RDU1603125

DEVELOPMENT OF A HIGH TEMPERATURE VISCOMETER FOR SEMISOLID METAL PROCESSING

(PEMBANGUNAN 'VISCOMETER' BERSUHU TINGGI UNTUK PEMPROSESAN BAHAN SEPARUH PEPEJAL)

IR DR ASNUL HADI BIN AHMAD
DR ZAKRI BIN GHAZALI
DR FADHLUR RAHMAN BIN MOHD ROMLAY
PROF MADYA IR DR HAJI NIK MOHD ZUKI BIN NIK MOHAMED
IR DR MOHD RASHIDI BIN MAAROF
PROF DR MAHADZIR BIN ISHAK@MUHAMMAD

RESEARCH VOTES NO:
RDU 1603125

FAKULTI KEJURUTERAAN MEKANIKAL
UNIVERSITI MALAYSIA PAHANG

2019

ACKNOWLEDGEMENTS

First and foremost, I would like to thank my research fellow which have supports both consciously and unconsciously with their knowledge in order to ensure this research can be completed. I appreciate all their contributions of time, ideas, knowledge, advice, guidance to make this research experience productive and stimulating. The joy and enthusiasm they have for their research was contagious and motivational for me, even during tough times in this research works. I am also thankful for the excellent example, they have provided as a successful researcher.

I would also grateful to all other researcher and industrial expertise for their help especially in materials characterization and experimental set-up. Special thanks also to UMP's Research and Innovation Department for their help in administrative works along this time.

ABSTRACT

This research presents the works on the development of a high temperature viscometer for semisolid metal processing.The objective of the project are to investigate the effects of pouring temperature and holding time on the formation of globular microstructure and to study the rheological behaviour of resulting microstructure by using new developed high temperature viscometer. Direct thermal method was used to produce feedstock billet and simple parallel plate compression viscometry was used to investigate the rheological behaviour of aluminium in term of viscosity. The feedstock billet was prepared by using direct thermal method. There were three different temperatures were used consist of 685℃, 665℃ and 645℃ while for holding time at 60s 40s and 20s. The rheological experiement were conducted by assuming the semisolid metal feedstock billet bahaving as Newtonian fluid. The different of semi-solid aluminium temperatures were used in this works were at 570℃ and 600℃ for rheological behaviour studies. The calculated viscosity number presented the capability of the semi solid metal slurry in filling the mould Furthermore, through parallel compression test presented the required force of semi solid metal slurry for deformation. Direct thermal method resulted, pouring the molten at low temperature close to the liquidus point provided an appropriate formation of globular microstructure. Rheological behaviour of material resulted, the magnitude of viscosity and applied force increases when reduction of semi solid temperature material. Lastly, the lower viscosity results in better movement of material with less machine pressure.

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

1.1 EXECUTIVE SUMMARY

Semi solid metal (SSM) processing is one of the modern casting that combines the advantage of traditional metal process which in liquid state (casting) and solid state (forging). SSM processing has commercially viable technology for production of metallic component with high integrity, improved mechanical properties, complex shape and tight dimensional control. SSM processing offers a great chance to manufacture net-shaped metal components in only one forming operation. In addition, elevated mechanical properties can be achieved because of the unique globular microstructure and flow behaviour within the die. In order to successfully perform semisolid forming process, it is required to establish a critical control on all process parameters, particularly feedstock preparation and forming process.

Semisolid metal (SSM) processing is based on the thixotropic behaviour of alloys with a non-dendritic microstructure in the semisolid state. If the microstructure consists of spheroids of solid or globular grains in a liquid matrix, its viscosity is time and shear rate dependent; if it is sheared it flows and if it is allowed to stand it thickens again. This behaviour leads to laminar rather than turbulent die fill and avoid defects such as porosity and improves mechanical properties. Different of the formation microstructure due to change in pouring temperature and holding time before quenching process is exposed in the behaviour of the SSM product during the application of the external forces. One of the main parameters to characterize the rheological behaviour of semisolid metal slurries is the viscosity and knowledge of viscosity is helpful in predicting die filling properties of commercial alloy.

1.2 PROBLEM STATEMENT

In comparison with cast aluminium alloys, wrought aluminium alloys have better mechanical properties and offering a wider application. There has been a strong demand to produce to near net shape components with aluminium 7xxx series alloys, which are mainly machined from the wrought state involving considerable waste. Semisolid forming can be a potential alternative near-net-shaping technology. The approach in this project is to first produce a starting material of 7075 alloy which is treated such that the microstructure consists of globular grains in a matrix of liquid phase. Direct Thermal Method (DTM) will be used for preparing the feedstock material.

A major problem for semisolid forming has been the lack of fundamental understanding of the rheological behaviour and the flow of alloys in the semisolid state that impairs our ability to simulate, optimize the processing and improve the repeatability. In order to achieve a deep understanding of rheological behaviour of wrought aluminium alloys, a fundamental study would be performed to develop a share rate-viscosity relationship in the semisolid state of the wrought aluminium 7xxx alloys by parallel plate rapid compression viscometer.

1.3 OBJECTIVES OF THE RESEARCH

a) To develop a die for thixoforming operation and form the components
b) To study on rheological aluminium 7075 behaviour produced via semi solid metal processing.
c) To design and fabricate the simple parallel plate compression testing machine for viscosity measurement

1.4 HYPOTHESIS

An important metallurgical characteristic that has significant effect during SSM processing is a fraction solid. The fraction solid determines material flowability and influence microstructure and defect formation. The low viscosity (low fraction solid volume) component helps material to flow inside die cavity. Meanwhile, high fraction solid volume helps to prevent various defects, a finer internal structure and a high quality product. This fraction solid however is found highly depended on material processing parameters. Hence, in order to produce better quality component with the combination of excellent flowability and superior mechanical properties, the fraction solid volume during SSM processing need to be controlled. This can be achieved by executing adequate processing parameters during SSM processing.

CHAPTER 2

LITERATURE REVIEW

2.1 Rheological Behaviour of Semi Solid Metal Processing

Rheology is the science of deformation and flow materials. The relationship between rheology and mechanical properties of the materials is closely related the materials. In discussion before, semi solid metal processing occurs in range of solidus and liquidus temperature, which mean contains both solid phase and liquid phase. The unique microstructure gives semi solid billets distinctive properties depending on the parameter control. The variation of the microstructure formation resulting from different parameter may be characterized by using the rheological test.

Generally, rheology can be divided into two categories, Newtonian fluid and non-Newtonian fluid. Newtonian fluid is defined as that the shear stress of the fluid arising from its flow is linearly proportional to its shear rate. The relation between shear stress and shear rate is clarify as viscosity. Viscosity is constant at a given temperature, independent of shear stress and shear rate. Non-Newtonian is defined as any other fluid that does not follow the Newtonian fluid relationship clarified. Non-Newtonian fluid also termed as thixotropic behaviour. The non-Newtonian fluid model is widely used to study the rheological behaviour of SSM slurries.

There were two broad categories that can be divided in semisolid metal slurries roughly; first a 'liquid-like' which mean contains dispersed solid particles and behave like a fluid under external forces although secondly a 'solid-like' which mean contains of an interconnected solid phase and behaves like a solid, exhibiting a well define yield strength. Therefore, the deformation mechanisms for these two types of slurry are fundamentally different to each other exactly. Semi solid metal slurries with a solid fraction of not exceed 0.6 and a globular solid morphology usually exhibit two unique rheological properties which are thixotropy and pseudoplasticity. Thixotropy is the time vulnerable shear thinning goods meanwhile the pseudoplasticity is where the behaviour of fluids in category of viscosity decreases under shear strain.

One of the main parameters to characterize the rheological behaviour of semi solid metal slurries is the viscosity. Viscosity value is an indication of semi solid materials capability in filling the die cavity during casting operation. Viscosity is equivalent to that of fluidity for liquid metal. A lot of research has already been done for the rheological characterization of the semi solid alloys. It was started by Joly and Mehrabian in 1976, study on the rheological phenomenon in stirred SSM slurries. They found that viscosity value is depends on the volume of fraction solid which viscosity increases with an increase of fraction solid volume. They also divided the rheological phenomenon into three categories which is pseudoplastic, thixotropic and continuous cooling behaviour. The viscosity of SSM slurries depends on the several metallurgical and process parameter including solid friction, particle size, chemical composition, mould characteristic and pouring temperature. From the previous research, it is generally believed that the apparent viscosity at the steady state is influenced by solid fraction and the shearing rate. The viscosity increases with an increasing solid fraction. The increasing is slight at the early stage, while it becomes dramatic at high solid fraction. The viscosity decreases with an increase in shear rate. With the increasing shear rate, the morphology becomes more globular and the liquid trapped in the solid particles is reducing, leading to a smaller solid fraction, resulting in decreasing viscosity.

