Doctoral Thesis

Process and alloy development for high quality aluminium components for lightweight construction - the new rheocasting process

Author(s):
Wabusseg, Heimo

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PROCESS AND ALLOY DEVELOPMENT FOR HIGH QUALITY ALUMINIUM COMPONENTS FOR LIGHTWEIGHT CONSTRUCTION - THE NEW RHEOCASTING PROCESS

A dissertation submitted to the
SWISS FEDERAL INSTITUTE OF TECHNOLOGY
for the degree of
Doctor of Technical Sciences

presented by

Heimo Wabusseg
Dipl.-Ing. mont.
born July 25, 1972
citizen of Austria

Accepted on recommendation of
Prof. Dr. P.J. Uggowitzer, examiner
Prof. Dr. S. Virtanen, co-examiner
Dr. H. Kaufmann, co-examiner
Prof. Dr. M.O. Speidel, co-examiner

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Christian Haslinger and Gottfried Rettenbacher, who supported me in the casting trials on the NRC-machine at LKR. And I will not forget the cool beers after the hot casting sessions.

Above all, I would like to thank my wife Alexia and our little bundle of joy Julia as well as my parents, to whom all I dedicate this thesis. Alexia, you made my written English really presentable and I bet it was not always easy for you and Julia to put up with a slightly grumpy hubby towards the tight deadline of my thesis. LUBAL - thank you!
In this work the principles behind the New Rheocasting process are described and its advantages over existing semi-solid forming processes are outlined. An overview of the theories that govern the development of the globular microstructure, essential feature of the New Rheocasting process, is given. In particular, the applicability of the wall mechanism theories with regard to nucleation are discussed. The effects of casting temperature and cooling rate on the resulting microstructures in the New Rheocasting process were investigated for three aluminium based alloys. The microstructures are described with the aid of parameters such as grain size, shape factor, contiguity and contiguity volume. The obtained microstructures were also compared to microstructures achieved with a "cellular automaton model". The alloys AlSi7Mg0.3, AlSi7Mg0.6 and AlMgSi1 as well as their modifications with Ba, and AlSi9Cu3, representing different categories of aluminium alloys and, therefore, different fields of applications, were investigated. More specifically, AlSi7Mg0.3 and AlSi7Mg0.6 are casting alloys and successfully implemented in existing semi-solid forming processes, whereas AlMgSi1 and AlSi9Cu3 are a wrought alloy and a secondary alloy, respectively. The latter is the most frequently used cast alloy and its production presents a significant cost advantage. The flow behaviour in the semi-solid state, in addition to the static and dynamic mechanical properties for each of the alloys are presented. Furthermore, for the secondary alloy AlSi9Cu3 and its adaptions with a reduced Si-content, wear and corrosion properties were investigated. The results of the investigations carried out in this work demonstrate the high applicability of the New Rheocasting process as a new semi-solid forming technology for the fabrication of complex components with outstanding mechanical properties. Moreover, an evaluation of possible new alloys, thanks to the New Rheocasting process for semi-solid forming is presented. This work began at the initial stages of the introduction of the process into the market and provided the base for the process implementation for aluminium alloys. The success obtained in the development of aluminium alloys is likewise foreseeable in the near future for magnesium alloys as well as for other high-melting metal alloys.
ZUSAMMENFASSUNG

In der vorliegenden Arbeit werden die Grundlagen zum Verständnis des New Rheocasting-Prozesses beschrieben und seine Vorteile gegenüber bereits existierenden Semi-solid-Formgebungsverfahren hervorgehoben. Neben einem Überblick über die Theorien zur Entwicklung der globularen Gefügeausbildung, die ein essentielles Merkmal des New Rheocasting-Prozesses darstellt, werden im besonderen die Theorien der Wandmechanismen zur Keimbildung erörtert.


Die Legierungen AlSi7Mg0.3, AlSi7Mg0.6, AlMgSi1 und deren Modifikationen mit Ba, sowie AlSi9Cu3, die verschiedenen Kategorien von Aluminiumlegierungen angehören und daher in unterschiedlichen Anwendungsgebieten eingesetzt werden, wurden untersucht. AlSi7Mg0.3 und AlSi7Mg0.6 sind Gusslegierungen und werden bereits erfolgreich in existierenden Semi-solid-Formgebungsverfahren verarbeitet, während AlMgSi1 zum Bereich der Schmiedelegierungen und AlSi9Cu3 zur Familie der Sekundärlegierungen zu zählen sind. Die Legierung AlSi9Cu3 ist die am häufigsten eingesetzte Gusslegierung und weist einen erheblichen Kostenvorteil auf.


Die im Rahmen dieser Arbeit durchgeführten Untersuchungen und Bewertungen zeigen deutlich, dass das vorgestellte New Rheocasting-Verfahren als neue Semi-solid-Technologie in hervorragendem Masse geeignet ist zur Herstellung komplexer Formteile mit herausragenden mechanischen Eigenschaften. Darüber hinaus werden in der vorliegenden
Arbeit Wege aufgezeigt, die eine zielführende Evaluation neuer, bisher für die Semi-solid-Formgebung noch nicht geeigneter Legierungen, erlauben.

Der Autor hofft, damit die Basis für eine neue, erfolgreiche Formgebungstechnologie geschaffen zu haben. Die grossindustrielle Umsetzung ist heute schon für Aluminiumlegierungen vielversprechend im Gange, eine Ausweitung auf Magnesiumlegierungen und höherschmelzende metallische Legierungen ist abzusehen.
# Table of Contents

1 Introduction .................................................................................................................. 1

2 New Rheocasting - Process .............................................................................................. 5
   2.1 General Overview ...................................................................................................... 5
   2.2 Material Flow and Recycling ..................................................................................... 9
   2.3 Metallurgical Aspects ............................................................................................... 11

3 Solidification Fundamentals ........................................................................................... 17
   3.1 Equiaxed Solidification ............................................................................................ 17
      3.1.1 Nucleation - Origins of the Equiaxed Zone ...................................................... 17
      3.1.2 Morphology of the Solid/Liquid Interface ....................................................... 24
   3.2 Instability of the Solid/Liquid Interface .................................................................... 25
      3.2.1 Interface Instability in Pure Substances ............................................................ 25
      3.2.2 Interface Instability in Alloys ............................................................................ 26
      3.2.3 Perturbations at a Solid/Liquid Interface .......................................................... 31
      3.2.4 Gibbs-Thomson Undercooling .......................................................................... 32
   3.3 Modeling of Globular Structure Evolution ............................................................... 34
      3.3.1 Cellular Automaton Model (MCA) .................................................................... 34
      3.3.2 Growth Kinetics and Orientation ...................................................................... 34
      3.3.3 Solute Redistribution ....................................................................................... 36
      3.3.4 Calculation of the Interface Curvature .............................................................. 37
   3.4 Simulated and Experimental Microstructures ............................................................. 39
      3.4.1 Effects of Pouring Temperature ........................................................................ 39
      3.4.2 Effects of Cooling Rate .................................................................................... 42
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.4 MECHANICAL PROPERTIES</td>
<td>99</td>
</tr>
<tr>
<td>7.4.1 STATIC MECHANICAL PROPERTIES</td>
<td>100</td>
</tr>
<tr>
<td>7.4.2 NON-EQUILIBRIUM SOLIDIFICATION</td>
<td>103</td>
</tr>
<tr>
<td>7.5 MODIFICATION WITH BARIUM</td>
<td>105</td>
</tr>
<tr>
<td>7.5.1 MICROSTRUCTURE</td>
<td>106</td>
</tr>
<tr>
<td>7.5.2 STATIC MECHANICAL PROPERTIES</td>
<td>107</td>
</tr>
<tr>
<td>7.5.3 DYNAMIC MECHANICAL PROPERTIES</td>
<td>108</td>
</tr>
<tr>
<td>8 SECONDARY ALLOY NRC - AlSi9Cu3</td>
<td>111</td>
</tr>
<tr>
<td>8.1 ALLOY CHARACTERISTICS</td>
<td>111</td>
</tr>
<tr>
<td>8.1.1 ADAPTATION OF AlSi9Cu3 FOR THE NRC-PROCESS</td>
<td>114</td>
</tr>
<tr>
<td>8.2 FLOW BEHAVIOUR IN THE SEMI-SOLID STATE</td>
<td>116</td>
</tr>
<tr>
<td>8.3 MICROSTRUCTURE</td>
<td>120</td>
</tr>
<tr>
<td>8.4 MECHANICAL PROPERTIES</td>
<td>121</td>
</tr>
<tr>
<td>8.4.1 STATIC MECHANICAL PROPERTIES</td>
<td>121</td>
</tr>
<tr>
<td>8.4.2 DYNAMIC MECHANICAL PROPERTIES</td>
<td>126</td>
</tr>
<tr>
<td>8.4.3 WEAR PROPERTIES</td>
<td>126</td>
</tr>
<tr>
<td>8.5 CORROSION PROPERTIES</td>
<td>128</td>
</tr>
<tr>
<td>8.5.1 CAUSES AND FORMS OF CORROSION</td>
<td>128</td>
</tr>
<tr>
<td>8.5.2 IMMERSION TESTS</td>
<td>130</td>
</tr>
<tr>
<td>8.5.3 POTENTIODYNAMIC POLARISATION MEASUREMENTS</td>
<td>132</td>
</tr>
<tr>
<td>8.5.4 ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY</td>
<td>135</td>
</tr>
<tr>
<td>9 CONCLUSIONS AND OUTLOOK</td>
<td>139</td>
</tr>
<tr>
<td>10 LITERATURE</td>
<td>143</td>
</tr>
</tbody>
</table>
The discovery of shear rate dependent viscosity for metals in the solidification range in the early 1970s [1] was the starting point for the development of semi-solid processing routes for light metal alloys, also known as „semi-solid metallurgy“ (SSM) or „semi-solid forming“. Over the last decade or more, the primary driving force for the development and enhancement of semi-solid forming processes has been the automotive industry in its search for energy efficient automobiles. Aluminium and magnesium usage in automobiles, especially in cast form, has increased dramatically and as a consequence the need for parts of higher strength and greater reliability has increased. SSM is one of the important processes filling this niche, and in the past 30 years a number of processes, namely Thixocasting, Thixoforging, semi-liquid Casting and Rheocasting have advanced to become manufacturing routes of industrial relevance.

The key feature of all these processes is the specific flow behaviour of semi-solid slurries, which contain a high volume fraction of non-dendritic solid phase. Such a semi-solid slurry allows a laminar, unbroken filling of the die, thanks to the increased viscosity compared with fully liquid melts. Since air entrapment can be avoided, heat treatable and weldable components with low porosity levels are obtained, which are superior in mechanical properties compared to conventional die-casting parts. Additionally, the die filling temperature as well as the heat content of the metal are lower, leading to longer die life and higher productivity.

Semi-solid processes are near-net shape forming technologies and allow the production of near-net shape castings with high integrity, i.e. excellent combination of strength and ductility, which are prerequisites for light weight component design. In addition, compared with conventional casting and forging processes the machining costs are reduced. Thanks to these advantages, semi-solid cast and forged parts have replaced conventional
forgings, permanent mould and investment castings, and in some instances even die castings. Applications include automotive suspension parts, wheels, master brake cylinders, antilock brake valves, pump housings, rocker arms, pulleys, clutch cylinders, knuckles, belt covers, pistons, engine mounts, electrical connector multiconductors, brass plumbing components and many more parts covering the field of automotive, aerospace and mechanical engineering components.

The key to a successful implementation of semi-solid forming techniques lies in the preparation of a semi-solid precursor material with an equiaxed or globular α-Al- or α-Mg-phase in the microstructure. Figure 1 shows the typical microstructure of an AlSi7Mg0.3 alloy (A356) produced by the New Rheocasting process.

![Figure 1: Typical microstructure of AlSi7Mg0.3 produced by the New Rheocasting process with globular α-Al-phase (bright) and eutectic phase (dark).](image)

Obviously, the α-Al-phase exhibits the required globular shape. A more detailed description of this coarse two-phase microstructure may be defined by a set of parameters, which are (i) average grain size, D°, of the α-Al-phase, (ii) shape factor, F°, and (iii) contiguity, C°, of the α-Al-phase (see Chapters 3.4.1 and 4.1.1).

At this point in time, the major SSM route employed on a small industrial scale is the conventional thixocasting process. In this process the globular structure of the precursor ma-
terial is achieved by stirring the melt during solidification at a continuous casting machine with a magnetohydrodynamic stirrer. The cast billets are cut into slugs representing the required portions for casting, placed into an induction heating carousel, heated into the semi-solid region, picked up by a robot, inserted into the shot sleeve of a high pressure die casting machine and then cast into a die. After solidification under pressure, the shot cluster is extracted from the die and parts and runner system are separated. Runner systems and overflows are then generally sent for out-source recycling.

However, as time progressed, a couple of disadvantages of the conventional thixocasting route became more and more apparent. First, it is difficult to obtain fully homogeneous billets in magnetohydrodynamically stirred continuous castings. In particular, they typically have some degree of inhomogeneity with respect to both microstructure and chemical composition. Furthermore, there is metal loss in the re-heating process of the billets which may account to as much as 10 percent of the total part weight. Gates and risers from formed parts cannot be recycled within the forming facility, but must be sent back to the ingot producer. Thus, the metal former pays a premium to the continuous caster, not only for the unique structure in the metal he sells to a customer but also for what he returns to the primary producer.

All of these disadvantages contributed to the fact that thixocasting never managed to grow out of its niche status. With productions of aluminium castings in the order of 1.500.000t in both North America and Europe and of 1.000.000t in Japan each year, thixocasting represents at most 1% of the total market, whilst die casting and permanent mould casting are at present overwhelmingly dominant [2]. The most frequently utilised alloy in thixocasting is AlSi7Mg0.5 (A357) with 11.200 t/y followed by AlSi7Mg0.3 (A356) with around 2.100 t/y [3].

If semi-solid forming wants to break out of its niche player status, it must overcome a very important challenge, namely that of becoming economically competitive in a wide spectrum of applications. This does not only mean meeting existing customer requirements, but rather helping the automotive industry to take advantage of the unique features of this
process, such as broadening the spectrum of semi-solid processable alloys as well as over-
coming most of its former drawbacks by optimising the different steps of existing proces-
ses in order to make them more robust and less expensive. Aside of the weight saving
potential and the achievable quality levels, the manufacturing cost of the resulting com-
ponent plays a decisive role in the success of a new process in the foundry market place.
Next to investment and material cost, productivity, scrap rate, process stability, die life,
heat treatment, machining and quality control are important factors influencing the part
cost in series production.

The search for more inexpensive processing routes for producing parts by semi-solid for-
ming directly from the liquid state (i.e. „Rheocasting“) has lead to a few processes ex-
ploited today, which fall into two general categories. In the first category, liquid metal is
poured into a container of approximately the size of the billet to be fed to the shot chamber
of the forming machine. The desired globular microstructure is then obtained in the billet
by some combination of cooling, grain refinement and/or convection. At the required tem-
perature, the billet is fed to the forming machine. In the second category, the desired slurry
structure is obtained within a bath of considerable size again by some combination of coo-
ling, grain refinement and stirring. Desired quantities of the semi-solid metal are then ex-
ttracted from the bath and formed by one or another process.

At the fair GIFA 99 in Düsseldorf, Germany, by far the most promising patented new way
of semi-solid forming was introduced by UBE Industries Ltd., namely the New Rheoca-
sting (NRC) process [4], which combines Squeeze Casting technology with a novel way
of slurry making.
Based on the crystal separation theory (see chapter 3.1.1) A. Ohno advocated the seed pouring casting method to produce equiaxed crystals [5]. As shown in Figure 2, in this process the molten metal is poured into the mould along a water cooled gutter. Following the idea to contrive the gutter surface in a way that crystals can easily form and separate, a low temperature melt containing the crystals will enter the mould and create a fine microstructure.

Figure 2: Seed pouring casting method [5].
Being at the bottom of the seed pouring cast method UBE Industries Ltd. developed the New Rheocasting (NRC) process, which is an innovative method for preparing the globular precursor material combined with the conventional vertical indirect squeeze casting technology. A fully liquid, but only slightly super-heated melt is taken from a holding furnace by a casting robot and poured into especially designed steel crucibles, which are placed on a carousel next to the NRC machine. By controlling the cooling of the melt into the semi-solid region a globular microstructure is attained. In the final step of the carousel, the temperature is equalised over the cross-section of the crucible. The semi-solid melt is then poured into the inclined sleeve of the squeeze casting machine, the sleeve docks to the die and the slug is cast into shape. The die filling is slow and runs from the bottom upwards, as in low pressure die casting. This allows laminar flow and the air to be pushed out of the die cavity without being trapped within the casting. During solidification, an adjustable over-pressure is applied. After part extraction the runner system is cut off and returned to the melt shop of the foundry for in-house recycling.

A schematic of the process steps involved in the New Rheocasting process and a photograph of the UBE HVSC PL-800 NRC machine installed in the foundry laboratory of LKR in Ranshofen, Austria, are shown in Figure 3 and Figure 4, respectively.

![Figure 3: Schematic of the New Rheocasting process.](image-url)
Table 1 gives an overview of some HVSC Pl-800 NRC machine characteristics. $H$ stands for horizontal die opening, such as found in conventional die casting systems, $V$ stands for vertical casting direction and $SC$ for squeeze casting. $PL$ means pitless and indicates that the installation of this casting machine does not require a pit and can be placed directly on the foundry floors just as in conventional high pressure die casting machines. Vertical squeeze casting technology combines characteristics of low pressure die casting, namely slow and bottom upwards die filling, with elements of high pressure die casting, such as solidification under high pressure. Crucial to the production of high quality parts is a slow and non-turbulent filling of the die with gate speeds around 0.5m/s, which are much lower than for pressure die casting.

The HVSC Pl-800 NRC machine can also be used as a conventional squeeze casting machine by pouring the melt straight into the inclined sleeve without using the cooling ca-
rousel. In semi-solid forming, however, the slurry has a higher viscosity than the fully liquid melt and, therefore, the transition point from laminar to turbulent flow moves to higher filling velocities. Furthermore, around 40 to 60% of the metal is already solidified, which also allows higher filling speeds than for squeeze casting. The maximum piston speed for the HVSC Pl-800 NRC machine is 500 mm/s. The maximum shot weight for aluminium alloys lies around 7.5 kg.

Table 1: HVSC PL-800 NRC machine characteristics.

<table>
<thead>
<tr>
<th>Machine properties</th>
<th>UBE HVSC PL-800 NRC</th>
</tr>
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<tr>
<td>Platen Size [mm]</td>
<td>1400 x 1400</td>
</tr>
<tr>
<td>Die bar distance [mm]</td>
<td>930 x 930</td>
</tr>
<tr>
<td>Clamping force [kN]</td>
<td>8000</td>
</tr>
<tr>
<td>Max. shot force [kN]</td>
<td>1230</td>
</tr>
<tr>
<td>Max. die filling force [kN]</td>
<td>336</td>
</tr>
<tr>
<td>Max. metal pressure [MPa]</td>
<td>171</td>
</tr>
<tr>
<td>Height of machine centre [mm]</td>
<td>2800</td>
</tr>
</tbody>
</table>

2.2 MATERIAL FLOW AND RECYCLING

Figure 5 shows a flow diagram comparing New Rheocasting and Thixocasting processing routes. In conventional Thixocasting the foundry receives continuous cast billets from an out-source supplier. These are then cut to the desired length for the required shot weight. During induction heating in vertical induction coils, the stability of the billet is essential for the final product quality and the overall economics of the process. Metal loss in the range of 8 to 10% during heating has to be taken into account in this heater configuration [2]. After induction heating and forming, the good parts are separated from runners, biscuit, overflows and scrap, which are then returned to the out-source supplier for recycling into a globular precursor material.
In the New Rheocasting process the melt is prepared in the foundry’s melt shop from conventional ingots and then transferred to the holding furnace at the squeeze casting machine. The ladling device pours the melt into the crucibles, which are then placed onto the carousel. When the semi-solid melt is ready, a robot picks up the crucible and allows the billet to slide into the vertical sleeve of the squeeze casting machine. There is no metal loss in this operation. After casting the runners and scrap are returned to the melt shop for in-house recycling.

