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Phase equilibria, calorimetric study and thermodynamic modeling of Mg–Li–Ca alloys

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Abstract

The binary Ca–Li and the ternary Ca–Li–Mg systems were studied experimentally by calorimetry and phase equilibration and also by thermodynamic calculations using the Calphad method. The enthalpy of formation of the binary phase $CaLi_2$ was investigated by drop solution calorimetry in a liquid aluminum bath. Ternary phase equilibria at 150 °C were studied with 54 different alloys annealed for 400 h and analyzed by X-ray diffractometry. A thermodynamic assessment of the binary Ca–Li system was performed. All available experimental data agree with the calculation in the binary Ca–Li system. In the ternary Ca–Li–Mg system, a complete ternary solubility of $CaMg_2$ and $CaLi_2$ at 150 °C was found. For this $Ca(Li,Mg)_2$ solution phase, a thermodynamic interaction parameter was assessed. Based on the present thermodynamic datasets and those of the binaries Ca–Mg and Li–Mg from literature, the phase equilibria in the entire ternary system were calculated. Isothermal and vertical sections of the phase diagram and the projection of the liquidus surface are shown. These calculated phase diagrams are well supported by the experimental data.

Keywords: Magnesium; Ca–Li; Calorimetry; Thermodynamics; Phase diagram

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1. Introduction

Calcium and lithium are both important alloying elements for magnesium. Calcium is known to be an effective grain refiner and to improve the creep resistance. Lithium additions decrease the density of the magnesium alloy. High-lithium magnesium alloys show cubic bcc structure and therefore excellent deformation properties. However, no constitutional data about the ternary Mg–Li–Ca system are known in the literature. For alloy development and controlling the behavior of this material system, the knowledge about phase relations and thermodynamic properties is essential. In the present work, these data are generated in a consistent manner by a combination of experimental methods with computational thermochemistry. The basic principle of these thermodynamic equilibrium calculations is the minimization of the total Gibbs energy of the system. For each phase—stable and metastable—an analytical expression of the Gibbs energy as a function of temperature, pressure and composition has to be selected and a least

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square optimization of the parameters using all available experimental information (phase equilibria, enthalpies of formation and transformation, activities, ...) is performed. Different experimental methods, such as phase identification by X-ray diffraction or solution calorimetry, were used in the present study.

2. The binary subsystems

The thermodynamic data sets of the binary subsystems Ca–Mg and Li–Mg were taken from the literature. For the Ca–Mg the assessment by Agarwal et al. [1] was used and for Li–Mg the evaluation by Saunders [2]. In these evaluations, the available experimental literature of these binary systems is discussed. For the binary system Ca–Li, no thermodynamic data set is published. Therefore, this subsystem was investigated in this work.

2.1. Experimental literature data for the Ca–Li binary system

The experimental knowledge based on the literature assessment of the Ca–Li system by Bale and Pelton [3] is also published in Handbook of Massalski [4]. They reported one congruent melting binary phase $CaLi_2$ which crystallizes in the hexagonal $MgZn_2$-type. The experimental investigations [5–8] were critically assessed and combined with the data about the allotropic transformation of Ca [4]. The invariant reactions of this assessment are given in Table 1. Some solubility data of the Ca–Li system are given by Borstedt and Guminski [9]. Hubberstey and Roberts [10] determined the Li-rich eutectic by both thermal analytical and resistance-temperature methods. They reported the Li–$CaLi_2$ eutectic at 142 °C with a liquid of 92 at.% Li. This agrees well with the assessed value by [4].

Considering this literature basis it becomes evident that the most crucial datum still missing for a reliable thermodynamic assessment is the enthalpy of formation of $CaLi_2$.

2.2. Calorimetric study

The enthalpy of formation of the binary phase $CaLi_2$ was determined by a calorimetric measurement. The technique employed in this study is drop solution calorimetry. The solvent for the dissolution is high purity molten aluminum (99.9999% Al, Pechiney) at 706 °C, which provides a sufficient solubility for Ca and Li. The equipment used is a double cell high-temperature Tian–Calvet type calorimeter (SETARAM HT1000). The calorimeter cells consist of silica tubes in which high density alumina crucibles are inserted. The experiments have been performed under a slight Ar overpressure to prevent any air contamination due to leakage of the system. In addition, titanium getters were placed just above the alumina crucibles inside the silica tubes. The gas has to pass over these getters before reaching the bath in order to prevent oxidation. The heat effects inside the calorimeter are monitored as a change of emf given by the detector, integrated with respect to time. The emf was recorded every second using a KEITHLEY multimeter connected to a PC via an IEEE-488 interface. The area underneath the thermogram was integrated using a specially developed software. Calibration of the calorimeter has been performed by dropping small pieces of pure aluminum (30–40 mg each) from room temperature into the aluminum bath. The total amount of the aluminum bath was 11.2 g. The heat content of aluminum between 25 °C and the temperature of the bath has been taken from the SGTE pure element database [11].

