Mg-Al-(Sr, Ca) SYSTEMS - AN EXPERIMENTAL ANALYSIS

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ABSTRACT

The phase equilibria in the Mg-Al-Ca system was investigated experimentally by differential scanning calorimetry (DSC) and X-ray diffraction (XRD) techniques similarly the Mg-Al-Sr system has been studied using DSC and XRD as well as scanning electron microscopy/energy dispersive spectrometer (SEM/EDS) and quantitative electron probe micro-analysis (EPMA). A new ternary solid solution $Mg_xAl_{4-x}Sr$ was confirmed in Mg-Al-Sr system, which is a substitutional solid solution of Mg atoms in Al_4Sr. Maximum solubilities of 21.3 at.% Al in $Mg_{17}Sr_2$ and 11.4 at.% Al in Mg were observed. It was also noticed that $Mg_{38}Sr_9$ dissolved 12.5 at.% Al. In the Mg-Al-Ca ternary system, one of the invariant transformations predicted by thermodynamic modeling was verified experimentally and found to occur at 512°C with composition close to 10.8 at.% Ca, 79.5 at.% Mg and 9.7 at.% Al. A ternary solid solution $(Mg,Al)_2Ca$ was confirmed where large solid solubility of Al in Mg_2Ca was observed.

MET SOC
CIM TICM
Magnesium Technology in the Global Age
45th Annual Conference of Metallurgists of CIM
Montreal, Quebec, Canada
Edited by M.O. Pekguleryuz and L.W.F. Mackenzie

INTRODUCTION

Magnesium has the best strength to weight ratio of common structural metals and has exceptional die-casting characteristics [1]. This makes magnesium alloys one of the most promising light-weight materials for automotive application. To date, most Mg applications in the auto industry are in the form of die-cast parts. However, wrought magnesium applications, particularly sheet, and power train applications represent tremendous growth opportunities for magnesium. Unfortunately, magnesium alloys face a challenge for power train applications because of their limited creep resistance at higher temperatures [2].

Calcium and strontium can improve the properties of magnesium alloys. It is well known that the addition of Ca up to 0.3% increases ductility through grain refinement [3]. Strontium like calcium is a very effective grain refiner [4,5]. In recent years, Mg-Al-(Sr,Ca) systems have emerged as potential heat-resistant Mg-alloys. The development of Mg-Al-(Sr,Ca) alloys was aimed to replace RE additions to Mg alloys. One of the main challenges of these alloy systems is to optimize the combinations of properties such as creep resistance, tensile yield strength and castability [6,7].

However, to date, little effort has been made to construct the phase relationships of Mg-Al-Sr and Mg-Al-Ca systems. The experimental work on the phase equilibria of the Mg-Al-Sr system was primarily originated from Makhmudov and coworkers [8-10]. Besides, inconsistency was noticed between their works. Makhmudov *et al.* [9] reported a ternary compound with stoichiometry of $Al_{34}Mg_6Sr_{60}$ (Al_6MgSr_{10}), which is different from the earlier reported X compound. The solubility limits for the binary compounds determined by Makhmudov *et al.* [10] do not agree with the 400°C isothermal section given by Makhmudov *et al.* [9] in 1981. Baril *et al.* [11], recently, investigated four samples in the Mg-rich region of the Mg-Al-Sr system and tentatively designated a ternary phase as $Al_3Mg_{13}Sr$. In their work, the stoichiometry of this phase is not clearly identified and the chemical composition is not compatible with the ternary compound reported by Makhmudov *et al.* [9]. The ternary system was modeled without using ternary phases or ternary interaction parameters. In 2003, Koray *et al.* [12] modeled Mg-Al-Sr, and obtained results very similar to Chartrand and Pelton [13] except for the extent of the Mg_2Sr field.

