CHAPTER 4 ASSESSMENT OF NET-SHAPE FORMING OF AL-25SI-2.5CU-1MG ALLOY BY POWDER THIXOCASTING

4.1 Motivation

The design concept of powder thixocasting has been described in Chapter 3. This Chapter will deal with our efforts in attempt to realize this concept, through net-shape forming of a high-quality hypereutectic Al-Si-X alloy.

This Chapter details the process optimization of powder thixocasting in fabricating Al-25Si-2.5Cu-1Mg alloy. The following main issues for powder thixocasting will be concerned and overcome by carrying out a series of experiments:

1. How to efficiently prepare the powder preforms for thixocasting.
2. How to prevent the fine grains of gas-atomized powder from grain coarsening during thixocasting.
3. How to reduce the formation of oxide films and intergranular pores in the powder preforms after powder thixocasting, for obtaining good strength of thixocast products.

Based on the above questions, the effects of processing parameters on the microstructure and strength of the thixocast products are also examined and discussed in this Chapter. The processing parameters include the particle size of Al-Si-Cu-Mg powder, the temperature at which the powder was consolidated and the temperature at which the consolidated powder preforms was thixocast. From these studies, a consolidation mechanism of powder thixocasting is also proposed. Finally, a scroll for air conditioner compressor was fabricated as a demo parts to demonstrate the feasibility of this process in net shape forming of the hypereutectic Al-Si-Cu-Mg alloy with fine microstructure and high integrity.

4.2 Experimental

4.2.1 Preparation of Al-Si-Cu-Mg powder

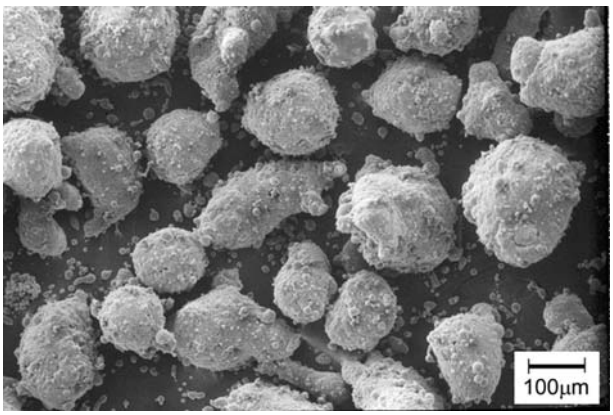
The hypereutectic Al-Si-X powder used, supplied by Valimet Inc, USA, were fabricated by a gas-atomization process. Their chemical compositions are given in Table 4.1. It has nominal compositions of Al-25Si-2.5Cu-1Mg- 0.5Mn (in weight percent). The nominal composition is similar to that of a commercialized compressor scrolls fabricated by conventional powder forging process.

The as-atomized powders were sieved and divided into three groups with different particle size ranges, namely fine (<45μm), medium (45~120μm) and coarse (120~300μm). Particle size distributions of the three fractions of the Al-Si-Cu-Mg powders were measured by a particle size analyzer, Beckman Coulter ^{TM}. The values of particle size are listed in Table 4.2, where d_{10}, d_{50} and d_{90} present the particle diameters such that 10%, 50% and 90%, respectively, of total powder volume is in particles of smaller diameter.

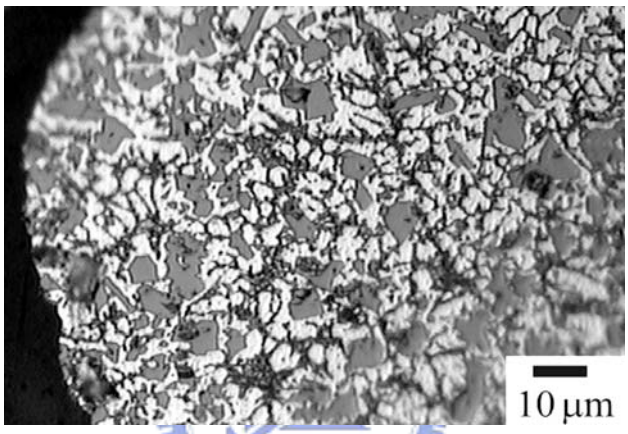
Since the gas-atomized Al-Si powder is rapidly solidified, it has near spherical shape in appearance (Fig. 4.1 a) and exhibits fine and non-dendritic microstructure (Fig. 4.1 b). The powder particles possess fine primary Si particles with average size of about 6μm, uniformly distributed in a matrix, comprising α-Al grains and black net-work eutectic phases, as illustrated in Fig. 4.1 b.

Table 4.1 Chemical compositions of the hypereutectic alloy powder

Element	Si	Cu	Mg	Mn	Fe	Ni	Ti	Al
Rate (wt%)	24.64	2.56	1.04	0.47	0.16	0.01	0.03	Bal.