$CaLi_2$ has been prepared by levitation melting starting from the pure elements (Ca: 99.5 mass%, Alfa; Li: 99.9 mass%, Chemetall) under controlled

Table 1
Compositions and temperatures of the invariant reactions in the binary system Ca–Li

Invariant reaction	Experimental [3,4]				Calculated (this work)			
	T (°C)	Phase compositions in at.% Li			T (°C)	Phase compositions in at.% Li		
βCa = αCa + liquid	370	9	8	43	369.5	8.6	6.6	43.5
Liquid = αCa + $CaLi_2$	230	59	10.5	66.67	233.4	57.1	9.6	66.67
Liquid = $CaLi_2$ + (Li)	141	92	66.67	~98	145	93.2	66.67	98.7

Table 2
Enthalpy of formation of the $CaLi_2$ phase at 25 °C determined in this work[a]

	Experimental (kJ/mol)	Calculated (kJ/mol)
$\Delta_f H(CaLi_2)$	−4.5 ± 1.6	−3.89

[a] Referred to the pure solid elements Ca-fcc and Li-bcc.

argon atmosphere. Mass loss was below 1%. Metallographic analysis verified that the sample was single phase. The sample was then cut into small pieces with a typical weight of 20 mg, which were sealed individually in silica tubes under vacuum to prevent any oxidation. The silica tubes were broken and the samples immediately dropped at room temperature into the calorimeter to minimize exposure to ambient atmosphere. From 10 individual drops, 9 have been selected and the heat effect value for dissolution at infinite dilution $Q^{\infty}(CaLi_2) = -54.0 \pm 4.0$ kJ/mol in liquid Al, has been determined by linear regression as a function of Li content in the solvent. The values for the pure elements have been taken from the literature. The heat effect at infinite dilution for Ca, $Q^{\infty}(Ca) = -58.3 \pm 1.6$ kJ/mol has been determined recently [12]. The value of pure Li is $Q^{\infty}(Li) = -4.6 \pm 1.2$ kJ/mol [13]. This gives for the enthalpy of formation at 25 °C: $\Delta_f H(CaLi_2) = 2 \times Q^{\infty}(Li) + Q^{\infty}(Ca) - Q^{\infty}(CaLi_2) = -13.5 \pm 4.9$ kJ/mol (-4.5 ± 1.6 kJ/mol of atoms). This experimental value is compared to the value calculated from optimized parameters in Table 2.

2.3. Thermodynamic modeling of the Ca–Li binary system

The Gibbs energy function $G_i^{0,\phi}(T) = G_i^{\phi}(T) - H_i^{SER}$ for the element i (i = Ca, Li) in the ϕ phase (ϕ = fcc (αCa), bcc (βCa) and (Li), or liquid) is described by the equation:

$$G_i^{0,\phi}(T) = a + bT + cT\ln T + dT^2 + eT^3 + fT^{-1} + gT^7 + hT^{-9} \quad (1)$$

where H_i^{SER} is the molar enthalpy of the stable element reference (SER) at 298.15 K and 1 bar, and T is the absolute temperature. The Gibbs energy functions for Ca and Li are taken from the SGTE compilation by Dinsdale [11].

The liquid, fcc (αCa) and bcc (βCa, Li) solution phases are described by the substitutional solution model. For the liquid phase the molar Gibbs energy is expressed by following equation:

$$G^{Liq} = (1-x)G_{Ca}^{0,Liq} + xG_{Li}^{0,Liq} + RT[x\ln x + (1-x)\ln(1-x)] + x(1-x)[L_{Ca,Li}^{0,Liq} + (1-2x)L_{Ca,Li}^{1,Liq} + \cdots] \quad (2)$$

in which R is the gas constant, and x is the molar fraction of Li. The interaction parameters L^0 and L^1 may be linearly temperature dependent and are to be optimized. The phases fcc (αCa) and bcc (βCa, Li) are described by analogous equations.

