temperature to 100 ^{o}C, 200 ^{o}C, 300 ^{o}C, and 400 ^{o}C.
   - Fixed pre –strain 10%.
   - Fixed recovery time to 1 hour.
   - Heating rate = 5 ^{o}C per minute

3. Different time:
   - Varying recovery time to 1 hour, 2 hour, 3 hour and 4 hour.
   - Fixed pre –strain 5 %
   - Fixed recovery temperature 300 ^{o}C.
   - Heating rate = 5 ^{o}C per minute

3.8 PRE – STRAIN RECOVERED SPECIMENS

The next step after recovery process is pre –strain again the specimens that had been recovered. The purpose of this process is to get the new yield strength which is the recovered yield strength. From the studied, the value of stress after recovery was changed compared to the value before recovery took place. The new yield strength or the recovery yield strength values had been recorded using the tensile test machine. The new stress was used to calculate the different stress value between before and after recovery process. This is also important to calculate the degree of recovery. The new recovered pre – strain values were the same with the value before recovered. This different of stress value is represent the amount of recovery that occurs in the specimen.

3.9 ANALYZING DATA

With all data that had been collected such as yield strength, recovery yield strength, time and temperature, the next step is to analyze all of these data. This is done by arranging the data in tables. In order to analyze the data, the degree of recovery, X_{rec} is calculated by using equation (2.1) for every specimen.

3.9.1 Plotting Graph

The graph was plotted to find the relationship between the variables and the degree of recovery. The graph also plotted to obtain the linear equation which is used to calculate the activation energy. The graph plotted for:

1. Degree of recovery versus pre – strain
2. Degree of recovery versus temperature
3. Degree of recovery versus time
4. Degree of recovery versus 1/ temperature
5. Degree of recovery versus ln(t)

The graph degree of recovery versus pre – strain, degree of recovery versus temperature and degree of recovery versus time were used to analyze the relationship between degree of recovery and these variables. While the other two graphs were used to calculated the activation energy.

3.9.2 Applying Friedel's Model

In this project, Friedel's model is applied to the data by using equation (2.2). The purpose of this calculation is to get the activation energy value, ***Q*.** This is done by comparing the linear equation that obtained from the graph degree of recovery versus 1/temperature and degree of recovery versus ln(t) to the Friedel's equation. From this value, the activation energy for recovery temperature and time can be found.

3.9.3 Result Comparison

The purpose of result comparison is to validate the result of this project. To make the comparison, several journals had been studied and research to prove the project's result. The value that had been comparing is activation energy together with relationship between degree of recovery, temperature and time.

3.10 Documentation

After finish all the test and result, all documentation regarding to result will be finished as a thesis for reference in the future.

CHAPTER 4

RESULT AND DISCUSSIONS

4.1 INTRODUCTION

All the things about this chapter are the findings that got from the experiment explained in the previous chapter. This chapter showed the result and data from the experiment that need to achieve the project's objective. The data is including the stress before and after recovery process for every specimen that had been tested. The activation energy also calculated in this chapter and compared to the value from other journal. All of the data will be calculated and discussed in order to valid it as the sources that found in the literature review. This is the important part to achieve the project's objective. The graphs will be plotted to obtain the relationship between the variables that that stated in the project's scope. The graphs also used to obtain the linear equations which are important to calculate the activation energy. The data will be analyzed through the discussion, so the last part of the project, it can be concluded and any recommendation can be suggested for further improvement.

4.2 RECOVERED STRESS BEFORE AND AFTER RECOVERY PROCESS

Recovery process is the most important process in this project. In order to analyze the recovery process, the way is finding the stress before and after recovery process. This has been done in the tensile test experiment. All of 50 specimens were tested in the tensile test before and after static recovery process. This is because the value obtained before and after static recovery was different. The difference value of the stress will represent the amount of recovery occurs in the material structure.

4.2.1 Varying Pre - Strain

Varying pre-strain was one of the project's scopes that need to be analyzed. The pre-strain was varying 5%, 10%, 15% and 20%. These pre – strain values were decided after the full tensile of plain carbon steel specimen was tested. The specimen of this material was test until fracture to find the maximum pre-strain that plain carbon steel can stand. After tested, the maximum pre – strain of plain carbon steel was 40%. So, the pre – strain was choose below 40% in order to be able the analysis. In this experiment, the temperature was fixed to 300^{o}C and recovery time fixed to 1 hour. Three specimens were tested for each pre – strain. The result which are maximum tensile strength σ_m , undeformed tensile stress σ_0 and recovery tensile stress σ_r were obtained in table below:

Table 4.1: Data for 5% pre-strain (1 hour, 300^{o}C)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	154.6	210.6	263.6
2	130.4	206.3	275.2
3	108.4	185.4	252.4