(a)



(b)

Figure 4.1 (a) SEM morphology and (b) optical microstructure of the gas-atomized hypereutectic Al-Si powder

Table 4.2 Particle size distributions of the three fractions of the gas atomized Al-Si powder

Fraction	Powder size (μm)		
	d_{10}	d_{50}	d_{90}
<45μm	14	32	42
45~120μm	50	78	114
120~300μm	135	175	240

4.2.2 Procedures of Powder Thixocasting

Figure 4.2 schematically depicts the novel process developed in this study. It includes the three steps: preparing a powder preform (Figs. 4.2 a and b), heating the powder preform into a semi-solid state (Fig. 4.2 c), and thixocasting the semi-solid preforms into net-shaped components (Fig. 4.2 d).

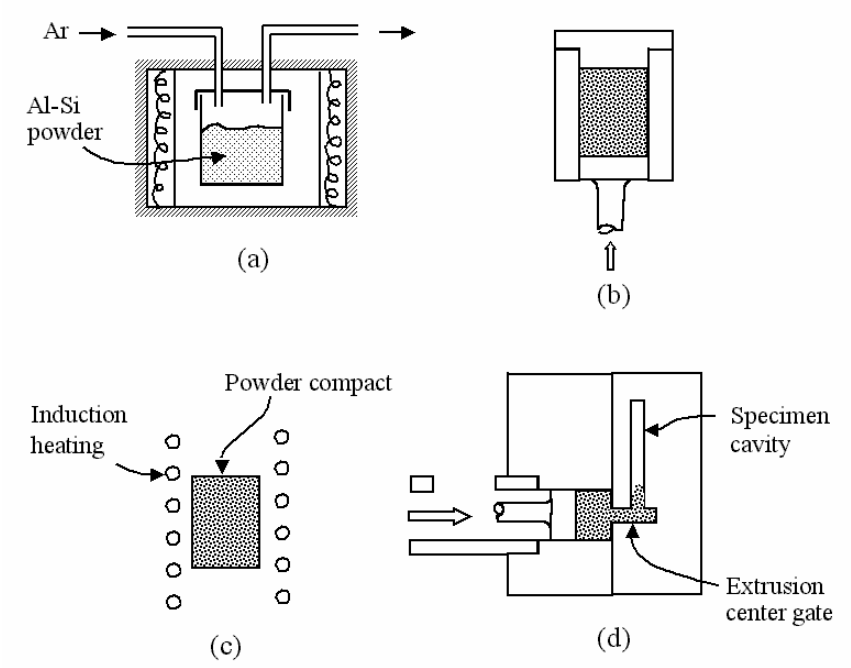


Figure 4.2 Schematic diagram of the powder thixocasting process

(a) preheating the hypereutectic pre-alloyed Al-Si powders, (b) hot consolidating the powders into compacts, (c) heating the compacts into semi-solid state with induction coils, and (d) thixocasting the semisolid compacts by a high-pressure die casting machine.

Figure 4.3 shows the layout of the apparatus used in this study for thixocasting process. The system comprises a forming machine, a robot and an induction heating station. The heating and forming of powder preforms was controlled by a computer. Non-dendritic feedstock was transferred using the robot, on the way from storage rail to heating station or from the station to shot sleeve.

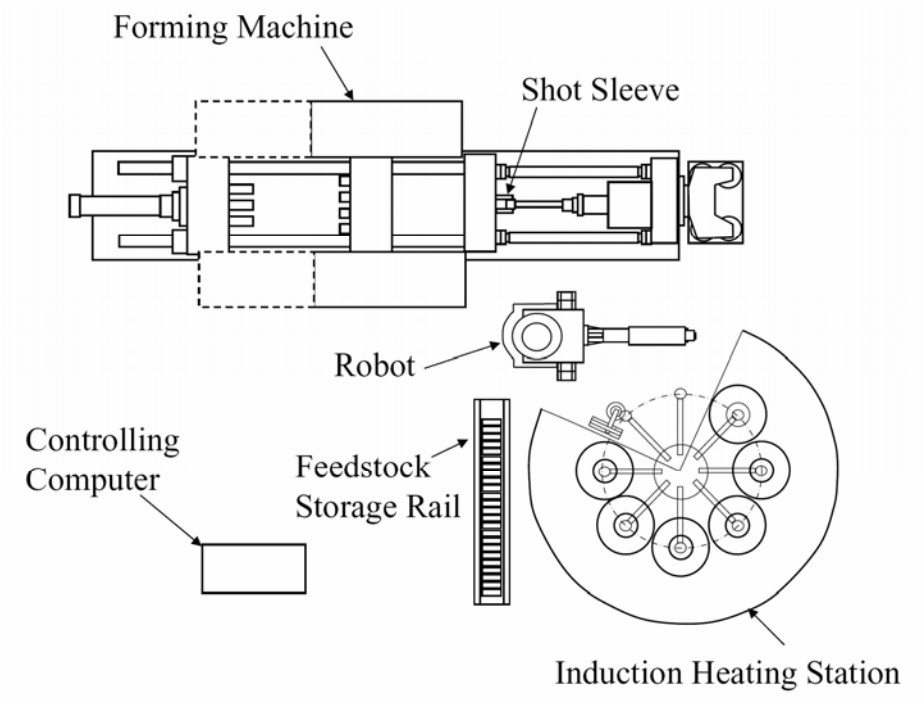


Figure 4.3 Schematic diagram of the apparatus layout for thixocasting process

Experimental procedures of powder thixocasting are detailed as follows.

