Phase Equilibria of the Constituent Ternaries of the Mg-Al-Ca-Sr System

M. Aljarrah, M. Medraj, Jian Li, and E. Essadiqi

Thermodynamic modeling of the Al-Ca-Sr, Mg-Ca-Sr, Mg-Al-Ca and Mg-Al-Sr systems was conducted using the modified quasichemical model. A self-consistent database has been established for these systems. Mg-Al-Ca and Mg-Al-Sr ternary systems were studied experimentally through microstructure characterization, phase identification, and thermal analysis and thermodynamic modeling based on these experimental findings. It has been observed that the intermetallic compounds in the Mg-Ca, Mg-Sr, Al-Ca, and Al-Sr binary systems dissolve the third component in the respective ternary phase diagrams. In addition, two ternary compounds, $Mg_{56}Al_{40}Sr_4$ and $Mg_2Al_4Ca_3$, have been reported.

INTRODUCTION

Magnesium alloys are being selected at an increasing rate for structural applications, particularly in the automotive and aerospace industries. The cause of this dramatic growth is driven by automotive companies' goals of reducing vehicle weight, optimizing fuel consumption, and minimizing emissions.[1,2] The high specific strength of magnesium alloys makes them attractive for various structural applications. However, the poor creep resistance of commercial magnesium alloys has prevented their use in major powertrain components, such as engine blocks, where the operating temperature can be as high as 250°C.[3] To address this, significant effort has been made to develop creep-resistant alloys and these efforts have resulted in a number of promising alloys. It has been shown that the high-temperature strength and creep resistance of Mg-Al alloys can be improved by alloying with Sr and Ca,[4–6] two elements that are particularly appealing to the carmakers due to their low cost.

In order to define the processing conditions for making Mg-Al-Ca-Sr-based alloys and subsequent treatments to obtain the optimum mechanical properties, knowledge of the phase diagram and thermodynamic properties of these alloys is essential. A precise description of the ternary sub-systems in the Mg-Al-Ca-Sr system provides an opportunity to apply the phase equilibria aspects of alloy development. This will enable tracking of individual alloys during solidification and subsequent heat treatment by calculating the phase distributions and compositions from the obtained data. This paper reviews the literature of these systems and extends that work theoretically and experimentally in the Mg-Al-Ca and Mg-Al-Sr ternaries.

See the sidebar for details on experimental procedures and thermodynamic modeling.

How would you…

…describe the overall significance of this paper?

Adding Ca and/or Sr to Mg-Al alloys has been found to be beneficial not only to keep cost low but also to improve mechanical properties, especially strength and creep resistance, at relatively high temperature. Therefore it is very important to understand the equilibrium in the Mg-Al-Ca-Sr system in order to develop the processing conditions and the subsequent heat treatments for such promising alloys.

…describe this work to a materials science and engineering professional with no experience in your technical specialty?

Combining computational thermodynamics with key experiments reduces the time and effort required to understand the phase equilibria in multicomponent systems. A preliminary thermodynamic model was developed for the constituent ternaries of the Mg-Al-Ca-Sr system based on the binary systems and the experimental results from the literature. This model was modified based on the current XRD, EPMA, and DSC results of key compositions in the ternary Mg-Al-Ca and Mg-Al-Sr systems.

…describe this work to a layperson?

Magnesium alloys have high specific properties due to their low density. Using Mg alloys in cars and planes will lead to reducing their weight, fuel consumption, and gas emission. This makes Mg alloys very attractive for the transportation industry. The current work provides an understanding of the equilibrium in the constituent four ternary systems of one of the very important Mg alloys systems.

LITERATURE REVIEW

Mg-Al-Ca System

Several researchers[7–19] studied the Mg-Al-Ca system experimentally. Among them, J. Gröbner et al.,[9] A.A. Luo et al.,[12] Y. Zhong et al.,[17] and M. Aljarrah et al.[19] suggested the presence of ternary solubilities of the third element in the binary compounds which are responsible for the improved creep resistance of the Mg-Al-Ca alloys.

A. Suzuki et al.[15] reported that C36, with the nominal composition $Mg_{52}Al_{30}Ca_{18}$, is a high-temperature phase existing between Mg_2Ca and Al_2Ca, but transforms to Mg and Al_2Ca at lower temperature. However, this chemical composition is different from the one reported later by A. Suzuki et al.[16] as $Mg_2Al_4Ca_3$. It is worth mention-

ing that the latter composition is similar to what was reported by S. Amerioun et al.[13] In addition, Suzuki et al.[16] found that C36 compound forms during eutectic reaction L↔Mg+C36+Mg_2Ca supporting their previous finding. Since the existence of this compound was not taken into account in the previous assessments, the reported isothermal sections in the literature claimed that Mg was in equilibrium with Al_2Ca. Therefore, considering the new ternary compound and the extended solubilities of the binary compounds in the thermodynamic modeling will alter the description of the Mg-Al-Ca system. Based on these findings and the experimental results of the current work, the liquidus projection and the isothermal section at 300°C of this system are presented.

