CHARACTERISATION AND MODELLING OF STATIC RECOVERY PROCESS OF STAINLESS STEEL

MOHD AIZAD BIN KAMAROL

UNIVERSITI MALAYSIA PAHANG

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MOHD AIZAD BIN KAMAROL

A report submitted in partial fulfillment
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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

Signature :

Name of Supervisor : Dr. Ahmad Syahrizan Bin Sulaiman
Position : Head of Manufacturing Programme
Date :

Signature :

Name of Panel :
Position :
Date :

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 concurrently submitted for award of other degree.

Signature……

Name: Mohd Aizad Bin Kamarol
ID Number: MA05036
Date: ……

To my beloved father and mother

Kamarol Bin Abu Bakar

Rosni Bte Ali

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ABSTRACT

Annealing is a process where a material undergoes heat treatment for an extended time period and then slowly cooled. This process is very useful to alter the structure of the material where the grain size of the material tested change. The mechanical properties such as ductility and toughness also change during the annealing process. In this research, the recovery stage which is one of the annealing processes is being investigated. The material that has been use is type 304 stainless steel. At the recovery temperature which is in the range of 100^{o}C-400 ^{o}C, the behavior of stainless steel being investigated and the degree of softening, X_{rec} is calculated using Friedel's model. Friedel's model is a mathematical model used to calculate the degree of softening. There are 36 specimens being tested and from this experiment, 3 graphs plotted which are Xrec vs Time, X_{rec} vs Temperature and X_{rec} vs Pre-strain. Using Friedel's model, the activation energy, Q is calculated and being compared with other journal. The Q value obtained from X_{rec} vs Time and X_{rec} vs Temperature graphs are 466kJ/mol and 154kJ/mol respectively. There is no comparison made with X_{rec} vs Pre-strain graph. The Q value obtained from the graph plotted and from the journal is almost the same. So, the Friedel's model is valid to calculate the degree of softening at static recovery temperature of stainless steel. From this whole research, the behavior of stainless steel at static recovery temperature can be predicted using the model. This is very useful to apply at the real life problem such as buildings and structure that using stainless steel as material.

ABSTRAK

Sepuh lindap ialah proses dimana bahan akan melalui proses pemanasan pada jangka masa tertentu dan disejukkan secara perlahan. Proses ini sangat berguna untuk mengubahsuai struktur bahan dimana saiz bijian logam bahan yang diuji akan berubah. Sifat mekanikal seperti kemuluran dan kekuatan bahan juga berubah semasa proses sepuh lindap berlaku. Dalam kajian ini, peringkat pemulihan iaitu satu daripada proses sepuh lindap disiasat. Bahan yang digunakan ialah keluli tahan karat jenis 304. Pada suhu pemulihan iaitu dalam lingkungan 100^{o}C-400^{o}C, kelakuan keluli ini diperhatikan dan darjah kelembutan, X_{rec} dikira menggunakan model Friedel. Model Friedel ialah model matematik yang digunakan untuk mengira darjah kelembutan. Sebanyak 36 spesimen telah diuji dan daripada eksperimen ini, 3 graf telah diplot iaitu Xrec vs Masa, X_{rec} vs Suhu dan X_{rec} vs Tegasan. Dengan menggunakan model Friedel, nilai tenaga pengaktifan, Q boleh dikira dan dibandingkan dengan nilai yang terdapat pada journal. Nilai Q yang didapati daripada graf X_{rec} vs Masa dan X_{rec} vs Suhu ialah 466kJ/mol dan 154kJ/mol setiap satu. Tiada perbandingan dapat dibuat dengan graf X_{rec} vs Tegasan. Nilai Q yang didapati daripada graf yang telah diplot dan nilai yang terdapat pada journal adalah hampir sama. Oleh itu, model Friedel adalah sah untuk mengira darjah kelembutan pada suhu pemulihan keluli tahan karat. Melalui keseluruhan kajian ini, sifat keluli tahan karat pada suhu pemulihan boleh dijangka menggunakan model ini. Ini adalah sangat berguna untuk diaplikasikan dalam masalah kehidupan seharian seperti bangunan dan struktur yang menggunakan keluli tahan karat sebagai bahan.

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Annealing is one of the important processes in industry. Through this process, the mechanical properties of metals can be altered. This process is a heat treatment process where the material is changing in properties such as strength and hardness. It is a process that produces conditions by heating and maintaining a suitable temperature, and then cooling. Annealing is used to induce ductility, relieve internal stresses, refine the structure and improve cold working properties.

One of the processes in annealing is static recovery. The static recovery process occurs before the recrystallization temperature. At this stage, thermal energy is supplied to allow the dislocation to rearrange themselves into lower energy configuration. Through this experiment, the stainless steel will be going through several annealing process at the static recovery temperature and the results of this experiment will then use to develop a mathematical model.

Type 304 stainless steels are the most and widely used of many stainless steel. Although they have a wide range of corrosion resistance, they are not the most corrosion resistant of austenitic stainless steels. The chemical compositions of type 304 stainless steel are 0.08% C, 2% Mn, 1% Si, 18%-20% Cr and 8%-12% Ni. The 304 series of stainless steels exhibit high temperature strength, oxidation resistance, ease of fabrication and weldability, good ductility and good impact resistance down to at least -183^{o}C.

Table 1.1: Mechanical and physical properties of types 304 stainless steel [5].

Property	Type 304
Modulus of elasticity (GPa)	193
Tensile strength (MPa)	515
Yield strength (MPa)	205
Percent elongation at failure (%)	40
Melting temperature (^{o}C)	1400 -1450

1.2 PROBLEM STATEMENT

- To investigate static recovery effect in type 304 stainless steel when subjected to different strain.

1.3 PROJECT OBJECTIVE

- To validate Friedel's model of static recovery process of stainless steel in compression test.

1.4 PROJECT SCOPES

- Use types 304 stainless steels as test specimen.
- Operate lathe machine to shape the stainless steel into compression test specimen.
- Use box furnace to perform annealing.
- Perform compression test using compression test machine and gather required information about the test (pre-strain at 2.5%, 5%, 7.5%, and10%).
- Plot the graph using Microsoft Excel.

1.5 PROJECT BACKGROUND

Mechanical properties of stainless steel can be change by thermo-mechanical processes. This process required the stainless steel to go through some mechanical and annealing process. Annealing is one of the heat treatment processes that will be use in this research. Through this process, the stainless steel properties will be change such as ductility and hardness. The ductility of the material will increase while the hardness will decrease. The annealing process includes heating the material at suitable temperature and then cooling it slowly. There are three stages of annealing process which are recovery, recrystallization and grain growth. The static recovery process occurs at the temperature below the recrystallization temperature. At this stage, the thermal energy is supplied to allow the dislocation to rearrange themselves into lower energy configuration. The hardness of the stainless steel is reduced while the ductility increased. The stainless steel behavior at the recovery stage is then used to make a mathematical model. Through the compression test at different pre-strain (2.5%, 5%, 7.5% and 10%), a graph is then will be plot using Microsoft Excel.

CHAPTER 2

LITERATURE REVIEW

2.1 ANNEALING

The term annealing refers to a heat treatment in which a material is exposed to an elevated temperature for an extended time period and then slowly cooled. Ordinarily, annealing is carried out to (a) relieve stresses, (b) increase softness, ductility, and toughness and/or (c) produce specific microstructure. A variety of annealing heat treatment is possible; they are characterized by the changes that are induced, which many times are microstructural and are responsible for the alteration of the mechanical properties. [1]

Any annealing process consists of three stages (a) heating to the desired temperature, (b) holding or soaking at the temperature, and (c) cooling, usually to room temperature. Time is an important parameter in these procedures. During heating and cooling, there exist temperature gradients between the outside and interior portions of the piece; their magnitudes depend on the size and the geometry of the piece. [1]

Full annealing is relatively straightforward heat treatment in which the steel is heated to a temperature above the A_3 critical temperature and held at the temperature long enough to allow the solution of carbon and another alloying elements in the austenite. [3] The alloy is then furnace cooled; that is the heat treating furnace is turned off and both furnace and steel cool to room temperature at the same rate, which takes several hours. [1]

2.2 RECOVERY

The most subtle stage of annealing is recovery. No gross microstructural change occurs. However, atomic mobility is sufficient to diminish the concentration of point defects within grains and in some cases, to allow dislocation to move to lower energy positions. This process yields a modest decrease in hardness and can occur at temperatures just below those needed to produce significant microstructural change. [2]

The internal energy of the recovered metal is lower than that of the cold worked state since many dislocations are annihilated or moved into lower energy configurations by the recovery process. During recovery, the strength of a cold worked metal is reduced only slightly but ductility is usually significantly increased. [4]

2.3 RECRYSTALLIZATION

Upon heating a cold worked metal to a sufficiently high temperature, new strain-free grains are nucleated in the recovered metal structure and begin to grow, forming a recrystallized structure. After a long enough time at a temperature at which recrystallization takes place, the cold worked structure is completely replaced with a recrystallized grain structure. [4]

Primary recrystallization occurs by two principal mechanisms; (a) an isolated nucleus can expand with a deformed grain or (b) an original high grain boundary can migrate into a more highly deformed region of the metal. [4]

2.4 GRAIN GROWTH

After recrystallization is complete, the strain-free grains will continue to grow if the metal specimen is left at the elevated temperature; this phenomenon is called grain growth, Grain growth does not need to be produced by recovery and recrystallization; it may occur in all polycrystalline materials, metals and ceramics alike.

As the grains increase in size, the total boundary area decrease yielding an attendant reduction in the total energy; this is the driving force for grain growth. [1]

Grain growth occurs by the migration of grain boundaries. Obviously, not all grains can enlarge, but large one grows at the expense of small ones that shrink. Thus, the average grain size increases with time, and at any particular instant there will exist a range of grain sizes. [1]

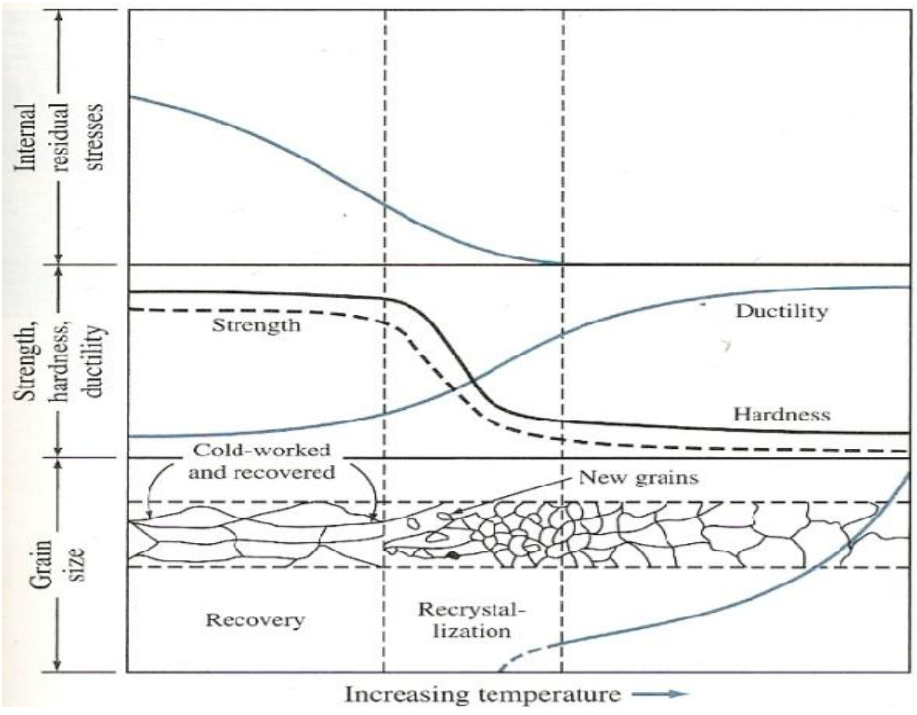

Figure 2.1: Effect of annealing on the structure and mechanical property changes of cold worked metal. [4]

2.5 COMPRESSION TEST

Compression test are determined by subjecting a specimen to an increasing compressive load until general yielding has occurred. Compression tests measure malleability (malleability is a measure of the extent to which material can withstand deformation in compression before failure occurs), and in compressive testing only compressive yield strength and compressive elastic modulus are measured. This is done in a manner similar method of tensile test. Theoretically, these values should be the same as tensile yield and modulus of elasticity values but, in reality, there is usually a small difference. [3]

In compression testing, the specimen barrels rather than necks. Because different factors are at work in barreling, malleable specimens flow in response to the load and actually compress rather than fracture. As the materials yield, it swells out (barrels), so that its increasing area continues to support the increasing load. [3]

Engineering stress, $\sigma = \frac{F}{A}$ [Eqn 2.1]

Where;

F= Instantaneous load applied perpendicularly to the cross section.