A. Preparing powder preforms

The powder preforms were consolidated at various elevated temperatures and at constant pressure, 85 MPa, to increase the compressibility of the powder and obtain sufficient strength of the powder preforms for handling.

Before pressing, around 950 grams of the powder was charged in a steel cup with inner diameter of 90 mm and was preheated in an electric furnace under the protection of an argon atmosphere. Two thermal couples were used to monitor the temperature of the powder during preheating, one was inserted at the center and the other was at outer side of the cup of powder, as is shown in the inset of Figure 4.4. At beginning, the temperature at outer side rises more quickly than that at center of the cup of the powder. After around 60 minutes, the two temperatures gradually get equivalent and are close to the furnace temperature. Therefore, in order to ensure that all of the powders used have homogenized temperature, the powders were preheated at least for 1 hour in each

test.

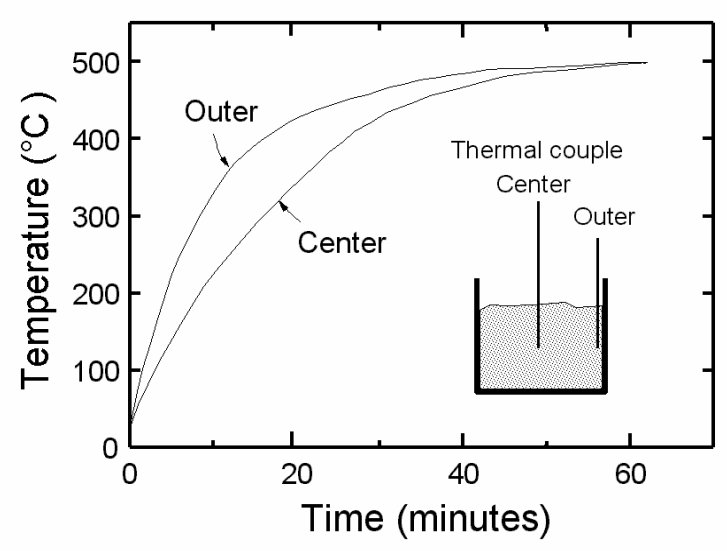


Figure 4.4 Temperature increasing curve of the Al-Si powder during preheating up to 500°C.

After preheating to a uniform temperature, the powders were immediately transferred to a uniaxial hydraulic press. Figure 4.5 shows the tonnage presser used for preparing powder preforms. The presser is an oil-hydraulic pressing machine that is indeed a commercialized squeeze casting machine, with clamping force of max.2500 kN and injection force of max. 650 kN.

The powder preforms were consolidated in air and at a pressure of 85 MPa. The preforms have a diameter of 76 mm, and their length vary from 300 to 550mm that is determined by the compressing force and temperature. Before the pressing proceeded, the compacting steel mould, preheated to a temperature of 250 °C, was spray coated with a graphite lubricant to reduce its gall against Al-Si-Cu-Mg powders during compacting.

In this stage, the effect of the final preheating temperature, referred as consolidating temperature, on the compressibility of the powder preforms was studied. The compressibility of perform is referred to their relative density, i.e. the ratio of the density of the preforms to theoretical density of powder.

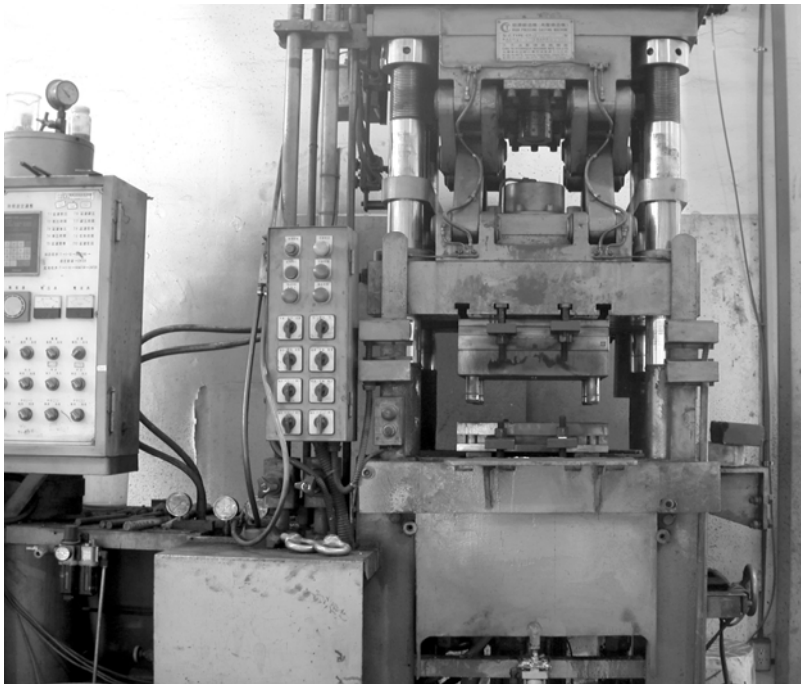


Figure 4.5 Oil-hydraulic pressing machine used for powder consolidation and squeeze casting.