The New Rheocasting process provides a so far unknown independence from metal suppliers in semi-solid forming and a cost advantage compared with the Thixocasting process. It is predicted that mainly due to the lower material cost an overall cost saving of up to 20% compared to conventional Thixocasting for aluminium alloys can be reached [6]. The cost saving can be further increased via the use of recycled material (secondary alloys) in New Rheocasting. Figure 6 illustrates a part-cost comparison between Thixocasting and New Rheocasting for primary and secondary alloys for one and three shifts.
It is foreseen that magnesium alloys will find a much wider industrial use if cost competitive manufacturing routes for structural, pressure-tight parts with improved ductility become available. The most frequently used high pressure die casting process is mainly suitable for thin walled castings, and the process fails when it comes to thicker walls and pressure tightness, which are required in most structural or hydraulic components. Semi-solid forming was considered to be one potential route toward the achievement of this development target, but the existing processes such as Thixocasting present a drawback in that precursor material is not readily available on the market in large quantities thus adding significant cost to the parts. The New Rheocasting process offers one essential advantage over all semi-solid forming processes especially in terms of magnesium alloys: it does not require any special precursor material. Furthermore, in the LKR laboratory in Ranshofen, Austria, a newly developed recycling unit from Rauch Fertigungstechnik Ltd, Austria, has been installed to provide in-house recycling by remelting magnesium scrap from the New Rheocasting machine by casting it into ingot or billet shape (see Figure 7). The fluxless continuous process uses mixtures of nitrogen and sulphur dioxide as protective gas and remelts up to 500kg of scrap per hour. In this pilot plant low amounts of dross and sludge are produced during the fluxless recycling process.
The continuous feed to the magnesium melting furnace is done with an electric conveyor belt, from which the melt is transported to the holding and dosing furnace. In this processing step the chemical composition of the melt can be adjusted and specific NRC requirements fulfilled. Since the quality of the resulting ingot does not differ significantly from the primary material, it can be directly re-used in the New Rheocasting process. In serial production foundries this transfer could be done in the molten state in order to avoid re-melting costs and to reduce energy consumption [7].

2.3 METALLURGICAL ASPECTS

Figure 8 gives a schematic illustration of the development of the thixotropic semi-solid structure in the New Rheocasting process. The essential core of this process is the numerous nucleation of α-grains which continue to grow in a globular shape due to controlled cooling. As soon as the slightly super-heated melt touches the wall of the steel crucible its temperature immediately drops into the semi-solid range and nucleation of α-particles starts. These nuclei are then distributed homogeneously over the whole volume during the
dosage of the remaining melt. During controlled cooling from the casting temperature, \( T_G \), down to the semi-solid forming temperature, \( T_F \), by the means of blowing air on the crucible, the nuclei grow to globular grains. Depending on the metal volume and on the alloy composition (aluminium or magnesium alloys) it takes between 2 and 5 minutes for this step of the process. For industrial applications, acceptable cycle times for economical processing are obviously much shorter and, therefore, the cooling process must take place on a carousel. The cooling carousel is illustrated in Figure 9.

\[
TG = TL + (10-50)\degree C
\]

\[
T_{SS1} > T_{SS2} > T_{SS3} = T_F
\]

Figure 8: Schematic illustration of the development of the thixotropic semi-solid structure in the NRC process. \( T_L \) is the liquidus temperature, \( T_G \) the temperature in the holding furnace, \( T_{SS1} \) to \( T_{SS3} \) the semi-solid temperatures during controlled cooling and \( T_F \) the semi-solid forming temperature.

Depending on the process cycle time the number of cooling stations can be varied up to 10 for short cycle times. The last station on the cooling carousel is an induction heating station where both the temperature in the semi-solid melt is homogenised over the cross section of the crucible and the contact layer of the slurry with the crucible is re-melted to guarantee that the slug can easily slide out of the crucible into the sleeve of the squeeze casting machine.
A very important aspect are the ceramic insulations on the top and bottom of the crucible whilst positioned on the cooling carousel. Figure 10 shows the temperature control effect on the temperature distribution of an AlSi7Mg0.3 (A356) slurry during cooling with ceramic covers after adjusting six thermocouples at different positions and heights in the slurry. In the absence of temperature control after pouring, the difference in temperatures between the six positions was very big (up to 15°C from the centre to the regions close to the crucible wall) and the slurry was difficult to discharge from the crucible. Via the use
of ceramic insulations, the maximum temperature difference between centre and regions close to the crucible wall was of only 3°C and the slurry was easily dischargable. (Note: crucible size $\phi=80\text{mm}$, $h=245\text{mm}$, wall thickness $t=2\text{mm}$).

Figure 10: Temperature control effect on temperature distribution of an AlSi7Mg0.3 slurry during cooling.
During the last few years, extensive efforts have been made in order to investigate the mechanism of the evolution of a globular structure. Some experimental observations such as fragmentation of large crystals, aggregation, coalescence and abrasion of the fragmented crystals are considered to be responsible for such a globular microstructure formed in processes like mechanical or electromagnetic stirring, intensive ultrasonic treatment during solidification or partial remelting of deformed alloys [8, 9, 10].

However, for the New Rheocasting process - in which one obtains the desired globular microstructure directly from the molten metal by control of casting temperature and cooling rate without any mechanical actions on the alloy slurry - there is a shortage of explanation about the mechanism of the globular structure formation. Moreover, this process has some significant advantages over the previous techniques and, therefore, it is necessary to clarify and understand quantitatively the evolution mechanism of globular structures, since it is closely associated with the control of semi-solid microstructures, of the quality of final semi-solid forming products as well as of the productivity of the process.
3.1 EQUIAXED SOLIDIFICATION

3.1.1 NUCLEATION - ORIGINS OF THE EQUIAXED ZONE

Depending on the casting conditions, a specified volume of molten alloy can solidify to produce any one of three characteristic grain microstructures. These grain macrostructures are generally termed chill, columnar and equiaxed. Figure 11 shows a general schematic picture of the type of structures obtained in ingots, where the three rather distinct zones appear.

In the chill zone the structure consists of many small roughly equiaxed grains. It has been suggested that nucleation begins from very many sites along the mould wall causing this
fine grain structure. However, this structure does not grow inward very far beyond the emergence of the columnar structure. The grains in the columnar zone are relatively large, long and crystallographically oriented, according to their dendrite directions parallel to the heat flow direction. The columnar grains grow into the centre of the ingot along the heat flow directions behind an advancing dendritic interface [11]. Within the liquids at the centre of the ingot there are generally many small equiaxed grains suspended throughout in the equiaxed zone. As freezing continues, these small grains begin to crowd together until finally they effectively block the inward motion of the columnar grains. This point is called the columnar to equiaxed transition.

The nucleation and growth mechanisms involved in the generation of chill and columnar crystals are generally well accepted and understood [12]; however, numerous theories have been developed to explain the origins of equiaxed crystals.

a) Constitutional Supercooling [13]

This theory is based on the idea that equiaxed crystals nucleate in the liquid ahead of a growing solid/liquid interface. Rejection of solute from the growing interface produces a solute diffusion layer in the liquid. This solute concentration gradient corresponds to a change in the liquidus temperature of the liquid. Thus, the liquid ahead of a growing interface can become constitutionally undercooled or supercooled, and the possibility arises of a critical degree of undercooling ($\Delta T_{\text{crit}}$) required for nucleation being exceeded in this liquid ahead of the interface (Figure 12). Heterogeneous nucleation can then occur on any suitable substrate particle in this region, i.e. those particles for which the degree of undercooling is sufficient. Equiaxed crystals nucleated ahead of the interface will be randomly orientated and rapid crystal growth can prevent the advance of the columnar zone.

b) Dendrite Arm Remelting [14]

The dendrite arm remelting theory is based upon observations of growth in casting experiments with Ni-Cu alloys and transparent organic analogues.
From these observations it was proposed that secondary dendrite arms grow with a necked shape, due to solute build up near their base. Diffusion of solute into the bulk liquid is restricted at the base by the solid primary arm, with the liquid in this region being already solute enriched from the growth of this arm. Due to the change in local equilibrium freezing temperature, the accumulated solute restricts growth at the base of the secondary arm, with growth occurring preferentially near the tip, where solute diffusion is relatively unrestricted. Detachment then occurs with fluid flow carrying the crystal fragments into the bulk liquid (Figure 13). It was suggested that the major forces of detached arms in castings are remelting through fluctuations in the growth rate and interaction between two diffusion fluxes: heat and composition.

c) **Showering from the Surface** [16]

This theory proposes that cast ingots contain four zones rather than the commonly acknowledged three (i.e. chill, columnar and equiaxed). The fourth zone is believed to be a coarse dendritic layer at the surface of the ingot exposed to the atmosphere. The coarse
dendritic grains in this region are thought to have nucleated independently from the solid nucleated on the mould walls. Heat transfer through radiation is suggested as the principle mechanism for undercooling at the surface, the resulting difference in heat loss producing a much coarser structure than is observed at the wall. Equiaxed nuclei are formed when dendrites from this surface layer fragment, and sink into the liquid ahead of the growing columnar zone. The mechanism of fragmentation is unclear, but it is suggested that the weight of a growing crystal might be sufficient to overcome surface tension forces holding it to the surface dendritic layer, and furthermore, that crystals might be dislodged through the formation of the shrinkage cavity.

**Crystal Necking and Separation at the Mould Wall** [17]

This suggested mechanism describes the formation of the equiaxed zone in which crystals nucleate on the mould wall immediately after pouring, before the formation of a stable solid skin. The crystals grow off the mould wall in a necked shape and are subsequently detached through mechanical breakage or thermal fluctuations in the liquid. Figure 13 illustrates this theory if the primary arm in the figure is now regarded as the mould wall and the secondary arm as a grain nucleated on the wall.

![Figure 13: Growth and detachment of secondary dendrite arms from primary arm](image-url)
Nucleation occurs on the mould wall since the thermal undercooling is at a maximum at this point. After a crystal has nucleated, growth at first occurs along the wall, since the temperature of the liquid is lowest in this region. The growing crystal rejects solute, which (if not physically restricted) diffuses into the liquid forming a concentration gradient. Due to the change in liquid composition, growth is restricted at the solid/liquid interface. Since there can be no diffusion of solute back towards the mould wall, further growth along the wall is suppressed. Growth occurs preferentially in the regions where diffusion of solute away from the interface is relatively un-restricted. The head of the crystal thus grows much faster than the root, and the crystal begins to take on a granular necked shape. Convection and turbulence play important roles in both crystal detachment and transport of crystals to the centre of the ingot. The mechanical force of thermal convection promotes the separation of crystals from the wall, and temperature fluctuations produced by convection may re-melt the crystal neck. Thermal convection is at its largest immediately after pouring, in the region adjacent to the mould wall when the temperature difference between the mould and the metal is largest. Crystal detachment and transport are therefore more pronounced in this initial period before the formation of a stable solid skin and occur most readily towards the upper part of the mould near the molten surface.

e) Free Chill Crystals [18]

The free chill crystal theory proposes that upon pouring, the equiaxed zone is formed by “free“ chill crystals which nucleate in a thermally undercooled region adjacent to the mould wall, and subsequently swept into the interior of the casting by fluid flow. Crystals nucleated directly on the mould wall, then grow into the melt and reject solute thus forming a small region of constitutionally undercooled liquid. This undercooled region protects some of the crystals previously nucleated, extending further into the liquid for lower pouring temperatures and higher solute contents. The crystals which survive the superheat are carried to the centre of the casting by turbulence and convection. Growth of these crystals continues in an equiaxed fashion until impingement and their presence inhibits further growth of the columnar zone. It is proposed that increased convection and turbulence
would increase the number of nuclei by presenting fresh liquid to the thermally undercooled region as well as distributing existing grains throughout the casting.

All these nucleation mechanisms are possible depending on the alloy composition, casting conditions and types of nucleating substrates present in the melt. It is also probable that more than one mechanism operates in any given casting. For Al-Si alloys it has been shown that two distinct nucleation mechanisms take place [19], namely one or both of the wall mechanisms (free chill crystals and the separation theory) and the constitutional supercooling mechanism. The dendrite arm re-melting and showering from the surface mechanisms are probably favoured in organic analogues where the thermal conditions, such as long range thermal changes, short range compositional changes, and slow cooling rates are more compatible. These mechanisms are less likely in metallic alloy systems as there are relatively more positive thermal gradients at the start of the solidification as well as shorter solidification times. On the other hand, the higher cooling rates in metallic systems favour the survival of crystals nucleated by the wall mechanisms, particularly if low superheat is used. In all systems the constitutional supercooling mechanism is favoured by the presence of effective nucleating substrates in the melt. Grain refinement of aluminium alloys by Ti-B grain refiners is an extreme case of this situation. However, even under these conditions it has been shown that the wall mechanisms can make a significant contribution to the final grain size of the equiaxed zone [19].

With regard to nucleation mechanisms in the New Rheocasting process it is reasonable to conclude that the wall mechanism theories are the most applicable. Both, the free chill crystal theory as well as the separation theory are very similar. However, there seems to be little acknowledgement of the necking and separation theory in the general literature. One of the attractions of the free chill crystal theory is that by introducing the concept of crystal survival, it accounts (at least superficially) for the variation in microstructure observed with pouring temperature. High pouring temperatures diminish the size of the thermally undercooled region as well as extend the time over which the mould absorbs the superheat. Hence, fewer crystals will be produced and a smaller percentage of these will survive. It also accounts for the effect of increased turbulence, which generally results in
a decrease in grain size and an enlarged equiaxed zone \( [20, 21, 22] \). The presence of small equiaxed grains within the columnar zone \( [18] \) and the settling of crystals to the base of the ingot is also consistent with the predictions of this theory. In a later study of equiaxed zone formation \( [23] \) it was proposed that the free chill crystal theory is valid provided that the rate of heat extraction is high and the pouring temperature is low. Furthermore, it was shown that the dissipation of superheat from a casting occurs relatively quickly, promoting the survival of those grains previously nucleated and transported to the centre of the ingot \( [11] \). The possibility of free chill crystals, provided that castings are poured with a small superheat and the columnar zone is absent, was acknowledged in \( [14] \). Moreover, it was observed that in a succinonitrile-ethyl alcohol solution copious nucleation of free chill crystals occurred for pouring temperatures close to the liquidus \( [24] \).

Objections to this theory focus on its dependence on large thermal undercoolings alone for the formation of equiaxed crystals. If rapid chilling at the mould wall is the sole producer of equiaxed nuclei, a systematic increase in the number of equiaxed grains would be expected with increasing undercooling or chilling at the wall. More specifically, the nucleation zone would extend further into the liquid and convective motions would be enhanced by presenting fresh liquid to the undercooled zone and transporting previously nucleated crystals to the centre of the casting. In \( [25] \) a fine grained structure was observed in Ni-Cu alloys for high and low undercoolings, whereas intermediate undercoolings produced coarse grains. Other evidence to support these observations can be found in \( [5] \) where experiments in order to test the effects of rapid chilling on the formation of the equiaxed zone were performed. An Al-0.1%Ti alloy was poured into two stainless steel crucibles; one cooled by air, the other cooled by ice water. The crucible cooled by air showed a fine equiaxed structure throughout, while the crucible cooled by ice water showed a central equiaxed zone surrounded by an outer columnar zone. Hence, it was concluded that in this alloy, rapid chilling alone does not promote the formation of the equiaxed zone. These observations could not be readily explained using the free chill crystal theory.
3.1.2 Morphology Of The Solid/Liquid Interface

Figure 14 illustrates the temperature fields of the various cast structures. These are planar interface (columnar grains - a) or thermal dendrites (equiaxed grains - b) in pure materials, and solutal (constitutional) dendrites in alloys (c, d).

In the case of a pure metal, columnar grains (a) possess an essentially planar interface and grow out from the mould in a direction opposite to that of the heat flow. Equiaxed grains (b) grow in a supercooled melt which acts as their heat sink and, therefore, the growth direction and the heat flow direction are the same. When alloying elements or impurities are present, the morphology of the columnar crystals (c) is generally dendritic. The equiaxed morphology in alloys (d) is almost indistinguishable from that in pure metals. However, the growth in pure metals is controlled by the heat flow, whereas the growth in alloys is mainly controlled by solute diffusion. In columnar growth the hottest part of the system is the melt, while in equiaxed solidification the crystals are the hottest part and, therefore, the melt must always be cooled below the melting point (i.e. undercooled) before equiaxed crystals are able to grow.
3.2 Instability of the Solid/Liquid Interface

3.2.1 Interface Instability in Pure Substances

Figure 15 schematically shows the development of a perturbation during columnar (a) and equiaxed (b) growth.

During columnar growth (a) of a pure substance, the temperature given by the heat flow, \( T_\text{q} \), increases with distance ahead of the interface and, \( G \), the interface temperature gradient, is positive. Therefore, the heat flow direction is opposite to that of solidification. When a perturbation of amplitude, \( \varepsilon \), forms at an initially smooth interface, the temperature gradient in the liquid increases while the gradient in the solid decreases. Furthermore, since the heat flow is proportional to the gradient, more heat flows into the tip of the perturbation and less flows out of it into the solid. As a result, the perturbation re-melts and the planar interface is stabilised.
In equiaxed solidification (b), however, the free crystals grow into an undercooled melt and the latent heat produced during growth flows along the negative temperature gradient in the liquid. A perturbation formed on the sphere will exhibit a steeper gradient and permit the tip to reject more heat. Therefore, the local growth rate is increased and the interface is always morphologically unstable.

### 3.2.2 Interface Instability In Alloys

In alloys the criterion for stable or unstable behaviour is more complicated because the local equilibrium melting point can vary along the solid/liquid interface. During solidification of an alloy there is a substantial change in the concentration ahead of the interface. This change will affect the local equilibrium solidification temperature, \( T_1 \), of the liquid, which is related to the composition by:

\[
T_1(C_0) - T_1 = m_1(C_0 - C_1)
\]  

(3-1)

where \( T_1(C_0) \) is the liquidus temperature corresponding to the initial alloy concentration, \( C_0 \), and, \( m_1 \), the liquidus slope. This relationship is shown in Figure 16. As the liquid concentration, \( C_1 \), decreases with distance, \( z \), the liquidus temperature, \( T_1 \), of the alloy will increase as indicated by the phase diagram. This means that if small volumes of liquid at various distances ahead of the solid/liquid interface were extracted by some means and solidified, their equilibrium freezing points would vary with position in the way described by the heavy curve in the lower left-hand diagram in Figure 16.

Depending upon the temperature gradient,

\[
G = (dT_q/dz)_{z=0}
\]

(3-2)

in the liquid at the solid/liquid interface there may or may not exist a zone of constitutional undercooling. This zone is defined to be that volume of melt ahead of the interface within which the actual temperature, \( T_q \), is lower than the local equilibrium solidification temperature, \( T_1 \). The melt in this zone is undercooled, i.e. in a metastable state.
The condition required for the existence of such a constitutionally undercooled zone is that the temperature gradient, $G$, at the interface in the liquid should be lower than the gradient of liquidus temperature change in the melt. Therefore, the interface is constitutionally undercooled, when:

$$G < m_l G_C$$  \hspace{1cm} (3-3)$$

where $G_C$ is the concentration gradient.

Figure 17 demonstrates the conditions for constitutional undercooling at the solid/liquid interface and the resulting structures. When the temperature gradient due to the heat flow is greater than the liquidus temperature gradient at the solid/liquid interface, the latter is stable (a). However, when the slope of the local melting point curve at the interface is greater than the slope of the actual temperature distribution, the planar interface is unsta-
ble (b), because the undercooling encountered by the tip of a perturbation advancing into the melt increases.

![Figure 17: Condition for constitutional undercooling at the solid/liquid interface [26].](image)

Furtermore, since the concentration gradient, \( G_C \), at the interface in the steady state is known as

\[
G_C = (dC_1/dz)_{z=0} = - (V/D) \Delta C_0
\]

(3-4)

where, \( V \), is the rate of interface movement and, \( D \), the diffusion coefficient in liquid.

Using Equation 3-3, the existence of constitutional undercooling can be written as:

\[
G < - m_1 V \Delta C_0 / D
\]

(3-5)
Also because

\[- m_1 \Delta C_0 = (T_1 - T_s) = \Delta T_0 \]  \hspace{1cm} (3-6)

the limit of constitutional undercooling can be expressed in its usual form:

\[ G/V = \Delta T_0 / D \]  \hspace{1cm} (3-7)

or

\[ V_C = GD / \Delta T_0 \]  \hspace{1cm} (3-8)

where instability will result, if \( G/V \) is smaller than \( \Delta T_0 / D \). \( \Delta T_0 \) can be replaced by the expression \(- m_1 \Delta C_0 \) or further by \(- m_1 C_0 (k-1)/k \) where the distribution coefficient, \( k \) (\( k = C_s / C_l \)), describes the difference in composition at the growing solid/liquid interface, assuming that local equilibrium exists in metals.

Figure 18 gives a schematic overview of the stability of the solid/liquid interface simulated at different rates of interface movement, \( V \). The parameters chosen in this simulation are for an Al-7\%Si alloy the temperature gradient, \( G \), of \( 10^{-3} \text{K/\mu m} \), a diffusion coefficient in liquid, \( D \), of \( 5000 \text{\mu m}^2/\text{s} \) and further an initial alloy concentration, \( C_0 \), of 1.37\%. The values for, \( k \), and, \( m_1 \), are 0.132 and -6.64K/\%, respectively.