The binary compound $CaLi_2$ was modeled as stoichiometric phase, and the Gibbs energy (per mol of atoms) is given by following expression with $x = 0.666667$:

$$G_{Ca:Li}^{0,CaLi_2} = (1-x)G_{Ca}^{0,fcc} + xG_{Li}^{0,bcc} + A_x + B_x T \quad (3)$$

The parameters of the binary system ($L_{Ca,Li}^{0,\phi}$, A_x and B_x) were optimized using all the experimental information detailed in Sections 2.1 and 2.2 and the program PANDAT [14]. The parameter for the metastable hcp phase, $L_{Ca,Li}^{0,hcp}$, has been set to a sufficiently high positive value, 5 kJ/mol, in the same order of magnitude as for the stable bcc and fcc phases. This prevents an artificial stable region of the hcp phase in the center part of the Ca–Li system that would occur if an ideal solution of the metastable hcp phase were assumed.

The calculated phase diagram compared to the experimental key points after [4] is shown in Fig. 1. The invariant reactions are compared in Table 1 and the enthalpy of formation in Table 2.

3. The ternary system Ca–Li–Mg

3.1. Literature data

The phase relations and thermodynamics of the ternary Ca–Li–Mg system are poorly known from the literature. Hsu and Saboungi [15] published a calculation of ternary isothermal sections using only extrapolation of binary data. No ternary experimental information was included. Since they used different binary data for the calculation and did not know about the complete ternary solubility of $CaMg_2$ and $CaLi_2$ these data are not considered further.

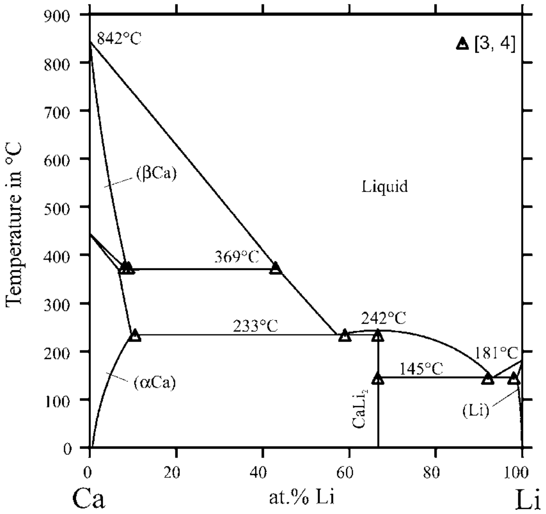

Fig. 1. Calculated binary Ca–Li phase diagram compared to experimental literature data [3,4].

3.2. *Own experimental investigations of ternary Ca–Li–Mg phase equilibria*

Since no information about the ternary phase relations exists, 45 alloys with different compositions (see Fig. 2) were prepared from mixture of pure metals (Li 98.2, Mg 99.9, Ca 99.2 mass%) by arc melting in argon atmosphere at pressure 1.01 bar on a water cooled copper mold. No reactions with the copper mold were visually observed. Alloys were preserved under purified oil. Control of alloy composition was carried out by comparison of alloy weight and weight of pure components. Annealing of alloys was carried out in tantalum containers placed in evacuated silica tubes at 150 ± 10 °C during 400 h. No reactions with the Ta-containers could be observed. Finally, the alloys were quenched in cold water. The X-ray powder method was used for phase analysis. Powder patterns were obtained by a DRON-2.0 diffractometer (Fe Kα radiation, $20° < 2\theta < 100°$, 2°/min speed of scanning). Using these powder diffraction results, the lattice parameters of the $Ca(Li,Mg)_2$ phase with different compositions were refined.

Thermal analysis was performed on one alloy ($Ca_{33}Li_{13}Mg_{54}$, at.%) with a composition on the pseudobinary section $CaMg_2$–$CaLi_2$ using a Netzsch DTA 404 apparatus. This composition was selected because preliminary thermodynamic calculations indicated a melting point maximum for this alloy. The elements were weighed, mixed and then pressed