Table 4.2: Data for 10% pre-strain (1 hour, 300^{o}C)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	136.5	252.1	317
2	134.5	258.1	327.1
3	130.4	245.8	325.5

Table 4.3: Data for 15% pre-strain (1 hour, 300°C)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	140.4	245.8	625.5
2	142.4	268.9	345.6
3	141	261.7	346.2

Table 4.4: Data for 20% pre-strain (1 hour, 300°C)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	141.0	264.7	546.2
2	141.7	258	362.6
3	151.7	263.3	389.9

4.2.2 Varying Recovery Temperature

In this project, varying recovery temperature also made to analyze the effect in static recovery. This is done to find the relationship between temperature and static recovery process. The selecting of temperature was important in this process. This is to make sure the temperature range is in the recovery temperature range which is below the recrystallization temperature. For plain carbon steel, the recrystallization temperature was 725°C. To analyze the static recovery process, the temperature range was set to 100°C, 200°C, 300°C, and 400°C. In this experiment, the pre – strain was fixed to 10% and recovery time was 1 hour.

The same technique in the varying pre - strain was used in this experiment to analyze the static recovery process which is finding the stress value before and after static recovery. After 12 specimens were tested, the result obtained as in table below:

Table 4.5: Data for temperature 100^{o}C (1 hour, 10% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	149.2	253	308
2	132.8	240.9	307.9
3	133.1	242.3	501.4

Table 4.6: Data for temperature 200^{o}C (1 hour, 10% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	132.8	242.9	507.9
2	123.1	242.3	301.4
3	130.4	244.1	298.7

Table 4.7: Data for temperature 300^{o}C (1 hour, 10% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	157.5	267.2	322
2	131	249.4	320.5
3	143.4	262.6	322.2

Table 4.8: Data for temperature 400^{o}C (1 hour, 10% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	123.4	242.6	424.2
2	124.6	233.9	266.7
3	151.4	249.9	287.3

4.2.3 Varying Recovery Time

The third variable need to be analyzed is the recovery time. The heating time was set differently which is the soaking time of the box furnace. The rate of temperature from ambient temperature to static recovery temperature was fixed to 5^{o}C/min. All of these specimens were cooled slowly in the furnace. There were 12 specimens used in this experiment which is 3 specimens for every recovery time. To analyze the different of time, the period of soaking time was varying to 1 hour, 2 hours, 3 hours and 4 hours. These ranges of time were selected to make sure the specimens did not enter the recrystallization region.

The recovery temperature was fixed to 300^{o}C. The pre – strain before and after heating was also fixed to 5%. After all of these specimens undergo static recovery process; they were subjected to pre – strain again. The stress value before and after static recovery obtained as in the tables below:

Table 4.9: Data for recovery time 1 hour (300^{o}C, 5% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	159.4	231.2	278
2	138.4	202.8	247.9
3	118.4	193.1	250.6

Table 4.10: Data for recovery time 2 hours (300^{o}C, 5% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	148.4	203.1	450.6
2	140.6	207.4	250.8
3	132.5	204.4	247.5

Table 4.11: Data for recovery time 3 hours (300^{o}C, 5% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	146.1	199.9	242.4
2	117.1	199.8	252.7
3	133.4	205.4	253.1

Table 4.12: Data for recovery time 4 hours (300^{o}C, 5% pre-strain)

Specimen	σ_0 (Mpa)	σ_m (Mpa)	σ_r (Mpa)
1	143.4	215.4	453.1
2	125.3	193.8	259.1
3	172.7	232.1	270.7

4.3 DEGREE OF RECOVERY CALCULATION

The degree of recovery was calculated using the Friedel's model as showed in the literature review's chapter. Degree of recovery was used to represent the amount of recovery occurs in the metal structure. Each of the specimens that had been tested was calculated by using the data obtained. According to Friedel's model, the degree of recovery can be calculated by using the equation (2.1) which is:

$$X_{rec} = \frac{\sigma_m - \sigma_r}{\sigma_m - \sigma_o} \tag{2.1}$$

Where :

- X_{rec} = Degree of recovery
- σ_m = maximum tensile strength
- σ_0 = undeformed tensile stress

- σ_r = recovery tensile stress

By using the data obtained which is the stress value before and after recovery, the degree of recovery was calculated as show below:

Sample of calculation for varying pre –stain data 5%:

$$X_{rec} = \frac{\sigma_m - \sigma_r}{\sigma_m - \sigma_o}$$

$$X_{rec} = \frac{(210.6 - 263.6)}{(210.6 - 154.6)}$$

$$X_{rec} = -0.95$$

4.3.1 Degree of Recovery for Varying Pre – Strain

From the sample calculation above, the degree of recovery for 5% pre – strain with 300^{o}C recovery temperature and 1 hour recovery time was – 0.95. If the value changed to percentage, the degree of recovery was 0.95%. This mean that the recovery occurs in this temperature (300^{o}C) and time (1 hour) was 0.95%. The degree of recovery for all of the specimens were calculated in order to find the relationship between amount of recovery with all variables. For the rest of pre - strain data calculation, the result was obtained in the table below:

