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Controlling Microstructure in Magnesium Alloys: A Combined Thermodynamic, Experimental and Simulation Approach

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Despite of a strong industrial demand for lightweight magnesium castings the actually available commercial alloys still do not satisfy all the requirements for mechanical properties e.g. in automotive or aeronautic applications, especially at higher temperatures. The mechanical properties of a castings are mainly controlled by microstructure, at least if major casting defects like shrinkage porosity or feeding problems can be avoided. In the present paper a combined research approach is presented which brings together thermodynamic chemistry, casting experiments and phase-field simulations with the general aim to derive microstructure quality criteria to assist the computational design of new optimized magnesium-based cast alloys.

Calphad analysis of phase equilibria for AZ series of Mg-alloys modified with Ca: The phase equilibria provide the most fundamental and indispensable information in understanding a variety of phenomena and in designing alloys with demanded properties. The Calphad approach is regarded as the most powerful method to predict the phase equilibria in multi-component system with quite a high accuracy.^[1] In this section,

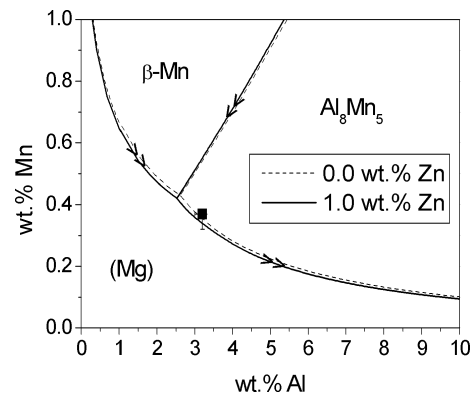


Fig. 1. Calculated primary crystallization fields, separated by monovariant reaction lines, for ternary Mg-Al-Mn alloys (dashed line) and quaternary Mg-Al-Mn-Zn alloys with 1 wt.% Zn (solid line). The arrows indicate decreasing temperature of the double-saturated liquid phase. The square mark indicates the alloy composition of AZ31 alloy employed in the casting experiments.

we present the Calphad analysis for the Mg-Al-Mn-Zn system which is the primary system in the AZ series of Mg-alloys. In addition, a tentative calculation for AZ31 alloy with Ca is demonstrated, which is associated with the later discussion of the casting experiments in Section 3. Most importantly, the present thermodynamic database can be effectively combined with the phase field simulation to describe the microstructural evolution process during solidification of the AZ31 alloy in Section 4.

Thermodynamic assessment of Mg-Al-Mn-Zn system: Within the Calphad methodology, the phase equilibria of high-order systems can be precisely described only if based on realistic thermodynamic parameters for its sub-binary and/or sub-ternary systems. A highly precise description of phase equilibria for Mg-rich Mg-Al-Mn-Zn alloys requires the thermodynamic descriptions of, especially, three ternary edge systems, Mg-Al-Mn, Mg-Al-Zn and Mg-Mn-Zn to be obtained, scrutinized or improved on the basis of the reliable experimental data.

In the present study, the thermodynamic description of Mg-Al-Mn system has been improved by focusing on Mn-solubility in Mg-Al liquid, which is reported by Ohno et al.^[2] Subsequently, performing own key experiments by means of DSC and DTA, we have demonstrated the reliability of the thermodynamic description of the Mg-Al-Zn system. Moreover, it has been clarified that the solidification process in most of Mg-rich Mg-Al-Zn alloys proceeds close to the Scheil condition. As for the Mg-Mn-Zn system, it has been shown that there exists some inconsistencies in the reported experimental works and the present thermodynamic calculation describes more reasonable phase equilibria, especially, the invariant reaction associated with the solidification of Mg-alloys. The details for Mg-Al-Zn and Mg-Mn-Zn systems will be reported elsewhere,^[3-4] respectively. Based on the thermodynamic descriptions of these sub-ternary systems, we carried out the thermodynamic calculation for the Mg-Al-Mn-Zn quaternary system. It has been shown that the calculated

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Table 1. Chemical composition of the present AZ31 alloys, balanced by Mg (wt.%). Zn and Mn in most of the samples are constants, 0.37 and 1.0 wt.%, respectively.