In the Mg-Al-Ca system, Gröbner *et al.* [14] performed thermodynamic calculations of the phase diagram combined with experimental investigations carried out by DTA and XRD. In their investigation, a consistent thermodynamic modeling of the ternary system was done involving the substantial ternary solid solubilities. Koray *et al.* [12], however, calculated the ternary Mg-Al-Ca diagram by combining the data of the three binary systems, Al-Mg, Ca-Mg, and Al-Ca, assuming no ternary solubility between the binary compounds. Powell *et al.* [15] suggested the presence of a ternary solid solution in this system. Recent investigation [16] pointed out that Al_2Ca and $CaMg_2$ are the primary precipitates responsible for the improvement of creep resistance in this system. There is limited number of publications on this system which mainly focus on

the calculations of the Mg-Al-Ca phase diagram without sufficient experimental work. And these calculations exhibited a considerable discrepancy among themselves and substantial disagreement with the experimental data. In this work, the phase equilibria in Mg-Al-(Ca,Sr) systems were studied experimentally and compared with the thermodynamic calculations.

EXPERIMENTAL PROCEDURES

Thermal analytical investigation and phase identification were carried out in the Mg-rich region of the Mg-Al-Sr and Mg-Al-Ca systems. For Mg-Al-Sr system, 22 alloys and for Mg-Al-Ca system, 21 alloys were chosen by critical assessment of the experimental and thermodynamic datasets that are available in the literature. The samples were prepared and analyzed chemically at MTL-CANMET and the nominal sample compositions remained in very close proximity with the actual compositions. Mg-Al-Sr and Mg-Al-Ca ternary diagrams with the investigated compositions in weight percentage are given in Figures 1 and 2. In preparing the alloys, magnesium of 99.8 wt%, aluminum of 99.9 wt.%, strontium of 99 wt.%, and calcium of 99 wt.% were used. The charge was melted in a graphite crucible in an induction melting furnace under argon with 1%SF6 to protect the melt from oxidation. The actual chemical composition was measured quantitatively by ICP atomic emission spectrometry.

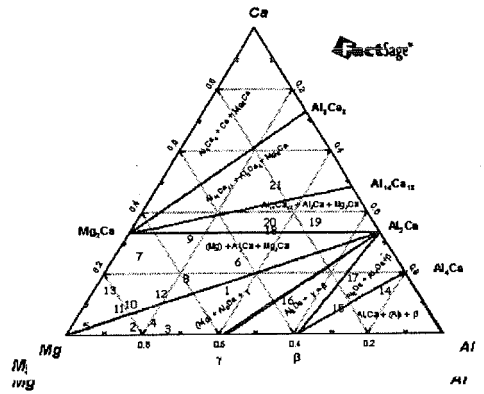


Figure 1 - Isothermal section at 25°C of the Mg-Al-Sr system with the investigated compositions in wt.%.

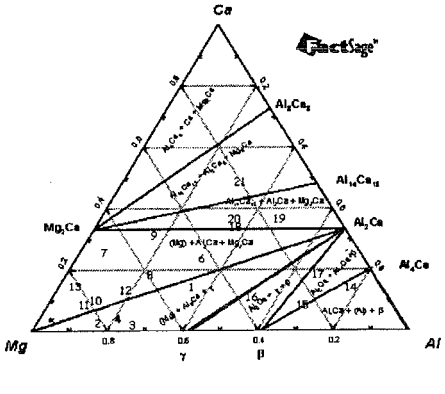


Figure 2 - Isothermal section at 25°C of the Mg-Al-Ca system with the investigated compositions in wt.%.

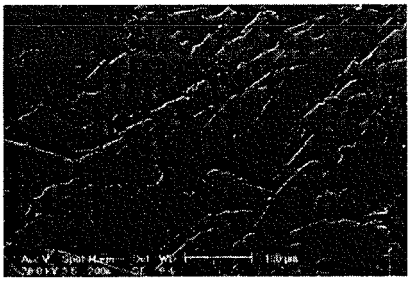
Thermal investigation of the systems was performed using a Setaram Setsys DSC-1200 instrument. The DSC measurements were carried with heating and cooling rates of 5°C/min from 25°C to 700°C. Slower heating rates were tried and were not found to reveal any other thermal arrests. The reproducibility of every measurement was confirmed by collecting the data during three heating and cooling cycles. More details on the interpretation of the DSC experiments were reported in our previous work [17]. Phase

