# THE ROLE OF RARE EARTH ELEMENTS IN STRUCTURE AND PROPERTY CONTROL OF MAGNESIUM DIE CASTING ALLOYS

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### Abstract

The performance of magnesium die cast parts is governed by the microstructure and by the distribution of structural features which occur as a result of the chemical composition and processing history of the alloy. The elevated temperature properties, especially mechanical strength under creep conditions, are primarily determined by the grain structure, the elements in solid solution and the effectiveness of second phase particles in stabilizing the grain boundaries. The current emphases in alloy development focus on the utilization of elements with low solubility in the solid state, leading to the formation of stable precipitates during solidification. Such elements include the rare earths, as well as silicon, strontium and calcium. A detailed analysis of the various microstructural features and attributes is given for a new family of rare earth-containing alloys. The optimization of alloy composition is addressed in terms of blending advantageous microstructural characteristics with phase equilibria considerations.

#### Introduction

An initial metallurgical screening [1,2] and a second screening based on raw material cost and availability, indicate that basically only silicon (Si), calcium (Ca), strontium (Sr) or rare-earth (RE) (in addition to Al, Zn and Mn) can be considered as alloying elements to magnesium die casting alloys. Possibly tin (Sn) and antimony (Sb) can also be added to this list. With Mg-Al-Si (AS) alloys being limited to service up to 150°C maximum [2], RE additions become the next most economical addition for higher temperature use. Extensive work on the Mg-Al-RE (AE) alloy system over the past several years has identified alloys with unique combinations of properties [1-6].

The beneficial effect of rare earth elements (Ce) to Mg was discovered in the 1930's [7,8]. Sand and gravity casting alloys e.g. EZ33 and ZE41, grain-refined with small additions of Zr and subsequently heat treated, have been in use for some time [9,10]. AE alloys for die casting were extensively examined in the 1970's [11-13], culminating with AE42 in the early 1990's [14-16].

Although AE42 is the current benchmark with respect to elevated temperature properties and corrosion, the die casting alloys of the AE system have not received the attention they probably deserve from the property and metallurgical points of view. One reason for this may be inaccurate rumors that the castability of these alloys is inferior. To the contrary, recent work comparing the performance of various creep resistant alloys rank AE42 as the alloy having the best combination of properties at room temperature [17] and die castability similar to AZ91D for large automotive (power train) components [18]. High cost has also been an argument against AE alloys. This was true in the 1990's when the prices of mischmetal (MM) were very high. New entrants and expansions by existing producers of MM have resulted in additional capacity at the mining, processing and

product levels, leading to a significant and sustainable price reduction [19]. Hydro Magnesium has recently patented a new family of AE die casting alloys including AE44, AE35 and AE63.

# Principles for alloy development - The toolbox

Alloy development is the art and science of combining an appropriate chemical composition with the possibilities of the processing method to create microstructures that provide the material properties desired. Figure 1 shows schematically the microstructural details that influence the alloy properties.



**Figure 1.** Schematic microstructure showing constituents that influence properties [6].

## Solid solution hardening

The  $\alpha$ -magnesium phase is strengthened by solid solution hardening. Potent elements include Al, Zn and some RE elements [20]. Since creep occurs preferentially in the grain boundary regions where any eutectic components would be located [21], it is important that the eutectic temperature is not too low. For example the Mg-Zn alloy system has a eutectic temperature of about 340°C, and ternary elements are required to increase the eutectic temperature. Alloying with Al provides a eutectic temperature around 430°C. Additions of elements like RE, Ca and Sr may further increase the eutectic temperature.

# Grain boundary hardening

Die casting is characterized by rapid cooling. Depending on the wall thickness, the cooling rate will typically be in the range  $10-1000^{\circ}$ K/s; the resulting grain size is very fine, typically between 5 and 100  $\mu$ m. This leads to a significant hardening effect predictable from the Hall-Petch relationship [22]. The fine grain size is harmful to creep properties, but it is known that ultrafine-grained magnesium alloys deform superplastically at elevated temperatures [23].