2.2 Measurement of Rheological Behaviour

The formation of different microstructure due to the change in pouring temperatures is exposed in flow behaviour of the SSM slurries during the application of the external forces. Based on reviewed articles, there are many techniques for measuring the viscosity were proposed by researcher before, rotational viscometry, drop-edge viscometry, extrusion methods and indentation tests. One of the famous technique was used is by parallel plate compression test.

Figure 2.5 shows schematic a simple parallel plate compression test machine. Parallel plate compression test consists of two disk in cylindrical cavity. In this technique, a dead weight is applied on the top surface of SSM billet. Variety of dead weight range was applied during the process and the deformation behaviour of SSM billet was measured by analysing strain variation with time. Based on reviewed articles, the actual designed machine was developed at the University of Quebec at Chicautimi. The machine consists of four main section; pneumatic weight motion control, axle of weight variation, roller bearings to control the straightness of motion and cylindrical resistant heating furnace to keep the temperature of billet constant.

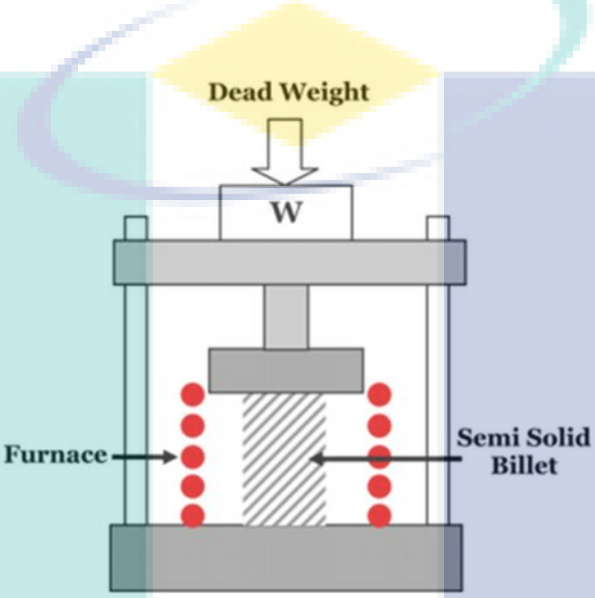

Figure 0.1 Schematic diagram of parallel plate compression test

Data acquisition system via a load cell was installed on the machine to record the data of the process. The load cell indicates the actual applied force which may be slightly different to the selected dead weight due to the friction or any other factor. The variation of billet height and applied load are registered against time to plot the strain-time graph. The resulted strain-time graph is further treated mathematically to characterize the viscosity. Assumption of semi solid slurries at early stage between Newtonian fluid and non-Newtonian fluid should be clearly clarified. If applied shear rate of less than $0.01(s^{-1})$, the resulted could be treated similar to the Newtonian fluid equation and vice versa for non-Newtonian fluid.

According to Newton's first law, the applied shear stress is gives as:

$$F = \frac{3\eta V^2}{2\pi h^5}\left(\frac{dh}{dt}\right)$$

For a Newtonian Fluid the average shear rate, γ_{av} at any instant during compression is given by equation below:

$$\gamma_{av} = \sqrt{\frac{V}{\pi}}\left(\frac{dh/dt}{2h^{2.5}}\right)$$

Where $v_x, \eta, V, h_0, h, F\ and\ t$ are deformation speed, viscosity$(Pa.s)$, volume of specimen (mm^3), initial height (mm), instantaneous height(mm), applied dead force (N) and deformation time(s).The viscosity is then calculated as the inverse slope of the graph.

If the semi solid slurry treated as a non-Newtonian fluid, the results are interpreted in term of the power law, relating to the shear stress to average shear stress. Based on the equation below, equation non-Newtonian fluid generally valid for $h \ll d$. m and n in the equation are the consistency factor and power index respectively material constant.

$$\tau = m(\gamma)^n$$

$$\frac{h_0}{h} = \left\{1 + \left(\frac{3n+5}{2n}\right) h_0^{(n+1/n)} kt\right\}^{2n/(3n+5)}$$

$$k = \left\{\left(\frac{2n}{2n+1}\right)^n \frac{4F}{\pi d^{n+3} m}(n+3)\right\}^{1/n}$$

Overview of result based on researcher before, presented in figure 2.6. The graph shows of strain-time graph obtained by Lashkari and Ghomashchi. They illustrate the effect of microstructure due to the different of superheat on visco-plastic behaviour of semi solid billet. Based on the graph, the globular microstructure has lower viscosity values than those of rosette and dendritic morphologies from higher superheats.

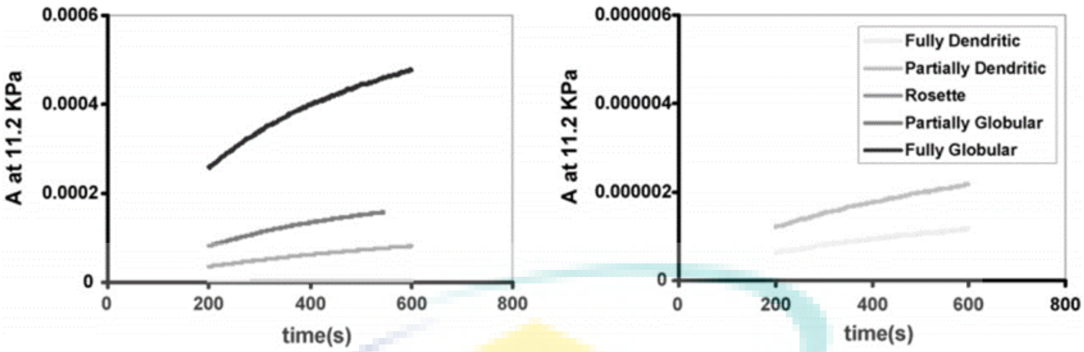

Figure 0.2 steady state part of strain-time graph at initial pressure 11.2 kPa

2.3 Direct Thermal Method for Feedstock Billet Preparation

Semi solid metal processing requires a binary micro-structure in which the primary phase is approaching a spheroidal (globular or non-dendritic) microstructure. Traditionally this is often achieved by stirring the alloy within the mushy state. Direct thermal method is an alternative method in rheocasting process for feedstock preparation. In direct thermal method, solidification condition control via active thermal management to yield non-dendritic solid in liquid matrix. Basically, direct thermal method is a process in which liquid alloy of low superheat is poured into cylindrical metallic mould of high thermal conductivity and low thermal mass.

Direct thermal method was introduced by University College Dublin in 2002. Direct thermal method process involves pouring low superheat liquid alloys into a cylindrical mould. At the early stage contact between molten metal and mould wall there is quick heat absorption from the melt to provide multiple nucleation. As a result of these conditions, the molten alloy cools very slowly at a very low rate by losing heat to the atmosphere to reach an equilibrium temperature below the liquidus (solidification range) of the alloy. Heat-matching between alloy and mould results in a pseudo-isothermal hold within the solidification range of the alloy, made possible by the very low rate of heat loss to the environment.

Direct thermal method is ideal for laboratory experiment environment due to low cost needed and limited size of billet used. The latest experiment done by using direct thermal method process is by A. Ahmad et al. (2014). In that experiment, different of pouring temperature and holding time before quenching process was used to analyse development of microstructure and Figure 2.7 shows the schematic diagram of experimental procedure of by using DTM method. Results of experiment shown different of formation microstructure was appear in the each of feedstock billet by referring on the Figure 2.8 with the different of properties.