Table 4.13: Degree of recovery for pre – strain 5% (1 hour, 300^{o}C)

Specimen	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_0$	X_{rec}
1	-53	56	-0.95
2	-68.9	75.9	-0.91
3	-67	77	-0.87

Table 4.14: Degree of recovery for pre – strain 10% (1 hour, 300°C)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-64.9	115.6	-0.56
2	-69	123.6	-0.56
3	-79.7	115.4	-0.69

Table 4.15: Degree of recovery for pre – strain 15% (1 hour, 300°C)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-379.7	105.4	-0.28
2	-75.9	126.5	-0.6
3	-84.5	120.7	-0.7

Table 4.16: Degree of recovery for pre – strain 20% (1 hour, 300°C)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-281.5	123.7	-2.28
2	-104.6	116.3	-0.9
3	-126.6	111.6	-1.1

4.3.2 Degree of Recovery for Varying Temperature

The calculation also had been made for varying temperature data. The calculation of degree of recovery was made using the Friedal's model as before. The result of calculation was obtained as table below:

Table 4.17: Degree of recovery for recovery temperature 100^{o}C (1 hour, 10% pre-strain)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-55	103.8	-0.53
2	-67	108.1	-0.62
3	-259.1	109.2	-2.37

Table 4.18: Degree of recovery for recovery temperature 200^{o}C (1 hour, 10% pre-strain)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-267	108.1	-2.47
2	-59.1	119.2	-0.5
3	-54.6	113.7	-0.48

Table 4.19: Degree of recovery for recovery temperature 300^{o}C (1 hour, 10% pre-strain)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-54.8	109.7	-0.5
2	-71.1	118.4	-0.6
3	-59.6	119.2	-0.5

Table 4.20: Degree of recovery for recovery temperature 400^{o}C (1 hour, 10% pre-strain)

Specimen	$\sigma_m - \sigma_r$	σ_m - σ_0	X_{rec}
1	-181.8	119.2	-1.53
2	-32.8	109.3	-0.30
3	-37.4	98.5	-0.38

4.3.3 Degree of Recovery for Varying Time

Table 4.21: Degree of recovery for recovery time 1 hour (300^{o}C, 5% pre-strain)

Specimen	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_0$	X_{rec}
1	-46.8	71.8	-0.65
2	-45.1	64.4	-0.70
3	-57.5	74.7	-0.77

Table 4.22: Degree of recovery for recovery time 2 hour (300^{o}C, 5% pre-strain)

Specimen	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_0$	X_{rec}
1	-247.5	54.7	-4.52
2	-43.4	66.8	-0.65
3	-43.1	71.9	-0.60

Table 4.23: Degree of recovery for recovery time 3 hours (300^{o}C, 5% pre-strain)

Specimen	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_0$	X_{rec}
1	-42.5	53.8	-0.79
2	-52.9	82.7	-0.64
3	-47.7	72	-0.66

Table 4.24: Degree of recovery for recovery time 4 hours (300^{o}C, 5% pre-strain)

Specimen	$\sigma_m - \sigma_r$	$\sigma_m - \sigma_0$	X_{rec}
1	-237.7	72	-0.66
2	-65.3	68.5	-0.95
3	-38.6	59.4	-0.65

4.4 GRAPH RESULT

Graph was the important in analyzing the data. In this project, graph was used to find the relationship between the variables and the degree of recovery. The graph was plotted by using the data that valid in degree of recovery value. For every variable value which is consist of three points, at least two points were taken if one of them was seem to be wrong compare to other points. There were three graphs that used to analyze the relationship of those variables with the degree of recovery which are:

- Degree of recovery versus pre-strain
- Degree of recovery versus temperature
- Degree of recovery versus time
-