	Averaged value	Composition variation in the samples
Al [wt.%]	3.2	3.0 – 3.3
Zn [wt.%]	1.0	1.0 – 1.1
Mn [wt.%]	0.37	0.32 – 0.38

phase equilibria are in excellent agreement with the experimental data and, also, the solidification behavior of Mg-alloys can be successfully described based on the present thermodynamic calculation. The detail for the quaternary system will be reported elsewhere.^[5] In the following, we present the calculated result for phase equilibria of AZ31 alloy, which is the main concern of this paper.

Shown in Figure 1 are calculated projections of the liquidus surface, indicating primary crystallization fields. These are separated by the monovariant three-phase reaction lines for liquid in equilibrium with two primary precipitates. The arrows indicate the direction of the reaction line along which the temperature decreases, and the primary precipitates are specified in the figure. The dashed lines correspond to the calculated result for the Mg-Al-Mn ternary alloy, while the bold lines represent the result for Mg-Al-Mn-Zn with constant 1 wt.% Zn in the liquid alloy. It should be stressed that these are described with quite a high degree of accuracy based on the most reliable experimental data. One can see that the addition of 1 wt.% Zn has no significant effect on these monovariant reaction lines. More importantly, it can be realized that the AZ31 alloy is in a delicate composition range with three possible primary precipitates. In analyzing the experimental result of as-cast AZ31 alloy, therefore, a careful attention should be paid to the fact that the solidification behavior is quite sensitive to small variations of Al and Mn compositions and a slight composition difference could lead to a significant difference in the resultant primary phase and, thus, the material properties. By contrast, this is not the case for the widely used AZ91 alloy. In Figure 1, the square mark indicates the actual alloy composition of AZ31 employed for the present casting experiments which are discussed later in Section 3. Although the variation in Mn-composition (0.32–0.38 wt.% Mn, see Tab. 1) touches the primary (Mg) region, the Mn-composition in most experiments was about 0.37 wt.% Mn. Therefore, it can be safely said that the Al_8Mn_5 phase is the primary equilibrium precipitate in casting experiments of the present AZ31 alloys.

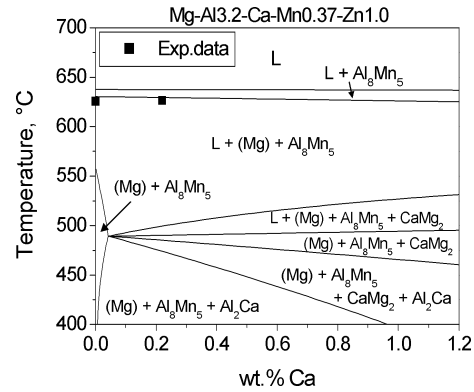


Fig. 2. Calculated vertical Mg-Al-Ca-Mn-Zn phase diagram section at constant 3.2 wt.% Al, 1.0 wt.% Zn and 0.37 wt.% Mn, which is the average composition of the present AZ31 alloys and corresponds to the square mark in Figure 1. The square marks indicate the experimental results obtained by thermal analysis.

Impact of Ca on the Mg-Al-Mn-Zn system: One focus in this paper is the analysis on the effect of Ca on the material properties of as-cast AZ31 alloy. To this end, the information on the phase equilibria of AZ31 alloy modified with Ca is of critical importance. A key system for the precise description/prediction is the Mg-Al-Ca system. Although the Calphad analysis on the Mg-Al-Ca system was reported in the literature,^[6] further experimental works suggest the improvement of the thermodynamic description to be required, which we are currently working on. The calculated result shown below is, therefore, still tentative; however, our tentative result is considered reasonable enough to demonstrate the effect of Ca on the phase equilibria of the Mg-Al-Mn-Zn system.