A= The original cross sectional area before any load is applied

Engineering strain, $\varepsilon = \frac{\left(l_i - l_o \right)}{l_o}$ [Eqn 2.2]

Where;

l_i = Original length before any load applied

l_o = The instantaneous length

Equation 1 and equation 2 are utilized to compute compressive stress and starain, respectively. By convention, a compressive force is taken to be negative, which yields a negative stress. Furthermore, since lo is greater than li , compressive strains computed from equation 2 are necessarily also negative. Compressive tests are used when material's behavior under large and permanent (i.e. plastic) strains is desired, as in manufacturing applications or when the material is brittle in tension.

2.6 STAINLESS STEEL

In all probability the most widely known and most commonly used material of construction for corrosion resistance is stainless steel. Stainless steels are iron based alloys containing 10.5% or more chromium. There are currently over 70 types of stainless steels. [5]

Stainless steel is not singular material, as its name might imply, but rather a broad group of alloys, each of which its own physical, mechanical, and corrosion-resistant properties. These steels are produced both as cast alloys [Alloy Casting Institute (ACI) types] and wrought forms [American Iron Steel Institute (AISI) types].

Generally, all are icon based with 12 to 30% chromium, 0 to 22% nickel, and minor amounts of carbon, columbium, copper, molybdenum, selenium, tantalum, and titanium. They are corrosion resistant and heat resistant, noncontaminating, and easily fabricated into complex shapes. [5]

2.6.1 Stainless Steel Classification

There are three general classification systems used to identify stainless steels. The first relates to metallurgical structure and places particular stainless steel into a family of stainless steels. The other two, namely, the AISI numbering system and the Unified Numbering System, which were developed by ASTM to apply to all

commercial metals and alloys, define specific alloy compositions. The various stainless steel alloys can be divided into seven basic families: [7]

a) Ferritic
b) Martensitic
c) Austenitic
d) Precipitation Hardenable
e) Superferritic
f) Duplex (ferritic-austenitic)
g) Super austenitic

2.7 FRIEDEL'S MODEL

Friedel's model is a mathematical equation proposed by J.Friedel, Professor of Solid State Physics, University of Paris. This model is written in his book entitled Dislocations and was published by Pergamon Press in 1964 The Friedel's model is very useful in modeling the static recovery behavior of the materials. The mathematical equation is used to determine the degree of softening of the materials when some test is carried out. The degree of softening, X, from this model is being calculated from the expression; [6]

$$X_{rec} = \frac{(\sigma_m - \sigma_r)}{(\sigma_m - \sigma_o)} \qquad \text{[Eqn 2.3]}$$

Where;

σ_m = Flow stress immediately before unloading.

σ_r = Initial flow stresses recorded during reloading.

σ_o = Initial flow stresses recorded during pre-straining.

The relationship between the amount of recovery, time and temperature was found, over a wide range of conditions to be; [7]

$$X = c_1 \ln t - \frac{Q}{kT} \qquad \text{[Eqn 2.4]}$$

Where;

Q = Activation energy

k = Gas constant

c_1 = Constant

T = Temperature

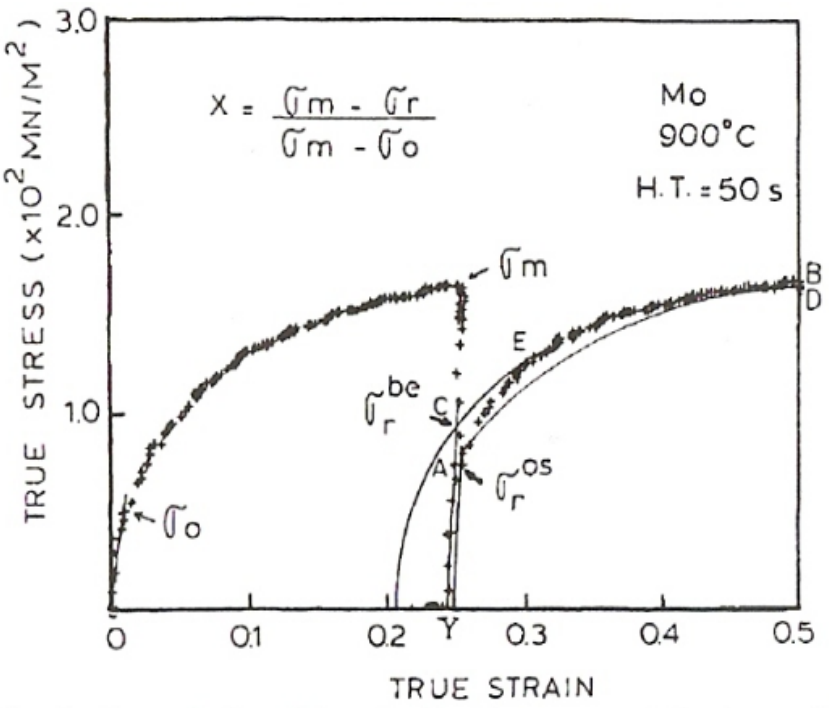

Figure 2.2: Determination of the reloading flow stress and the degree of softening by the back extrapolation and offset method. [10]

2.8 JOURNAL COMPARISON

To make sure this experiment works, a comparison with other journal being made. This is important to compare the calculation that being made with other established journal. Some of the journals are;

- H.L Andrade, M.G Akben, and J.J Jonas, Metallurgical Transaction. Effect of Molybdenum, Niobium, and Recrystallization and on Solute Strengthening in Microalloyed Steels **14(**1983),pp 1967-1977 [6]
- F.J Humphreys and M. Hatherly, Recrystallization and Related Annealed Phenomena Second Edition, UK, Elsevier Ltd, 2004 [7]
- T.Furu,R.Orsund and E.Nes,Subgrain Growth In Heavily Deformed Aluminium-Experimental Investigation and Modelling Treatment,Vol.**43**(1995),No.6,pp. 2209-2232 [8]

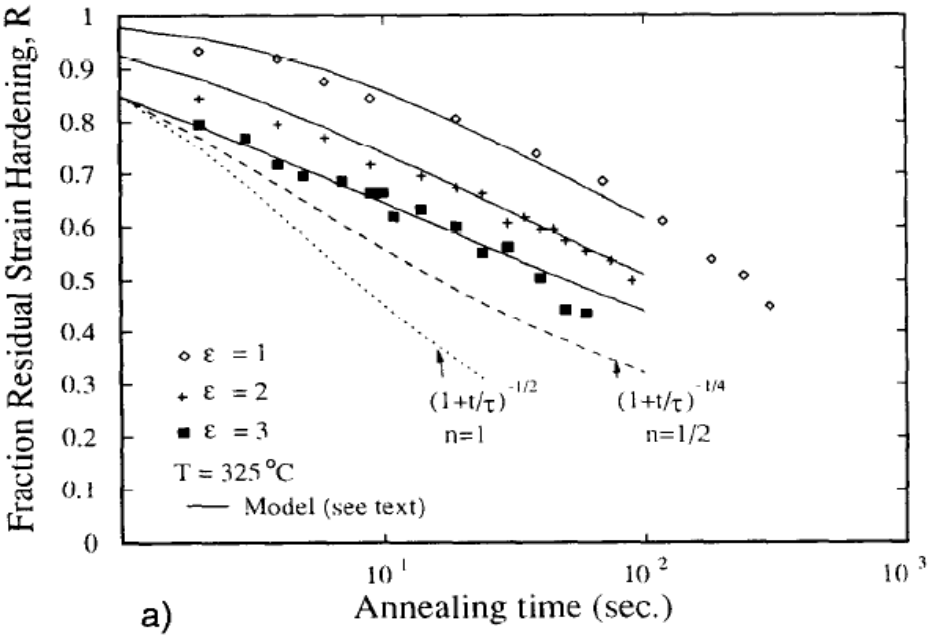

Figure 2.3: T.Furu, R. Orsund and E. Nes. Subgrain Growth in Heavily Deformed Aluminium-Experimental Investigation and Modelling Treatment, 1995 [8]

Table 2.1: Data collected from T.Furu, R. Orsund and E. Nes graph [11]

R (degree of softening)	Time(sec)
0.45	390
0.5	300
0.52	220
0.6	120
0.69	70
0.75	45
0.8	24
0.84	9
0.86	6
0.91	3.8
0.95	1.6

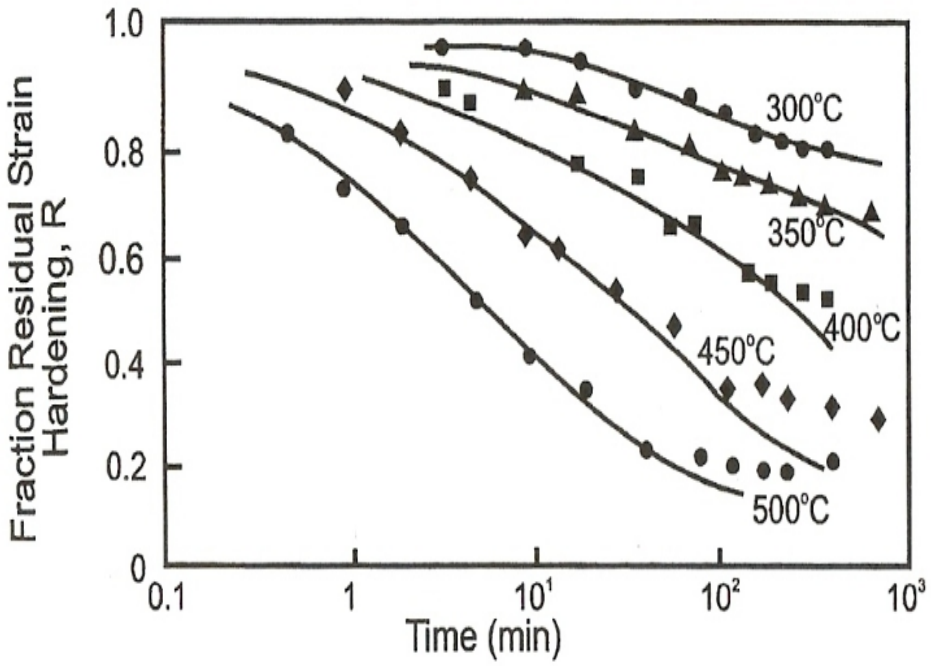

Figure 2.4: Modelling the recovery by Michalak and Paxton (Iron) [13].

Table 2.2: Data collected from J.T Michalak and H.W Paxton

R	Time(sec)
0.98	180
0.98	540
0.97	1200
0.9	2100
0.89	4200
0.87	6000
0.85	10800
0.83	13200
0.81	17400
0.81	27000

Table 2.3: Calculated values of Q from different journals

Material	Journal	Q (kJ/mol)
Stainless Steel	G.R. Stewart,J.J Jonas and F.Montheillet	405
Iron	J.T Michalak and H.W Paxton	105

CHAPTER 3

METHODOLOGY

3.1 FLOW OF METHODOLOGY PROCESS

Figure 3.1 shows the overall process of the experiment that being conducted. The function of this flow chart is to give guideline and information about the project. From this flow chart, the critical part of this research can be determined. The critical part of this research is the annealing process at the static recovery temperature. This part of this experiment must be conducted very well to get the accurate data. This is because, this research must be conducted at the temperature below the recrystallization temperature which is at the recovery state range between 100^{o}C and 400^{o}C.

