EFFECT OF TEMPORAL THERMAL FIELD ON QUALITY OF SEMI-SOLID METAL FORMED COMPONENTS

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A thesis submitted for the degree of Doctor of Philosophy

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DECLARATION

I hereby certified that this material, which I now submit for assessment on the programme of study leading to the award of Doctor of Philosophy is entirely my own work, that I have exercised reasonable care to ensure that the work is original, and does not, to the best of my knowledge breach any law of copyright, and has not been taken from the works of others save and to the extent that such work has been cited and acknowledged within the text of my work.

PREFACE

This thesis presents the original works on semi-solid aluminium alloy processing which has not been submitted for a degree in Dublin City University or at any other universities. The research has been conducted in the School of Mechanical and Manufacturing Engineering, Dublin City University, during the period of December 2011 to December 2014. This work has been disseminated through the following publications and conferences.

Book Chapter:

A.H. Ahmad, S. Naher, S.N. Aqida and D. Brabazon, Routes to spheroidal starting material, *Chapter 5, Volume 5 in Comprehensive Materials Processing Technology, Elsevier, Ed. S. Hashmi, pp* 135-148, 2014.

Journal Articles:

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A.H. Ahmad, S. Naher and D. Brabazon, Direct thermal method of aluminium 7075. *Advanced Materials Research, Vols 939, pp 400-408, 2014.*

A.H. Ahmad, S. Naher and D. Brabazon, Injection tests and Effect on Microstructure and Properties of Aluminium 7075 Direct Thermal Method Feedstock Billets. *Key Engineering Material. Vols.* 611-612, pp 1637-1644, 2014.

A.H. Ahmad, S. Naher and D. Brabazon, Effects of cooling rates on thermal profiles and microstructure of aluminium 7075. *International Journal of Automotive and Mechanical Engineering. Vols 9, pp 1685-1694, 2014.*

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International Conference Papers:

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NOMENCLATURE AND ABBREVIATION

Symbols/	Description	Unite	
Acronym	Description	Units .	
SSM	Semisolid Metal	-	
DTA	Differential Thermal Analysis	-	
DSC	Differential Scanning Calorimetry	-	
DTM	Direct Thermal Method	-	
CTE	Coefficient of Thermal Expansion	x10 ⁻⁶ K ⁻¹	
f_s	Fraction Solid	-	
T _a	Temperature	°C	
T _m	Melting point of the solvent	°C	
T_L	Liquid temperature of the alloy	°C	
k	Equilibrium partition ratio	-	
f_s^{eff}	Effective fraction solid	-	
π	Pai	-	
D_f	Fractal dimension	mm	
V	Volume of an atom	mm ³	
η_o	Apparent viscosity of liquid	Pa.s	
ρ	Density	g/cm ³	
Т	Cooling Rate	°C/s	
Ŷ	Shear rate	s ⁻¹	
α	Alpha	-	
β	Beta	-	
q _e	External Heat Flux	W/m ²	
ΔH_f	Latent Heat	J	
v _m	Volume	mm ³	
	Cooling rate	°C/s	
Α'	Area	mm ²	
SCR	Shearing/cooling roll	-	
HJN	Hollow jet nozzle	-	

CRM	Centre de Recherches Metallurgiques	-
EMS	Electromagnetic Stirring	-
CREM	Refining Electromagnetic Caster	-
MHD	Magnetohydrodynamics	-
SSTT	Semi-Solid Thermal Transformation	-
NRC	New Rheocasting Process	-
SEED	Swirled Enthalpy Equilibrium Device	-
CRP	Continuous Rheoconversion Process	-
LSPSF	Low Superheat Pouring with a Shear Field	-
RCP	Rheo-Container Process	-
CS	Cooling Slope	-
SIMA	Strain Induced Melt Activation	-
RAP	Recrystallization And Partial melting	-
SIM	Self-Inoculation Method	-
HPDC	High Pressure Die Casting	-
CDF	Close Die Forging	-
PM	Permanent Mould	-
σ _{th}	Theoretical Cohesive Strength	N/m ²
Е	Young Modulus	GPa
¥s	Specific Surface Energy	J/m ²
a _o	Lattice Parameter	mm
UTS	Ultimate Tensile Strength	MPa
YS	Yield Strength	MPa
SHT	Solution Heat Treatment	-
HRB	Hardness Brinell	HB
CCA	Cooling Curve Analysis	-
DCP	Dendritic Coherency Points	-
∂T	Temperature Derivation	°C
L	Length of the specimen	mm
CNC	Computer Numerical Control	-
EDM	Electrical Discharged Machine	-
Cp	Specific Heat Coefficient	JK ⁻¹
$\left. \frac{dT}{dt} \right _{bc}$	Rate of change of the base line curve	°Cs ⁻¹

k	Thermal Conductivity	W/mK
\dot{q}_{G}	Energy Generation per unit volume	J/mm ³
HR	Hardness Rockwell	HR
SEM	Scanning Electron Microscope	-
EDXS	Energy-dispersive X-ray Spectroscopy	-
С	Circularity	-
AR	Aspect Ratio	-
W _{fb}	Weight measure by electronic scale	gram
W _{H2} 0	Submerged measurement readings	ml
ANOVA	Analysis of Variance	-
σ _{TS}	Tensile Strength	MPa
σ _y	Yield Strength	MPa
σ _f	Elongation	%
Sig.	Significant	-
C.Rate	Cooling Rate	°C/s
Liq. Temp.	Liquidus Temperature	°C
Eut. Temp.	Eutectic Temperature	°C
Sol. Temp.	Solidus Temperature	°C

ABSTRACT

This thesis presents an investigation of the effect of the semi-solid processing parameters on aluminium alloy 7075. In particular, this thesis focuses on the effect of the temporal thermal fields. Thermal analysis (TA) was initially performed in order to determine suitable processing conditions and understand the relationship between fraction solid and temperature. A specially designed chamber was used to achieve the cooling rates of 0.13 °C/s and different cooling medium were used to replicate the cooling rates of 0.32 °C/s, 0.36 °C/s and 0.39 °C/s. Different cooling rates had a marginal effect on the liquidus, eutectic and solidus phase change temperatures. The coherency point temperatures were also found to be weakly dependent on the cooling rates within the range investigated within this work. The fraction solid was however found to be strongly dependent on temperature. At temperatures higher than the coherency point (approximately 0.20 f_s for all cooling rates), the slopes of the fraction solid curves were not very steep which indicates that the fraction solid is sensitive to temperature fluctuation within this region. Based on this information gained from TA, globular microstructure feedstock billets suitable for SSM forming were produced by the direct thermal method (DTM). Spheroidal primary phase microstructures were best produced with the combination of 665 °C pouring temperature and 60 s holding time. The average microstructure primary phase diameter from DTM was measured to be within the range of 82 to 123 µm and circularity between 0.55 and 0.67. Higher pouring temperature was found to produce larger amount of secondary phase which was also determined in this work to lead to greater fluidity of the semi-solid material. The fluidity of the DTM feedstock billets were evaluated by a compression test at 185 kN. The formability of the feedstock billets was significantly influenced by the primary grain circularity and secondary (liquid) phase content within feedstock billets, with grain size having least effect. The average primary phase diameter and circularity after the forming operation were found to be in the range of 110 to 136 µm and 0.59 to 0.71 respectively. Tensile, hardness and porosity were examined for the feedstock billet after forming. Structures with greater amount of secondary phase were found to have the lowest strength and hardness properties. Processing parameters that led to samples with increased density (685 °C pouring temperature and 20 s holding time) were found to have increased strength and hardness. Smaller grain structures produced higher CTE and thermal dimension expansion values. The findings of this work have established the DTM feedstock production processing conditions which can enable the thixoforming of semi-solid 7075. Such enhanced formability is a priority when processing alloys of low fluidity such as 7075 wrought alloy.

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CHAPTER 1

INTRODUCTION

1.1 Overview

The use of the lightweight material such as aluminium alloy becomes one of the most important engineering materials in automotive and aerospace industries. Most of the automotive components such as cylinder heads, pistons, intake manifolds and chassis application in automotive power trains are made from aluminium alloy, typically by using a conventional metal casting process [1]. There are several potential drawbacks are associated with conventional casting process, for instance shrinkage porosity formation, gas entrapment and hot cracking which leads to product rejection. Nevertheless, some of these conventional casting process weaknesses can be overcome by applying a semisolid metal processing technique.

The semisolid metal (SSM) processing is an attracting technology, which offers the ability to produce aluminium and to some extend steel components. This process has many advantages such as good mechanical properties, die-life extension, less filling defects and less solidification time [2-6]. From the productivity aspect, the semi-solid casting process is a beneficial process that can provide the same or a greater production rate than the conventional highpressure die casting process. Semisolid casting processes are quickly developing as an alternative to traditional casting processes.

The SSM involves either with semi solid slurries or feedstock billets that have globular or non-dendritic microstructures surrounded within a liquid matrix. This special characteristic slurries or feedstock billets typically has a fraction solid in the range of 0.5 to 0.7 which able to flow smoothly when injected into a die cavity due to their morphology structure. The final part which manufactured are high in quality, eliminated most of the casting defects and be able to produce a near net shape product. These slurries or feedstock billet needs special

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techniques to prepare consist of a liquid metal route, an electromagnetic field, thermal profile control process, solid state routes and combination methods. The technique which is utilized to produce the slurries or feedstock billets depended on the size of the final product, primary phase structure and cost effectiveness.

The liquid metal routes involve with melting raw material above its liquidus temperature and then processing to produce the spheroidal microstructure. Mechanical stirring among the example of the liquid metal routes which uses auger, impellers or multiple agitators to produce a laboratory feedstock billet. An electromagnetic field is used in metal melting of electromagnetic stirring and for moving the metal fluid from one location to another. This allows the production of continuous SSM feedstock with no contact between agitators and metal that use low energy. Thermal profiles control process exploited the rapid cooling condition which the melt is heated just above the liquidus and rapidly cooled to below liquidus in order to produce a large number of primary phase particles. Swirled enthalpy equilibrium device for instance, uses a metallic container in order to replicate a rapid thermal equilibrium which in turn forms a large number of nuclei within bulk melt. Solid state routes process the material to spheroidal microstructure without melting the raw material. Strain Induced melt activation method is one of the methods which applies to a metal at hot working condition and then cold working it at room temperature. Combination method is the method which associates with two or more other methods to produce spheroidal microstructure. Self-inoculation method, for example is a combination of swirled enthalpy equilibrium device and cooling slope method which able to use a higher superheating temperature.

There are two possible methods which are used in semi-solid metal (SSM) processing consist of rheo and thixo routes. In rheo routes, a raw material which is originally from ingot processes without an intermediate solidification step. The molten metal which is slightly above the liquidus temperature is poured into a steel crucible and then treated to form a globular microstructure before moving into a forming machine. Thixo routes on the other hand cooled a raw material into a semisolid state with an intermediate solidification step. Thixo routes required preparation of SSM feedstock which has been initially treated in such a

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way when it is heated to semisolid range temperature it will form globular microstructures. The success of these processes merely depended on high quality raw material, size and morphology of primary phase and fraction solid volume during processing. Rheological behaviour of materials in SSM processing and the role of thixo routes which influences formed component quality are very crucial; therefore, it motivates many researches which are at the present conducted in large or laboratory scale around the world.

1.2 Research Objectives

The main objectives of the thesis were to evaluate the effect of temporal thermal field on wrought aluminium 7075 in order to comprehend the relationship between processing parameters and quality of semi-solid metal (SSM) formed component. The thesis addresses seven separate goals as follows:

- 1. To understand the relationships between the solidification rate, metallurgical behaviour, and fraction phase evolution of aluminium 7075.
- To determine the liquidus, eutectic and solidus temperature, fraction solids, and dendritic coherency point resulting from the different cooling conditions of aluminium 7075.
- To investigate the effect of pouring temperature and holding time during the DTM process on solidification rate and fraction solid, and in turn the microstructure of produced SSM 7075 feedstock billets.
- 4. To design, manufacture, test and commission a compression test processing unit and processing of SSM feedstock billets.
- 5. To analyse and understand the relationship between microstructure and formability of SSM feedstock billets.
- 6. To evaluate the ultimate tensile strength (UTS), yield strength (YS), elongation, stiffness (E) and hardness (HV) of the formed billet.
- 7. To evaluate microstructure, Coefficient of Thermal Expansion (CTE) and dimensional expansion of the SSM feedstock billets after forming.

1.3 Research Design

The main objective of the research was to evaluate the effect of temporal thermal field on aluminium 7075 quality processed by SSM technique. In order to achieve this, the research was designed and divided into several experiments which interconnected to each other. Lacks of information on 7075 thermal profiles in literature motivated to conduct the Thermal Analysis (TA) experimental works. The bulk metal with a volume of 750 gram was used in this experimental work to determine the thermal profiles. The bulk material was used to replicate the real application at real casting floor. In SSM processing research, the thermal profiles typically were gained by either DTA or DSC which used smaller volume of sample. The thermal analysis experiments conducted in this work also give deeper information on fraction solid temperature relations and dendritic coherency point. The thermal profile data in this experiment were important as it was used as an input to other experimental works. The results from TA experiment responded to research objectives 1 and 2.

Direct Thermal Method (DTM) is one of the methods used to produce feedstock billets for thixoforming. This method was selected due to the small feedstock billets size, which 22 mm in diameter and 70 mm in length needed to fit the requirement for forming die and its also a very cost effective technique. The limited information on the subject of material behaviour, especially aluminium 7075 which was used as a raw material in DTM was the motivation for this experimental works. Most of the researchers in DTM merely concentrate on aluminium A356 that higher in silicon contents which assist the material to flow. The feedstock billets which produced by DTM were used as the starting material for filling distance test. The results obtained from DTM experimental works were responded to research objectives 3.

Compression Test unit was used to evaluate the performance of feedstock billets which produced by the DTM. Flowability tests which conducted by the compression test unit in semisolid range temperature gives brief information regarding feedstock billets behaviour. The experimental works were conducted due to the insufficient information about the behaviour of DTM feedstock billets in literature. Most of the previous research merely concentrated on feedstock billets production, with less details experimental works have been done regarding reheating back the feedstock in semi-solid temperature especially for aluminium 7075. The feedstock billets after forming were used for the subsequent experimental works. The results were used to respond to research objectives 4 and 5.

The Mechanical Properties and Coefficient of Thermal Expansion (CTE) tests were conducted in order to evaluate the performance of the feedstock billets after forming such as ultimate tensile strength, yield strength, modulus of elasticity, elongation, hardness and thermal dimension expansion. The experimental work was conducted in order to give detailed explanations on the feedstock billets behaviour after forming. There were lacks of information regarding mechanical properties and CTE was the motivation of this experimental works. The results for these experimental works were used to respond to research objectives 6 and 7.

Overall, the research design of this thesis was designed with the aim to give a better understanding to readers about the correlation between the novel methodologies used in these experimental works and research objectives.

1.4 Thesis Outline

The thesis outline was laid out in a progressive manner with the intention to present the reader the flow of the experimental works started with the introduction followed by literature review, methodologies, results, discussion and conclusions. The thesis contents are presented as follows:

Chapter 1: Introduction

This chapter presents a brief overview and background that discussed the importance of this research followed by the objectives of this research; and research design that demonstrated how this research was designed in order to answer the research objectives.

Chapter 2: Literature Review

This chapter aims to give the reader a brief fundamental knowledge of the subject. This chapter focuses on the literature regarding semisolid metal, solidification, routes to spheroidal microstructure feedstock, mechanical properties of semisolid forming, aluminium and its alloys, thermal analysis, coefficient of thermal expansion and a brief description about motivation of the research.

Chapter 3: Methodologies

The research methodologies for this research were presented in chapter 3. The method which was used consists of material composition and preparation, thermal analysis, direct thermal method, compression test unit, mechanical properties, grain measurements, energy-dispersive X-ray spectroscopy and coefficient of thermal expansion and statistical analysis.

Chapter 4: Results

This chapter presents the results which were obtained from the experimental works. The results consist of as received sample, cooling curve analysis, direct thermal method feedstock billets microstructure and grain size measurement, compression test, mechanical properties and coefficient of thermal expansion.

Chapter 5: Discussion

Chapter 5 presents the discussion regarding the results which were obtained from chapter 4. Detail discussion was concentrated on the effect of cooling rate on solidification, fraction solid, dendritic coherency point and microstructure. The discussion chapter was also deliberated the effect of pouring temperature and holding time to feedstock billets; the effect of the feedstock billets properties on compression test and microstructures; effect of forming process to mechanical properties and coefficient of thermal expansion.

Chapter 6: Conclusion

This chapter presents overall findings of the research with recommendation for future work. This chapter also highlights the significance of this works and the most important findings of this work.

CHAPTER 2

LITERATURE REVIEW

2.1 Semi-Solid Metal

In conventional liquid alloy solidification processes, several processes occur that change the formation of the molten metal structure. With increasing time and decreasing temperature, the liquid metal changes to solid form. This process can be broken into a number of stages. At the beginning of this process, the dendrites (tree-like in morphology) form independently from one another. After this, the dendrites grow and eventually begin to spread through the whole area of the bulk metal and form interconnecting networks [7, 8]. Once solidus temperature is reached, any remaining liquid solidifies the instantaneously and traps the networks and their structures in place. At the end of the solidification process, there would typically be hundreds of millions to trillions of dendrites depending on exact cooling conditions and volume considered. This dendritic microstructure typically results from conventional liquid metal solidification processes. The exact size, shape, and number of primary phase components have a strong influence on the fluid consistency and processability within the semi-solid state. These characteristics depend on the processing technique and associated parameters applied during the solidification stage. This formation of the primary phase in turn determines the final properties of the material and produced component.

In 1971 during experimental work on the measurement of semi-solid metal (SSM) viscosity of Sn 15 %wt Pb alloys, it was noted that the viscosity was much reduced within the semi-solid state if shearing of the melt was continued during solidification [9-13]. The molten metal was sheared during cooling from above the liquidus temperature into semi-solid temperature regime. The associated microstructure alteration from dendritic to spheroidal was found to be the cause of this effect and the semi-solid fluid was in some cases noted as being orders of magnitude less viscous after this alteration. Within this early work, it was noted that the spheroidal microstructure was formed due to the

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shearing action leading to the release of the secondary dendrite arms from the dendrite arm roots leading to the structure fragmentation and spherodisation [13]. Later, the process of obtaining these structures was termed 'semi-solid metal' (SSM) forming. To date much research work has been carried out in order to improve the methods of obtaining the required spherical microstructure. Figure 2.1 shows schematically how the grain size and shape vary irreversibly with time during mechanical shearing in the semi-solid state.

SSM processing occurs between the liquidus and solidus temperature, a range within which the fluidity of molten metal can change greatly. Instead of a dendrite microstructure from the conventional liquid casting, a globular or spheroidal microstructure can be achieved by controlling process parameters, such as the temperature of the melt, cooling rate, stirring time, stirring type, and stirring speed [14, 15]. Both dendritic microstructure and spheroidal microstructure are shown in Figure 2.2.



Figure 2.1: Evolution of the primary phase structure during solidification with the vigorous agitation: (a) initial dendritic fragment; (b) dendritic growth; (c) rosette; (d) ripened rosette and (e) spheroidal [13].



Figure 2.2: Micrographs showing a (a) dendritic and (b) spheroidal microstructure [14, 15].

2.1.1 Principle of Thixoforming

There are two methods which are used in SSM processing consist of rheo and thixo routes. A rheo route involved with an ingot is processed without an intermediate solidification step. The liquid alloy which is marginally above the liquidus temperature is poured into a steel crucible and treated to form globular microstructures before transferring into a forming machine. Thixo route in contrast, is processed with an intermediate solidification step which a raw material heated to the semisolid state range temperature before injected into a die cavity. Thixo route required preparation of SSM feedstock which has been initially treated in such a way when it is heated to semisolid range temperature, it forms globular microstructures [15, 16]. The examples for rheo and thixo routes are presented in Figure 2.3.



Figure 2.3: Schematic illustration for variation of SSM processing routes [15].

The combination of two techniques in SSM processing is called semi-solid metal forming (SSF). This technique is a combination of both casting and forging operations. Forming process is typically involved with the operation in fully solid (forging) or in the fully liquid state (casting). The solid state forging operation involved with the production of a high performance part with a long production line, therefore, it required high machining and tooling costs and high energy consumption as well.

The liquid state casting operation produces a complex geometry component with fewer production steps, but in comparison with solid state forming, it generally produces a less quality performance part. Thixoforming of SSM is a possible method which combines the higher formability of liquid metal with the excellent quality of solid state formed component. There are several technique designations which are used SSM processing on thixotropic or feedstock billets in solid state shaping operation such as extrusion (thixoextrusion), forging (thixoforging) and rolling (thixorolling).

The success of thixoforming operation is much depended on feedstock billet temperature during the reheating process. The feedstock billet must be reheated to an appropriate SSM temperature before thixoforming operation. The formability and flowability of the feedstock billet are influenced by fraction solid volume within a feedstock billet during operation. Therefore, heating temperature is a crucial parameter in thixoforming. The relationship between fraction solid and heating temperature were discussed details in section 2.1.2.

SSM billet heating temperature should be kept as low as possible in order to keep the original shape and size of the feedstock billet. Material losses during the heating process should be avoided. Incapability to maintain the original shape of feedstock billet contributes to difficulty in billet transfer process from heating furnace into forming dies and also die filling problem. Furthermore, excessive heating operations also introduced inclusions and defects that related to heating [17]. Proper controls for a heating furnace are required in SSM heating to obtain homogenous temperature distribution over the entire cross sectional area. This is important in order to avoid micro-segregation and skin effect without grain coarsening. The uniform temperature distribution, uniform globular structure and optimum fraction solid volume were perquisite for SSM heating [18].

2.1.2 Fraction Solid

An important metallurgical characteristic that has significant effect during SSM processing is a fraction solid. Several studies highlighted it was important to obtain a low viscosity and a high fraction solid in SSM processing [19-21]. The fraction solid determines material flowability and influence microstructure and defect formation. The low viscosity component helps material to flow inside die cavity and high fraction solid helps to prevent various defects, a finer internal structure and a high quality product. The suitable fraction solid volume within material helps the grains to rotate, slip and move freely under a small external working force as presented in Figure 2.4.



Figure 2.4: Movement of the solid grains under an external working force [19].

The fraction solid volume in SSM forming typically occurred in the range of 0.30 to 0.50 f_s [16, 22] and in some other cases in the range of 0.60 to 0.70 f_s [23, 24]. There were a large number of published studies described various techniques which were used to evaluate high volume fraction solid flowability such as forward [25] and backward extrusion [26], and simple [27] and triaxial compression [28]. The variation on fraction solid which is used in SSM processing significantly affects the part properties. The results from one of the studies indicated that the tensile properties for aluminium 6061 processed with fraction solid in the range of 0.68 to 0.94 were brittle and elastic while with 0.56 f_s produced higher elongation and ductile value [25].

In SSM processing studies, several methods were used to estimate fraction solid volume within the molten alloy consist of the Scheil equation and the lever rule [29-31]. The estimation of fraction solid is essential in order to access the temperature-fraction solid ($T - f_s$) relations which later determines the success of SSM processing. Fraction solid volume in the melt is determined by Scheil as in Equation 2.1 as follows [20, 32]:

$$f_s = 1 - \left(\frac{T_m - T_L}{T_m - T}\right)^{1/(1-k)}$$
 Equation 2.1

where f_s is fraction solid volume, T_m is melting point of the solvent (for instance AI-Si alloy is at 660 °C, melting point of AI), T_L is liquidus temperature of the alloy and *k* is the equilibrium partition ratio.

Liquid segregation, primary and secondary phase distribution, grain size and grain rate coarsening were much influenced by the fraction solid volume within SSM sample. The liquid segregation occurred because more liquid flowing to the edge of SSM sample when the compression rate and fraction liquid increased and compression viscosity decreased. The liquid segregation and, primary and secondary phase distribution was homogenous while the grain size was increased when SSM sample was compressed at 0.5 f_s and punch velocity at 0.5 m/min [21]. The increased in temperature that leads to lower fraction solid volume was also found to increase the grain rate coarsening within samples [33].

2.1.3 Primary Phase Morphology and Particle Size Distribution

The flow behaviour of SSM was influenced by the primary phase morphology of SSM slurries [13, 14, 34]. Study on SSM flowability shows dendritic structures tend to have the lower flowability than equiaxed structures [20]. The dendritic structures have a tendency to interlock each other and prohibited material from flows when external force is applied. Furthermore, non-dendritic or globular structures were able to flow better which they tend to rotate and slip during forming operation [9, 35].

A finer microstructure produced a better flowability due to better movement, less collisions among particles and lower viscosity [13, 14, 36]. Several rheological tests were performed in order to characterise SSM behaviour. Simple model and defined parameters to characterise rheological of the SSM slurries were used in several research with variance in particle sizes to calculate the effective fraction solid, f_s^{eff} as presented in Equation 2.2 [37]:

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$$f_s^{eff} = \frac{\pi}{6} \left(\frac{\frac{D_f}{\pi^2}}{2^{D_f} \operatorname{x} \left(\frac{D_f}{2} \right)} \right)^{-3/D_f} \left(\frac{V}{d^3} \right)^{(3-D_f)/D_f} f_s^{3/D_f}$$
Equation 2.2

where D_f is fractal dimension, V is the volume of an atom, d is the average grain size and f_s is fraction solid. In order to get a detailed understanding, the variance of apparent viscosity versus particle diameter is presented in Figure 2.5 which calculated by using Equation 2.2. The apparent viscosity values that affect flowability for all samples in Figure 2.5 decreased with the increasing of particle diameter.



Figure 2.5: Calculated apparent viscosity for SSM slurries with the variance of particle size [38].

2.1.4 Alloy Compositions

The alloy chemical composition is found to influence the amount of primary phase solidified in SSM sample. Addition of small solute elements concentration enhanced the volume of melt during earlier stages of solidification. The addition of alloying element interferes with grain growth and allowed new finer grains to growth. These solutes produced boundary layer ahead solidification that affects the constitutional undercooling zone, which generally associated with dendritic growth. The type and percentage of solute elements, constitutional undercooling and thus the growth rate and morphology of the primary phase, dendritic or equiaxed growth may be controlled by controlling alloy chemical composition [39].

Higher solute concentration in the alloys produced higher rosette type feature and at the same time increase fraction liquid trapped between primary structures. This leads to the higher viscosity of SSM slurry that affects the fraction solid volume. The relationship between apparent viscosity of semi-solid alloy η , and compositional factors is presented in Equation 2.3 as follows [39].

$$\eta = \eta_o \left\{ 1 + \frac{\alpha \rho T^{\frac{1}{3}} \gamma^{-\frac{4}{3}}}{2 \left(\frac{1}{f_s} - \frac{1}{0.72 - \beta T^{\frac{1}{3}} \gamma^{-\frac{1}{3}}} \right)} \right\}$$
 Equation 2.3

where η_o is the apparent viscosity of liquid, ρ is the density of the alloy, *T* is the cooling rate, f_s is the fraction solid, Υ is the shear rate and α and β are the values which dependent on chemical composition of the alloy and become larger with an increment of solute content. The amount of alloy compositions not just altering the viscosity of the material, but also the dendritic coherency point by delaying it with an increment of solute concentration.

2.1.5 Advantages of Semi-Solid Metal Processing

SSM casting processes have developed as a niche casting process where high mechanical properties or a complex shape, or both, are required. It is an advanced technology which offers the ability to produce various components to be used in different industries, mostly within the automotive and aerospace industries. This process has many advantages, including low processing temperature, making it energy efficient, which contributes to part cost saving [15, 40, 41]. Due to increased fluidity during forming, provide from the spheroidal microstructure, parts formed have lower porosity levels and associated improved mechanical properties [42]. Other benefits include die-life

extension, less filling defects, and faster solidification. From a productivity viewpoint, SSM processing can provide the same or a greater production rate compared to conventional high-pressure die casting processes. Several advantages of SSM processing are summarised in Table 2.1 [13].

Behaviour	Advantages		
Lower Heat Content	Increase processing time		
	Reduce mould erosion		
	Able to form ferrous part		
	Able to form high melting point material		
	Able to form reactive materials		
High Fraction Solid	Reduce shrinkage		
	Reduce feeding		
	Reduce macrosegregation		
	Finer grain structure		
High Viscosity	Improved machineability		
	Improved surface finish		
	Reduce entrapped mould gases		
	Reduced oxidisation		
Lower flow stress	Produced complex parts		
than solid metals	Lower manufacturing cost		
	Higher manufacturing speed		
Incorporate with	Able to incorporate with		
other materials	composites		
Separation between	Purification		
liquid and solid			

Table 2.1: Behaviour and advantages of SSM processing [13].

