

3. EXPERIMENTAL METHODS

3.1. Semi-solid metal processing

3.1.1. SSM-HPDC of rectangular plates

The SSM-processing of each batch of alloy A356 or F357 commenced by melting a master melt of A356 (with composition of 0.36% Mg in Table 3.1) in a 20 kg resistance heated tilting furnace. Additions of Mg and Al-10Sr were made to the melt to obtain the desired composition and thereafter the melt was degassed with argon. A356 alloys with Mg-contents less than that of the master melt ($< 0.36\%$ Mg) were obtained by allowing evaporation of Mg from the melt followed by subsequent addition of Sr to compensate for evaporation losses. A sample was poured from the melt, cooled and the chemical composition analysed by optical emission spectroscopy (Thermo Quantis OES). Table 3.1 shows the chemical compositions for each of the alloys. It can be seen that almost the whole Mg-composition range of these alloys is covered (Table 2.1).

The thermodynamic properties (liquidus and solidus) of the alloys were then calculated with an aluminium thermodynamic database (ProCAST) from the specific OES compositions by employing the Scheil equation (or non-equilibrium lever rule) [91]. The Scheil equation was used since solidification during SSM-HPDC does not occur under equilibrium conditions due to the fast cooling rates that are achieved. These thermodynamic properties were used to determine SSM processing parameters namely pouring temperature and SSM casting temperature. A pouring temperature of approximately 40°C above the liquidus and a SSM processing temperature corresponding to a solid fraction of $\sim 30\%$ were used from experience with this specific SSM processing system. For example, Figure 3.1 shows the solid fraction as a function of temperature for a typical A356 composition of Al-7Si-0.3Mg-0.1Fe-0.03Sr and F357 with composition of Al-7Si-0.6Mg-0.1Fe-0.03Sr. Table 3.2 summarises the calculated thermodynamic properties and derived rheocasting parameters for these compositions. It is seen that the processing parameters do not differ much for A356 and F357. This procedure was used for each of the compositions shown in Table 3.1.

Table 3.1: Chemical composition (wt%) of alloys A356 and F357 used for producing SSM-HPDC plates.

Al	Si	Mg	Fe	Cu	Mn	Ti	Sr
A356							
Balance	7.10	0.25	0.14	0.01	0.01	0.08	0.04
Balance	7.21	0.28	0.13	0.01	0.01	0.12	0.04
Balance	7.02	0.30	0.07	0.01	0.01	0.15	0.0002
Balance	7.15	0.31	0.13	0.01	0.01	0.09	0.02
Balance	7.14	0.34	0.14	0.01	0.01	0.08	0.03
Balance	7.14	0.36	0.10	0.01	0.01	0.07	0.02
Balance	7.08	0.38	0.10	0.01	0.01	0.12	0.02
Balance	7.08	0.40	0.10	0.01	0.01	0.08	0.02
F357							
Balance	6.81	0.45	0.12	0.01	0.01	0.09	0.02
Balance	6.80	0.49	0.13	0.04	0.01	0.13	0.02
Balance	7.20	0.52	0.08	0.02	0.01	0.12	0.0014
Balance	7.01	0.62	0.10	0.01	0.01	0.13	0.02
Balance	7.10	0.63	0.09	0.01	0.01	0.14	0.02
Balance*	7.20	0.67	0.25	0.01	0.01	0.16	0.04

* This alloy also contained 0.04% Ni - an order of magnitude higher than any of the other alloys. This was due to inadvertent contamination of the melt with stainless steel during degassing.

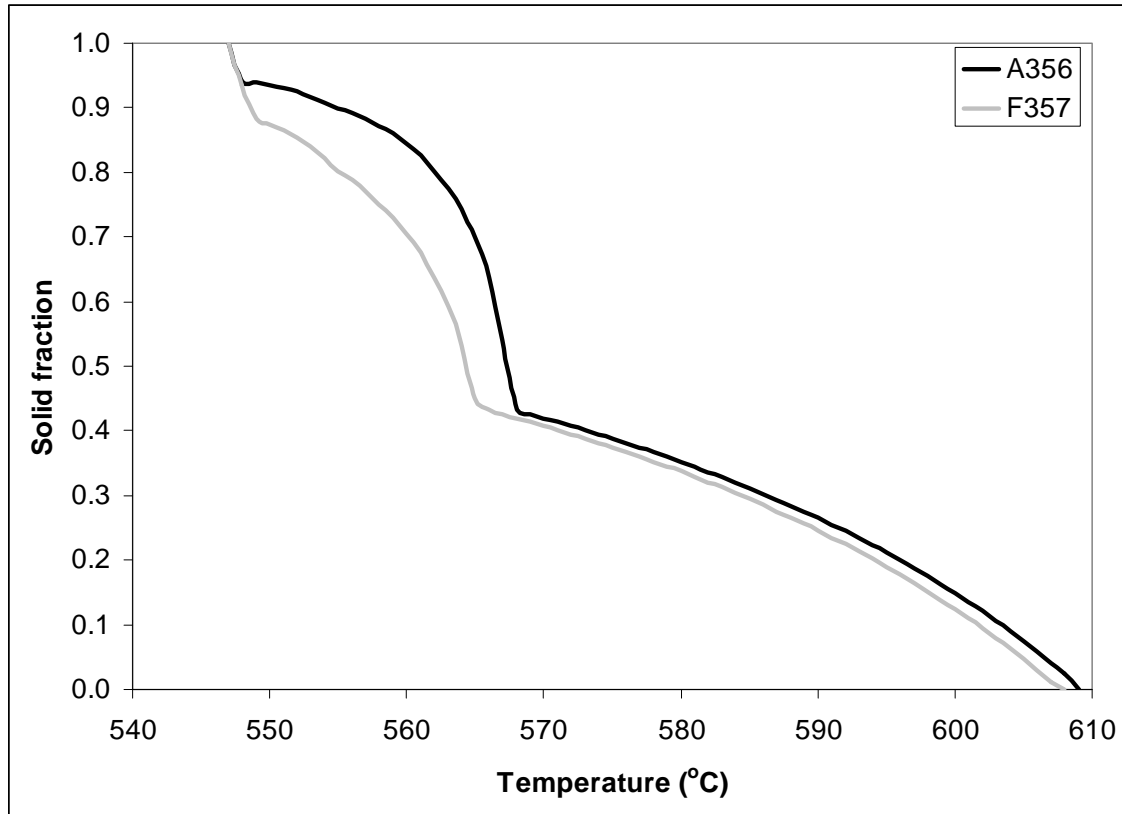


Figure 3.1: Solid fraction as a function of temperature calculated by ProCAST for typical A356 (Al-7Si-0.3Mg-0.1Fe-0.03Sr) and F357 (Al-7Si-0.6Mg-0.1Fe-0.03Sr).