Figure 2 represents the calculated vertical phase diagram section at constant 3.2 wt.% Al, 1.0 wt.% Zn and 0.37 wt.% Mn which is the average composition of the present AZ31 alloys and corresponds to the square mark in Figure 1. The variation in Ca-content in Figure 2 is balanced by Mg. One can see that the primary precipitate is always the Al_8Mn_5 phase and the liquidus temperature does not significantly change with increasing Ca composition. The intermetallic phases including Ca such as $CaMg_2$ and Al_2Ca appear in low temperature re-

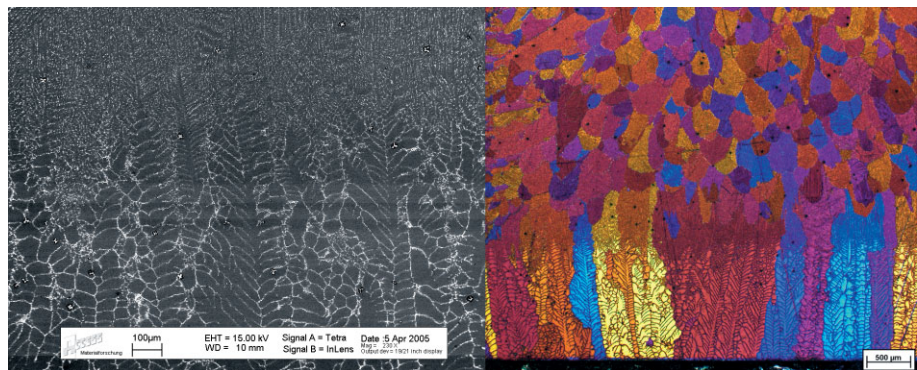


Fig. 3. EDX analysis and colour etching of a directionally solidified AZ31 sample.

gion. A substantial mutual solid solubility of these two phases is considered in the thermodynamic calculation. A small addition of Ca (up to 0.042 wt.% Ca) drastically decreases the equilibrium solidus temperature, which corresponds to the phase boundary $L+(Mg)+Al_8Mn_5/(Mg)+Al_8Mn_5$, and then, the further addition slightly increases the solidus temperature corresponding to $L+(Mg)+Al_8Mn_5+CaMg_2/(Mg)+Al_8Mn_5+CaMg_2$. The liquidus temperature measured by conventional thermal analysis for two alloys in the casting experiments is superimposed as data points in Figure 2. It is seen that the signals do not correspond to the true liquidus temperature of primary Al_8Mn_5 precipitation. They are closely associated with the phase boundary $L+Al_8Mn_5/L+Al_8Mn_5+(Mg)$, i.e., the precipitation of Mg-solid solution. Our calculations for the solidification process demonstrated that the amount of Al_8Mn_5 precipitate is quite small in these alloys, which cannot be detected in the thermal analysis, and this is quite consistent with our findings in the work on the Mg-Al-Mn-Zn quaternary system.^[5] It should be noted that this fact can be elucidated only based on the well-established thermodynamic calculation. The measured "liquidus" signal, corresponding to the immense growth of (Mg) commencing at the lower equilibrium line in Figure 2, shows only a small supercooling and is quite consistent with the calculation.

In later sections, the experimental results will be discussed in the light of the present calculated result. It is noted that calculations performed with a higher Mn-content of 0.5 wt.% indicate that Ca addition stabilizes the primary precipitate β -Mn at the expense of Al_8Mn_5 .

Casting Experiments. Directional solidification of the AZ31 alloy: In the present study, directional solidification of AZ31 samples was carried out in order to calibrate input parameters necessary in quantitative phase field simulation and to perform a fair comparison between the experimental and simulated results. 8 mm thick samples have been processed in a Bridgman type furnace, featuring powerful graphite heaters and a gallium-indium liquid metal quenching bath.

The high reactivity of liquid magnesium has to be taken in account, when processing the samples. The obligatory argon

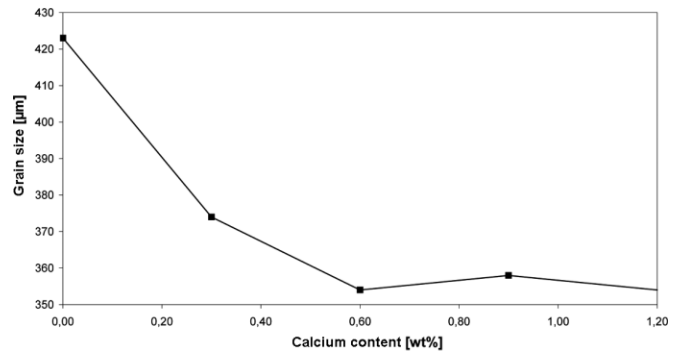


Fig. 5. Grain size as a function of calcium content.