2.2 Solidification

Solidification is a process which occurs during the transformation temperature of molten alloys from fully liquid of melt to fully solid of metal. The solidification process can be observed by the change of the physical form of metal from fully liquid into fully solid. The solidification process is a phenomena in which play significant roles in many aspects of daily life from manufacturing to solid state physics field. The solidification process, for example, occurs in the production of a sheet metal for forging which is usually cast before shaped to final dimensions. Solidification starts with the development of nuclei which followed by their subsequent growth. The nucleus is a simple unit of the crystal lattice structure that contained arranged atom for metal. During the solidification process, the heat must be lost in order to allow molten alloys solidify. The temperature needs to drop below the equilibrium freezing temperature that is also known as undercooling in order to make a metal to solidify. The high degree of undercooling will produces spontaneous or homogeneous nucleation that can only be achieved by rapid cooling. However, the homogeneous nucleation is difficult to achieve due to moderate cooling rate that commonly used in most of the solidification process. The heterogeneous nucleation typically occurs with moderate cooling rate that contained irregularities such as cracks on mould wall and solid impurities. The large number of nuclei was produced by the higher undercooling rate in both homogeneous and heterogeneous nucleation [7]. This nucleation in later stage produces many independent solid crystals that after solidification become the grain structure of the metal.

A finer grain structure which typically obtained large number of grains per unit volume occurs with higher cooling rates. Large grain boundary surface within solidified sample is useful to block the slip of crystal structure defects producing a better mechanical property component.

The higher cooling rate typically produces more dendritic structures. The dendritic structure formation during solidification is influenced by a solute gradient ahead of the solid/liquid interface that controlled by solute diffusion and is a much slower process. Due to less time for solute diffusion at a higher cooling rate, large solute concentrations occur in the liquid, ahead of the solid/liquid front. This leads to larger growth of undercooling [7, 32].

The initial stage that solidified first in the alloy is called a primary phase. Meanwhile, an alloy which is solidified last which occurs around a primary phase is called a secondary phase. The primary phase nucleates and glows from the smaller particles when the alloy is held at a high temperature. These small initial grains have higher surface interfacial energy per unit volume and surrounded by a higher local matrix solute content compare to larger particles. The energy content reduced as solidification continues with diffusion flow occurs from small particles that melt and disappear to the large growing particles. This process, in which the mean radius of the average primary article increases, is designated as coarsening.

Coarsening occurs by agglomeration or coalescence of the solid particles during solidification. During the semi-solid state, solid particles may collide with each other by several factors such as particle sedimentation, natural convective currents and induced shear. These particles may agglomerate through diffusion controlled re-crystallisation when they collided with well aligned crystal lattice orientations. The disagglomeration of these particles may also formed by the method of joining necked region with melted particles. The disagglomeration produced large numbers of nucleation site or called grain multiplication, which dendritic arms particle were melted by inducing flow in semi-solid melts [7, 43]. Particles which obtained higher miss-orientation angle between two crystal structure are likely susceptible to melting.

Much of the delay in exploiting cast materials probably originated from the lack of understanding on the solidification nature and how a microstructure is produced. In particular, the face of fracture surfaces is always taken to indicate the nature of the crystals, which a casting is composed. In the absence of an adequate picture of the solidification process, casting was bound to remain a crucial area to be explored and still remain until today.

2.2.1 Heat Extraction

Solidification is impossible without heat extraction. The melt must be cooled down to solidification temperature and after that the latent heat of solidification which occurs at the solid/liquid interface must be extracted. There are two methods of heat extraction during solidification that change an internal energy either solid or liquid metal phases. The first method is by decreasing in enthalpy due to cooling and the second is by transformating metal from liquid to solid state which is equal to the latent heat of fusion [7].

The heat extraction occurs when a suitable means of cooling applied to the melt in order to produce an external heat flux, q_e . Resultant cooling rate, dT/dt, is gathered from a simple heat balance when the metal has a low cooling rate and equilibrium specific heats between liquid and solid. In order to imitate with the dimensions and other factors, the heat extraction behaviour can be presented in mathematical model by using the latent heat per unit volume $\Delta h_f = \Delta H_f / v_m$ which is positive for solidification and specific per unit volume, *c*, as presented in Equation 2.4 and Equation 2.5 as follows [7]:

$$q_e\left(\frac{A'}{v}\right) = -c\left(\frac{dT}{dt}\right) + \Delta h_f\left(\frac{df_s}{dt}\right)$$
 Equation 2.4

So that cooling rate, \dot{T} :

$$\dot{T} = \frac{dT}{dt} = -q_e \left(\frac{A'}{vc}\right) + \left(\frac{df_s}{dt}\right) \left(\frac{\Delta f_s}{c}\right)$$
 Equation 2.5

The first right side of Equation 2.5 represents the response of casting geometry (ratio of surface area A' over volume v) upon the extraction of heat. Meanwhile, the second right side of Equation 2.5 is the continuing evolution of latent heat of fusion during solidification. Base on the Equation 2.5, the heating during solidification only can occur if the second right side of the equation $\left(\frac{df_s}{dt}\right)\left(\frac{\Delta f_s}{c}\right)$, is higher than the first one $-q_e\left(\frac{A'}{vc}\right)$. This phenomenon is known as recalescence. Recalescence is the temperature increase, which occurs during a metal cooling due to change of a structure and an enthalpy. The variation of fraction solid within an alloy during solidification with the temperature as a function of time can be calculated from the relationship in Equation 2.6 as follows:

$$\frac{df_s}{dt} = \left(\frac{dT}{dt}\right) \left(\frac{df_s}{dT}\right)$$
 Equation 2.6

Since volume fraction of solid f_s is a function of temperature. In this case:

$$\dot{T} = \frac{-q_e \left(\frac{A'}{vc}\right)}{1 - \left(\frac{\Delta h_f}{c}\right) \left(\frac{df_s}{dT}\right)}$$

Equation 2.7

Solidification should decreases the cooling rate since $\left(\frac{df_s}{dT}\right)$ is negative.

There are two basic heat extraction processes happen during solidification. These are either by fix rate movement of a crucible or via chill (see Figure 2.6). The fix rate movement of the crucible during solidification is typically used for single crystal growth or directional solidification. In this process, the growth rate of the solid and the temperature gradient are separately controlled. The unidirectional crystal growth and heat flux occur when the growth rate of the solid is too high. Another process which involved with chill solidification, crystal growth occurs in parallel and opposite to the heat flux direction. The example of heat extraction by fix rate movement and via chill is presented in Figure 2.6 [7].



Figure 2.6: Schematic diagram for heat extraction via (a) fix rate movement of crucible and (b) chill [7].

2.2.2 Conditions for Nucleation

During the early stage of solidification, the melt was not immediately solidified at the freezing temperature as shown in Figure 2.7 [44]. The nucleation begins at some degree of undercooling which is generally very small in practical situations for metals. The undercooling is defined as the temperature difference between an equilibrium temperature of a system and its actual temperature. The nuclei are lower than the equilibrium temperature when the melt is undercooled. In this case, undercooling is greater than zero. Since nuclei effect is not present and because some undercooling is needed to supply a driving force for a nucleation and growth processes. The liquid must first be undercooled to the actual nucleation temperature where nucleating particles naturally present in the melt become active. When these nuclei start to develop into small crystals, a latent heat is evolved causing the temperature of the surrounding melt to rise. The continuing growth of the crystals is observed to occur at about constant temperature, the actual growth temperature, which under the condition is usually between 1 and 2 °C below the equilibrium liquidus temperature [7].



Figure 2.7: Schematic of cooling curve during solidification [44].

Higher undercooling during solidification leads to a larger number of nuclei formation which produced a finer grain size due to a large number of grains per unit volume. Larger grain boundary surface areas are more efficient to block the slip of crystal structure defects that subsequently produced the better mechanical properties component. Larger solidification ranges are typically produced more dendritic growth [45]. Thermal diffusion controlled a thermal gradient at the solid/liquid interface which later influenced the dendritic growth, structure and solidification rate for a metal. Solute diffusion, which is a much slower process in contrast, controlled a solute gradient a head of solid/liquid interface that characterise the dendritic formation. Large solute concentration is found in the liquid ahead of solid/liquid front due to little time for solute diffusion. This produced a larger growth of undercooling which reveal the fact that higher cooling rates are favourable to produce more dendritic structure.

Small grain, which initially begin to grow are not modifying the cooling rate that imposed by the external heat flux, q_e . The increment in undercooling significantly increases the nucleation rate, *I*, and also the growth rate, *V*, of the dendrites [7, 46]. The overall solidification rate approaches a maximum value when the internal heat flux (q_i), which is proportional to the latent heat of fusion and the volume rate of transformation, $\dot{f}_s = (df_s/dt)$, is equal to the external one (q_i) (Equation 2.7). Here, $\dot{T} = 0$ [7].

The early stage of equiaxed solidification that is crucial and nucleationcontrolled, fraction solid of the melt volume is still very small. Slightly after that, the melt temperature has increased beyond the nucleation temperature and at this stage, which known as the second stage of solidification, is growthcontrolled. The number of grains presents that constantly evolved and the solidification proceeds first by the lengthening of dendrites and then by dendrite arm thickening when the grains are in contact. The nucleation is assumed to be the dominant process at the early stage of solidification that considered to influence the establishment of the final grain population with each nucleus forming one equiaxed grain. Even though in the case of columnar solidification, the first stage of solid in casting typically in the form of equiaxed grains. The conditions leading to nucleation are therefore of utmost importance in determining the characteristics of any cast microstructure [7].

2.2.3 Solidification and Microstructures

Solid nuclei initially are appearing in the liquid at close to mould wall. This due to the mould wall is the coldest compare to other locations, the resulting the heat transfer from the hot to the cold surface. These nuclei are called heterogeneous nuclei. These nuclei advance rapidly with a short period of time, thus increase in size and form at the outer equiaxed zone, which can grow

either parallel or opposite to the heat flow direction. The formation of columnar zones which occur due to mutual competition, transformed from other overgrown orientations. Beyond a certain stage in the development of columnar dendrite branches, which become detached and grows independently. These are likely to form an equiaxed shape due to latent heat that is extracted by the undercooled melt. The region, which contains solidified element is then called an inner equiaxed zone. The change from columnar to equiaxed growth merely relies on the liquid degree of convection. In continuous casting machines, the finer structure at the ingot centre was produced by electromagnetic stirring which promoted this transition [7]. The example columnar and equaixed grains within a material are presented in Figure 2.8.



Figure 2.8: Columnar and equiaxed grain structure occurred at mould wall and the centre [47].

2.3 Routes to Spheroidal Microstructure Feedstock

This section presents several techniques that have been used either within the laboratory or within the industry in recent years for producing feedstock for SSM processing. All of these techniques have the particular aim to produce the spherical microstructure required for SSM processing. The methods of

producing this feedstock are presented in the five following sections describing respectively liquid metal routes, electromagnetic field techniques, thermal profile control processes, solid state routes and routes which utilise a combination of methods.

2.3.1 Liquid Metal Routes

The liquid metal routes involve melting the raw material above its liquidus temperature and then processing to produce the spheroidal microstructure [48].

a. Mechanical Stirring

Mechanical stirring is achieved using augers, impellers or multiple agitators that are attached to a rotating shaft [49-53]. An advantage of mechanical stirring is that the torque and rotational speed can be recorded during stirring and used in real time to determine the fluid viscosity as well as for process control. A Searle viscometer set-up in this manner, in which the inner rotor turns, can be simultaneously used to determine fluid rheological properties and produce SSM feedstock [35, 54]. With such a set-up rheological properties can be determined with respect to processing time and shear rate. The measurements of torque and speed are easily logged against time from sensors on the shaft of the mechanical stirrer. This method is a cost effective method for the laboratory scale feedstock production and investigation.

The mechanical stirring process can be sub-categorised into active and passive stirring techniques. Direct inducing shear in mechanical stirring is the active stirring method. Passive stirring, on the other hand, is shear of solidifying melt by forcing it through stationary narrow channels under close temperature control within the semi-solid range [55-57]. A continuous mechanical stir-caster is grouped under active stirring methods while reciprocating injection screw, shearing/cooling roll (SCR), and hollow jet nozzle (HJN) methods are categorised as passive stirring techniques. Thixotropic billet material for thixocasting was previously produced by STAMPAL SPA in Italy using a passive stirring technique [56]. The metal alloy was forced to flow into a cooled

static mixer (consisting of refractory spheres) by an electromagnetic induction pump. The production rate for thixotropic feedstock was around 1000 kg per day using this technique [34]. Other designs of passive stirring used in processing of semi-solid metal have also been patented [58, 59].

b. Continuous Mechanical Stir-Casters

The semi-solid metal thixotropic nature of Sn-Pb and aluminium based alloys were studied at MIT from 1971 where the first batch caster was developed [13]. Low temperature continuous stir-casters, which required tight temperature control, were subsequently developed specifically for non-ferrous low melting point alloys [60]. A stir-caster with a reservoir capacity of 18 kg of steel was constructed in MIT [12]. The high temperature continuous stir-caster required even higher levels of temperature control which was implemented by closely controlling both heating elements as well as heat extraction rates. The materials that could be processed using this system were not limited to aluminium but copper alloys, iron and its alloys could also be processed.

These techniques were also used for compocasting, a technique where ceramic reinforcement is distributed uniformly within a metal matrix within the semi-solid state. For example, consistent SiC reinforcement distributions are regularly achieved within an aluminium matrix via this route [61-64]. The mechanical properties of materials are significantly improved with optimum stirring operations that mix the particles uniformly inside SSM. For example, the ultimate tensile strength of AA6061-T6-AIN_p composite formed via compocasting was found to be 47 % higher than the un-reinforced monolithic alloy [65].

c. Reciprocating Injection Screw

The reciprocating injection screw is a mechanical method for producing thixotropic slurry that combines the technologies of plastic injection moulding and thixotropic metal processing [66-68]. Alloy chips passing through the injection barrel are heated up via the shearing action of the screw and external

heaters. Semi-solid slurry is accumulated in front of the screw and is held at a desired fraction solid. Rotation of the screw is then stopped and the semi-solid metal is injected into a die cavity with the assistance of a non-return valve. Important process control parameters include temperature, injection speed and pressure, and holding time. The process is carried out in a controlled atmosphere to prevent air from reaching the semi-solid metal. Due to the reactivity of magnesium alloys with air during melting, this technique is particularly suitable for magnesium alloy forming. Close control of barrel temperature and shot velocity is required in order to provide increased strength and ductility of the formed material [69]. Cast alloys produced by this process have superior mechanical properties due to close-to-zero-porosity, as well as a fine and uniform microstructure, compared to the conventional processes. It is difficult to process aluminium alloys by this method as they react with the screw and the barrel materials when in the semi-molten state.

d. Shearing/Cooling Roll

The Shearing/Cooling Roll (SCR) method involves pouring molten metal into a gap between a roll-shoe and a rotating roll [70, 71]. A schematic diagram for SCR is presented in Figure 2.9. Process parameters include melt pouring temperature, roll temperature, shoe height, and rotational speed. These can be adjusted in order to manage the shearing action in the metal during the solidification stage. A higher pouring temperature within the liquid range can produce periodical cracks at the part surface, whereas a temperature within the semi-solid range, even that of a relatively low fraction solid can provide smooth flowing and defect free cast plates [72]. In order to create different shear rates, the gap distance between the rollers can be adjusted. The process output is also seen to transform from a dendritic type structure to a structure with fine equiaxed grains with the decrement of roll-shoe cavity height, a situation leading to increase shearing and cooling rate [73-75]. This method is simple and easy with common tooling materials readily available and allows the attainment of a high cooling rate [70]. A drawback of the process may be the relatively restricted form of thin sheet produced which limits the form of parts which could be subsequently produced.



Figure 2.9: Schematic diagram of Shearing/Cooling Roll (SCR) [74].

e. Hollow Jet Nozzle

Hollow Jet Nozzle (HJN) is a technique in which a high temperature melt is poured through a cooled hollow nozzle where powder is injected into the melt stream such that a low superheat is achieved before metal solidification within a continuous casting process. It was developed by Centre de Recherches Metallurgiques (CRM) in Liege, Belgium [76]. A schematic diagram depicting HJN is illustrated in Figure 2.10. Researchers have examined this as a useful technique for producing spheroidal microstructures in steel alloys [77]. The spheroidal microstructure in this case is obtained due to the increased in the number of nucleation sites from the seed particles injected and due to the low superheat resulting in rapid solidification. The formed crystals soon impinge on each other preventing further crystal growth [77].



Figure 2.10: Principle of the injection through the Hollow Jet Nozzle (HJN) [77].

2.3.2 Electromagnetic Field Techniques

Electromagnetic fields are used in metal melting of electromagnetic stirring (EMS) and for moving the metal fluid from one location to another. These techniques have commonly been used within the industry to produce a fine spheroidal grained structure for thixoforming [78, 79].

a. Electromagnetic Stirring

The electromagnetic stirring (EMS) technique has been applied to SSM to produce a high fluid stirring velocity in the semi-solid materials. The basic concept of this process is that strong electromagnetic fields induce electrical currents in a moving conductive fluid, which in turn creates forces on the fluid. This process allows continuous production of SSM ingot feedstock with no contact between the agitator and metal, and requires relatively low energy consumption [78].

EMS was developed by a company called ITT in the USA, and patents were held by Alumax [34, 80, 81]. The technique is capable of producing a fine structure with grain sizes of 30 µm in continuous cast billet, free of inclusions and gas pick up. However, it was originally limited to the billet of no more than 40 mm diameter. Another technique based on a refining electromagnetic caster (CREM) was designed and patented later by Pechiney SA in 1990 [82]. EMS was the main technique used by industry to produce feedstock for SSM forming. EMS is suitable not only for non-ferrous alloys, but it has also been successfully used to process ferrous alloys [83, 84]. As for another stirring technique, the combination of temperature control for lower pouring temperature and faster cooling rate promotes a finer structure in EMS. Other parameters that are important to provide a uniform fine spheroidal microstructure include the setting of the gap between the electromagnets and the melt, the electromagnetic current, and the frequency [85, 86]. A study of magnetohydrodynamics (MHD) is needed in order to fully understand and model how the electric and magnetic fields interact with the metal during EMS. MHD involves a simultaneous and iterative solution to the differential Navier-Stokes equations of fluid dynamics and Maxwell's equations of electromagnetism. This is required as the applied magnetic field induces an electrical current within the moving conductive fluid which provides a force and in turn alters the magnetic field.

b. Permanent Magnet EMS

Permanent magnet EMS produces an intense stirring in semi-solid alloy slurries. Rotation of the permanent magnet is achieved by rotation with an electric motor either inside a hollow crucible or circumferentially outside of an EMS stir caster. The rotation of the permanent magnets at speeds of 500 rpm can be used to produce a required stir-cast microstructure in a 150 mm diameter billet. Permanent magnet EMS has the advantage of low electrical power consumption at approximately 2 kWhr⁻¹ per tonne [87]. This significantly reduces the cost of electrical facilities. Another advantage of this technique is its small space requirements compared with the bulkiness of the electromagnetic stirring systems. Some disadvantages of permanent magnet EMS are the large

initial cost of the permanent magnet, maintenance requirements for the rotating components, and the need to achieve rotation.

c. Osprey Process and Spray Deposition

The early concept of spray forming was introduced in the early 1970s by the name of spray rolling [88]. This early research was concentrated on spray forming of aluminium alloys. The findings triggered the development of the technique and commercialisation thereof, which was then licensed by Osprey Metals, Neath, Wales in the early 1980s. This spray forming technique was hence known as the 'Osprey process' [89]. The rapid solidification that occurred in this technique produced a microstructure which was refined, equiaxed and with low segregation. The technique was used for production of bulk semifinished products in a single integrated operation. This process applies a high velocity inert gas jet to a stream of liquid metal in order to atomise it. This stream is then deposited in the semi-solid state onto a target where it builds up as a non-dendritic structure. The operation of atomisation and droplet merging on the collector mandrel is continuous. The microstructure resulting from this process is homogenous, fine grained and contains uniform precipitation of the second phases, all of which help to improve the workability and properties of the produced preform [90-93].

d. Grain Refinement by Addition of Refiner

The uniform distribution of fine particulate seed refiners in the melt provides nucleation points for new grains to form [94, 95]. This process is used normally in the production of conventional ingot. In order to produce a spheroidal starting material, a greater quantity of the refiner is used than in conventional alloy production. A small rounded microstructure can then be obtained after reheating process within a short time of isothermal holding. As an example, the addition of titanium diboride (TiB₂) into liquid aluminium alloy results in a finer grain microstructure. This results from the low solubility of TiB₂ even in the absence of any dissolved Ti [96]. The TiB₂ plays an important role in grain nucleation where it introduces an activity gradient at the TiB₂/melt interface.

A fine and equiaxed structure with an average grain size of 102 μ m was found with the addition of AI-5Ti-1B by reacting with supplemental refiners of K₂TiF₆ and KBF₄ into the molten aluminium alloy [97]. The different microstructures formed from untreated and treated materials are shown in Figure 2.11.

e. Vibration

Either by sonic or ultrasonic vibration produced by mechanical vibration elements solidification can be applied during to refine the microstructure [98, 99]. Such vibration is normally provided via magnetostrictive or piezoelectric transducers [98]. A schematic of the process is shown in Figure 2.12. Contact between the molten metal and transducer probe (normally made of titanium) is provided via a high temperature material coupling (typically ceramic based). Some disadvantages include that it can only be applied to small volumes as the cavitation is focussed near the transducer. High temperature coupling material may dissolve with time when immersed in the liquid metal where it may also contaminate the melt [98].



Figure 2.11: Grain form of pure aluminium (a) untreated and (b) grain refined with AI-5Ti-1B [94].



Figure 2.12: Schematic of a system for grain refinement using vibration [98].

2.3.3 Thermal Profile Control Processes

Thermal profile control during solidification is now the most common technique for production of semi-solid metal feedstock commercially. In this process, the alloy temperature is initially brought to just about the liquidus so that there is a small amount of superheat. After this, the alloy is rapidly cooled to below the liquidus temperature in order to nucleate a large number of primary phase crystals. The alloy is then held at a set temperature, for a predefined amount of time, within the semi-solid range in order to spheroidal nucleated crystals. The semi-solid material can then be directly formed into the final component shape without the requirement for solidification and re-heating. Within the semi-solid range, the alloy fraction solid is very sensitive to temperature and composition variations. Thermal profile control processes are also referred to in the literature as Semi-Solid Thermal Transformation (SSTT) processes [100-103]. There are many variations on this technique discussed below, including New Rheocasting Process (NRC), Swirled Enthalpy Equilibrium Device (SEED), Continuous Rheoconversion Process (CRP), Low Superheat Pouring with a Shear Field (LSPSF), and the Direct Thermal Method (DTM).

a. New RheoCasting

Figure 2.13 presents a schematic diagram depicting the steps in the NRC process. The steps in the New RheoCasting (NRC) process are as follows [104-106]. The process starts with melting the raw material and pouring with low superheat into a chill environment so that more nuclei occur throughout the melt volume. Superheating the molten alloy to less than 50 °C above the liquidus temperature is used within this process [104]. The molten metal is then cooled slowly to avoid dendritic nucleation. After this, the molten metal is held for some time within the semi-solid temperature range. After it has reached the appropriate fraction solid, it is then transferred from the cooling station and charged into an inclined sleeve. This semi-solid metal lastly is cast into the required part mould by die casting. The dimension of the charge holding wall was seen as crucial where an increment of wall thickness produced the unwanted dendritic microstructures [107]. This process has been used for aluminium and magnesium casting.



Figure 2.13: Schematic diagram for the UBE new rheocasting process (NRC) that produces globular microstructure from pouring until the casting process [104].

b. Swirled Enthalpy Equilibrium Device

The Swirled Enthalpy Equilibrium Device (SEED) process uses a metallic container to create a rapid thermal equilibrium in order to form a large number of nuclei within the bulk metal [108]. The process can be used to produce slugs of aluminium in different sizes to allow production of over 18 kg of product weight [109, 110]. The process can be divided into two important steps, which are heat extraction to achieve the required fraction solid and the swirling

process which is used to achieve thermal equilibrium and uniform distribution of nuclei, see Figure 2.14. In one version of the process, excess liquid is drained to produce a self-supporting SSM feedstock which is then formed under pressure. Heat extraction has to take account of superheat and the evolution of latent heat in order to generate an equilibrium state between the crucible and the slurry.

Other important parameters in the process that influences the formation of finer grains are the isothermal holding period and the addition of solid alloying elements during swirling [111]. The temperature gradient at the initial stage of cooling is large, due to the engagement of superheated molten metal with the cooler wall surface of the mould. The temperature difference between the molten metal and mould decreases significantly during the swirling process as the temperature of the insulated mould increases.



Figure 2.14: Process flow of SEED begins with (a) pouring liquid metal into a container, (b) addition of solid alloy while swirling, (c) de-moulding and (d) isothermal holding [111].

c. Continuous Rheoconversion Process

The Continuous Rheoconversion Process (CRP) process, shown in Figure 2.15, uses the concept of passive liquid mixing within the designed reactor system and provides for the controlled formation of nuclei and growth of primary alloy

phase [112-114]. This reactor provides extraction of heat, copious nucleation and forced convection during solidification which results in a fine globular grain structure. The mentioned reactor is placed at the top of a metal slope leading to a shot sleeve within a typical die casting machine. The melt is then pumped into the inlet of the reactor from the holding furnace. As the melt flows through the reactor, the set fraction solid is achieved by controlling the temperature gradient controlled via cooling water flow rate within the reactor heat exchanger [112, 115, 116]. The evolution of microstructure for CRP depends on the pouring temperature, the amount of heat extracted by the reactor, and slope angle.



Figure 2.15: Schematic design of the Continuous Rheoconversion Process (CRP) [112, 115, 116].

d. Low Superheat Pouring with a Shear Field

The Low Superheat Pouring with a Shear Field (LSPSF) process manipulates the solidification condition to control nucleation formation, nuclei survival and grain growth. A schematic diagram of LSPSF slurry machine is shown in Figure 2.16. In this process, the melt is poured into a rotating stainless steel barrel which is temperature controlled via simultaneous induction heating and air cooling in order to achieve rapid cooling of the metal to slightly below its liquidus. This melt, then with numerous nuclei flows into the slurry holder where it is cooled slowly to achieve the required fraction solid and microstructure for SSM forming [117]. The basic concepts of this process are similar to the cooling slope except for the inclusion of the rotating barrel. The rapidly cooling alloy was mixed in this process by using the rotating barrel. The combination of lowered pouring temperature and rapid cooling at the beginning of the process provides for the high required degree of nuclei formation. Similar to the other thermal processes, this process aims to achieve a maximum nucleation rate at the start of solidification and a maximum nuclei survival rate. This process has been demonstrated for aluminium alloy processing [118].



Figure 2.16: Schematic diagram of a laboratory scale Low Superheat Pouring with Shear Field (LSPSF) slurry maker [106, 107, 119].

e. Direct Thermal Method

The novel process of Direct Thermal Method (DTM) was discovered initially by Brabazon and co-workers in 1997 [120-124]. In this process the aluminium alloy was cooled from a low superheat within thin walled cylindrical copper moulds. The superheated aluminium was rapidly cooled into the semi-solid state by the heat extraction provided by the copper mould. This initial rapid cooling gave rise to copious nucleation. At the same time a thermal equilibrium was rapidly reached between the copper mould and the aluminium alloy which in effect provided an isothermal arrest which enabled the diffusion dominated processes of ripening to yield a globular microstructure. The concept of this process is different from the others in that it uses only natural cooling. An example of the spheroidal microstructure produced from DTM using a copper mould is shown in Figure 2.17.



Figure 2.17: Spheroidal microstructure of A356 produced from the Direct Thermal Method [120].

Important processing parameters in DTM that shape the desirable microstructure include pouring temperature, holding time before quenching or forming, and size of cooling mould utilised. Lower pouring temperature and shorter holding periods produced more spherical structures [121]. The more spheroidal primary phase would increase the fluidity of the material during SSM forming. Higher cooling rates resulting from lower pouring temperatures above to below the liquidus temperature provide less superheat to be extracted by cylindrical copper mould. The resulting increased undercooling of the alloy during the solidification stage promotes the formation of more nuclei which in turn results in a smaller grain size [121]. An obvious advantage of this process is that it needs no special equipment and provides for low processing cost [120, 122-124]. However, there are limitations on the size of billet that can be produced.

The DTM allows the formation of a globular microstructure by concept of natural cooling between superheat melt and mould material. The concept of natural cooling permits thermal equilibrium arrest, which rapidly achieved due to the rapid cooling action, raises the formation of nucleation. This action enabled the diffusion dominated processes of ripening to yield a globular microstructure.

Copper was chosen as a mould material due to its thermal conductivity characteristic. The thermal conductivity was an important factor in order to make sure heat from the melt during solidification rapidly convicted to the environment. The thermal properties comparison between copper and other materials are presented in Table 2.2. The copper thermal conductivity is at 385 W/mK as shown in Table 2.2 which relatively higher than other typical materials that used as a mould material.

