MECHANICAL PROPERTIES AND MICROSTRUCTURE EVOLUTION OF THIXOFORMED ALUMINUM ALLOYS

by

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ABSTRACT

MECHANICAL PROPERTIES AND MICROSTRUCTURE EVOLUTION OF THIXOFORMED ALUMINUM ALLOYS

Thixoforming is an attractive process for the manufacture of complex parts with substantial savings of time and cost. Different from the conventional metal forming technologies which use either solid metals (forging) or liquid metals (casting) as starting materials, thixoforming deals with semi-solid slurries Thixoforming is composed of three main processes, i.e. thixotropic feedstock production, reheating, and thixoforming. In this study, three steps of the thixoforming process were carried out for A357 and AA6082 alloys. The production of a fine, equiaxed, globular microstructure (often referred to as “thixotropic) is a must for the success of the thixoforming process. In the first step, cooling slope (CS) casting process was carried out to obtain such a feedstock. The effect of casting temperature and pouring length on the formation of thixotropic microstructure were investigated. In the reheating step, as-cast billets were partially remelted at semi-solid temperature intervals and isothermally held for suitable durations with an induction furnace in order to observe the microstructural evolution. In the forming step of the process, as-cast billets were inductively reheated to the temperatures practiced in the second step and then thixoformed between dies. Microstructural evolution and mechanical properties (hardness, tensile strength, yield strength, elongation to fracture) of the thixoformed samples were investigated. In order to use the advantage of low viscosity presented in the thixotropic feedstocks, thixoforming was performed with lower press loads comparing to the loads used in literature.

ÖZET

YARI-KATI HALDE ŞEKİLLENDİRİLEN ALÜMİNYUM ALAŞIMLARININ MEKANİK ÖZELLİKLERİ VE MİKROYAPISAL EVRİMİ

Yarı-katı halde şekillendirme, karmaşık şekilli parçaların daha az sayıda süreç adımı ve daha düşük maliyetlerle üretilmeleri için cazip bir yöntemdir. Hammadde olarak katı metalleri (dövme) veya sıvı metalleri (döküm) kullanan geleneksel işlemlerden farklı olarak; yarı-katı halde şekillendirme işlemi yarı-katı durumdaki malzemeleri şekillendirir ve hammadde üretimi, tekrar ısıtma ve şekillendirme olmak üzere üç ana prosesden oluşur. Bu çalışmada yarı-katı halde şekillendirmenin üç basamağıda A357 and AA6082 alüminyum alaşımları için uygulanmıştır. Eşeksenli ve yuvarlak ince taneli bir mikroyapının (tiksotropik olarak adlandırılır) üretilmesi yarı-katı halde şekillendirme sürecinin ilk işlemini oluşturur. Bu tür bir yapının oluşturulması için eğimli plakadan döküm işlemi uygulanmıştır. Döküm sıcaklığının ve döküm uzunluğunun tiksotropik yapı oluşumu üzerindeki etkisi incelenmiştir. Tekrar ısıtma işleminde, dökülen kütükler indüksiyon fırını kullanılarak yarı-katı sıcaklık aralıklarında kısmen eritilmiş ve bu sıcaklıklarda bazı bekleme sürelerine tabi tutulmuşlardır. Isıtılan kütüklerde mikroyapısal evrim gözlemlenmiştir. Üçüncü basamak olan şekillendirme işleminde, dökülen kütükler ikinci basamakta uygulaması yapılmış sıcaklıklara tekrar ısıtılıp, özel olarak tasarlanmış kalıplarda yarı-karı halde şekillendirilmişlerdir. Şekillendirilmiş örneklerin mikroyapısal evrimleri ve mekanik özellikleri (sertlik, çekme dayanımı, akma dayanımı, uzama) tespit edilmiştir. Tiksotropik hammaddenin doğasında olan düşük vizkozite özelliğinden yararlanmak amacıyla şekillendirme işlemi literatürde uygulanan kuvvetlere oranla çok daha düşük yükler altında gerçekleştirilmiştir.

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LIST OF SYMBOLS/ABBREVATIONS

ΔC	Solid solubility difference between semisolid temperature and aging temperature
dT_p/dt	Variation of temperature via time
F_g	Particle shape factor
f_s	Solid fraction
N_A	Particle density
RC	System parameter
s	Structural parameter
γ_o	Shear rate
CS	Cooling slope
LTP	Low pouring temperature
MHD	Magneto-hydrodynamic
RAP	Recrystallization and partial melting
SCR	Shearing-cooling roll
SIMA	Strain induced melt activation
SSM	Semi-solid metal
SSP	Single slug production method
TSRM	Laboratory scale twin screw rheomoulding

1. INTRODUCTION

The new trend in automotive industry to produce more fuel-efficient vehicles has resulted in the increased use of aluminum alloys. Especially for light weight structures aluminum is on advance. Furthermore, firms are looking for cost reduction due to shorter process routes and fewer forming operations [1]. A necessary condition for the manufacture of low-cost lightweight aluminum parts is the ability to process this expensive material into near net shape components of complex geometry and high strength [2]. Die casting and permanent mold methods have been used in manufacturing critical parts in aluminum, but have a number of quality and weight problems to overcome. Soundness, property uniformity, and near-net-shape limitations (part weight) are some of the issues that have troubled the users of such parts. Over the past five years, thixoforming has developed into a mature, high volume production method capable of satisfying the need for near-net-shape, high quality, low cost aluminum parts [3]. The advantage of this new process over die casting as an economic casting method becomes clear when the component cannot be satisfactorily produced by conventional die casting (thick-walled component, special alloys) or if additional stages in production can be saved. Compared to forming in a solid state, the main advantage is the high flowability, enabling complex shapes and a complicated material distribution in only one forming step. The concomitant low investment costs and material savings due to burr-free forming often more than compensate the extra costs and work for the very exact heating which is required [4].

Thixoforming process is a relatively new method for forming alloys in the semisolid state to near net shaped products. This recently developed technology was derived from basic studies initiated by Flemings and collaborators in the 1970s [5]. Conventional forging is done in the solid state, whilst conventional casting is done in the liquid state. Thixoforming takes place in the solidus-liquidus region and has the advantages of giving possibility of forming complex parts with cast and wrought alloys with shorter process routes [6]. Thixoforming takes advantage of the unique thixotropic behavior found in the semi-solid state. In this state, the alloy generally exhibits decreasing viscosity with increasing shear. This unique viscosity behavior is a result of the buildup and breakdown

of the solid phase within the slurry. With no shear stress applied on the slurry, the semi-solid slurry behaves like a solid allowing the operator or robot to handle the slurry prior to forming. Once shear is applied to the slurry (during casting) the structural buildup of the solid breaks down allowing the slurry to flow like a high-viscosity liquid. This unique thixotropic nature of semi-solid slurry is exhibited in Figure 1.1 where a semi-solid slug was cut like butter with a knife [7].

Figure 1.1. Slug in semi-solid state being cut with a knife exhibiting the unique thixotropic nature of the semi-solid material [7]

Today, thixoforming has established itself as a scientifically sound and commercially viable technology for production of metallic components with high integrity, improved mechanical properties, complex shape, and tight dimensional control [8]. Although there will initial extra capital costs involved in providing specialized new equipment and higher running costs in using specially prepared feedstock, there will be many applications where the benefits cited above will ensure that thixoforming is a commercially viable technology. As well as automobile wheels, these presently include aluminum master brake cylinders, fuel systems, electrical connectors, valve bodies and brass plumbing fittings complete with

threads [5]. Figure 1.2 shows some of the components produced by thixoforming at Stampal for an Alfa Romeo car [9]. Economically interesting applications already exist for aluminum casting alloys. The fundamental feasibility of such processes has also been proven for aluminum wrought alloys and a wide range of refractory materials [4].

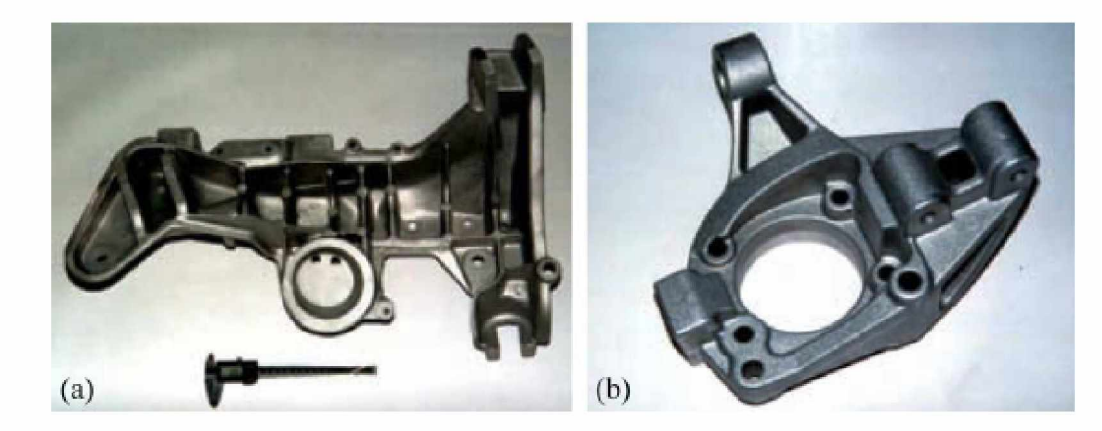

Figure 1.2. Automotive components produced by STAMPAL for the Alfa Romeo car: a) multi-link, rear suspension support 8.5 kg, A357, T5; b) steering knuckle A357, T5 substitution of cast iron part [9]

Thixoforming basically consist of three phases: (i) manufacturing of material with non-dendritic microstructure, (ii) heating the material to the forming temperature, and (iii) forming the material in a die casting press (thixocasting) or forging press (thixoforging) as shown in Figure 1.3 [10]. In thixoforming, specially produced non-dendritic billet is heated - as a rule inductively - to a temperature between solidus and liquidus. This material then ideally consists of a fine and evenly distributed globular solid phase in a matrix melt. In this condition the material can be handled like a solid and placed into a forming die. Depending on whether forming takes place in a die casting machine or a forging press, one talks of thixocasting or thixoforging. The shear stresses which occur during forming lead to a reduction in viscosity, the billet behaves in the same way as a viscous fluid despite its high share of solid phases (approximately 40 - 60 per cent) and permits the production of complicated geometries at relatively low forming forces [4].

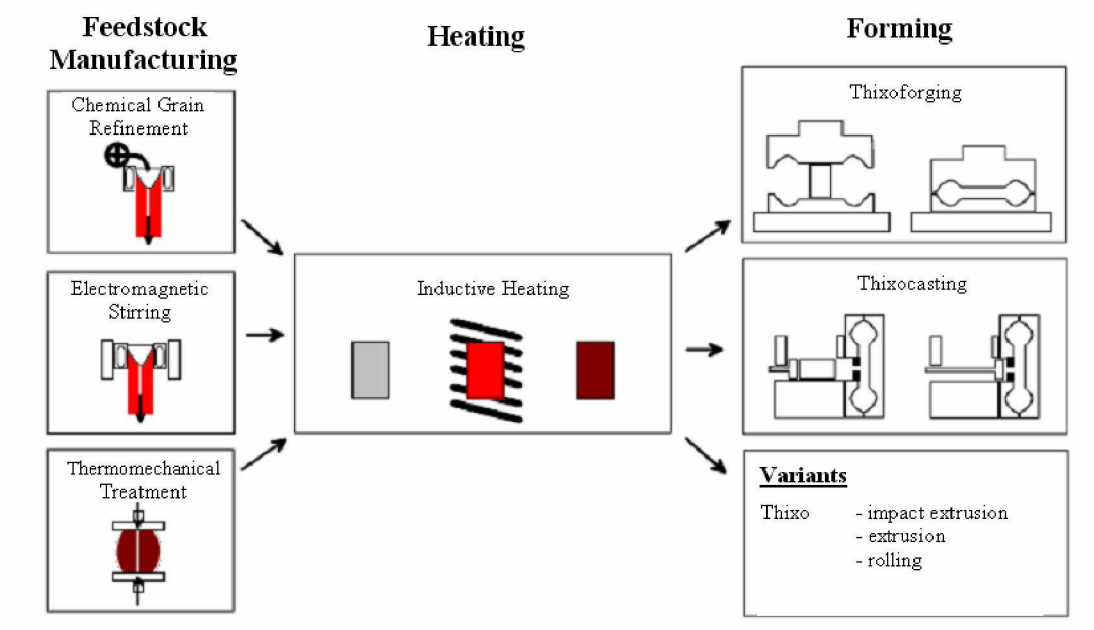

Figure 1.3. Diagrammatic representation of thixoforging and thixocasting [4]

Semisolid billets production, which is to obtain billets with a non-dendritic microstructure and behaving with thixotropic nature after proper partial remelting, becomes the basis and the key to the whole process [11]. Feedstock can be produced by either solid or liquid state routes. Feedstock is produced mainly by magneto-hydrodynamic (MHD), strain induced melt activation (SIMA), recrystallization and partial melting (RAP), or spray casting. In addition, there have been several attempts to employ ultrasonic, low pouring temperature (LTP), grain refining, and cooling slope casting (CS) for feedstock preparation. However, there have been few published reports on the latter process [12]. A cooling slope is the simplest process to make the ingot for the thixoforming. Only pouring the melt on the cooling slope and solidifying the metal, the ingot for thixoforming is obtained [13]. In this study, cooling slope casting process was carried out in order to obtain thixotropic feedstock for the alloy groups A357 and AA6082. After producing feedstock via pouring down the molten metal through a cooling slope plate, the produced billets were inductively heated to semi-solid interval and formed with a hydraulic press.

The principal objectives of the present study are:

- To obtain thixotropic feedstock via cooling slope casting process and to investigate the effect of pouring temperature and pouring distance on the formation of thixotropic microstructure.
- To investigate the morphologic evolution of as-cast billets via induction heating and isothermal holding practices.
- To investigate microstructural evolution and mechanical properties (hardness, tensile strength, yield strength and elongation to fracture) of the thixoformed samples.

2. THIXOFORMING

2.1. Semi-Solid Forming

Semisolid metal (SSM) processing is a hybrid manufacturing method that incorporates elements of both casting and forging [14]. It is a relatively new technology for metal forming. Different from the conventional metal forming technologies which use either solid metals (solid state processing) or liquid metals (casting) as starting materials, SSM processing deals with semisolid slurries, in which non-dendritic solid particles are dispersed in a liquid matrix [8]. Forming in the semisolid state requires that a metal or alloy have a roughly spherical and fine-grain microstructure when it enters the forming die [14].

The term SSM processing is usually confused with the terms rheocasting and thixoforming. Rheocasting and thixoforming are two types of SSM processing. In brief, rheocasting involves the application of shearing during solidification to produce non-dendritic semisolid slurry that can be transferred directly into a mould or die to give a final product. On the other hand, thixoforming is used to describe the near net shaping of a partially melted non-dendritic alloy slug within a metal die: thixocasting if in a closed die, or thixoforging if in an open die [8]. In rheocasting, slurry is formed when 60 to 70 per cent of the material is liquid and in thixoforming, non-dendritic billet is formed when 30 to 40 per cent of the material is liquid [14].

SSM processing offers to industry parts of complex shapes that have substantially higher quality than, for example, die casting but are lower in cost than those produced by alternative methods such as forging and machining. The higher quality arises primarily from the higher viscosity of the SSM processed material as compared with liquid material [15].

2.1.1 Background of Semi-solid Forming

Semisolid metal forming is based on a discovery made during research on hot tearing undertaken at MIT in the early 1970s. Seeking to understand the magnitude of the forces involved in deforming and fragmenting dendritic growth structures, MIT researchers constructed a high temperature viscometer. They poured model lead tin alloys into annular space created by two concentric cylinders and measured the forces transmitted through the freezing alloy when the outer cylinder was rotated. During the course of these experiments, it was discovered that when the outer cylinder was continuously rotated, the semisolid alloy exhibited remarkably low shear strength even at relatively high fractions solidified. This unique property was attributed to a novel non-dendritic microstructure [14]

The research expanded, and the MIT engineers coined the term rheocasting to describe the process of producing this unique microstructure. They showed that sheared and partially solidified alloys could be assigned an apparent viscosity and that they possess many of the characteristics of thixotropy. Most notably, the semisolid alloys displayed viscosities that depend on shear rate and that rose to several hundred, even thousands, of poise (approaching the consistency of table butter) when at rest and yet decreased to less than 5.0 Pa .s or 50 P (poise) (the range of machine oils) upon vigorous agitation or shearing. For the first time, therefore, these results afforded an opportunity to control viscosity of alloy melts from that of fully liquid to any desired upper limit [14]

The feasibility of SSM processing of various alloys has been investigated in the past 30 years. Initially, the primary focus was on high temperature alloys, notably steels, and practically no attention was given to aluminum and magnesium alloys. This was mainly due to the drive for perfection of steel die casting technology for military applications. Semisolid processing was considered to be an effective means reduce the casting temperature. However, because the oil crises in the 1970s, and increasing environmental concerns since the 1980s, automobile market forces have been pressing hard for weight reduction using high performance light metal parts. As a consequence, since the 1990s, SSM processing has predominantly concentrated on aluminum alloys. In the past decade, SSM processing has experienced intensive research, development, and commercialization. Today, SSM processing has established itself as a scientifically sound and commercially

viable technology for production of metallic components with high integrity, improved mechanical properties, complex shape, and tight dimensional control. Perhaps more importantly, it has demonstrated a great potential for further technological development and commercial exploitation [8].

2.1.2. Advantages and Disadvantages of Semi-solid Forming

Alloy slurries used in thixoforming may contain as much as 80 per cent solid: this has a number of consequences for SSM processing as compared with conventional liquid metal casting into permanent metal dies, leading to both technical and economic benefits [5]. The main specific advantage of the thixoforming route is that the forming facility is free from handling liquid metal, and the process can be highly automated using approaches similar to those employed in forging and stamping. This basic concept of completely separating the two main parts of the process (forming of the desired structure and forming of the part) has been intuitively appealing and much work has been done in developing this process route industrially [14].

Particularly significant for higher-melting alloys, semisolid metal working afforded lower operating temperatures and reduced metal heat content (reduced enthalpy of fusion). The lower temperature and short dwell time lead to longer die life. In addition, the energy required to heat aluminum alloys for casting (ignoring losses) is 35 per cent greater than the energy required to heat the same aluminum alloy to the condition ready for semisolid forming (Figure 2.1). When this is adjusted for the yield differences, the actual advantage of SSM processing can be observed to be substantially greater [14].

The viscous flow behavior could provide for a more laminar cavity fill than could generally achieved with liquid alloys. This could lead to reduced gas entrainment. And also, fluid flow filling die cavity under high final pressure enables the filling of thinner sections and the forming of lighter parts [14].

Solidification shrinkage would be reduced in direct proportion to the fraction solidified within the semisolid metal working alloy, which should reduce both shrinkage porosity and tendency toward hot tearing [14].

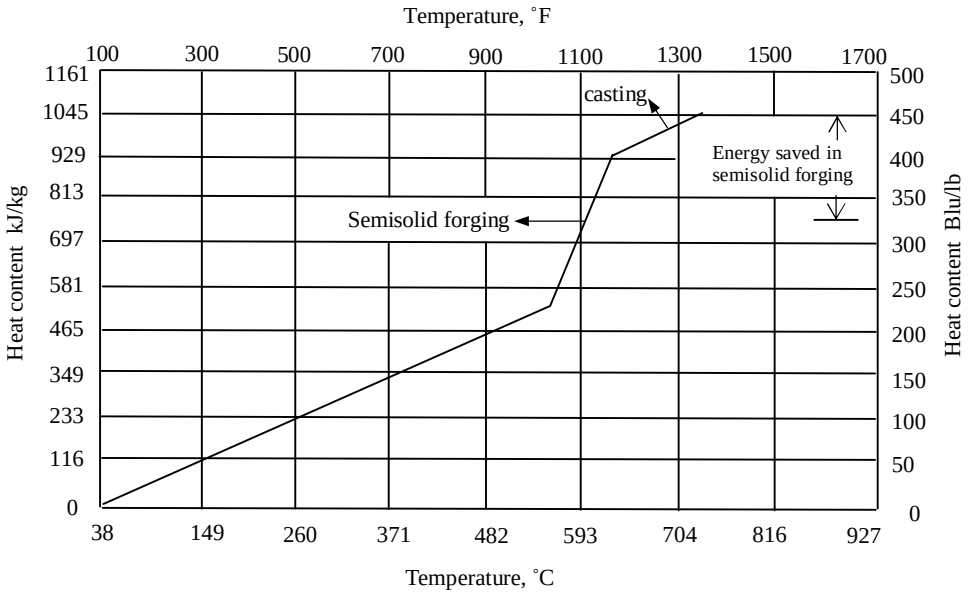

Figure 2.1. Heating curves showing energy savings possible in semisolid forging versus casting of A357 aluminum alloy [14]

Rapid quench in the forging press which is one of the special characteristics of semisolid forming avoids expensive solution treatment to obtain higher properties. This press quench, along with the very fine spherical grains, can be used with some aluminum alloys in order to achieve better mechanical properties with only aging treatment [14].

Beside these advantages, SSM processing has other benefits such as [8]:

- Provide high integrity components, which are heat treatable.
- Provide improved mechanical properties of finished components.
- Produce components with complex shapes and tight dimensional control.
- Produce castings with thin walls.
- Provide high production rate.

In comparing SSM processing with shaping in the solid state, the most significant advantage is that the forming stresses are perhaps 10^{-4} lower in the semisolid state. This means that more intricate shapes can be formed faster, using smaller presses, and with lower finishing costs [5].

With these enabling features in mind, there are several areas where thixoforming process finds most of its applications [8]:

- Existing permanent mould parts where the near net shape capability of SSM can eliminate much of the machining and finishing.
- Parts that must be pressure tight and cannot be produced in conventional die casting, such as master brake cylinders, fuel rails, air conditioner compressor housing, and safety-critical parts.
- High strength parts, such as engine mounts, steering knuckles, alloy wheels, tie rods, control arms, and seat belt retainer housings.
- Wear resistant parts that require hypereutectic alloys, such as unanodised master cylinder, compressor piston, brake drums, and gearshift levers.
- Parts that are forged requiring excessive tooling.

Table 2.1 gives an example of a comparison of an aluminum automobile wheel made by two routes: semisolid forging and gravity die casting. Many of the points above are well illustrated: there is raw material weight saving of one third and the greater integrity and superior alloy properties have allowed the design weight to be reduced by almost a third again, added to this the production rate is increased by a factor of over 7 [5].

Table 2.1. Comparison of semisolid forging (thixoforging) and gravity diecasting for production of aluminum automobile wheels [5]

Process	Weight from Mould/die, kg	Finished Part weight, Kg	Production Rate per Mould/die Pieces, h^{-1}	Aluminum Alloy	Heat treatment	Tensile Strength MN m^{-2}	Yield Strength MN m^{-2}	Elongation %
Semisolid forging	7.5	6.1	90	357 (Al-7Si-0.3Mg)	T5	290	214	10
Gravity diecasting	11.1	8.6	12	356 (Al-7Si-0.5Mg	T6	221	152	8

On the other hand, SSM processing has some disadvantages [9]:

- The cost of raw material can be high and the number of suppliers small.
- Process knowledge and experience has to be continually built up in order to facilitate

application of the process to new components.

- This leads to potentially higher die development costs.
- Initially at least, personnel require a higher level of training and skill than with more traditional processes.
- Temperature control. Fraction solid and viscosity in the semisolid state are very dependent on temperature. Alloys with a narrow temperature range in the semisolid region require accurate control of the temperature.
- Liquid segregation due to non-uniform heating can result in non-uniform composition in the component.

2.1.3. Rheology of Semi-solid Alloy Slurries

Conventional liquid casting processes face many potential problems. These include gas entrapment during turbulent filling operations, hot tearing of the solidifying metal and shrinkage porosity formation. Hot tearing (cracking of the stressed solidifying metal) occurs during the solidification of alloys as a result of the dendritic network limiting the alloy's ductility in the semi-solid state. Shrinkage porosity can also be produced due to the reduced feedability of liquid metal through the dendritic network. These problems are greatly alleviated in alloys with spheroidal primary phase morphologies. Under the action of shear these alloys can fill all areas of a die cavity in a laminar manner, reducing gas entrapment, shrinkage porosity, and hot tearing. It is the rheology of the metal slurries with this spheroidal solid morphology that promote their use in semi-solid forming operations. It is, therefore, important to understand the rheology of these fluids [16].

The modeling of slurry flow into die cavities during thixoforming requires fundamental understanding of their rheological behavior and knowledge of the basic parameters which control the process. Concentric cylinder viscometers or rheometers are usually employed for this purpose. Two types of cylindrical viscometers are possible: the Searle type in which the inner cylinder rotates and the outer remains stationary, and the Couette which has a rotating outer cylinder which inhibits the onset of turbulent flow [5].

Semisolid metal slurries can be roughly divided into two broad categories; 'liquid-like' slurry contains dispersed solid particles and behaves like a fluid under external forces,

while a ‘solid-like’ slurry contains an interconnected solid phase and behaves like a solid, exhibiting a well defined yield strength. The deformation mechanisms for these two types of slurry are fundamentally different. Semisolid metal slurries with a solid fraction less than 0.6 and a globular solid morphology usually exhibit two unique rheological properties: thixotropy and pseudoplasticity. Thixotropy describes the time dependence of transient state viscosity at a given shear rate, while pseudoplasticity refers to the shear rate dependence of steady state viscosity [8].

The first investigation of the rheology of SSM slurries was conducted on the Sn–Pb system by Spencer et al [17]. They showed that the stirred SSM slurry at a solid fraction higher than 0.2 behaves like a non-Newtonian fluid with an apparent viscosity orders of magnitude less than that of an unstirred dendritic slurry. It is this first observation which initiated numerous rheological studies on stirred SSM slurries. Among them is the very extensive study by Joly and Mehrabian [18] on the Sn–Pb system. As demonstrated by Joly and Mehrabian, the rheological phenomena in stirred SSM slurries can be approximately divided into three categories:

- Continuous cooling behavior, which describes the viscosity evolution during continuous cooling at constant cooling rate and shear rate.
- Pseudoplastic behavior, which describes the shear rate dependence of steady state viscosity, or shear thinning behavior.
- Thixotropic behavior, which describes the time dependence of transient state viscosity.

The continuous cooling behavior gives the first insight into the effects of solid fraction, shear rate, and cooling rate on the rheological behavior of SSM slurries. In particular, it is more relevant to the practical conditions set in SSM processing techniques. Figure 2.2 shows an example of results obtained from the continuous cooling experiments on Sn–15Pb alloy carried out by Joly and Mehrabian [18]. Generally, for a given cooling rate and shear rate, the measured apparent viscosity increases with increasing solid fraction, slowly at low solid fraction and sharply at high solid fraction. At a given solid fraction, the apparent viscosity decreases with increasing shear rate and decreasing cooling

rate. This is because both increasing shear rate and decreasing cooling rate promote more spherical particle morphology [18].

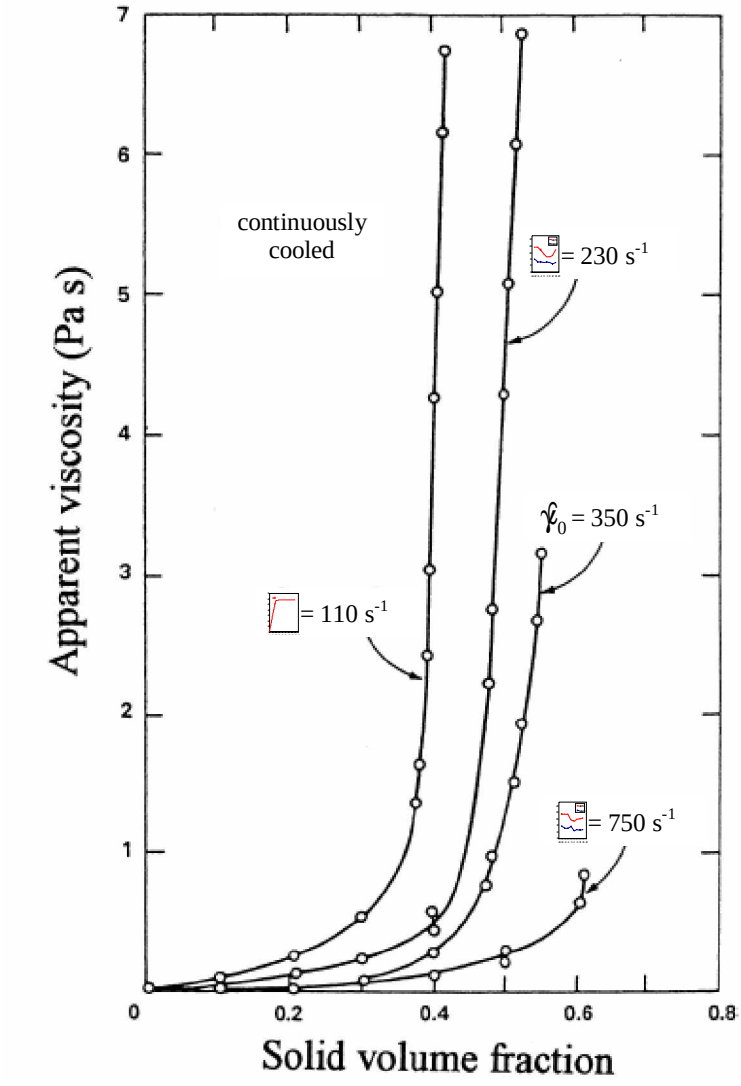

Figure 2.2. Apparent viscosity versus solid fraction f_s of Sn–15Pb alloy sheared continuously and cooled at 0.33 K min−1 at different shear rates γ_o [18]

The isothermal steady state experiments lead to more precise rheological characterization and are a first step towards the determination of a constitutive equation. The steady state is usually defined as a state at which the viscosity of a SSM slurry with fixed volume fraction and shear rate does not vary with prolonged shearing time. Thus, for a given alloy system, steady state viscosity is a function of solid fraction and shear rate.

Joly and Mehrabian [18] showed on Sn–15Pb alloy that the behavior is shear thinning (or pseudoplastic), where the apparent steady state viscosity decreases with increasing shear rate. This shear thinning was demonstrated more generally by Turng and Wang [19], as shown in Figure 2.3. For a SSM slurry with a fixed solid fraction, the steady state viscosity decreases with increasing shear rate, approaching an asymptotic value when the shear rate becomes infinite. It is now generally accepted that the steady state viscosity at a given shear rate depends on the degree of agglomeration between solid particles, which, in turn, is the result of a dynamic equilibrium between agglomeration and deagglomeration processes [8].

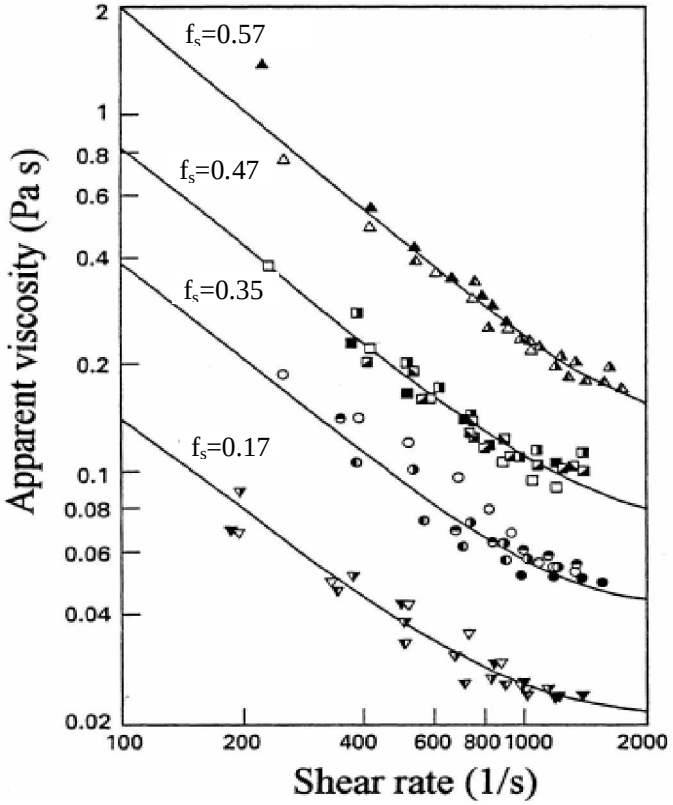

Figure 2.3. Steady state apparent viscosity versus shear rate in Sn–15Pb alloy for various solid fractions f_s [19]

The thixotropic behavior of SSM slurry was first demonstrated by Joly and Mehrabian [18] on Sn–15Pb alloy by measuring the hysteresis loops during a cyclic shear deformation. However, such a procedure is not sufficient to quantify the kinetics of agglomeration and deagglomeration processes. To overcome this shortcoming, special

experimental procedures involving an abrupt shear rate jump or a shear rate drop were developed to characterize the kinetics of structural evolution [8]. An example of such thixotropic experiments obtained by Mada et al. [20] for Sn–15Pb alloy is shown in Figure 2.4. It has been found that the agglomeration process dominates after shear rate drop, whereas the deagglomeration process dominates after a shear rate jump.

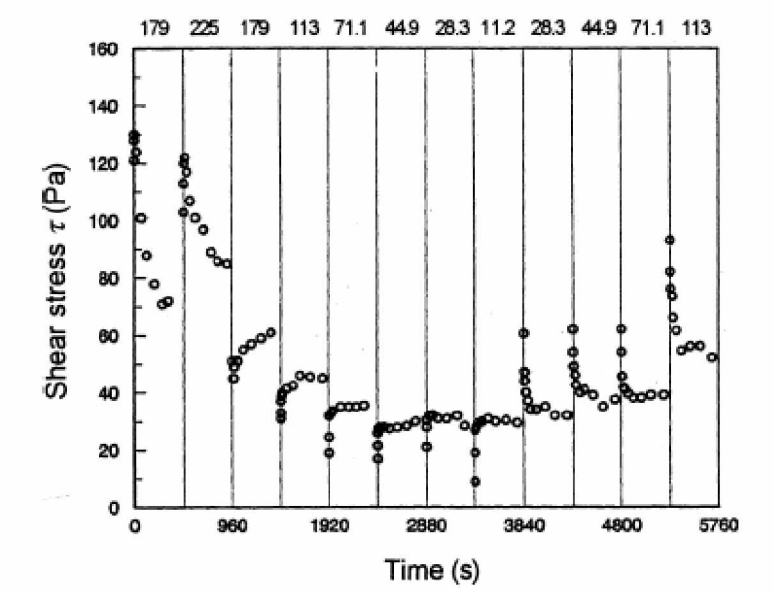

Figure 2.4. Results obtained from shear rate transient experiments on Sn-15Pb alloy with solid fraction of 0.45: along top of figure are shear rate (unit s^{-1}) values that apply to vertical sections beneath [20]

2.2. Thixoforming

Thixoforming is a general term coined to describe the near net shape forming processes from a partially melted non-dendritic alloy slug within a metal die. If the component shaping is performed in a closed die, it is referred to as thixocasting, while if the shaping is achieved in an open die, it is called thixoforging, as schematically illustrated in Figure 2.5 [8]. This distinguished thixoforming from rheocasting which has come to be known as the process used for producing semisolid structures or forming parts from slurry without an intermediate freezing step [14].

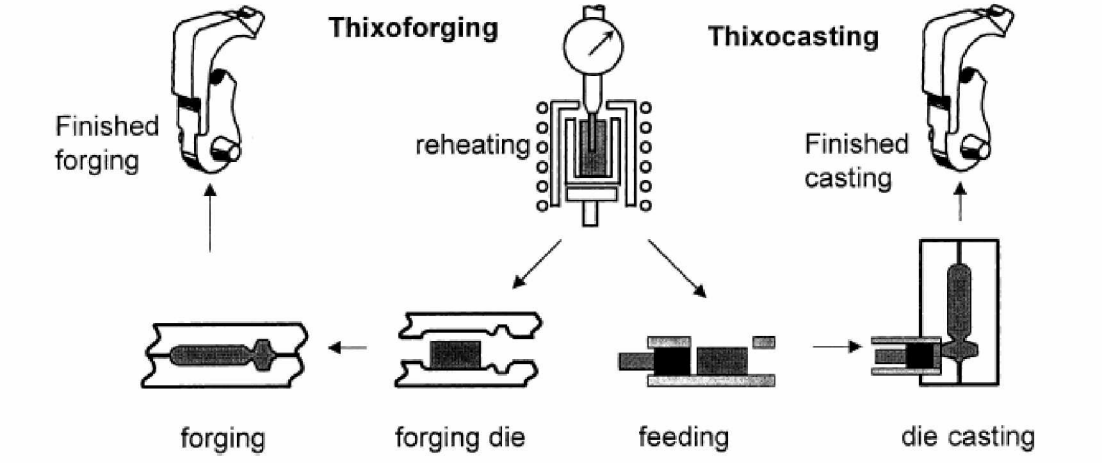

Figure 2.5. Schematic illustration of thixoforming process [8]

There have been several attempts in the United States and abroad to commercialize rheocasting process, but none of these ventures is known to have been commercially successful. On the other hand, thixoforming which exploits the manufacturing advantages of thixotropic semisolid alloy bars began commercial production in 1981. It is a now rapidly expanding commercial process [14]. Thixoforming is now highly automated, with billets being progressively heated under computer control and then transported by robot arms to the forming operation for automatic shaping and subsequent removal from the die. Commercial interest today is primarily in high-integrity aluminum components, especially for the automotive industry [15].