The other critical part of this experiment is the design or dimension of the specimen for the compression test. The specimen's dimension for all 36 specimens must be the same. This is because the dimension is important during the compression test is being conducted. For this research, the dimension of the specimen must be 10mm in diameter and 25mm in length. The tolerance of the specimen is only $\pm 0.1mm$ If the dimension is not the same, the data obtained from the test is not accurate. So, the sequence of the flow chart must be followed to make sure this research is done and all the data from the test is accurate.

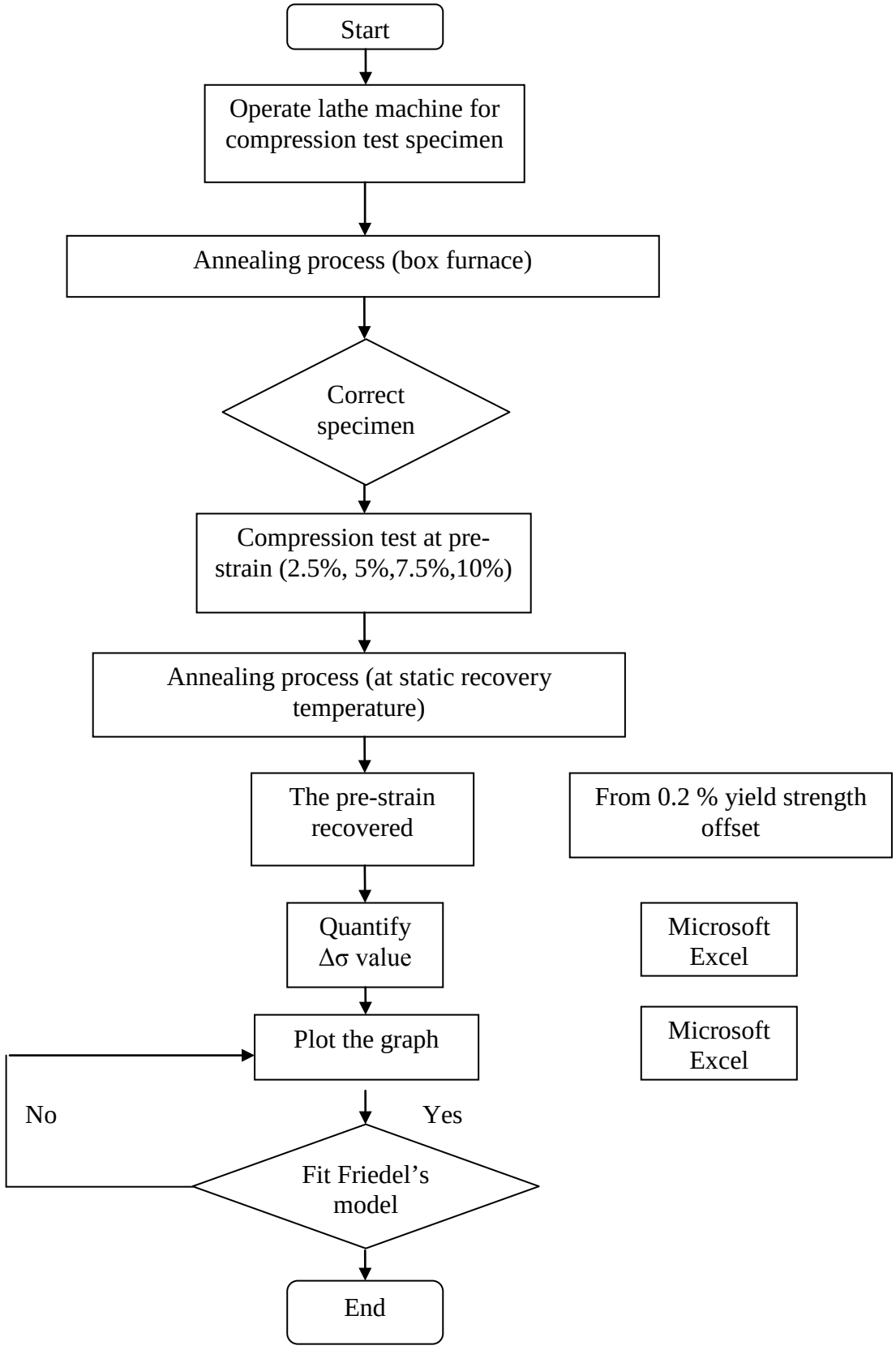

Figure 3.1: Flow chart of overall process

3.2 MACHINING PROCESS

Before the machining process can be start, the dimension of the specimen is being draw using Solid Work software. The entire specific dimension for the specimen must be known. The dimension of the specimen is 10mm in diameter and 25mm in length. The raw material used which is type 304 stainless steel must be cut with the band saw before using the lathe machine. This is because, the raw material comes from manufacturer in a long cylinder rod. So, to operate it using the lathe machine, the raw material must be cut first.

Figure 3.2: Bandsaw machine.

To use the band saw machine, cutting speed is a very important parameter. If not, the end product is not good. If the material used is hard, the cutting speed must be set lower. This is very important to avoid the saw from broke. The use of coolant is a must. This is because the coolant can protect the material and the saw from overheating.

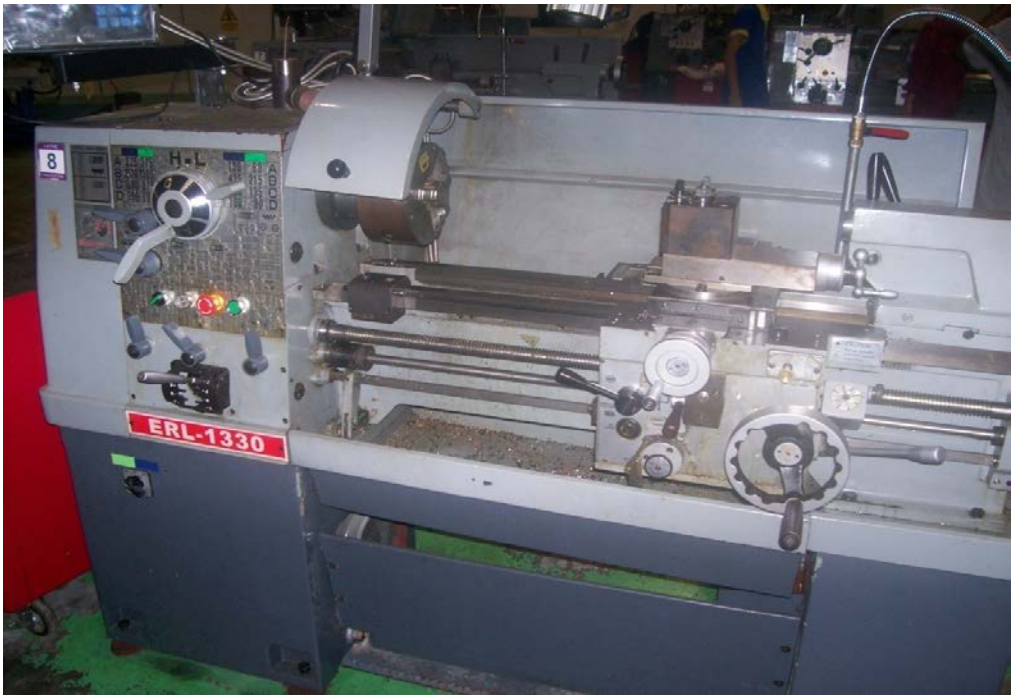

Figure 3.3: Lathe machine

Figure 3.3 shows the conventional lathe machine used to prepare the specimens for the compression test. The important thing in handling the lathe machine is the speed needed to cut the specimen. If the speed of the lathe machine is correct, then the machining process can be carried out. If not, the specimen will damage and the finishing product is bad.

The dimension of the specimen must accurate which is 10mm in diameter and 25 mm in length. So, to produce a good specimen, the machine must feed slowly and the dimension of the specimen must be measure every time using the vernier caliper. Because of the length of the material which is stainless steel needed is only 25 mm, the machining process must be carried out very careful. Stainless steel is a hard material. So, it will take a long time to cut it little by little. The edge of the material which is very sharp must be taken out. The material will then undergo chamfering process to make sure it is safe for the user to use during the experiment.

3.3 ANNEALING PROCESS

After the correct specimen and dimension obtained, the next step is the annealing process. This process required a furnace and an operating temperature which is;

Annealing temperature = 760^{o}C

Static recovery temperature = 400^{o}C

Firstly, all the specimens will be put in the furnace and being heat at 760^{o}C. This process required a specific time, how long the specimen will be heat up. This can be set at the furnace itself. At the program controller, there are three things that should be setup which is time 1, time 2, and the maximum temperature. Time 1 is the time where the maximum temperature will be achieved which is 760^{o}C. Time 2 is the soaking time which is how long the maximum temperature must be maintained and the maximum temperature is the temperature that we need to heat the specimen. The program controller is shown in **Figure 3.5**.

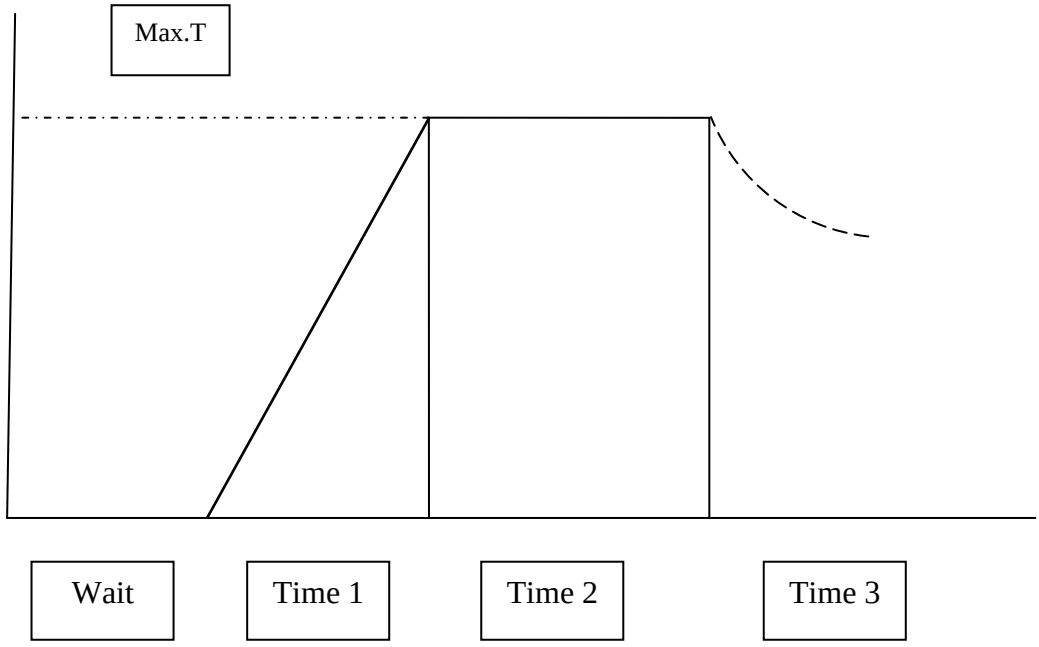

Figure 3.4: Box furnace program controller setup graph

Figure 3.4 shows the time and temperature that need to be setup before the overall process of annealing being conduct. This is important because the overall process of annealing is depends on the time and the temperature being set at the controller. As mentioned before, one of the critical parts of this research is the temperature that being set. If the temperature is not right, the whole experiment will failed because the date produce is not accurate.

Max. T = Maximum temperature for the annealing process.
Time 1 = Time the maximum will achieve.
Time 2 = Soaking time which the specimen is being heated at constant temperature.
Time 3 = Cooling time which the time needed for the specimen to cool in the furnace.

