J. Phase Equilib. Diffus. (2023) 44:679–686
https://doi.org/10.1007/s11669-023-01077-5

SHORT COMMUNICATION

Solubility Data of Eight Common Alloying Elements in Magnesium

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Submitted: 10 August 2023 / in revised form: 15 November 2023 / Accepted: 17 November 2023 / Published online: 3 December 2023

Abstract The solubility values of eight common alloying elements Al, Ca, Ce, Gd, Nd, Sn, Y and Zn in hcp Mg are experimentally measured from diffusion profiles obtained from diffusion multiples and liquid-solid diffusion couples (LSDCs) using electron probe microanalysis. These solubility values are used to stablish solidus and solvus lines and compared with the experimental results reported in the literature as well as the computed phase boundaries using two CALPHAD (CALculation of PHAse Diagrams) databases. Our experimental values for Mg-Ca (530, 580, 600, 630 °C), Mg-Ce (605, 630 °C), Mg-Gd (570, 600, 630 °C) and Mg-Nd (615, 630 °C) are the first ever measurements of the hcp solidus for these four binary systems. Additional solubility data obtained from our experiments are reported for Mg-Al (375, 420, 450, 500, 550, 600 °C), Mg-Sn (375, 420, 500, 550, 600 °C), Mg-Y (590, 610, 630 °C), and Mg-Zn (275, 450, 500, 550 °C). Our experimental data are valuable input to future thermodynamic reassessments of the eight binary systems. This study also clearly shows the effectiveness of measuring solidus data using the elegant LSDCs.

Keywords CALPHAD · magnesium · phase diagram · solubility · solidus · solvus

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1 Introduction

Magnesium alloys are highly attractive lightweight metallic materials for vehicles to improve the energy efficiency in the transportation sector as well as biocompatible implant materials.[1–8] The Integrated Computational Materials Engineering (ICME) approach is now widely used to accelerate the design of Mg alloys.[9–11] CALPHAD (CALculation of PHAse Diagrams) is one of the most important ICME tools as it predicts the phase equilibria for given alloy compositions to promote the desirable phases to improve the mechanical properties of Mg alloys.[12–16] The parameters in the underlying CALPHAD Mg thermodynamic databases are optimized from experimental data such as solubility values of alloying elements in Mg. Efficient approaches of determining multiple solidus and solvus solubility data along a temperature gradient in a single experiment was developed by Zhao et al. and Engelhardt et al., respectively.[17,18] In this study, we obtained solubility values using diffusion-couple based experimental methods.

The experimental solubility data of eight solutes Al, Ca, Ce, Gd, Nd, Sn, Y and Zn in hcp magnesium are reported in this short communication and compared with literature experimental data as well as computed phase boundaries using two commercial CALPHAD software packages-Pandat (database used: PanMg2019) and Thermo-Calc (database used: TCMG5). Our solubility data were extracted from diffusion profiles obtained using electron probe microanalysis (EPMA) on both liquid-solid diffusion couples (LSDCs) and diffusion multiples. Those samples were initially prepared for studying the diffusion coefficients of Mg-based binary systems[19–23] and the experimental profiles are directly employed for this study without collecting more EPMA data. The process of obtaining the solubility data from LSDCs is explained in Fig. 1 and the detailed process of making LSDCs can be found in Refs.[19–22] LSDCs are designed to promote the formation of a liquid phase inside (being contained) a block of pure Mg. Diffusion of a solute element from the liquid phase into Mg forms a solid solution gradient to allow the solubility to be well defined as shown in Fig. 1. Below the eutectic temperatures of the binary systems, diffusion multiples are very efficient ways to determine isothermal phase diagrams, including the solubility values.[23–28] The process of using diffusion multiples to obtain the solubility data is illustrated in Fig. 2 and the detailed process of making the Mg-containing diffusion multiples can be found in Ref. [23]

Figure 3 summarizes all the solubility values of Al in hcp Mg from measured diffusion profiles of four Mg-Al LSDCs above the eutectic temperature and four diffusion multiples (two sets of different diffusion multiples and each annealed at two temperatures) below the eutectic temperature. The diffusion profiles at above and below eutectic point are shown in Fig. 3(b) and (c), respectively. Our experimental solubility data points are presented as red dots in the Mg-rich part of the Mg-Al phase diagram in Fig. 3(a) in comparison with the literature data and the computed solidus and solvus lines using Pandat.