4.4.1 Degree of Recovery Versus Pre-Strain

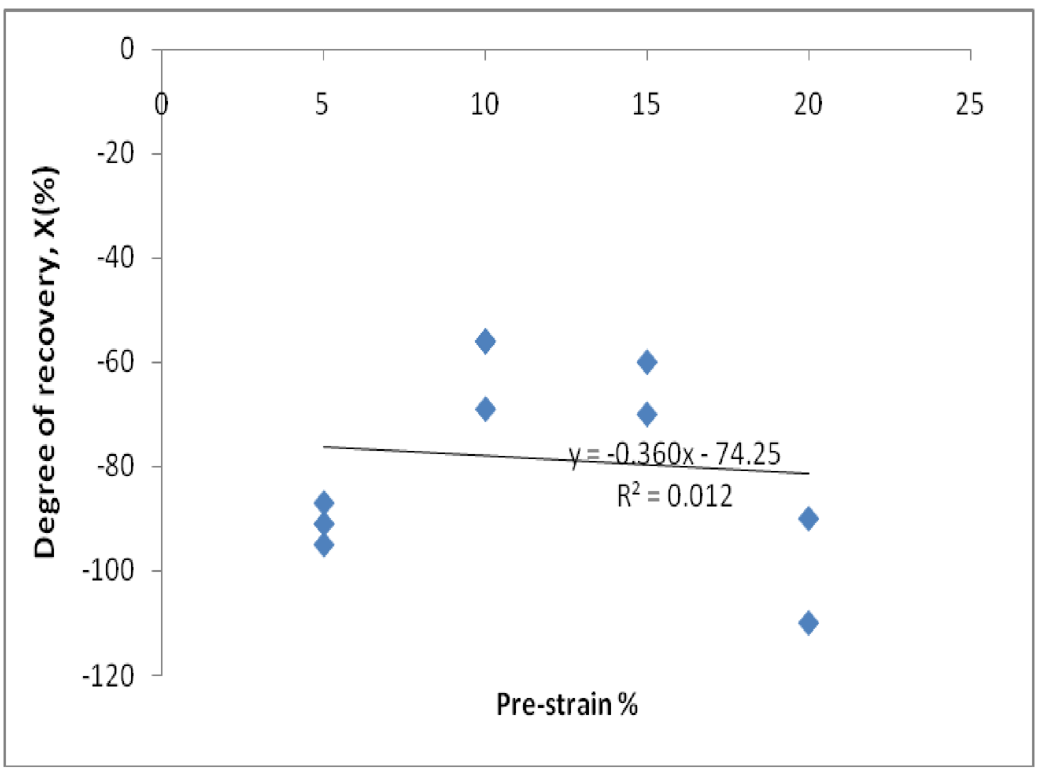

Figure 4.1: Graph degree of recovery versus pre – strain

From figure 4.1 which is graph degree of recovery versus pre – strain, the trendline was made to find the trend of the data. According to the graph, the degree of recovery was decrease as the pre – strain value increase.

4.4.2 Degree of Recovery Versus Temperature

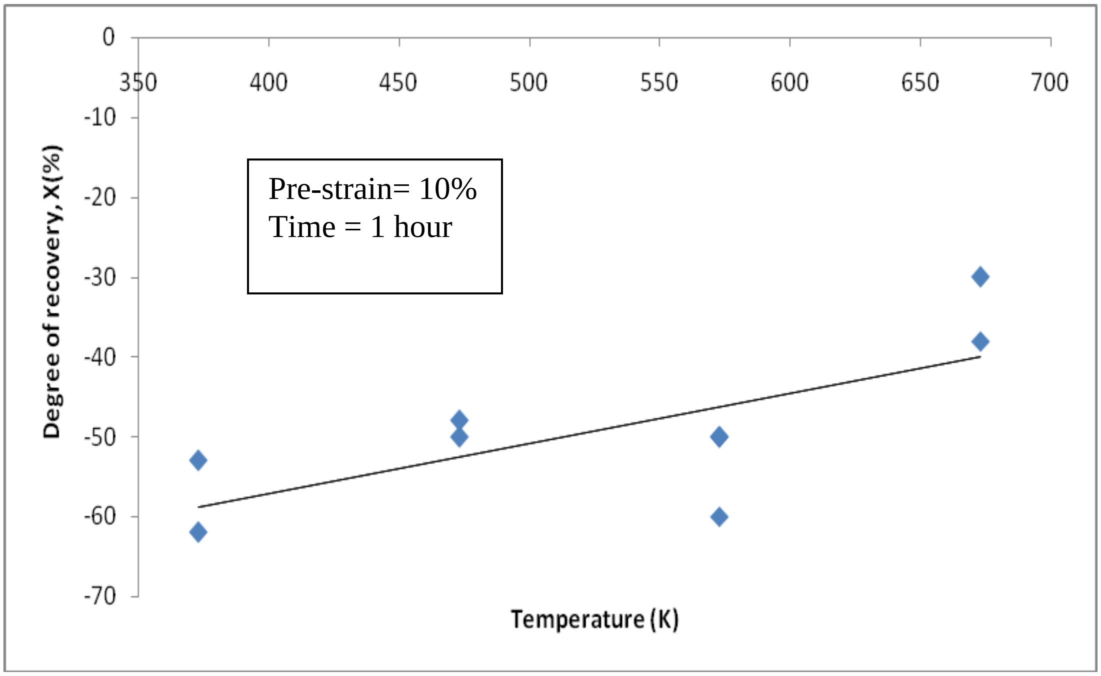

Figure 4.2: Graph degree of recovery versus temperature

Graph in figure 4.2 shows degree of recovery versus temperature with fixed pre – strain 10% and time 1 hour. The trend of the data showed that as the temperature increase, the degree of recovery also increase. This was explained as in the literiture review where the the effect of increasing temperature will affect the internal energy that supplied to the structure.