Figure 3.5: Program controller

Figure 3.6: Furnace

After all the perimeters being set, the specimens are put in the box furnace. The soaking time for the specimens is one hour. A simple calculation is done to predict how long the overall operation take time. The process start at room temperature and hit the maximum temperature which is 760^{o}C (annealing temperature). The time needed to achieve the maximum temperature is being calculated using formula below.

Time needed to achieved maximum temperature = $\frac{\left(T_{\max} - T_{ambient}\right)}{I}$

Where;

T_{max} = Maximum temperature desired (^{o}C)

$T_{ambient}$ = Ambient temperature (^{o}C)

I = Increment of the box furnace (5^{o}C/minute)

Overall process time = Time to achieved maximum temperature + Soaking time + Cooling time

Cooling time is determined manually. When the specimen cooled down in the box furnace, the temperature is taken every 5 minutes for 20 minutes. Then we can predict how long it will take to cool at room temperature before the specimen can be taken out from the furnace.

Figure 3.7: Specimen in the box furnace

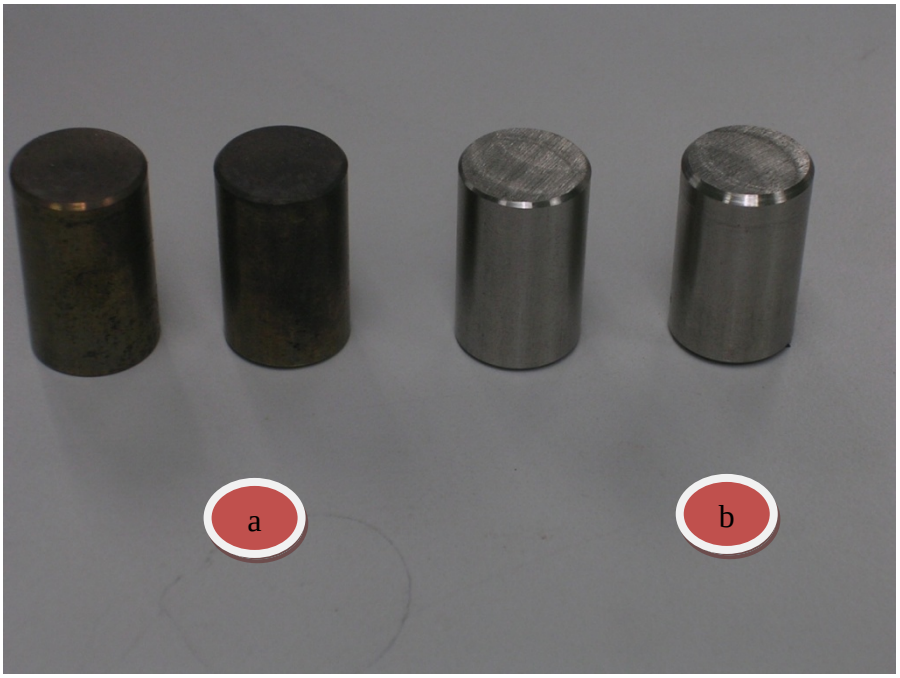

Figure 3.8: The test specimens a) after annealing at 760^{o}C b) before annealing

3.4 COMPRESSION TEST

After annealing process done, all the specimens will undergo compression test. This compression test required to get different pre-strain. There are 36 specimens and will be divided into certain group to undergo different pre-strain. The compression test is set at 2.5%, 5%, 7% and 10% pre-strain. To make sure this entire specimen did not mix with each other, the specimen is marked. This will make the process going smoothly and the specimen can be easily recognized.

The data from the compression test which is σ_{max} and σ_o is recorded. Some of the data cannot be shown from the computer. This is because of the specimen length is not proportionally decrease with the increasing load. So, we have to get the σ_{max} and σ_o values manually from the graph that already plotted by the computer. **Figure 3.9** show a compression test machine that being use during the test conducted. It is an automatic compression test machine that will plot the graph needed during the experiment being run.

Figure 3.9: Compression test machine

3.5 ANNEALING (RECOVERY TEMPERATURE)

The next step for this experiment is to recover the specimen. The annealing process is done again but with different temperature. At this stage, the specimen will be heat up in the box furnace at the temperature below the recrystallization temperature which is 1/3 from the melting point temperature of stainless steel ($1400^{o}C$). This is the critical parts where the temperature plays an important role. If the temperature is not at the recovery temperature, the structure of the material is going to be change. This is because the structure will be recrystallized. When the material recrystallized, the grain boundary of the material is growing bigger and this will results a wrong experiment.

The temperatures selected for this experiment are $100^{o}C$, $200^{o}C$, $300^{o}C$ and $400^{o}C$. These temperatures are chosen because at this stage, the recovery will occur. The process is exactly the same like previous annealing but the different only at the temperature selected.

3.6 RECOVERED PRE-STRAIN

The specimen is then being test again under the different pre-strain using the compression test machine. This time, the σ_r which is the recovered stress value being recorded. From this recovered stress data, we can quantify the value of $\Delta\sigma$, which is the different between the stress before and after recovery. This value is identified at 0.2% yield strength offset. Because of the used of computerized machine, the offset already set up in the program. The data from the compression test machine already fit this law.

The same problem occurs during earlier compression test happened again during recovered pre-strain. So, the manual method is used to get the $\Delta\sigma$ value from the computer.

3.7 ANALYZING DATA

To analyze these data, three graphs are plotted using Microsoft Excel. The data gathered will put in a table. These results will be discussed in chapter 4. The graphs are;

- X_{rec} vs Pre-strain
- X_{rec} vs Temperature
- X_{rec} vs Time

These three graphs are being compared with the results in the journals produced by other researchers. To make sure the results can be used or to valid the Friedel's model, the value of Q which is the activation energy calculated. This value is calculated and the percentage of different between the experimental and the value from the journal is done to see the different of error.

From the graph, the behavior of stainless steel at different temperatures and times can be predicted. This results is then can be use as a model for real life problem such as buildings structures that use stainless steel as a material.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 RESULTS

There are 36 specimens that have been tested. The results have been plotted into 3 graphs. From this graphs, a comparison using Fridel's model with the linear equation from the graph is made. The data produced is then being compared with other journal proposed from other researchers. There are 3 graphs that have been plotted:

1. X (degree of recovery) vs Time (300^{o}C at 5% pre-strain).

2. X (degree of recovery) vs Pre-epsilon (300^{o}C at 1 hour).

3. X (degree of recovery) vs Temperature (5% pre-strain at 1 hour).

Each graph is constructed using 12 specimens and each point in the graph is made with 3 specimens. To get the exact point, an average reading is taken from the 3 specimens. This is done because to avoid some problem that occurs during the machining process or during the compression test being conducted. The equation from the graph plotted is compared with Friedel's model;

$$X_{rec} = c_1 \ln t - \frac{Q}{kT}$$

Where;

Q = Activation energy (kJ/mol)
k = Gas constant (8.314 J/mol.K)
c_1 = Constant
T = Temperature (K)

During the experiment, there are several precaution steps that should be considered. This is very important to make sure the result is accurate. The precaution steps are;

1. The specimen must be cool at room temperature in the furnace before taking out.
2. Check the furnace temperature before heating the specimen.
3. The dimension of the specimen must be same.

Environmental expect such as room temperature also play an important role in this experiment. This is because the structure of the materials can be change if there is big different between the furnace temperature and the room temperature. That is why the specimens must be cool in the furnace at room temperature before it can be taken out.

Time also one of the important factors. The specimen must be heated at specific time according to the graph that going to be plot. If the specimen is heated longer than it supposed to be, the result is going to be inaccurate. This is because when the specimen is heated longer, the grain or the materials structure changing a lot. This will affect the whole experiment and the value needed for the test is not accurate.

4.2 RELATIONSHIP BETWEEN X AND t (TIME)

Table 4.1: X vs Time (300 degree Celcius/5% pre-strain) at 1 hour

σ_o	σ_m	σ_r	X	%X
200.2	354.4	299.2	0.358	35.8
200.5	352.5	310.9	0.274	27.4
194.3	367.6	306.5	0.353	35.3

Table 4.2: X vs Time (300 degree Celcius/5% pre-strain) at 2 hours

σ_o	σ_m	σ_r	X	%X
227	358.5	314.5	0.33	33
232.8	352.4	309.6	0.357	35.7
205.4	361	307.4	0.344	34.4

Table 4.3: X vs Time (300 degree Celcius/5% pre-strain) at 3 hours

σ_o	σ_m	σ_r	X	%X
222.5	351.8	299.6	0.403	40.3
195.4	380.3	303	0.418	41.8
224.9	371.7	303.8	0.463	46.3

Table 4.4: X vs Time (300 degree Celcius/5% pre-strain) at 4 hours

σ_o	σ_m	σ_r	X	%X
204.6	365.3	286.2	0.492	49.2
210.4	398.2	281.5	0.62	62
204.9	398.8	286.2	0.58	58

From the data collected, a graph X vs Time (at 300^{o}C, 5% pre-strain) is plotted. The graph is shown in **Figure 4.1**;

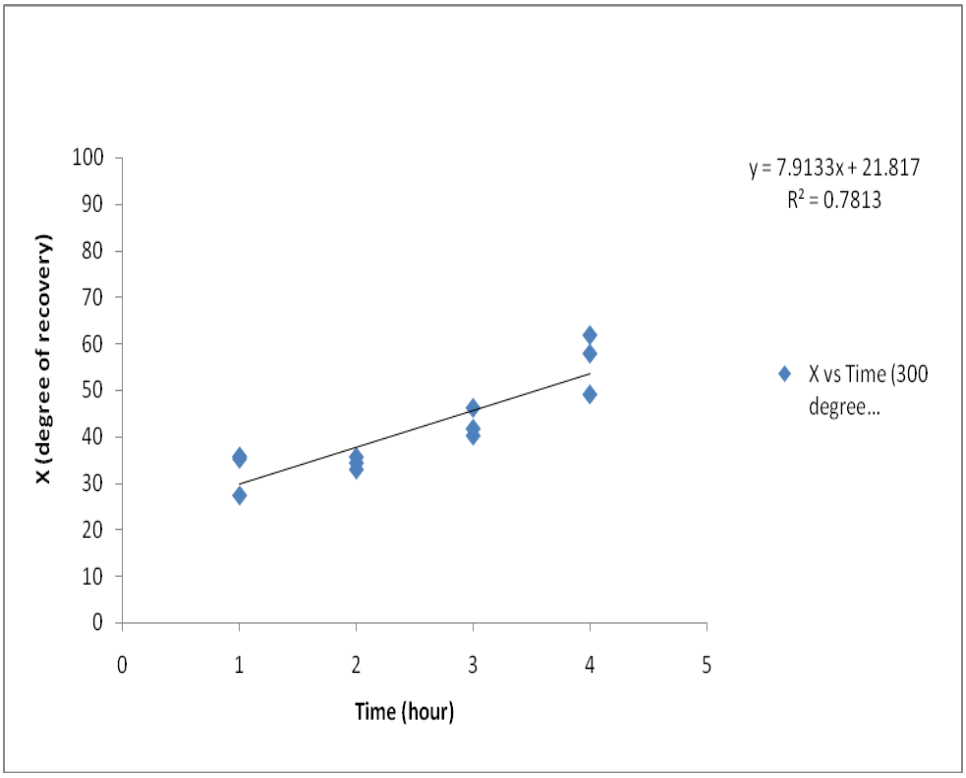

Figure 4.1: Graph X_{rec} vs Time (hour)

From the graph plotted above, there is a relationship between X value and Time. The average line get from the graph shows a linear line which shows that the values of X increase with increasing time. The linear equation of the graph is shown in the graph.

To make sure the data from the graph is suitable with the Friedel's model, the graph is plotted again. This is because the model proposed by Fridel use "lnt" instead of "t" which is time. The graph that will be plot again is X vs lnt (at 300^{o}C, 5% pre-strain). The graph is shown in **Figure 4.2** .