The solubility values of Al, Sn, Y and Zn in hcp Mg for both the solidus and solvus curves obtained from both LSDCs and diffusion multiples are summarized in Fig. 4. The solubility values of Al in hcp Mg have been well studied in the literature.[29–36] Six new data points from this study are shown in Fig. 4(a) and (b) in comparison with the solubility data from the literature as well as the computed solidus and solvus curves. Our four solubility values above the eutectic temperature are in excellent agreement with the literature data and the computed results. Our other two data points below the eutectic temperature are slightly lower than the literature data and the computed solvus curves, but still in overall good agreement. Since our

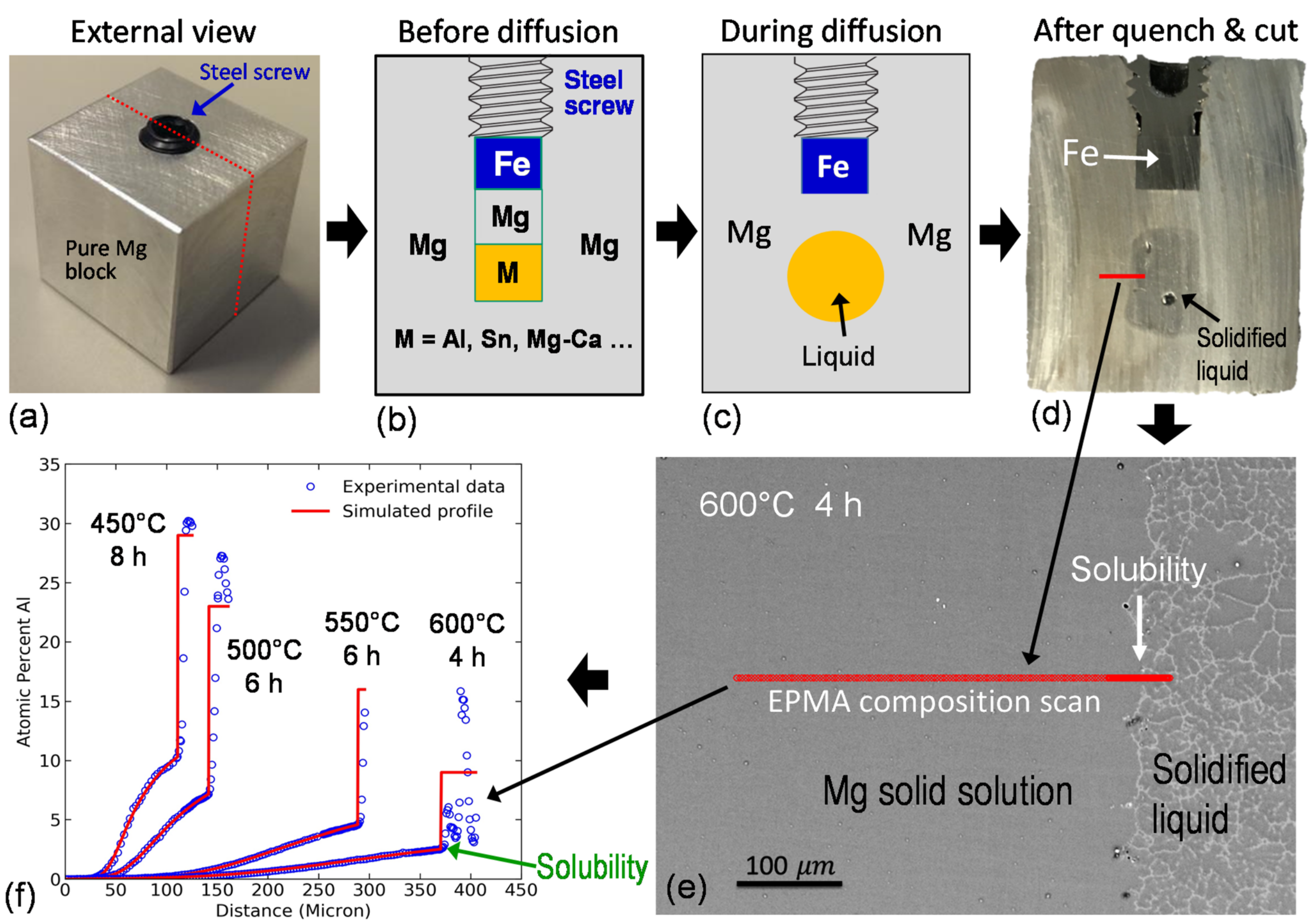

Fig. 1 Application of LSDCs to the determination of solubility values of elements in Mg above the eutectic temperatures: (a) to (d) Preparation of a LSDC; (e) Example of an EPMA scan superimposed on an SEM backscattered electron image of an Mg-Al LSDC that was diffusion annealed at 600 °C for 4 h; and (f) diffusion profiles of four LSDCs superimposed together including the one that was annealed 600 °C for 4 h where the solubility is marked with an arrow. The detailed process of making LSDCs can be found in Refs. [19–22]

