CHARACTERIZATION AND MODELING OF STATIC RECOVERY PROCESS OF BRASS (COPPER ZINC) ALLOY

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BORANG	• PENGESAHAN STATUS TESIS
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CHARACTERIZATION AND MODELING OF STATIC RECOVERY PROCESS OF BRASS (COPPER ZINC) ALLOY

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A report submitted in partial fulfillment of the requirements for the award of the degree of Bachelor of Mechanical Engineering

> Faculty of Mechanical Engineering UNIVERSITI MALAYSIA PAHANG

> > NOVEMBER 2008

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 concurrently submitted for award of other degree.

Name: MOHAMAD HAFIZUL HISYAM BIN YAHYA ID Number: MA 06004 Date: 03 NOVEMBER 2008 To my beloved mother and knowledge of human kind

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ABSTRACT

Static recovery process is a process of restoration of material properties back to original state. This process only occurs just below recrystallization temperature. In static recovery condition the dislocation of material is annihilated so the internal stress of material could be released. To obtain fraction of recovery, brass will be pre strain in various amount of value before go through annealing process in various static recovery temperature. This experimental process starts with machining the specimens by using manual lathe machining. Then all the specimens will be annealed in recrystallization temperature which is 570°C. Next process is pre strain in various pre strain performed by compression and tensile test machine. After that process, specimens will be annealed in recovery temperature which is below 290°C. Then final pre strain will be performed to obtain yield strength of recovered material. Finally based on the experimental data Friedel's model is used to acquire activation energy. From the calculation some of the activation energy value could be acquire such as -30 kJ/mol, 516 kJ/mol, 276 kJ/mol and 349 kJ/mol. These values of activation energy are compared with value obtained by using same method that used by Martinez which is in range of 216-357 kJ/mol. From the comparison percent of difference can be acquire. Since this percent of difference could be acquired, the significant value can be obtained and this is how Friedel's model is validated.

ABSTRAK

Proses pemulihan statik merupaka satu proses pemulihan sifat bahan kembali kepada keadaan asal. Pemulihan statik hanya berlaku dibawah suhu penghabluran semula. Pada keadaan pemulihan statik, logam terherot akan dihapuskan supaya tegasan dalaman bahan tersebut dapat dilepaskan. Untuk mendapatkan darjah pemulihan, loyang akan di pra terikan dalam beberapa nilai sebelum disepuh lindap dalam beberapa nilai dalam suhu pemulihan statik. Proses ujikaji ini dimulakan memesin bahan ujikaji dengan menggunakan mesin larik secara manual. Kemudian semua bahan ujikaji akan disepuh lindap pada susu penghabluran semula iaitu pada suhu 570°C. Proses seterusnya ialah pra terikkan dalam beberapa nilai dengan menggukan mesin mampatan dan mesin teikkan. Setelah proses tersebut dijalankan, bahab ujikaji akan disepuh lindap pada suhu pemulihan static iaitu dibawah suhu 290°C. Pra terikkan terakhir akan dijalankan utuk mengetahui nilai kekuatan alah bahan yang telah pulih. Akhir sekali daripada data ujikaji permedelan Friedel digunakan untuk mendapatkan nilai tanaga pengaktifan. Daripada pengiraan nilai tenaga penaktifan dapat diperolehi antaranya ialah-30 kJ/mol, 516 kJ/mol, 276 kJ/mol and 349 kJ/mol. Niali-nilai ini akan dibandingkan dengan nilai yang diperolehi dengan menggunakan cara yang sama yang digunakan oleh Martinez iaitu dalam julat 216-357 kJ/mol. Daripada perbandingan tersebut peratusan pembezaan dapat diperolehi. Oleh kerana peratusan pembezaan dapat diperolehi, nilai yang berkaitan dapat diperolehi dan inilah cara pemoldelan Fridel dapat di buktikan.

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LIST OF SYMBOLS

ε	Strain
$\Delta \sigma$	Stress difference
$\sigma_{_m}$	Yield stress of deformed material
$\sigma_{_o}$	Yield stress of recover material
σ_{r}	Yield stress of undeformed material
X _{rec}	Fraction of recovery
Q	Activation energy
t	Time
R	Gas constant
Т	Temperature
c ₁	Constant
a	Constant
b	Constant

LIST OF ABBREVIATIONS

ASTMAmerican Society for Testing and MaterialsCuCopperZnZincCNCComputer Numerical Control

CHAPTER 1

INTRODUCTION

1.1 Project Background

Mechanical properties of metals can be changed by heat treatment process. This process typically done by combining varied types of mechanical deformation and annealing processes. Static recovery only occurs below recrystallization temperature and involves motion and extermination of point deflects as well as extermination and re arrangement of dislocation. It will result the formation of subgrain and subgrain boundaries. The unique feature of static recovery process is that it does not involve any change in grain structure of cold worked metal, the only changes is the dislocation disarrangement within existing grain. It is proposed that a series of heat treatment experiments are performed brass alloys in order to properly characterize the static recovery process. In this research tensile test will used to determine the difference of stress of static recovery between varies pre-strain specimens. Mathematical model will produced from the static recovery behavior. The mathematical will be useful tool to predict commercial end product mechanical properties.

1.2 Problem Statement

Static recovery is process of grain recovery below recrystallization temperature. Friedel's model states that the degree of recovery depends on the amount of pre strain, time and temperature. It is important to validate this Friedel's model so the manufacturer can easily predict end of the result after recovery process.

1.3 Objective

To validate Friedel's model static recovery process for brass (cooper zinc) alloy

1.4 Project Scopes

This focus area is done based on the following aspect:

- (i) Analyze stress difference using tensile and compression test.
- (ii) Material used only Brass (copper zinc).
- (iii) Pre strain at 2.5% 12.5%.
- (iv) Analyze static recovery.
- (v) Specimens for tensile and compression test machine by manual machining.