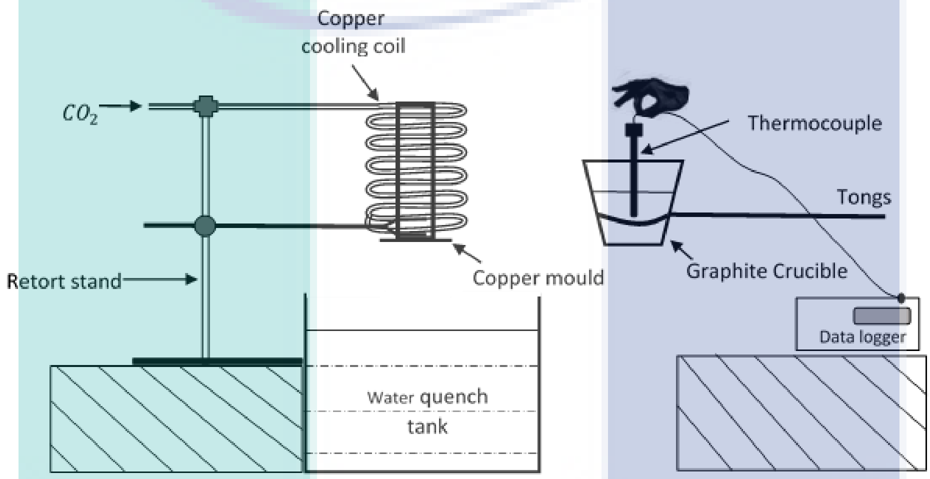

Figure 0.3 Schematic diagram of experimental procedure of direct thermal method

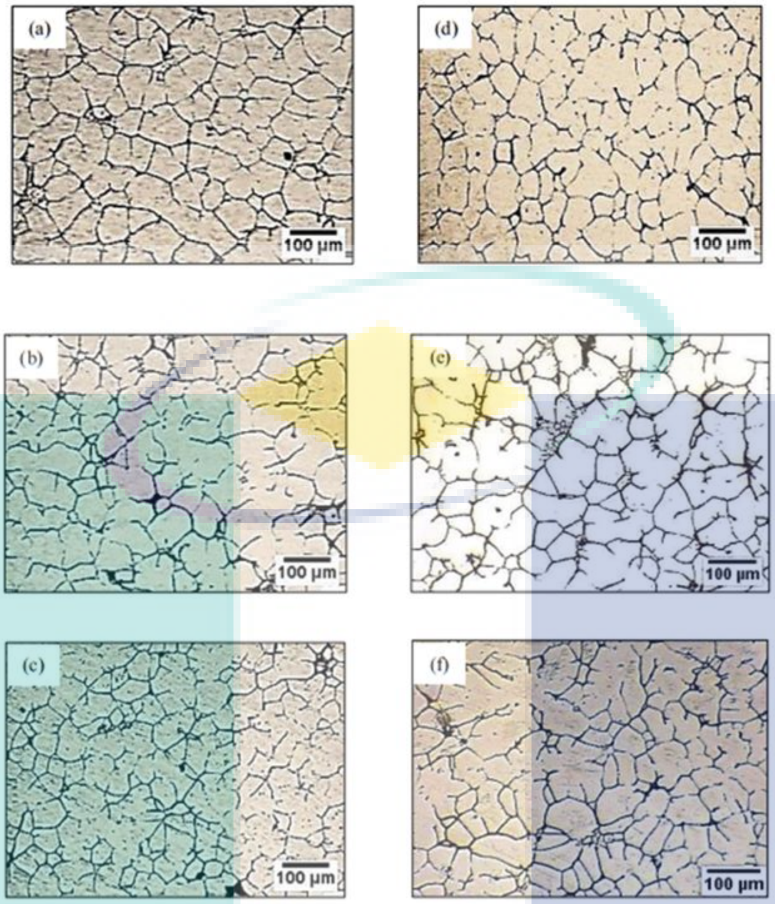

Figure 0.4 Microstructure for different pouring temperature and holding time with (a) 685°C/60s (b)685°C/40s (c) 685°C,20s (d)665°C/60s (e)665°C/40s (f)665°C/20s

Based on the result of experiment, there are several factor can be considered as control parameter to determine the formation of microstructure which are pouring temperature and holding time.

2.4 Pouring Temperature

Pouring temperature is where the temperature of molten alloy needed to raised up to a specific temperature before being poured into mould for cooling. Pouring temperature also must be taking care carefully of the effect that lead to heat loss. This can due to the transfer of alloy through ladles as long as the distance between the furnace and mould. Another reason that lead to heat loss was due to the heat absorbed by ladles. So, if the distance ladles were too long it may lead to the specific temperature have been set drop down and may give an effect to its properties after fully solidify.

Table 2.1 the parameter used among researcher that have been study about the parameter and the effect given by the parameter. A suitable pouring temperature can give a unique morphology of a materials. Lashkari and Ghomashchi has been studies the effect of pouring temperature on the morphology of aluminium A356. Based on the experiment, they found that the lower pouring temperature, the smallest the structure and more globular will achieved. Figure 2.9 shows the different of formation structure of alloy between two pouring temperature.

Researcher	**Pouring temperature, T_p (℃)**			
A.H.Ahmad	680	665	660	630
O.Lashkari	695	675	645	615
D.J.Browne	700	660	640	-

Table 0-1 pouring temperature parameter used among reseacher

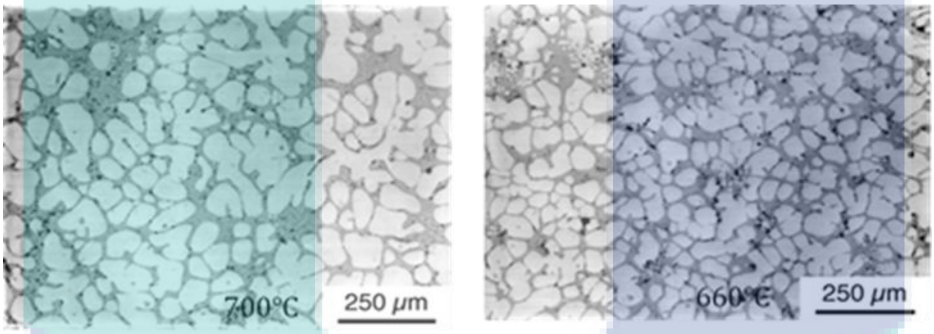

Figure 0.5 Different of formation structure of alloy between two pouring temperature.

2.4 Holding Time

Within exact pouring temperature and holding time, in can influence to the globular structure making. Existing research recognizes the critical role played by lower pouring temperature and holding time somehow affected the creation of globular structure Z. L. Ning, H. Wang, and J. F. Sun, 2010 have studied on deformation behaviour of semisolid A356 alloy prepared by low temperature pouring. In their research, they have been proved that by using of finite element, the prediction of the heat distribution has successfully success work during solidification progress. Big range of casting parameter was used in that research which were in range of 30 minutes to 10 minute.

In 2014, A.H Ahmad have done research on the combination between pouring temperature and holding time parameter in range of not exceeding 1 minutes. Table 2.2

below shows the different combination of pouring temperature and holding time used. The different of that lead to different of outcome for morphology of aluminium 7075. The result describes that the lower pouring temperature with the lower holding time given the microstructure viewed be most globular and smallest structure can be achieved.

Pouring temperature, T_p **(℃)**	**Holding time,** t_h **(s)**
685	60
	40
	20
665	60
	40
	20

Table 0-2 Diffrent of pouring temperature and variable holding time used

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, the method of organizing the project has been discussed. It involves specific component used in the experiment, tools, and measurement collector. The framework of flow process has been clarified clearly as in order to make sure the project will smoothly progress and objective of the experiment can be achieved.

3.2 Flow Chart

Methodology was constructed based on the scope of project as a guiding principal to formulate this project successfully and achieve the objective. Experiment from oldest researcher before has been used as a references and guider to get the procedure of the experiment process. The terminology of the work and planning for this project was shown in the flow chart Figure 3.1. This is very important to make sure that the experiment in the right direction.