The industrial thixoforming process is subdivided into three independent sections, as schematically shown in Figure 2.6: the raw material production, the reheating and the actual forming process. The aim of the raw material production is to prepare the feedstock in such a way that, after reheating it to the semi-solid processing temperature, a globular solid-phase structure will be obtained. Commonly, the characteristic structure is generated by shearing the liquid alloy during solidification, either mechanically or electromagnetically. Due to shearing forces, the dendritic structures are globularised. Other possible treatments to achieve the desired globular microstructure are the Single Slug Production method (SSP) or chemical grain refinement through crystallization nuclei additions. After total solidification, the structure will be retained during reheating. Reheating takes place immediately before the actual forming process. Since fast

adjustment of a homogeneous temperature field in the billet is required, inductive heaters are usually applied. The actual forming is carried out with modern forging or high-pressure casting machines. Thus, one can distinguish between thixoforging and thixocasting. One of the main advantages of thixoforming is the production of complex-geometry components within a single production step. Because of the small volume shrinkage during solidification, parts can be produced in near-net-shape quality without additional cutting and shaping, thus saving time and energy. Furthermore, in thixoforging, the driving forces are small compared to conventional forging, leading to smaller plant dimensions and an increase in the forging tool durability. Thixocasting guarantees, in comparison to its conventional counterpart, a laminar filling of complex geometries. Remarkable characteristics of thixoformed products are excellent mechanical properties as well as the possibility of thermal treatment. Especially, the automobile industry already produces complex light body components as well as high-pressure-resistant components for fuel injections and braking systems. Alloys already used by thixoforming are mainly aluminum based alloys (e.g., Al-Si-Mg alloys A356, A357). Other materials, like copper-based alloys, metal matrix composites and steels are of industrial interest and are under scientific investigation [21].

2.2.1. Feedstock Production

In thixoforming process, production of appropriate raw material is a key step. These billets are cast from semisolid fluids possessing the rheocast non-dendritic microstructure. The final freezing of these bars captures this microstructure. The bars then represented a raw material that could be heated to at a later time or remote location to the semisolid temperature range to reclaim the special rheological characteristics [14].

The objective of feedstock production is to provide a material with a characteristic thixotropic microstructure where a non-dendritic (or globular) primary phase with a fine grain size is uniformly distributed in a matrix of lower melting point. Although thixotropic feedstock materials in their semisolid state may be directly used for component shaping, they are often used as a raw material in the solid state for subsequent reheating into the semisolid state and component shaping through thixoforming. Thixotropic feedstock production can start either from a liquid alloy through controlled solidification under

specific conditions, or from solid state through heavy plastic deformation and recrystallization. Currently, there is little choice in commercially available feedstock materials, and alloys are usually limited to aluminum based materials, mostly A356 and A357 type cast alloys with 3–6 inch billet diameter, produced by MHD stirring. However, there are a number of other production techniques, which are at different stages of research and development. The basic features of those techniques available for feedstock production are summarized in Section 3 [8].

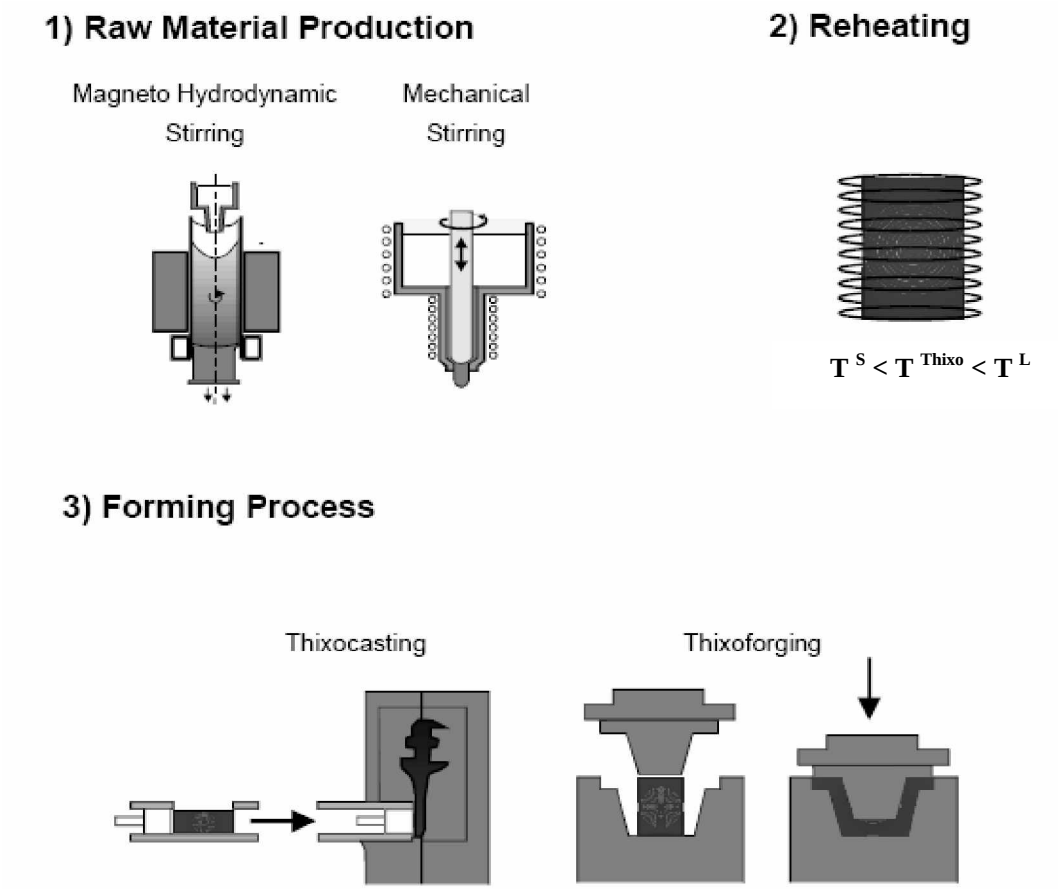

Figure 2.6. Steps of thixoforming process [21]

Alloys Used for Thixoforming: The alloys commonly used for thixoforming are limited to a few casting alloys, only a limited number of trials have been carried out on wrought alloys. If the materials are properly cast and the mould is well designed, the resulting mechanical properties are generally better than those of conventionally cast alloys. However, the mechanical properties offered by thixoformed cast alloys may not be

sufficient for specific applications when higher strength, especially higher fatigue strength, is required. Wrought alloys could be an alternative for such applications. However, wrought alloys are very difficult to cast or shape in the semisolid state owing to the very strong temperature dependence of the liquid volume fraction, lower fluidity compared with silicon containing cast alloys, and problems related to hot tearing. Table 2.2 summarizes the calculated thermal properties of some conventional wrought and cast aluminium alloys, which have been used in the past for thixoforming processing. For the wrought alloys, a small variation in temperature induces a large change in solid fraction. Therefore, a small decrease in temperature can lead to a considerable increase in solid fraction resulting in a microstructure absolutely not favorable to thixoforming. In this case, deformation is inhomogeneous and liquid segregation occurs often during mould filling. On the other hand, when temperature increases, the liquid fraction becomes too high, which makes slug handling very difficult due to the reduced shape stability [8].

Table 2.2. Thermal properties of aluminum alloys [8]

	T, °C (Ref. 184)			T, °C (Scheil model)			T, °C (Equilibrium)			Slope of f_s–T curve	
Alloy/nominal composition	T_L	T_S	ΔT_{S-L}	T_L	T_S	ΔT_{S-L}	T_L	T_S	ΔT_{S-L}	$f_s = 0{\cdot}3$	$f_s = 0{\cdot}6$
Wrought aluminium alloys											
2024/Al–4·4Cu–1·5Mg–0·6Mn	638	502	136	640·6	507·0	133·6	640·6	515·8	124·8	0·0339	0·014
3004/Al–1·2Mn–1·0Mg	654	629	25	653·8	522·0	131·8	653·8	640·8	13·0	0·149	0·084
4032/Al–12·2Si–1·0Mg–0·9Cu–0·9Ni	571	532	39	571·5	519·0	52·5	571·5	535·4	36·1	0·0676	0·0244
5056/Al–5·0Mg–0·1Mn–0·1Cr	638	568	70	635·7	298·0	337·7	635·7	578·8	56·9	0·0335	0·0188
6061/Al–1·0Mg–0·61Si–0·30Cu–0·20Cr	652	582	70	652·1	532	120·1	652·1	590·5	61·6	0·0707	0·0331
7075/Al–5·6Zn–2·5Mg–1·6Cu–0·23Cr	635	477	158	634·9	471·4	163·5	665·4	517·7	147·7	0·031	0·0147
Cast aluminium alloys											
296.0/Al–4·5Cu–2·5Si	635	530	105	632·5	525·1	107·4	632·5	527·8	104·7	0·0192	0·0078
356.0/Al–7Si–0·3Mg	615	555	60	615·6	557·2	58·4	615·6	567·5	48·1	0·0118	0·201
357.0/Al–7Si–0·5Mg	615	555	60	614·9	557·2	57·7	614·9	560·7	54·2	0·0117	0·121
390.0/Al–17·0Si–4·5Cu–0·6Mg	650	505	145	661·4	510·2	151·2	661·4	510·2	151·2	0·06	0·0241
520.0/Al–10Mg	605	450	155	608·7	450·1	158·6	608·7	508·0	100·7	0·016	0·0094
771.0/Al–7Zn–0·9Mg–0·13Cr	645	605	40	644·8	468·2	176·6	644·8	612·8	32·0	0·0586	0·0349

A356 and A357 are well adapted for the thixoforming process. Shape retention is improved by decreasing silicon concentrations with some loss in fluidity and strength. Magnesium additions (0.2 - 1.0 wt per cent can be used to provide product strength through heat treatment. Eutectic modifications where necessary are accomplished with small additions of strontium (~0.02 wt per cent). Since, the 0.2 per cent offset yield strength is reached in the early stage of dislocation motion; yield strength is only moderately affected by minor defects and grain size. This property is primarily a function of alloy composition and heat treatment [22]. The first commercial attempt at alloy

development especially suited for thixoforming was carried out by Pechiney. To optimize the strength and ductility in the T5 condition, an Al–6Si–1Cu–Mg alloy was developed based on the A357 compositions. The properties of Al–6Si–1Cu–Mg alloy after thixoforming match or exceed those of permanent mould cast A356 alloy under T6 conditions. There have also been trials on modified hypereutectic alloys based on A390 alloy. The other approach for alloy design for thixoforming is based on existing wrought alloys for improving processability and maintaining the good combination of mechanical properties. Efforts in this direction have been mainly concentrated on the Al–Mg–Si system with increased magnesium and silicon contents and minor additions of other alloying elements. It was found that such alloys have sufficient thixoformability and good combinations of mechanical properties. In addition, hot cracking was eliminated [8].

Most of the published work concerning semi-solid forming of aluminum is related to the casting alloys A356 and A357. For reheating usually wrought Al-alloys, the different freezing range and solidification characteristics as well as the different thermophysical properties have to be taken into account. For example a billet made from 6082 can be reheated to homogeneous semisolid condition much faster than a billet made from A356 because 6082 allows faster homogenization of the temperature field [23].

2.2.2. Reheating

Slug reheating is a critical stage in the thixoforming process. However, from a fundamental point of view, remelting has been much less frequently studied than feedstock production step in thixoforming. Its purpose is not only to obtain the desirable nominal liquid fraction, but also to ensure transformation of the solid phase to a spheroidal morphology with fine particle size. The driving force for such morphological evolution in the semisolid state is the reduction of the interfacial energy between the solid and liquid phases. To achieve this semisolid microstructure, the important processing parameters during the reheating process include accuracy and uniformity of heating temperature and heating duration. It is the heating temperature that determines the solid fraction in the slug. Too high a heating temperature causes instability of the slug resulting in difficulties for slug handling, while too low a heating temperature leads to unmelted, coalesced, polyhedral silicon phase in the slug in the case of hypoeutectic cast aluminum alloys

having a detrimental effect on the rheological properties during die filling and on the ductility of the finished parts. Furthermore, a uniform temperature distribution throughout the slug is important, because a non-uniform distribution of temperature may lead to fluctuation in solid fraction and rheological characteristics, which in turn may cause solid/liquid separation during mould filling. Finally, the heating duration has to be optimized; too long a heating time will cause structural coarsening, while too short a heating time will lead to incomplete spheroidization of the solid particles compromising the rheological properties and leading to difficulties during mould filling [8].

A variety of methods can be used to reheat the thixotropic slug to the semisolid temperature interval. Selection of the method depends on the alloys involved, the costs of different forms of energy and the levels of heat control required. Furnaces can employ convection or radiant electric or gas heat, electrical induction, or resistance. The time and energy requirements may vary widely depending on the alloy and the size of the slug [14]. Reheating of the billet before forming must be accurate and homogeneous and should be quick to avoid excessive grain growth. Induction heating is to be preferred because of its high heat penetration and good controllability [23]. Induction heating has advantages over conventional radiant-heated furnace heating in the reduction of the amount of scaling and scrap due to the lesser billet heating time [24]. In industry and research laboratories only induction heating units are used to obtain heated parts within the range of temperatures between the solidus and liquidus state with a semi-solid microstructure. Through the use of induction heating it is possible to induce high energy in the part, to obtain heated parts at the right temperature in a short time. Considering the possible output of a thixoforming process, the time for the heating process is also important. Furthermore, the duration of the heating process influences the quality of the microstructure [25].

Another feature of induction heating is its repeatability. For a given material, the power input repeats accurately over the heating cycle. This allows time to be used as the measure of temperature, since the material, heated for a measured time, can be assumed to be at the given temperature with high accuracy. This is useful where there is a constant production rate, using automatic feeding and ejection. Because the initial billet temperature of the forming process is the key parameter to filling results in the semi-solid forming

process, an accurately controllable induction heating method must be selected for the reheating process [24].

During the heating process with induction system, the whole part of the billet has to be heated homogeneously up to the temperature needed for the process. It is important that the induction current, conditioned by the working frequency of the induction heater, heats up only the outer part. This effect is called skin effect. The necessary homogenization of the temperature of the whole part is achieved by heat conduction. Therefore some time is needed in order to reach the same temperature. Even after this time, there is a radial temperature gradient. This gradient can be minimized by optimizing the parameters of the heating process. With regard to the time needed for the induction heating process, it is necessary to have several heating units for one thixoforming press [25].

Controlling the temperature of the billet during heating via induction system is very important because thixoforming is carried out within a small range between the solidus and liquidus temperature. A small variation in the temperature especially for aluminum wrought alloys lead inadequate solid fraction and microstructure for thixoforming. Controlling the temperature with Ni-Cr-Ni thermocouple (K-type) is the most conventional method. However the capability of thermocouples is limited if aluminum wrought alloys are used. This is due to the fact that aluminum wrought alloys only have a very narrow temperature range for processing, which exceeds the accuracy of the commonly used Ni-Cr-Ni thermocouples [26]. An alternative method was developed in order to control the temperature more accurately which is based on the changes of the magnetic and electric properties that occur during the heating process. These changes occur both in the solid and in the semi-liquid state. The unit works with a constant coil voltage. The effective current changes when the temperature changes. This change of the current can be measured with the help of the tests that are being done before series production starts. During these tests, the temperatures as well as the effective current are measured during the heating process. After this, during the series production, it is possible to use the recorded changes of the effective current to indicate the temperature during the heating process and also to indicate when the material is in the semi-solid state. The beginning of the partial melting process is clearly indicated by a considerable current drop [25].

Induction heating is currently implemented in two different ways: vertical and horizontal heating. A vertical heating system has been conventionally used. It suffers from the slug instability problem when the height/diameter ratio is not correctly chosen. The horizontal heating system is a relatively new development, in which the slug lies in a tray and is heated to the optimal processing state monitored by an automatic control loop. Advantages of the horizontal heating system include reduction of the shape stability problem, possibly using higher liquid fractions and alloys with a short freezing range. However, it has a higher system cost and higher space requirement. The relatively low energy efficiency of the induction heating station is a drawback. Possible improvement of energy efficiency can be achieved by preliminary heating to a critical temperature in a convection furnace followed by induction heating for temperature homogenization [8]. For vertical type induction heating, if too much thermal energy and heating time are provided to heat the billet to the required temperature, undesirable phenomena such as liquid segregation, ‘‘the elephant-foot effect’’ by its own weight and an ‘‘electromagnetic end effect’’ may occur. For the horizontal-type induction coil, the ‘‘elephant foot effect’’ by its own weight seldom appears and outflow of the liquid decreases remarkably [27].

Structural Evolution During Partial Remelting and Isothermal Holding:. During reheating, microstructural evolution is a diffusion controlled process. On the one hand, the holding time should be long enough to complete the morphological transition from dendritic (or rosette) to spherical, but, on the other hand, the holding time should be short enough to prevent excessive grain growth, which is detrimental to the mechanical properties of thixoformed parts. So the reheating process needs to be optimized to achieve the most desirable slurry characteristics for thixoforming. Understanding the structural evolution would be a critical step towards such optimization. In contrast to solidification from the liquid state, the variety of solid state structures that can be encountered during remelting give rise to a rich range of melting behavior. However, the microstructural evolution during remelting and subsequent holding before thixoforming has received only limited attention. In the section above, the information available about structural evolution during partial remelting and isothermal holding in the literature is summarized [8].

The kinetics of structural evolution during isothermal holding in the semisolid state has been subjected to a number of studies. Sannes et al. [28] investigated the structural evolution of semisolid ZE33 magnesium alloy at different solid fractions. They found that

the coarsening rate increases with decreasing solid fraction. Based on the experimental results, the authors proposed that at high solid fraction, growth by coalescence ripening makes a major contribution to the total microstructural coarsening in the semisolid state, while at low solid fraction; Ostwald ripening is the dominant mechanism for structural coarsening. Loue and Suery [29] studied the influence of thermomechanical history on the microstructural evolution of A357 alloy during partial remelting and isothermal holding. They found that during the coarsening process, particle density for the initially globular structures decreases with increasing isothermal holding time, while that for initially dendritic structures remains fairly constant, as shown in Figure 2.7. They also found that longer solidification time and smaller initial grain size accelerate the coarsening kinetics.

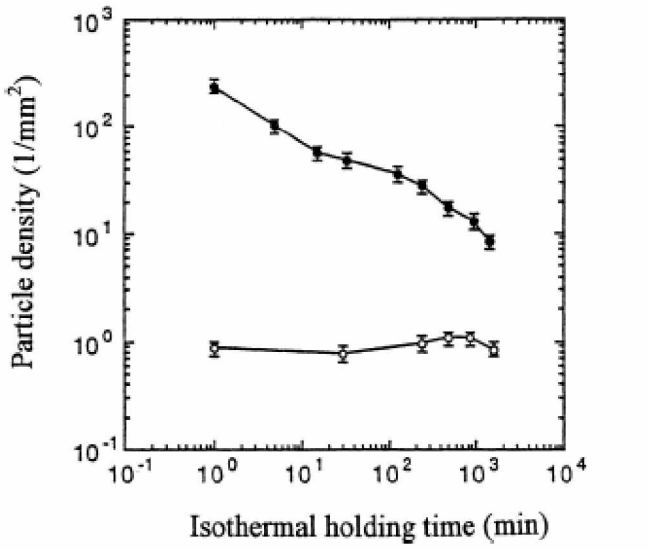

Figure 2.7. Particle density N_A as function of isothermal holding time during partial remelting at 580 °C (f_s=0.45) of conventional, initially globular (○) and MHD stirred, initially dendritic (●) Al–7Si–0.6Mg alloy [29]

The effects of initial particle morphology on the coarsening process were investigated by Blais et al [30]. During isothermal holding in the semisolid state, regardless of the initial particle morphology, the solid phase always evolves towards a spherical morphology. However, the kinetics of such evolution is governed by the initial microstructure; as shown in Figure 2.8, the larger the initial shape factor Fg, the faster the kinetics for spheroidisation, and the longer the holding time required to obtain spherical

particles [30]. The kinetics of coarsening of a MHD stirred A357 alloy follows the prediction of Ostwald ripening (Figure 2.9) [31].

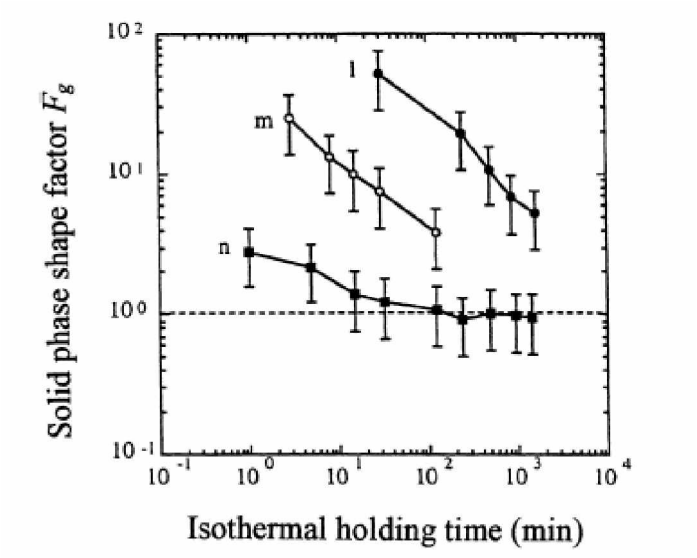

Figure 2.8. Particle shape factor F_g as function of holding time at 580˚C (f_s=0.5) for Al-Si–0.6Mg alloy obtained by classical continuous casting, without (l) and with (m) grain refining, and by MHD stirring (n) [30]

Another phenomenon observed during microstructural coarsening is the entrapped liquid inside the solid particles. Based on the available experimental results, it appears that both Ostwald ripening and particle coalescence contribute to the process of entrapping liquid, but with opposite effects. On the one hand, Ostwald ripening leads to a loss of entrapped liquid, since the small particles with entrapped liquid will dissolve and the entrapped liquid will join the bulk liquid. On the other hand, coalescence of complex shaped particles results in liquid entrapment. In the latter case, the liquid entrapped in the solid particle will remain entrapped. This makes the evolution of entrapped liquid rather complex, especially during short holding time [32].

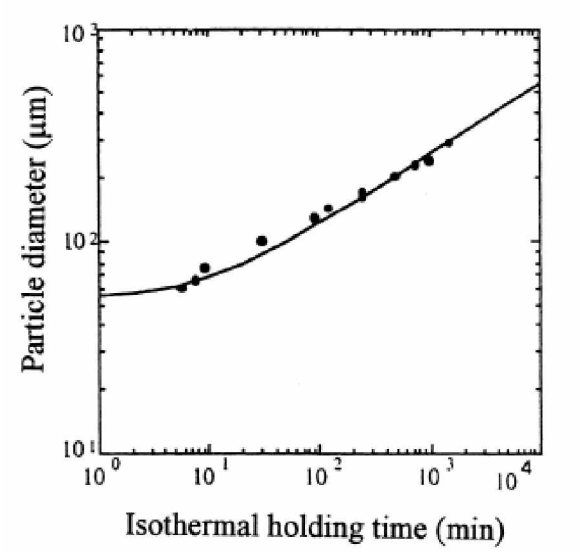

Figure 2.9. Average particle size as function of holding time at 580˚C (f_s=0.5) for Al–7Si–0.6Mg alloy obtained by MHD stirring [31]

Plastic deformation before reheating has been found to be an important factor as regards the coarsening kinetics. Loue and Suery [29] found that cold working before partial remelting of A356 alloy allows the most rapid globularization of the solid phase once the threshold for crystallization is surpassed.

Tzimas and Zavaliangos [33] compared the reheating behavior of three different initial microstructures obtained by MHD stirring, the SIMA (stress induced and melt activated process), and spray casting. They concluded that at medium liquid fraction, the microstructure of spray cast and SIMA alloys consists of discrete equiaxed grains uniformly dispersed in the liquid matrix, while the corresponding microstructure of MHD alloys exhibits extensive agglomerates consisting of incompletely spheroidized grains. Their results also demonstrated that the reheated MHD microstructures are less equiaxed compared with SIMA and spray cast alloys even after 5 min soaking in the semisolid state.

The kinetics of semisolid thermal transformation of initially dendritic structure has been investigated by a number of workers. On increasing the temperature, partial remelting was found to start at grain boundaries, followed by an apparent decrease in the proportion of eutectic phase. During partial remelting and the subsequent isothermal holding in the semisolid state, coarsening first proceeds predominately through coalescence of dendritic

arms. As the dendritic arms of the same cell have a perfectly matching crystallographic orientation, coalescence of the dendrite arms is supposed to be extremely rapid. Such a mechanism of rapid coalescence causes substantial entrapment of the liquid phase in the interdendritic region, while in the case of electromagnetically stirred alloys the coalescence of short dendritic arms leads to a more spherical morphology. After this rapid coalescence stage, the much slower coarsening by diffusion of solid atoms from areas of high curvature to areas of low curvature can take place. As long as the particles are not spherical, coarsening will lead to an increase in the mean free path in the solid phase, but the number of particles remains constant until eventually the solid phase becomes spherical. Further coarsening will then take place through dissolution of the small globules and the particle density will then decrease [8].

It can be summarized that the microstructural evolution during reheating and subsequent isothermal holding is characterized by the following processes [8]:

- Partial remelting of the low melting point phase starting at grain boundaries.
- Rapid coalescence of dendritic arms resulting in liquid entrapment.
- Spheroidization of individual particles due to atomic flux from the areas of high curvature to the areas of low curvature.
- Coarsening by both dissolution of smaller particles and coalescence between particles, resulting in a decrease in particle density.

2.2.3. Forming

The process of forming a partially melted non-dendritic alloy slug into a near net shape component within metal dies has been termed thixocasting, thixoforging, or more generally, thixoforming. Thixocasting usually refers to the operation of injecting the slug into the die by a ram or plunger as in die casting, and in fact the early work on SSM employed die casting machines and dies. Placing the slug within open dies and squeezing the two halves together is often referred to as thixoforging. However, the deformation and flow of the semisolid alloy within the die is different from that of pure liquid or pure solid in either process, and the term thixoforming is generally preferred [5]

There are two separate stages involved in thixoforming a slug of the appropriate non-dendritic structure into a shaped component within a die. The first is the uniform heating and partial melting of the alloy slug which is carried out with induction heating in order to avoid steep temperature gradients. When the slug is known to be in the correct softened condition, either by adopting a heating program established experimentally, or by temperature measurement or some other non-contacting device monitoring softness, the slug may be transferred to the die shot chamber by robot handling where it is then injected into the die by a hydraulic ram. Alternatively, the induction heating may be carried out with the slug seated on a pedestal attached to the ram and injected directly into the die. This technique avoids handling of the semisolid slug and delay involved, and permits both heating and injection to be carried out within a vacuum or controlled atmosphere chamber, this has obvious advantages when dealing with easily oxidized alloys at high temperatures [5]. During the experiments performed by Kang et al. [34], 7-8 seconds delay in feeding of reheated billet from induction furnace to the dies lead increase in solid fraction before applied pressure which reduced mechanical properties of the thixoforged part.

In thixoforming, the material flow begins with the impact of the ram on the slug and the thixotropic nature of the slug allows the metal to flow into the cavity at very low pressures. Only at the end of the forming stroke, the pressure increases to the selected level to form fully dense component. The time of containment under pressure depends on alloy and part dimensions and is normally only a few seconds in duration because heat transfer to the die is extremely efficient at the high pressure. At the completion of the forming cycle, dies are opened and the part is ejected [14]. Figure 2.10 shows typical thixoforming arrangements. The slug is generally heated in an induction heater until it reaches the required semi-solid state and then forced into the die by a ram. Slugs are usually orientated vertically during heating but there are some horizontal arrangements where the slug is placed in a container to catch any escaping liquid. Horizontal heating arrangements tend to operate with liquid fractions around 0.6, vertical with 0.4 [35].

At present, thixocasting through horizontal cold chamber die casting is the dominant process. A robot arm transfers the semisolid slug into the shot chamber and the plunger injects the materials into the die cavity. All the thixocasting machines are real-time controlled and thus permit a reaction to possible fluctuation during the forming process. At

this stage, smooth laminar mould filling is the crucial step for the forming process. This can be achieved by an optimised shot profile tailored for specific alloys and their physical conditions [8]. In the alternative process, whereby the semisolid slug is forged between two die halves (thixoforging), similar conditions will hold, but it is more difficult to predict and control the flowrates of the slurry in different regions of the closing dies [5].

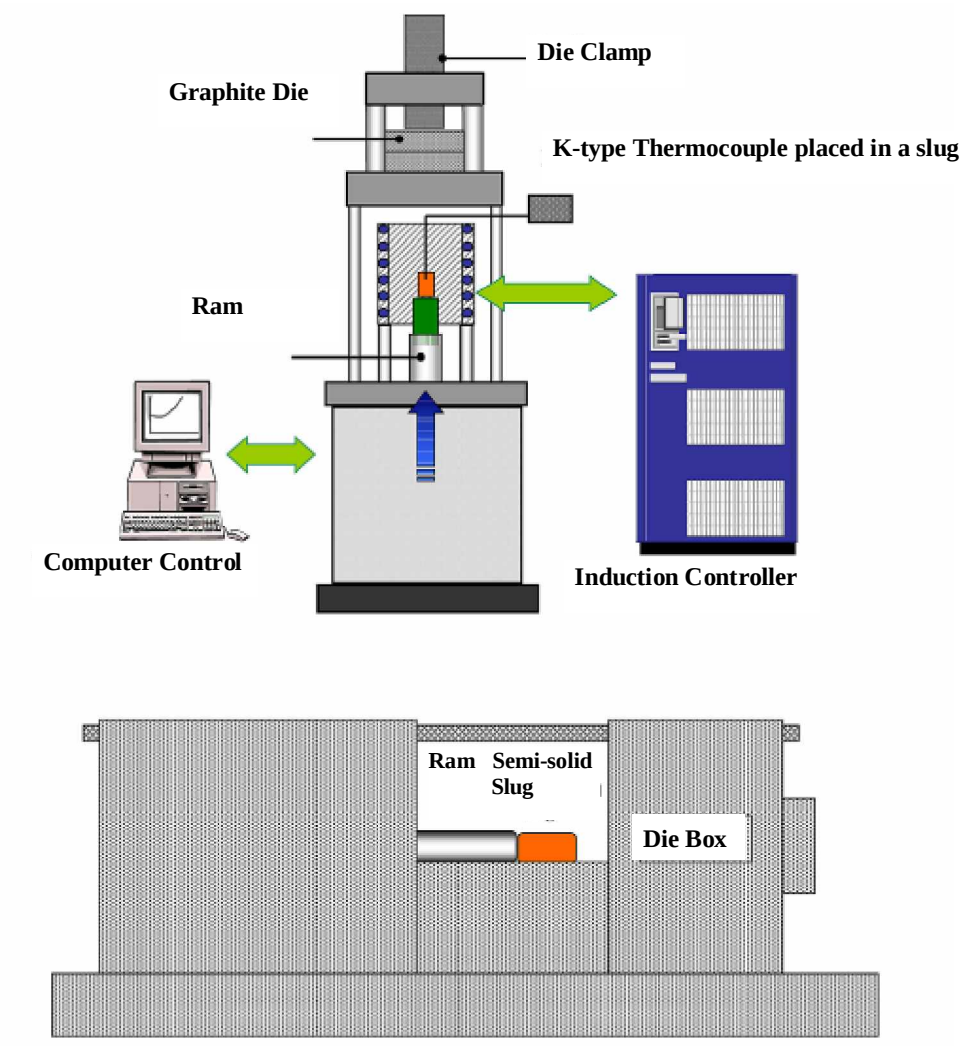

Figure 2.10. Top: computer controlled hydraulic vertical thixoforming press and bottom: typical commercial hydraulic horizontal thixoforming press [35]

In thixoforging process, forging presses may vary, but the ability to control the forming speed and the pressure precisely is essential if the press is to be used to forge a variety of parts. Depending on part size, geometry, alloy and quality specified, forming

speeds may range from a few millimeters per second to 1270 mm/s and mold pressure from a few hundred kilograms per square centimeter to 140 MPa or more [14]. Because of the shear-rate dependent viscosity of the metal, the thixoforging technique requires extremely low punch forces at the beginning of the forging process. Only towards the end of the process is a higher punch force needed, because the material cools down to temperature which is closer to the solidus temperature and because of the surface of the work piece gets larger. The punch force of the traditional forging process, however, increases during the whole punch stroke as shown in Figure 2.11. After the cavity has been filled, the press works with a constant force over a certain period of time during which the workpiece has to solidify completely. The punch velocity should be as high as possible in order to close the dies as fast as possible. This is necessary because the differences in temperature of the material and die cause a quick cooling of the workpiece. The stroke of the punch should be as short as possible because the cycle time should also be kept short. The punch velocities should be optimized to fill the cavity with a laminar flow of the semi-solid material. If there are large variations in the cross sections of a workpiece, the punch velocity will have to be adjusted during the filling process. After the cavity has been filled, the material of the workpiece should solidify completely under a pressure load of approximately 1000 bar. This pressure is needed to ensure a microstructure without inner porosities [25]. Cho and Kang [36] observed the filling behavior of thixoforged products and they investigated effect of pressure on microstructure and mechanical properties of these products. They carried out experiments with ram speed of 200 mm/s and applied pressure of 100 and 150MPa. The results showed that the higher applied pressure, the denser microstructure and the higher mechanical properties.

Another important aspect during the forming process concerns the design of the gating system and die cavity and the correct choice of die temperature. Such a design process has to consider the flow characteristics of the semisolid metals. The forming process can be optimized through process simulations using various computer modeling techniques [8]. When simulating the die filling, the moving upper die as well as the thixotropic properties of the work material has to be considered. The work material is described by a shear rate and shear time dependent viscosity. The die filling of a simple part, calculated with different viscosity parameters is, compared with the experimental results. Within these simulations, the forming forces were calculated and compared with

the experimentally measured forces. The simulation is verified with these quantitative data. With a proper choice of the viscosity parameters it is possible to fit the results of the simulation to the reality [37]. Kapranos et al. [38] studied a process for producing and assessing a high-quality thixoformed component, using a standard aluminum die casting alloy, forced into a non-hardened steel die. They showed that die designs based on conventional pressure die casting practice are inadequate. In particular, the higher viscosities of the metal slurries used in thixoforming require modified runners and gates. The design of these can be aided greatly by CFD modeling and this tool will be used more and more in all aspects of die design for thixoforming.

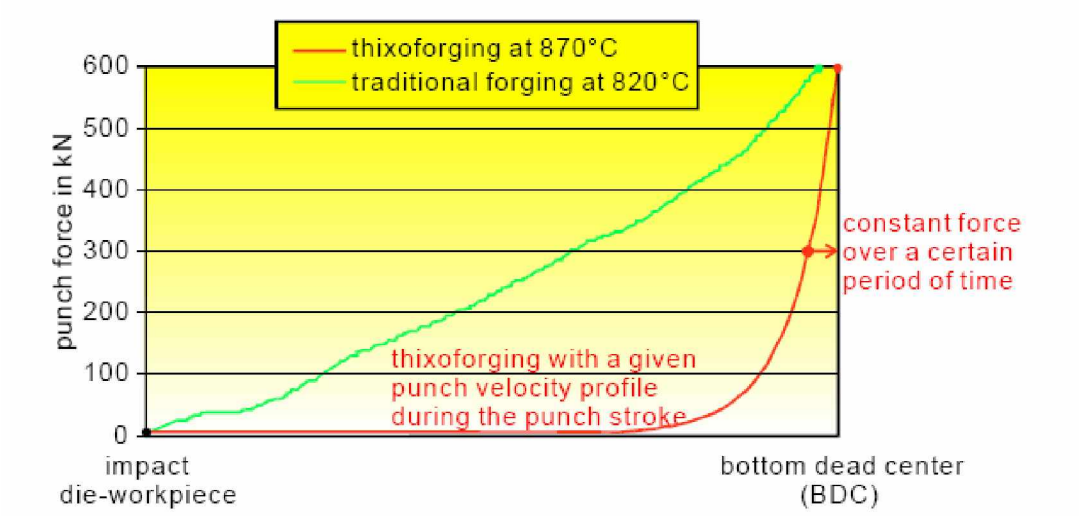

Figure 2.11. Punch-force punch-stroke diagram of a traditional forging process and thixoforging process [25]

The practice in thixoforming of aluminum and copper alloys has been to use conventional die steels (H-13, H-21) and die lubricants to prevent sticking [5]. Dies have hardness of 45 to 48 HRC. Die cavities are ground and electric discharge machined; tolerances and shrink rule are approximately the same as for die casting. Polishing is frequently advised to improve metal flow, to ease part ejection and to optimize surface quality [14]. Generally the dies are preheated to about 250˚C-300˚C, so the rate of heat transfer from the aluminum to the die will be reduced, allowing the semi-solid metal to flow a longer distance before the critical solid fraction preventing flow is reached [39].

3. FEEDSTOCK PRODUCTION FOR THIXOFORMING

3.1. Properties of Feedstock for Thixoforming

Forming in the semisolid state requires that a metal or alloy have a roughly spherical and fine-grain microstructure uniformly dispersed in a liquid matrix when it enters the forming die [14]. This final structure is suitable for semi-solid processing. In this case, the structure that presents minor grain size, minor shape factor (roundness), and the most homogeneous and globular size of the primary phase has the best behavior in semi-solid forming as well as the best mechanical final properties [40]. A primary goal of slurry preparation is to create such a structure to ensure the favorable rheological characteristics to facilitate the subsequent component shaping process. Semisolid metal slurries exhibit distinctive rheological characteristics: the steady state behavior is pseudoplastic (or shear thinning), while the transient state behavior is thixotropic [8]. For example, mascara, honey and certain kinds of paint are all thixotropic. When they are sheared they flow, when allowed to stand they thicken up again; their viscosity is shear rate and time dependent. Such behavior in SSM alloys was firstly discovered in the early 1970s during the investigation of hot tearing with a rheometer. If the material was stirred continuously during cooling from the fully liquid state to the semisolid state the viscosity was significantly lower than if the material was cooled into the semisolid state without stirring. Stirring breaks up the dendrites which would normally be present so that the microstructure in the semisolid state consists of spheroids of solid surrounded by liquid (Figure 3.1). It is this microstructure which is a requirement for thixotropic behavior and for semisolid processing. When such a semisolid microstructure is allowed to stand, the spheroids agglomerate and the viscosity increases with time. If the material is sheared, the agglomerates are broken up and the viscosity falls. In the semisolid state, with between 30 per cent and 50 per cent liquid, if the alloy is allowed to stand it will support its own weight and can be handled like a solid. As soon as it is sheared, it flows with a viscosity similar to that of heavy machine oil [9].