CHAPTER 2

LITERATURE REVIEW

2.1 ANNEALING

Annealing is a process of heat treatment used to eliminate the effect of cold working. Annealing in low temperature condition may be used to remove residual stress during cold work process [3]. Annealing also know as stress relief by relieving internal strain from cold work, welding or some fabrication process [2]. Two common practice annealing process is full annealing process annealing. Full annealing involves heating the material to austenite region. For process annealing the material involve heating to a point just below the austenite transition temperature. Upon annealing several processes occur such as there is a large decrease in the number of point defects. There are also dislocations of opposite sign attract and exterminate each other. Then the dislocations rearrange themselves into lower energy configurations. Finally both point defects and dislocations are absorbed by grain boundaries migrating through the material. In this case, there is reduction will occur in the grain boundary area. Figure 2.1 show effect of annealing temperature on strength and ductility of Brass alloy. It's shown that most of the softening alloy occurs during the recrystallization stage. For brasses annealing temperature usually in range of 430-650°C and stress relief usually perform in 1 hour with temperature range of 204-260°C [2].



Figure 2.1 Schematic illustration of effect of annealing temperature on Brass [4].

2.2 COLD WORK

Cold work means mechanically deformation of a metal at low temperature [4]. The amount of cold work is defined by the relative of area changes at deformation area. Normally the amount of deformation defining by percent of cold work [3]:

Percent cold work =
$$\left[\frac{A_o - A_f}{A_o}\right] \times 100$$
 (2.1)

Where A_o is the original cross-sectional area of the metal and A_f is the final cross-sectional area after deformation. When a metal having cold work, many of strain energy expended in the plastic deformation is stored in the metal. The strain energy can be form of dislocations and other form such as point of defect [1]. The density of dislocation can be express as the length of dislocation lines per unit volume (net units of m⁻²). A heavily cold worked alloy can have a dislocation density as high as 10^{16} m⁻² with a much higher hardness and stress [4]. When cold work increases, both yield and tensile strength increase. However, the ductility of the material will decrease and approach to zero. Example of cold work is rolling, forging, extrusion, wire drawing and stamping.



Figure 2.2 A cold worked Brass [4].

2.3 RECOVERY

Recovery is a process that some of physical properties of the materials are recovered. Recovery also states as the finest annealing process because there are no gross micro structural change occur [4]. If a material having a cold work, the microstructures of the material will contain lager number of dislocation. When heat is supply in recovery temperature which the range is just below recrystallization temperature range, internal stress in the material will relief allow the dislocation move from the boundaries of a polygonized subgrain structure as show in figure 2.3. this recovery process are called polygonization and its occur before recrystallization phase [1]. During this recovery process the mechanical properties of the material is not changing because the number of dislocation is not reduced. Recovery also knows as stress relief anneal. Recovery usually improves the corrosion resistance of the material.



Figure 2.3 (a) Deform metal crystal showing dislocation. (b) After recovery heat treatment, dislocations move to small angle grain boundaries [1].

2.4 RECRYSTALLIZATION

Recrystallization is a process of formation of new grain by heat treating a cold worked material. Recrystallization occur when enough heat supply and new strain free grain are nucleated in the recovered metal structure and begin to grow and forming recrystallization structure. Recrystallization will rapidly recovery eliminates residual stress and polygonized dislocation structure. Because quantity of dislocation is greatly reduced, the recrystallization material has low strength but high in ductility [3]. Primary recrystallization occurs by two principal which is an isolated nucleus can expand with deformation grain or original high angle grain boundary can travel into more highly deform region of material. Recrystallization will occur if there is a minimum deformation of the material. If the degree of deformation is small (above minimum) the temperature for recrystallization occurs is higher. To decrease the time for recrystallization the temperature should be increase.

The degree of deformation will affect the final size of grain size. If there are more deformation, the lower annealing temperature for recrystallization and the smaller grain size. Grater amount of deformation needed to produce an equivalent recrystallization if the grain size is big. The purity of material will decrease the recrystallization temperature. Figure 2.4 show recrystallization process occurs in Brass in various times.

The figure 2.4 shown Brass grain after having cold work. Then new grain starts to appear after 3 seconds at temperature 580°C. The process continues after 4 seconds and many more grain appear. The complete recrystallization occurs after 8 second and substantial grain growth occur after 1 hour.



Figure 2.4 (a) A cold worked Brass. (b) After 3 s at 580°C. (c) After 4 s at 580°C. (d) After 8 s at 580°C. (e) After 1 hour at 580°C. [4]

2.5 STRESS VERSUS STRAIN TESTING

Strain stress testing give basics information on the stiffness, strength and ductility of the material as figure 2.5. Usually the data could get by performing tensile test. From the strain stress graph the parameter could get is show in table 2.6 [2].



Figure 2.5 Stress strain curve. [3]

Table 2.1: Summary of	Stress strain	testing va	lues.
-----------------------	---------------	------------	-------

Parameter	Description
Modulus of elasticity	Used to measure the relative stiffness of the materials
	Design stress should be lower than yield strength to ensure
Yield strength	that a part does not fail by plastic deformation. Shear
	strength could estimate from yield strength.
	The ratio of yield strength to ultimate tensile strength
Ultimate tensile stress	provides an indication of degree of work hardening that has occur.
Percent of elongation	Indicate materials ductility and toughness
Percent of reduction in	Indicate materials ductility and toughness
area	

General shape of the	Area under curve provides a relative indication of material
curve	toughness.

2.6 BRASS (COPPER ZINC) ALLOYS

Brass are widely use in daily application such as automotive radiator, coins, cartridge casing, musical instrument and jewelry [4]. Brasses consist of copper with addition of zinc from 5 to 40 percent of zinc. Solid solution of cooper and zinc called alpha brasses and if percent of zinc up to 40% the form will be in alpha and beta form as shown in figure 2.7 [1]. Yellow Brass usually added with small amount of lead to improve machinability [2].