Materials	Thermal Conductivity (W/mK)	Specific Heat Capacity (J/gC)		
Aluminium, (Pure)	210	0.900		
Copper, (Pure)	385	0.385		
Iron, (Pure)	76.2	0.440		
High Carbon Steel	52	0.669		
H13 Tool Steel	24.3	0.460		

Table 2.2: Thermal properties of several materials [125].

Heat conduction and heat transfer behaviour between the molten metal and copper mould were expressed in an analytical and a mathematical approach by the Fourier's Law. The principle of conservation of energy can be defined as the total rate of heat conduction into and rate of a heat generation inside the system are equal to the total rate of a heat conduction out of and rate of energy storage inside the control volume [126].

The definition is illustrated in details as shown in Figure 2.18 with the assumption that the temperature inside material is a function of *X* coordinate and time; that is T=T(x, t) and a conductivity *k*, a density ρ and a specific heat *c* of the solid are all constant. While *A* is a volume, \dot{q}_G is rate of the energy generation per unit volume inside the system and *q* is the rate of energy.



Figure 2.18: The principle of heat conduction and heat generation into and out of the system [126].

In order to get a deeper understanding on the conduction behaviours, it can be expressed in a mathematical form as presented in Equation 2.8:

$$-kA \left. \frac{dT}{dx} \right|_{x} + \dot{q}_{G} A \Delta x = -kA \left. \frac{dT}{dx} \right|_{x+\Delta x} + \rho A \Delta xc \left. \frac{dT(x + \frac{\Delta x}{2}, t)}{dt} \right|_{x+\Delta x}$$
Equation 2.8

.

Dividing Equation 2.8 by the control volume and rearranging; it produced Equation 2.9:

$$k \frac{\left(\frac{dT}{dx}\right)_{x+\Delta x} - \left(\frac{dT}{dx}\right)_{x}}{dx} + \dot{q}_{G} = \rho c \frac{dT(x + \frac{\Delta x}{2}, t)}{dt}$$
 Equation 2.9

In the limit as $\Delta x \rightarrow 0$, the first term on the left side of Equation 2.9 can be expressed in the form of Equation 2.10:

$$\frac{dT}{dx}\Big|_{x+dx} = \frac{dT}{dx}\Big|_{x} + \frac{d}{dx}\Big(\frac{dT}{dx}\Big|_{x}\Big)dx = \frac{dT}{dx}\Big|_{x} + \frac{d^{2}T}{dx}\Big|_{x}dx \qquad \text{Equation 2.10}$$

The right side of Equation 2.10 can be expended in a Taylor series as in Equation 2.11:

$$\frac{dT}{dt}\left[\left(x+\frac{\Delta x}{2}\right),t\right] = \left.\frac{dT}{dt}\right|_{x} + \left.\frac{d^{2}T}{dxdt}\right|_{x}\frac{\Delta x}{2} + \cdots$$
 Equation 2.11

Equation 2.11 then becomes Equation 2.12 to the order of $\Delta x_{;}$

$$k \frac{d^2T}{dx^2} + \dot{q}_G = \rho c \frac{dT}{dt}$$
 Equation 2.12

Physically, the first term on the left side represents the net rate of heat conduction and the second term on the right side represents the net rate of energy generation. Base on the Equation 2.12 the thermal conductivity plays the important rules to catalyse the heat conduction of the system. The thermal conduction rules explain in details the phenomenon which happens during solidification of melt within a copper mould. The high thermal conductivity of the copper mould, increase the heat losses thus increase the cooling rate. The increment in cooling rate later produced a rapid cooling condition that affects the formation of nucleation during solidification. This action affects the under cooling behaviour which, eventually affects the formation of a globular microstructure.

f. Rheo-Container Process

The Rheo-Container Process (RCP) method was developed for processing reactive materials such as magnesium or aluminium-lithium alloys [127, 128]. It has a special encapsulation that prevents the reaction of processed materials with surrounding gasses, see Figure 2.19. The reactive molten metal is poured directly into an aluminium container of 1 mm wall thickness in order to prevent reaction between the atmospheric gasses and the highly oxygen-sensitive alloy. The initial reaction between the molten metal and the wall of the container which is at room temperature produces seed crystals that scatter inside the container due to fluid flow. The heat capacity of the container rapidly brings the metal below the liquidus temperature to start solidification. This chilling process of the molten metal produces a globular microstructure (cooling channel process). After filling, the container is placed directly into a cooling device, covered immediately, and filled with argon gas in an effort to prevent alloy

reaction. In order to reduce processing time, pressurised air is applied to cool the container. The container, together with the semi-solid billet, is transferred into the shot chamber of die-casting machine after it has reached the required temperature and fraction solid before it is pressed into the die.



Figure 2.19: Schematic diagram for the Rheo-Container Process (RCP) [109, 110].

g. Cooling Slope

Cooling slope (CS) as shown in Figure 2.20 is another low cost method that can be used to produce ingots for thixoforming. The basic process of the CS method is that of pouring the molten metal into water cooled cooling slope [129]. The molten metal is poured onto the cooling slope, close to its liquidus temperature, which quenches it into the semi-solid state and results in the formation of many nuclei. The CS can be easily mounted as part of a conventional casting machine.

The primary crystals of the semi-solid slurry at the cooling slope became more spherical after being maintained within the semi-solid state [129]. As with the other thermal profile controlled processes, the CS method can be used to produce feedstock directly for SSM forming or can be used to solidify billet for subsequent SSM processing. The primary crystals of the ingot cast by these techniques will also be transformed back to a globular microstructure when the ingot is re-heated into the semi-solid state. Within the CS method, it was noticed that the primary crystal size became smaller when the thermal conductivity of the slope was higher. Experimental results have shown that this cooling rate is a dominant factor in the globularisation of primary crystals [129].



Figure 2.20: Schematic diagram of CS method where the molten metal is poured onto a cool incline surface in order to create a chill environment [129].

Aluminium alloy A390 that has been SSM formed using this technique has shown that the part was metallurgically sound, free from porosity and attained higher hardness than conventional processing [130]. Disadvantages from the cooling slope method could however, result due to the temporal and dimensionally extended nature of the flow, which could result in gas pick, oxide formation and associated porosity like defects within the cast components.

2.3.4 Solid State Routes

Solid state routes provide the formation of a spheroidal microstructure without melting the raw material. The material is deformed by the conventional forging type processes and then reheated into semi-solid state. These processes are termed Strain Induced Melt Activation (SIMA) [131-136] and Recrystallization And Partial melting (RAP) [137-142]. In the SIMA process this deformation is achieved by hot working the metal (above the recrystallisation temperature) and then cold working it at room temperature, as indicated in Figure 2.21. The RAP process, on the other hand, involves warm working below the recrystallization temperature. After this stage, the rest of the process is similar for either SIMA or RAP. Upon reheating into the semi-solid state and isothermal holding the deformed metal transforms to a fine spheroidal microstructure suitable for SSM

forming. Straining methods which may be used in the SIMA and RAP processes include extrusion, rolling and forging. This processing route has been demonstrated for aluminium, magnesium, steel, and copper alloys [131-142]. SIMA and RAP both have advantages in their cost effectiveness, simple operation, and ability to prepare feedstock from alloys with a high melting point [131, 132]. However this RAP technique is somewhat limited for small section billets due to the level of deformation required. Appropriate isothermal heat treatment contributes to the success of the process [133, 139]. Increased isothermal holding time in the partial re-melting process in particular has been found to contribute to the formation of globular primary phase morphology [139]. On the other hand, increased isothermal holding periods for magnesium alloys have been found to increase the average size of the solid particles [139]. A comparison has been made between different techniques involved with RAP and semisolid thermal transformation using ZK60 magnesium alloy where a finer spheroidal microstructure was obtained by the RAP route [141]. The effects of the prolonged holding time during isothermal holding not only affect the microstructure but also the mechanical properties. In this case, the ultimate tensile strength for the sample with longer holding time decreased compared with the sample with shorter holding time.

The degree of deformation has been found to be a controlling parameter for the recrystallization, where higher strain results in decreased grain size [134]. The cooling rate during ingot formation prior to SIMA is also one of the important parameters in order to create finer microstructure. A higher cooling capacity has been shown to allow the creation of a finer microstructure of α -Mg primary phase surrounded by a thin layer of eutectic mixtures [135]. The application of SIMA and RAP for high melting point material such as steel has been successful. This technique is more appropriate to control globular microstructure deformation due to simple process compared to the stirring method [136]. Other steels like M2 tool steel and HP9/4/30 of high performance category were also processed [142]. A partial melting experiment shows that temperature of 1360 °C is suitable for processing these steels with 0.7 fractions solid and a grain size of around 30 µm [142].



Figure 2.21: Process flow for SIMA showing (a) melting and casting, (b) preheating, (c) extrusion, (d) quenching, (e) cold working, (f) reheating, and (g) forming and quenching [131].

2.3.5 Combinations of Methods

A number of lab and industrial scale production methods for SSM feedstock combine together elements of two or more other methods. These illustrate the diversity of possible routes to achieve a spheroidal microstructure for SSM forming. Presented below are some of these methods, including Self-Inoculation Method (SIM), NRC with grain refinement, SEED with grain refinement, CS with vibration, and mechanical stirring with ultrasonic vibration.

a. Self-inoculation method

The Self-Inoculation Method (SIM) was a combination technique that applied SEED and cooling slope methods [143, 144]. The SIM process is started with the melting of the raw material, addition of the self-inoculation into the melt with stirring action and lastly pouring the melt into the multi-stream mixing cooling channel. A schematic diagram for SIM is presented in Figure 2.22. This technique has the advantage being able to use a higher superheating temperature in systems that may require this in order to obtain the spheroidal

microstructure [143]. Research results to date show that this technique was applied to Mg-alloys, Al-alloys, Zn alloys and Cu-alloys [143, 144].



Figure 2.22: Schematic diagram of Self-Inoculation Method (SIM) [143].

b. NRC with grain refinement

Grain refinement has been combined with the NRC method of feedstock production [145, 146] The addition of AI-5Ti-1B chemical grain refinement into aluminium alloy AI-7Si-0.4Mg reduces the size of the grains where the system requires a high melt pouring temperature. The reaction of grain refiner in this case was found to not only depend on the material used as the refiner but also processing temperature. For example, the addition of AI-5Ti-1B grain refiner into molten aluminium alloys has been found to be less effective at lower pouring temperatures in the range of 630 to 650 °C compared to a higher pouring temperature range from 670 to 720 °C [145]. By applying the higher pouring temperature the grain refiner was able to provide a finer and more homogeneous microstructure with small variation in grain size between the centre and the edge of the slug [145]. In this latter work, it was noted that best nucleation formation results were obtained for inoculation periods lasting 2 minutes or more.

c. SEED with grain refinement

The nucleation temperature was increased by 5 °C with the combination of SEED process and grain refinement [146]. The process flow of this technique starts with the base alloy being melted in a resistance furnace and refined with AI5Ti1B. The treated billet then was re-heated again with the conventional heating equipment and the SEED processes applied. The addition of Ti-B grain refiner into the SEED process increased the density of α -AI primary phase and resulted in smaller grain sizes. The same technique (SEED and grain refinement) was applied to the aluminium alloy A356 with the addition of Sr, Ti and B as modifiers [147]. The addition of these modifiers catalyses the nucleation of primary α -Al during earlier solidification period and result in a much higher degree of nucleation. The immediate addition of Ti, B and Sr in conjunction with SEED reduced the grain size efficiently compared with when the grain refiner was added on its own. The results showed that the final grain size was reduced by 56 % compared to just a 33 % reduction when only the refiner was used [147]. The eutectic Si of in particular was well modified in this process by the addition of Sr content from a flaky Si structure to one of a more nodular and fibrous type [147].

d. CS with vibration

The combination of the cooling slope (CS) and vibration techniques for aluminium alloy A380 has been shown to result in a α -AI sphericity of 89 % [148]. The inclined surface in this case was made from mild steel and equipped with an integrated water cooling system (CS) with a vibration unit during processing billet for thixoforming. During the casting process, the cooling slope was mechanically vibrated. The process was carried out using a lower pouring temperature in order to create globular microstructure and with a 5.75 Hz vibration frequency. The microstructure attained from this process showed an increase in grain number due to nucleation on the slope and dendrite fragmentation caused by shearing during flow on the slope. Despite the very low vibration frequency used, the vibration was noted in this work to have

produced a more uniform cooling and to have increased the amount of nucleation and to have increased the sphericity of the primary phase.

e. Mechanical stirring with ultrasonic vibration

Mechanical stirring and ultrasonic vibration have also been combined as an alternate route to produce spheroidal starting material. This technique was used to fabricate reinforced magnesium matrix nanocomposites [149]. In this process the first step was the melting of magnesium alloy AZ91 under an atmosphere containing a gas mixture CO_2/SF_6 , then reduction of the temperature into the semi-solid range at which point SiC nanoparticles (preheated to 550 °C) were added into the AZ91 semi-solid alloy. The mixture then was stirred for a period of time in order to ensure a uniform mixing. After a mechanical stirring, ultrasound was applied via a transducer with a maximum power of 2 kW and frequency of 20 kHz before the final part shaping under 100 MPa pressure. The results from this research showed that a finer microstructure could be achieved with the combined sequential application of mechanical stirring and ultrasonic vibration. A higher localised fluid flow from the combined effects of vibration and mechanical stirring was noted to have mobilised the insoluble solid nanoparticles and promote more nucleation.

2.4 Mechanical Properties of SSM Forming

A large and growing body of literature has investigated the mechanical properties of SSM forming, which includes the cast and wrought aluminium alloy. Several data for mechanical properties were presented which primarily related to permanent mould casting as presented in Table 2.3. Moreover, a considerable amount of literature likewise has been suggested several factors that give a significant effect to the mechanical properties of SSM forming part such as primary and secondary phase grain size and morphology, also porosity.

The primary phase grain size and morphology influenced the mechanical properties with smaller size and globular grain morphology improved the properties of a metal [150-154]. Elongation and strength properties were

increased not just because the grain size reduction, but also due to a finer microstructure distribution within a sample. The tensile strength and elongation for the uniform and a globular primary structure was found increased than a conventional HPDC part. The uniform and globular microstructure was found effectively to reduce stress concentration at the grain boundary under an applied stress, thus increased tensile strength and ductility [150, 155]. Tensile strength was also reduced with a larger average size of primary phase aluminium, while the coarse microstructure and large precipitates increased the diffusion time of elements during saturation [151, 156]. Furthermore, grain coarsening was initiated to the weak mechanical properties of the material [154]. The smaller primary phase α-Al size and more globular microstructure increase the mechanical properties of the material. The average globular primary phase size, which performed by the SSM processing technique was in the range of 54 to 140 µm [151]. In order to increase the material properties, thixforming material need to produce smaller and more spherical microstructure feature.

Table 2.3: Mechanical properties for some aluminium alloys; with the CDF is for closed die forging; PM is for permanent mould casting; SSM semisolid metal

Alloy	Process	Tensile	Yield	Elongation	Hardness	Temper
		Strength (MPa)	Strength (Mpa)	(%)	(HB)	
Casting Alloys						
356	SSM	296	193	12	90	T6
(AI-7Si-0.5Mg)	SSM	300	256	11.4	-	T6
	SSM	320	240	12	105	T6
	SSM	310	260	9	100	T7
	SSM	234	172	11	89	T5
	SSM	225	180	5 - 10	80	T5
	PM	262	186	5	80	T6
	CDF	340	280	9	-	T6
	PM	186	138	2	-	T51
357, A347	SSM	358	290	10	100	T6
(Al-7Si-0.3Mg)	SSM	330	260	9	115	T6
(3)	SSM	330	290	7	110	T7
	SSM	296	207	11	90	T5
	SSM	285	200	5 - 10	90	T5
	PM	359	296	5	100	T6
	PM	200	145	4	-	T51
Wrought Alloys						
2017 (al-4Cu-Mg)	SSM	386	276	8.8	88	T4
2017 2024 (Al-4Cu-	W	427	275	22	105	T4
	N22	366	277	0.2	_	Te
1Mg)	00101	500	211	5.2		10
2024	CDF	420	230	8	-	T6
2024	W	476	393	10	-	T6
2024	W	469	324	19	120	T4
2219(Al-6Cu)	SSM	352	310	5	89	T8
2219	W	400	260	8	-	T6
6061 (Al-1Mg-Si)	SSM	330	290	8.2	104	T6
6061	W	310	275	12	95	T6
7075 (Al-6Zn-Mg-	SSM	496	421	7	135	Т6
CU)	0.014	105	0.04			To
7075	SSM	405	361	6.6	-	16
/075	CDF	560	420	6	-	16
7075	VV	570	505	11	150	16

and W is for wrought [34].
The amount of liquid (secondary) phase inside material was significantly influenced the variation of mechanical properties for thixoforming parts [151]. Inhomogeneous structures which consist of liquid (secondary) phase concentrated in the inter-globular regions during the reheating process before thixoforming was found to reduce the mechanical properties [138]. On the other hand, the reduction of liquid (secondary) phase within the treated sample significantly improved the material properties [157]. The liquid (secondary) phase increased the gap between grains, which easily generated crack when the material is stretched. The close contact between grains that typically compact influenced material property which exhibits isotropic along the deformation direction [158]. Typically, a large number of liquidus (secondary) phase reduces the material properties by increasing the gap between structures that block the break within the sample. The inhomogeneous of liquid (secondary) phase, usually occurs during the heating before forming process. The post-treatment after the thixoforming operation seem to be the solution to homogenous the liquidus (secondary) phase.

The existence of large pores caused by entrapped gas and shrinkage pore reduce the mechanical properties of diecasting samples [150, 159]. Material properties of SSM processing parts were found to improve mainly due to the reduction of porosity content. Shrinkage porosity usually occurs from the lack of metal flows and low feedability inside die cavities while gas porosity occurs from various gasses entrained during solidification. The porosity also may occur from hydrogen gasses which induced during heating, such for aluminium when heated higher than 600 °C [160]. The porosity reduced the material properties by allowing a hollow structure within alloy which unable to prevent crack propagation between grains. The porosity defect is typically associated with processing parameters. The less the turbulence flow during filling can reduce the porosity and increase the mechanical properties by reducing gas entrapment. Some other treatment methods such as degassing were also found to reduce the porosity content within sample [160].

2.4.1 Fractography

Fracture by definition is the separation or fragmentation of a solid body into two or more parts under the action of stress [161]. The fracture process is made from two components consist of crack initiation and propagation. The fracture can be classified into two general categories known as a ductile and brittle fracture. The ductile structure is characterised by plastic deformation prior to and during the crack propagation with gross deformation occurs at the fracture surface. The brittle structure is characterised by rapid crack propagation without gross deformation and little microdeformation. The brittle structure is similar to cleavage in ionic crystals. The brittle structure is catalysed by decreasing temperature, increasing strain rate and triaxial stress conditions. The brittle fracture needs to be avoided because it suddenly happens without any warning. The difference between ductile and brittle fracture is presented in Figure 2.23. The fracture deformation can be predicted by using an analytical approach. Classical Griffith crack is normally used to predict the crack. The strength of a brittle material which exhibited linear elastic behaviour is given by Equation 2.13 [161, 162]:

$$\sigma_{th} = \left(\frac{2E\gamma_s}{a_o}\right)^{1/2}$$
Equation 2.13

With E is young modulus, γ_s is specific surface energy, a_o is lattice parameter and σ_{th} is theoretical cohesive strength.



Figure 2.23: Photographs of fractured specimens with (a) ductile and (b) brittle fractures [161].

2.5 Aluminium and Aluminium Alloys

In aluminium alloy casting, the broad range of mechanical properties is achievable by Al-Si (A356/357) alloy system, but in some application, it needs specific properties which could be better achieved by other alloy system [109]. Al-Si (A356/357) has the excellent flow ability as the rich silicon elements content make it suitable for the complex and thin wall components. It has been used widely in the automotive industries for a various apparatuses. It also can be observed by the studies along these recent years that used Al-Si alloy systems in their research [35, 163, 164]. The challenges in producing near-net-shape component with the excellent properties make the superior properties of aluminium alloy become more essential. In aluminium alloy systems, the wrought aluminium alloy has significant advantages in the ultimate tensile strength (UTS) and yield strength compare with other aluminium alloy systems. The 2xxx (Al-Cu), 5xxx (Al-Mg) and 7xxx (Al-Zn) among the desirable wrought aluminium alloy. Each of the alloys has their own advantages and fitted for their own specific purposes.

2.5.1 Aluminium Alloy Designations

Aluminium are categorised into two main groups consist of wrought and cast alloys which depended on its initial fabrication process. The group is further divided into several families of alloy based on their chemical composition and temper designation. The chemical composition is the type of elements within a material which based on weight percentage amount. Meanwhile, temper designation is the condition of materials which undergo a heat treatment process.

Typically, 75 to 80 % of aluminium alloys are used to produce products such as rolled plate, sheet, foil, extrusion, tube, rod, bar and wire [165]. The wrought alloys are obtained from cast ingots which working operations and thermal treatment made to material changed its original structure. The wrought alloys are classified into several classes which its working characteristics and properties are much depended on alloying composition and structure. The relationship between the classes of wrought aluminium alloys and alloying composition are presented in Figure 2.24 [165].

The wrought aluminium alloys used a four-digit system which differentiate composition for each alloy classes. Major alloying element is identified by the first digit while the remaining digits are used to identify alloying type. The following discussed the four-digit systems which are used for wrought aluminium alloys:

AA1*xxx* series alloys consist of 99 % unalloyed pure aluminium or higher. Aluminium AA1*xxx* series has a high corrosion resistance, thermal and electrical conductivity but low in mechanical properties.



Figure 2.24: The range of available wrought aluminium alloys [165].

AA2*xxx* series alloys with copper as the principal alloying element is a heattreatable material with a high strength property. However, it has a low corrosion resistance which is exposed to intergranular attack and difficult to inhibit [165].

AA3xxx series alloys have manganese as its principal alloying elements which are a non-treatable material [165]. The AA3xxx series has a higher corrosion resistance and moderate properties strength.

AA4xxx series alloys comprise silicon as its principal alloying elements. The material melting temperature is low due to the addition of silicon without changing its material properties [165]. These alloys are able to be either heat or non-heat treatable. The heat treatable AA4xxx series alloys comprise silicon and other elements such as magnesium and copper that allowed its react to a heat treatment process. The non-heat treatable AA4xxx series alloys on the other hand, consist with solely silicon as principal alloying elements without other elements which allowed the materials to react to heat treatment in order to increase its strength. The AA4xxx are excellent in corrosion resistance and can be inhibited [165].

AA5*xxx* series alloys consist of magnesium as principal alloying elements. The alloys are non-heat treatable material. The special characteristic of magnesium is a high solubility in aluminium. Good corrosion resistance and moderate strength are advantages of AA5*xxx* series alloys. Conversely, this alloy is experiencing stress corrosion cracking under certain conditions of loading.

AA6*xxx* series alloys contain magnesium and silicon as principal alloying elements with moderate strength, good ductility and heat-treatable. The AA6*xxx* series alloys are excellent in corrosion resistance.

AA7*xxx* series alloys consist of zinc as the principal alloying element with other elements such as copper, magnesium, chromium and zirconium may be specified [165]. The AA7*xxx* series alloys are divided into two major families which is depending on the copper as a third alloying element. The highest strength properties, which occurred after heat treatment is in AI-Zn-Mg-Cu alloys but poor in corrosion resistance. AI-Zn-Mg alloys are on the other hand good in corrosion resistance.

AA8xxx series alloys contain various compositions which consist of tin and some lithium. The characteristic of AA8xxx series alloys is much depended on the major alloying elements.

Cast aluminium alloys are the common foundry metals which normally are used in automotive products and applications. The advantages of cast aluminium alloys for castings are low melting temperature, negligible solubility and good surface finish of the final product. The cast aluminium alloy designations are consisted of four-digit number system. The first digits indicate the alloy group which belong to and the second and third digits represent the specific aluminium alloy. The decimal point which is used after the third digit to differentiate between initial condition, whether casting or ingot. The production form, for example *xxx*.0 representing castings meanwhile *xxx*.1 representing ingot that compromise alloying element limit the same or similar alloying to casting. The designation for cast aluminium alloys is presented in Table 2.4.

Alloys Series	Principal Alloying Elements			
1 <i>xxx.x</i>	99.00 % minimum Aluminium			
2 <i>xxx.x</i>	Copper			
3 <i>xxx.x</i>	Silicon + Copper and/or Magnesium			
4 <i>xxx.x</i>	Silicon			
5 <i>xxx.x</i>	Magnesium			
6 <i>xxx.x</i>	Unused Series			
7 <i>xxx.x</i>	Zinc			
8 <i>xxx.x</i>	Tin			
9 <i>xxx.x</i>	Other Elements			

Table 2.4: Designation system in cast aluminium alloy [165].

2.5.2 Aluminium 7075

Most aluminium semi-solid formed components are made from casting alloys such as A356 and A357 due to their fluidity behaviour. The cast aluminium alloys, though have relatively poor mechanical properties compared to conventional wrought aluminium alloys. The wrought aluminium alloys have significant advantages in terms of higher ultimate tensile strength (UTS) and yield strength. The wrought aluminium 7075 is one of the aluminium alloys that have the highest tensile properties [166]. Components produced using this alloy includes aircraft fittings, gears and shafts, fuse parts, regulating valve parts, and worm gears [125]. STAMPAL-Italy [167], Grove Aircraft Landing Gear Systems Inc.-USA [168], ALCOA-USA [169] and JJ Churchill Ltd-UK [170] are among the list of companies who produce a wide range of the components using 7075.

Zinc (Zn) and Magnesium (Mg) are two important elements of aluminium 7075. Both elements give significant effect for the material properties. The addition of Zinc and Magnesium will develop the potential strength and greater response to a heat treatment process in aluminium systems, especially in the range of 3 to 7.5 % Zn [125].

The ultimate tensile strength (UTS) and yield strength of aluminium 7075-O are 228 and 103 MPa respectively. These values were increased significantly for aluminium 7075-T6, which has been undergoes a heat treatment process

respectively at 572 and 503 MPa [171]. Meanwhile, for the thermal properties, aluminium 7075 has a liquidus temperature at 635 °C and a solidus temperature between 477 and 486 °C [125, 172]. For the comparison, the UTS of 7075-T6 is higher than a grey cast iron [173]. This indicates that aluminium 7075 is able to replace a grey cast iron where light weight performance is required.

Aluminium 7075 is widely being used in the thixoforming and rheocasting [16, 157, 174-177]. Typically, it undergoes a heat treatment process to increase the mechanical properties. The T6 Solution Heat Treatment (SHT) is the normal procedure for it. The Ultimate Tensile Strength (UTS) result shows that the T6 SHT increased significantly compared with the as-cast component.

Figure 2.25 shows the effect of SHT for rheocasting aluminium 7075 [157]. The best UTS value was found in the SHT at 450 °C and four hours SHT time. The temperature and duration of aging also gives a substantial effect on the performance of aluminium 7075. The lower temperature and long duration time for treatment are crucial. Figure 2.26 shows that Hardness (HRB) value for aluminium 7075 increased with the lowest setting temperature and the longest aging time. The peak HRB value was found at 120 °C for 72 hours [157].



Figure 2.25: Ultimate Tensile Strength (UTS) of aluminium 7075 increased with the Solution Heat Treatment (SHT) for As-Cast, SHT for 1 hour (SHT 1) and SHT for 4 hours (SHT 4) [157].



Figure 2.26: Hardness (HRB) varies at different temperatures and times for aging [157].

Aluminium 7075 has recently been investigated for use in the SSM processing [16, 138, 159, 175, 178, 179]. The performance of aluminium 7075 was investigated by using both thixoformed cooling slope and recrystallization and partial melting (RAP) route in order to compare their mechanical properties [138]. It was noted in this work that thixoformed RAP route produced better tensile properties.

2.5.3 Aluminium 7075 Solidification Sequence

In order to estimate material behaviour and understand the related processing parameters which involved during solidification, information about material solidification sequence is vital. There are two crucial information for material solidification consist of liquidus and solidus temperature, which material is in fully liquid (completely melt) for liquidus and fully solid state (completely solid) for solidus. The liquidus and solidus temperatures were at 630 to 634 °C and 470 to 477 °C which depended on the source of references and material compositions [171, 180]. The molten alloys are entirely in the liquid state after the melt passed its liquidus temperature. Alloy temperature later is allowed to drop towards solidus temperature due to heat lost and convection which

indicate the start of the solidification process. The solidification sequence and phase precipitated of Aluminium 7075 are shown in Table 2.5 as follow.

Solidification sequence of aluminium 7075 starts with the formation of dendritic aluminium network. This formation is known as the primary phase of the aluminium systems. The formation of the dendritic network is started just below the liquidus temperature with this formation is much depended on the cooling rate of the melt. This reaction is believed to occur at the temperature in the range of 630 to 638 °C. The second reaction of phase precipitation is Al₃Fe. The Al₃Fe phase is started to turn into solid in the range of the temperature of 601 to 618 °C. The next phase to precipitate is AI + Mg_2Si which is believed to occur at the temperature in the range of 560 to 568 °C. The eutectic phase precipitation, which is the last to solidify phase are AI + Al_2Cu + $MgZn_2$ + $Al_2Mg_3Zn_3$. This reaction is believed to occur in temperature range of 468 to 470 °C. The phase diagram for Al-Zn-Mg-Cu system is presented in Appendix A.

