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Properties of MgCaZr Alloys

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The use of most commercial magnesium alloys is limited to working at normal temperatures. The excellent ratio between the mechanical properties and the density of magnesium alloys necessarily leads to the development of new types of alloys that would be usable even at elevated temperatures. This would significantly increase the applicability of these alloys where steels or aluminum alloys are still used, especially in the transport industry. The problem with today's high temperature resistant magnesium alloys is the need to use expensive rare earth alloys. Significantly cheaper alloys of magnesium with zirconium and calcium are studied in this work. The microstructure, mechanical properties under pressure at the temperatures of 20, 150, 200 and 250 ° C were studied for several alloys with different contents of Zr and Ca. Furthermore, the stability of alloys during their long-term temperature exposure was studied. A very positive effect of the studied additives on the properties of alloys was found, which gives these alloys a very promising perspective in the future.

Keywords: Magnesium Alloys, Thermal stability, Mechanical Properties, Microstructure, Alloying

1 Introduction

Metallic magnesium was firstly prepared in small quantities by H. Davy in 1808. The industrial production of magnesium began in France in 1857. The production of magnesium was initially difficult and expensive. Therefore, the magnesium produced was first used for special applications (pyrotechnics, chemical reagent in organic synthesis, etc.). As a structural material, magnesium alloys were first used to build the skeleton of the Zeppelin airships [1]. Other applications of magnesium alloys in the transport industry followed, such as rims and car bodies. The rapid development of the production and processing of magnesium alloys did not occur until after the end of World War II in Europe and USA. Current global magnesium production is estimated at more than 1,000,000 tons per year. Today, about 90% of this production is in China [2]. The end of the World War II significantly started the further development of magnesium alloys. Due to the high reactivity of magnesium, the development of magnesium alloys was initially focused on alloys that will not be exposed to elevated temperatures and aggressive chemical environments during their use. Only in recent decades has the development focused on new types of magnesium alloys with increased resistance to elevated temperatures. Stabilization of the microstructure of magnesium alloys at elevated temperatures was made possible by relatively expensive alloys based on rare earth metals, thorium, zirconium, and others [1-5]. Unfortunately, the price of these alloys has a significantly increasing tendency in the current unfavorable global situation [5]. Long-term use of magnesium alloys at elevated temperatures is conditioned by their high creep resistance and oxidation resistance [6]. Current scientific research focuses on the development of magnesium alloys, where a substantial part of precious alloys is replaced by more affordable elements [7-10]. Calcium and strontium [8-13] appear to be very promising in this context. In this work, calcium in combination with zirconium was used for preparation of studied alloys.

1.1 Magnesium alloys with enhanced resistance at elevated temperatures

The development of alloys with increased resistance at elevated temperatures is based on the overall stabilization of their microstructure. The microstructure of magnesium alloys is formed by primary dendrites of a magnesium solid solution and possible intermediate phases formed in the interdendritic regions. To achieve the highest possible mechanical properties of the alloy, it is necessary to ensure the smallest possible size of dendrites of the magnesium solid solution. A solid solution of magnesium should show the highest possible strength, which can be achieved with suitable alloys. For some types of magnesium alloys, precipitation hardening can also be used for solid solution reinforcement. Furthermore, it is important to stabilize the interdendritic regions with possibly formed intermediate phases, so that creep mechanisms are reduced to a minimum as much as possible at elevated temperatures [12-18]. The selection of suitable alloys to produce these is necessarily based on several aspects. The prerequisites for achieving a stable microstructure of the alloy at elevated temperatures are the lowest possible diffusion coefficients of the alloying elements in magnesium, the availability and favorable price of the alloying elements and, finally, the possibility to use established foundry technologies to produce these magnesium alloys. Tab.1 shows the diffusion parameters of selected elements in magnesium. The diffusion coefficient D for a given temperature can be calculated according to the Arrhenius equation, see Eq.1 [19].

$$D = D_0 \,.\, exp(Q/R.T) \ [m^2.s^{-1}] \qquad (1)$$

Where:

D_0…Temperature independent pre-exponential constant,

Q…Activation energy of diffusion,

R…Universal gas constant,

T…Thermodynamic temperature.