Figure 2.6 Binary phase diagrams of Copper Zinc. [3]

2.7 FRIEDEL'S MODEL

Friedel's model is an important mathematical equation to determine the extent of recovery, X_{rec} . The fraction of recovery can define as [6]:

$$X_{rec} = \left(\frac{\sigma_m - \sigma}{\sigma_m - \sigma_o}\right) \tag{2.2}$$

Where,

- σ = Yield stress of recovered materials.
- σ_m = Yield stress of deformed materials
- σ_o = Yield stress of undeformed materials

Base on Friedel's model state that there are relationship between amount of recovery, time and temperature. The relationship can be defined as [6]:

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$
(2.3)

Where,

- $C_1 = Constant$
- t = Time(s)
- R = Gas constant (8.314 J K-1 mol-1)
- T = Temperature (K)
- Q = Activation energy (kJ mol-1)

2.8 MODELING RECOVERY KINETIC

The recovery kinetics can be determined using annealing temperature data which there is no relations within the recrystallization. Modeling of the recovery process can simply be determined by using fraction of residual strain versus logarithmic scale of the annealing time. The relationship of the annealing time and fraction of residual strain can be express by [7]:

$$1 - X_{rec} = b - a \ln t \tag{2.4}$$

Where,

 X_{rec} = Fraction of recovery

- a = Constant
- b = Constant
- t = Time (s)

The constants value of a and b could be determined by using square fitting of the experimental data. Note that the value of the a and b is vary according to the annealing temperature. Figure 2.7 shows the relation between fractions of residual strain versus annealing time in logarithmic scale.



Figure 2.7 Evolution of the fraction of residual strain versus function of the annealing time in vary temperature on a logarithmic time scale [7].

By using figure 2.7 the function of each temperature used to determined constant a and b. The constant relation can be express by figure 2.8 and figure 2.9 with constant value the kinetic modeling of the recovery can easily predicted.

Temp	$1 - Y = -b - a \ln t$	0	h
(°C)	$I = A_{rec} = U = U III I$	a	u
300	y = -0.0207x + 0.9592	0.0207	0.9592
400	y = -0.0273x + 0.852	0.0273	0.852
450	y = -0.0296x + 0.777	0.0296	0.777
500	y = -0.0262x + 0.6817	0.0262	0.6817



Figure 2.8 The relation between a and temperatures in degree Celsius.



Figure 2.9 The relation between b and temperatures in degree Celsius.

The rate of recovery can be express as a function of the instantaneous amount of recovery and purposed by Martinez [7]

$$\frac{dX_{rec}}{dt} = a \exp\left(-\frac{b}{a}\right) \exp\left(\frac{1-X_{rec}}{a}\right)$$
(2.5)

Where,

 X_{rec} = Fraction of recovery

- a = Constant
- b = Constant

By using equation 2.5 value a and b will be substitute and graph recovery rate can be plotted. Together with the graph we can get value $\ln (c_1)$ from the intercept of y axis of graph recovery rate versus temperature.



Figure 2.10 The natural logarithm of the instantaneous rate of recovery calculated at various X_{rec} value vs. the inverse temperature.

From the figure 2.10 the value of c_1 will be substitute in equation 2.6. Equation 2.6 is an arrhenius type behavior for the rate of recovery and can be defined as:

$$\frac{dX_{rec}}{dt} = c_1 \exp\left(-\frac{Q_{app}}{RT}\right)$$
(2.6)

Where,

- $c_1 = Constant$
- R = Gas constant (8.314 J K-1 mol-1)
- Q = Activation energy (kJ mol-1)
- T = Temperature (K)



Figure 2.11 The relations between activation energy and fraction of recovery.

The relationship between activation energy can be showed after the value of c_1 substitute in equation 2.6. From this graph show that the activation energy range is about 177.66 - 400 KJ/mol.

2.8.1 Arrhenius Rate Method

The relationship between energy of molecule in a gas had found by Arrhenius who is a Swedish who experimentally studied rate of reaction. The rate of many chemical reaction as a function of temperature ex press by [1]

Arrhenius rate equation: Rate of reaction =
$$Ce^{-Q/RT}$$
 (2.7)

Arrhenius equation (2.7) commonly rewritten in the natural logarithmic as

$$\ln \text{ rate} = \ln \text{ constant} - \frac{Q}{RT}$$
(2.8)

From the figure 2.7 shows the evolution of the fraction of residual strain versus function of the annealing time in varies temperature on a logarithmic time scale [7] a graph relation of time of reaction and inverse temperature. Using this graph, the slope of each reaction in percent will be acquired to obtain activation energy. The relation between slope with activation energy can be describe as:

$$slope = -\frac{Q}{R} \tag{2.9}$$



Figure 2.12 The relations between reaction time and inverse temperature

By using equation 2.9, activation energy could be determined at 60%, 70% and 80%. By using linear equation the activation energy at wanted point could be determined. The relationship of activation energy and fraction of recovery could express by figure 2.13.



Figure 2.13 The relations between activation energy and fraction of recovery
CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

Methodology is one of most important thing to be considered in order to complete any researches. The reasons is because with the methodology its will make the job is following the right step until the research is completed. Methodologies also use to ensure the research follow the objective that had been stated earlier or in other word, to follow the guideline based on the objective.

In methodology, the step or the structure of the research is the most important thing to consider. Methodology also can be described as the structure of the research because it has working steps based on the objective and scopes. With methodology, it is easier to facilitate the works and to view all the working process of the research. If there any problems in the working step it is easy to know the next step to do. The research will go smoother and faster with structured methodology.



3.3 LATHE MACHINING

Lathe machining is the first step of this process. Since the shape of the specimens of both tensile and compression are cylindrical shape lathe machining are be used. The specimen will be machine to standard size for tensile testing. The dimension of specimens is very important to ensure that the result will be significant. But in manual lathe machining it is almost impossible to get precise and consistent dimension, so there is tolerance during lathe machining. The tolerance is +0.5 mm have been chosen for all of the specimens. The shape of specimens must follow standard size and shape and for this experiment the shape and size follow ASTM E8 standard. Figure 3.1 show drawing of tensile test specimens and figure 3.2 show drawing of compression test specimens.