Figure 2. SEM-BEC of typical die cast microstructure of AE42.



AE42									RE/	Likely
	Mg	AI	Ce	La	Nd	Pr	Mn	ΣRE	(RE+AI)	phase
1	98.65	1.18	0.00	0.00	0.00	0.00	0.10	0.00	0.00	
2	87.28	8.80	1.81	0.96	0.57	0.33	0.08	3.67	0.29	Al <sub>11</sub> RE <sub>3</sub>
3	91.33	5.22	1.66	0.79	0.44	0.23	0.00	3.12	0.37	Al <sub>2</sub> RE
4	80.33	14.42	1.68	1.14	0.28	0.24	1.82	3.34	0.19	Al <sub>11</sub> RE <sub>3</sub>
6	86.56	10.43	1.41	1.04	0.09	0.04	0.35	2.58	0.20	$AI_{11}RE_3$
7	84.31	12.38	1.66	1.07	0.22	0.18	0.19	3.13	0.20	$AI_{11}RE_3$

Figure 3. SEM-EDS analyses of dispersoids in AE42.

## **Dispersion** hardening

Alloying elements that form a dense distribution of intermetallic particles during solidification plays a key role in providing creep resistance to die casting alloys. Such particle distributions may have a high dispersion level, f/r, where f is the volume fraction of particles and r is the mean particle radius. These particle dispersions become increasingly efficient in reducing the creep rate arising from thermal activation by forming three-dimensional networks along the grain boundaries [24]. An additional positive effect is observed when the dispersoid-forming element combines with the solid solution hardening element to reduce the amount of



Figure 4. SEM-BEC of typical die cast microstructure of AE44.



AE44									RE/	Likely
	Mg	AI	Ce	La	Nd	Pr	Mn	ΣRE	(RE+AI)	phase
1	98.26	1.40	0.03	0.00	0.03	0.07	0.12	0.13	0.08	
2	88.56	7.69	1.68	1.10	0.47	0.33	0.09	3.58	0.32	Al <sub>2</sub> RE
3	86.11	10.33	1.71	1.19	0.30	0.24	0.02	3.44	0.25	Al₃RE
4	85.88	10.79	1.38	1.25	0.25	0.20	0.19	3.08	0.22	$AI_{11}RE_3$
5	90.23	6.66	1.39	0.82	0.39	0.22	0.15	2.82	0.30	Al <sub>2</sub> RE
6	88.30	8.18	1.60	0.87	0.52	0.25	0.10	3.24	0.28	Al₃RE
7	90.67	6.67	1.18	0.89	0.26	0.25	0.00	2.58	0.28	Al₃RE

Figure 5. SEM-EDS analyses of dispersoids in AE44.

low melting eutectic along the grain boundaries. Rare earth elements, Ca and Sr in Mg-Al alloys are examples of this.

# Age hardening

Age hardening in the usual sense of performing a full T6 heat treatment is generally not considered to be compatible with the desired properties and high volume production rates of die cast parts. Among the reasons for this are a) during solution treatment, the loss in strength due to grain coarsening can be larger than the increase caused by subsequent age hardening, and b) the formation of blisters due to expansion of gas porosity upon heating can be a problem unless special precautions are taken.

However, age hardening is commonly used for coarse-grained, sand cast parts; an example is provided by WE43 alloy, where age hardening precipitates are formed in extremely high densities during a final age hardening [25]. These particle dispersions provide both strength and creep resistance, due to the high thermal stability of the particles formed. This explains the paradox that while magnesium die casting alloys commonly show lower creep resistance than aluminium alloys, some magnesium sand casting alloys have superior creep strength to the aluminium casting alloys [26].

## Experimental

Ingots of AE42 and AE44 were melted in a 15 kW resistance heated furnace. The special AE alloys, containing Ce, Nd and combinations, rather than MM, were prepared from a basis of molten AM20 to which pure Al, Ce and/or Nd were added. The various AE alloys were die cast on a 420 ton Bühler SC42D Evolution machine, with a five cavity plate/bar die containing two 3 mm test plates and three test bars (tensile bar, creep bar and fatigue bar [2].