At \( V=0.05 \mu\text{m/s} \) (a) the condition \( V<V_C \) with \( V_C=0.08 \mu\text{m/s} \) is fulfilled and the interface exhibits a planar morphology. At higher \( V \) values of \( 1 \mu\text{m/s} \) when \( V>V_C \) (b) and \( 2.8 \mu\text{m/s} \) when \( V>>V_C \) (c) the interface becomes unstable and depending on the amount of constitutional undercooling, dendritic growth is more or less pronounced with possible formation of side branches leading to a tree-like morphology.
Figure 18: Solid/liquid interface morphology as a function of the rate of interface movement $V$ [27].
3.2.3 Perturbations At A Solid/Liquid Interface

A drawback of the constitutional undercooling criterion is that it ignores the effect of the surface tension of the interface, which should have a marked influence upon interface stability by tending to inhibit the formation of perturbations. The constitutional undercooling theory does not give any indication of the scale of the perturbations which will develop if an interface becomes unstable. Figure 19 illustrates a perturbed solid/liquid interface schematically. Experimental observations show that the initial form of the morphology is approximately sinusoidal, and therefore, perturbation analysis permits the calculation of the wavelength of the instabilities which develop [26].

The perturbed interface can be described by a simple line function:

\[ z = \varepsilon \sin[\omega y] \]  

(3-9)

where, \( \varepsilon \) is the amplitude and, \( \omega (=\frac{2\pi}{\lambda}) \), is the wave number. \( \Delta T_d \) is the difference between the melting point, \( T_f \), and the temperature of the depression of the interface, \( T_d \). \( \Delta T_t \) is the difference between, \( T_f \), and the temperature of the tip of the interface, \( T_t \).

Figure 19: Interface perturbations at a solid/liquid interface [26].
Figure 20 illustrates the rate of development of a perturbation at a constitutionally undercooled interface. The parameter, $\dot{e}/e$, describes the relative rate of development of the amplitude of a small sinusoidal perturbation. At very short wavelengths, the value of this parameter is negative due to curvature damping and the perturbation will tend to disappear. At wavelengths of $\lambda_i$ and above, the sinusoidal shape will become more accentuated. The wavelength having the highest rate of development is likely to become dominant. The reason for the tendency to stability at high $\lambda$-values is the difficulty of diffusional mass transfer over large distances. When the interface is completely stable, the curve will remain below the $\dot{e}/e=0$ line for all wavelengths. This implies disappearance of perturbations having any of these wavelengths.

![Graph showing development rate of a perturbation at a constitutionally undercooled interface](image)

Figure 20: Development rate of a perturbation at a constitutionally undercooled interface [26].

3.2.4 Gibbs-Thomson Undercooling

With any solid/liquid interface of area, $A$, an excess interface energy required for its creation is associated. Therefore, heterogeneous systems or parts of systems which possess a high $A/v$ ratio will be in a state of higher energy and unstable with respect to a system of lower $A/v$ ratio with $v$, as the volume of the body. The relative stability can be expressed by the equilibrium temperature between both phases (melting point).
The change in melting point due to this curvature effect, often called the curvature or Gibbs-Thomson undercooling, is given by:

$$\Delta T_r = \Gamma K$$

(3-10)

The curvature, $K$, and the Gibbs-Thomson coefficient, $\Gamma$, are here defined so that a positive undercooling or decrease in equilibrium melting point is associated with a portion of solid/liquid interface which is convex towards the liquid phase. The curvature can then be expressed as:

$$K = \frac{dA}{dv} = \frac{1}{r_1} + \frac{1}{r_2}$$

(3-11)

where $r_1$ and $r_2$ are the principal radii of curvature which are the minimum and maximum values for a given surface. It can be shown that they are always perpendicular to each other [26]. However, the total curvature of a sphere is $2/r$ and that of a cylindrical surface is $1/r$. The Gibbs-Thomson coefficient is given by:

$$\Gamma = \frac{\sigma}{\Delta s_f}$$

(3-12)

where, $\Delta s_f$, is defined as the entropy of fusion per volume. For most metals, $\Gamma$ is of the order of $10^{-7}$Km. Hence, the effect of the solid/liquid interface energy, $\sigma$, only becomes important for morphologies which have a radius less than about 10$\mu$m. These include interface perturbations, dendrites, eutectic phases, and - which is very important for the development of the globular microstructure in the New Rheocasting process - especially nuclei and the growth of nuclei.

For instance, assuming the radius of a growing nucleus is about 1$\mu$m, a perturbation with the radius of $<1/10\mu$m would result in a Gibbs-Thomson undercooling, $\Delta T_r > 1^\circ$C, which could be sufficient to make the perturbation disappear.

With respect to constitutional undercooling only, the crystal is supposed to exhibit dendritic growth, in additional consideration of the Gibbs-Thomson undercooling, however, comprehension for the globular growth of the $\alpha$-grains in the New Rheocasting process is obtained.
3.3 MODELING OF GLOBULAR STRUCTURE EVOLUTION

In [28] the morphology evolution of the primary \( \alpha \)-Al phase, i.e. dendritic, intermediate rosette-like and globular, at the solidification of an Al-7\%Si alloy using a modified cellular automaton (MCA) model was studied. The casting experiment was similar to the New Rheocasting process.

3.3.1 CELLULAR AUTOMATON MODEL (MCA)

The cellular automaton model for nucleation consists of the geometry of the cell, the state of the cell, the neighbourhood configuration, and several transition rules determining the state of a given cell during one time step. In [28] the computational domain is divided into a uniform orthogonal arrangement of cells and the selected neighbourhood configuration contains the first eight neighbours (Neumann model). Each cell is characterised by several variables such as temperature, concentration, crystallographic orientation, and solid fraction as well as state (e.g. solid or liquid). The cellular automaton evolves in discrete time step, and the state of a cell at a particular time is calculated from the local rule.

3.3.2 GROWTH KINETICS AND ORIENTATION

In this simulation [28] the growth velocity of a dendrite tip at a certain undercooling in the melt is calculated with the aid of a model in [29]. The total undercooling in the solid/liquid interface, \( \Delta T \), is considered to be the sum of four contributions [30]:

\[
\Delta T = \Delta T_c + \Delta T_t + \Delta T_r + \Delta T_k
\]  

(3-13)

where \( \Delta T_c, \Delta T_t, \Delta T_r, \Delta T_k \) are the undercooling contributions associated with concentration, temperature, curvature and attachment kinetics, respectively. The last term, \( \Delta T_k \), the kinetic undercooling, becomes significant only at rapid solidification and is negligible under normal solidification conditions [30].
Therefore the local undercooling of the solid/liquid interface at a given time $t$, $\Delta T(t)$, is given by:

$$\Delta T(t) = T_j + [C(t) - C_0]m - \Delta T(t) - \Gamma \bar{K}(t) \quad (3-14)$$

where $\bar{K}(t)$, $C(t)$ and $T(t)$ are the mean curvature, the concentration and the temperature of the solid/liquid interface at time $t$.

According to [30] and an iteration method, the relationship between the growth velocity, $v\{\Delta T(t)\}$, and the local undercooling, $\Delta T(t)$, can be expressed as:

$$v\{\Delta T(t)\} = k_1 \Delta T(t)^2 + k_2 \Delta T(t)^3 \quad (3-15)$$

where $k_1$ and $k_2$ are the coefficients: $k_1 = 2.9 \times 10^{-6} \text{ m/sK}^2$ and $k_2 = 1.49 \times 10^{-6} \text{ m/sK}^3$, for an Al-7%Si alloy, respectively (see Figure 21).

![Figure 21: Growth kinetics of a dendrite tip, as calculated with the KGT model, for Al-7Si% and Al-5%Si [30].](image)

Once a cell has nucleated, it then grows with a preferential direction corresponding to its crystallographic orientation. The growth velocity is determined by the local undercooling, $\Delta T(t)$, and the growth length, $l(t)$, is therefore given by:
where $\Delta t$ is the time step, $\theta$ is the angle of the preferential growth direction of a solid cell with respect to the linking line between this solid cell and its liquid neighbour cell. $N$ indicates the iteration number. Furthermore, the solid fraction of a given cell at a certain time, $f^s(t)$, can be expressed by:

$$f^s(t) = \frac{l(t)}{L}$$

where, $L$, is the spacing of the cellular automaton network. When $f^s(t) = 1$, which means the growth front of the solid cell touches the centre of its neighbouring liquid cell, the neighbouring cell transforms its state from liquid to solid and gets the same orientation index as this solid cell.

3.3.3 SOLUTE REDISTRIBUTION

It is assumed that the solid and liquid compositions at the solid/liquid interface are in equilibrium. When the solidification at the solid/liquid interface occurs, the solute partition between solid and liquid is:

$$C_s^* = kC_l^*$$

where, $k$, is the partition coefficient, $C_s^*$ and $C_l^*$ are the interface equilibrium concentration in solid and liquid phases, respectively.

When a cell transforms its state from liquid to solid by nucleation or growth, its concentration will change and it will liberate the amount of solute, $dC=C_l^*-kC_l^*$, which is assumed to be distributed to its liquid neighbouring cells. As the solidification proceeds, the
solidified cells reject solute to their neighbouring liquid cells. The governing equation for the solute redistribution in the liquid region is given by:

$$\frac{\partial C_l}{\partial t} = \frac{\partial}{\partial x} \left[ D_l \frac{\partial C_l}{\partial x} \right] + \frac{\partial}{\partial y} \left[ D_l \frac{\partial C_l}{\partial y} \right] + C_l (1 - k) \frac{\partial f^s}{\partial t}$$  \hspace{1cm} (3-19)

where $t$ is time, $D_l$ is the solute diffusion coefficient in liquid, $f^s$ is the solid fraction, and $k$ is the partition coefficient. $C_l (1 - k) \frac{\partial f^s}{\partial t}$ indicates the amount of solute rejected at the solid/liquid interface.

The governing equation for diffusion in the solid is given by:

$$\frac{\partial C_s}{\partial t} = \frac{\partial}{\partial x} \left[ D_s \frac{\partial C_s}{\partial x} \right] + \frac{\partial}{\partial y} \left[ D_s \frac{\partial C_s}{\partial y} \right]$$ \hspace{1cm} (3-20)

where, $D_s$, is the solute diffusion coefficient in solid.

### 3.3.4 Calculation of the Interface Curvature

The interface curvature in a cell, $A$, with the solid fraction, $f^s$, can be calculated from the following expression [31]:

$$\overline{K}(A) = \frac{1}{dx} \left[ 1 - 2 \left( \frac{f^s(A) + \sum_{i=1}^{n} f^s(i)}{n + 1} \right) \right]$$ \hspace{1cm} (3-21)

where, $dx$, is the cell size, and, $n$, is the number of neighbouring cells which includes the first layer surrounding neighbour cells, $n=8$. The values for the curvature vary from $1/dx$ to 0 for convex surfaces, and from 0 to $-1/dx$ for concave surfaces.

Considering all the possible cases for the interface curvature resulting from Equation 3-21, Figure 22a-b shows the example of a solidified cell, $A$, which lies at the solid/liquid
interface. There must exist at least one liquid cell within its eight neighbours including the four nearest neighbours and the four corner neighbours.

When there is only one neighbouring solid cell around A, Equation 3-21 becomes:

\[ K = \frac{1}{9dx} \left[ 1 - 2 \left( \frac{1 + \frac{1}{9}}{9} \right) \right] = \frac{5}{9dx} \]

where \( \frac{5}{9dx} > 0 \) and therefore indicating a convex surface of the curvature as well as for two or three neighbouring solid cells with the results \( \frac{3}{9dx} \) and \( \frac{1}{9dx} \), respectively.

![Figure 22a: Schematic illustration of the cases leading to a convex curvature surface according to Equation 3-21.](image)

When there are four neighbouring solid cells around A, Equation 3-21 becomes:

\[ K = \frac{1}{9dx} \left[ 1 - 2 \left( \frac{1 + \frac{4}{9}}{9} \right) \right] = \frac{-1}{9dx} \]

with \( \frac{-1}{9dx} < 0 \) and therefore indicating a concave surface of the curvature. For five, six and seven surrounding solid cells the corresponding results are \( \frac{-3}{9dx} \), \( \frac{-5}{9dx} \) and \( \frac{-7}{9dx} \), respectively.
3.4 SIMULATED AND EXPERIMENTAL MICROSTRUCTURES

3.4.1 EFFECTS OF POURING TEMPERATURE

Figure 23 shows the predicted and experimental microstructures of an Al-7%Si alloy with a cooling rate of 0.3°C/s for various casting temperatures [28]: (a) 620°C, (b) 630°C, (c) 640°C, and (d) 650°C.
The white phase indicates the primary $\alpha$-Al phase and the dark matrix is the eutectic phase. At low casting temperatures of 620°C, the primary $\alpha$-Al phase exhibits the globular morphology as shown in (a). However, with the increase in casting temperature, the primary $\alpha$-Al phase transforms gradually to an intermediate rosette-like morphology (b-c) and finally to a dendritic structure (d). These phenomena are considered to be due to different nucleation conditions under various casting temperatures. Furthermore, it can be stated that at low casting temperatures, close to the liquidus, the nucleation occurs rapidly since the liquid is still in motion during pouring. Therefore, the remelting of necked shape crystals and the fracturing of crystals can all contribute to the formation of new crystals, which promotes the formation of abundant crystal seeds in the melt. At this point, crystals can grow simultaneously throughout the liquid region because the temperature field in the liquid is uniformly undercooled. The distribution of crystals and the spacing among them is uniform due to a large number of nuclei that are created simultaneously, leading to the suppression of dendritic growth in the melt. In contrast, if the number of nuclei is relatively low as it is the case at higher casting temperatures, the distance between the nuclei enlarges. Consequently, the solute concentration inbetween the nuclei is lower, resulting in an increase of $\Delta T_c$ and, therefore, leading to a more pronounced dendritic growth.

Figure 24 illustrates typical New Rheocasting microstructures of an AlSi7Mg0.3 alloy depending on the casting temperature employed (between 630°C and 700°C). The results correspond with observations published in [28]. However, comparing the microstructures obtained at a casting temperature of 650°C (Figure 23d) a significant difference is noted in the New Rheocasting process. In fact, when pouring the melt along the wall of the crucible the wall mechanisms for nucleation produce enough nuclei to impede dendritic growth and to exhibit a globular microstructure even at higher casting temperatures (more than 30°C above the liquidus temperature for AlSi7Mg0.3). In [28] the melt was not poured along the wall but directly into the bottom at the centre of the crucible leading to a dendritic microstructure by mostly suppressing the wall mechanisms for nucleation for the same casting temperature. Therefore, it can be stated that the essence of the "New Rheocasting recipe" is the casting of a relatively "cold" melt along the wall of the crucible, in a similar fashion to pouring "Weissbier" into a glass.
Figure 24: NRC microstructures of AlSi7Mg0.3 as a function of the casting temperature.

All micrographs in Figure 24 represent the morphological state at a semi-solid temperature of 580°C. After filling of the crucible, the latter was cooled with air and water-quenched at the moment the semi-solid metal reached 580°C. To quantify the NRC microstructure of AlSi7Mg0.3 as a function of the casting temperature, $T_G$, the microstructural parameters $D^e$ and $F^e$ of the $\alpha$-Al phase are listed in Table 2. The parameters clearly indicate the effects of increasing casting temperatures on the shape of the grains, from globular, to rosette-like and further to dendritic.

The shape factor, $F^\alpha$, is given by:

$$F^\alpha=(S^{\alpha d})^2/4\pi A^\alpha$$  \hspace{1cm} (3-22)
with, \(A^\alpha\), as average area of \(\alpha\)-Al-grains and, \(S^\text{al}\), as \(\alpha\)-liquid-phase boundary. Normally \(F^\alpha\) is \(>1\), however in case of fully spherical grains \(F^\alpha\) tends to \(1\). For dendritic microstructures, the shape factor \(F^\alpha\) is \(>3\) and a thixotropic state cannot be achieved, while with \(F^\alpha<2\), which is typical for SSM precursor material, the shape requirements for thixotropic behaviour are met [32]. In general, the closer the shape factor \(F^\alpha\) approaches \(1\), the more the rheological behaviour improves in the semi-solid state.

The average grain size \(D^\alpha\) of the \(\alpha\)-Al-phase, however, should not exceed \(150\mu\text{m}\) depending on the specific alloy.

| Table 2: NRC microstructure as a function of the casting temperature. |
|-----------------|--------------|--------------|--------------|--------------|
|                 | 630°C        | 650°C        | 675°C        | 700°C        |
| \(D^\alpha[\mu\text{m}]\) | 71           | 92           | 105          | 119          |
| \(F^\alpha[-]\)    | 1.32         | 1.28         | 1.59         | 2.18         |

3.4.2 Effects of Cooling Rate

The evolution of the primary \(\alpha\)-Al phase during solidification is not only closely related to the casting temperature but also to the cooling rate. Figure 25 illustrates typical New Rheocasting microstructures of an AlSi7Mg0.3 alloy cast at 630°C and solidified with two different cooling rates: (a) 0.6°C/s and (b) 4°C/s.

Figure 25: NRC microstructures of AlSi7Mg0.3 as a function of cooling rate.
At the lower cooling rate, the solute distribution in the liquid in the vicinity of the solid/liquid interface becomes more uniform compared to the higher cooling rate. A lower $\Delta T_c$ value, however, enforces the effect of curvature undercooling and gives rise to the interface stability. The curvature effect enhances the spherical growth trend of crystals and it is evident that lower cooling rates promote microstructures exhibiting a globular $\alpha$-Al phase.

Figure 26 shows the predicted and experimental microstructures of an Al-7%Si alloy with a casting temperature of 620°C for various cooling rates [28]: (a) 0.3°C/s, (b) 3°C/s, (c) 10°C/s, and (d) 30°C/s. It is obvious that with higher cooling rates the morphology of the primary $\alpha$-Al phase exhibits a gradual change from globular to rosette-like and finally to dendritic structure.

Figure 26: Simulated and experimental microstructures of an Al-7%Si alloy for different cooling rates [28].
4.1 SEMI-SOLID MICROSTRUCTURE

A successful implementation of semi-solid techniques requires a semi-solid precursor material with a globular microstructure. A closer description and quantification of such a microstructure can be given by a set of parameters, namely average grain size, $D_\alpha$, the shape factor, $F_\alpha$ (see Chapter 3.4.1), contiguity, $C_\alpha$, or contiguity volume, $V_{c_\alpha}$, of the solid $\alpha$-phase.

4.1.1 CONTIGUITY AND CONTIGUITY VOLUME

The conventional semi-solid forming processes, such as Thixocasting or Thixoforging, are based on the fact that in the un-perturbed state the semi-solid slugs can be handled like solid rods, but attain fluid-like properties during shearing. It is generally accepted that the solid-like characteristic is due to the presence of a solid skeleton consisting of interconnected grains. During the forming operation the bonds between grains break under shear stress, resulting in a significant decrease of viscosity [1, 33].

A useful microstructural parameter describing the magnitude of the solid skeleton is the contiguity of the solid phase, $C_\alpha$. The contiguity describes the interconnection of the $\alpha$-Al-phase and therefore, is a measure for the amount of solid-solid contact in a semi-solid structure and is defined as the average fraction of the surface area shared by one grain with all neighbouring grains of the same phase.

$$C_\alpha = \frac{2S_{\alpha\alpha}}{2S_{\alpha\alpha} + S_{\alpha}}$$  \hspace{1cm} (4-1)
with, $S^{\alpha \alpha}$, being the $\alpha \alpha$-grain boundary and, $S^{\alpha l}$, being the boundary between $\alpha$-Al and the liquid phase. Usually $C^\alpha$ is $<1$, unless in the case of a fully connected solid phase where $C^\alpha$ tends to 1, and in the case of fully isolated $\alpha$-Al-grains where $C^\alpha$ is 0. However, the rheological behaviour for structures with contiguities, $C^\alpha$, of 0 and 1 do not show thixotropic properties.

The contiguity, i.e. the „grade of skeletonisation“, is a parameter, which quantitatively helps to describe the distribution of phases in coarse two-phase alloys. For alloys in the semi-solid state, it is appropriate to standardise the contiguity by multiplying it by the fraction of solid, $f^\alpha$, to yield the contiguity volume, $V_C^\alpha$, which quantifies the connecting portion of the solid phase. In [34] the continuous volume fraction is calculated in terms of the contiguity and the volume fraction of the solid particles. The representative structure model is shown in Figure 27.

In Figure 27, the volume $V_{iC}^\alpha$ of the element, i, is given by:

$$V_{iC}^\alpha = a_i^{\alpha \alpha_i} \cos \theta_i$$  \hspace{1cm} (4-2)
where $a_i^{\alpha\alpha}$ is the area of particle-particle contact and $l_i$ is the length of the volume element and $\theta_i$ the angle between the normal to the area and the direction of the element.

The volume of the interconnected $\alpha$-phase volume, $V_{VC}^{\alpha}$, per unit volume is the sum of the volumes of all elements $i$.

$$V_{VC}^{\alpha} = \sum_{i=1}^{n} a_i^{\alpha\alpha}l_i\cos\theta_i$$  \hspace{1cm} (4-3)

$$= \frac{1}{2}l\cos\theta\sum_{i=1}^{n} 2a_i^{\alpha\alpha}$$

with $\overline{l}$ and $\overline{\cos\theta}$ being the average values of $l_i$ and $\cos\theta_i$.