identification was carried out by X-ray diffraction (XRD) with a Philips diffractometer (Cu$K\alpha$ radiation) equipped with a PW 1050/25 focusing gonimometer with steps 0.02° of 2θ diffraction angle and 1s exposure time. All the samples were investigated in the powder form in the as-cast condition at room temperature. PowderCell 2.3 [18] was used to calculate the diffraction patterns for different phases and to identify their peaks. SEM/EDS and EPMA were employed to examine the phase compositions for 14 alloys in Mg-Al-Sr system. EPMA analysis was carried out at three different locations for each phaseand the average was used for the present analysis. Chemical compositions of the phases were measured using a CAMECA SX51 EPMA. Microstructural observations were made using optical microscope (Olympus BX60M). The samples were etched using 1vol% nital solution (HNO_3 in ethanol) for a short period of time (~ 5s) to prevent dissolving of Mg grains.

RESULTS AND DISCUSSIONS

Experimental investigation was carried out using DSC, XRD, SEM/EDS and EPMA analysis on the Mg-Al-Sr system whereas DSC and XRD were used to study the Mg-Al-Ca system. Twenty two different ternary samples were studied in the Mg-Al-Sr system whereas twenty one samples were studied in the Mg-Al-Ca system. However, in this paper, only four compositions for the Mg-Al-Sr system and three compositions for the Mg-Al-Ca system will be discussed and the results of the other alloys are summarized in Tables 1 and 2.

In the present investigation, a new ternary solid solution has been found in twelve samples and represented by $Mg_xAl_{4-x}Sr$. This solid solution was detected by XRD as well as EPMA and found not to be a separate phase but it is due to the substitution of Al by Mg atoms in the Al_4Sr binary compound. Moreover, the solid solution of Mg in Al_4Sr must be considered as substitutional solid solution because of the following reasons: (i) Al/Sr ratio is not constant as proven by the EPMA analysis of all the samples containing this phase, and (ii) The numerical simulation of the X-ray spectra assuming substitutional solid solution agrees well with the experimental spectra but not if the solution is assumed to be a interstitial. For instance, Figure 3 shows SEM image, XRD pattern and EPMA analysis of sample 5 (22.53/43.75/33.72 Sr/Mg/Al wt.%). Al_4Sr has been identified as plate-like structure that dissolves 9.2 at.% Mg. Three phases; (Al_4Sr), (Mg) and γ have been identified by XRD and EPMA analyses. DSC spectra of sample 5 with heating and cooling runs are shown in Figure 3(IV). The onset, peak, melting temperatures and the melting enthalpy were registered. During heating of this sample, two thermal arrests, corresponding to the invariant reaction at 449°C and the univariant reaction at 490°C are observed. For this sample, the liquidus temperature is observed during cooling which is 686°C. In cooling runs, three thermal arrests have been observed. However, the invariant and univariant reactions occurred at quite close temperature. The experimental results were compared with the thermodynamic calculations to confirm the transformation temperature along with the associated reaction. For this purpose, the phase assemblage diagram shown in Figure 3(III) was calculated using FactSage [19] and the database developed by Chartrand and Pelton [13].



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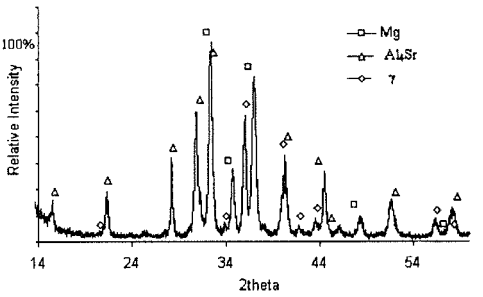

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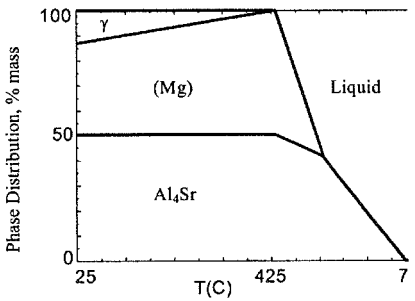

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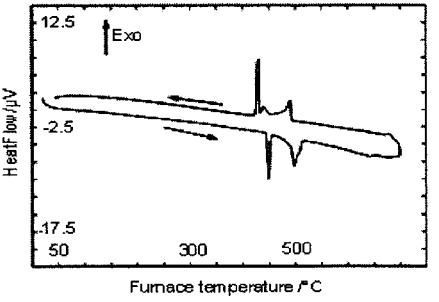