Table 3.2: Calculated thermodynamic and derived rheocasting parameters of typical A356 and F357 (from Fig. 3.1) for the CSIR-RCS-HPDC cell.

Parameter	Property	A356 temperature (°C)	F357 temperature (°C)
Thermodynamic	Liquidus	609	608
	Solidus	548	548
Rheocasting	Pouring	645	645
	SSM	586	584

The sequence for casting was then as follows: liquid metal was poured from the tilting furnace into the stainless steel processing cup (about 400 g), which was then manually transferred to a single coil version of the CSIR-RCS (induction stirring with simultaneous forced air cooling [31]), where processing started once the cup entered the coil (Fig. 3.2).



Figure 3.2: Single coil version of the CSIR rheocasting system.

The semi-solid temperature of the alloy in the cup was measured by a thermocouple. Processing stopped after the thermocouple signal reached the preset SSM temperature calculated as shown in Table 3.2. At this point, the cup was ejected from the coil and manually transferred to the HPDC machine (initially with an Edgewick machine with a 50 ton clamping force, later replaced with an LK machine with a 130 ton clamping force – Fig. 3.3). The injection shot was manually triggered when the SSM billet was in the shot sleeve. The piston followed the set computer controlled injection velocity profile. Plates having dimensions of $4 \times 80 \times 100 \text{mm}^3$ for the Edgewick machine and $6 \times 55 \times 100 \text{mm}^3$ for the LK machine were cast. The dies used to cast the plates are shown in Fig. 3.4 (with the $4 \times 80 \times 100 \text{mm}^3$ plate cavity insert) and Fig. 3.5 (with the $6 \times 55 \times 100 \text{mm}^3$ plate cavity insert).



Figure 3.3: LK DCC130 shot controlled high pressure die casting machine.

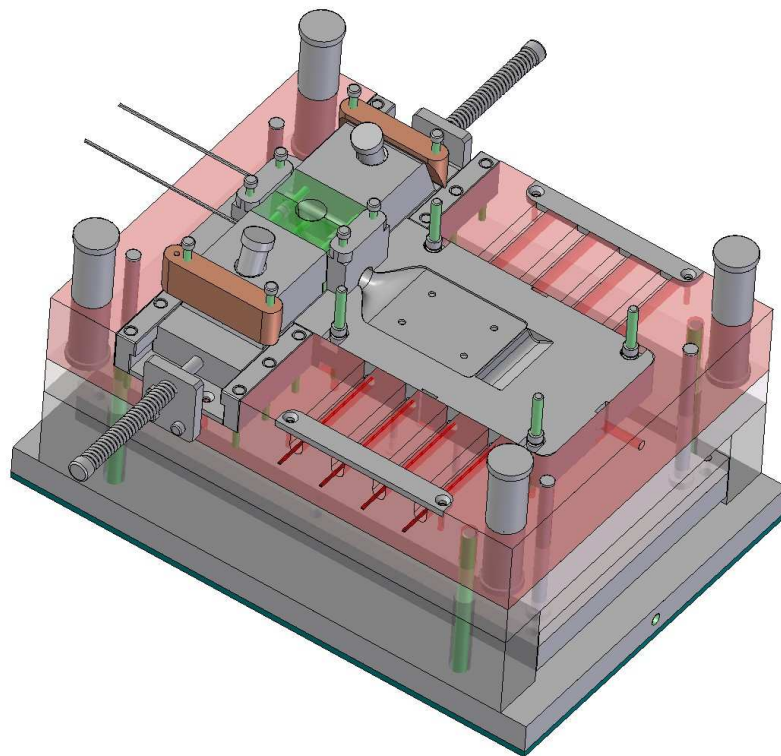


Figure 3.4: Moving half of the plate die (containing the $4 \times 80 \times 100 \text{mm}^3$ cavity insert) used with the 50 t Edgewick HPDC machine.