By substituting the linear mean length of the $\alpha$-phase particles, $\overline{L}^{\alpha}$, for $\overline{l}$, and by using the relation $\overline{\cos\theta}=1/2$, one obtains:

$$V_{VC}^{\alpha} = \frac{\overline{L}^{\alpha}}{4}\sum_{i=1}^{n} 2a_i^{\alpha\alpha}$$  \hspace{1cm} (4-4)

$$= \frac{S_V^{\alpha}\overline{L}^{\alpha}}{4}\sum_{i=1}^{n} 2a_i^{\alpha\alpha}$$

where $S_V^{\alpha}$ is the total surface area of the $\alpha$-phase particles per unit volume of alloy.

By using the Tomkeieff-equation

$$\overline{L}^{\alpha} = \frac{4V_{VC}^{\alpha}}{S_V^{\alpha}}$$  \hspace{1cm} (4-5)

and

$$C^{\alpha} = \frac{2a_i^{\alpha\alpha}}{S_V^{\alpha}}$$  \hspace{1cm} (4-6)
the final result is

\[ V_{VC}^\alpha = V_{VC}^\alpha C^\alpha \]  

(4-7)

Thus, the fraction of continuous volume is equal to the volume fraction of the \( \alpha \)-phase multiplied by the contiguity.

4.1.2 COMPARISON OF SEMI-SOLID PRECURSOR MATERIALS

As mentioned in Chapter 1 the conventional Thixocasting (CTC) process requires re-heating of the precursor material into the semi-solid state. During this procedure, holding times at the semi-solid temperature lead to changes in the microstructure and the \( \alpha \)-Al-phase gets more globular and coarser and more connected. The same effect is observed at the NRC-process while maintaining the material at the semi-solid temperature.

Figure 28 represents the microstructure of an AlSi7Mg0.3 alloy after a holding time of 5 minutes at 580°C (\( t^\alpha = 0.5 \)) for both the NRC- and the CTC-slug. At a position in the centre \((x/r = 0)\) as well as close to the surface \((x/r = 0.9)\) of the slug, the NRC material exhibits a more globular microstructure than the CTC material, which is shown in a more detailed comparison in Table 3. For the CTC-material there is an apparent change in the Si-content from lower values in the centre to higher concentrations in regions close to the surface of the slug (Figure 29). This means that at a specific semi-solid temperature due to the increased amount of Si-rich eutectic close to the surface the solid fraction, \( f^\alpha \), is lower than in the centre of the slug, i.e. the CTC microstructure is less homogeneous than the NRC structure.
Table 3: Comparison of the NRC- and CTC-microstructural parameters in the centre \((x/r = 0)\) and close to the surface \((x/r = 0.9)\) of the slug.

<table>
<thead>
<tr>
<th></th>
<th>(F^\alpha) [-]</th>
<th>(D^\alpha) [(\mu)m]</th>
<th>(C^\alpha) [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRC - centre</td>
<td>1.39</td>
<td>88</td>
<td>0.38</td>
</tr>
<tr>
<td>NRC - surface</td>
<td>1.41</td>
<td>86.5</td>
<td>0.39</td>
</tr>
<tr>
<td>CTC - centre</td>
<td>1.91</td>
<td>69.3</td>
<td>0.32</td>
</tr>
<tr>
<td>CTC - surface</td>
<td>1.98</td>
<td>78</td>
<td>0.39</td>
</tr>
</tbody>
</table>

Figure 28: NRC- and CTC-microstructures in the centre and close to the surface of the slug.
The change in the $\alpha$-Al-phase morphology during a holding period at the semi-solid temperature is documented in Figure 30 and quantitatively illustrated in Figure 31 and Figure 32, where the data for the CTC-material reflects the microstructure in the centre of the slug.

As demonstrated in Figure 30, the $\alpha$-Al-phase coarsens and exhibits a more globular shape with longer isothermal holding times. Furthermore, according to the Ostwald-mechanism a reduction of the number of grains per unit area is accompanied by an increase of the average $\alpha$-Al grain size.
Figure 31 shows the development of the shape factor, $F_a$, with increasing holding time in the semi-solid state at 580°C where the advantage of the NRC-structure is evident. As explained in Chapter 3.4, already during solidification on the cooling carousel of the New Rheocasting machine and after short holding periods respectively, the microstructure exhibits an almost globular shape of the $\alpha$-Al-phase, with some improvement at longer holding times. The stirred continuous cast CTC precursor material, however, shows a significant spheroidisation with increasing holding time though it is less globular even after a long holding period at the semi-solid temperature.

![Diagram](image)

**Figure 31: Shape factor, $F_a$, as a function of holding time in the semi-solid state.**

The coarsening of the $\alpha$-Al-phase and its increase in contiguity, $C^\alpha$, during isothermal holding in the semi-solid state is illustrated in Figure 32.

It is important to note that the investigations into the development of the microstructure illustrated in Figures 31 and 32 are only of "academic" relevance to the New Rheocasting process, due to the fact that the slugs cool continuously on the carousel and, therefore, cannot be held in the semi-solid state for 30min.
Figure 32: Grain size, $D^\alpha$, and contiguity, $C^\alpha$, as a function of holding time in the semi-solid state.

For both materials the coarsening can be described according to Ostwald's law for a diffusion control:

$$d^3 - d_0^3 = k \cdot t$$  \hspace{1cm} (4-8)

where $t$ is the time, $k$ is the rate constant, $d$ and $d_0$ are the current and initial grain size, respectively. [35] pointed out that larger particles grow at the expense of smaller ones so as to reduce the surface energy of the system.

The development of the grain size for the NRC- and CTC-materials, however, differ in their coarsening kinetics. With a rate constant, $k$, of 166 $\mu m^3/s$ the NRC microstructure exhibits a much slower coarsening kinetics during isothermal holding than the CTC microstructure with a value of 430 $\mu m^3/s$ for $k$.

In the case of purely diffusion-driven Ostwald ripening, $k$ has the form:
where $\Gamma$ represents the Gibbs-Thomson coefficient, $D$ the liquid interdiffusion coefficient, and $m$ the slope of the liquidus curve. $C_s$ and $C_l$ are the equilibrium compositions of the solid and liquid interface at the coarsening temperature and $g(\Theta)$ is a function describing the solid and liquid interface.

For an AlSi7Mg0.3 alloy at 580°C with $g(\Theta) = 3.17$, $\Gamma = 2 \times 10^{-7}$ mK, $D = 3 \times 10^{-9}$ m$^2$/s, $m = 6.8$ K/at-% Si, $C_s = 1.3$ at-% Si and $C_l = 10.8$ at-% Si, a calculated rate constant, $k$, of 210 $\mu$m$^3$/s [36] is obtained. This value is higher than the value experimentally observed for the NRC-material but much smaller than the value obtained for the CTC-material. [37] and [38] report on the globulisation of non-spherical (rosette-shaped) grains of a precursor material for the conventional Thixocasting process and refer to a more accelerated coarsening of $252 \leq k [\mu m^3/s] \leq 1320$ compared to ideal spheroids. This corresponds to the CTC-result of $430 \mu m^3/s$ for the alloy with a shape factor $F_s > 1.6$. In [39], however, a value of $148 \mu m^3/s$ is determined for $k$ for the coarsening of spherical grains in the semi-solid state, which corresponds very well with the data achieved for the NRC-structure.

According to [39], the coarsening mechanism of grains in the semi-solid state is a cooperation of Ostwald ripening and grain coalescence. As pure coalescence is supposed to show the same time dependence as pure diffusion driven coarsening [40, 41], it is suggested that the contribution of both mechanisms is independent and that the effective rate constant $k$ can be written as [39]:

$$k = k_C + k_O$$  \hspace{1cm} (4-10)

with $k_C$ and $k_O$ being the rate constants due to coalescence and Ostwald ripening, respectively.

The observed values of the rate constant $k$ suggest that for the coarsening of non-spherical (rosette-shaped) $\alpha$-Al-grains, coalescence is an important if not dominating coarsening
mechanism, whereas for spherical grains the grain coalescence mechanism is suppressed and therefore, Ostwald ripening is predominant. Similar to the coarsening, the contiguity, \( C^\alpha \), of the \( \alpha \)-Al-phase increases with holding time. Furthermore, the \( C^\alpha \)-graphs in Figure 31 point out that the grade of „skeletonisation“ may be controlled by the same mechanism as the coarsening. According to [41], coalescence starts when at first two spherical particles are connected through a neck which contains a grain boundary with low specific energy. Due to the concaveness of the neck, surface material is dissolved at other particle surface points and subsequently re-precipitated at the neck area. The neck area grows and the grain boundary dissapears leading to a more coarsened and connected structure. Figure 33 illustrates the coalescence mechanism schematically.

The NRC-material is more connected than the CTC-material and, therefore, exhibits higher values for \( C^\alpha \). However, even after a holding time of 30 minutes in the semi-solid state the values for \( C^\alpha \) and \( V_C^\alpha \) (with \( V_C^\alpha=0.5C^\alpha \) with \( f=0.5 \)) are within the requested range of 0.3 to 0.6 and 0.15 to 0.3, respectively, for a successful implementation of semi-solid forming [42], which is determined by the flow behaviour of the semi-solid material (see Chapter 4.2.3).

Figure 33: Increase of connectivity due to coalescence [43].
4.2 **Flow Behaviour in The Semi-Solid State**

When semi-solid slurries are placed under shear stress, the steady-state viscosity decreases as the applied shear rate increases, reaching values in the order of 100 mPas [44]. This behaviour is called Rheo-fluidisation and can be explained through the interaction between the solid particles. It is a very common phenomenon observed in suspensions, metal alloys in the semi-solid state, colloids and latexes. Their characteristic behaviour is qualitatively interpreted as a competition between the aggregating (Coulomb attraction, van der Waals' forces, surfaces forces, etc.) and break-up (shear) forces. At a low shear rate (typically lower than 1 s⁻¹), individual particles can aggregate into "clusters" which are able to form a more or less rigid network and the slurry is considered as solid. At very high shear rates, the motion of the particles prevents bonding between particles and leads to a more dispersed suspension that behaves like a fluid. Figure 34 illustrates a typical shear rate dependence of the viscosity for a suspension of interacting particles.

![Figure 34: Rheo-fluidisation (\(\dot{\gamma}_g > \dot{\gamma}_s\)). 1-percolating network, 2-dispersed suspension, 3-suspension of clusters [45].](image)

Usually the rheological behaviour of partially re-melted alloys is examined by parallel plate compression and consequently the applied shear rates are generally limited to \(\dot{\gamma} < 10^{-1} \text{ s}^{-1}\). However, in order to investigate the rheological behaviour of semi-solid alloys under conditions close to those encountered in industrial environments, i.e. under...
higher shear rate conditions (\(\dot{\gamma} > 10 \text{s}^{-1}\)), a backward extrusion technique was developed initially by [46], and subsequently adapted by [43] at the Institute of Metallurgy, ETH Zurich.

4.2.1 BACKWARD EXTRUSION TECHNIQUE

Figure 35 shows a schematic diagram of the experimental apparatus for semi-solid state backward extrusion. Within the stainless steel container the cylindrical specimens (\(\varnothing = 26 \text{mm, } h = 35 \text{mm}\)) of the NRC-material were heated and partially re-melted using a vertical IR tube furnace. After a homogenisation time of 3 minutes the specimens were isothermally backward extruded around the stationary plunger at constant ram speed of 200mm/s. Ram displacement and extrusion forces were measured, and the extruded product was quenched in water.

![Experimental apparatus for semi-solid state backward extrusion](image)

Figure 35: Experimental apparatus for semi-solid state backward extrusion [43].
4.2.2 Shear Rate

For Newtonian fluids, it was deduced that the extrusion force increases linearly with extruded length, due to the likewise linear increase of the area of contact between plunger and fluid. In case of a constant extrusion speed, the apparent viscosity, $\eta_{\text{app}}$, of the fluid can be extracted from the slope, $dF/dt$, of the linear part of the extrusion curve using [46]:

$$\eta_{\text{app}} = \frac{1}{2\pi\lambda C_1 v_r} \cdot \frac{dF}{dt} \quad (4-11)$$

where $\lambda$ is the extrusion ratio (defined as $\frac{R_c^2}{R_c^2 - R_p^2}$, where $R_p$ and $R_c$ are the radii of the plunger and the container, respectively), $v_r$ the ram speed and $C_1$ and $C_2$ constants defined as:

$$C_1 = \frac{1}{\ln\left(\frac{R_c^2}{R_p^2}\right)} \cdot \left(C_2 \cdot (R_c^2 - R_p^2) - v_r\right) \quad (4-12)$$

$$C_2 = \frac{v_r}{(R_c^2 - R_p^2) - \left((R_c^2 + R_p^2) \cdot \ln\left(\frac{R_c}{R_p}\right)\right)} \quad (4-13)$$

The corresponding mean value of the shear rate, $\dot{\gamma}_{av}$, in the gap between the container and the plunger can be calculated from the velocity gradients and is given by:

$$\dot{\gamma}_{av} = \frac{C_1 \cdot \left\{ \ln\left(\frac{-C_1}{2C_2 R_c R_p}\right) - 1 \right\} - (C_2 \cdot (R_c^2 + R_p^2))}{R_c - R_p} \quad (4-14)$$

According to Equation 4-14 for the chosen ram speed of 200mm/s in this work one obtains a shear rate, $\dot{\gamma}_{av}$, of 70s$^{-1}$, which is a good approximation to industrial shear rates.
4.2.3 Flow Characteristics

Figure 36 shows the effect of the liquid fraction, \( f^1 \), on the flow characteristics, measured by backward extrusion at a ram speed of 200mm/s and an isothermal holding time of 3 minutes, for the alloy AlSi7Mg0.3 as a function of temperature. Figure 37 illustrates the decreasing contiguity volume with increasing liquid fractions up to \( f^1=0.5 \).

At a liquid fraction, \( f^1 \), of 0.05 (\( V_c^0 = 1 \)) the alloy shows almost a typical hot forming flow characteristic with a steep increase of the force at small displacements followed by a strong deformation of the specimen. A similar behaviour is observed for specimens with liquid fractions, \( f^1 \), lower than 0.3. In this range of liquid fractions, the bridging of the solid skeleton is highly pronounced, and the contiguity volume, \( V_c^0 \), exceeds the requested range of 0.3. The microstructural examination of the deformed specimens after backward extrusion at \( f^1=0.3 \) revealed an expulsion of the liquid phase and a corresponding densification of the solid at the bottom of the container with only a small area of homogeneous deformation in the middle of the extruded product where solid globules can be found. This is illustrated in Figure 38 and reported in [46]. This suggests that the deformation process is essentially confined to the skeleton. Extensive disruption of the solidly bound particles does not take place and, therefore, no thixotropic behaviour is observed [33].

Figure 36: Extrusion curves for AlSi7Mg0.3 for different liquid fractions (0.05<\( f^1 <0.5 \)).
Figure 37: $C^\alpha$ and $V_C^\alpha$ for different liquid fractions (0.05<$t^l$<0.5).

Figure 38: Evolution of the microstructure within the extruded product at $t^l=0.3$. 

$500 \text{ \mu m}$
At liquid fractions, $f^l$, of 0.4 and 0.5, on the other hand, the slugs are sufficiently stable to maintain their cylindrical shape, nonetheless deform without any remarkable increase in force, indicating that the contiguity volume, $V_C^{c^a}$, is low enough to permit the required disruption of the bridges between the solid grains. Consequently, the flow characteristic can be assumed to be thixotropic at $V_C^{c^a} < 0.3$ (Figure 37). The microstructural evolution within the extruded product at the same positions as shown in Figure 38 at a liquid fraction, $f^l$, of 0.5 and furthermore, the corresponding run of the hardness curves for $f^l = 0.5$ and $f^l = 0.3$ in comparison are illustrated in Figure 39.

![Figure 39](image)

**Figure 39:** Evolution of the microstructure within the extruded product and run of the hardness curve at $f^l = 0.5$.

Solid globules can be found throughout the extruded product thanks to the homogeneous deformation. The run of the hardness curve for $f^l = 0.5$ exhibits a slight increase towards
the upper zone which corresponds to the higher amount of the Si-rich eutectic liquid fraction found in this sector. This can be explained with some expulsion of the liquid phase at the very beginning of the extrusion process until the required critical force is established to break the bridging of the solid skeleton apart. However, for \( f^l = 0.3 \) the hardness values show a much bigger increase than for \( f^l = 0.5 \) the further the distance is from the bottom of the extruded product. This is in agreement with the evolution of the microstructure within the extruded product as shown in Figure 38, in which it can be clearly seen that the upper zones mostly contain the expelled Si-rich eutectic phase and no \( \alpha \)-globules.

For contiguity volume levels below 0.1 at \( f^l > 0.6 \), however, very low extrusion forces are measured, because the solid-phase particles are only weakly interconnected in a 3D network and the billet becomes unstable and looses its shape stability. Therefore, it is of significant importance for a successful implementation of semi-solid forming to exactly adjust the level of the solid and the liquid fraction, respectively.

### 4.3 Solid/Liquid Fraction

Equilibrium solidification takes place when the cooling rate is slow enough to allow extensive solid-state diffusion (diffusion coefficient in the solid \( D_s = \infty \)) and therefore, the solid and liquid will always be homogeneous with compositions following the solidus and liquidus lines illustrated in Figure 40.

The relative amounts of solid and liquid at any temperature are simply given by the lever rule:

\[
 f^s = 1 - f^l = \frac{1}{1 - k} \left( \frac{c_1 - c_0}{c_l} \right) = \frac{1}{1 - k} \left( \frac{T_1 - T}{T_f - T} \right) \tag{4-15}
\]

where \( k = c_s/c_l \) is constant, \( c_0 \) is the alloy composition, \( T_1 \) is the liquidus temperature and \( T_f \) the melting point of the pure substance.
However, very often the cooling rate is too rapid to allow substantial diffusion in the solid phase \((D_s = 0)\) and non-equilibrium solidification takes place as illustrated in Figure 41.
When the first solid containing $k_0$ mol of solute forms at $T_1$, it is purer than the liquid from which it forms ($k_0 < k$) so that solute is rejected into the liquid and raises its concentration above $c_0$. The temperature of the interface must therefore decrease below $T_1$ before further solidification can occur, and the next layer of solid will be slightly richer in solute than the first. As this sequence continues the liquid becomes progressively richer in solute and solidification takes place at progressively lower temperatures. At any stage during solidification, a local equilibrium can be assumed to exist at the solid/liquid interface, i.e. for a given interface temperature the compositions of the solid and the liquid in contact with each other will be given by the equilibrium phase diagram. However, since there is no diffusion in the solid, the separate layers of solid retain their original composition. Furthermore, the mean composition of the solid is always lower than the composition at the solid/liquid interface, as shown by the dashed line in Figure 41.

According to the non-equilibrium lever rule or the Scheil equations [47] the relative amounts of solid and liquid at any temperature are given by:

$$f^s = 1 - f_l = \frac{1}{1 - \left(\frac{c_l}{c_0}\right)^{k-1}}$$

(4-16)

[48] advanced this model by accounting for the effect of partial diffusion in the solid and documented deviations from the results calculated by the Scheil equation depending upon solid diffusion and cooling rate, respectively:

$$f^s = 1 - f_l = \frac{1}{1 - 2\alpha k} \left(1 - \left(\frac{T_f - T}{T_f - T_l}\right)^{k-1}\right)$$

(4-17)

with

$$\alpha = \frac{D_s t_s}{d_p^2}$$

(4-18)

where $t_s$ is the solidification time and $d_p$ the primary dendrite arm spacing or grain size.
Figure 42 illustrates the deviations in the results obtained from ThermoCalc calculations of the amount of liquid phase, $f_l$, as a function of the temperature, $T$, for the alloy AlSi7Mg0.3 using both the Scheil model and the assumption of equilibrium solidification. (Note: $m_f = \text{const}$ for Scheil-ThermoCalc).

In Table 4, the corresponding temperatures for the liquid fraction values $0.1 < f < 0.6$ illustrated in Figure 42 are listed.

<table>
<thead>
<tr>
<th>$f_l = 0.2$</th>
<th>$f_l = 0.3$</th>
<th>$f_l = 0.4$</th>
<th>$f_l = 0.5$</th>
<th>$f_l = 0.6$</th>
<th>$f_l = 0.7$</th>
</tr>
</thead>
<tbody>
<tr>
<td>equilibrium</td>
<td>570°C</td>
<td>572.5°C</td>
<td>574.5°C</td>
<td>578°C</td>
<td>587.5°C</td>
</tr>
<tr>
<td>Scheil-TC</td>
<td>571.5°C</td>
<td>573°C</td>
<td>574°C</td>
<td>577°C</td>
<td>588°C</td>
</tr>
</tbody>
</table>

The deviations in the semi-solid range ($0.4 < f < 0.6$) are very small for this alloy, i.e. $\Delta T \approx 1^\circ \text{C}$, hence in this case, equilibrium solidification can be presumed for the calculation of the fraction solid and fraction liquid, respectively, to adjust the corresponding temperatures.
Figure 43: Enthalpy H as a function of temperature, T, for AlSi7Mg0.3 (by ThermoCalc).