Mg-Al-Sr System

In order to resolve the discrepancies in the Mg-Al-Sr system obtained by previous researchers,[23–26] M.A. Parvez et al.,[21] A. Janz et al.,[22] and M. Aljarrah et al.[27] studied the Mg-Al-Sr system and provided comprehensive discussions of their experimental results. According to Janz et al.[22] and Aljarrah et al.,[27] ternary solubilities of the binary compounds in the Mg-Sr and Al-Sr systems extended into the ternary system and new ternary compounds have been reported. H. Cao et al.[30] studied the Mg-Al-Sr system using three alloys in the Mg-Al rich region. They reported extended solubilities of three binary compounds (Al_4Sr, $Mg_{17}Sr_2$, and $Mg_{38}Sr_9$) and the calculated liquidus projection and isothermal section were based on these solubilities. In their work,[30] thermal analysis of the studied alloys was not carried out and they did not investigate the solubility of the other binary compounds.

In our work, key experiments were chosen to measure the extended solu-

Table I. Optimized Thermodynamic Parameters of the Ternary Compound and Solid Solubilities

Phase	Parameter (J/mol·atom)
(Mg_2Ca)	$G^{0,Mg_2Ca}_{Al:Ca} = 146.3$
	$L^{0,Mg_2Ca}_{Mg,Al:Ca} = -121100 + 13T$
(Al_2Ca)	$G^{0,Al_2Ca}_{Mg:Ca} = 13946.7$
	$L^{0,Al_2Ca}_{Mg,Al:Ca} = -33611.3$
(Al_3Ca_8)	$G^{0,Al_3Ca_8}_{Mg:Al} = 38036.4$
	$L^{0,Al_3Ca_8}_{Ca,Al:Mg} = -83680$
$Mg_2Al_4Ca_3$	$G^{0,C36}_{Mg:Al:Ca} = -26044 - 36 * T$

EXPERIMENTAL PROCEDURES AND THERMODYNAMIC MODELING

Twelve Mg-Al-Ca and 11 Mg-Al-Sr alloys were prepared by melting stoichiometric amounts of the constituent elements in an induction furnace under flowing argon with 1%SF_6 to protect the melt from oxidation. In preparing the alloys, magnesium of 99.8 wt.%, aluminum of 99.9 wt.%, strontium of 99 wt.%, and calcium of 99 wt.% purities were used. The melt was stirred during melting and the alloys were re-melted three times to ensure homogeneity. Every sample was cut into two halves; an as-cast sample for thermal analysis using differential scanning calorimetry (DSC) experiment and a sample for heat treatment to construct the isothermal sections at 400°C and 300°C for the Mg-Al-Sr and Mg-Al-Ca systems, respectively. For this purpose, the samples were analyzed using x-ray diffraction, scanning electron microscopy, and electron probe micro-analyzer.

The chemical compositions of the as-cast alloys were analyzed using inductively coupled plasma-atomic emission spectrometry. Alloys containing high levels of Ca and Al were susceptible to vaporization losses of these elements. Accordingly the actual composition was used for the comparison with the thermodynamic calculations. The loss of Ca and/or Sr was below 3 wt.% for alloys containing less than 40 wt.% Ca and/or Sr. In alloys containing more than this concentration, the loss of these elements was in the range of 4–5 wt.%. In addition, below 2 wt.% loss of Al was found in alloys containing more than 50 wt.% Al. Differential scanning calorimetry experiments were performed using a Setaram Setsys DSC-1200 instrument. Temperature calibration of the DSC was done using pure Mg and Al. The samples were cut and mechanically polished to remove any possible contaminated surface layers. Afterwards, they were cleaned with acetone and placed in a graphite crucible with a lid cover to contain Mg vapors and protect the apparatus. To avoid oxidation, multiple evacuations followed by rinses with pure argon were done. The DSC measurements were performed at 5°C/min. heating and cooling rates. Slower heating rates were tried and were not found to reveal additional thermal arrests. The weight of the sample was 40–50 mg. During the calibration, it was made certain that the geometrical dimensions, the mass (40–80 mg) and the surface quality did not show any visible effects on DSC spectra. The reproducibility of every measurement was confirmed by collecting the data during three heating and cooling cycles. The estimated error between the repetitive heating and cooling is ±1°C or less. However, the solidification behavior can be revealed much better with the cooling scans.

In the binary sub-systems, thermodynamic modeling has been carried out using compound energy formalism for the binary intermediate solid solution. The liquid phase was modeled using the modified quasichemical model and the disordered solution model was used for the terminal solid solutions. The optimized binary thermodynamic parameters were published by the same authors.[29,31,32]

In the current work, ternary solubility of Al in all the binary compounds of the Mg-Sr and Mg-Ca systems was observed. Similarly, some of the Al-Sr and Al-Ca binary compounds dissolve Mg. These ternary solubilities are modeled with two sublattices with Sr atoms occupying the first lattice. In view of the fact that atomic size of Mg and Al are quite similar, these two elements replace each other in the Mg-Sr and Mg-Ca compounds and their mixing is allowed on the second sublattice. However, for the Al_3Ca_8, Mg and Ca are found to replace each other. For example, the Gibbs energy of $(Mg,Al)_{17}Sr_2$ is modeled using the compound energy formalism as follows:

$$G^{(Mg,Al)_{17}Sr_2} = y_{Mg} G_{Mg:Sr}^{\,0,(Mg,Al)_{17}Sr_2} + y_{Al} G_{Al:Sr}^{\,0,(Mg,Al)_{17}Sr_2} + \frac{17}{19} RT(y_{Mg} \ln y_{Mg} + y_{Al} \ln y_{Al}) + y_{Mg} y_{Al} L_{Mg,Al:Sr}^{0,(Mg,Al)_{17}Sr_2}$$