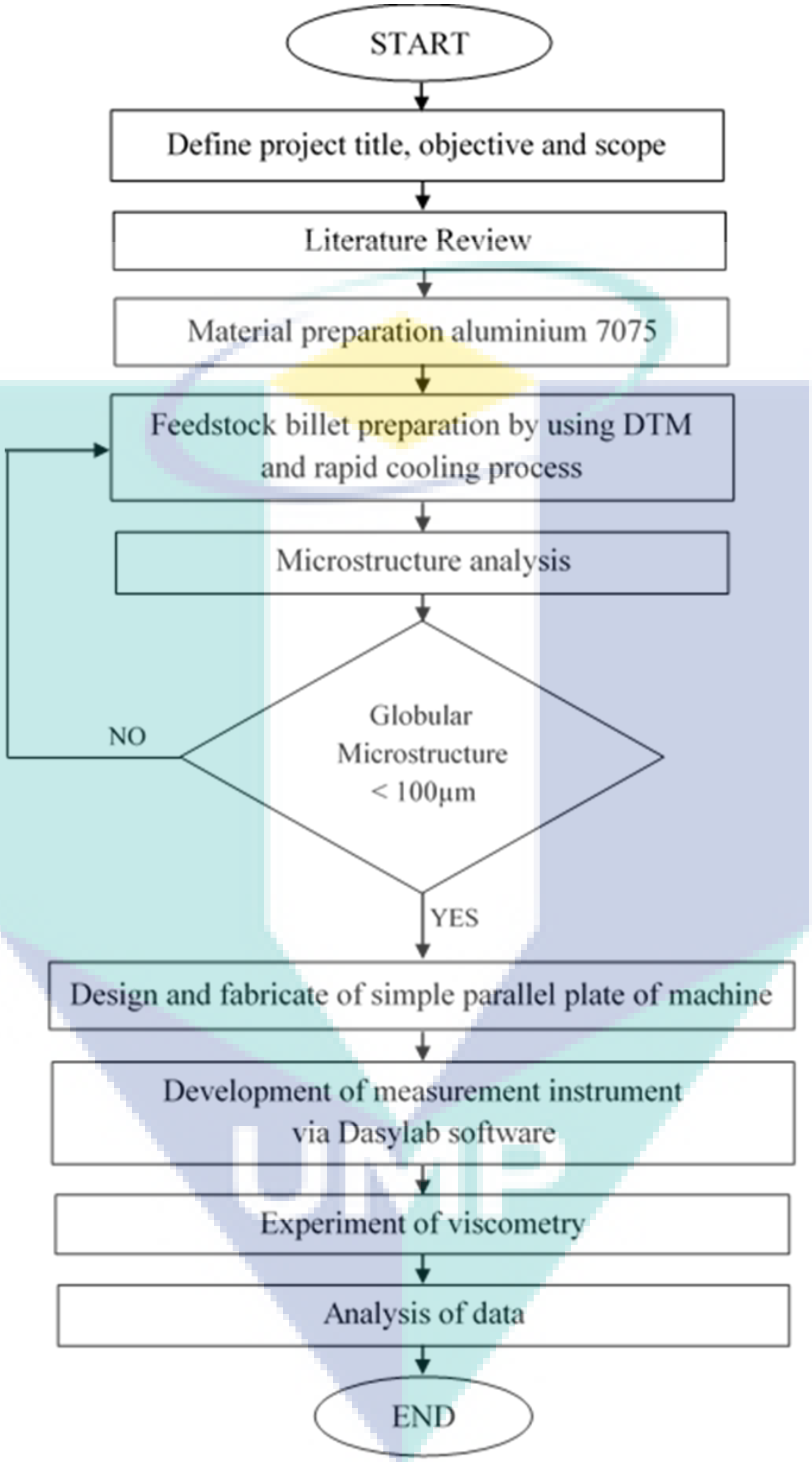

Figure 0.1 Flow chart of process

3.3 Material Preparation

In this study, aluminium 7075 is used as the basic material of the study. Aluminium 7075 is used because this aluminium has a lot of use inside industry in transport applications, including marine, automotive and aviation. Besides, due to their high strength-to-density ratio 7000 series alloys, it also lead to use in rock climbing equipment, bicycle components, inline skating-frames and hang glider airframes.

In the early stages, this aluminium was obtained from the factory at standard conditions ie in aluminium rods measuring $25mm$ for diameter and $2m$ for length. Next, this aluminium need be cut using a disc cutter machine to a shorter size which is $100mm$ and $50mm$. In this way, aluminium will be incorporated into the graphite crucible container for the melting process inside the furnace. Figure 3.2 shows the aluminium rods obtained from the factory and figure 3.3 shows a graphite crucible container used in this study.

Figure 0.2 As received aluminium 7075

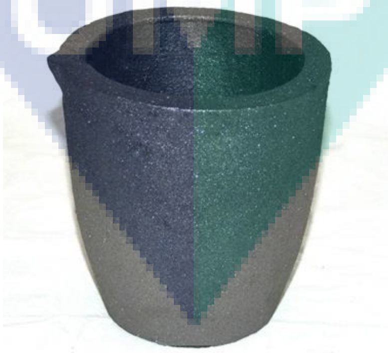

Figure 0.3 Graphite crucible container

There are some important things to be prepared and done in feedstock billet preparation. For example, the preparation of copper mould, fabrication of copper cooling coil for the rapid cooling process and the arrangement of tools used to ensure the process can be done with success.

The copper material used as a mould for aluminium billet is because of the copper has a high conductivity of heat transfer rate compared to other materials. Indirectly, this will accelerate the hardening process or heat release of aluminium to form of globular microstructure itself. Table 3.1 shown the value of conductivity between the differences of material selection and aluminium have the highest value of conductivity.

Material	Conductivity value at 20℃
aluminium	$\mathbf{3.5 \times 10^7}$
Iron	$\mathbf{1 \times 10^7}$
Carbon steel	$\mathbf{1.43 \times 10^7}$
Stainless steel	$\mathbf{1.45 \times 10^6}$

Table 0-1 Conductivity of material at 20 ℃

Two types of copper are used to produce the mould, copper plate with 1mm thickness and copper tube with $27mm$ outer diameter and $1mm$ thickness. Copper plate is used as base plate. The copper plate cut into smaller shapes and according to the size of the set is $50mm \times 50mm$. In the meantime, for copper tube, it will be cut at a length of $120mm$ each. This set of measurements is based on a study conducted by previous researchers and taking into account the areas to be used in this study. Figure 3.4 shown the dimension of copper mould.

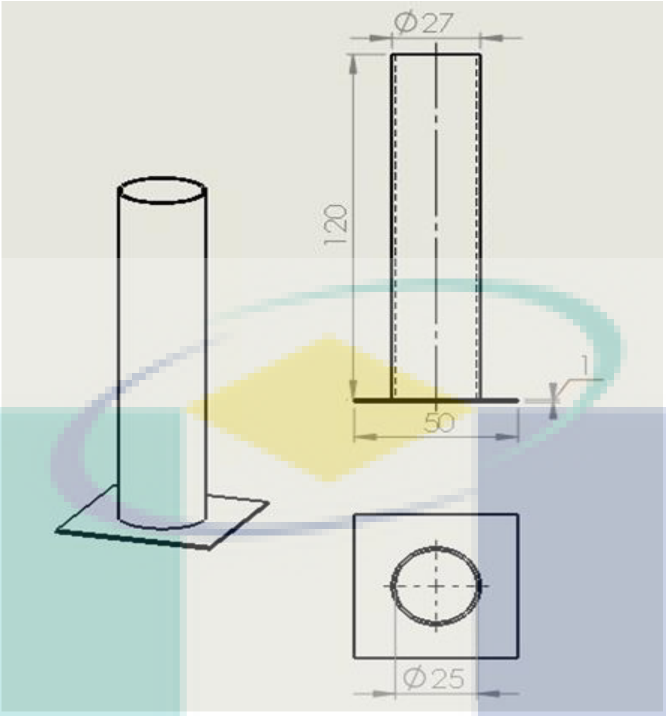

Figure 0.4 schematic dimension of copper mould

Next, joining the process between copper plate and copper tube is done using brazing method. Brazing is one of the most commonly used methods for copper materials. Generally, brazing is a metal-joining process in which two or more metal items are joined together by melting and flowing a filler metal into the joint. Figure 3.5 shown the fabrication of mould by using brazing process.

Figure 0.5 Fabrication of copper mould by using brazing process

Next, for fabrication of cooling coil, copper tube with diameter $3/8$" inch or $9.53mm$ was used. The detail of cooling coil dimension shown in figure 10. High of cooling coil is

$140mm$ m and it contain of 5 coil of “O” channel. The distance between the coils is $20mm$ and each coil has 7 holes opposite copper mould. Fabrication of cooling coil takes a long time because it involves many steps that need to be done to form the "O" channel. Among them are $90°$ elbow of copper tube and also T-joint copper. Figure 3.6 and figure 3.7 shown the copper cooling coil that have been fabricate. The whole process of connecting the parts involved in the fabrication of copper cooling coil is using the brazing method.