Tab. 1 *Diffusion parameters of selected elements in Mg [19]*

Element	D_0, m^2s^{-1}	Q, $Jmol^{-1}$	Element	D_0, m^2s^{-1}	Q, $Jmol^{-1}$
Ag	2.0×10^{-4}	131 494	**Li**	5.5×10^{-5}	125 983
Al	1.2×10^{-4}	141 814	**Mg**	2.9×10^{-5}	125748
Be	5.3×10^{-6}	156 486	**Mn**	1.2×10^{-4}	153 217
Ca	6.0×10^{-6}	103 780	**Nd**	2.1×10^{-6}	116 354
Cd	6.4×10^{-5}	135 369	**Ni**	9.8×10^{-10}	100 275
Ce	2.7×10^{-6}	108 116	**Pu**	1.5×10^{-4}	139 933
Cu	3.5×10^{-7}	99 693.2	**Sb**	3.3×10^{-4}	139 068
Fe	3.8×10^{-10}	87 820.8	**Sn**	1.1×10^{-4}	140 925
Ga	1.2×10^{-4}	134 188	**U**	1.2×10^{-9}	109 587
Gd	9.9×10^{-6}	127 804	**Y**	8.1×10^{-6}	126 726
In	4.0×10^{-5}	132 459	**Zn**	8.7×10^{-5}	125 073
La	3.1×10^{-6}	104 307			

Tab. 2 *Basic types of magnesium alloys for work at elevated temperatures [20-34]*

	Composition	Example	Maximum long-term operating temperature, °C
AZ alloys	Mg-Al-Zn	AZ91	150
AM alloys	Mg-Al-Mn	AM60	150
ZC alloys	Mg-Zn-Cu-Mn	ZC62, ZC63	150
AE alloys	Mg-Al-RE	AE42, AE44	150
ACM alloys	Mg-Al-Ca-RE	ACE522	160
ZACE alloys	Mg-Zn-Al-Ca-RE	ZACE05613, ZACE05411	170
AJ alloys	Mg-Al-Sr	AJ52	175
AS alloys	Mg-Al-Si	AS21, AS33, AS41	175
AJX alloys	Mg-Al-Sr-Ca	AJX500, AJX531, AJX621	175
experimental*	Mg-Ba2-Ca2		180
MRI alloys*	Mg-Al8- Ca1-Sr1 Mg-Al7-Ca2-Sn1-Sr	MRI 153, MRI 230	190
AX alloys	Mg-Al-Ca	AX51, AX52, AX33	190
JM alloys	Mg-Sr-Mn	JM51, JM52	200
QE alloys	Mg-Ag-RE	QE22	200
WE alloys	Mg-Y-RE-Zr	WE43, WE54	250
GW alloys	Mg-Gd-Y-Zr	GW103	250
EZ alloys	Mg-RE-Zn-Zr	E733	260
HZ alloys	Mg-Th-Zn	HZ32	300
H alloys	Mg-Th	H3	320
HK alloys	Mg-Th-Zr	HK31	350

* the designation of the alloys is outside the scope of ASTM

As can be seen from Tab. 1, mentioned elements are characterized by significantly different values of diffusion coefficients in magnesium. Fe, Ni and U are characterized by the lowest values of diffusion coefficients [19]. Compared to magnesium, these elements have significantly higher melting temperatures and practically negligible solubility in solid magnesium. This is the main reason why the mentioned elements cannot be used as alloying elements to produce magnesium alloys resistant to elevated temperatures using conventional foundry techniques. The very poor castability of Fe, Ni and U with Mg does not allow this. In Tab. 1, there are several elements that have several orders of magnitude higher diffusion coefficients compared to Fe, Ni and U. These are Ca, Ce, Gd, La, Nd and Y. They are mainly rare earth metals, which are key elements in magnesium alloys, which enable their use at elevated temperatures, although the castability of Ce, Gd, La, Nd and Y with magnesium is not easy. Currently however the mineral resources of these raw materials are decreasing, and their price is increasing significantly. Therefore, there is currently an effort to replace rare earth metals with cheaper alternatives. Therefore, in current magnesium alloys for work at elevated temperatures, elements such as Ca, Sr and Ba are increasingly used, which partially or completely replace hard to find rare earth metals [5,11,16-18, 20-31].