Specimens for microstructure studies were cut and prepared from one arbitrary test plate in the as cast condition. The specimens were examined on a JSM5600, Pioneer, Scanning Electron Microscope (SEM) system. Energy Dispersive Spectroscopy (EDS analysis) was performed at 8000X magnification with an accelerating voltage of 15 kEV.

Specimens from a series of alloys were tensile creep tested for minimum 150 h in accordance with ASTM E139, at e.g.  $175^{\circ}C/40$  MPa and  $175^{\circ}C/75$  MPa. Tensile creep testing was also performed on samples that were aged at  $175^{\circ}C$  for 500 h. A minimum of two specimens was tested at each condition.

#### **Results and discussion**

## Dispersoid phases in AE alloys

The microstructure of die cast Mg-Al based alloys generally consists of a grain interior of  $\alpha$ - Mg(Al) solid solution, and a grain boundary zone (grain "mantle") which is eutectic Mg-Al (Alenriched  $\alpha$  Mg with  $\beta$  - Mg<sub>17</sub>Al<sub>12</sub> embedded).

The rare earth used in the AE alloys is usually mischmetal (MM), a mixture of Ce, La, Nd and Pr, where Ce (approx. 50%) and La (about 30%) are the main constituents. AE42 contains 2-3 wt% Ce-rich MM (RE). Intermetallic phases (dispersoids) are distributed in the grain boundary zone as an almost continuous network, Fig. 2. SEM-EDS analysis indicates that the intermetallic phase formed as die cast mainly is Al<sub>11</sub>RE<sub>3</sub>, Fig. 3. (RE/(Al+RE) close to 0.21). This is also reported in the literature [27, 28]. The formation of  $Al_{11}RE_3$  reduces the effective amount of Al, which otherwise would lead to Al-enrichment and subsequent formation of Mg<sub>17</sub>Al<sub>12</sub> in the grain boundary zone. The effect on creep is primarily to inhibit grain boundary sliding. The grain interior is not significantly influenced by the addition of RE [1]. This is also indicated in Fig. 3, pos. 1. In alloys with a RE/Al ratio corresponding to AE42, the Al<sub>11</sub>RE<sub>3</sub> phase has a lamellar structure with a relatively large extension from the grain boundaries. With increasing Al content the Al<sub>11</sub>RE<sub>3</sub> phases are coarsening, and more concentrated close to the grain boundaries. The positive effect on creep is also reduced since the intermetallic particles then increasingly are embedded in eutectic Mg-Al.

At higher RE/Al ratios the ductility is reduced due to formation of a continuous skeleton of an intermetallic phase decorating the grain boundaries [1,2]. On the other hand, the creep strength will improve.

AE44 contains around 4 wt% RE. As seen from Fig. 4, the continuous network of intermetallic phases on the grain boundaries is even more predominant than in AE42. SEM-EDS analysis indicates that in AE44 the grain boundary phase formed in the die cast specimens is mainly Al<sub>2</sub>RE, rather than Al<sub>11</sub>RE<sub>3</sub> (and Al<sub>3</sub>RE). (in Al<sub>2</sub>RE the RE/(Al+RE) ratio is close to 0.33). This means that with an increasing RE/Al ratio, Al<sub>2</sub>RE gradually becomes the more dominant phase relative to Al<sub>11</sub>RE<sub>3</sub>. It seems that at a RE/Al ratio (wt% basis) of slightly less than unity, the Al<sub>2</sub>RE phase is the more dominant. The phase diagrams of Al-Ce, Al-La, Al-Nd and Al-Pr all indicate stable phases of Al<sub>3</sub>RE and Al<sub>2</sub>RE [29]. The transition from Al<sub>11</sub>RE<sub>3</sub> to Al<sub>2</sub>RE (possibly through Al<sub>3</sub>RE) will be investigated in more detail in a future paper [30].