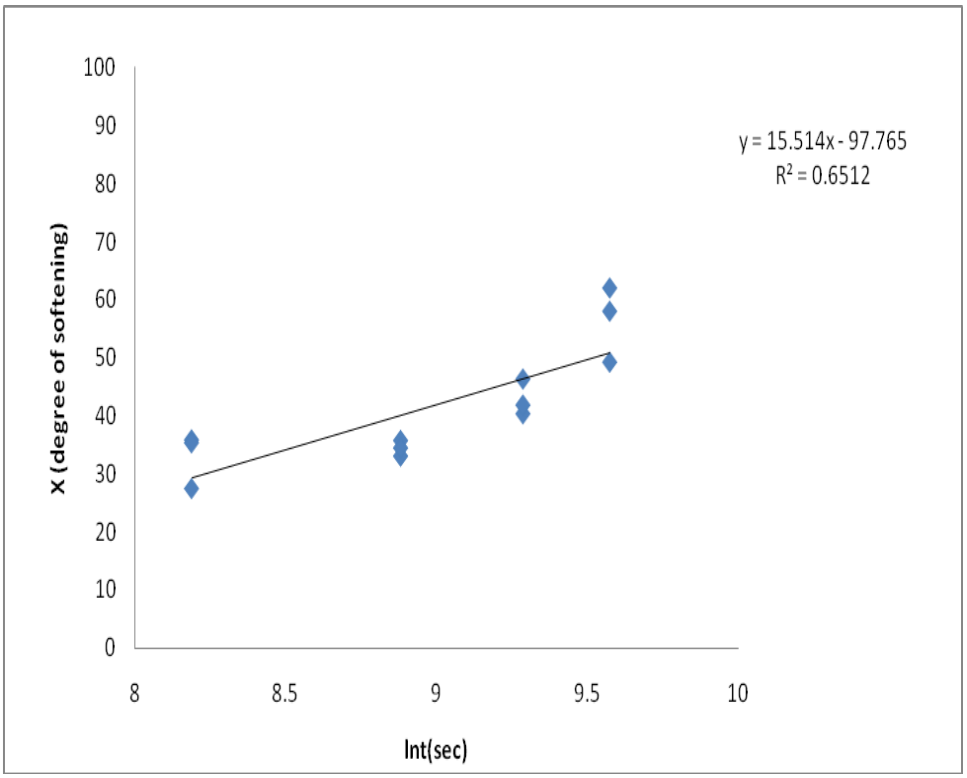

Figure 4.2: Graph X_{rec} vs lnt (sec)

From this data, the Q value which is the activation energy can be calculated. Using the Friedel's model, Q value can be found from the graph.

From the graph : $y = 15.514x - 97.765$ (1)

Frisdel's model : $X = c_1 \ln t - \frac{Q}{kT}$ (2)

A comparison between the first (1) and second (2) equation is made. Using the Friedel's model, the value of Q which is the activation energy is calculated. The value that fit with the Q value from Friedel's model is the intersection at y axis of the graph which is -97.765. So;

$$\frac{Q}{kT} = 97.765$$

$$Q = 97.765 \times 8.314 J/mol.K \times 573K$$

$$\therefore Q = 466 kJ/mol$$

4.3 RELATIONSHIP BETWEEN X AND T (TEMPERATURE)

Table 4.5: X vs Temperature at 100^{o}C (1 hour/5 % pre-strain)

σ_o	σ_m	σ_r	X	%X
199.9	361.8	275.5	0.533	53.3
196.2	355.3	269.6	0.539	53.9
246.4	367.1	280	0.722	72.2

Table 4.6: X vs Temperature at 200^{o}C (1 hour/5 % pre-strain)

σ_o	σ_m	σ_r	X	%X
219	359.3	274.7	0.603	60.3
195.6	373.3	256.8	0.656	65.6
209.1	353.9	236.3	0.812	81.2

Table 4.7: X vs Temperature at 300^{o}C (1 hour/5 % pre-strain)

σ_o	σ_m	σ_r	X	%X
241.6	354.2	263.7	0.803	80.3
234.2	361.7	278.5	0.653	65.3
244.2	352	261.7	0.838	83.8

Table 4.8: X vs Temperature at 400^{o}C (1 hour/5 % pre-strain)

σ_o	σ_m	σ_r	X	%X
250.2	340.2	277.3	0.7	70
257.4	361	274	0.837	83.7
238	349.5	246.6	0.924	92.4

From the data collected, a graph of X vs Temperature (1 hour/5 % pre-strain) plotted using Microsoft Excel. The same method applied at X vs Time graph is use again at this results.

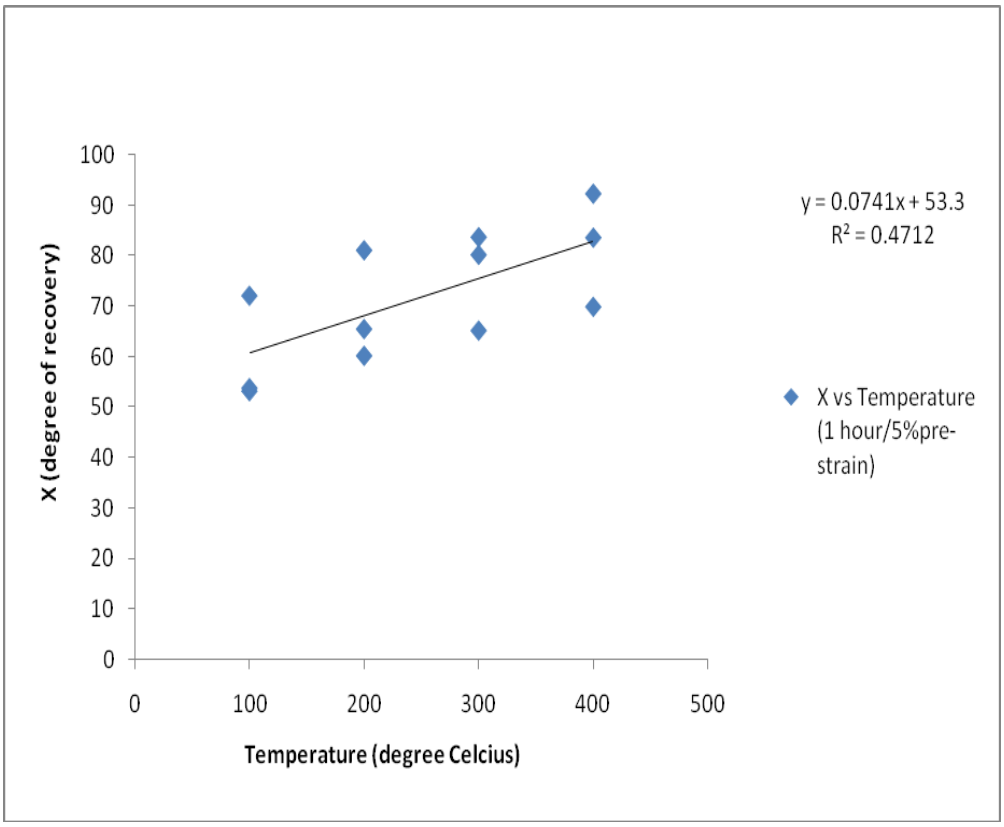

Figure 4.3: Graph X_{rec} vs Temperature (^{o}C)

Once again the graph shows a linear equation. This is the same as what is proposed by Friedel's model which is a linear equation. So, this graph linear equation can be compared to the one that Friedel's proposed. But there is a different value of temperature use in the equation which is in Celcius and not in Kelvin. A little modification is made to make sure the linear equation can be compared to Friedel's model.

From Friedel's model, the temperature is being plotted in Kelvin not in Celcius. So, the graph must be plotted again but this time using Kelvin value. The graph shows X vs Temperature, but from Fridel's model the graph must be 1/Temperature. So, the graph must be plotted again and the graph is X vs 1/Temperature (1 hour/5% pre-strain). The graph is shown in **Figure 4.4**.

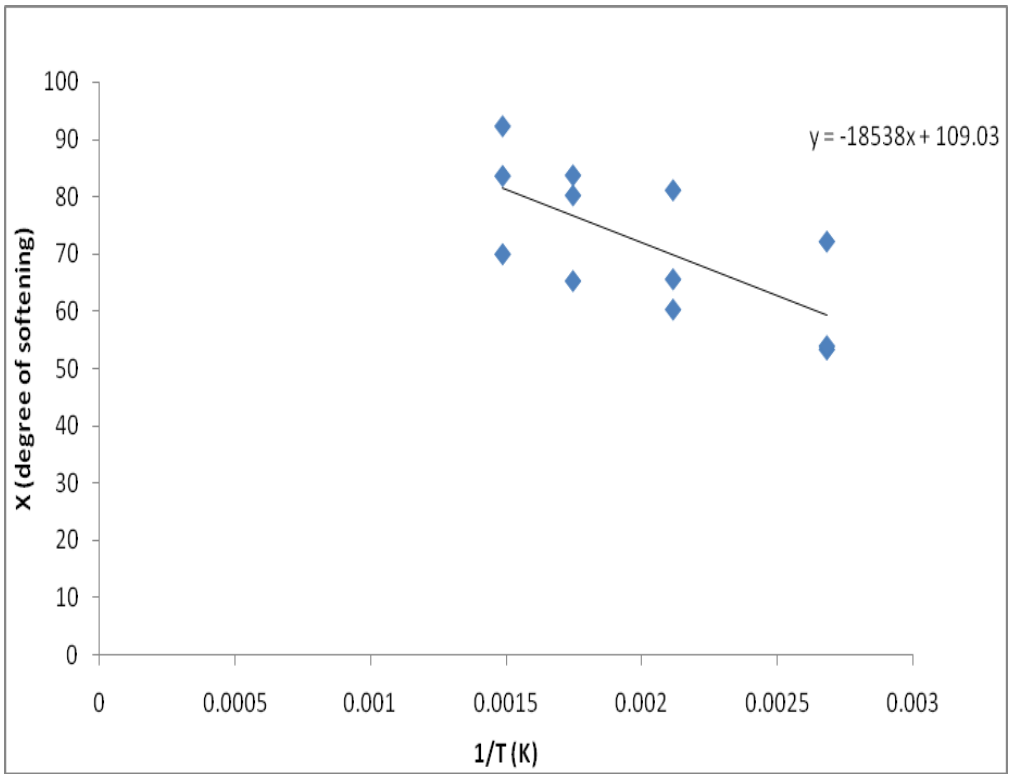

Figure 4.4: Graph X_{rec} vs 1/T (K)

Again, the linear function of the graph is being compared with Friedel's model. The value of Q which is the activation energy is calculated according to the experimental value of the linear function;

From the graph : $y = -18538x + 109.03$

Frisdel's model : $X = c_1 \ln t - \frac{Q}{kT}$

So, the slope of the graph is being used to calculate the value of Q, where the value for x-axis from the linear function is $\frac{1}{T}$.

$$\frac{Q}{k}\left(\frac{1}{T}\right) = 18538$$

$$Q = 18538 \times 8.314 J / mol.K$$

$$\therefore Q = 154 kJ / mol$$

4.4 RELATIONSHIP BETWEEN X AND PRE-STRAIN

Table 4.9: X vs Pre-strain at 2.5% (1 hour/300^{o}C)

σ_o	σ_m	σ_r	X	%X
147.7	294.8	182.7	0.762	76.2
147.1	288.2	215.3	0.517	51.7
100.8	131.3	162.9	-1.036	-103.6

Table 4.10: X vs Pre-strain at 5% (1 hour/300^{o}C)

σ_o	σ_m	σ_r	X	%X
156.6	358.2	244.5	0.564	56.4
63.59	167.3	271	-1	-100
165.1	354.8	260.7	0.496	49.6

Table 4.11: X vs Pre-strain 7% (1 hour/300^{o}C)

σ_o	σ_m	σ_r	X	%X
150.2	430.7	274.7	0.556	55.6
211.1	420.6	265.7	0.739	73.9
145.2	429.2	265	0.578	57.8

Table 4.12: X vs Pre-strain 10% (1 hour/300^{o}C)

σ_o	σ_m	σ_r	X	%X
187.1	484.4	246.6	0.8	80
158.4	473.5	242.9	0.732	73.2
196.3	470.8	245.7	0.82	82

The graph of X_{rec} vs Pre-strain is plotted using the data from the experiment. The graph is shown in **Figure 4.5**.