B. Heating the powder preform to semi-solid state

Because the preforms were heated in air, the heating to semi-solid state should be as quick as possible to prevent from formation of excess oxide films on the powder surface. In this work, an induction heating station, as shown in Fig. 4.6, was adopted. The induction coils of the station has clear internal diameter of 145 mm, overall coil length of 310 mm and is driven by a converter with rated frequency of 280 Hz.

Figure 4.6 Semi-solid feedstock heating station with five induction coils

A precise control of heating process is essentially required to obtain the exact liquid fraction and temperature homogeneity in the preform in a semisolid state.

Temperature homogeneity was achieved by heating with an induction coil governed by a power-temperature program. During heating, the variation of the preform temperature was measured using a thermal couple, which was inserted into the preform at the location as shown in the inset of Fig.4.7. The measured temperature was employed to simultaneously adjust the induction input power and finally heat the preform to a preset temperature.

Inadequate setting of the power-temperature heating program results in either excess melting on the preform surface or the need for time-consuming heating to achieve temperature homogeneity throughout the preform. Several trials obtained an acceptable power-temperature region.

Figure 4.7 illustrates an example of the reheating program, which is suitable for the powder preforms consolidated at 550°C and with density of 90%. At the start of heating, a high power (45kW) was used to quickly heat the preform. Once the preform reached around 500°C, the power was reduced stepwise to homogenize the temperature through the perform thickness.

In Fig.4.7, the total heating time was only about 200 seconds. It was found that short heating time is important for obtaining good strength of thixocast products. This is because the short heating time could assure minimization of the surface oxidation on the Al-Si powder since the induction heating was conducted in air. However, the total heating time is strong depended on the density of the powder preforms and a higher density a shorter heating time; the reasons will be discussed in Section 4.3.

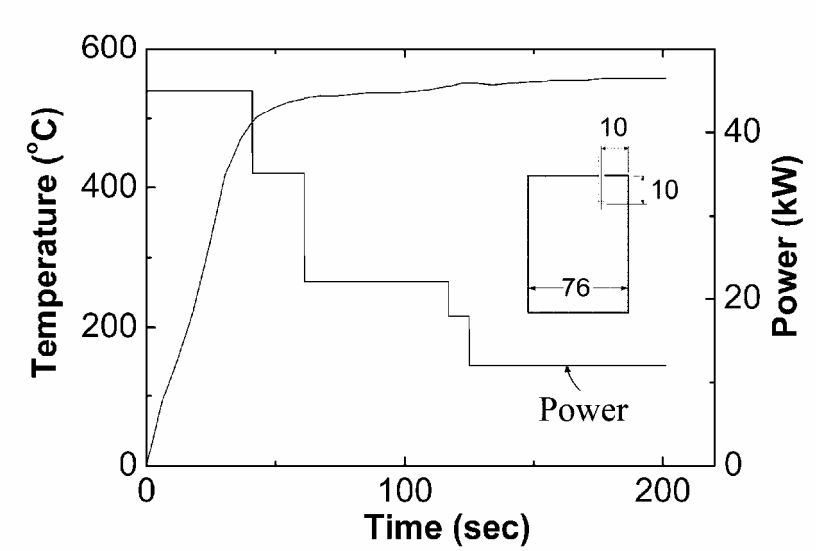


Figure 4.7 A suitable power-time heating program used in heating the powder performs into semi-solid state.

The program controls the input power of induction coil, so that temperature rising of the powder performs is under controlled. An inset shows where the thermocouple was inserted in the preform.

After heating to semi-solid state, the powder preforms normally have several melt beads on surfaces, as shown in Fig. 4.8. When the melt beads began to be squeezed out from the interior to the preform surface, a semi-solid temperature on preform surface was reached. Besides measuring the temperature, this study also adopted a simple method of ascertaining the extent to which the temperature was homogenized throughout the preform. A steel rod with diameter of approximately 1.5mm was used to pierce the heated preform. Since the semi-solid temperature range containing low fraction of liquid was very narrow, if the steel rod could easily pierce to the center of the

preform, the preform was assumed to have approximately uniform temperature through its thickness.

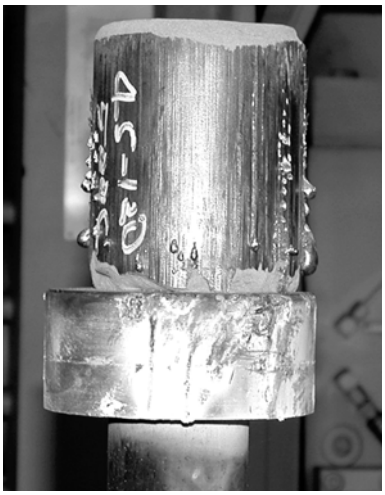


Figure 4.8 Melt beads formed on a semi-solid powder preform

The preform that standing on a ceramic pedestal were fabricated at a consolidating temperature of 500 ^{o}C and using the powder with particle size of 45-120 μm.