where $G_{Mg:Sr}^{\,0,(Mg,Al)_{17}Sr_2}$ is the Gibbs energy of formation of $Mg_{17}Sr_2$ in the binary Mg-Sr system and $G_{Al:Sr}^{\,0,(Mg,Al)_{17}Sr_2}$ represents the metastable end member of the solid solution in the binary Al-Sr phase diagram and it was given high positive value (10^5 J/mol). $L_{Mg,Al:Sr}^{0,(Mg,Al)_{17}Sr_2}$ describes the ternary interaction parameter within the sublattice, and this term was iteratively obtained so that the calculated phase transformation temperatures are consistent with those obtained from the DSC measurements.

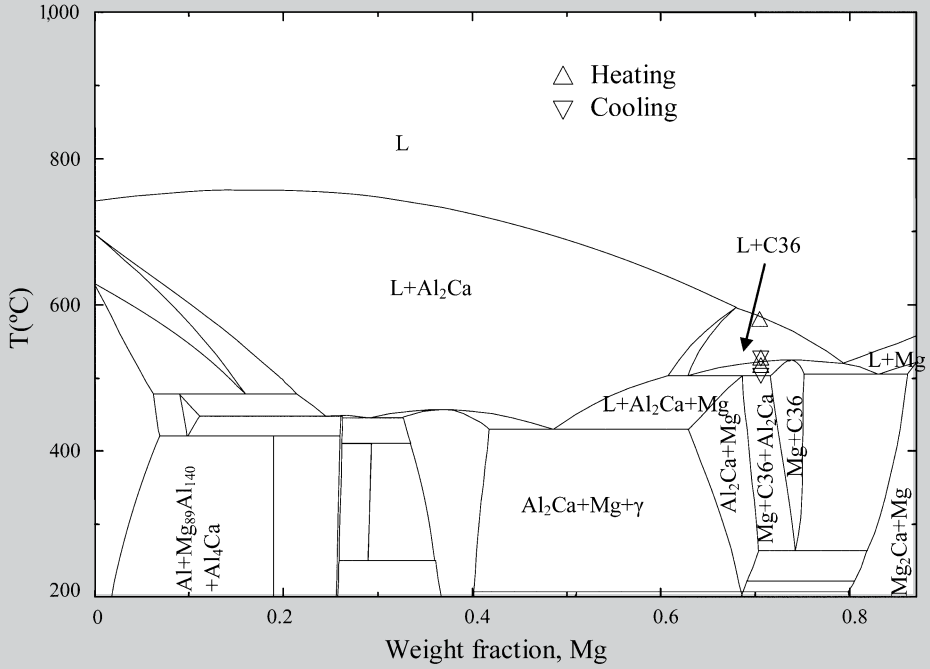
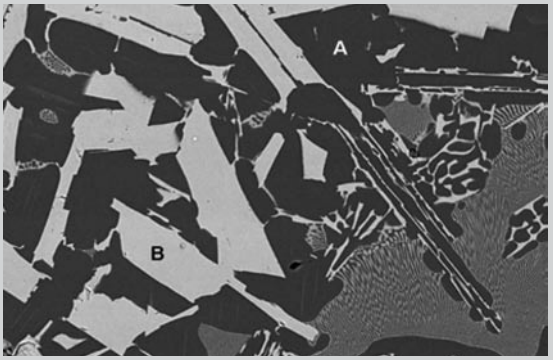


Figure 1. A calculated vertical section at 13 wt.% Ca with DSC signals from heating and cooling curves of (72/15/13 wt.% Mg/Al/Ca) alloy.



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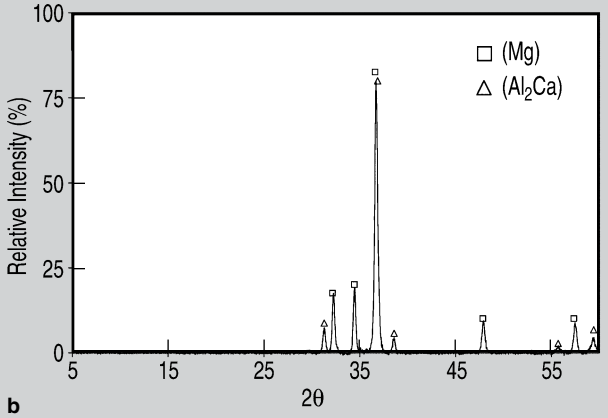
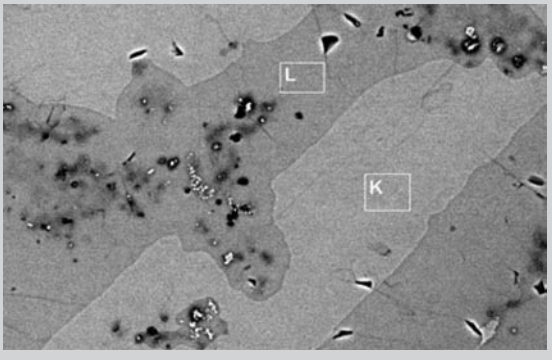


b

Region	Mg	Al	Ca	Phases
A	98.2±0.1	1.8±0.1	0.0±0.1	(Mg)
B	6.3±0.2	60.4±0.1	33.3±0.1	(Al_2Ca)

c

Figure 2. (a) SEM image, (b) XRD pattern, and (c) EPMA analysis of composition (72/15/13 wt.% Mg/Al/Ca).