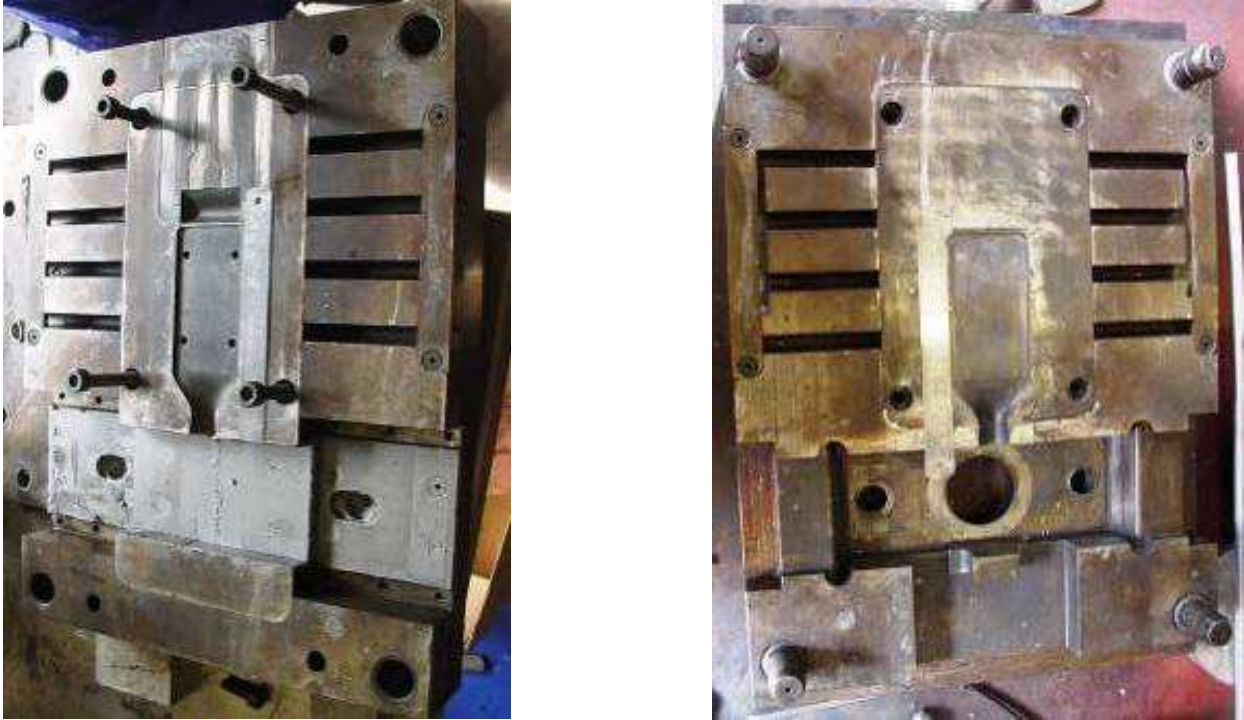
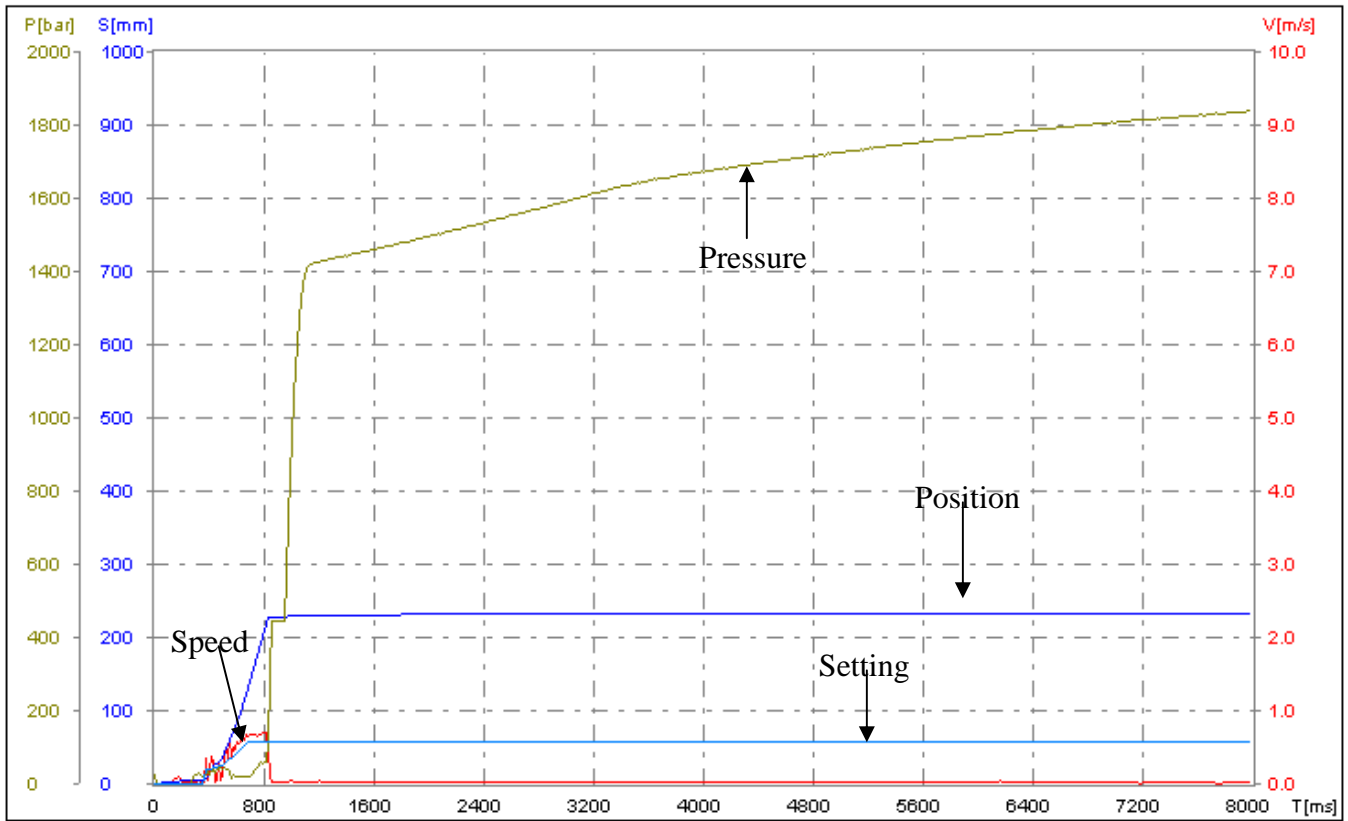


Figure 3.5: Moving half (on the left) and fixed half (on the right) of the plate die (containing the $6 \times 55 \times 100 \text{mm}^3$ cavity insert) used with the LK DCC130 HPDC machine.

The tool material used was W302 (AISI H13, DIN 1.2344) hot working die steel with a hardness of HRC = 46. It was cartridge heated to a temperature of 250°C and lubricated using ISOLAT ENVIRO 911N from Fochem International (Pty) Ltd.

Both the Edgewick and LK DCC130 machines have shot control. However, the Edgewick machine is relatively old and the shot control was not very sophisticated. The shot control system was mainly used to control the injection speed and for multiple step injection profiles. The shot profile was composed of two phases, a low speed of $\sim 0.3 \text{ m/s}$ and a high speed of $\sim 0.5 \text{ m/s}$. In order to achieve the full intensification pressure available, the stroke length was set to a distance longer than that required to achieve intensification after complete filling. A low speed and high speed of ~ 0.45 and 0.85 m/s respectively were used with the LK DCC130 machine. With this machine, intensification could be triggered using a preset back pressure or plunger position. The plunger position was used for the casting of the A356/7 plates. Typical speed and pressure profiles during SSM-HPDC of a F357 plate ($6 \times 55 \times 100 \text{mm}^3$) using the LK DCC130 machine are shown in Fig. 3.6.