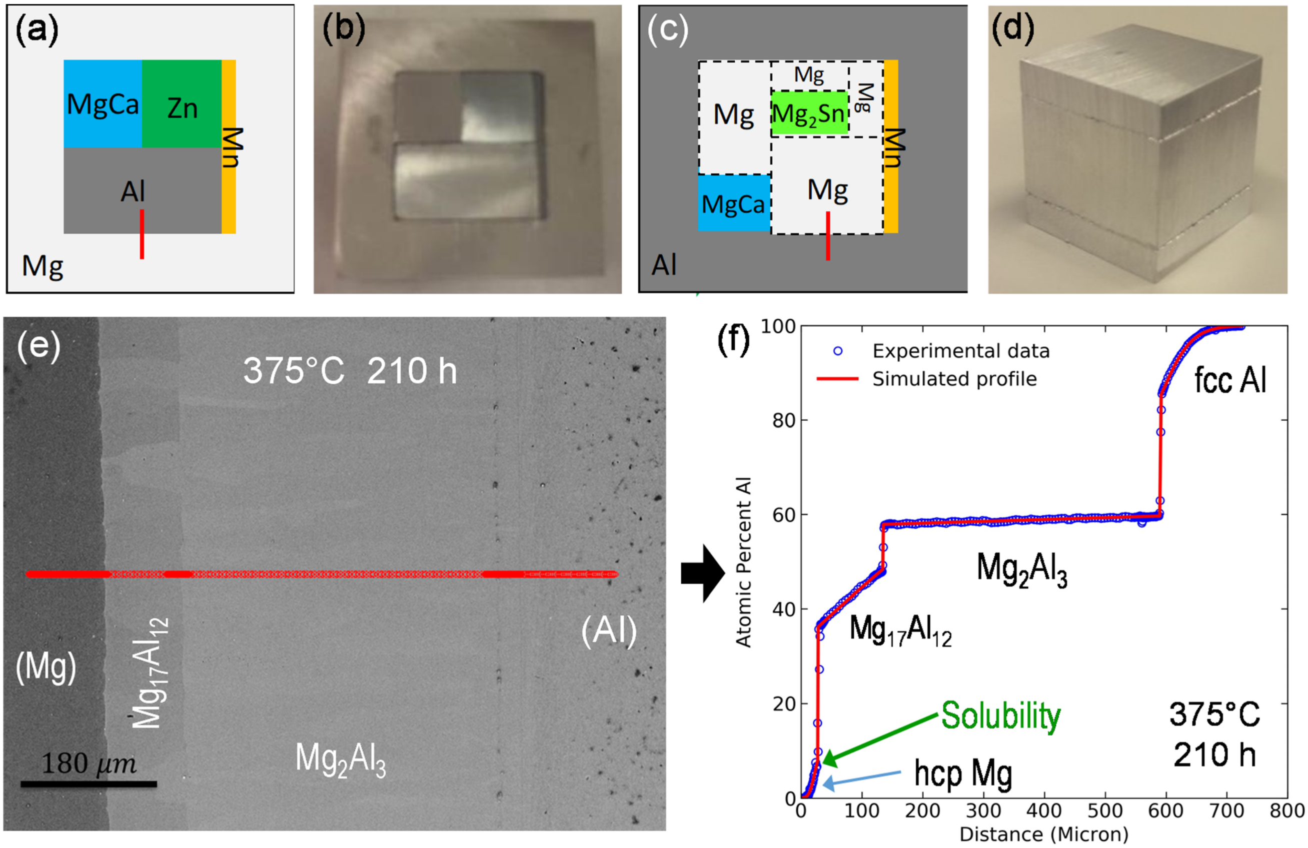

Fig. 2 Application of two diffusion multiples to the determination of solubility of elements in Mg below the eutectic temperatures: (a) and (b) A diffusion multiple containing Mg-Al-Ca-Mn-Zn [(b) is a cross-sectional photo of (a)]; (c) and (d) A diffusion multiple containing Al-Ca-Mg-Mn-Sn [(d) is a photo of diffusion multiple in (c)]; (e) An EPMA scan line (red) crossing the diffusion zone of the Mg-Al binary diffusion couple region of a diffusion multiple that was annealed at 375 °C for 210 h (SEM backscattered electron image); and (f) Composition profile crossing the red line in (e) showing the compositions of the phases and the solubility data. The detailed process of making these two diffusion multiples can be found in Ref. [23] (Color figure online)

diffusion multiples were annealed for up to 1760 h, it is likely that our data are quite reliable.

Figure 4(c) and (d) summarize the solubility values of the Mg-Sn system. In the region above eutectic temperature, all the data points show good agreement with two computed solidus curves. Below the eutectic temperature, our solubility data are in excellent agreement with the results reported by Raynor, Vosskuhler, and Willey.[37–39] The solubility data reported by Grube and Vosskuhler are lower than our data at temperatures below 500 °C,[40] and solubility values reported by Nayak and Oelsen[41] using calorimetric method are substantially higher than our solubility values at 500 °C, 420 °C, and 375 °C. Near