(IV)

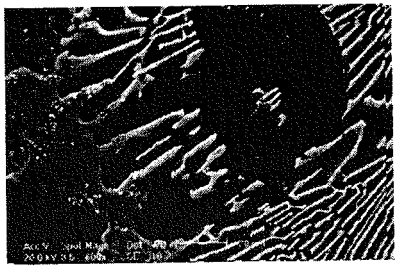
Location	at.% Mg	at.% Al	at.% Sr
A	9.2	69.8	21
B	61.34	38.61	0.05
C	89.39	10.6	0.01

(V)

Figure 3 - (I) SEM image; (II) XRD pattern; (III) Phase assemblage; (IV) DSC spectra and (V) EPMA analysis of sample 5.

Spot analysis of composition 2 (8.65/76.15/15.20 Sr/Mg/Al wt.%) was carried out at three different locations as shown in Figure 4. The microstructure is characterized as dendrites. EDS analysis indicated that the dark phase have both Mg and Al. Two types of secondary phases were observed in contrast to a single lamellar-type secondary phase in composition 1 reported in [17]. The eutectic morphology is more evident in this alloy as shown in Figure 4(II). Both types of secondary phases contain all the three elements; Mg, Al and Sr. Figure 4(III) shows that composition 2 has been identified positively with two phases (Mg), (Al_4Sr) and a very small amount of ($Mg_{17}Sr_2$). It can be seen by the EPMA analysis that the dark phase is Mg dissolving 7.4 at.% Al and the large precipitate is identified as $Mg_{17}Sr_2$ dissolving 19.3 at.% Al. Baril *et al.* [11] reported the existence of a bulky phase with chemical composition 78.10 ± 1.18 at.% Mg, 4.58 ± 0.37 at.% Sr and 17.32 ± 0.99 at.% Al in AJ52x alloy. This is not close to the chemical composition of the large precipitate observed in sample 2. However, the stoichiometry of this bulky phase

was not clearly identified and they tentatively designated the phase as $Al_3Mg_{13}Sr$. Table-1 summarizes the calculated transformation temperature and associated reactions with DSC signals and the phase contents identified by XRD at room temperature.



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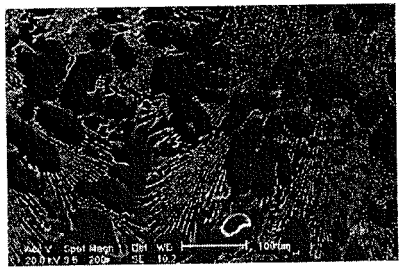

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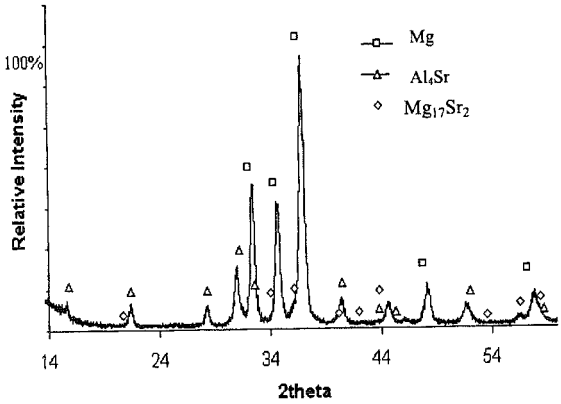


(III)

Location	at.% Mg	at.% Al	at.% Sr
A	92.6	7.4	0.00
B	71.40	19.32	9.28

(IV)

Figure 4 - SEM Image (I) 800x; (II) 200x; (III) XRD pattern and (IV) EPMA analysis of composition 2.

Figure 5 shows SEM micrographs, XRD patterns and EPMA analyses of compositions 10 (22.78/54.39/22.83 Sr/Mg/Al wt.%) and 11 (27.83/42.89/29.28 Sr/Mg/Al wt.%). (Mg), (Al_4Sr) and ($Mg_{17}Sr_2$) were identified in the XRD patterns for samples 10 and 11 as shown in Figure 5(III) and (IV). In the present EPMA analysis shown in Figure 5(V) and (VI), the light grey precipitate is identified as $Mg_{17}Sr_2$

dissolving 21.3 and 20.2 at.% Al, respectively. XRD pattern and EPMA analysis indicate that ($Mg_{17}Sr_2$) are present in both alloys which is not predicted by thermodynamic calculations of [12,13]. From the EPMA analyses, Al_4Sr dissolves 14.1 at.% Mg in sample 10 and 12.5 at.% of Mg in sample 11.

