Air gap formation and hot tearing in solidification processing of Al- and Cu-base alloys

by

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Abstract

Shrinkage in a solidifying metal has been studied experimentally as well as theoretically. The main focus has been to examine the mechanisms causing an air gap to form between a casting and a mould, and hot crack formation to occur in a solidifying metal.

The formation of an air gap has been experimentally studied during solidification of Al- and Cu-based alloys in a cylindrical mould. The displacements of the casting and the mould causing an air gap have been measured during solidification and cooling of the casting. The temperature distribution was measured simultaneously. Mathematical modelling has been performed to increase the understanding of the solidification process and the strains formed in the solidifying metal contributing to the formation of an air gap between casting and mould. Most of the work was dedicated to develop a new model to describe the strain during solidification, but traditional theory was used for the modelling work as well.

The model suggested in this work includes non-equilibrium effects on the solidification process and the shrinkage. The formation and condensation of lattice defects formed in the solid phase during solidification and its effect on the solidification process as well as on the material shrinkage resulting in air gap formation was considered. The results from the modelling work show good agreement with the experimental results. The conclusion is that it is important to include these non-equilibrium effects in modelling of shrinkage during solidification.

The same conclusion was drawn from results of experimental work with high temperature tensile testing of in situ solidified samples and the development of a new theory for hot crack formation. It was found that a super saturation of lattice defects formed during the solidification process enhances the nucleation and growth of hot cracks during cooling.
”När jag blir nyfiken står tiden still
Då blir jag pigg och orkar lite till
En enkel fråga: Varför är det så?
Jag har ingen aning, men det roar mig ändå”

Bob Hund
Düsseldorf
Omslag: Martin Kann
This thesis is based on the following supplements:

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Supplement 2

Supplement 3

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Chapter 1

Introduction

1.1 Background to air gap formation

To be able to understand a casting process, knowledge about the heat transport between the casting and the mould is crucial. This heat transfer is usually described by a heat transfer coefficient. Factors influencing the size and shape of the heat transfer coefficient are the roughness of the mould surface, coating properties, the conductivity of the mould and the cast metal and more. But above all it is the air gap formed between the casting and the mould due to shrinkage of the casting and expansion of the mould that decides the heat transfer. The connection between variations in the heat transfer coefficient and the formation of an air gap has been studied in a number of articles about mould casting.\textsuperscript{1,6}

It has been shown that the air gap formation starts when the solidifying metal, in going from a plastic to an elastic behaviour, gets strong enough to withstand the pressure from the liquid metal.\textsuperscript{7} Before the formation of a macroscopic air gap, heat transfer occurs mainly through conduction. When the air gap starts to grow, the conduction is gradually reduced and the heat transfer can be described by a simple superposition of the radiation and conduction terms.\textsuperscript{8} For high temperature casting like steel, radiation through the gap must be included in the heat transfer,\textsuperscript{9} whereas for aluminium and copper based alloys, as are used in this work, the heat transfer due to radiation has been proved to be insignificant compared to conduction. Since the air gap size is very small the convection can be neglected.\textsuperscript{10}

In mathematical modelling of a casting process, governing equations, thermophysical properties and boundary conditions, are needed. For most practical casting processes the thermal boundary conditions are difficult to decide thoroughly for all points of the mould/metal interface. The reason for this is that the heat transfer can vary considerably with time and temperature along the face of the casting. Still these boundary conditions decide the results of thermal as well as thermomechanical modelling of the process. In figure 1 the most important phenomena related to solidification processing are shown. Many of the currently accepted numerical models claim to be able to model a process including all the
properties in the figure, but the agreement of the results with reality is often not quantitatively correct since there is a lack of material data at elevated temperatures and the physical models are not always sufficient.\textsuperscript{11}

![Diagram of phenomena related to solidification processes.\textsuperscript{11}](image)

The mechanism behind the material shrinkage resulting in a gap between the solidifying metal and the inner mould wall is not yet fully understood. A general explanation is that the shrinkage is caused by thermal contraction in the solidified shell due to a decreased temperature and thermal gradients. Difficulties arise in the treatment of the effect from the phase transformation on the shrinkage, but there are also different opinions about how to describe the thermomechanical behaviour of the solid and the mushy zone. According to Kristiansson an elastic model is sufficient for modelling of strain.\textsuperscript{13} Other researchers claim however that for description of thermal stress and strain, a viscoplastic/elastic model is needed although the use of these more advanced models has not yet lead to any convincing breakthrough in the modelling of air gap formation.\textsuperscript{11,13-15}

Experimental observations of the air gap formation have been made for a long time. Savage could 1962 estimate the instant of the on-set of air gap formation indirectly from temperature measurements in the mould near the interface towards the casting.\textsuperscript{16} Majumdar et al were able to see the growth of the air gap as a function of time by measuring the capacitance between a sensor on the mould wall and the cast metal.\textsuperscript{17} Modern researchers, however, seem to agree that the best method for measuring air gap formation is by using linear variable differential transducers (LVDT:s).\textsuperscript{4}

Theoretical and computational work made on experiments similar to the ones that will be presented in this work, but with other alloys and various mould shapes or materials have been made earlier with different aims.\textsuperscript{11,13-15,18-22} It has been
concluded that more work is needed before a true correlation between model
descriptions and measured air gaps can be established.\textsuperscript{11}

1.2 Background to hot tearing

A phenomenon closely related to air gap formation is hot tearing, or hot crack
formation as it is also called. Hot tearing is caused by a combination of low
strength and low ductility in a metal at elevated temperatures and applied tensile
stress. In a cast material the tensile stress can be developed thermally as well as
mechanically. Materials are often brittle at high temperatures and undergo a
transition to a ductile behaviour at a specific temperature called the transition
temperature or the zero ductility temperature. The transition temperature is
generally found near or below the solidus temperature. Above this temperature the
material is very sensitive for hot tearing when tensile stress is applied.