Name: Time Position Speed Castpress Setting
 Color: — — — —
 Value:

Figure 3.6: Speed and pressure profiles during SSM-HPDC of a F357 plate (6x55x100mm³) using the LK DCC130 machine.

Figure 3.7 shows the whole casting including the biscuit and the runner of the plate cast using the LK DCC130 machine.

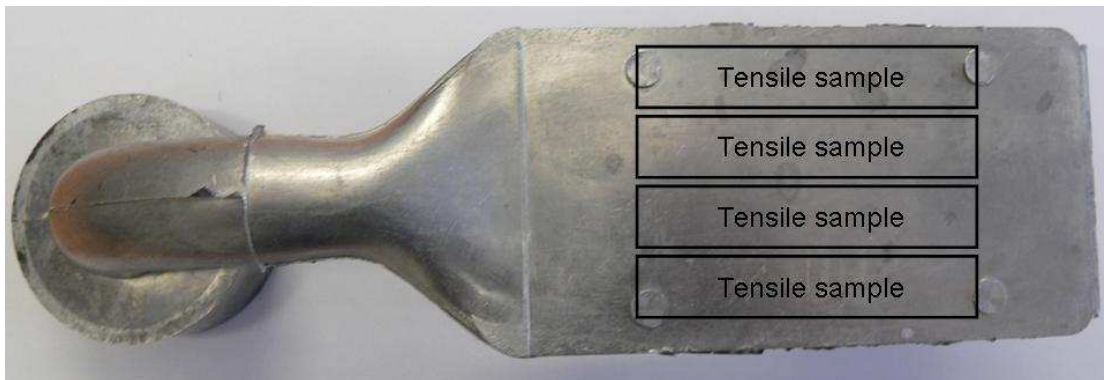
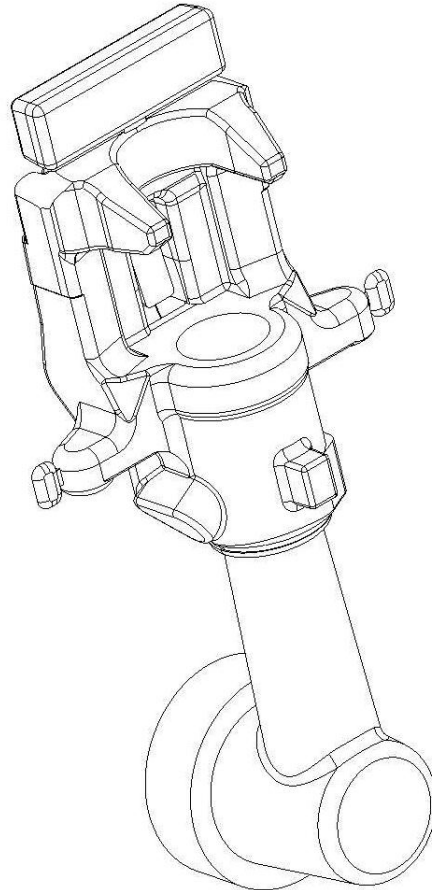


Figure 3.7: SSM-HPDC plate (6x55x100mm³) including the runner and biscuit, also indicating where the tensile samples were machined from.

3.1.2. SSM-HPDC of automotive brake callipers

Automotive brake callipers were also produced with SSM-HPDC to determine whether the heat treatment cycles that were developed for the small rectangular plates could also be applied successfully to a larger (and more complex) casting. Figure 3.8 shows the brake calliper casting in drawing (including the runner and the biscuit) [92]. The overall dimensions for the casting in Figure 3.8 are 327mm high \times 163mm wide \times 95mm thick with the biscuit and gate diameters 78mm and 35mm respectively.



SIZE : A4

Figure 3.8: Automotive brake calliper including runner and biscuit [92].

To accomplish SSM-HPDC of the automotive brake calliper, the industrial scale version of the CSIR-RCS-HPDC cell was used (Fig. 3.9). The main components that make up the cell are: a 240 kg dosing furnace (WESTOFEN WESTOMAT W25S), an automated robotic arm (FANUC ROBOT M-16iA), a three-coil version of the CSIR-RCS (induction stirring with simultaneous forced air cooling [31]) processing unit and a 630 ton clamping force HPDC machine with full shot control (LK DCC630). The mechanical hardware operation of the cell runs automatically through computer

control software (NATIONAL INSTRUMENTS LABVIEW8.6). The 240 kg dosing furnace was filled by melting commercial aluminium alloy A356 ingots in charges of approximately 40 kg each with a custom-made induction heating ladle furnace. The metal temperature in the dosing furnace was left to equilibrate after the last charge (with additional Sr) was added. A sample was poured into a cup and chilled for chemical analysis by OES. The composition of the aluminium alloy A356 melts are given in Table 3.3 and the pouring temperature and SSM-processing temperature were determined as described before for the plates.