Figure 3.1 Tensile test specimen dimensions.



Figure 3.2 Compression test specimen dimensions.

For tensile test specimens brass will be cut by using band saw cutting before undergo lathe machining. The length for each cut is of specimens is according to dimension which is 160 mm each. After that each specimens will be rough machining until it reach outside diameter 10 mm. Before specimens go through angle cutting process, each of specimens will be marked so it is easy to machining. The angle cutting done by goes through side by side for each specimen.

For compression test specimens, brass will be cut to 380 mm before going to lathe machine. The purpose of this cutting is because if brass bar cut to 25 mm it is hard to machine it until diameter of the specimen reach 15 mm. After been cut by band saw, specimen will go through lathe process until the diameter of the bar reach 15 mm. After that the bar will be cut using band saw in to 25 mm for each cutting. After the cutting, specimens will be chamfered to remove sharp edges. This process will be done using lathe machining.

3.4 ANNEALING THE SPECIMEN

In order to make sure the entire specimen having same strength, the specimen will undergo annealing process. The entire specimens will undergo annealing so all of the specimens will have consistent strength before undergo cold work process. The important apparatus for annealing process is furnace. All the specimens must be place in furnace before annealing process start. To make sure the specimen will heated constantly, the specimens will be placed not above each other.

Annealing temperature for this experiment is 570° C since the annealing temperature of brass alloy is in range of 425-700°C. Annealing process will start with heating until 570°C in constant rate which is 5°C per minute. The time for the heating rate 5°C can be calculated by using equation 3.1. Then there are soaking time which is the specimens will be heated in constant temperature 570°C in an hour before the specimens cooled in furnace atmosphere. Figure 3.3 show annealing graph for brass. Heating time for 5°C rise can express by:

Heating time
$$=\frac{C-c}{5}$$
 (3.1)

Where,

C = Final temperature

c = Furnace ambient temperature



Figure 3.3 Annealing graph for brass.

3.5 PRE STRAIN

The static recovery only happened in worked material. So it is important step before the specimen annealed in recovery temperature. Before pre strain value is considered the specimen must be tested first. The purpose of the test is to determine the maximum tensile strength or maximum compressive strength. When the value of this parameter determined then the justification of how much pre strain can easily be made. If there are no test, the specimens could fail before can get required amount of pre strain.

There are also could be the machine could not support the required amount of pre strain due to the toughness of the materials. For the brass material the amount of pre strain of the tensile test has been set to 5%, 7.5%, 10% and 12.5% since the maximum elongation for this material using tensile machine is 30%. For the compression test the value of the pre strain is 2.5%, 5%, 7.5% and 8.3% since the compression machine could support the compressive load until 8.3% of strain.

3.6 ANNEALING IN STATIC RECOVERY TEMPERATURE

In this step the specimen will be annealed below the recrystallization temperature of the material. This step is similar to the annealing the specimens step but the difference is this annealing temperature is below 290°C. The reason the annealing temperature below 290°C is because the static recovery is only occur below recrystallization temperature which is the recrystallization temperature for brass is at 290°C.

The purpose of this process is to soften the material so the degree of recovery could be determined. If the pre strain is variable the temperature is constant if temperature, if the temperature is variable the time and pre strain is constant and if the time is variable the temperature and pre strain is constant. For the variable temperature for compression test the temperature is in 50°C difference start with 320°C, 270°C, 220°C and finally 170°C and for tensile test it starts with 300°C, 250°C, 200°C and finally 150°C. The purpose 50°C interval is to see the differential of recovery effect. If the interval is too small it is hard to distinguish the effects. Figure 3.4 show brasses after be annealed.



Figure 3.4 Brasses after annealed in static recovery temperature.

3.7 TENSILE OR COMPRESSION TEST OF THE RECOVERED MATERIAL

This step is important to find the recovered yield stress. In this step the specimen will be tested to find the yield strength of recovered material. For this experiment there are two type of stress versus strain testing used which is compression and tensile test. For both of the testing the strain values have been setup so the yields strength could be determined.

If the value of the strain is too small the yield strength value will not accurate because the materials have not reach plastic deformation. The yield strength will be got by an intercept of offset 0.2 % of elastic deformation with stress versus strain graph. For brasses it is enough to set 5% of strain to get yield strength value by using tensile and also compression method. This value is important to proceed to next process.

3.8 FIND FRACTION OF RECOVERY

The fractions of recovery also know as X_{rec} . It is the degree of softness of the recovered material. The fraction of recovery could be determined using equation 2.2 this equation consist of several parameter such as yield stress of recovered materials, yield stress of deformed materials and yield stress of undeformed materials. The value yield stress of deformed materials and yield stress of undeformed materials could be getting by referring data during pre strain.

During pre strain the machine can show the yield strength of material which is equivalent to yield stress of undeformed materials. For data yield stress of deformed materials it is equivalent to maximum yield strength during pre strain. By referring data tensile or compression test after material is recovered yield stress of recovered materials could be determined. The value of yield stress of recovered materials is equivalent to yield strength of the testing.

3.9 GRAPH PLOTTING

Graph plotting is the most important thing to do because with the graph the data can be interpreted. To interpret the experimental data, several of graphs need to be plotted. Scatter graph is one of the method can be used to view the pattern of the data. By using scatter plot, the gradient, regression and interception of the axis could be view so the data could easily interpret.

For compression test there are several data, to distinguish the data three graph could be plotted. First graph is graph of fraction of recovery versus pre strain. This graph is consist varies pre strain start from 2.5%, 5%, 7.5% and 8.3%. The purpose of this data is to show the relationship between extent of recovery and pre strain. For tensile test there also have is graph of extent of recovery versus pre strain. But the different is the value of varies pre strain is start from 5%, 7.5%, 10% and 12.5%.