Reaction	Phase Precipitation	Temperature (°C)
1 st	liquid 🛛 Al, dendritic network	630 - 638
2 nd	liquid \longrightarrow AI + Al ₃ Fe	601- 618
3 rd	liquid \longrightarrow AI + Mg ₂ Si	560 - 568
4 th	liquid \longrightarrow AI + Al ₂ Cu + MgZn ₂ + Al ₂ Mg ₃ Zn ₃	468 - 470

Table 2.5: Phase precipitated during solidification of Aluminium 7075 [44].

2.5.4 Aluminium 7075 Limitations

There have been various attempts to use aluminium 7075 in SSM processing, but they suffered from a very narrow temperature window, limited flow length and a tendency to form hot cracks, tearing and decreased hot workability [109, 181]. Furthermore, the stress-corrosion cracking likewise becomes one of the weaknesses of aluminium 7075 [165]. Figure 2.27 shows the stress-corrosion defects of aluminium 7075 after quenched in cold-water for forging process. Despite of the weaknesses, its however, was also has been able processed by SSM processing and the mechanical properties of this

material increased significantly after the post treatment method [138, 153, 182, 183].



Figure 2.27: Stress-corrosion cracks in cold-water quenched aluminium 7075 forging [165].

A common problem that normally occurred during processing of conventional 7075 extrusion tubes was the formation of a welding line defect. However, with thixoextrusion process, homogenous and equiaxed grain structure occurred within the sample and eliminated the welding line defects, and used lower energy consumption for processing [178]. The material performance was influenced by processing parameters used in thixoforming operation. Superior 7075 mechanical properties occurred from the use of a higher tool temperature setting and an appropriate tool punch speed. Furthermore, the setting resulted in crack reduction, which was due to overly fast cooling rate [159]. Aluminium 7075 has also a low sensitivity to temperature variation at a high fraction solid which makes it more suitable for the high fraction solid thixoforging compared to other alloy systems [24]. While the difficulties in processing this material in SSM arise, yet there are still researches able to overcome the problem and manipulated the properties of aluminium 7075. The near-shape rheocasting of aluminium 7075 was successfully processed with CSIR-RCS and HPDC with the increment of UTS and without hot tearing defect occurred [172]. This shows that with excellent processing route, the enhancement of the aluminium 7075 mechanical properties is still achievable in SSM.

2.6 Thermal Analysis

Thermal Analysis (TA) is a technique which is used to record physical properties of the sample as a function of temperature versus time or thermal curve either sample is heated or cooled by using a scheduled program [184]. The main purpose of making a TA measurement is to evaluate the recorded properties of the thermal analysis curve in order to analyse the changes that happened in the sample in both physical and chemical aspects. The interpretation of the thermal analysis curve consist with the related feature of a property-temperature curve such as discontinuities, changes of slope and peak that represents a thermal event in the sample that usually involve with chemical reaction or physical transition effect from the change in sample temperature. Thermal analysis (TA) is useful and important characterisation method which allows determination of the quality of a melt batch via a recording of phase change temperatures and fraction solid-temperature profiles relations [185].

Information regarding thermal profiles during SSM processing is a crucial aspect in order to control the processing parameters. The thermal profiles provide information regarding fraction solid-temperature relations which contributes to a significant effect on the selection of processing temperature in semisolid range temperature. A considerable amount of literature has been published on SSM processing highlighted the important of TA to estimate a suitable temperature during processing to avoid over or under approximation for forming temperatures [186-189]. The optimum forming temperature, which selected during the forming operation is found to produce a better quality product and eliminates most of the casting related defects.

2.6.1 Differential Thermal Analysis and Differential Scanning Calorimetry

Differential thermal analysis (DTA) and differential scanning calorimetry (DSC) are among a list of thermal analysis methods. Both of these techniques use a sample to be tested and a reference sample. During testing, the change of temperature and energy between these two samples are plotted against time. DTA measures changes of temperature in the sample and reference samples [184]. The sample is heated at a constant rate and the temperature variances are compared with reference sample which not experience thermal event. The thermal event is a process which both physical and chemical processes accompanied by changes in enthalpy or specific heat. The temperature variation between sample and reference sample is too small to be observed, hence in order to measure the differences in sample and reference sample temperature; it is plotted against the furnace temperature.

DSC on the other hand, measures directly the energy difference between a sample and a reference sample [184]. Both of the samples are heated and hold at similar temperatures, which determined initially during a thermal event in the sample. The thermal analysis curves are obtained from the amount of the energy supplied or withdraw to the sample in order to maintain zero temperature difference between sample and reference sample.

In recent years, a large volume of published studies on SSM processing research which used DSC in order to obtain material thermal profiles such as liquidus, eutectic, solidus temperature and as well as fraction solid-temperature relations. Wider range for maximum to a minimum temperature between heating and cooling during DSC processing was performed by Yucel [190] for aluminium A357. The temperature variation in this process altered material thermal profiles. The increment at minimum of 2.27 to maximum 181 °C/s heating rate temperature significantly changed liquidus temperature from 621 to 702 °C. For this reason, the fraction solid-temperature relation also was affected simultaneously by liquidus alteration.

The fraction solid-temperature relations were likely depended on the heating rate for DSC used for aluminium 7075. Figure 2.28 shows the relation between fraction solid and temperature, which was obtained by DSC for SSM processing at 15 °C/min heating rate of aluminium 7075. Several studies had also revealed by using a heating rate of 15 °C/min, fraction solid that occurred at 620 °C was at 0.7 [24, 159, 191]. Heating rate at 18 °C/min produced 0.60 fractions solid at 620 °C which gives indication that the higher heating rate delayed the formation of phases inside material [192]. The variant of heating or cooling rates used in DSC gives a different result which varies from each other. Furthermore, the variant in cooling and heating by using a small sample, typically 10 mg may deviate from original bulk metal which is used during processing, since the high volume of material are typically used for melting the raw material. In fact, cooling rate also much depended on material volume [180, 193].



Figure 2.28: Fraction solid vs temperature curves for aluminium 7075 with 0.25 °C/s heating rate obtained by DSC [191].

2.6.2 Cooling Curve Analysis

Cooling curve analysis (CCA) is one of the thermal analysis methods which are straightforward, low processing cost and gives a consistent result. CCA determine the relationship between melt treatment, alloy composition, cooling curve parameters and properties. This method has been widely used to determine material thermal profiles in these recent years. The determination of a baseline is crucial in CCA method as it will decide the fraction solid-temperature relations. The CCA method interprets latent heat that evolved during solidification by the first derivation of cooling curve graph. This latent heat, which was characterised by the temperature change inside molten metal, represented the phase changes [185, 194]. CCA is a suitable method to analyse the large volume of material which reflects the real shop floor application.

A number of studies in the literature have used the CCA method to determine the fraction solid-temperature relations and solidification behaviour of various materials. Most of the CCA method was widely used in the aluminium alloy system to predict the material solidification behaviour. Divergent in cooling rate behaviour which was studied by using CCA method contribute a better understanding of the evolution of dendritic arm spacing that affected the material mechanical properties [195]. Furthermore, deeper analysis on the effect of grain refinement and different processing conditions towards solidification behaviour were analysed and CCA method was used as a tool to characterise the heat evolution within melt [186, 196, 197]. The CCA method assists in allowing useful information in order to control the processing parameters. The CCA was also used to estimate the fraction solid-temperature relations and solidification behaviour during solidification such as eutectic growth temperature which was crucial in SSM processing [198-200].

The CCA method is also used in the investigation of solidification behaviour of magnesium alloy system. The solidification and microstructure formation were comprehensively evaluated in order to estimate the processing parameters and the phase changes in magnesium material [201, 202]. Figure 2.29 shows one of

the CCA method examples, which was used to estimate the solidification behaviour of aluminium alloy AA1050.



Figure 2.29: Cooling curve of aluminium alloy AA1050 with the first derivation curve and a baseline curve by suing cooling curve analysis method [203].

2.6.3 Dendritic Coherency Point

Another thermal analysis method uses two thermocouples to measure the heat change in a single sample. This method uses a large volume of sample during measurement which reflects the real industrial application. The thermocouples are placed at two different locations which are connected to the data capturing system. The first thermocouple is embedded inside the molten alloy at the centre of the crucible and the second thermocouple is located near the crucible wall. The cooling curves for the sample is captured and compared to evaluate the heat change during the solidification process. This method required derivation of the cooling curve to capture the transformation of heat change event and fraction solid. In this method the temperature difference between the solidification cooling curves at two separate points within the melt can be used to determine dendritic coherency points (DCP) [180].

The DCP is the point where during solidification the dendrites begin to impinge on one another across the solidifying system and where the metal strength begins to develop [44, 204]. The solidifying metal has negligible shear strength before DCP compared to after this coherency point occurs where the dendritic network has formed. This point is therefore accompanied by a significant increase system viscosity and strength [204]. Figure 2.30 presents the dendrite at a different fraction solid which it evolved at a high fraction solid volume.



Figure 2.30: Grain size effect on coherency point with (a) a low fraction solid and (b) high fraction solid [205].

2.7 Coefficient of Thermal Expansion

Coefficient of thermal expansion (CTE) is a thermal property which is used to characterise the dimensional variation of alloys. CTE was typically influenced by several factors consist of secondary phase volume [206-208], microstructure [209], magnitude and distribution of internal stresses produced at the interfaces [210], thermal history [211] and porosity [212]. The CTE at the certain temperature can be derived by using the relation which is in Equation 2.14 as follow where *L* is the length of the specimen at a temperature of *T* [213]:

$$\mathsf{CTE} = \frac{\partial}{\partial T} \left(\frac{\Delta L}{L} \right)$$
 Equation 2.14

Previous research has reported the CTE was much influenced by the microstructure. Sample with large and coarse structure was likely exhibited lower CTE. Figure 2.31 shows the CTE of AI-50Si alloy which produced by different methods. The larger and coarse microstructure features typically occurred in cast alloy produced the lowest value of CTE [214]. The CTE is also affected by the inter-atomic distance between structures. The increases in inter-atomic structure distance within sample increase the CTE value. This was due to the un-rigid phase structure expansion, which happened between primary phase structures lead to structure to expand [215]. In view of all that has been mentioned so far, one may suppose that CTE are much influenced by the microstructure within a sample. The lower CTE is desirable to allow better dimensional control of product design. With the increment of structure within sample increase the CTE, thus the suitable microstructure size is needed in SSM processing.



Figure 2.31: CTE curves for the AI-50Si alloy which prepared by other methods [214].

2.8 Motivation/Scope of work

There is interest to process aluminium 7075 within the semi-solid state due to the enhanced properties available from this alloy compared to its cast alloy alternatives. The properties of aluminium 7075 are superior to conventional cast alloy. Aluminium 7075 however is more difficult to process within the semi-solid state due to its narrow solidification range and higher propensity for hot tearing. Although extensive research within the literature has been carried out on aluminium 7075, less attention was given to detailed experimental investigation on thermal profiles and microstructure at various solidification rates. There is also currently a lack of detailed experimental investigation within the literature into the thermal profiles for aluminium 7075 at various solidification rates which may occur during thixoforming. In order to understand the relationship between solidification rate, metallurgical behaviour, and fraction phase evolution, the experimental work which presented in this thesis was conducted. In particular, the liquidus, eutectic and solidus temperature, fraction solid, dendritic coherency point effect resulting from the different cooling conditions of aluminium 7075 was then determined.

In the literature, it was found that the direct thermal method (DTM) was able to produce a spherical microstructure for the semi-solid metal (SSM) feedstock. Publications in recent years demonstrated the effects of different mould material used and the wall thickness investigations. However, the previous research on DTM has not focussed on use of this technique for this raw material. Previous studies have concentrated on the aluminium silicon alloy which has advantages in terms of increased fluidity, but has lower mechanical properties compared with 7075. In DTM, the combination of a suitable pouring temperature and a holding time produce finer microstructure is still not fully understood due also to the vast range of possible casting conditions in terms of alloy, holding time, pouring temperature, mould materials and quenching medium used. The focus of this DTM research was to investigate the relationship between the pouring temperature and holding time of DTM using aluminium 7075, in order to produce a spheroidal microstructure for the SSM feedstock. In addition, the

effects of solidification rate and fraction solid resulting from the different pouring temperature and holding time used were determined in order to allow for setting of these parameters for the thixoforming operation.

There is a lack of detailed information within the literature for the thixoforming of 7075. This is particularly relevant for feedstock billets resulting from the DTM. This thesis is focussed on the design of compression test processing units and the processing of SSM feedstock billets within a semisolid temperature range. This thesis also presents an evaluation of SSM feedstock billet formability. The relationship between microstructure and formability of SSM feedstock billets was analysed and related to primary grain diameter, circularity and secondary phase area measurements.

Mechanical properties characterisation for SSM processing is crucial, especially in order to understand the relationship between microstructure and material properties. A considerable amount of literature has been published on relationship between microstructure formation and mechanical properties of aluminium alloys. However, there has been a lack of information on the mechanical properties of aluminium 7075 DTM feedstock billets which produced by thixoforming. In this thesis, mechanical properties of the DTM feedstock billet after thixoforming were analysed include ultimate tensile strength, yield strength, elongation and hardness. Statistical analysis between processing parameters, microstructure, mechanical properties and formability were particularly performed.

Coefficient of thermal expansion (CTE) is an important property which determines the dimensional design tolerance for the material. It is a very important to understand the relationship between SSM processing parameter that affected the CTE. There are a huge number of publications in the literature studied regarding CTE and its relation between microstructure and processing parameters. Nevertheless, there is very little similar research has performed for SSM thixoforming components. This thesis presented experimental work which conducted on the variation of thixoforming feedstock billets and its effect on CTE. Statistical analysis between related processing parameters and CTE were also determined.

Figure 2.32 shows the outline and flow of the work completed and presented in this thesis. This outline presents input and outputs for each experiment and their relationship to each other. The thermal analysis experimental work resulted in defined thermal profiles within calculated statistical limits. Thermal profiles provide useful information such as liquidus, eutectic, solidus and DCP temperatures which were used as the initial setting parameters for the DTM experiments. Thermal profile information such as liquidus temperature was used to determine pouring temperature in the DTM. This provided the selection of minimum pouring temperature (645 °C) in DTM, approximately 5 °C higher than the liquidus temperature. Thermal profiles information was also used to determine fraction solid volume during the holding period and quenching temperature in the DTM. Thermal profiles information obtained from the thermal analysis experiment was also used as the parameters setting for the compression test experiment. DCP and fraction solid data from the thermal analysis experiment were used to determine the heating temperature of the feedstock billets in compression test. Heating temperature for the feedstock billet was set at 620 °C, 14 °C lower than DCP. Fraction solid volume during heating and compression test of feedstock billet was found approximately at 0.5 and 0.6 respectively. DTM experiment was evaluated in details two components consist with image analysis and statistical analysis. DTM provided SSM feedstock billets which were used for the compression test experiments. The compression test experiments were evaluated in detail with three components consisting of filling distance, image analysis of formed microstructure and statistical analysis. The feedstock billet after the compression test sectioned and tested for resultant mechanical properties and CTE experiment. Properties evaluated included tensile, hardness, density, fractography and statistical analysis. Finally, CTE experiments were conducted in detail to determine the thermal dimensional expansion and repeatability thereof.



Figure 2.32: Outline and relationship between thesis sections.

CHAPTER 3

METHODOLOGIES

3.1 Material Compositions and Sample Preparation

During these years, there was an increased demand to produce components by using lightweight materials with reasonable mechanical properties. Wrought aluminium alloys which withstand higher strength and mechanical properties than aluminium silicon alloys A356 become prevalent in SSM processing research. There were also the vast interests in literature showed that these materials able to be processed by SSM techniques [159, 175, 177, 191]. In view of that, wrought aluminium 7075 was chosen in this experimental work. The 7075 is the aluminium alloys with the highest mechanical properties in aluminium system. The 7075 becomes more favourable than A356 especially in producing aerospace components that required the material to withstand higher stresses.

The chemical composition of the aluminium 7075 alloys used in this experimental work, as determined with Optical Emission Spectroscopy, Foundry Master Oxford Instruments and from the literature, is presented in Table 3.1. The 7075 was supplied by Impact Ireland (Metals) Ltd, Ireland. The chemical composition test was repeated five times in order to acquire accurate results. The test sheet for the chemical composition is presented in Appendix B.

Source (wt%)	AI	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn
Experiment	88.5	0.2	2.02	0.24	2.38	0.12	0.14	0.09	6.04
Bäckerud [44]	Bal	0.19	1.36	0.28	2.49	-	0.11	-	-
ASM [171]	87.1- 91.4	0.18- 0.28	1.2- 2.0	<0.5	2.1- 2.9	<0.3	<0.4	<0.2	5.1- 6.1

Table 3.1: The chemical composition of the aluminium 7075.

3.1.1 Starting Material

Starting material was originally received with dimension of 50 mm in diameter and 1000 mm in length. The starting material was then sliced into smaller pieces with 50 mm in diameter and 20 mm in length by a bend saw. After that, metallography samples were sectioned from the starting material by Delta AbrasimMet abrasive cutter wheel 245 mm in diameter and 1.78 mm thick at 3800 rpm with continuous flowing coolant. The coolant was used in order to avoid deformation and overheating during sectioning. The starting material samples were cut at three different locations as shown in Figure 3.1. The three different locations were chosen in order to observe different microstructure formation at centre and outer areas of the starting material.



Sectioned area

Figure 3.1: Three cross sectioned areas which were used for the starting material analysis.

3.1.2 Thermal Analysis Sample

The thermal analysis solidified alloys which transferred out from a graphite crucible were sliced at the centre by a bend saw. Later, the specimens were sectioned at a centre and both of crucible walls at 45 mm from the bottom of the crucible. The three different locations were selected to examine differences in microstructure formations. The selected locations were also the place where the end tip of the thermocouples located. During solidification, the microstructure is supposed to solidify first at both walls of graphite crucible. This happened as a result of heat convection to the environment through the crucible wall. The solidification at the centre of the melt occurs in the later stages. The schematic for the thermal analysis, specimen sectioned areas are presented in Figure 3.2.



Figure 3.2: Cross sectioned areas for thermal analysis specimens.

3.1.3 Direct Thermal Method Sample

Solidified alloy of the DTM sample with initial dimensions of 25 mm in diameter and 100 mm in length were removed from the copper moulds and later machined by a Computer Numerical Control (CNC) lathe machine at 20 mm from the top and 5 mm from the bottom of the DTM sample. These locations were selected in order to give a minimum balance of 75 mm for the final DTM feedstock billets. The micrograph samples for each solidified alloy were obtained from the end of surface which sliced at 20 mm from the top. The schematic for DTM feedstock billet machining dimensions are shown in Figure 3.3.



Figure 3.3: Schematic for feedstock billet after DTM with initial and final dimensions.

3.1.4 Filling Distance Sample

Feedstock billets after forming were machined by Electrical Discharged Machine (EDM) wire cut across the horizontal line at the conical area as shown in Figure 3.4. The feedstock billets were machined as such in order to obtain the microstructure formation at the end of the feedstock billets after forming. The specimens for micrograph were gained from the surface of these conical areas. The remaining balance from the feedstock billets were used for tensile test specimens.



Figure 3.4: Sectioned area for the filling distance sample for micrographs.

3.1.5 Mounting, Grinding and Polishing Sample

Samples were mounted to provide a protection to sample and to create a uniform surface for subsequent automatic grinding and polishing processes. The samples mounted with Bakelite thermoset phenolic resin by using Buehler Simplimet 2000 Mounting Press hot mounting machine. The curing temperature was set at 180 °C for 8 minutes and cooled for 4 minutes.

Mounted samples were ground by using 240, 800, and 1200- grit size silicon carbide (SiC) for 4 minutes, at 10 N force pressure and at a 200 rpm grinding wheel speed with flowing water. The flowing water helps to remove heat and flush any loose particles of metals and abrasive. These initial polishing steps are called planar grinding.

Ground samples were then polished by the Ultra Pad with 9 and 3 micron; and Acetate Silk with 1 micron size diamond suspension. The samples were then given a final polish with Chemomet with 0.05 micron size alumina polishing suspension for 6 minutes each, at 10 N pressures and the polishing speed of 150 rpm. Buehler Motopol 2000 grinder/polishing machine were used to perform both of grinding and polishing works. Table 3.2 summarised the grinding and polishing work.

Process	Surface	Abrasive	Lubricant	Duration	Base Speed	Head Speed/ Direction	Force
				(min)	(RPM)		(N)
Planar grinding	SiC	600, 800, 1200- grit	H2O	4	200	120/complementary	10
	Ultra Pad	9 μm Diamond suspension		6	150	120/complementary	10
Sample integrity	Ultra Pad	3 μm Diamond suspension		6	150	120/complementary	10
	Acetate Silk	1 μm Diamond suspension		6	150	120/complementary	10
Final Polish	Chemomet	0.05 µm Alumina suspension		4	150	120/complementary	10

Table 3.2: Parameters for Aluminium 7075 grinding and polishing.

3.1.6 Chemical Etching and Micrographs

Chemical etching was used to reveal the grain boundaries of the material under optical microscope after polishing process. During the grinding and polishing processes, thin layer on material surface was formed. The chemical etching removed this thin layer by attacking the surface with the highest energy leading to surface relief that to be distinguished under reflective light. The samples then were etched by using Keller's etch for approximately 30 seconds [216]. Compositions of the etchant were 95 ml water, 1 ml hydrofluoric acid HF, 1.5 ml hydrochloric acid HCL and 2.5 ml nitric acid HNO_3 .

Reichert ME F2 universal camera optical microscope was used to view the microstructures. Buhler Omnimet Enterprise software was then used to capture the microstructure images by using a 8x, 10x, 20x and 40x magnification.

3.2 Thermal Analysis

A variety of methods are used in these recent years to assess thermal profiles in SSM processing which has its own advantages and drawbacks. In current experimental works, thermal analysis experiments were conducted to accurately determine thermal profile information, especially the relationship between Temperature - Fraction Solid ($T_a - f_s$) and dendritic coherency point (DCP). The thermal profile information was used to determine initial processing parameters which were used for DTM and Compression Test. The thermal profile information was also used to predict the f_s volume within feedstock billets during experimental process. Higher f_s volume amount within sample makes it impossible to flow well into a die cavity. Meanwhile, lower f_s volume amount within sample makes it difficult to transfer from the furnace to a forming die.

The estimation of liquidus, DCP, eutectic and solidus temperatures therefore are crucial. The selection of proper temperature during feedstock billets reheating process avoids the excessive primary α -AI phase change which typically happens between DCP and liquidus range temperature. The change of primary α -AI phase during SSM feedstock billets reheating process was reduced feedstock billets flowability during injection processing [44].Since, several SSM research in the literature showed DSC was used to estimate the thermal profiles during processing, but a lack of information about DCP was presented [186, 199]. Furthermore, the size of the sample used in DSC was too small compared to typical sample at a casting floor. The thermal analysis in present experimental works provides a wide range of useful information regarding aluminium 7075 thermal properties during solidification.

Thermal analysis results merely depended on the cooling rate which was used during solidification [184]. In order to replicate similar conditions that were used in both DTM and Compression Test, the cooling rates were fixed at four different cooling rate conditions. These conditions were meant to achieve a slow, an intermediate, high and the highest cooling rate conditions base on previous works presented by Båckerud et al [44]. The experiment setup for all conditions was constructed by the following procedures.

Graphite crucible of 80 mm in diameter and 100 mm in height was used to melt the metal. An aluminium 7075 billet with a weight of 750 g was placed into the graphite crucible before each experiment. The crucible was then heated to a temperature of 750 °C by using Ambrell Easy Heat 2.4 kW Induction Heating. The crucible with molten metal then was transferred to the kaowool chamber in order to achieve the slow cooling rate. The specially designed chamber with kaowool insulation contained 100 mm of kaowool beneath the crucible, 50 mm side walls, and a 40 mm thick top layer of kaowool in order to ensure very slow cooling. Chromel-alumel K-type thermocouples were located at two different locations, one at the centre of the crucible and one at 35 mm from the centre closer to the crucible wall. Both thermocouples were immersed within the metal to a depth of 45 mm from the top of the melt. The experimental setup for the slow cooling rate is presented in Figure 3.4.

The similar size of the graphite crucible with 750 g aluminium 7075 billet were heated to a temperature 750 °C and allowed to cool naturally without a kaowool blanket for the intermediate cooling rate, placed in a minimum forced rate for high cooling rate or in a maximum air flow to achieve the highest cooling rate. The chromel-alumel K-type thermocouples also were located at similar places to the slow cooling rate setup.



Figure 3.4: Schematic for thermal analysis experiment set-up for slow cooling rate by using two thermocouples method.

Experiment Number	Conditions	Medium		
1				
2	Slow Cooling	Kaowool Blanket		
3				
4				
5	Intermediate Cooling	Cool Naturally		
6				
7		Minimum Force Air Flow		
8	Higher Cooling			
9				
10		Maximum Force Air Flow		
11	Highest Cooling			
12				

Table 3.3: Overall experimental layout for thermal analysis.

After the cooling curves were captured, the cooling rates were calculated from the portion of the cooling curve above the liquidus temperature (between 10 and 50 °C above the liquidus temperature). The thermocouples were calibrated before each recording to ensure accurate readings. The thermal analysis experiments for each condition were repeated three times to ensure reproducibility of the results. The layout of the overall experimental works for thermal analysis is presented in Table 3.3.

The temperature versus time cooling curve data was recorded by using an NI 6036 data acquisition (DAQ) card with a cold junction compensation. A series of programs developed in LabVIEW allowed control of data acquisition rate, as well as calculation of the cooling curve differential (dT/dt curves) and fraction solid with respect to time. A DAQ rate of 500 Hz was set. For the slow cooling rate conditions this equated to 5,000,000 data points captured during the experimental period, approximately of two and a half hours. For the intermediate, high and highest cooling rate conditions there were approximately 1,500,000 data points (50 minutes), 1,150,000 data points (38 minutes) and 1,000,000 (33 minutes) respectively. The examples of LabVIEW programming used in thermal analysis experiments are presented in Figure 3.5 and Appendix C.



Figure 3.5: Example of the LabView programming which was used in thermal analysis experiments.

A base line was constructed on the dT/dt curves to represent the cooling rate which would have occurred in the absence latent heat evolution. The area between this curve and the actual cooling curve differential was calculated at each time point and divided by the total area between these curves in order to determine the fraction solid versus time curves, as per Equation 3.4. In the solidification process, the heat loss may be equated to the heat conducted away and can be formulated according to Equation 3.1 as follows [44]:

$$V \rho C_p \frac{dT}{dt}\Big|_{bc} = V k(T_{metal} - T_{mould})$$
 Equation 3.1

where V is the volume, ρ is the density, C_p is the specific heat coefficient, $\frac{dT}{dt}\Big|_{bc}$ is the rate of change of the base line curve, k is the thermal conductivity and $(T_{metal} - T_{mould})$ is the difference between the temperature of the metal and mould. The latent heat, H that evolves during solidification can be represented by Equation 3.2 as follows:

$$\frac{dH}{dt} - V \rho C_p \left. \frac{dT}{dt} \right|_{bc} = V k (T_{metal} - T_{mould})$$
Equation 3.2

Adding equation 1 and 2 gives a relation between the effects of latent heat on dT/dt curve that can be written as in Equation 3.3:

$$\frac{dH}{dt} = V \rho C_p \left[\frac{dT}{dt} \Big|_{cc} - \frac{dT}{dt} \Big|_{bc} \right]$$
 Equation 3.3

The fraction solid f_s , present at a given time (t_1) during solidification is expressed by Equation 3.4 where the ratio of the amount of latent heat evolution until time t_1 , to the total amount of latent heat evolution during solidification can be written as:

$$f_{s} = \frac{\int_{t_{0}}^{t_{1}} dH}{\int_{t_{0}}^{t_{f}} dH} = \frac{\int_{t_{0}}^{t_{1}} \left[\frac{dT}{dt}\Big|_{cc} - \frac{dT}{dt}\Big|_{bc}\right] dt}{\int_{t_{0}}^{t_{f}} \left[\frac{dT}{dt}\Big|_{cc} - \frac{dT}{dt}\Big|_{bc}\right] dt}$$
Equation 3.4

In order to determine the fraction solid at specific times (Δ t), area integration value at Δ t was divided with the total area integration values measured from start to end of solidification. The area integration value was calculated between dT/dt and baseline curve by using the area under graph concept. The value of fraction solid for each cooling rate at any point between the start and end of solidification was determined by using this method. Figure 3.6 as follows shows the example of the dT/dt and the base line curve for fraction solid calculation.



Figure 3.6: Cooling curve from the centre with derivative and calculated baseline [44].