C. Semi-solid forming using a die casting machine

Figure 4.9 shows the die-casting machine that carried out the net-shape forming of semi-solid powder preforms.

As soon as the preform was heated to a semi-solid state, it was transferred into a sleeve of a high-pressure die-casting machine, as shown in Fig. 4.10. Afterward, a plunger extruded the semi-solid powder preforms into mold cavities to form net-shape components. The extrusion final maximum pressure was 90 MPa and plunger velocity was 0.5 m/sec, which corresponds to a center gate velocity of 3.9 m/sec. The plunger was 84mm in diameter. During thixocasting, the mold temperature was kept at 250°C by using circulating oil heating system.

Figure 4.9 Thixocasting machine with clamping force of 5,000 kN

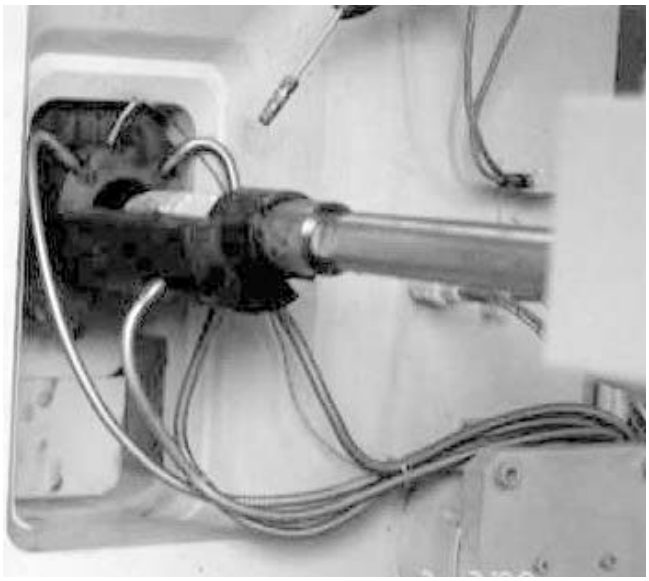
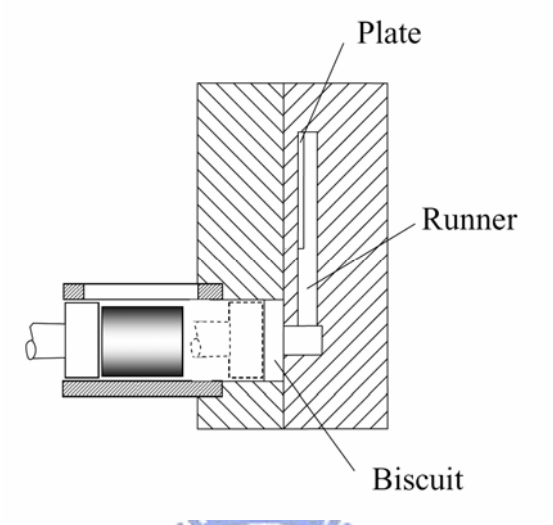


Figure 4.10 Injection system comprising a sleeve and a plunge

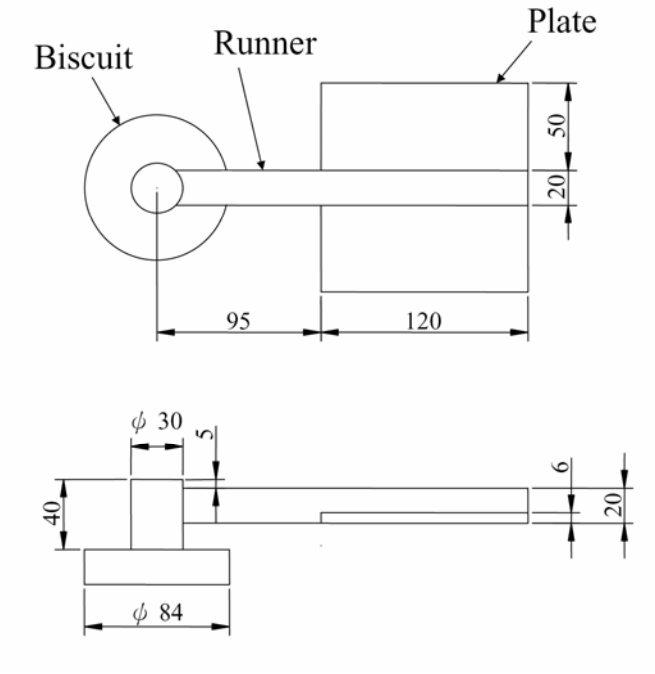
Figure 4.11 displays the dimensions of the mold cavity used, and schematically draws how the sample was divided into three segments, Biscuit, Runner, and Plate. When the plunger extruded the semi-solid powder preform at the position of “Biscuit”, the powder particles or slurries passed through a central gate to a “Runner”, and finally filled the “Plate” mould cavity.