There are numerous methods for experimental investigations of high
temperature tensile properties of a material. The method of using a tensile testing
machine with a heating device is widely accepted. Kinoshita, Kasai and Emi used
an induction furnace inserted in a tensile testing machine to melt a sample and let
it solidify ‘in situ’ to the testing temperature.\textsuperscript{23} This technique makes it possible to
create conditions similar to the ones found in a casting process regarding
microstructure and cooling rate. Rogberg\textsuperscript{24} and Hansson\textsuperscript{25} have used a mirror
furnace in order to achieve the same conditions in a tensile testing machine. In
these studies it has been found that the transition temperature between brittle and
ductile is well below the solidus temperature. Rogberg\textsuperscript{24} and Hansson\textsuperscript{25} also found
that the transition temperature depends on the cooling rate and that ‘in situ’
solidified samples give a higher transition temperature in true strain and hot
ductility. The mechanical testing of ‘in situ’ solidified samples also showed a
lower strength than testing of ‘as cast’ samples.

Generally hot tearing is explained by the presence of liquid films in the
interdendritic areas. The liquid films can be formed by segregation of impurities or
alloying elements during solidification. Shrinkage and feeding problems in the
 mushy zone have been suggested to cause the hot cracks and different models for
hot tearing criteria have aroused from this theory.\textsuperscript{26–28}

The results from Kinoshita et al., Rogberg,\textsuperscript{24} and Hansson\textsuperscript{25} have shown that
there is a need for a theory that explains hot tearing without assuming the presence
of liquid. It has been observed that the transition from brittle to ductile behaviour
in austenitic steel occurs at a higher temperature with increasing cooling rate.\textsuperscript{25}
Since the microsegregation of impurities usually increases with increasing cooling
rate the behaviour should be the opposite if liquid films were to be the only cause
of hot crack formation.
1.3 Aim and content of thesis

A solidification process is always connected to density changes due to the variations in temperature and the phase transformations. The temperature changes in a solidifying and cooling material is often uneven which causes stresses and strains. In a casting process these stresses and strains can give practical problems like hot tearing, air gap formation, macrosegregation and more.

The main aim of this work has been to examine the mechanisms of the material shrinkage causing air gap formation during solidification. This has been done experimentally by measuring the temperature and the displacements of a casting simultaneously during a solidification process in a model set-up of simple geometry allowing the measured heat transfer to be applied to the whole boundary. Various alloys with different solidification modes have been used in the study to map the material behaviour. The experimental work regarding air gap formation is showed in supplements 1 to 5.

Mathematical modelling of the casting process with regard to temperature distribution, solidification and shrinkage has been done according to traditional theory in supplement 3. Most of the work has however been concentrated to the development of a new model that can explain the experimental observations better. In a number of recently published papers, one has discussed that the fraction of lattice defects in the solid during solidification exceeds the equilibrium fraction. This affects the material properties and amongst other things, leads to a latent heat that varies with cooling rate. The connection between condensation of lattice defects and material shrinkage has earlier been discussed by Simmons and Balluffi and by Wang and Reeker. The model developed and tested in supplements 1, 2, 4, and 5 aims to couple these effects to the shrinkage in a solidifying metal and its influence on the air gap formation.

Thermal strain developed during a solidification process is generally mathematically described as a product between the thermal gradient and the linear expansion coefficient. Traditional models also include the solidification shrinkage. This is often done by addition of an extra term in the expression for the total strain or by modification of the tabulated linear expansion coefficient. This model was checked in the present work and found not to fit the experimental results. Instead the hypothesis was that the extra term contributing to the shrinkage depends on the formation and condensation of lattice defects.

To study if the non-equilibrium model, considering the formation and condensation of lattice defects to contribute to the shrinkage, is reasonable other experiments were done as well. The mechanisms explaining air gap formation should also be applicable to explain for example hot tearing. Therefore high temperature tensile testing was done to see the high temperature tensile properties of some of the alloys examined regarding air gap formation. The results of these experimental studies are shown in supplement 6 and to some extent in supplement 7. These kinds of tests also give valuable information about the thermomechanical
data at elevated temperatures, like the coherence temperature for example. This data is needed for modelling of air gap formation.

Experimental results from high temperature tensile testing showed that there is a need for a new theory to explain hot crack formation. A model for explaining hot tearing for pure metals and alloys solidifying over a small temperature interval was developed in supplement 7. This model is closely connected to the theoretical work concerning air gap formation since it couples the supersaturation of lattice defects in the solid to the nucleation and growth of cracks in a casting process.
Chapter 2

Air gap formation

2.1 Experimental work

In order to measure the air gap formed during solidification an experimental set-up was constructed. The shape of the mould was chosen to be cylindrical to avoid corner effects and to make the heat transfer axisymmetric which keeps the mathematical modelling of the problem simple.

The metal was poured from the top in the cylindrical mould with a core in its centre. The mould was made from a low alloy steel (0.14% C, 0.35% Si and 1.2% Mn) and the core was made from a quartz glass tube filled with oil bound sand. The set-up was insulated in top and bottom to prevent from heat loss in z-direction. The height of the mould was 100 mm, and the external diameters of the casting and the mould was 150 and 250 mm respectively. The core diameters varied in some cases but in most of the experiments it was 24 mm. Figure 2 shows the geometry of the experimental set-up.

Thermocouples were inserted 50 mm from the top of the mould, the core and the melt. Five thermocouples were placed in the mould and four to five in the melt depending on what kind of metal that was used in the experiment. The radial spacing between the thermocouples was about 10 mm in the mould and 10-15 mm in the melt. In all the experiments with Al-based alloys as cast metal, K-type thermocouples inserted in alumina tubes were used. For the experiments with Cu-based alloys, B-type thermocouples of 0.1 mm in diameter were used. The wires were separated with one-hole alumina tubes and inserted in protective quartz glass tubes with an outer diameter of 2 mm. The small dimension of the thermocouples was necessary since the high thermal conductivity of the Cu-based alloys gives fast temperature changes.

Cooling curves were recorded throughout the solidification process. In figure 3 an example of cooling curves from this kind of experiment is shown. These curves are from an experiment performed with Al-4.5%Cu as cast alloy.
Figure 2: The experimental set up containing an aluminium casting.

Figure 3: Measured temperatures in the mould and the casting for Al-4.5%Cu.
Linear variable differential transducers (LVDT:s) were used for measuring displacements of the mould and the casting. The LVDT:s were placed at a depth of 50 mm from the top of the mould and silica rods were used to connect between the LVDT:s and the measuring points. The total error in the displacement measurements is estimated to be maximum 5μm. The measuring points were chosen so that the displacement of the cast metal and the outer wall of the mould were measured at three locations throughout the ring with 120° in between, to control any deviations from the radial symmetry of the casting. A deviation from the symmetry of about 20 μm was accepted in the end of the measurements.