The higher internal energy supplied to the structure will increase the dislocations to rearrange into lower configuration order. When more dislocations turn to lower configuration order, the recovered grain will increase in the structure. This mean that the degree of recovery will increase when the temperature increase.

4.4.3 Degree of Recovery Versus Time

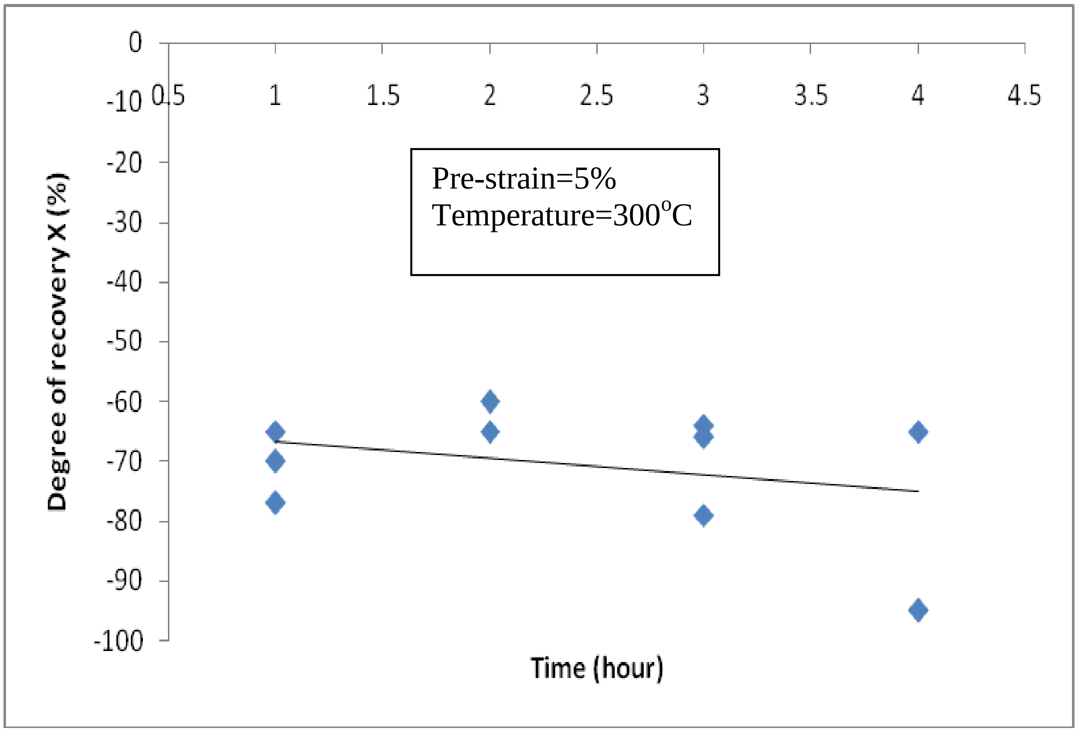

Figure 4.3: Graph degree of recovery versus time

The graph in figure 4.3 shows the relationship between the recovery time and degree of recovery. From the experiment, the data seem to be decrease slightly as the recovery time increase. From the literiture review, the degree of recovery will increase when the recovery time is increase. This can be explained when the soaking time increase, the internal energy that supplied to the structure is increase. The increasing of internal energy will relocated more dislocations to lower configuration energy in the structure. This will makes the recovery of the material is increase.

From graph obtained, the trend is slightly decrease. This was the effect of the experiment error which is explained further in the error discussion. The graphs that showed before were used to obtain the relationship between degree of recovery with the variables. The analysis of the project is proceed to validate the static recovery process.

4.5 Equation Obtained from Graph

In order to validate the static recovery process of plain carbon steel, the equation from the graph for each variable must be obtained. The graphs for variable time and temperature were replotted to obtain the equation. This is because the equation was important for comparison to Friedel's equation which is:

$$X_{rec} = C \ln t - \frac{Q}{RT} \tag{2.2}$$

Where:

X_{rec} = Degree of recovery

C = Constant

t = Time (s)

Q = activation energy (kJ/mol)

R = Gas constant (8.314 J/mol.K)

T = Temperature (K)

For temperature variable, the graph need to be constructed which is degree of recovery versus 1/temperature. Mean while, for recovery time variable, the graph degree of recovery versus ln(t) had to be plotted. For the pre – strain variable, there was no equation comparison made to the Friedel's model equation. This is because there was no pre – strain variable in the equation relationship. This graph was used only to get the relationship between degree of recovery.

This equation had to be obtained because the activation energy needs to be calculated. The activation energy was important to validate the static recovery process by using the Friedel's model equation. The equation was obtained in the next graphs.