the eutectic temperature of ~ 550 °C, the solubility value from our experiment and that of Grube and Vosskuhler are significantly higher than other literature data. The extrapolated composition of the hcp solid solution at the eutectic temperature using our experimental solubility values will be higher than that using other datasets. Two solvus curves computed using Pandat and Thermo-Calc differ appreciably, possibly due to the use of different thermodynamic assessments that have relied on different experimental datasets. The excellent agreement of our data with those reported by Raynor, Vosskuhler, and Willey[37–39] indicates that the thermodynamic parameters used in the Thermo-Calc TCMG5 database could predict the solvus line of the hcp Mg-Sn solid solution better than that using the Pandat PanMg2019 database. These four consistent datasets also indicate that Sn can be a very effective element to induce precipitation-strengthening due to the pronounced solubility decrease with decreasing temperatures.

Our solubility data of Y in hcp Mg at both 590 °C and 610 °C are lower than the literature data[42–47] and the computed solvus lines from both Pandat and Thermo-Calc but with a better agreement with the Thermo-Calc result, as shown in Fig. 4 (e) and (f). Our solubility value at 630 °C agrees well with the computed results. The solidus and solvus curves from both Pandat and Thermo-Calc are overall consistent, despite a slightly higher eutectic temperature from Pandat than that from Thermo-Calc.