The silica rods freeze into the solidifying shell and follow it as the air gap is formed. The movement of the inner mould wall was measured to give the size of the air gap as the relative movements of the metal and the mould. The rows of thermocouples were placed nearby this location.

Typical curves from displacement measurements are shown in figure 4. Initially the mould expands thermally when it gets heated by the melt. The casting follows this expansion until a shell of sufficient strength has formed. As the temperature in the shell decreases, the strain in the solid shell brings the casting to depart from the mould and an air gap is formed. When this happen the contact between the casting and the mould gets deteriorated and the rate of thermal expansion of the mould decreases.

![Figure 4: Measured displacements for Al-4.5%Cu.](image-url)
Evaluation of the cooling curves recorded in the experiments gives necessary information about the boundary conditions needed to model the solidification process. By fitting of the measured temperatures to third order polynomials, temperatures as well as temperature gradients at the interfaces of the casting and the mould could be extrapolated as functions of time. With this information a heat transfer coefficient, \( h \), could be calculated as a function of time using the expression in equation 1.

\[
-k_{\text{casting}} \left( \frac{\partial T}{\partial r} \right)_{\text{casting}} = h \cdot (T_{\text{metal}} - T_{\text{mould}}) = -k_{\text{mould}} \left( \frac{\partial T}{\partial r} \right)_{\text{mould}}
\]  

(1)

Here \( k \) is the conductivity of the material and the subscript \( i \) means interface. To define the boundary conditions, information about the heat transfer coefficient or the interface temperature of the casting is needed. The heat transfer coefficient will vary with time due to the change in contact between the casting and the mould. For Al-4.5\%Cu the result is shown in figure 5.

![Figure 5: Calculated heat transfer coefficient for Al-4.5\%Cu.](image)

The shape of the calculated heat transfer coefficient is somewhat misleading. The initial up-going trend is due to the fact that it takes some time before the temperature gradient in the mould could be measured since it is initially localised to the very near vicinities of the interface where there are no thermocouples. This
means that we start our calculation with a measured temperature gradient that is zero which leads to a heat transfer coefficient that is zero. Physically however the value should be higher than this result shows.

When the temperature has reached the locations of all the thermocouples a maximum value of the heat transfer coefficient is reached. The amplitude of this value is determined by thermal resistance from mould coating or mould roughness and the ability of the cast metal to form contact points with the mould. It is reasonable to believe that the heat transfer coefficient should have at least the maximum value from time zero. The decrease of the heat transfer coefficient after its maximum comes mostly from thermal shrinkage that occurs in the solid shell during solidification, creating an air gap between the casting and the mould wall. The shell cools on the outside whilst the temperature at the solidification front is relatively constant. This causes thermal gradients in the shell and shrinkage of the material. At the same time the mould expands thermally which contributes to the air gap.

The experimental method described here has been used for numerous alloys and is shown in supplement 1 to 5. All the examined alloys are tabulated in table 1 together with the number of the supplement where it could be found. In the third column it is indicated if additional experiments also have been performed on the alloy with additions of grain refinement or Sr.

Table 1: The alloys examined regarding air gap formation.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Supplement</th>
<th>Grain refinement/modification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>1, 2</td>
<td>No</td>
</tr>
<tr>
<td>Al-12.7%Si</td>
<td>1</td>
<td>Sr-mod</td>
</tr>
<tr>
<td>Al-4.5%Mg</td>
<td>4</td>
<td>No</td>
</tr>
<tr>
<td>Al-2.1%Si</td>
<td>4</td>
<td>No</td>
</tr>
<tr>
<td>Al-4.5%Cu</td>
<td>2, 4</td>
<td>TiB₂</td>
</tr>
<tr>
<td>Al-6.2%Cu</td>
<td>2, 4</td>
<td>TiB₂</td>
</tr>
<tr>
<td>Al-7%Si-0.3%Mg</td>
<td>4</td>
<td>TiB₂</td>
</tr>
<tr>
<td>7827</td>
<td>4</td>
<td>TiB₂</td>
</tr>
<tr>
<td>Cu</td>
<td>5</td>
<td>No</td>
</tr>
<tr>
<td>Cu-6%Sn</td>
<td>5</td>
<td>No</td>
</tr>
<tr>
<td>Cu-Te</td>
<td>5</td>
<td>No</td>
</tr>
</tbody>
</table>

2.2 Theoretical analysis of solidification

The mathematical evaluation of the experiments has been made in three different ways. In supplement 1, 2 and parts of supplement 5 a simple model was used assuming a solidification process with a planar solidification front. This model is valid in the case of pure aluminium and Al-Si of eutectic composition as in supplement 1, and pure Cu and Cu with small additions of Te as in supplement 5.
In the case of Al-4.5%Cu and Al-6.2%Cu with and without grain refinement as in supplement 2, this model can only serve as a first approximation. A more thorough analysis of alloys solidifying over a temperature interval has been done in supplement 4 and 5. Both methods presented there include non-equilibrium effects on the solidification process. In supplement 3 traditional models have been tested for some experiments using three commercial softwares and one in-house code used for research. The main features of the mathematics behind the different models are shown here.

The solidification process could be described by solving the Fourier heat equation, generally written as in equation 2.

\[ \rho \cdot C_p \frac{\partial T}{\partial t} = k \cdot \nabla^2 T + q \]  \hspace{1cm} (2)

In equation 2, a source term \( q \) was introduced. For solidification under equilibrium conditions, like in all the programs used in supplement 3, this term could be described as:

\[ q = \begin{cases} 
0 & \text{for } T > T_f \\
\rho_s \cdot \Delta H_{ab} \frac{\partial f_s}{\partial T} \frac{\partial T}{\partial t} & \text{for } T_f < T < T_i \\
0 & \text{for } T < T_s 
\end{cases} \]  \hspace{1cm} (3)

where the evolution of the tabulated value of the latent heat, \( \Delta H_{ab} \), is distributed over the solidification range. The symbol \( \rho \) represents the density and \( f_s \) is the solid fraction.