The alloys used as a feedstock material for thixoforming should owe some basic properties. First of all, solidification range is an important factor and needs to be optimum.

On the one hand, too narrow solidification ranges leads poor castability. On the other hand, too wide a solidification range could lead to poor resistance to hot tearing and poor fluidity of the liquid alloy. The temperature sensitivity of solid fraction is another important criterion for feedstock materials. The feedstock material's temperature sensitivity of solid fraction should be as small as possible. In addition, to take full advantage of the fact that thixoformed components are heat treatable, alloys for thixoforming need to have large ΔC which is defined as the solid solubility difference between semisolid temperature and aging temperature. Ideal slurry for thixoforming has a controlled volume fraction of fine and spherical solid particles distributed uniformly in a liquid matrix with good fluidity. Such slurry can ensure smooth mould filling and fine and uniform microstructure after solidification. However, the ease with which the fine and spherical particles can form during thixoforming depends on the actual alloy composition. Lastly, the characteristic rheological properties of SSM slurries makes thixoforming unique and advantageous compared with other conventional casting techniques. The processing condition, and the morphology, size, and distribution of the solid phase in the liquid matrix, all have great influence on the viscosity of SSM slurries. The variation in viscosity, on the other hand, will influence the castability. In addition, particle agglomeration strongly affects the rheology of SSM slurries. A reduced tendency for particle agglomeration will facilitate thixoforming and can be achieved through addition of alloying elements [8].

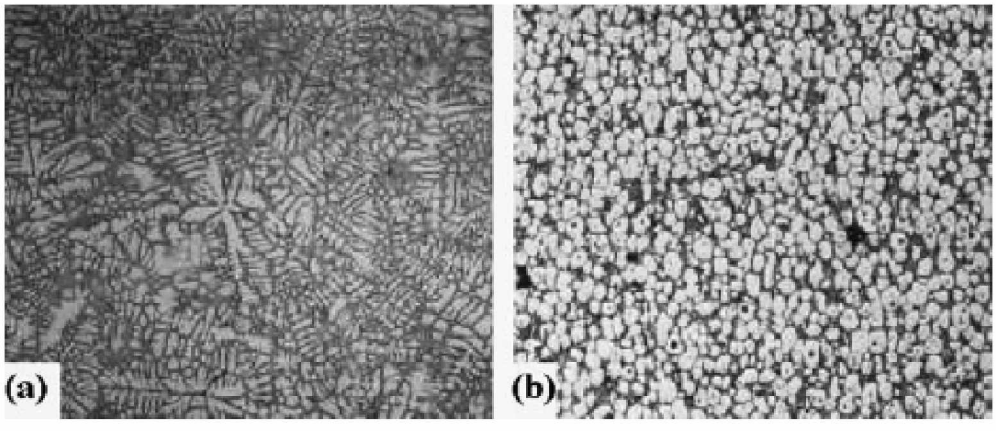

Figure 3.1. Micrograph of a typical a) dendritic microstructure in an as-cast sample and b) globular microstructure in a semisolid alloy sample [9]

3.2. Microstructural Evolution During Feedstock Production

3.2.1. Microstructural Characterization

For a fixed alloy composition, complete description of the microstructure of semisolid slurry involves quantifying the volume fraction, size, shape, and distribution of the solid particles. This is normally done with a solid material quenched from the semisolid temperature. So far, nearly all the quantifications have been done on a two-dimensional (2D) polished surface using conventional metallographic techniques [8].

The behavior of a semisolid material is very sensitive to the volume fraction of solid. A large volume fraction of solid results in increased apparent viscosity and significant internal damage during deformation, as a result of strain localization which in turn causes difficulties in die filling and defects in the final product. Although no specific limits have been explicitly suggested in the literature, it is generally accepted that the volume fraction of solid during processing should be at most 0.6 [41]. Accurate knowledge of solid fraction in semisolid slurry is critical for both scientific understanding and effective process control. For a given alloy, solid fraction can only be defined uniquely at a given temperature under equilibrium conditions. In any other case, it depends on the prior thermal history. Practically, utilization of thermodynamic data (equilibrium phase diagram), thermal analysis techniques and quantitative metallography on microstructures are three effective methods for the evaluation of solid fraction. Although these three methods are all approximate, each one has its own significance and unique advantages: the use of thermodynamic data is a fast tool for alloy design, thermal analysis provides one with comparable information and reveals the prior thermal history of an alloy, and quantitative microscopy after quenching reveals more microstructural information, such as morphology and distribution of the solid phase in the semisolid state [8].

During feedstock production in thixoforming, it is important to control whether the microstructure is composed of adequate spherical grains. In order to quantify the particle morphology, an object-specific shape factor F is usually used. Two different expressions for the shape factor F has been used in literature:

$$F_1 = \frac{4\Pi A}{P^2} \tag{3.1}$$

$$F_2 = \frac{P^2}{4\Pi A} \tag{3.2}$$

where A and P represent the area and perimeter of the object, respectively. In both cases, F=1 refers to a perfectly spherical morphology, while for very complex shapes, $F_1 \rightarrow 0$ and $F_2 \rightarrow \infty$.

For a given slurry system with a fixed solid fraction, particle distribution in the liquid matrix has a pronounced influence on the slurry rheology and has a strong implication regarding the quality of the SSM processed components. Despite such importance, currently there is no mature method for the quantification of particle distribution in the liquid matrix. To model the deformation behavior of SSM slurries, a structural parameter s has often been used to describe the particle distribution. In a completely agglomerated state s=1, while for a completely dispersed state s=0. Thus, $0 \leq s \leq 1$. However, this parameter is not well defined and is extremely difficult to measure experimentally. More recently, it was proposed that using the average number of particles in agglomerates as a parameter to describe the degree of particle agglomeration in SSM slurry. Obviously, this parameter can be determined experimentally using a standard metallographic method. The concerned parameter has been successfully used for the development of a microstructural model for SSM slurries. It was demonstrated that the viscosity of specified SSM slurry has a one-to-one correspondence to the average number of particles in agglomerates [8].

3.2.2. Nucleation and Formation of Equiaxed Grains

During dendritic solidification of alloys, a number of processes take place simultaneously within semisolid region. These include crystallization, solute redistribution, ripening, interdendritic fluid flow and solid movement. The dendritic structure which forms is greatly affected by convection during the early stages of solidification. In the limit of vigorous convection and slow cooling, grains become spheroidal. Alloys with this

microstructure posses rheological properties in the semisolid state which are quite different from those of dendritic alloys [15].

Nucleation and growth are the two main mechanisms during solidification. Homogeneous and heterogeneous nucleations are two ways in which nuclei can form. Once nucleation has occurred, solidification proceeds by the movement of an interface. The process may generate heat if the enthalpy of the solid is less than that of the liquid. Similarly, solute may partition into the liquid if its solubility in the solid is less than that in the liquid. The accumulation of solute and heat ahead of the interface can lead to circumstances in which the liquid in front of the solidification front is supercooled. The interface thus becomes unstable and in appropriate circumstances solidification becomes dendritic. The driving force of dendritic growth is proportional to the constitutional supercooling. In general, solidification occurs by heterogeneous nucleation, either on impurity particles or wherever the liquid comes into contact with the container surface [42]. Dendritic growth occurs by three separate processes: (a) The initial propagation of the primary dendrite stem, which forms driving by the large constitutional supercooling (b) evolution of dendrite branches, and (c) coarsening and coalescence of the dendrite stem and arms. Depending on the above conditions, the microstructure in the semisolid state can have columnar dendritic, equiaxed dendritic, rosette or spheroidal morphology.

A number of theories have been developed to explain the origins of equiaxed crystals during solidification. The first of these is the constitutional supercooling theory which is based on the idea that equiaxed crystals nucleate in the liquid ahead of the moving solid/liquid interface. Owing to solid segregation, this liquid is constitutionally undercooled, and growth of the columnar region is restricted by diffusion ahead of the interface. The possibility thus arises for undercooling in the liquid ahead of the solid/liquid interface to be sufficient to allow nucleation on any suitable substrates present in the melt. The second theory is the free chill crystal theory. This proposes that, on pouring, the equiaxed zone is formed by free chill crystals that nucleate in the thermally undercooled region adjacent to the cold mold. These equiaxed crystals are carried into the bulk liquid by convection and turbulence. Rejection of solute from the growth of solid material nucleated on the wall (rather than in the adjacent liquid) produces a region of constitutionally undercooled liquid. The undercooled zone preserves those crystals which would otherwise

have remelted. It extends further into the liquid for lower pouring temperatures and higher solute contents, increasing the number of free crystals that are able to survive and grow until impingement. One of the most widely accepted theories is the dendrite arm remelting theory. Based on observations of growth in transparent organic analogues, it was proposed that secondary arms of columnar dendrites form a necked shape because of solute buildup at the base of the arm. Diffusion of solute into the bulk liquid is restricted by the presence of neighboring dendrite arms. This decreases the local equilibrium freezing temperature and growth occurs preferentially in the purer liquid near the tips of the arms where solute diffusion is relatively unrestricted. Necks are comparatively rich in solute, and those that grow in thermally undercooled regions can be remelted if latent heat conducted through the solid raises the local temperature above the liquidus temperature. Another theory proposed that a coarse layer, formed from grains that nucleate independently of the mould wall can exist at the surface of the ingot exposed to the atmosphere. Equiaxed grains are formed when dendrites from this surface layer fracture and detach during the formation of the shrinkage pipe. Fragments sink into the liquid until they come into contact with columnar crystals growing in from the mould wall. They are then able to grow under the same conditions as the columnar zone with heat being transferred through the solid in the direction of heat extraction. The fifth mechanism for equiaxed grain formation is the separation theory which suggests that crystals nucleate on the mould wall as a result of thermal undercooling immediately after pouring but before the formation of a stable solid skin. The crystals grow out from the wall in a necked shape, owing to solute segregation, and are subsequently detached through mechanical break-age and/or thermal fluctuations in the liquid. Convection and turbulence play important roles in both crystal detachment and transportation in the bulk liquid. Survival of crystals in any of these five theories is dependent upon local thermal conditions and degree of constitutional undercooling in the liquid. All mechanisms are possible, but whether a particular mechanism becomes operative depends on the alloy composition, casting conditions and the type of nucleating substrates present in the melt [43].

In general, as-cast metal exhibits three distinct zones of grain structures: chill zone, columnar zone and equiaxed zone. Columnar to equiaxed zone transition is found to be affected by such factors as superheat, alloy system, composition, fluid flow, mechanical disturbance, inoculation, addition of grain refiner and casting size [44]. Vibration has been

shown to influence formation of new grains. An interpretation favored by many some decades ago that the vibration promoted heterogeneous or even homogeneous nucleation. Equiaxed grains are favored over columnar grains by low pouring temperature. As pouring temperature is lowered further, the equiaxed grains become finer. More important from a practical standpoint, introducing convection by mechanical or electromagnetic means during the early stages of solidification favors formation of fine, equiaxed grains. The early growth is dendritic, but with continuous shear and time during solidification, the dendrite morphology becomes that of a rosette, because of ripening, shear and abrasion with other grains. With sufficiently slow cooling and high shear, the grains become spheroidal, usually with a small amount of entrapped liquid. Figure 3.2 shows schematically the evolution of the morphology of the primary phase during the rheocasting process. There is a general belief today that, vibration, low pouring temperature and externally induced convection all promote grain refinement primarily by a dendrite fragmentation mechanism. Some possible dendrite fragmentation mechanisms are: (a) dendrite arm fracture, in which arms shear off as a result of the force on the arm from the fluid flow; (b) remelting of the arm at its root because of normal ripening; (c) remelting as above, enhanced by thermal perturbation, which results from turbulent convection; (d) remelting as above, but where the melting at the root is accelerated by the stress introduced at the dendrite root because of the force of fluid flow; (e) as in (c), but where the melting at the root is further enhanced by a high solute content in the solid at the dendrite root; (f) recrystallization as a result of the stress introduced by the force of the fluid flow, with rapid liquid penetration along the new grain boundaries [15].

3.2.3. Solidification Behavior Under Forced Convection

As it was concluded in the previous section, stirring or forced convection leads to production of equiaxed morphologies. The early work by Spencer et al. [17] and Joly and Mehrabian [18] on the Sn–Pb system using rotational rheometers confirmed that the solid phase in the semisolid state has either a degenerated dendritic structure or rosette morphology. With prolonged stirring time, such particles change to a more or less spherical morphology containing entrapped liquid by a ripening process. Increasing the shear rate accelerates this morphological transition and reduces the amount of entrapped liquid inside the solid particles [8].

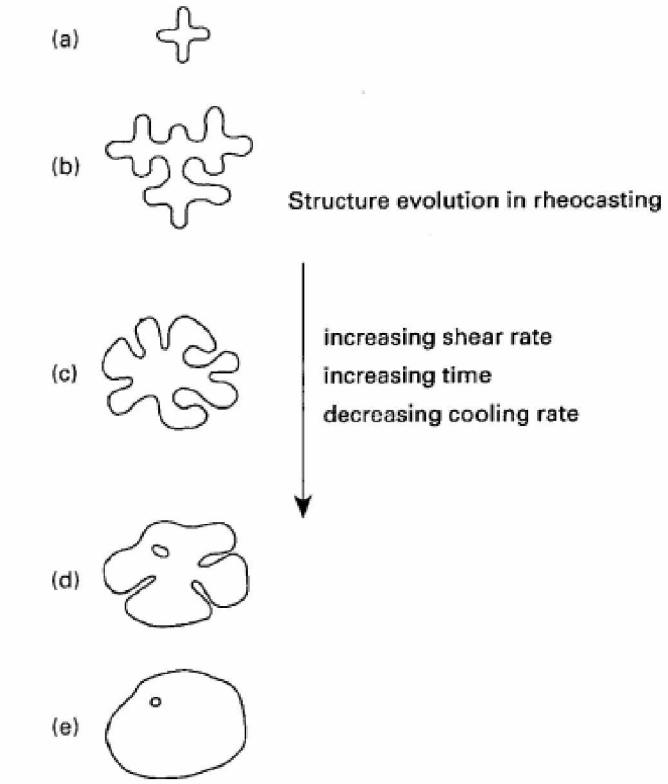

Figure 3.2. Schematic illustration of evolution of structure during solidification with vigorous agitation: (a) initial dendritic fragment; (b) dendritic growth; (c) rosette; (d) ripened rosette; (e) spheroid [15]

For Al–Cu alloys, Vogel et al. [45] observed that with applied shear the primary particles grow as rosettes until a certain limit beyond which further growth does not occur but subsequent solidification takes place by formation of more (new) particles. Molenaar et al. [46] observed rosette type and radially grown cellular particles in intermediate and fast cooled and sheared Al–Cu alloys. At slow cooling rate, rosettes were observed. Similar to the observation by Vogel et al. [45], stirring speed was not found to influence the particle density or size significantly. The cell spacing was considerably greater than the secondary dendrite arm spacing from unstirred melt, indicating that stirring promotes crystal growth. Smith et al. [47] studied the microstructural evolution during solidification of a stirred Al–19Si alloy. They found that with increasing shear rate the average particle diameter decreases, while the particle density increases. It should be mentioned that in all those investigations on melt stirring the flow conditions are generally characterised by largely

laminar flow with relatively low shear rate achieved by using rod or simple impeller as stirrer [8].

Ji and Fan [48] studied the effect of turbulent flow on the solidification morphology of Sn–15Pb alloy using a laboratory scale twin screw rheomoulding (TSRM) machine developed recently. They found that under intensive turbulent flow, the solidification morphology is spherical even at the very early stage of solidification. Their isothermal shearing experiments showed that the size, shape factor, and density of the solid particles are almost constant with increasing isothermal shearing time (Figure 3.3). This is in good agreement with the early work by Ryoo and Kim [49] on Mg–Al–Zn–Si alloys. The particle size distribution was found to be very close to that of randomly dispersed monospheres. They also found that in the low shear rate region, increasing shear rate increases particle density and decreases particle size, while in the high shear rate region both particle size and density reach a plateau solid/liquid interface. Following this work, Das et al. [50] have recently conducted a systematic study on the growth morphology under various fluid flow conditions using different stirring devices. Cylindrical rod with low rotation speed creates essentially simple laminar flow; propellers induce predominantly laminar flow with limited degree of turbulence, while a TSRM machine produces basically turbulent flow [8].

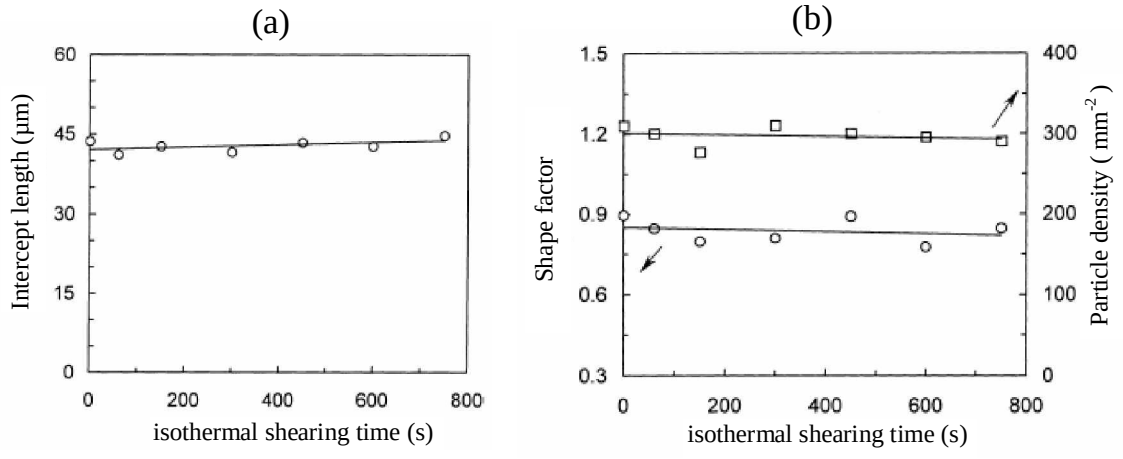

Figure 3.3. Effect of isothermal shearing time on intercept length on a) intercept length, b) shape factor and particle density of primary particles of Sn-15Pb alloy processed in TSRM machine [48]

The experimental findings so far on solidification under forced convection can be summarized as follows [8]:

- Forced convection promotes finer particles with a non-dendritic morphology.
- Forced convection accelerates crystal growth.
- Turbulent flow influences much more significantly the particle size and morphology than does laminar flow.
- With increasing shear rate and intensity of turbulence the particle morphology changes from dendritic to spherical via rosette.

3.3. Feedstock Production Methods

The ideal microstructure for a thixotropic feedstock before the component shaping process would be an accurately specified volume fraction of fine and spherical solid particles uniformly dispersed in a liquid matrix [8]. There are many different routes for obtaining such a microstructure as described in the following sections.

3.3.1. Mechanical Stirring

The original MIT research showed that a very effective method of producing the semisolid metalworking microstructure was to mechanically agitate an alloy vigorously during the solidification process [14]. The vigorous agitation of metal alloys during solidification leads to formation of spheroidal solid particles suspended in the liquid matrix through a process of dendrite-arm detachment and coarsening [51]. Melt agitation is commonly generated by means of augers, impellers, or multipaddle agitators mounted on a central rotating shaft. Shear rate can be roughly estimated by the ratio of the velocity of the impeller extremity to the clearance between the impeller tip and the mould wall. The shear offered by the stirrer during solidification promotes the formation of non-dendritic structure rosettes [8].

The mechanical stirring approach was developed from a batch process in which a crucible of molten liquid is mechanically mixed while cooling. It has been incorporated in vacuum or inert atmosphere chambers for use with high melting point metals or to reduce

air entrapment [15]. Continuous rheocaster process was improvement of batch process, as illustrated in Figure 3.4. In the continuous process, superheated liquid in the holding vessel flows down into an annulus between the stirring rod and the outer cylinder where it is simultaneously stirred and cooled. Slurry flows from the bottom of the rheocaster either to be cast directly to shape (rheocasting), or to be solidified as feedstock material for subsequent reheating and thixoforming. The resulting solid particles are usually coarse rosettes [8]. High solidification rates produced finer particle diameters, and high shear rates produced more rounded particle shapes with less clustering particles [14]. Efforts have been made to scale up the continuous rheocaster to industrial production level but apparently without success. The reason for this are the unacceptable erosion of the ceramic stirrer (particularly with high melting point alloys), the contamination of the slurry by dross and gas entrapment, low productivity and the difficulty in process control [4].

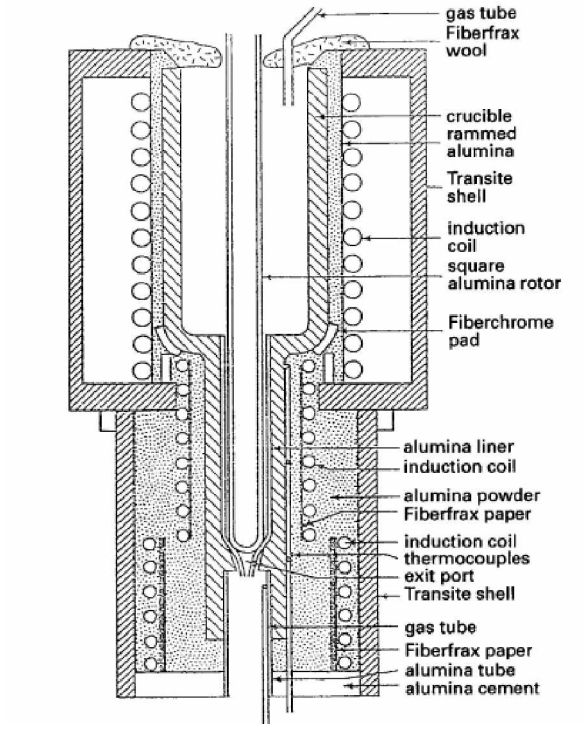

Figure 3.4. Schematic diagram of continuous caster [8]

Recently, due to the technical and commercial concerns associated with other methods for feedstock production, there has been renewed interest in this technology. Modifications to the early version of the rheocaster were made to develop a process for

feedstock production employing mechanical stirring such as SCR process (shearing-cooling roll) and passive stirring [5]. In these modified rheocasting processes, shearing and solidification are caused to occur in separate volumes, and thus are effectively decoupled. The objective of the modification was to improve the microstructural uniformity in the cross-section of the continuous cast billet [8]. Brabazon et al. [52] designed a novel stir caster for processing aluminum alloys in the mushy state. In their experiments, stir cast A356 alloy showed significant improvements in mechanical properties and reduced porosity in comparison to conventional gravity permanent mold castings.

3.3.2. Magneto Hydrodynamic (MHD) Casting

The industrial application of semi-solid metal working to metal parts used in military, aerospace, automotive or other high-quality or safety-critical applications demands integrity of the materials. Equivalence to the materials used in the conventionally cast or forged parts is a minimum specification [14]. The mechanical stirring route of producing feedstock for semisolid processing has a number of drawbacks; the most obvious being that it is only suitable for batch rather than continuous production [51]. In order to meet these requirements and overcome the problems associated with direct mechanical stirring, the MHD caster was developed. The development of this equipment recognized the need for the exclusion of gasses, oxides and nonmetallic inclusions and the avoidance of other discontinuities. It also showed that there is an important relationship between the stirring shear rate and the solidification rate, and this relationship determines the type of semisolid metal working microstructure that is generated [53].

The design of the MHD casting system incorporates the feed of filtered and degassed metal into the direct chill mold well below a quiescent surface in the delivery vessel or tundish. The metal near the freezing point in the mold is vigorously stirred by dynamic electromagnetic field, which creates the necessary shearing action. At the same time and location, controlled conductive heat transfer through the mold wall to a surrounding water jacket induces freezing. The MHD casting process therefore provide the ability to control precisely the shearing action and the rate of heat removal and thus deliver the desired solidified microstructure with a grain size that is normally about 30 μm. This compares favorably with the 100 to 400 μm grain size produced by mechanical mixers [14].

Electromagnetic stirring can be achieved through three different modes: vertical flow, horizontal flow, and helical flow, with the helical mode being ultimately a combination of the vertical and horizontal modes. In the horizontal flow mode, the motion of the solid particles takes place in a quasiisothermal plane so that mechanical shearing is probably the dominant mechanism for spheroidisation. In vertical flow mode, the dendrites located near the solidification front are recirculated to the hotter zone of the stirring chamber and partially remelted, and therefore thermal processing is dominant over mechanical shearing [8].

MHD casting systems are installed in the primary aluminum plants of one company on both vertical and horizontal continuous casters, producing a variety of fine-grained semisolid metalworking aluminum alloys from 38 to 152 mm in diameter [14]. The major advantages of horizontal continuous casting include better economy, continuous production, and low investment costs, but the quality of the billet is influenced by the gravity. On the other hand, the vertical casting system benefits from symmetrical solidification and there is no limitation of the billet diameter. However, the vertical system suffers from drawbacks such as discontinuous production, high investment costs, and high production costs [8]. The MHD equipment is essentially superimposed on the mature direct chill casting technology [14].

The MHD casting process has been engineered to deliver aluminum billets suitable for thixoforming at commercially competitive rates [14]. Much of the commercial production of aluminum alloy components to date has been based on MHD material supplied by Pechiney and SAG [9]. The essential requirements of the billet are a fine-grain non-dendritic microstructure and freedom from oxides, nonmetallic inclusions and gas. [5].

The major hurdles to the wide acceptance of this technology for thixotropic feedstock production include high production costs, microstructural non-uniformity in the cross-section of the cast billet, and non-spherical (although non-dendritic) particle morphology. Such a microstructural deficiency will cause prolonged reheating time and difficulties during subsequent thixoforming, consequently resulting in a further increase of the production costs [5]. Tzimas and Zavaliangos [33] found that MHD-cast alloys exhibit

a non-uniform initial microstructure, with dendritic features dominant at the perimeter of the casting.

3.3.3. Strain Induced Melt Activated (SIMA) Process

Small diameters materials (smaller than 38 mm) and some wrought alloys are difficult and expensive to cast and an alternative method is needed for the production of raw materials for thixoforming [14]. An effective approach utilizing wrought processing technology was perfected in 1981. This technique has been named strain induced melt activated (SIMA) by its inventors [54].

The SIMA process involves the partial melting of a heavily deformed alloy in order to obtain a fine equiaxed microstructure. The process consists of four discrete stages. First, the alloy is cast in convenient sizes to obtain a typical dendritic microstructure. Subsequently, it is hot worked so a directional microstructure is introduced and the thickness of the casting is decreased. The third stage involves the introduction of a critical level of stored energy in the alloy by cold working. Finally, the deformed alloy is partially remelted, within the range of 15–50 vol. per cent liquid, and held isothermally for a short time [24]. A schematic process sequence for the SIMA process can be seen in Figure 3.5. [55].

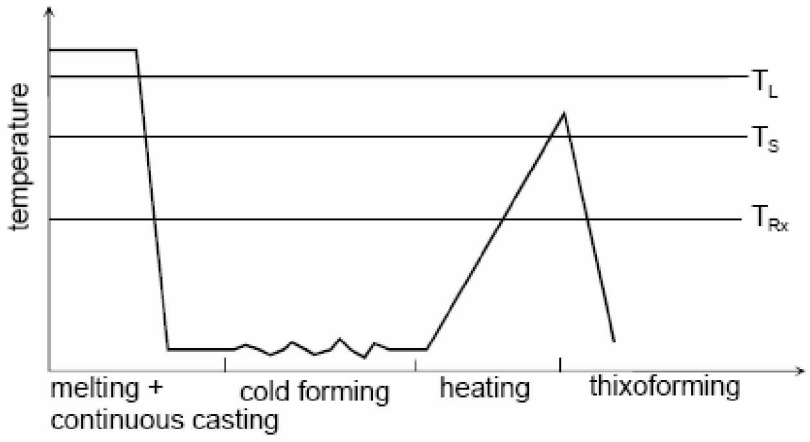

Figure 3.5. Schematic process sequence for SIMA method [55]

The transformation of the deformed dendritic microstructure into equiaxed could be explained by the grain deformation–recrystallization model: when the alloy is deformed during the third stage of the process, its dislocation density increases. During the final stage, as the temperature increases and while the deformed alloy is still in the solid state, it is subjected to recovery and recrystallization. New, strain-free grains nucleate and grow at the expense of strained grains. These new grains are characterized by high-angle grain boundaries. Upon heating above the solidus temperature, the high-energy grain boundaries are penetrated by liquid, causing the fragmentation of the original grains. The presence of liquid phase enhances grain growth and spheroidization of the newly formed grains. The soaking time and temperature in the semisolid state are expected to control the extent of grain growth and the degree of spheroidization [33].

The SIMA process produces high quality feedstock for thixoforming and has the potential for wrought alloys and high melting temperature alloys such as steel and superalloys [8]. In SIMA process, particle can be as small as 30 μm, depending on the degree of cold work and the rate of heating. A minimum of 10 per cent cold work would seem to be required for effective fragmentation, which sets a limit on the maximum practical billet achievable by extrusion to around 50 mm. The solid state production route via recrystallisation would appear to offer a competitive route to the liquid MHD process, using simpler technology and equipment generally available [5]. However, the SIMA process requires plastic deformation and recrystallisation of conventionally cast dendritic materials by thermomechanical treatments that are energy and processing intensive making it cost approximately 3–5 times more than the MHD stirring process. The SIMA process is therefore only effective for small niche applications and for small diameter feedstock [8].

3.3.4. Spray Casting

Spray forming has successfully demonstrated its ability to produce materials having superior properties compared to that of ingot metallurgy. The major advantage of spray forming is that it can produce near-net-shape preform of alloys or composites with refined equiaxed microstructure, low segregation and sometimes, increased solubility [56].

In spray casting, the metal is melted in an induction furnace and poured through a nozzle. A jet of high pressure inert gas (usually nitrogen or argon) disintegrates the stream of liquid metal into micron-sized droplets that experience very high cooling rates during flight, of the order of 10^3 K s−1 (gas atomization). While the largest droplets remain fully liquid during atomization and the smallest solidify, droplets of intermediate sizes become semisolid. The droplets are collected on a substrate moving underneath, are consolidated and form a coherent preform. A second stage of solidification takes place on the substrate at the beginning of deposition, and subsequently on the upper surface of the preform. Liquid and semisolid droplets with high liquid content (>40 vol. per cent) splat upon impact, while solid and semisolid droplets with high volume fraction of solid fragment. A portion of the solid grains is subjected to remelting and resolidifies slowly. Typical local solidification times on the preform surface are of the order of 100 s indicating that, more than 90 per cent of the solidification time of a spray cast preform occurs on the deposit, at high volume fractions of solid [24].

The microstructure of the as-spray formed preform consists of fine equiaxed grains with no microsegregation, but the mechanism by which this microstructure evolves from the dendritic droplets is complex. The dendrites contained within the droplets fragment on impact with the top surface, and then spheroidise and coarsen in a sump of semi-solid material at the top of the growing preform before final solidification [57].

The spray forming process is in commercial operation and is able to deliver clean alloy with controllable grain size down to about 20 μm [5]. Various alloys produced by spray casting have been evaluated experimentally as feedstock materials for thixoforming. [8]. A present limitation of this route is the minimum size of billet that may be sprayed, around 60 mm, though this can be reduced by a further operation such as extrusion. In total, this could be a more expensive production route compared with, says MHD; however it may have particular advantages with high temperature alloys such as steels and super alloys [5].

3.3.5. Chemical Grain Refining

In some alloy systems grain refiners are added before casting and can be potent in suppressing dendritic growth. In this case, the alloy having a fine dendritic microstructure is heated to and maintained at temperature above the solidus temperature, while retaining its shape. After the dendritic networks have been thermally transformed into spheroidal solid particles, the alloy in its semi-solid condition is then thixoformed [58]. However, chemical grain refining is not used alone, but used in conjunction with other feedstock production methods, such as MHD stirring and liquidus casting [8]. Compared to MHD techniques, the grain refinement approach is more flexible and is relatively cost-effective. Therefore, grain refined aluminum alloys are being employed for semi-solid processing by many die casters. However, there are some drawbacks inherent to the current Al-Ti/Al-Ti-B grain refined materials such as lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles in the melt. These can negatively affect the quality and productivity of SSM castings and the operation [59]. Another disadvantage of the chemical grain refinement method is that nucleation agents are only effective to specific alloy systems; another is that in some cases these additives will remain present in the product as non-metallic inclusions, which may impair both the processability of the semi finished stock and the mechanical properties of the final product [8]. Although suitable structures have been produced in aluminum alloys using higher additions of standard grain refiners, it appears to be difficult to obtain grain sizes less than 100μm and questions of recyclability of the alloys have been raised [5].

In the recent past, a permanent grain refining technology has emerged. This new technology is termed SiBloy®, and has been patented by Elkem Aluminum. Unlike traditional grain refining techniques, the grain refining effect of SiBloy® is achieved by adding Si-B master alloy into the melt. During cooling, fresh AlB_2 particles (instead of insoluble TiB_2, $TiAl_3$ etc.) precipitate out from the melt just above the liquidus temperature, which, in turn, serve as potent nucleating agents, and thus grain refining the melt. Extensive experiments with Al-Si cast alloys were carried out by Per-Arne Tondel which showed that the Si-1B additive gives rise to the finest grain structure with a small boron addition level (~0.015wt per cent B) in contrast to traditional grain refiners such as Al-6Ti, Al-5Ti-1B, Al-5Ti-0.2B, Al-4B etc. Moreover, the grain refining effect was found

to be: 1) independent of holding time (no fading); 2) unaffected by remelting treatments (permanent effect), 3) almost independent of cooling rate in the range between 0.5 and 15°C/s, and 4) effective for Al-Si alloys with Si content between 5 and 11 per cent [59].

3.3.6. Liquidus Casting Process

Liquidus casting, also known as low superheat casting, has been developed recently as an alternative technique for production of thixotropic feedstock. In liquidus casting, melt with a uniform temperature just above its liquidus is poured into a mould for solidification. The resulting microstructures are usually fine and non-dendritic. Upon reheating, the liquidus cast microstructure spheroidises rapidly to produce microstructural features suitable for thixoforming operations. So far, this technique has been tested on both cast and wrought aluminium alloys [8].

Xia and Tausig [60] made experiments about liquidus casting process of a wrought aluminum alloy 2618 by casting from its liquidus temperature They found that the resulting microstructure consisted of fine equiaxed, non-dendritic grains and the grains in the liquid cast material were fine with a mean size of 44μm and fairly globular in shape with a mean CSF of 18. Moreover, they have discussed the mechanism responsible for formation of the equiaxed, non-dendritic primary grains in liquidus casting process as mentioned below. When superheat is sufficiently low, the whole melt may be undercooled and copious heterogeneous nucleation may take place throughout the melt, and this may lead to the complete elimination of the columnar zone in the casting and to the formation of fine, equiaxed grains in the entire casting. Another possibility is that equiaxed grains may form through some crystal multiplication mechanisms such as melting of dendrite arms and deformation of dendrites caused by fluid motion from convection or pouring action. With liquidus casting, the melt before pouring has no superheat and the whole melt would certainly be undercooled after casting (some crystals might even be formed during pouring). Therefore, nucleation will take place in the entire volume of the melt and the growth of the nuclei will be equiaxed as the melt is undercooled and heat flows from the solidifying solid to the liquid surrounding it. This would most probably be responsible for the formation of the fine, equiaxed structure in the liquidus cast material although other mechanisms may also play some role [60]. Dong et al. [61] studied liquidus semi-

continuous casting of wrought aluminum alloy 7075 and they found that lowering the casting temperature changed the shape of the primary grains from dendritic ones to non-dendritic and net globular ones.

Liquidus casting is gaining more attention as a simple and cost effective technique for feedstock production and seems to have a promising future. However, the major obstacles for industrial application may arise from difficulties related to accuracy and uniformity of temperature control, and consistency and uniformity of resulting microstructure in large scale production [8].

3.3.7. Cooling Slope (CS) Casting Process

Cooling slope (CS) casting process is a recently developed method to produce raw material required for further thixoforming route. It is a simple continuous casting process and based on pouring molten metal with a suitable superheat through a cooling slope plate into the mold. A schematic illustration of this method is shown in Figure 3.6.