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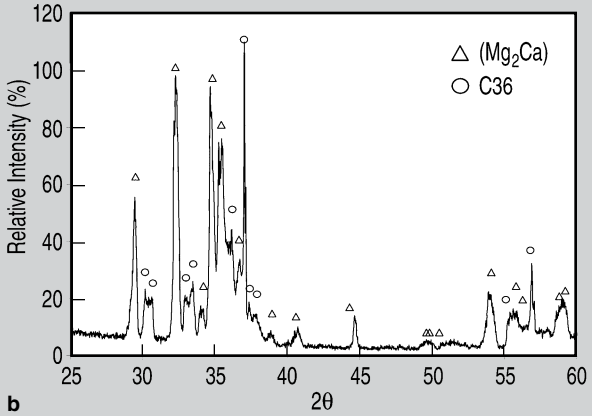

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Region	Mg	Al	Ca	Phases
L	51.3±0.7	16.9±0.4	31.8±0.3	(Mg_2Ca)
K	21.3±1.3	45.2±1.3	33.5±0.1	C36

c

Figure 3. (a) SEM image, (b) XRD pattern, and (c) EPMA analysis of composition (29/23/48 wt.% Mg/Al/Ca).

bilities of the binary compounds in the ternary system and to verify the chemical composition of the ternary compound. In addition, heat-treated samples were chosen to construct the 400°C isothermal section of the Mg-Al-Sr system.

RESULTS AND DISCUSSION

The phase transformation temperatures of Mg72Al15Ca13 alloy obtained through the differential scanning calorimetry (DSC) measurements are labeled on the vertical section shown in Figure 1. In this sample, the predicted phase transformation temperatures are consistent with the experimental results. Scanning electron microscopy (SEM) image, x-ray diffraction (XRD) pattern, and electron probe micro-analyzer (EPMA) analysis positively identified two phases (Al_2Ca) and (Mg), as can be seen in Figure 2. According to the EPMA analysis, (Al_2Ca) dissolves 6.3±0.2 at.% Mg, whereas (Mg) dissolves 1.8±0.1 at.% Al. Due to the slow kinetics of formation, C36 could not be observed in this sample. Thermodynamic predictions and the experimental results are consistent in terms of phase transformation temperatures. Ternary thermodynamic parameters of the

Table II. Ternary Solubility of the Binary Compounds in the Ternary Mg-Al-Ca System

	Solubility of the Third Element in the Binary Compounds (at.%)		
Phase	**From Ref. 9**	**This Work**	**Calculation**
(Al_2Ca)	5.0 at.% Mg	8.6 at.% Mg	7.6
(Al_3Ca_8)	10.0 at.% Mg	13.5 at.% Mg	12.5
(Mg_2Ca)	22.0 at.% Al	22.3 at.% Al	15.0

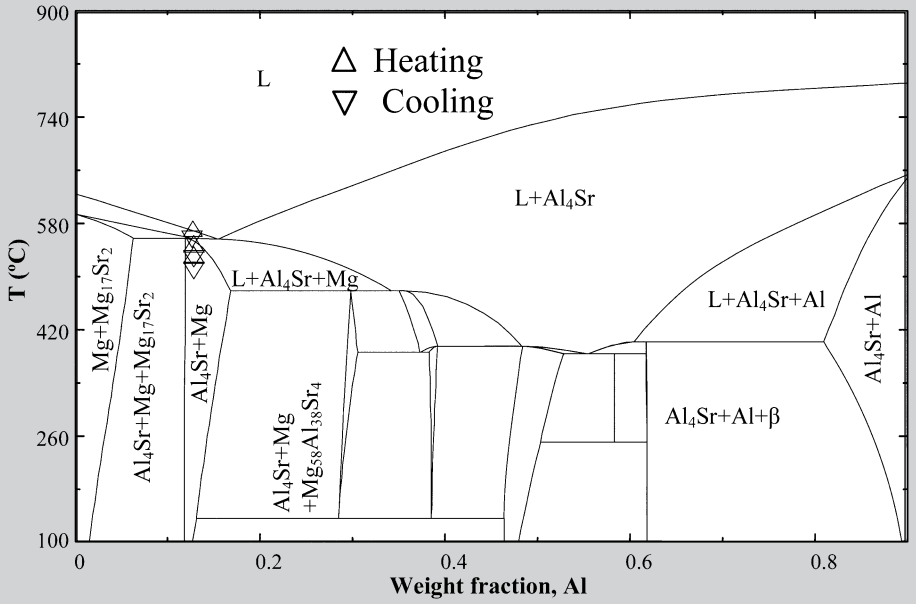
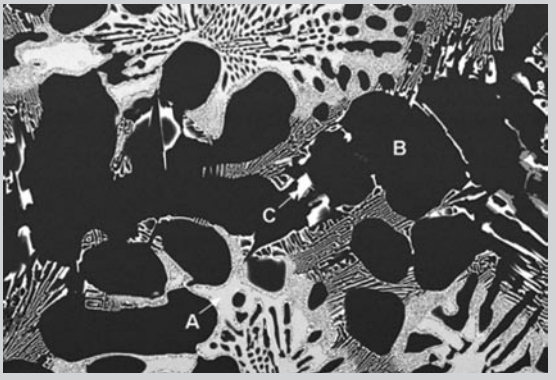


Figure 4. A calculated vertical section at 9.6 wt.%Sr with DSC signals from heating and cooling curves of Mg78Al12Sr9.6.