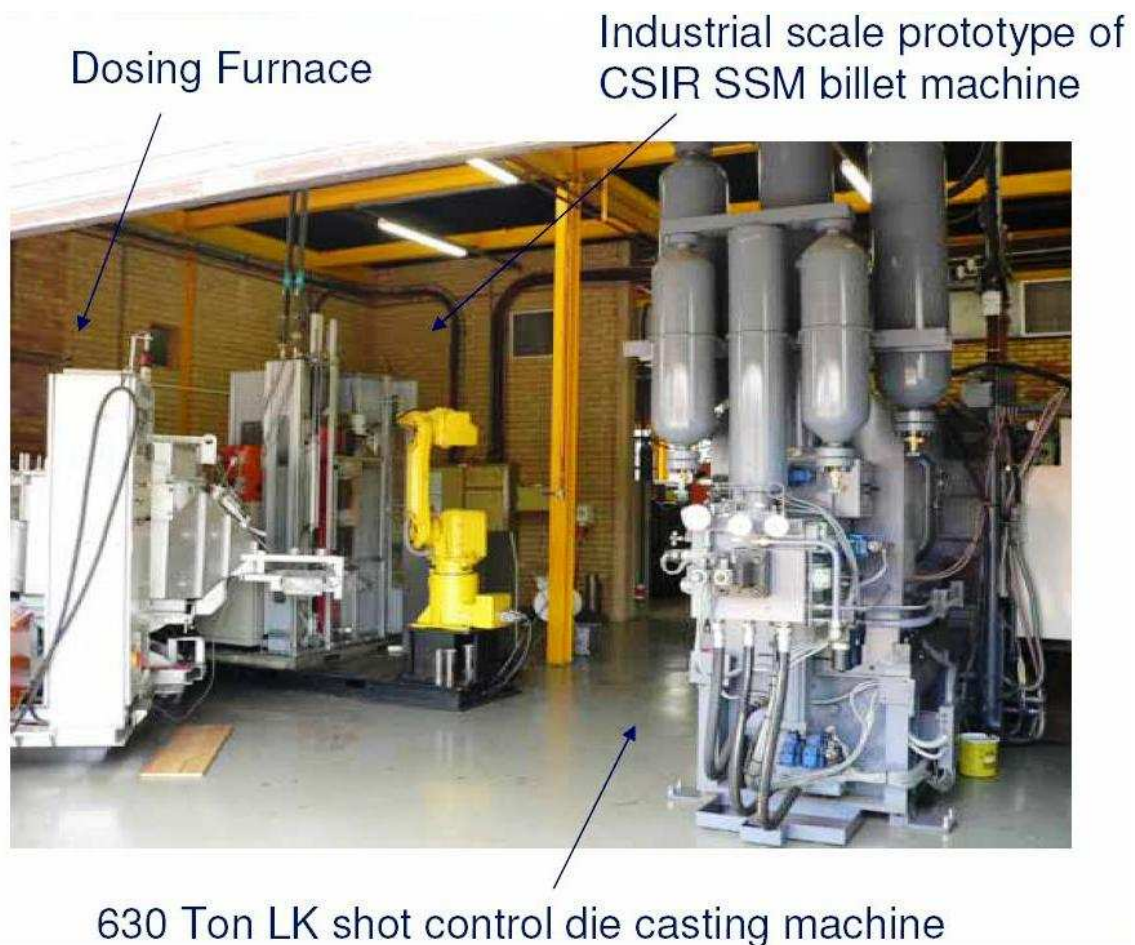


Figure 3.9: Industrial scale version of the CSIR-RCS-HPDC cell with dosing furnace and LK DCC630 HPDC.

Table 3.3: Chemical composition (wt%) of alloy A356 used for producing SSM-HPDC and gravity die cast (GDC) automotive brake callipers.

Method	Al	Si	Mg	Fe	Cu	Mn	Ti	Sr
SSM-HPDC (1)	Balance	7.3	0.31	0.14	0.01	0.01	0.16	0.03
SSM-HPDC (2)	Balance	7.0	0.35	0.14	0.01	0.01	0.14	0.02
GDC	Balance	6.6	0.36	0.27	0.03	0.01	0.06	0.02

The sequence for casting was as follows: liquid metal was poured from the dosing furnace into a stainless steel processing cup (approximately 2 kg) which was then robotically transferred to the CSIR-RCS unit and entered the coil train at the bottom. All three coils in the processing unit were inductive while forced air cooling was only used (manually activated) on the top coil to control the SSM temperature which was measured with a thermocouple in the semi-solid metal contained in the cup. When this cup at the top of the train had been processed after reaching the SSM temperature, it was robotically transferred to the shot sleeve of the HPDC machine and the injection shot was triggered automatically to fill the die. The die temperature was controlled by two oil heaters (REGLOPLAS 300 L), one for each half of the die, and was set at 170°C. The piston followed a set computer-controlled injection velocity profile to achieve the best filling result [92]. To make comparisons between the heat treatment response of globular and dendritic A356, automotive brake callipers of similar composition (Table 3.3) and exact design (shape, size, mass) were cast by a local manufacturer using gravity die casting (GDC).

3.2. Investment casting of rectangular plates

To make further comparisons of the heat treatment response of globular and dendritic A356, rectangular plates (95x30x4mm³) with a composition given in Table 3.4 were cast using investment casting (IC) to obtain a coarser dendritic microstructure than achieved in the brake calliper cast using GDC.

Table 3.4: Chemical composition (wt%) of alloy A356 used for producing investment cast (IC) plates.

Al	Si	Mg	Fe	Cu	Mn	Ti	Sr
Balance	6.70	0.25	0.10	0.01	0.01	0.06	0.03

Wax pattern assemblies consisted of 6 of the plates assembled with 3 plates per opposite side, spaced 40 mm apart. A vertical 25mm x 20mm rectangular runner bar was used (250mm long) as shown in Fig. 3.10. The plates were assembled at an angle of 75 degrees inclined to the runner bar. The runner bar extended below the lowest plate's tip to form a dross trap. A pouring cup was attached to the top of the runner bar. The wax used was Remet 289B green wax. The preferred bottom pouring method for aluminium was not used to simplify the assembly and to ensure symmetry in the mould. Wax assemblies were prepared by washing with Pattern Wash 6 from Remet and rinsed with de-ionised water. The wax assemblies were left for 24 h to dry and to stabilise at the dipping room temperature of 21°C. The primary slurry consisted of Ransom & Randolph Primecote, colloidal silica 30% binder and Zircon flour (Zircon silicate) -325mesh as refractory filler. The secondary slurry consisted of Ransom and Randolph Customcote colloidal silica 25% binder with fused silica (-325mesh) as the refractory filler. The face coat stucco used was Zircon sand P109 (mean 109µm) and the back-up coat stucco was Chamotte (Aluminosilicate). The shell making facility was temperature controlled to 21°C±1°C and the humidity was monitored – the relative humidity was measured as ±60%. The dipping procedure is given in Table 3.5.