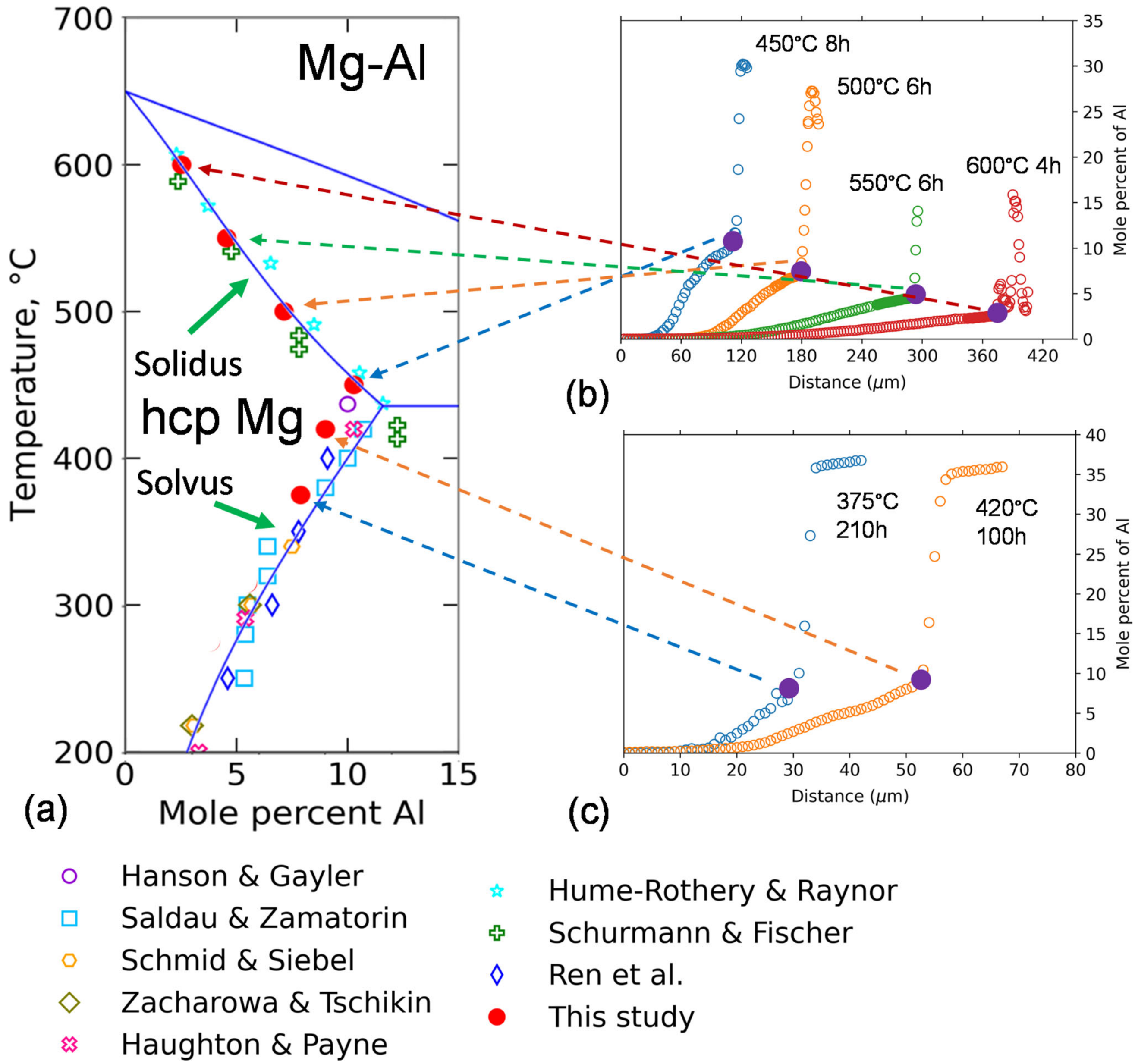

Fig. 3 The process of extracting solubility values from diffusion profiles: (a) Solubility data from this study and the literature [29–36] together with the computed solubility lines using the Pandat software; (b) Diffusion profiles from four LSDCs at temperature above the eutectic temperature; and (c) Mg-rich part of the diffusion profiles from two diffusion multiples that were diffusion annealed at temperatures below the eutectic temperature