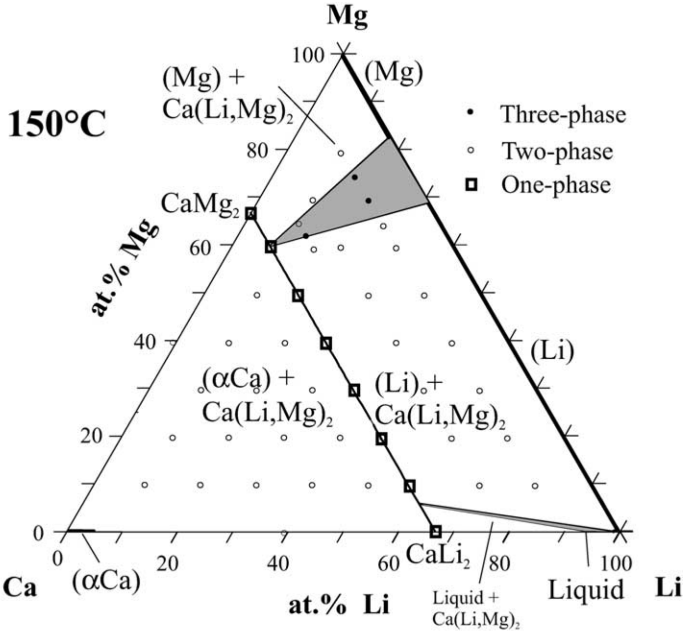

Fig. 2. Ca–Li–Mg phase diagram at 150 °C: calculated isothermal section including the investigated sample compositions.

under a pressure of 100 MPa into small pellets of around 1 g. The alloy was prepared by levitation melting under argon atmosphere. Weight loss was found less than 1 mass%. The sample was sealed under pure argon at 1 bar in specially adapted tantalum containers using electric arc welding. This procedure entirely eliminated evaporation and oxidation of the samples during the long DTA runs. The DTA measurements were carried out under vacuum (to protect the Ta-containers) up to 750 °C with heating/cooling rates of 5 and 1 K/min. The sample was first heated and cooled with 5 K/min and subsequently twice with 1 K/min for the precise determination of peak positions. The sample was also investigated metallographically and was found to be single phase. No reaction with the Ta-crucible was observed.

3.3. Experimental results

A complete ternary solubility of $CaMg_2$ and $CaLi_2$ at 150 °C was found. This phase is called $Ca(Li,Mg)_2$ in the present work. The change of the lattice parameters from $CaMg_2$ to $CaLi_2$ is very small and in the range of the observed experimental error. No additional ternary phase or ternary solubility was observed. The joint solubility of Mg and Ca in (Li) was found to be very small. The three-phase equilibrium composition of $Ca(Li,Mg)_2$ with the phases (Li) and (Mg) was found at 58 at.% Mg, 9 at.% Li, 33 at.% Ca at 150 °C.

A sharp thermal signal was observed during the DTA runs of the alloy with a composition $Ca_{33}Li_{13}Mg_{54}$ (at.%) at 658 °C (heating, maximum) and 657 °C (cooling, onset). This signal was interpreted as liquidus temperature of the $Ca(Li,Mg)_2$ phase. It was reproducibly within ±1 K in three repeated heating cycles, the total estimated error is ±4 K from calibration. The solidus temperature of this alloy was taken from the heating peak onset at about 600 °C ± 20 K. The large error of ±20 K is due to the flat solidus signal.

3.4. Ternary thermodynamic modeling

The ternary system was calculated using the binary data for Ca–Mg [1] and Li–Mg [2] together with the own data set for Ca–Li described above. The two binary phases $CaLi_2$ and $CaMg_2$ actually form one single phase $Ca_1(Li,Mg)_2$ which is modeled by a simple substitutional sublattice model with complete solid solution on the second sublattice. The Gibbs energy (per mol of atoms) is expressed by

$$\begin{aligned} G^{\mathrm{Ca(Li,Mg)_2}} = {} & y_{\mathrm{Li}} G^{0,\mathrm{CaLi_2}}_{\mathrm{Ca:Li}} + y_{\mathrm{Mg}} G^{0,\mathrm{CaMg_2}}_{\mathrm{Ca:Mg}} \\ & + 0.666667RT(y_{\mathrm{Li}} \ln y_{\mathrm{Li}} + y_{\mathrm{Mg}} \ln y_{\mathrm{Mg}}) \\ & + y_{\mathrm{Li}} y_{\mathrm{Mg}} L^{0,\mathrm{Ca(Li,Mg)_2}}_{\mathrm{Ca:Li,Mg}} + \cdots \end{aligned} \quad (4)$$