In Figure 43 the enthalpy evolution, H, as a function of the temperature, T, is shown, where $\Delta H$ is the amount of heat which must be removed during cooling from the casting temperature, $T_G$, to the semi-solid forming temperature, $T_F$, in the New Rheocasting process. For the alloy AlSi7Mg0.3, $\Delta H$ is 6506J/mol or 240.5J/g of alloy, respectively, which has to be discharged via the walls of the NRC crucible on its way on the cooling carousel. As mentioned in Chapter 2.3, at the semi-solid forming temperature, $T_F$, of 580°C, corresponding to a liquid fraction of $f^1 = 0.5$, this alloy exhibits a temperature gradient of $\Delta T = 3^\circ C$ from the centre of the slurry to the crucible wall for a melt volume of about $10^3$cm$^3$.

According to Figure 42, for both calculation methods (Scheil and equilibrium calculation) a temperature variation, $\Delta T$, of 3°C at 580°C hardly affects the homogeneity of the slurry over the cross-section concerning the solid/liquid fractions. (Note: ThermoCalc gives liquid fractions, $f^1$, of 0.452, 0.476 and 0.497 for the alloy AlSi7Mg0.3 for equilibrium calculation at 583°C, 580°C and 577°C, respectively).
Nonetheless, as will be discussed later in Chapter 7 and Chapter 8, the temperature gradient, $\Delta T$, varies for different alloys, such as the wrought alloy AlSiMg1 and the secondary cast alloy AlSi9Cu3, under the same environmental conditions in the New Rheocasting process.
Aluminium In The Automotive Industry

With more than 50 million cars and light trucks manufactured each year in the automotive industry the use of aluminium currently averages from 100 to 115kg/vehicle, and forecasts suggest that the aluminium content of an average car will reach 160kg/vehicle by 2005 and up to 272kg/vehicle by 2010 [49]. Aluminium products include rolled products, such as aluminium sheets for heat exchangers, heat shields, bumper stock as well as closure sheet and structural sheet for complete body assemblies. Aluminium alloy extrusions offer designers unparalleled freedom from standard shape restrictions, and the extruded applications include space frames, suspension systems, seat frames and rails, sun roofs, window and door frames as well as aluminium/aluminium metal matrix composite drive-shafts. Forged aluminium products are structural forgings for chassis and suspension parts and forged wheel and airbag components. Nonetheless, more than 75 percent of the aluminium content in cars is in castings. Die castings are used for pistons, transmission housings, suspension components and aluminium metal matrix brake drums and rotors. Sand castings are used for engine blocks, cylinder heads and manifolds, whereas in structural castings cross-members and body structures are produced. Structural die castings and structural permanent mould castings are used for body structures and subframes. The former is currently employed for wheels in 45 percent of new passenger vehicles today.

Semi-solid formed automotive parts are also already on the market and these include parts such as suspension control arms, suspension multilinks, fuel rails, steering knuckles, brake discs and drums, engine brackets, rocker arm pedestal mounts, air conditioning compressor heads, actuator shaft components, air bag cannisters, automotive pulleys, petrol collectors, brake command main cylinders, automobile wheels, power steering valve bo-
xes, brake system components, brake disc crimpers, valve bodies, multiconductor electric connectors, space frame junction nodes and wheel rims [50].

Prior to the appearance on the market of Audi A8 and other cars such as Honda NSX, the use of aluminium in series production of automobiles was generally limited to individual components or modules. The reasons for this were the higher price of aluminium compared to steel and the fact that aluminium could not at the time be processed in robot-intensive assembly plants. However, with the begin of the Audi A2 project in 1995, in which an aluminium compact car was to be designed and produced with reduced fuel consumption thanks to its reduced weight, and with the required safety standards. Furthermore, the car was also envisaged to be competitive in the compact car price segment. This was realised within the aluminium space frame concept which was the starting point for the development of Audi A2. It is a construction in which large aluminium sheets are integrated into a highly stable aluminium frame structure. Furthermore, in addition to Audi, which was a pioneer and remains a leader in the use of aluminium in the construction of auto bodies, other car manufacturing companies also started utilising aluminium to an increased extent in their new car developments. Figure 44 and Figure 45 show the quota of materials used in a Porsche 911 Carrera and in a VW Lupo 3L, respectively, each of them representing a different category of cars.

Due to fierce global competition, the automotive industry has been forcing the rapid advancement of technology. Today's cars are significantly safer, cleaner and more fuel efficient than those of only a few years ago - with an increasing rate of improvement in time. The automotive industry has to consider many factors, the most important ones being fuel economy, emissions, safety and cost. Whilst fuel economy, emissions and safety are government restricted, the cost or value factor is customer mandated. The constantly changing political focus and global regulations cause the automotive companies to constantly re-assess the relative importance of each of these factors and consequently the amount of research effort put into the different areas.
Porsche 911 Carrera

Figure 44: Quota of materials used in a Porsche 911 Carrera.

VW Lupo 3L

Figure 45: Quota of materials used in a VW Lupo 3L.
The impact on energy saving is becoming increasingly important due to a depletion of fossil fuels and an increase of environmental pollution. The US government has therefore promoted the development of a so-called “supercar“ - an environmentally friendly car with up to triple the fuel efficiency of today’s mid-size cars - without sacrificing affordability, performance, or safety [51]. To achieve the fuel economy goal, the reduction of vehicle weight by substituting iron and steel with low density materials such as aluminium, magnesium and composites, has been at the centre of research efforts.

However, since the automotive engineering industry is at present the most important consumer of aluminium and is likely to remain so in the future, developments in car manufacturing are of particular interest for the aluminium industry. The fact that in the latest motor shows the trend towards lightweight construction was dominant, shows that the branch has a hope for the future. It cannot be denied that the aluminium industry can claim to be the first to have responded to the thematic of lightweight construction and that the increased use of aluminium in automobiles gave its impetus to it. Nonetheless, lightweight construction is not necessarily linked to any one material, it is purpose-related and, at least at first instance, independent of particular techniques or materials. Therefore, more lightweight construction does not necessarily mean a higher aluminium consumption due to possible competition from other materials.

The steel industry was provoked into a creativity offensive leading to the development of new materials and improved designs. For example, the development of an UltraLight Steel Auto Body (ULSAB) structure was carried out. In the concept phase of a global steel industry study the structure was proved to be lightweight, structurally sound, safe, executable and affordable. The ULSAB structure weighed 25 percent less than the average benchmarked in the concept phase of the study, and torsion and bending tests showed remarkable improvements. In addition, the fabrication of the ULSAB structure should not cost more than typical auto body structures in its class and could even yield potential cost savings, according to economic analysis [52]. Moreover, perpetuating studies such as ULSAS (UltraLight Steel Auto Suspension) and ULSAC (UltraLight Steel Auto Closure) were released, with the objectives to reduce the mass of a new steel suspension by at least
20 percent versus benchmarked conventional steel-intensive designs without a cost penalty.

In addition, plastics are also gaining ground and it must be borne in mind that its drawbacks of lower mechanical and thermal load capacity along with insufficient shape stability will no longer be true in the future [53].

Meanwhile magnesium alloys will in the medium term become established as another lightweight material offering weight reduction of about 30 percent compared to aluminium alloys and, therefore, further reducing vehicle weight and fuel consumption. Magnesium alloys are amongst the lightest structural materials available, however, they do cost more per unit mass than the competing materials and the automotive industry is reluctant to pay a premium for weight saving. However, the material has considerable potential and one should not be deceived by its problematic processing properties, which are typical for a newcomer to this range of materials.

An important factor for the use of a material in motor vehicle construction is its ability to be reused at the same level of utility at the end of its lifetime. In the case of aluminium, the primary extraction of the metal is energy-intensive, but the energy consumption for melting secondary metal is only about 5 percent of that used for primary metal extraction. The EU published an old automobiles guideline which specifies recycling targets for all old vehicles in the member states. Thus, by January 2006 at the latest, the fraction of materials recovered for re-use from all old vehicles must be increased to at least 85 percent of the average vehicle weight per year. Within the same time limit the fraction reutilised and recycled must increase to at least 80 percent of the average vehicle weight per year. By January 2015, these fractions must increase to 95 percent and 85 percent, respectively. The most important prescription from the standpoint of automobile producers is the obligation to take back old vehicles from their last holders without cost. The guideline encourages recycling friendly design and construction and therefore, this is a great opportunity for aluminium to establish itself in further applications in automotive engineering, because if old automobiles consisted mainly of aluminium there would be no need for prescrip-
tions about recycling them due to their still existing economic value which would ensure their complete recovery and use [54].

One of the most significant challenges for aluminium is definitely to make it more affordable for the vehicles of tomorrow. In the case of semi-solid forming this implies a lowering of the process costs by increasing process stability and enabling in-house recycling, which can be implemented in the New Rheocasting process as explained in Chapter 2.2. Moreover, the use and adaption of more inexpensive secondary alloys for certain components as well as the substitution of forging by semi-solid casting of wrought alloys will lead to a diminution of costs. Furthermore, alloys must be developed or adapted to fulfill the mechanical requirements without the need for costly full heat treatments, solution annealing and artificial aging, which lead to distortion or blistering of castings.
6.1 ALLOY CHARACTERISTICS

The standard casting alloys AlSi7Mg0.3 (A356) and AlSi7Mg0.6 (A357) have been accepted for years as standard semi-solid forming alloys because of their high eutectic content, $\tau^{\text{eut}}$, close to 0.5, their good flow characteristics and corrosion resistance and, in particular, their excellent mechanical properties in the heat-treated condition. For the alloy AlSi7Mg0.3 the microstructural parameters as a function of the casting temperature and as a function of the holding time in the semi-solid state in the New Rheocasting process are given in Chapter 3.4.1 and Chapter 4.1.2, respectively. The flow characteristics as a function of temperature measured by backward extrusion are described in Chapter 4.2.3. The increased Mg-content in AlSi7Mg0.6, however, is intentionally added to induce enhanced age hardening through Mg$_2$Si-precipitation without showing any significant variations in the microstructural properties and flow behaviour in the semi-solid state compared with the alloy AlSi7Mg0.3.

Table 5 shows the chemical composition in weight-percentage of both alloys, AlSi7Mg0.3 and AlSi7Mg0.6, investigated in this work as well as the standard compositions of these alloys.

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Sr</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlSi7Mg0.3</td>
<td>6.97</td>
<td>0.14</td>
<td>&lt;0.01</td>
<td>0.02</td>
<td>0.32</td>
<td>&lt;0.01</td>
<td>0.07</td>
<td>0.015</td>
</tr>
<tr>
<td>A 356</td>
<td>6.5-7.5</td>
<td>&lt;0.2</td>
<td>&lt;0.2</td>
<td>&lt;0.1</td>
<td>0.3-0.45</td>
<td>&lt;0.1</td>
<td>&lt;0.2</td>
<td>-</td>
</tr>
<tr>
<td>AlSi7Mg0.6</td>
<td>6.91</td>
<td>0.12</td>
<td>&lt;0.01</td>
<td>0.02</td>
<td>0.66</td>
<td>&lt;0.01</td>
<td>0.07</td>
<td>0.012</td>
</tr>
<tr>
<td>A 357</td>
<td>6.5-7.5</td>
<td>&lt;0.2</td>
<td>&lt;0.2</td>
<td>&lt;0.1</td>
<td>0.4-0.7</td>
<td>&lt;0.1</td>
<td>&lt;0.2</td>
<td>-</td>
</tr>
</tbody>
</table>
6.2 MECHANICAL PROPERTIES

6.2.1 EXPERIMENTAL PROCEDURE

The melt in the holding furnace, $T_G$, was varied between 630°C and 705°C to investigate the influence of the pouring temperature on the mechanical properties of the formed component. After batching the molten alloy manually into the cup ($Ø=80$mm, $h=120$mm) at a "single cup cooling station" (see Figure 46), it was cooled by air blowing down to the semi-solid forming temperature, $T_F$, of ca. 580°C ($f_s=0.5$) which was measured on-line by a thermocouple in the centre of the slug.

![Figure 46: Single cup cooling station.](image)

After inserting the semi-solid slug into the shot sleeve of an UBE-HVSC-350 squeeze casting machine it was cast into a step plate, which is illustrated in Figure 47. The die tem-
perature was held constant at 270°C and the pressure applied during solidification was 72MPa.

![Figure 47: Step plate.](image)

The step plate exhibits wall thicknesses of 2mm, 6mm, 10mm and 14mm, respectively. After removing the step plate from the die, it was immediately water-quenched and then heat treated.

Table 6 gives an overview of the heat treatment parameters for AlSi7Mg0.3 and AlSi7Mg0.6.

<table>
<thead>
<tr>
<th>condition</th>
<th>heat treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>as cast</td>
</tr>
<tr>
<td>T4</td>
<td>solution annealed 520°C/2h + water quenched</td>
</tr>
<tr>
<td>T5</td>
<td>artificially aged 160°C/7h + water quenched</td>
</tr>
<tr>
<td>T6</td>
<td>T4 + artificially aged 160°C/7h + water quenched</td>
</tr>
</tbody>
</table>
For the examination of the static mechanical properties by uniaxial tensile strength tests at room temperature, round tensile test specimens were taken from the 14mm, 10mm, and 6mm thickness steps and flat tensile test specimens from the 2mm step.

6.2.2 Static Mechanical Properties

The effect of the pouring temperature on the microstructural parameters $D_\alpha$ and $F_\alpha$ is described in Chapter 3.4.1. Figure 48 illustrates the influence of the pouring temperature on the static mechanical properties in the T6-condition for each step of the step plate.

![Figure 48: Mechanical properties of AlSi7Mg0.3 in the T6-condition for the four steps of the step plate as a function of the pouring temperature.](image)

A high level of ductility, of around $13\pm2\%$ can be obtained for steps 1 (14mm), 2 (10mm) and 3 (6mm), at a casting temperature, $T_G$, of 630°C, whereas the values for elongation to fracture for step 4 (2mm) in comparison decrease significantly to $6\pm1\%$. However, a casting temperature, $T_G$, of 705°C leads to a striking deterioration of the mechanical pro-
properties. Furthermore, the fourth step exhibits elongation to fracture values of about 2%, i.e. the specimens partly break with low plastic deformation.

Table 7 gives a summary of the average results achieved in the T6-condition for the three thickest steps of the step plate for different casting temperatures, \( T_G \), and forming temperatures, \( T_F \).

Table 7: Average mechanical properties of the steps 1 (14mm), 2 (10mm) and 3 (6mm).

<table>
<thead>
<tr>
<th>( T_G ) [°C]</th>
<th>630</th>
<th>650</th>
<th>675</th>
<th>705</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_F ) [°C]</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>580</td>
<td>581</td>
<td>582</td>
<td>580</td>
<td>580</td>
</tr>
<tr>
<td>YS [MPa]</td>
<td>247±8</td>
<td>245±8</td>
<td>243±9</td>
<td>240±9</td>
</tr>
<tr>
<td>UTS [MPa]</td>
<td>313±3</td>
<td>310±3</td>
<td>310±4</td>
<td>309±7</td>
</tr>
<tr>
<td>A [%]</td>
<td>12.7±1</td>
<td>12.4±1.5</td>
<td>12.3±1.2</td>
<td>12.9±1</td>
</tr>
</tbody>
</table>

The values for yield strength, YS, and ultimate tensile strength, UTS, for the alloy AlSi7Mg0.3 in the T6-condition are high with 250MPa and 310MPa, respectively, for all variations of casting and forming temperatures. As a vital aspect of quality the values for elongation to fracture, A, are of particular interest. They are on a high level in a range of 12% for casting temperatures, \( T_G \), up to 650°C (i.e. 30°C above liquidus temperature, \( T_L \)) and exhibit a significant decrease to 5% at casting temperatures of 705°C (i.e. 85°C above liquidus temperature, \( T_L \)).

Figure 49 illustrates, in contrast, the microstructures of step 2 (10mm) and step 4 (2mm) of the step plates fabricated with casting temperatures, \( T_G \), of 630°C and 705°C, respectively, and a constant forming temperature, \( T_F \), of 580°C.
Figure 49: Microstructures of steps 2 and 4 for AlSi7Mg0.3 in the T6-condition with $T_G=705^\circ$C and 630°C.

For the microstructure of step 4 and the combination of parameters $T_G=630^\circ$C/$T_F=580^\circ$C one can observe a higher eutectic fraction, $f_{^\text{eut}}$, compared to step 2 ($\Delta f_{^\text{eut}}=0.08$) leading to a significant decline of elongation in step 4. This increase in $f_{^\text{eut}}$ in the thinnest step can be explained by some expulsion of liquid eutectic phase at the beginning of forming, when a certain force has to be applied to deform the solid skeleton to transform the slug into a solid-liquid suspension. Therefore, in front of the die-filling semi-solid front a higher amount of liquid eutectic fraction can be expected. For the variation $T_G=705^\circ$C/$T_F=580^\circ$C the microstructure features an insufficient globulisation of the $\alpha$-Al-phase. The material exhibits scarce thixotropic properties and the eutectic fractions, $f_{^\text{eut}}$, of around 0.45 are high for step 2 as well as for step 4. Moreover, in the casting trials it became ap-
parent, that the thinnest step of the step plate could not be cast without defects with this set of parameters and thus showed very poor elongation properties.

Figures 50 to 53 illustrate the static mechanical properties of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 for all four steps of the step plate in the F-, T4-, T5- and T6-condition, respectively. The "ideal" parameters chosen for these alloys in the New Rheocasting process were $T_G = 630^\circ C$ and $T_F = 580^\circ C$.

![Graph of mechanical properties](image)

Figure 50: Mechanical properties of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the F-condition.
Figure 51: Mechanical properties of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the T4-condition.

Figure 52: Mechanical properties of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the T5-condition.
For all four heat treated conditions there is a remarkable increase visible in the yield strength and ultimate tensile strength in the thinnest fourth step (2mm), which is accompanied by a significant loss of ductility due to the increased amount of eutectic phase in this step. However, there exists an apparent constancy of the mechanical properties in the three thicker steps of the step plates indicating the uniformity of the microstructure in these areas.

The higher amount of magnesium in the alloy AlSi7Mg0.6 leads to a significant improvement compared to AlSi7Mg0.3 in terms of strength as well as in terms of ductility, especially in the T5- and T6-condition (i.e. for the first three steps of AlSi7Mg0.6 values for yield strengths of 190±10 MPa and 280±10 MPa with elongations of 12±2 % and 16±2 % in the T5- and the T6 -condition, respectively, compared to yield strengths of 155±10 MPa and 235±10 MPa, and elongations of 9±1 % and 13±2 % for AlSi7Mg0.3). This is surprising because an increase of the magnesium content usually entails a decrease of ductility.
coming along with elevated strength values. Therefore, the better elongation values for the alloy AlSi7Mg0.6 could lead to the assumption that the chosen casting parameters for this alloy were perfect.

Figure 54 shows the obtained data for yield strength and elongation to fracture for the first three steps for the alloy AlSi7Mg0.3 in the T6-condition compared with selected "best-values" in literature for conventional thixocasting and with standard values achieved with this alloy by chill casting, sand casting and high pressure die casting, respectively.

![Figure 54: Mechanical properties of cast components of the alloy AlSi7Mg0.3 in the T6-condition.](image)

The mechanical properties obtained in the NRC process are located at the upper end of the scatter of the results and therefore, indicating that with this process components exhibiting a striking quality can be fabricated.

To verify the excellent static mechanical properties attained for the step plate, a complex and harder to cast component (see Figure 55) was fabricated on the HVSC PL-800 NRC machine with the alloy AlSi7Mg0.3. The formed part exhibited a wall thickness from
2mm to 5mm on a length of 55cm at a casting weight of about 3kg. The pressure applied during solidification was 140MPa, whereas all the other process parameters were identical as for the step plate. The mechanical properties achieved in the T6-condition were a yield strength of 238±10 MPa, an ultimate tensile strength of 316±10 MPa at elongation values of 15±1.5 % consistently in all areas of the component [62].

![Component fabricated on the HVSC PL-800 NRC machine with the alloy AlSi7Mg0.3.](image)

6.2.3 Dynamic Mechanical Properties

The specimens for testing the dynamic mechanical properties, i.e. fatigue strength, were cylindrical hourglass-shaped with a minimum diameter $\varnothing=6$mm and taken from the two thickest steps. The tests were conducted at room temperature on a servo-hydraulic tensile/compression test machine with $R=0.1$ and $f=50$Hz.

Figures 56 to 59 show the results of the fatigue tests for the 14mm and the 10mm step of the step plate of both alloys, AlSi7Mg0.3 and AlSi7Mg0.6 in the F-, T4-, T5- and T6-condition, respectively. The fatigue limit was reached at $10^7$ cycles.
Figure 56: Fatigue strength of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the F-condition. R=0.1.

Figure 57: Fatigue strength of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the T4-condition. R=0.1.
Figure 58: Fatigue strength of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the T5-condition. R=0.1.