atmosphere used for conventional samples would not reliably suppress reactions at the high processing temperatures, so a closed steel crucible system was adapted to the furnace. The directional solidification was performed with a gradient of $\sim 12 K/mm$ at a velocity of 4.2 mm/min, which is the same condition with the phase field simulation as discussed later in the Section 4.

Figure 3 shows an EDX BSE analysis and a colour etched micrograph of the longitudinal section along the quenched melting front. As a benefit of the colour etching technique, the single dendrite arms can be identified and allocated to the particular dendrite by the different colours. The quantitative analysis of the dendrite arm spacing and the angle of growth direction are used for the calibration of alloy parameters and described in the Section 4.

Grain size distributions with different Ca contents: Solidification morphology may be modified by micro alloying elements or different cooling conditions. Trials have been conducted with a cylindrical shaped sample, solidifying in a furan hardened sand mold at a freezing rate of 6.9 K/s. The sample was cut in the middle for metallographic analysis.

The solidification morphology can be described as shell forming for commercial pure Magnesium AZ31, becoming mushy with a content of 0.3% Ca. In detail the morphology was examined up to 1.0% Ca with results from castings of a coil shaped cavity used for studies on the flow length. Figure 4 shows the average grain size diameter scaled to the maximum grain size of each sample. As an effect of calcium addition the grains become more uniform in size and shape from the edge to the centre. Furthermore grain size reduces with increasing calcium content, shown by previous results^[7] of trials in permanent mold. (Fig. 5)

As discussed in Figure 2, the small addition of Ca (up to 0.042 wt.%) decreases the equilibrium solidus temperature, while the liquidus temperature does not drastically change. The further addition has no significant influence on both the equilibrium liquidus and solidus temperatures. Our thermodynamic calculation demonstrated that the non-equilibrium (Scheil-type) solidification process depends on the Ca-composition, especially, the precipitation of Al_2Ca phase is largely affected by the addition of Ca. The precipitation temperature

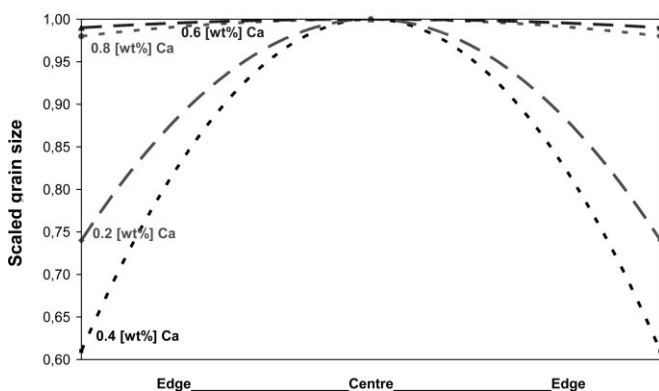


Fig. 4. Scaled grain size distribution along the cross section of samples with different calcium amounts.



Fig. 6. Oil heated hot work steel mold with spiral cavity.

of Al_2Ca phase during the solidification process increases from 431 °C at 0.1 wt.% Ca to 498 °C at 1.0 wt.% Ca which is higher than the equilibrium precipitation temperature, 475 °C. And, the amount of the Al_2Ca precipitate increases with addition of Ca. As mentioned in the Section 2, the present thermodynamic description regarding Ca modification is still tentative and should be accompanied by a careful attention to the quantitative discussion. However, it is suggestive that the effect of Ca-addition on grain size and its distribution could, at least partly, be related to the precipitation of Al_2Ca phase.

Studies on casting properties in industrial scale: In developing or modifying a casting alloy, castability may be influenced for example by new phases, generated as a result of a different chemical composition. A benefit by the modification can only be reached, when the new alloy features increased mechanical properties combined with an acceptable castability.