Our experimental solubility data of Zn in hcp Mg at 550 °C, 500 °C, and 450 °C obtained from three LSDCs are appreciably higher than those reported by Park and Wyman,[48] as shown in Fig. 4(g) and (h). The results call for more experimental measurements in the future to see whether the higher solubility values are reliable. Our solubility value measured at 275 °C from a diffusion multiple agrees well with the other literature data.[49–51]

Our solubility data of the other four solutes Ca, Ce, Gd, and Nd were all obtained from LSDCs and are summarized in Fig. 5. All these data are first measurements in the world of the solidus of the hcp Mg phase for the Mg-Ca, Mg-Ce, Mg-Gd, and Mg-Nd systems. The new experimental data fill the gap of missing data for these systems and will be very valuable input to future enhanced thermodynamic reassessments of these binary systems. The calculated phase diagrams using the current TCMG5 and PanMg2019 databases are also included in Fig. 5 for comparison together with the literature experimental data below the eutectic temperatures for Mg-Ca,[52–56] Mg-Ce,[48,57–61] Mg-Gd system,[47,62–64] and Mg-Nd[48,59,65–67] systems.

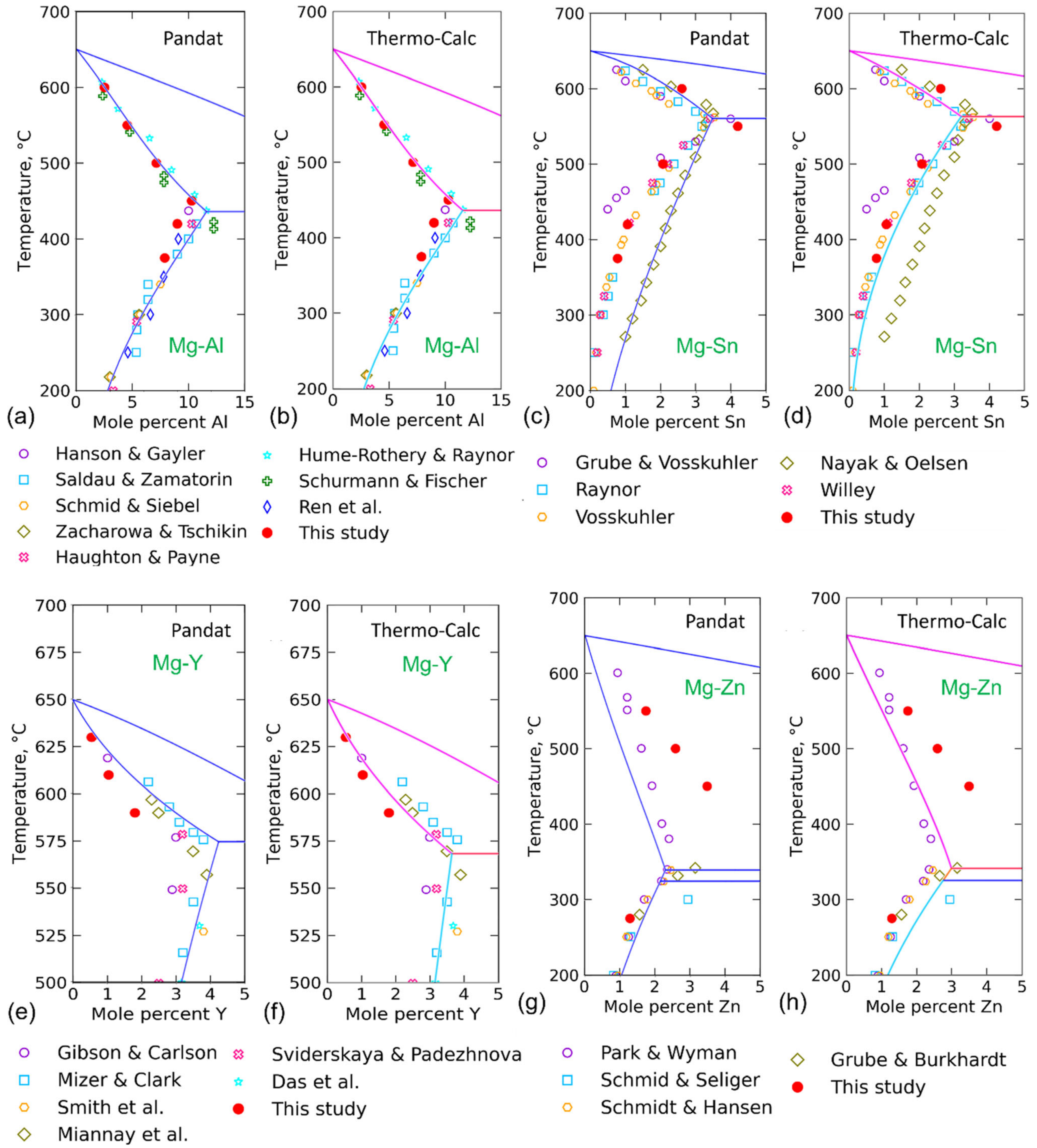

Fig. 4 Comparison of the experimental solubility data from the current study with the literature experimental data as well as the computed phase boundaries using both Pandat and Thermo-Calc and their associated databases: (a) and (b) for Mg-Al, (c) and (d) for Mg-Sn, (e) and (f) for Mg-Y, and (g) and (h) for Mg-Zn, respectively

In summary, solubility values of eight solutes Al, Ca, Ce, Gd, Nd, Sn, Y and Zn in hcp Mg at various temperatures are measured using experimental diffusion profiles collected from diffusion multiples and LSDCs. The results clearly show that LSDC is a very effective way to measure the solidus values. The solidus data for the hcp Mg phase for Mg-Ca, Mg-Ce, Mg-Gd, and Mg-Nd are the first such experimental measurements in the world and will be very valuable input to future reassessment of the thermodynamic parameters of these binary systems. Our data and the