4.5.1 Graph Degree of Recovery Versus 1/temperature

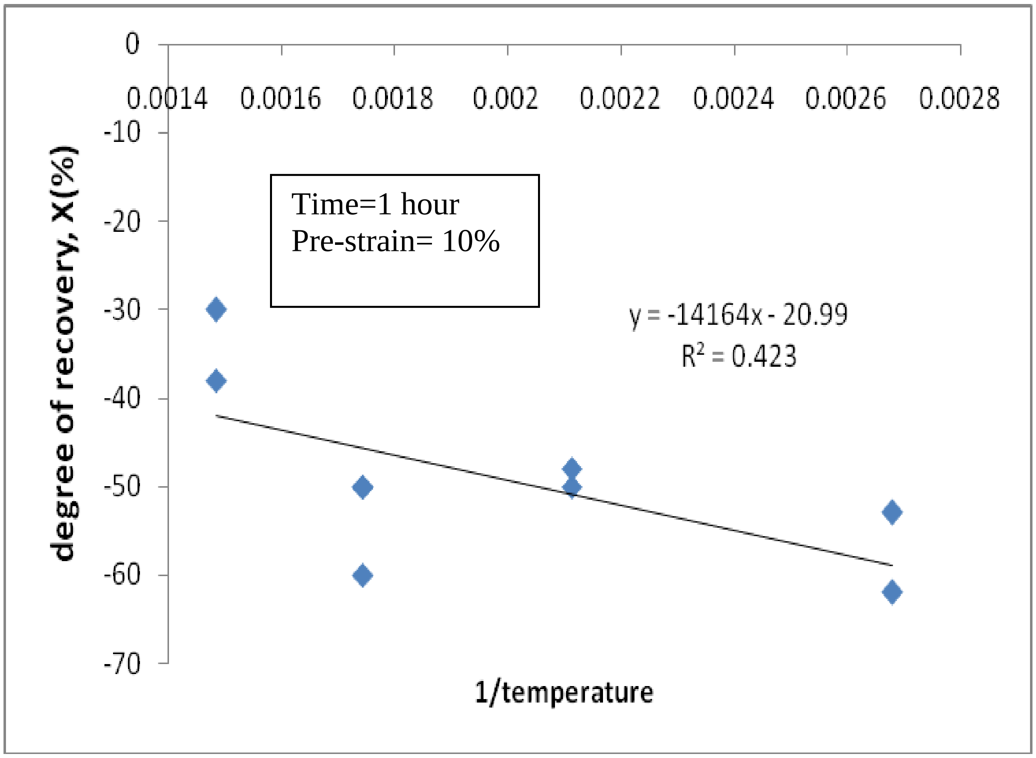

Figure 4.4: Graph degree of recovery versus 1/temperature

From the graph degree of recovery versus 1/temperature, the linear equation was obtained which is:

$$y = -14164x - 20.99 \qquad (4.1)$$

This linear equation was used to calculate the activation energy by comparing equation (4.1) to Friedel's model equation (2.2). In this experiment, the recovery time was fixed to 1 hour. So, the activation energy for 1 hour recovery process for plain carbon steel calculation showed below.

$$y = -14164x - 20.99 \text{ ....(1)}$$

$$X = C\ln t - \frac{Q}{RT} \text{ ....(2)}$$

Equation (1) compare with equation (2) which is:

$$-14164x = -\frac{Q}{RT}$$

Since,

$$x = \frac{1}{T}$$

Then,

$$-14164 = \frac{Q}{R}$$

Q = (-14164)R

Q = (-14164)(8.314)

Q = 118 kJ/mol

From the calculation above, the value for activation energy for I hour is 118 kJ/mol.