Figure 59: Fatigue strength of NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 in the T6-condition. R=0.1.
The fatigue strength values for NRC-AlSi7Mg0.6 are higher than for NRC-AlSi7Mg0.3 in all heat treatment conditions, thus corresponding to the results obtained in the static mechanical tests. The fatigue limit for the alloy with the higher magnesium content achieved in the F- and T4-condition exhibits a maximum stress of 155MPa, whereas in the T5- and T6-condition more than $10^7$ load cycles were attained at a maximum stress of 165MPa. For the alloy AlSi7Mg0.3, however, fatigue limits of 125MPa, 140MPa, 150MPa and 155MPa were reached in the F-, T4-, T5- and T6-condition, respectively.

Table 8: Fatigue strengths of AlSi7Mg0.3 at $10^7$ load cycles.

<table>
<thead>
<tr>
<th>condition</th>
<th>R=0.1 maximum stress [MPa]</th>
<th>R=-1 maximum stress [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRC</td>
<td></td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>125</td>
<td>-</td>
</tr>
<tr>
<td>T4</td>
<td>140</td>
<td>-</td>
</tr>
<tr>
<td>T5</td>
<td>150</td>
<td>100 [x]</td>
</tr>
<tr>
<td>T6</td>
<td>155</td>
<td>120 [x]</td>
</tr>
<tr>
<td>Badiali [61] - thixo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>-</td>
<td>135</td>
</tr>
<tr>
<td>Bieri [56] - thixo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T4</td>
<td>145</td>
<td>-</td>
</tr>
<tr>
<td>T6</td>
<td>145</td>
<td>-</td>
</tr>
<tr>
<td>Gabathuler [63] - thixo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>150</td>
<td>-</td>
</tr>
<tr>
<td>- chill</td>
<td>T6</td>
<td>120</td>
</tr>
<tr>
<td>- die</td>
<td>T6</td>
<td>80</td>
</tr>
<tr>
<td>- sand</td>
<td>T6</td>
<td>80</td>
</tr>
<tr>
<td>Wang [64] - chill</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>130</td>
<td>90</td>
</tr>
<tr>
<td>Davidson [65] - squeeze</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>-</td>
<td>130</td>
</tr>
<tr>
<td>Davidson [66] - chill</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>-</td>
<td>111</td>
</tr>
</tbody>
</table>

Table 8 illustrates the obtained fatigue limits for the first two steps for the alloy AlSi7Mg0.3 under different heat treatment conditions compared with data reported in the literature achieved by conventional thixocasting, squeeze casting, chill casting, sand casting and high pressure die casting. One can perceive that the fatigue strength is significantly higher for components fabricated in both semi-solid forming techniques, New Rheocasting and Thixocasting, than for sand cast, high pressure die cast or chill cast parts.
This can obviously be traced back to the fact that in comparison semi-solid formed components are classified as free of pores and voids. As reported in [64] the fatigue life of cast alloys containing defects can be one or two orders of magnitude lower than in defect-free components. The presence of casting defects shortens not only the fatigue crack propagation period, but also the initiation period.

6.3 Modification With Barium

Besides strontium, also barium was described as a modifying agent for the eutectic phase in aluminium cast alloys [67] creating a fibrous or lamellar morphology of the silicon. However, [43] modified an AA6082 alloy to tailor it specifically for semi-solid processing by adding barium to reduce grain growth and coalescence of contacting grains. It was found in [68] that small amounts of barium diminish the solid/liquid interface energy, \( \gamma_{\text{SL}} \), and consequently increase the penetration of the liquid phase between the \( \alpha \)-Al grains, which in turn leads to lower contiguity values, \( C^\alpha \). Furthermore, in case of the alloys AlSi7Mg0.3 and AlSi7Mg0.6, a diminution of the connectivity of the \( \alpha \)-Al grains could lead to the reduction of the force which must be applied to deform the solid skeleton to transform the slug into a solid-liquid suspension. Therefore, the amount of liquid eutectic fraction in front of the die-filling semi-solid front can be expected to be lower, leading to enhanced cavity filling behaviour for the thinnest step of the step plate and thus evoking improved mechanical properties.

Figure 60 illustrates the static mechanical properties of a NRC-AlSi7Mg0.3 alloy additionally modified with 0.05 weight-% barium in the T6-condition in the different steps of the step plate. One can observe a more balanced distribution of the mechanical properties over all four steps of the step plate with the barium-modified alloy which in turn leads to the attainment of high elongation values, \( 10.5 \pm 1.5 \% \) in the 2mm step, due to the reduced eutectic phase compared to the unmodified alloy which has a value of \( 6.2 \pm 1 \% \). For the alloy AlSi7Mg0.6, however, it appears that a modification with 0.05 weight-% barium
leads to a deterioration of the ductility in all the steps as shown for the T6-condition in Figure 61.

**Figure 60:** Mechanical properties of AlSi7Mg0.3 and AlSi7Mg0.3Ba in the T6-condition.

**Figure 61:** Mechanical properties of AlSi7Mg0.6 and AlSi7Mg0.6Ba in the T6-condition.
One reason for this diminution of elongation properties in the alloy with an increased amount of magnesium could be the accumulated occurrence of barium containing intermetallic compounds in the microstructure of NRC-AlSi7Mg0.6Ba as illustrated in Figure 62. A detailed analysis by SEM revealed Ba, Mg, Si and Al as constituents of these compounds. The barium-containing intermetallic phases could not be found in the barium-modified AlSi7Mg0.3 alloy and, therefore, this leads to the assumption that only the increased amount of magnesium promotes the formation of these compounds. This corresponds very well with observations made in [69], in which magnesium was said to react with strontium to form a complex intermetallic compound Mg₂Sr(Si₃Al₄) in a strontium-modified AlSi7Mg-alloy with magnesium contents in excess of 0.6%. The formation of such compounds is expected to happen in the liquid before the eutectic reaction.

The alloys NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 proved to exhibit excellent mechanical properties. The strength and ductility levels as well as the fatigue properties in all heat treatment conditions achieved with the New Rheocasting process - representing standard values for this process - can be reached in the conventional Thixocasting process only under very favourable process conditions.
7.1 ALLOY CHARACTERISTICS

The aluminium wrought alloy AlMgSi1 (AA6082) is widely used for extrusion in structural applications and offers attractive mechanical properties, such as high strength and elongation, and is characterised by good weldability and corrosion resistance. A typical feature of this alloy is the high strength developed after heat treatment including solution annealing and artificial aging due to precipitation of Mg₂Si and additional Si particles. Sometimes wrought alloys are requested for optical reasons, because casting alloys turn into a dark grey colour during anodising. On the other hand, wrought alloys show substantial tendency for hot cracking after being cast and their flow properties are much worse than those of the silicon containing casting alloys such as AlSi7Mg.

Semi-solid forming could overcome these drawbacks of cast wrought alloys, if stable semi-solid processing conditions can be attained. However, to achieve reproducible part properties, it is normally considered as necessary to use alloys with a large solidification range. Figure 63 indicates that the temperature range for liquid fractions, f, between 0.4 and 0.6 (calculated by using the Scheil equation in ThermoCalc) is 19°C for the casting alloy AlSi7Mg0.3 whereas it is only 5°C for the wrought alloy AlMgSi1. Table 9 shows the chemical composition in weight-percent of the alloy NRC-AlMgSi1 investigated in this work as well as the standard composition of this alloy.

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlMgSi1</td>
<td>1</td>
<td>0.23</td>
<td>0.05</td>
<td>0.42</td>
<td>0.69</td>
<td>0.09</td>
<td>0.07</td>
<td>0.1</td>
</tr>
<tr>
<td>AA 6082</td>
<td>0.7-1.3</td>
<td>&lt;0.5</td>
<td>&lt;0.1</td>
<td>0.1-0.45</td>
<td>0.6-1.2</td>
<td>&lt;0.2</td>
<td>&lt;0.15</td>
<td>&lt;0.1</td>
</tr>
</tbody>
</table>
In conventional thixoforging this narrow processing window causes some difficulties in establishing a homogeneous solid/liquid distribution in the reheated slugs [70]. For the New Rheocasting process, however, the temperature distribution at the semi-solid forming temperature, \( T_F \), showed a temperature gradient of only \( \Delta T \approx 1 \, ^\circ C \), from the centre of the slurry to the crucible wall, for a melt volume of about \( 10^3 \, \text{cm}^3 \), indicating a very homogeneous precursor material. (Note: for AlSi7Mg0.3 \( \Delta T \approx 3 \, ^\circ C \) as reported in Chapter 4.3).

Figure 64 illustrates the enthalpy, \( H \), as a function of the temperature, \( T \), for the alloy AlMgSi1 with \( \Delta H \) being the amount of heat which must be removed during cooling from the casting temperature, \( T_G \), of 660°C, i.e. 10°C above liquidus temperature, to the semi-solid forming temperature, \( T_F \), of 643°C. The enthalpy, \( \Delta H \), which must be discharged via the walls of the NRC crucible on its way on the cooling carousel within a temperature range, \( \Delta T_{GF} \), of only 17°C, is of 6625J/mol or 245.3J/g for AlMgSi1, which is comparable to the \( \Delta H \) value for AlSi7Mg0.3 (6506J/mol, see Chapter 4.3) within a \( \Delta T_{GF} \) of 50°C. Therefore, a lower temperature gradient and a more uniform temperature distribution over
the cross-section of the slurry can be expected for AlMgSi1 at the same environmental conditions for the New Rheocasting process.

A temperature variation, ΔT, of 1°C at 643°C, however, maintains the homogeneity of the solid/liquid fraction of the slurry over the cross-section in the requested range and, therefore, guarantees the process stability. (Note: ThermoCalc for the alloy AlMgSi1 for equilibrium calculations at 644°C, 643°C and 642°C yielded liquid fractions, f1, of 0.572, 0.536 and 0.494, respectively).

Figure 64: Enthalpy, H, as a function of temperature, T, for AlMgSi1 (by ThermoCalc).
7.2 SEMI-SOLID MICROSTRUCTURE

7.2.1 EFFECT OF POURING TEMPERATURE

Figure 65 shows the experimental New Rheocasting microstructures for the alloy AlMgSi1 for different casting temperatures between 660°C and 700°C (i.e. 10°C to 50°C above liquidus temperature for this alloy). All micrographs in Figure 65 represent the morphological state at a semi-solid temperature of 643°C. As for the alloy AlSi7Mg0.3 described in Chapter 3.4.1, after filling the crucible, it was cooled with air and water-quenched at the moment the semi-solid metal reached 643°C. To quantify the microstructures illustrated in Figure 65, Table 10 lists the microstructural parameters $D^\alpha$, $F^\alpha$ and $C^\alpha$.

![Figure 65: NRC-microstructure of AlMgSi1 as a function of the casting temperature.](image)

Table 10: NRC-microstructure as a function of the casting temperature for AlMgSi1.

<table>
<thead>
<tr>
<th></th>
<th>$660^\circ$C</th>
<th>$680^\circ$C</th>
<th>$700^\circ$C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$D^\alpha$ [µm]</td>
<td>107</td>
<td>109</td>
<td>143</td>
</tr>
<tr>
<td>$F^\alpha$ [-]</td>
<td>1.25</td>
<td>1.28</td>
<td>1.31</td>
</tr>
<tr>
<td>$C^\alpha$ [-]</td>
<td>0.31</td>
<td>0.27</td>
<td>0.25</td>
</tr>
</tbody>
</table>

The microstructural parameters for the $\alpha$-Al phase exhibit values in the required range for successful semi-solid forming for casting temperatures, $T_G$, up to 680°C (i.e. 30°C above liquidus temperature). However, at a $T_G$ of 700°C, a deterioration of the shape factor, $F^\alpha$,
is observed and the grain size, $D^\alpha$, is close to the upper limit of 150\(\mu\text{m}\). Therefore, a casting temperature, $T_G$, not exceeding 30°C above liquidus temperature is taken as a "recommended" NRC casting parameter for the alloy AlMgSi1 as well as for the alloy AlSi7Mg0.3 (see Chapter 3.4.1).

7.2.2 Effect Of Holding Time In The Semi-Solid State

Figure 66 documents the change in the $\alpha$-Al phase morphology during a holding period of 1 minute, 5 and 30 minutes, respectively, at the semi-solid temperature ($f^\alpha=0.5$) of 643°C after a casting temperature, $T_G$, of 660°C. Table 11 lists the microstructural parameters $D^\alpha$, $F^\alpha$ and $C^\alpha$ to quantify the microstructures illustrated in Figure 66.

![Figure 66: NRC-microstructure of AlMgSi1 after different holding periods in the semi-solid state.](image)

<table>
<thead>
<tr>
<th></th>
<th>1 min</th>
<th>5 min</th>
<th>30 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>$D^\alpha$ [(\mu\text{m})]</td>
<td>125</td>
<td>143</td>
<td>182</td>
</tr>
<tr>
<td>$F^\alpha$ [-]</td>
<td>1.17</td>
<td>1.14</td>
<td>1.12</td>
</tr>
<tr>
<td>$C^\alpha$ [-]</td>
<td>0.31</td>
<td>0.36</td>
<td>0.49</td>
</tr>
</tbody>
</table>

It can be seen that at long holding times, the grain size, $D^\alpha$, by far exceeds the requested limits of 150\(\mu\text{m}\) for semi-solid forming and that due to grain coalescence the value for the contiguity of the $\alpha$-Al phase, $C^\alpha$, indicates a strongly inter-connected solid skeleton lea-
96

Ding to a deterioration of the material's flow behaviour. However, as mentioned in Chapter 4.1.2 for the alloy AlSi7Mg0.3, a holding time of 30 min is not possible on the cooling carousel in the New Rheocasting process and, therefore, this investigation is only of "academic" relevance.

7.3 Flow Behaviour In The Semi-Solid State

The rheological properties of the re-melted alloy, originally cast at a T_G of 660°C, were studied by means of backward extrusion technique as shown in Chapter 4.2.1. Figure 67 shows the effect of the liquid fraction, f_L, on the flow characteristics for the alloy NRC-AlMgSi1 as a function of temperature. The specimens were isothermally backwards extruded after an isothermal holding time of 3 minutes. Figure 68 illustrates the decreasing contiguity volume, V_C, with increasing liquid fractions up to f_L of 0.5.

![Figure 67: Extrusion curves for NRC-AlMgSi1 at different liquid fractions (0.1 < f_L < 0.5).](image)

For specimens with liquid fractions f_L < 0.3 the alloy shows a typical hot forming flow characteristic with a steep increase of the force at small displacements (quasi-elastic range) followed by deformation of the specimen before reaching a steady-state plateau. In this
range of liquid fractions (as for the alloy AlSi7Mg0.3 described in Chapter 4.2.3), the bridging of the solid skeleton is highly pronounced, and the contiguity volume, $V_C^\alpha$, exceeds 0.3.

![Figure 68: Contiguity, $C^\alpha$, and contiguity volume, $V_C^\alpha$, as a function of the liquid fraction, $f^\ell$.](image)

The microstructure of the deformed specimens (see Figure 37) after backward extrusion shows expelled liquid phase and a corresponding densified solid phase at the bottom of the container, as shown in Figure 69 on the left. The deformation process is essentially confined to the skeleton, and extensive disruption of the solidly bound particles does not take place and, therefore, no thixotropic behaviour is observed for liquid fractions $f^\ell < 0.3$ for the alloy AlMgSi1. At liquid fractions, $f^\ell$, of 0.4 and 0.5, on the other hand, the slugs exhibit thixotropic properties. They deform without any remarkable increase in force, indicating that the contiguity volume, $V_C^\alpha$, is low enough to permit the required disruption of the bridges between solid grains. The microstructural evolution within the extruded product is illustrated in Figure 69 on the right, where solid globules can be found from top to bottom due to the homogeneous deformation.
Figure 69: Evolution of the microstructure within the extruded product at $f^1=0.1$ (left) and $f^1=0.5$ (right).

To accentuate the correctness of the observed borderline of 0.3 for thixotropic forming in terms of the amount of fraction liquid, $f^1$, Figure 70 shows the average static mechanical properties in the T4-condition (see Table 12) of the first three steps of the step plate (14mm, 10mm and 6mm) as a function of the forming temperature, $T_F$. The casting temperature, $T_G$, was 660°C and the forming temperatures were 638°C, 640°C and 644°C corresponding respectively to $f^1$ values of 0.3, 0.35 and 0.45.
For the sample formed at a fraction liquid, $f_1$, of 0.3 there is an apparent loss of ductility compared to the specimens fabricated with a forming temperature, $T_F$, corresponding to liquid fractions of 0.35 and 0.45 (i.e. 17±2 % and 16±2 % for $f_1$ of 0.45 and 0.35, respectively and 7.5±1 % for the $f_1$ value of 0.3). This dramatic diminution of the elongation properties can be explained by the disturbed die-filling behaviour due to the expulsion of the liquid phase in front of the semi-solid front.

### 7.4 Mechanical Properties

The experimental procedure was similar to the one described in Chapter 6.2.1 for the alloy NRC-AlSi7Mg0.3. The casting temperature, $T_G$, and the semi-solid forming temperature, $T_F$, for the alloy NRC-AlMgSi1, however, were kept constant at 660°C and 643°C, respectively. Table 12 gives an overview of the heat treatment parameters for AlMgSi1.
Table 12: Heat treatment conditions for AlMgSi1.

<table>
<thead>
<tr>
<th>condition</th>
<th>heat treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>as cast</td>
</tr>
<tr>
<td>T4</td>
<td>solution annealed 540°C/5h + water quenched</td>
</tr>
<tr>
<td>T5</td>
<td>artificially aged 170°C/10h + water quenched</td>
</tr>
<tr>
<td>T6</td>
<td>T4 + artificially aged 170°C/10h + water quenched</td>
</tr>
</tbody>
</table>

7.4.1 Static Mechanical Properties

In contrast to the experiments with the alloys NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6, with the alloy NRC-AlMgSi1 a proper and constant filling could not be achieved for the thinnest step of the step plate (2mm) because of the alloy’s known tendency to feature “hot cracks“ in thin wall-sections and transitions between different wall thickness sections. Therefore, in Figures 71 to 74 the static mechanical properties of NRC-AlMgSi1 in the F-, T4-, T5- and T6-condition, respectively, are given for the first three steps of the step plate only.

The mechanical properties appear to be homogeneous over all three steps with a slight drift towards higher strength values in the third step in all heat treatment conditions. Average values for yield strength, ultimate tensile strength and elongation in the F- and T6-condition, for example, are between 80MPa and 340MPa, 160MPa and 390MPa and 17% and 5%, respectively. Therefore, the requested standards for forged components with this alloy, which are a minimum yield strength of 260MPa and a minimum ultimate tensile strength of 315MPa are clearly exceeded by the NRC-AlMgSi1 properties in the T6-condition. However, the required elongation values of 6% cannot be reached. The gap must be due to the fact that at least the initial liquid portion of the alloy solidifies in non-equilibrium, which results in the formation of eutectic and undesired secondary phases in the as-cast microstructure of the step plate as indicated in Figure 75.
Figure 71: Mechanical properties of NRC-AlMgSi1 in the F-condition.

Figure 72: Mechanical properties of NRC-AlMgSi1 in the T4-condition.
Figure 73: Mechanical properties of NRC-AlMgSi1 in the T5-condition.

Figure 74: Mechanical properties of NRC-AlMgSi1 in the T6-condition.
Figure 75: Microstructure with eutectic silicon phase (dark areas) of NRC-AlMgSi1 in the F-condition after casting into the step die.

7.4.2 NON-EQUILIBRIUM SOLIDIFICATION

Figure 76 gives a schematic illustration of the ternary Al-Si-Mg system.

Figure 76: Schematic illustration of a pseudo-binary section of the ternary Al-Si-Mg system. Bold lines reflect the equilibrium, dashed line represents non-equilibrium.
While in equilibrium (bold lines in Figure 76), the eutectic phase occurs at a Si-content of
>1.49% ($c_{\text{max}}$), the real NRC-cast structure must be considered to be formed in non-equilibrium (dashed line in Figure 76). However, normally, a non-equilibrium state is observed at fast cooling conditions and, therefore, in the New Rheocasting process at a fraction solid, $f^\alpha$, of 0.5 before forming, the system is considered to be in equilibrium. To explain the appearance of eutectic phase in the microstructure of the final component, one can suppose a strongly suppressed silicon diffusion during solidification in the die. Assuming that "no diffusion in solid, perfect mixing in liquid" takes place, the non-equilibrium solidification (from a fraction solid, $f^\alpha$, greater than 0.5) can be predicted by the Scheil equation (see Chapter 4.3). Figure 77 shows the fraction solid, $f^\alpha$, as a function of the temperature, $T$, for semi-sold processing of the alloy AlMgSi1 for equilibrium and non-equilibrium solidification as calculated by Scheil.