In solidification processes occurring under non-equilibrium conditions the source term has to include the effects of the formation and condensation of lattice defects during the solidification process. The dominating defect is the vacancy in the materials relevant for this work and the model includes only this defect.\(^{36}\)

A fraction of vacancies exceeding the equilibrium fraction will increase the inner free energy of the solid, influencing the solidification process and some material properties. As a result, the source term \( q \) in the Fourier equation must be written according to equation 4:

\[ q = \begin{cases} 
0 & \text{for } T > T_f \\
\rho \cdot \Delta H_{sol} \frac{\partial f_s}{\partial T} \frac{\partial T}{\partial t} + \rho \cdot \Delta H_{vac} \frac{\partial X_{vac}}{\partial t} & \text{for } T_f < T < T_i \\
\rho \cdot \Delta H_{vac} \frac{\partial X_{vac}}{\partial t} & \text{for } T < T_i 
\end{cases} \]  \hspace{1cm} (4)
where $\Delta H_{\text{sol}}$ is the enthalpy for the vacancies and $X_{\text{vac}}$ is the fraction of vacancies in the solid. This model is used in supplement 4 and 5 for evaluation of the experimental work.

In order to solve this equation a model for the variation of the fraction of solidified metal as a function of temperature is needed. This could be described by differentiating Scheil’s equation or the lever rule.

The numerical treatment of the source terms in equations 3 and 4 is problematic in some cases. If the alloy has a very narrow solidification interval or is a pure metal or a eutectic, $T_s$ will be close to or equal to $T_l$. The numerical solution will in this case give no contribution from the evolution of the latent heat. In this case an artificial temperature interval has to be assumed. This temperature interval should be as small as possible to be near the real material behaviour, but large enough to avoid the risk of falling from above the liquidus temperature to below the solidus temperature within a volume element during one time step in the numerical solution. This method is used in supplement 3, 4 and 5.

Another method for calculations of solidification without solidification interval is to put up a heat balance over the casting/mould interface. This renders an expression where the location of the solidification front, or the growth rate, depends on the heat evolved at solidification and the heat transported away from the casting over the boundary. In cylindrical coordinates, with no variations in angular or height direction, this is written:

$$
\frac{dy}{dt} = \frac{h \cdot R}{(R - y)} \cdot \rho \cdot (\Delta H_{\text{sol}} + \Delta H_{\text{vac}} \cdot \Delta H_{\text{vac}}) \cdot k_{\text{casting}} \cdot \left( \frac{T_{\text{melt}} - T_{\text{mould}}}{h \cdot R \cdot \ln \frac{R}{R - y} + k_{\text{casting}}} \right)
$$

(5)

where $y$ is the location of the solidification front. This model was used in supplement 1, 2 and parts of supplement 5 for evaluation of the solidification process.

In the two latter models, where non-equilibrium effects are taken into account, the latent heat of solidification, $\Delta H_{\text{sol}}$, is lower than the tabulated value. The latent heat of solidification is dependent upon the fraction of vacancies formed in the solid at solidification, $X_{\text{vac}}$, the equilibrium fraction of vacancies, $X_{\text{eq}}$, and the enthalpy for vacancies as:

$$
(\Delta H_{\text{sol}}) = (\Delta H_{\text{tab}}) - (X_{\text{vac}}^{\text{inc}} - X_{\text{vac}}^{\text{eq}}) \cdot (\Delta H_{\text{vac}})
$$

(6)

Mathematical modelling of the solidification process with Fourier’s equation using the source term in equation 4, or by solving equation 5 implies to take into account the heat evolved at the solidification front, and the heat released due to the
condensation of vacancies in the solid state. The faster the solidification goes the more lattice defects are incorporated in the solid and the larger is the divergence from the equilibrium model.

The vacancies will condense in grain boundaries and dislocation loops, striving towards an equilibrium fraction that gives a minimum in free energy. The variation of the fraction of vacancies with time could be described with a square wave solution to Fick’s second law with Fourier analysis according to equation 7.

\[
X_{\text{vac}} = X_{\text{vac}}^{\text{eq}} - X_{\text{vac}}^{\text{inc}} = (X_{\text{vac}}^{\text{inc}} - X_{\text{vac}}^{\text{eq}}) \exp\left(-\frac{4 \cdot \pi^2 \cdot D}{\lambda^2} \cdot t\right) \frac{2}{\pi} \quad (7)
\]

\(D\) is the diffusion coefficient and \(\lambda\) is the diffusion distance. This equation is often used to describe homogenisation processes.

### 2.3 Theoretical analysis of shrinkage

The part of the air gap formation that is a result of the contraction of the casting could be described through thermal strain calculations. The temperature variations in longitudinal direction and the metalostatic pressure could be neglected. Considering elastic stress and strain only is justified since the focus in this work is on the strains which are not greatly influenced if plastic deformation is included in the calculations.\(^\text{12}\)

Before the solidification fronts have met, the problem can be treated as a cylinder with a concentric hole of radius \(a\), which is the location of the solidification front. The outer radius is \(R\). Then the following equations could be used to describe the thermal displacement, \(u_r\), assuming planar stress conditions.\(^\text{17}\) The relation between stress and strain is given by Hooke’s generalised law which give the coefficients in equation 8.

\[
u_r = \frac{\alpha}{r} \left\{ (1 - \nu) \int_0^{R-r} r dr + \frac{(1-\nu) R^3 + (1+\nu) a^3}{(R^2 - a^2) \int_0^{R-r} r dr} \right\} \quad (8)
\]

A relation coupling the contribution to the total displacement from vacancy condensation, \(u_v\), to the fraction of condensed vacancies, \(\Delta X_{\text{vac}}\), is given in equation 9. This equation was obtained from a volume balance.

\[
\Delta X_{\text{vac}} = \frac{2 \cdot u_v \cdot R - \frac{u_v^2}{R^2} \cdot R}{R^2 - \frac{u_v^2}{L}} \quad (9)
\]
The total material shrinkage will thus be the sum of the displacement due to thermal gradients, $u_T$, and the vacancy induced shrinkage, $u_V$.

This model should be compared to traditional models where the thermal strain either consists only of the expression showed in equation 8 but the linear expansion coefficient is increased in the solidification interval in order to include the solidification shrinkage. The other frequently used method is to add a term in the expression for thermal strain that includes a part of the solidification shrinkage. These two approaches are tested in supplement 3 where the models also include plastic deformation and in one case also viscoplastic/elastic deformation.