In the present paper, the influence factors on castability have been investigated by the determination of the flow length. As an established method, a mold featuring a coil shaped cavity has been used for the quantitative description of castability in gravity casting. In order to reproduce industrial scale conditions and also to attain data which allow a

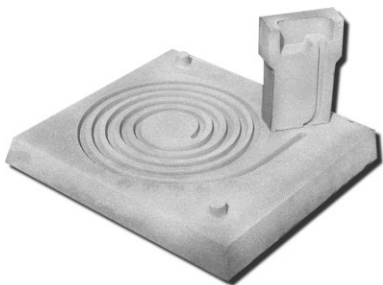


Fig. 7. Lower half of furan resin hardened sand mold with spiral cavity

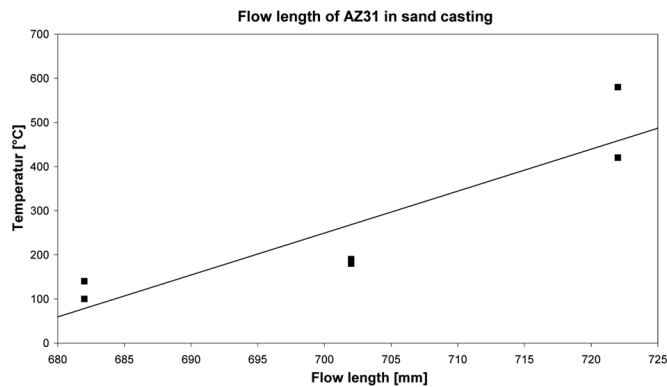


Fig. 8. Flow length of AZ 31 as a function of temperature.

comparison with literature values, the geometric shape of the coil was designed following VDG standard (Verein Deutscher Gießereifachleute e.V.).^[8] Furthermore trials were conducted in a heated hot work steel mold and furan resin hardened sand molds to examine the influence of the mold material.

The permanent mould (Fig. 6) features a powerful heating/cooling system for the upper and lower part of the die. A number of drilled ducts are leading tempered heat transfer oil, which ensures an absolutely equal temperature distribution over the whole mold including the modular ingate. Casting in the metallic mold has been carried out at a constant melt temperature of 750 °C into the preheated die, which was tempered constantly at 280 ± 5 °C and also flooded with protective gas before casting.

The furan resin hardened sand molds (Fig. 7) were enriched with 1.0 % elemental sulfur and 0.5 % boric acid to suppress reactions between magnesium and the sand mold. The molds always had room temperature during casting and were flooded with protective gas before casting.

Numerous factors affect the castability and flow length. Among several factors, the melt temperature is one of the major influences, as examined in a previous work^[9] for AZ31 in sand casting (Fig. 8) from temperatures of about 680 °C up to 720 °C. Within a temperature interval of 40 K, the passed distance rises up to a value six times above the original flow length of 100 mm. The observed linear correlation can be explained by the extended interval from casting to freezing temperature, affecting the amount of specific heat and also lowering the melt viscosity. Like the melt temperature, the heat transfer coefficient and the temperature gradient between mold and liquid metal influence the flow length. Comparing sand and steel mold, these factors are greatly differing, clearly illustrated by the values for flow length in permanent mold casting (Fig. 9) and sand casting (Fig. 10). The flow lengths plotted against the content of calcium and set in relation to the average grain size show a positively developing characteristics up to 0.8 % Ca in permanent mold and 0.4 % Ca in sand mold. The impact of the decreasing grain size on the flow ability seems to be dominated by the forming of precipitates, hindering free flow when reaching a certain calcium

level. This effect is observed earlier in sand casting than in permanent mold casting, presumably because of stronger segregation in this case. The grain size behavior of AZ31 in sand and permanent mold casting shows, that there is nearly no refining effect in the permanent mold at a wall thickness of about 5 mm with increasing calcium content. Furthermore the grain sizes linearly decrease in sand and steel mold, which is in contrast to the behavior shown in Figure 5. This might be related to the shearing force applied by the melt onto the growing dendrites or grains during casting, which produces particles acting as new nucleants.