# Phases in ACe and ACeNd alloys

In order to further study the dispersoid phases in grater detail, "synthetic" AE alloys were prepared, die cast and examined. In these alloys MM was replaced by pure Ce and Nd, and combinations. From Fig. 6 it is seen that the die cast microstructure of ACe44 is very similar to that of AE44 (Fig. 4). Looking at the phases, Fig. 7, SEM-EDS analysis indicates that in the alloy with 4% Ce mainly  $Al_{11}Ce_3$  and/or  $Al_3Ce$  is found, and not  $Al_2Ce$  as might be expected from AE44.

In a second alloy Ce and Nd were added in equal amounts to produce ACeNd422. The microstructure of this alloy is also very similar to that of AE44 (Fig. 4) and ACe44 (Fig.6). The SEM-EDS analysis indicates that also in the ACeNd422 alloy mainly  $Al_{11}$ (Ce,Nd)<sub>3</sub> is found, and not  $Al_2$ (Ce,Nd) (or  $Al_3$ (Ce,Nd)) as might be expected from the study of AE44 (Fig.3).

In a third alloy only Nd was added to produce ANd43. Also in this alloy there are strong indications that  $Al_{11}Nd_3$  and possibly  $Al_3Nd$  are present. (Nd/(Al+Nd) ratios corresponding to 0.21 and 0.25, respectively). One may speculate if lanthanum (La) in the AE alloys is instrumental in the promotion of  $Al_2RE$ , or if it is the mixture of the different RE elements (Ce, La, Nd and Pr) in a certain ratio that are important. This is presently subjected to further investigations [30].

# Stability of dispersoids in AE alloys

It has been reported that sharply decreasing creep strength of AE42 above 150°C is attributed to a partial decomposition of Al<sub>11</sub>RE<sub>3</sub> into Al<sub>2</sub>RE and Al, subsequently forming Mg<sub>17</sub>Al<sub>12</sub> [26]. As a practical consequence, prolonged exposure of AE42 to elevated temperatures could have a negative influence on the creep properties. This could be highly relevant for e.g. engine blocks or other high temperature exposed automotive components in service. With a higher RE/Al ratio than that of AE42, possibly producing Al<sub>2</sub>RE during solidification, this unwanted phase transformation may no longer occur.

To study this experimentally, die cast specimens of AE44 and AE42 were subjected to a special heat treatment (aging) at  $175^{\circ}$ C for 500 hours. Tensile creep tests of as cast and aged specimens were undertaken at 40 and 75 MPa at  $175^{\circ}$ C. The results are shown in Figs. 10 and 11, respectively. From Fig. 10 it is seen that at  $175^{\circ}$ C/40 MPa the aging of AE42 has a negative effect on the creep properties. On the other hand, AE44 seems to remain unaffected by the aging process, indicating that no phase



Figure 6. SEM-BEC of die cast microstructure in ACe44.



Ace	**					LIKEIY
	Mg	AI	Ce	Mn	Ce/(Ce+Al	) phase
1	98.25	1.45	0.12	0.10	0.08	
2	89.36	8.14	2.26	0.18	0.22	Al <sub>11</sub> Ce <sub>3</sub>
3	85.88	10.10	3.75	0.02	0.27	Al <sub>3</sub> Ce
4	91.13	6.84	1.81	0.15	0.21	Al <sub>11</sub> Ce <sub>3</sub>
5	88.56	8.50	2.71	0.11	0.24	Al <sub>3</sub> Ce
6	92.37	5.82	1.62	0.10	0.22	Al <sub>11</sub> Ce <sub>3</sub>
7	96.65	2.40	0.81	0.15	0.25	Al <sub>3</sub> Ce

Figure 7. SEM-EDS analyses of dispersoids in ACe44.

transformation has taken place. It is noticed that at this condition AE42 (as cast) exhibits creep properties at the level of AE44.

From Fig 11 where the load has been increased to 75 MPa, it is seen that AE44 in aged and as cast conditions remains the same. Furthermore, it is noticed that at this condition AE44 exhibits significantly better creep strain and creep rates than AE42. Tensile creep tests at  $150^{\circ}$ C/50 MPa indicate that AE42 in the as cast and aged condition behaves similarly.