2.4 Mathematical evaluation of the experimental work

The modelling work was focused on the solidification process and the strain leading to shrinkage of the solidifying metal. The behaviour of the mould and its effect on the air gap formation was in all cases, except for supplement 3, not considered since the mechanics describing the mechanical behaviour of steel up to 600°C is known.

In supplement 1, 2 and partly in supplement 5 the solidification process was modelled by numerical solution of equation 5. The procedure for the solution is thoroughly described in supplement 1. The solution gave a location of the solidification front as a function of the time. From knowledge about the interface temperature of the casting and the temperature at the solidification front the thermal shrinkage was modelled according to equation 8 with the assumption of a linear temperature distribution in the solid shell. A typical result from this kind of calculation is shown in figure 6.

In the figure the measured displacement of a casting of pure aluminium is shown, as well as the calculated thermal contraction and the shrinkage due to vacancy condensation and the sum of the two. Only inward movement of the casting is considered.
In supplement 4 and 5 the Fourier equation was solved numerically with the finite difference method (FDM) with Dirichlet boundary conditions defined by the extrapolated interface temperatures of the casting at the outer boundary. It was assumed that there were no variations in the heat transfer in angular direction or along the height of the casting. Scheils equation was used to describe the precipitation of solid fraction as a function of temperature.

When the solidification had been modelled the temperature distribution was used to make a polynomial, describing the variation of the time derivative of the temperature over the radius of the casting. A coherence temperature was chosen in order to define when the material can take up stress. Assuming that solidification is slow it is allowed to differentiate equation 8 and 9, which could be solved numerically.

In this work the point of special interest is at the interface towards the mould where the results could be directly compared to experimental displacement measurements. A typical result from the calculations of the temperature distribution and the displacements with the FDM program are shown in figure 7 and 8. The alloy used in this case was Al-4.5%Cu.
Figure 7: Calculated temperature in Al-4.5%Cu at the radius 25 mm and measured temperature at the same point. The interface temperature is shown as well.

Figure 8: Calculated and measured displacements of the Al-4.5%Cu casting.
In figure 7 the measured temperature with the thermocouple closest to the core is displayed together with the calculated temperature closest to this point. The interface temperature is displayed as well. The further away you get from the boundary, the larger is the divergence between the calculated and measured results; thus the worst results are shown in the figure.

In figure 8 the measured displacement of the interface of the casting towards the mould could be seen. The calculated displacement due to thermal strain and vacancy condensation are shown, as well as the sum of these two contributing to the total displacement.

The start of air gap formation occurs when the solid shell is stable enough to compensate for the pressure from the melt. The on-set of air gap formation was found from the experiments. A material property that is crucial to know when the shrinkage should be calculated is the coherence temperature, which indicates when the material can take up stress and thereby starts to behave mechanically as a solid rather than a liquid. The coherence temperature could be found experimentally by high temperature tensile testing of ‘in situ’ solidified samples. For some of the alloys this property has been experimentally measured by high temperature tensile testing which can be seen in supplement 6 and 7. In other cases literature data have been used.

Furthermore it is important to know the maximum fraction of vacancies that is frozen into the solid during solidification. This fraction of vacancies can be estimated from the undercooling found from the experiments. The maximum fractions of vacancies formed in the solid were found to vary between 2 and 10 times the equilibrium fraction of vacancies in aluminium at the melting point, and between 5 and 25 for copper. The diffusion distances were estimated from the structure of the material.

The computational results with the use of this model are generally very good and the curves in figures 7 and 8 are representative for the results in supplement 4. In the case of Cu-based alloys in supplement 5 the model shows acceptable results. Compared to the measured values the calculated displacements get slightly overestimated initially and underestimated later. This is probably due to inelastic behaviour of the metal. Creep is not included in the model used here. A creep process would increase the supersaturation of vacancies and delay the vacancy condensation.

In supplement 3 four different softwares were used for the modelling work. Here not only the displacements of the casting were modelled but also the mould distortions. The codes all solve the Fourier equation with the equilibrium source term with the finite difference method (FDM) or the finite element method (FEM). In most cases the full three dimensional geometry was modelled. In addition to the elastic contraction the plastic deformation is included in the models. The shrinkage is described by an increase in the thermal expansion coefficient in the two FDM-programs. In the FEM-programs the literature data of the linear expansion coefficient is used, but in the expression for total strain an extra term is added in
order to include the solidification shrinkage in the thermal strain. The result mostly gives an overestimation of the shrinkage. In this work the condensation of lattice defects was not considered. Further information about the models used by the different commercial softwares used in supplement 3 could be found there.

One example of the results from this modelling work is shown in figure 9. The alloy examined in these calculations is Al-7%Si-0.3%Mg. In figure 9 the shrinkage of the casting is regarded as negative whilst the positive curves show the mould expansion. The figure shows experimental displacements as well as four calculated displacements of the casting and two calculated displacements of the mould.

![Figure 9: Calculated displacements of mould and an Al-7%Si-0.3%Mg casting according to four commercial softwares.](image)

In supplement 3 calculations were also performed with one code including inelastic behaviour of the metal. These results were better than the ones shown in figure 9, but still deviated from the measured curve during solidification process.
2.5 Discussion

The experimental data has been evaluated with three different approaches. The traditional way of solving the Fourier equation under equilibrium conditions in commercial softwares showed that there is a lack of models to thoroughly describe the shrinkage during solidification. The macroscopic thermomechanical approach where the shrinkage is described by a modified thermal expansion coefficient or by the sum of the thermal contraction and an estimation of the solidification shrinkage can not reproduce the experimental results. The need for a better way to describe the shrinkage in a solidification process was clear from the work in supplement 3.

The theory that the condensation of lattice defects contributes to the shrinkage is not new. The assumption that this effect can contribute to the air gap formation, however, is. In supplement 1 and 2 a model was presented where the metal is considered to solidify with a planar front. The results gave a first indication that this model could explain the measured shrinkage of pure metals and eutectics. The development and modification of the model to cover the two-phase region of the solidifying and cooling metal gave results found to fit very well to the experimental findings for Al-based alloys. The results were acceptable for Cu-based alloys, but it was concluded that the model needs to be developed in order to include creep mechanisms.
Chapter 3

Hot tearing

3.1 Introduction

There is a lack of data for thermomechanical properties of materials at elevated temperatures. Metals at high temperatures often undergo a transition from brittle to ductile behaviour when the temperature is decreased from the melting point. Information about the temperature at which this transition occurs as well as the size of the ultimate tensile stress before cracking could be found from high temperature tensile testing of the metal.