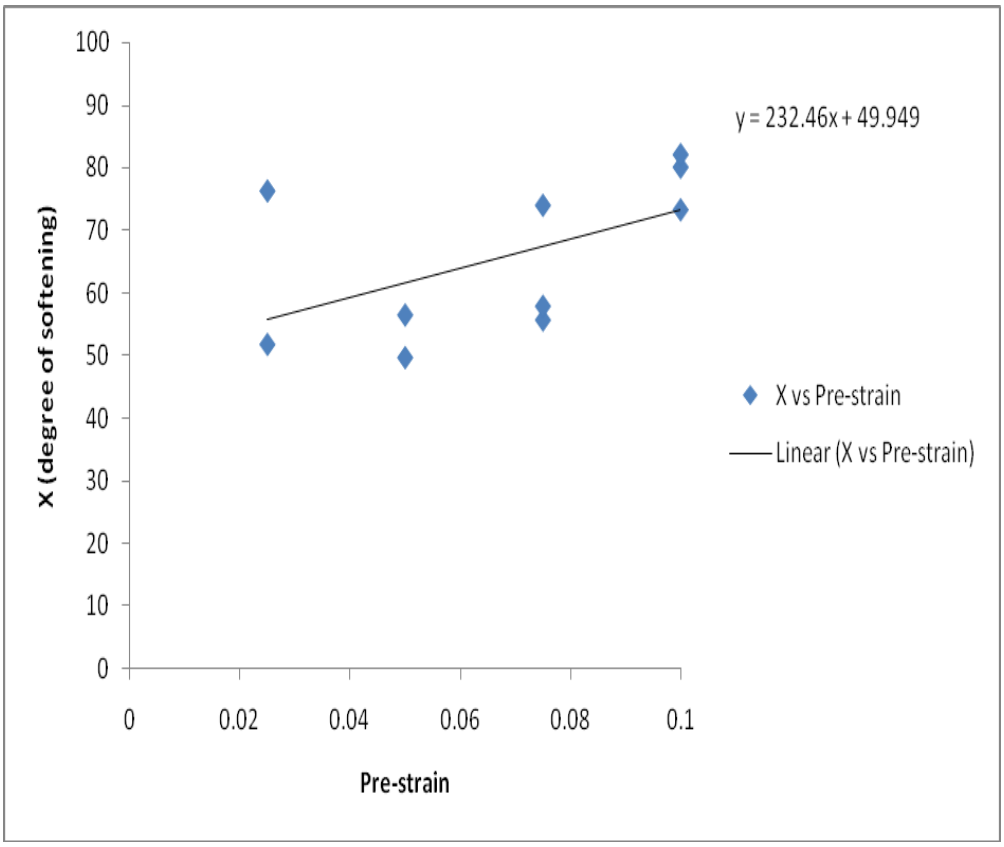

Figure 4.5: Graph X_{rec} vs Pre-strain

There is no comparison can be made between Friedel's model and pre-strain. This is because, the relationship shown from Friedel's model only for temperature, time and activation energy. In this research, the relationship of pre-strain with the degree of recovery is carried out. So, to make sure the graph is valid, a journal comparison is made.

In a journal written by E.Nes, *Recovery Revisited,* Acta Metall.mater, Vol.43, No.6, pp 2189-2207, 1995, stated that activation energy, Q, decrease with increasing cold work. The pre-strain is a cold work. So, when pre-strain increase, the value of Q which is activation energy should be decrease.

From Friedel's model,

$$X_{rec} = c_1 \ln t - \frac{Q}{kT}$$

The value of X which is degree of softening is directly proportional to the value of Q which is activation energy. This means the degree of softening increase with the increase of activation energy. E.Nes stated that activation energy is decrease with increasing cold work. This two statement is related to each other because there is the similarities of X and Q.

From Friedel's model;

$$X \propto Q$$

From E.Nes journal;

$$Coldwork \propto \frac{1}{Q}$$

From these two statements, a conclusion can be made that when X which is the degree of softening decrease, the cold work is increasing. This is because there is a relationship between X and Q values. So, it can be relate to what E.Nes proposed which is the activation energy decrease with increasing cold work.

From the relationship shows above, Friedel's model can be relate to E.Nes's statement through the value of X. From the equation, X is directly proportional to Q value which means when the value of Q increase, X also increase. This can be relate to E.Nes's statement about the cold work although there is no value of X present, but it still can be compared with the value of Q since there is a relationship between Q and X. From this, the relationship of Friedel's model and E.Nes and be made into one equation. The equation is shown below;

$$X \propto Q \propto \frac{1}{coldwork}$$

The graph that being plotted from the experiment result shows that the values of X increase with increasing cold work. There is a different from what proposed by Friedel and E.nes. The average line of the graph shows a linear equation. This is true and can be compared to Friedel's model but the relationship is exactly wrong. So, this result cannot be used for the comparison.

The result is wrong because there is some errors occur during this experiment being carried out. There is a probability of specimens error or experimental error.The actual graph for X vs Pre-strain according to E.Nes and Friedel's model should be like the red line shown in **Figure 4.6**.

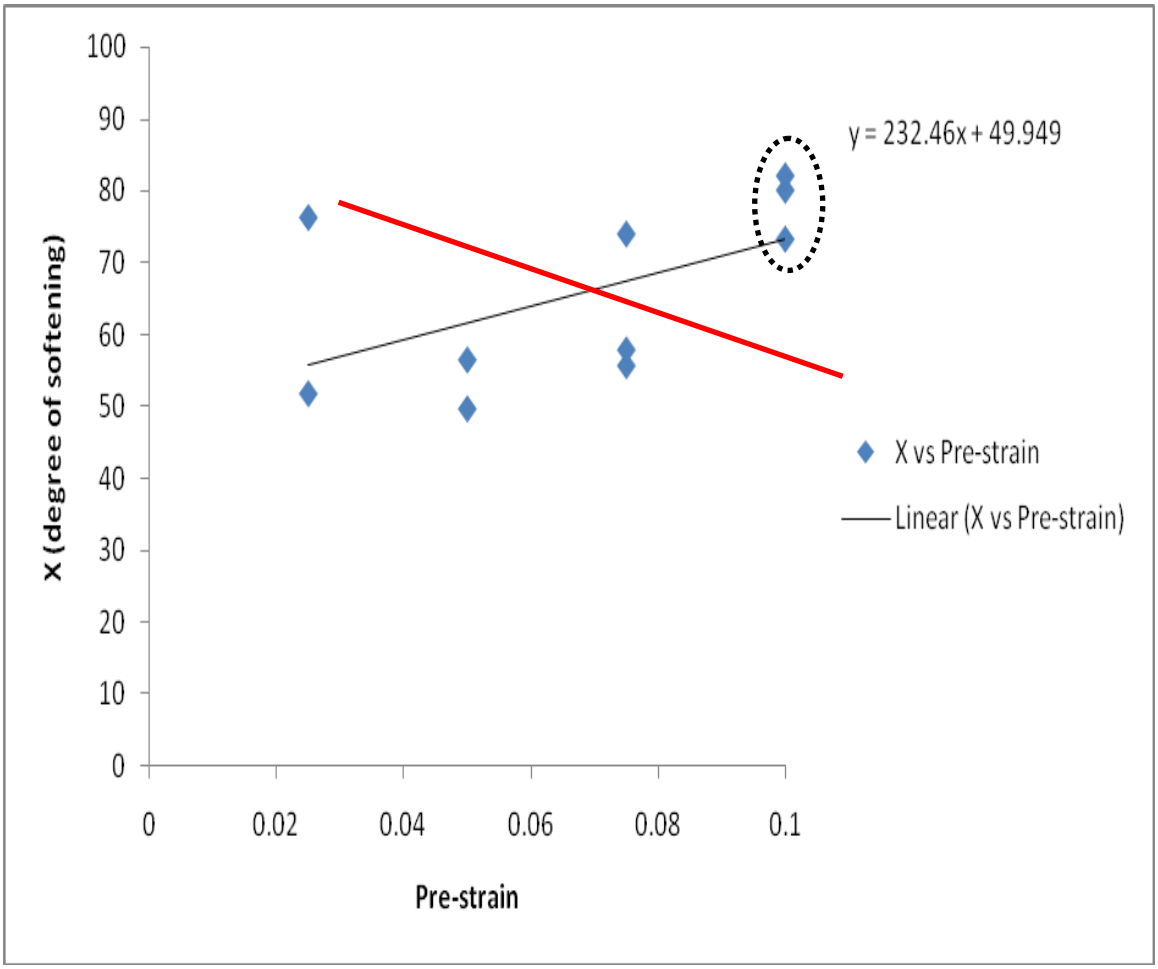

Figure 4.6: Comparison between experimental graph and the actual line graph (red).

The three points in the dashed circle in the graph are not correct. Although this three specimens are consistent which means the error produced is not big because the average reading of X% values for the specimens is in the range of 73% and 82%. This is because the value of X at 0.1 pre-strain must be lower than the value of X at 0.075 pre-strain according to what is proposed by E.Nes. The conclusion can be made is there must be specimens error during the experiment being conducted.

4.5 SPECIMENS ERROR

From the X_{rec} vs Pre-strain graph, the error occurred at 10% pre-strain. This is because, the specimens size which is 10mm in diameter and 25mm in length cannot support the load that the compression test machine produced. When the specimens is being tested until 10% pre-strain, the specimens begin to crack. That is why at 10% pre-strain, the value of X is more than the value of X at 7.5% pre-strain.

Stainless steel is a hard material. At 10mm diameter it only can stands until the range below 10% pre-strain. If the diameter of the specimen is bigger than 10mm, for example 15mm, the compression test machine that produced 50kN load cannot compress the stainless steel. That is why this experiment being carried out at 10mm in diameter.

From the data collected, there is also error occurred at 2.5% and 5% pre-strain. The value of X that have been calculated is a negative (-ve) value. So, the point or the data plotted at the graph is not counted. Only two points is being shown in the graph at 2.5% and 5% pre-strain. This is because, the other two points is a negative(-ve) value which is not in the data range. This happened because the value of σ_r which is the yield strength during reloading is bigger than σ_m which is the yield strength before unloading. So, according to the equation to get the value of X, it will produced a negative(-ve) value.

The purpose of three specimens being tested at each pre-strain is to avoid the data from getting out of range. So, the problem that occurred at 2.5% and 5% pre-strain can be avoided. The other two points are out of range which is the X value is negative(-ve). So, an average value of the other two points can be used and the negative value can be neglected But there is a different from the problem occurred at 10% pre-strain. As mentioned before, although the range of each data is very closed but still there is error. This is because of the relationship of X and coldwork is different from Friedel's model and what is propsed by E.nes.

Figure: 4.7: Specimens that crack after being tested at 10% pre-strain.

Figure: 4.7 show two specimens that being tested at 10% pre-strain. From the figure, the stainless steel specimen is bending at the middle. This is because the specimens cannot stand the load produced by the compression test machine and this will result the error produced in the graph. There is also a loud cracking sound heard during the experiment being conducted. So, the data produced from these specimens cannot be used as the test result.

4.6 VALIDATE FRIEDEL'S MODEL

To validate Friedel's model, a sample calculation is made with other journals. The activation energy, Q, is calculated using Friedel's model from the graph taken from other researchers.

Table 4.13: Activation energy values proposed from other researchers and the experimental value

Material	Journal	Q (kJ/mol)
Iron	J.T Michalak and H.W. Paxton	105
Stainless steel	G.R. Stewart,J.J Jonas and F.Montheillet	405
Stainless steel	Friedel's Model	466

The Q value that has been proposed by J.T Michalak and H.W Paxton is 105kJ/mol. To validate Friedel's model, a calculation using this model is made. If the value calculated using Friedel's model is the same as the proposed value, so this model is valid for this experiment to determine the effect of recovery with time, temperature and pre-strain.