in which y_{Li} and y_{Mg} are the site fractions of Li and Mg on the second sublattice. The parameters $G^{0,\phi}_{Ca:*}$ (also called compound energies) are expressed relative to the Gibbs energies of the pure elements at the same temperatures. These parameters are taken from the binary descriptions of the $CaLi_2$ and $CaMg_2$ phases. The L^0 parameter represent the interactions primarily within the sublattice. Just one ternary interaction parameter for the $Ca(Li,Mg)_2$ phase, $L^{0,\mathrm{Ca(Li,Mg)_2}}_{\mathrm{Ca:Li,Mg}} = A + BT$, was used to fit the experimentally found phase relation at 150 °C and the measured liquidus temperature. This parameter is about zero in the range of the liquidus temperature and −13,000 J at 150 °C, indicating an increased stability of this phase at lower temperature. No ternary parameter was necessary for the liquid phase and the standard Redlich–Kister type (Muggianu) extrapolation was used, which is justified for these chemically not too different components. For calculation the software PANDAT [14] was used. The calculated isothermal section of the Ca–Li–Mg system at 150 °C is given in Fig. 2. The investigated sample compositions are shown in different symbols. Calculated isothermal sections at 250 and 500 °C are given in Figs. 3 and 4. The liquidus surface with the dominating field of primary crystallization of the $Ca(Li,Mg)_2$ phase is shown in Fig. 5. The ternary invariant reactions are detailed in Table 3. The calculated pseudobinary section from $CaMg_2$ to $CaLi_2$ with the experimentally found liquidus and solidus temperatures is given in Fig. 6. The calculated vertical section at constant $x_{\mathrm{Li}} = 0.13$, parallel to the Ca–Mg system and including the DTA results, is shown in Fig. 7.

4. Discussion

The enthalpy of formation of the $CaLi_2$ phase was determined by solution calorimetry to enable well

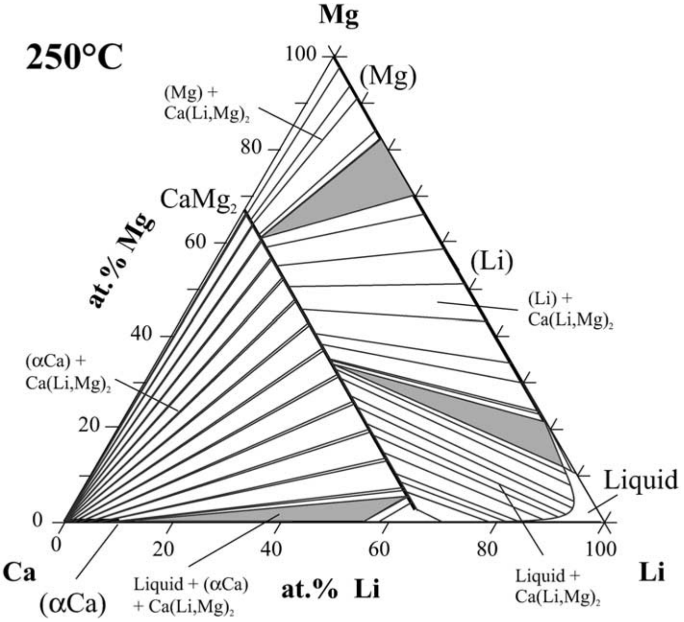

Fig. 3. Ca–Li–Mg phase diagram at 250 °C: calculated isothermal section.

defined extrapolation of the Gibbs energy function of this binary phase into ternary system. In the thermodynamic assessment of the binary system, the value of $\Delta_f H(CaLi_2)$ was selected somewhat more positive than the experimental mean value to enable a better agreement with the temperatures of the two binary eutectic reactions involving the phase $CaLi_2$, see Fig. 1 and Table 1. The assessed value of $\Delta_f H$ is still well within