Second graph is graph fraction of recovery versus temperature. In this graph the constant value is annealing at recovery temperature time and amount of pre strain which is an hour annealing time and 5% pre strain. The graph plotted is fraction of recovery versus inverse temperature. For compression test varies of temperature is start from 170°C, 220°C, 270°C and 320°C. For tensile experiment the value of temperature is start from 150°C, 200°C, 250°C, 300°C.

Third graph is fraction of recovery versus annealing in static recovery temperature time. The constant temperature of compression is annealing in static recovery temperature and amount of pre strain which are 280°C and 5% of pre strain. For tensile test experiment annealing in static recovery temperature and amount of pre strain which are 230°C and 5% of pre strain

To compare the activation energy using Martinez method a graph of fraction of residual stress versus ln time must be created. The value of fraction of residual stress could get by one minus fraction of recovery. Then the gradient and y axis intercept relation plotted versus annealing in static recovery temperature plotted. This relation is named as a and b. By using this relation a graph of recovery rate versus inverse of temperature could be plotted before graph of relation of activation energy with the fraction recovery could be made.

3.10 FRIEDEL'S MODELING

With the result of the tensile and compression test Friedel's equation will be used to modeling and characterization of static recovery process of the brass. To modeling and characterization of static recovery process of the brass first is to find the activation energy based on experimental value.

There are two way to find activation energy which is by using graph fraction of recovery versus time and graph fraction of recovery versus inverse of temperature. To

find activation energy by using graph fraction of recovery versus time there are trendline linear type need to be added to the graph. From the linear equation of the trendline the gradient value is equal to c_1 and the intercept of y axis is equal to activation energy divide by gas constant and temperature. The relation can describe by figure 3.5.

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

$$y = mx + c$$

Figure 3.5 Relation of Friedel's model with linear equation on graph fraction of recovery versus time.

By using graph fraction of recovery versus inverse of temperature the value of activation energy could be determined. The approach to find activation energy is similar to find activation energy by using graph fraction of recovery versus time. The relation can describe by figure 3.6.

$$X_{rec} = C_1 \ln t - \left(\frac{Q}{R}\right) \left(\frac{1}{T}\right)$$
$$\uparrow \qquad \uparrow \qquad \uparrow$$
$$y = c + mx$$

Figure 3.6 Relation of Friedel's model with linear equation on graph fraction of recovery versus inverse of temperature.

After the value of activation energy could determined by using Friedel's model the result of activation energy determined by using Friedel's model will be compared with the result using Martinez method. The reason is to know the percent of difference between those methods.

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

In this chapter the result from the testing using tensile and compression test machine will interpret. The data have been converting to scatter graph and added with trendline to get the linear equation. By using the linear trendline, activation energy could be determined by comparing it with Friedel's model.

To validate Friedel's model the value of activation energy will be compared with Martinez method and the percent of difference will be show to evaluate whether Friedel's model can be validate or not. The relation of the result will discuss in the end of the chapter.

4.2 RESULT

4.2.1 Results for Compression Test on Recovery Vs Time

The relation between fraction of recovery and time had been describe as figure 4.1 and table 4.1 show the data from the experiment. For this relation there are eight specimens have tested using compression test machine. Each pair of specimens annealed to varies time from two hour until five hour. In this experimental constant temperature 230°C and pre strain is used.

The result of the experiment used to validate Friedel's model. If the value of the result is unreasonable the result will not used in data plotting. The data from the graph will substitute in Friedel's model to find the activation energy of the material in this condition. After the data interpret, by using Friedel's model the value of activation energy for this material is -30 kJ/mol.

X _{rec} vs.	X _{rec} vs. Times (5% pre strain / 280 Degree Celsius)							
	Times (Hour)	$\sigma_{_0}$	$\sigma_{\scriptscriptstyle M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}	In Times (s)		
Exp1	2	126.9	215.1	143.2	81.51927	8.8818363		
Exp2	2	123.5	231	146.4	78.69767	8.8818363		
Exp1	3	122.7	220.1	139.9	82.34086	9.2873014		
Exp2	3	128.5	230.8	130.8	97.75171	9.2873014		
Exp1	4	127.1	222	141.1	85.24763	9.5749835		
Exp2	4	121.6	222.5	154.1	67.78989	9.5749835		
Exp1	5	86.8	235.2	130.8	70.3504	9.798127		
Exp2	5	116.1	227.3	129.1	88.30935	9.798127		

Table 4.1: Data Recovery vs. Times at 280°C and 5% pre strain



Figure 4.1 Graph fraction of recovery versus time at 280°C and 5% pre strain.

Below is the step to calculate activation energy by using Friedel's model and the data from the graph.

We know that

$$\mathbf{X}_{rec} = \mathbf{C}_1 \ln t - \frac{\mathbf{Q}}{\mathbf{R}\mathbf{T}}$$

From the graph we get

$$y = 8.5302 + 6.4392$$

The relation from the equation:

$$X_{rec} = y$$
; $C_1 = 8.5302$; $\ln t = x$; $\frac{Q}{RT} = 6.4392$; $R = 8.314$ J K-1 mol-1

Q = -30 kJ/mol

4.2.2 Results for Compression Test on Recovery Vs Temperature

The relationship of the fraction of recovery with the temperature can be described by figure 4.2. The variable temperature is used to show the relationship of fraction of recovery and temperature. The range of temperature selected is increment of 50° C start from 170° C until 320° C. Data of this graph is developing base on eight specimens by using compression test. Figure 4.2 is a graph of fraction of recovery versus variable inverse temperature. The purpose the x axis of the graph 4.2 in inverse temperature is because with this unit, data from the graph can be used in Friedel's model.

The graph 4.2 is created based on experimental data from table 4.2. From the graph 4.2, the linear equation will be used in determining activation energy. The linear equation will compare with Friedel's model. From the comparison the activation energy for this relation is 276 kJ/mol.