Figure 8. SEM-BEC of die cast microstructure in ACeNd422.



ACeN	ld422					(Ce+Nd)/	Likely
	Mg	AI	Ce	Nd	Mn	(Ce+Nd+AI)	phase
1	98.55	1.27	0.00	0.00	0.13	0.00	
2	97.80	1.79	0.15	0.16	0.06	0.15	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>
3	88.67	8.99	1.14	0.82	0.28	0.18	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>
4	84.83	12.36	1.27	1.09	0.36	0.16	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>
5	91.44	6.75	0.86	0.76	0.09	0.19	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>
6	93.82	4.67	0.75	0.56	0.11	0.22	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>
7	91.39	6.55	0.98	0.80	0.21	0.21	Al <sub>11</sub> (Ce,Nd) <sub>3</sub>

Figure 9. SEM-EDS analyses of dispersoids in ACeNd422.

Notice that the frequency of data recording during the start-up of the creep tests has not been sufficient to produce reliable data during the initial stages of the phase I creep. As a consequence, the value of the creep strain may be affected by a constant error throughout the experiments. Alternatively, the creep rates should be used as a measure of creep performance. Creep rates obtained at 100 h are listed in Table 1. These are, however, not the same as the minimum creep rate during phase II. During the lowest stress level (40 MPa) it is not likely that AE44 has fully reached the



**Figure 10.** Creep strain as a function of time at  $175^{\circ}$ C/40 MPa; as cast and aged (HT) specimens of AE42 and AE44. The aging consisted of exposure to  $175^{\circ}$ C for 500 h.



Figure 11. Creep strain as a function of time at  $175^{\circ}$ C/75 MPa; as cast and aged (HT) specimens of AE42 and AE44. The aging consisted of exposure to  $175^{\circ}$ C for 500 h.

175°C							
	40 MPa	75 MPa					
AE42	1.1E-06	8.4E-06					
AE42 HT	5.8E-06	n/a					
AE44	2.5E-06	1.8E-06					
AE44 HT	2.3E-06	1.5E-06					

**Table 1** Creep rates at  $100 \text{ h} (\text{h}^{-1})$ .

steady state phase II creep. This may explain why the creep rates of AE44 at 100 h apparently are higher than that of as cast AE42.

## Conclusions

This initial study indicates that at a critical RE/Al ratio the dispersoid phase stabilizing the grain boundaries of AE alloys changes from  $Al_{11}RE_3$  (and  $Al_3RE$ ) to  $Al_2RE$ . In the alloy AE44, recently patented by Hydro Magnesium, the  $Al_2RE$  phase is dominant. However in the "synthetic" alloys ACe44, ACeNd422 both containing 4wt% rare earth elements the dominating phases still seems to be  $Al_{11}Ce_3$  (possibly with some  $Al_3Ce$ ) and  $Al_{11}(Ce,Nd)_3$ , respectively. The reason for this is not yet understood.

Furthermore, it is verified experimentally at  $175^{\circ}$ C/40 MPa that the creep properties of AE42 are significantly better in the as cast condition than in the aged condition. Most probably this is attributed to a phase change in AE42 from the metastable Al<sub>11</sub>RE<sub>3</sub> to Al<sub>2</sub>RE with subsequent formation of the brittle Mg<sub>17</sub>Al<sub>12</sub>. The creep properties of AE44 at 175°C, 40 and 75 MPa, are not affected by aging.

#### Acknowledgements

The following persons are acknowledged for contributing to this work: Darryl Albright, Kari Straume Andersen, Sten Bjørneboe, Per R. Gjertsen, Haavard Gjestland, Jan Ivar Klovland, Ketil Pettersen, Lisabeth Riopelle, Stian Sannes, Vidar Sjøberg, Jan Ivar Skar, Eva Thorjussen, Jørild M. Svalestuen, Chenguo Tian, Arve Wilhelmsen, Jo Willekens.

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