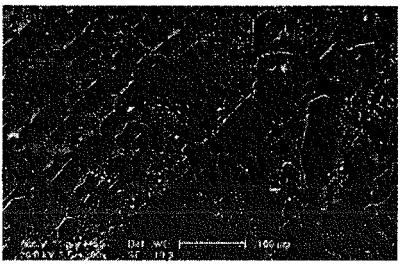
Table 1 - DSC and XRD measurements compared with thermodynamic modeling of the Mg-Al-Sr system.

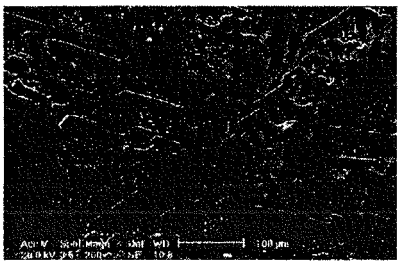
Sample	DSC Thermal Signals (°C)	Predicted temp. based on [13] (°C)	Identified phases (XRD)
10	618h 544c/560h 513c/523h	605 528 494	(Al_4Sr), γ and β
11	571h 546c/561h 513c/524h	664 525 496	(Al_4Sr), γ and β
12	643c 632h 599c/615h	642 520 499	(Al_4Sr), (Al_2Sr) and ($Mg_{17}Sr_2$)
13	- 605h/599c 579h/570c	696 605 579	(Al_4Sr), (Al_2Sr) and ($Mg_{17}Sr_2$)
14	643c 632h 599c/615h	783 716 499	(Al_4Sr), (Al_2Sr) and ($Mg_{17}Sr_2$)
15	662c 458c 459h/448c	662 - 454	(Al_4Sr), γ and β
16	675h/670c 453h/441c	670 446	(Al_4Sr), γ and β
17	- 492h 454h	970 - 431	(Al_4Sr), (Al) and β
18	- 472h/444c 452h	674 - 450	(Al_4Sr), (Al) and β
19	- 607h/597c 455h/442c -	633 601 525 371	(Al_4Sr), (Al) and β
20	- 505h/497c 454h/440c	868 498 484	(Al), (Al_4Sr) and β
21	584h/572c 564h/557c -	- 535 487	(Mg) and ($Mg_{17}Sr_2$)
22	613h/590c -	551 502	($Mg_{17}Sr_2$) and ($Mg_{38}Sr_9$)

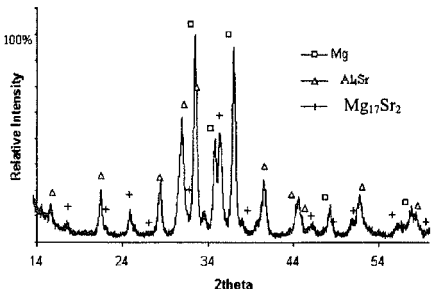
Sample	DSC Thermal Signals (°C)	Predicted temp. based on [13] (°C)	Identified phases (XRD)
1	609c 596c/605h 517c/535h -	591 - 535 222	(Mg) and (Al_4Sr)
2	563h 531c 516c/536h 510c/491h 427c/441h -	551 533 - - - 282	(Mg), (Al_4Sr) and ($Mg_{17}Sr_2$)
3	531c 528c 516c/523h 487c/510h 427c/442h -	540 530 - - - 398	(Mg), (Al_4Sr) and ($Mg_{17}Sr_2$)
4	514c/524h 472c 435c/494h 422c/442h	591 - 496 429	(Mg), (Al_4Sr) and γ
5	647c/652h 510c/513h 453c/489h 431c/445h -	648 535 525 - 318	(Mg), (Al_4Sr) and γ
6	686c 499c/490h 448c/449h 432c	703 532 - 433	(Al_4Sr) and γ
7	676c 510h 444c/466h	703 443 429	(Mg), (Al_4Sr) and γ
8	- 489c/507h 445c/457h -	802 - - 446	(Al_4Sr), γ and τ
9	- 506h 445c/457h	846 501 429	(Al_4Sr) and γ