Separately, the temperature recorded from the thermocouple at the crucible wall was subtracted from the temperature reading from the centre thermocouple at each time. This temperature difference was plotted against time. The DCP was then determined by identifying the maximum difference between these two readings.

3.3 Direct Thermal Method

There is a large volume of published works in the literature describing methods to produce SSM feedstock for thixoforming operation. In this experimental works, the DTM was chosen because this method only required an easy experimental step-up and a low processing cost to produce multiple feedstock billets. Furthermore, there is a lack of information in the literature on aluminium 7075 which used the DTM to produce a SSM feedstock. Results from this experimental work provides useful information regarding aluminium 7075 behaviours prepared with the DTM for thixoforming feedstock.

The experiment setup for DTM was conducted by the following procedures. A 1 kg aluminium 7075 ingots was placed in a graphite crucible and was heated to a temperature of 700 °C by using the resistance heated Carbolite 1600 box furnace. Once the desired temperature of the melt was obtained, it was poured into a cylindrical copper mould with 1 mm wall thickness, 25 mm in diameter, and 100 mm in height. The different pouring temperatures were set at 645 °C, 665 °C or 685 °C. After pouring molten metal into the mould, it was held for 20 s, 40 s or 60 s respectively. After the holding time for each copper mould was achieved, it later quenched into room temperature water. In order to capture microstructure for the billet with the normal solidification condition, another billet which poured with 685 °C molten alloy were allowed to solidify without quenching. Schematic diagram for DTM apparatus is presented in Figure 3.7 as follows:



Figure 3.7: Schematic diagram for DTM used in the experimental work with (a) experimental set-up and (b) dimension for the copper mould.

The copper moulds were later machined by using a lathe to separate the aluminium billets and copper moulds. The condition of copper moulds after quenching is shown in Figure 3.8. The experiment layout for DTM is presented in Table 3.4. The experiment was repeated three times to ensure repeatability of the result with every single cycle of the experiment consist of 10 experiment numbers.



Figure 3.8: The copper moulds after quenching, containing solidified aluminium 7075 billets.

Experiment Number	Pouring Temperature (°C)	Holding Time (s)		
1	005	60		
2	685	40		
3		20		
4		60		
5	665	40		
6		20		
7		60		
8	645	40		
9		20		
10	685	Without quenching		

Table 3.4: The DTM experiment layout with 10 different processing parameters.

3.4 Compression Test Unit

Various methods in literature have been developed and introduced to evaluate SSM feedstock formability. In this present work, a compression test unit was used to evaluate the performance of the DTM feedstock billets. The principle of thixo route was applied in this experiment which the feedstock billet heated to a SSM range temperature in order to catalyse the formation of a globular microstructure. At present, there is limited information in previous studies
regarding the behaviour of DTM feedstock billets that undergoes a thixo route. Most of the research purely concentrates on the microstructure characterisation of the DTM feedstock billets especially on aluminium A356. The present experimental works evaluate the behaviour of aluminium 7075 DTM feedstock billets that processed with a thixo route. The findings from these works provide a brief explanation regarding 7075 behaviours during and after the thixo process.

The machine was designed, built and commissioned in order to be used to evaluate various types of DTM billet microstructures. The machine was the combination of a hydraulic press machine and a custom-made forming die. The die inlet area was designed according to the DTM feedstock billets dimension. The custom-made forming die had also a conical shape cavity in order to access the formability of feedstock billets.

3.4.1 Design Overview

The design and construction of the machine consist of four main major components. The components are a hydraulic press machine, an upper and a lower plate and a forming die. The compression test unit is placed near to the heating furnace to allow a shorter transportation period of feedstock billets from the furnace, shortly after the heating process. The machine was designed with specific requirements in order to make sure the success of the operation.

The die material was chosen from a high heat resistant material because it needs to process the feedstock billets in the range of temperature between 600 to 620 °C. The H13 tool steel was chosen for the die material as it has the favourable characteristics. Properties of the material also required a high deformation resistant when heat is applied. This is to make sure the plunger movement is smooth when entering the forming die. The hydraulic press machine also needs to able to apply a sufficient pressure in order to inject the feedstock billet into a die cavity.

The basic concept operation of compression test unit is just similar to a conventional press machine which uses a comprehensive force to impulse material into a die cavity. The plunger which is located at the upper plate area on the top of machine forming area is being used to push a feedstock billet directly into a die cavity. The operation was started with the plunger was fetched to the start position without force. The die was later heated in order to avoid excessive heat lost from the billets which affect the f_s volume within feedstock billet. The feedstock billet after that was placed into the forming die for compression operation after it was heated by a box furnace. The start button was then triggered in order to make sure the upper plate was moved down to the lower plate. After several minutes, the plunger was lifted automatically back to its original position. The die lastly was opened to remove the injected billet.

The top of the compression test has a control unit which controls the overall operation of the machine. Schematic of the compression test unit which was used in this work is presented in Figure 3.9. A more detailed engineering drawing of the compression test unit is shown in Appendix D.



Figure 3.9: Schematic view of the semi-solid processing unit used in this work.

3.4.2 Hydraulic Press Machine

The machine used a hydraulic system to transmit force with the aim to inject feedstock billet into a die cavity. The machine was designed in a way that the force applied from the top to the bottom direction. This machine has the maximum force capacity of 215 kN which controlled by a pressure gauge which able to regulate the force in the range of 31 to 215 kN. The operation of the machine is controlled by the electrical switch panel, which is attached on the top of the machine area. The machine also has six K-type thermocouples which are mounted at the top of the machine. The six K-type thermocouples are connected to a machine controller by using a 1 m wire. The machine has a packing time controller, which determines the period for the plunger to stay inside the forming die before retract back to its original position. The packing time controller is capable to control the time period in the range of 1 to 90 seconds. Two controlled switches equipped with a sensor as a machine guarding to ensure the safety of operator.

3.4.3 Upper Plate

The upper plate was located at the top of the machine forming area as shown in Figure 3.9 (b). The function of the upper plate was to assemble several parts consist of a plunger and four guide posts. The plunger was used as a pushing rod that transfers the force from the hydraulic system to the SSM feedstock billet with the aim to inject the SSM feedstock billet to the forming die cavity shape. The guide posts were used to align the upper die position in order to make sure the plunger was correctly entered the forming die during operation. The upper plate and guide post were made from the stainless, steel while plunger from H13 Tool Steel due to its high temperature resistance characteristic.

3.4.4 Lower Plate

The lower plate was located at the bottom of machine forming area as shown in Figure 3.9 (b). The lower plate was used to assemble the forming die and four

guide bushes. The forming die was the important part in this system which used to form the SSM billets. The guide bushes were used to make sure the guide posts were well aligned before the plunger entered the forming die. The forming die schematic with detail dimensions is presented in Figure 3.10. The lower plate and guide bushes were made from the stainless steel, while forming die from H13 Tool Steel due to its high temperature resistance characteristic.



Figure 3.10: The schematic of forming die which was used to form SSM feedstock billet.

3.4.5 Filling Distance Test

The feedstock billet was placed into a 35 mm in diameter and 50 mm in height steel crucible. The steel crucible was later placed into a box furnace. The furnace was set to a temperature of 640 °C and remains constant for 30 minutes at that temperature. Concurrently, the forming die was heated to a temperature of 115 °C by using a flammable torch. A K-type thermocouple was placed into the die opening to measure the temperature inside the die. The packing time of the die which determines the plunger to stay inside the die was set for 5 s. The machine force was set at 185 kN from the maximum capability of 215 kN for safety purposes. The steel crucible which contains feedstock billet was transferred into the forming die and transport time was timed. The press button which operated the upper plate into the lower plate was pushed in order to start the forming process. Once the billet and the forming die were at room temperature, the billet was removed from the forming die. The dimensions of the formed billet were measured by using height gauge. The formed billets were measured from the base to the highest billets end for overall length. Another length was measured from the base to the highest end before the conical area. The experiments were performed with three experiment cycles with 10 samples for each experiment which in total 30 experiments were conducted.

3.5 Mechanical Properties

The mechanical properties of SSM components were evaluated with the intention to characterise their mechanical properties behaviours after forming process. The feedstock billets evaluation, which was obtained after the compression consists of tensile and hardness test. The feedstock billets after forming were machined by CNC lathe according to specific dimensions required for each consequence test. In total, 30 feedstock billets after forming were prepared for tensile test specimens. The hardness test specimens were obtained from the grip area of tensile tests. The specimen with 12 mm in diameter and 12 mm in length were cut by an abrasive cutter wheel and were polished to obtain flat, smooth and parallel surface. The schematic and detail dimensions for tensile sample are presented in Figure 3.11.

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Figure 3.11: Schematic for the tensile specimens.

3.5.1 Tensile Test

Tensile tests were conducted by using a Zwick Z50 BT1-FB050TN 50kN Universal Testing Machine equipped with TestXpert 2 V 2.1 software. Tensile specimen with dimension of 8 mm in diameter, 10 mm gauge length and 2.5 mm transition radius were clamped to the machine grips. In order to reduce error in displacement readings, the extensometer was used to replace crosshead displacement readings. The crosshead displacement readings are taken from the motor encoder which incorporates any movement in the grips, pulleys, screws and belts. The use of crosshead displacement readings produces a significant larger crosshead travel reading. The extensometer with 10 mm gauge length and 1 mm extension was used. The steel spring was used to mount the extensometer to the specimen to avoid knife edge slippage during the test. The special extension knife edge with 28 mm length was used to ensure enough clearance between upper and lower grips. Test speed was set at 1 mm/min due to the small size of the specimen with the preload were set at 6 MPa. The tensile specimen with an extensometer mounted is presented in Figure 3.12.



Figure 3.12: Extensometer mounted to the tensile sample with a long knife edge to ensure enough clearance between grips before the start of test.

3.5.2 Hardness Test

The hardness of the feedstock billets after forming was tested by a MAT10/RAB Rockwell and Brinell Hardness Model 206RT machine. This machine uses a diamond cone having an included angle of 120 ° and a radius of curvature at the tips of 0.2 mm is forced into the surface of the test specimen. A 100 kgf load and 20 seconds holding time were used during these tests. The Rockwell hardness value, HR reading was obtained directly from the machine and it also can be calculated by using the formula in Equation 3.5 as follows [217]:

$$HR = N - \frac{h}{s}$$
 Equation 3.5

where N is the number specific to Rockwell hardness scale; 100 for scales A, C, D 15 N, 30 N, 45 N, 15 T, 30 T and 45 T, and 130 for scales B, E, F, G, H and K. Meanwhile, h is the permanent depth of indentation, in mm under preliminary test force (minor load) just after removal of the additional test force. S is the scale unit specific to the Rockwell hardness scale; 0.002 mm for scales A, B, C, D, E, F, G, H and K, and 0.001 mm for scales 15 N, 30 N, 45 N, 15 T, 30 T and 45 T.

3.5.3 Fractography

The fracture surface of the tensile specimen was examined by using an EVOLS15 Scanning Electron Microscope (SEM) machine in order to determine the cause and mode of sample fracture. The specimens were directly placed in the SEM chamber after the tensile test to avoid oxidation of the sample surface. The driving voltage was set at 15 kV which an optimum setting for aluminium alloys. Composition study was then performed by using EDX probe and software. The working distance was set at 11 mm in order to obtain sufficient data.

3.6 Grain Size Measurements

Primary phase grain size diameter, circularity, aspect ratio and secondary phase area measurement were determined by Image J software. The circularity is an indication of a perfect circle which occurs within a microstructure. The value that approaches a value of 1.0 is considered as the perfect circle with decreasing number toward 0.0 is indicates an increasing elongated shape. The aspect ratio is an indication for circular or square morphology, which aspect ratio value increases with an elongated particle. The circularity, C and aspect ratio, AR were calculated by using the following formula as in Equation 3.6 and Equation 3.7 which P and A are representing a perimeter and an area of the particle respectively:

$$C = 4\pi/P^2$$
 Equation 3.6

AR = major axis/minor axis Equation 3.7

The measurement was performed by the 'analyse particles' menu in the Image J software. The micrographs initially were converted to a Red, Green, Blue (RGB) Colour format before the brightness and contrast were properly adjusted to distinguish between grain boundaries and solid particles. 'Threshold' menu was then selected and used to adjust the high contrast micrographs in order to

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measure the number of grains. The type of measurement and scale setting on the micrograph were determined before analyse the particles. The example of the threshold image and analysis are presented in Figure 3.13. The circularity and aspect ratio measurement are only involved with the selection of suitable particles which grain boundaries were not touched the micrograph frame.





3.7 Energy-dispersive X-ray Spectroscopy Analysis

The Energy-dispersive X-ray Spectroscopy (EDXS) analysis was performed by an Oxford Instrument Inca Energy 350XT machine as shown in Figure 3.14 to measure the chemical composition of the direct thermal method and compression test samples. This system uses a Carl-Zeiss EVO-LS15 Scanning Electron Microscope (SEM) to observe the sample structure while EDXS detectors disperse chemical composition data. The EDXS detector and sample surface were set less than 11 mm in order to collect sufficient data. The data which obtained from the EDXS detector were then analysed by Inca Software.



Figure 3.14: Carl-Zeiss EVO-LS15 SEM which was used for EDXS analysis.

3.8 Porosity Measurement

Porosity levels for each feedstock billets after forming were determined by density measurements. The feedstock billets after forming were mass weighted by an electronic weight scale. After that, the feedstock billets after forming were submerged into water and the water readings before and after placed feedstock billets were recorded. The density of feedstock billets after forming later was calculated by Archimedes' principle as presented in Equation 3.8 as follows:

$$\rho_{\rm fb} = \frac{W_{\rm fb} \times \rho_{\rm H_2O}}{W_{\rm H_2O}}$$
 Equation 3.8

Where $\rho_{\rm fb}$ is the density of feedstock billet after forming in g/cm³; W_{fb} is the weight of the feedstock billet measure by electronic scale in g and W_{H₂O} is the weight of feedstock billets fully submerged measurement readings in ml. Meanwhile, $\rho_{\rm H_2O}$ is the density of the water with the value of 1 g/cm³.

3.9 Coefficient of Thermal Expansion Measurement

The selected feedstock billets after compression test consist of sample 1, 2, 3 and 10 were cut by an abrasive cutter wheel from the end of the tips of tensile sample. The samples were selected in order to ensure the vast changes between grain sizes give a significant effect to thermal dimension expansion result. The dimensions for each feedstock billet which were used in the thermal expansion experiment are presented in Table 3.5. The thermal dimension expansion measurement was conducted with NETZSCH DIL 402 PC dilatometer. The schematic of the dilatometer is presented in Figure 3.15. Sample was placed into the machine cylindrical container between two stamps. The temperature was set to a temperature of 500 °C with heating and cooling rate was set at 5 °C/min. During the measurement the nitrogen gas was applied to the systems with a flow rate of 100 ml/min.

Table 3.5: Dimensions for each sample which was used in thermal dimension expansion experiment.

Sample No	Original Length (mm)	Original Diameter (mm)	Grain Size (µm)
1	12.24	12.02	124
2	12.28	12.05	116
3	12.11	12.08	110
10	12.17	12.09	136



Figure 3.15: Schematic view for the NETZSCH DIL 402 PC dilatometer.

3.10 Statistical Analysis

IBM SPSS STATISTICS (version 21) software was used to perform statistical analysis of the results. The main purpose of a two-way Analysis of Variance (ANOVA) was to understand if there was an interaction between the two independent variables on the dependent variable [218]. The two-way ANOVA was also used to compare the mean differences between groups which were divided into two independent variables (called factors). The two independent variables which were used in DTM experimental work consist of pouring temperature and holding time. On the other hand, the dependent variables consist of average primary grain diameter, circularity, aspect ratio and secondary phase area. The interaction in a two-way ANOVA reflected the effect of one of the independent variables or vice versa. The significant interaction indicated there were simple main effects and what the effects were.

One-way ANOVA was used to determine whether there were any significant differences between the means of an independent variable [218]. The one-way ANOVA was used to understand whether the average overall length A-B was differed based on transfer time on Compression Test. The one-way ANOVA was also used to recognise whether the overall length A-B was changed by variation of average primary grain diameter, circularity and average secondary phase area.

Pearson product moment correlation coefficient or Pearson's correlation was used to measure the strength and direction of association which existed between the two results, for instance between Ultimate Tensile Strength and Density. Correlation coefficients range from -1 to 1, with stronger correlations represented by high absolute correlation coefficients. The positive correlation coefficient is represented by the two variables trend in the same direction which, if one variable increase another variable is also increasing. Meanwhile, negative correlation shows the two variables trend in the opposite direction which, if one variable is increased another variable is decreased. A Pearson's correlation is

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also provided the line of best fit through the data of two variables which gives further understanding on the trend of the results.

CHAPTER 4

RESULTS

4.1 As received sample

Microstructure for a starting material is shown in Figure 4.1. The grain structure formation was elongated and un-recrystallised. These grain structure formations were shown by the contrast of variations with the formation of elongated grains, with several porosities detected.



Figure 4.1: Optical microscope image for as received sample of aluminium 7075.

4.2 Cooling Curve Analysis

4.2.1 Slow Cooling Rate at 0.13 °C/s

Cooling curve for a slow cooling rate condition (at the cooling rate of 0.13 °C/s which calculated at several points before liquidus temperature) is shown in Figure 4.2 (a). The results which obtained from Figure 4.2 (a) show the liquidus, eutectic and solidus temperature occurred at temperature of 638 °C, 474 °C and 472 °C respectively. Further analysis was made to the cooling curve, which later produced the first derivation of the cooling curve with a baseline graph as presented in Figure 4.2 (b). This derivation curve allowed the identification of phase changes which occurred within alloys during solidification. The vertical lines as in Figure 4.2 (b) represent the start (liquidus point) and ends (solidus point) of solidification. The calculated fraction solid versus temperature graph is shown in Figure 4.2 (c). The information in Figure 4.2 (c) is particularly useful to determine a suitable temperature (T_a), fraction solid (f_s), $T_a - f_s$ relations for SSM processing.

4.2.2 Intermediate Cooling Rate at 0.32 °C/s

Cooling curve for an intermediate cooling rate condition (at the cooling rate of 0.32 °C/s which calculated at several points before liquidus temperature) is shown in Figure 4.3 (a). As can be seen in Figure 4.3 (a), the liquidus, eutectic and solidus temperature occurred at temperature of 638 °C, 474°C and 472 °C respectively. Figure 4.3 (b) shows the first derivation of the cooling curve with a baseline as well as vertical lines which indicate the start and end of the solidification. The corresponding calculated relations of $T_a - f_s$ is presented in Figure 4.3 (c).

4.2.3 High Cooling Rate at 0.36 °C/s

Cooling curve for a high cooling rate condition (at the cooling rate of 0.36 °C/s which calculated at several points before liquidus temperature) is shown in Figure 4.4 (a). The liqudus, eutectic and solidus temperature was at a

temperature of 640 °C, 476 °C and 472 °C respectively. Figure 4.4 (b) shows the first derivation of the cooling curve with a baseline as well as vertical lines which indicate the start and end of the solidification. The corresponding calculated relation of $T_a - f_s$ is presented in Figure 4.4 (c).

4.2.4 Highest Cooling Rate at 0.39 °C/s

Cooling curve for the highest cooling rate condition (at the cooling rate of 0.39 °C/s which calculated at several points before liquidus temperature) is shown in Figure 4.5 (a). The liquidus, eutectic and solidus temperature was at temperature of 639 °C, 474 °C and 471 °C respectively. Figure 4.5 (b) shows the first derivation of the cooling curve with a baseline as well as vertical lines which indicate the start and end of the solidification. The corresponding calculated relation of $T_a - f_s$ is presented in Figure 4.5 (c).



Figure 4.2: For a cooling rate of 0.13 °C/s the (a) cooling curve, (b) cooling curve derivation with respect to time (with a baseline) and (c) calculated temperature-fraction solid relation.



Figure 4.3: For a cooling rate of 0.32 °C/s the (a) cooling curve, (b) cooling curve derivation with respect to time (with a baseline), and (c) calculated temperature-fraction solid relation.



Figure 4.4: For a cooling rate of 0.36 °C/s the (a) cooling curve, (b) cooling curve derivation with respect to time (with a baseline), and (c) calculated temperature-fraction solid relation.



Figure 4.5: For cooling rate of 0.39 °C/s the (a) cooling curve, (b) cooling curve derivation with respect to time (with a baseline), and (c) calculated temperature-fraction solid relation.

4.2.5 Dendritic Coherency Point

Two thermocouples method is designed specifically to measure the coherency point (DCP) of the sample. The DCP is determined by measuring the first maximum temperature difference which occurred between the thermocouple at the centre and wall of crucible ($T_{Difference} = T_{wall} - T_{centre}$) [180, 219]. The temperature differences between the first and second thermocouple are shown in Figure 4.6 (a) and Figure 4.6 (b) for the slow and intermediate cooling rate while Figure 4.7 (a) and Figure 4.7 (b) for the high and highest cooling rates.

The DCP for the slow cooling rate occurred at 675 s as shown in Figure 4.6 (a). This value was then compared with the cooling curve formation in Figure 4.2 (a) which indicated DCP for the slow cooling rate was at temperature of 634 °C. Similarly, the DCP for the intermediate cooling rate was determined accordingly, which occurred at 466.4 s with a temperature of 634 °C as shown in Figure 4.6 (b) and Figure 4.3 (a). The high cooling rate DCP was appeared at 414.1 s (see Figure 4.7 (a)) which corresponded to a temperature of 632 °C (Figure 4.4 (a)). Finally, the coherency point for the highest cooling rate was at 391.2 s and temperature of 634 °C, see in Figure 4.7 (b) and Figure 4.5 (a).



Figure 4.6: Temperature differences between thermocouples located at the centre and wall of the crucible with different cooling rates of (a) 0.13 °C/s and (b) 0.32 °C/s.



Figure 4.7: Temperature differences between thermocouples located at the centre and wall of the crucible with different cooling rates of (a) 0.36 °C/s and (b) 0.39 °C/s.

4.2.6 Microstructure

In order to assess the effects of cooling rate on the microstructure, the qualitative comparison method was used. The cross-sectional area of microstructure images for different cooling rate conditions at the centre, a crucible wall (right) and a crucible wall (left) of solidified aluminium 7075 after thermal analysis is presented in Figure 4.8, Figure 4.9 and Figure 4.10 respectively. The images show the microstructure formation was different to each other due to the cooling rate variant applied. It was also apparent from these results, the obvious formation between primary (solid) and secondary (liquid) phase. The primary phase was the structure which first to precipitate during solidification, while secondary phase occurred at the boundary of the primary phase which precipitated last within the solidified alloy.



Figure 4.8: Microstructure formations at the centre of solidified material after removed from a graphite crucible with different cooling rate conditions consist of (a) 0.13 °C/s, (b) 0.32 °C/s, (c) 0.36 °C/s and (d) 0.39 °C/s.



Figure 4.9: Microstructure formations at the wall (right) of the solidified material after removed from a graphite crucible with different cooling rate conditions of (a) 0.13 °C/s, (b) 0.32 °C/s, (c) 0.36 °C/s and (d) 0.39 °C/s.



Figure 4.10: Microstructure formation at the wall (left) of the solidified material after removed from a graphite crucible with different cooling rate conditions of (a) 0.13 °C/s, (b) 0.32 °C/s, (c) 0.36 °C/s and (d) 0.39 °C/s.

Microstructure formation for the slow and the highest cooling rate condition were viewed closely in order to observe the substantial changes. Cross-sectional area for microstructure images for the slow and the highest cooling rate at the centre of solidified aluminium 7075 after thermal analysis are presented in Figure 4.11. The result shows that the difference between the slow and the highest cooling rate microstructure was evident. It was apparent that smaller size microstructure was formed within the highest cooling rate condition sample.



Figure 4.11: Higher magnification images for microstructure at the centre of solidified aluminium 7075 after removed from a graphite crucible with different cooling rate conditions of (a) 0.13 °C/s, and (b) 0.39 °C/s.

4.2.7 Grain Size Measurement

In order to assess the effect of the different cooling rate used to microstructure formation, the quantitative method was used. The quantitative method involved with the grain size measurement which was performed to evaluate the average primary grain structure diameter, perimeter, circularity and aspect ratio of the microstructure. The grain size measurement results for microstructure at the centre solidified material are shown in Figure 4.12 and Figure 4.13. Likewise, the grain size measurement for microstructure at the wall (right) of solidified material are presented in Figure 4.14 and Figure 4.15. The grain size measurements for microstructure at the wall (left) are presented in Figure 4.16 and Figure 4.17.

The grain size measurement for microstructure at different cooling rate conditions shows that the increment in cooling rate (from the slow (0.13 °C/s) to the highest (0.39 °C/s) cooling rate), the average primary grain diameter and perimeter was decreased. In contrast, it was apparent from the results obtained the average microstructure circularity and aspect ratio was increased.

The average grain diameter and perimeter results also present the significant value between 0.13 and 0.39 °C/s cooling rates (see Figure 4.12). The significant value was presented by the results that did not overlapped to each other. However, the insignificant value was found when comparison between 0.13 and 0.32 °C/s, 0.32 and 0.36 °C/s, 0.36 and 0.39 °C/s cooling rates was made. The results were found overlapped to each other. Moreover, the average circularity and aspect ratio results were found insignificant due to overlapped results with each other (see Figure 4.13). This gives an indication that with the variance of cooling rate conditions used in this experiment, the circularity of the sample was not changed much.



Figure 4.12: Grain size measurement for microstructure at the centre of solidified material with (a) average microstructure diameter and (b) average microstructure perimeter (errors are 95% confidence intervals).







Figure 4.14: Grain size measurement for the microstructure at wall (right) of solidified material with (a) average microstructure diameter and (b) average microstructure perimeter (errors are 95% confidence intervals).



Figure 4.15: Grain size measurement for the microstructure at wall (right) of solidified material with (a) average microstructure circularity and (b) average microstructure aspect ratio (errors are 95% confidence intervals).



Figure 4.16: Grain size measurement for the microstructure at wall (left) of solidified material with (a) average microstructure diameter and (b) average microstructure perimeter (errors are 95% confidence intervals).



Figure 4.17: Grain size measurement for the microstructure at wall (left) of solidified material with (a) average microstructure circularity and (b) average microstructure aspect ratio (errors are 95% confidence intervals).

4.3 Direct Thermal Method

4.3.1 Microstructure formation

Comparison between DTM samples was made by examining the microstructure formation which occurred within DTM samples. The microstructure for pouring temperature of 685 °C, 665 °C and 645 °C with holding time of 60 s, 40 s and 20 s are presented in Figure 4.18 to Figure 4.20 respectively. Likewise, the microstructure for pouring temperature of 685 °C which was allowed to solidify without quenching is shown in Figure 4.21.

The role of holding time in DTM was to ensure an adequate temperature was achieved before quenching. The quenching temperature for each mould was estimated accordingly as the temperature in the copper mould was dropped at 0.7 °C/s, which was obtained from a separate experimental work. The first calculated quenching temperatures for pouring temperature of 685 °C by the holding time of 60 s, 40 s and 20 s were at 643 °C, 657 °C and 671 °C respectively. The second calculated quenching temperatures for pouring temperatures for pouring temperature of 665 °C by the holding time of 665 °C by the holding time of 60 s, 40 s and 20 s were at 623 °C, 637 °C and 651 °C. The last calculated quenching temperatures for pouring temperature of 645 °C by the holding time of 60 s, 40 s and 20 s were at 603 °C, 617 °C and 631 °C respectively. These quenching temperatures later were used to determine $T_a - f_s$ relations within copper mould which were based on the results obtained from a previous experiment (see section 4.2.4).

There was apparent indication from these figures that there was a significant microstructure difference between samples that quenched at different time period. Another obvious finding with these results was the sample which allowed to solidify without quenching produced a bigger size structure than other samples. The microstructure between the sample with pouring temperature of 685 °C, holding time of 60 s and sample without quenching show this evidence as presented in Figure 4.18 (a) and Figure 4.21.

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Figure 4.18: Microstructure for sample with pouring temperature of 685 °C and at different holding time with (a) 60 s (sample 1), (b) 40 s (sample 2) and (c) 20 s (sample 3) before quenching.


Figure 4.19: Microstructure for sample with pouring temperature of 665 °C and at different holding time with (a) 60 s (sample 4), (b) 40 s (sample 5) and (c) 20 s (sample 6) before quenching.



Figure 4.20: Microstructure for sample with pouring temperature of 645 °C and at different holding time with (a) 60 s (sample 7), (b) 40 s (sample 8) and (c) 20 s (sample 9) before quenching.



Figure 4.21: Microstructure for sample with pouring temperature of 685 °C (sample 10) and allowed to solidify without quenching.

4.3.2 Grain Size Measurement

Comparisons between microstructure of the samples were made by using a grain size measurement method. The grain size measurements consist with the average values for primary grain diameter, circularity, aspect ratio and secondary phase area. The results obtained from the grain size measurement are presented in Figure 4.22, Figure 4.24, Figure 4.26 and Figure 4.28.

