Doctoral Thesis

Processing of aluminum alloys in the semi-solid state
basic principles and constitutive models

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Processing of Aluminum Alloys in the Semi-Solid State
Basic Principles and Constitutive Models

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presented by
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"In most of mankind gratitude is merely a secret hope for greater favors."

Duc de la Rochefoucauld

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"Don't fear failure so much that you refuse to try new things. The saddest summary of life contains three descriptions: could have, might have, and should have."

Louis E. Boone

Summary

Thixoforming, an innovative technique in which metals are formed in their semi-solid state, permits the cost-efficient production of components with improved properties and performance compared to conventional pressure die casting. First developed in the USA based on a discovery made in the early 1970s at the Massachusetts Institute of Technology, thixoforming is now practised on a worldwide scale. Since this process is still relatively new, great technical advances can be expected in the near future. These advances should allow production of near-net-shape components with good mechanical properties, combined with low manufacturing cost. Due to the distinct thixotropic flow pattern of the semi-solid slurry, high production speeds similar to those of conventional pressure die casting can be achieved, yet the disadvantages of the latter are eliminated: thixoformed components possess a good combination of strength and ductility, and they are heat-treatable and weldable.

Thixotropy is a property of some materials of becoming fluid when stirred or sheared, and setting solid again when left standing. The requirement for thixotropic flow behavior is a particular microstructure in the semi-solid state. At the onset of forming, the solid phase of the material must exhibit a globular morphology, a feature that can be produced with rigorous stirring during solidification. The first part of the thesis deals with the production of such stir-cast feedstock material using a prototype caster with a mechanical stirring device. Cast billets of AlSi7 and resulting microstructures show the feasibility of the chosen process variant, and a mass and heat transfer model facilitates the optimal settings of the process parameters during the casting runs.

In the second part of the thesis, the governing constitutive models for the distinct flow behavior of thixotropic aluminum alloys in the semi-solid state are derived with respect to temperature, strain rate and microstructure. Experimental techniques such as deformation tests, backward extrusion experiments, differential thermal analysis and quantitative metallurgy provide the necessary data to calibrate the materials models. For the implementation in a special-purpose FE software package, constitutive models for the thixotropic flow stress, the specific heat capacity and the thermal conductivity of cast and wrought aluminum alloys in the semi-solid state are provided.

The objective of the third part of this thesis is to implement these models in the FE software package ThixoForm for the optimization of the thixoforming manufacturing process. The constitutive flow model as well as the knowledge of related characteristic materials properties such as the agglomeration ratio of the solid phase in the semi-solid metal, the amount of fraction liquid and other thermal variables enable the materials scientist to optimize the thixoforming process significantly. The simulation results are compared with industrial experiments and used for process control with the objectives.
of high-quality parts and maximum productivity. They prove that the software package *ThixoForm* has promise as a tool in the thixoforming industry.

Because of insufficient fundamental knowledge, the focused research on specific topics and the lack of process control stability of this complex forming process, there exists a demand for a holistic research approach. The aim of this thesis is to fill this gap: it should be become possible to control the whole thixoforming process chain with models and simulations in order to *virtually* process thixoformed components from the stir-cast billet to the formed component.
Zusammenfassung


Das thixotrope Fließverhalten des halbflüssigen Metalls ermöglicht hohe Fertigungsgeschwindigkeiten wie beim Druckgussverfahren, die produzierten Teile verfügen aber nicht über die bekannten Nachteile von gegossenen Bauteilen: Thixogeformte Bauteile sind gekennzeichnet durch eine gute Kombination von Festigkeit und Zähigkeit, sie können wärmebehandelt werden, sind schweißbar und darüber hinaus relativ kostengünstig.


Für das eigentliche thixotrope Umformen im halbflüssigen Zustand werden im zweiten Teil der Arbeit die für eine Beschreibung des Fließverhaltens notwendigen Werkstoffmodelle entwickelt, unter Berücksichtigung der Einflüsse von Temperatur, Umformgeschwindigkeit und Gefügezustand. Experimentelle Untersuchungen wie Stauchversuche am Umformdilatometer, Rückwärts-Fließpressversuche, Differential-Thermoanalysen und quantitative Metallographie liefern die notwendigen Messdaten und Informationen für die Kalibrierung dieser Werkstoffmodelle. Für eine nachträgliche Implementierung in ein
Finite-Element-Softwarepaket werden Modelle bereitgestellt, welche sowohl die Fließspannung im halbflüssigen Zustand, als auch thermische Eigenschaften wie die spezifische Wärmekapazität und die Wärmeleitfähigkeit von verschiedenen Guss- und Knetlegierungen aus Aluminium beschreiben.

Der dritte Teil der Arbeit beschreibt die Implementierung dieser Werkstoffmodelle in das FE-Softwarepaket ThixoForm, mit dem Ziel vor Augen, beliebige Thixoforming-Prozesse simulieren zu können. Die Simulationsergebnisse werden mit experimentellen Versuchen im Industriemassstab verglichen, um die Übereinstimmung zu überprüfen. Die Simulation dient als Prozesskontrolle, um mit maximaler Produktivität hochwertige Produkte herzustellen. Die Simulationsergebnisse zeigen auf, dass das Softwarepaket ThixoForm als nützliches Werkzeug in der Thixoforming-Industrie eingesetzt werden kann.

Wegen dem immer noch lückenhaften Basisswissen und der häufig allzu fokussierten Ausrichtung der Forschung auf ein gewisses Spezialgebiet besteht dringender Bedarf an einer ganzheitlichen Betrachtungsweise dieser Thematik. Das Ziel dieser Arbeit ist es, diese Lücke zu schließen: Es soll mit Hilfe der vorgestellten Werkstoffmodelle und Grundprinzipien in Zukunft möglich sein, die gesamte Thixoforming-Prozesskette zu beherrschen dank einer virtuellen Betrachtungsweise der Herstellung thixogener Bauteile, beginnend mit der Modellierung der Vormaterialherstellung bis hin zur Simulation der Bauteilherstellung.
Introduction

One goal of innovative manufacturing technologies is the development and production of new materials with improved properties and performance. To compete with existing technologies, such methods of production should also become increasingly less expensive. A relatively new class of innovative forming technology that fulfills these qualities is thixoforming, a technique in which metals are formed in their semi-solid state. Based on a discovery made by a Ph. D. student at the Massachusetts Institute of Technology (MIT) in the early 1970s [1], the USA led in the development of thixoforming manufacturing techniques [2]. Nowadays, the efforts in thixoforming are made on a worldwide scale; several end users now consider innovative thixoforming as a competitive process variant which contributes both to manufacturing efficiencies and component quality. Because thixoforming is still at the beginning of its development, great technical advances can be expected in the near future compared to conventional techniques; for example, the industrial development of other alloy compositions in the domain of aluminum and magnesium alloys, bridging the gap to higher melting alloys such as copper and iron alloys, the extension of the thixoforming technology to larger parts and components, and a further improvement of profitability and operating efficiency.

The expression “thixo” stands for thixotropy, a term that derives from the Greek words θίξις—contact, touch, and τροπή—change, modification. Hence, thixotropy describes the property of some materials of becoming fluid when stirred or shaken, and setting solid again when allowed to stand. This reduction in viscosity is due to a temporary breaking down of an internal structure of a system under shear. The viscosity of thixotropic systems depends on the shear history, i.e. the extent of previous mechanical agitation to which the material has been subjected.

The basic principle of thixoforming is the forming of components within the solidus-liquidus interval of the investigated alloy system. In this interval, one part of the material is already liquid, whereas other parts are still fully solid. To exhibit thixotropic behavior, the solid phase must consist of globular particles that are embedded in the liquid phase. This particular microstructure can be accomplished with rigorous mechanical or electromagnetic stirring during the continuous casting of the billets.

Prior to forming in the semi-solid state, the cooled-down billets are sectioned to the desired dimensions and then heated up rapidly, using inductive heating devices. Depending on the chosen process variant, the temperature of the workpiece is adjusted corresponding to a fraction liquid between 30 and 60%. When the desired temperature is reached, the workpiece is soaked for several minutes in order to ensure a homogeneous temperature distribution. During this soaking period also the microstructure is modified; the solid particles will grow and coalescence procedures take place. Depending on the soaking time
the solid particles build a more or less coherent skeleton resulting in a flow behavior that is a strong variable of the strength of the skeleton. This strength depends strongly on the agglomeration ratio of the globular solid particles.

Interestingly enough, the slug keeps its shape during the soaking period, although about half of the material is liquid. This shape stability is even robust enough to permit a transfer of the workpiece into a forming tool. Only when shear forces are applied to the slug does the solid behavior change to a viscous suspension flow. This structural viscosity, or pseudo-plasticity, is the main principle of the thixoforming process; when sheared, the robust skeleton of solid particles is disrupted and the now isolated globular primary particles float in the viscous melt, resulting in much lower viscosities than the bulk solid skeleton [3]. When rested after shearing, the inter-particle network builds up again, forming a skeleton resulting in "solid" state behavior of the thixoformed component. In other words, during forming (e.g. the filling of the cavity of a pressure-die casting) a remarkable liquefying of the slug can be observed. Under the high shear rates of the filling process, the viscosity of the alloy drops several orders of magnitude and flows almost like a fully liquid melt into the cavity. Because of the thixotropic properties of the suspension, the component can be ejected from the casting machine after a very short solidification time, i.e. during the rebuilding of the solid skeleton. Figure 1 shows a general view of the whole thixoforming process, consisting of the production of the feedstock material, the reheating of sectioned specimens and the actual thixoforming. As we will see, the modeling and simulation of the whole thixoforming process is mandatory if we want to control the forming of metals in their semi-solid state.

From a manufacturing point of view, the main advantage of the thixoforming process lies in the fact that it allows a substantial amount of the solidification process to occur outside the mold or cavity. Not only does this reduce defects related to solidification within the components, but since the semi-solid formed components show this particular structure buildup at rest, they can be handled much like solids in the absence of shear.
forces. For these reasons, the thixoforming process is, functionally speaking, much more like plastic injection molding than liquid metal die casting.

Since the material entering the thixoforming process has only a fraction liquid of 30 to 60%, volumetric contraction is substantially reduced. This reduced solidification permits near-net-shape forming of alloys that were usually restricted to conventional hot forming processing. When thixoformed, wrought aluminum alloys such as AA6082 provide comparable performance to the extruded and machined versions.

Moreover, viscous flow behavior during thixoforming has a substantial impact on the quality of thixoformed components in contrast to the liquid fill pattern of a conventional casting process. The structure buildup at rest of thixoformed parts and its resistance to sudden shear rate change (i.e. thixotropy) allows handling actions while still retaining a solid-like viscosity. Only when the material is continuously sheared does thinning occur, which allows the slurry to enter the cavity. Not only does the almost smooth filling process eliminate or at least reduce the gas entrapment common to many casting processes but, since the material flow is predictable, the filling mode is laminar and not turbulent. Not only is solidification shrinkage reduced, but also gas entrapment is effectively eliminated such that thixoformed parts can be manufactured in a fully heat treated condition. Similar shaped conventional cast parts cannot be solution heat treated because of surface blistering resulting from expansion of entrapped gas at the solutionizing temperature.

![Figure 2: Positioning the thixoforming process among conventional forming techniques with regard to invested manufacturing costs and resulting mechanical properties](image-url)
In recent years, it has become increasingly important to take into account the reduction in environmental impact effected by thixoforming; compared to conventional casting, possible energy savings approach 35% [2]. These savings result from the fact that the slugs are only partially melted without superheat. Furthermore, thixoforming involves no liquid metal handling and there is reduced heat loss associated with liquid metal furnaces. This, together with the other attributes mentioned above, is another reason that thixoforming plays an important role in manufacturing automation [4].

The thixoforming process is therefore very well suited for large series of light but high-quality components: there are, for example, applications in the automotive industry where the high demands concerning the mechanical properties and the effective production cost are very well suited for the thixoforming process. But thixoforming must not be considered as competition for conventional hot forging or pressure die casting, but as a new possibility to achieve more than adequate mechanical properties with less than comparable production costs (see Figure 2). Therefore, the cost-efficient production of near-net-shape manufactured parts through thixoforming possesses a significant potential, not only because of very good mechanical properties and low production cost but also because of the development of completely new geometries of formed parts that were formerly impossible or difficult to achieve.
"A technique succeeds not by a clever trick or a happy accident, but because it expresses some aspect of a physical truth."

O.G. Sutton

Rheocasting Techniques

A key problem in the manufacturing of thixoformed parts is the feedstock material. In order to "thixoform" a part, a well defined input material has to be produced. It has a fundamental and thus decisive influence on the quality of thixoformed components. The quality, i.e. the microstructure, of the feedstock material determines the flow characteristics of the suspension in the semi-solid range during subsequent thixoforming. To guarantee sufficient flow properties, the dendritic structure of conventionally cast metals must be replaced with a globular fine-grained structure that can be obtained by rigorous stirring during the casting process. This stir-casting is often referred to as rheocasting because it has its origin in the field of rheological investigations of metal melts. The structure transformation that takes place during stirring reduces the entanglement between the solid particles when formed and is, as mentioned before, indispensable for pseudo-plastic and thixotropic behavior in the semi-solid temperature range.

The first step in the process chain thixoforming, the production of feedstock material with a globular microstructure, can be achieved using different kinds of processing techniques: Almost all investigated techniques cause the formation of the globular microstructure by applying shear forces on the solidification front. Although often referred to as dendrite fragmentation it can be shown (see Chapter 2.3) that the globular microstructure does not develop by fragmentation of dendrite arms, but that the globular structure originates isotropically under the influence of a stirring device. Molenaar et al. observed [5] that the conditions for a dendritic growth of solid particles are not met in agitated melts. The model for the prediction of the evolving primary particle size during casting in Chapter 2.4 thus was established based on that assumption.

One of the earliest procedures for the production of rheocast billets is stirring with a mechanical agitator (see Figure 1.1) that originates from the viscosimeters used in the field of rheology for the evaluation of rheological properties of liquid matter. The agitation is realised by a cylindrical or conical stirrer positioned in the freezing range of a continuous casting machine. Detrimental to this technique is contact of the stirrer
with the melt, the necessary preheating of the stirrer and the all-over thermal load of the whole stirring device. But contamination of the stirrer with the melt can be reduced using graphite as material. Advantageous to mechanical rheocasting is the very homogeneous microstructure over the whole cross-sectional area, the casting of almost any given billet diameter and the simple adjustment of desired microstructure properties with process parameters such as stirrer speed or gap width between stirrer and cooling device.

![Diagram of mechanical agitation and MHD technique](image)

**Figure 1.1:** Two possible agitation processes for producing rheocast billets

The rheocasting method most used at present in industrial applications is the Magneto-Hydrodynamic (MHD) Technique. The generation of the necessary shear forces on the solidification front is accomplished by an electromagnetic stirring device in the mold area (see Figure 1.1) that develops a strong fluid flow in the semi-solid mushy zone [6]. This fluid flow can be oriented parallel to the feeding direction or perpendicular to it [7,8]. The advantage of electromagnetic rheocasting is certainly contact-free stirring of the semi-solid melt but the adjustment of desired microstructure properties is not as simple as using mechanical agitation. Furthermore, the resulting microstructure is not as homogeneous over the cross-sectional area as the mechanically stirred one.

Other techniques for the production of feedstock material are the Strain-Induced Melt Activation (SIMA) Technique or the use of chemical grain refiners (see Figure 1.2). These two processes do not require agitation on the solidification front for the development of the desired globular microstructure. In the case of the SIMA process a conventional cast billet is cold formed, e.g. extruded or forged. Because of the high strain induced, the dislocation density is increased by several orders of magnitude. During the subsequent heating and soaking in the temperature range between the solidus and liquidus temperature, this high dislocation density acts as a driving force for the globularization of the primary
Figure 1.2: The SIMA and the grain refining process: two other techniques to produce rheocast billets

particles [9]. The SIMA material shows a very fine-grained microstructure and can be advantageous compared to the MHD technique if the material is difficult to cast. Its restrictions are the limited fields of applications; typically only small billet diameters can be processed using the SIMA technique and with an additional process step the production costs rise.

Chemical grain refinement offers a simple alternative using special-purpose grain refiners such as AlTi5B. Because of the elevated amount of heterogeneous nucleation sites a globular microstructure is achieved during the heating and soaking period in the semi-solid temperature regime [10].

UBE Industries Ltd. introduced in 1999 a patented new rheocasting technique, the New Rheocasting (NRC) process [11]. In the NRC process the conventional squeeze casting technology is combined with an innovative method for preparing the globular microstructure of the feedstock material. A fully liquid melt is taken from a holding furnace and poured into specially designed crucibles that are placed on a cyclic conveyor system next to the NRC machine. A globular microstructure is attained by a controlled cooling of the melt into the semi-solid region. Subsequently, the temperature is equalized over the cross-sectional area 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. Die filling is slow and runs from the bottom upwards. That allows a laminar flow pattern and the air is pushed out of the die cavity without being entrapped within the casting [12].
Rheocasting Billets with ThixoStruct

Industrial manufacturing of the feedstock material, usually cylindrical cast billets, means that the continuous process of preparing the semi-solidified metal slurry must be as simple as possible, easy to control, and must result in a reproducible high quality of the thixotropic structure of the as-cast billets. And finally, the fabrication of the billets should be at reasonable cost.

These general requirements were leading to the decision for a mechanical agitation unit for the preparation of the semi-solid slurry, based on earlier experience [13] that heat flow and shear rate—the two major control parameters to achieve an optimum in thixotropic structure and properties—would be easier to control than in the alternative processes with electromagnetic stirring devices.

2.1 Mechanical Agitation and Heat Exchange Unit

In Figure 2.1 the design layout and a photograph of the ThixoStruct rheocaster prototype can be seen. Compared to conventional mechanical stirring devices, ThixoStruct uses a conical stirrer. Combined with an adjustable height of the agitator unit, the conical shape enables the user to adjust the gap width between the stirrer and the heat exchange unit within an interval of 1 to 7.5 mm without interrupting the casting and stirring process. The taper of the conical stirrer and the annulus of the heat exchange unit is a 1/10 of the vertical distance for a simple correlation of the gap width and the vertical height adjustment. At a given rotation speed, the shear rate in the annular gap is given by the following relationship:
Figure 2.1: Design layout and photograph of the \textit{ThixoStruct} rheocaster

\begin{equation}
\dot{\gamma} = \frac{4\pi \cdot \dot{\omega}}{1 - \left( \frac{r_{avr}}{r_{avr} + \Delta x_m} \right)^2}
\end{equation}

where $\dot{\gamma}$ is the shear rate, $\dot{\omega}$ denotes the rotational speed of the stirrer, $r_{avr}$ is its average radius, and $\Delta x_m$ is the annular gap width.

Therefore, control of the shear rate can be accomplished either by a change of the gap width (i.e. the vertical position of the stirrer) or by variation of the rotational speed. The \textit{ThixoStruct} rheocaster provides selection and control of both. The motor direct drive is equipped with high-sensitive control of the rotation speed and the possibility of torque measurements.

Furthermore, the rheocaster is equipped with thermocouples to measure actual temperatures of the metal at different positions in the heat exchange unit during the casting runs, allowing permanent observation of heat transfer conditions.