Figure 5 shows SEM micrographs, XRD patterns and EPMA analyses of compositions 10 (22.78/54.39/22.83 Sr/Mg/Al wt.%) and 11 (27.83/42.89/29.28

Sr/Mg/Al wt.%). (Mg), (Al_4Sr) and ($Mg_{17}Sr_2$) were identified in the XRD patterns for samples 10 and 11 as shown in Figure 5(III) and (IV). In the present EPMA analysis shown in Figure 5(V) and (VI), the light grey precipitate is identified as $Mg_{17}Sr_2$ dissolving 21.3 and 20.2 at.% Al, respectively. XRD pattern and EPMA analysis indicate that ($Mg_{17}Sr_2$) are present in both alloys which is not predicted by thermodynamic calculations of [12,13]. From the EPMA analyses, Al_4Sr dissolves 14.1 at.% Mg in sample 10 and 12.5 at.% of Mg in sample 11.







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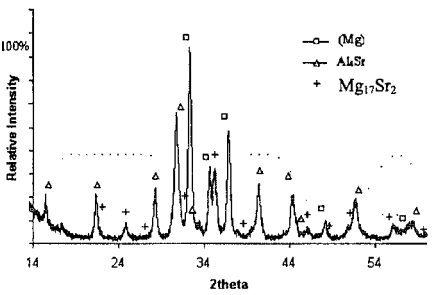


(IV)

Location	at.% Mg	at.% Al	at.% Sr
A	88.61	11.21	0.18
B	14.1	64.5	21.4
C	70	21.3	8.7

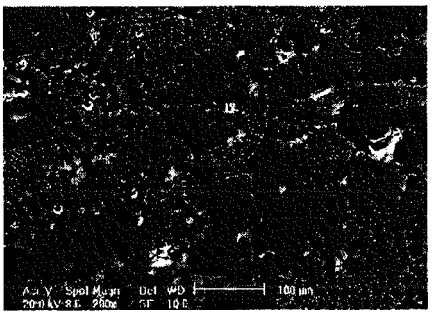
(V)

Location	at.% Mg	at.% Al	at.% Sr
A	12.5	66.6	20.9
B	70.6	20.2	9.2

(VI)

Figure 5 - (I) SEM Image of sample 10; (II) SEM Image of sample 11; (III) XRD pattern of sample 10; (IV) XRD pattern of sample 11; (V) EPMA analysis of sample 10 and (VI) EPMA analysis of sample 11.

Figure 6 shows SEM image XRD pattern and EPMA analysis of sample 22 (32.74/60.55/ 6.71Sr/Mg/Al wt.%). Regions (A) and (B) were identified by EPMA analysis as ($Mg_{17}Sr_2$) and ($Mg_{38}Sr_9$) which was supported by XRD results as shown in Figure 6(II). In this alloy, EPMA analysis shows that $Mg_{17}Sr_2$ dissolves 6.4 at.% Al and $Mg_{38}Sr_9$ dissolves 12.5 at.% Al. This suggests that the extent of the $Mg_{38}Sr_9$ phase field in the calculated ternary Mg-Al-Sr system by [13,14] is predicted narrower thus the system needs to be re-optimized.



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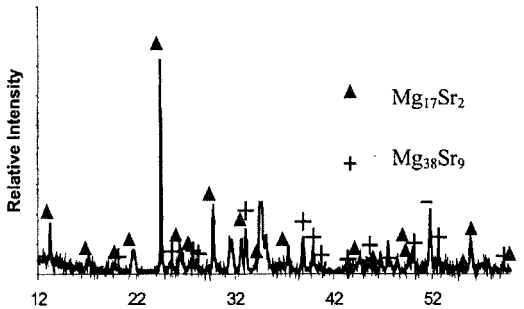


(II)

Location	at.% Mg	at.% Al	at.% Sr
A	83	6.4	10.6
B	70.8	12.5	16.7

(III)

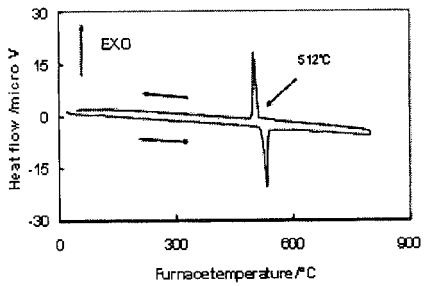
Figure 6-(I) SEM image; (II) XRD pattern and (III) EPMA analysis of sample 22.