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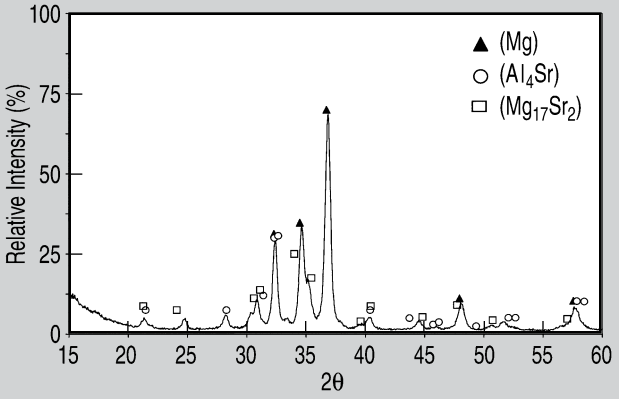

b

Location	at.% Mg	at.% Al	at.% Sr	Phases
A	76.2±0.2	14.2±0.1	9.6±0.1	($Mg_{17}Sr_2$)
B	98.0±0.3	2.0±0.3	0.0	(Mg)
C	9.4±2.2	70.0±2.2	20.6±0.6	(Al_4Sr)

c

Figure 5. (a) SEM image, (b) XRD pattern, and (c) EPMA analysis of Mg78Al12.4Sr9.6

Table III. Comparison between the Calculated and the Measured Solubility of the Binary Compounds in the Mg-Al-Sr System

	Solubility of the Third Element in the Binary Compounds (at.%)				
Phase	**Ref. 30**	**Ref. 25**	**Ref. 22**	**This Work**	**Calculation**
(Al_4Sr)	15.6	25.0	16.2	19.9	15.9
(Al_2Sr)	—	19.0	33.6	26.1	30.8
($Mg_{17}Sr_2$)	13.8	13.0	15.4	20.0	13.1
($Mg_{38}Sr_9$)	20.4	—	17.5	20.3	23.1
($Mg_{23}Sr_6$)	—	27.0	22.6	—	21.9
(Mg_2Sr)	—	12.0	22.2	26.1	18.6

Mg-Al-Ca system are presented in Table I.

The SEM image in Figure 3a shows two distinct phases: C36 and (Mg_2Ca), which are located in regions K and L, respectively. These phases were verified by the XRD and EPMA analysis as can be seen in Figure 3b and c. The chemical composition of the new ternary compound is quite close to that reported by Amerioun et al.[13] and Suzuki et al.[16]

In summary, ternary solubilities of three binary intermetallic compounds extended into the Mg-Al-Ca ternary system have been found and denoted as (Al_2Ca), (Al_3Ca_8), and (Mg_2Ca), as can be seen in Table II. In this work, the maximum solubility of Mg in Al_2Ca is found to be 8.6 at.%, compared to Gröbner et al.'s[9] results of 5.0 at.%. It was also observed that Mg_2Ca dissolves 22.3 at.% Al in accord with Gröbner et al.'s [9] work who reported 22.0 at.% Al. The binary compound Al_3Ca_8 in the Al-Ca system dissolves 13.5 at.% Mg, compared to Gröbner et al.'s[9] values as 10.0 at.% Mg.

The vertical section at 9.6 wt.% Sr with indicated DSC signals from cooling and heating curves are shown in Figure 4. The DSC spectra showed three phase transformations, two of which (the liquidus and solidus) were in good accord with the current calculations. As for the third signal, it represents the solid phase transformation which is due to a steep phase boundary and is not possible to detect precisely by DSC due to the small change in heat capacity associated with such transformation.

Three phases, (Mg), (Al_4Sr), and ($Mg_{17}Sr_2$), were positively identified in the XRD pattern as well as in the microstructure using EPMA, as shown in Figure 5. The SEM image shows that the dark Mg-matrix was separated by gray phase (region A) and bright precipitate (region C). (Al_4Sr) and ($Mg_{17}Sr_2$) are located at the grain boundaries region and appear to be lamellar. Quantitative EPMA analysis in Figure 5c shows that (Mg) and ($Mg_{17}Sr_2$) dissolve 2.0±0.3 and 14.2±0.1 at.% Al, while (Al_4Sr) dissolves 9.4±2.2 at.% Mg.

In this work, ternary solubilities of six binary compounds extended into the ternary Mg-Al-Sr system have been found and denoted as: (Al_2Sr), (Al_4Sr), ($Mg_{17}Sr_2$), ($Mg_{38}Sr_9$), and ($Mg_{23}Sr_6$), (Mg_2Sr). As can be seen in Table III, the maximum solid solubility of Mg in Al_4Sr is found to be 19.9 at.%, compared to M.M. Makmadohv et al.'s,[25] Janz et al.'s,[22] and Cao et al.'s[30] results as 25.0 at.%, 16.2 at.%, and 15.6 at.%, respectively.