X _{rec} vs.	X _{rec} vs. Temperatures (5% pre strain / 1 Hour)								
	Temperature (°C)	$\sigma_{_0}$	$\sigma_{_M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}	$\frac{1}{T}(K)$			
	(-)								
Exp1	170	124.1	227.5	187.8	38.39458	0.002257			
Exp2	170	132.9	220.8	176.2	50.73948	0.002257			
Exp1	220	133.2	223.6	185.4	42.25664	0.002028			
Exp2	220	134.3	233.7	172	62.07243	0.002028			

Table 4.2: Data Recovery vs. Temperatures at 5% pre strain in 1 Hour

Exp1	270	132.32	233.7	172	60.86013	0.001842
Exp2	270	134.8	238.2	168.2	67.69826	0.001842
Exp1	320	125.5	218.6	170.1	52.09452	0.001686
Exp2	320	140.9	220.6	164.77	70.05019	0.001686



Figure 4.2 Graph fractions of recovery versus temperature in an hour and 5% pre strain.

Below is the step to calculate activation energy by using Friedel's model and the data from the graph.

We know that

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

From the graph we get

$$y = -33234x + 120.44$$

The relation from the equation:

$$X_{rec} = y$$
; $C_1 \ln t = 120.44$; $\frac{Q}{R} = -33234$; $\frac{1}{T} = x$; $R = 8.314$ J K-1 mol-1

So we can get

$$Q = 276 \text{ kJ/mol}$$

4.2.3 Results for Compression Test on Recovery Vs Pre Strain

The value of fraction of recovery is based three elements which is the recovery temperature, annealing time and amount of pre strain. There are eight specimens have been tested to show the relation between various pre strain with constant value of annealing time and recovery temperature. On this experimental the amounts of pre strain have been setup to 2.5%, 5%, 7.5% and 8.3%.

This experimental is conduct by performing compression test to find the stress difference. The data show that with increase of amount of pre strain the fraction of recovery is increase. There is limitation to Friedel's model which is it cannot show in its equation the relationship of the amount of pre strain with the fraction of recovery. Since the linear regression of this experimental is very far from one, the reliability of the data can't fully be used.

Tab	le 4.3:	Data	Recovery	/ VS.	Times a	t 280°C	and 5%	pre strain
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X _{rec} vs. Pre-Strain (280 Degrees Celsius/ 1 Hour)							
	Pre-Strain	$\sigma_{_0}$	$\sigma_{_M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}		
Exp1	0.025	124.2	147.2	131.3	69.13043		
Exp2	0.025	114	165	137.9	53.13725		
Exp1	0.05	135.9	224.4	166.1	65.87571		

Exp2	0.05	125	223.9	164.7	59.85844
Exp1	0.075	132.9	261.5	187.6	57.46501
Exp2	0.075	134.8	262.8	180.9	63.98438
Exp1	0.083	153.8	267.7	193.5	65.14486
Exp2	0.083	156	290.6	193.5	72.13967



Figure 4.3 Graph fraction of recovery versus pre strain at 280°C in an hour.

4.2.4 Results for Tensile Test on Recovery Vs Time

Figure 4.4 show relation between fraction of recovery and time. The graph from figure 4.4 is based on table 4.4 which is data from the experiment. For this relation there are eight specimens have tested using tensile test machine. Each pair of specimens annealed to varies time from two hour until five hour. In this experimental constant temperature 230°C and pre strain is used. The result of the experiment used to validate

Friedel's model. If the value of the result is unreasonable the result will not used in data plotting.

The data from the graph will substitute in Friedel's model to find the activation energy of the material in this condition. After the data interpret, by using Friedel's model the value of activation energy for this material is 349 KJ/mol. In this case it shows if the annealing temperature is decrease the activation energy is increase. This statement is based on comparison with activation energy done by compression test.

X _{rec} vs. 7	X _{rec} vs. Times (5% pre strain / 230 Degree Celsius)							
	Times (H)	$\sigma_{_0}$	$\sigma_{\scriptscriptstyle M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}	In Times (s)		
Exp1	2	138.5	242.4	223	18.6718	8.881836305		
Exp2	2	150.9	255.6	239.1	15.75931	8.881836305		
Exp1	3	144.8	247.5	219	27.75073	9.287301413		
Exp2	3	155.4	261.5	243.9	16.58812	9.287301413		
Exp1	4	130.7	233.9	194.8	37.8876	9.574983486		
Exp2	4	159.4	266	299.9	-31.8011	9.574983486		
Exp1	5	153.2	258.4	242.3	15.30418	9.798127037		
Exp2	5	151.3	256.5	220.8	33.93536	9.798127037		

Table 4.4: Data Recovery vs. Pre-Strain at 230°C and 5% pre strain.



Figure 4.4 Graph fraction of recovery versus time at 230°C and 5% pre strain.

Below is the step to calculate activation energy by using Friedel's model and the data from the graph.

We know that

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

From the graph we get

$$y = 11.448x - 83.436$$

The relation from the equation:

$$X_{rec} = y$$
; $C_1 = 11.448$; $\ln t = x$; $\frac{Q}{RT} = -83.436$; $R = 8.314$ J K-1 mol-1

Q = 349 KJ/mol

4.2.5 Results for Tensile Test on Recovery Vs Temperature

The relationship of the fraction of recovery with the temperature can be described by figure 4.5. The variable temperature is used to show the relationship of fraction of recovery and temperature. The range of temperature selected is increment of 50° C start from 150° C until 300° C. Data of this graph is developing base on eight specimens by using tensile test. Figure 4.5 is a graph of fraction of recovery versus variable inverse temperature. The purpose the x axis of the graph 4.5 in inverse temperature is because with this unit, data from the graph can be used in Friedel's model.

The graph 4.5 is created based on experimental data from table 4.5. From the graph 4.5, the linear equation will be used in determining activation energy. The linear equation will compare with Friedel's model. From the comparison the activation energy for this relation is 276 kJ/mol. From the graph shows if the inverse temperature increase, the fraction of recovery is decrease but the value using tensile and compression test is different. The difference is the value of fraction of recovery done by compression test is higher than tensile test.