Currently, mainly the alloys listed in Tab. 2 are used to produce magnesium alloy castings with increased re-sistance at elevated temperatures.

The alloys with the highest resistance at elevated temperatures do not contain aluminum. The formation of the intermediate phase $Mg_{17}Al_{12}$, which magnesium together with aluminum forms, is the cause of the thermal instability of common commercial magnesium alloys at elevated temperatures [3,31,34-36]. The most effective alloying element in magnesium alloys to achieve their increased resistance at elevated temperatures is thorium. It forms with magnesium stable intermediate phases that reinforce interdendritic regions. The disadvantage of thorium is its radioactivity, preventing the use of these alloys on a large scale. [20,33]. Traditional magnesium alloys for use at elevated temperatures are based on additions of rare earth metals. The price of these elements is constantly increasing. For that reason, modern magnesium alloys have the content of precious rare earth metals largely replaced by Ca or Sr. [7-9]. Both Ca and Sr form with magnesium the stable binary phase Mg_2Ca, resp. Mg_2Sr. These are present in the form of eutectics at the interdendritic regions and thus stabilize the interdendritic regions. The Mg-Ca phase diagram is in Fig. 1.

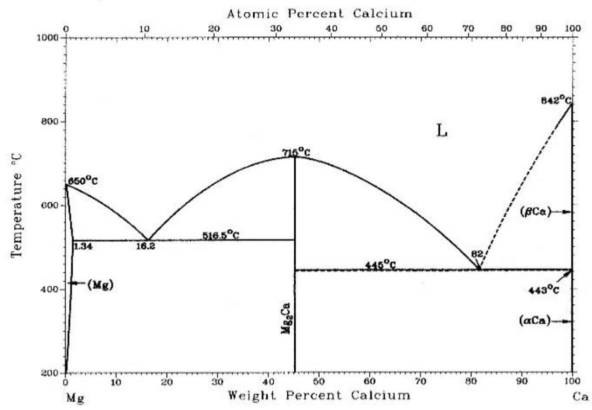

Fig. 1 *Mg-Ca phase diagram [1]*

Another element that is a typical alloy of magnesium alloys with increased resistance at elevated temperatures is zirconium. This element could significantly refine the microstructure of the primary dendritic phase in several alloys [20-31]. Its use is typical for aluminum alloys. However, this element is also very effective for refining of magnesium alloys. The part of the Mg-Zr phase diagram near to magnesium is shown in Fig. 2.

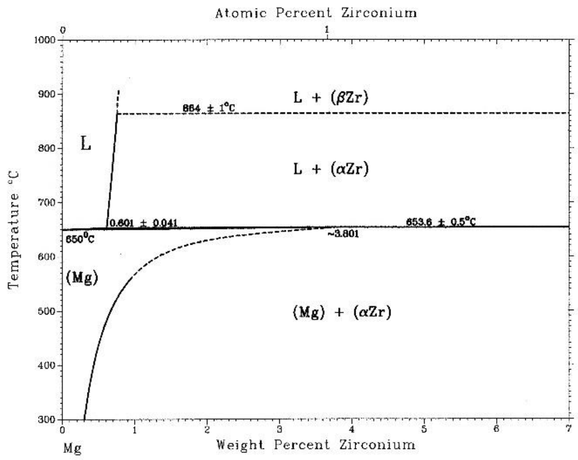

Fig. 2 *Mg-rich part of Mg-Zr phase diagram [1]*

In the experimental part of this work, magnesium alloys containing no rare earth metals alloyed with different amounts of Zr and Ca were studied. The aim of Zr addition was to refine the microstructure of the primary magnesium solid solution. The aim of Ca addition is stabilization of interdendritic regions by formation of calcium-containing phases.