There was a significant difference between average microstructure primary grain diameters for sample 1, 2 and 3 which are presented in Figure 4.22. Moreover, the significant difference was also found with sample 10 that allowed to solidify without quenching when comparison was made with other samples. Sample 10 which processed with pouring temperature of 685 °C obtained the highest average diameter value. This gives the indication that sample 10 contained large microstructure features. There was insignificant difference for grain diameter with sample 3 to 9 which processed at different processing conditions. The results were found overlapped to each other. Together, these results provide an important indicator that the relationship between processing parameters and microstructure formation.



Figure 4.22: Grain size measurement for the average primary grain diameter of 10 samples (errors are 95% confidence intervals).

The analysis of variance (ANOVA) table as presented in Table 4.1 shows the significant (sig.) values for all processing parameters were less than 0.05. The low significant values indicated the significance of these factors and their interactions. The F values from the ANOVA table are used to determine factor ranks that influence factor/interaction on the average primary grain diameter. The F values in Table 4.1 show the pouring temperature was the highest at 96.085 and indicated pouring temperature to be the most influential factor in the change of average primary grain diameter values. The interaction between pouring temperature and holding time was the second rank with the F value of 23.322. The holding time had the least influence to average primary grain diameter value with 10.673 F value.

Table 4.1: Analysis of variance (ANOVA) table for processing parameters and

Source	Sum of Square	Mean Square	F	Sig.
Pouring Temperature	272.094	136.047	96.085	0.000416
Error (Pouring Temperature)	5.664	1.416		
Holding Time	45.466	22.733	10.673	0.025
Error (Holding Time)	8.520	2.130		
Pouring Temperature * Holding Time	524.088	131.022	23.322	0.000182
Error (Pouring Temperature*Holding Time)	44.944	5.618		

primary grain diameter.

Table 4.2: Estimated mean by levels of pouring temperature

Pouring Temperature	Mean (µm)	Std. Error	95% Confidence Interval	
(°C)			Lower Bound	Upper Bound
645	86.473	0.392	83.256	92.050
665	87.243	0.384	83.646	92.951
685	93.147	0.607	82.408	99.734

for primary grain diameter.

Table 4.3: Estimated mean by levels of holding time for primary grain diameter.

Holding Time	Mean (µm)	Std. Error	95% Confidence Interval	
(s)			Lower Bound	Upper Bound
20	86.487	0.793	84.117	92.953
40	89.373	0.380	83.252	99.735
60	91.174	0.234	83.646	97.311

Table 4.2 presents the model-estimated means and standard errors of average primary grain diameter at the factor levels of pouring temperature. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.2 was useful in order to explore the differences between levels of factor (pouring temperature). The average primary grain diameter was formed at 86.473, 87.243 and 93.147 μ m with pouring temperature of level 1 (645 °C), level 2 (665 °C) and level 3 (685 °C) respectively. The results in Table 4.2 also indicate there were changes in average primary grain diameter value when the comparison was made between levels. The mean value for the average primary grain diameter for level 3 (685 °C) was higher than other levels that indicated some changes happened parallel with the value changes.

Table 4.3 shows the model-estimated means and standard errors of average primary grain diameter at the factor levels of holding time. The results in Table 4.3 indicated the average primary grain diameter occurred at 86.487, 89.373 and 91.174 µm with the holding time of level 1 (20 s), level 2 (40 s) and level 3 (60 s) respectively. The change in average primary grain diameter value was not obvious when the comparison was made between levels. Even though the mean value of level 3 was the highest but the differences between levels were relatively small.

Table 4.4 provides the model-estimated mean and standard errors of average primary grain diameter at the combination factor levels of pouring temperature and holding time. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.4 was useful in order to explore the interaction effect between pouring temperature and holding time, which was found in the tests of model effects. The highest average primary grain diameter values were formed at 99.735 μ m by the combination parameters between pouring temperature at level 3 (685 °C) and holding time at level 2 (40 s). The lowest value for average primary grain diameter occurred at 82.401 μ m by the combination parameters between pouring temperature at level 1 (20 s). A group which obtained the highest changes was in the group with pouring temperature at level 3 (685 °C).

A graph for model-estimated mean relations between processing factors is presented in Figure 4.23. The results showed the average primary grain diameter value which processed by pouring temperature and holding time with respect to different levels. The combination between pouring temperature at level 3 (685 °C) and holding time at level 2 (40 s) produced the highest average primary grain diameter value. Meanwhile, the combination of pouring temperature at level 3 (685 °C) and holding time at level 1 (20 s) produced the smallest average primary grain diameter value.

Table 4.4: Estimated mean by levels of pouring temperature and holding time for primary grain diameter.

Pouring Temperature	Holding Time	Mean (µm)	Std. Error	95% Coi Inte	nfidence rval
(°C)	(s)			Lower Bound	Upper Bound
	20	84.114	1.376	82.152	88.736
645	40	83.255	0.673	82.580	83.782
	60	92.059	0.317	91.034	93.130
	20	92.951	0.553	91.034	92.956
665	40	85.142	0.539	84.661	86.992
	60	83.649	1.756	82.454	88.213
685	20	82.401	1.758	81.345	87.477
	40	99.735	0.308	99.024	100.676
	60	97.313	1.267	94.651	97.555



Figure 4.23: Estimated mean relations between processing parameters and primary grain diameter.

In order to assess the globular microstructure, the average circularity and aspect ratio measurement were used. The results of the particular analysis between microstructure are summarised in Figure 4.24 and Figure 4.26 which represents an average microstructure circularity and aspect ratio values respectively. The results, as shown in Figure 4.24, indicate that the sample with the highest circularity was in sample 4 that significantly difference compared to other samples. This gives an obvious indicator that the sample which was processed with pouring temperature of 665 °C and holding time of 60 s produced more globular microstructure compare with other samples. This evident was supported by the average aspect ratio result shown in Figure 4.26.



Figure 4.24: Grain size measurement for the average microstructure circularity of 10 samples (errors are 95% confidence intervals).

Table 4.5: Analysis of variance (ANOVA) table for processing parameters and

Source	Sum of Square	Mean Square	F	Sig.
Pouring Temperature	0.007	0.004	7.304	0.046
Error (Pouring Temperature)	0.002	0.001		
Holding Time	0.023	0.012	30.971	0.004
Error (Holding Time)	0.002	0.000		
Pouring Temperature * Holding Time	0.009	0.002	6.610	0.012
Error (Pouring Temperature*Holding Time)	0.003	0.000		

primary grain circularity.

Table 4.6: Estimated mean by levels of pouring temperature

Pouring Temperature	Mean	Std. Error	95% Confidence Interval	
(°C)			Lower Bound	Upper Bound
645	0.601	0.004	0.584	0.618
665	0.614	0.011	0.566	0.663
685	0.574	0.014	0.512	0.637

for primary grain circularity.

Table 4.7: Estimated means by levels of holding time

for primary grain circularity.

Holding Time	Mean	Std. Error	95% Confidence Interval	
(s)			Lower Bound	Upper Bound
20	0.557	0.013	0.502	0.611
40	0.607	0.010	0.565	0.648
60	0.627	0.008	0.591	0.663

The ANOVA table as shown in Table 4.5 presents the significant (sig.) values for all processing parameters were less than 0.05. The low significant values indicated the significance of these factors and their interactions. The F values from the ANOVA table are used to determine factor ranks that influence factor/interaction on the average primary grain circularity. The F values in Table 4.5 show the holding time was the highest at 30.971 and indicated holding time to be the most influential factor in the change of average primary grain circularity values. The pouring temperature was the second rank with the F value of 7.304. The interaction between pouring temperature and holding time had the least influence to average primary grain circularity value with 6.610 F value.

Table 4.6 presents the model-estimated mean and standard errors of average primary grain circularity at the factor levels of pouring temperature. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.6 was useful in order to explore the differences between levels of factor (pouring temperature). The average primary grain circularity was formed at 0.601, 0.614 and 0.574 with pouring temperature of level 1 (645 °C), level 2 (665 °C) and level 3 (685 °C) respectively. The results in Table 4.6 also indicate there were quite small changes in average primary grain circularity value when the comparison was made between levels. The average value for primary grain circularity for level 2 (665 °C) was higher than other levels. However, the changes were not significantly different from each other.

Table 4.7 shows the model-estimated means and standard errors of average primary grain circularity at the factor levels of holding time. The results in Table 4.7 indicated the average primary grain circularity occurred at 0.557, 0.607 and 0.627 with the holding time of level 1 (20 s), level 2 (40 s) and level 3 (60 s) respectively. The results also indicate there were changes in average primary grain circularity value when the comparison was made between levels. The mean value for average primary grain circularity for level 3 (60 s) was higher than other levels that indicated some changes happened parallel with the value changes.

Table 4.8 provides the model-estimated mean and standard errors of average primary grain circularity at the combination factor levels of pouring temperature and holding time. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.8 was useful in order to explore the interaction effect between pouring temperature and holding time, which was found in the tests of model effects.

The highest average primary grain circularity values were formed at 0.670 by the combination parameters between pouring temperature at level 2 (665 °C) and holding time at level 3 (60 s). The lowest value for average primary grain circularity occurred at 0.550 by the combination parameters between pouring temperature at level 2 (665 °C) and holding time at level 1 (20 s). A group which obtained the highest changes was in the group with pouring temperature at level 2 (665 °C).

A graph for model-estimated mean relations between processing factors is presented in Figure 4.25. The results showed the average primary grain circularity value which processed with pouring temperature and holding time at different levels. The combination of pouring temperature at level 2 (665 °C) and holding time at level 3 (60 s) produced the highest average primary grain circularity value. Meanwhile, the combination between pouring temperature at level 2 (665 °C) and holding time at level 2 (665 °C) and holding time at level 1 (20 s) produced the smallest average primary grain circularity value.

Pouring Temperature	Holding Time	Mean	Std. Error	95% Confidence Interval	
(°C)	(s)			Lower Bound	Upper Bound
	20	0.563	0.019	0.483	0.643
645	40	0.630	0.010	0.587	0.673
	60	0.610	0.006	0.585	0.635
	20	0.550	0.020	0.464	0.636
665	40	0.623	0.007	0.595	0.652
	60	0.670	0.010	0.627	0.713
	20	0.557	0.018	0.481	0.633
685	40	0.567	0.017	0.495	0.638
	60	0.600	0.012	0.550	0.650

Table 4.8: Estimated mean by levels of pouring temperature and holding timefor primary grain circularity.



Figure 4.25: Estimated mean relations between processing parameters and primary grain circularity.

Figure 4.26 presents grain size measurement results for the average microstructure aspect ratio of 10 samples. The results indicated that the sample with the highest average aspect ratio value was in sample 2. Meanwhile, the smallest average aspect ratio value was in sample 4. The higher average aspect ratio value indicates sample with elongated and smaller average aspect ratio value indicates circular morphology. However, these results show that the results were overlapped to each other.



Figure 4.26: Grain size measurement for the average microstructure aspect ratio of 10 samples (errors are 95% confidence intervals).

Table 4.9: Analysis of variance (ANOVA) table for processing parameters andprimary grain aspect ratio.

Source	Sum of Square	Mean Square	F	Sig.
Pouring Temperature	0.010	0.005	0.612	0.586
Error (Pouring Temperature)	0.032	0.008		
Holding Time	0.022	0.011	1.481	0.330
Error (Holding Time)	0.030	0.008		
Pouring Temperature * Holding Time	0.009	0.002	0.429	0.784
Error (Pouring Temperature*Holding Time)	0.041	0.005		

Pouring Temperature	Mean	Std. Error	95% Confidence Interval	
(°C)			Lower Bound	Upper Bound
645	1.510	0.017	1.436	1.584
665	1.507	0.030	1.377	1.637
685	1.549	0.025	1.441	1.657

Table 4.10: Estimated mean by levels of pouring temperaturefor primary grain aspect ratio.

Table 4.11: Estimated mean by levels of holding time

Holding Time	Mean	Std. Error	95% Confidence Interval	
(s)			Lower Bound	Upper Bound
20	1.544	0.029	1.419	1.670
40	1.540	0.014	1.480	1.600
60	1.481	0.026	1.369 1.594	

for primary grain aspect ratio.

The ANOVA table as shown in Table 4.9 presents the significant (sig.) values for all processing parameters were more than 0.05. The high significant values indicated the insignificance of these factors and their interactions. These can be observed from the values between results in Figure 4.26 that overlapped with each other. The F values from the ANOVA table are used to determine factor ranks that influence factor/interaction on the average primary grain aspect ratio. The F values in Table 4.9 show the holding time was the highest at 1.481 and indicated holding time to be the most influential factor in the change of average primary grain aspect ratio values. The pouring temperature was the second rank with the F value of 0.612. The interaction between pouring temperature and holding time had the least influence to average primary grain aspect ratio value.

Table 4.10 presents the model-estimated mean and standard errors of average primary grain aspect ratio at the factor levels of pouring temperature.

The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.10 was useful in order to explore the differences between levels of factor (pouring temperature). The average primary grain aspect ratio was formed at 1.510, 1.507 and 1.549 with pouring temperature of level 1 (645 °C), level 2 (665 °C) and level 3 (685 °C) respectively. The results in Table 4.10 also indicate there were quite small changes in average primary grain aspect ratio value when the comparison was made between levels. The mean value for average primary grain aspect ratio for level 3 (685 °C) was higher than other levels. However, the changes were not significantly different from each other.

Table 4.11 shows the model-estimated means and standard errors of average primary grain aspect ratio at the factor levels of holding time. The results in Table 4.11 indicated the average primary grain aspect ratio occurred 1.544, 1.540 and 1.481 with the holding time of level 1 (20 s), level 2 (40 s) and level 3 (60 s) respectively. The results also indicate there were changes in average primary grain aspect ratio value when the comparison was made between levels. The mean value for average primary grain aspect ratio for level 1 (20 s) was the highest and level 3 (60) was the smallest. The obvious differences between these two levels indicated some changes happened parallel with the value changes.

Table 4.12 provides the model-estimated mean and standard errors of average primary grain aspect ratio at the combination factor levels of pouring temperature and holding time. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.12 was useful in order to explore the interaction effect between pouring temperature and holding time, which was found in the tests of model effects. The highest average primary grain aspect ratio values were formed at 1.570 by the combination parameters between pouring temperature at level 3 (685 °C) and holding time at level 2 (40 s). The lowest value for average primary grain aspect ratio was at 1.433 by the combination parameters between pouring temperature at level 3 (60 s). A group which obtained the highest changes was in the group with pouring temperature at level 2 (665 °C).

A graph for model-estimated mean relations between processing factors is presented in Figure 4.27. The results showed the average primary grain aspect ratio value which processed with pouring temperature and holding time at different levels. The combination of pouring temperature at level 3 (685 °C) and holding time at level 2 (40 s) produced the highest average primary grain aspect ratio value. Meanwhile, the combination between pouring temperature at level 2 (665 °C) and holding time at level 3 (60 s) produced the smallest average primary grain aspect ratio value.

Pouring Temperature	Holding Time	Mean	Std. Error	95% Confidence Interval	
(°C)	(s)			Lower Bound	Upper Bound
	20	1.517	0.078	1.181	1.852
645	40	1.517	0.024	1.413	1.620
	60	1.497	0.035	1.345	1.648
	20	1.553	0.058	1.305	1.802
665	40	1.533	0.009	1.495	1.571
	60	1.433	0.042	1.254	1.613
685	20	1.563	0.023	1.463	1.664
	40	1.570	0.049	1.358	1.782
	60	1.513	0.038	1.352	1.675

Table 4.12: Estimated mean by levels of pouring temperature and holding time for primary grain aspect ratio.



Figure 4.27: Estimated mean relations between processing parameters and primary grain aspect ratio.

Figure 4.26 presents grain size measurement results for the average secondary phase area of 10 samples. The results indicated that the sample with the highest secondary phase area value was in sample 3. Meanwhile, the smallest secondary phase area value was in sample 10. There was a significant difference in sample 3 compared with sample 4 to 10 but insignificant difference was found between sample 1 to 3. Sample with higher average secondary phase area value indicates that the sample has a higher liquid content volume surrounded the primary phase structure.



Figure 4.28: Grain size measurement for the secondary phase area of 10 samples (errors are 95% confidence intervals).

Source	Sum of Square	Mean Square	F	Sig.
Pouring Temperature	1177950667.873	588975333.937	116.067	0.0003
Error (Pouring Temperature)	20297789.005	5074447.251		
Holding Time	786231865.750	393115932.875	336.192	0.00004
Error (Holding Time)	4677274.066	1169318.516		
Pouring Temperature * Holding Time	103410186.338	25852546.584	14.730	0.001
Error (Pouring Temperature*Holding Time)	14041055.761	1755131.970		

Table 4.13: Analysis of variance (ANOVA) table for processing parameters and secondary phase area.

Pouring Temperature	Mean (µm²)	Std. Error	95% Confidence Interval	
(°C)			Lower Bound	Upper Bound
645	22787.969	777.245	19443.755	26132.183
665	29091.523	628.594	26386.899	31796.146
685	38844.164	518.371	36613.793	41074.536

Table 4.14: Estimated mean by levels of pouring temperature for the secondary phase area.

Table 4.15: Estimated mean by levels of holding time

Holding Time	Mean (µm²)	Std. Error	95% Confidence Interval	
(s)			Lower Bound	Upper Bound
20	36657.293	265.569	35514.642	37799.944
40	30611.595	574.054	28141.638	33081.551
60	23454.768	840.795	23416.927	23492.610

for the secondary phase area.

The ANOVA table as shown in Table 4.13 presents the significant (sig.) values for all processing parameters were less than 0.05. The low significant values indicated the significance of these factors and their interactions. The F values from the ANOVA table are used to determine factor ranks that influence factor/interaction on the average secondary phase area. The F values in Table 4.13 show the highest holding time was at 336.192 and indicated holding time to be the most influential factor in the change of average secondary phase area values. The pouring temperature was the second rank with the F value of 116.067. The interaction between pouring temperature and holding time had the least influence to average secondary phase area value with 14.730 F value.

Table 4.14 was useful in order to explore the differences between levels of factor (pouring temperature). The average secondary phase area was formed at 22787.969, 29091.523 and 38844.164 μ m² with pouring temperature of level 1 (645 °C), level 2 (665 °C) and level 3 (685 °C) respectively. The results in Table 4.14 also indicate there were changes in average secondary phase area value when the comparison was made between levels. The mean value for the average secondary phase area for level 3 (685 °C) was higher than other levels.

Table 4.15 shows the model-estimated means and standard errors of the average secondary phase area at the factor levels of holding time. The results in Table 4.15 indicated the average secondary phase area occurred at 36657.293, 30611.595 and 23454.768 μ m² with the holding time of level 1 (20 s), level 2 (40 s) and level 3 (60 s) respectively. The results also indicate there were changes in average secondary phase area value when the comparison was made between levels. The mean value for the average secondary phase area for level 1 (20 s) was the highest and level 3 (60) was the smallest. The obvious differences between these two levels indicated some changes happened parallel with the value changes.

Table 4.16 provides the model-estimated mean and standard errors of the average secondary phase area at the combination factor levels of pouring temperature and holding time. The estimated mean show the mean response for each factor, adjusted for any other variables in the model. The information in Table 4.16 was useful in order to explore the interaction effect between pouring temperature and holding time, which was found in the tests of model effects. The highest average secondary phase area values were formed at 45235.370 μ m² by the combination parameters between pouring temperature at level 3 (685 °C) and holding time at level 1 (20 s). The lowest value for the average secondary phase area was at 15478.195 μ m² by the parameters combination between pouring temperature at level 1 (645 °C) and holding time at level 1 (645 °C) and holding time at level 1 (645 °C).

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A graph for model-estimated mean relations between processing factors is presented in Figure 4.29. The results showed the average secondary phase area value which processed with pouring temperature and holding time at different levels. The combination of pouring temperature at level 3 (685 °C) and holding time at level 1 (20 s) produced the highest secondary phase area value. Meanwhile, the combination between pouring temperature at level 1 (645 °C) and holding time at level 3 (60 s) produced the smallest average secondary phase area value.

Pouring Temperature	Holding Time	Mean (µm²)	Std. Error	95% Confidence Interval	
(°C)	(s)			Lower Bound	Upper Bound
	20	32411.842	499.582	30262.315	34561.369
645	40	20473.870	1327.445	14762.336	26185.405
	60	15478.195	586.949	12952.759	18003.631
	20	32324.668	702.651	29301.404	35347.931
665	40	31882.913	1080.606	27233.442	36532.384
	60	23066.987	1004.220	18746.177	27387.797
	20	45235.370	979.876	41019.305	49451.435
685	40	39478.000	480.825	37409.177	41546.823
	60	31819.123	840.317	28203.529	35434.718

Table 4.16: Estimated mean by levels of pouring temperature and holding timefor the secondary phase area.



Figure 4.29: Estimated means relations between processing parameters and secondary phase area.

4.3.3 Energy Dispersive X-ray Spectroscopy Analysis

In order to assess the chemical compositions of the sample, an Energydispersive X-ray Spectroscopy (EDXS) analysis was used. The test was performed on two different areas consist of primary and secondary phases. The primary phase was represented by a solid grain structure. Meanwhile, the secondary phase was represented by liquid structure that occurred at the grain boundary of the solid grain structure. The chemical compositions of one of DTM samples at primary phase, which analysed by using EDXS are shown in Table 4.17. Three major elements were detected consist of aluminium (AI), Zinc (Zn) and Magnesium (Mg). The selected area of the primary phase, which was used in EDXS analysis are presented by micrographs in Figure 4.30 and Figure 4.31. The EDXS quantitative analysis spectrum of phases detected in the selected area is shown in Figure 4.32.

Table 4.17: Chemical composition of DTM specimen analysed from the selectedprimary phase area.

Elements (wt%)	AI	Zn	Mg
Spectrum	92.71 - 100	3.40 - 5.95	1.59 – 1.89



Figure 4.30: Selected primary phase area in one of the DTM samples with line spectrum measurements.



Figure 4.31: Selected primary phase area in one of the DTM samples with line spectrum measurements.



Figure 4.32: EDXS qualitative analysis spectrum of phases for DTM sample with their elements.

Chemical compositions for one of the DTM samples which analysed by EDXS for a secondary phase is shown in Table 4.18. Two major elements were detected from DTM specimen consist of aluminium (AI) and copper (Cu). The selected secondary area which was used in EDXS analysis for DTM specimen is presented by micrograph in Figure 4.33 to Figure 4.35. The EDXS quantitative analysis spectrum of phases detected in the selected area is shown in Figure 4.36. There was a high copper element volume detected in secondary phase within samples which this element initially was not detected in the primary phase.

Table 4.18: Chemical composition of DTM specimen analysed from the selectedsecondary phase area.

Elements (wt%)	AI	Cu
Spectrum	45.75 – 63.18	36.82 - 54.25



Figure 4.33: Selected secondary phase area in one of the DTM samples with (a) normal and (b) higher magnifications.



Figure 4.34: Selected secondary phase area in one of the DTM samples with line spectrum measurements.



Figure 4.35: Selected secondary phase area in one of the DTM samples with line spectrum measurements.



Figure 4.36: EDXS qualitative analysis spectrum of phases for DTM sample with their major elements.

4.4 Compression Test

4.4.1 Filling Distance

Figure 4.37 illustrates several examples of the feedstock billets which were formed after the compression test. This figure gives a better understanding about the measurement of overall length A and B. There is a significant difference between the formed feedstock billets at conical section as shown in Figure 4.37, that indicate the success of compression test.

The relationship between microstructure and formability for each sample were evaluated by using a compression test unit. In this compression test, feedstock billets which initially reheated injected into the forming die. Figure 4.38 shows several examples of feedstock billets after the compression test. There was strong evidence that formability strongly depended on microstructure.



Figure 4.37: The overall length of the feedstock billets after the compression test.



Figure 4.38: The feedstock billets condition inside the forming die slightly after cooled to room temperature with (a) billet was filled 30 % (b) billet was filled 50 % and (c) billet was filled 100 % of filling area.

	Feedstock Process Conditio	Billets ing ons	Average Overall	Average Overall	Average Transfer
Sample No.	Pouring Temperature (°C) (s)		Length A (mm)	Length B (mm)	Time (s)
1		60	53.12	47.04	10.52
2	685	40	51.71	46.73	11.87
3		20	54.77	44.19	12.05
4		60	51.27	43.86	11.03
5	665	40	50.77	45.55	12.76
6		20	51.96	47.07	11.38
7		60	51.87	46.41	10.71
8	645	40	51.75	46.43	10.30
9		20	52.69	47.45	11.96
10	685	-	53.18	47.45	10.63

Table 4.19: The average overall length A, overall length B and transfer timeresults for compression test of 10 samples.

The average compression test results for 10 feedstock billets which were formed after compression test are shown in Table 4.19. The results consist of overall length A and B after and transfer time during the compression test. The feedstock billets initially were processed with 10 different processing parameters which repeated three times in order to gain repeatability of the results. It is apparent from Table 4.19 that the average overall lengths A of the feedstock billets were changed after the compression test. The feedstock billets were changed after the compression test. The feedstock billets decreased approximately 25 % in overall length compared with the original length. Meanwhile, average diameter for the feedstock billets were increased by 18 % compared with the original billet diameter.

In order to get clear information about the compression test, the results were summarised and presented in Figure 4.39. The results show that the billets which were grouped in sample 3 represent the highest value of overall length A-B. This shows that billets in sample 3 were effectively filled into the conical area of the forming die which indicates the ability of the feedstock billets to form. The feedstock billets in sample 3 were the highest in formability compared with other feedstock billets. The significance difference was found when the comparison was made between sample 3 and other samples. Meanwhile, the results show that insignificance was found when the comparison was made between sample 1, 2 and 4 to 10.



Figure 4.39: Overall length results for 10 group samples which measured after compression test (errors are 95% confidence intervals).

Transfer time represents the duration of feedstock billets after transported out from furnace until the start button on the machine was triggered. This transfer time was used to determine the actual temperature of feedstock billets at the initial stage of compression test. Base on separate experimental work, it shows that cooling rate during the time of feedstock billets transferred out from the furnace was at 0.88 °C/s. The temperature at the respective time (base on transfer time) was calculated by using this cooling rate. The feedstock billet temperature after transported out from the furnace was at 620 °C. The temperature for sample 3 during the start button was triggered which calculated based on 0.88 °C/s cooling rate was approximately at 609 °C. The temperature then was used to estimate the fraction solid within feedstock billets.

Table 4.20: One way analysis of variance (ANOVA) table for average overall length A-B (mm).

Source	Sum of Square	Mean Square	F	Sig.
Between Group	81.699	9.078	7.346	0.000109
Within Group	24.714	1.236		
Total	106.413			

Table 4.21: Pearson correlation coefficient analysis for overall length A-B (mm) and transfer time (s) results.

		Overall Length A-B (mm)	Transfe r Time (s)
Overall Length A-B	Pearson Correlation	1	0.147
(mm)	Sig. (2-tailed)		0.686
Transfer Time (s)	Pearson Correlation	0.147	1
	Sig. (2-tailed)	0.686	

The One way ANOVA table as shown in Table 4.20 presents the significant (sig.) value of F test was less than 0.05. The low significant value indicated the overall length A-B results between sample groups were statistically significant to each other. Meanwhile, Table 4.21 presents the Pearson correlation coefficient analysis results between overall length A-B and transfer time. The Pearson correlation coefficient measures the linear association between two scale variables. The correlation between overall length A-B and transfer time was positive with 0.147. The correlation was not statistically different from 0 due to the significant value of 0.686 was greater 0.05. This result suggests the transfer time was not given an appreciable effect on overall length A-B. Furthermore, Figure 4.40 shows a correlation coefficient indicated that with the increment of transfer time, the overall length A-B was also increased.



Figure 4.40: A graph for correlation between overall length A-B and transfer time (s).

4.4.2 Microstructure After Compression Test

Changes in microstructure after compression test were compared by using qualitative analysis method. The microstructure after compression test for sample with pouring temperature of 685 °C, 665 °C, 645 °C and holding time 60 s, 40 s, 20 s are presented in Figure 4.41 to Figure 4.43 respectively. On the other hand, the microstructure for pouring temperature of 685 °C which allowed to solidify without quenching is presented in Figure 4.44.

The microstructure difference between samples was evident. Three apparent differences were detected among samples consist of the amount of secondary around primary phase, microstructure morphology and size. The microstructure for variance pouring temperatures either at 685 °C, 665 °C or 645 °C with a holding time of 20 s produce higher secondary phase amount. The microstructures with higher secondary phase are shown in Figure 4.41 (c), Figure 4.42 (c) and Figure 4.43 (c). Furthermore, sample with a holding time 40 s with various pouring temperatures produced more spheroidal microstructure which presented in Figure 4.41 (b), Figure 4.42 (b) and Figure 4.43 (b) respectively. The primary phase size for each sample seems was evolved from its original size. The microstructure for sample which allowed to solidify without quenching produces the highest structure size compared to other samples as shown in Figure 4.44. The feedstock billets microstructure size was increased significantly after the compression test and these results suggested that feedstock billets microstructure was significantly changed after heating and forming operations.