This design of mold cavity was based on the hypothesis that severe plastic deformation helps to eliminate the oxides and pores in powder preforms. When powder

preform was injected through Biscuit to Runner, it suffered an extrusion and equal channel angular extrusion (ECAE). If injected further to Plate, the powder would be plastic deformed once more. Therefore, it is expected the consolidation strength in Plate would be the best. This expectation will be verified and described in Section 4.3.



(a)



(b)

Figure 4.11 (a) Schematic diagram and (b) dimensions of the mold cavity for powder thixocasting

Figure 4.12 shows a typical photograph of the powder thixocast specimens. It shows that the specimen is sound and has good surface appearance.

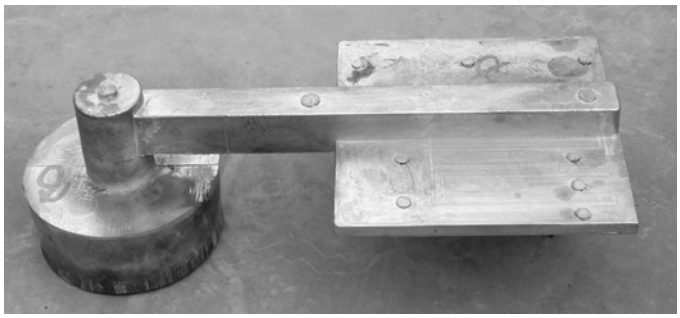


Figure 4.12 Photo of a powder thixocast specimen

4.2.3 Examinations of Material Properties

Through the following tests, the influenced of powder size, processing temperatures and heat-treatment time were investigated.

A. Microstructural observations

The microstructures of the powders before and after thixocasting were examined using an optical microscope. The etching solution for the observation was 0.5% HF water solution.

B. Temperature range for semi-solid state

To determine suitable heating parameters, the liquidus and solidus temperatures of the powder were revealed by using a differential thermal analysis (DTA), Du Pont Thermal Analyst 2100. In performing each DTA experiment, 40 mg powder was charged in an alumina crucible and was heated with heating rate of 10°C /min from 30°C up to 800°C and cooling rate of 10°C /min down to 30°C.

C. Isothermal Heat-treatment and Growth of Si particle size

Isothermal heat treatment tests were performed to elucidate the grain growth of primary Si particles during semi-solid processing of the Al-Si-Cu-Mg prealloyed powder.

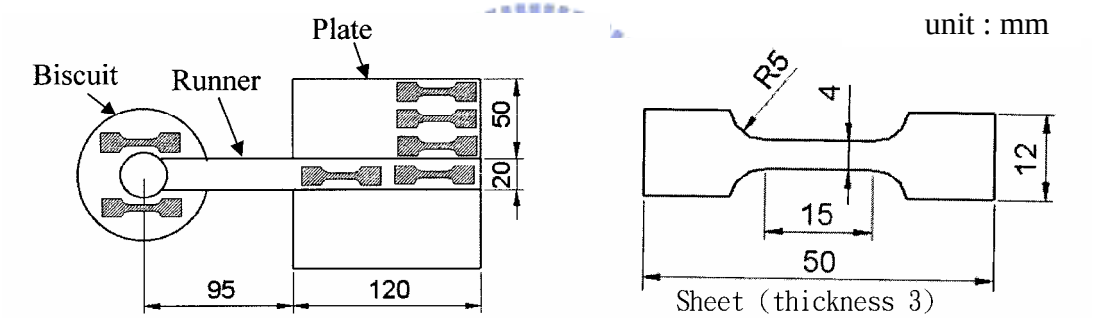
Around 1g of the powder charged in an alumina crucible was isothermally heated

in an electric furnace for various time intervals. The heated powder was then cooled immediately. The coarsening of Si particles was studied using an optical microscope.

The Si grain sizes were measured by the method of linear intercept. The mean linear intercepts L were the average of the linear intercepts of at least 100 Si grains measured for atomized powders with different areas, obtained using an optical microscope. The polyhedral Si grains were assumed to be uniform spheres, whose diameters *d* were calculated according to the relationship $d = 3L/2$, to simplify the calculation.

D. Tensile strength tests

Tensile specimens were machined from the three sections of the thixocast specimens, Biscuit, Runner and Plate, as shown in Fig. 4.13, which also shows the dimensions of the tensile specimens.



(a) (b)

Figure 4.13 (a) Locations and (b) dimensions of the tensile specimens.

E. Density test

The relative density of consolidated preforms were estimated from the volume of preforms and based on a theoretical density of the powder of 2.56.

The densities of powder thixocast products were measured by applying Archimedes' Principle. The density testing samples in cubic volume size of 5×5×5 mm^3 were machined from the three sections of the thixocast products, Biscuit, Runner and Plate.