Mg-Al-Ca SYSTEM

A eutectic point in the Mg-Mg_2Ca-Al_2Ca field has been investigated through compositions 10 to 13 shown in Figure 2. Sample 10 was prepared with the calculated composition of the eutectic point predicted by thermodynamic modeling of [20]. The phase assemblage diagram in Figure 7(II) shows the eutectic transformation, i.e. liquid phase transforms into three phases simultaneously: (L/(Mg) + Al_2Ca + Mg_2Ca) during cooling. DSC spectra of sample 10 in Figure 7(I) show a sharp, narrow and unique peak. This indicates that the infinite heat transfer occurs during an invariant transformation. The optical micrograph of sample 10 in Figure 7(III) shows typical lamellar eutectic feature and some plate-like precipitates. This indicates that the sample is quite close to the eutectic composition. The XRD pattern in Figure 7(IV) shows the coexistence of the (Mg), Al_2Ca and Mg_2Ca phases. This is in agreement with the phase assemblage diagram shown in Figure 7(II). Therefore, it is concluded that this eutectic transformation takes place at 512°C.

The XRD pattern of sample 7 (35.62/58.07/6.30 Ca/Mg/Al wt%.) shows strong peaks for Mg_2Ca as can be seen in Figure 8(I). The micrograph of this sample in Figure 8(II) shows several islands of Mg_2Ca, but no traces of Al_2Ca plates. This suggests that

Al_2Ca is embedded in the lamellar structure. In sample 7, the XRD peaks are better matched with the $(Mg,Al)_2Ca$ solid solution with 22 at.% Al than pure Mg_2Ca. Therefore, it is confirmed that a ternary solid solution $(Mg,Al)_2Ca$ exists in this sample. The identified and the calculated phases in samples 7 and 8 are in a agreement except for Mg_2Ca being a solid solution rather than a pure compound in these samples.



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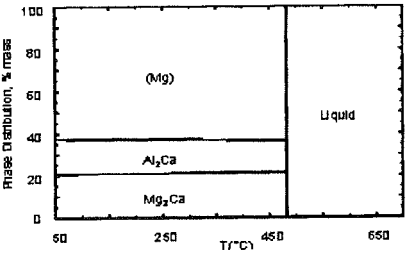

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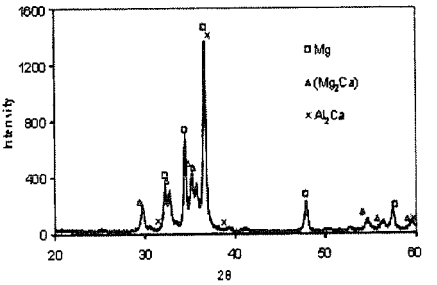
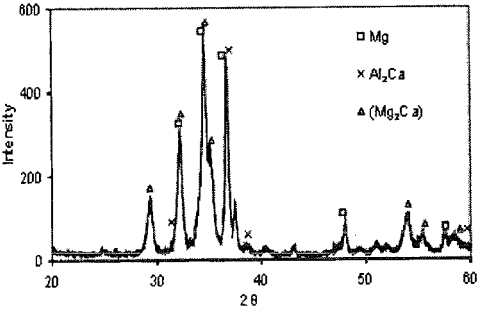


(IV)

Figure 7 - (I) DSC spectra; (II) Phase assemblage; (III) Optical micrograph and (IV) XRD pattern of sample 10 (16.44/73.61/9.95 Ca/Mg/Al wt%).



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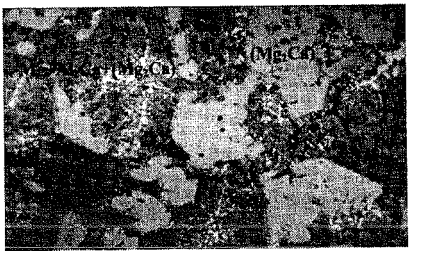

(II)

Figure 8-(I) XRD pattern and (II) Optical micrograph of sample 7.