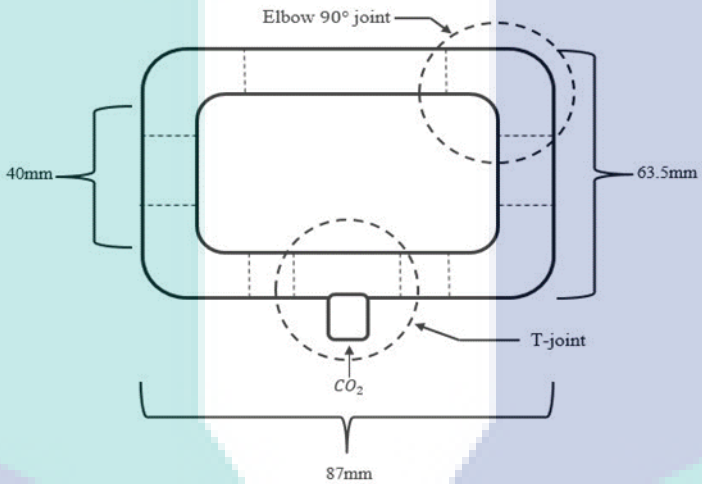

Figure 0.6 Top view schematic design of copper cooling coil

Figure 0.7 Fabricated of copper cooling coil

3.3 Feedstock Billet Preparation via DTM and Rapid Cooling Process

Based on previous studies, there are several ways to produce feedstock billet and direct thermal method is one of them. Direct Thermal Method (DTM) is a technique which involves pouring the molten alloy into a cylindrical copper mould to produce a globular microstructure. In the early stages, the provision of tools used was systematically arranged so that the DTM process ran smoothly.

Figure 3.8 shown the schematic of DTM apparatus set up. Based on the perimeter used in Table 3.2, there are some important tools to use. Among them are K-type thermocouple sensors to measure the temperature of material, data loggers as data collector, and timer to measure the holding time of cooling process. Carbon dioxide gas, CO_2 as medium for rapid cooling process and channeled through copper cooling coil which has been made.

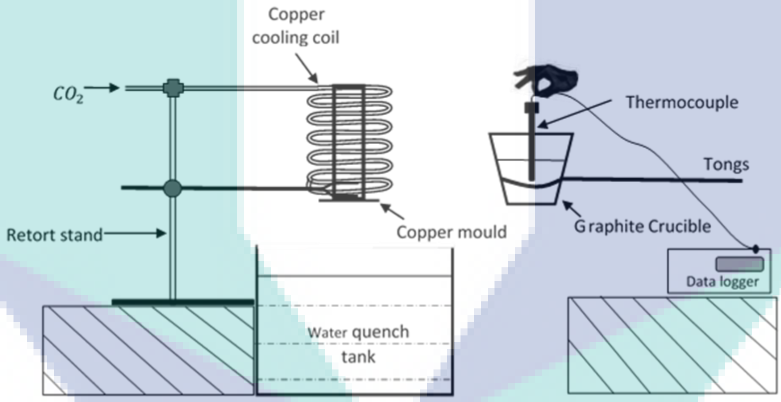

Figure 0.8 Schematic of direct thermal method process

No. billet	Temperature, ℃	Holding time, s
1	685	60
L	685	40
3	685	20
4	665	60
5	665	40
6	665	20
7	645	60
8	645	40
9	645	20
10	685	without quenching

Table 3.2: Parameter of the feedstock billet with different of pouring temperature and holding time

Graphite crucible is used as a container for inserting aluminum ingot to be melted in the furnace. 4 ready-cut of aluminium 7075 ingots were placed in the graphite crucible in the early stages. It will be heated in the furnace until the aluminum ingot changes to the liquid phase above melting point temperature. The furnace temperature starts at room temperature and is heated to 900˚C. The graphite crucible is removed when the furnace temperature at around 850˚C and the K-type thermocouple is used to take aluminium molten temperature readings. Figure 3.9 shown the process of measurement temperature inside the crucible.

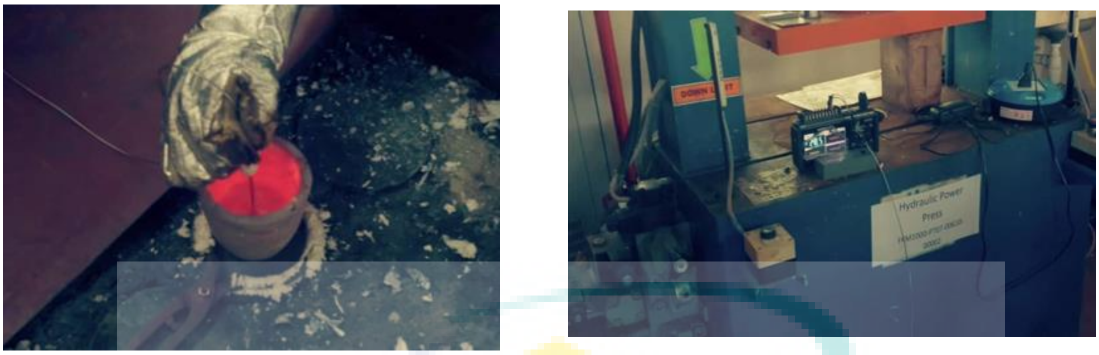

Figure 0.9 Measurement of temperature by using K-type thermocouple

Meanwhile, the copper mould is burned on the inside using two mixture of materials between the graphite powder and the liquid thinner. This is to facilitate billet processing after being cooled. This process should be done with caution and minimum mixture content. This is to prevent graphite from mixing with aluminum liquids when in the mould.

Next, the molten aluminum temperature is measured before it is poured into copper mold. As a precautionary step, the temperature set forth in this study will be added at a rate of 10℃to ensure the exact temperature of the molten aluminium during the process of pouring into the copper mould. For example for a billet sample 1, the pouring temperature for molten aluminium is at a temperature of 695˚C. In this study, there were difference of pouring temperature which are 675˚C for 665˚C, 655˚C for 645˚C and 695˚C for 685˚C. Pouring process is done quickly and well trained.

CO_2 gas is continuously discharged through copper cooling coil to the mold after the pouring process is completed as a rapid cooling process. The time for the discharged gas will be recorded based on the perimeter set at each temperature, holding time. The holding time set is 60s, 40s and 20s at each temperature. 60s holding time has been set for billet sample 1. After that, the mould will continue to be inserted into the water for the quenching process. The water used for this quenching process should be in room temperature.

Aluminium billets will be removed from the mould. The aluminium billet length is measured to ensure that the billet reaches the specified size of $120mm$ and $25mm$ for diameter. If there is a defect on the billet such as shrinkage, the DTM process is re-run to form the perfect aluminium billet. Figure 13 shown the feedstock billet of aluminium from the DTM process.

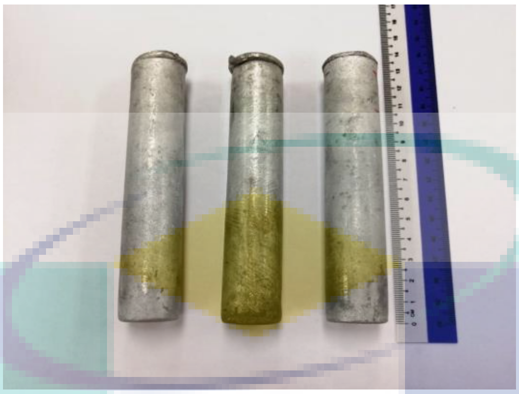

Figure 0.10 Aluminium feedstock billet

3.4 Design of Parallel Plate of Compression Test

Rheological behavior of aluminium 7075 in this project was done by using parallel plate compression machine. Rheology is the science of deformation and flow of materials and to characterize the rheological behavior of semi-solid metal slurries is the viscosity. There are consist of three main components in design of parallel plate compression test which are hydraulic press machine, an upper plate and a based plate.

High heat resistant material was chosen for the parallel plate of machine because of the process needs required high temperature of feedstock billet. Besides, material that have chosen required deformation resistant when heat is applied to make sure able to support the pressure during the compression session. According this parameter, tool steel material was chosen to design and fabricate the parallel plate of machine. Detailed dimensions of parallel plate are taken based on the compression machine used and the size of the sensor was used to measure the force during the experiment. Figure 3.18 shown the dimension of upper plate of machine.