Phase-Field Simulation. Background: The phase field method is getting more and more popular for simulation of microstructure evolution in solidification processes.^[10–14] One of its main feature is the description of moving phase or grain boundaries using a continuous phase field parameter, corresponding to a diffuse interface. The phase field equation, expressing the evolution of the phase field parameter in time and thus the movement of the front, can be derived in a thermodynamically consistent way by local minimization of the Gibbs free energy.^[15]

In this paper the multi-phase field code MICRESS[®]^[16] is used which can be online coupled to thermodynamic databases. This allows using all types of CALPHAD databases^[17] with an arbitrary number of elements and phases. These databases are connected via the Fortran TQ interface of ThermoCalc. For the simulation in this paper the database for Mg-Al-Zn-Mn-Ca, which has been described above, was used. Ca has been omitted in this first stage, reducing the thermodynamic description to the quaternary system Mg-Al-Zn-Mn for AZ31.

For sake of calculation speed multi-binary extrapolation is used to reduce the frequency of thermodynamic data access, which needs complex iterations for Gibbs energy minimization. Details about this model are described elsewhere.^[15]

Hexagonal Anisotropy: Besides thermodynamic data, numerous physical quantities are necessary to describe the solidification process of a complex technical alloy. Especially the energies and mobilities of the phase boundaries and their

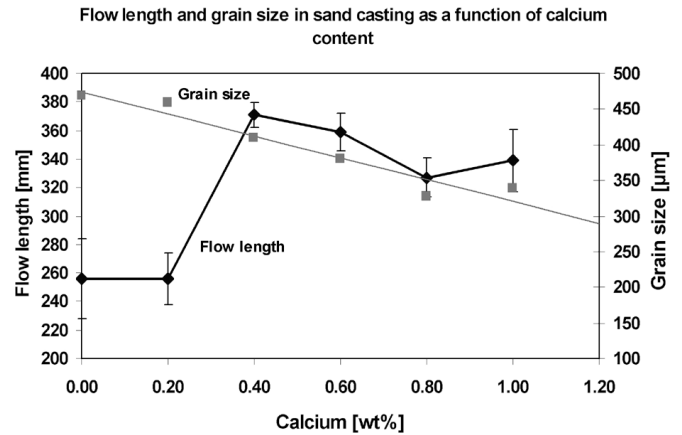


Fig. 10. Flow length of AZ 31 in sand mold as a function of calcium content.

specific anisotropy are decisive for the solidification microstructure.

In Mg alloys the hexagonal lattice symmetry leads to a different dendritic morphology compared to the typical cubic dendrites observed in most technical fcc or bcc alloys. This needs to be described by a three-dimensional anisotropy function as shown in Figure 11. The use of this function for the anisotropy of the interface mobility and the surface energy for the hcp-liquid interface leads to typical hexagonal dendrites as shown by a 3D simulation of equiaxed growth of pure Mg (Fig. 12).

Calibration of Alloy Parameters: The experimental results of directional solidification discussed in the previous section can be used for calibration purposes. Diffusion coefficients in the melt as well as the surface energy are difficult to measure and so no accurate literature values are available. By comparison of the primary and secondary arm spacings between simulation and experiments these values can be adjusted (Fig. 13). Unknown anisotropy parameters have to be adjusted to guarantee the correct growth and selection behavior.

Equiaxed Growth: For prediction of grain sizes a seed-density model is used which assumes a given density-radius-distribution of seeding particles (inoculants) which may start growing if a certain critical undercooling is reached. According to Böttger et al.^[18] isothermal conditions with global release of latent heat can be assumed, due to the huge differ-

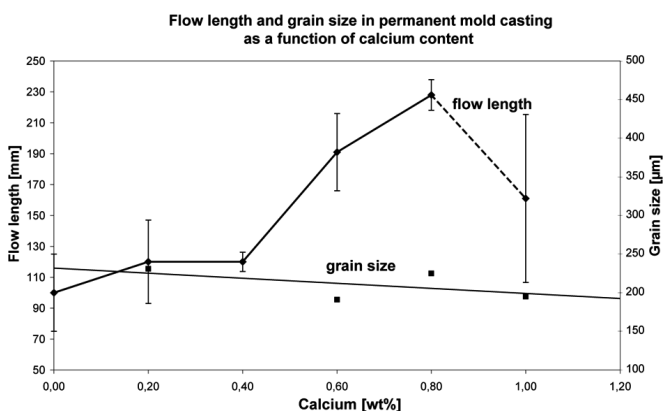


Fig. 9. Flow length of AZ 31 in permanent mold as a function of calcium content.