4.5.2 Graph Degree of Recovery Versus ln(t)

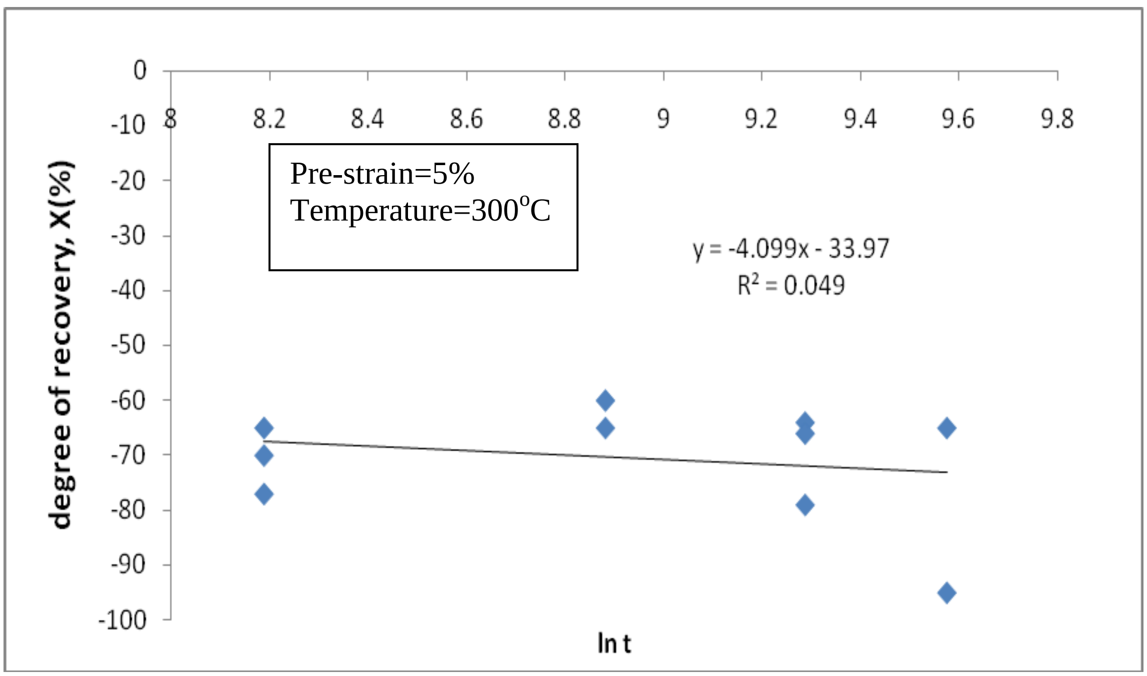

Figure 4.5 : Graph degree of recovery versus ln(t)

From the graph degree of recovery versus ln(t), the linear line was obtained with linear equation:

$$y = -4.099x - 33.97 \qquad (4.2)$$

Tis graph represents the data with static recovery process temperature 300^{o}C. From the linear equation obtained, the activation energy can be calculated by comparing Friedel's model equation as calculated before. Hence, the activation energy for 300^{o}C was calculated as showed below.

$$y = -4.099x - 33.97 \text{ ....(1)}$$

$$X = C\ln t - \frac{Q}{RT} \text{ ....(2)}$$

Equation (1) compare with equation (2) which is:

$$-33.97 = -\frac{Q}{RT}$$

$$Q = (33.97)(RT)$$

Substitute the value R=8.314 J/mol.K and T= 573K:

Q = (33.97)(8.314)(573)

Q = 162 kJ/mol

From the calculation, the activation energy for 300^{o}C was 162 kJ/mol.

4.6 ACTIVATION ENERGY COMPARISON

In order to validate the static recovery process by using the Friedel's model, the activation energy that had been calculated need be compared. The comparison was made with the value of activation energy that had been taken from the other journal. The journal was taken from the literature review which is the activation energy of carbon steel for 300^0C and 1 hour recovery process. Below is the table of activation energy comparison:

Table 4.25: Activation energy comparison with literature review

Activation energy	Experiment data	Literature review
Q for 300^oC	162 kJ/mol	174 kJ/mol
Q for 1 hour	118 kJ/mol	136 kJ/mol

Source: Ali Smith, Haiwen Luo, David N. Hanlon, Jilt Sietsma and Sybrand Van Der Zwaag, Recovery Processes In the Ferrite Phase In C-Mn Steel (2004) [8]

4.6.1 Different Error Calculation

From the comparison of activation energy in table 4.25, there were errors for both values. The percentage of error was calculated below.

Error Q for 300^oC:

$$Error = \frac{174 - 162}{174} x100\%$$

$$= 6.9\%$$

Error Q for 1 hour:

$$Error = \frac{136 - 118}{136} x100\%$$

$$= 13.2\%$$

Based on the calculation above, the error of activation energy for 300^oC was 6.9% and for 1 hour was 13.2%. From these values, the error values were small compared to the literature review's value. Hence, the activation energy for experiments data were accepted. That mean the static recovery process for plain carbon steel was valid by using Friedel's model.

The errors in the comparison value was due to the experimental error. This error was occurs during the experiment process. The major error that occurs was the oxidation of the specimen. This is because, the oxidation of plain carbon steel was high compared to the other metals. In this project, the oxidation occurs to the specimen during the annealling and recovery process. During these process, the reaction at the metal was occur due to high temperature. The result was the oxidation layer occured at the specimen's surface.