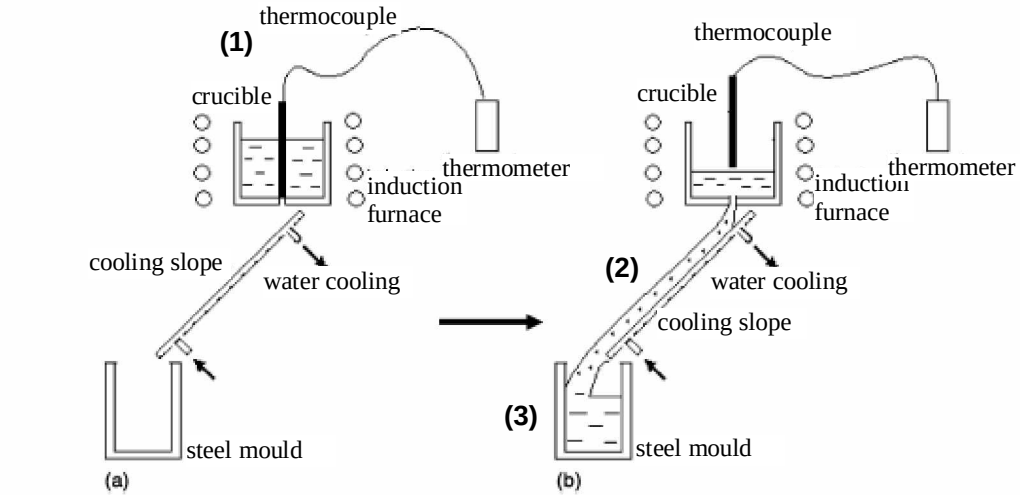

Figure 3.6. Cooling slope casting system: a) melting, b) casting via the cooling slope [62]

In the literature, two mechanisms were suggested to explain the formation of non-dendritic microstructure after CS casting process. According to Kapranos et al. [12], dendrite fragmentation mechanism plays an important role during microstructural evolution in CS casting process. The fragmentation of weak dendrite arms may occur near

the contact surface of cooling slope samples because dendritic crystals in the partially solidified melt collide under gravitational forces on the inclined CS. These crystals, formed by detachment of weak dendrite arms along the cooling slope plate based on dendrite fragmentation mechanism, then grow in the mould [12]. On the other hand, according to Motegi at al. [63, 64, 65, 66], crystal separation theory is responsible for the formation of non-dendritic morphology as-cast CS microstructures. According to this theory, granular crystals nucleate and grow on the cooling end like a mold wall and separate from it due to fluid motion. They believe that metal crystals were generated on the cooling plate and moved with molten alloy into a heating mold, and subsequently became granular in the mold. The inclination governs the flow rate and contact time between the molten alloy and the cooling plate. If a low angle is employed, the molten alloy flows slowly and a solid shell forms easily on the cooling plate. In contrast, if a high angle is employed, the alloy flows faster and fewer crystals are formed. As a result, the dendrite crystals are produced.

The cooling slope casting process is composed of the following main process sections (Figure 3.6):

a) A melt pouring section (1), which consists of a crucible which melts the alloy and pours down it through the cooling slope plate.
b) A nucleation section (2), which generates crystal nuclei in the melt as it is flowing through the cooling slope plate.
c) A crystal generating section (3) which metal obtained from the nucleation section is cooled down in the metal mold.

Cooling slope casting provides globular or spheroidal microstructure when heated to the semisolid temperature. However, a drawback of this process may be the formation of casting defects, such as pores and oxide. This would cause the mechanical properties of thixoformed CS products to be lower [12]. Studies about this process were reviewed below. Most of the studies were carried out by the leadership of Motegi and Haga in Japan.

Motegi et al. [63, 64, 65, 66] performed many experiments using a water cooled slope made of pure copper in order to obtain thixotropic Al-Si-Mg feedstock for thixoforming. They modified a vertical continuous casting machine as shown in Figure 3.7.

Crystal seeds of the primary α aluminum were nucleated on a specially designed cooling plate. This was followed by pouring the molten alloy into the mold. The seeds of primary crystals existing in the molten alloy grew granular grains. In their first study, Al 7.64wt per cent Si0.303wt per cent Mg alloy was poured at 624 to 724˚C with a cooling plate of 470 mm long and 70˚ inclination. They observed that granular crystals appear at pouring temperature of 654˚C and cooling length of 100 mm. They found that the cooling plate was effective in generating crystal seed in the molten alloy. In order to explain the presence of granular crystals in the solidified structure, they applied crystal separation theory which proposes that granular crystals nucleate and grow on the mold wall, and then are separated from there by fluid flow. They also found evidence that lower casting temperatures in the mold yield more granular crystals of α aluminum [63, 64]. In their following experiments, they poured an Al 1.63wt per cent Si0.54wt per cent Mg alloy at 656m to 696˚C. They found that the cooling slope is useful for generating numerous crystal seeds, and the best conditions were tilt angle 60˚, length 200 mm. If length in cooling slope is too long, the molten alloy flowing onto it forms a solid shell, on the other hand, short distance generate small amount of crystals seeds, because there are less position for seed to be generated, and finally the best pouring temperature was 656˚C. They found evidence that increase in pouring temperature resulted in a bigger particle size [65]. Motegi and Tanabe [66] used a copper alloy with different Sn contents in order to obtain semisolid feedstock with inclined cooling plate. Their results were generally consistent with the previous studies and they found that the best casting temperature of each Cu-Sn alloy was liquidus temperature + 30 K and the optimum plate inclination was 60 degrees with a cooling length of 280 mm. Moreover, semisolid slurry must be held in an isothermally heating mold in order to produce granular crystals.

Haga and Suzuki [67] cast A356 alloy via a cooling slope made of mild steel at a range of temperatures of 640 to 680^{o}C and investigated the factors which affect the spheroidicity of αAl. In order to analyze the effect of cooling rate on the formation of primary αAl particles, they used two mould materials (metal and insulator) and they found that the factor which mostly affects the globularization of the primary crystal is the cooling rate in the mold. When metal mold was used, finer and more globular grains were obtained comparing to insulator mold. Haga and Kapranos [68] poured A356 and A390 aluminium alloy ingots via cooling slope casting through pouring distances of 150 and 250 mm, with a

tilt angle of 60°, and superheats of 20 and 40°C. They used two molds made of copper and ceramic. The as-cast billets were reheated at 570, 580 and 590°C and then thixoformed. With copper die, if the ingot was heated up at 570°C the primary particles did not spheroidise, but if the temperature is 580 or 590°C the particles became spheroidal, with a larger particle size for the higher temperature. With the insulating die, when the ingot was heated up to 570°C the primary particles spheroidised, for 580 or 590°C, the particles also became spheroidal, with a larger particle size for the higher temperature. Haga and Kapranos [69] poured A356 aluminum alloy into preheated lower part of a die and immediately an upper die at room temperature, containing an internal cavity, was inserted in the lower die half. The results were in agreement with previous work and they found again that low superheat temperature promoted the formation of spheroidal grains.

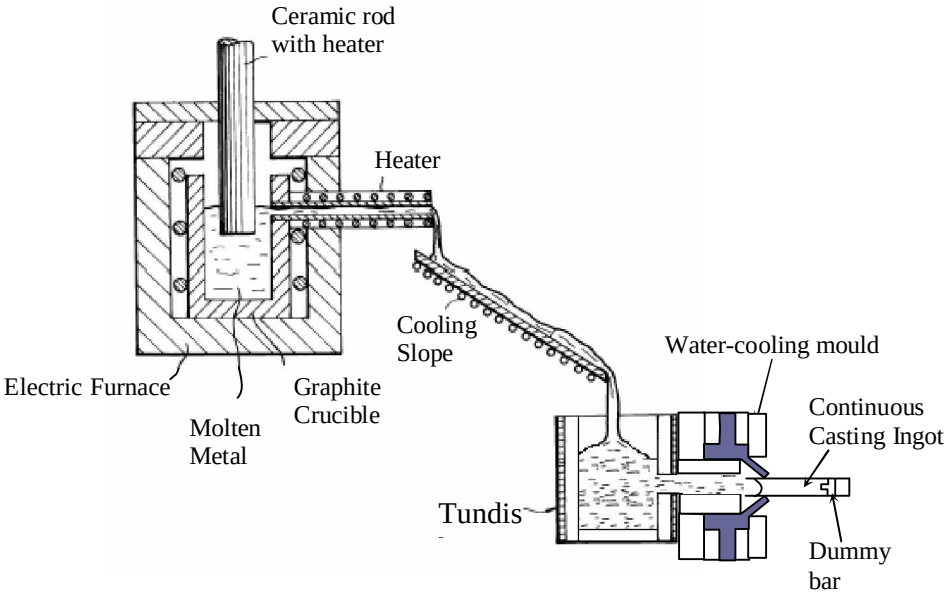

Figure 3.7. Schematic illustration of horizontal continuous casting of semi-solid aluminum alloy [64]

Different from the previous studies, Liu at al. [62] used cooling slope casting technology for the preparation of thixoformable feedstock from aluminum wrought alloys instead of conventional cast alloys. They poured 2014, 6082, 7075 and 7010 aluminum wrought alloys at a temperature 10-20 K above the liquidus via a water cooled slope and reheated as-cast samples to the semi-solid intervals in order to observe the morphologic

evolution. Cooling slope cast material showed coarsened rosette morphology with a relatively high proportion of entrapped liquid within globules after heating to the semi-solid state. Thixoforming for all alloys resulted in successful filling of die and the tensile results of all thixoformed alloys were in agreement with the results in the literature.

4. EXPERIMENTAL STUDY

4.1. Materials Used in Thixoforming Experiments

The alloys used for thixoforming process are limited to a few casting alloys, only a limited number of trials have been carried out on wrought alloys. Al-Si7-Mg (A356 and A357) cast alloys are the most common aluminum alloys used as raw material for thixoforming applications. Commercially, MHD route is processed for A356 and A357 aluminum cast alloys to be used as feedstock materials for thixoforming applications by SAG in Austria. The application of aluminum wrought alloys in thixoforming industry has been growing during last years. AA6082 and AA7075 wrought alloys are the most widely tried alloys for thixoforming. SIMA route is the most appropriate method for processing wrought alloys to obtain non-dendritic feedstock material.

In experimental studies, A357 (Al-Si7-Mg) aluminum cast alloy and AA6082 (Al-Mg-Si1) aluminum wrought alloy were used. A357 alloy is also designated as Etial 177 according to Etibank designation system. A357 alloy is commercially fabricated by Etibank as ingot forms. AA6082 alloy was supplied from ASAŞ ALÜMİNYUM as in billet shape for the experimental study. Al-Si based A357 alloy has wide solidification interval and good fluidity and due to these properties, it is widely used in thixoforming processes especially in automotive applications. AA6082 aluminum alloy is heat treatable, high strength wrought alloy and it is used in Europe for structural extrusions in automotive parts. However, AA6082 alloy has very strong temperature versus liquid volume fraction dependence, lower fluidity compared with silicon containing cast alloys and narrow solidification interval so there are some difficulties in using this alloy for thixoforming applications.

The chemical compositions of A357 and AA6082 alloys are given in Table 4.1. In order to obtain solidus and liquidus temperatures of these alloys, differential scanning calorimeter (DSC) technique was carried out in TÜBİTAK MAM Alloy Development Laboratory. Samples of about 30 mg weight were cut from both alloys and put in an argon

atmosphere for DSC tests A357 sample was heated to 650˚C and cooled to 450˚C at 2.5 ˚C/min while AA6082 sample was heated to 700˚C and cooled to 500˚C at 2.5 ˚C/min. Liquidus and solidus temperatures of both alloys are the starting and ending point of the endothermic reaction occurred during phase transformation as shown in Figure 4.1 and Figure 4.2. According to these DSC curves, solidus and liquidus temperatures of A357 and AA6082 alloys are given in Table 4.2.

Table 4.1 Chemical composition A357 and AA6082 alloys (wt per cent)

Alloy	Al	Si	Mg	Cu	Fe	Mn	Ti
A 357	92.72	6.629	0.347	0.016	0.167	0.002	0.066
AA6082	97.25	1.194	0.677	0.004	0.189	0.625	0.014

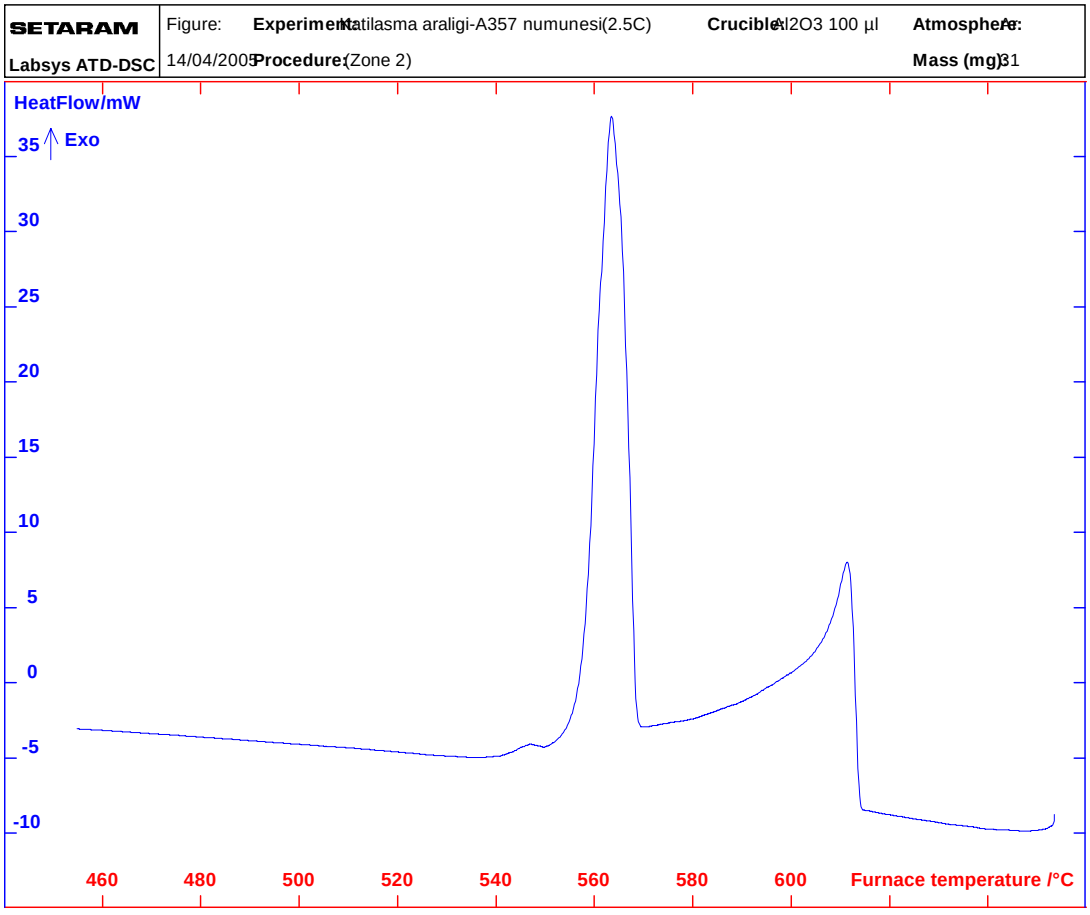

Figure 4.1. DSC curve of A357 alloy

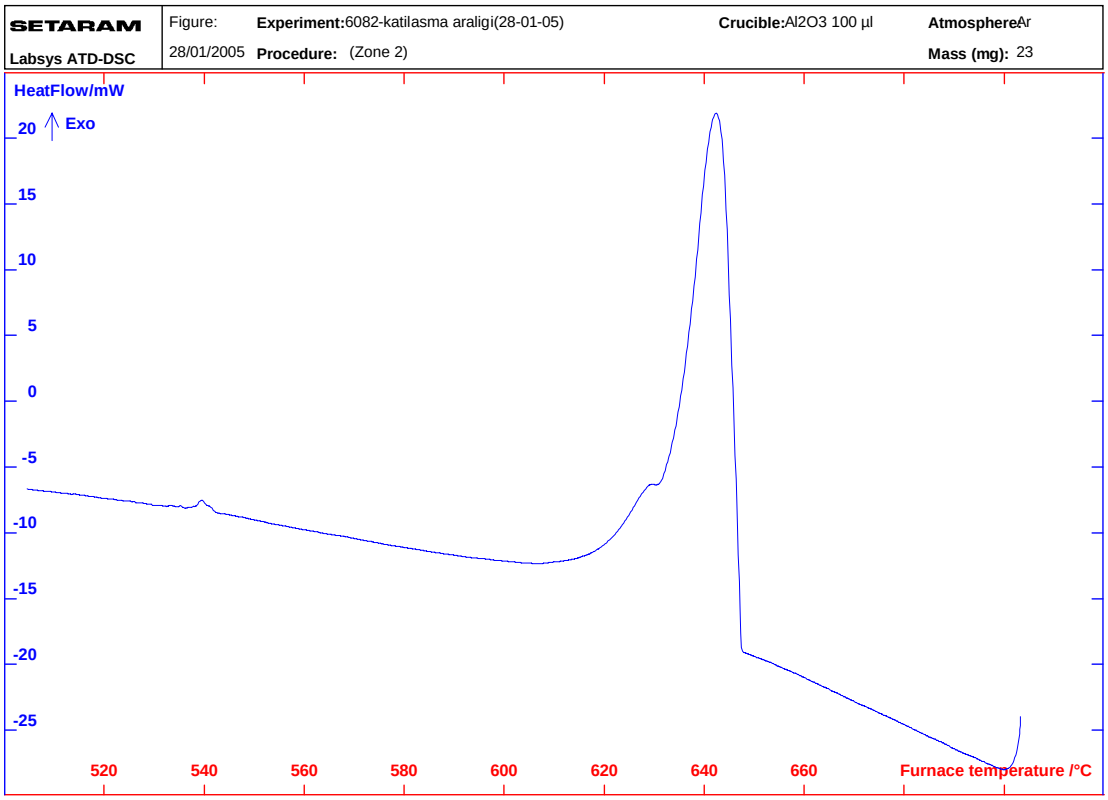

Figure 4.2. DSC curve of AA6082 alloy

Table 4.2 Solidus and liquidus temperatures of A357 and AA6082 alloys

Alloy	solidus temperature (^{o}C)	liquidus temperature (^{o}C)
A357	549	614
AA6082	603	649

4.2. Experimental Methods

In the experimental study, thixoforming of A357 and AA6082 alloys were carried out in three steps. Production of the non-dendritic raw material for thixoforming process was the first step in the experimental study. Cooling slope casting process was carried out as a feedstock production method. The second step of the thixoforming process was reheating practice in order to observe the microstructural evolution of as-cast billets in the semi-solid interval. In this step of experiments, as-cast billets were heated to temperatures

between solidus and liquidus and optimum cooling slope casting parameters were determined for further experimental step. The last step of the thixoforming experiments was heating the chosen billets to semi-solid temperature interval and giving a final shape in that phase. After casting, reheating and forming practices, metallographic studies were carried out in order to investigate microstructures of the specimens. Moreover, in order to determine mechanical properties of the thixoformed specimens, hardness measurements and tensile tests were carried out. T6 heat treatment practice was performed to improve the mechanical properties of the thixoformed specimens.

4.2.1. Feedstock Production via Cooling Slope Casting Process

Thixoforming is an advantageous process for the production of complex shaped parts with less process steps and lower cost. Before the forming operation, microstructure should consist of immersed solid particles in liquid matrix and during reheating, lower melting point phase should disperse to the grain boundaries. There are various methods for obtaining such a structure and among them; cooling slope casting process is attractive due to its practical and economical application. In the experimental study, non-dendritic feedstock was produced via cooling slope casting process. In the cooling slope casting process, low superheat molten metal was poured through water cooled inclined plate into a mold. The molten metal starts solidification on the cooled plate and continues solidification in the mold. With this method, dendritic morphology of the material turns into a non-dendritic microstructure.

In the experimental study, firstly, A357 and AA6082 alloys were melted in a graphite crucible. Resistance and induction furnaces were used to melt the material. An induction furnace and coils were designed for melting and heating processes during the experiments. A photo of induction furnace for melting operation is shown in Figure 4.3. Dimensions of the induction coil for melting operation are below:

- Coil wire diameter.....7 mm
- Coil diameter.....103 mm
- Coil height.....100 mm
- Number of turns.....5

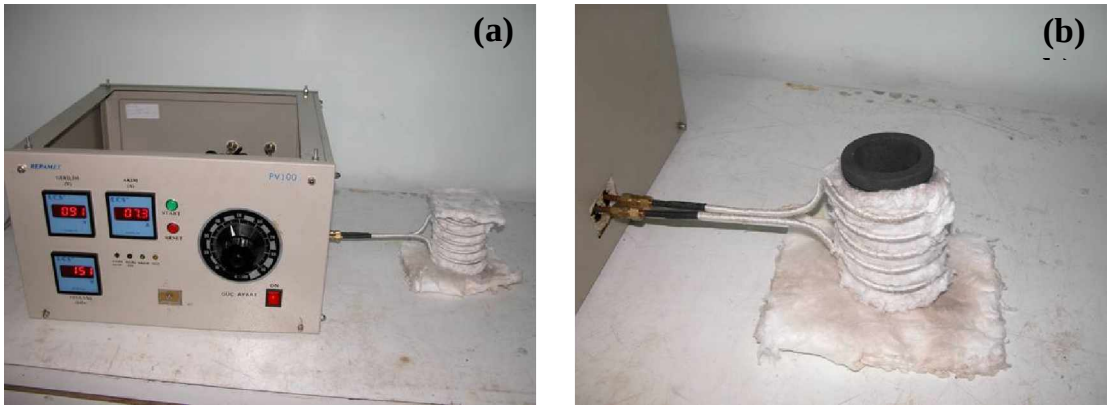

Figure 4.3. Induction melting unit a) induction machine b) graphite crucible inside the melting coil

A357 alloy was melted at 680^{o}C and AA6082 alloy was melted at 710^{o}C. The oxide layer on the molten material was cleaned with a metal rod before casting. Then, molten metals were allowed to cool down in the air to the suitable superheat temperatures which were varying from 10^{o}C to 40^{o}C. A K type thermocouple was used to monitor the temperature in the melt. When the molten metal reached the desired superheat temperature, it was poured through a specially designed cooling slope plate into a metal mold. Tilt angle of the cooling slope was chosen as 60^{o}. The molten material was poured from three different pouring distances for each casting temperature in order to investigate the effects of casting temperature and pouring distance on the formation of non-dendritic microstructure. Experimental conditions for casting with the cooling slope are given in Table 4.3.

Table 4.3 Experimental conditions of cooling slope casting process

Alloy	A357		AA6082
Pouring temperature range	620^{o}C to 640^{o}C		665^{o}C to 680^{o}C
Tilt angle (α) of the cooling slope	60^{o}		
Mould material	Mild steel		
Pouring distance on the cooling slope	200 mm	300 mm	400 mm

The cooling slope plate was produced from U profile made of mild steel and has dimension of 10 x 50 x 500 mm. The plate was cooled with city water that has a flowrate of 5 liter/minute. In order to prevent sticking of molten aluminum to the cooling slope plate, boron nitride (BN) spray was coated on the plate before starting the casting practice. Steel was chosen as mold material because in cooling slope casting process after the nucleation stage, one of the important aspects is to control the cooling rate in the mould and the mould must provide first a cooling rate high enough to promote the nucleation process. Steel mold has dimensions of 100 x 100 x 150 with a core of 30 mm in diameter. The amount of aluminum cast during the process was approximately 300 gram and the resulting as-cast billet from the cooling slope casting process had 30 mm diameter and 120 mm height. The width of the plate is 50 mm and the diameter of the core inside the mold is 30 mm so a cone was designed to collect the flowing aluminum and ease the filling of the mold with out spreading outside. The cone was produced from graphite because graphite has a property of conserving the heat. The graphite cone was heated to 500C^{o} in a resistance furnace in order to prevent freezing of the aluminum pouring from the plate before filling the mold and hot graphite cone was inserted on the mold before starting to cast. The photo and schematic illustration of the cooling slope casting process experimental set up is shown in Figure 4.4.

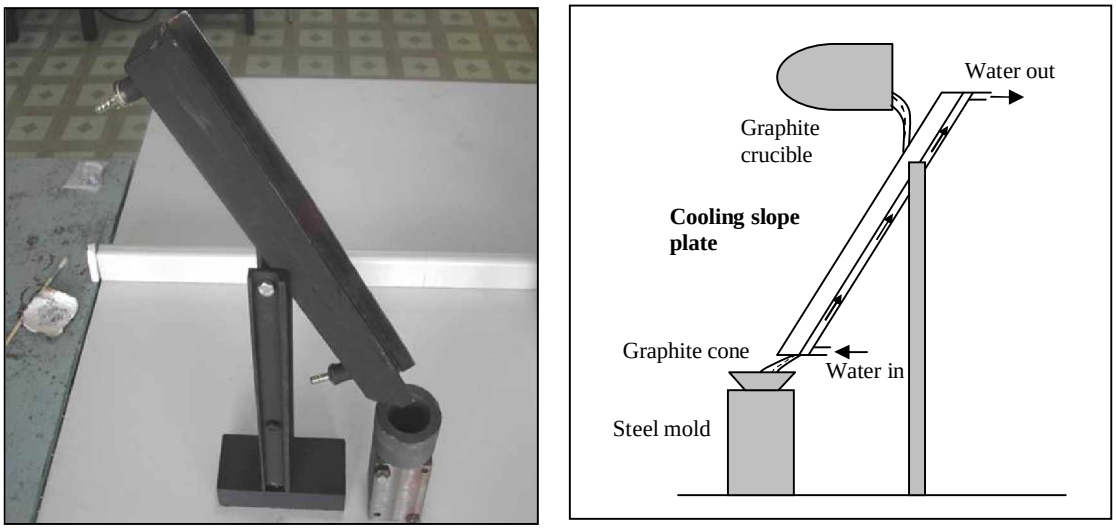

Figure 4.4. Cooling slope casting process experimental set up

4.2.2. Reheating and Isothermal Holding of As-cast Billets

Grain coarsening and liquid fraction in the semi-solid state depend on both temperature and time. Therefore, with the aim of examining the microstructural evolution in the semisolid state as a function of holding time, samples of as-cast A357 and AA6082 billets were subjected to isothermal exposure at various times and temperatures after cooling slope casting process.

Before starting the reheating practices, solid fraction versus temperature curves of A357 and AA6082 alloys should be evaluated in order to determine the isothermal holding temperatures. For the determination of volume fraction of solid, the DSC curves (Figure 4.1 and Figure 4.2) and heat transfer model of Gray (Equation 4.1) were used [70].

$$\frac{dh}{dt} = -\frac{dq}{dt} + (C_s - C_r)\frac{dT_p}{dt} - RC\frac{d^2q}{dt^2} \tag{4.1}$$

where h is the enthalpy, q is the heat flux represents the heat flux between sample and reference pan and it was determined by DSC experiment. Scanning velocity, dT_p/dt, is the variation of temperature via time while $(C_s - C_r)\ dT_p/dt$ is the deviation in the baseline. RC is the system parameter. So Equation (4.1) can also be written as:

$$\frac{dh}{dt} = -\frac{dq}{dt} - RC\frac{d^2q}{dt^2} \tag{4.2}$$

By using finite differences method, Equation (4.2) can be rewritten as:

$$\frac{\Delta h}{\Delta t} = -\frac{\Delta q}{\Delta t} - \frac{RC\left\{\left[\frac{\Delta q}{\Delta t(t+\Delta t)} - \frac{\Delta q}{\Delta t(t)}\right]\right\}}{\Delta t} \tag{4.3}$$

Scanning velocity, dT_p/dt, did not change so Δt was known for specific T_p temperature. RC was the system parameter while h was the area under the DSC curve and represents the enthalpy value. Assuming that, enthalpy value was directly proportional

with amount of solidifying alloy, solid fraction was determined. In order to obtain solid fraction curve, firstly, RC value which was the only unknown term in the right side of the Equation 4.3 was calculated by trial and error method. During this method, the enthalpy value corresponded to 100 per cent solid fraction was obtained from the software presents in the DSC machine. In order to calculate real RC value, iteration method was carried out such as an initial RC value was given in Equation 4.3 and the resulting enthalpy value was compared to the enthalpy value obtained from the software. The real RC values were calculated as 33.79 for A357 alloy and 12.66 for AA6082 alloy. After finding the only unknown term in the right side of the Equation 4.3 (RC), volume fraction of solid versus temperature curves of both alloys were obtained by calculating enthalpy values for each temperature. Figure 4.5 shows the solid fraction versus temperature curves of A357 and AA6082 alloys. In accordance with this figure, reheating temperatures were determined as 580˚C for A357 alloy which corresponds to solid fraction of 50 per cent and 640˚C for AA6082 alloy which corresponds to solid fraction of 52 per cent.

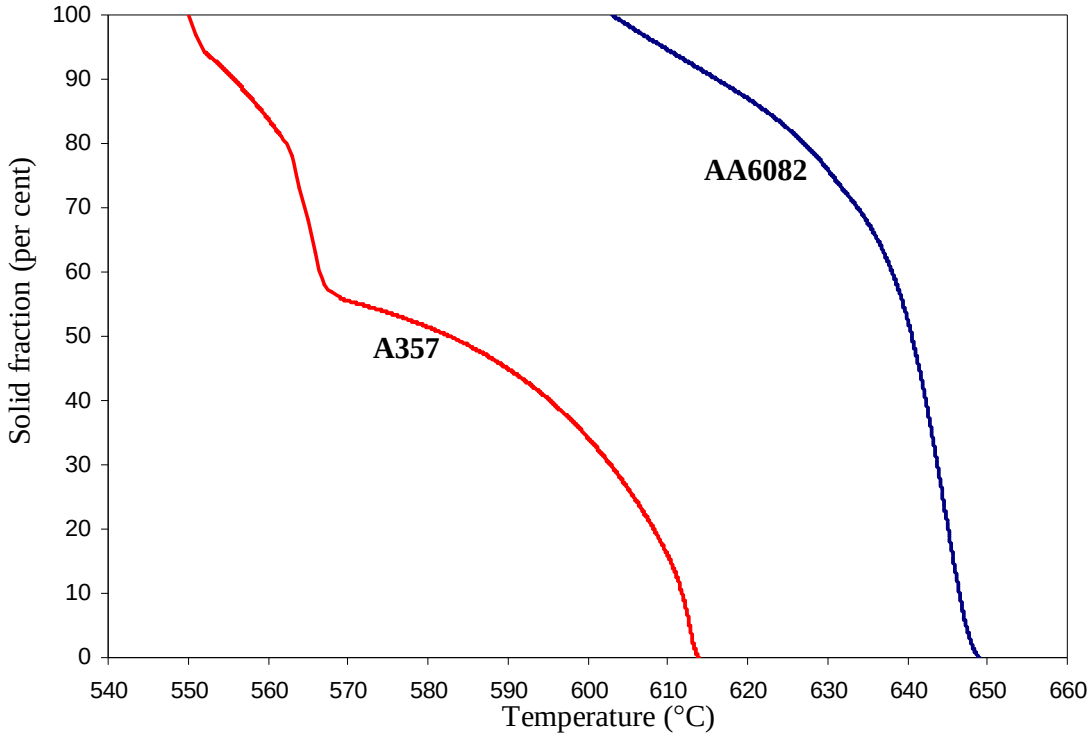

Figure 4.5. Solid fraction versus temperature curves of A357 and AA6082 alloys

During the reheating practice, billets should be heated to semi-solid temperature interval as quickly and as homogeneously as possible. Induction heating units are widely accepted heating systems for thixoforming. In induction heating system, energy is transferred from a coil to the billet via an alternating magnetic field. The induced electro-magnetic force (e.m.f) produces a circulating current that in turn, generates heat on the billet. So if energy on the coils is cut, the heating of the billet is stopped immediately. In resistance furnaces, there is no such option to cut the heat on the billet immediately. So, another advantage of induction heating unit over resistance furnace is the more accurate controlling of the temperature on the billet.

For the reheating experiments, a laboratory scale induction furnace was designed. It is a high frequency induction unit with the characteristics: 3 kW maximum power and frequency up to 130 kHz. This induction furnace was designed to perform melting and heating functions during the experimental studies. For each function, special coils were designed. Designed coil for the melting function was mentioned in the previous section. Dimensions of the induction coil for the heating function are below:

- Coil wire diameter.....7 mm
- Coil diameter.....65 mm
- Coil height.....50 mm
- Number of turns.....5

The as-cast billets were cut into small billets with a diameter of 30 mm and height of 35 mm for the reheating experiments. 3 mm diameter hole was drilled from the center of the surface through a depth of 15 mm in order to insert the K-type thermocouple. The machined as-cast billets were placed in the center of the coil on a refractory type material Experimental set-up of the reheating system is shown in Figure 4.6.

During the reheating practice, controlling of the temperature is very important. Temperature controlling unit should own at most ± $1^{o}C$ sensitivity in order to avoid undesired temperature fluctuations during the isothermal holding step of reheating. In the reheating experiments, temperature of the billet was controlled directly via an inserted K-

type thermocouple. During reheating, temperature was displayed with a digital thermometer that has sensitivity of ± 1°C.

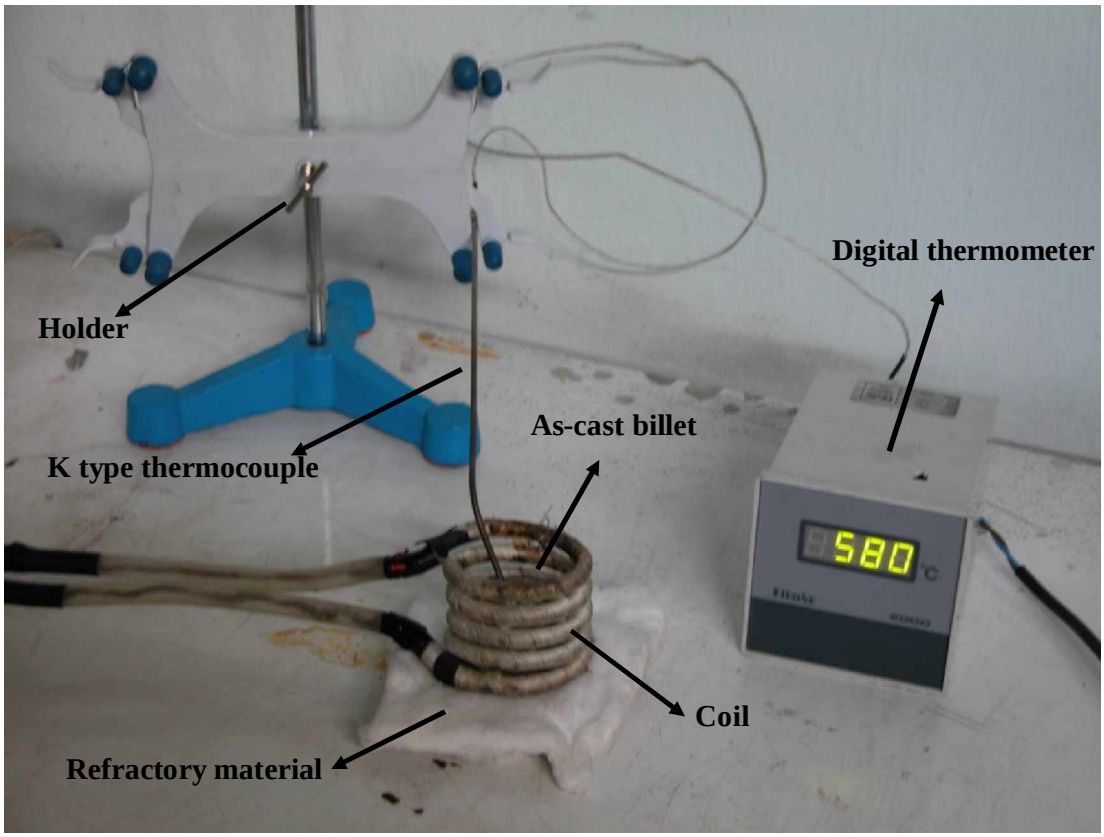

Figure 4.6. Experimental set-up of the reheating and isothermal holding practices

The reheating duration is directly proportional with the used power of the induction heating furnace. In the experiments, the as-cast billets were heated by induction furnace with power of 1.3 kW. The current of the furnace was 7 A and the voltage was 190 V. A357 billets were heated to semi-solid temperature (580˚C) in 3 minutes while AA6082 billets were heated to semi-solid temperature (640˚C) in 4 minutes. This time difference arises from the different semi-solid temperatures of the alloys. During the reheating, the billets were subjected to different isothermal holding times varying from 5 minutes to 30 minutes. Figure 4.7 and Figure 4.8 show reheating curves of the A357 and AA6082 as cast billets with isothermal holding time of 5 minutes.

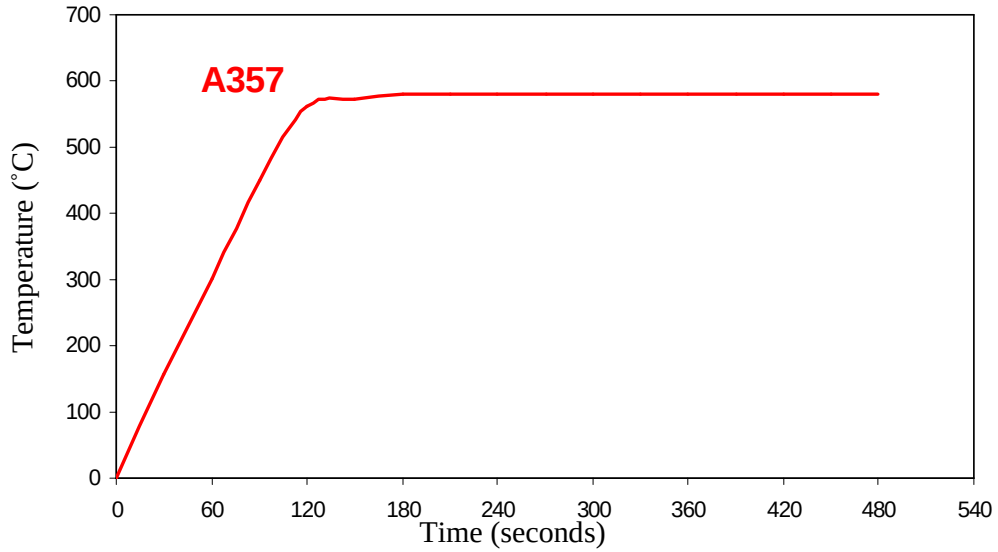

Figure 4.7. Reheating curve of A357 alloy with isothermal holding time of 5 minutes

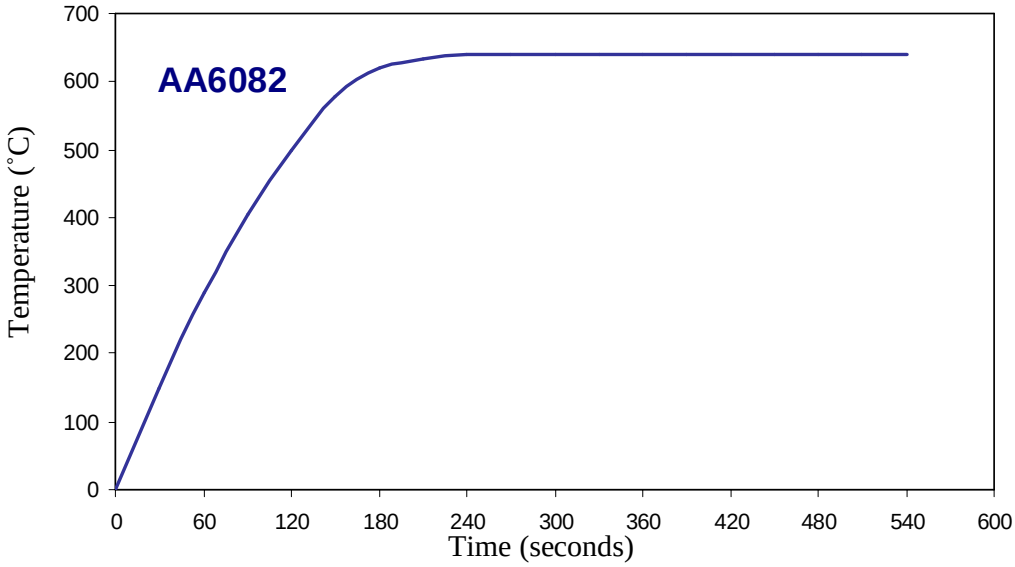

Figure 4.8. Reheating curve of AA6082 alloy with isothermal holding time of 5 minutes

The parameters used in cooling slope casting and reheating practices are given in Table 4.4 and Table 4.5. Codes were given for each parameter to ease designation in further experimental studies.