But before billets can be cast, the heat exchange unit, the tundish, and the graphite stirrer must be heated up to temperatures as near as possible to the temperature of the aluminum melt (approx. $680 \, ^\circ \text{C}$). This will prevent freezing of the material within the gap between stirrer and cooling unit when the liquid metal is poured through the narrow gap. The heat exchange unit and the tundish are equipped with shielded electrical heating coils that make it possible to heat up these two sections to temperatures as high as $700 \, ^\circ \text{C}$. The stirrer is heated indirectly as it is lowered down until it comes in contact with
the inner surface of the heat exchange unit. Therefore, the stirrer is heated up only through contact heat conduction and radiation from the heat exchange unit. However, temperature measurements at the surface of the stirrer have shown that this procedure is fast and reliable enough to generate a homogeneous temperature distribution at the outer surface of the stirrer.

The temperatures of the attached thermocouples are recorded by a laptop computer and when the ones nearest the inner surface of the heat exchange unit reach the melt temperature, the rheocaster is ready for operation. The heating-up sequence takes about 2 hours.

The furnace that contains the aluminum melt (alloy designation A356) is lowered and the melt is poured into the tundish. At the same time, the stirrer starts to rotate with a speed of 100 to 500 revolutions per minute. When liquid metal reaches the hot top area, i.e. the end of the heat exchange unit, the casting platform is being lowered with a constant velocity (e.g. 5 to 7 cm per minute). The filling capacity of the liquid aluminum is kept at a constant level to ensure constant metallostatic pressure throughout the whole casting process.

As soon as the system reaches thermal equilibrium, the heating coils are switched off and the heat exchange unit is cooled with a constant but adjustable air flow (from 0 to 20 liters per minute). The cooling circuit consists of a helical copper tube that is integrated into the heat exchange unit (see Figure 2.2). Because of the now high radial heat transfer the alloy solidifies partially. The aim of the cooling process is to reach the liquidus temperature of the alloy system (i.e. 613 °C for A356) within the heat exchange unit. Because of the agitated metal flow, the first solid particles solidify rosette-like, resulting in the desired near-globulitic microstructure. Attention must be paid to a possible freeze-
back from the direct chill mold. Therefore, the amount of water flow through the direct chill, and the velocity of the casting platform have to be chosen accordingly, in order to prevent freezing of the alloy in the hot top region.

If these process parameter settings are robust enough, the billet can be cast without further changing the settings.

![Image](image_url)

Figure 2.3: Undisturbed oxide layer on the surface of the stirred melt

In Figure 2.3 the oxide layer on the upper metal surface in the tundish is shown during a period of 8 seconds. The sequence shows that no (undesired) oxide layer material is stirred in the liquid metal; the oxide layer remains stationary.

The monitoring of the casting process is provided by the control unit ThixoControl. It is placed right next to the ThixoStruct rheocaster and collects, stores and controls all data and process parameters.

Because of the limited depth of the casting ditch, the casting process must be interrupted whenever the casting platform reaches the floor of the ditch. The flow of the melt is stopped, the billet is removed from the casting ditch and transported to a storage depot (see Figure 2.4). The resulting billets are approximately 6 m long and have a diameter of 22 cm.

To investigate the microstructure, disks are cut at different positions. To achieve a certain desired microstructure, several process parameters can be altered, such as stirrer revolution speed, lowering velocity and air flow etc. But it would also be possible to model the mass and heat flow of the ThixoStruct rheocaster and to predict virtually with which process parameter settings a certain microstructure can be achieved. This modeling is subject of the next chapter.

2.2 Modeling the Mass and Heat Transfer

In the particular process of rheocasting, control of fraction solid and heat and mass flow is even more important than shear rate control. Especially for alloys with a narrow
freezing range\(^1\), simple temperature control between input metal temperature and exit temperature can lead to substantial differences in the fraction solid achieved at the exit of the heat exchange unit. Therefore, precise knowledge of the heat flow conditions in the heat exchange unit is a significant requirement for the reliable operation of the process.

### 2.2.1 Heat Transfer Model

With the goal of implementing the heat flow model into the process control equipment, the objective is to create a model that is as simple yet accurate as possible. For this purpose, the following assumptions were taken:

1. No heat flow into the stirrer.
2. Metal temperature in the annular gap only varies in the vertical direction.
3. Heat flow in the heat exchange unit is only in the radial direction, i.e. heat flow in the vertical direction is negligible.
4. No vertical and no radial heat flow in the hot top area.

The finite difference model based on these assumptions is then described for steady-state conditions with no heat generation by the following heat equation [14]:

\[
\frac{1}{r} \frac{\partial}{\partial r} \left( kr \frac{\partial T}{\partial r} \right) = 0
\]

\(^1\)Difference between liquidus and solidus temperature
where $r$ is the radial position, $k$ the thermal conductivity of the metal, and $T$ the temperature.

Neglecting the interfacial contact resistance between the cast steel heat exchange unit and the copper cooling tube, the radial heat transfer rate $q_r$ can be expressed by

$$q_r = UA(T_m - T_c)$$  \hspace{1cm} (2.3)$$

where $U$ is the overall heat transfer coefficient, $A$ is the interface area normal to the direction of heat transfer, $T_m$ is the metal temperature in the gap, and $T_c$ is the temperature of the coolant. If $U$ is defined in terms of the inside area of the heat exchange unit $A_i = 2\pi r_i L$, equation 2.3 may be equated to yield

$$U = U_t = \frac{1}{\frac{1}{h_1} \cdot \ln \frac{r_o}{r_i} + \frac{1}{k_s} \cdot \ln \frac{r_o + t_c}{r_o} + \frac{1}{r_i \cdot h_2}}$$  \hspace{1cm} (2.4)$$

where $h_1$ and $h_2$ are the convection heat transfer coefficients aluminum $\rightarrow$ cast steel and copper $\rightarrow$ coolant, respectively; $r_i$ and $r_o$ are the radial distances of the inner and outer side of the heat exchange unit; $t_c$ is the thickness of the copper tube; $k_s$ and $k_c$ are the thermal conductivities of steel and copper.

The definition $U = U_t$ in Equation 2.4 is arbitrary, and the overall coefficient may also be defined in terms of $A_o$, the outer area of the heat exchange unit, or of the intermediate area. Note that $U_t A_i = U_o A_o$.

To simulate the heat flow of the ThixoStruct rheocaster, the heat exchange unit is divided into 100 vertical segments with equal heights, each having its specific dimension of $r_i$. The values of $r_o$ and $t_c$ are constant. With known data of the thermal conductivities $k_s$ and $k_c$, the overall heat transfer coefficient $U_t$ depends only on variations of $r_i$, $h_1$, and $h_2$. The latter are determined from experimental temperature measurements at steady-state conditions.

The modeling of the heat transfer is a recursive procedure. So, let's consider for the time being the first segment. The first segment has, like each segment, the height $\Delta z$. The calculation of the radial heat flow results from the known temperature difference $\Delta T$ between the entry temperature $T_m^{(1)}$ of the liquid metal in the first segment, and the temperature $T_c^{(1)}$ of the coolant (i.e., water or compressed air). Other values such as heat transfer coefficients and thermal conductivities of the considered materials must be known to calculate the radial heat transfer rate through the first segment $q_r^{(1)}$ according to Equation 2.3.

$$q_r^{(1)} = U^{(1)} A^{(1)} (T_m^{(1)} - T_c^{(1)})$$  \hspace{1cm} (2.5)$$

where $A^{(1)} = 2\pi r_i^{(1)} \Delta z$ is the conical surface area of the heat exchange unit in the first segment, $T_m^{(1)}$ and $T_c^{(1)}$ are the known entry temperatures of the metal and the coolant respectively. The number in brackets denotes the segment number.
2.2. MODELING THE MASS AND HEAT TRANSFER

Heat Exchange Unit

1. Segment

Figure 2.5: Division of the heat exchange unit into representative segments
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The overall heat transfer coefficient \( U^{(1)} \) in the first segment is defined as, according to Equation 2.4,

\[
U^{(1)} = \frac{1}{ \frac{1}{h_1} + \frac{r_i^{(1)}}{k_s} \cdot \ln \frac{r_o}{r_i^{(1)}} + \frac{r_i^{(1)}}{k_c} \cdot \ln \frac{r_o + t_c}{r_i} + \frac{1}{r_o + t_c} \cdot \frac{1}{h_2} }
\]  

(2.6)

With the given geometry and the known temperatures, the radial heat transfer rate in the first segment \( q_r^{(1)} \) can be calculated. And if \( q_r^{(1)} \) is known, the temperatures at the boundary surfaces can be calculated, e.g. the temperature \( T_3^{(1)} \) on the surface of the heat exchange unit in the first segment:

\[
T_3^{(1)} = T_m^{(1)} - \frac{q_r^{(1)}}{A^{(1)} \cdot h_1}
\]  

(2.7)

where \( h_1 \) is the convection heat transfer coefficient aluminum → cast steel.

Let’s move on to the next segment. The liquid metal transferred the heat \( q_r^{(1)} \) to the coolant in the first segment. Consequently, the metal temperature drops \( \Delta T_m \). On the other hand, the coolant’s temperature rises \( \Delta T_c \). Therefore, there is a new temperature difference between metal and coolant \( \Delta T^{(2)} \) in the second segment. This smaller temperature difference influences the radial heat transfer rate in the second segment \( q_r^{(2)} \). Moreover, the overall heat transfer coefficient \( U^{(2)} \) changes as well because the stirrer has a conical geometry and the wall thickness of the heat exchange unit increases.

The radial heat transfer rate \( q_r^{(2)} \) and the overall heat transfer coefficient \( U^{(2)} \) in the second segment can be expressed similarly as in Equations 2.5 and 2.6

\[
q_r^{(2)} = U^{(2)} A^{(2)} \left( T_m^{(2)} - T_c^{(2)} \right)
\]  

(2.8)

\[
U^{(2)} = \frac{1}{ \frac{1}{h_1} + \frac{r_i^{(2)}}{k_s} \cdot \ln \frac{r_o}{r_i^{(2)}} + \frac{r_i^{(2)}}{k_c} \cdot \ln \frac{r_o + t_c}{r_i} + \frac{1}{r_o + t_c} \cdot \frac{1}{h_2} }
\]  

(2.9)

To calculate the radial heat transfer rate in the second segment, the temperature difference \( \Delta T^{(2)} = [(T_m^{(1)} - \Delta T_m) - (T_c^{(1)} - \Delta T_c)] \) must be known. But how can we calculate \( \Delta T_m \) and \( \Delta T_c \)? If the temperature of the liquid metal is above the liquidus temperature (in the case of A356, the liquidus temperature \( T_L \) is 613 °C), the temperature drop can be calculated as follows:

\[
\Delta T_m = \frac{q_r^{(1)}}{c_{p,m} \cdot \rho_{m} \cdot V_m^{(1)}} \Delta T_{m} \quad \forall \ T_m \geq T_L
\]  

(2.10)
2.2. MODELING THE MASS AND HEAT TRANSFER

where \( c_{p,m}^L \) is the specific heat of the liquid metal, \( \rho_{m}^L \) is the mass density of the liquid metal, \( \Delta t_{m}^{(i)} \) is the duration of stay of the metal in the first segment (i.e. the flow rate), and

\[
\Delta V_{m}^{(i)} = \pi \cdot \Delta z \cdot \left[ \left( r_{i}^{(i)} + \Delta x_{m} \right)^2 - \left( r_{i}^{(i)} \right)^2 \right]
\]  

(2.11)

is the segment volume of the liquid metal.

If the temperature of the metal falls below the liquidus temperature \( T_L \), solid primary phase particles build. Because of solidification, the latent heat \( L_A \) of the primary phase is released and counteracts the temperature drop.

\[
q_{p,m} \cdot \Delta t_{m}^{(i)} \cdot \Delta V_{m}^{(i)} \cdot L_A \cdot \Delta f_{S}^{(1\rightarrow 2)} \geq \frac{f_{S}^{(1\rightarrow 2)}}{f_{S}^{(1\rightarrow 2)}}
\]

(2.12)

where \( f_{S}^{(1\rightarrow 2)} \) is the fraction solid solidified in the first segment.

The model assumes a buffer effect of the upper segments, meaning that the maximal temperature in a segment can be the same as the one in the preceding segment. Thus, the second term in Equation 2.12 must not be greater than the first one.

\[
\frac{L_A}{c_{p,m}^L} \cdot \Delta f_{S}^{(1\rightarrow 2)} \leq \frac{q_{r}^{(i)}}{c_{p,m}^L \cdot \rho_{m}^L} \cdot \frac{\Delta t_{m}^{(i)}}{\Delta V_{m}^{(i)}}
\]

(2.13)

If the temperature in the annular gap reaches the solidus temperature \( T_S \), the temperature stays constant until all of the eutectic melt has solidified. But this scenario should not be attained because a rheocaster can freeze if the fraction liquid falls below 50%.

For the temperature rise of the coolant, analogous considerations can be made:

\[
\Delta T_{c} = \frac{\Delta t_{m}^{(i)}}{\rho_{c}^L} \cdot \frac{1}{c_{p,c}^L} \cdot \Delta f_{S}^{(1\rightarrow 2)}
\]

(2.14)

where \( c_{p,c}^L \) is the heat capacity, \( \rho_{c}^L \) is the mass density, and \( \Phi_{c} \) is the flow rate of the coolant.

With the smaller temperature difference \( \Delta T^{(2)} \) but the higher overall heat transfer coefficient \( U^{(2)} \) compared to the first segment, the radial heat transfer rate \( q_{r}^{(2)} \) can be calculated as well as the temperatures at the interfaces.

With this recursive procedure, the radial heat transfer rate \( q_{r}^{(i)} \) of each segment \( i \) can be calculated.

2.2.2 Solidification Model

The parameter of most interest, the fraction solid \( f_{S} \), is calculated step by step from the thermal balance of the actual heat content of each segment, starting at the top of the
heat exchange unit with the incoming liquid metal at temperature $T_m^{(1)}$, with the initial heat content $Q_m$, considering specific heat content as well as latent heat of fusion.

The fraction solid $f_S$ of the melt is directly linked to the actual metal temperature and can be calculated using Scheil's equation:

$$f_S^{(n)} = 1 - f_L^{(n)} = 1 - \left( \frac{T_M - T_L}{T_{M} - T_{m}^{(n)}} \right)^{1/k}$$  \hspace{1cm} (2.15)

where $f_S^{(n)}$ and $f_L^{(n)}$ are the solid and liquid fractions of the melt in the $n$-th segment, $T_M$ is the melting point of the pure metal (in our case pure aluminum), $T_L$ is the liquidus temperature of the investigated alloy (in our case A356, i.e. AlSi7), $T_{m}^{(n)}$ is the metal temperature in the $n$-th segment, and $k$ is the partition coefficient of the alloy system.

Van’t Hoff proposed an equation for the calculation of the partition coefficient $k$:

$$k = 1 - \frac{m_L \cdot L_M}{R \cdot T_M^2}$$  \hspace{1cm} (2.16)

where $m_L$ is the slope of the liquidus line in the hypo-eutectic alloy system, $L_M$ is the latent heat of fusion of the pure metal, and $R$ is the universal gas constant.

How well the calculated fraction liquid $f_L$ corresponds to experimental data is shown in Figure 2.6. An interesting effect is the undercooling in the experiment, caused by a defined cooling rate and also by alloy composition. According to Scheil, the remaining eutectic melt at the solidus temperature $T_S$ solidifies isothermally, which is only the case when the cooling rate is indefinitely slow.

The experimental data were obtained with a Differential Thermal Analysis (DTA) experiment (see Figure 2.7) that is explained in Chapter 6.1. The fraction solid $f_S$ can be calculated at a specific temperature between the solidus and liquidus temperature according to:

$$f_S = \int_{T_S}^{T} (y + q) \cdot d\vartheta / \int_{T_S}^{T_L} (y + q) \cdot d\vartheta \quad \forall \quad T_S \leq T \leq T_L$$  \hspace{1cm} (2.17)

where $y$ is the DTA signal, $q$ is the offset of the DTA curve, and $\vartheta$ is the integration variable for the temperature $T$.

Scheil's equation (Equation 2.15) is only valid for the temperature region between liquidus and solidus temperature. When the temperature is still above the liquidus temperature of the alloy system $T_L$, the fraction solid $f_S$ equals zero:

$$f_S^{(n)} = 0 \quad \forall \quad T_{m}^{(n)} > T_L$$  \hspace{1cm} (2.18)

If the temperature reaches the solidus temperature $T_S$, the temperatures keep constant because of the isothermal latent heat release at the eutectic temperature. The
Figure 2.6: Comparison of measured fractions liquid and calculated values according to Scheil's equation

Figure 2.7: DTA curve recorded during the cooling down from the fully liquid state, cooling rate: 2 K/min
temperature can only drop further when the last melt has solidified. The amount of eutectic melt that has solidified in the $n$-th element $\Delta f_{S,E}^{(n)}$ is:

$$\Delta f_{S,E}^{(n)} = \frac{q^{(n)}}{L_E \cdot \rho_m} \cdot \frac{\Delta t_m^{(n)}}{\Delta V_m^{(n)}}$$  \hspace{1cm} (2.19)

where $L_E$ is the latent heat of fusion of the eutectic phase.

If the eutectic solidification begins in the $n$-th segment, the overall fraction solid $f_S$ in the $m$-th segment ($m > n$) equals:

$$f_S^{(m)} = f_S^{(n-1)} + \sum_{i=n}^{m} \Delta f_{S,E}^{(i)}$$  \hspace{1cm} (2.20)

As mentioned above, the temperature can only drop further, when the fraction solid equals 1:

$$\max \left(f_S^{(n)}\right) = 1$$  \hspace{1cm} (2.21)

Of course this equation serves only as a theoretical limit because no rheocaster should be confronted with a fully solidified metal within the heat exchange unit.

### 2.3 Formation of the Globular Microstructure

Figure 2.8 shows a typical example of a solidified rheocast structure of A356. The microstructure is non-dendritic, the primary particles show a globular or rosette-like shape. Figure 2.9 shows the structures of a solidified unstirred sample, showing the typical dendritic structure. Why are those structures so different? There exist theories that explain the rosette-like structure in rheocast samples with the mechanical fragmentation of dendrite arms. These theories assume that the melt would solidify in a dendritic morphology but the shear rate inducing agitator destroys the dendrites and thus, rosette-like particles originate from the dendrite fragments. But with a stirred solidification model [5] it can be shown that solid particle growth in stirred melts is likely to be globular already in the early stage of solidification.

The principal difference between stirred and unstirred (i.e. directional) solidification, with respect to the heat flow, is schematically shown in Figure 2.10. In the unstirred case, heat flow is unidirectional from the liquid through the solid towards the (cooled) surface. The thermal gradient at the solid-liquid interface is positive. In contrast, during rheocasting, the latent heat evolved is dissipated from the solid particles through the liquid. To make this possible, the liquid has to be undercooled. The thermal gradient at solid-liquid interface in that case is therefore negative and no constitutional supercooling can take place.