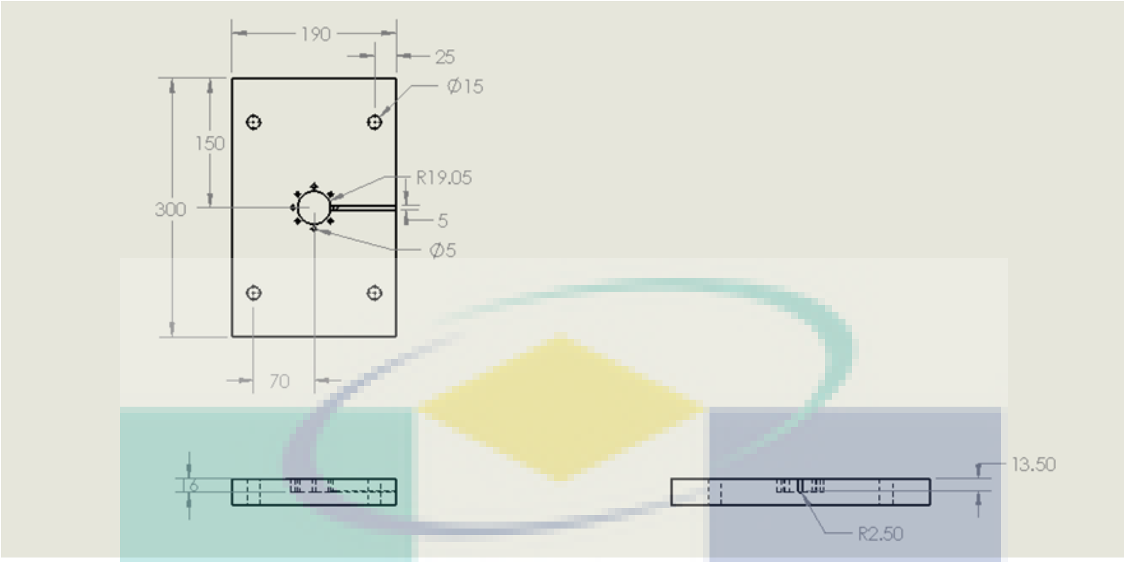

Figure 0.11 Dimension of upper parallel plate design

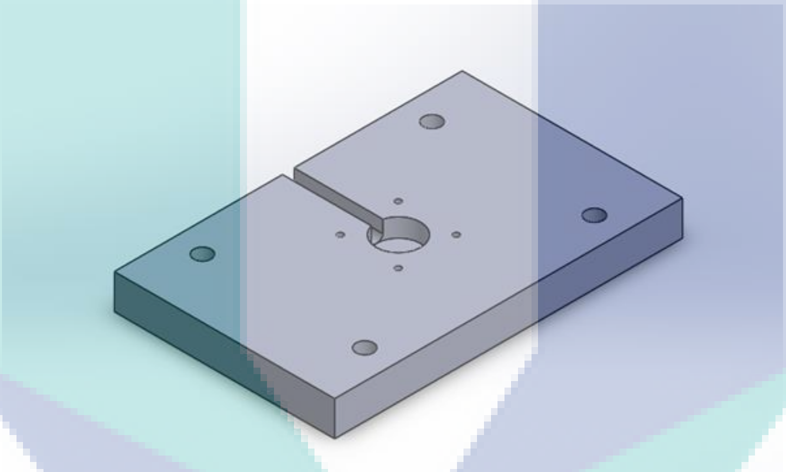

Figure 0.12 Isometric view of upper parallel plate

There is a slot for the sensor load on the upper plate. Load cell sensor is used to take the force measurement applied during the process occurs. The weakness of each sensor is the high temperature resistance. Therefore, a sheet metal with a thickness of 1.5 mm of mild steel material is used to protect the sensor. Upper plate was located at the top of hydraulic machine as shown in Figure 3.20. Upper plate will act pressure on the billet and the base plate at the bottom will withstand.

Figure 0.13 Parallel plate compression machine

Hydraulic press machine used to compress the billet with the aim to measure the force and displacement of billet. The force is applied from the top to the bottom. The operation of machine was controlled by electrical switch panel manually. To measure the displacement, the machine is equipped with a distance sensor and the measurement and controls of sensor are displayed on the control panel.

3.5 Development of Measurement for Viscometry test

Viscosity value in this study is calculated based on the following equation below. The simplest way to analyse the results obtained during compression test is assume the squeezed semi-solid alloy behaving like Newtonian fluid. There are several instrumental instruments required such as load cell sensors to measure force imposed, displacement sensor to measure displacement changes in billet, k-type thermocouple to measure the temperature of billet, and NI 9219 data acquisition, DAQ data logger to record the data. Equation below was used to calculate the magnitude of viscosity value.

$$\left(\frac{3Vh_0}{8\pi P_0}\right)\left(\frac{1}{h^4}-\frac{1}{{h_0}^4}\right)=\frac{t}{\eta}$$

$$P_0=\frac{Fh_0}{V}$$

Whereη, V, h_0, F, P_0 and t are viscosity $(Pa.s)$, volume of specimen (mm^3), initial height (mm), instantaneous height (mm), applied dead load (N), initial pressure and deformation time (s), respectively.

The force versus time data was recorded by using an NI 9219 data acquisition (DAQ). A program has been develop by using dasylab software to control data acquisition rate, and force value. The example of dasylab programming are presented in figure 3.21 and Figure 3.22 The data will be stored in the excel file for analysis to the next level.

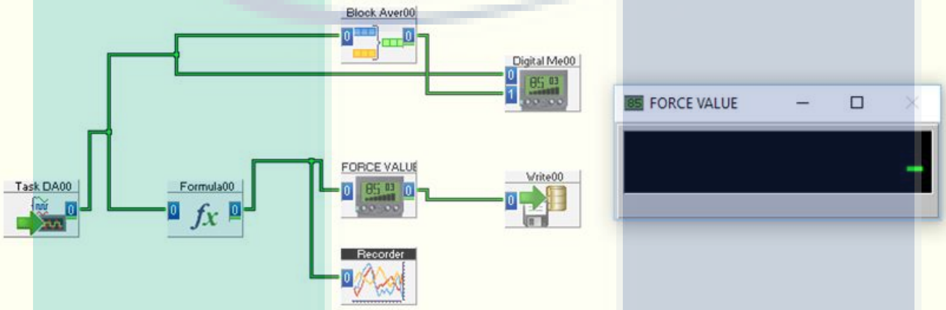

Figure 0.14 Dasylab programming layout

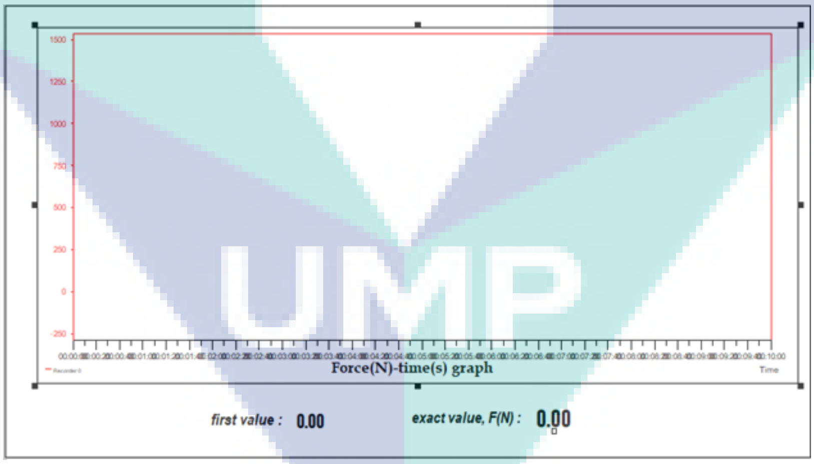

Figure 0.15 Dasylab layout for graph record

3.6 Viscometry Test

There are several ways to study rheological in the specific a viscosity value based on previous researcher and parallel plate compression test is one of them and used in this study. Safety precautions are stressed during the experiment.

Figure 3.23 shown the setup of parallel plate machine. The machine pressure used is at $10bar$ for each replication experiment. In this study, the difference in reheating temperature billet is used as a variable. Displacement billet is measured using displacement sensor on the machine. The initial height of billet in this process is $80mm$.