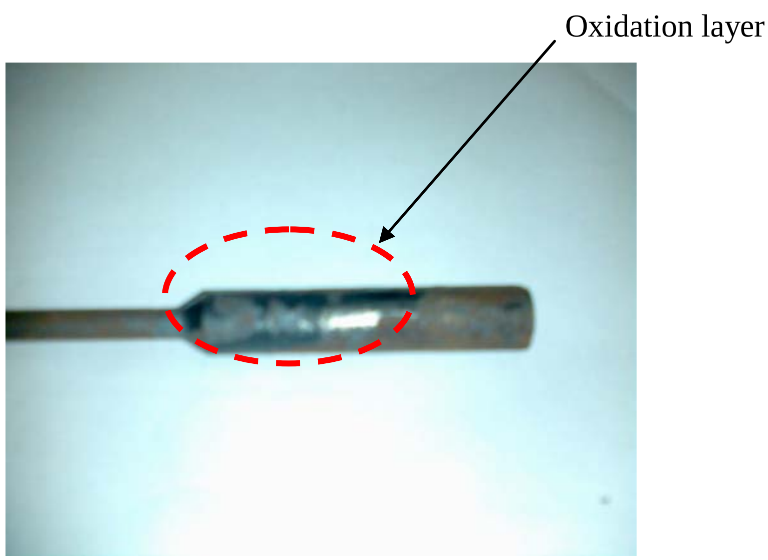

Figure 4.6: Oxidation at the specimen's surface

Figure 4.4 shows the oxidation layer occurred at the specimen's surface which is the black region in the red spot. This oxidation layer had affect the result in the tensile test process. This is because the layer was fragile and most of them peel off from the surface. Although this layer can be removed, about 40% of the oxidation still adhesive permanantly at the surface.During the tensile test process, slip was occured between the specimen and the gripper as the machine pull apart. Although the slip was very small, it can affect the result of the stress.

Futhermore, the range of temperature and time variable were small in this project. This range will result the less effect of degree of recovery between the variables. Due to this effect, the trend of the data when the graph was plotted will cause the less accuracy and precision.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Based on the analysis that has been discussed in the previous chapter, the activation energy, Q that obtained from experiment calculation was accepted by comparing with journal purpose by (Ali Smith, Haiwen Luo, David N. Hanlon, Jilt Sietsma and Sybrand Van Der Zwaag, *Recovery Processes In the Ferrite Phase In C-Mn Steel* [8]) . According to this project, the activation energy of plain carbon steel for variable time (fixed temperature 300^{o}C and 5% pre – strain) is 162 kJ/mol. Mean while, the activation energy for variable temperature (fixed recovery time 1 hour and 10% pre – strain) of this material is 118 kJ/mol. From the comparison, these values were close to the journal values where the errors were 6.9% for time variable and 3.2% for temperature variable. Due to these values, static recovery process for plain carbon steel has been validated by using Friedel's model equation.

From the relationship between static recovery process with pre – strain, (5%, 10%, 15% and 20%) the degree of recovery was slightly decreased with increasing pre – strain. There was no activation energy calculated from this variable because there was no pre – strain value in the Friedel's model equation. This research found that degree of recovery was increased with increasing recovery temperature (100^{o}C, 200^{o}C, 300^{o}C and 400^{o}C). This was satisfied with the literature review which is recovery increase with increasing temperature. For time variable (1 hour, 2 hours, 3 hours and 4 hours), recovery process that obtained was decrease with increasing time. This result was inversed with the theory. This is due to errors that had been discussed in previous chapter.

5.2 RECOMMENDATIONS

After this project was completed, there are some recommendations ideas that can be used for further study. This is to make some improvements to the research analysis for better result obtain.

Firstly, the improvement that can be made is increasing the range of recovery temperature variables. This is because the higher range of temperature, the effect of the degree of recovery will increase. The effect of recovery in different temperature is important in the analysis where the trend of the data can obtain more clearly. This improvement also can be used for time and pre - strain variables.

The other improvement can be made is by taking more data for each variable. This is to make sure the analysis is more accurate for better result. Furthermore, taking more variables also can improve the analysis. For example, the different temperature variable can be analyzed by fixing more pre – strain variables in one graph. This is used to find better characteristic and behavior of plain carbon steel in the static recovery process.

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APPENDIX A

Data reverse calculation for journal Ali Smith, Haiwen Luo, David N. Hanlon, Jilt Sietsma and Sybrand Van Der Zwaag, 2004, *Recovery Processes In the Ferrite Phase In C-Mn Steel.*

Temperature 600^{o}C

Time(s)	ln(t)	X(%)
300	5.703782	82
200	5.298317	80
110	4.70048	76
60	4.094345	70
30	3.401197	62
20	2.995732	60
0.6	-0.51083	20

Temperature 550^{o}C

Time(s)	ln(t)	X(%)
400	5.991465	80
100	4.60517	60
17	2.833213	40
6	1.791759	30
2	0.693147	20
0.5	-0.69315	15
0.2	-1.60944	10

Temperature 500^{o}C

Time(s)	ln(t)	X(%)
400	5.991465	62
300	5.703782	60
100	4.60517	50
60	4.094345	40
20	2.995732	30
10	2.302585	29
2	0.693147	20

Temperature 450^{o}C

Time(s)	ln(t)	X(%)
500	6.214608	42
300	5.703782	40
60	4.094345	30
30	3.401197	20
5	1.609438	16
1	0	10