The fluid flow around floating particles in a field of constant shear can be expressed as a superposition of the externally applied velocity field $\mathbf{v}^0$ and an additional flow velocity $\mathbf{v}^1$. 
2.3. FORMATION OF THE GLOBULAR MICROSTRUCTURE

Figure 2.8: Sample micrograph of a rheocast structure with globular or rosette-like particles, 200× [15]

Figure 2.9: Sample micrograph of a conventionally cast structure with dendrites, 200× [15]
Assuming that the radial component $v_r$ of the combined flow field $v^0 + v^1$ contributes to the heat and mass transport at the interface, it is possible to determine the distance from the surface of the sphere where the convection predominates the diffusion. The criterion for this is that the Peclet number for the radial flow, which is defined by

$$\text{Pe} = \frac{v_r d}{D}$$  \hspace{1cm} (2.22)

must be greater than 1. In Equation 2.22, $d$ is the diameter of the globular primary particle, and $D$ is the diffusion coefficient. The radial component $v_r$ of the flow field can be described best with a spherical coordinate system where the origin is the center of the considered primary particle [5]:

$$v_r = \frac{\gamma R}{2} \sin(2\phi) \sin^2 \theta \left[1 - \frac{5}{2} \frac{R^3}{r^3} + \frac{3}{2} \frac{R^5}{r^5}\right]$$  \hspace{1cm} (2.23)

where $\gamma$ is the shear rate, and $\phi$, $\theta$ and $r$ are the spherical coordinates. Using a Cartesian coordinate system $x,y,z$ for the location in the flow field near the particle yields $r^2 = x^2 + y^2 + z^2$. $R$ is the radius of the globular primary particle.

Substituting $\varrho = r/R$, solving Equation 2.22 for $\text{Pe} = 1$, and considering the global maximum of the radial components\(^2\) leads to:

$$\varrho = \frac{5}{2} \varrho^2 + \frac{3}{2} \varrho^4 = \frac{D}{\gamma R^2}$$  \hspace{1cm} (2.24)

Evaluating $\delta_d = r - R$, it is found that $\delta_d$ approaches the value

\(^2\text{i.e. for angles } \phi = 45^\circ \text{ and } \theta = 90^\circ\)
provided \( r \ll (D/\dot{\gamma})^{1/2} \). At small \( R \), i.e. when \( R \ll (D/\dot{\gamma})^{1/2} \), it follows from Equation 2.24 that \( \delta_d = D/\dot{\gamma}R \). The function \( \delta_d(R) \) is shown in Figure 2.11 for different shear rates \( \dot{\gamma} \), and a mass diffusivity value \( D \) of \( 5 \cdot 10^{-9} \text{ m}^2/\text{s} \) [16]. The function \( \delta_d(R) \) defines under which conditions the mass transport is dominated by diffusion or convection. With decreasing particle radius \( R \), the distance \( \delta_d \) over which transport by diffusion predominates, increases. This is because the influence of the additional flow field \( \mathbf{v} \) is small for small particles. As stated above, the particles that grow in the bulk liquid have a temperature slightly higher than the temperature of surrounding liquid. This enables the particle to dispose of the latent heat released at the surface, which means that the particle can grow. For a particle with a radius \( R \) of 100 \( \mu \text{m} \), Figure 2.11 shows that for a shear rate of 100 \( \text{s}^{-1} \) the mass transport by convection predominates that by diffusion at distances greater than 2.5 \( \mu \text{m} \). Provided the diffusion boundary layer thickness is 2.5 \( \mu \text{m} \), the solute peak at the solid-liquid interface of a floating particle is reduced due to the fluid flow around the particle. As a consequence, the equilibrium temperature distribution ahead of the interface will be almost uniform. Combined with the small negative thermal gradient at the interface, the amount of constitutional supercooling ahead of the interface is small. The interface of the particle is stable when the thermal gradient at the interface equals 0, a condition that cannot be achieved completely, so long as the growth rate \( G \) of the primary particles is positive. But based on this analogy, the growth of a solid particle in a stirred bulk liquid will be cellular when the thermal gradient at the interface has become sufficiently small. The interface of the particle is stable when the thermal gradient at the interface equals 0, a condition that cannot be achieved completely, so long as the growth rate \( G \) of the primary particles is positive. But based on this analogy, the growth of a solid particle in a stirred bulk liquid will be cellular when the thermal gradient at the interface has become sufficiently small.

\[
\delta_d^\infty = \left( \frac{2 D}{15 \dot{\gamma}} \right)^{1/2}
\]  

(2.25)

2.4 Calculation of the Primary Particle Size

Experimental investigations [17–21] showed that the primary particle size depends on the fraction solid \( f_s \), the cooling rate \( C_R = \Delta T/\Delta t \), and the shear rate \( \dot{\gamma} \). Other variables such as the temperature of the melt \( T_m^{(1)} \) at the entry of the heat exchange unit, the gap width \( \Delta x_m \), the entry of the heat exchange unit, the gap width \( \Delta x_m \) between stirrer and reactor, the revolution speed \( \dot{\omega} \) of the stirrer, and the lowering velocity \( v_z \) of the casting platform are further parameters that are derived from the primary process parameters. The following calculation scheme by Prasad et al. [22] assumes that the primary particles do not result from the fragmentation of dendrites but evolve directly globularly from the melt. The fraction solid \( f_s \) built in time \( t \) is, according to the kinetic law of phase transformation:

\[
f_s = 1 - \exp \left[ -\frac{\pi}{3} \cdot N \cdot G^3 \cdot t \right]
\]  

(2.26)
where $N$ is the nucleation rate and $G$ the growth rate. $N$ and $G$ are considered to be constant. The number of primary particles $n_p$ formed in time $t$ is

$$n_p = N \cdot t \cdot V_u$$  \hspace{1cm} (2.27)

where $V_u$ is the untransformed (=still liquid) volume. If in this time $t$ the primary particles grow to an average diameter $d$, then

$$V \cdot f_S = \frac{\pi}{6} \cdot n_p \cdot d^3$$  \hspace{1cm} (2.28)

where $V$ is the total volume. Eliminating $n_p$ from Equations 2.27 and 2.28 and introducing $V_u/V = 1 - f_S$ yields

$$N = \frac{6 \cdot f_S}{\pi \cdot d^3 \cdot (1 - f_s) \cdot t}$$  \hspace{1cm} (2.29)

For a model that considers only single particles, the mass balance at the solid–liquid interface is

$$\frac{\partial m}{\partial t} = \frac{4 \cdot \pi \cdot R_i^2 \cdot D_L \cdot \rho \cdot (C_L - C_0)}{\delta_c}$$  \hspace{1cm} (2.30)
2.4. CALCULATION OF THE PRIMARY PARTICLE SIZE

where \( m \) is the transformed mass, \( D_L \) the liquid diffusion coefficient of the solute alloying element, \( R_t \approx Gt \) the radius of the solidified particle at time \( t \), \( \rho^L_a \) the mass density of the liquid aluminum alloy, \( C_L \) the liquid composition at the solid-liquid interface, \( C_0 \) the bulk liquid composition, and \( \delta_h \) the hydrodynamic boundary layer thickness. If liquid melt flows past a solidified primary particle the thickness of the hydrodynamic boundary layer yields

\[
\delta_h = \frac{D_L^{1/3} \cdot \nu^{1/6} \cdot q^{1/2}}{v^{1/2}}
\]

(2.31)

where \( \nu \) is the kinematic viscosity and \( v \) is the flow velocity of the liquid phase.

The total amount of mass transformed \( m \) at time \( t \) can be described as

\[
m = \frac{4\pi}{3} \cdot R_t^3 \cdot \rho^L_a \cdot (C_0 - C_S)
\]

(2.32)

where \( C_S \) is the average composition of the solid particles formed. Based on Equations 2.30, 2.31 and 2.32, the average growth rate \( G \) can be written as

\[
G = \kappa \left( \frac{D_L^{4/9} \cdot v_0^{1/3}}{\nu^{1/9} \cdot q^{1/3}} \right) \left( \frac{C_L - C_0}{C_0 - C_S} \right)^{2/3}
\]

(2.33)

where \( \kappa = 0.896 \) [22] is a non-dimensional constant.

When the revolving speed of the stirrer is increased, the flow velocity of the liquid phase \( v \) is also increased and therefore the average growth rate \( G \) also increases (according to Equation 2.33) resulting in a larger primary particle size. But that contradicts known experimental results [17–21], where primary particles are smaller with increasing revolution speed. The reason for this discrepancy is that the solid particles do not remain stationary during agitation, but move under the action of fluid forces. Consequently, the relative velocity between solid particles and fluid phase decreases drastically. Therefore it is recommended that the velocity \( v \) of the liquid phase should be replaced with a relative or slip velocity \( v_s \). Although the exact relationship between \( v \) and \( v_s \) is not known [22], an inverse relationship is assumed:

\[
v_s = \frac{\alpha}{v} = \frac{\alpha}{\pi \cdot r_{aav} (1 - \beta^2) \cdot \dot{\gamma}}
\]

(2.34)

where \( \alpha \) is a proportional constant, \( r_{aav} \) the average radius of the stirrer, \( \beta \) the radius ratio of the inner radius of the heat exchange unit \( r_{aav} + \Delta x \) and the agitator radius \( r_{aer} \), and \( \dot{\gamma} \) the shear rate.

Noting that

\[
t = \frac{T_L - T_R}{C_R}
\]

(2.35)
where $T_L$ is the liquidus temperature of the alloy system, $T_R$ a temperature in the semi-solid range, and $C_R$ the cooling rate, a universal equation for the primary particle size $d$ can be obtained:

$$d = \frac{p \cdot D_{L_0}^{1/9} \cdot (T_L - T_R)^{3/3}}{C_R^{1/3} \cdot \gamma^{1/3}} \cdot \left(\frac{-f_S}{(1 - f_S) \cdot \ln(1 - f_S)}\right)^{1/3} \cdot \left(\frac{C_L - C_0}{C_0 - C_S}\right)^{2/3} \quad (2.36)$$

The fraction solid $f_S$ is correlated with temperature according to Scheil's equation:

$$f_S = 1 - \left(\frac{T_M - T_L}{T_M - T_R}\right)^{1/k} \quad (2.37)$$

where $k$ is the partition ratio (see Equation 2.16) and $T_M$ the melting temperature of aluminum. The value of $p$ cannot be found from theoretical considerations because its value depends on the exact relationship between the velocity $v$ of the fluid phase and the relative slip velocity $v_s$ between primary particles and melt. A detailed computer analysis [22] of the experimental data suggests that for a cylindrical stirrer geometry the following relationship can be derived:

$$p = \frac{\kappa'}{C_0^{3/3}} \quad (2.38)$$

where $\kappa' = 0.119$ [22] is an empirical non-dimensional constant.

Substituting for $T_R$ the metal temperature in the hot top region, Equation 2.36 yields the primary particle size $d_a$ at the exit of the heat exchange unit. Figure 2.12 shows the relationship between the hot top temperature and the resulting primary particle size $d_a$. For the calculation, a gap width of 7.4 mm, a revolution speed of 400 rpm and a platform lowering velocity of 5 cm/min was considered.

Unfortunately, the calculated size of the primary particles at the exit of the heat exchange unit cannot be compared with experimental values because the solidification progresses after the exit of the heat exchange, resulting in a coarsening of the particles. But substituting $T_R$ with the solidus temperature $T_S$ and the fraction solid $f_S$ with the maximal volume fraction of primary particles $f_S^P = 46.4\%$ (according to the lever rule) in Equation 2.36, the average size $d_p$ of the primary particles just before solidification of the remaining eutectic melt can be calculated.

With the same process parameters as mentioned above, the average size of the primary particles $d_p$ at the solidus temperature $T_S$ equals 59 $\mu$m. In contrast to the particle size at the exit of the heat exchange unit the size of the primary particles at $T_S$ can be determined experimentally. If we compare the calculated value with the chord length distribution of a sample micrograph (see Figure 2.13) the agreement is exceptional. The mean value of this chord length distribution is 36.8 $\mu$m. The relationship between chord length and particle size is given by [23]

$$d_p = \frac{3}{2} \cdot \bar{l} \quad (2.39)$$
2.4. **CALCULATION OF THE PRIMARY PARTICLE SIZE**

![Graph](image)

Figure 2.12: Size $d_a$ of primary particles at the exit of the heat exchange unit versus the temperature in the hot top region

where $\bar{l}$ is the mean chord-length. Therefore the experimental mean particle size $d_p$ equals 55 $\mu$m, which is in good agreement with the calculated value $d_p$ of 59 $\mu$m.

A more thorough discussion of the achieved microstructures is presented in the following chapter.

![Graph](image)

Figure 2.13: Chord-length distribution of a sample micrograph
CHAPTER 2. RHEOCASTING BILLETS WITH THIXOSTRUCT
Results of the Casting Runs

During a two-week casting campaign ten rheocast billets were produced. The billets had a diameter of 22 cm and a length of approximately 6 m each. The pursued goals of this campaign were:

1. to prove the industrial utilizability of the ThixoStruct rheocaster,
2. to verify the mass and heat transfer model derived in Chapter 2.2,
3. to guarantee a homogeneous microstructure throughout the entire billet, and
4. to ensure a feedstock material quality comparable with that of industrial manufacturers.

The first goal was performed extremely well. The rheocaster proved very robust without breakdowns and the total mass of rheocast aluminum A356 yielded 2 metric tons. In the following sections the performance of the other goals is summarized.

3.1 Heat Extraction Capability of the ThixoStruct Rheocaster

In order to achieve a semi-solid slurry in the hot top mold, the melt temperature at the exit of the heat exchange unit must be lower than the liquidus temperature $T_L$ of the alloy system. To ensure this temperature, the heat extraction capability of the heat exchange unit must be known. The radial heat transfer rate was measured with a custom-made temperature probe (see Figure 3.1). The probe consists of three thermocouples that are radially offset by 7.5 mm. With this design, three temperatures can be measured simultaneously at different radial positions and the radial heat transfer rate can be evaluated according to Equation 2.3:
$q_r = U \cdot A \cdot (T_m - T_c)$

where $U$ is the overall heat transfer coefficient, $A$ is the interface area of the heat exchange unit normal to the direction of heat transfer, $T_m$ is the (average) metal temperature in the gap, and $T_c$ is the (average) temperature of the coolant. With a constant pressurized air flow of 11 liters per minute an average heat extraction of $q_{\text{calc}} = 4.61 \times 10^3$ J/s was computed. To verify this calculated heat flow with experimental results, the melt temperature in the tundish and in the hot top region was measured. The temperature difference indicates the heat extracted within the heat exchange unit. The mass flow rate $\dot{m}_a$ of the aluminum through the heat exchange unit was 100.35 g/s for a gap width $\Delta x$ of 5 mm and a billet lowering velocity $v_z$ of 5.5 cm/min. The extracted heat can be calculated as follows:

$$q_{\text{exp}} = \dot{m}_a \cdot c_{p,a}^L \cdot \Delta T$$

where $\dot{m}_a$ is the mass of the metal in the annular gap of the heat exchange unit, $c_{p,a}^L$ is the heat capacity of the aluminum alloy. For the sake of simplification a constant heat capacity of 0.98 J/gK was assumed. The average temperature difference $\Delta T$ between the melt temperature in the tundish and in the hot top region was 47 °C.

According to Equation 3.1 the experimental heat transfer rate yields $q_{\text{exp}} = 4.62 \times 10^3$ J/s. The very good agreement with the calculated value of $q_{\text{calc}} = 4.61 \times 10^3$ J/s proves that the established heat transfer model is very accurate.

In Figure 3.2 the connection between the entry temperature $T_m\text{(1)}$ and the outlet temperature $T_m^{(n)}$ is visualized. For comparison, some experimental data are shown that

---

1 $n$ denotes the number of the last segment of the heat exchange unit, in this case $n = 100$
are obtained during the casting runs. Close agreement with the simulated temperatures justifies the assumptions and considerations on which the heat transfer model is based.

![Graph showing comparison of calculated and experimental entry and outlet temperatures of the ThixoStruct rheocaster](image)

**Figure 3.2:** Comparison of calculated and experimental entry and outlet temperatures of the ThixoStruct rheocaster

### 3.2 Microstructures of Rheocast Billets

#### 3.2.1 Describing Microstructures

Micrographs of rheocast aluminum alloys with so-called thixotropic behavior often show great variety in the shape and connectivity of primary particles. In order to classify these alloys, guidelines for an ideal microstructure have to be established. Rheological assumptions can lead to criteria that define such an ideal thixotropic microstructure. Once established, these criteria can be used as a guideline in the rheocasting process of semi-solid aluminium alloys. Structural parameters such as particle size, form factor, connectivity and agglomeration ratio of primary particles have a great influence on the thixotropic behavior of an aluminium alloy. Once the relationship between process parameters of a rheocasting process and properties of the microstructure is known, the semi-solid casting process can be optimized. The main influences that change the structural parameters of a semi-solid cast aluminum alloy are:

- the rotation speed of the stirrer,
- the gap width between stirrer and heat exchange unit,
and the temperature of the liquid aluminum in the tundish.

The globular morphology of primary particles is fundamental to the microstructure of a feedstock material billet. It is this specific morphology that guarantees the intrinsically viscous flow behavior in the semi-solid temperature range. With conventionally cast billets having a dendritic morphology, this behavior cannot be observed. Because the dendrites in the molten eutectic entangle with each other under shearing or compression forces, there is no decrease in viscosity. Of course, the boundary between dendritic and globular morphologies is quite arbitrary and therefore decisive criteria have to be established that predict the order of intrinsic viscosity. For this reason, it is desirable to specify the investigated microstructure with as few quantities as possible.

Size Distribution of Primary Particles

Primary information on rheological behavior gives the mean particle size and its distribution. They can be determined by different methods, i.e. linear analysis or object oriented methods. The measured mean chord length can be converted to a mean particle size using Equation 2.39 if assuming spherical particles. For a detailed characterisation of microstructures the consideration of the distribution of the chord lengths reveals more insight. Independent of the chosen method (i.e. linear analysis or object oriented methods), particle size distributions of planar cross sections vary from the spatial distribution of the particles for the following reasons:

- **Reduction effect**: the plane of section cuts off segments of particles that are smaller than the area of a center cut. Therefore the diameters of the segments are smaller than the equivalent diameters of the particles.

- **Recording effect**: the plane of section cuts big particles more often than their actual frequency predicts, whereas smaller particles are cut more seldom.

With the knowledge of the size distribution, some basic assumptions about rheological behavior can be made. Although an upper boundary for the maximal particle size does not exist in literature, experience shows that the maximal particle size should not exceed a twentieth of the minimal wall thickness of the final part [24], i.e. for thixoformed parts this is approx. 3 mm. Therefore, a maximal particle size of 150 μm should not be exceeded.

Form Factor

Characteristic viscosity features such as the shear rate sensitivity cannot be described using only the size distribution of the primary particles. It is possible to class each cross section of a primary particle in a micrograph with a form factor \( F \), which is defined as

\[
F = \frac{4\pi \cdot A}{P^2}
\]  

(3.2)
3.2. MICROSTRUCTURES OF RHEOCAST BILLETS

where $A$ is the area and $P$ the perimeter of the particle [25]. Globular primary particles have a form factor of 1 whereas dendritic fragments yield lower values\(^2\) (see Figure 3.3). Knowing the form factor and the particle size distribution of primary particles in a micrograph, it is possible to establish the following requirements for an ideal thixoform microstructure:

1. The microstructure should have small primary particles (maximal 150 $\mu$m) with a narrow size distribution.

2. Primary particles should be as globular as possible ($F > 0.5$) in order to facilitate shearing in the semi-solid range. Besides, unlike dendritic fragments, globular particles trap less liquid phase, therefore as much fluid as possible contributes to the viscous flow.