Figure 3.10: Wax pattern assembly used for investment casting (IC) of alloy A356.

De-waxing (wax removal) was performed using a standard LBBC steam boilerclave with 200°C steam at 8 bar pressure for 15 min. The moulds were pre-fired in a gas fired furnace with a 5% oxidising atmosphere to a temperature of 800°C, kept for 2 h, and furnace cooled. The mould was inspected and vacuumed to ensure that the mould was clean and preheated to the required casting temperature and soaked for 1 h in an electric kiln furnace. The metal and mould temperature during casting was 720°C to ensure a relatively coarse microstructure. Aluminium alloy A356 was melted in a SiC crucible in an electric furnace. Melting was timed to reach 720°C as the mould reached the 1 h soaking time. This was to reduce metal time at temperature to reduce hydrogen pick-up as no de-gassing was done due to the small volume of the melt. The mould and metal was removed from the furnace at the same time. The mould was

suspended in still air on a mould stand and the metal surface was skimmed to remove dross and slowly poured into the mould. The mould was left to cool to room temperature and mould removal was done by hand.

Table 3.5: The dipping procedure for investment casting of alloy A356.

Coate No:	Slurry	Stucco	Drying Time	Drying Method
Primary 1	Zircon Primary	Zircon Sand	8 hours	Air dry
Primary 2	Zircon Primary	Zircon Sand	8 hours	Air dry
Secondary 1	Customcote	Chamotte 0.25- 0.7mm	45 Min	Fan
Secondary 2	Customcote	Chamotte 0.25- 0.7mm	45 Min	Fan
Back-up 1	Customcote	Chamotte 0.7- 1.2mm	45 Min	Fan
Back-up 2	Customcote	Chamotte 0.7- 1.2mm	45 Min	Fan
Back-up 3	Customcote	Chamotte 0.7- 1.2mm	45 Min	Fan
Back-up 4	Customcote	Chamotte 0.7- 1.2mm	45 Min	Fan
Seal coate	Customcote	-----	24 Hours	Air

3.3. SSM-HPDC of Al-Mg-Si wrought alloys 6082 and 6004

To make direct comparisons of SSM-HPDC Al-7Si-Mg casting alloys with SSM-HPDC Al-Mg-Si wrought alloys, SSM slurries of alloys 6082 and 6004 (chemical compositions of the melts are given in Table 3.6, as well as the upper and lower limit specifications for the different elements [33]) were prepared using the CSIR-RCS single coil version. Rectangular plates ($6 \times 55 \times 100 \text{mm}^3$) were cast in steel moulds with an LK DCC130 HPDC machine (Fig. 3.3). Special emphasis was placed on the effects of prior natural aging on subsequent artificial aging and alloy 6082 was selected as a

reference alloy in which natural aging has a negative influence on artificial aging, whereas a second reference alloy 6004 is expected to show the opposite effect (Fig. 2.32). For microscopy, the 6082 and 6004 were etched using Barker's anodizing method.

Table 3.6: Chemical composition limits for wrought Al-Mg-Si alloys 6082 and 6004 [33], as well as the compositions of the alloys used in this study.

Alloy	Si	Mg	Fe	Mn	Cu	Ti
6082	0.82	0.84	0.22	0.58	0.03	0.02
Spec low	0.70	0.60	-	0.40	-	-
Spec high	1.30	1.20	0.50	1.00	0.10	0.05
6004	0.47	0.45	0.16	0.22	0.02	0.01
Spec low	0.3	0.40	0.10	0.20	-	-
Spec high	0.6	0.70	0.30	0.60	0.10	0.05

3.4. Thermo-Calc

Thermo-Calc (a commercially available software package used to perform thermodynamic and phase diagram calculations for multi-component systems of practical importance) was used to investigate the possible effects of variable compositions on the equilibrium phases in some of the alloys, using the Al-DATA ver. 2 database.

3.5. Heat treatment of castings

3.5.1. Solution treatment

All solution treatments were performed in a CARBOLITE HRF 7/22 air circulating furnace with a fan to ensure a uniform temperature distribution. Solution treatment temperature was not considered as a variable in this study since a temperature of 540°C gives the best compromise between shortening heat treatment time as well as minimising the risk of blistering and distortion [72,90]. However, solution treatment times were varied between 0.5 to 24 h.

3.5.2. Quench after solution treatment

All samples were quenched in room temperature water after the solution heat treatment, except for samples from the batches with 0.36 and 0.40% Mg (Table 3.1), where quenching was also performed in 70°C water.

3.5.3. Natural aging and the T4 temper

Natural aging curves were determined by measuring Vickers hardness (VHN) using a FUTURE-TECH FV-700 Vickers hardness tester by employing a 10 kg load from the average of at least four readings per sample. The average hardness values were reproducible within ± 3 VHN for all samples tested.

3.5.4. Artificial aging and the T5 and T6 tempers

All artificial aging heat treatments were performed in the same furnace as above. The T5 temper condition was investigated using the batch containing 0.63% Mg in Table 3.1. The variables that were used to test the SSM-HPDC F357 in the T5 temper condition are shown in Table 3.7.

Table 3.7: Heat treatment parameters for SSM-HPDC F357-T5.

Quench after SSM-HPDC	Natural pre-aging	Artificial aging
Air (AQ)	25°C-120h	180°C-4h
Water (WQ)	-	180°C-4h
Water (WQ)	25°C-120h	180°C-4h
Water (WQ)	25°C-120h	160°C-28h

The T6 temper condition was studied by determining artificial aging curves at temperatures ranging from 160–190°C, following natural pre-aging periods ranging from 0–120 h. Vickers hardness measurements were done as above. In certain samples, the micro-Vickers hardness of the primary α -Al globules and the eutectic regions were also determined by using a FUTURE-TECH FM-700 Vickers microhardness tester by employing a 50 g load.

Samples used for the aging curves were cut from the plates and were deliberately kept relatively small at $\sim 4 \times 20 \times 20 \text{ mm}^3$ to avoid variations in quench rates achieved after the solution heat treatments.