2 Materials and methods

In this work, the microstructure, selected mechanical properties (hardness, properties in uniaxial pressure) and thermal stability of 5 magnesium alloys with different amounts of Ca and Zr were studied. Pure magnesium was also used for comparison.

Tab. 3 *Chemical composition of studied alloys*

Alloy	Chemical composition, wt.%		
	Zr	Ca	Mg
A		0.02	rest to 100%
B	0.10	1.07	rest to 100%
C	0.39	1.67	rest to 100%
D	0.41	3.17	rest to 100%
E	0.69	3.24	rest to 100%
F	1.82	0.93	rest to 100%

All studied alloys were prepared from pure magnesium (4N), calcium (2N5) and MgZr20 master alloy by melting in an electric induction furnace under a protective atmosphere of Ar. The melt was cast into a massive brass mold. Cooling rate during solidification was about 10 Ks^{-1}. After cooling, the cylindrical castings were machined into samples for further experiments. The chemical composition of alloys was determined using X-ray fluorescence spectrometry (XRF). The composition of the alloys is in the Tab. 3.

All samples for microstructure study by light microscope (Olympus PME3) and electron microscope (TescanVega3 LMU equipped with an energy dispersive spectrometer Oxford Instruments Inca 350) were prepared by a classical procedure including grinding, polishing and etching with 2% nital.

Hardness was repeatedly measured using the Brinell method with a WC ball with a diameter of 2.5 mm in full compliance with the valid ČSN EN ISO 6506-1 standard. The hardness of each sample was measured at least 5 times.

The uniaxial pressure test was performed using the LabTest 5.250SP1-VM universal machine. Each sample was measured three times at temperatures of 20, 150, 200 and 250 °C. The compressive yield strength and compressive strength were evaluated.

The thermal stability of the alloys was determined at temperatures of 150, 200 and 250°C. At certain intervals, the hardness of the alloys was measured by the Brinell method.

3 Results and discussion

3.1 Microstructure

Microstructures of studied alloys are in the Figs. 3-8. In each series of images, the microstructure of the alloy in the as-cast state, documented by a light microscope (a), is shown first. Another, detailed picture of the microstructure of the alloy in the as-cast state is taken with an electron microscope (b). The last picture shows a picture of the microstructure of the alloy after heat exposure (250°C/128h) taken with an electron microscope (c).