Table 4.4. Experimental conditions used in cooling slope casting process and reheating practice for A357 alloy

Sample code	Casting Temperature (°C)	Casting Distance (mm)	Holding temperature (°C)	Holding time (minute)
CA1	640	400	-	-
CA1.1	640	400	580	5
CA1.2	640	400	580	15
CA1.3	640	400	580	30
CA2	630	400	-	-
CA2.1	630	400	580	5
CA2.2	630	400	580	15
CA3	620	400	-	-
CA3.1	620	400	580	5
CA3.2	620	400	580	15
CA4	640	300	-	-
CA4.1	640	300	580	5
CA4.2	640	300	580	15
CA5	630	300	-	-
CA5.1	630	300	580	5
CA5.2	630	300	580	15
CA6	620	300	-	-
CA6.1	620	300	580	5
CA6.2	620	300	580	15
CA7	640	200	-	-
CA7.1	640	200	580	5
CA7.2	640	200	580	15
CA8	630	200	-	-
CA8.1	630	200	580	5
CA8.2	630	200	580	15
CA9	620	200	-	-
CA9.1	620	200	580	5
CA9.2	620	200	580	15

Table 4.5. Experimental conditions for AA6082 alloy

Sample code	Casting Temperature (^{o}C)	Casting Distance (mm)	Holding temperature (^{o}C)	Holding time (minute)
C1	680	400	-	-
C1.1	680	400	640	5
C1.2	680	400	640	15
C2	675	400	-	-
C2.1	675	400	640	5
C2.2	675	400	640	15
C3	670	400	-	-
C3.1	670	400	640	5
C3.2	670	400	640	15
C4	665	400	-	-
C4.1	665	400	640	5
C4.2	665	400	640	15
C5	680	300	-	-
C5.1	680	300	640	5
C5.2	680	300	640	15
C6	675	300	-	-
C6.1	675	300	640	5
C6.2	675	300	640	15
C7	670	300	-	-
C7.1	670	300	640	5
C7.2	670	300	640	15
C8	665	300	-	-
C8.1	665	300	640	5
C8.2	665	300	640	15
C9	680	200	-	-
C9.1	680	200	640	5
C9.2	680	200	640	15
C10	675	200	-	-
C10.1	675	200	640	5
C10.2	675	200	640	15
C11	670	200	-	-
C11.1	670	200	640	5
C11.2	670	200	640	15
C12	665	200	-	-
C12.1	665	200	640	5

4.2.3. Forming Operation

After obtaining feedstock material for thixoforming via cooling slope casting process and reheating practices, the last step of the thixoforming was to give a final shape to produced billet in the semi-solid state.

The thixoforming operation was carried out with a hydraulic press as shown in Figure 4.9. The maximum load of the press is 7.8 ton-f and the maximum speed of the ram is 89 mm / sn. In thixoforming presses, the speed of the ram is very important and it must be as fast as possible in order avoid temperature drop in the reheated billet before the pressure is applied.

Figure 4.9. Photo of hydraulic power unit

For the thixoforming experiment, two kinds of dies were designed with Solid Works 3D software. The material of the dies was 2344 steel. Hardness of the dies was increased to 52 HRC via heat treatment in order to minimize dimensional changes because of thermal expansion and applied load and in order to extend life of the dies. The two die groups were designed for different purposes. Each die group is composed of three dies: main die, upper

half die and lower half die. First die group was designed to thixoform a part for metallographic investigations and hardness tests while the second die group was designed to obtain tensile specimen from the thixoformed product to investigate tensile mechanical properties. Figure 4.10 shows photo of each die group. Thixoformed samples are also shown in the same figure in order to describe the cavity of the dies. The design of die groups was similar but the cavity of the second group is deeper comparing to the first group in order to obtain much longer specimens needed for tensile tests.

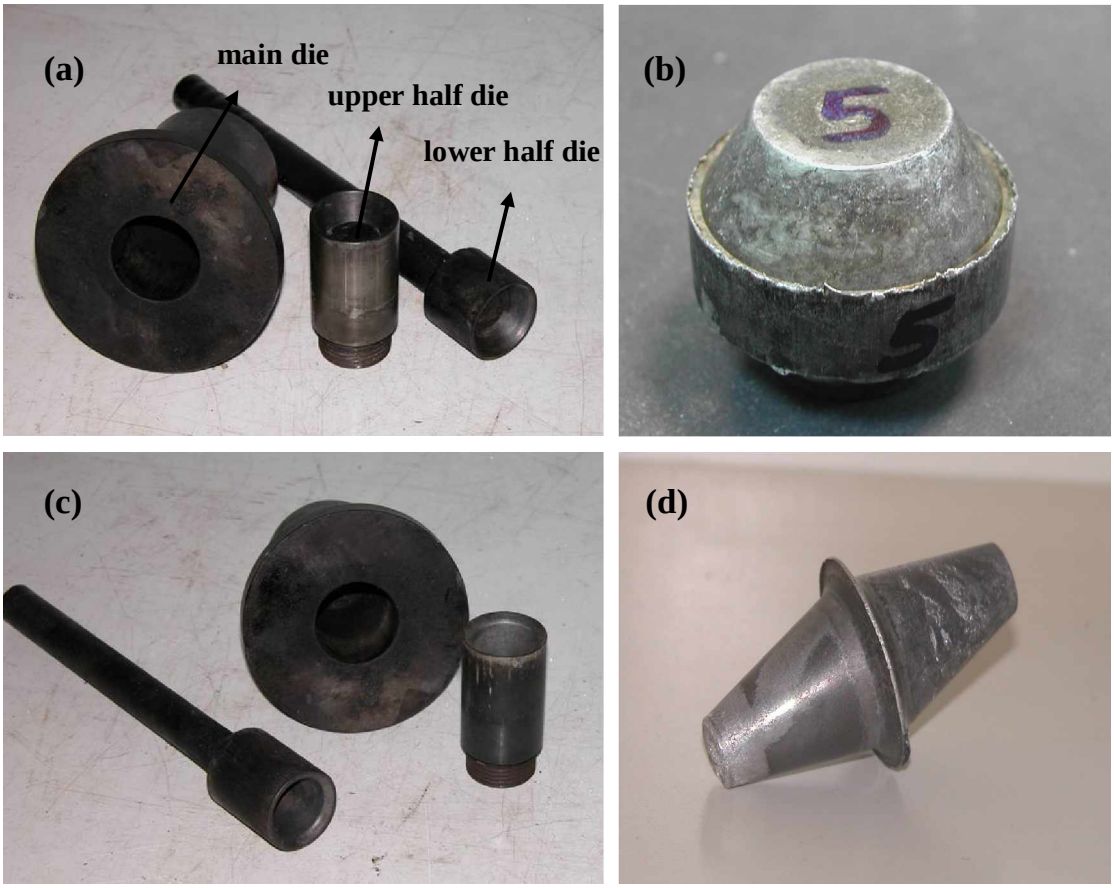

Figure 4.10. Photo of the die groups and thixoformed sample a) first die group, b) sample obtained after thixoforming with first die group, c) second die group, d) sample obtained after thixoforming with second die group

Before thixoforming experiment, some as-cast billets were chosen according to their microstructural evolution during the reheating practices. For A357 alloy, CA4 and CA5 coded as-cast billets were chosen while for AA6082 alloy, C7 coded as-cast billet was

chosen for forming practices. During the thixoforming experiment, the chosen billets were reheated to semi-solid temperature by induction furnace. In order to retain the billet shape and to prevent heat loss, it is an important part of the forming process to transfer the re-heated material, having the desirable solid fraction, to the die cavity. Transferring of the reheated billet from induction coil to the die cavity is a major problem in thixoforming process because handling of the semi-solid material is very different. In the experimental study, this transferring problem was solved with the design of the die. As mentioned before, two die groups were designed to obtain different results from the thixoformed samples. As the design and working principles of the die groups were similar, only first die group is explained. The first die group was composed of three parts: main die, lower half die and upper half die. Two methods were carried out in order to solve the semi-solid transferring problem.

In the first method; the coil of the induction furnace was placed on the surface of the main die while the chosen as-cast billet were placed in the cavity of the lower half die as shown in Figure 4.11a. After reheating route of these billets with induction furnace, the lower half die was pulled down by hand while the upper half die was moved down into the cavity of the main die by hydraulic press as shown in Figure 4.11b. The semi-solid material was forged between upper and lower half dies. After completion of the forging route, the upper half die was moved up and the forged aluminum billet was ejected by pulling up of the lower die as shown in Figure 4.11c. In this method, the transferring of the reheated billet was successfully performed however; the microstructure of the thixoformed sample was not uniformly distributed. Because during the reheating of the billet, the bottom of the billet was in contact with lower half die and this lead heat extraction from the contact surface so temperature difference occurred between upper and lower part of the reheated billet. To provide uniform temperature distribution during the reheating of the billet, a new transferring method was designed.

In the second method, a refractor and non-inductive material was placed between the coil of induction furnace and main die while as-cast billet was placed on this material during reheating route as shown in Figure 4.12a. After completion of reheating and isothermal holding routes in semi-solid temperature, the refractor material was pulled in order to allow the semi-solid aluminum billet to drop in the cavity of lower half die. At the

same time, the upper half die was moved down into the cavity of the main die and aluminum billet was forged in the semi-solid state between lower and upper half dies as shown in Figure 4.12b. Then, the upper half die was moved up and the forged aluminum was ejected by pulling up of the lower die as shown in Figure 4.12c. In this method, both transferring and homogenous reheating of the billet was successfully performed due to the fact that refractor material avoided heat extraction from contact surface.

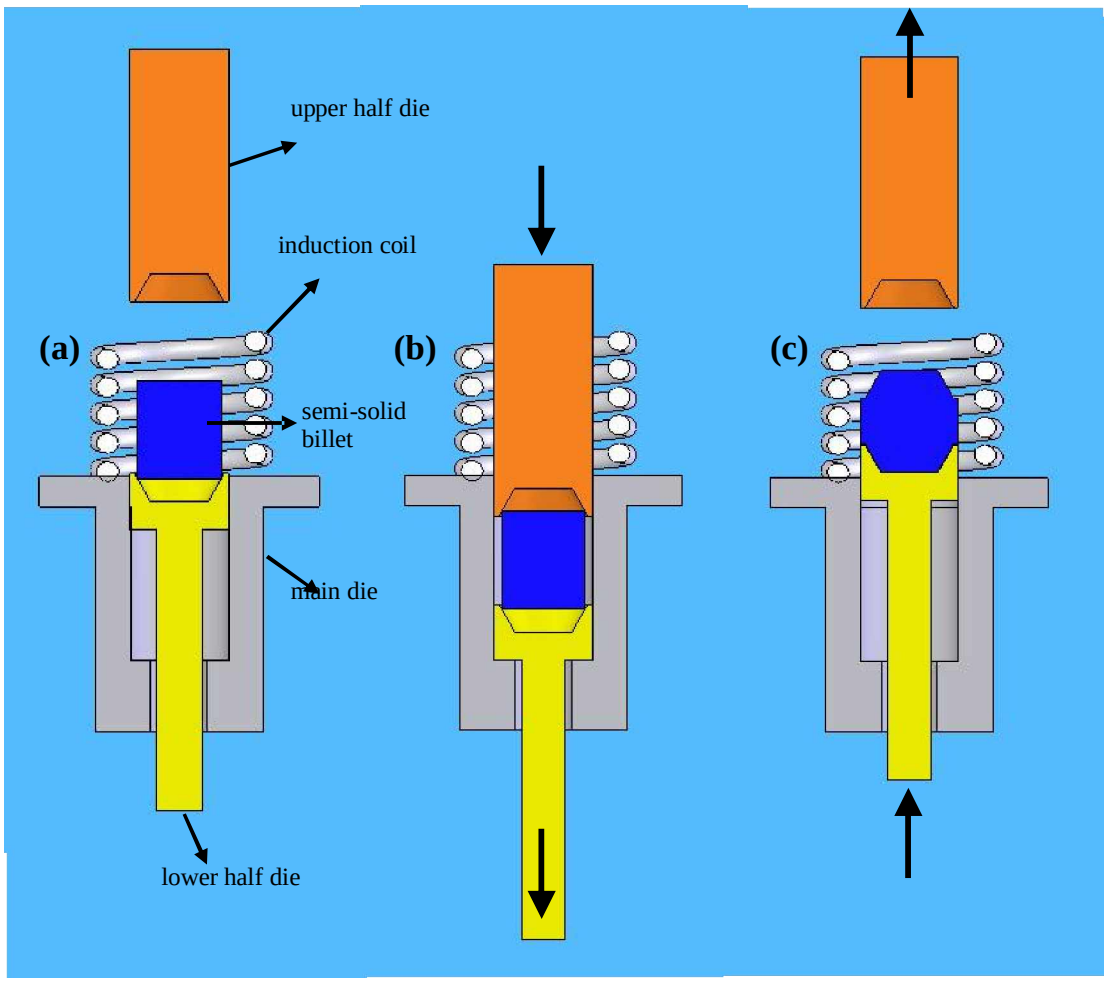

Figure 4.11. Simulation of the first method thixoforming experiment designed with Solid Works 3D software a) reheating of as-cast billet. b) forging of semi-solid billet. c) ejecting of the thixoformed sample

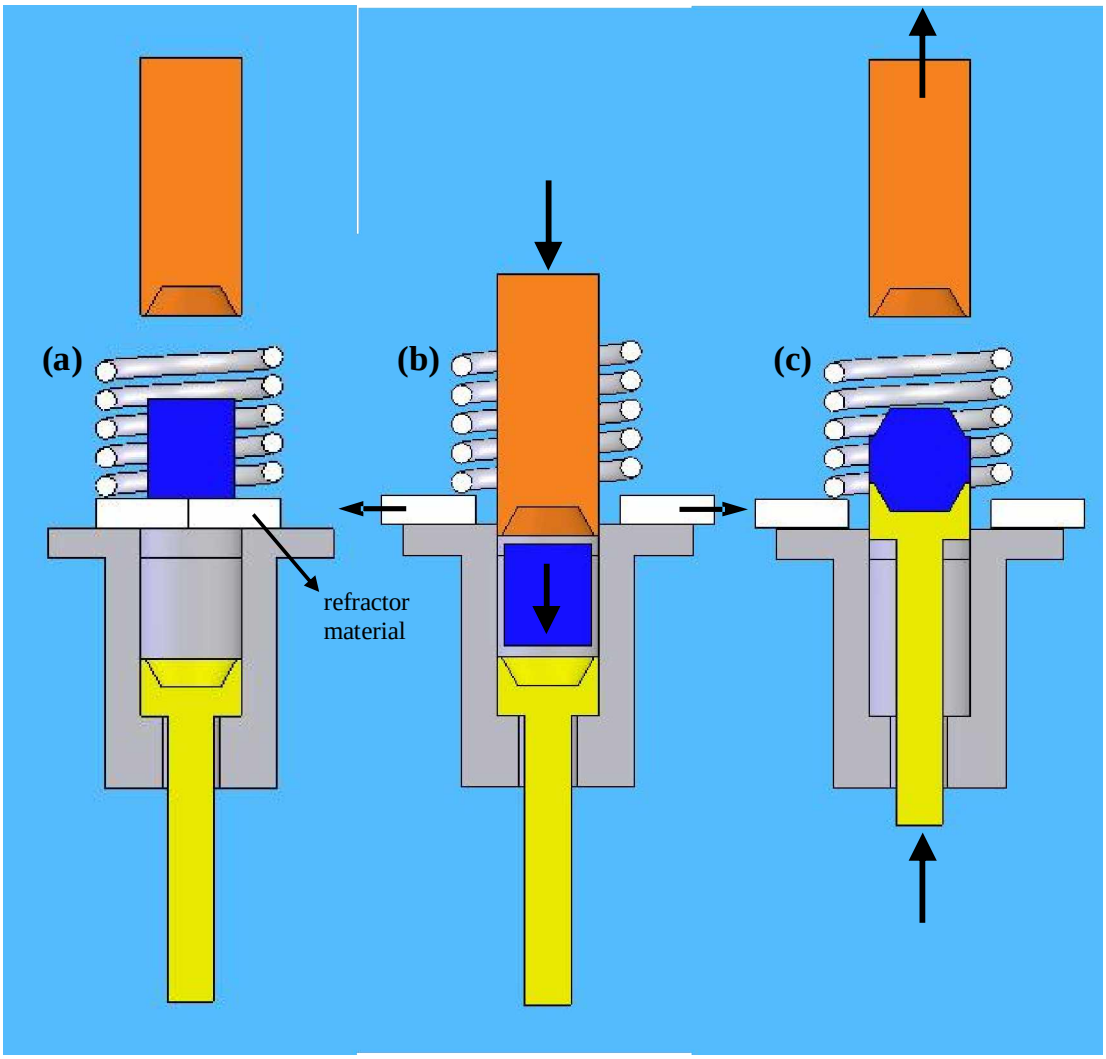

Figure 4.12. Simulation of the second method thixoforming experiment designed with Solid Works 3D software a) reheating of as-cast billet, b) forging of semi-solid billet, c) ejecting of the thixoformed sample

In the experimental study, the as-cast billets were cut into small billets with diameter of 30 mm and height of 35 mm. 3 mm diameter and 15 mm deep hole was drilled on the center of the surface of the billets. The temperature of the billet during the reheating route was controlled by a K-type thermocouple inserted on these holes of the billets and displayed via a digital thermometer.

During the experimental study, the dies were preheated by a ring heater in order to minimize the temperature drop during semi-solid billet was in contact with lower half. The

ring heater was fixed to the main die and the temperature was controlled by a closed circuit controlling unit composed of a digital thermometer and a K-type thermocouple as shown in Figure 4.13. The dies were preheated to 350˚C for AA6082 as-cast billets because low amount of liquid phase presented in the microstructure of this alloy in the semi-solid temperature lead poor die filling especially in the experiments with the second die group. On the other hand, A357 as-cast billets showed excellent die filling property in the experiments with both die groups at the preheating temperature of 300˚C.

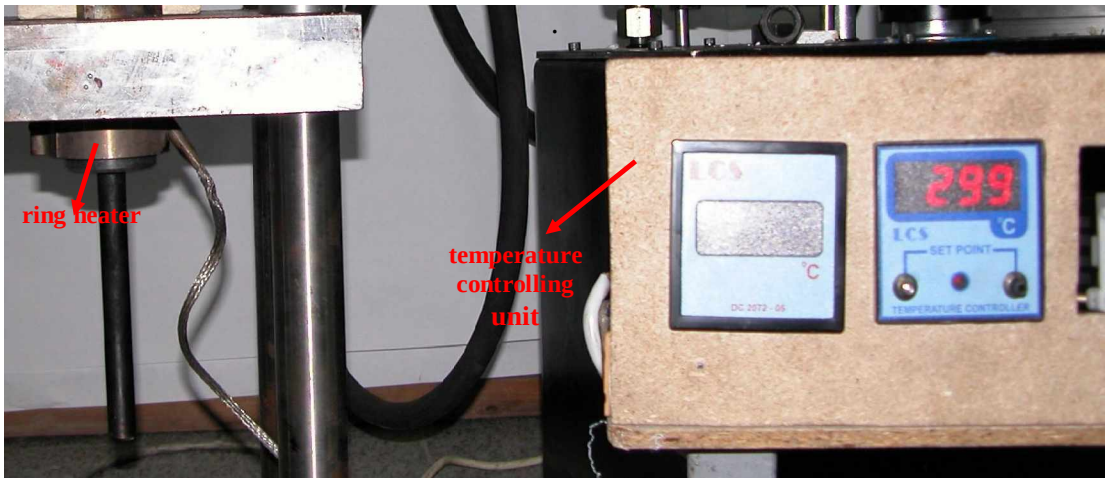

Figure 4.13. Photo of the ring heater and temperature controlling unit

The cavity of the second die group is deeper comparing to first die group and in order to ease the flow of the semi-solid material through the second die group, the surface of dies were coated with boron nitride spray. In addition, lower and upper half dies of the both die groups were in contact with main die during forging and ejecting practices of the thixoforming experiment, so graphite oil layer was coated on the surface of dies to ease the moving of the dies and ejection of the material.

As shown in Figure 4.14, thixoforming unit was composed of a hydraulic power unit, a hydraulic press, an induction furnace (induction coil), die group (upper half die, lower half die, main die), a ring heater and temperature controlling systems (K-type thermocouples, digital thermometers).

Figure 4.14. A photo of the thixoforming unit

Parameters used in thixoforming experiments are given in Table 4.6. Codes were given for each condition to ease designation.

4.2.4. Metallographic Study

All metallographic studies were carried out in TÜBİTAK MAM Alloy Development Laboratory. Three sets of metallographic studies were carried out. In the first set, the as-cast billets obtained by cooling slope casting process were cut into smaller billets as shown in Figure 4.15. All samples were polished using 500, 1000 and 2000 grinding papers, followed by 3 micron diamond paste and finally polished to ¼ micron by using 'Silco' liquid (silica powder suspended in water) with Struers polishing machine. After this, samples were chemically etched with 0.5 ml HF in 100 ml water for 20 seconds. Micrographs from samples were captured using an Olympos camera driven by Spot Insight QE software from three parts; center, mid-radial and edge (Figure 4.15).

Table 4.6 Parameters used in thixoforming experiments

Sample Code	Alloy	As-cast billet code	Holding temperature (˚C)	Holding time (minute)	Die group	Die temperature (˚C)
P1	A357	CA4	580	5	First	300
P2	A357	CA4	580	15	First	300
P3	A357	CA5	580	5	First	300
P4	A357	CA5	580	15	First	300
P5	AA6082	C7	640	5	First	350
P6	AA6082	C7	640	15	First	350
P7	A357	CA4	580	5	Second	300
P8	A357	CA4	580	15	Second	300
P9	A357	CA5	580	5	Second	300
P10	A357	CA5	580	15	Second	300
P11	AA6082	C7	640	5	Second	350
P12	AA6082	C7	640	15	Second	350

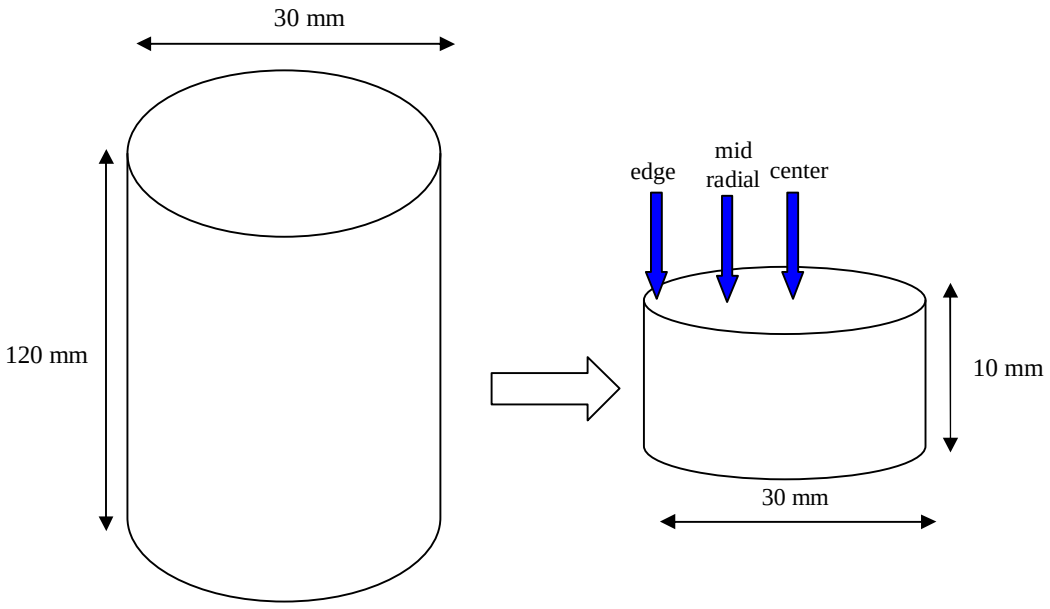

Figure 4.15. Scheme for ingot machining and location of samples for metallographic characterization.

In the second set of studies, the reheated billets were polished and etched in order to investigate microstructural evolution. In the last set of experiments thixoformed samples were polished and etched to investigate microstructure and microhardness of the forged billets. The Vickers hardness of the sample was measured under 500 gf load and 15 sn dwelling time with Future-Tech micro hardness tester before and after T6 heat treatment in order to investigate the effect of heat treatment.

4.2.5. T6 Heat Treatment

To improve the mechanical properties of thixoformed samples, T6 heat treatment practices were performed for samples made of A357 and AA6082 alloys according to ASTM B-917M-01 standard [71]. T6 heat treatment was started with solution hardening process with heating and isothermal holding the samples in the resistance furnace. After quenching the samples, age hardening process was carried out by reheating and isothermal holding. Final step was the cooling down of the samples at room temperature. T6 heat treatment conditions for A357 and AA6082 alloys are given in Table 4.7.

Table 4.7. T6 heat treatment conditions for A357 and AA6082 alloys

Alloy	Holding temperature in solution hardening practice (˚C)	Holding time in solution hardening practice (hours)	Holding temperature in age hardening practice (˚C)	Holding time in age hardening practice (hours)
A357	538	10	171	6
AA6082	560	1	185	6

4.2.6. Tensile Test

For tensile test, circular tensile specimens were machined following ASTM E 8M-04 standard from the samples thixoformed in second die group (coded as P7-P12) as shown in Figure 4.16 [72]. The specimens had gage length of 24 mm and diameter of 4 mm. The tensile tests were performed in KOSGEB with Testometric Micro-500 tensile test device

and tensile strength, yield strength and elongation to fracture properties of the specimens were obtained.

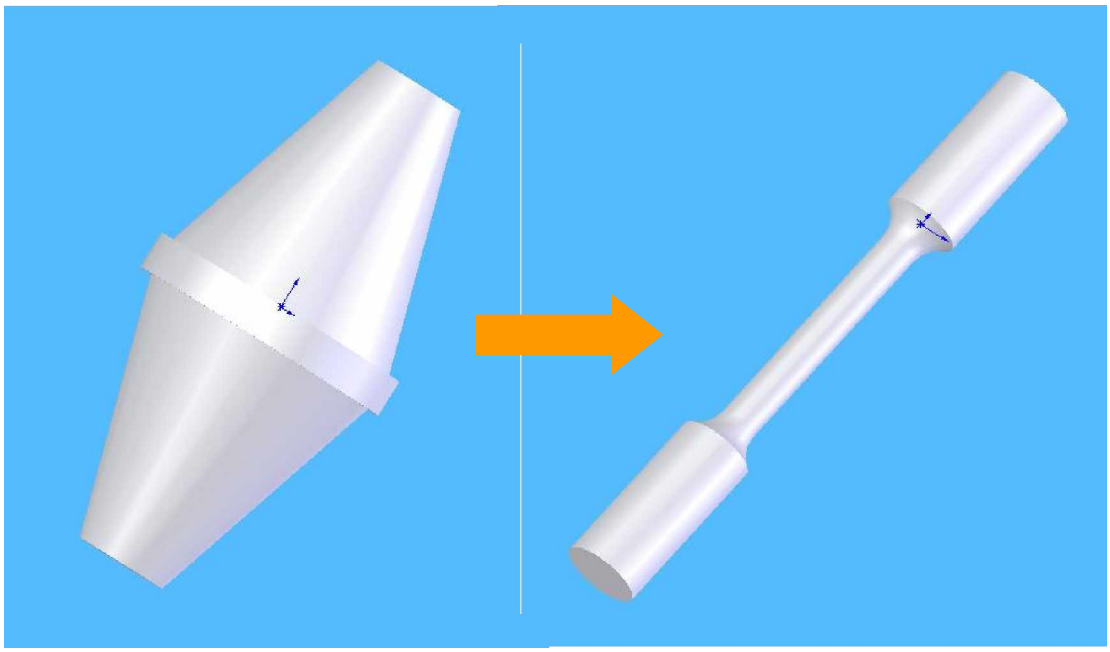

Figure 4.16. Sample thixoformed with second die group and tensile test specimen machined from this sample (designed with Solid Works 3D software)

5. RESULTS AND DISCUSSION

5.1. As-received Materials

Figure 5.1 shows the microstructures of as-received A357 and AA6082 aluminum ingots before cooling slope casting process. The microstructure of A357 alloy presents the conventional dendrite structure of α phase surrounded by the eutectic. Due to the fact that AA6082 alloy contains low alloy composition, there are less second phase in AA6082 alloy comparing to A357 alloy and this second phase including Al-Fe(Mn)-Si intermetallic compound causes discontinuous structure in dendrite boundaries as shown in Figure 5.1b.

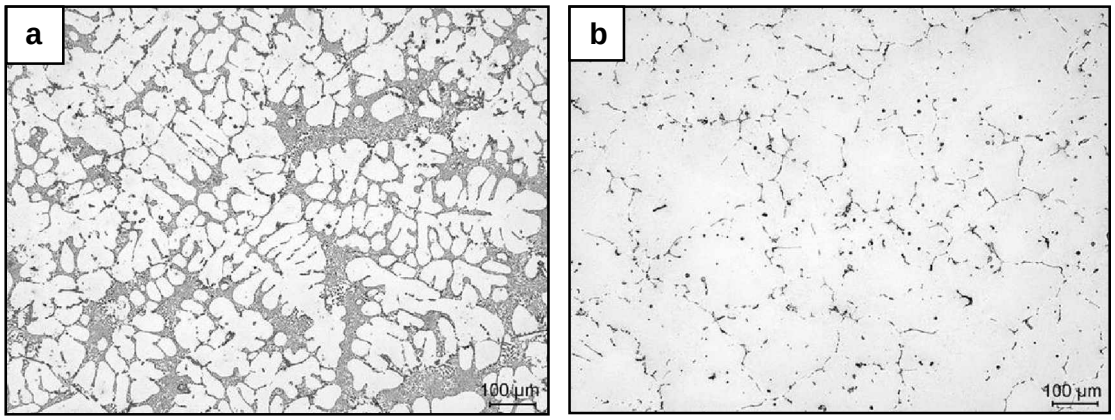

Figure 5.1. Microstructures of a) as-received A357 alloy ingot, b) as-received AA6082 alloy billet

5.2. As-cast Billets after Cooling Slope Casting Process

Figure 5.2 shows the microstructures of all as-cast billets obtained when the molten metal cast via the cooling slope into a metal mold. All of the pictures were taken from mid-radial position of as-cast billets. The dendritic morphology of the as-received material turned into non-dendritic and equiaxed fine grains after cooling slope casting practice for casting temperatures and pouring distances shown in Figure 5.2.

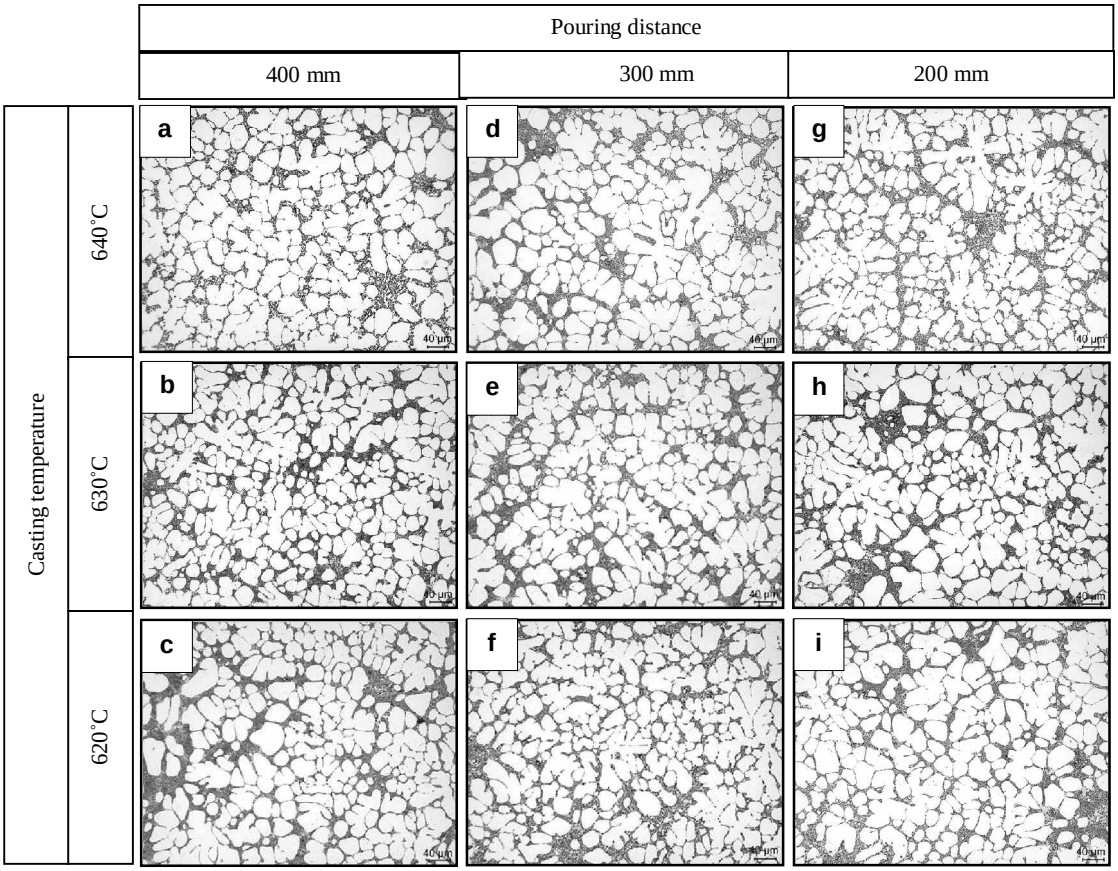

Figure 5.2. Micrographs of A357 as-cast billets obtained after cooling slope casting process a) CA1, b) CA2, c) CA3, d) CA4, e) CA5, f) CA6, g) CA7, h) CA8, i) CA9.

The microstructures of the as-cast billets were not uniformly distributed. It is possible to detect three areas in the cross section of the billets. First, there is a narrow dendritic area which was in contact with the mould wall, then, a rosette morphology area in mid-radial position and finally at the centre of the ingot a spheroidal morphology. Figure 5.3 shows examples of such microstructures in such a billet. These different morphologies were resulted from different cooling behaviors in such areas.

Figure 5.4 shows the initial microstructure produced by pouring A357 alloy at 620^{o}C, 630^{o}C, 640^{o}C down the cooling slope for a distance of 200 mm into a steel mould. The resulting α–Al shape changed from spheroidal to rosette and then to dendritic, with increase of pouring temperature as shown in Figure 5.4. It was found that low superheat

pouring temperature promotes the formation of fine spheroidal grains in cooling slope casting process.