Contiguity Volume

One important criterion for thixoforming is the presence of a yield strength in the semi-solid temperature range in order to handle a reheated billet. An increase in yield strength can be obtained when some of the primary particles are connected with each other. But the cohesive forces in this skeleton should not exceed the shear forces applied and therefore hinder the disruption of the connected particles during the thixoform process. The volume fraction of these skeletal connected particles can be evaluated by means of the contiguity $C$ [26]. It is a measure of solid-solid interfaces in a semi-solid structure and is defined as the fraction of surface area shared by one specific grain with all neighboring grains of the same phase. For the solid phase the contiguity $C_S$ can be defined as:

$$C_S = \frac{2S_{SS}}{2S_{SS} + S_{SL}}$$

(3.3)

where $S_{SS}$ is the interface area in the solid phase, i.e. the interface between connected particles that are not separated by liquid phase, $S_{SL}$ is the interface area between the solid and the liquid phase. In the case of $C_S = 0$ all the particles are isolated and completely

\(^2\)In literature, often the inverse representation of the form factor can be found where $F_{alt} = 1/F$ and $0 \leq F_{alt} < \infty$. The main advantage of the form factor $F$ used in this thesis is the possibility of a graphical visualization that is linear in $F$. 
surrounded by melt. With increasing $C_S$ the particles get more connected and the skeletal agglomeration increases. Low values of $C_S$ are not desired since the semi-solid structure does not possess form stability. On the other hand, when $C_S \to 1$ and the solid phase is fully agglomerated, the transition to suspension cannot be achieved. Since the strength of the skeleton depends not only on the contiguity, but also on the fraction solid $f_S$ it is recommended that the product $f_S \cdot C_S = C_V$ be introduced as a decisive term. It describes the volume fraction of connected solid particles and is called contiguity volume [27].

The distinctive thixotropic properties are only observed within a certain interval of $C_V$. Uggowitzer suggests [28] an upper boundary of 0.3 and a lower boundary of 0.1 for $C_V$: $0.1 < C_V < 0.3$

### 3.2.2 Experimental Microstructures

In Figure 3.4 micrographs sampled from the center and from the fringe of a rheocast billet are shown. Although there are regions with globular particles, there exist still regions with coarse dendrites. This is probably due to too high a temperature (i.e. about 1 or 2 °C above the liquidus temperature $T_l$) in the hot top region during casting.

The mean particle size at the center is 117 μm. At the fringe of the billet it is only slightly smaller: 95 μm. The mean form factor $F$ in the center and at the fringe yields the same value: 0.54. More information reveals the distributions of these two microstructural parameters: the median$^3$ of the particle size distribution at the center is 83 μm whereas at the fringe it is only 61 μm. Thus, at the fringe there are more "small" particles than in the center and less but huger "large" particles. This is a clear sign of more dendritic solidification at the fringe of the billet because of direct chill solidification, and hence the more severe temperature gradient at the outer surface of the billet. The form factors $F$ of the two investigated microstructures show almost a perfect linear distribution in $F$ (see Figure 3.4) and no difference in origin can be seen. Apparently the metal expelled from the heat exchange unit was distributed homogeneously over the whole cross-section and the solidification took place only seconds later. Further confirmation of the homogeneous microstructure across the cross-section shows the local chemical composition. With the Glow Discharge Optical Emission Spectroscopy (GDOES) method the local chemical composition was measured. Table 3.1 shows the results for silicon and magnesium contents. The fluctuations in chemical composition along the radius are below ±5%.

Although the microstructure is very homogeneous across the cross-section there exist still dendritic regions that could be diminished with a lower pouring temperature of the liquid melt.

### 3.2.3 Comparison with Commercially Cast Billets

The microstructures of the rheocast billets were compared with commercially rheocast billets of different manufacturers. Unlike the billets produced with ThixoStruct, the commercially produced billets were all cast using the MHD technique. Furthermore, the diameter

$^3$The median is the value of the middle item when data are arranged in order of size; it is a measure of central tendency.
Figure 3.4: Experimental microstructures together with their chord length and form factor distributions, material: A356
CHAPTER 3. RESULTS OF THE CASTING RUNS

<table>
<thead>
<tr>
<th>Radial position</th>
<th>Silicon</th>
<th>Magnesium</th>
</tr>
</thead>
<tbody>
<tr>
<td>12 mm</td>
<td>6.97 w-%</td>
<td>0.43 w-%</td>
</tr>
<tr>
<td>39 mm</td>
<td>7.16 w-%</td>
<td>0.44 w-%</td>
</tr>
<tr>
<td>66 mm</td>
<td>6.69 w-%</td>
<td>0.44 w-%</td>
</tr>
<tr>
<td>93 mm</td>
<td>6.54 w-%</td>
<td>0.45 w-%</td>
</tr>
</tbody>
</table>

Table 3.1: Chemical composition of alloying elements at different radial positions, material A356

of these billets was only half the size of the *ThixoStruct* cast billets, i.e. 10 cm instead of 22 cm. Common to all MHD cast billets is the very globular microstructure at the center of the billet. Figure 3.5 shows sample micrographs at the center of all commercially cast billets as well as a micrograph of a *ThixoStruct* billet. The form factor distributions show clearly that the commercially cast billets have a more globular microstructure at the center than the *ThixoStruct* billet. Apparently, the MHD technique builds up higher shear rates at the center of the billet than the mechanical agitation technique used with *ThixoStruct*.

However, looking at the micrographs of the fringe position reveals a completely different picture (see Figure 3.6). At the fringe, the form factor distribution shifts to a substantially lower value for the MHD cast billets, whereas the form factor distribution of the *ThixoStruct* sample is similar to that at the center position. Because all the material went through the narrow gap, the homogeneity of the form factor distribution across the cross section is not surprising. Whereas with the MHD technique, the whole cross section is sheared constantly. Together with a non-uniform radial and axial velocity distribution this results in a non-uniform form factor distribution of the agitated solid particles. Although the discrepancy in form factors is equalized during the subsequent reheating process, there is need for better control of the induced agitation.

Table 3.2 summarizes the microstructural quantities average form factor $\bar{f}$ and average particle size $\bar{d}$ together with their standard deviations.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Center position</th>
<th>Fringe position</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\bar{f}$</td>
<td>$\sigma_f$</td>
</tr>
<tr>
<td>A</td>
<td>0.57</td>
<td>0.25</td>
</tr>
<tr>
<td>B</td>
<td>0.59</td>
<td>0.24</td>
</tr>
<tr>
<td>C</td>
<td>0.60</td>
<td>0.24</td>
</tr>
<tr>
<td><em>ThixoStruct</em></td>
<td>0.54</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 3.2: Average form factor $\bar{f}$, mean particle size $\bar{d}$, and their standard deviations $\sigma_f$ and $\sigma_d$ for different feedstock material producers, material A356

Another drawback of MHD cast billets is their inhomogeneity with regard to the distribution of the alloying elements. Table 3.3 shows the chemical composition at different radial positions for all commercially cast billets compared with the one of a *ThixoStruct* billet. As mentioned above, the fluctuations in chemical composition of the *ThixoStruct*
3.2. MICROSTRUCTURES OF RHEOCAST BILLETS

Figure 3.5: Comparing microstructures and form factor distributions of commercially MHD and ThixoStruct cast billets, center position, material: A356
CHAPTER 3. RESULTS OF THE CASTING RUNS

Figure 3.6: Comparing microstructures and form factor distributions of commercially MHD and ThixoStruct cast billets, fringe position, material: A356
3.3. SUGGESTED IMPROVEMENTS

Billet are below ±5%, whereas the fluctuations of the MHD cast billets are much higher. This inhomogeneity regarding macrosegregations is much more frightening than the inhomogeneity in the form factor distribution. Macrosegregations lead to locally different forming behavior when formed in the semi-solid state. Moreover, other materials properties such as corrosion behavior strongly depend on local chemical composition.

Although the MHD technique has gained a vast propagation in the casting industry, these issues must be kept in mind when producing rheocast billets in great quantities.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Radial position</th>
<th>Silicon</th>
<th>Magnesium</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0 mm</td>
<td>6.63 w-%</td>
<td>0.37 w-%</td>
</tr>
<tr>
<td></td>
<td>15 mm</td>
<td>7.11 w-%</td>
<td>0.39 w-%</td>
</tr>
<tr>
<td></td>
<td>30 mm</td>
<td>6.96 w-%</td>
<td>0.39 w-%</td>
</tr>
<tr>
<td></td>
<td>45 mm</td>
<td>7.42 w-%</td>
<td>0.40 w-%</td>
</tr>
<tr>
<td>B</td>
<td>0 mm</td>
<td>6.52 w-%</td>
<td>0.33 w-%</td>
</tr>
<tr>
<td></td>
<td>15 mm</td>
<td>6.74 w-%</td>
<td>0.36 w-%</td>
</tr>
<tr>
<td></td>
<td>30 mm</td>
<td>7.50 w-%</td>
<td>0.38 w-%</td>
</tr>
<tr>
<td></td>
<td>45 mm</td>
<td>7.25 w-%</td>
<td>0.38 w-%</td>
</tr>
<tr>
<td>C</td>
<td>0 mm</td>
<td>7.17 w-%</td>
<td>0.38 w-%</td>
</tr>
<tr>
<td></td>
<td>15 mm</td>
<td>7.09 w-%</td>
<td>0.37 w-%</td>
</tr>
<tr>
<td></td>
<td>30 mm</td>
<td>7.29 w-%</td>
<td>0.35 w-%</td>
</tr>
<tr>
<td></td>
<td>45 mm</td>
<td>7.89 w-%</td>
<td>0.39 w-%</td>
</tr>
<tr>
<td>ThixoStruct</td>
<td>12 mm</td>
<td>6.97 w-%</td>
<td>0.43 w-%</td>
</tr>
<tr>
<td></td>
<td>39 mm</td>
<td>7.16 w-%</td>
<td>0.44 w-%</td>
</tr>
<tr>
<td></td>
<td>66 mm</td>
<td>6.69 w-%</td>
<td>0.44 w-%</td>
</tr>
<tr>
<td></td>
<td>93 mm</td>
<td>6.54 w-%</td>
<td>0.45 w-%</td>
</tr>
</tbody>
</table>

Table 3.3: Chemical composition of alloying elements at different radial positions for different feedstock material producers, material A356

3.3 Suggested Improvements

Although the ThixoStruct rheocaster yielded very promising results it must still be considered as a prototype. To produce effectively feedstock material with optimal thixotropic properties (i.e. a globular microstructure), the mass flow rate must be increased. One possible approach is a central heat exchange unit that feeds, for example, six symmetrically positioned hot top molds with the semi-solid slurry. To achieve this the heat exchange unit must be redesigned. The active cooling length must be increased to meet the higher demand for mass flow rate.

But there are also some possible simplifications. For example, there is no need for mounting several thermocouples because the radial heat transfer is now considered as well known. Furthermore, a high-sensitive control of the rotation speed of the motor direct drive and the possibility of torque measurements will not be necessary in an industrial rheocaster. It suffices to provide a motor drive with a constant revolution speed. Also the
variable adjustment of the gap width is negligible because the constant revolution speed of the agitator and the desired shear rate yields the optimal gap width.

Irrespective of the actual design of an industrial-sized rheocaster the cast billets must be processed further. They are sectioned to the desired size, heated up to the semi-solid temperature range and thixoformed to near-net-shape components. The next part of the thesis will deal with the distinctive forming behavior of the material in its semi-solid state.
Part II

Thixoforming: The Forming of Feedstock Material in the Semi-Solid State
"The grand aim of all science is to cover the greatest number of empirical facts by logical deduction from the smallest number of hypotheses or axioms."

Albert Einstein

4

Heating Up Feedstock Material

An important condition for thixoforming processing is a reliable reheating of the feedstock material. Prior to heating up, the material is sectioned corresponding to the part volume to be formed. To heat up the tailored slugs, mainly convective or inductive heating devices are used. The main advantage of the inductive heating process is the direct generation of heat within the slug due to induction by eddy currents. This effect provides a heating procedure that is rapid and relatively easy to control.

4.1 Processing Techniques

Although heat input within the solidus and liquidus temperature $T_S$ and $T_L$ for the partial melting of the slug is huge compared to heating in the fully solid state, this input yields only a small temperature increase of the slug. But a temperature increase of a few degrees centigrade in the semi-solid temperature range results in a considerable increase in fraction liquid. Therefore, besides exact temperature control, homogeneous temperature distribution within the slug must be ensured to guarantee uniform flow behavior during the subsequent forming process. Because it is not economical, and because there will be undesired particle growth during a long soaking period in the semi-solid state, temperature equalization should not take too long. Therefore, the slug should be submitted to a rapid heating up phase with high energy input. For the temperature equalization process in the semi-solid temperature range, heat input is reduced and the slug is soaked at constant temperature. During this soaking period the solid particles undergo microstructural evolutions that are a prerequisite for the distinctive thixotropic flow behavior. The next section will deal with these evolution processes.

The duration of the reheating process of a single slug is normally longer than the pulse period of forming machines. To ensure high productivity, several slugs must be heated up simultaneously, but staggered. One possible approach is the use of several generators,
each working with a constant capacity, where the slugs are transported with a conveyor system from one generator to the next. Corresponding to the necessary heating scheme, the first generators are operated with high capacities, whereas the following generators are operated with intermediate and lower capacities.

Because of the limited validity of the temperature in the semi-solid temperature range, measuring temperatures is not an appropriate method for getting information on the current state of the partial remelting process. Hirt et al. [29] presented a non-contacting measuring device that yields a signal correlating to the fraction liquid of the reheated slug. The robustness of the process can be increased significantly if this concept is introduced in the control of the reheating stage.

4.2 Microstructural Evolutions

The reheating stage does not only affect the fraction solid \( f_s \) but also the degree of microsegregation and the morphology of the solid phase. Current industrial practice uses rapid heating of the order of 200 \( K/\text{min.} \) This heating rate is so high that homogenization and melting of eventual non-equilibrium eutectic phases are negligible as long as the material is still in its solid state. But as soon as the temperature of the alloy exceeds the non-equilibrium eutectic temperature, non-equilibrium liquid is formed. It is common practice to soak the alloys at the forming temperature in order to promote globulization of the solid particles and to achieve uniform distribution of the liquid phase. Examples of homogenization times for various binary alloy systems with a globular microstructure can be found in [30].

The driving force for the microstructural evolution in the semi-solid state is the reduction of the interface area between the solid and the liquid phase. During partial remelting and subsequent isothermal soaking in the semi-solid state, coalescence processes precede coarsening phenomena. Coalescence takes place between parts of a non-ideal globular particle. As crystallographic orientation is the same throughout a particle, the process of intra-particle solid-state aggregation is extremely rapid. In the case of not perfectly globular particles, the mechanism of coalescence results in a relatively high quantity of intra-particle liquid that will not contribute to viscous flow in subsequent forming in the semi-solid state. Figure 4.1 shows this situation with a three-dimensional Scanning Electron Microscope (SEM) picture very well.

After the rapid coalescence phase the slower coarsening process by diffusion of solid matter can take place. During coarsening, the shape factor \( F \) of the solid particles rapidly approaches unity, corresponding to a perfectly globular microstructure. The classical relationship for coarsening is Ostwald's ripening law:

\[
d^3 - d_0^3 = Kt
\]  

where \( d \) is the mean diameter of the solid particles after coarsening time \( t \), \( d_0 \) is the original diameter of the solid particles, and \( K \) is a rate constant. In the case of purely diffusion-driven Ostwald ripening, \( K \) has the form [31]:

[31]:
4.2. MICROSTRUCTURAL EVOLUTIONS

Figure 4.1: SEM micrograph showing the occurrence of intra-particle liquid in the semi-solid. The liquid phase was etched away using Schmid’s tincture

\[ K = \frac{8}{9} \cdot \frac{T \cdot \Gamma \cdot D_L}{m_L \cdot (C_S - C_L)} \]  \hspace{1cm} (4.2)

where \( T \) is the temperature, \( D_L \) is the liquid diffusion coefficient, \( \Gamma \) is the capillary length, \( m_L \) is the slope of the liquidus curve and \( C_S \) and \( C_L \) are the equilibrium compositions of the solid and the liquid interface at the coarsening temperature. As pure coalescence is supposed to show a similar time dependence as diffusion-driven coarsening \([32,33]\), Wan and Sahm \([34]\) suggest that the contribution of both mechanisms is independent and that the effective rate constant \( K \) can be written as:

\[ K = K_C + K_O \]  \hspace{1cm} (4.3)

where \( K_C \) and \( K_O \) are the rate constants due to coalescence and Ostwald ripening, respectively.

The choice of processing parameters during reheating is therefore restricted by these two phenomena: the soaking time must be long enough to ensure a minimum of globulization of the solid particles, yet must be as short as possible in order to prevent too large a coarsening of the particles (see Figures 4.2 and 4.3).

During soaking in the semi-solid state, some alloys such as AA6082 show strong solid phase coarsening and coalescence effects with increasing soaking times. Gullo \([35]\) presented modified AA6082 alloys that show almost no microstructural changes during isothermal soaking, allowing the user to soak the billets over a longer period. This is due
to small additions of Ba, Fe, Cr, and Zr. Barium reduces solid-liquid interface energy, leading to an increased penetration of the liquid phase between contacts of solid particles. Moreover, these elements form dispersoids that inhibit grain growth. The combination of both effects leads to less sensitive microstructural evolutions during the soaking phase.

When cast alloys are reheated, an additional factor must be considered: in the eutectic phase melting should be complete and uniform. This boundary condition results in a minimal soaking time that is practically independent of the energy supply, as the evolution of the partial remelting is only dependent on heat absorption into the material. The following empirical dependence of the minimal soaking time \( t_s \) on the billet radius \( r \) was established [24]:

\[
t_s \approx C \cdot r^2
\]

(4.4)

where \( C \) is an empirical constant \((C = 0.3 \text{ for A356 and A357 alloys and } [ts] = s, [r] = mm)\). The minimal soaking time \( t_s \) is proportional to the square of the billet radius, which is normal for diffusion-controlled phenomena such as heat transfer.

When the material has the desired temperature and microstructure the actual thixoform process, forming in the semi-solid state, can take place. Modeling of this characteristic flow behavior is the subject of the next chapter.
Figure 4.2: Micrographs showing the effect of different soaking times on the coalescence and the coarsening of the solid particles, material A356, soaking temperature 580 °C, soaking times 0 minutes (top), 7 minutes (middle) and 15 minutes (bottom)
Figure 4.3: SEM micrographs of primary particles, showing the coalescence process between two neighboring particles
Models are to be used, not believed.

H. Theil

Modeling the Thixotropic Flow Behavior

A very important variable in thixoforming processes is the flow behavior of the semi-solid slurry. In order to model this characteristic feature, it is particularly important to investigate the factors that influence flow behavior: temperature is by far the most important influence because it determines the relative fractions liquid and solid. At low solid fractions the liquid/solid mixture acts as a nonlinear slurry whereas in the range of fraction solid above 0.5 there is a transition where the mechanism of flow changes from a suspension of solid particles to the plastic deformation of a connected particle network. Other factors such as particle morphologies will dictate the fraction solid where the transition into the plastic deformation regime takes place. The prior shear rate and temperature-time history is also a strong determinant of flow behavior. The aim of the proposed models is to combine all these influences with the help of a microstructure-based approach that incorporates the agglomeration ratio of the near-globulitic primary particles.