Table 4.5: Data Recovery vs.	Temperatures	at 5% pre	strain in 1	Hou
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X _{rec} vs. T	X _{rec} vs. Temperatures (5% pre strain / 1 Hour)							
	Temperature	$\sigma_{_0}$	$\sigma_{_M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}	$\frac{1}{T}(K)$		
Exp1	150	142.6	249	244.4	4.323308	0.002364		
Exp2	150	151.6	257.5	256.7	0.75543	0.002364		

Exp1	200	159.8	264	246.5	16.79463	0.002114
Exp2	200	146	247.9	235.6	12.07066	0.002114
Exp1	250	140	244.5	220.2	23.25359	0.001912
Exp2	250	141	245.6	226.9	17.87763	0.001912
Exp1	300	155.1	263.8	212.9	46.82613	0.001745
Exp2	300	152.4	255.6	212.7	41.56977	0.001745



Figure 4.5 Graph fractions of recovery versus temperature in an hour and 5% pre strain.

Below is the step to calculate activation energy by using Friedel's model and the data from the graph.

We know that

$$X_{rec} = C_1 \ln t - \frac{Q}{RT}$$

From the graph we get

$$y = -62096 + 146.73$$

The relation from the equation:

$$X_{rec} = y$$
; $C_1 \ln t = 146.73$; $\frac{Q}{R} = -62096$; $\frac{1}{T} = x$; $R = 8.314$ J K-1 mol-1

So we can get

$$Q = 516 \text{ kJ/mol}$$

4.2.6 Results for Tensile Test on Recovery Vs Pre Strain

From experimental data using compression test show with increase of amount of pre strain value of fraction of recovery is increase. This experiment is used to compared data with previous experimental result which is performing using compression test.. There are eight specimens have been tested to show the relation between various pre strain with constant value of annealing time and recovery temperature. On this experimental the amounts of pre strain have been setup to 5%, 7.5%, 10% and 12.5%. This experimental is conduct by performing tensile test to find the stress difference.

The data show that with increase of amount of pre strain the fraction of recovery is decrease. There is limitation to Friedel's model which is it cannot show in its equation the relationship of the amount of pre strain with the fraction of recovery. Since the linear regression of this experimental is very far from one, the reliability of the data can't fully be used. Since the linear regression of this data is higher from experimental using compression test, the reliability of this data is higher than data performing using compression test.

X _{rec} vs. P	X _{rec} vs. Pre-Strain (230 Degrees Celsius/ 1 Hour)							
	Pre-Strain	$\sigma_{_0}$	$\sigma_{\scriptscriptstyle M}$	$\sigma_{\scriptscriptstyle R}$	X _{rec}			
Exp1	0.05	139.1	242.1	222.5	19.02913			
Exp2	0.05	101.3	208.6	225.7	-15.937			
Exp1	0.075	158	292.9	247.3	33.80282			
Exp2	0.075	137.5	272.6	242.4	22.35381			
Exp1	0.1	142.4	302.3	286.4	9.943715			
Exp2	0.1	140.8	297.8	261.8	22.92994			
Exp1	0.125	92.15	262.6	310.9	-28.337			
Exp2	0.125	142.9	321.4	296.4	14.0056			

Table 4.6: Data Recovery vs. Pre-Strain at 230°C in 1 Hour



Figure 4.6 Graph fraction of recovery versus pre strain at 230°C in an hour.

4.3 MODELING RECOVERY KINETIC

This type of modeling is used by Martinez to show relationship between activation energy with fraction of residual stress. Based on data fraction of recovery versus time, graph fraction of residual strain has been defined as figure 4.7. Equation 2.4 is used to develop parameter a and b which is dependent to temperature. This parameter is show as figure 4.8 and figure 4.9.



Figure 4.7 Evolution of the fraction of residual strain versus function of the annealing time in vary temperature on a logarithmic time scale of brass.



Figure 4.8 The relation between a and temperatures in degree Celsius.



Figure 4.9 The relation between b and temperatures in degree Celsius.

Figure 4.8 and 4.9 show that the effect of temperature on both parameter and b is decrease with an increment of temperature.



Figure 4.10 The natural logarithm of the instantaneous rate of recovery calculated at various X_{rec} value vs. the inverse temperature of brass material.

Rate of recovery can be express as a function of the instantaneous amount of recovery in the way used by Martinez is equation 2.5. This equation 2.5 can be used by add the substituting value a and b in equation 2.5. Using data by substituting a and b value a graph natural logarithm of instantaneous rate of recovery versus inverse temperature can be created. Figure 4.10: show the graph of natural logarithm of the instantaneous rate of recovery calculated at various X_{rec} value vs. the inverse temperature of brass material.

The graph on figure 4.10 can be used to determine activation energy of the material at difference fraction of recovery. Using graph on figure 4.10 value of activation energy could be determined. Value $\ln c_1$ could be read based on y axis

interception of graph in figure 4.10. By substituting those value, equation 4.5 and equation 4.6 can be solve using simultaneously equation to get the activation energy of the material.



Figure 4.11 The relations between activation energy and fraction of recovery of brass.

After equation 2.6 solves graph relationship between activation energy versus fraction of recovery can be plotted. Figure 4.11 show relations between activation energy and fraction of recovery of brass. In this graph the value of activation energy is in range of 216 -357 kJ/mol. Its show with increasing of fraction of recovery the activation energy is decreasing.

The activation energy value is more consistent by using same method that Martinez used. Value activation energy by using Friedel's model will compared with activation energy by using Martinez method. It will show the percent of difference and conclusion can be made based on the comparison. From the results for compression test on recovery versus time, the activation energy for this experimental procedure is -30kJ/mol. The percent of differences results for compression test on recovery versus time can calculate as,

$$\frac{216 - (-30)}{216} \times 100 = 114\%$$

Furthermore the results for tensile test on recovery versus temperature show the activation energy for this experimental procedure is 516kJ/mol. The percent of differences results for tensile test on recovery versus temperature can calculate as,

$$\frac{516 - 355}{516} \times 100 = 45\%$$

Since the percent of difference of the results for compression test on recovery versus time and the results for tensile test on recovery versus temperature are too high, the value of activation energy by using Friedel's model for this type of experiment is unreliable. However the activation energy of results for compression test on recovery versus temperature and results for tensile test on recovery versus time is in range of the result by using Martinez method. It means this type of experiment has proved the validation of Friedel's model.