Table 2 summarizes the experimental results for 21 samples in comparison with the thermodynamic calculations. The thermodynamic calculations are consistent with the experimental results in some samples, especially in the solidus temperature, whereas discrepancy was observed in several cases especially in the liquidus phase transformation temperature. This suggests that Mg-Al-Ca system should be re-optimized considering the new experimental findings.

Table 2 - DSC and XRD measurements compared to thermodynamic modeling of Mg-Al-Ca system.

Sample	DSC Thermal Signals (°C)	Predicted tmp. based on [20] (°C)	Identified phases (XRD)
1	472h/442c	430	(Mg), Al_2Ca and γ
	540h/523c	496	
	750c	728	
2	442h/431c	427	(Mg), γ and τ
	-	485	
	556h/542c	539	
3	441h/431c	428	(Mg), γ and τ
	-	450	
	488h/460c	476	
4	445c	428	(Mg), Al_2Ca and γ
	524c	494	
	584c	502	
5	-	195	(Mg) and Al_2Ca
	534h/524c	501	
	585h/605c	616	
6	516h/512c	481	(Mg), (Mg_2Ca) and Al_2Ca
	-	600	
	772c	748	
7	512h/506c	480	(Mg_2Ca), (Mg) and Al_2Ca
	-	569	
	640h/621c	650	
8	514h/519c	482	(Mg), (Mg_2Ca) and Al_2Ca
	-	631	
	-	666	
9	514h/519c	482	(Mg), Mg_2Ca and Al_2Ca
	-	631	
	-	666	
10	516h/512c	480	Mg_2Ca and Al_2Ca
11	515h/506c	482	(Mg), Mg_2Ca and Al_2Ca
	-	493	
	545h/532c	512	
12	516h/513c	481	(Mg),
	-	491	
	678h/626c	587	
13	520h/513c	480	(Mg), Mg_2Ca and Al_2Ca
	-	502	
	580/548c	572	
14	-	444	(Al), Al_4Ca and Al_2Ca
	-	492	
	542c/548h	540	
	620c/628h	618	
	-	838	
15	450c/456h	445	(Al), Al_2Ca and β
	505c/496h	491	
	633c/640h	714	
16	454c/464h	454	(Mg), (Al) and Al_2Ca
	774c/783h	752	
17	446c/452h	446	Al_2Ca, γ and β
	488c/482h	469	
	-	884	
18	506c/512h	550	Al_2Ca, Mg_2Ca and $Al_{14}Ca_{13}$
	-	631	
	-	810	
19	506c/520h	550	(Mg_2Ca), Al_2Ca and $Al_{14}Ca_{13}$
	-	620	
	-	909	
20	514h/506c	550	(Mg_2Ca), Al_2Ca and $Al_{14}Ca_{13}$
	-	627	
	-	785	
21	-	633	(Mg_2Ca), Al_3Ca_8 and $Al_{14}Ca_{13}$
	-	577	
	512h/507c	550	
	-	491	

SUMMARY

The equilibria in Mg-Al-Ca system have been investigated using DSC and XRD similarly the Mg-Al-Sr system has been studied using DSC, XRD SEM/EDS and EPMA analysis. In the present investigation, a new ternary solid solution $Mg_xAl_{4-x}Sr$ has been found. It was also observed that $Mg_{17}Sr_2$ and $Mg_{38}Sr_9$ dissolved 21.3, 12.5 at.% Al, respectively. In the Mg-Al-Ca system, a ternary eutectic point in the Mg-rich corner was verified. The eutectic temperature is determined as 512°C. Mg_2Ca was found to exist as a solid solution with a certain amount of Al dissolution. Many samples match with the thermodynamic findings especially in the Mg-Al-Ca system. Also, a few samples show a large discrepancy with the liquidus temperature compared with the calculated values. This suggests that the Mg-Al-(Ca,Sr) systems need to be re-optimized.

ACKNOWLEDGEMENTS

This research was carried out with the support of NSERC grants, Canada. The authors wish to express their appreciation for their supports.

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