In order to fully exploit the advantages of thixoforming, the flow behavior of semi-solid alloys during the forming process must be understood. Although considerable research has been done on optimizing the forming technologies and the specific globular microstructure of the feedstock material, much less is known about how the flow behavior of the semi-solid slurries influences the quality of the formed part. For this reason, the objective of this part of the thesis is to model the thixotropic flow behavior of cast and wrought aluminum alloys (A356, AA6082 and AA7075). The constitutive model, as well as knowledge of related characteristic materials properties such as the agglomeration ratio of the primary particles, the specific heat capacity, the fraction liquid or thermal conductivity in the semi-solid temperature range, enable the materials scientist to optimize the thixoform process.

The constitutive behavior of thixotropic alloys in the semi-solid temperature regime
depends on a number of factors, including temperature, strain rate, temperature-time history and the degree of agglomeration among the primary particles.

![Graph showing thixotropic flow behavior](image)

Figure 5.1: Thixotropic flow behavior of A356 resulting from a compression test

It is well accepted that the flow stress $\sigma$ of alloys in their semi-solid state conforms with the thixotropic behavior shown schematically in Figure 5.1, due to agglomeration and disagglomeration phenomena during forming. The yield or peak stress $\sigma_{\text{max}}$ is a function of the initial microstructure, which itself is strongly dependent on the strain rate as well as the thermal and mechanical history of the investigated specimen. The steady-state stress $\sigma_{\infty}$ and the characteristic time $\tau$ also depend on the agglomeration and disagglomeration mechanisms. Such cross-linked phenomena require fundamental approaches and extensive experiments. A first possible level of simplification is to neglect the structural changes of the microstructure during forming and to assume a strain-independent flow stress that only varies with temperature and strain-rate [36].

### 5.1 Steady-State Approach

When the fraction solid $f_s$ is high, the plasticity theory can be modified to model the material as solid, with pockets of liquid phase. Therefore, to model the steady-state flow stress $\sigma_{\infty}$ in the semi-solid range, a constitutive hot forming approach, for example by Zener-Hollomon, [37] that describes the bulk hot forming behavior of metals can be used:

$$\sigma_{ZH} = \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\varepsilon_0} \cdot \exp \left(\frac{Q}{RT}\right) \right]^n$$

(5.1)
5.1. STEADY-STATE APPROACH

where $\sigma_0$ is the true stress at the solidus temperature $T_S$, $\dot{\varepsilon}$ the strain rate, $\dot{\varepsilon}_0 = 1 \text{ s}^{-1}$ serves for unit equation purposes, $Q$ is the activation energy for self-diffusion, $R$ the universal gas constant, and $m$ the materials exponent. The Zener-Hollomon approach was enhanced by a semi-solid scale factor $\delta$ [38] that describes the transition from the plastic deformation regime to nonlinear viscous slurry behavior:

$$\delta = 1 - (\beta f_L)^{2/3} \tag{5.2}$$

where $f_L$ is the fraction liquid. For ellipsoidal liquid pockets in a hexagonal representative volume element, $\beta$ yields 1.428 [38].

With the scaling factor $\delta$, the steady-state flow stress $\sigma_\infty$ can be described as follows:

$$\sigma_\infty = \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m \cdot \left[ 1 - (\beta f_L)^{2/3} \right] \tag{5.3}$$

Because the fraction liquid is, according to Equation 2.15, a function with discontinuities at the solidus $T_S$ and the liquidus temperature $T_L$, Equation 5.3 is difficult to describe and to handle in simulations. Furthermore, the assumption of ellipsoidal liquid pockets, on which the model is based, is no longer valid when the liquid fraction reaches a certain value $f_L^*$ where the percolation of the solid phase disappears, i.e. when there is no coherent solid skeleton anymore. For these reasons, the liquid fraction $f_L$ is replaced with a coefficient $\tilde{f}_L$ that is a) a continuous function of temperature and b) considers the percolation threshold $f_L^*$ above which the assumption of ellipsoidal liquid pockets is no longer valid and where the solid particles flow in a viscous liquid irrespective of the actual fraction liquid.

$$f_L \mapsto \tilde{f}_L = \frac{f_L}{1 + \exp \left( -a [T - T_C] \right)} \tag{5.4}$$

where $T_C = \frac{T_L + T_S}{2}$ and $a$ describes the slope of $\tilde{f}_L$ at $T_C$. Figure 5.2 shows the different concepts of the conventional fraction liquid $f_L$ and the modified coefficient $\tilde{f}_L$.

Substituting $f_L$ with $\tilde{f}_L$ yields for the steady-state flow stress $\sigma_\infty$:

$$\sigma_\infty = \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m \cdot \left[ 1 - \left( \beta \cdot \frac{f_L}{1 + \exp \left( -a [T - T_C] \right)} \right)^{2/3} \right] \tag{5.5}$$

Figure 5.3 shows the course of theoretical steady-state flow stress $\sigma_\infty$ for two aluminum alloys as a function of the forming temperature $T$.

The one-phase approach derived above considers the flow stress to be a function of temperature $T$ (or fraction liquid $f_L$) and strain rate $\dot{\varepsilon}$ only. It ignores thixotropy and the evolution of microstructural parameters. The next section will cover the topic of implementation of the thixotropy effect and structural evolution in the semi-solid state.
Figure 5.2: Comparison of the fraction liquid $f_L$ and the modified coefficient $\tilde{f}_L$.

Figure 5.3: Course of the flow stress $\sigma_\infty$ as a function of the forming temperature $T$ for A356 and AA6082, strain rate $\dot{\varepsilon} = 0.1 \, \text{s}^{-1}$. 
5.2 Transient Approach

In common thixoforming processes the partially remelted slug is rapidly formed or injected into a die cavity. Therefore, the strain rate changes rapidly from zero to large values depending on the forming velocity and geometry of the future component. The question now is whether the strain is great enough for the thixotropic material to reach steady-state conditions or whether it is still in a so-called transient behavior phase. In the first case, forming behavior can be described with a single-phase approach as discussed in the previous section. In the latter case, time-dependent mechanisms caused by structural changes must be considered [36]. In Figure 5.4 the flow stress $\sigma$ of A356 as a function of true strain $\varepsilon$ is shown. The specimen was compressed between two parallel plates.

![Flow curve of A356 resulting from a compression test, showing transient and steady-state behavior](image)

Figure 5.4: Flow curve of A356 resulting from a compression test, showing transient and steady-state behavior

During forming, the structure of the semi-solid slurry will change due to time-dependent processes that occur, including the agglomeration or disruption of particle bonds between particle agglomerates.

The time dependence of semi-solid slurries indicates that it is important to understand the different kinetic processes that govern slurry structure. Several investigators have shown that semi-solid slurries change flow stress much more rapidly after a strain rate increase than after a strain rate decrease [39,40]. It is generally accepted that the rate of particle agglomeration is substantially slower than the rate of disagglomeration. Furthermore, particle deformation and agglomeration can cause the fluid phase to be entrapped within and between particles. This increases the apparent fraction solid, resulting in an increase of flow resistance.
To capture these phenomena, an internal variable model\(^1\) was established [40]. Such a model uses two sets of equations, one set that represents the flow stress \(\sigma\) at a given structure \(s\) and a second that represents the evolution \(\dot{s}\) of the structure.

\[
\begin{align*}
\sigma &= f(\dot{\varepsilon}, T, t, s) \\
\dot{s} &= f(\dot{\varepsilon}, T, t, s)
\end{align*}
\]

where \(\dot{\varepsilon}\) is the strain rate and \(s\) is a scalar parameter indicating the degree of agglomeration \((0 \leq s \leq 1)\), e.g., the contiguity \(C_s\).

Ito et al. [42] observed that within the liquid phase of alloys in the semi-solid temperature regime there exist large agglomerates of solid primary particles. These agglomerates interact hydrodynamically with each other but also undergo plastic deformation. Therefore, the measured flow stress \(\sigma\) is due to hydrodynamic flow of agglomerates and deformation of solid particles within these agglomerates. \(s\) represents the degree of this agglomeration. When all particles are connected in a skeleton-like network then \(s = 1\), when none of the particles are connected then \(s = 0\). The effective flow stress \(\sigma\) derives from two independent mechanisms:

- Viscous effects due to hydrodynamic flow of solid particles in a fluid phase
- Disruption of bonded primary particles within the agglomerates

\(^1\) Internal variables are distinct from external variables such as temperature or strain rate in that internal variables cannot be directly imposed on the semi-solid slurry but instead evolve according to specific kinetic processes. Given these internal variables at any point, however, the state of the material can be completely characterized irrespective of its prior history [41].
5.2. TRANSIENT APPROACH

5.2.1 Hydrodynamic flow of solid particles

The relationship used for the effect of relative particle motion is governed by Krieger’s empirical law of viscosity $\eta$ for dense suspensions [43]:

$$\eta = \eta_0 \left(1 - \frac{f_s}{f_s^*}\right)^{-k} \quad (5.8)$$

where $\eta_0$ is the viscosity of the fluid, $f_s$ is the solid fraction of the suspension and $f_s^*$ is the onset of an interconnected network of primary particles. For $k$, Krieger suggests $k \approx 2.5f_s^*$ but for globular non-compact clusters, the value of $k$ is close to 2.5 [44].

Since this is the first mention of viscosity, a short theoretical input is appropriate.

Viscosity is defined as the resistance to flow in a fluid or, to express it more scientifically, a measure of the intensity of dissipation of energy that is necessary to maintain the irreversible deformation of a fluid [45]. For a Newtonian fluid the viscosity $\eta$ is linked to the stress $\sigma$ and shear rate $\dot{\gamma}$ by Newton’s law of viscosity:

$$\sigma = \eta \dot{\gamma} \quad (5.9)$$

Because all plasticity models described and derived within this thesis are based on the (elongational) strain rate $\dot{\varepsilon}$ and not on the shear rate $\dot{\gamma}$, the viscosity must be introduced as elongational viscosity. The relationship between shear viscosity $\eta_{\text{shear}}$ and elongational viscosity $\eta_{\text{elong}}$, can be described with the Trouton ratio $T$. For uniaxial extension or compression a Trouton ratio of $T = 3$ is obtained [45]:

$$\eta_{\text{elong}} = 3 \cdot \eta_{\text{shear}} \quad (5.10)$$

and therefore

$$\sigma = 3\eta \dot{\varepsilon} \quad (5.11)$$

Agglomerates of primary particles usually contain liquid phase between particles. This entrapped liquid decreases the effective fraction liquid and does not contribute to the viscous flow. Therefore the effective fraction solid $f_s^{\text{eff}}$ is higher than the actual fraction solid $f_s$. Kumar et al. [46] estimate the effective fraction solid with a model of a fully agglomerated structure. The effective fraction solid $f_s^{\text{eff}}$ can be expressed as:

$$f_s^{\text{eff}} = f_s + \frac{6(\sqrt{3} - \frac{2}{3})}{3\pi + (\sqrt{3} - \frac{2}{3})} \cdot s \cdot f_s \approx f_s (1 + 0.1s) \quad (5.12)$$

Combining Equations 5.8, 5.11 and 5.12 yields for the hydrodynamic part $\sigma_h$ of the flow stress $\sigma$:

$$\sigma_h = 3\eta_0 \dot{\varepsilon} \left(1 - \frac{f_s (1 + 0.1s)}{f_s^*}\right)^{-2.5} \quad (5.13)$$
5.2.2 Disruption of agglomerated primary particles

Besides the viscous effects due to hydrodynamic flow of solid particles in the fluid phase expressed by $\sigma_h$, there is also a plastic contribution $\sigma_p$ due to the disruption of bonded primary particles within the agglomerates. For $\sigma_p$, the approach by Zener-Hollomon is used, but keeping in mind that not the whole volume contributes to the plastic flow but only the bonds of connected particles:

$$\sigma_p = \int \frac{dV_b}{V_b} \cdot C_b \cdot \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\varepsilon_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m$$  \hspace{1cm} (5.14)

where $V_b$ is the volume of deforming metal in each bond\(^2\) and $C_b$ is the volume concentration of bonds. We assume that the effective deforming volume is a sphere with the radius $R_b$ of the bond:

$$V_b = \frac{4\pi}{3} R_b^3$$  \hspace{1cm} (5.15)

$C_b$ can be determined by simple consideration of the volume of connected particle pairs:

$$C_b = \frac{3}{8\pi} \frac{f_s \cdot s}{R^3}$$  \hspace{1cm} (5.16)

where $R$ is the radius of the globular solid particle.

Assuming a homogeneous distribution of particle bonds within a representative volume yields

$$\int \frac{dV_b}{V_b} = V_b$$  \hspace{1cm} (5.17)

and therefore, the plastic contribution $\sigma_p$ of the flow stress can be written as:

$$\sigma_p = \frac{f_s \cdot s}{2} \left( \frac{R_b}{R} \right)^3 \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\varepsilon_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m$$  \hspace{1cm} (5.18)

Finally, combining the effect of viscous flow of solid particles and the contribution due to plastic disruption of agglomerated particles yields the transient flow stress $\sigma_t$:

$$\sigma_t = \sigma_h + \sigma_p$$

$$\sigma_t = 3\eta_0 \dot{\varepsilon} \left( 1 - \frac{f_s (1 + 0.1s)}{f_s^2} \right)^{-2.5} + \frac{f_s \cdot s}{2} \left( \frac{R_b}{R} \right)^3 \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\varepsilon_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m$$  \hspace{1cm} (5.19)

\(^2\)Definition: when two globular particles are connected, they form a bond
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5.2.3 Evolution equation for the degree of agglomeration

The evolution of structure in a thixotropic semi-solid slurry can be described with static hardening (i.e. agglomeration) and flow softening (i.e. disagglomeration). We therefore assume that the evolution equation for the degree of agglomeration \( s \) is of the form:

\[
\frac{ds}{dt} = \dot{s} = A(s, t, T) + D(s, \dot{\varepsilon}, t) \tag{5.20}
\]

with the agglomeration function \( A \) and the disagglomeration function \( D \)

**Agglomeration function \( A \)**

The agglomeration of particles is assumed to result from particles bonding together. In the presented formulation, we assume that only welded pairs form. It is further assumed that the frequency of contact \( f_c \) is proportional to the shear rate \( \dot{\gamma} \). The longer two particles are in contact, the higher is the probability that they will form a bond strong enough to prevent subsequent disruption due to strain gradients. The important parameters for agglomeration are therefore:

- frequency of contact \( f_c \) between two particles,
- contact time \( t_c \), and
- volume fraction of unagglomerated particles

The probability of agglomeration \( p_A \) is considered to be proportional to the product of the frequency of particle contact \( f_c \), the time of contact \( t_c \) and the volume fraction of unagglomerated particles \((1 - s)\):

\[
p_A \propto f_c \cdot t_c \cdot (1 - s) \tag{5.21}
\]

For the frequency of contact \( f_c \), Mason and Bartok [47] proposed:

\[
f_c = \frac{8 f_s \cdot \dot{\gamma}}{\pi} \tag{5.22}
\]

Although Equation 5.22 was derived for small fractions solid \( f_s \) and collisions between single spheres it captures the characteristic feature that \( f_c \) increases with fraction solid \( f_s \) and shear rate \( \dot{\gamma} \). Mason and Bartok [47] also evaluated the average contact time \( t_c \) between non-interacting particles and determined the following relation:

\[
t_c = \frac{\pi}{\dot{\gamma}} \tag{5.23}
\]

During the contact, a rigid bond will be built between the two particles if a favorable crystallographic orientation is established. Martin et al. [48] suggest that only a low-angle
grain boundary ($\leq 0.25$ rad) favors bond growth. So, the probability for two particles to 
collide with a favorable crystallographic orientation is only $p_b = 0.02$ [49,50].

The initial contact between two isolated particles rapidly leads to a bond, i.e. a small 
neck. After the adhesion process, a diffusion-controlled neck growth follows. Neck growth 
is driven by difference in surface curvature. Solid matter will flow into the neck region 
from regions with a higher chemical potential. The step that controls the growth rate is 
the diffusion of solute atoms through the melt. One possible model for diffusion of solute 
atoms near the neck region is provided by Martin et al. [48]. The resulting formation time 
$t_f$ for a neck with a radius $R_b$ is

$$t_f = \frac{R^3 \cdot m_L \cdot (C_L - C_S)}{4D_L} \cdot \frac{\Delta S}{\gamma_{SL}} \cdot f\left(\frac{R_b}{R}\right)$$

(5.24)

where $R$ is the radius of a particle, $m_L$ the liquidus slope, $C_L$ the solute concentration 
in the liquid, $C_S$ the one in the solid, $\Delta S$ the entropy change per volume unit, $D_L$ the 
diffusion coefficient of the solute atoms in the liquid, and $\gamma_{SL}$ the solid-liquid interface 
tension.

$$f\left(\frac{R_b}{R}\right) = \frac{10}{3} + \frac{1}{1 - \frac{R_b}{R}} + 4 \ln \left(1 - \frac{R_b}{R}\right) - 6 \left(1 - \frac{R_b}{R}\right) + 2 \left(1 - \frac{R_b}{R}\right)^2 - \frac{1}{3} \left(1 - \frac{R_b}{R}\right)^3$$

(5.25)

Considering the assumptions and derived parameters mentioned above, the following 
formulation of the agglomeration function $A$ is proposed:

$$A = p_b \cdot f_c \cdot \frac{t_c}{t_f} \cdot (1 - s) \quad \therefore \quad A \neq f(\dot{\gamma})$$

(5.26)

With the same diffusion model Barbé et al. [44] were able to predict the maximal neck 
size $R^\text{max}_b$ and the maximal cluster size $R^\text{max}_c$ of agglomerated particles as a function of 
 shear rate $\dot{\gamma}$:

$$R^\text{max}_b \propto \dot{\gamma}^{-4/7}$$

(5.27)

$$R^\text{max}_c \propto \dot{\gamma}^{-3/7}$$

(5.28)

For shear rates $\dot{\gamma}$ in the order of $10^3$ s$^{-1}$, $R^\text{max}_b$ and $R^\text{max}_c$ are close to 10 $\mu$m and 100 
$\mu$m respectively.

Disagglomeration function $D$

The disruption of an agglomerate is due to the mechanical forces generated by stress on 
the agglomerated cluster. Therefore, the bond deforms by viscoplastic flow. A separation 
of the particles will occur when the strain reaches a critical value $\epsilon_{\text{crit}}$. The time $\tau_{\text{crit}}$ 
required for the strain to reach $\epsilon_{\text{crit}}$ is
5.2. TRANSIENT APPROACH

\[ \tau_{\text{crit}} = \frac{\epsilon_{\text{crit}}}{\dot{\epsilon}} \]  
(5.29)

Assuming that the change of aggregation \( \Delta s \) due to disruption of particle bonds is proportional to the actual agglomeration ratio \( s \), and considering that any period of time \( \Delta t \) scales with the ratio of \( \Delta \tau \) to the total time \( \tau_{\text{crit}} \) for complete separation, we obtain:

\[ \Delta s = -s \frac{\Delta t}{\tau_{\text{crit}}} \]  
(5.30)

Combining Equations 5.29 and 5.30 we obtain

\[ \frac{\Delta s}{\Delta t} = -\frac{\dot{\epsilon}}{\epsilon_{\text{crit}}} s \]  
(5.31)

Therefore, the disagglomeration function \( D \) of the evolution equation (5.20) is given by

\[ D = \dot{s} = \lim_{\Delta t \to 0} \frac{\Delta s}{\Delta t} = -\frac{\dot{\epsilon}}{\epsilon_{\text{crit}}} s \]  
(5.32)

Combining all equations concerning the evolution equation yields

\[ \frac{ds}{dt} = \dot{s} = p_b \cdot \frac{s f_s}{t_f} \cdot (1 - s) - \frac{\dot{\epsilon}}{\epsilon_{\text{crit}}} s \]  
(5.33)

Equation 5.33 is a differential equation linear in \( s \), and a linear evolution equation will lead to a simple exponential law for the structural parameter \( s \):

\[ s(t) = \left( s_0 - \frac{a}{a - d} \right) \cdot \exp \left( [d - a] t \right) + \frac{a}{a - d} \]  
(5.34)

\[ a = \frac{8p_b f_s}{t_f} \]  
(5.35)

\[ d = -\frac{\dot{\epsilon}}{\epsilon_{\text{crit}}} \]  
(5.36)

with \( s_0 \) as the initial degree of agglomeration.