It was also observed that Al_2Sr dissolves 26.1 at.% Mg, whereas Mak-

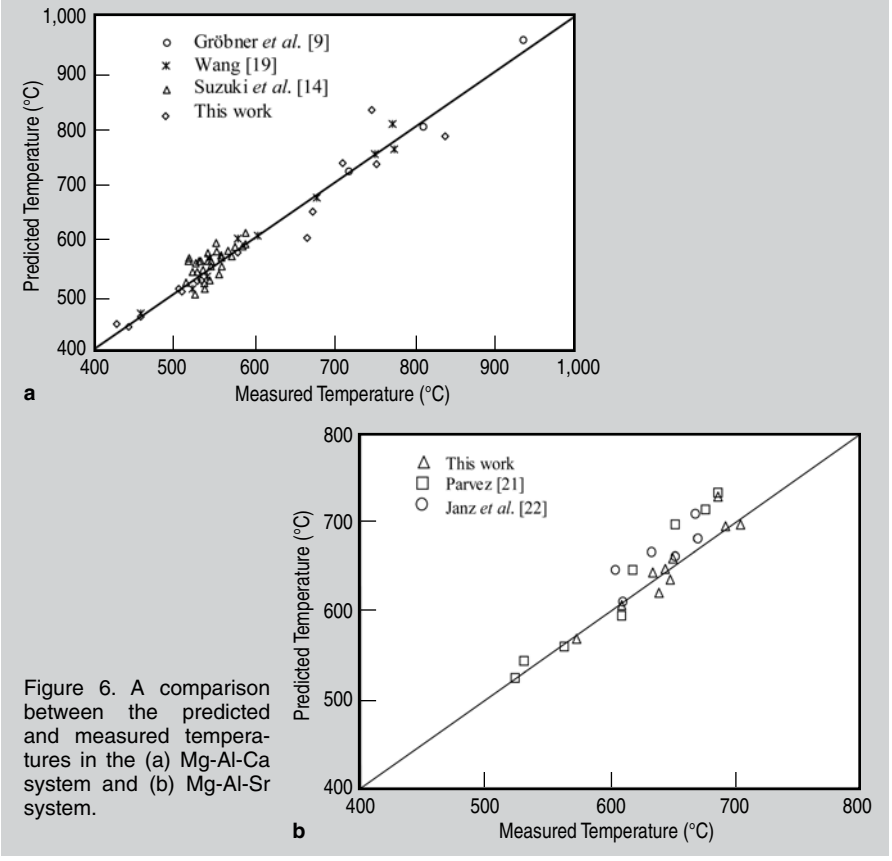

Figure 6. A comparison between the predicted and measured temperatures in the (a) Mg-Al-Ca system and (b) Mg-Al-Sr system.

Table IV. Ternary Thermodynamic Parameters of the Mg-Al-Sr System

Phase	**Parameter (J/mol·atom)**
Liquid	$\Delta g^0_{Mt,Al,Sr} = -45187 + 42T$
(Mg_2Sr)	$G^{0,(Mg,Al)_2Sr}_{At:Sr} = -27339.67 + 6.67T$
($Mg_{23}Sr_6$)	$G^{0,(Mg,Al)_{23}Sr_6}_{Al:Sr} = -28992 + 17.17T$ $L^{0,(Mg,Al)_{23}Sr_6}_{Mg,Al:Sr} = -5541.8$
($Mg_{38}Sr_9$)	$G^{0,(Mg,Al)_{38}Sr_9}_{Al:Sr} = -29679.4 + 17.1T$ $L^{0,(Mg,Al)_{38}Sr_9}_{Mg,Al:Sr} = -3790.6$
($Mg_{17}Sr_2$)	$G^{0,(Mg,Al)_{17}Sr_2}_{Al:Sr} = -13161.1 + 3.68T$ $L^{0,(Mg,Al)_{17}Sr_2}_{Mg,Al:Sr} = -1061.6$
(Al_4Sr)	$G^{0,(Mg,Al)_4Sr}_{Mg:Sr} = -9020.8 + 10T$
(Al_2Sr)	$G^{0,(Mg,Al)_2Sr}_{Mg:Sr} = -10069.7 + 2.83T$ $L^{0,(Mg,Al)_2Sr}_{Mg,Al:Sr} = 300$
$Mg_{58}Al_{38}Sr_4$	$G^{0,Mg_{58}Al_{38}Sr_4}_{Mg:Al:Sr} = -6010 + 200T$

madohv et al.[25] and Janz et al.[22] reported this solubility as 19.0 at.% and 33.6 at.% Mg, respectively. The ternary solubility obtained in this work is used in the current thermodynamic modeling of the Mg-Al-Sr system. The ternary thermodynamic parameters of the Mg-Al-Sr system are shown in Table IV.