The graph R, which is fraction residual strain hardening vs time (min) proposed by J.T Michalak and H.W Paxton is being plotted again. This is because, from Friedel's model, only X which is degree of softening can be determined not the value of R. However, there is a relationship between the values of R with X. The time used also being changed from minute to second. This will make sure the calculation become easy and the equation produced can be compared to Friedel's model.

To compare the graph proposed by J.T Michalak and H.W Paxton, the relationship of X with R must be define first. As proposed by A.Martinez-de-Guerenu, F. Arizti, I.Guitierrez, in their journal *Recovery During Annealing In A Cold Rolled Low Carbon Steel*, Acta Meterialia **52** (2004), pp. 3665-3670, the fraction of residual stress can be defined as ;

$$1 - R_y = \frac{H_c}{H_c^{def'}} \qquad \text{[Eqn 4.1]}$$

Where;

R_y = Fraction of recovery.

H_c = The coercive field of the material after an isothermal recovery annealing at fixed temperature.

H_c^{def} = The coercive field of the cold rolled material.

From the equation proposed by A.Martinez-de-Guerenu, F. Arizti, and I.Guitierrez, a relationship between R and X can be made. R_y is a value of fraction of recovery which means it is the same as X value from Friedel's model. The different is just A. Martinez used a different term in describing the fraction of recovery. The relationship is as follows;

$$R = 1 - X \qquad \text{[Eqn 4.2]}$$

Where;

R = Fraction of residual stress

X = Degree of softening (fraction of recovery)

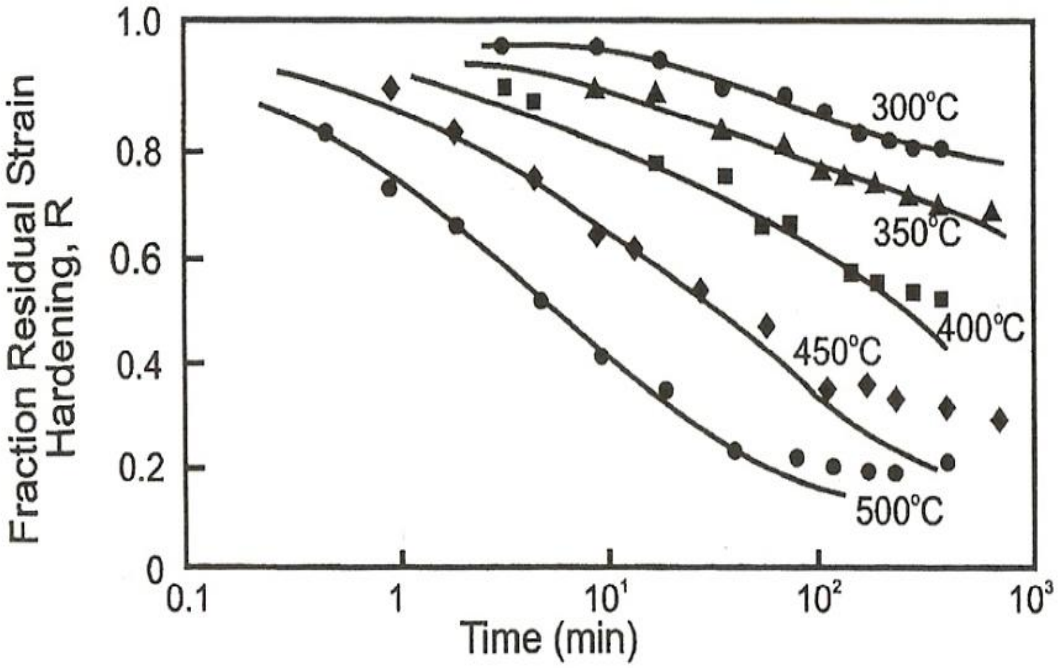

Figure 4.8: Actual graph proposed by J.T Michalak and H.W Paxton [13].

Figure 4.8 show the actual graph of R, fraction residual strain hardening vs Time (min) proposed by J.T Michalak and H.W Paxton. The specimens that being used in the experiment is iron. From the graph, the researchers use temperature in the range of 300^{o}C and 500^{o}C. Only data for the selected temperature will be used as a comparison. From this graph, the data for 300^{o}C will be compared with Friedel's model.

The data from the graph in **Figure 4.8** is collected and being plotted again using Microsoft Excel but with X, degree of softening vs Time (sec). So, the data collected must be change to X value first before it can be compared to Friedel's model. To determine the value of X, equation 4.2 is used. The time also must be change from minute to second. The rebuild graph for J.T Michalak and H.W Paxton for X, degree of recovery vs Time (sec) is shown in **Figure 4.9.**

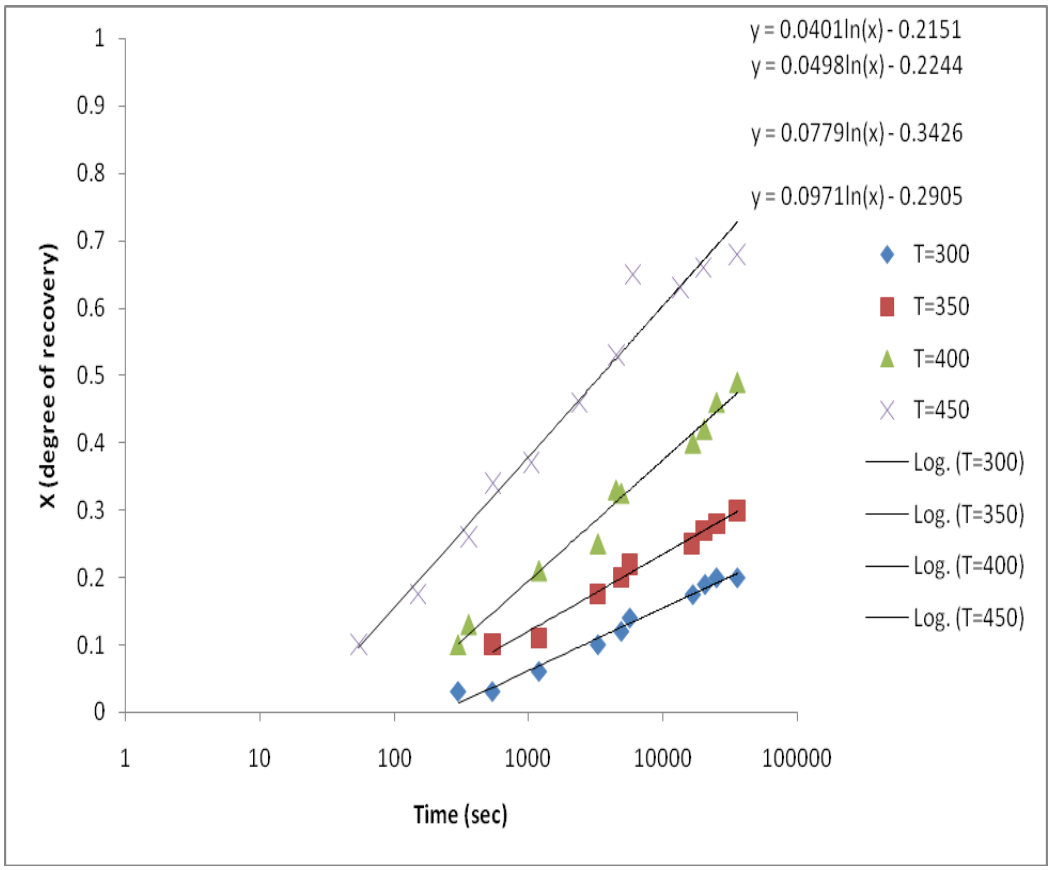

Figure 4.9: Graph X vs Time (sec) proposed by J.T Michalak and H.W Paxton that have been plotted again using X value.

The value of Q, which is activation energy of the material (iron) at 300^{o}C is calculated using Friedel's model. The result from this calculation is then can be compared with the Q value that has been proposed by J.T Michalak and H.W Paxton. The linear equation produced from the graph is then can be compared with Friedel's model and the value of Q can be determined. The calculation is shown as follow;

The linear equation from the graph ; $y = 0.0401\ln(x) - 0.2151$

Friedel's model ; $X = c1 \ln t - \frac{Q}{kT}$

From the comparison, the value of Q is the intersection of y-axis from the linear equation, where the value of T and k are temperature (573K) and gas constant (8.314kJ/mol) respectively. The calculation is made as follow;

$$\frac{Q}{kT} = 0.2151$$

$$Q = 0.2151 \times 8.314 kJ/mol \times 573K \times 100$$

$$\therefore Q = 103 kJ/mol$$

Activation energy, Q calculated from the graph is approximate to the value proposed by J.T Michalak and H.W Paxton which is 105kJ/mol. The different only 2kJ/mol. So, it can be conclude that Friedel's model is valid for the experiment proposed by J.T Michalak and H.W Paxton.

4.7 PERCENTAGE OF DIFFERENT

Through this whole research, the value of Q, activation energy that has been calculated is being compared to the journal proposed by G.R Stewart, J.J. Jonas and F.Montheillet. This is because, the material that has been used by these researchers is also stainless steel. The proposed value is 405kJ/mol which is different from this research which is 466kJ/mol. The different of error between the proposed value by the researchers and this experiment is calculated as follows;

$$\%Different = \frac{466 - 405}{405} \times 100\%$$

$$\%Different = 15\%$$

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Through this whole research, Friedel's model is valid to use to determine X (degree of softening). This is because the activation energy, Q calculated from this research is almost the same as the one proposed by G.R Stewart, J.Jonas and Montheillet, in their journal. The value of Q proposed by the researchers is being calculated using Zener-Hollomon parameter. So, this can be conclude why there is percentage of different between this two values which is from this research and the proposed value.

From E.Nes, in his journal *Recovery Revisited*, Acta Metall.mater, Vol.**43** (1995),No.6, pp 2189-2207 stated that activation energy, Q, decrease with the increasing of cold work. This is also satisfied in this research. The results can be seen from the graph X_{rec} vs Pre-strain. Although there is some error during the experiment, but the variation of the graph is satisfied with the statement.

From this research, the behavior of stainless steel at static recovery temperatures range from 100^{o}C until 400^{o}C can be predicted. This is very useful to predict the failure of the material at the specific temperature stated. But in this research, Friedel's model only valid at the recovery state of the material. So, the behavior of stainless steel above the recovery state of the material can not be predicted using this model.

5.2 RECOMMENDATIONS

To make sure the research value is accurate, some modification must be made. One example of the modification is the used of many parameters to make a comparison. The graph used by previous researcher used a lot of parameters. In this research, for example graph X_{rec} vs lnt (at 300^{o}C/5% pre-strain), the constant temperature used only at 300 ^{o}C. This is not enough to produce a good result and get an accurate value. So, the usage of many temperatures range may produce an accurate result.

The use of the right dimension for the specimen is also one of the factors that affect this research. This is because, at 10% pre-strain, the specimen cracked and the data produced was wrong. So, in the future, the use of bigger specimen for the compression test can be made. This will make sure the specimen can sustain the force produced by the compression test machine which is 50kN.

When the specimen dimension use is bigger, the force needed to pre-strain the specimen is also big. Stainless steel is a hard and tough material. This is because, from this research the 15mm diameter of stainless steel already being tested using the compression test machine. Only small amount of pre-strain can be produced from the compression test machine. So, it cannot produce a lot of pre-strain data. So, to make sure the test going smoothly, the use of compression test machine that can support more than 50kN force is highly recommended.