4.3 Results and Discussions

4.3.1 How to Prepare the Powder Preforms Efficiently

A. The powder preforms were consolidated at high temperatures

In conventional aluminum PM, powder preforms are typically prepared at room temperature. However, to compress the preform at room temperature always requires a high compacting pressure, for example over 250 MPa, to ensure about 90% density, and additive of lubricant such as wax are required to ensure an acceptable compacting density of aluminum powder preforms. However, this high compacting pressure requires a high tonnage presser when a large preform is fabricated, which is considered to be uneconomic. Besides, the addition of lubricant always retards the bonding between powder particles and results in less strength of products.

In this study, the powder preforms were tried to be compacted at various elevated temperature. Thus the cost for making the powder preforms can be decreased, because they can be consolidated using low tonnage presser, for example a compacting pressure of only 85 MPa was needed in this study.

B. The relative densities of the preform increases with consolidating temperatures

Figure 4.14 presents a photograph of the powder preforms consolidated at various temperatures. The preforms consolidated at 250°C and 400°C exhibited cracks on their corners and interior. These cracks were generated when the preforms were extracted out from the mould, indicating that the powder preforms did not have enough strength to be handled if the consolidating temperatures were too low.

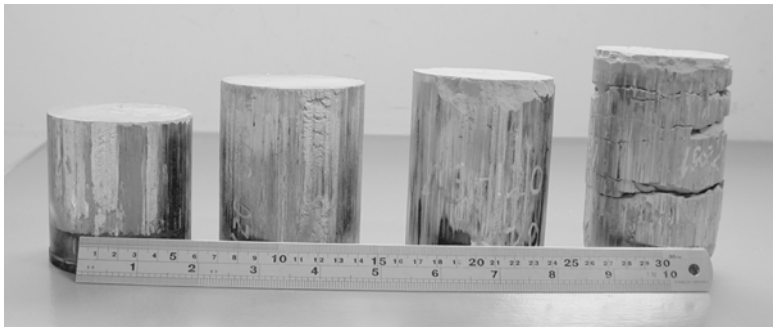


Figure 4.14 Photograph of the high-temperature consolidated powder preforms.

From left to right, they were hot consolidated under preheating temperatures of 550 ^{o}C, 500 ^{o}C, 400 ^{o}C, and 250 ^{o}C, respectively.

Table 4.3 lists the relative density of the powder compacts as a function of powder size and powder consolidation temperature. The relative density is the ratio of the compact density to the theoretical density of the bulk materials. Table 4.3 shows that the relative density increased quickly with the consolidating temperature, but fell slightly as the size of the powder increased. This increase in density with temperature is clearly due to the fact that the powder becomes softening at high temperature, causing the compressibility of the preforms to increase with temperature.

The compacts consolidated at 250°C and 400°C had relative density values that were too low, such that they were too weak to be handled. However, the compacts consolidated at 600°C showed the detrimental effect of Si grain growth. Consequently, only two powder consolidating temperatures, 500°C and 550°C, were used for thixocasting herein. The effects of the two consolidating temperatures on the thixocast specimen are considered below.

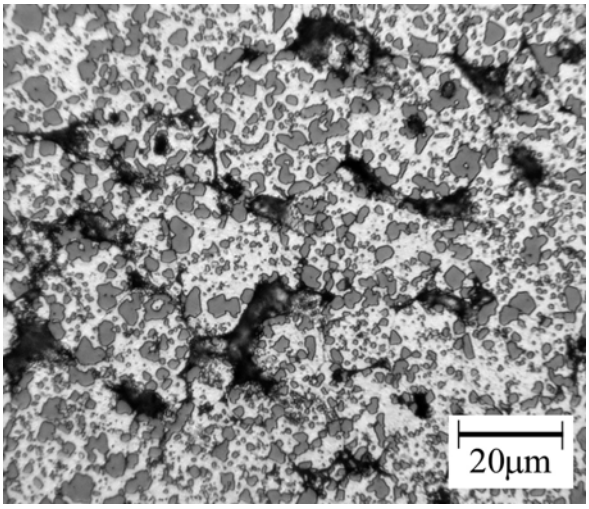
This compressibility improvement was similar to that reported in Low [4], who also introduced elevated temperature consolidation to increase the compressibility of hypereutectic Al-Si powders. Nevertheless, it is also worth to note in this study that the preform consolidated at 550°C has much higher density than that consolidated at 500°C. This great improvement should be attributed to semi-solid compressing, since the powder at 550°C is in a semi-solid state, as described in Section 4.3.2.