Large solubilities of the Al in four binary compounds in the Mg-Sr system were observed. $Mg_{17}Sr_2$ dissolves 20.0 at.% Al, compared to Makmadohv et al.'s,[25] Janz et al.'s,[22] and Cao et al.'s[30] values as 13.0 at.%, 15.4 at.%, and 13.8 at.% Al, respectively. $Mg_{38}Sr_9$ dissolves 20.3 at.% Al compared to Janz et al.'s[22] and Cao et al.'s[30] results as 17.5 at.% and 20.4 at.% Al, respectively. Furthermore, it was found that Mg_2Sr dissolves 26.1 at.% Al, compared to Makmadohv et al.'s[25] and Janz et al.'s[22] results as 10.5 at.% and 22.2 at.% Al, respectively. Since the samples prepared to study the solubility of $Mg_{23}Sr_6$ were not successful because they were oxidized, the maximum solubility of Al in $Mg_{23}Sr_6$ was taken from the more recent work of Janz et al.[22] as 22.6 at.% Al.

As can be seen in Figure 6a and b, the measured liquidus temperatures obtained by DSC measurements and those predicted using thermodynamic modeling are consistent especially at low temperature. Based on the experimental results from this work and the literature, Mg-Al-Ca and Mg-Al-Sr isothermal sections at 300°C and 400°C, respectively, were drawn in Figure 7a and b. As can be seen in these figures, isothermal sections with and without extended solubilities of the binary compounds in the ternary systems are calculated and compared. The extended solubility of the binary compounds in the ternary system resulted in significant deviations between these isothermal sections.

Based on the experimental results obtained in this work and the literature, the liquidus projections of the Mg-Al-Ca and Mg-Al-Sr systems were drawn in Figure 8. Since experimental thermodynamic data for the Al-Ca-Sr and Mg-Ca-Sr systems could not be found in the literature, thermodynamic descriptions of these systems were established based on the optimized binary sub-systems and combined with the Mg-Al-Ca and Mg-Al-Sr systems in one database for the Mg-Al-Ca-Sr system.

CONCLUSIONS

A thermodynamic database of the Mg-Al-Ca and Mg-Al-Sr ternary systems was constructed by incorporating the experimental findings from this work and the literature. In the Mg-Al-Sr system, ternary solid solubilities of six binary compounds extended into the ternary system (i.e., (Mg_2Sr), ($Mg_{38}Sr_9$), ($Mg_{23}Sr_6$), ($Mg_{17}Sr_2$), (Al_4Sr), and (Al_2Sr)) and one ternary compound