Such data is very useful in practical casting applications. In order to know when hot crack formation is likely to occur in a casting process these data combined with the temperature distribution in the casting give the necessary information to see if there is a risk of hot crack formation. With the knowledge of the solidification behaviour and heat transfer between the metal and the mould it is possible to get information about the thermal gradients in the solid metal shell giving thermal stresses and strains during solidification. If the thermal stress exceeds the measured maximum stress for the material in the tensile tests and this occurs above the transition temperature from ductile to brittle, a crack will most probably form. If the stress distribution reaches the maximum strength of the material below the transition temperature it might not necessarily be so severe. The material is ductile below the transition temperature and the tensile stress could then result in plastic deformation.

Another aspect of the usability of results from high temperature tensile tests is that the coherence temperature can easily be detected. In modelling of air gap formation during a solidification process this temperature is essential for forming a criterion for when the air gap can start to form and how much of the mushy zone that contributes to the shrinkage of the solid. Since both air gap formation and hot tearing are phenomena related to the shrinkage behaviour of the material during solidification there is a close connection between them.
3.2 Experimental method

High temperature tension testing is an experimental method often used to decide the susceptibility for hot tearing of a material. With some techniques the samples are heated to the testing temperature without complete melting and then tested. This method is excellent for measuring the hot ductility relevant for a hot rolling process for example. The results from the ‘as cast’ samples can however not be applied to hot tearing during a solidification process. The mechanical properties would be overestimated. \(^{24},^{25}\) In this work the high temperature tensile testing was done on ‘in situ’ solidified samples instead.

The experimental work was performed in a tensile testing machine. A mirror furnace with three ellipsoidal reflectors was mounted on the set-up. The mirror furnace gives a limited heating zone by its focus which is located at mid height of the testing rod. Inserted in the sample at the same height there was an S-type thermocouple. This makes it possible to melt a small part of the sample and let it solidify and cool to the test temperature under controlled conditions. The grips were water-cooled giving a structure formed by dendrites growing in the pulling direction during solidification. The samples were 8 mm in diameter and the length of the molten zone was about 10 mm.

A drawing of the experimental set-up is shown in figure 10. On the left the grips are shown containing a sample, on the right the mounting of the lamps is illustrated. In figure 11 there is a photograph showing an overview of the equipment.

![Drawing of the experimental set-up](image)

**Figure 10:** Drawing of the experimental set-up. To the left the sample mounted in the grips with a thermocouple and to the right a top-view of the three ellipsoidal lamps and the sample.\(^{24,25}\)
The high temperature tensile properties were studied for a number of aluminium alloys. The examined alloys were pure aluminium, Al-4.5%Mg, Al-6.2%Cu, AA6061 and a commercial aluminium alloy quite similar to AA6061. The used alloy compositions are shown in table 2.

<table>
<thead>
<tr>
<th>nr</th>
<th>Alloy</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mg</th>
<th>Ti</th>
<th>Mn</th>
<th>Cr</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AA6061</td>
<td>0.60</td>
<td>0.25</td>
<td>0.29</td>
<td>0.94</td>
<td>0.021</td>
<td>0.10</td>
<td>0.20</td>
<td>0.02</td>
</tr>
<tr>
<td>2</td>
<td>7827</td>
<td>0.65</td>
<td>0.25</td>
<td>0.30</td>
<td>0.45</td>
<td>0.15</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>Al-4.5%Mg</td>
<td>0.8</td>
<td>0.14</td>
<td>&lt;0.01</td>
<td>4.5</td>
<td>-</td>
<td>&lt;0.01</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>Al-6.2%Cu</td>
<td>0.09</td>
<td>0.099</td>
<td>6.2</td>
<td>2.4e^{-3}</td>
<td>1e^{-3}</td>
<td>&lt;3e^{-5}</td>
<td>2e^{-4}</td>
<td>4e^{-3}</td>
</tr>
<tr>
<td>5</td>
<td>Al</td>
<td>0.035</td>
<td>0.071</td>
<td>1.3e^{-5}</td>
<td>7e^{-4}</td>
<td>5.1e^{-5}</td>
<td>1.3e^{-3}</td>
<td>1.3e^{-3}</td>
<td>3.3e^{-3}</td>
</tr>
</tbody>
</table>
In this work the aluminium based alloys have been tested at varying cooling rates or strain rates. A typical temperature cycle of a tensile testing experiment of an Al-base alloy in the mirror furnace equipment is shown in figure 12. The sample was heated to 500°C at a rate of about 360°C/minute. Then the rate of heating was decreased to 180°C/minute until the temperature was 620-635°C when the rate was slowed down even more to 30°C/minute until the liquidus temperature was reached. At this point the temperature was decreased. The cooling rates used here were 0.1 K/s, 1 K/s and 2 K/s. The pulling rate was 0.05 s⁻¹ in all cases except in the case of the commercial alloy AA6061 which was tested for varying strain rates 0.05 s⁻¹, 0.005 s⁻¹ and 0.0005 s⁻¹.

The machine is regulated at zero force below the solidus temperature, but above this temperature the positions of the grips were locked.

![Temperature cycle diagram](image)

**Figure 12:** The temperature cycle during a tensile test of an aluminium based alloy.

### 3.3 Measured properties

From the experimental data the ultimate tensile stress, the strain to fracture and the hot ductility could be calculated as functions of the testing temperature. The ultimate tensile stress, \( \sigma_b \), was defined as the maximum measured force, \( F_{\text{max}} \), divided by the original area of the cross section of the sample, \( A_0 \).

\[
\sigma_b = \frac{F_{\text{max}}}{A_0}
\]  

(10)
The true strain, or the strain to fracture, was obtained from the knowledge of the length of the heated zone, \( l_0 \), and the measured elongation, \( \Delta l \), from start of the test to fracture of the sample. The relationship is shown in equation 11.