Figure 4.41: Microstructure of sample with pouring temperature of 685 °C and at different holding time with (a) 60 s (sample 1), (b) 40 s (sample 2) and (c) 20 s (sample 3) after the compression test.



Figure 4.42: Microstructure of sample with pouring temperature of 665 °C and at different holding time with (a) 60 s (sample 4), (b) 40 s (sample 5) and (c) 20 s (sample 6) after the compression test.



Figure 4.43: Microstructure of sample with pouring temperature of 645 °C and at different holding time with (a) 60 s (sample 7), (b) 40 s (sample 8) and (c) 20 s (sample 9) after the compression test.


Figure 4.44: Microstructure of sample with pouring temperature of 685 °C (sample 10) and allowed to cold without quenching after the compression test.

4.4.3 Grain Size Measurement

In order to assess the differences between microstructure of the samples after the compression test, a grain size measurement method was used. The grain size measurement consists of the average primary phase structure diameter, circularity and secondary phase area. Figure 4.45, Figure 4.47 and Figure 4.49 presents the summary of grain size measurement results.

A clear evidence of structural transformation was found in average primary grain diameter for all samples which is shown in Figure 4.45. The average primary grain diameter in the sample 10 which processed with pouring temperature of 685 °C and solidifies without quenching was the highest. This shows that large microstructure occurred within this sample. Significance difference was found when the comparison was made between sample 1, 2, 3, 5 and 10. Meanwhile, sample 4, 6 to 9 was found insignificance by the overlapped results. Overall, these results indicate that there are a significant relation between processing parameters and microstructure formation.



Figure 4.45: Grain size measurement for the average microstructure diameter of 10 samples (errors are 95% confidence intervals).

Table 4.22: One way analysis of variance (ANOVA) table for average primary grain diameter (µm) after the compression test.

Source	Sum of Square	Mean Square	F	Sig.
Between Group	1804.54	200.50	18.47	0.001
Within Group	217.08	10.85		
Total	2021.62			

The One way ANOVA table as shown in Figure 4.22 presents the significant (sig.) value of F test was less than 0.05. The low significant value indicated the average primary grain diameter results between sample groups were statistically significant to each other. Meanwhile, Table 4.23 presents the Pearson correlation coefficient analysis results between average grain diameter and overall length A-B. The correlation between average grain diameter and overall length A-B was negative with -0.249. The correlation was not statistically different from 0 due to the significant value of 0.488 was greater 0.05. This result suggests the primary grain diameter was not given an appreciable effect on overall length A-B.

		Grain diamete r (µm)	Overall length A-B (mm)
Grain diameter (µm)	Pearson Correlation	1	-0.249
	Sig. (2-tailed)		0.488
Overall length A-B	Pearson Correlation	1	-0.249
(mm)	Sig. (2-tailed)	0.488	

Table 4.23: Pearson correlation coefficient analysis for grain diameter (µm) and overall length A-B (mm) results.



Figure 4.46: : A graph for correlation between grain diameter (μ m) and overall length A-B (mm).

Figure 4.46 shows a correlation graph between average primary grain diameter and overall length A-B. The negative correlation coefficient indicated that with the increment of average primary grain diameter, the overall length A-B was decreased. The globular microstructure of the sample was assessed by measuring average microstructure circularity. The average microstructure circularity for all samples is presented in Figure 4.47. The results obtained from grain size measurement analysis as shown in Figure 4.47 demonstrate sample 2 circularity was the highest. This sample initially was processed with pouring temperature of 685 °C and holding time of 40 s. Taken together, these results suggest that there was a significant change in microstructure formation after the compression test that altered the initial microstructure which less spheroidal to more spheroidal.



Figure 4.47: Grain size measurement for the average microstructure circularity of 10 samples (errors are 95% confidence intervals).

Table 4.24: One way analysis of variance (ANOVA) table foraverage primary grain circularity.

Source	Sum of Square	Mean Square	F	Sig.
Between Group	0.036	0.004	2.665	0.033
Within Group	0.030	0.001		
Total	0.066			

		Circ.	Overall Length A-B (mm)
Circularity	Pearson Correlation	1	-0.809
	Sig. (2-tailed)		0.005
Overall length A-B	Pearson Correlation	-0.809	1
(m m)	Sig. (2-tailed)	0.005	

Table 4.25: Pearson correlation coefficient analysis for grain circularity and overall length A-B (mm) results.

The One way ANOVA table as shown in Table 4.24 presents the significant (sig.) value of F test was less than 0.05. The low significant value indicated the average primary grain circularity results between sample groups were statistically significant to each other. Meanwhile, Table 4.25 presents the Pearson correlation coefficient analysis results between average primary grain circularity and overall length A-B. The correlation between primary grain circularity and overall length A-B was negative with -0.809. The correlation was statistically different from 0 due to the significant value of 0.005 was less than 0.05. This result suggests the primary grain circularity was given an appreciable effect on overall length A-B.

Figure 4.48 shows a correlation graph between average primary grain circularity and overall length A-B. The negative correlation coefficient indicated that with the increment of average primary grain circularity, the overall length A-B was decreased.



Figure 4.48: A graph for correlation between circularity and overall length A-B (mm).

The secondary phase was assessed by measuring the average secondary phase area. The average microstructure area for all samples is presented in Figure 4.49. The results obtained from grain size measurement analysis as shown in Figure 4.49 demonstrate sample 3 secondary phase was the highest. This sample initially was processed with pouring temperature of 685 °C and holding time of 20 s. Taken together, these results suggest that there was a significant change in microstructure formation after the compression test that altered the initial secondary phase structure.





Table 4.26 : One way analysis of variance (ANOVA) table for average area of secondary phase (μm^2).

Source	Sum of Square	Mean Square	F	Sig.
Between Group	719099055.975	79899895.108	2.737	0.029
Within Group	583815139.663	29190756.983		
Total	1302914195.638			

Table 4.27: Pearson correlation coefficient analysis for average area of secondary phase (μm^2) and overall length A-B (mm) results.

		Secondary Phase (µm²)	Overall Length A- B (mm)
Circularity	Pearson Correlation	1	0.711
	Sig. (2-tailed)		0.021
Overall length A-B	Pearson Correlation	0.711	1
(11111)	Sig. (2-tailed)	0.021	

The One way ANOVA table as shown in Table 4.26 presents the significant (sig.) value of F test was less than 0.05. The low significant value indicated the average secondary phase area results between sample groups were statistically significant to each other. Meanwhile, Table 4.27 presents the Pearson correlation coefficient analysis results between average secondary phase area and overall length A-B. The correlation between secondary phase area and overall length A-B was positive with 0.711. The correlation was statistically different from 0 due to the significant value of 0.021 was less than 0.05. This result suggests the secondary phase area was given an appreciable effect on overall length A-B.

Figure 4.50 shows a correlation graph between average secondary phase area and overall length A-B. The positive correlation coefficient indicated that with the increment of the average secondary phase area, the overall length A-B was increased.



Figure 4.50: A graph for correlation between average of secondary phase area (μ m²) and overall length A-B (mm).

4.4.4 EDXS Analysis

The EDXS analysis was performed on two different areas consist of primary and secondary phases of the sample which were processed after the compression test. The chemical compositions are shown in Table 4.28. Three major elements were detected from the specimen consist of aluminium (Al), Zinc (Zn), and Magnesium (Mg). The selected area which was used in EDXS analysis of the specimen is presented by micrographs in Figure 4.51 and Figure 4.52. The EDXS quantitative analysis spectrum of phases detected in the selected area is shown in Figure 4.53.

Table 4.28: Chemical composition of DTM specimen analysed from the selectedprimary phase area.

Elements (wt%)	AI	Zn	Mg
Spectrum	90.12 - 100	3.76 – 6.68	2.17 – 3.22



Figure 4.51: Selected primary phase area in one of the samples with line spectrum measurements.



Figure 4.52: Selected primary phase area in one of the samples with line spectrum measurements.



Figure 4.53: EDXS qualitative analysis spectrum of phases for sample with their major elements.

The chemical compositions of secondary phase one of the samples which analysed by using EDXS are shown in Table 4.29. Four major elements were detected from specimen after the compression test consist of aluminium (AI), Copper (Cu), Zinc (Zn), and Magnesium (Mg). The selected area which was used in EDXS analysis is presented by micrograph in Figure 4.54 to Figure 4.57. The EDXS quantitative analysis spectrum of phases detected in the selected area is shown in Figure 4.58.

Table 4.29: Chemical composition of DTM specimen analysed from the selectedsecondary phase area.

Elements (wt%)	AI	Cu	Zn	Mg
Spectrum	29.83-68.5	23.51-67.72	10.51-27.70	1.21-6.32



Figure 4.54: Selected secondary phase area in one of the samples with (a) normal and (b) higher magnifications.



Figure 4.55: Selected secondary phase area in one of the samples with line spectrum measurements.



Figure 4.56: Selected secondary phase area in one of the samples with (a) normal and (b) higher magnifications.



Figure 4.57: Selected secondary phase area in one of the samples with line spectrum measurements.



Figure 4.58: EDXS qualitative analysis spectrum of phases for sample with their major elements.

4.5 Mechanical Properties

4.5.1 Tensile and Hardness

The results of average mechanical properties for 10 feedstock billets after compression test are presented in Table 4.30. The results consist of Ultimate Tensile Strength (σ_{TS}), Yield Strength (σ_y), Elongation (ϵ_f), Modulus of Elasticity (E) and Hardness. Sample 1 was found to obtain the highest properties compared with other samples. In order to get vibrant information about the mechanical properties test, the results were summarised and presented in Figure 4.59 to Figure 4.62.

Sample	σ _{TS}	σy	٤ _f	Е	Hardness
NO.	(MPa)	(MPa)	(%)	(GPa)	(HV10)
1	213	206	1.5	71	115
2	170	148	2.7	71	108
3	83	72	2.4	71	85
4	127	94	2.7	75	95
5	133	124	2.5	66	97
6	93	67	2.3	69	94
7	99	64	1.7	70	98
8	121	111	1.8	71	106
9	142	128	1.5	72	105
10	106	94.2	0.7	72	96

Table 4.30: Average mechanical properties results for 10 samples after the compression test.



Figure 4.59: Average Ultimate Tensile Strength (UTS) results for 10 samples after the compression test.



Figure 4.60: Average Yield Strength (YS) results for 10 samples after the compression test.



Figure 4.62: Average Hardness results for 10 samples after the compression test.

4.5.2 Fractography

Images from the SEM for the tensile test specimen fracture surfaces for all samples are presented in Figure 4.63 to Figure 4.72. The images indicated that failure in all specimens occurred by ductile and intergranular fracture (the crack propagated along the grain boundaries). Porosity, primary phase grain size, grain morphology and secondary phase area which are among the important factor that influenced the mechanical properties. Porosity in the images was presented by the dimples and the sample with high number of dimples produced lower mechanical properties. The high porosity content was evident in sample 3 as shown in Figure 4.65 (a). Closer view of the Figure 4.65 (b), there was evident several numbers of porosity occurred and it significantly degraded the properties of the sample.



Figure 4.63: Tensile fracture surface images for one of the sample 1 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.64: Tensile fracture surface images for one of the sample 2 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.65: Tensile fracture surface images for one of the sample 3 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.66: Tensile fracture surface images for one of the sample 4 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.67: Tensile fracture surface images for one of the sample 5 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.68: Tensile fracture surface images for one of the sample 6 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.69: Tensile fracture surface images for one of the sample 7 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.70: Tensile fracture surface images for one of the sample 8 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.71: Tensile fracture surface images for one of the sample 9 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.



Figure 4.72: Tensile fracture surface images for one of the sample 10 produced by SEM with (a) a dimpled fracture surface, (b) a closer view of the dimple and (c) a close up view for the pores of the dimples.

4.5.3 Density

Average densities and porosity levels of 10 samples which were determined by Archimedes' principle are presented in Table 4.31. Some of the densities obtained from this experimental works were slightly higher than the reported density for aluminium 7075 in literature which typically at 2.81 g/cm³ [125]. The porosity level in the sample was determined by comparing the density obtained from the experimental works and the literature. The average density of the sample which obtained slightly higher than 2.81 g/cm³ indicated lower porosity and sample with marginally lower average density showed higher porosity content. The results in Table 4.31 show that the lowest porosity was found developed in sample 1 and the highest porosity content occurred in sample 3. The better mechanical properties were also observed with a low porosity content sample.

Sample No.	Average Density (g/cm ³)	Porosity
1	3.01 <u>+</u> 0.14	Lowest
2	2.81 <u>+</u> 0.21	Low
3	2.37 <u>+</u> 0.06	Highest
4	2.68 <u>+</u> 0.22	Moderate
5	2.68 <u>+</u> 0.28	Moderate
6	2.52 <u>+</u> 0.27	High
7	2.60 <u>+</u> 0.21	High
8	2.73 <u>+</u> 0.28	Moderate
9	2.87 <u>+</u> 0.21	Low
10	2.63 <u>+</u> 0.18	High

Table 4.31: Average density and porosity level of 10 samples determined byArchimedes' principle.

4.5.4 Statistical Analysis

A Pearson's correlation coefficient and a scatter diagram were used to determine the correlation between mechanical properties, primary phase grain diameter, primary phase circularity, secondary phase area, density and compression test results. The Pearson's correlation coefficient results are presented subsequently in Table 4.32.

The strongest correlation for all samples was observed between properties and density as in Table 4.32 with a stronger correlation coefficient is represented by high absolute correlation coefficients. The results show the important fact that mechanical properties of the sample were much influenced by the porosity content. This can be seen from Table 4.30 wherein sample 1 which obtained the highest average tensile property value has the lowest porosity content (see Table 4.31). Furthermore, sample 3 which degrade in tensile property has the highest porosity level. The circularity, secondary phase area and compression test values produced a moderate correlation with the UTS, YS and elongation but a higher correlation with the hardness values.

The plotted scatter diagram as presented in Appendix E allowed the factorial analysis between results. This is important to predict the overall results trend. Interestingly, the incremental of primary grain diameter and density (lower porosity level) in general increased the UTS, YS, and hardness values while elongation decreased. Moreover, the increment in primary circularity increased the UTS, YS, elongation and hardness respectively. Increment trends in the secondary phase area and compression test values decreased the UTS, YS, and hardness while elongation increased.

		UTS (MPa)	YS (MPa)	Elongation (%)	Hardness (HV)
Grain Size (µm)	Pearson Correlation	0.237	0.305	-0.571	0.088
	Sig. (2-tailed)	0.510	0.391	0.084	0.808
Circularity	Pearson Correlation	0.447	0.393	0.263	0.685
	Sig. (2-tailed)	0.194	0.260	0.462	0.029
Secondary Phase Area	Pearson Correlation	-0.426	-0.309	0.016	-0.527
(µm)	Sig. (2-tailed)	0.219	0.384	0.964	0.117
Density (g/cm ³)	Pearson Correlation	0.921	0.891	-0.246	0.842
	Sig. (2-tailed)	0.001	0.001	0.493	0.002
Overall Length A-B (mm)	Pearson Correlation	-0.315	-0.259	0.236	-0.694
_ 、 、	Sig. (2-tailed)	0.375	0.470	0.512	0.026

Table 4.32: Pearson correlation coefficient for grain size, circularity, secondary phase area, density and overall length A-B with results ofmechanical properties test.

4.6 Coefficient of Thermal Expansion

In order to obtain relationship between the coefficients of thermal expansion (CTE) and microstructure, several samples after the compression test were analysed by a thermal dilatometer. The CTE and thermal dimension expansion during heating for sample 1, 2, 3 and 10 are presented in Figure 4.73 and Figure 4.74 respectively. The CTE value of all samples was much influenced by microstructure morphology and size. The results, as in Figure 4.73 show the CTE value was higher for a sample with smaller microstructure size. Sample 2 has the highest CTE value in comparison with other samples and the lowest was in sample 10. The differences of CTE value were obvious when a comparison was made between sample 2 and 10 but the small gradient was found between CTE for sample 1, 2 and 3. This was due to the small microstructure variance between sample 1, 2 and 3. Similarly, the differences of thermal dimension expansion values between the small and the large microstructure size was evident, especially for sample 2 and 10 as shown in Figure 4.74. However, the difference between sample 1, 2 and 3 was not too obvious.



Figure 4.73: Coefficient of thermal expansion (CTE) curves for all samples.



Figure 4.74: Thermal dimension expansion curves during heating.

4.6.1 Statistical Analysis

A Pearson's correlation coefficient and a scatter diagram were used to determine the correlation between coefficient of thermal expansion (CTE) and thermal dimension expansion with primary grain diameter, primary grain circularity and secondary phase area results. The Pearson's correlation coefficient results are presented in Table 4.33.

The strongest correlation for all samples was observed between CTE and primary grain diameter as in Table 4.33. The results show the important fact that CTE of the sample was much influenced by grain size. This can be seen from Figure 4.73 where sample 1, 2 and 3 which obtained small average primary grain diameter has a higher CTE value. Additionally, sample 10 which obtained a low CTE value has a large primary grain diameter. A strong correlation was also found between thermal dimension expansion and primary grain circularity. However, the stronger correlation was also found between thermal dimension expansion and primary grain circularity. The morphology of sample primary grain, which has a more globular structure, increased the thermal dimension expansion. Another factor, secondary phases area were likely not strongly affected the CTE and thermal dimension expansion results.

The plotted scatter diagram as presented in Appendix F allowed the factorial analysis between results. This is important to predict the overall results trend. The results show that the incremental of primary grain grain diameter in general decreased the CTE and thermal dimension expansion. Moreover, the increment in primary grain circularity increased the CTE and thermal dimension expansion respectively. Increment trends in the secondary phase area were found to increase CTE but decreased the thermal dimension expansion value.

		CTE (x10 ⁻⁶ K ⁻¹)	Expansion (µm)
Grain Siza (um)	Pearson Correlation	-0.844	-0.646
Grain Size (µm)	Sig. (2-tailed)	0.156	0.354
Circularity	Pearson Correlation	0.400	0.700
Circularity	Sig. (2-tailed)	0.600	0.300
Secondary	Pearson Correlation	0.219	-0.201
Phase Area (µm²)	Sig. (2-tailed)	0.781	0.799

Table 4.33: Pearson correlation coefficient for grain size, circularity and secondary phase area with CTE and Thermal dimension expansion results.

CHAPTER 5

DISCUSSION

5.1 As Receive Sample

In reviewing the literature [182], initial as-received aluminium 7075 microstructure features were similar to the microstructure of the hot-worked metals as shown in Figure 5.1. The results show that the grains and intermetallic particle were elongated and aligned with the extrusion direction. This may be due to the extrusion process which was used to produce the raw material.



Figure 5.1: Microstructure for aluminium 7075 with (a) as-received sample and (b) elongated grains and aligned to extrusion direction [182].

5.2 Cooling Curve Analysis

5.2.1 Effect of Cooling Rate on Solidification and Fraction Solid

The cooling curve analysis was designed to determine the $T_a - f_s$ relations for aluminium 7075 in the thermal analysis experiment. Moreover, the cooling curve analysis was also performed to observe thermal profile changes with a variance of cooling rates used. These thermal profile data were subsequently applied to predict the precise control parameters during processing including heating, holding and quenching temperatures.

In this experimental work, the bulk of material was used to replicate the real industrial application. This enabled the precise thermal profile data captured due to the large amount of material used. Similar thermal analysis method for 7075 previously was performed, but with a different weight of alloy [44, 193]. There were also several publications in literature where similar methods to determine thermal profile were used, merely with different materials [200, 201]. The slow cooling rate was used to simulate a stabilised temperature condition to be used during forming. In the scope of SSM processing research, DSC at fast cooling rates was used to gain thermal profile data with 50 to 200 gram samples [190, 220].

The liquidus, eutectic, and solidus temperatures for various cooling rates of this work and other reports are shown in Table 5.1. The liquidus temperature was found slightly higher than other reported experimental results [44, 125]. This is commonly observed, though, due to variations in chemical composition of the alloy as received from different suppliers or at different times from the same supplier. The variation in alloying elements affects the overall solidification process and results in deviation in temperature at which phase changes occur [44].

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Source	C. Rate (°C/s)	Liq. Temp. (°C)	Eut. Temp. (°C)	Sol. Temp. (°C)
This work	0.13	638.5	474.3	472.6
This work	0.32	638.5	474.3	472.7
This work	0.36	640.1	476.6	472.8
This work	0.39	639.1	474.8	471.4
Bäckerud [44]	0.30	630.0	469.0	469.0
Bäckerud [44]	0.70	630.0	470.0	470.0
ASM [171]	-	635.0	-	477.0

Table 5.1: Liquidus, eutectic and solidus temperature at different cooling rates.

Several works regarding thermal analysis in the literature show different methods were used to determine the cooling rates [200, 221-223]. The different cooling rates resulted in marginal altered solidification characteristics for 7075. The liquidus, eutectic and solidus temperatures were noted to increase weakly with the increasing cooling rate. These differences can be seen clearly between the liquidus, eutectic and solidus temperatures for the cooling rates of 0.13 and 0.36 °C/s. However, the difference between 0.13 and 0.36 °C/s was not too significance to each other.

The fraction solid values were slightly changed by the use of different cooling rates. The fraction solid deformation at different cooling rates for aluminium 7075 is shown in Figure 5.2. The results show that the increment of cooling rate slightly has increased the fraction solid deformation temperatures. The difference between the fraction solid formation for a slow cooling (0.13 °C/s) and the highest cooling rate (0.39 °C/s) was apparent from 0.7 to 0.9 fraction solid (see Figure 5.2). However, fraction solid deformation temperature changes were not too substantial compared to each other. In comparing these results with other reported results, similar trends were found [44]. These results allowed a better experimental temperature control for reheating process.

The results presented in Figure 5.2 also revealed a narrow temperature window that affects the fraction solid for SSM processing. The fraction solid of SSM processing based on previous studies was found in the range of

0.5 to 0.7 [16, 23, 24]. The cooling curve, which was obtained in this present work, shows the liqudus temperature occurred at a temperature of 639 °C. In the meantime, the temperature for 0.5 to 0.7 fractions solid was in the range of 625 to 605 °C. This allowed only 14 to 34 °C temperature range drops from the liquidus before the SSM processing temperature. This narrow temperature window behaviour seems a disadvantage of aluminium 7075 compared to used extensively for SSM processing. aluminium A356 which was Comparatively, the liquidus temperature for A356 is at 613 °C and the temperature for 0.5 to 0.7 fractions solid was at 580 to 565 °C respectively [44]. This gives temperature range of 33 to 48 °C drops from the liqudus temperature, which gives A356 more processing flexibility and times for SSM processing. A possible explanation for this might be due to aluminium 7075 is not a cast alloy which explained the narrow temperature window for processing behaviour [16]. Thus, a better precaution is required during processing aluminium 7075 in semi-solid range temperature to make sure the success of SSM processing.



Figure 5.2: Fraction solid formation of different cooling rates.

5.2.2 Effect of Cooling Rate on Dendritic Coherency Points

The cooling rate shows a strong relation to the formation of DCP. Prior studies that have been noted the importance of DCP with several aluminium alloys were evaluated at different cooling rate conditions [180, 224-226]. The DCP temperature was used to determine the limitation temperature, which was later used for heating the feedstock billets in compression test. In addition, it was important to control the heating process in order to make sure the heating was not exceeded the DCP temperature, which consequently changes the primary grain of a structure.

The use of the slower cooling rate (in this work 0.13 °C/s) accelerates formation of DCP and allowed it to occur close to liquidus temperature. This finding supports previous research linked cooling rate and DCP [44]. Figure 5.3 shows the temperatures at which coherency occurred in the different cooling rates from the work of Bäckerud et al. [44] and this work.



Figure 5.3: Measured Dendritic Coherency Point (DCP) temperatures at different cooling rates and were compared with the literature.

The measured results show that the DCP temperatures were not significantly dissimilar to each other. This may be explained by the range of cooling rate used to be too small to each other. These findings were then compared with Bäckerud et al. [44] for the cooling rate in the range of 0.3 to 0.7, and the formation of DCP temperature difference was also not so significance (see Figure 5.3). Despite this, the results which obtained in this work were higher than Bäckerud et al. findings [180].

The calculated fraction solids at the coherency points occurred approximately at 0.20 for all cooling rate conditions. In SSM processing, knowledge of the coherency point is important. The shear strength of the semi-solid material increases greatly after this point. Knowledge on this point, therefore, allows a better understanding of material behaviour and determination of a suitable temperature for SSM processing. The formation of DCP was delayed with a higher cooling rate [227]. This was due to the formation of dendritic arm growth rate influenced by the cooling rate which also affected the DCP. The use of cooling rate in the range of 0.7 to 2.7 °C/s was found significantly increased the formation of DCP. Nevertheless, various studies were also indicated the disputed trend with a higher cooling rate increased the formation of DCP [44, 180]. These differences can be explained by the variation of method, material chemical compositions and weight that much influenced the overall findings. Based on the experimental results obtained from this experimental work, the DCP formation temperature was found not significantly different to each other which indicate that heating control may not be altered much within the cooling rate range investigated.

5.2.3 Effect of Cooling Rate on Microstructure

The formation of microstructure was much depended on the cooling rate. The image analysis and grain size measurement results show the microstructure formation which was processed with a higher cooling rate condition produced a smaller primary grain structure. Grain size for the microstructure of the highest cooling rate (0.39 °C/s) was smaller compared to the microstructure for the slow cooling rate (0.13 °C/s) (see Figure 4.11). The findings were also supported by

the grain size measurement results (see Figure 4.12 to Figure 4.17) with average primary grain diameter for the highest and slow cooling rate conditions were between 158 and 278 µm respectively.

The present findings seem to be consistent with other researchers which found changes in the microstructure were mainly caused by the different cooling rates [42, 228-230]. This may be explained by the fact that the higher cooling rate used and short solidification time resulted smaller microstructure features. The increase in cooling rate increased the nucleation temperature within the material which reflected the grain size formation [199, 223]. Furthermore, a higher cooling rate condition was also generally refined the microstructure and catalysed solid solubility of alloying elements within solid solution [231]. The incremental in cooling rate not just created a finer microstructure, but at the same time reduce the shape factor. This finding has important implications for developing the initial parameters for SSM processing.

5.3 Direct Thermal Method

5.3.1 Effect of Pouring Temperature

The variation of pouring temperature used in DTM experiment was intentionally established to determine the effect of processing parameters to microstructure formation. The results of this experimental work indicated that the grain sizes of the sample were strongly influenced by the processing parameters applied. Prior study has noted that the pouring temperature was among the important factors in DTM which determined the success of the process [232].

The pouring temperature is related to the cooling rate. Lower pouring temperature is the important parameter in producing a globular microstructure in the DTM. The lower pouring temperature for DTM retards the formation of microstructure [121, 232]. The lower pouring temperature leads to a higher cooling rate as less superheat has to be extracted from the mould and the melt. The low pouring temperature, which was used in this experimental work, developed small grain size diameter and more globular microstructure as the low pouring temperature helps to increase the cooling rate. The microstructure which was executed with the pouring temperature of 645 °C produced smaller diameter size and more spherical primary grain structure than the pouring temperature of 685 °C. This was evident with the result obtained which is presented in Figure 5.4. Two samples with the obvious deviation were selected to compare. The average primary grain diameter and circularity were used to determine the size and shape of the microstructure to support this finding. The average primary grain diameter of sample 2 was higher while circularity was lower than sample 8. Sample 8 produced by the lower pouring temperature, which was executed at pouring temperature of 645 °C. The higher pouring temperature leads to the slower cooling rate as more time needed by the systems to extract the heat from the above to below the liquidus temperature.



Figure 5.4: Comparison between microstructures with (a) average diameter for sample 2 (pouring temperature at 685 °C) and 8 (pouring temperature at 645°C), (b) microstructure and (c) average circularity for sample 2 and 8.

Lower pouring temperature produced smaller grain structure can be explained by the fact that the formation of microstructure merely depended on cooling rate which was used for material processing [233-235]. The cooling rate affects the formation of microstructure by the magnitude of undercooling temperature during solidification [236]. The undercooling is the difference between the equilibrium transformation temperature and temperature, which the material cools before the start of phase transformation. The undercooling is influenced by the solidification rates, which are influenced by the type of a mould material, mould thickness and similar. Therefore, the formation of a microstructure which evolves within material depends on the degree of undercooling. The undercooling becomes larger when the cooling rate is higher. The increment in undercooling increases the amount of nucleation, which is ultimately resulting in a smaller grain size. During the solidification process, as the undercooling temperature and time increase, the melt potential nucleation decreases resulting in a coarse-grained structure deformation. The higher cooling rate is therefore associated with a smaller grain size and a globular microstructure.