![Scheil calculation predicts non-equilibrium formation of eutectic phase.](image)

One can see that before reaching the eutectic temperature in non-equilibrium solidification there is still a remainder of liquid phase present, thus causing the occurrence of non-equilibrium eutectic in the microstructure of the step plates and therefore, leading to deteriorated elongation properties.
7.5 MODIFICATION WITH BARIUM

As mentioned in Chapter 6.3 and reported in [43], small amounts of barium diminish the solid/liquid interface energy, \( \gamma_{\text{al}} \), and consequently increase the penetration of the liquid phase between the \( \alpha \)-Al grains, which in turn lead to lower contiguity values, \( C^\alpha \). Figure 78 indicates the contiguity volume, \( V_C^\alpha \), as a function of the isothermal holding time at the semi-solid temperature compared with a 0.2 weight-% barium-containing and an unmodified NRC-AlMgSi1 alloy. The contiguity volume, \( V_C^\alpha \), of the solid \( \alpha \)-Al-phase of the alloy modified with barium remains in the range of 0.2 to 0.25 and does not exceed the critical value of 0.3, thus forming a weakly inter-connected solid skeleton, and consequently allowing a homogeneous deformation of the semi-solid slug even after longer homogenisation times. On the other hand, the contiguity volume of the unmodified NRC-AlMgSi1 alloy exceeds a value of 0.3 and loses its shear thinning behaviour.

Furthermore, a diminution of the connectivity of the \( \alpha \)-Al grains in the Ba modified NRC-AlMgSi1 alloy should lead to an easier deformation of the solid skeleton during the forming process and, therefore, improve the cavity filling behaviour especially for the thinnest step of the step plate.
7.5.1 MICROSTRUCTURE

Figure 79 and Figure 80 illustrate typical sections of the microstructure of the unmodified and Ba-modified alloy, respectively. It can be clearly seen that the structure of both alloys is characterised by the presence of secondary phases. NRC-AlMgSi1 features local liquid pocket segregations with needle shaped silicon ("non-equilibrium eutectic" due to non-equilibrium solidification), which is accompanied by polygonal $\alpha-(\text{AlFeSi})$ compounds, where Fe is partly substituted by Mn. The microstructure of NRC-AlMgSi1Ba exhibits more rounded and rosette-like shaped phases along the grain boundaries. According to EDX-measurements these phases are of the same composition as the Si and $\alpha$-phases; additionally, a strong barium signal is obtained. The morphology of the secondary phases indicates that barium acts as a modifying element. As mentioned in Chapter 6.3, [67] reported that elements such as barium, modify the eutectic silicon flakes in aluminium-silicon cast alloys to branched fibres with high twin densities, and primary flakes to nearly spherical shapes.

Figure 79: Microstructure of NRC-AlMgSi1 in the T6-condition, characterised by the presence of secondary phases along grain boundary. Sections of liquid pocket segregation (right) show pronounced formation of needle shaped Si and $\alpha$-(AlFeSi) compounds.
7.5.2 Static Mechanical Properties

Figure 81 shows the yield strength, YS, the ultimate tensile strength, UTS, and the elongation to fracture, A, for both the Ba-modified and unmodified NRC-AlMgSi1 alloy in the T6-condition.
However, the modification of the alloy with barium did not enhance the cavity filling behaviour to enable the fabrication of the thinnest fourth step of the step plate of a consistent quality. In addition, for the 14mm, 10mm and 6mm step no significant difference in the static mechanical properties between the two alloys were noted. There was only a slight decrease in strength visible accompanied by a small increase in ductility for the alloy modified with barium. This increase may be attributed to the modified eutectic phase.

7.5.3 Dynamic Mechanical Properties

Although the effect of barium addition on the morphology of the secondary phases did not significantly affect the static mechanical properties, a significant influence on the dynamic properties was observed. Figure 82 shows the results of the fatigue tests for the two thickest steps (14mm and 10mm) of the step plate in the T6-condition. The Ba-modified alloy exhibited significantly higher fatigue strength with a value of 185MPa compared to the unmodified AlMgSi1 alloy which has a maximum stress, $\sigma_{\text{max}}$, of 135MPa.

![Graph showing fatigue strength comparison](image)

Figure 82: Fatigue strength of NRC-AlMgSi1Ba and NRC-AlMgSi1 in the T6-condition. $R=0.1$. 

In the alloy NRC-AlMgSi1, the needle-shaped eutectic Si particles, which are up to 30μm long, may act as fatigue crack initiation sites and may reduce the fatigue strength, whereas crack nucleation at the rounded phases in the alloy NRC-AlMgSi1Ba is assumed to be less dominant and therefore, the fatigue limit is increased compared to barium free AlMgSi1.

*The alloy NRC-AlMgSi1 proved to be “processable“ with the New Rheocasting process. Due to the alloy’s tendency towards “hot cracks“ in thin-walled sections, applications for this alloy are restricted to more thick-walled parts. The level of ductility for this alloy in the wrought or forged condition could not be reached, because the cast microstructure solidified in non-equilibrium, promoting the appearance of non-equilibrium eutectic phase and, therefore, reducing the elongation values.*
8.1 Alloy Characteristics

AlSi9Cu3 is a very frequently employed die-casting alloy. It is used in almost all fields of pressure die casting, especially in the fabrication of pumps, engine housings and gearings. AlSi9Cu3 is easily castable, possesses high mechanical strength values at elevated temperatures and is inexpensive because it is a secondary alloy. In order to reduce the tendency towards sticking, pressure die casting alloys require a higher content of Fe or, alternatively, Mn than the corresponding sand or gravity die casting alloys. However, the usual amount of Fe or Mn of 0.5 to 1 weight-% in pressure die casting alloys gives rise to the precipitation of intermetallic phases containing Fe or Mn during solidification. The presence of Fe results in the formation of acicular and plate structured β-phase (Al₅FeSi), whereas an increased Mn content primarily leads to the formation of α-phase (Al₁₅Fe₃Si₂) in the form of polygonal hard phases or so-called Chinese-script, whereby Fe is partially substituted by Mn or Cr ((Al, Cu)₁₅(Fe, Mn, Cr)₃Si₂) [71]. On account of the notch effect due to these intermetallic phases in the microstructure, only relatively low ductility values can be achieved with this alloy and, therefore, AlSi9Cu3 finds no application in safety components.

Table 13 gives the chemical composition (in weight-%) of the NRC-AlSi9Cu3 alloy investigated in this work in comparison with the nominal composition of AlSi9Cu3 (A226).

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ni</th>
<th>Ti</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRC-AlSi9Cu3</td>
<td>8.2</td>
<td>2</td>
<td>0.8</td>
<td>0.2</td>
<td>0.2</td>
<td>0.97</td>
<td>0.07</td>
<td>0.03</td>
<td>0.11</td>
</tr>
<tr>
<td>A226</td>
<td>8-11</td>
<td>2-3.5</td>
<td>0.8</td>
<td>0.1-0.5</td>
<td>0.1-0.5</td>
<td>&lt;1.2</td>
<td>&lt;0.3</td>
<td>&lt;0.15</td>
<td>&lt;0.2</td>
</tr>
</tbody>
</table>
Figure 83 shows that the temperature range for the liquid fraction, $f_l$, between 0.4 and 0.6 (calculated by using the Scheil equation in ThermoCalc) is only 3°C for the secondary alloy NRC-AlSi9Cu3, which is an even smaller solidification range than for the wrought alloy NRC-AlMgSi1 (i.e. 5°C, see Chapter 7.1).

For the New Rheocasting process, the temperature distribution at the semi-solid forming temperature, $T_F$, showed for NRC-AlSi9Cu3 a temperature gradient of $\Delta T = 3$ °C, from the centre of the slurry to the crucible wall, for a melt volume of about $10^3$ cm$^3$. (Note: for AlSi7Mg0.3 $\Delta T = 3$ °C, and for AlMgSi1 $\Delta T = 1$ °C as mentioned in Chapter 4.3 and Chapter 7.1).

Figure 84 illustrates the enthalpy, $H$, as a function of the temperature, $T$, for the alloy AlSi9Cu3 with $\Delta H$ being the amount of heat which must be removed during cooling from the casting temperature, $T_G$, of 615°C, i.e. ca.13°C above liquidus temperature, to the semi-solid forming temperature, $T_F$, of 570°C. The enthalpy, $\Delta H$, which has to be discharged via the walls of the NRC crucible on its way on the cooling carousel within a temperature range, $\Delta T_{GF}$, of 45°C, is 6587 J/mol or 234 J/g, respectively. A temperature variation, $\Delta T$, of 3°C at 570°C, however, does not maintain the homogeneity of the solid/liquid fraction of the slurry over the cross-section in the requested range and, therefore, it
can be stated that reproducible part properties cannot be achieved. (Note: ThermoCalc yielded for the alloy AlSi9Cu3 for equilibrium calculation at 573°C, 570°C and 567°C li-
quid fractions, $f^l$, of 0.613, 0.523 and 0.305, respectively).

Figure 84: Enthalpy, $H$, as a function of temperature, $T$, for AlSi9Cu3.

Figure 85 illustrates the solid/liquid fraction, $f^s$ and $f^l$, as a function of the temperature, $T$, for NRC-AlSi9Cu3. Due to the Si content of 8.2 weight-%, the alloy exhibits a high por-
tion of eutectic phase and the volume content of primary $\alpha$-Al at the eutectic temperature is only about 0.35. Therefore, when adjusting the solid/liquid fraction between 0.4 and 0.6 for the alloy NRC-AlSi9Cu3 as required for successful semi-solid forming, in addition to $\alpha$-Al-phase also eutectic phase will be present. This presence of solid eutectic phase at semi-solid temperature leads to the formation of undesired coarse Si particles, $f^{Si}$, as well as intermetallic Fe containing phases, $f^{Fe}$, in the microstructure, thus leading to a deterio-
ration of the mechanical properties.

Figure 86 shows the microstructure of an NRC-AlSi9Cu3 precursor material fabricated at a casting temperature, $T_G$, of 615°C and quenched in water at 570°C.
8.1.1 ADAPTATION OF AlSi9Cu3 FOR THE NRC-PROCESS

In order to increase the volume content of primary $\alpha$-Al at the eutectic temperature, i.e. to avoid the formation of both coarse eutectic Si and intermetallic phases, an adaption of
the NRC-AlSi9Cu3 was performed by adding primary aluminium. Figure 87 illustrates the "recipe" for the adaption on the basis of a ThermoCalc analysis. It can be seen that the dilution with primary aluminium leads to a reduction of the Si content in two steps to 7 and 6 weight-%, respectively. The Cu content, however, was kept constant at 2 weight-% by adding the appropriate amount of this element, and the content of all other elements was reduced proportionally.

![Figure 87: Liquid/solid fraction, fₘ and f₁, as a function of temperature, T, for NRC-AlSi9Cu3 and the adapted alloys NRC-AlSi7Cu2 and NRC-AlSi6Cu2.](image)

In the case of the two adapted alloys NRC-AlSi7Cu2 and NRC-AlSi6Cu2, a measured temperature variation, ΔT, of 3°C at the forming temperature, Tₚ, (i.e. 572°C for NRC-AlSi7Cu2 and 583°C for NRC-AlSi6Cu2) enabled the obtainment of a homogeneous slurry over the cross-section keeping the solid/liquid fractions in the requested range of 0.4 to 0.6 for semi-solid processing. (Note: ThermoCalc for the alloy NRC-AlSi7Cu2 for equilibrium calculation at 575°C, 572°C and 569°C yielded liquid fractions, f₁, of 0.563,
0.539 and 0.431, respectively, and for the alloy NRC-AlSi6Cu2 for equilibrium calculation at 586°C, 583°C and 580°C, liquid fractions, \( f_1 \), of 0.532, 0.506 and 0.483, respectively).

8.2 Flow Behaviour In The Semi-Solid State

The rheological properties of the re-melted Al-Si-Cu alloys, originally cast at casting temperatures, \( T_G \), of 615°C (NRC-AlSi9Cu3), 620°C (NRC-AlSi7Cu2) and 630°C (NRC-AlSi6Cu2) were studied by means of backward extrusion as shown in Chapter 4.2.1. Figures 88 to 90 show the effect of the liquid fraction, \( f_1 \), on the flow characteristics for the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 as a function of temperature, T. The specimens were isothermally backwards extruded after an isothermal holding time of 3 minutes.

![Figure 88: Extrusion curves for NRC-AlSi9Cu3 at different liquid fractions (0\(<f_1<0.35).](image)

For all three alloy specimens with liquid fractions \( f_1 < 0.3 \), as shown in Figures 88 to 90, the typical hot forming flow characteristic is observed with a steep increase of the force...
at small displacements (quasi-elastic range), followed by deformation of the specimen before reaching a steady-state plateau. Therefore, no thixotropic behaviour is observed for liquid fractions $\tau^l<0.3$ for the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2. The microstructure of the deformed specimens (see Figure 37) for all three alloys after backward extrusion showed expelled liquid phase and a corresponding densified solid phase at the bottom of the container, as demonstrated in Figure 91.

Figure 89: Extrusion curves for NRC-AlSi7Cu2 at different liquid fractions ($0<\tau^l<0.45$).

Figure 90: Extrusion curves for NRC-AlSi6Cu2 at different liquid fractions ($0<\tau^l<0.45$).
At liquid fractions, $f^l$, of 0.3 and 0.35, on the other hand, the slugs fabricated with the alloy NRC-AlSi9Cu3 exhibited thixotropic properties. The low forming forces at higher liquid fractions ($f^l > 0.35$) could not be measured properly with the load cell in the experimental set-up used in these investigations, because of the alloy's high eutectic portion (due to the Si content of 8.2 weight-%). The volume content of primary $\alpha$-Al at the eutectic temperature is only about 0.35 and a slight increase of the temperature (of only 1-2°C) led to a steep rise of the liquid fraction, $f^l$, up to ca. 0.55, which gave imperceptible low forces for the utilised load cell. The slugs of the alloys NRC-AlSi7Cu2 and NRC-AlSi6Cu2, howe-
ver, exhibited thixotropic properties at liquid fractions, \( f^1 \), of 0.4 and 0.45, which is illustrated in Figure 92, where solid globules can be found from top to bottom in the microstructure within the extruded product, due to the almost homogeneous deformation.

Figure 92: Evolution of the microstructure within the extruded product for \( f^1 \) of 0.45.
8.3 MICROSTRUCTURE

Figure 93 illustrates the microstructure of all three types of alloy (i.e. NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 specimens taken from the step plate) in the T6 heat treated state (see Table 14). It is obvious that with a decreasing Si-content the fraction of eutectic phase (i.e. the dark phase in Figure 93) decreases. Furthermore, another beneficial feature of the dilution with primary aluminium is the reduction of the amount of coarse eutectic Si and undesired intermetallic phases such as the acicular and plate structured β-phase (Al$_5$Fe$_3$Si) and the polygonal or so-called Chinese-script α-phase (Al$_{15}$Fe$_3$Si$_2$). Although no quantitative analysis was performed, the observation of several micrographs clearly demonstrate the reduction of ductility degrading inclusions with decreasing Si-content in the alloy.

As shown in Table 14 the average grain size, $D_\alpha$, and shape factor, $F_\alpha$, of the primary α-Al-phase were similar for all three alloys. No significant differences could be evaluated for all the different wall thicknesses of the cast step plate.

<table>
<thead>
<tr>
<th></th>
<th>$F_\alpha$ [-]</th>
<th>$D_\alpha$ [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRC-AlSi9Cu3</td>
<td>1.48</td>
<td>65</td>
</tr>
<tr>
<td>NRC-AlSi7Cu2</td>
<td>1.41</td>
<td>75</td>
</tr>
<tr>
<td>NRC-AlSi6Cu2</td>
<td>1.51</td>
<td>92</td>
</tr>
</tbody>
</table>

Figure 93: Microstructure of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the T6-condition.
8.4 Mechanical Properties

The experimental procedure was the same as described in Chapter 6.2.1 for the alloy NRC-AlSi7Mg0.3. The casting temperature, \( T_G \), and the semi-solid forming temperature, \( T_F \), for the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2, however, were kept constant at 615°C, 620°C, 630°C and at 570°C, 572°C, 583°C, respectively. Table 15 gives an overview of the heat treatment parameters for these alloys.

<table>
<thead>
<tr>
<th>condition</th>
<th>heat treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>as cast</td>
</tr>
<tr>
<td>T4</td>
<td>solution annealed 520°C/2h + water quenched</td>
</tr>
<tr>
<td>T5</td>
<td>artificially aged 160°C/6h + water quenched</td>
</tr>
<tr>
<td>T6</td>
<td>T4 + artificially aged 160°C/6h + water quenched</td>
</tr>
</tbody>
</table>

8.4.1 Static Mechanical Properties

In Figures 94 to 97, the static mechanical properties of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the F-, T4-, T5- and T6-condition, respectively, are given as average values for the 14mm and 10mm step as well as for the 6mm and 2mm step of the step plate. All three types of alloys exhibited better mechanical properties with decreasing wall thickness, i.e. the best values were achieved in the 6mm and 2mm step, in all heat treatment conditions, which is in agreement with data published in [72]. One reason for this are the different cooling rates during solidification in the single steps due to their variations in wall thickness. Especially the growth of the intermetallic phases and the coarse Si phases in the eutectic phase is apparently suppressed, leading to the formation of a fine eutectic phase thanks to the high solidification rate, and therefore leading to higher elongation to fracture values. In addition it is important to note that within the four steps investigated the elongation increases with decreasing Si-content. This is due to the presence of higher amounts of ductile \( \alpha \)-Al-phase in the microstructure.
Figure 94: Mechanical properties of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the F-condition.

Figure 95: Mechanical properties of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the T4-condition.
AISi9Cu3/AISi7Cu2/AISi6Cu2 - T5

Figure 96: Mechanical properties of NRC-AISi9Cu3, NRC-AISi7Cu2 and NRC-AISi6Cu2 in the T5-condition.

AISi9Cu3/AISi7Cu2/AISi6Cu2 - T6

Figure 97: Mechanical properties of NRC-AISi9Cu3, NRC-AISi7Cu2 and NRC-AISi6Cu2 in the T6-condition.
Figure 98 shows the microstructure of NRC-AlSi9Cu3 in the 10mm step in comparison with the microstructure in the 2mm step in the T6-condition, which clearly indicate the reduction of the amount of coarse intermetallic phases in the thinnest step of the step plate.

Figure 98: Microstructure of NRC-AlSi9Cu3 of step 2 and step 4 of the step plate in the T6-condition.

A promising combination of strength and ductility values was obtained in the T4-condition in the two thinnest steps of the step plate (6mm and 2mm) with yield strengths of 210, 200 and 170MPa, ultimate tensile strengths of 310, 300 and 255MPa and elongation values of 8, 10 and 12% for NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2, respectively. In the F-, T5- and T6-condition, however, the obtained average mechanical data for the two thinnest steps of the three alloys were yield strengths of 151-178, 137-160 and 225-255MPa, ultimate tensile strengths of 243-268, 222-263 and 290-320MPa at elongation to fracture values of 4.3-5.6, 4.4-4.7 and 4.1-6.1%, respectively.

The average mechanical properties achieved in the different heat treatment conditions with the NRC process for the two thinnest steps of the step plate of the three investigated alloys are compared with published data (for cast components with a wall thickness between 2mm and 6mm) in Table 16. In terms of elongation to fracture the obtained values for the NRC-alloys exhibited a significant improvement versus the ductility properties of the die cast components. The elongation values increased with decreasing Si-content of the NRC-alloys thanks to the diminution of the amount of ductility degrading inclusions as mentioned in Chapter 8.3.
Table 16: Static mechanical properties of AlSi9Cu3 alloys.