3.6. Tensile testing

The tensile properties of the alloys were determined using an INSTRON 1342/H1314 with 25 kN load cell capacity and an INSTRON Model 2620-602 extensometer with gauge length of 12.5mm. To determine the 0.2% proof stress, a stress rate of 10MPa/s was used and for the ultimate tensile stress (UTS) determination a displacement rate of 10mm/min. These parameters were selected based on the American Society for Testing and Materials (ASTM) standard E8M-04. The extensometer was removed during tensile testing after reaching the 0.2% plastic strain value (typically after 1-2% strain) to prevent damage to the extensometer in the case of premature fracture. The % elongation after fracture was calculated in accordance with ASTM standard E8M-04, where gauge marks were drawn with ink on the tensile specimens. After fracture, the ends of the fractured specimen were fitted together carefully and the distance between the gauge marks measured. Tensile specimens that were machined from the plates and brake callipers are shown in Figures 3.11 and 3.12 respectively.

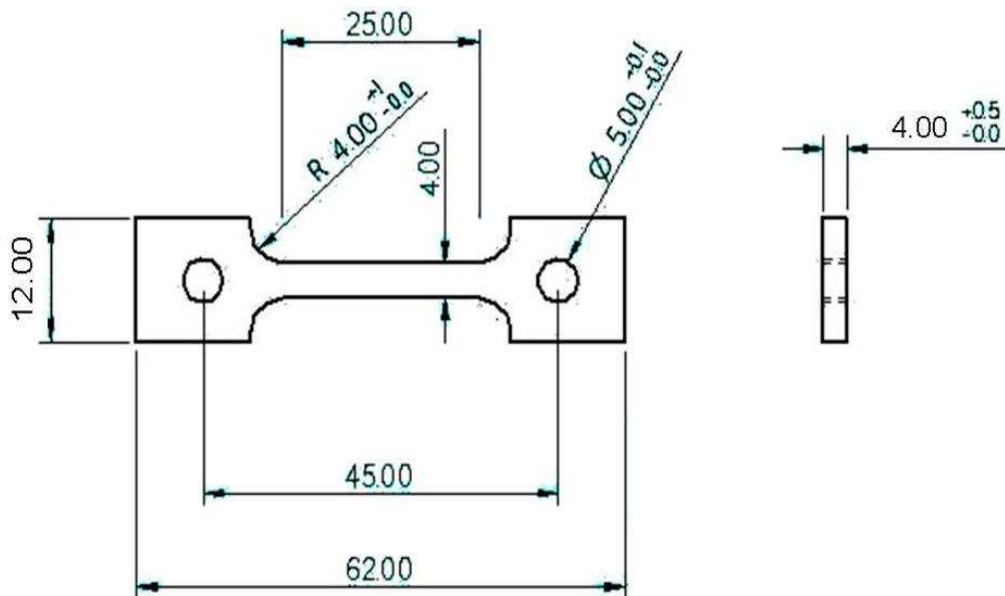


Figure 3.11: Tensile specimens with dimensions in mm machined from plates.

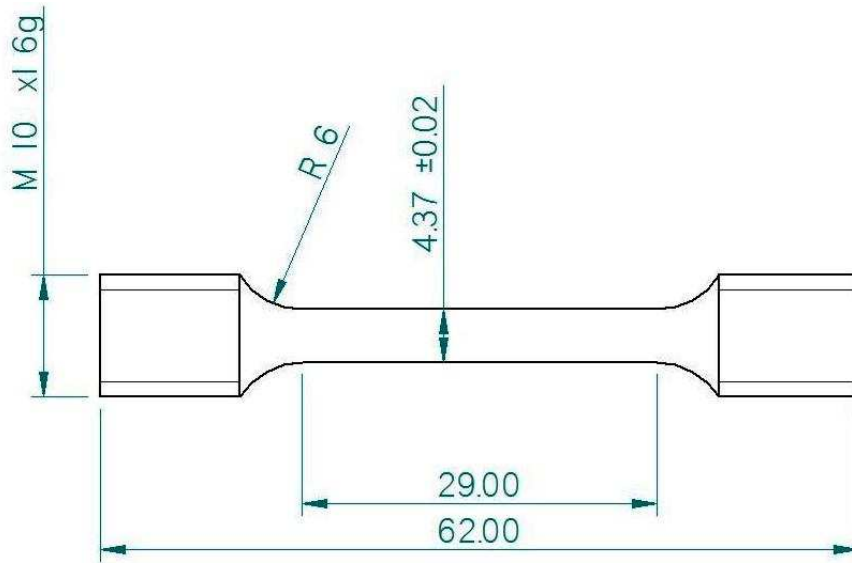


Figure 3.12: Tensile specimens with dimensions in mm machined from brake callipers.

Four tensile samples were machined from each $6 \times 55 \times 100 \text{ mm}^3$ plate (Fig. 3.7) and five from each $4 \times 80 \times 100 \text{ mm}^3$ plate. The positions in the brake callipers where the tensile samples (Fig. 3.12) were machined from are shown in Fig. 3.13.