Figure 0.16 Parallel Plate compression test set up

Furnace is used for the reheating process of feedstock billet to semi solid aluminum temperature and k type thermocouple used to measure temperature. The first step, the furnace will be heated at $830°\mathrm{C} - 850°\mathrm{C}$ before the billet are inserted. The heating process in the furnace takes 6 minutes to prevent the billet from changing to the liquid phase. The billet will be removed from the furnace and the latest temperature reading is taken. Temperatures on the outer surface are recorded at $450°\mathrm{C} - 500°\mathrm{C}$. Next, gas fire torch is used to further heating process to increase temperature of billet. Outer surface of billet temperature is recorded over time. Figure presented the position of billet in the parallel plate of machine before compress. The feedstock billet were compressed uniaxially in a parallel plate machine. The pressure movement is controlled pneumatically with capacity 10 bar. The applied force, resulting displacement of billet and time are recorded.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Viscometry Test

Viscometry test is conducted to study the rheological behavior of aluminium 7075 in semi solid metal processing. The viscosity value is an indication of the capability of the material in filling the mould and to determine the required force for deformation and flow of materials. In this study, the sample number 7 was used because it had a small grain size of globular microstructure. In addition, the sample number 7 is the lowest pouring temperature of 645℃ and the holding time of the old 60s. The billets with more globular and finer particles, resulting due to lowest pouring temperature, render better flow and superior deformation.

The concept of thixo-casting, which is reheating of the cylindrical samples to mushy zone before deformation was the basis of viscometry test in this project. The instantaneous change in billet height, force applied are recorded during the parallel plate compression test to convert into engineering graph. Figure 4.9 represented the force-time graph at temperature of billet at 570℃. The graph shown the force used to form a SSM billet at a temperature of 570℃ is at a rate of 700N up to 800N. Fraction solid billet at temperature 570℃ is at 0.83 which is 83% solid phase. Next, engineering strain, which was termed quasi-steady-state is used for viscosity calculation and the result presented in the Figure 4.10. Three times the repeat of the experiment was done at 570℃ to ensure that the results were equally proportional. The overall resulting data for force applied, displacement height of billet and viscosity value are presented in Table 4.4 and Table 4.5.

Time, s	A	B	C
0.00	0.00	0.00	0.00
1.00	258.13	412.64	249.92
2.00	495.54	615.36	564.18
3.00	658.55	708.44	698.13
4.00	753.26	700.38	729.12
5.00	779.59	726.60	792.81
6.00	700.99	663.74	623.68
temperature	570	570	570
initial height, h_0, mm	80	80	80
displacement, mm	55.14	56.01	56.85
final height, h, mm	24.86	23.99	23.15
diameter billet, mm	25.00	25.00	25.00

Table 0-1 Table analysis of viscometry test

Billet Number	A	B	C
Max. force, N	779.590	726.600	792.810
diameter billet, mm	25.000	25.000	25.000
area of billet, mm^2	4.91E+02	4.91E+02	4.91E+02
volume of billet, mm^3	3.93E+04	3.93E+04	3.93E+04
pressure, Pa (N/m^2)	1.59E+06	1.48E+06	1.62E+06
deformation time, s	5	5	5
viscosity, Pa.s	6.120E-07	7.586E-07	8.032E-07

Table 0-2 Table analysis of viscometry test

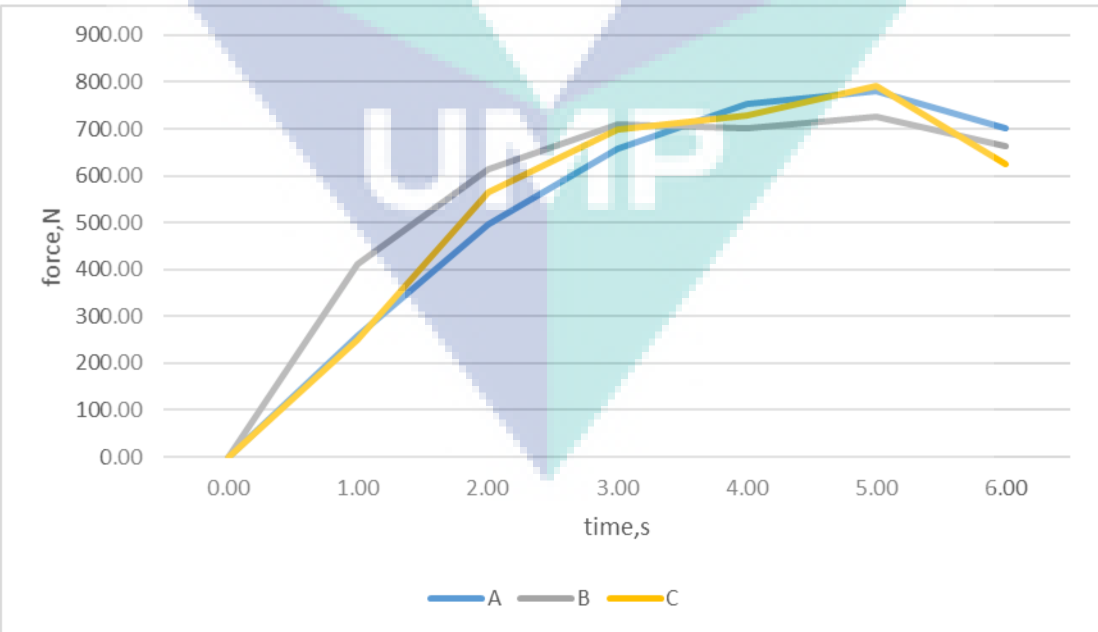

Figure 0.1 Force-time graph

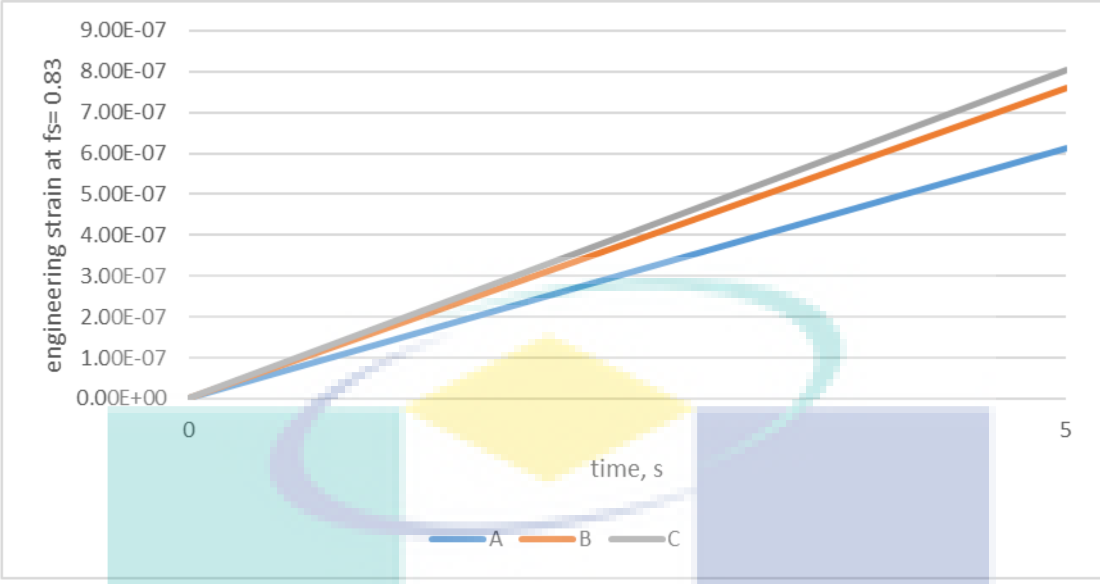

Figure 0.2 Magnitude of viscosity value - time graph

Next, the experiment of viscometry test goes on to two different temperatures on the sample number 7. The set temperature is guided by the fraction solid graph of aluminum that is presented in the Figure 4.11. This study was conducted to investigate the relationship between temperature differences, fraction solid, force applied and viscosity value. Initial pressure set on compression machine was set at 10 bar. The overall resulting data for force applied, displacement height of billet and viscosity value are presented in Table 4.6 and Table 4.7 and Figure 4.12.