Mada and Ajersch [51] formulated a similar kinetic evolution equation using the analogy of the chemical kinetics to express the rate of disagglomeration and agglomeration:

\[ -\frac{ds}{dt} = k_1 (s - s_e)^p - k_2 (s_0 - s)^q \]  
(5.37)
where $k_1$ and $k_2$ are the corresponding kinetic constants, $p$ and $q$ are the reaction orders of the disagglomeration and agglomeration process respectively, and $s_e$ is the agglomeration ratio at equilibrium or steady-state conditions. It was experimentally determined [39] that $p$ equals 1 and that the rate of agglomeration is negligible with respect to the rate of disagglomeration when sheared, so that $k_1 > k_2$. This kinetic equation can be integrated, assuming that at $t = 0$, $s = s_0$:

$$s(t) = (s_0 - s_e) \cdot \exp(-k_1 t) + s_e \quad (5.38)$$

Comparing Equation 5.38 with Equation 5.34 we obtain for the kinetic constant $k_1$ and for the steady-state agglomeration degree $s_e$:

$$k_1 = a - d = \frac{8p_b f_s}{l_f} + \frac{\dot{\varepsilon}}{\varepsilon_{crit}} \quad (5.39)$$

$$s_e = \frac{a}{a - d} \quad (5.40)$$

### 5.2.4 Thixo-Elastic Behavior

In the case of rapid transient experiments such as compression tests where the strain is large enough to promote structural rupturing of the material, the need for a model that combines viscoelasticity and thixotropy appears obvious. But, is it possible to distinguish the viscoelastic contribution from the thixotropic one? It depends on the relative importance of the three different characteristic times of the processes involved, i.e.:

- the Maxwell time $\tau_M$ for initial viscoelastic relaxation,
- the thixotropic time $\tau_T$, characterizing structural change,
- and the experimental time $\tau_E$, the duration of strain rate application.

The viscoelastic part $\sigma_v$ of the total flow stress $\sigma$ can be described with:

$$\sigma_v = \sigma_0 \cdot (1 - \exp(-t/\tau_M)) \quad \text{with} \quad \tau_M = \eta/E \quad (5.41)$$

where $E$ is the Young’s modulus and $\eta$ is the viscosity of the investigated material.

The thixotropic time $\tau_T$ is, according to Equation 5.38:

$$\tau_T = \frac{1}{k_1} = \frac{1}{a - d} \quad (5.42)$$

Finally, the experimental time $\tau_E$ can be calculated in the case of compression tests (with final strain $\varepsilon_{\text{final}}$) with:

$$\tau_E = \frac{\varepsilon_{\text{final}}}{\dot{\varepsilon}} \quad (5.43)$$
5.3. TWO-PHASE APPROACH

If $\tau_M$ and $\tau_T$ are very different (generally, $\tau_M \ll \tau_T$) it will be very easy to separate the elastic behavior (observed on short time scales, $\tau_E \approx \tau_M$) from the thixotropic one (observed on large time scales, $\tau_E \approx \tau_T$). On the contrary, if $\tau_M$ and $\tau_T$ have the same order of magnitude, viscoelasticity and thixotropy will be superimposed. In the case of the compression test (see also Figure 5.4), the following values of the characteristic times were derived:

- $\tau_M \approx 0.25 \text{ s}$
  with $\eta \approx 10^7 \text{Pa} \cdot \text{s}$ and $E \approx 40 \text{ GPa}$ [52] for $T = 570 \degree \text{C}$ and $f_S = 100\%$
- $\tau_T = 1.5 \text{ s}$ for a compression test with $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ and $T = 570 \degree \text{C}$
- $\tau_E = 5.0 \text{ s}$

The difference between $\tau_M$ and $\tau_T$ is less than an order of magnitude and therefore we are confronted with a superposition of viscoelastic and thixotropic processes during compression tests. Combining the viscoelastic concept of $\sigma_v$ (see Equation 5.41) with the plastic contribution $\sigma_p$ of the transient response $\sigma_t$ (see Equation 5.19) we obtain:

$$\sigma_v = \sigma_0 \cdot \exp \left( -\frac{\dot{\varepsilon}}{\dot{\varepsilon} \cdot \tau_M} \right)$$ (5.44)

$$\sigma = 3 \eta \dot{\varepsilon} \left( 1 - \frac{f_S (1 + 0.1s)}{f_S} \right)^{-2.5} +$$

$$\frac{f_S \cdot s}{2} \left( \frac{R_b}{R} \right)^3 \sigma_0 \left[ \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \cdot \exp \left( Q/RT \right) \right]^m \left( 1 - \exp \left( -\frac{\dot{\varepsilon}}{\dot{\varepsilon} \cdot \tau_M} \right) \right)$$ (5.45)

Figure 5.6 shows the close agreement of the proposed thixoelastic model with experimental data of a compression test.

5.3 Two-Phase Approach

During semi-solid processing, the segregation of liquid phase is often observed. This phenomenon is pronounced when the primary particles deviate from the globular form (e.g. dendrites) or when the fraction solid $f_S$ is high. There is a characteristic transition from liquid-like behavior where solid particles are embedded in the liquid phase and two-phase solid/liquid behavior. In this case, a percolated solid skeleton has formed that responds to applied forces by plastic deformation. The reduction of interstitial liquid volume will lead to the development of hydrostatic pressure in the liquid phase. When there are gradients of this hydrostatic pressure, phase segregation will occur. The relationship between the gradient in the hydrostatic pressure and the different velocity between liquid and solid phases is expressed by Darcy's law that introduces the concept of permeability $k$. The permeability describes the ease of liquid flow through the percolated solid skeleton.

3The hydrodynamic part $\sigma_h$ of $\sigma_t$ is irrelevant of time-dependent viscoelastic and thixotropic structural changes.
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Transient model

Thixoelastic model

Experimental data

Figure 5.6: Comparison of the thixoelastic model with experimental data (compression test with strain rate $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ and forming temperature $T = 570 \degree \text{C}$)

\[
q = -\frac{k}{\mu} \nabla p
\]  

(5.46)

where $q$ is the flux of the liquid phase through the porous solid skeleton, $k$ is the permeability, $\mu$ is the dynamic viscosity of the fluid, and $p$ is the fluid pressure.

The main disadvantage of the two-phase approach is that the experimental characterization is very complex and difficult. Although the next part of the thesis, the simulation of thixoforming processes, is based on the one-phase transient approach and not on the two-phase approach, some conditions should be mentioned that promote segregations [36]:

- High fractions solid $f_s$
- Large and non-globular primary particles
- Low strain rates

5.4 Experimental Verification

For the determination of the mechanical properties of a semi-solid alloy, various types of experiments can be conducted. Although there exist straightforward possibilities to determine these properties, the following boundary conditions must be kept in mind [36]:

5.4. EXPERIMENTAL VERIFICATION

- The experiment must be representative for the investigated forming process,
- the occurrence of liquid segregation and strain localization must be paid attention to when the determination of flow stresses is involved,
- the microstructures of the specimens should not be different to the processed ones.

In the following sections an overview of the two used experiments is presented together with the main advantages and drawbacks of each one.

5.4.1 Compression Tests

Flow Stress Determination

In the conventional compression test, a cylindrical sample with an initial height \( h_0 \) is compressed between two dies. The compression force \( F \) and the decreasing height \( h(t) \) as a function of time \( t \) are measured. The cylindrical compression test is a common method of investigating materials in Forming technology. Its essential advantages are the rather simple manufacturing of the specimens as well as the direct conversion of the decreasing height \( h \) and the compression force \( F \) into true strain \( \varepsilon \) and true stress \( \sigma \). In the ideal case, an uniaxial stress affects the circular surface of the cylindrical specimen. Radial and tangential stresses are nil. Neglecting corrections of the Tresca flow criterion, the true strain \( \varepsilon \) can be written as

\[
\varepsilon = \ln \frac{h_0}{h(t)} \quad (5.47)
\]

The flow stress \( \sigma \) can be written in an approximation as

\[
\sigma = \frac{F(t)}{A(t)} \quad (5.48)
\]

where

\[
A(t) = \pi \cdot \frac{D(t)^2}{4} \quad (5.49)
\]

where \( D(t) \) is the time dependent mean diameter of the specimen. According to the law of constancy of volume

\[
D_0^2 h_0 = D(t)^2 h(t) \quad (5.50)
\]

where \( D_0 \) is the initial diameter of the uncompressed specimen. Assuming that the deformation is homogeneous, the flow stress \( \sigma \) can be described as a function of the compression time \( t \):
\[ \sigma = \frac{4 \cdot F(t) \cdot h(t)}{\pi \cdot D_0^2 \cdot h_0} \quad (5.51) \]

Thus, the flow stress \( \sigma \) and the true strain \( \epsilon \) can be calculated from the experimentally measured force-time curve \( F(t) \) and the decreasing height \( h(t) \) using Equations 5.47 and 5.51.

**Experimental Procedure**

Cylindrical compression tests of various alloy designations (e.g. A356, AA6082, AA7075 etc.) with a height of 10 \( \text{mm} \) and a diameter of 5 \( \text{mm} \) were machined from the as-cast billet. Isothermal hot uniaxial compression tests at constant strain rates \( \dot{\varepsilon} \) were carried out on a high-speed deformation dilatometer Bähr DIL805A/D. Figure 5.7 shows a general view of the dilatometer.

![Figure 5.7: High-speed deformation dilatometer Bähr DIL805A/D](image)

It is able to carry out tests with a controlled constant strain-rate of up to 12 \( s^{-1} \). In order to reduce friction between the specimen and the alumina dies, Molykote HSC plus was used as lubricant. To ensure a homogeneous temperature, molybdenum platelets with a diameter of 8 \( \text{mm} \) and a thickness of 0.3 \( \text{mm} \) were applied on the faces of the sample in order to minimize the heat flow into the dies. The tests were conducted in a vacuum chamber equipped with an inductive heating system (see Figure 5.8).

As atmosphere, high purity argon gas was chosen with a pressure of approximately 800 \( \text{mbar} \), inflated after the evacuation of the chamber to 10^{-4} \( \text{mbar} \). The length change was measured by a Linear Variable Differential Transducer (LVDT). The specimens were
5.4. EXPERIMENTAL VERIFICATION

Figure 5.8: Vacuum chamber of the deformation dilatometer (left) and detail of specimen with thermocouples, alumina dies and inductive heating coil (right)

heated with a constant heating rate of 5 K/s. Prior to compression, each specimen was soaked for 5 minutes at the test temperature to equalize thermal gradients within the sample. During the compression test, load and length change were recorded by a computer and later converted to flow stress curves. The samples were deformed to a true strain $\varepsilon$ of 0.5. Immediately after the compression test, the specimens were cooled down with forced argon gas.

Results

Figure 5.9 shows three flow curves of the wrought aluminum alloy AA7075 recorded at three different temperatures. It shows transient behavior of the flow stress with a characteristic peak stress and the asymptotic approach to a steady-state value. The higher the temperature $T$, i.e. the fraction liquid $f_L$, the lower the peak and the steady-state stress.

The aluminum alloy AA7075 is suitable for showing the disadvantages of the compression test. Plotting the steady-state stress values as a function of temperature (see Figure 5.10) reveals an abrupt and unexpected decrease of the steady state stress at a temperature of approx. 475 °C. The reason for this behavior is the melting of the ternary eutectic phase in the unhomogenized sample. A DTA measurement confirms the suspicion. At a temperature of 480 °C approx. 4 vol.-% of the material melts (see Figure 5.11). This partial melting leads to a lubrication between the solid phase and results in a strain localization along this liquid film. And because the deformation of the compressed specimen is not hindered in the radial direction, the deformation is not homogeneous anymore, resulting in the observed drop of the flow stress. If the process to be modeled does not possess the possibility of an unresisted flow in the direction perpendicular to the main forming direction (e.g. extrusion) an alternative way of providing flow stress data must be ensured.
Figure 5.9: Flow curves of AA7075 at three different temperatures in the semi-solid range, strain rate: $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$

Figure 5.10: Unexpected softening at $T=475^\circ\text{C}$. Plot of the steady-state stress as a function of temperature $T$, strain rate $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$
5.4. EXPERIMENTAL VERIFICATION

The experimental setup for the backward extrusion experiments is also known as Gullometer.

5.4.2 Backward Extrusion Experiments

One possible alternative experimental technique to the compression tests are backward extrusion experiments. A cylindrical specimen with a diameter of 30 mm and a length of 30 mm is heated and partially remelted in a vertical infrared tube furnace. After soaking at predefined temperatures the specimens were extruded backwards against a stationary plunger with a constant ram speed. The ram displacement $x$ and the extrusion force $F$ were measured as a function of time $t$. Figure 5.12 shows the experimental setup\(^4\). The main advantages compared to the compression test are:

- The material flow is similar to the one in industrial thixoforming processes, i.e. the material flow is restricted to the extrusion direction,
- the resulting strain rates correspond well to the ones aimed at the industrial processes, and
- the specimen volume is approx. 30 times larger than that of the compression specimens, resulting in a more plausible adaption of experimental flow stresses to real forming conditions.

But, the backward extrusion process possesses also some drawbacks:

- Contrary to the compression tests, there exists friction in the axial direction along the outside wall of the plunger. The experimental determination of the friction

\(^4\)The experimental setup for the backward extrusion experiments is also known as Gullometer.
Figure 5.12: Experimental setup of the backward extrusion experiments [28]
forces is not possible and therefore friction models must be incorporated in order to extract the flow stress values.

- The temperature distribution in the backward extrusion setup is not isothermal. Neither the punch nor the plunger possess the same temperature as the specimen.

Despite the disadvantages of unknown heat transfer into the tools and unknown friction coefficients along the plunger, the backward extrusion experiments yield very valuable information concerning the onset of thixotropic behavior, and comprise an appropriate forming process for modeling issues. That is why the thixotropic flow model derived above was first checked and calibrated with backward extrusion experiments (see Chapter 8). Figure 5.13 shows different backward extrusion experiments with the aluminum alloy A356 at different temperatures $T$, showing the onset of pronounced thixotropic flow behavior at temperatures above 570 °C.

![Figure 5.13: Extrusion forces $F$ of backward extruded specimens as a function of punch displacement $x$, material A356, punch speed 20 mm/s](image)
"Hell must be isothermal; for otherwise the resident engineers and physical chemists (of which there must be some) could set up a heat engine to run a refrigerator to cool off a portion of their surroundings to any desired temperature."

Henry A. Ben

Modeling other Thermal Variables

6.1 Fraction Solid

Although the flow stress of an alloy in the semi-solid state is a very important modeling variable, the accurate evaluation of the fraction solid \( f_S \) as a function of temperature is necessary, since this parameter controls as well to a large extent thixotropic behavior in the semi-solid state. The fraction solid can be evaluated either directly or through its effects on a physical property. The following experimental techniques are possible methods [53]:

- utilization of thermodynamic data (equilibrium phase diagrams, ThermoCalc),
- thermal analysis techniques,
- quantitative metallography of microstructures quenched from the semi-solid state, and
- measurement of mechanical response (compression tests, backward extrusion experiments).

The disadvantage of the last method is the requirement of calibration and the lack of correspondence between the mechanical response and the fraction solid \( f_S \), because the mechanical properties of semi-solid slurries also depend strongly on the microstructure. For these reasons this thesis focuses on the first three methods.

6.1.1 Thermodynamic Data

For alloys that melt or solidify under equilibrium conditions, the fraction solid \( f_S \) can be calculated directly based on phase diagrams. For simple binary eutectic alloys, the lever
rule yields the fraction solid at a given temperature $T_R$ in the semi-solid range. Assuming that the solidus and liquidus lines of the phase diagram are linear yields:

$$f_S^{eq} = \frac{(T_M - T_R) - m_L C_0}{(T_M - T_R)(1 - k)} \quad (6.1)$$

where $T_M$ is the melting point of the pure solvent, $m_L$ is the slope of the liquidus line, $C_0$ is the alloy composition and $k$ is the partition coefficient of the investigated alloy.

The phase diagram can be used for non-equilibrium conditions as well when there is complete diffusion in the liquid phase and no diffusion in the solid phase. For determination of the solid fraction $f_s$ at a certain temperature $T_R$ the non-equilibrium lever rule, or Scheil equation, can be used:

$$f_s^{Sch} = 1 - \left(\frac{T_M - T_R}{T_M - T_L}\right)^{-\frac{1}{1-k}} \quad (6.2)$$

where $T_L$ is the liquidus temperature of the alloy.

For calculation of the fraction solid $f_s$ in complex multi-component alloy systems there exist thermodynamic databases such as ThermoCalc. ThermoCalc utilizes equilibrium phase diagrams and the Scheil equation for the prediction of solid fractions. Figure 6.1 shows the calculated pseudo-binary phase diagram and the evolution of the solid fraction of the aluminum alloy AlSi7Mg0.38.

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Figure 6.1: Calculated pseudo-binary phase diagram for AlSi7Mg0.38 (left) and the evolution of the fraction solid $f_s$ as a function of temperature $T$

1http://www.thermocalc.com
Thermodynamic databases can be used to calculate the solid fraction during melting if the alloy is near equilibrium or if a model that governs the thermal history of the alloy exists. Therefore, there is need for experimental techniques that predict directly the evolution of the fraction solid for conditions similar to them of the investigated process. Nevertheless, this method is a valuable tool for materials selection and alloy design.

6.1.2 Thermal Analysis Techniques

Thermal analysis techniques such as Differential Scanning Calorimetry (DSC) and DTA can be considered as "traditional" methods to determine the evolution of fraction solid during melting or solidifying. After the measurement of the latent heat of melting, the fraction liquid \( f_L \) is calculated using the method of partial areas (see Figure 6.2).

Although DSC has an advantage compared with DTA, as it measures directly the evolution of the heat of melting during the solid-liquid phase transformation, the DTA was used because of the capability to test larger specimens, which minimizes the possibility of obtaining non-representative results due to local variations of composition. Although the determination of the fraction solid evolution during heating up and cooling down is straightforward, there are disadvantages. If we compare the high heating rates used in semi-solid processing (up to 200 \( K/min \)) and the rather slow rates used in the DTA tests (1–20 \( K/min \)), we find that thermal analysis techniques do not simulate industrial conditions very accurately, as they cannot achieve such high heating rates. But the DTA and DSC methods provide useful information on characteristic temperatures such as the liquidus and solidus temperature \( T_L \) and \( T_S \) or the eutectic temperature \( T_E \), which can be used to identify the occurrence of non-equilibrium phases in an alloy (see Figure 5.11). Thermal analysis can also be used to compare the melting kinetics of similar alloys and to provide information that otherwise would be impossible to obtain.