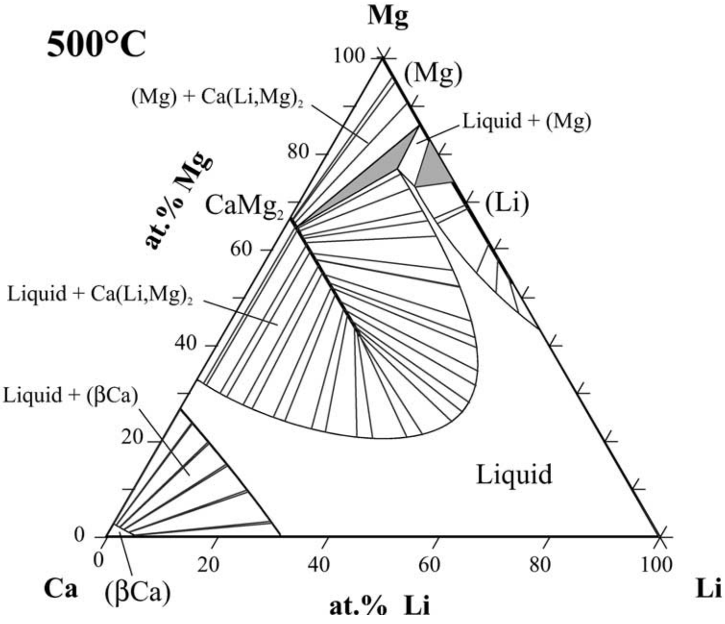

Fig. 4. Ca–Li–Mg phase diagram at 500 °C: calculated isothermal section.

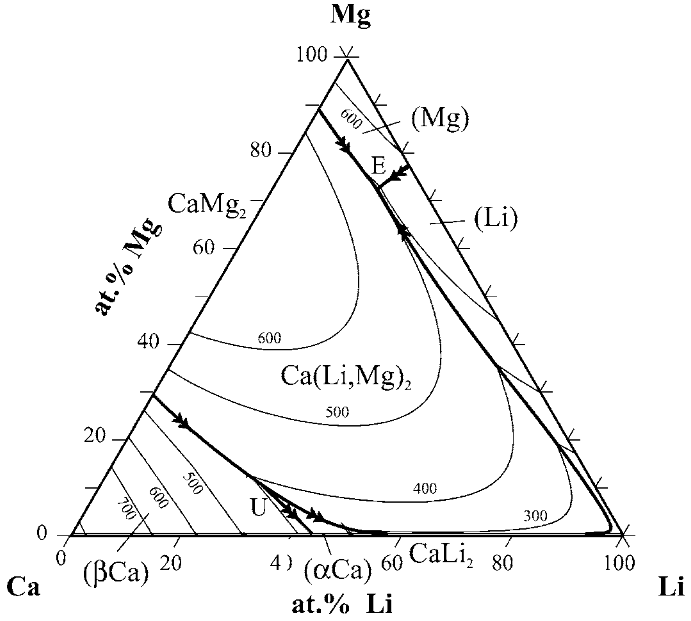

Fig. 5. Calculated Ca–Li–Mg liquidus surface.

the experimental error as seen in Table 2. The overall agreement of the calculated binary phase diagram with the available experimental data is satisfying. The calculation also agrees with the reviewed data of Borstedt and Guminski [9].

From this experimental investigation in the ternary system Ca–Li–Mg, a continuous solid solution between $CaLi_2$ and $CaMg_2$ was found. This is a most important finding for the phase diagram of the Ca–Li–Mg system. The lattice parameters within this solid solution change very little, while substituting Li by Mg atoms in the crystal lattice. This phase is also the dominating phase in the liquidus surface. Only one ternary interaction parameter ($A + BT$) of this solid solution phase is necessary to fit the experimentally found three-phase equilibrium composition at 150 °C and the measured liquidus temperature. All experimental results show agreement with the calculation, both in the binary Ca–Li and the ternary Ca–Li–Mg system. The section $CaMg_2$–$CaLi_2$ is definitely a pseudobinary system.

Based on this consistent dataset, a variety of ternary phase diagram sections or phase amount charts can be calculated. Most interesting for applications may be

Table 3
The ternary invariant reactions

Invariant reactions	Type	Calculated temperature in (°C)	Calculated compositions			
			Phase at.%	Ca	at.%Li	at.%Mg
$L = (Mg) + (Li) + Ca(Li,Mg)_2$	E	491	L	8.4	19.1	72.5
			(Mg)	0.4	17.2	82.4
			(Li)	0.3	25.9	73.9
			$Ca(Li,Mg)_2$	33.3	3.0	63.7
$\beta Ca + Ca(Li,Mg)_2 = \alpha Ca + L$	U	391	L	60.6	28.8	10.5
			(βCa)	93.8	5.4	0.8
			$Ca(Li,Mg)_2$	33.3	31.1	35.6
			αCa	95.2	4.1	0.7