The high difference of percent of activation energy is occur because there is error occur during the test. These errors occur because of several factors such as type of machine use, specimens' preparation method and calibration of the machine. This factor is contributed to all errors. By eliminate this factors more consistent data could be acquire.

CHAPTER 5

CONCLUSION

5.1 CONCLUSION

The purpose of this study is to validate Friedel's model static recovery process of brass (copper zinc) alloy due to varies pre strain. Through this study the behavior of brass (copper zinc) alloy under static recovery process could be predicted by using Friedel's model. The behavior of brass (copper zinc) alloy under static recovery process is dependent on several factors such as amount of pre strain, annealing temperature and annealing time.

During the study there are several data are obtain such as the data for compression test on recovery versus time, the data for tensile test on recovery versus temperature, data for compression test on recovery versus temperature and data for tensile test on recovery versus time. Through this data Friedel's model is used to find the activation energy of all this data. Data for compression test on recovery versus time give result to activation energy is -30kJ/mol and the data for tensile test on recovery versus temperature show the activation energy is 516kJ/mol. The other data that acquire from this study is data for compression test on recovery versus temperature which is give result on activation energy 276kJ/mol and data for tensile test on recovery versus time show the activation energy is 349kJ/mol.

Comparing the result of compression test on recovery versus time and the result for tensile test on recovery versus temperature, found that the value of activation energy of these result is insignificant because there are lot of different in percentages value if comparing with activation energy by using Martinez method. However the data from compression test on recovery versus temperature and tensile test on recovery versus time give more significant value because it is in the range of value obtain by using Martinez method.

5.2 **RECOMMENDATION**

Significant data in important in every study because with significant data it show the study is in the right path. From this study there are two insignificant values obtain after comparing with other value. These insignificant may cause by errors during experimental procedure. To get more reliable value these are some recommendations:

- (i) All specimens prepared by using CNC machine
- (ii) Only one type of furnace used which is having automatic temperature controller.
- (iii) Test must execute just after specimen reach room temperature to prevent other factor that could change the result of recovery process.
- (iv) The machine must be calibrate and maintenance more frequently to prevent errors during stress strain analysis.
- (v) The higher capability of compression machine must be used to support higher size of material and accuracy. The machine may have extensometer to give more accurate strain value.

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APPENDIX A

Drawing of tensile test specimen



APPENDIX B



Drawing of compression test specimen

APPENDIX C

-	Temperature 300°C			Temperature 400°C			
1-Ry	Times (s)	In t	1-Ry	Times (s)	In t		
0.95	3	1.098612	0.83	3	1.098612		
0.92	6	1.791759	0.81	6	1.791759		
0.9	10	2.302585	0.78	10	2.302585		
0.88	50	3.912023	0.75	50	3.912023		
0.85	120	4.787492	0.7	120	4.787492		
0.83	900	6.802395	0.68	900	6.802395		
0.8	2000	7.600902	0.64	2000	7.600902		
0.79	4000	8.29405	0.63	4000	8.29405		
0.78	6000	8.699515	0.6	8000	8.987197		
0.77	8000	8.987197	0.59	10000	9.21034		
0.76	10000	9.21034	0.59	14000	9.546813		
0.75	25000	10.12663	0.6	25000	10.12663		
0.74	50000	10.81978	0.55	50000	10.81978		

Data collected from Martinez journal

-	Temperature 4	50°C	Temperature 500°C							
1-Ry	Times (s)	In t	1-Ry	Times (s)	In t					
0.78	3	1.098612	0.67	3	1.098612					
0.73	6	1.791759	0.64	6	1.791759					
0.69	10	2.302585	0.63	10	2.302585					
0.66	50	3.912023	0.57	50	3.912023					
0.62	120	4.787492	0.53	120	4.787492					
0.58	900	6.802395	0.5	900	6.802395					
0.53	2000	7.600902	0.46	2000	7.600902					
0.51	5000	8.517193	0.45	5000	8.517193					
0.49	8000	8.987197	0.45	8000	8.987197					
0.5	10000	9.21034	0.45	10000	9.21034					
0.49	20000	9.903488	0.45	20000	9.903488					
0.48	35000	10.4631								
0.47	50000	10.81978								
0.47	70000	11.15625								

Temp (°C)	1-Ry=-a Ln t + b	а	b
300	y = -0.0207x + 0.9592	0.0207	0.9592
400	y = -0.0273x + 0.852	0.0273	0.852
450	y = -0.0296x + 0.777	0.0296	0.777
500	y = -0.0262x + 0.6817	0.0262	0.6817

APPENDIX D

Gantt chart final year project 1

PROJECT	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
ACTIVITIES	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Literature review																
Project briefing.																
Specimen drawing.																
Band saw and lathe																
demonstration.																
Request material.																
Specimens																
machining																
Tensile test.																
Annealing																
Proposal writing.																
Submit proposal.																
Presentation																
preparation.																
FYP 1 presentation.																

Gantt chart final year project 2

PROJECT ACTIVITIES	W 1	W 2	W 3	W 4	W 5	W 6	W 7	W 8	W 9	W 10	W 11	W 12	W 13	W 14	W 15	W 16
Report Writing																
Machining Specimens																
Annealing Heat Treatment																
Pre Strain																
Annealing in Recovery Temperature																
Tensile Test (find Δσ)																
Graph Plotting																
Friedel's Modeling																
Submit FYP 2 Report																
Presentation Preparation																
Final Presentation																
APPENDIX E

Picture during final year project