6.1.3 Quenching Experiments

Quenching experiments are used to freeze the microstructure in the semi-solid state and to permit the evaluation of the fraction solid \( f_S \) using quantitative metallography. To minimize the extent of dendritic solidification, the cooling rate during quenching must be very fast. To calculate the fraction solid from images of the quenched microstructure, image analysis is used in combination with optical microscopy. The experimental setup is shown in Figure 6.3.

A vertical infrared tube furnace similar to that used for the backward extrusion experiments was used to heat the sample to the desired temperature. The cubic sample with a side length of approx. 1 cm was placed inside the furnace on top of a small steel plate. A thermocouple was placed inside a hole located on the top surface of the sample to ensure that the sample reached the predefined temperature. The material was heated to the quenching temperature and soaked for 5 to 15 minutes before quenching, to ensure uniform partial melting. After soaking, the plate was removed and the sample fell into the water quencher. Besides the use for the measurement of the fraction solid this method offers a significant advantage: it reveals the microstructure in the semi-solid state, i.e. the morphology of the solid phase and the spatial distribution of the phases. This informa-
Figure 6.2: DTA curve of A356 (top) and the evaluation of fraction liquid $f_L$ using the method of partial areas (bottom)
6.2 Specific Heat Capacity

For a model that describes thixotropic behavior, the effect of latent heat release must be incorporated as well. Two schemes have been widely used: they are the effective specific heat capacity and the thermal conductivity.
Figure 6.4: Quenched microstructures of A356, soaking time 15 minutes, soaking temperatures: 570 °C (top), 575 °C (middle) and 580 °C (bottom)
heat method [54,55] and the enthalpy method [56,57]. Usually, in the literature only a total amount of latent heat release during the entire solidification process is given, but the actual rate of latent heat release is not available. The energy equation for an alloy undergoing solidification\(^2\) can be expressed by [58]:

\[
\rho c_p \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left( k \frac{\partial T}{\partial x} \right) + \rho G
\]  

(6.3)

where \(\rho\) is the density, \(c_p\) is the specific heat, \(k\) is the thermal conductivity, and \(G\) is the rate of energy generation owing to solidification. It is commonly assumed that latent heat is released proportionally to the rate of formation of the solid fraction \(f_s\). Hence \(G\) can be expressed by

\[
G = L \frac{\partial f_s}{\partial t}
\]  

(6.4)

where \(L\) is the total latent heat release during solidification.

If the enthalpy method is used, the two equations above can be combined:

\[
\frac{\partial H}{\partial t} = \frac{\partial}{\partial x} \left( k \frac{\partial T}{\partial x} \right)
\]  

(6.5)

where \(H\) is the enthalpy of the alloy that is defined by

\[
H = \int c_p \partial T + L(1 - f_s)
\]  

(6.6)

On the right hand side of Equation 6.6 the first term represents sensible heat, whereas the second term is latent heat.

If the modified specific heat method is used, by combining Equations 6.3 and 6.4 the governing equation becomes

\[
\rho c_p^* \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left( k \frac{\partial T}{\partial x} \right)
\]  

(6.7)

where \(c_p^*\) is the modified specific heat including both sensible heat and latent heat, and which is defined by

\[
c_p^* = c_p - L \frac{\partial f_s}{\partial T}
\]  

(6.8)

To include latent heat effects in both methods, a relationship between the temperature \(T\) and the fraction solid \(f_s\) must be provided, especially near the eutectic point for a

\(^2\)The same assumptions are valid for melting, but for the sake of simplicity only the solidification process is considered here.
eutectic alloy. As described in Section 6.1 the total latent heat of an alloy can be obtained through thermal analysis instruments such as DSC or DTA.

The procedure for calculating the latent heat from the experimental DTA heating and cooling curves can be summarised as follows: collection of curve data; calculation of the first derivative of temperature with respect to time; identification of the solidus and liquidus temperatures $T_S$ and $T_L$; obtaining the null curve\(^3\); and calculation of latent heat.

With the calculated latent heat $L$, the modified specific heat can be evaluated directly from the DTA experiments. Figure 6.5 depicts such a measured curve. For the approximation of measured specific heat capacities $c_p$, a set of Lorentz functions are suitable:

\[
c_p = c_p^0 + \sum_{i}^{n} \frac{a_i}{k_i + (T - T_i)^2}
\]

(6.9)

Where $c_p^0$ is the specific heat capacity of the investigated alloy at room temperature, $n$ the number of peaks, $a_i$ the relative peak maxima, $k_i$ the peak form parameter, and $T_i$ are the temperatures at the respective peak maxima.

![Figure 6.5: Measured course of the specific heat capacity $c_p$](image1)

In Figure 6.6 an approximated curve using Lorentz functions is shown together with an experimental DTA curve.

---

\(^3\)The null curve is an experiment with empty crucibles to measure intrinsic DTA signals caused by the experimental setup.
6.3. THERMAL CONDUCTIVITY

Another possible way to obtain information on the fraction solid with respect to temperature, as described in Chapter 6.1, is the integration of the specific heat curve. The amount of fraction liquid \( f_L = 1 - f_S \) as a function of temperature can easily be calculated by integration of the temperature-dependent specific heat capacity \( c_p \), assuming that the latent heat is evenly released over the whole temperature range, i.e. the amount of latent heat of the eutectic phase is considered as the same as the one of the primary phase.

\[
    f_L = 1 - f_S = \frac{\int_{T_S}^{T} (c_p - c_p^0) \cdot d\theta}{\int_{T_S}^{T_L} (c_p - c_p^0) \cdot d\theta} \quad (6.10)
\]

\[
    f_L = \frac{1}{2} + \frac{1}{L} \sum_{i}^{n} \left( \frac{a_i}{\sqrt{k_i}} \cdot \arctan \left( \frac{r - T_i}{\sqrt{k_i}} \right) \right) \quad (6.11)
\]

The results of this approximation are in agreement with results from DTA experiments (see Figure 6.7).

6.3 Thermal Conductivity

The change of aggregation (solid to liquid and vice versa) must be considered also with regard to the thermal conductivity: molten matter possesses a lower thermal conductivity than solid matter. To model this drop of thermal conductivity at temperatures above the solidus temperature \( T_S \), a sigmoidal course of the thermal conductivity \( k \) is suggested (see Figure 6.8):
CHAPTER 6. MODELING OTHER THERMAL VARIABLES

Figure 6.7: Evolution of fraction liquid $f_L$ using Equation 6.11 compared with results from DTA experiments

\[ k = k_L + (k_S + k_L) \cdot \left( 1 - \frac{1}{1 + \exp \left( -b [T - T_c] \right)} \right) \quad (6.12) \]

Where $k_L$ and $k_S$ are the thermal conductivities of the liquid phase and the solid phase respectively, and $b$ is the $c$-constant.

The critical issue here is how to account for convection in the liquid and in the semi-solid temperature regime. During thixoforming, thermal energy is transported not only by conduction but also by convection. Complete modeling of this convective transport requires solving of the difficult fluid-flow problem in the liquid phase. This complexity has not been included in this thesis. The enhanced heat transfer due to convection could be modeled by artificially increasing the value of the thermal conductivity $k$. This increase defines an effective thermal conductivity $k_{\text{eff}}$ and increases the rate of heat transfer, having an effect similar to that of actual convective heat transfer. For temperatures greater than the liquidus temperature $T_L$, Lally et al. [60] proposed for the effective thermal conductivity:

\[ k_{\text{eff}} = k_c \cdot k_S(T) \quad \forall \ T \geq T_L \quad (6.13) \]

where $k_c$ is the multiplying factor that accounts for convection and has a value between 5 and 10 [61,62] and $k_S(T)$ is the temperature dependent thermal conductivity of the
6.3. THERMAL CONDUCTIVITY

Figure 6.8: Suggested course of the thermal conductivity $k$ of the aluminum alloy A356 compared with experimental data [59]

solid. For temperatures between the solidus $T_S$ and the liquidus $T_L$, Gabathuler et al. [63] proposed:

$$ k_{\text{eff}} = k_S(T) \cdot \left[ 1 + (k_c - 1) \cdot \left( \frac{T - T_S}{T_L - T_S} \right)^2 \right] \quad \forall \ T_S \leq T < T_L \quad (6.14) $$

In the next chapter, the implementation of all the derived temperature-dependent and thixotropic variables and models into the special-purpose FE software package ThixoForm will be described.
Part III

Process Simulation: The Virtual Processing of Thixoformed Components
"It would appear that we have reached the limits of what it is possible to achieve with computer technology, although one should be careful with such statements, as they tend to sound pretty silly in 5 years."

John von Neumann (1949)

Simulating Thixoforming Processes

The Finite Element (FE) method has been recognized as a powerful tool for simulating forming processes for more than twenty years. Based on the computational results, the engineer is able to predict the failures that may occur during processes and to change the parameters to improve the processes. Due to the rapid advances in computer technology, the computation time of FE simulations has been shortened significantly and the accuracy of the simulated results is continually increasing. However, the application of FE simulation in the forming industry, especially in the thixoforming industry, is still limited. Difficulties arise, on the one hand, from the enormous computational costs, and, on the other hand, from the large deformations taking place inside the component during the forming process. When the mesh is updated according to the material flow, it quickly becomes strongly distorted. This phenomenon causes the computation to break down or leads to unreliable results. So, for the FE simulation of thixoforming processes the Arbitrary Lagrangian-Eulerian (ALE) formulation can be applied. The nodes are divided up into two groups, namely surface nodes and internal nodes. The surface nodes are updated as a Lagrangian mesh and the internal nodes act as an Eulerian mesh. The distortion of the mesh is eliminated since the Eulerian mesh remains regular and it is relatively easy to adjust the surface nodes. The FE program ThixoForm has been developed according to this concept as an easy to use special-purpose package taking into account the distinctive flow behavior of thixotropic alloys in their semi-solid temperature regime. ThixoForm is a further development of the well-established PressForm software package. The preprocessor of PressForm is able to generate the computational mesh as well as the discretization of the tools. A preconditioned conjugate gradient iterative solver together with the element-by-element method is used to solve large systems of linear equations. For the simulation of thixoforming processes the mesh is also automatically controlled and regenerated where necessary.
CHAPTER 7. SIMULATING THIXOFORMING PROCESSES

7.1 Principles of Finite Element Simulations

7.1.1 Finite Elements

It is not the aim of this thesis to provide a thorough introduction into the method of FE. More information on this topic and the basics of PressForm in particular can be found in [64]. Nevertheless, two FE-specific characterizations should be mentioned:

- ThixoForm applies the implicit FE method.
- ThixoForm is based on the ALE formulation.

In order to describe the deformation as well as various state variables, two methods have been introduced in continuum mechanics, namely the Lagrangian and the Eulerian methods. The Lagrangian method uses the undeformed state as a reference configuration. Because the initial configuration always refers to the same material particle, it is also called the material description. The Lagrangian method uses a material related mesh; each node in the FE mesh has a corresponding material point. When the material point moves, the node must follow and moves to the same position. Therefore, the movements of the domain are well simulated by the FE mesh and a deformation history-dependent constitutive relation such as the thixotropic flow behavior is conveniently established.

Another way to describe continua is to express the initial configuration as a function of the current position. Hence, in the Eulerian formulation, a position \( x \) is occupied by different material particles as the material moves continually. \( x \) works as a reference frame in space. Therefore, the Eulerian method is also called the spatial description; the Eulerian method uses a stationary mesh which is independent of material movement. The computational results are the velocities at the nodes and the deformation rates in the elements. The velocities at the nodes are also the velocities of the material particles that currently occupy the position of the nodes. Because the elements are occupied by different parts of material, the deformation history of the material is not directly included. Hence, the ALE method has been developed [65] to combine the advantages of both methods and to remedy their deficiencies. The basic idea of the ALE method is to uncouple the updating of the mesh nodes from the material flow in order to avoid strong mesh distortion. Compared with the Eulerian description the ALE method provides more flexibility.

7.1.2 Stress and Strain Tensors

ThixoForm uses the rigid-plastic description of the stress tensor. The rigid-plastic description ignores elastic strains \( \epsilon_{ij} \) and connects directly the strain increments \( \Delta \epsilon_{ij} \) with the deviatoric stresses \( \sigma'_{ij} \):

\[
\sigma'_{ij} = \frac{2\sigma_v}{3\Delta \epsilon_v} \cdot \Delta \epsilon_{ij}
\]

where \( \sigma_v \) is the equivalent stress and \( \Delta \epsilon_v \) is the equivalent stress increment.

In the theory of plasticity, the equivalent stress \( \sigma_v \) and the equivalent strain increment \( \Delta \epsilon_v \) are defined as:
7.1. PRINCIPLES OF FINITE ELEMENT SIMULATIONS

7.1.3 Flow Stress

The flow behavior can be implemented into ThixoForm using the models derived in Chapter 5. Because the steady-state regime of the flow stress is reached after strains \( \varepsilon \) of approx. 0.4 and the achieved strains during bulk forming processes are often larger, only the steady-state flow stress \( \sigma_\infty \) was implemented (see Equation 5.5):

\[
\sigma_\infty = \sigma_0 \cdot \left[ \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^n \cdot \left[ 1 - \left( \beta \cdot \frac{f_L}{1 + \exp \left( -a [T - T_C] \right)} \right)^{2/3} \right]
\]

To get information on transient flow behavior, the evolution equation for the degree of agglomeration \( s \) was implemented as well, to learn the actual degree of agglomeration \( s \) during the forming process (see Equation 5.34).

\[
s(t) = \left( s_0 - \frac{a}{a - d} \right) \cdot \exp \left( [d - a] t \right) + \frac{a}{a - d}
\]

\[
a = \frac{8 \rho_h f_s}{t_f}
\]

\[
d = -\frac{\dot{\varepsilon}}{\varepsilon_{crit}}
\]

The agglomeration ratio \( s \) was introduced as a new post value in the post processor xpf of ThixoForm.

For each element \( k \) there exists for each time increment \( i \) an individual time \( t_{R,k}^{(i)} \). The decision as to whether the skeleton of solid particles in each element is disrupted or not during forming is made according to the following two cases:

Case 1

\[
\dot{\varepsilon}_{(i)} > \varepsilon^* \rightarrow s_{0,k} = \begin{cases} s_0 & \text{at the beginning of simulation} \\ s_i & \text{during simulation} \end{cases}
\]

\[
t_{R,k} = 0
\]

\[
t_{F,k}^{(i+1)} = t_{F,k}^{(i)} + \Delta t
\]

\[
s_k^{(i+1)} = \left( s_{0,k} - \frac{a}{a - d} \right) \cdot \exp \left( [d - a] t_{F,k}^{(i+1)} \right) + \frac{a}{a - d}
\]
CHAPTER 7. SIMULATING THIXOFORMING PROCESSES

Case 2

\[ \dot{\epsilon}_k^{(i)} \leq \dot{\epsilon}^* \rightarrow s_{0,k} = s_k^{(i)} \]  
(7.8)

\[ t_{r,k}^{(i+1)} = t_{r,k}^{(i)} + \Delta t \]  
(7.9)

\[ t_{F,k} = 0 \]  
(7.10)

\[ s_k^{(i+1)} = \left( s_{0,k} - \frac{a}{a - d} \right) \cdot \exp \left( d \cdot \frac{t_{r,k}^{(i+1)}}{t_{r,k}^{(i)}} \right) + \frac{a}{a - d} \]  
(7.11)

where \( \dot{\epsilon}^* \) is the critical strain rate to initiate the disruption of the solid skeleton, \( t_{r,k} \) is the recovery time, i.e. the time where no critical forming takes place, and \( t_{F,k} \) is the local forming time for each element \( k \).

7.1.4 Description of Friction

Friction is an important factor in forming processes. It influences not only the geometric form of the components but also the distribution of deformations. From the physical point of view, friction is a very complex phenomenon. Various models have been developed to describe friction. Although establishing a friction model is beyond the scope of this thesis, frictional forces have to be reasonably evaluated in the simulation of forming processes. In ThixoForm a simple but effective description is implemented.

According to Coulomb's law, the friction force is proportional to the pressure between two bodies,

\[ \tau = \mu \cdot \sigma_N \]  
(7.12)

where \( \tau \) is the (frictional) shear stress, \( \mu \) is the friction coefficient and \( \sigma_N \) is the normal stress.

But the pressure applied on the surface of a workpiece can be much higher than the yield stress in bulk forming processes. Coulomb's law may overestimate the friction forces although \( \mu \) may seem to be reasonable. Under these circumstances Coulomb's law is no longer valid. If the pressure exceeds a certain value, the shear friction model is applied:

If \( \mu \cdot \sigma_N > m \cdot \sigma_y \) then \( \tau = m \cdot \sigma_y \)  
(7.13)

The shear factor \( m \) may vary between 0 and 1 according to different stress states. Both the friction coefficient \( \mu \) and the shear factor \( m \) are constant during a simulation and they are determined by experiments.

7.1.5 Thermal Analysis

The heat flow \( q \) and the internal energy \( E \) in the workpiece can be described with
7.2. STRUCTURE OF THIXOFORM

\[ q = -k \nabla T \] \hspace{1cm} (7.14)

\[ E = c_p \rho \Delta T \] \hspace{1cm} (7.15)

where \( k \) is the thermal conductivity, \( T \) is the temperature field, \( c_p \) is the specific heat capacity, and \( \rho \) is the mass density. For \( k \) and \( c_p \) the models derived in Chapter 6 were implemented into ThixoForm:

\[ c_p = c_p^0 + \frac{a_1}{k_1 + (T - T_1)^2} + \frac{a_2}{k_2 + (T - T_2)^2} \]

\[ k = k_L + (k_S + k_L) \cdot \left( 1 - \frac{1}{1 + \exp(-b(T - T_C))} \right) \]

The parameters are described in Equations 6.9 and 6.12. The gentle reader may have noticed that the implemented specific heat model considers only two possible phases and not an infinite number of peaks as proposed in Equation 6.9. Because thixotropic alloys possess mostly only two phases, i.e. eutectic and primary phase, computation time can be kept at a minimum. With the used concept of the effective specific heat method, attention must be paid when the incremental temperature steps during simulation are greater than the solidus-liquidus interval. Contrary to the enthalpy method, effective specific heat peaks can be "swallowed".

With the implemented temperature-dependent specific heat model, the fraction liquid \( f_L \) can be computed at any time in any element according to Equation 6.11:

\[ f_L = \frac{1}{2} + \frac{1}{L} \left( \frac{a_1}{\sqrt{k_1}} \cdot \arctan \left( \frac{T - T_1}{\sqrt{k_1}} \right) + \frac{a_2}{\sqrt{k_2}} \cdot \arctan \left( \frac{T - T_2}{\sqrt{k_2}} \right) \right) \]

The fraction liquid \( f_L \) was introduced as a new post value in the post processor xpf of ThixoForm.