<table>
<thead>
<tr>
<th></th>
<th>condition</th>
<th>YS [MPa]</th>
<th>UTS [MPa]</th>
<th>A [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NRC-AlSi9Cu3</td>
<td>F</td>
<td>151</td>
<td>243</td>
<td>4.3</td>
</tr>
<tr>
<td></td>
<td>T4</td>
<td>210</td>
<td>310</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>T5</td>
<td>157</td>
<td>263</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>255</td>
<td>320</td>
<td>4.1</td>
</tr>
<tr>
<td>NRC-AlSi7Cu2</td>
<td>F</td>
<td>178</td>
<td>268</td>
<td>5.1</td>
</tr>
<tr>
<td></td>
<td>T4</td>
<td>200</td>
<td>300</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>T5</td>
<td>160</td>
<td>239</td>
<td>4.4</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>250</td>
<td>318</td>
<td>5.9</td>
</tr>
<tr>
<td>NRC-AlSi6Cu2</td>
<td>F</td>
<td>163</td>
<td>248</td>
<td>5.6</td>
</tr>
<tr>
<td></td>
<td>T4</td>
<td>170</td>
<td>255</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>T5</td>
<td>137</td>
<td>222</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>225</td>
<td>290</td>
<td>6.1</td>
</tr>
<tr>
<td>Heusler [71] - HP die</td>
<td>F</td>
<td>158</td>
<td>265</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>-</td>
<td>338</td>
<td>4.2-7.5</td>
</tr>
<tr>
<td>Schindelbacher [72] - HP die</td>
<td>F</td>
<td>140</td>
<td>240</td>
<td>1.6</td>
</tr>
<tr>
<td>Schneider [73,74,75,76] - HP die</td>
<td>F</td>
<td>190</td>
<td>320</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>T4</td>
<td>180</td>
<td>320</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>T5</td>
<td>200</td>
<td>300</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>330</td>
<td>410</td>
<td>3.8</td>
</tr>
<tr>
<td>Wang [77] - HP die</td>
<td>F</td>
<td>141-221</td>
<td>309-346</td>
<td>2.5-6.5</td>
</tr>
<tr>
<td>Samuel [78] - chill</td>
<td>F</td>
<td>175</td>
<td>220</td>
<td>1</td>
</tr>
<tr>
<td>Parton [79] - HP die</td>
<td>F</td>
<td>-</td>
<td>235</td>
<td></td>
</tr>
<tr>
<td></td>
<td>T5</td>
<td>240</td>
<td>262</td>
<td></td>
</tr>
<tr>
<td>Henning [80] - squeeze</td>
<td>F</td>
<td>-</td>
<td>250</td>
<td>1.5</td>
</tr>
<tr>
<td></td>
<td>T4</td>
<td>-</td>
<td>310</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>T6</td>
<td>-</td>
<td>300</td>
<td>2.5</td>
</tr>
</tbody>
</table>
8.4.2 Dynamic Mechanical Properties

Figure 99 shows the results of the fatigue tests for the two thickest steps (14mm and 10mm) of the step plate for the investigated alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the T6-condition. The achieved fatigue strength values were a maximum stress, $\sigma_{\text{max}}$, of 115, 125 and 125 MPa, respectively. The dilution of NRC-AlSi9Cu3 with primary aluminium clearly led to a reduction of the amount of coarse eutectic Si particles and intermetallic phases which can act as fatigue crack initiation sites which in turn increased the fatigue limit for both alloys, NRC-AlSi7Cu2 and NRC-AlSi6Cu2.

![Figure 99: Fatigue strength of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the T6-condition. R=0.1.](image)

8.4.3 Wear Properties

One of the pressure die casting applications of the alloy AlSi9Cu3 is in the fabrication of pumps, where a certain amount of wear resistance is demanded. The alloy's adequate wear resistance properties are mainly confined to the relatively high amount of Si and, fur-
thermore, to the amount of intermetallic phases in the microstructure. To investigate the
effect of the decreasing Si-content in the adapted alloys NRC-AlSi7Cu2 and NRC-
AlSi6Cu2 on the wear properties, the alloys were subjected to a dry sand/rubber wheel
abrasion test [81]. A schematic diagram of the test apparatus is given in Figure 100.

The dry sand/rubber wheel abrasion test involved the abrading of the NRC-AlSi9Cu3-,
NRC-AlSi7Cu2- and NRC-AlSi6Cu2-specimens (l=25mm, w=24.9mm, h=5mm) in the
F- and T6-condition with sand. The SiO₂-sand with an average grain size of 0.1-0.3mm
was introduced between the specimen and the rotating rubber wheel (Ø=50mm), whereas
the specimen was pressed against the rotating wheel at a specified force by means of a
lever arm (applied weight: 860g). The rotation of the wheel was such that its contact face
moved in direction of the sand flow (sand flow rate: 300g/min). The test duration was de-
termined by the number of wheel revolutions, i.e. by reaching the distance of exactly 1km.
Figure 101 illustrates the results of the abrasive wear tests for the alloys NRC-AlSi9Cu3,
NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the F- and T6-condition.

In both heat treatment conditions the abrasion slightly increased with decreasing Si-con-
tent. In the as-cast condition, F, the alloys showed a higher wear resistance than in the T6-
condition. The least abrasion was measured for the alloy NRC-AlSi9Cu3 in the F-condi-
tion with 15.864±0.315mm³, whereas the highest abrasion rate was found for the alloy NRC-AlSi6Cu2 in the T6-condition with 18.023±0.115mm³. However, this slightly reduced abrasion resistance is assumed not to be relevant with respect to an application of the alloys NRC-AlSi7Cu2 and NRC-AlSi6Cu2 as pump material.

![Bar graph showing abrasion rates for NRC-AlSi9Cu3, NRC-AlSi7Cu2, and NRC-AlSi6Cu2 in F and T6 conditions.](image)

Figure 101: Abrasion of NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the F- and T6-condition.

### 8.5 Corrosion Properties

#### 8.5.1 Causes and Forms of Corrosion

Aluminium owes its excellent corrosion resistance to the barrier oxide film strongly bound to its surface. The spontaneous passive film formed in air at ambient temperature is only about 5nm in thickness, and when damaged, it re-forms immediately in most environments and continues to protect the aluminium from corrosion.

Uniform or general corrosion of aluminium is rare, except under highly acidic or alkaline conditions such as phosphoric acid or sodium hydroxide [82]. However, in environments
where the surface film is insoluble, localised corrosion can take place at weak spots on the oxide film in the presence of specific aggressive ions, such as for example pitting corrosion in the presence of chloride ions. Other forms of localised corrosion that can occur are crevice, filiform, galvanic, intergranular, exfoliation or biological corrosion.

Localised corrosion can be caused by differences in corrosion potential on the metal surface due to for example the presence of cathodic microconstituents such as insoluble intermetallic compounds or single elements. The most common in aluminium alloys are CuAl2, FeAl3 and silicon [83]. The solution-potential (or corrosion-potential) of an aluminium alloy is primarily determined by the composition of the aluminium-rich solid solution, which constitutes the predominant volume fraction and area fraction of the alloy microstructure [84]. The effects of the principal alloying elements on the corrosion potential of high-purity aluminium are shown in Figure 102. The effects of multiple elements in solid solution on the corrosion potential are approximately additive.

![Figure 102: Effect on principal alloying elements on electrolytic solution potential of aluminium. Potentials are for high purity binary alloys solution heat treated and quenched. Measured in a solution of 53g/l NaCl + 3g/l H2O2 at 25°C [83].](image-url)
In the case of the investigated alloys of the type NRC-Al-Si-Cu, silicon as an alloying element is cathodic to the aluminium solid-solution matrix and accounts for a considerable volume fraction. However, the effects of silicon on the corrosion resistance of these alloys are minimal because of low corrosion current density resulting from the fact that the silicon particles are highly polarised [83]. The Fe- and Mn-containing intermetallic α- and β-phases have solution potentials of almost the same level as that of the solid-solution matrix [85] and therefore, these constituents are not significant sites for corrosion initiation.

An increasing copper content, on the other hand, leads to a decrease in general corrosion resistance which is attributable to galvanic cells created by formation of minute copper particles or films deposited on the alloy surface as a result of corrosion. As corrosion progresses, copper ions, which initially go into solution, replate onto the alloy to form metallic copper cathodes. Reduction of copper ions and increased efficiency of O₂ and H⁺ reduction reactions in the presence of copper lead to an increase of the corrosion rate [83].

In order to investigate the corrosion properties of the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the different heat treatment conditions F, T4, T5 and T6, respectively, immersion tests and electrochemical experiments such as potentiodynamic polarisation measurements and electrochemical impedance spectroscopy (EIS) were carried out.

8.5.2 IMMERSION TESTS

The specimens of the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2 and NRC-AlSi6Cu2 in the F-, T4-, T5- and T6-condition were hand polished with different grades of emery paper (down to grade 500), cleaned with isopropanol in an ultrasonic bath, and finally rinsed with ethanol. NRC-AlSi7Mg0.3 in the F-condition was also used in the investigation as a reference. All specimens were immersed in a 5% NaCl-solution at a temperature of 35°C. After 2h, 5h, 24h, 168h (one week) and 504h (three weeks) the specimens were taken out of the immersion bath and weighed after cleaning in an ultrasonic bath containing 65% HNO₃ [86].
Figure 103 shows the mass loss/unit area of the different specimens after an immersion time of three weeks.

In first instance it can be observed that the mass loss is highest for all three NRC-Al-Si-Cu alloys in the T5- and F-condition, whereas the alloys in the solution annealed conditions (T4 and T6) exhibit a reduced mass loss rate. This clearly indicates that the heat treatment and, therefore, the different resulting microstructures play an important role in determining the corrosion rate. In fact the T4- and T6-conditions yield a more uniform microstructure in which the alloying elements are more evenly distributed. In addition it is important to note that the Si-content within the different heat treatment conditions also plays a role in the mass loss rate. It would appear that in the F- and T5-condition an increasing Si-content leads to an improved corrosion resistance. The reference alloy NRC-AlSi7Mg0.3 in the F-condition showed the best corrosion resistance in the immersion tests, despite the mass loss rate increased after approximately 200h, reaching values simi-
lar to the NRC-alloys in the T4- and T6-condition with $10^{-3}\text{g/cm}^2$ mass loss after three weeks.

8.5.3 POTENTIODYNAMIC POLARISATION MEASUREMENTS

Although under conditions of passivity, aluminium does not undergo uniform corrosion, localised corrosion in the form of pitting corrosion may still occur, particularly in the presence of aggressive ions such as Cl⁻ ions. The pitting-potential, $E_p$, is defined as the potential above which pitting will initiate on the surface for specific solutions and temperature conditions.

The specimens for the potentiodynamic polarisation measurements were hand polished down to 1µm. The measurements were performed at 25°C in a 0.1M Na₂SO₄ and in a 0.1M Na₂SO₄ + 0.001M NaCl solution. A standard electrochemical three electrode set-up was employed, with a Pt gauze as secondary electrode and a Saturated Calomel Electrode (SCE) as reference electrode. The working electrode was polarised starting at -1V vs. SCE and scanning anodically to 1.5V vs. SCE. The current-potential curves were acquired via the use of a Zahner IM5d potentiostat. The scan rate employed in all experiments was 1mV/s. Figure 104 shows the results of the potentiodynamic polarisation measurements for the alloys NRC-AlSi₉Cu₃, NRC-AlSi₇Cu₂, NRC-AlSi₆Cu₂ and NRC-AlSi₇Mg0.3 in the F-condition in a 0.1M Na₂SO₄ + 0.001M NaCl solution.

From the curves it can be seen that there is quite a small variation of the corrosion properties between the different alloys in the F-condition. The open circuit potential for the reference alloy is the most cathodic at -0.5V vs. SCE, whilst all other alloys are in close range around -0.3V vs. SCE. In the anodic direction it can be seen that pitting corrosion takes place on all the alloys, except for NRC-AlSi₆Cu₂, in the order NRC-AlSi₉Cu₃ with $E_p=0.6V$, NRC-AlSi₇Mg0.3 with $E_p=1.2V$ and NRC-AlSi₇Cu₂ with $E_p=1.4V$. It is interesting to note that the reference alloy has the highest passive current throughout the anodic range. In contrast NRC-AlSi₆Cu₂ has low passive currents and does not pit under the potential range employed.
Figure 104: Potentiodynamic polarisation curves of NRC-AlSi9Cu3, NRC-AlSi7Cu2, NRC-AlSi6Cu2 and NRC-AlSi7Mg0.3 in the F-condition in a 0.1M Na2SO4 + 0.001M NaCl solution.

Figure 105 illustrates the pitting potentials of the NRC-Al-Si-Cu alloys in all heat treatment conditions for both investigated solutions. A potential of 1.6V vs. SCE signifies that "no pitting" events were recorded in the investigated potential range.

Surprisingly, the specimens in the T4- and T6-condition showed improved corrosion resistance in the presence of added 0.001M NaCl than in the pure 0.1M Na2SO4 solution. This behaviour could be explained by the adsorption of Cl⁻ ions on the surface of the specimen, which can occur at undercritical concentrations of Cl⁻. Therefore, the electrochemically active surface becomes partly blocked, thus protecting the surface and preventing pitting corrosion from occurring. Nonetheless, it is interesting to note that only in the T4- and T6-condition the alloys undergo pitting corrosion in Na2SO4. These two heat treatments have similar effects on the microstructure of the alloys, but a clear explanation is...
here not possible. In the T5- and F-condition, however, the addition of 0.001M NaCl led to lower pitting corrosion potentials. In particular, the pitting potentials decreased with increasing Si-content. This could be explained by the fact that an increase in Si-content in the F- and T5-condition gives rise to a larger number of possible pitting corrosion initiation sites on the metal surface. These conditions, in fact, exhibit a more heterogeneous microstructure. In contrast, in the T4- and T6-heat treatment condition, an increase in Si-content improves the resistance to pitting corrosion because the latter is evenly dissolved.

For most practical situations, values of pitting potential >1V are not dangerous, since pitting corrosion in this case would only take place in the presence of strong oxidising species in the solution. Hence, only the alloy NRC-AlSi9Cu3 in the F-condition shows a
pitting potential which could potentially lead to pitting corrosion without strong oxidisers in the solution under open-circuit conditions (oxygen-containing aqueous solutions could suffice to polarise the alloy to values above its pitting potential). Nevertheless, it must be taken into consideration that the values measured are only valid for these experimental conditions. For instance, an increase of the Cl⁻ content or temperature would be expected to decrease the pitting potential.

8.5.4 ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

Electrochemical impedance spectroscopy (EIS) measurements were recorded in the frequency range of $10^{-2}$ Hz to $10^5$ Hz for all specimens in the same two solutions as described above. The spectra were acquired via the use of a Zahner IM5d potentiostat and the applied potential was the open circuit potential $\pm 10^{-2} V$.

Impedance spectra give information on the metal/electrolyte interface. In general, at low frequencies the impedance, $Z$, approaches the polarisation resistance, $R_p$, where $R_p$ is proportional to the inverse of the corrosion current, $i_{corr}$, (i.e., the higher $R_p$ or $Z$ at low frequencies, the lower the corrosion rate).

Figure 106 shows the results of the impedance measurements of the alloys NRC-AlSi9Cu3, NRC-AlSi7Cu2, NRC-AlSi6Cu2 and NRC-AlSi7Mg0.3 in the F-condition in a 0.1M Na$_2$SO$_4$ + 0.001M NaCl solution.

It can be seen that the alloys display quite different spectra. In particular, NRC-AlSi9Cu3 and NRC-AlSi7Cu2 appear to have very similar phase shift and impedance behaviour. The phase shift response clearly shows two time constants. This may indicate the presence of a heterogeneous surface, where the different surface sites show a strongly different corrosion behaviour. NRC-AlSi6Cu2 and NRC-AlSi7Mg0.3, in contrast, have a different phase shift behaviour and much higher impedance values. Once again the alloy with the lowest Si-content, NRC-AlSi6Cu2, showed the best corrosion resistance in the F-condition.
Figure 106: Impedance measurements of NRC-AlSi9Cu3, NRC-AlSi7Cu2, NRC-AlSi6Cu2 and NRC-AlSi7Mg0.3 in the F-condition in a 0.1M Na₂SO₄ + 0.001M NaCl solution.

Figure 107 gives an overview over the impedances of the NRC-Al-Si-Cu alloys in all heat treatment conditions for both types of solutions at a frequency of $10^{-2}$Hz. This value was taken as an indication of the corrosion resistance of the alloys.

Overall, all impedance values measured are in the range of $10^6\Omega$, which signifies that at the potential investigated all the alloys are passive (for actively corroding metals and alloys values in the range of $10^3\Omega$ or less would be typically expected). The values shown in Figure 107 clearly indicate that both alloy composition and microstructure play an important role in determining the corrosion behaviour. For example for NRC-AlSi9Cu3, a heat treatment seems to improve the corrosion resistance in the order T4, T6 and T5 compared to the as-cast F-condition. However, a direct correlation of the effect played by both these factors is difficult, since the nature of the attack on the surface is not known. In fact,
Impedance values measured only represent an average value over the whole surface, which does not describe for example a non-uniform attack of the surface.

Figure 107: Impedances at 0.01Hz of NRC-AlSi9Cu3, NRC-AlSi7Cu2, NRC-AlSi6Cu2 in the F-, T4-, T5- and T6-condition and of NRC-AlSi7Mg0.3 in the F-condition in 0.1M Na2SO4 +/- 0.001M NaCl solution.

The alloy NRC-AlSi9Cu3 proved to be not "processable" with the standard New Rheocasting equipment, while the adapted versions NRC-AlSi7Cu2 and NRC-AlSi6Cu2 proved nicely "processable". The adapted alloys exhibited mechanical properties clearly exceeding the properties achieved with high pressure die casting. The reduced Si-content of the adapted alloys did not affect the wear resistance of the alloy. Thanks to the fact that AlSi9Cu3 is a secondary alloy and therefore inexpensive and part of the aluminium recycling system, the increased use of this alloy entails ecological and economical advantages.
CONCLUSIONS AND OUTLOOK

In the work presented, the mechanism behind the development of the globular microstructure, which is the essential aspect of semi-solid processing, was explained. Based on the fact that the essence of the “New Rheocasting” recipe is the casting of a relatively “cold“ melt along the wall of a crucible, in a similar fashion to pouring “Weissbier“ into a glass, the wall mechanisms for nucleation produce enough nuclei to impede dendritic growth and to exhibit a globular microstructure. The stability of the process under consideration of industrial conditions was found to be very high in terms of variations of the casting temperature (up to more than 30°C above liquidus temperature) as well as in terms of longer holding periods in the semi-solid state.

The New Rheocasting process provides a so far unknown independence from metal suppliers in semi-solid forming - because the melt is prepared in the foundry's own melt shop from conventional ingots and then transferred to the holding furnace at the casting machine - and a significant cost advantage compared with the Thixocasting process. In contrast to the conventional Thixocasting process there is no metal loss in this operation, and after casting, the runners and scrap are returned to the melt shop for in-house recycling. It is predicted that, mainly due to the lower material cost, an overall cost saving of up to 20% compared to conventional Thixocasting for aluminium alloys can be reached. The cost saving can be further increased via the use of recycled material (secondary alloys) in New Rheocasting.

The alloys investigated in this work were AlSi7Mg0.3, AlSi7Mg0.6, AlMgSi1 as well as their modifications with Ba, and AlSi9Cu3, representing different categories of aluminium alloys and, therefore, different fields of applications.
The alloys NRC-AlSi7Mg0.3 and NRC-AlSi7Mg0.6 proved to exhibit excellent mechanical properties. The strength and ductility levels as well as the fatigue properties in all heat treatment conditions achieved with the New Rheocasting process - representing standard values for this process - can be reached in the conventional Thixocasting process only under very favourable process conditions.

The alloy NRC-AlMgSi1 proved to be "processable" with the New Rheocasting process. Due to the alloy's tendency towards "hot cracks" in thin-walled sections, applications for this alloy are restricted to more thick-walled parts. The level of ductility for this alloy in the wrought or forged condition could not be reached, because the cast microstructure solidified in non-equilibrium, promoting the appearance of non-equilibrium eutectic phase and, therefore, reducing the elongation values.

The alloy NRC-AlSi9Cu3 proved to be not "processable" with the standard New Rheocasting equipment, while the adapted versions NRC-AlSi7Cu2 and NRC-AlSi6Cu2 proved nicely "processable". NRC-AlSi9Cu3 has a very narrow solidification range between \( T_f \) from 0.4 to 0.6, which makes it nearly impossible to fabricate parts with reproducible properties in a rough foundry environment. Therefore, compositional changes were required to stabilise the process through alloy adaptation. These adapted alloys exhibited me-
chanical properties clearly exceeding the properties achieved with high pressure die casting. The reduced Si-content of the adapted alloys did not affect the wear resistance of the alloy. Thanks to the fact that AlSi9Cu3 is a secondary alloy and therefore inexpensive and part of the aluminium recycling system, the increased use of this alloy entails ecological and economical advantages.

Thanks to the New Rheocasting process, in the near future the variety of alloys applicable for semi-solid processing should be enlarged significantly because of the uncomplicated production of its precursor material. Beside metal matrix composites (MMC's) and the family of high strength aluminium foundry alloys, magnesium based alloys above all will be the subject of intensive research activities.

The New Rheocasting process was practically unknown at the start of this work in 1999, but at this point in time more than 10 New Rheocasting machines have already been sold worldwide. The authors of [87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98] partly see that as a visible expression of their successful research and cooperation.
10

LITERATURE


CURRICULUM VITAE

PERSONAL DATA

Name: WABUSSEG, Heimo
Birth date: 25th July 1972
Birth place: Wagna, Austria
Nationality: Austrian

EDUCATION

1978 - 1982 Volksschule Leibnitz, Austria
1982 - 1990 Gymnasium Leibnitz, Austria
1991 - 1998 Montanuniversität Leoben, Austria, Studies of Metallurgy
1998 Graduation to “Dipl.-Ing. mont."
1999 - 2002 Federal Institute of Technology, Zurich, Institut of Metallurgy, PhD student under supervision of Prof. Dr. Peter J. Uggowitzer

WORK EXPERIENCE

1995 - 1997 Wolfram Bergbau- und Hütten-GmbH, Bergla, Austria; practical studies
1998 Treibacher Industrie AG, Althofen, Austria; R&D
1998 – 1999 Manganese Metal Company, Nelspruit, South Africa; R&D

LIST OF PUBLICATIONS