Table 4.3 The relative densities (%) of the green powder compacts consolidated at different temperatures and with different sizes of powders

Powder size	Powder Consolidation Temperature (^{o}C)				
	250	400	500	550	600
<45μm	-	-	79	93	97
45~120μm	60	67	75	92	96
120~300μm	-	-	74	90	95

Figures 4.15 (a)-(c) show a series of optical micrographs with representative microstructures of the green compacts consolidated at various temperatures. Comparing Figs. 5(a) and 5(b) reveals that the compacts consolidated at 500°C had much more inter-granular pores than those consolidated at 550°C. According to the DTA results, which will be detailed in Section 4.3.2, the powders are in a solid state at 500°C and in a semi-solid state at 550°C. Thus Fig. 4.15 (a) and (b) revealed clearly that the powders in the semi-solid state were much more compressible than those in the fully solid state. Figure 4.15 (c) presents the micrograph of the powder compact consolidated at 600°C, and shows that some liquid was squeezed out of the powders, filling the inter-granular pores of the compacts. In this case, although the density of the compacts became very high, the Si grains became much coarser. This severe Si grain growth was consistent with the results of the isothermal heat-treatment tests of the powder.

(a)



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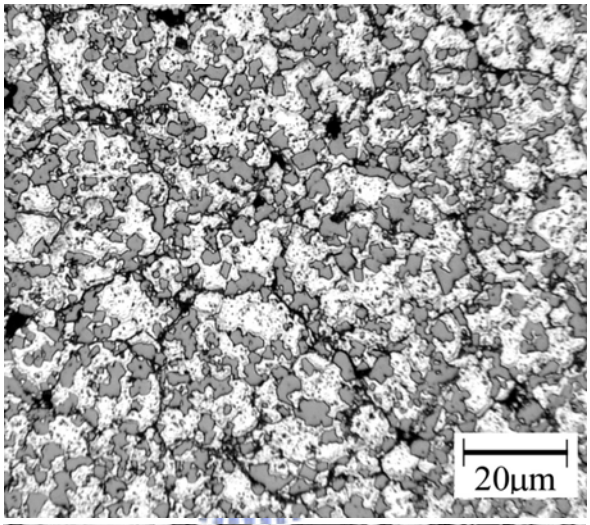

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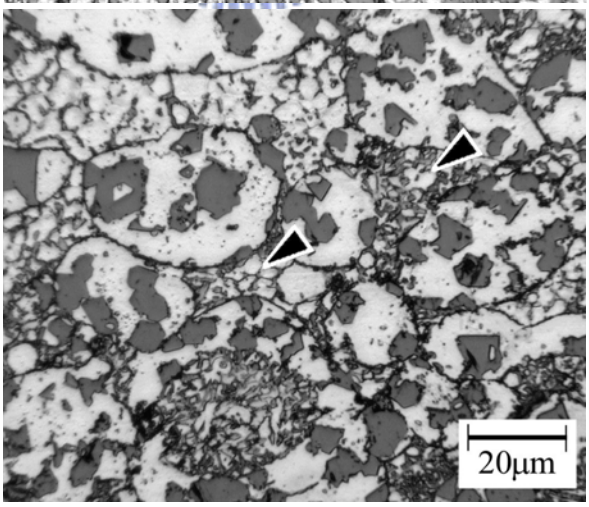

Figure 4.15 Optical microstructures of the green powder compact Those compacts were consolidated at (a) 500 ^{o}C, (b) 550 ^{o}C, and (c) 600 ^{o}C and using the hypereutectic Al-25Si-2.5Cu-1Mg -0.5Mn powders in size of< 45μm; the arrows indicate the region where the melt was squeezed out of the powder.

C. Low relative density of the preforms retard the efficiency of an induction heating

A high relative density of powder preform was found crucial to success the powder thixocasting in this study. The preforms of low relative density were found too weak to be handled; besides, such low density was also found to decrease the heating rate of the preforms by an induction coil to a semi-solid state. For example, despite numerous attempts to accelerate the heating rate, we were surprised to find that over 20 minutes continued to be necessary for achieving the successful homogeneity throughout the preforms that were consolidated at 500 °C, of relative density about 74%; whereas, the heating time was found able to shorten to less than 4 minutes for the preforms that were consolidated at 550 °C, of relative density about 90%;

In inductive heating, the heat input is concentrated only on the surface of a metal due to the “skin” effect of induction heating [79]. Penetration depth of the oscillating induction field into the metal is limited; for example, the penetration depth for aluminum alloys is generally just 3~5 mm [79]. In this study, therefore, as induction heating proceeded, only the exterior layer of the preform volume is rapidly heated by the oscillating induction field. The induction input heat then transfers from exterior to center of the preforms via heat conduction. However, if the relative density of the powder preform is too low, the excess porosities in the preforms may retard the transfer of heat from preform surface to its interior.

In addition, too much heat concentrated on the surface during induction heating would cause excess melting on the preform surface. In order to prevent the excess surface melting, the heating power should decrease stepwise. However, as the heating power was lowered down, it would take very long time to homogenize the temperature through the thickness of a preform of low relative density. Therefore, this may explain why a time-consuming heating process was always needed for the low-density preforms. In other words, the preforms of high volume of porosities are not acceptable for powder thixocasting because the inductive heat on the “skin” of such preforms will difficultly transfer into its interior, causing time-consuming or unevenly heating of the powder preforms. Consequently, the powder preforms were preferred to be consolidated at 550°C to obtain a high density of about 90% in this study.