Strain to fracture:

\[
\varepsilon = \ln \left( \frac{l_0 + \Delta l}{l_0} \right)
\]  

(11)

The area reduction, or hot ductility as it is often called, was calculated through division of the change in cross sectional area and the original area of the sample.

Hot ductility or area reduction:

\[
RA = \frac{A_0 - A_1}{A_0}
\]  

(12)

3.4 Results for varied strain rate

In figure 13 and 14 the measured ultimate tensile stress and the true strain for AA6061 are shown. The figures show high temperature tensile testing of ‘in situ’ solidified samples at the strain rates 0.05, 0.005 and 0.0005 s\(^{-1} \) and a cooling rate of 2 K/s. For comparison there are also results from testing of ‘as cast’ samples under the same conditions. The tensile testing of the ‘as cast’ samples was made with a conventional Gleeble test equipment by Twite et al.\(^{38} \) It is evident from the results that the testing of the ‘as cast’ samples gives better mechanical properties. The transition temperatures are increased and the levels of ultimate tensile stress are decreased for all the ‘in situ’ solidified experiment series. The conclusion is that data resulting from high temperature tensile testing of ‘as cast’ samples should not be used for modelling of solidification processes. The thermomechanical properties get overestimated. For modelling of hot rolling for example those results are excellent.
Figure 13: Ultimate tensile stress for AA6061 performed with ‘in situ’ solidified samples (this work) and ‘as cast’ samples (Twite et al)\(^{19}\).

Figure 14: True strain for AA6061 performed with ‘in situ’ solidified samples (this work) and ‘as cast’ samples (Twite et al)\(^{19}\).
3.5 Results for varied cooling rate

Interesting for casting applications is also to vary the cooling rate. This was done for several aluminium based alloys. One example is shown below and that is the results from a very crack sensitive commercial alloy of a composition close to AA6061. The results from the high temperature tensile testing of this alloy (marked nr 2 in table 2) tested at cooling rates 0.1 K/s and 1 K/s, are shown in figure 15 to 17. It can be seen in the figures that the variation in cooling rate gives almost no effect on the ultimate tensile stress. The transition temperatures in the true strain and the hot ductility however are lowered considerably with higher cooling rate. Of the other alloys examined in this work only Al-4.5%Mg gave a similar but less pronounced shift in the transition temperatures. For Al-6.2%Cu and pure aluminium no shift was detected.

![Graph](image)

*Figure 15: The ultimate strength as a function of test temperature for alloy nr 2 at cooling rates 0.1 and 1 K/s.*
Figure 16: True strain as a function of test temperature for alloy nr 2 at cooling rates 0.1 and 1 K/s.

Figure 17: Hot ductility or area reduction as a function of test temperature for alloy nr 2 at cooling rates 0.1 and 1 K/s.
The solidification data has been experimentally determined with differential thermal analysis for the aluminium based alloys tested in this work. The results are given in Table 3 together with the transition temperatures from brittle to ductile for the different cooling rates at strain rate 0.05 s\(^{-1}\).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Name</th>
<th>(T_L)</th>
<th>(T_S)</th>
<th>(T_E)</th>
<th>(dT/dt)</th>
<th>(T_{DB})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AA6061</td>
<td>650</td>
<td>568</td>
<td>-</td>
<td>2</td>
<td>550-557</td>
</tr>
<tr>
<td>2</td>
<td>-</td>
<td>660</td>
<td>580</td>
<td>-</td>
<td>0.1</td>
<td>540-550</td>
</tr>
<tr>
<td>3</td>
<td>Al-4.5%Mg</td>
<td>631</td>
<td>542</td>
<td>450</td>
<td>0.1</td>
<td>540</td>
</tr>
<tr>
<td>4</td>
<td>Al-6%Cu</td>
<td>641</td>
<td>548</td>
<td>548</td>
<td>0.1 &amp; 1</td>
<td>535</td>
</tr>
<tr>
<td>5</td>
<td>Al</td>
<td>660</td>
<td>660</td>
<td>-</td>
<td>0.1 &amp; 1</td>
<td>640-642</td>
</tr>
</tbody>
</table>

For all of the alloys listed above, the transition temperatures were found below the solidus temperatures at strain rate 0.05 s\(^{-1}\). Alloy nr 2 shows a transition far below the solidus temperature which indicates that this alloy is very prone to cracking. In the case of Al-6.2\%Cu the transition occurs below the eutectic temperature.

The theory generally used today to explain hot tearing is based upon the assumption that microsegregation of impurities or alloying elements forms a material with lower melting-point creating a liquid film where the cracks are formed. The transition temperatures are however found at so low temperatures that the theory suggested by Fredriksson et al. concerning the formation and condensation of vacancies is more reasonable as an explanation of hot tearing behaviour.\(^{39}\)

3.6 Theoretical analysis of hot tearing

Since the transition temperature is affected by the cooling rate as well as the strain rate in the casting, quite extensive investigations are demanded in order to map the tendency for hot crack formation in a metal. Several experimental studies made on different alloys have given results showing that hot tearing can occur well below the solidus temperature.\(^{23-25}\) This is confirmed in this work. Results from Hansson show that the transition temperature in austenitic Fe-Ni was decreased with decreased cooling rate.\(^{25}\) This result could not be explained by the presence of liquid films. A theory is needed to explain the formation of hot cracks without assuming the presence of liquid.

The model used in chapter 2 for explanation of air gap formation could also be used to explain the formation of hot cracks. Lattice defects are formed in the solid
during solidification due to differences in density in the liquid and solid state, resulting in a supersaturation of lattice defects in the solid. A theory where homogeneous nucleation of cracks is considered to be caused by vacancy condensation is presented in detail in supplement 7. The main features will be reviewed here.

The supersaturation, $f$, of vacancies in a solidifying material depends on the cooling rate, which also affects the structure of the material and thereby the diffusion distances. The rate at which vacancy condensation occurs depends on the diffusion coefficient and the diffusion distance among other things. In figure 18 the supersaturation of vacancies as a function of the temperature at three different cooling rates for Al, Cu, and Fe-10%Ni is shown. Additionally Fe-2%Ni was examined. The diffusion in this alloy was however so fast that the equilibrium level of vacancies was reached within 1°C and thus the graphs would not be visible in figure 18. The fraction of vacancies formed in the solid during solidification is found from thermal analysis of the materials where the growth undercooling gives the maximum fraction of vacancies.