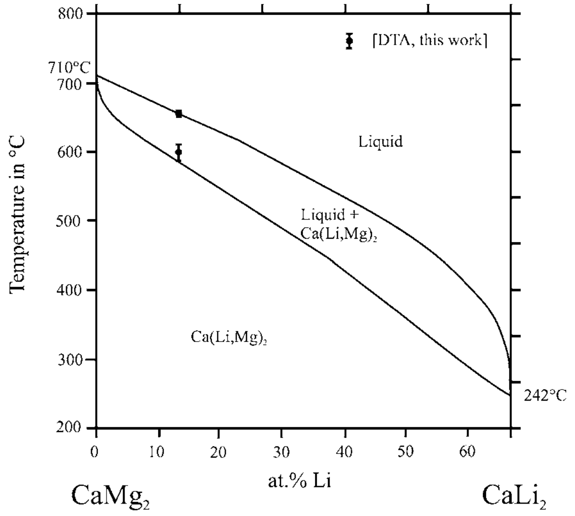

Fig. 6. Calculated pseudobinary section $CaMg_2$–$CaLi_2$ including own DTA results of liquidus and solidus.

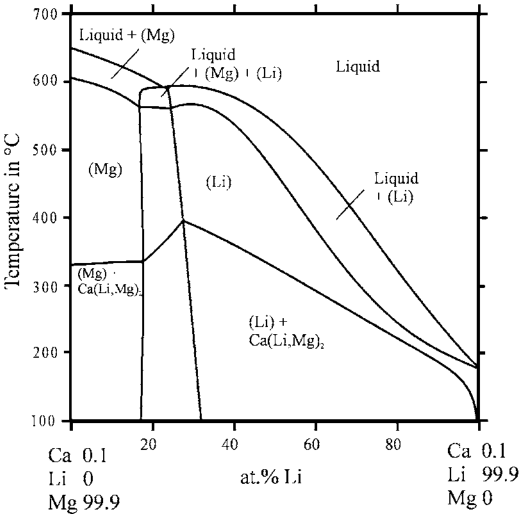

Fig. 8. Calculated ternary phase diagram section Mg–Li with constant 0.1 at.% Ca.

the vertical sections Mg–Li with constant additions of 0.1 at.% Ca and 0.5 at.% Ca in Figs. 8 and 9. It should be noted that in Fig. 8, the limit of the (Mg) single phase field at 330–334 °C represents the cut through the ternary solvus surface of the $Ca(Li,Mg)_2$ phase. That line originates at 330 °C in the binary Mg–Ca system where the assessed [1] solubility limit is 0.1 at.% Ca.

The solidification behavior of Mg–Li alloy with only 0.1 at.% Ca addition, Fig. 8, resembles qualitatively that of the binary Mg–Li system. The liquidus lines are virtually unchanged, whereas, the solidus

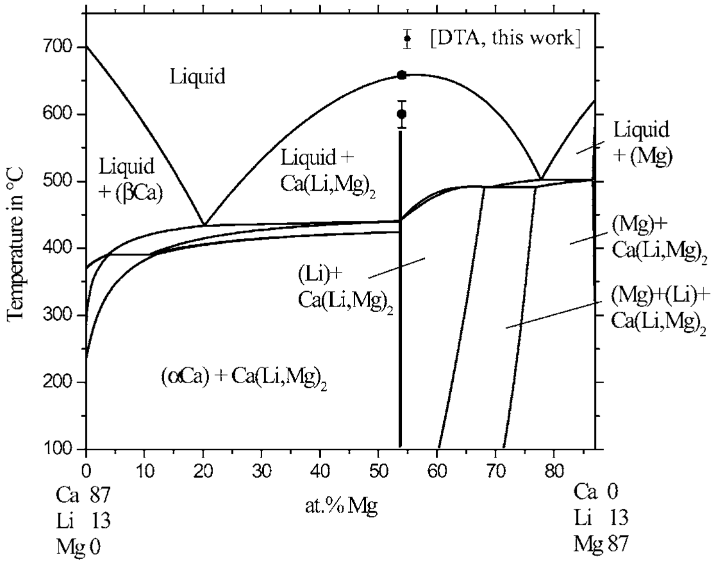

Fig. 7. Calculated ternary phase diagram section Ca–Mg with constant 13 at.% Li.