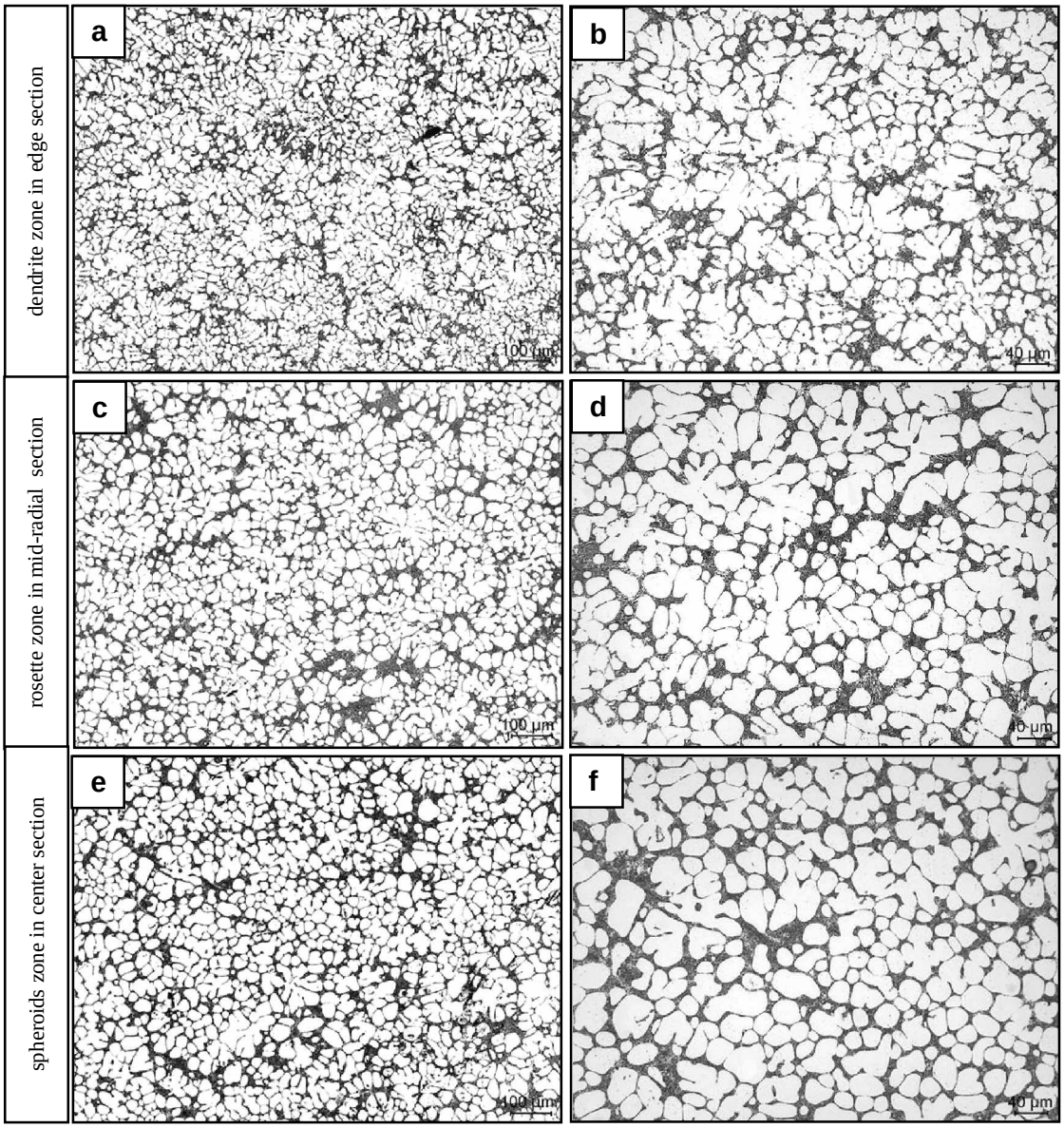

Figure 5.3. Microstructure zones of CA2 codedA357 as-cast billets poured at 630˚C through 400 mm cooling length a) and b) edge sections which were in contact with the mould wall, c) and d) mid-radial sections, e) and f) centre sections.

Figure 5.5 shows the corresponding microstructures after casting A357 alloy at 640^{o}C through poring distances of 200, 300 and 400 mm. As the pouring distance

increased from 200 to 400 mm, the microstructure has changed from dendrite to spheroidal morphology as shown in Figure 5.5. Longer pouring distance promoted the formation of spheroidal α–Al morphology because as the contact length increased, the contact time of the melt with the cooling slope increased and this increases lead much more crystals to generate on the plate and to separate from it.

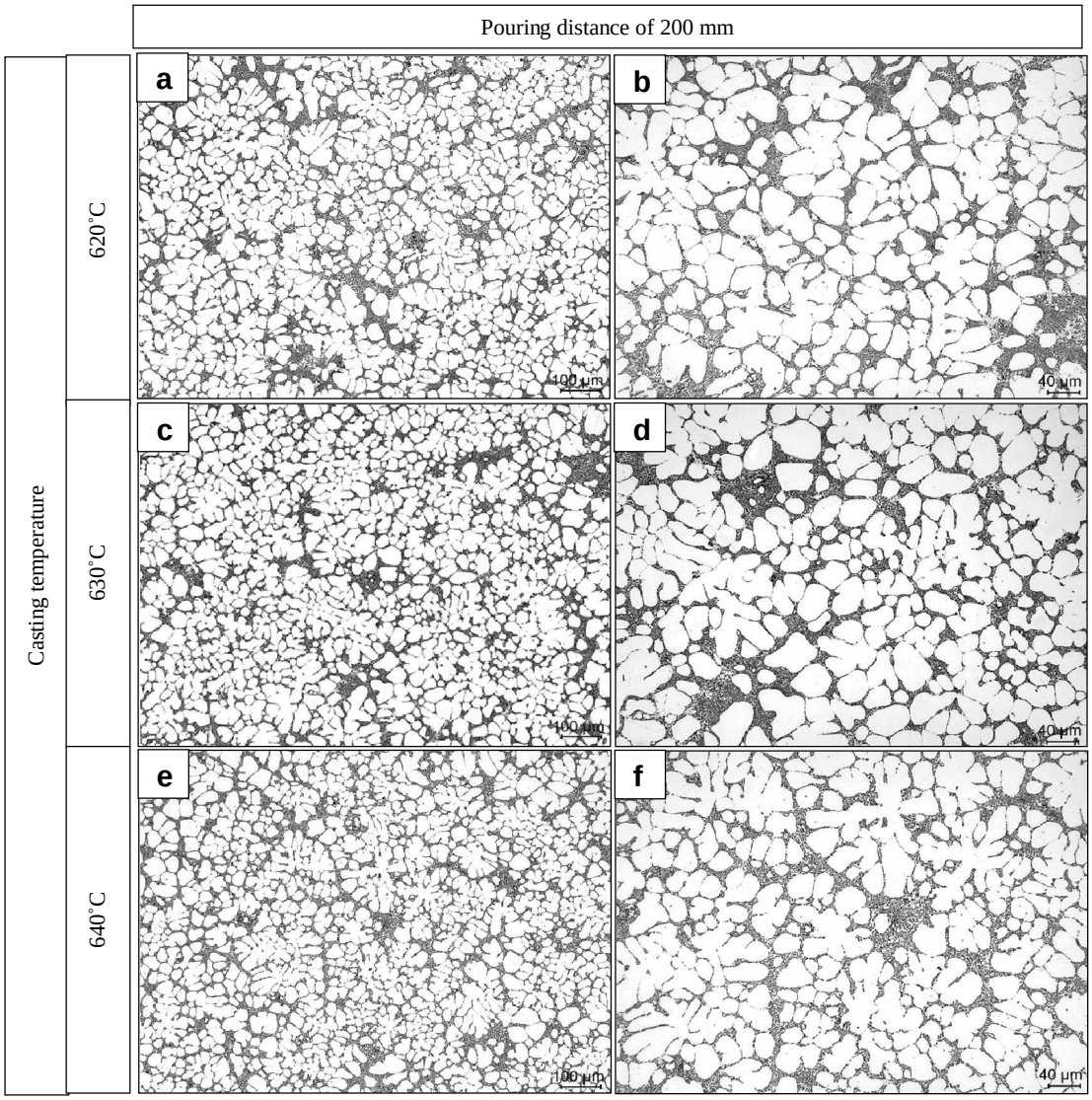

Figure 5.4. α–Al particle morphologies of as-cast billets obtained after pouring through 200 mm on the cooling slope into a steel mould a) and b) globular from low temperature pouring (at 620 °C), c) and d) rosette-like from medium temperature pouring (630°C), e) and f) dendritic structure from high temperature pouring (640°C).

Figure 5.6 shows the microstructures of all as-cast billets obtained after cooling slope casting process of AA6082 alloy. All of the pictures were taken from mid-radial position of as-cast billets. After cooling slope casting process, discontinuous structure presented in the dendrite boundaries turned into more homogeneous morphology consisting of equiaxed grains and the grain boundaries became clearer as shown in Figure 5.6.

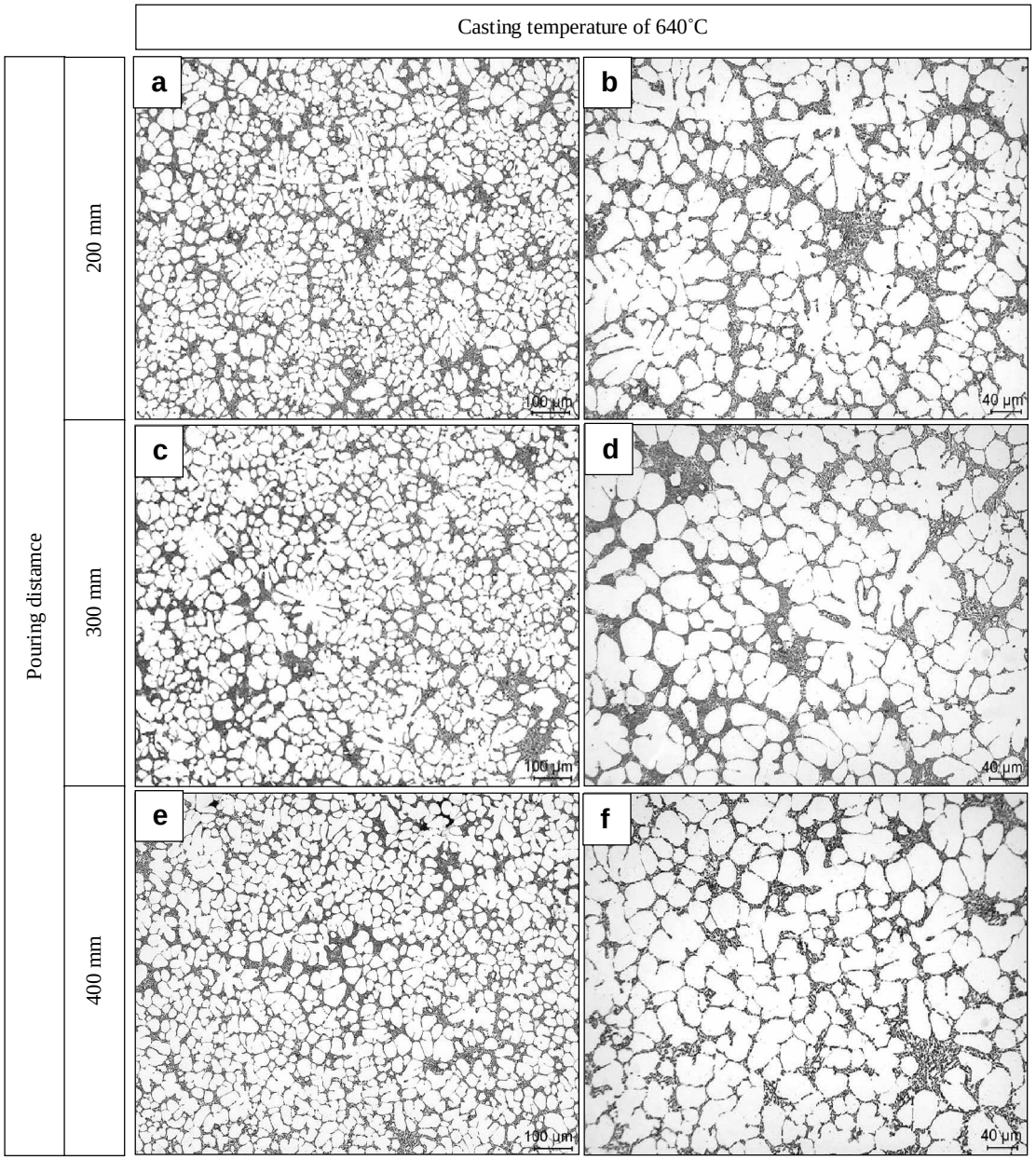

Figure 5.5. Microstructures of as-cast billets poured at 640°C through cooling distances of a) and b) 200 mm, c) and d) 300 mm, e) and f) 400 mm.

Figure 5.7 shows the effect of pouring distance on the formation of equiaxed grains after cooling slope casting process of AA6082 alloy. As the pouring distance was increased from 200 to 400 mm, the grains became more equiaxed and the amount of second phase in the grains boundaries increased as shown Figure 5.7. This improvement is directly related with the increasing contact time of the molten metal on the cooling plate as mentioned before for A357 alloy.

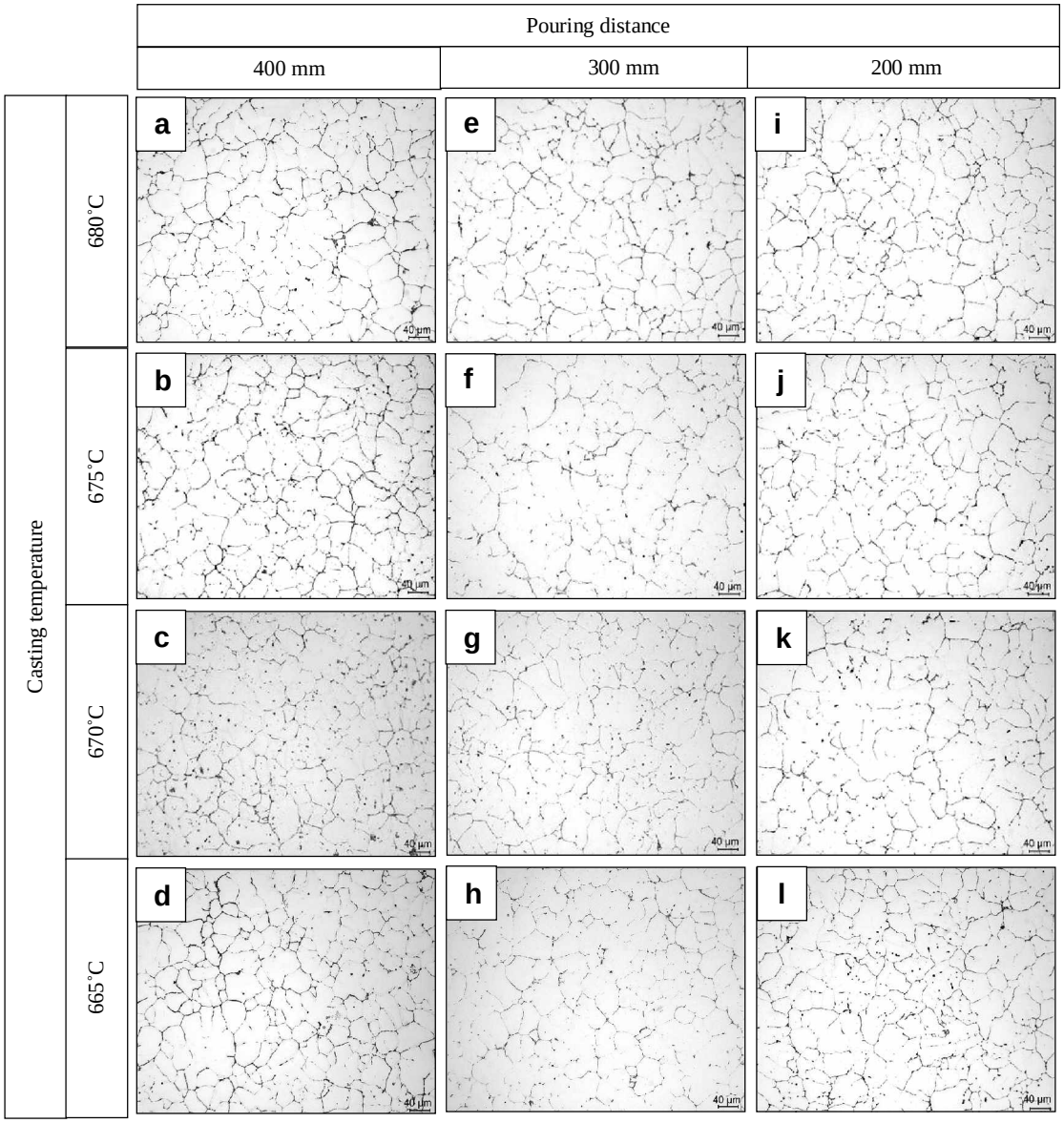

Figure 5.6. Microstructures of AA6082 as-cast billets obtained after cooling slope casting process a) C1, b) C2, c) C3, d) C4, e) C) 5, f) C6, g) C7, h) C8, i) C9, j)C10, k) C11, l) C12.

Figure 5.8 shows the effect of pouring temperature on the morphology of as-cast billets. Figure 5.8a to Figure 5.8c show the non-uniform microstructure of the C1 coded as cast billet obtained after pouring at 680^{o}C down the cooling slope for a distance of 400 mm. As the pouring temperature was decreased from 680^{o}C to 665^{o}C, the grains became more equiaxed and the morphology became more uniform as shown in Figure 5.8d to 5.8f.

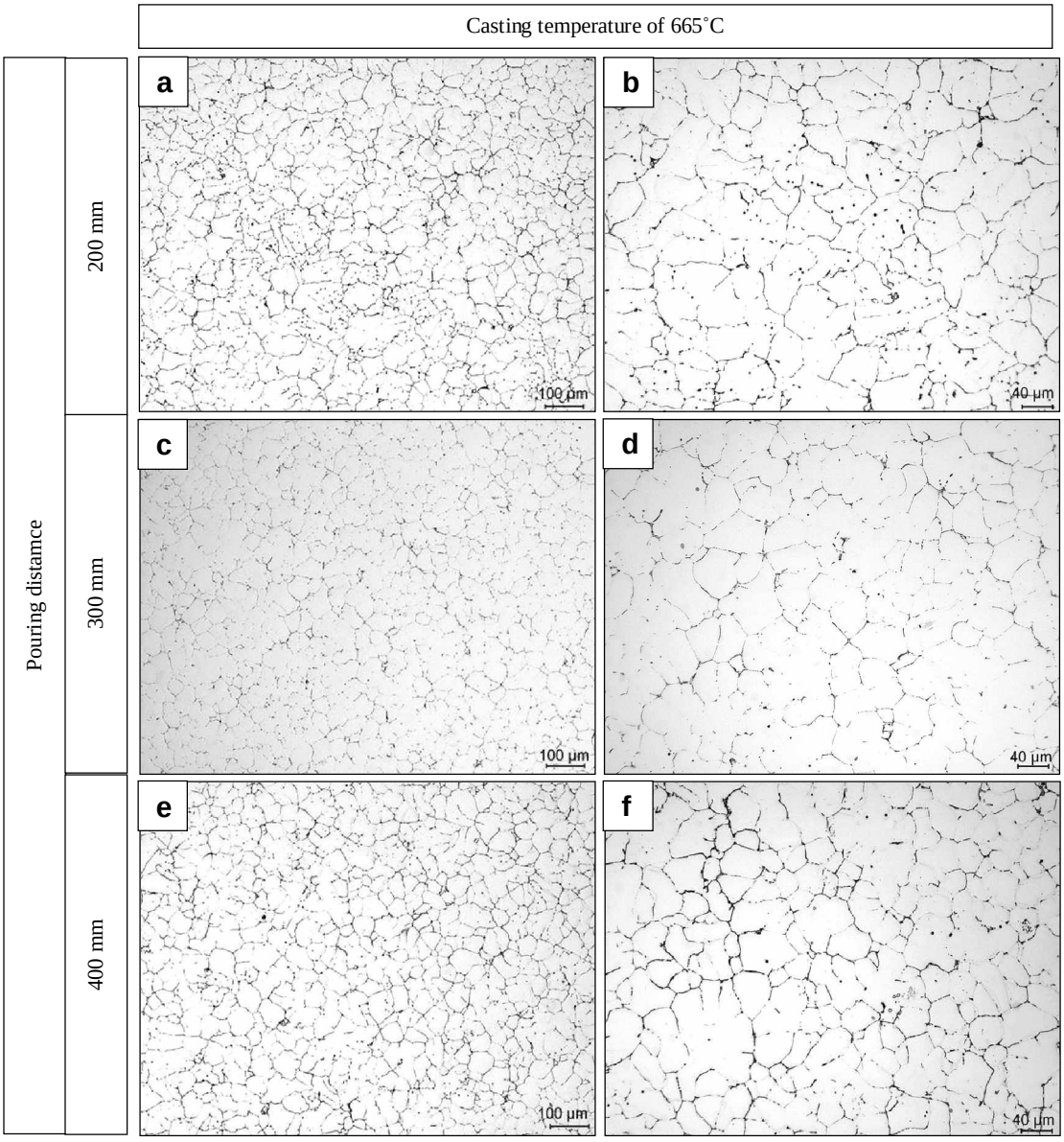

Figure 5.7. Microstructures of AA6082 as-cast billets poured at 665°C through cooling distances of a) and b) 200 mm, c) and d) 300 mm, e) and f) 400 mm.

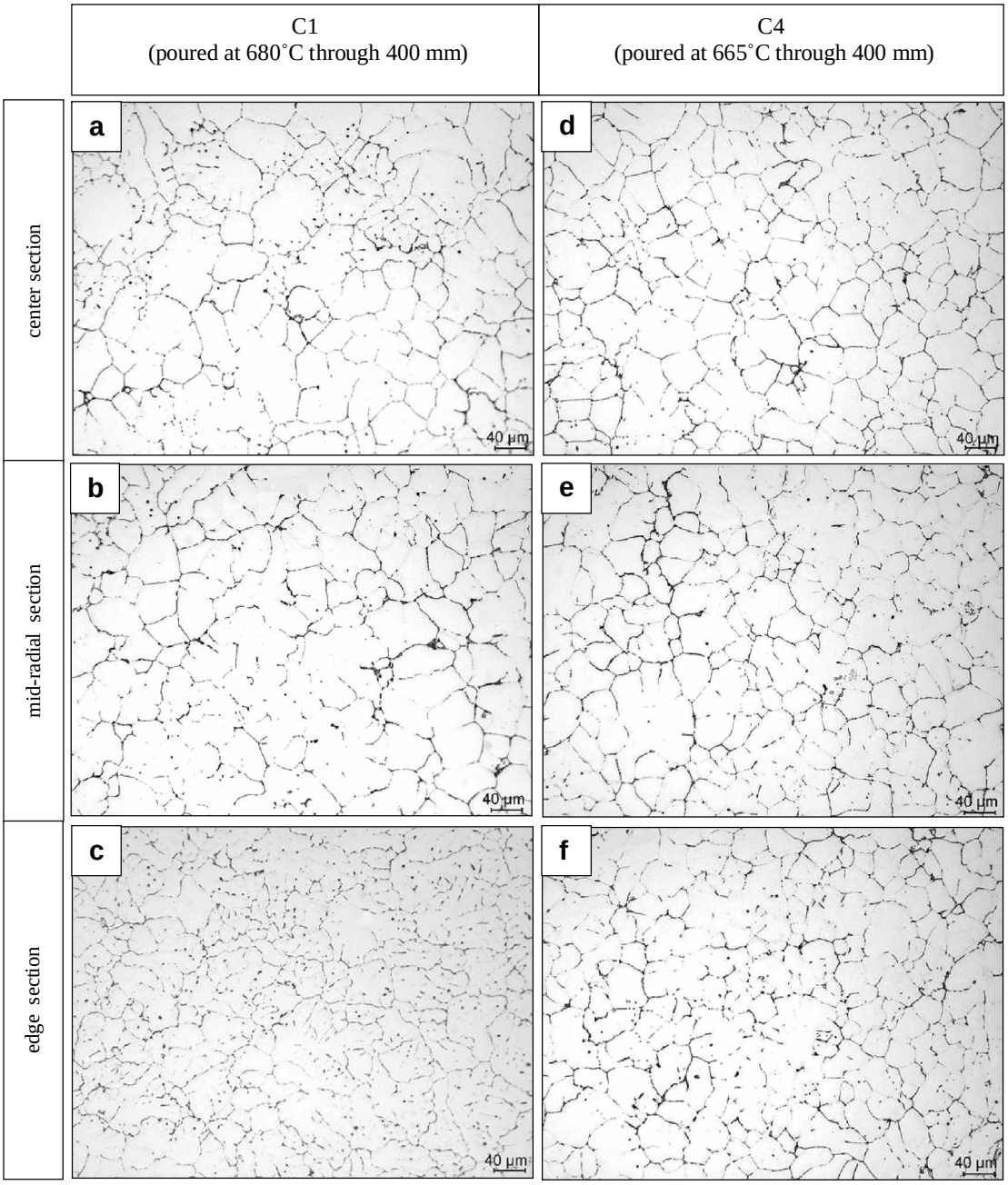

Figure 5.8. Microstructure zones of C1 and C4 coded AA6082 as-cast billets poured through 400 mm cooling length at a), b) and c) 680˚C, d), e) and f) 665˚C

5.3. Reheated Billets after Isothermal Holding

Billets with diameter of 30 mm and height of 35 mm from A357 and AA6082 aluminium alloy ingots produced by cooling slope cast into steel mould were heated in an

induction furnace and isothermally held at 580 °C and 640 °C. The photos of the reheated and isothermally held A357 as-cast billets are shown in Figure 5.9. The bottom of the samples deformed and changed its shape while being held in 580˚C for various holding times. The bottom part of the reheated semi-solid sample could not resist the weight of the remaining upper part and that's why the cylindrical shape of the as-cast billets changed into elephant foot like shapes.

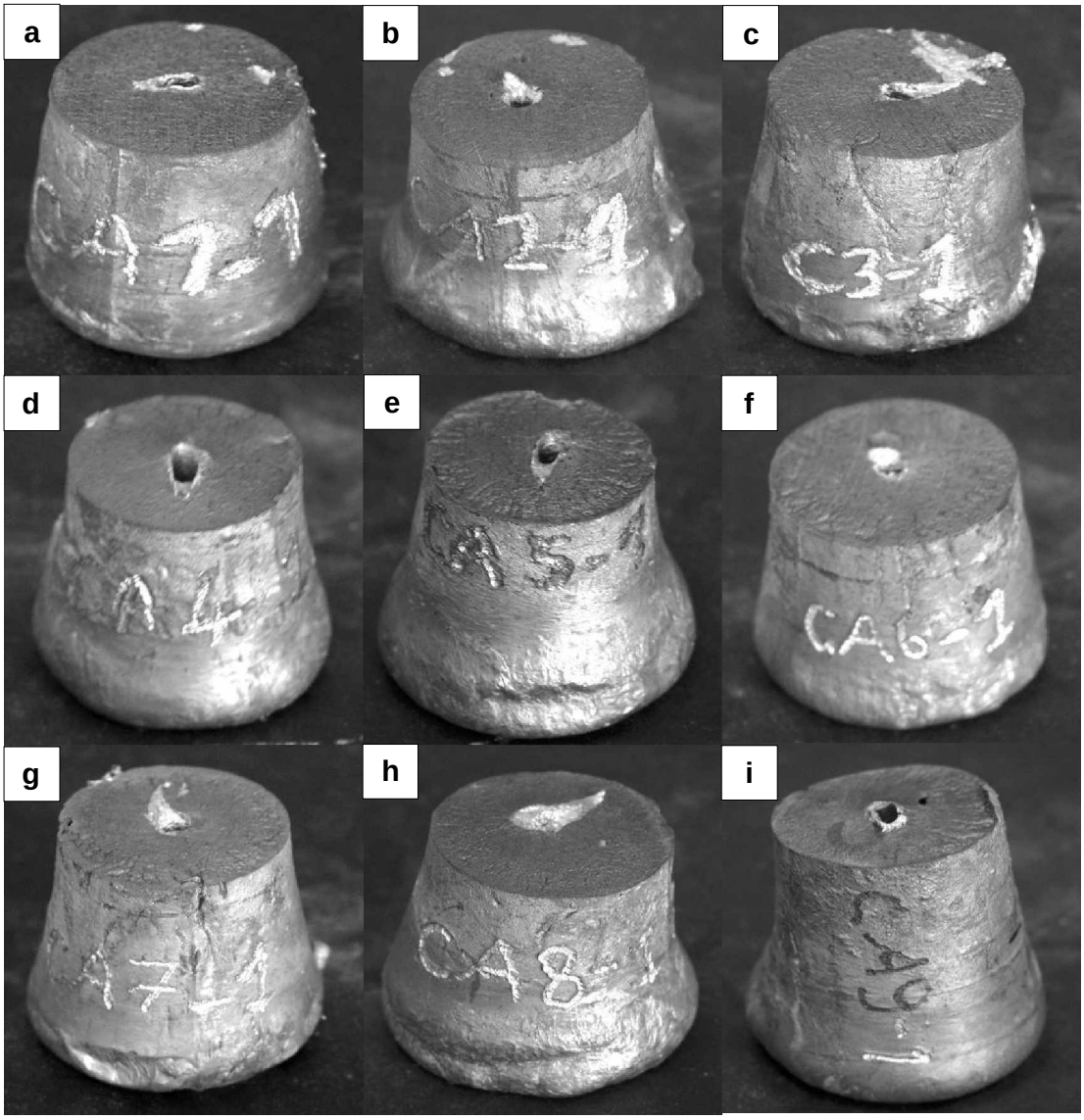

Figure 5.9. Photos of reheated A357 as-cast billets obtained after isothermally held at 580˚C for 5 minutes a) CA1.1, b) CA2.1, c) CA3.1, d) CA4.1, e) CA5.1, f) CA6.1, g) CA7.1, h) CA8.1, i) CA9.1.

For A357 alloy, at 580˚C, the eutectic constituent is remelted while almost all the primary phase remains solid. The samples were isothermally held for 5 and 15 minutes. Representative optical micrographs of the resulting microstructural evolution with increasing isothermal holding time are shown in Figure 5.10 and Figure 5.11. The samples had been heated into the semi-solid range in the induction furnace and then rapidly quenched so are assumed to give a reasonable indication of the evolution of semi-solid microstructure at the temperature of holding. Non-uniform morphology of the as-cast billets turned into uniformly distributed globular particles after reheating and isothermal holding practices.

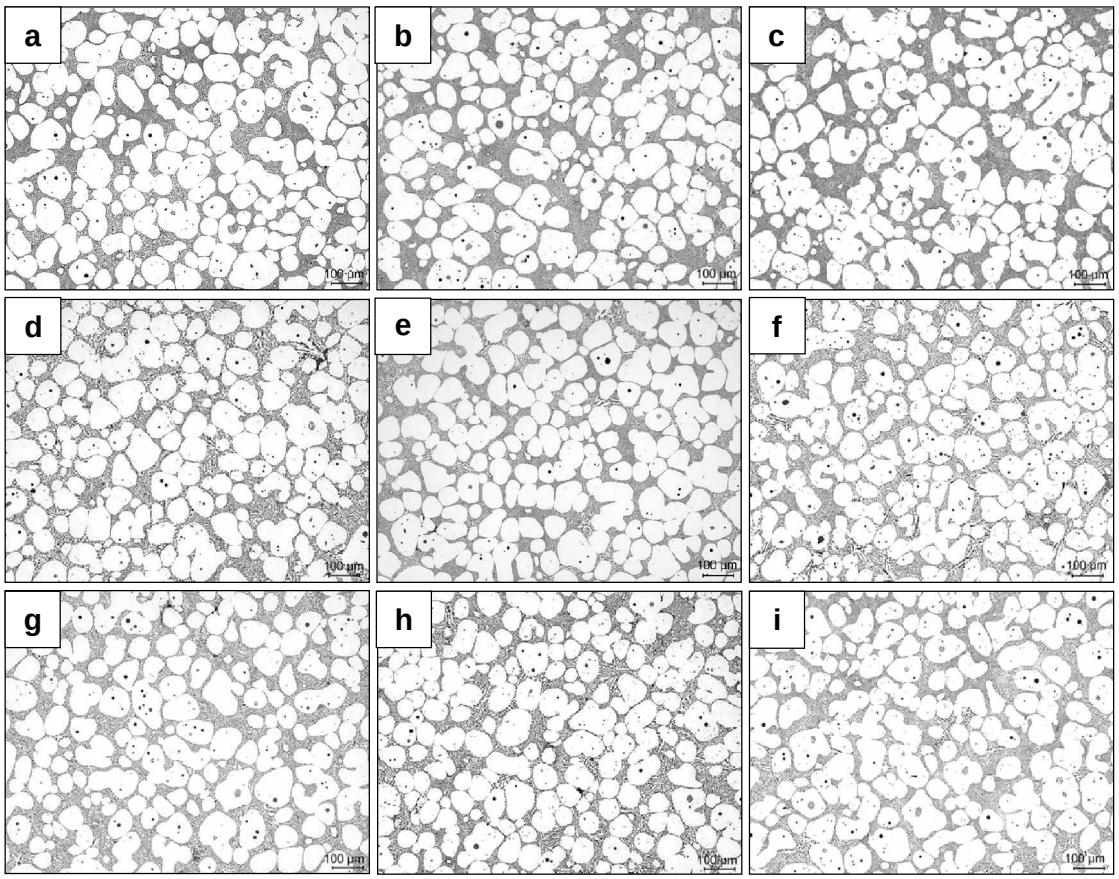

Figure 5.10. Microstructures of reheated A357 as-cast billets obtained after isothermally held at 580˚C for 5 minutes a) CA1.1, b) CA2.1, c) CA3.1, d) CA4.1, e) CA5.1, f) CA6.1, g) CA7.1, h) CA8.1, i) CA9.1.

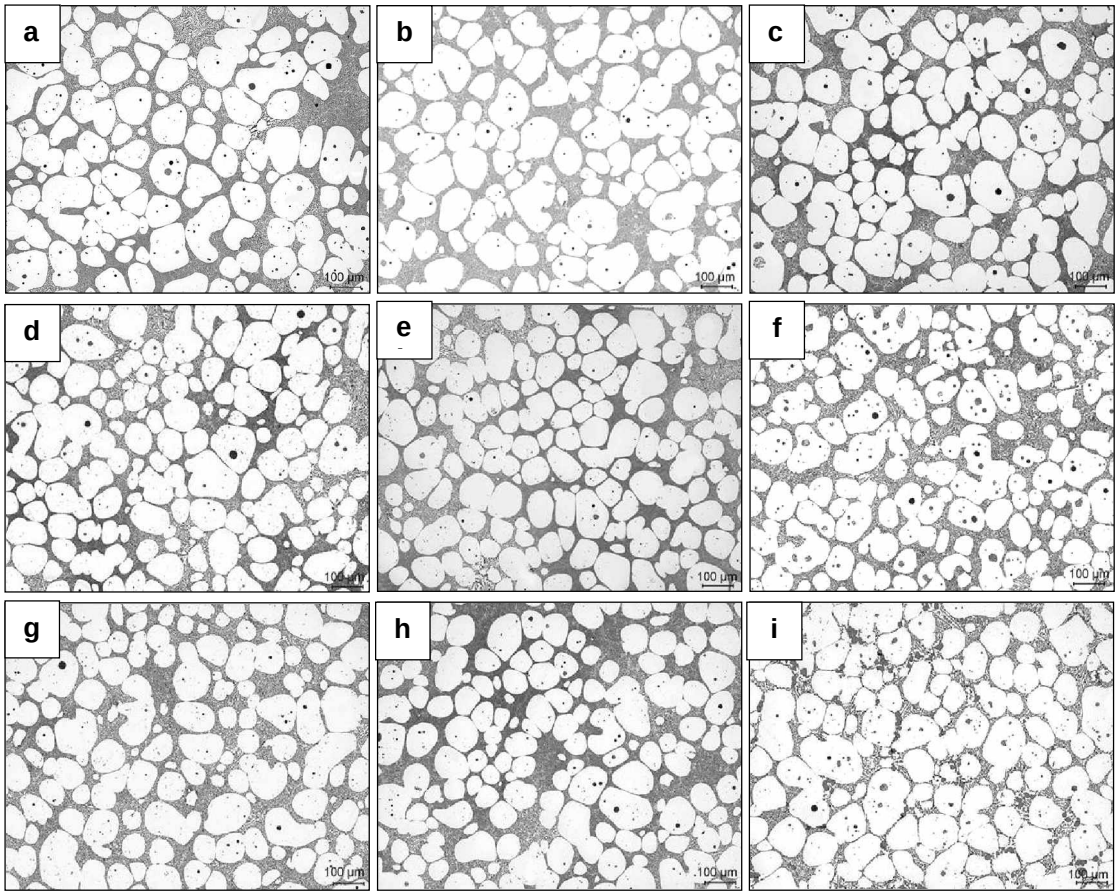

Figure 5.11. Microstructures of reheated A357 as-cast billets obtained after isothermally held at 580˚C for 15 minutes a) CA1.2, b) CA2.2, c) CA3.2, d) CA4.2, e) CA5.2, f) CA6.2, g) CA7.2, h) CA8.2, i) CA9.2.

Figure 5.12 shows the microstructural evolution of the CA5 coded as-cast billet poured at 630^{o}C with 300 mm pouring distance then isothermally held at 580^{o}C. After 5 minutes of isothermal holding, the fine dendritic-rosette structure from cooling slope casting process developed into a clearly globular microstructure and the α–Al phase has completely transformed to spheroidal elements. After 15 minutes of isothermal holding, the microstructure became quite globular and uniform in particle size. At the end of the process, spheroidal α-Al particles were uniformly distributed in the quenched liquid matrix.

Figure 5.13 shows the corresponding microstructures after isothermal holding at 580 °C of the CA1 coded as-cast billets obtained with casting temperature of 640^{o}C and

pouring distance of 400 mm. As the holding time was increased from 5 minutes to 30 minutes, α-Al particle size and its spheroidicity increased. Microstructural evolution proceeded first with a small increase in the spheroidicity and an increase in the size of α-Al particles. After 15 and 30 minutes of isothermal holding, the microstructure became more globular with an increase in the size of α-Al particles.