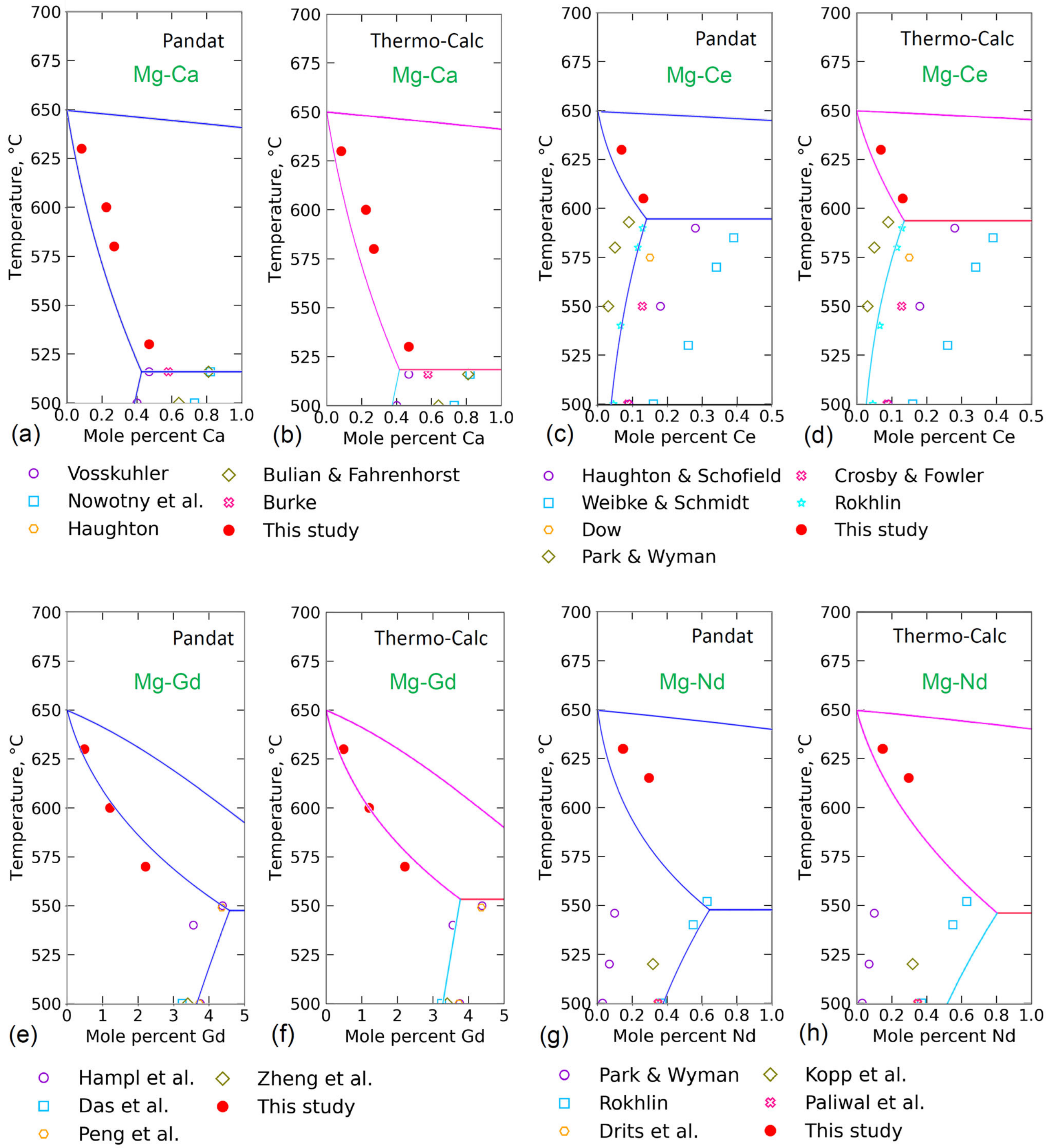

Fig. 5 Comparison of the experimental solubility data from the current study with the literature experimental data as well as the computed phase boundaries using both Pandat and Thermo-Calc and their associated databases: (a) and (b) for Mg-Ca, (c) and (d) for Mg-Ce, (e) and (f) for Mg-Gd, and (g) and (h) for Mg-Nd, respectively. The solidus data are the first experimental measurements for the hcp Mg phase of these four binary systems

good agreement with the data reported by Raynor, Vosskuhler, and Willey[37–39] show a more pronounced temperature dependence of the Sn solubility in hcp Mg, which is good for promoting precipitation strengthening. The above four consistent datasets indicate the solubility data reported by Nayak and Oelsen[41] at 500 °C, 420 °C, and 375 °C may not be very reliable. We also reported much higher solubility of Zn in hcp Mg than data reported by Park and Wyman above the eutectic temperature.

Acknowledgment This study was funded by the US. National Science Foundation (NSF) Division of Civil, Mechanical and Manufacturing Innovation (CMMI) through award #2004979 with Dr. Alexis Lewis as the Program Director. The original experimental samples of LSDCs and diffusion multiples were made during a study funded by the Vehicle Technology Program of the Office of Energy Efficiency and Renewable Energy (EERE) of the U.S. Department of Energy under contract number DE-EE0006450 with Dr. William Joost as the Program Manager.

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