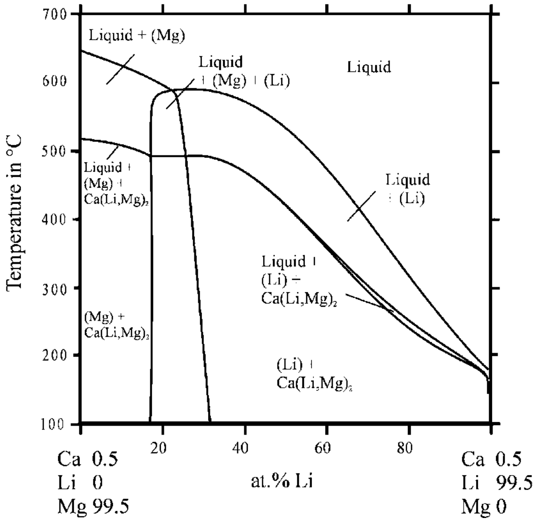

Fig. 9. Calculated ternary phase diagram section Mg–Li with constant 0.5 at.% Ca.

lines are shifted to lower temperatures. This generates substantial solidification intervals at the melting "point" of "Mg", the eutectic reaction $L = (\text{Mg}) + (\text{Li})$ and the melting maximum of the (Li) solution phase. Precipitation of the Ca(Li,Mg)_2 compound occurs in the solid state only, below 415 °C.

This is quite different for an addition of 0.5 at.% Ca as given in Fig. 9. The primary solidification of (Mg) or (Li), starting at almost the same liquidus temperatures as before, extends now all the way down to the eutectic type three-phase equilibria where Ca(Li,Mg)_2 precipitates in secondary solidification. This secondary solidification of Ca(Li,Mg)_2 occurs in a very narrow temperature range above 506 °C in Mg-rich alloys. The ternary eutectic reaction E ($L = (\text{Mg}) + (\text{Li}) + \text{Ca(Li, Mg)}_2$) at 506 °C is observed for alloys between 17.1 and 25.5 at.% Li in Fig. 9. Almost, the same phase diagram section is obtained at higher Ca-content, 1 at.% Ca, except for the Li-rich end. However, the phase amounts, which cannot be read from Fig. 9, differ significantly between alloys with 0.5 or 1 at.% Ca addition. It is interesting to note that the phase diagram sections of Figs. 8 and 9 depend only very little on the modeling of the Ca(Li,Mg)_2 phase. Even if the interaction parameter given in Table 4 is changed from $-24000 + 25T$ J/mol to a constant value of -13000 J/mol, the phase boundaries involving the Ca(Li,Mg)_2 phase are shifted to at most 25–35 °C higher temperatures in the intermediate composition range of Figs. 8 and 9.

Table 4
Parameters assessed in the present work for the binary Ca–Li and the ternary Ca–Li–Mg system in (J/mol), phase descriptions bcc = (βCa) and (Li), fcc = (αCa), hcp = metastable

$$L^{0,\text{Liquid}}_{\text{Ca,Li}} = -5641.2 - 3.2065T$$
$$L^{0,\text{bcc}}_{\text{Ca,Li}} = 5918.9$$
$$L^{0,\text{fcc}}_{\text{Ca,Li}} = 6458.3$$
$$L^{0,\text{hcp}}_{\text{Ca,Li}} = 5000$$
$$G^{0,\text{CaLi}_2}_{\text{Ca:Li}} = -3893.97 + 2.132T + 0.333333\cdot G^{0,\text{fcc}}_{\text{Ca}} + 0.666667 G^{0,\text{bcc}}_{\text{Li}}$$
$$L^{0,\text{Ca(Li,Mg)}_2}_{\text{Ca:Li,Mg}} = -24000 + 25T$$

5. Conclusion

The combination of experimental measurements with thermodynamic calculations facilitate the assessment of ternary phase relations. Results from ternary samples at one temperature (150 °C) and a DTA key experiment combined with polythermal thermodynamic assessments of the binary systems, based on experimental enthalpy data, are sufficient for the description of the entire ternary system.

The ternary system Ca–Li–Mg is part of the development of a multicomponent magnesium database for the system Mg–Al–Ca–Li–Si. It is relevant for the focused design of new Mg alloys supported by computational thermochemistry. Especially in magnesium alloys, controlling the precipitation of phases like Ca(Li,Mg)_2 requires these well established thermodynamic data. The ternary Ca–Li–Mg is one module of our long-term investigation for technical multicomponent magnesium alloys.

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