![Graph showing supersaturation of vacancies](image)

Figure 18: Supersaturation of vacancies in Al (solid line), Cu (dotted line) and γ-Fe (dashed line) as a function of temperature difference from melting point at three different cooling rates.

A clustering of condensing vacancies at grain boundaries or at the interface between liquid and solid nucleates the crack. This is the way a brittle fracture starts to form. Mathematically this could be described as follows:
\[ \Delta \sigma = \sqrt{\frac{55 \cdot R \cdot T}{\pi \cdot N_A} \left( E \delta^2 + \frac{RT \ln(f)}{V_{m}^{\text{vac}}} \right)^2} \]  

(13)

where \( f \) is the supersaturation of vacancies, \( R \) is the gas constant, \( N_A \) is Avogadro's number, \( V_{m}^{\text{vac}} \) is the molar volume of the vacancies, \( E \) is Young's modulus, \( \delta \) is the strain or the average mismatch between two crystal lattices and \( \Delta \sigma \) is the difference in surface tension between the grain boundary and the newly formed interfaces of the crack.

The surface tension as a function of the supersaturation of vacancies according to equation 13 is shown in figure 19. The strain is assumed to be zero. If strain is considered, the curves will move upwards towards increasing values of the surface tension.

![Figure 19: Surface tension as a function of supersaturation of vacancies for Al, Cu, γ-Fe and δ-Fe.](image)

By comparing the necessary supersaturation of vacancies for the nucleation of a crack, given in figure 19, with the supersaturation of vacancies that is possible to achieve from figure 18, it can be concluded that it is possible to nucleate cracks by vacancy condensation.

A modified model of crack growth during a creep process was used in this work. It has earlier been presented by Fredriksson et al. crack growth by the diffusion of vacancies from the bulk solid to a growing plate-like crack was assumed. The driving force is the difference in the free energy between the bulk vacancy concentration and its equilibrium value. This force drives the diffusion of
vacancies to the tip of the growing crack as well as to form new surfaces. The growth rate, \( v \), can be written:

\[
v = \frac{D}{4V_m \cdot RT \cdot \Delta \sigma} \left[ RT \ln(f) + E \delta \gamma_m \right]
\]

(14)

where D is the vacancy diffusion coefficient.

In figure 20 the growth velocity as a function of the supersaturation of vacancies is shown assuming no strain. Strain will move the curves upward towards higher values of growth velocity. A decrease of the surface tension will also increase the growth velocity. Practically this can easily be achieved by looking at the crystal orientation giving a minimum in surface tension or by addition of impurities to the alloy, like for example sulphur to iron.

![Figure 20: The growth rate of a crack as a function of the supersaturation of vacancies for Al, Cu, \( \gamma \)-Fe and \( \delta \)-Fe.](image)

3.7 Discussion

The calculations together with experimental data show in supplement 7 that at a large supersaturation of lattice defects or a low difference in surface tension between the solid and the vacuum, homogeneous nucleation of a crack is possible. The condensation of the lattice defects in a material under stress is suggested to initiate and favour the growth of a crack. In that case no liquid is needed to explain
the crack formation. It is also clear from the results in supplement 7 that a decrease in surface tension decreases the supersaturation of vacancies necessary for nucleation of a crack. As long as there is liquid between the dendrite arms or in the grain boundaries, it is most probable that the crack nucleates on the interface between the solid and the liquid. When the material has completely solidified nucleation and growth of cracks will mainly take place in the grain boundaries.
Chapter 4

Concluding remarks

Experimental work including measurements of displacements and temperatures during a solidification process of Al- and Cu-based alloys was performed in this work. The shrinkage related to a solidification process contributing to air gap formation was studied for several alloys in a simple geometry which could be modelled mathematically. A theory was developed taking non-equilibrium effects into consideration regarding the solidification process as well as the thermomechanical behaviour of the metal. The theory included the formation and condensation of lattice defects formed in the solid and its effect on solidification and shrinkage. The model was shown to fit very well to the experimental results, whilst the traditional non-equilibrium theory could not explain the experimental results. The work showed that it is important to consider the formation and condensation of lattice defects in modelling of shrinkage leading to air gap formation during solidification.

High temperature tensile testing was performed on several alloys in order to decide the hot crack susceptibility. The transition temperatures were decided from the ultimate tensile stress curves, the hot ductility and the strain to fracture. It was found from the experimental work that the transition from ductile to brittle behaviour often occurs below the solidus temperature for the alloy. A theory for explanation of hot crack formation without the presence of liquid was tested at pure elements and alloys solidifying with a narrow solidification interval. It was found that a supersaturation of vacancies formed during the solidification process enhances the nucleation and growth of hot cracks during cooling. The condensation and annealing rate of vacancies determines the transition temperature from brittle to ductile fracture during cooling after the solidification process.
Chapter 5

Future work

This thesis contains basic information about the solidification process and shrinkage behaviour for some aluminium and copper base alloys. For some of the aluminium based alloys the thermomechanical properties have also been investigated. New mathematical models for description of the shrinkage have been developed. These models have been proved to fit better to experimental observations than the traditional theories. The new theories could also be used to spread some light over the cause of hot tearing.

The models used in this work for description of the strain in a solidifying metal needs to be developed in order to achieve a better fitting to the experimental data and be made more general for different and more complicated geometries. A possible way of doing this is to add the effect from the condensation of lattice defects in the more advanced softwares that were used in supplement 3, and not use the solidification shrinkage as an adjusting parameter which is doubtful. Another way is to develop the FDM-program written in this work and used in supplement 4 and 5 to also include creep.

Experimentally the next step is to apply the knowledge of the material behaviour to a real industrial continuous or semi-continuous casting process. This will be done in the plant of Outokumpu in Västerås, Sweden, according to plans. In such a process the shrinkage would be studied by temperature measurements in strip and mould to investigate air gap formation and mechanisms for crack formation on some of the alloys investigated in this work. The knowledge about the shrinkage behaviour found from the model experiments of air gap formation as well as the material properties found from the high temperature tensile testing will be important base data in that work.

The theory developed here for explanation of hot crack formation in pure metals and alloys solidifying over a narrow temperature interval, will be extended to cover also alloys solidifying with an extended two-phase region.
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Bibliography