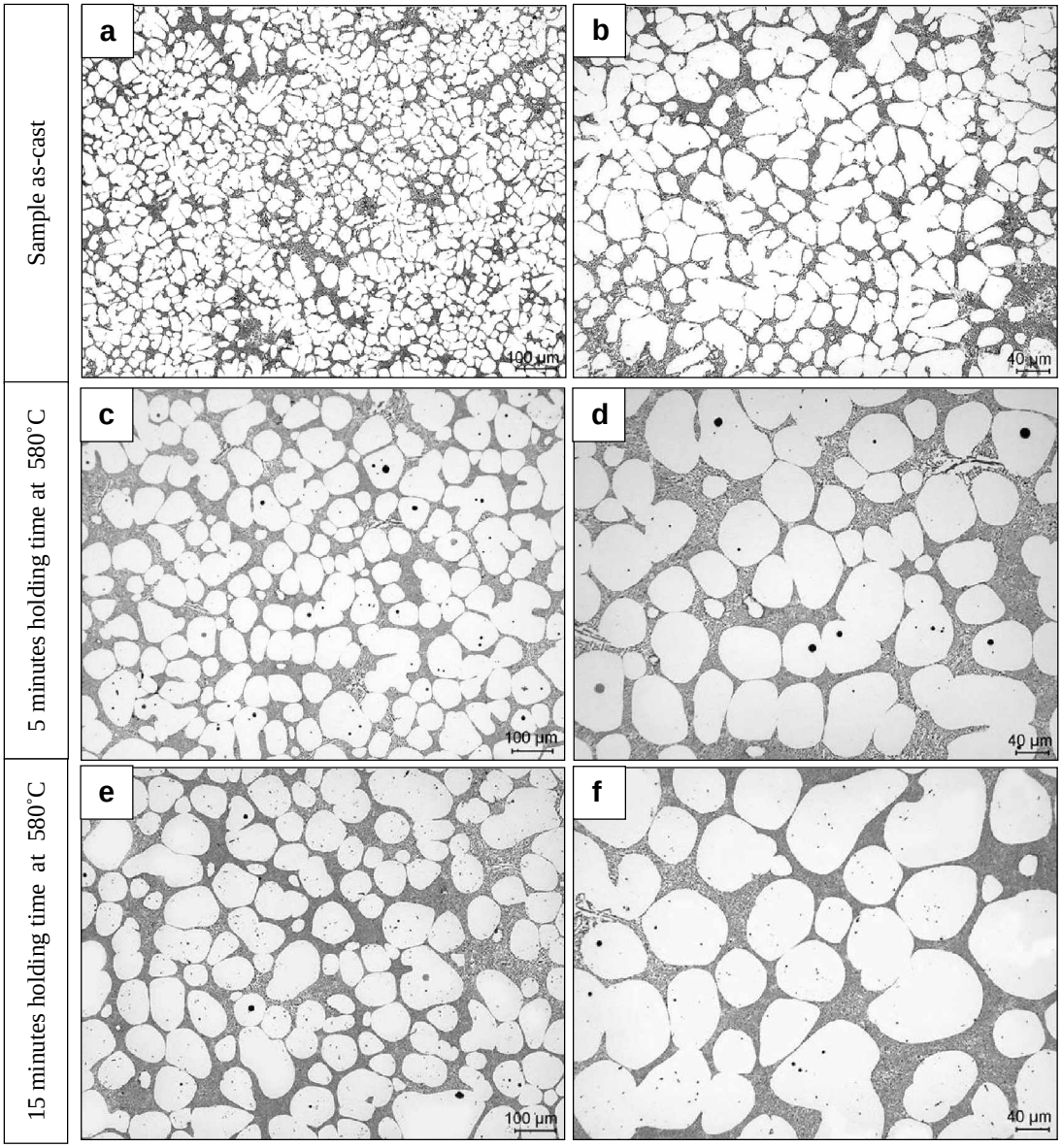

Figure 5.12. Microstructural evolution of the CA5 coded as-cast billets poured at 630°C with 300 mm pouring distance then isothermally held at 580°C for a) and b) 0 minute (CA5), c) and d) 5 minutes (CA5.1), e) and f) 15 minutes (CA5.2)

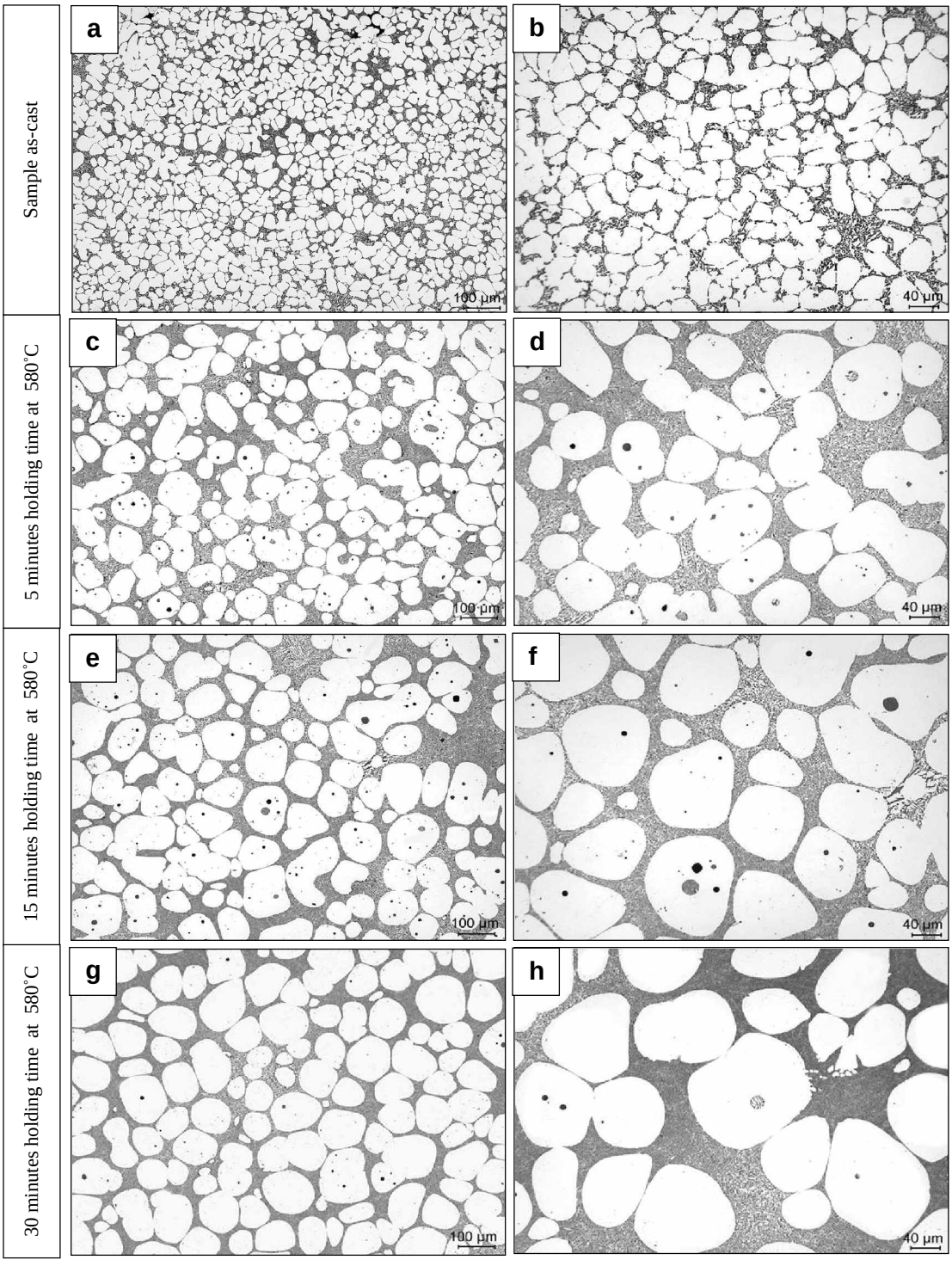

Figure 5.13. Microstructural evolution of the CA1 coded as-cast billets poured at 640°C with 400 mm pouring distance then isothermally held at 580°C for a) and b) 0 minute (CA1), c) and d) 5 minutes (CA1.1), e) and f) 15 minutes (CA1.2), g) and h) 30 minutes (CA1.3)

During isothermal holding in the semisolid state, the equiaxed particles are vulnerable to coalescence and spheroidization. Figure 5.14 shows the growth of particles by coalescence and developing spheroidization after isothermal holding at 580 °C of the CA3 coded as-cast billets obtained with casting temperature of 620°C and pouring distance of 400 mm. The coalescence process is an active mechanism present in the growth of the α-Al particles. As shown in Figure 5.14a, even in a short holding time of 5 minutes in the semi-solid condition, the initial rosette morphology developed a more spheroidal morphology with the developing of coalescence necks. When the holding time was increased to 15 minutes, α-Al particles grew by coalescence mechanism and spheroidicity improved as shown in Figure 5.14b.

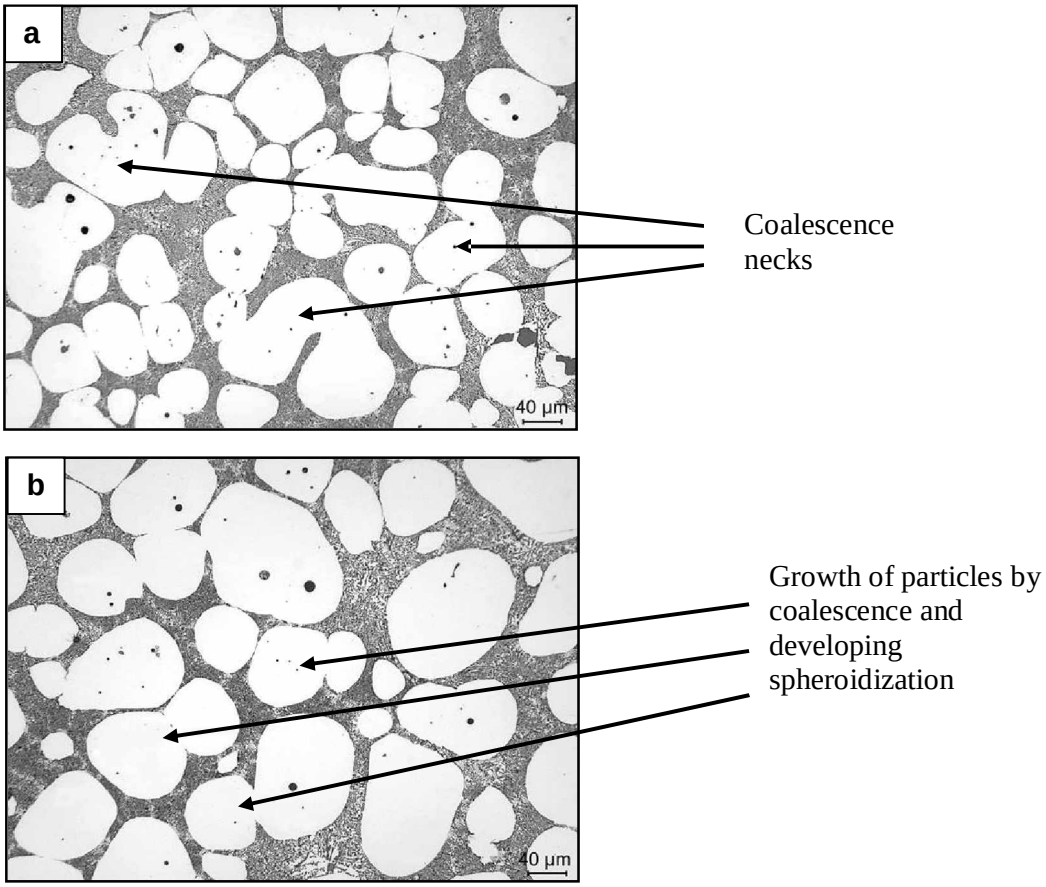

Figure 5.14. The coalescence mechanism and spheroidization process of the CA3 coded as-cast billets poured at 620°C with 400 mm pouring distance then isothermally held at 580°C for a) 5 minutes and b) 15 minutes.

Figure 5.15 shows the photos of the reheated AA6082 as-cast billets. Similar to A357 alloy, AA6082 as-cast billets turned into elephant foot shape and the diameter of bottom part increased after reheating and isothermal holding practices. However, shape and dimension changes were not significant as in A357 alloy due to the low amount of liquid phase formation in the structure.

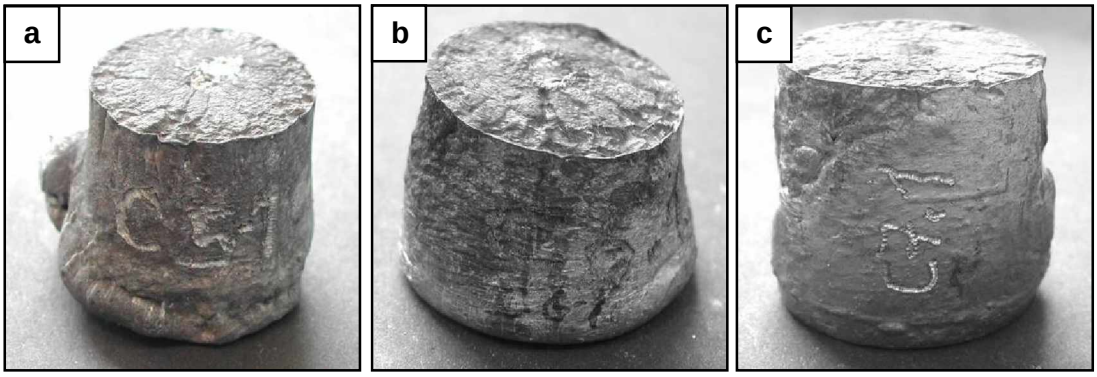

Figure 5.15. Photos of reheated AA6082 as-cast billets obtained after isothermally held at 640˚C for 5 minutes a) C5.1, b) C6.1, c) 7.1

Figure 5.16 and Figure 5.17 show all resulting microstructures formed after reheating of AA6082 as-cast billets to 640˚C and then isothermally held for 5 and 15 minutes. Reheating temperature of 640˚C corresponds to solid fraction of 52 per cent according to Figure 4.2. Different from the micrographs of the reheated A357 billets, the grains were hexagonal and the amount of the remelted eutectic phase was small. This is a frequently occurred difference between cast and wrought aluminum alloys.

Figure 5.18 shows the microstructural evolution of the C4 and C8 coded as-cast billets at 640°C obtained after poured at 665°C. As mentioned in previous section, the initial as-cast microstructures of these billets were different resulted from different pouring distances as shown in Figure 5.18a and Figure 5.18b. But after reheating to 640°C and isothermal holding for 5 and 15 minutes, these two different microstructures turned into similar thixotropic structure as shown from Figure 5.18c to Figure 5.18f. However, hexagonal grains were formed rather than equiaxed and globular particles. These hexagonal grains are frequently formed during heating of wrought aluminum alloys. It is found that, inadequate spheroidization in grain boundaries were resulted from insufficient

amount of remelted eutectic phase in AA6082 alloy and this situation is directly related with the low alloy composition of AA6082 alloys. For example, large amount of remelted eutectic phase formed in the reheated samples of A357 alloy due to the large amount of silicon present in the alloy composition.

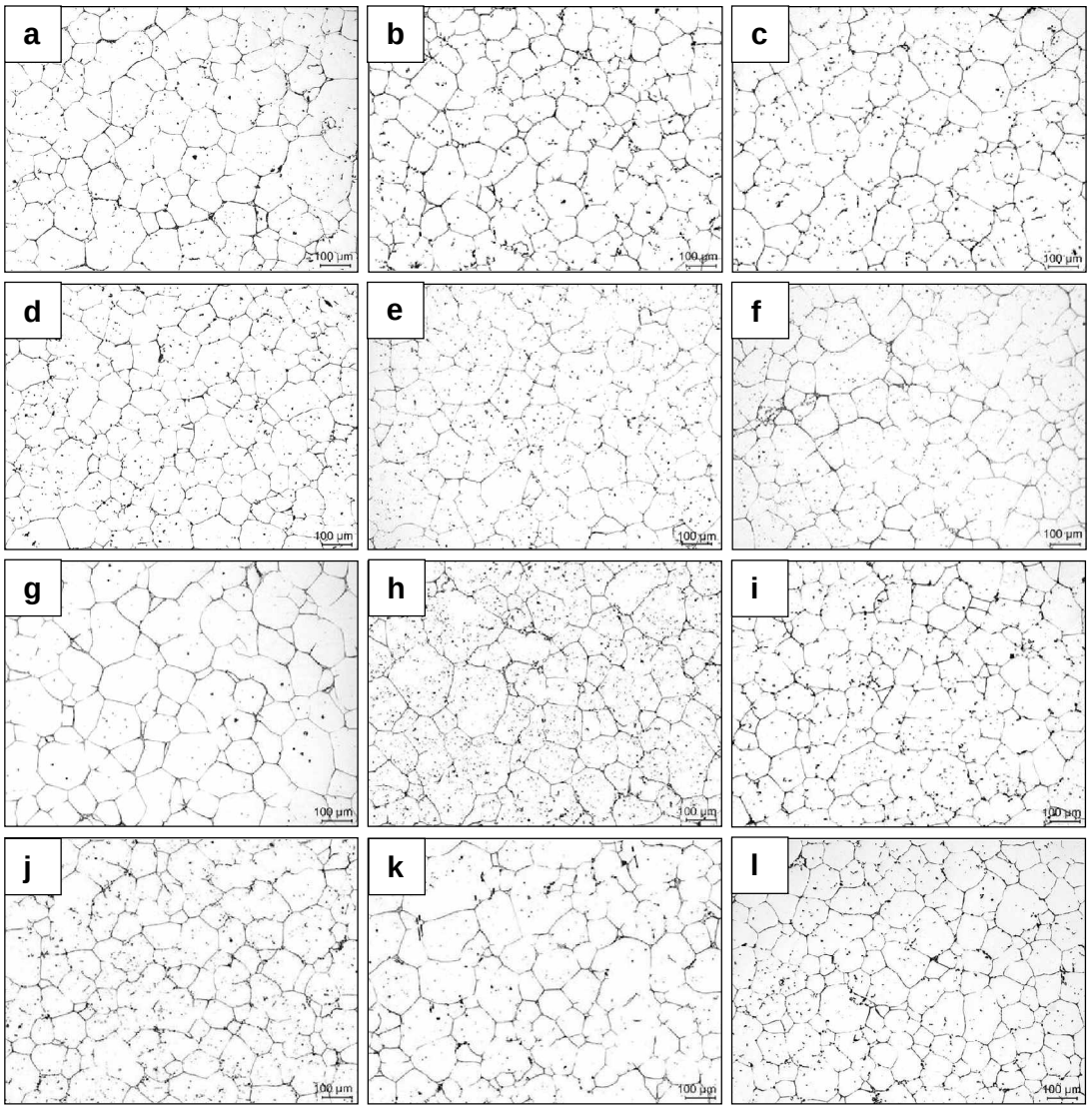

Figure 5.16. Microstructures of reheated AA6082 as-cast billets obtained after isothermally held at 640˚C for 5 minutes a) C1.1, b) C2.1, c) C3.1, d) C4.1, e) C) 5.1, f) C6.1, g) C7.1, h) C8.1, i) C9.1, j) C10.1, k) C11.1, l) C12.1

Figure 5.19 shows the increase in grain size with increase in holding time at 640°C for AA6082 alloy. For thixoforming process, feedstock with coarse grains is not a

preferable situation due to the negative effects on mechanical properties. As the holding time increased from 5 to 15 minutes, the grains became coarser and the grain boundaries became clearer as shown from Figure 5.19c to Figure 5.19f. In addition, the amount of remelted eutectic phase increased as the holding time increased as shown in figure 5.19e and figure 5.19f.

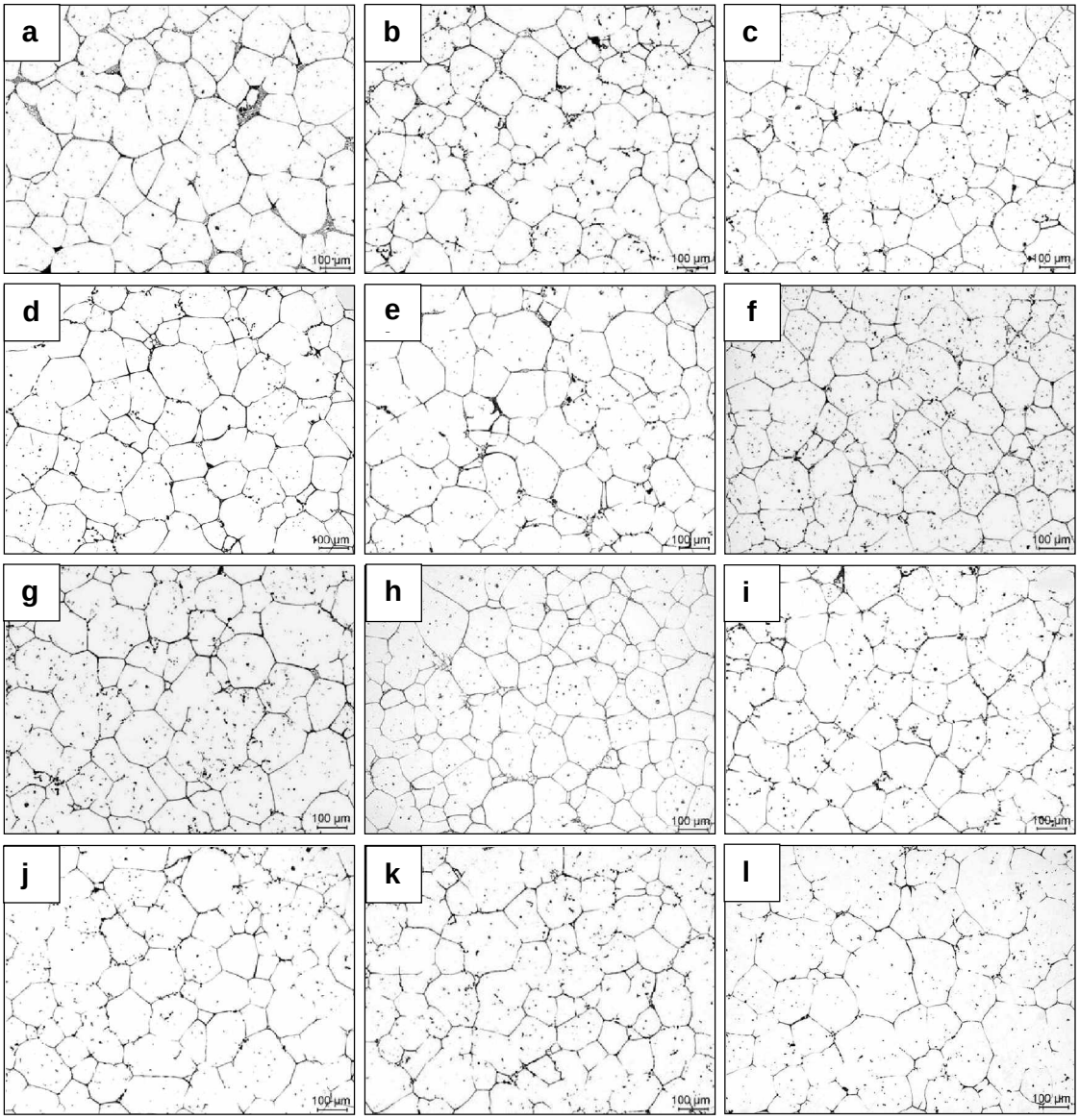

Figure 5.17. Microstructures of reheated AA6082 as-cast billets obtained after isothermally held at 640˚C for 15 minutes a) C1.2, b) C2.2, c) C3.2, d) C4.2, e) C) 5.2, f) C6.2, g) C7.2, h) C8.2, i) C9.2, j) C10.2, k) C11.2, l) C12.2

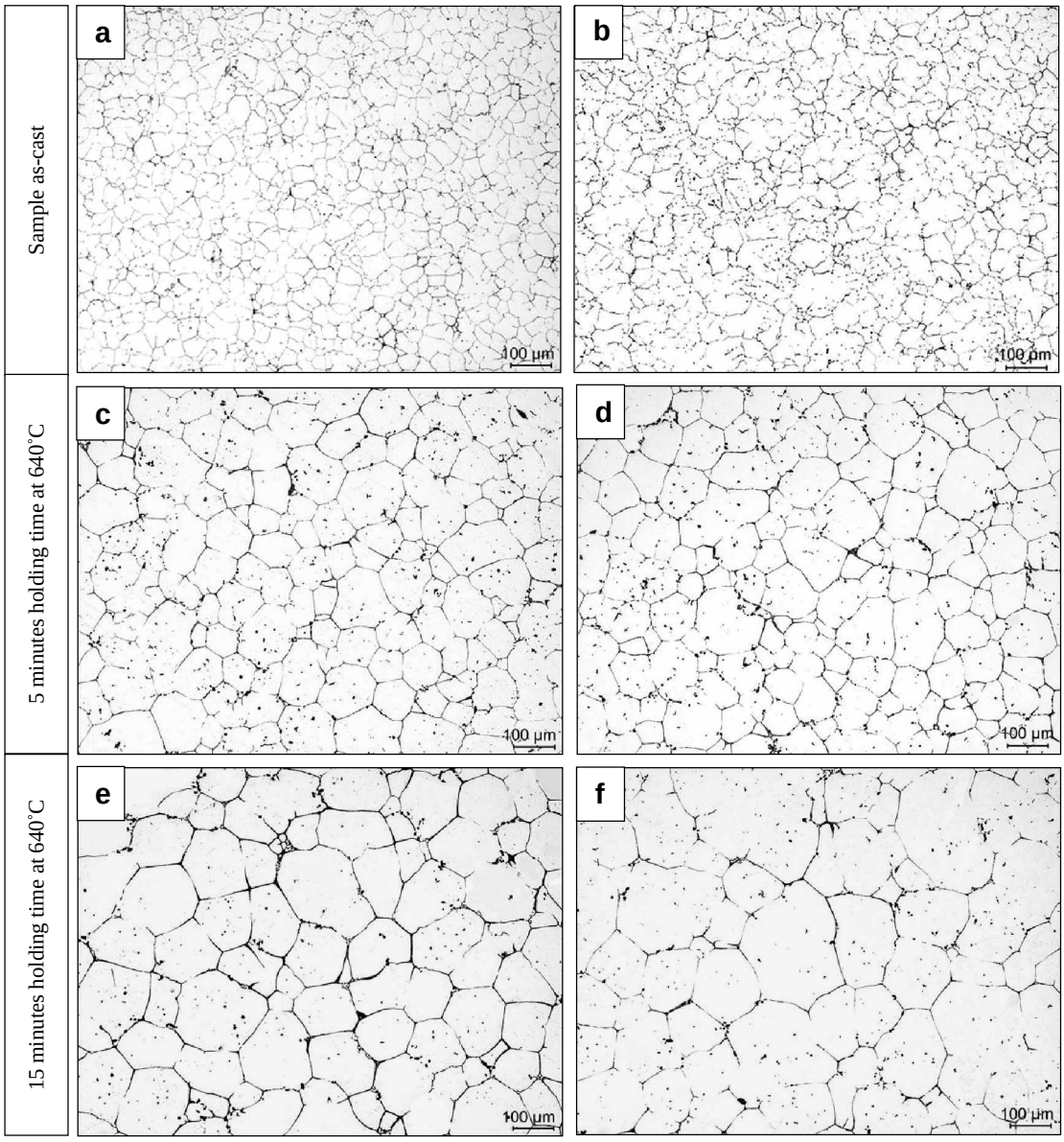

Figure 5.18 Microstrcutural evolution of AA6082 alloy as-cast billets obtained after poured at 665˚C and isothermally held at 640˚C a) as-cast sample poured through 400 mm distance (C4), b) as-cast sample poured through 200 mm distance (C8), c) poured through 400 mm distance and isothermally held for 5 minutes (C4.1), d) poured through 200 mm distance and isothermally held for 5 minutes (C8.1), e) poured through 400 mm distance and isothermally held for 15 minutes (C4.2), f) poured through 200 mm distance and isothermally held for 15 minutes

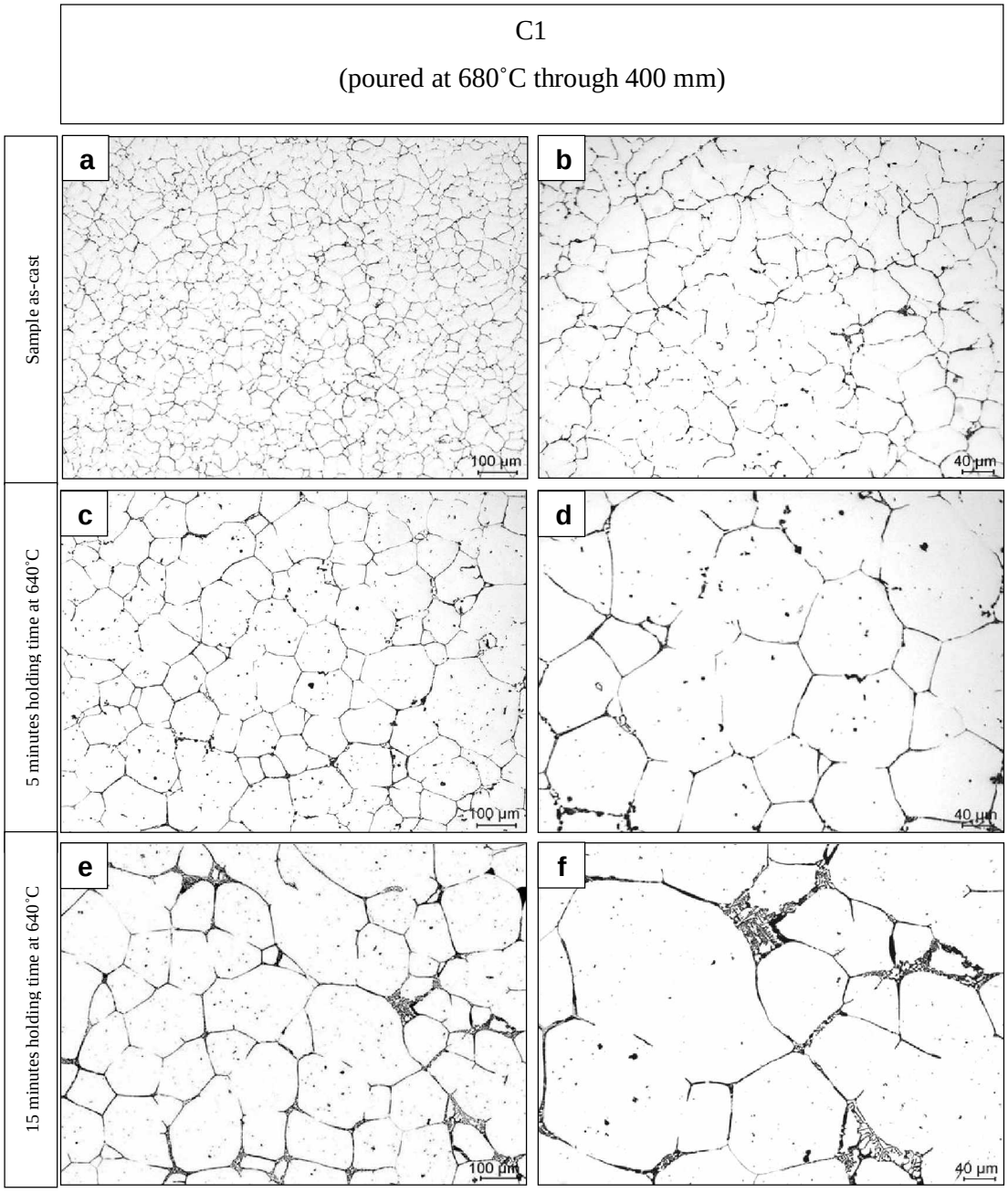

Figure 5.19. Microstructural evolution of AA6082 alloy as-cast billets obtained after poured at 680˚C through 400 mm and then isothermally held at 640˚C for a) and b) as-cast samples (C1), c) and d) 5 minutes (C1.1), e) and f) 15 minutes (C1.2).

5.4. Thixoformed Samples

For thixoforming practices, CA4 (casting temperature of 640˚C and pouring distance of 300 mm) and CA5 (casting temperature of 630˚C and pouring distance of 300 mm) coded as-cast billets were used for A357 alloy while C7 (casting temperature of 670˚C and pouring distance of 300 mm) coded as-cast billet was used for AA6082 alloy. Figure 5.20 and Figure 5.21 show the photos of thixoformed samples obtained from these as-cast billets. The samples in Figure 5.20 were thixoformed for microstructural investigation and hardness tests with first die group while the samples in Figure 5.21 were thixoformed for tensile tests with second die group. As given in Table 4.6, in order to investigate six conditions for each die group, totally twelve types of samples were thixoformed and codes from P1 to P12 were given. Both sample types showed successful die filling characteristics resulted from their thixotropic nature. Although the forming experiment was designed for die filling without any flash, small amount of flashes formed due to tolerance between main die and lower half die. These undesired flashes made difficulties during ejecting of the thixoformed samples.

Figure 5.20. Samples thixoformed with first type die group for microstructural investigation and hardness tests

Figure 5.21. Samples thixoformed with second type die group for tensile tests

5.4.1. Microstructure of the Thixoformed Samples

As mentioned in section 4.2.3, two methods were carried out in order to solve handling and transferring problem of semi-solid material during thixoforming practice. Figure 5.22 shows the micrographs of the thixoformed sample obtained with the first method experiments after CA5 coded as-cast billet was formed at 580˚C with holding time of 5 minutes. Micrographs were taken from different parts through longitudinal cross section of the thixoformed sample in order to represent the non-uniform structure of the α-Al particles. Bottom part of the thixoformed sample was not thixotropic and did not contain globular grains suspended in liquid matrix. The bottom part was in contact with the lower half die and due to the heat extraction, temperature was less than 570˚C. That is why eutectic phase was not remelted and the grains did not spherodize in the parts close to the bottom. Moving from bottom to the upper parts, the structure became thixotropic, globular grains formed and eutectic phase was remelted as illustrated in Figure 5.22. Although handling and transferring problem of semi-solid material was solved in this method, non-uniform microstructure was obtained. So, second method was carried out in order to solve both problems.

Figure 5.23 shows the corresponding microstructures of P3 coded sample obtained in the second method after forming of CA5 coded as-cast billet at 580˚C with holding time of 5 minutes. As shown in Figure 5.23, different from first method experiment, globular grains were suspended in liquid matrix which was distributed uniformly in all parts of the sample. Eutectic phase was remelted and the temperature was approximately 580˚C in all over the specimen.

In the second method, both transferring and uniform distribution problems were solved, so the remaining thixoforming experiments were carried out with the second method. Figure 5.24 shows the microstructures of P1, P2 and P4 coded samples obtained after thixoforming of CA4 and CA5 coded A357 as-cast billets at 580˚C with holding time of 5 and 15 minutes. In all thixoformed samples, thixotropic structure was formed with fine globular grains distributed uniform in eutectic phase. However, the eutectic phase was not uniform and in lamellar form instead of fine structure. The lamellar eutectic phase was formed in the thixoformed samples because the formation of the eutectic silicon is strongly

affected by the addition of strontium and there is not strontium element in the chemical composition of A357 alloy as given in Table 4.1. As the holding time was increased from 5 to 15 minutes, the size of the globular grains increased. Coarse grains and lamellar eutectic phase were not desirable in thixoforming process due to adverse affect on mechanical properties.

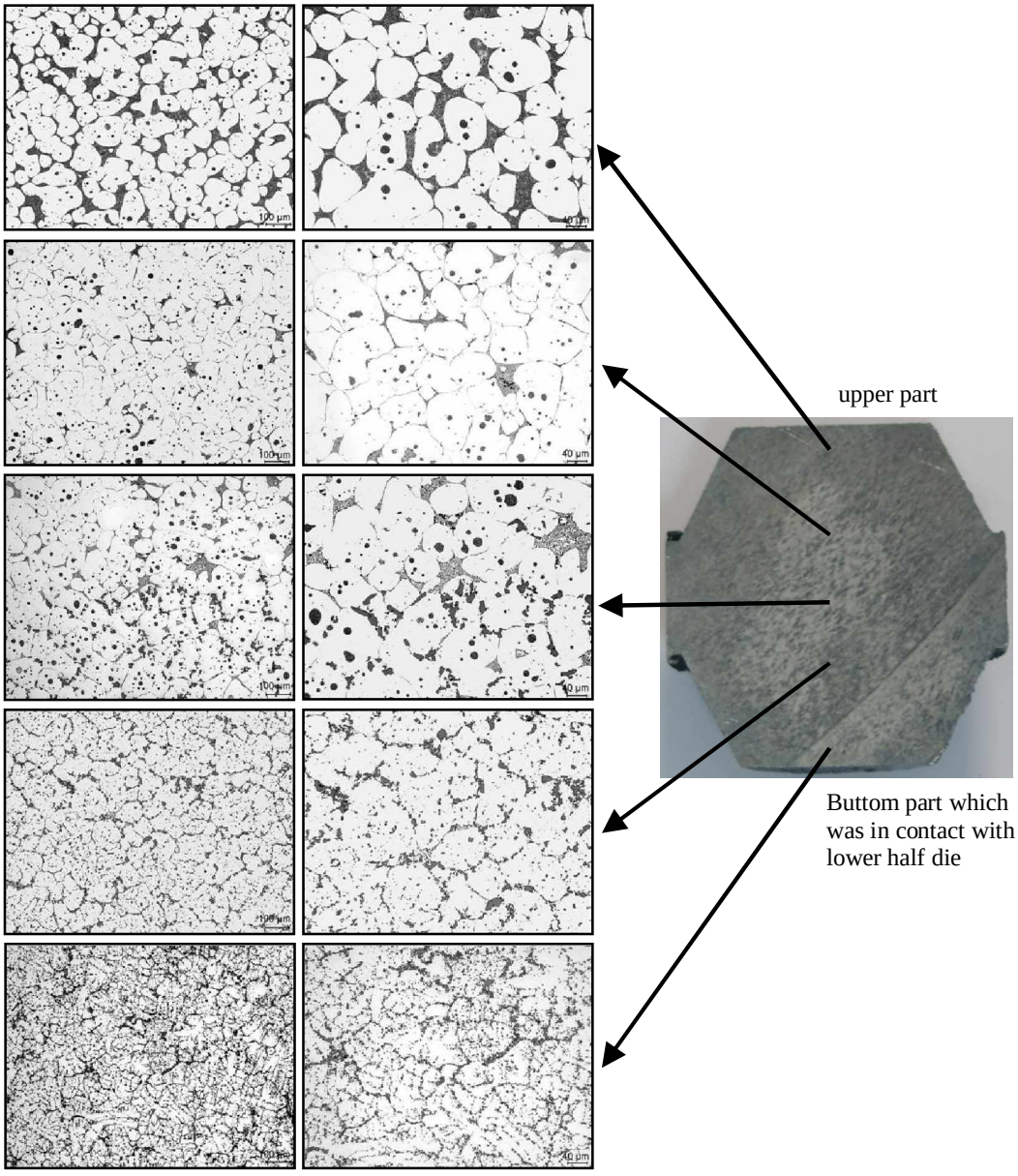

Figure 5.22. Micrographs were taken from different parts through longitudinal cross section of the thixoformed sample after first method. Non-uniform morphology presented in the sample.