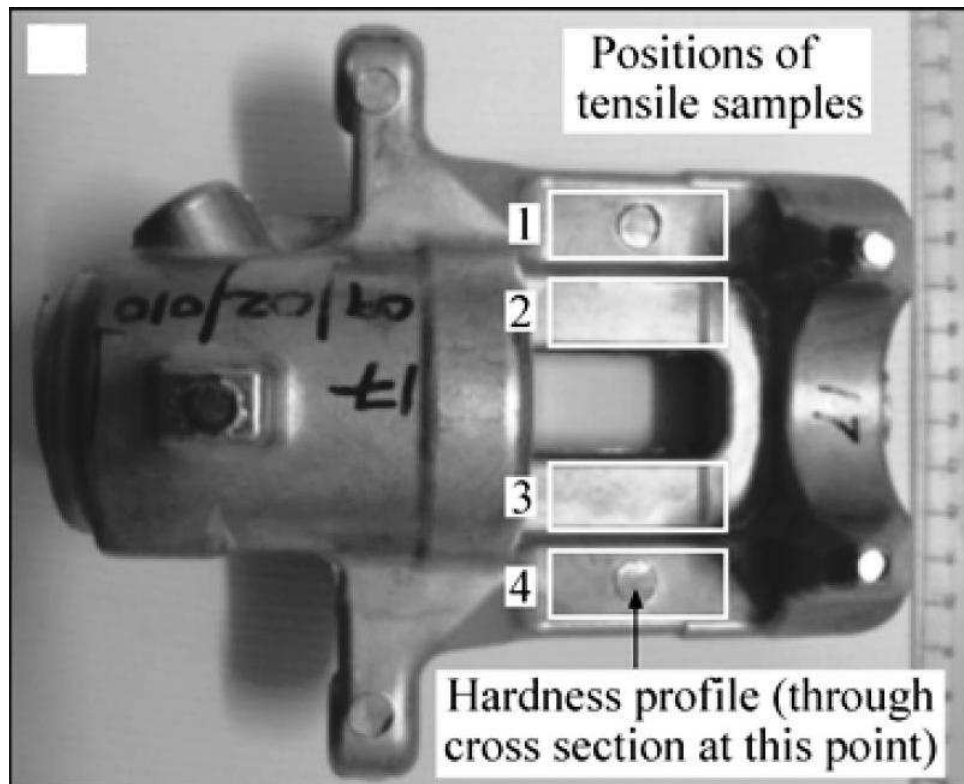


Figure 3.13: Positions where 4 tensile specimens/brake calliper were machined from.

3.6.1. The Quality Index (QI)

The “Quality Index” (QI) was used in this work to allow comparison of different compositions and heat treatments. The quality index relates the ductility (% elongation or %A) and strength (ultimate tensile strength or UTS) into a single term. It was originally developed by Drouzy and co-workers [93]. The quality index (specifically for alloys A356/7) is given by equation 3.1:

$$\text{QI (MPa)} = \text{UTS (MPa)} + 150\log(\% \text{ elongation}) \quad (3.1)$$

The rationale behind this equation lies in the well known phenomenon that for most mechanisms of strengthening in alloys (except for grain refinement) one has to sacrifice some ductility or toughness and a high QI, therefore, aims to find a combination of high strength and high ductility or toughness in the alloy.

3.7. Impact testing

The impact strengths of samples from batches with 0.25, 0.31 and 0.45% Mg respectively (Table 3.1) were determined using a ZWICK impact tester with a 40 kpcm hammer. Non-standard sized Charpy specimens (55x10x3mm³ with a 45° V-notch of 2mm depth) were machined from the plates. Subsequently, the impact properties of samples from the batch with 0.38% Mg (Table 3.1) were determined by an INSTRON DYNATUP 9210 drop weight impact tester using the sub-standard size Charpy specimens for each temper condition (F, T4, T5, T6). The mass of the weight for the drop weight test was 5.5 kg. In order to achieve a total projectile energy of 5 J, a drop height of 9.3 cm was required. This resulted in a speed at impact of 1.35 m/s. Maximum sensitivity was obtained by using a 15 kN load cell.

The validity of using Charpy impact testing to measure the fracture properties of aluminium alloys has been questioned before [94]. Therefore, the impact results in this thesis are considered only as a comparative means to rapidly differentiate between different conditions.

3.8. Optical microscopy and scanning electron microscopy

The samples used for optical microscopy and scanning electron microscopy were mounted in an electro-conductive resin prior to grinding on SiC papers from P120 to

P2400 grit. This was followed by polishing using 9 to 1 μm diamond suspension, finishing with 50 nm colloidal silica. An optical microscope (LEICA DMI5000 M) equipped with a camera (LEICA DFC480) and imaging software (Image-Pro MC v6.0) was used to study the microstructures of the alloys. Scanning electron microscopy (SEM) was performed with a JEOL JSM-6510. Etching was performed using an aqueous solution containing 0.5% HF.

3.9. Transmission electron microscopy (TEM) and Atom probe tomography (APT)

The TEM and APT investigations were conducted at the Australian Centre for Microscopy & Microanalysis (ACMM), The University of Sydney, Australia. Thin foil samples of the alloy from the batch with 0.49% Mg (Table 3.1) were prepared from punched 3 mm discs using electrochemical jet polishing in a solution of 30% nitric acid in methanol at -30°C . TEM studies were performed using a PHILIPS CM120 operated at 120 kV. Needle-like samples with fine tips with radii ~ 50 nm for APT were prepared from blanks with dimensions of $0.3 \times 0.3 \times 15 \text{mm}^3$ using a two-stage electropolishing technique [95]. The first stage involved electropolishing in a room temperature solution of 25% perchloric acid in acetic acid at 15V, whereas the second stage was conducted in a room temperature solution of 5% perchloric acid in 2-butoxyethanol at 20V. APT analysis was done using a Local Electrode Atom Probe (LEAP®) manufactured by Imago Scientific instruments, under ultrahigh vacuum ($\sim 1 \times 10^{-8}$ Pa) at a sample temperature of ~ 20 K, with a proven detector efficiency of this instrument of 57% [96]. The pulse fraction or ratio of pulse voltage to DC standing voltage employed was 20% with a pulse repetition rate of 200 kHz. In spite of concerns regarding preparation of APT samples for casting alloys, measurements with more than 8 million atoms were attained, which is significantly more than what has been reported for these alloys before [11]. Reconstruction and visualization of the APT data was done with the Imago Visualization and Analysis Software (IVAS™) in combination with advanced calibration techniques [96,97]. For quantification of clusters, the maximum separation algorithm was employed [98]. Mg, Si and Cu atoms were identified as clusters if they had a separation distance of less than 0.6 nm. A surrounding distance from these atoms of 0.5 nm was chosen to include all other elements. The smallest solute clusters taken into account were designated to contain at

least $n = 10$ solute atoms in order to minimise the effect of small solute clusters that exist in the alloy having a random distribution of solutes [99]. Areas within the primary α -Al grains were used for study for both TEM and APT, rather than the eutectic α -Al grains. The rate of nucleation and growth of precipitates has been shown to be different in the eutectic region of Al-Mg-Si alloys, which is a high solute density region of the material, compared to the primary α -Al grains [100].