On the surface of the workpiece, the heat transfer to the environment \( q_a \) and the tools \( q_t \) is governed by:

\[ q_a = -h_a(T - T_a) \] \hspace{1cm} (7.16)

\[ q_t = -h_t(T - T_t) \] \hspace{1cm} (7.17)

where \( h_a \) and \( h_t \) are the heat transfer coefficients to the environment and the tools respectively; \( T_a \) is the ambient temperature, and \( T_t \) is the tool temperature.

7.2 Structure of ThixoForm

Like most FE programs, ThixoForm consists of different modules as shown in Figure 7.1. The modules are called repeatedly according to the definitions of the problem defined in the input file:
Chapter 7: Simulating Thixoforming Processes

Preprocessing
- Tools geometry
- Workpiece geometry
- Materials data
- Contacts definition
- Process definition

Inputfile *.inp

ThixoForm
- ALE method
- Iteration solver

Feedback

Evaluating Simulation Results

Outputfile *.pst

Postprocessing with xpf

Figure 7.1: Structure of ThixoForm
Title BackwardExtrusion

WorkpieceDefinition
UseFile pf2d.pf

ToolDefinition Matrize
UseFile Matrize.af
ToolDefinition Stempel
UseFile Stempel.af
ToolDefinition Container
UseFile Container.af

MaterialDefinition
Alu
YoungsModulus 77000.0
PoissonsRatio 0.35
InitialTemperature 570.0
ThermExpansion 0.000001
ThermConductivity 60. 150. 0.2
SpecificHeat 965000.0 214046000. 348400000. 42. 857. 97200000. 35.6 885.0
Density 0.0000027
EnvironmentFlux 0.05
ApproachCoefficient -25.0
Thixo 0.52 163200. 0.154197 0.6375 0.55312 840.34 0.3 0.02 18. 0.1 0.01

ContactSpecification
ShearFriction 0.20 Matrize Alu
ShearFriction 0.20 Stempel Alu
ShearFriction 0.20 Container Alu
HeatFluxCoefficient 1.0 ToolTemperature 550.0 Matrize Alu
HeatFluxCoefficient 1.0 ToolTemperature 550.0 Stempel Alu
HeatFluxCoefficient 1.0 ToolTemperature 550.0 Container Alu

ProcessStepDefinition
BackwardExtrusion
DisplacementControlledTool 0.0 0.0 20.0 Stempel
DisplacementControlledTool 0.0 0.0 0.0 Matrize Container
EnvironmentTemperature 55.0
UntilTime 1.50
MaxDisplacement 0.025
PostIncrement 50
For the mesh discretization, the remeshing and the rigid-plastic element formulation in ThixoForm please refer to [64].

7.3 Description of the Commands

In this section only the commands in the input file are explained that are specific for ThixoForm. For the description of all other commands please refer to the user's guide of PressForm [66].

An input file consists of five components:

- **Title** denotes the title of the simulation.
- **WorkpieceDefinition** introduces the section with geometry data of the workpiece.
- **ToolDefinition** defines the tools geometries.
- **MaterialDefinition** describes the material of the workpiece and its elastic, plastic and thermal behavior.
- **ContactSpecification** defines the contact between workpiece and tools.
- **ProcessStepDefinition** controls the sequences of the process.

With the option **UseFile** the data for a specific section can be inserted from a separate file.

7.3.1 Thermal Conductivity

The thermal conductivity \( k \) is modeled according to Equation 6.12. The critical temperature \( T_C = \frac{T_L - T_S}{2} \) will be entered within the Thixo environment.

\[
k = k_L + (k_S + k_L) \cdot \left( 1 - \frac{1}{1 + \exp(-b[T - T_C])} \right)
\]

\[\text{ThermConductivity} \quad k_L \quad k_S \quad b\]

Units: \( [k_L] = [k_S] = W/mK, \quad [b] = 1 \)

7.3.2 Specific Heat Capacity

For the modeling of the specific heat capacity \( c_p \) the melting and solidification of two phases can be considered.

\[
c_p = c_p^0 + \frac{a_1}{k_1 + (T - T_1)^2} + \frac{a_2}{k_2 + (T - T_2)^2}
\]
7.4. EXECUTING THIXOFORM SIMULATIONS

The new post-value fraction liquid $f_L$ is introduced using

$$f_L = \frac{1}{2} + \frac{1}{L} \left( \frac{a_1}{\sqrt{k_1}} \cdot \arctan \left( \frac{T - T_1}{\sqrt{k_1}} \right) + \frac{a_2}{\sqrt{k_2}} \cdot \arctan \left( \frac{T - T_2}{\sqrt{k_2}} \right) \right)$$

Specific Heat \( c_p^L \) \( L \) \( a_1 \) \( k_1 \) \( T_1 \) \( a_2 \) \( k_2 \) \( T_2 \)

Units: \( c_p^L = 10^6 \, J/gK, \) \( L = 10^6 \, J/g, \) \( a_1 = 10^6 \, JK/g, \) \( k_1 = K^2, \) \( T_1 = 10^6 \, J/g, \) \( k_2 = K, \) \( T_2 = K \)

7.3.3 Thixotropic Flow Stress

For the description of the flow stress the steady-state approach was chosen, enhanced with the evolution equation of the structural parameter $s$:

$$\sigma_\infty = \sigma_0 \left[ \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \cdot \exp \left( \frac{Q}{RT} \right) \right]^m \left[ 1 - \left( \frac{f_L^*}{\beta \cdot \exp \left(-a\left[ T - T_C \right]\right)} \right)^{2/3} \right]$$

$$s(t) = \left( s_0 - \frac{a}{a - d} \right) \cdot \exp \left( \left( d - a \right) t + \frac{a}{a - d} \right)$$

$$a = \frac{8p_b f_S}{t_f}$$

$$d = \frac{\dot{\varepsilon}}{\varepsilon_{crit}}$$

Thixo $\sigma_0$ \( Q \) \( m \) \( f_L^* \) \( a \) \( T_C \) \( s_0 \) \( p_b \) \( t_f \) \( \varepsilon_{crit} \) \( \dot{\varepsilon}^* \)

Units: \( \sigma_0 = N/mm^2, \) \( Q = J/mol, \) \( m = \left[ f_L^* \right] = \left[ a \right] = \left[ s_0 \right] = \left[ p_b \right] = \left[ \varepsilon_{crit} \right] = 1, \) \( T_C = K, \) \( t_f = s, \) \( \dot{\varepsilon}^* = s^{-1} \)

The fraction solid $f_S = 1 - f_L$ is calculated according to Equation 6.11. $\dot{\varepsilon}^*$ is the critical strain rate introduced in Equations 7.4 and 7.8.

7.4 Executing ThixoForm Simulations

The name of the ThixoForm input file must possess the suffix *.inp. Using an input file with the designation Example.inp, ThixoForm is executed as follows:

thixoform -job Example
The post-file with the simulation results is generated automatically by *ThixoForm* and has the same prefix as the input file but the suffix *.*.pst, in the example above Example.pst. These post-files can be visualized with the post-processor *xpf*.

Together with the job name, alternative options can be specified. The following list shows the possible options:

<table>
<thead>
<tr>
<th>Option</th>
<th>Function</th>
<th>Default</th>
</tr>
</thead>
<tbody>
<tr>
<td>-job</td>
<td>Job name</td>
<td>Job name must be entered</td>
</tr>
<tr>
<td>-bg</td>
<td>Run job in background</td>
<td>Off</td>
</tr>
<tr>
<td>-L</td>
<td>Lagrange multiplier technique</td>
<td>Off</td>
</tr>
<tr>
<td>-R</td>
<td>Euler rezoning technique</td>
<td>On</td>
</tr>
<tr>
<td>-RS</td>
<td>Write the restart files</td>
<td>Off</td>
</tr>
<tr>
<td>-S</td>
<td>Separation check by contact</td>
<td>Off</td>
</tr>
<tr>
<td>-T</td>
<td>Thermal computation</td>
<td>On</td>
</tr>
<tr>
<td>-U</td>
<td>Updated Lagrange method</td>
<td>On</td>
</tr>
<tr>
<td>-V</td>
<td>Write the PostScript velocity vector field</td>
<td>Off</td>
</tr>
</tbody>
</table>
8.1 Motivation

In order to verify the constitutive models and the simulation technique, cylindrical specimens of a rheocast billet (A356) with a globular microstructure were formed in a backward extrusion process (see Figure 8.1) at different temperatures in the semi-solid temperature range. For the experimental setup please refer to Chapter 5.4.2.

8.2 Results

The measured forces were then compared with the simulation using ThixoForm. Figure 8.2 shows the course of the measured and simulated forces as a function of deformation $x$. 
for different temperatures within the semi-solid regime of A356.

![Diagram showing measured and simulated courses of the forming forces for different temperatures.](image)

**Figure 8.2:** Measured and simulated courses of the forming forces for different temperatures.

The results confirm the idea that the derived constitutive models and their implementation are able to predict well the transition temperature at approx. 570 °C for A356, where the forming behavior changes from the plastic deformation of a connected-particle network to a viscous flow of a suspension of solid particles. But there are still some caveats:

1. At lower forming temperatures the predicted forces are too high because the simulation proceeds from a homogeneous initial temperature distribution in the specimen that cannot be achieved with the infrared radiation furnace geometry.

2. The measured increase of the forming force towards the end is due to the heat transfer into the tools. Further experiments would be necessary to specify the heat transfer coefficients more accurately.

The ThixoForm simulation code also yields post values that are not accessible during the backward extrusion experiment such as the agglomeration ratio $s$ of the solid particles. Because the Forming industry demands not only simulation results that show the feasibility of the process, but also the exact prediction of possible failures occurring during these processes, such non-accessible simulation data are very important for process control. For example, areas with a low agglomeration ratio $s$ tend to show macro-segregations that result in local porosity or cracks (see Figure 8.3).
Figure 8.3: Regions in a backward extrusion specimen with macro-segregation due to different agglomeration ratios $s$ of the primary particles, material A356
Figure 8.4 shows the course of the agglomeration ratio $s$ during the backward extrusion process: light areas correspond to regions where the primary particles are separated, darker areas to those where the particles form a connected network.

8.3 Conclusions

As the results indicate, the thixotropic constitutive models are in close agreement with experimental results, and hence the simulation of forming processes in the semi-solid range has promise as a tool in the thixoforming industry. In the future development of ThixoForm, main emphasis should be put on more realistic thixoforming processes. They capture problems of wider temperature ranges, larger deformations, lubrication of the dies etc. Only if all these boundary conditions are correctly determined can reliable solutions be expected. In the next chapter such a more realistic case study is presented.
Case Study: ThixoniX

The backward extrusion experiments served as a first calibration of the constitutive models and the simulation technique. Because the size and the forming geometry of the backward extrusion experiments could not compete with realistic thixoforming processes, the main reason for the ThixoniX experiments [67] was to test the ThixoForm software with components of realistic dimensions and enhanced complexity.

9.1 Experimental Setup

The experimental setup of ThixoniX is shown in Figure 9.1. A 250 kN servo-hydraulic tensile tester served as forming press. The upper and lower forming tool were fixed between the hydraulic jaws of the press. To allow isothermal forming experiments, both forming tools could be heated up to temperatures of 600 °C using custom-made heating platforms that could withstand compressive forces of up to 250 kN. To prevent overheating of the forming press due to radiation of the heating platforms, all machine elements that were next to the forming tools were isolated using insulation cladding (see Figure 9.2). The fixation platform of the upper and the lower forming tools themselves were provided with a pressurized air cooling circuit (see Figure 9.3) to prevent overheating by conduction. To prevent further damage due to overheating of the hydraulic fixations, ceramic plates with high compressive strengths were inserted between the jaws and the T-type fixation platform. Although the heating platforms and the forming tools were heated up to 570 °C, the temperature of the jaws never exceeded 50 °C.

The forming tools were fixed to the fixation platforms with 12 mm screws that were thermally isolated with custom-made zirconia washers. Five Pt100 thermometer probes were inserted in the heating platforms and the upper forming tool to guarantee continuous thermal control of the ThixoniX setup. The temperatures were measured and stored in a laptop computer. After the experiment, the temperature data were calibrated with the
measured forces and displacements of the forming press.

Both forming tools were rotation-symmetrical, simplifying the fabrication and the simulation efforts, yet providing a workpiece geometry that is comparable to realistic component geometries. At the upper forming tool, three different angles (see Figure 9.4) were realized to observe different forming regimes. The upper forming tool has the function of a punch: it is lowered during the forming process against the stationary lower forming tool. The lower forming tool represents the outer contour of the workpiece and had a conical geometry for easier removal of the workpiece after forming. To center the lower tool, four adjusting screws were placed on the lower heating platform. The upper forming tool possessed a pin that allowed centering. The upper forming tool was fixed and secured with two mounting brackets.

9.2 Forming Experiments

Rheocast aluminum disks (material A356) with a diameter of 22 cm and a height of 5 cm were heated up together with the lower forming tool in a convective furnace to a temperature of 575 °C. To shorten the reheating process, the furnace was overheated with a temperature difference of 10 °C. To control the temperature evolution of the disk, two thermocouples were inserted into two previously bored holes (depth 20 mm), one at the center of the disk, the other at its fringe. When the forming temperature of the disk was reached, the lower forming tool with the inlaid disk was taken from the furnace with a
9.2. FORMING EXPERIMENTS

Figure 9.2: Insulated heating platforms and cladded spindle drive

Figure 9.3: Cooling circuit of the upper fixation platform
Figure 9.4: Upper forming tool: tool together with heating platform, insulation and fixation (top), view from below (middle), and sketch of the tool (bottom)
handle and was positioned on the lower heating platform of the forming press. During the reheating process of the disk, the heating circuits of the upper and lower heating platform were switched on and the upper forming tool was heated by conduction until thermal equilibrium of the system was established. When the lower forming tool with the inlaid disk was placed on the heating platform, the forming process was started immediately with the desired forming speed, i.e. 0.5 mm/s, 1 mm/s and 2 mm/s. After the forming process, the upper part of ThixoniX retracted automatically in its reset position and the formed workpiece could be extracted. For easier extraction, the two forming tools were coated with a graphite layer prior to heating, and the upper frontal area of the disk was layered with graphite powder immediately previous to the forming process. The extracted workpiece was chilled with a fine water spray until it could be handled and stored on a rack. The measured punch loads $F$ with respect to displacement $x$ for different forming speeds $v$ are shown in Figure 9.5.

![Figure 9.5: ThixoniX: Punch loads $F$ with respect to displacement $x$ for different forming speeds $v$, forming temperature 573.5 °C, material A356](image)

9.3 ThixoForm Simulations

As mentioned previously, the ThixoniX campaign served as verification of the constitutive models implemented into the ThixoForm simulation software for components with realistic dimensions. The calibration of the software was strictly restricted to process parameter variations. Modifications of the model parameters were excluded, because the calibration of the model parameters were already done during the simulation of the backward
extrusion experiments. This restriction allows the validation of the implemented models independent of dimensions. To simulate the forming process, the tools and the workpiece meshes had to be defined. Because of the rotational symmetry of the tools and the workpiece, only half of the cross section had to be described. Figure 9.6 shows the resulting meshes of the upper and lower tool as well as the mesh of the workpiece itself.

Figure 9.6: The generated meshes for the upper tool, lower tool and workpiece

The process parameters valid for calibrations were: the workpiece temperature $T_W$, the heat transfer coefficient tools-workpiece $h_{T-W}$, the tools temperature $T_T$, and the shear factors $m_U$ and $m_L$ for the contact between workpiece and upper and lower tool respectively. The comparison between simulation and experiment suggested for the shear factor $m_U$ a value of 0.2, while the shear factor $m_L$ for the lower tool was higher, i.e. $m_L = 0.4$. Because of heat loss during transport from the furnace to the ThixoniX device, the workpiece temperature was the second quantity to calibrate. The best correspondence resulted from a workpiece temperature $T_W$ of 573 °C. In Figure 9.7 the experimental punch loads $F$ are compared with the simulation results for three different velocities, 0.5, 1.0, and 2.0 $mm/s$.

9.4 Discussion of Simulation Results

Even without calibration of the process parameters mentioned above, the qualitative correspondence of the simulation was surprising. Slight changes of the process parameters influenced the simulation results substantially; therefore, only few iterations were necessary until a quantitative correspondence was achieved. Also, as already observed during the experiments, the simulation results showed practically no influence of the forming speed on the punch load evolution. At the end of the forming process the simulation yields different results. The reason for these deviations is the cavity that builds during
Figure 9.7: Experimental punch load evolution with respect to displacement $x$ compared with ThixoniX simulation results, deformation velocities 0.5 mm/s (top), 1.0 mm/s (middle), 2.0 mm/s (bottom)
the last forming stage. Because the experimental setup had no venting holes, a counter-pressure formed, resulting in different form filling behavior as predicted by the simulation. Nevertheless, the *ThixoForm* software proved to be very efficient and reliable.
The important thing is not to stop questioning.

Albert Einstein

Outlook

The ThixoniX campaign revealed that the implemented constitutive models and hence the simulation of forming processes in the semi-solid temperature range has promise as a tool in the thixoforming industry. But the results indicate that the modeling of the distinctive flow behavior is by no means trivial. More research in the field of thixotropic flow behavior is needed; for example, a better understanding of the transient response of thixotropic materials in the semi-solid range. Also, disagglomeration and reagglomeration processes trigger the occurrence of macrosegregations and crack formation due to hot tearing. Therefore, a better understanding of the mechanisms involved during forming must be formulated. Second, the development of more profound materials models and their full application in simulation packages are needed, in order to establish control of overall component production.

But independently of suggestions for further research, it seems clear that in the near future the primary market for semi-solid formed components will be the light alloys aluminum and magnesium. Although considerable research has been done on the thixoforming of steel and copper alloys, the high temperatures involved restrict their production and application. Market applications for light alloys will be primarily for high-end automotive and electronic components. Several car manufacturers already produce one or two thixoformed components for their cars, be it only for the sake of individual technology leadership. Processing routes will continue to evolve, with emphasis in the coming years on the direct production of the semi-solid slurry from the fully liquid state such as the NRC technique or the Vexocast® [68] process. There is strong need for more economical ways of producing the original slurry structure and transferring that structure to a forming machine without freezing it in and reheating it again. Also the development of alloys specially tailored for thixoforming will evolve, and with that the forming of components at higher fractions solid and faster forming velocities.

Since the first discovery of the thixotropic behavior of metals in their semi-solid state, over thirty years have passed. Some may think that this is too long a time to establish
a new forming process, but many problems have had to be solved to make it happen, for example the definition and the development of a clearly defined market. Nowadays, the most important factors seem to be present for expansion and further development of the production and application of thixoformed components.
"Books are the quietest and most constant of friends; they are the most accessible and wisest of counsellors, and the most patient of teachers."

Charles W. Eliot

Bibliography


"Concern for man himself and his fate must always form the chief interest of all technical endeavor. Never forget this in the midst of your diagrams and equations."

Albert Einstein

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Acronyms

MHD Magneto-Hydrodynamic
SIMA Strain-Induced Melt Activation
NRC New Rheocasting
DTA Differential Thermal Analysis
DSC Differential Scanning Calorimetry
ALE Arbitrary Lagrangian-Eulerian
FE Finite Element
LVDT Linear Variable Differential Transducer
SEM Scanning Electron Microscope
GDOES Glow Discharge Optical Emission Spectroscopy
"I was born not knowing and have had only a little time to change that here and there."

Richard Feynman

Resume

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Selected Publications