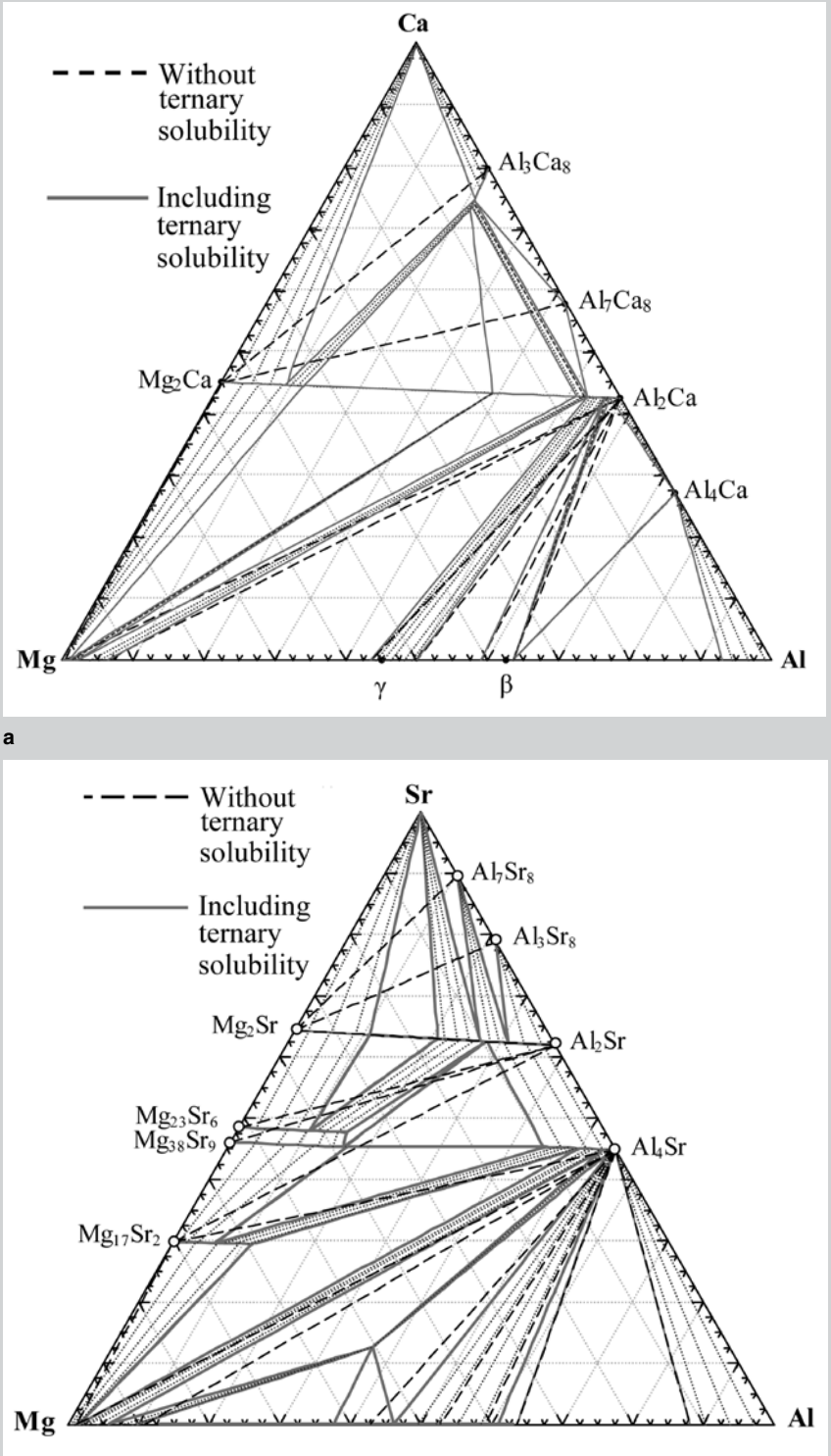

Figure 7. Calculated isothermal sections of (a) Mg-Al-Ca at 300°C and (b) Mg-Al-Sr at 400°C in wt.%.

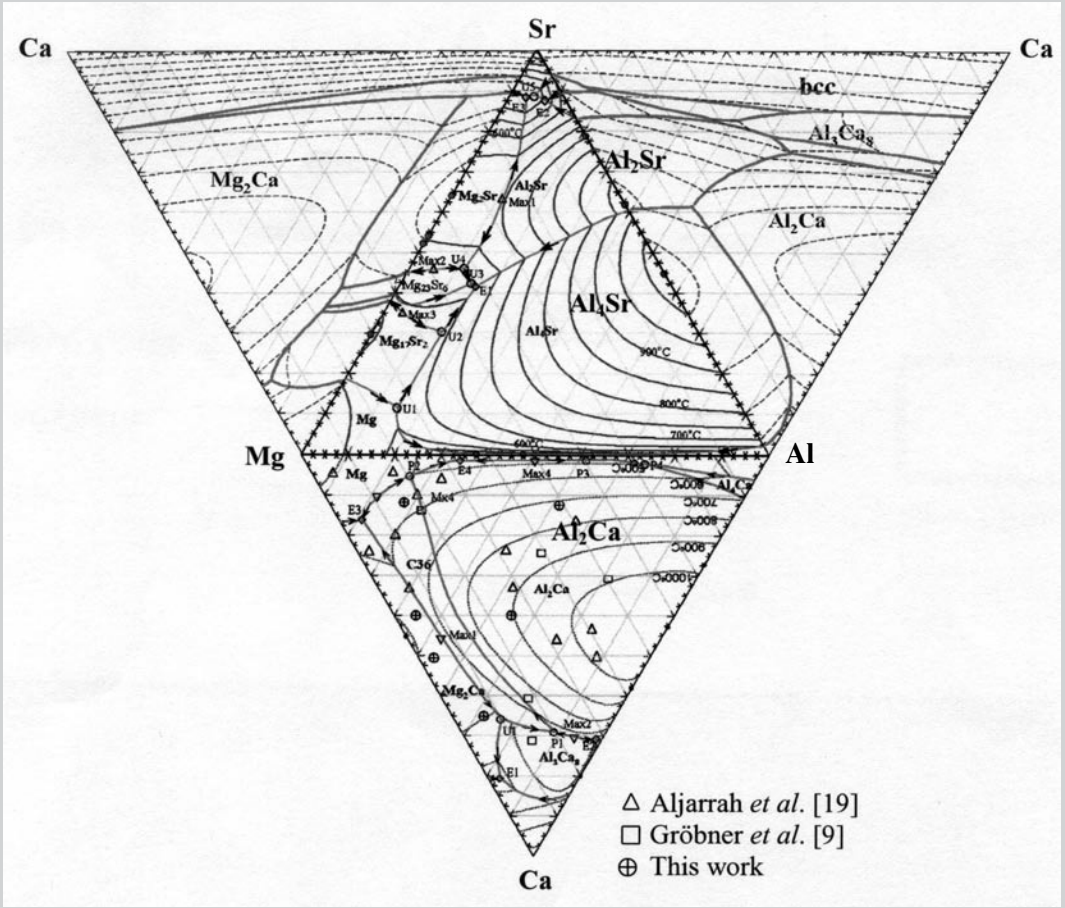

Figure 8. A liquidus projection of the constituent ternaries of a Mg-Al-Ca-Sr system.

with stoichiometry $Mg_{58}Al_{38}Sr_4$ have been found. Besides, three binary compounds (i.e., (Al_2Ca), (Al_3Ca_8), and (Mg_2Ca)) in the Mg-Al-Ca ternary system dissolve the third component in the ternary system. A new ternary compound with chemical composition $Mg_2Al_4Ca_3$ has been observed. Further, isothermal sections of the Mg-Al-Sr and Mg-Al-Ca systems have been constructed at 400°C and 300°C, respectively. Thermodynamic descriptions of the Mg-Ca-Sr and Al-Ca-Sr systems were established based on the optimized binary sub-systems and combined with those of Mg-Al-Ca and Mg-Al-Sr to establish a self-consistent thermodynamic database for the Mg-Al-Ca-Sr system.

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M. Aljarrah and M. Medraj are with the Department of Mechanical Engineering, Concordia University, Montréal, Canada; Jian Li and E. Essadiqi are with CANMET-MTL, Ottawa, Ontario, Canada. Dr. Medraj can be reached at (514) 848-2424 ext. 3146; fax: (514) 848-3175; e-mail mmedraj@encs.concordia.ca.