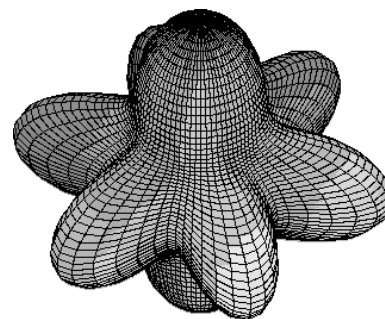


Fig. 11. Hexagonal anisotropy function for the hcp-liquid interface.

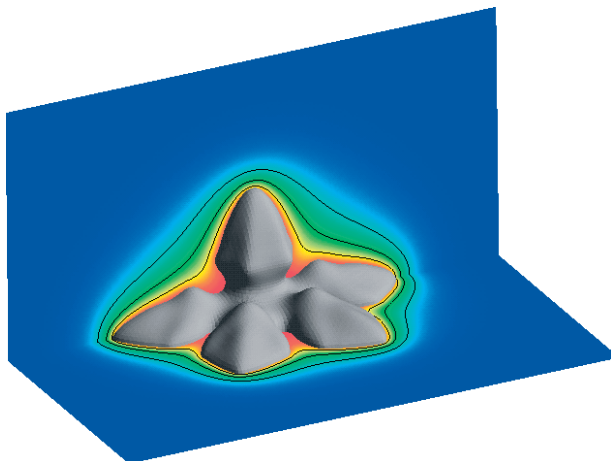


Fig. 12. 3D Phase-field simulation of a pure Mg dendrite using the hexagonal anisotropy function of Figure 11.

ence between the thermal and solute diffusion length. As the typical radii of the inoculant particles are small compared to their average distance and much smaller than the resolution of the finite-differences grid curvature is calculated analytically from the phase amount as long as the growing seeds cannot be accurately resolved by the phase field method. This is explained with more detail in the literature.^[19]

As demonstrated in Figure 14, this model is able to reproduce the influence of various parameters on grain size like the inoculants mean radius, its density distribution, the effect of segregating solutes as well as the cooling conditions. In all three cases a grain refinement is observed. There is also a clear impact on the dendritic morphology: While an increased seed density leads to a more globulitic microstructure (Fig. 14(c)), the stronger cooling in Figure 14(d) results in a higher branching of the dendrite arms. This behaviour depends on a subtle interplay of the thermodynamic properties and the density distribution of the seeding particles and may be of major importance for castability. The effect of the primary Al_8Mn_5 particles as possible seeding agents has not yet been taken into account.

Conclusions: A combined approach to investigate microstructure formation in AZ31 under typical casting conditions has been presented. First as an important basis for experiments and simulation a precise Calphad analysis has been performed for the Mg-Al-Mn-Zn system. It could be shown that AZ31 lies in a critical composition range where the precipitation of three primary phases, namely Mg, Al_8Mn_5 and β -Mn, is possible. This may be of major importance for the solidification microstructure, if the two Mn phases act as seeding agents for the Mg grains. Furthermore the effect of Ca, which is also known to have a refining effect in Mg alloys, has been included into the Calphad analysis.

For simulation of microstructure formation during solidification an existing phase-field model has been extended to perform spatially resolved microstructure simulations directly coupled to these new assessed Mg-Al-Mn-Zn data-

bases. Simulations of directional growth have been performed to understand the implications of the hexagonal lattice symmetry for the solidification microstructure and to calibrate unknown physical properties like diffusion coefficients and interfacial energies by comparison to corresponding experiments. On the other hand equiaxed solidification simulation was aimed to understand the mechanisms of grain size selection as well as the effects of grain refinement on the dendritic morphology.