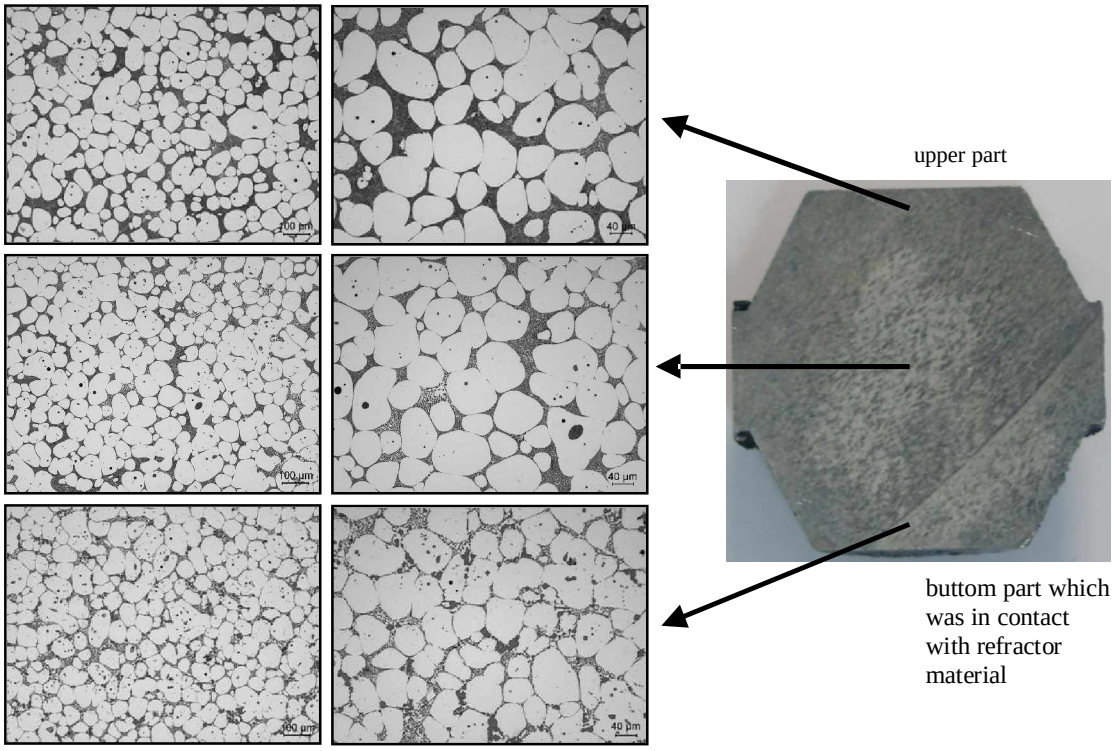

Figure 5.23. Micrographs were taken from different parts of the longitudinal cross section of the P3 coded thixoformed sample after second method. Globular α-Al grains were uniformly distributed in the sample.

Figure 5.25 shows the micrographs of the P5 coded sample obtained after forming of C7 coded AA6082 as-cast billet at 640˚C with 5 minutes holding time. It is possible to detect three zones in the cross section of the thixoformed sample. In the first zone, the grain boundaries were not clear and liquid phase was not present. The second zone was a transition zone and it was similar to the results obtained in reheating practices. In this zone, hexagonal grains were formed with clear grain boundaries but there was not any remelted eutectic phase due to the low alloy composition of AA6082 alloy. The third zone was composed of fine globular grains suspended in liquid matrix. This structure is thixotropic and preferable for thixoforming process. Uniform distribution of microstructure in this thixoformed sample was resulted from the macrosegregation of the liquid phase during forming. In the previous section, the results of the reheating practices showed that only small amount of remelted liquid phase formed for AA6082 alloy and it is obvious from Figure 5.25 that all this remelted liquid phase segregated from the first and the second zone to the third zone during thixoforming process.

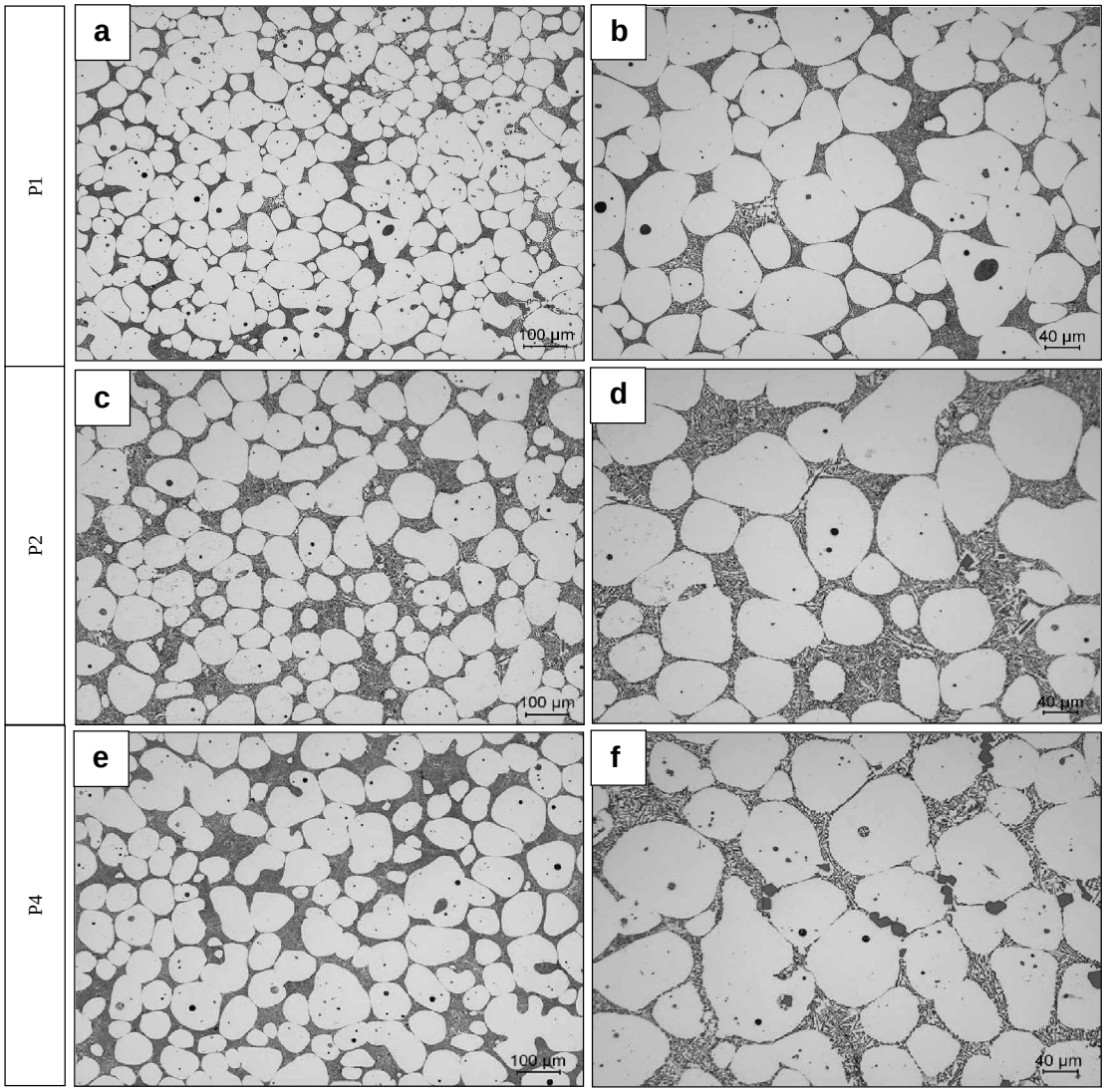

Figure 5.24. Microstructures of the thixoformed A357 samples a) and b) thixoformed after CA4 coded as-cast billet isothermally held at 580˚C for 5 minutes, c) and d) thixoformed after CA4 coded as-cast billet isothermally held at 580˚C for 15 minutes, e) and f) thixoformed after CA5 coded as-cast billet isothermally held at 580˚C for 15 minutes

Figure 5.26 shows the micrographs of the P6 coded sample obtained after forming of C7 coded as-cast billet at 640˚C with 15 minutes holding time. Similar to microstructure of P5 coded sample, a key feature of this microstructure was the segregation of liquid phase and formation of different zones. Figure 5.26a and Figure 5.26b represents the microstructure of one zone which was composed of unclear grains without any liquid

phase. On the other hand, Figure 5.26c and Figure 5.26d represents the microstructure of another zone in which liquid phase was formed among globular grains.

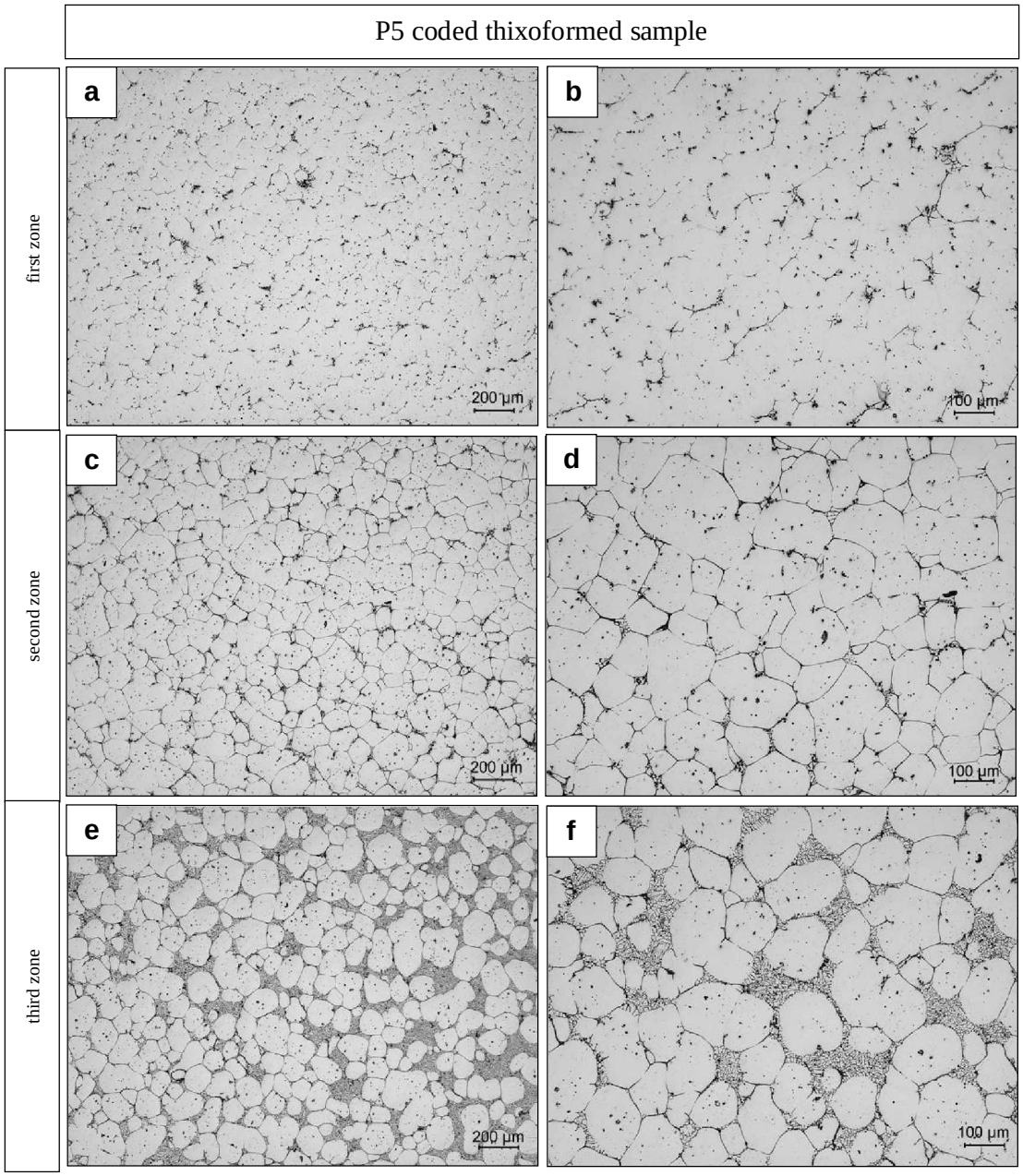

Figure 5.25. Microstructure zones of P5 coded sample thixoformed after C7 coded AA6082 as-cast billet isothermally held at 640˚ for 5 minutes a) and b) first zone, c) and d) second zone, e) and f) third zone.

5.4.2. Hardness Measurements of Thixoformed Samples

The microhardness of the thixoformed samples were measured in 4 mm increments through x and y axis of the cross section as shown in Figure 5.27. The center of the sample was chosen as base point and indicated as “0 mm”. The other points were indicated as positive and negative values respectively according to the base point and axes.

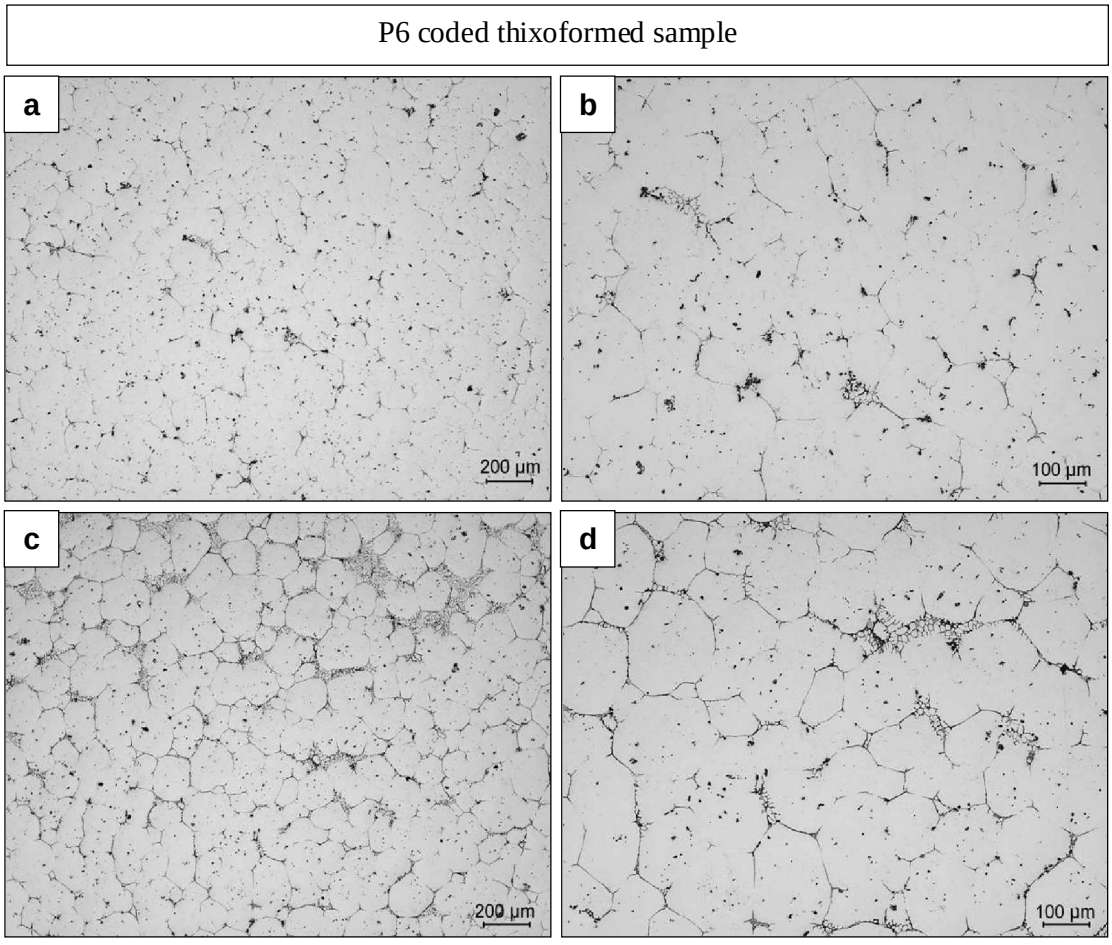

Figure 5.26. Microstructure of P6 coded sample thixoformed after C7 coded AA6082 as-cast billet isothermally held at 640˚ for 15 minutes.

Figure 5.28 to Figure 5.33 show the hardness measurements of the thixoformed samples coded from P1 to P6. As expected, T6 heat treatment increased the hardness values of the samples. Another consequence of these measurements was the effect of isothermal holding times on the hardness values. Thixoformed samples which were held

isothermally for 5 minutes (P1, P3 and P5) had better harness values comparing to samples which were held isothermally for 15 minutes (P2, P4 and P6). Grain coarsening mechanism could be responsible for such a decrease because as the holding time was increased, the grain size was also increased due the coalescence of the particles.

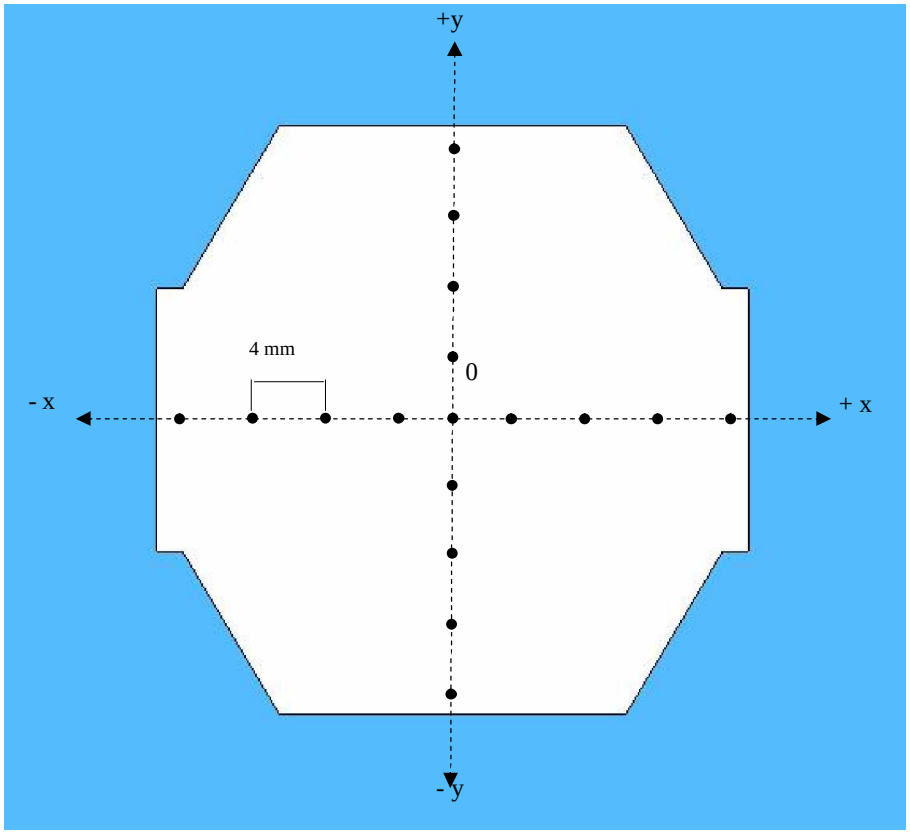

Figure 5.27. Schematic illustration of the points taken for hardness measurements

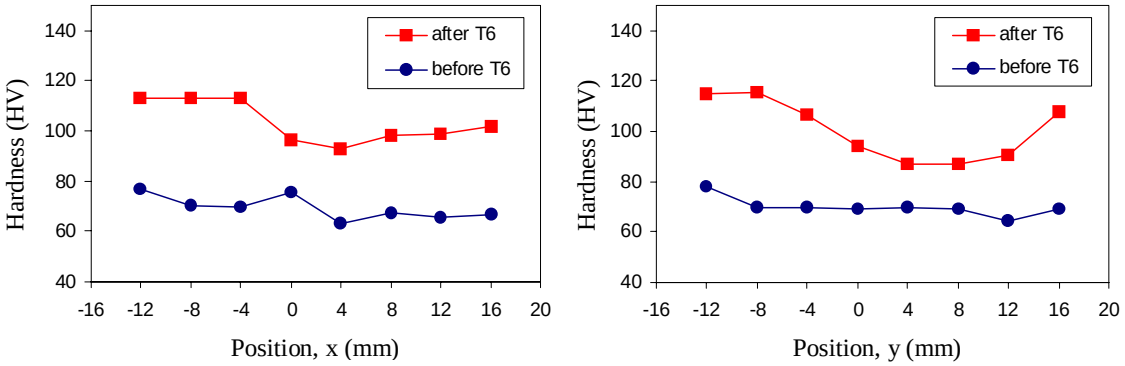

Figure 5.28. The microhardness measurements of the P1 coded thixoformed sample

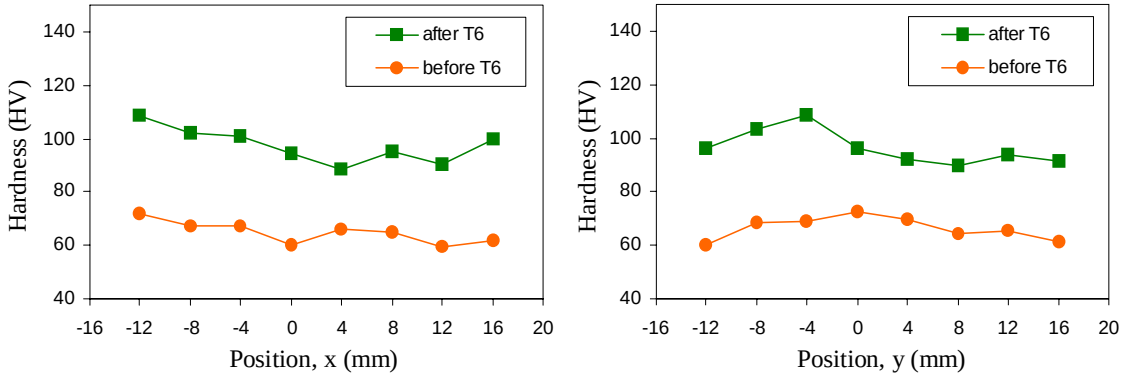

Figure 5.29. The microhardness measurements of the P2 coded thixoformed sample

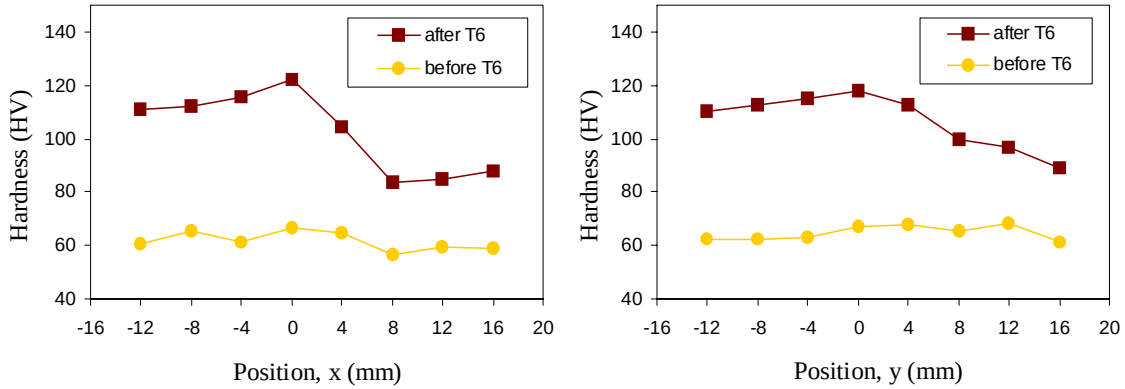

Figure 5.30. The microhardness measurements of the P3 coded thixoformed sample

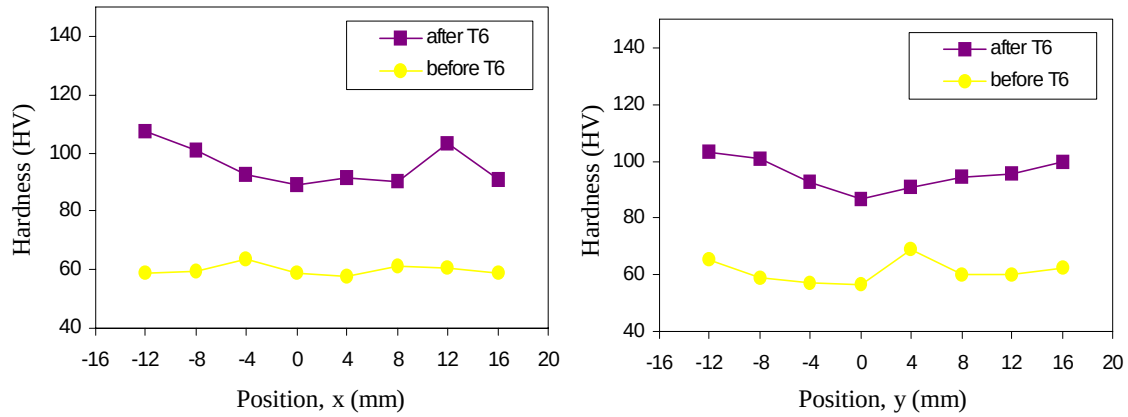

Figure 5.31. The microhardness measurements of the P4 coded thixoformed sample

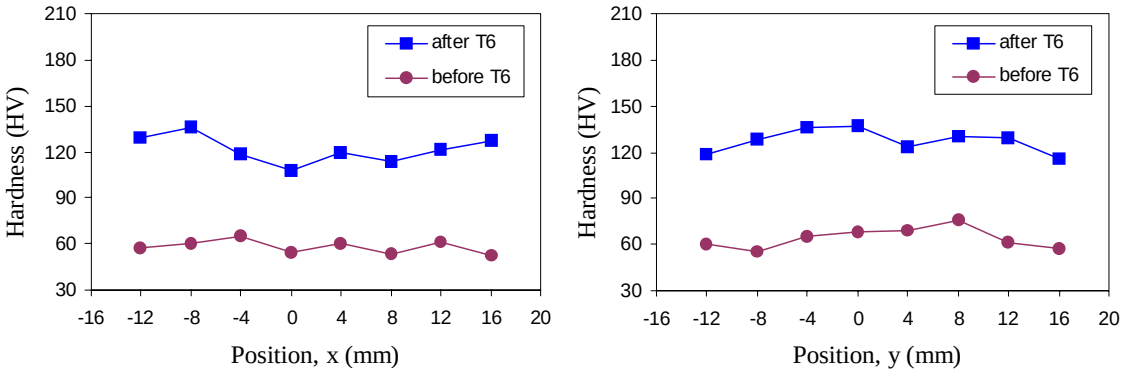

Figure 5.32. The microhardness measurements of the P5 coded thixoformed sample

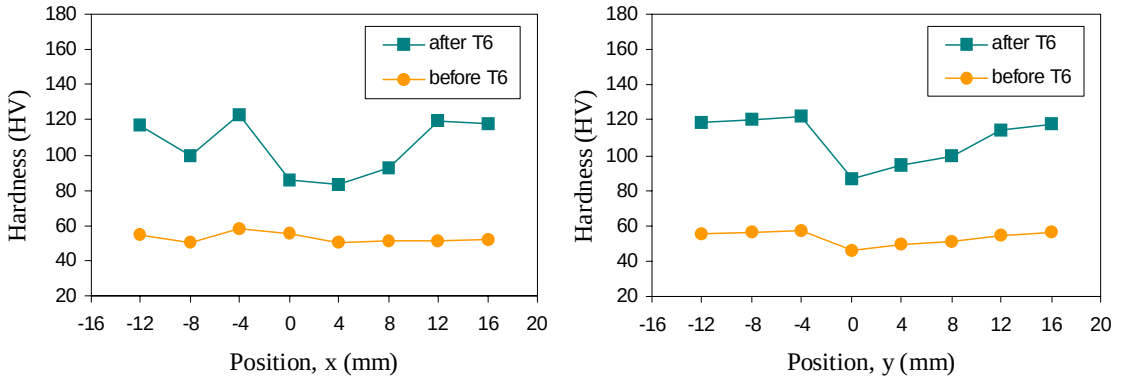

Figure 5.33. The microhardness measurements of the P6 coded thixoformed sample

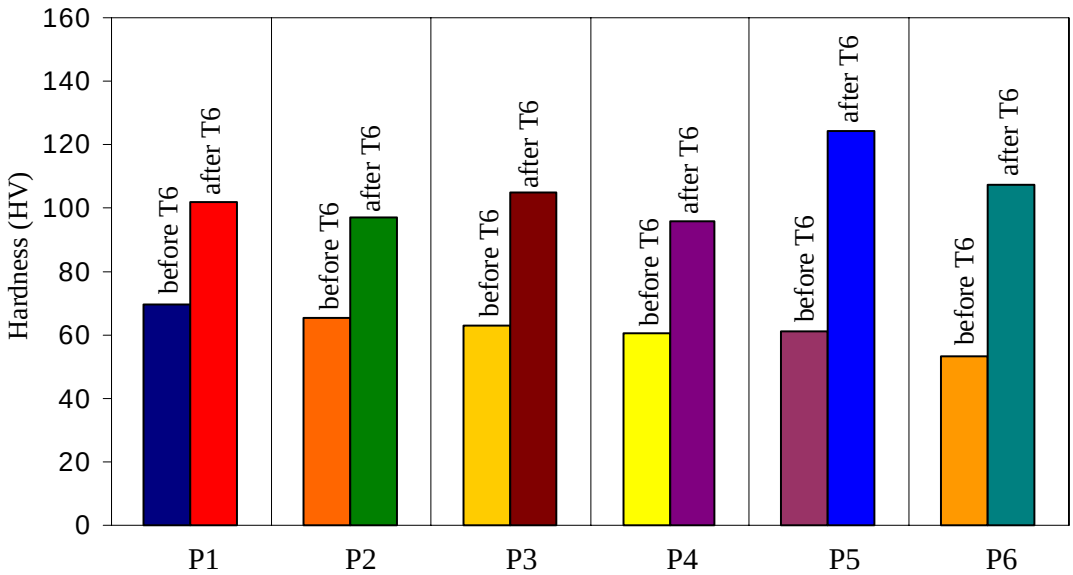

Figure 5.34. Average hardness values of the thixoformed samples coded from P1 to P6

Figure 5.34 shows the average hardness values of the thixoformed samples. For A357 alloy (P1, P2, P3 and P4 coded samples), measured values of around 100 HV (after T6 heat treatment) exceeded the results of similar studies in the literature [27, 36]. On the other side, after T6 heat treatment, values around 120 HV were measured for AA6082 alloy (P5 and P6 coded samples); however, there is not any similar study in the literature for AA6082 alloy to compare the results.

5.4.3. Tensile Mechanical Properties of Thixoformed Samples

Figure 5.35 shows the ultimate tensile strength, yield strength and elongation to fracture values of the A357 and AA6082 thixoformed samples. The samples were coded from P7 to P12 and conditions correspond to each code were given in Table 4.6. For comparison of these results, similar studies in the literature were reviewed in Table 5.1.

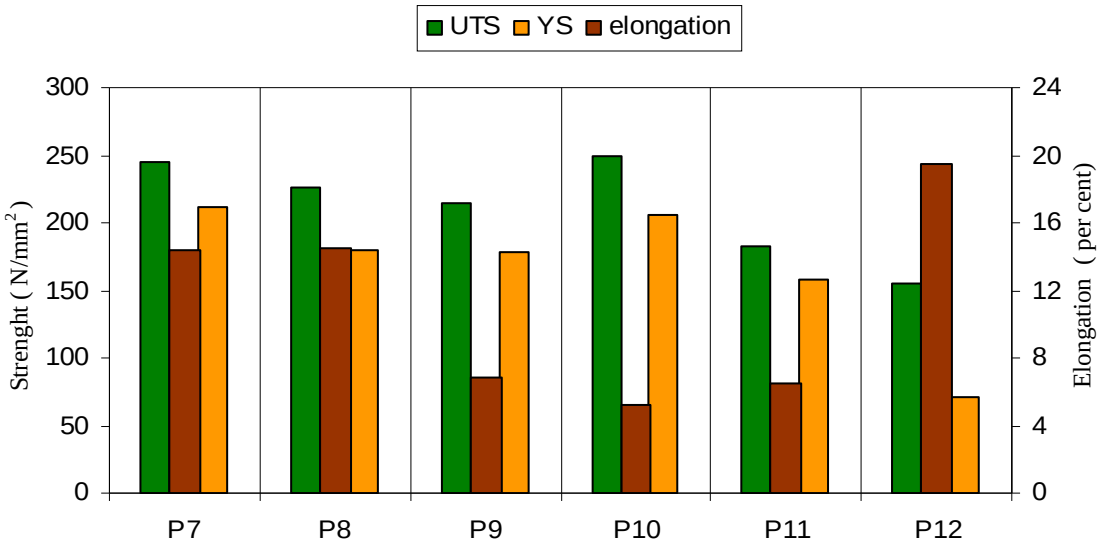

Figure 5.35. Tensile mechanical properties of the thixoformed samples

For A357 alloy (P7-P10), the results were in good agreement with literature. However, best mechanical properties were not obtained due to the facts listed below:

- Formation of casting defects such as pores and oxides during CS casting process
- Formation of lamellar eutectic phase during reheating due to the lack of strontium element in alloy composition

- Lower press load (1.5 ton) during thixoforming process comparing to the loads in the literature (200-500 tons).

Table 5.1. Mechanical properties of the thixoformed samples in the literature

Process	Alloy	Feedstock production method	Heat treatment	Ultimate tensile strength (MPa)	Yield strength (MPa)	Elongation (per cent)
Thixoforging [68]	A356	CS	T6	293	234	15
Thixoforging [36]	A356	MHD	T6	295 208	253 150	5.4 7.8
Rheocasting [73]	A356		T6	290	229	11.7
Thixoforging of master cylinder [3]	A357	MHD	T6	269	230	6.2
Thixoforging of steering knuckle [25]	A357 AA6082 AA6082 AA6082		T6 T6 T6 T6	250 280 250 210	200 250 200 150	6.5 7 7.5 11
Thixoforging [23]	AA6082	MHD	T6	370	330	6
Thixoforging of steering knuckle [74]	A357 A357 AA6061 AA6061	MHD MHD SIMA SIMA	T6 T6 T6 T6	346 337 313 235	280 220 270 193	4 4.2 10.2 11.5

On the other hand, the results for AA6082 alloy were not in agreement with literature. In addition to the facts listed above, macrosegregation of remelted liquid could be responsible for such a decrease in the strength of the thixoformed sample. For the wrought alloys, a small variation in temperature induces a large change in solid fraction. Therefore, a small decrease in temperature can lead to a considerable increase in solid fraction resulting in a microstructure absolutely not favorable to thixoforming. In this case,

deformation is inhomogeneous and liquid segregation occurs often during mould filling. In the tensile specimen of AA6082 alloy, non-uniformly distributed liquid phase, especially liquid pools, could form weak points and these weak points could lead to lower tensile and yield strength values. The results also show that tensile mechanical properties were dependent on holding times. As a result of longer holding times, the tensile and yield strengths decrease and the elongation to fracture increases.

6. CONCLUSIONS

Thixoforming is an attractive process for the manufacture of complex parts with substantial savings of time and cost and it is composed of three main processes, i.e. thixotropic feedstock production, reheating, and thixoforming. In this study, three steps of the thixoforming process were carried out for A357 and AA6082 alloys. Feedstock for thixoforming ought to have a non-dendritic microstructure where equiaxed, globular grains are uniformly distributed in a liquid matrix. Cooling slope (CS) casting process was employed to obtain such a feedstock as it is both economical and practical. Microstructure of the as-cast samples showed that decreasing the superheat and increasing the cooling length in CS casting process promoted the formation of non-dendritic and equiaxed grains. Moreover, the morphology of α-Al phase changed through the cross section of as-cast samples. Dendrite morphology was dominant in the regions close to edge of the sample while equiaxed morphology was observed in sections near to the centre. This non-uniform microstructure was resulted from different cooling rates in different regions.

In the reheating step of the experiment, A357 and AA6082 as-cast samples were partially remelted at 580˚C and 640˚C respectively and isothermally held for 5 and 15 minutes with an induction furnace. The results of the reheating practices showed that, the non-uniform morphology presented in the as-cast samples turned into a more homogeneous and globular microstructure after partial remelting and isothermal holding for A357 alloy. Moreover, as a result of a longer holding time, the grain size increased and the grains became more globular. However, hexagonal grains were formed rather than equiaxed and globular particles in the microstructure of the reheated AA6082 samples. It is found that, inadequate spheroidization in grain boundaries were resulted from insufficient amount of remelted eutectic phase in AA6082 alloy and this situation was directly related with the low alloy composition of AA6082 alloys. The external appearance of the billets turned into elephant foot shape during reheating practices for both alloys.

In the forming step of the experiment, as-cast billets were reheated inductively to the temperature practiced in reheating step and then thixoformed between dies to investigate microstructural evolution and mechanical properties. Thixoforming for both alloys resulted

in successful filling of the dies. The microstructure of the thixoformed A357 samples were in good agreement with the results obtained in the reheating step while macrosegregation of the liquid phase was observed in the microstructure of the thixoformed AA6082 samples. T6 heat treatment was carried out to improve the mechanical properties of the samples. Mechanical properties of the thixoformed A357 samples were in good agreement with results in the literature, however; formation of defects during CS process, lamellar eutectic phase formation during reheating step and lower press loads comparing to forces in the literature avoided to obtain better yield and tensile strengths. On the other hand, yield and tensile strength of the thixoformed AA6082 sample was lower than the values in the literature due to the segregated and non-uniformly distributed liquid phase in the sample.

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