Cooling rate determines the success of DTM to produce smaller primary phase structure and spheroidal microstructure [199, 235, 237]. The principle of DTM which allows for a quick heat change retards the formation of a microstructure. The chilling effect which occurs within the copper mould produced smaller nuclei and affects the formation of a spheroidal microstructure. The results in DTM suggest that all the samples except sample 10 were suitable for thixoforming operation. The suitable thixoforming feedstock primary grain structure was found in the range of approximately 100 μ m [175].

5.3.2 Effect of Holding Time

The role of holding time in DTM was to ensure an adequate fraction solid before quenching. The formation of a smaller size and a globular microstructure by using DTM was influenced by fraction solid volume in molten metal before quenching [22, 238, 239]. This fraction solid was estimated by determining the temperature drops during the holding period. Based on separated experimental works, the temperature inside DTM mould during holding time was dropped at 0.7 °C/s. The different fraction solids used in this experimental work show the apparent changes in microstructure evolution. The formation of smallest average primary grain diameter in the DTM experimental work occurred at pouring temperature of 685 °C and holding time of 20 s (see Figure 4.22). This was then supported by the statistical analysis results as shown in Figure 4.23. The sample was quenched at the temperature of 671 °C which it was in fully liquid state condition.

The observed correlation between holding time and fraction solid might be explained in this way, during rapid solidification after molten alloy was poured into a copper mould, the nuclei was started to deform from the liquid condition. The nuclei were evolved and impinged to each other within the material and later produced a dendritic microstructure as in typical solidification process. During this process (from the liquid to dendritic microstructure formation), the fraction solid which occurred in the material was increased as it approached the solidus temperature. The formation of the nuclei which was small at this stage, become larger due to the increment of fraction solid volume. The quenching technique which was performed before nuclei become larger captured a smaller and finer globular shape microstructure.

Sample 3 was quenched into ambient temperature water while it still in fully liquid condition (temperature at 671 °C). The quenching action had provided chilling environment when copper mould and molten alloy reached water. This chilling action allows rapid solidification of the melt that later catalysed nuclei formation and produced large number of small size structures. Proper selection of a holding time in DTM is crucial to allow the formation of a desire

microstructure feature due to fraction solid effect. Results in this experimental work show the fraction solid has influenced a microstructure formation. The fraction solid is one of important parameters in SSM, which determines material flowability, similar like in conventional casting process. A different fraction solid which was used in this experimental work has showed the apparent changes in microstructure evolution.

The formation of a globular microstructure in DTM was typically influenced by the raw material used. In order to show this, a comparison was made between aluminium A356 and 7075 which processed by using a DTM [122]. The microstructure for A356 from the literature and 7075 from this work are presented in Figure 5.5. Both of the samples were processed at nearly similar pouring temperature in the range of 640 to 645 °C and holding time in the range of 40 to 75 s. The A356 microstructure was slightly more globular than 7075 with obvious distinction for the primary and secondary phase formation. Even though some of the research indicated that there was likely to process 7075 in semi-solid range temperature, but with this experimental work results, the A356 DTM feedstock billets showed more superior than 7075 in the scope of globular microstructure and the distinction between primary and secondary phase. Furthermore, it was also found that in the case of casting alloy 7075, the eutectic phase and globular structure were less to appear than for the casting alloy A356 [152].



Figure 5.5: Microstructure formation for two different raw materials which processed by using DTM with (a) aluminium A356 [122] and (b) aluminium 7075 (this work).

5.4 Compression Test

5.4.1 Effect of properties of feedstock billets on compression test

A strong relationship was found between different types of feedstock billets used and compression test. The compression test results show that sample 3 was obtained the highest length A-B value. This was an indicator the success of compression test for feedstock billets in sample 3. This result may be explained by the fact that sample 3 feedstock billets initially was produced with higher liquid particles or secondary phase compared to other samples.

In SSM processing, the success of the operation was depended on two major components consist of globular microstructure and liquid а matrix [159, 240]. This can be explained in details as the formation of primary solid phase which in a globular microstructure is surrounded by secondary liquid matrix. During the DTM, sample 3 was guenched while it still in liquids condition (temperature at 671 °C). This explained high liquid content within sample 3 which gives a significant effect to the compression test result. The formation of high liquid content inside sample while processing assist the microstructure to slip between others and easily formed into a die cavity shape.

Literature study shows one of the methods used to examine secondary phase was by examining microstructure with a backscattered SEM which highlighted the differences between primary and secondary phases [241]. In current experiment works, similar concept was used except rather than using a backscattered SEM, ImageJ software was used. Initial microstructure for the specimens shortly after the DTM for sample 1 and 3 are presented in Figure 5.6. The arrows in Figure 5.6 show the secondary (liquid) phase, which occurred within the sample. The results show that the secondary phase was higher within sample which quenched at a temperature of 671 °C (sample 3). This phenomenon was supported by the filling test results that sample 3 obtained the highest length value due to the higher secondary phase with average diameter and circularity of the structure before the compression test

was at 82 µm and 0.56 respectively. Furthermore, the highest value of the average secondary phase area before and after the compression test was also dominant in sample 3 (see Figure 4.28 and Figure 4.49 respectively). This gives indication that initial feedstock billet with a high liquid matrix content catalysed the secondary phase during the heating process.



Figure 5.6: Microstructure features for back layer contents were highlighted to enhance the secondary (liquid) phase with arrows to indicate secondary phase for (a) sample 1 and (b) sample 3 before the compression test.

Transfer time results (see Table 4.19) indicated that the transfer time gives a minimum effect to the overall length of the sample. Statistical analysis (Table 4.21) indicated that several correlations existed between transfer time and overall length of the sample. However, the correlation was not so significance. The feedstock billets relations between transfer time and formability was the fraction solid. During transport from the furnace to forming die, the temperature of the feedstock billets was released to the environment by convection. This temperature lost changed the fraction solid content within samples. The relation between fraction solid and temperature drops within aluminium 7075 were discussed in detail in several publications [21, 242], and with other materials [240].

5.4.2 Effect of Filling Test on Microstructure

In order to give a better understanding of changes in microstructure between initial feedstock billet after DTM and compression test, the microstructure images are compared in Figure 5.7. The general microstructure formation after compression test were more globular and larger in size compared to initial feedstock billet (after the DTM). The microstructure in Figure 5.7 (b) consists of Al solid grain, which is surrounded with the secondary (liquid) phase. This result shows the microstructure from one of the sample 3 which exceed the highest average overall length A-B value. The microstructure was changed to a more globular structure with a higher average grain size after the feedstock billets were re-heated to semisolid range temperature.



Figure 5.7: Aluminium 7075 microstructure for the feedstock billets sample 3 with (a) after the DTM and (b) after the compression test.

Table 5.2 shows average grain size measurements for sample 3 microstructure before and after the compression test consists of primary grain diameter, circularity and secondary phase area. It is apparent from this table that microstructure formations after the compression test was larger in size and more globular than microstructure before the compression test. The average grain diameter was increased to 34 % while the average grain circularity was increased to 5 % compared to initial feedstock billet before the compression test. The average secondary phase area was also increased at 9 %.

Possible explanations for this might be the heating process of feedstock billets before and during the compression test. The feedstock billets were heated to the semisolid temperature range and during this time eutectic phase, which is the lowest melting temperature melted first. This reaction believed was occurred at 477 °C [242]. Microstructure in Figure 5.8 shows the microstructure of sample 3 with higher magnifications with primary α -Al solid grain and secondary (liquid) phase. The liquid phase infiltrated solid particles which split microstructure into several large grains, which known as primary α -Al solid grain phase as in Figure 5.8.

	Diameter (µm)	Circularity	Secondary Phase Area (µm²)
Before Compression Test	82	0.56	46915
After Compression Test	110	0.59	51191

Table 5.2: Average primary phase grain size measurements for microstructure of sample 3 before and after the compression test.



Figure 5.8: Microstructure for sample 3 with higher magnifications.

During heating process (temperature at 620 °C) it has been seen that primary α -Al was surrounded by the eutectic liquid phase. As the temperature increased, fraction liquid increased and fraction solid decreased in feedstock billets. This resulted the melting of sharp corner of grains and produced more globular structure of primary α -Al within the secondary (liquid) phase. This is evident in Figure 5.8 which contains a sharp corner of the primary α -Al. The findings of the current study are consistent with most of the results reported in literature which found final microstructure of SSM slurry by thixoforming consists of primary α -Al grains surrounded with secondary (liquid) phase [33, 41, 138, 192]. During the time feedstock billet was transferred into a forming die, the feedstock billet temperature was approximately at 609 °C which was related to a 0.6 fraction solid that an evidence of the presence of high secondary phase content [242]. As the initial microstructure of feedstock billets in sample 3 was surrounded by a higher secondary phase content, it catalysed

the formation of more α -Al solid grains as in Figure 5.8. A similar phenomenon was also found in several publications [175, 243-246].

5.5 EDXS Analysis

The EDXS results showed the significant composition difference between the DTM and the compression test samples, especially for the element at the grain boundaries. The analysis also revealed compositions for primary phase (α -Al) for DTM samples consist with AI, Zn and Mg such as in Table 4.17. Likewise, the same composition was found in the primary phase of the compression test sample shown in Table 4.28. Further analysis was made on both samples at the grain boundary area or secondary (liquid) phase. The grain boundary compositions for the DTM specimen were contained only with AI and Cu as shown in Table 4.18. Meanwhile, the compositions for the compression test sample were consisted with AI, Cu, Zn and Mg as shown in Table 4.29. The difference in composition at the grain boundary for both specimens was likely because of the precipitation sequence. The complete precipitation sequence within the sample was with the compression test sample because the sample was allowed to solidify within a forming die. The solidification process for the DTM was unable to provide a complete precipitation sequence because the samples were quenched into ambient temperature water. The precipitation sequence of Aluminium 7075 was started with α -Al, followed by the Al + Al₃Fe and then AI + Mg_2Si . The next reaction was the eutectic phase, which precipitate last during solidification and melt first during heating consist of Al + Al₂Cu + MgZn₂ + Al₂Mg₃Zn₃. The eutectic phase occurred between the primary phases (α -Al) at the grain boundaries [44, 175]. This phenomenon explained the cause of different compositions between DTM and compression Test sample at the grain boundary which allowed only AI and Cu elements within the DTM sample as a complete precipitation sequence not occurred.

5.6 Mechanical Properties

The mechanical properties for some of the feedstock billets were close to those reported in the literature [176], especially the results for sample 1 presented in Table 4.30. The mechanical properties for SSM components were typically lower than for wrought formed components in terms of tensile strength, yield strength, elongation and hardness [138]. Some previous researchers have used SIMA to fabricate feedstock and compare the thixoformed microstructural features with the process parameters and resulting component properties [176]. Table 5.3 presents the mechanical properties results obtained from the literature which used SIMA and this work.

The highest values for tensile, yield strength and hardness for sample 1 compared with other samples can be attributed to its lower porosity content. The porosity content in sample 1 was the least compared to other samples. The result correlated well with the corresponding density measurement which was the highest for this sample, shown in Table 4.31. The high porosity content in SSM processed component has been reported as a common process problem [138, 176, 183]. The porosity within the sample is believed to have come from the DTM process due to pouring method used which could entrain air due to the narrow entrance to the mould. Furthermore, the density in sample 3 which obtained the lowest density value (higher porosity) was processed with a pouring temperature of 685 °C and a holding time of 20 s. This sample was at temperature of 671 °C when it was quenched into ambient temperature water. A similar result was found for sample 6 which had a density of 2.52 g/cm³ and was quenched at temperature of 651 °C. Both of the samples (3 and 6) holding periods were 20 s and quenched far above the liquidus temperature which still in complete liquid state.

Source	Grain Size (µm)	UTS (MPa)	YS (MPa)	8 (%)	Hardness (HV10)
This Work (sample 1)	124	213	206	1.5	115
Mohammadi [154]	70	220	-	5.1	130
ASM [172]	-	221	96.5	9 -10	-
Farshid [177]	113	234	191	2	87

Table 5.3: Comparison between grain size, UTS, YS, **£** and hardness values from this work and the literature.

One of the causes for the porosity within material was the trapped air during processing [247]. Porosity would be expected to occur in a structure due to dissolve hydrogen in liquid and insufficient mass feeding of liquid into spaces between dendritic arms [32, 247, 248]. During the dendritic growth slightly after the molten was poured into a copper mould, the liquid in front of the solid-liquid interface becomes enriched with hydrogen. The hydrogen between dendritic arms could therefore be expected to increase continuously as the temperature drops which in turn results in generated hydrogen bubbles as solid forms. The higher amount of dissolved hydrogen is rejected and diffuses into bubbles. Formed bubbles which moved, form inter dendritic regions toward remaining liquid during solidification would have not sufficient time to exit to the top of the copper mould. Therefore, they remain in the non-solidified liquids and such bubbles form large porosities in the sample.

Grain size affects the mechanical properties with smaller size resulting in higher mechanical properties. The results show that the lowest grain size was in sample 3 (110 μ m) as presented in Figure 4.45. However, this sample also exhibited lower tensile test at 83 MPa. This may be due to the fact that the porosity content in sample 3 was high which would have an overriding effect on the tensile properties. This finding shows that the feedstock billets were able to be processed with a smaller grain size but that porosity was a more dominant factor that effected mechanical properties than the grain size. Previous studies also show that a more globular structure is less effective to prevent crack initiation and results in lower strength values. This was not found within the range of casting conditions examined within this work.

Another factor that affected the mechanical properties within the sample was the secondary phase area. It was found that sample with the highest value of secondary phase area exhibited the lowest mechanical properties as this component was less effective to block the crack propagation. The results show that the highest average secondary phase area was in sample 3 as presented in Figure 4.49 which was also obtained the lowest mechanical properties value.

The statistical analysis, which indicated the correlation between results, shows the relationship between properties and factor which affect the properties (see Table 4.32). Strong correlation coefficient (0.70 to 0.99) was found between UTS–density, YS–density and hardness–density. Moderate correlation coefficient (0.4 to 0.69) was also found between elongation–grain size, UTS–circularity, hardness-circularity, UTS–secondary phase area, hardness–secondary phase area and hardness–overall length A-B. Rest of the results were found at weak correlation coefficient (0.1 to 0.39).

In order to have higher mechanical properties of thixoforming components, it was suggested to have the post-treatment process shortly after the thixoforming operation. It has been shown and is well known that the tensile properties significantly increase when the heat treatment process is applied after thixoforming. The eutectic phases are concentrated in the inter-globular regions. Solution treatment must therefore homogenise this inhomogeneous structure [138].

5.7 Coefficient of Thermal Expansion

The measured CTE and thermal dimension expansion for all samples show the increase in CTE and thermal dimension expansion with the smaller microstructure size sample as presented in Figure 4.73 and Figure 4.74. The results were found similar to the trend in the literature [249-251]. The coarsening microstructure of original Cu-Zn alloy was found to produce lower CTE value compare with refined alloy that had grain size in the range of 3 to 6 μ m [249]. Moreover, the difference between thermal dimension expansion value for as-cast crystalline coarse-grained and 7.5 nm samples was evident at

1.6 μ m (Δ I) [250]. However, due to lack of information in literature for CTE and thermal dimension expansion properties which processed with SSM, the comparison was made with the similar aluminium 7075 materials. The T6 tempered 7075 was found at 21 x10⁻⁶ K⁻¹ [251].

The CTE and thermal dimension expansion values were mostly influenced by the microstructure size and circularity due to the expandability of the structure depended on allowable tolerance between structures. Previous researchers have found that thermal dimension expansion was larger within samples that had smaller grain structures [250]. This was evident in sample 1, 2 and 3 where microstructure size was in the range of 110 to 124 µm which had CTE values from 35 to 37 $\times 10^{-6}$ K⁻¹ compared with sample 10 at 136 µm grain size which had a CTE value of 34 x10⁻⁶ K⁻¹ (see Figure 4.73). Meanwhile, the thermal dimension expansion value for sample 1, 2 and 3 was in the range of 175 to 183 µm and sample 10 at 171 µm (see Figure 4.74). Furthermore, the liquid (secondary) phase was found to be lower in sample 10 compared to sample 3 as presented in Figure 4.49. The higher content of secondary phase has previously been linked with increased ability for structure expansion [214]. Thermal dimension expansion of material was considered as the increment in inter-atomic distance [215, 249]. During heating process, structure of material expands due to heat effects. The expansion for a smaller structure size such as in sample 3 was higher than sample 10 due to more spaces for a structure to expand. In fact, the higher (liquid) secondary phases were found in sample 3 which then assisted these structures to move. It was found that the microstructure features such as size, shape, phase distribution and composition was among the factor that influenced the CTE [252].

The statistical analysis showed the relationship between CTE, thermal dimension expansion and factor which affect thermal expansion (see Table 4.33). Strong correlation coefficient (0.70 to 0.99) was found between CTE– grain size and thermal dimension expansion-circularity. Moderate correlation coefficient (0.4 to 0.69) was also found between thermal dimension expansion-grain size, thermal dimension expansion–circularity and CTE-circularity. Weak correlation coefficient (0.1 to 0.39) was found between CTE–secondary phase area and thermal dimension expansion–secondary phase area.

CHAPTER 6

CONCLUSIONS

6.1 Thermal Analysis

In order to gain useful information of phase transformation temperatures from thermal analysis experimental work, a steady heat flow rate from the metal to the surrounding environment must be achieved to avoid cooling curve distortions which are not related to latent energy release effects. This was achieved in this research with a specially designed insulated chamber to achieve a slow cooling rate, cooling of the melt within the crucible in a quiescent open atmosphere for the intermediate cooling rate, and with use of a set air flow rate over the crucible to achieve the highest cooling rate. Furthermore, the novel processing conditions used in the research (750 g material weight and data sampling at 500 readings/second) provided a close to actual application in casting floor and detailed information on $T_a - f_s$ and dendritic coherencytemperature relation for SSM processing. Cooling rate and fraction solid are important parameters for thixoforming. The thermal analysis performed on the captured experimental cooling curves within this research has shown that the different cooling rates have had a marginal effect on the liquidus, eutectic and solidus phase change temperatures. Temperatures of coherency point were also found to be weakly dependent on the cooling rates within the range investigated within this work. The fraction solid was found to be strongly dependent on temperature. Below the coherency points (approximately 0.20 f_s for all cooling rates), the slopes of the fraction solid curves were not very steep which indicates that the fraction solid is sensitive to temperature fluctuation within this region. The thermal profiles which obtained from this experimental work were used to determine the pouring temperature and fraction solid volume in DTM. Furthermore, the thermal profiles were also used to determine heating temperature and fraction solid volume in compression test experiment. These results contribute vital information and were encouraging to conclude that

suitable processing conditions can be applied for the thixoforming of 7075 for achieving optimised properties.

6.2 Direct Thermal Method

The combination of an adequate pouring temperature and a holding time produced an appropriate smaller size and a globular microstructure. In particular, the cooling rate and the fraction solid volume also play the important rules in determining the formation of a spheroidal microstructure and finer structure. The wrought aluminium 7075 which tend to have a narrow temperature window and high flowability resistance used in this research produced average primary microstructure phase diameter in the range of 82 to 123 µm and circularity in the range of 0.55 to 0.67. Higher pouring temperature was found significantly produced higher secondary phase that led to greater fluidity within sample. The higher cooling rate that was used for SSM feedstock processing has produced a small, globular and a uniform microstructure. The combination of pouring temperature at 685 °C and the holding time of 20 second produced the smallest primary phase structure size. Likewise, the combination between pouring temperature and holding time of 665 °C and 60 s respectively, produced the highest circularity value at 0.5 f_s . The finding presented in this research contribute details understanding and information regarding the behaviours of the wrought aluminium 7075 processed by DTM for thixoforming.

6.3 Compression Test

The determined important characteristics such as microstructure formation and flowability of DTM aluminium 7075 feedstock billets which were processed by using a compression test unit within the semisolid temperature range were presented in this research. The novelty of flowability test through aluminium 7075 DTM feedstock billets with the compression test determines formability of the material. The average primary grain diameter, which formed after the forming operation was found in the range of 110 to 136 μ m that increased from

its initial size. Meanwhile, the average primary grain circularity and average secondary (liquid) phase area were in the range of 0.59 to 0.71 and 32704 to 51191 μ m² respectively. The experimental results show that formability of the feedstock billets was significantly influenced by the primary grain circularity and secondary (liquid) phase content within the feedstock billets. The primary grain size was found gives less effect to material formability. The microstructure, as examined after compression test, showed the formation of more globular α -Al solid grain surrounded by higher secondary (liquid) phase within the sample that assists the feedstock billet formability. The largest secondary phase within sample leads the larger length of flowability. The significant findings in this research work contribute novel information regarding microstructure formation, such as primary grain size, circularity and secondary (liquid) phase area for aluminium 7075 DTM feedstock billets after the heating process.

6.4 Mechanical Properties

The mechanical properties of some feedstock billets after compression test have the similar properties such for aluminium 7075 thixoformed component. The novelty of this mechanical test was at the test samples, which used aluminium 7075 feedstock billets formed by DTM, produced the tensile properties (UTS) between 83 and 213 MPa. The yield strength (YS) was in the range of 64 to 206 MPa, elongation was between 0.7 to 2.7 % and hardness was in the range between 85 to 115 HV10. The material properties were decreased significantly by the higher porosity and secondary (liquid) phase within the sample. This was evident by the density and fractography test that indicated density values were in the range of 2.31 to 3.01 g/cm³ and void formation in fracture surfaces. Structure with greater amount of secondary phase was found significantly has the lowest strength and hardness properties. Processing parameters that led to samples with increased density led to higher strength and hardness. Processing parameters that led to samples with increased circularity led to higher hardness. The findings in this research contribute novel information on mechanical properties such as UTS, YS, elongation, hardness, density and fractography for aluminium 7075 DTM feedstock billets after the forming operation.

6.5 Coefficient of Thermal Expansion

The CTE and thermal dimension expansion values for aluminium 7075 DTM feedstock billets after compression test were higher with smaller structure size. The novelty of this research that used SSM feedstock billets after forming operation provided the useful information on the CTE and thermal dimension expansion, which occurred in the range of 34 to 37 $\times 10^{-6}$ K⁻¹ and 171 to 183 µm. The factors which affect the CTE and expansion were average primary phase grain diameter and circularity. The CTE was much influenced by the size of the microstructure, which indicated smaller grain diameter significantly increased the CTE values. Furthermore, the more globular microstructure features were also found to increase the CTE. The findings contribute detail information about SSM feedstock billets behaviour in relation to dimensional related thermal dimension.

6.6 Suggestion for Future Work

Further research work needs to be done to establish more understanding on improved parameters such as pouring temperature and holding time to produce a finer and more globular microstructure by using DTM. The concentration should be given to the external cooling medium on copper mould which accelerates heat convection from the molten alloy to the environment in order to increase nucleation rates. Moreover, detail experimental works should be conducted on different wrought aluminium alloys to understand their behaviour in SSM range temperature produced by DTM. The high porosity content within sample should be controlled to improve mechanical properties and such treatment process should be conducted for the molten alloy before pouring molten alloys into the copper mould. Other work which will need further investigation is the simulation of heat flow within copper mould during the solidification process in order to give deeper relevant information such as heat flow and pressure. More broadly, research is also needed to determine the detail compression test parameters effect to material behaviour with different parameters setting such as compression force, packing time and mould temperature. More concentration should be given on improving heating element

at the forming die to reduce the temperature difference between a feedstock billet and a forming die. A future study on investigation of the material flow in forming by using simulation would also be advantageous to allow for more indepth understanding and prediction of material flow behaviour.

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APPENDICES

A. Phase diagram for Al-Zn-Mg-Cu system [253].



Table A.1: The chemical composition for aluminium 7075.

Source (wt%)	AI	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn
ASM [171]	87.1- 91.4	0.18- 0.28	1.2- 2.0	<0.5	2.1- 2.9	<0.3	<0.4	<0.2	5.1- 6.1

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	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr
1	88,6	0,138	0,258	2,10	0,125	2,18	5,94	0,216
2	88,5	0,134	0,233	2,00	0,121	2,43	6,05	0,192
3	88,4	0,141	0,224	2,00	0,125	2,44	6,14	0,203
4	88,6	0,142	0,241	2,02	0,122	2,35	6,04	0,200
5	88,5	0,143	0,218	1,99	0,116	2,49	6,01	0,200
					•			
	Ni	Ti	Be	Ca	Li	Pb	Sn	Sr
1	0,0261	0,0945	0,0002	0,0033	0,0001	0,0620	0,0599	0,011
4	0,0180	0,0891	0,0002	0,0019	< 0.0001	0,0565	0,0345	0,000
4	0.0113	0.0837	0,0002	0,0017	< 0.0001	0,0462	0.0361	0,004
5	0,0147	0,0843	0,0002	0,0017	< 0,0001	0,0502	0,0326	0,001
Ave	0,0168	0,0871	0,0002	0,0021	0,0001	0,0530	0,0400	0,003
	v	Na	Bi	Zr	в	Ga	Cd	Co
1	0,0143	0,0061	0,0224	0,0128	0,0041	0,0147	0,0019	< 0,003
2	0,0166	0,0033	0,0098	0,0067	0,0021	0,0132	0,0010	< 0,003
3	0,0142	0,0041	0,0085	0,0097	0,0025	0,0131	0,0016	< 0,003
4	0,0169	0,0084	0,0058	0,0070	0,0024	0,0120	0,0019	< 0,003
Ave	0,0156	0,0060	0,0109	0,0083	0,0026	0,0129	0,0016	< 0,003
	Ag	Hg	In					
1	0,0045	< 0,0030	< 0,0100					
2	0,0037	< 0,0030	< 0,0100					
3	0,0038	< 0,0030	< 0,0100					
4	0,0044	< 0,0030	< 0,0100					
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C. LabVIEW Programming for Thermal Analysis Experiment



Figure C.1: LabVIEW programming for data capture.





Figure C.2: LabVIEW programming for cooling curve and temperature difference.





Figure C.3: LabVIEW programming for 1st derivation of cooling curve with respect to time.





Figure C.4: LabVIEW programming for 1st derivation of cooling curve with respect to time (with baseline).

D. Drawing of The Compression Test Unit



Figure D.1: The schematic diagram of die with the feedstock billets inserted.



Figure D.2: The process flow of the compression test with (a) die without feedstock billet, (b) feedstock billets inserted and (c) upper plate shifted down and pushed plunger toward feedstock billets.

E. Pearson Correlation Coefficient Graph for Mechanical Test



Figure E.1: Pearson correlation coefficient between average UTS (MPa) and average primary phase grain diameter (µm).



Figure E.2: Pearson correlation coefficient between average YS (MPa) and average primary phase grain diameter (µm).



Figure E.3: Pearson correlation coefficient between average elongation (%) and average primary phase grain diameter (µm).



Figure E.4: Pearson correlation coefficient between average hardness (HV) and average primary phase grain diameter (µm).



Figure E.5: Pearson correlation coefficient between average UTS (MPa) and average primary phase grain circularity.



Figure E.6: Pearson correlation coefficient between average YS (MPa) and average primary phase grain circularity.



Figure E.7: Pearson correlation coefficient between average elongation (%) and average primary phase grain circularity.



Figure E.8: Pearson correlation coefficient between average hardness (HV) and average primary phase grain circularity.



Figure E.9: Pearson correlation coefficient between average UTS (MPa) and average secondary phase area (μm^2).



Figure E.10: Pearson correlation coefficient between average YS (MPa) and average secondary phase area (μ m²).



Figure E.11: Pearson correlation coefficient between average elongation (%) and average secondary phase area (µm²).



Figure E.12: Pearson correlation coefficient between average hardness (HV) and average secondary phase area (μm^2).



Figure E.13: Pearson correlation coefficient between average UTS (MPa) and density (g/cm³).



Figure E.14: Pearson correlation coefficient between average YS (MPa) and density (g/cm³).



Figure E.15: Pearson correlation coefficient between average elongation (%) and density (g/cm³).



Figure E.16: Pearson correlation coefficient between average hardness (HV) and density (g/cm³).



Figure E.17: Pearson correlation coefficient between average UTS (MPa) and overall length A-B (mm).



Figure E.18: Pearson correlation coefficient between average YS (MPa) and overall length A-B (mm).



Figure E.19: Pearson correlation coefficient between average elongation (%) and overall length A-B (mm).



Figure E.20: Pearson correlation coefficient between average hardness (HV) and overall length A-B (mm)



F. Pearson Correlation Coefficient Graph for Thermal Expansion Test

Figure F.1: Pearson correlation coefficient between coefficient of thermal expansion ($x10^{-6}K^{-1}$) and grain size (μ m).



Figure F.2: Pearson correlation coefficient between coefficient of thermal expansion $(x10^{-6}K^{-1})$ and circularity.



Figure F.3: Pearson correlation coefficient between coefficient of thermal expansion ($x10^{-6}K^{-1}$) and secondary phase (μm^2).



Figure F.4: Pearson correlation coefficient between thermal dimensional expansion (µm) and grain size (µm).



Figure F.5: Pearson correlation coefficient between thermal dimensional expansion (µm) and circularity.



Figure F.6: Pearson correlation coefficient between thermal dimensional expansion (μ m) and secondary phase (μ m²).