Additionally, casting experiments have been carried out in order to verify the correlation between microstructure and process parameters like cooling conditions, addition of inoculants for grain refinement or alloy modifications. A first qualitative comparison has proven the principle ability of this combined approach to describe the mechanisms of microstructure formation and grain refinement in AZ31.

A systematic quantitative analysis shall help to optimize model parameters with the aim of achieving quantitative agreement between experiments and simulation. Furthermore interesting details about the grain refining effects of Al_8Mn_5 and β -Mn and about the relation between basal and prismatic growth kinetics in hexagonal Mg dendrites is ex-

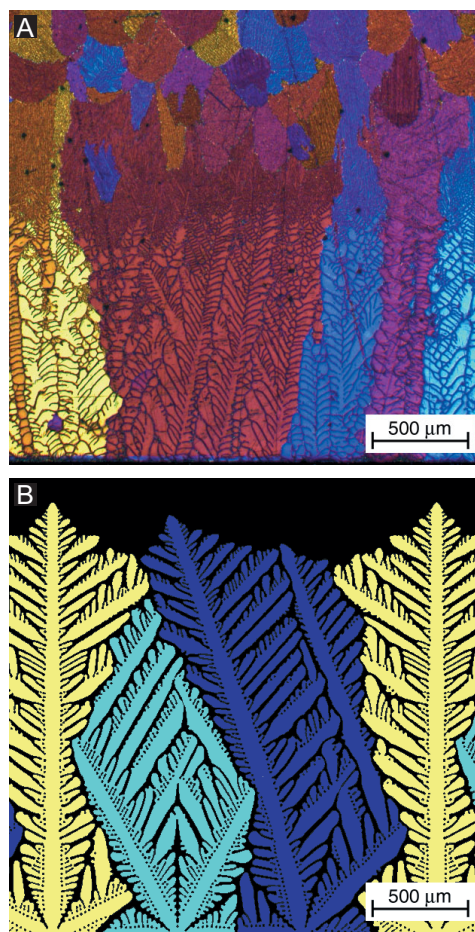


Fig. 13. Directional growth of AZ31 in experiment and qualitative comparison to simulation.

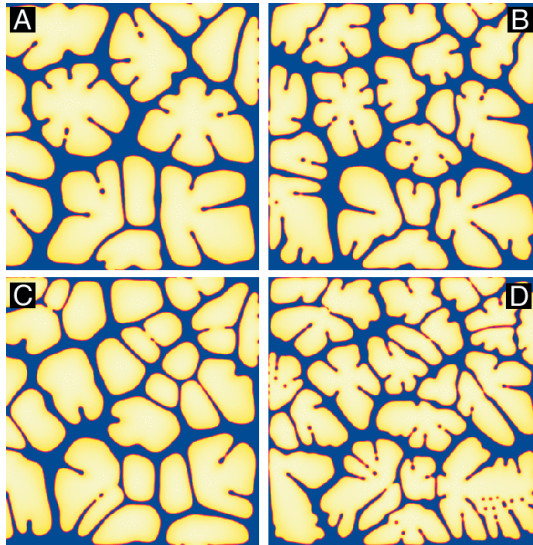


Fig. 14. Different microstructures resulting from different grain refinement mechanisms: A: AZ31 reference, B: Al composition raised from 3 to 6 wt%, C: Seed density function raised by factor 5, D: Heat extraction rate increased from 25 to 100 $\text{Jcm}^{-3}\text{s}^{-1}$

pected from further systematic experimental work in combination with improved phase-field simulations.

In a later stage the study will be extended to Sr and Ce additions which could also act as nucleating agents.

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Improvement of Mechanical Properties of Powder Metallurgical NiTi Shape Memory Alloys**

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Powder metallurgy (PM) is a promising method for the production of near-net-shape components of NiTi. Its advantages include avoiding expensive thermomechanical working, the machining of cast alloys and high losses of material.^[1,2] Two well-known processes for the production of powder metallurgical shape memory parts are Hot Isostatic Pressing (HIP) and Metal Injection Moulding (MIM). HIP is used to achieve nearly dense parts of simple geometries. Complex parts can be produced by MIM, where densification

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