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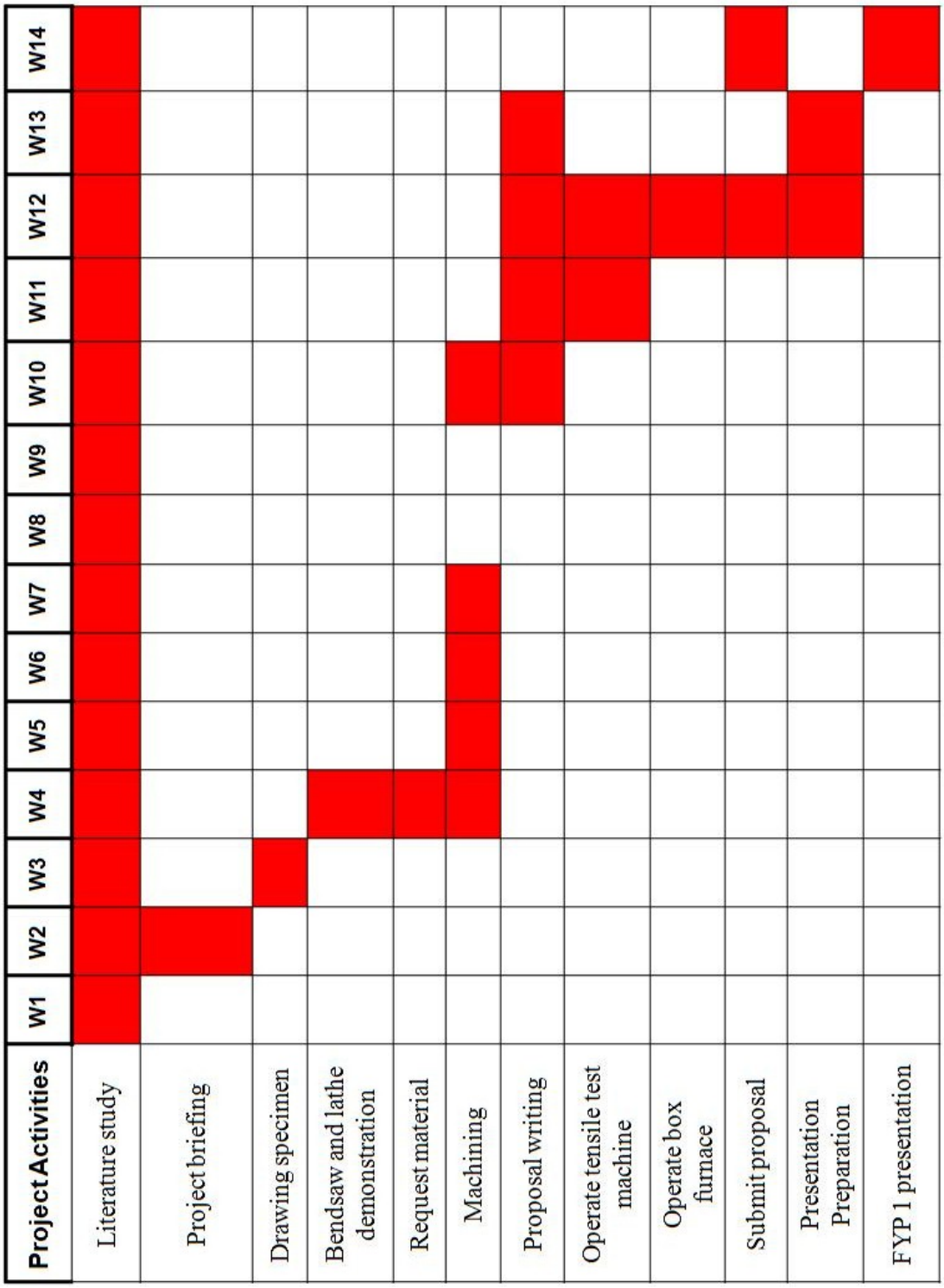
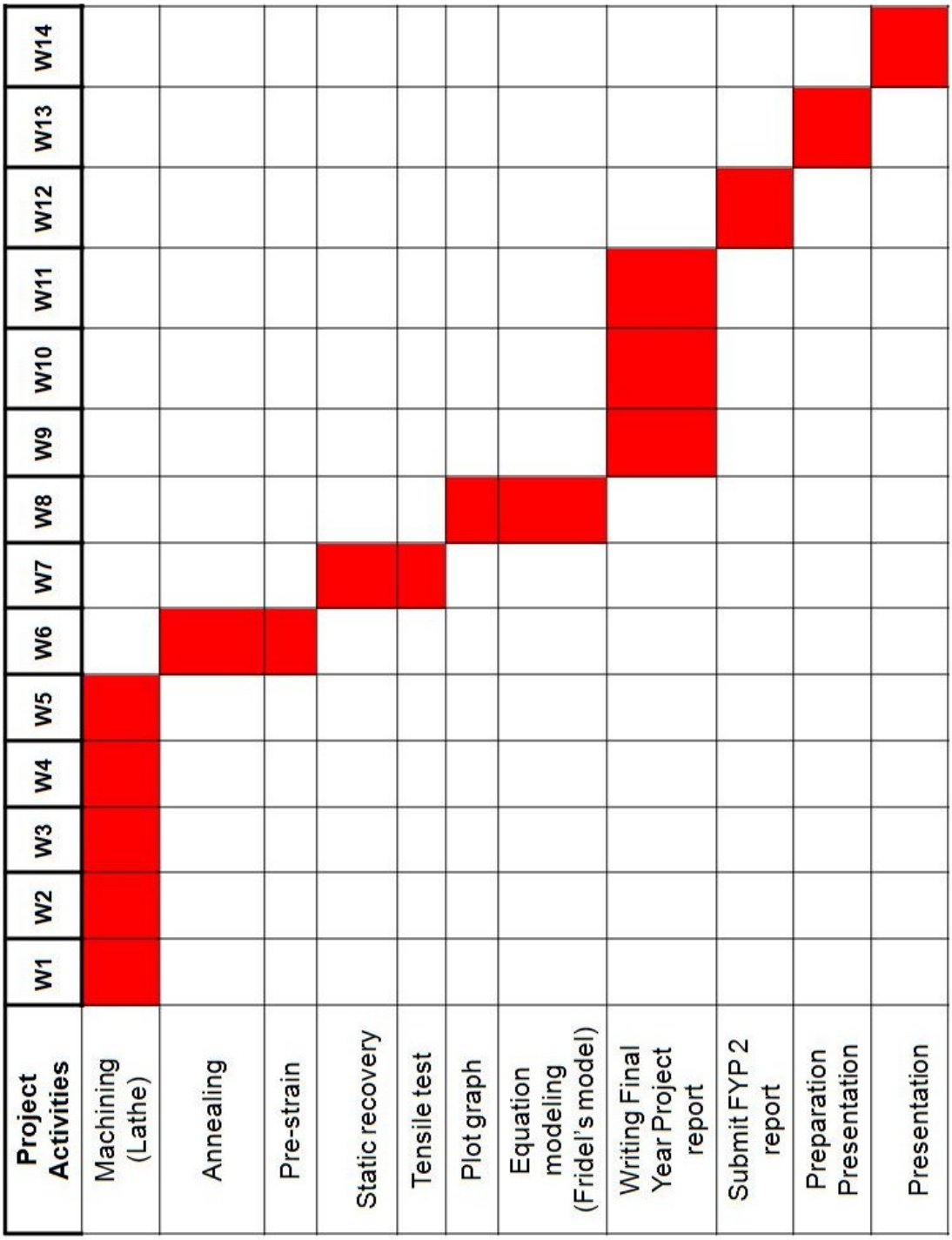
APPENDIX A

(Gantt chart for FYP 1)



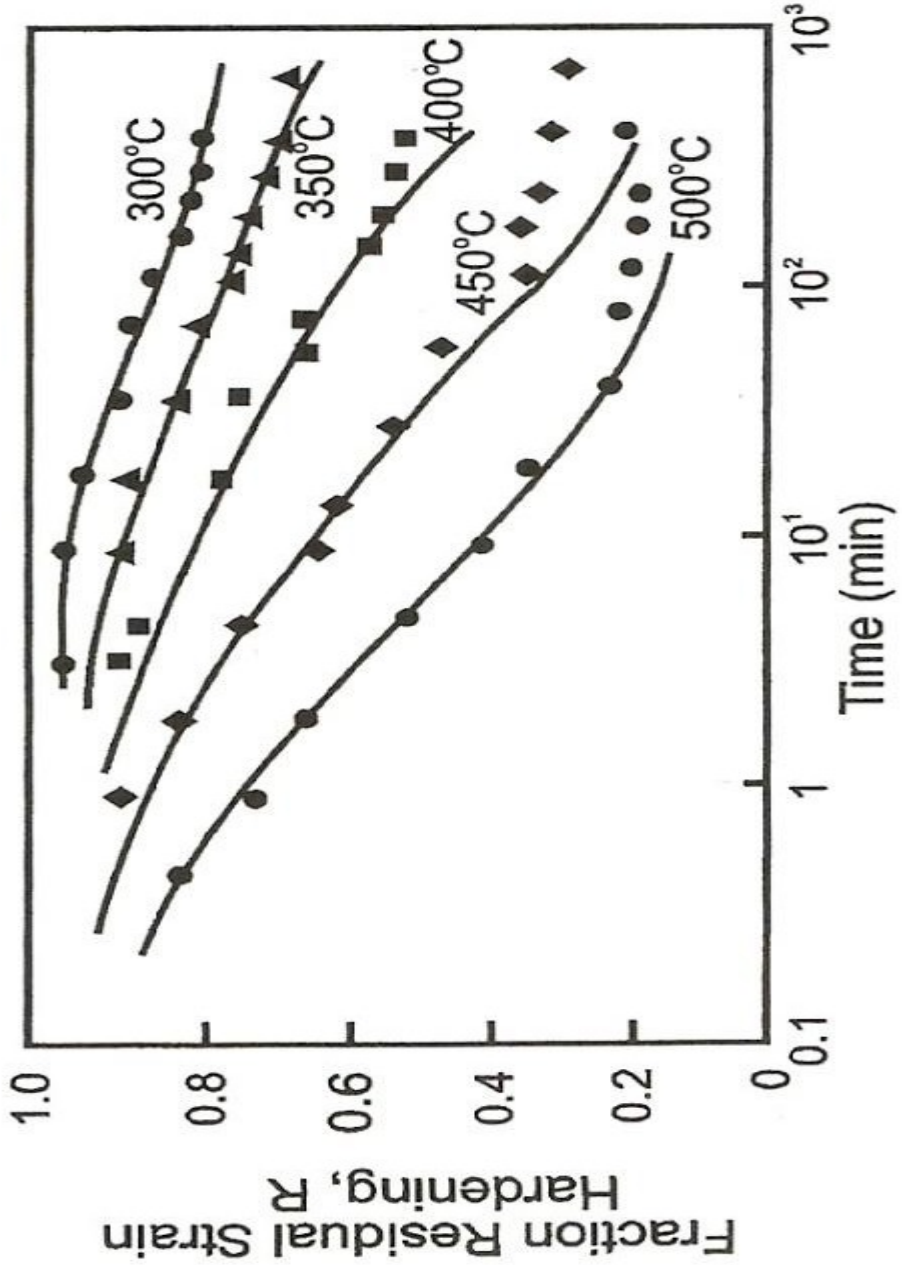
APPENDIX B

(Gantt chart for FYP 2)



APPENDIX C

(Graph proposed by J.T Michalak and H.W Paxton)



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LIST OF SYMBOLS

σ	Engineering stress
ε	Engineering strain
l_i	Original length before any load applied
l_o	The instantaneous length
X_{rec}	Degree of recovery
σ_m	Flow stress immediately before unloading
σ_r	Initial flow stresses recorded during reloading
σ_o	Initial flow stresses recorded during pre-straining
Q	Activation energy
T	Temperature
k	Gas constant
R_y	Fraction of recovery
H_c	The coercive field of the material after an isothermal recovery annealing at fixed temperature
H_c^{def}	The coercive field of the cold rolled material
R	Fraction of residual stress

LIST OF ABBREVIATIONS

C	Carbon
Mn	Manganese
Si	Silicon
Cr	Chromium
Ni	Nickel
ACI	Alloy Casting Institute
AISI	American Iron Steel Institute
ASTM	American Society for Testing and Materials