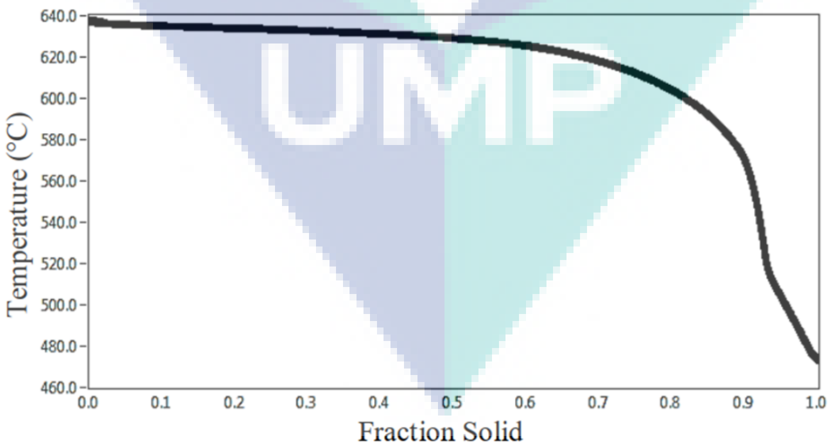

Figure 0.3 Graph of thermal analysis aluminium 7075

Billet sample	A	B
Max. force, N	779.590	575.760
Diameter billet, mm	25.000	25.000
Area of billet, mm^2	4.91E+02	4.91E+02
Volume of billet, mm^3	3.93E+04	3.93E+04
Pressure, Pa N/m^2	1.59E+06	1.17E+06
Deformation time, s	5	5
Viscosity, Pa.s	6.120E-07	1.175E-07

Table 0-3 Table analysis of viscometry test

Billet sample	A	B
Max. force, N	779.590	575.760
Diameter billet, mm	25.000	25.000
Area of billet, mm^2	4.91E+02	4.91E+02
Volume of billet, mm^3	3.93E+04	3.93E+04
Pressure, Pa N/m^2	1.59E+06	1.17E+06
Deformation time, s	5	5
Viscosity, Pa.s	6.120E-07	1.175E-07

Table 0-4 Table analysis of viscometry test

Figure 0.4 Magnitude of viscosity value - time graph

Based on experiment results, the magnitude of viscosity for fraction solid 0.90 is higher than 0.83 and force applied also higher at fraction solid 0.90 which is at 779 N compared to the fraction solid 0.83 at 575N. The magnitude of viscosity increases with increasing fraction solid or reduction of the applied pressure. This indicates a low pressure is required if SSM billets are heated to temperatures close to the liquidus temperature of material. The higher temperature results in lower fraction solid and better deformability and flow ability.

CHAPTER 5

CONCLUSION

Viscometry test is conducted to investigate the capability of the material within the semi solid temperature in filling the mould and to determine the required force for deformation and flow of materials. The magnitude of viscosity value and force aluminium 7075 feedstock billet was recorded in this study through parallel plate compression test. Feedstock billet with combination pouring temperature at 645 and holding time at 60s has been selected for this experiment. This is because based on the previous study, the billets with more globular and finer particles, render better flow and superior deformation. The viscosity values are calculated for the billets by assuming the semi solid aluminium billets behaving as Newtonian fluid respectively. Two semi-solid temperatures were set at 570℃ and 600℃. The experimental results showed, feedstock billet at temperature 570℃ required higher force for deformation compared to the feedstock billet at temperature 600℃. The magnitude of viscosity decrease when the temperature of feedstock billet close to the liquidus point. The magnitude of viscosity decrease with increasing the temperature of billet and required force for deformation will be decrease. The significant finding shows that the lower viscosity resulted a better movement of material through the die with less required force at the temperature of billet close to the liquidus point.

REFERENCES

(Ahmad, Naher, & Brabazon, 2014)Ahmad, A. H., Naher, S., & Brabazon, D. (2014). Direct Thermal Method of Aluminium 7075. *Advanced Materials Research*, *939*, 400–408. https://doi.org/10.4028/www.scientific.net/AMR.939.400

Binesh, B., & Aghaie-Khafri, M. (2016). RUE-based semi-solid processing: Microstructure evolution and effective parameters. *Materials and Design*, *95*, 268–286. https://doi.org/10.1016/j.matdes.2016.01.117

DINSDALE, A. T., & QUESTED, P. N. (2004). The viscosity of aluminium and its alloys — A. *Journal of Materials Science*, *39*, 7221–7228.

Fan, Z. (2002). *Semisolid metal processing The feasibility of SSM processing of various alloys*. *International Materials Reviews* (Vol. 47). https://doi.org/10.1179/095066001225001076

Husain, N. H., Ahmad, A. H., & Rashidi, M. M. (2017). An overview of thixoforming process. *IOP Conference Series: Materials Science and Engineering*, *257*(1). https://doi.org/10.1088/1757-899X/257/1/012053

Kolahdooz, A., & Aminian, S. (2018). Effects of important parameters in the production of Al-A356 alloy by semi-solid forming process. *Journal of Materials Research and Technology*, (x x), 1–10. https://doi.org/10.1016/j.jmrt.2017.11.005

Lashkari, O., Ajersch, F., Charette, A., & Chen, X. G. (2008). Microstructure and rheological behavior of hypereutectic semi-solid Al-Si alloy under low shear rates compression test. *Materials Science and Engineering A*, *492*(1–2), 377–382. https://doi.org/10.1016/j.msea.2008.05.018

Lashkari, O., & Ghomashchi, R. (2007a). A new machine to characterize microstructural evolution of semi-solid metal billets through viscometery. *Materials and Design*, *28*(4), 1321–1325. https://doi.org/10.1016/j.matdes.2006.01.023

Lashkari, O., & Ghomashchi, R. (2007b). The implication of rheology in semi-solid metal processes: An overview. *Journal of Materials Processing Technology*, *182*(1–3), 229–240. https://doi.org/10.1016/j.jmatprotec.2006.08.003

Lashkari, O., & Ghomashchi, R. (2008). Deformation behavior of semi-solid A356 Al-Si alloy at low shear rates: Effect of fraction solid. *Materials Science and Engineering A*, *486*(1–2), 333–340. https://doi.org/10.1016/j.msea.2007.09.009

Lashkari, O., Nafisi, S., & Ghomashchi, R. (2006). Microstructural characterization of rheo-cast billets prepared by variant pouring temperatures. *Materials Science and Engineering A*, *441*(1–2), 49–59. https://doi.org/10.1016/j.msea.2006.05.075

Li, Y.-G., Mao, W.-M., Zhu, W.-Z., Yang, B., & Zhu, D.-P. (2013). Rheological behavior of semi-solid 7075 aluminum alloy in continuously cooling process. *Zhongguo Youse Jinshu Xuebao/Chinese Journal of Nonferrous Metals*, *23*(12).

Nafisi, S., Lashkari, O., Ghomashchi, R., Ajersch, F., & Charette, A. (2006). Microstructure and rheological behavior of grain refined and modified semi-solid A356 Al-Si slurries. *Acta Materialia*, *54*(13), 3503–3511. https://doi.org/10.1016/j.actamat.2006.03.016

Pola, A., Tocci, M., & Kapranos, P. (2018). Microstructure and Properties of Semi-Solid Aluminum Alloys: A Literature Review. *Metals*, *8*(3), 181. https://doi.org/10.3390/met8030181

Razak, N. A., Ahmad, A. H., & Rashidi, M. M. (2017). Investigation of pouring temperature and holding time for semisolid metal feedstock production. *IOP Conference Series: Materials Science and Engineering*, *257*, 012085. https://doi.org/10.1088/1757-

899X/257/1/012085
Song, Y., Won, C., Kang, S. hoon, Lee, H., Park, S. J., Park, S. H., & Yoon, J. (2018). Characterization of glass viscosity with parallel plate and rotational viscometry. *Journal of Non-Crystalline Solids*, *486*(February), 27–35. https://doi.org/10.1016/j.jnoncrysol.2018.02.003
Wajid, M., & Shah, A. (2006). Investigation of semi-solid metal processing route, (Semisolid Metal Processing), 111.
WANG, Y. fei, ZHAO, S. dun, & ZHANG, C. yang. (2018). Microstructures and mechanical properties of semi-solid squeeze casting ZL104 connecting rod. *Transactions of Nonferrous Metals Society of China (English Edition)*, *28*(2), 193–219. https://doi.org/10.1016/S1003-6326(18)64656-4