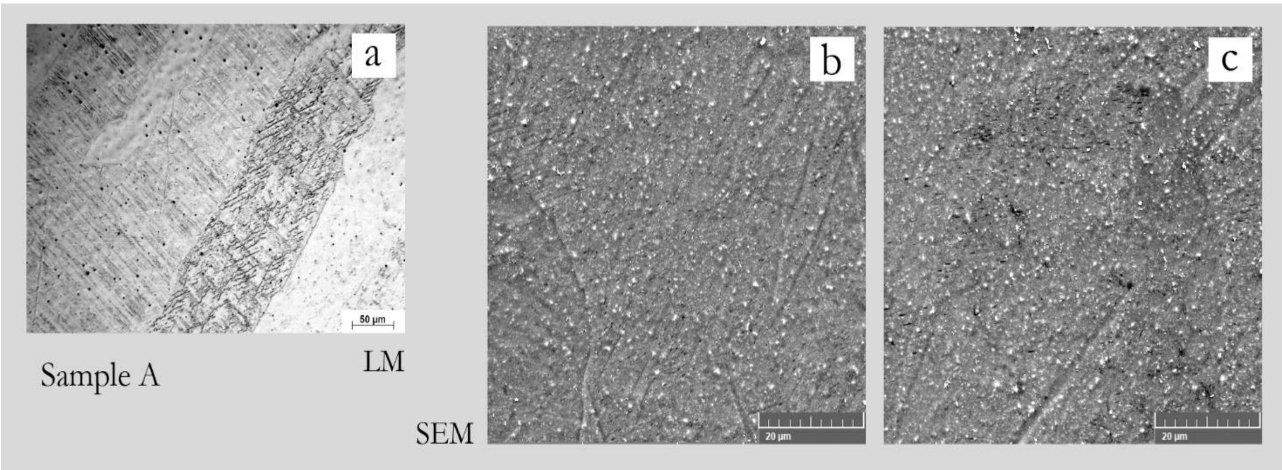

***Fig. 3** Microstructure of sample A, a) and b) as-cast, c) after heat treatment (250°C/128h)*

Fig. 3 shows the microstructure of magnesium. It consists of very large equiaxed grains. In some grains, there is visible twinning (see Fig. 3a), which is also observed in other samples. The magnesium microstructure was not visibly affected even by long-term annealing at a temperature of 250°C, as indicated by the microstructures in Fig. 3b and c.

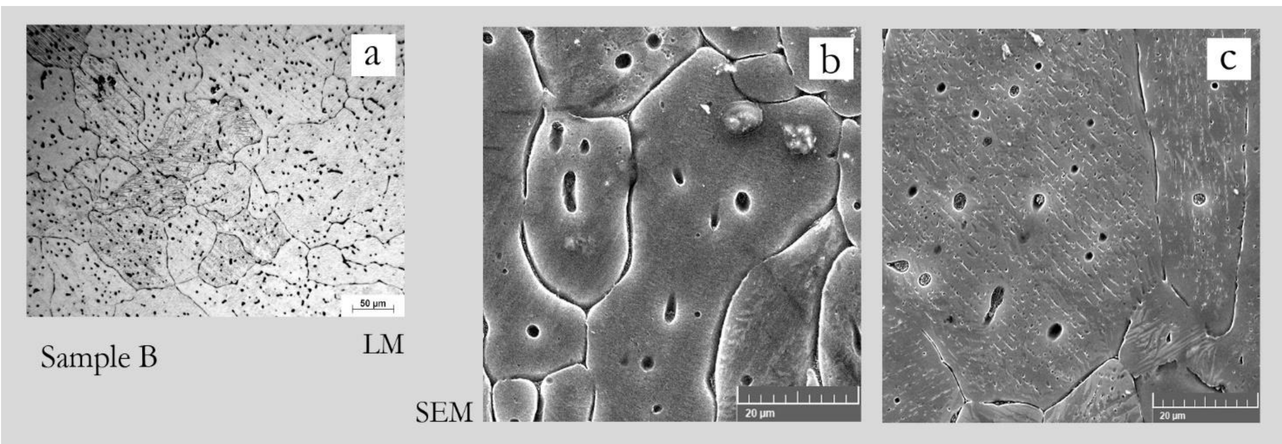

***Fig. 4** Microstructure of sample B, a) and b) as-cast, c) after heat treatment (250°C/128h)*

Figure 4 shows the microstructure of A alloy with the lowest Zr content (0.1 wt.%) and Ca content of 1.07 wt.%. The microstructure consists of dendrites of the primary Mg solid solution and phases with a high calcium content at the grain boundaries. The character of the phases in the interdendritic regions looks like an eutectic, however, based on the Mg-Ca phase diagram, the eutectic should appear in the microstructure of the alloys only when the calcium content exceeds 1.34 wt.%. The calcium content in this alloy is 1.07 wt.% and due to the high cooling rate during the preparation of the alloy (approx. 10 Ks^{-1}), it can be assumed that it is a non-equilibrium eutectic. In alloys B-E (Figs. 4-7), the calcium content is higher than its maximum solubility at the eutectic temperature, and the appearance of the eutectic in these alloys no longer contradicts the equilibrium phase diagram, however, their non-equilibrium character must also be considered in these alloys. Significant changes in microstructure are not visible even after long-term annealing of the alloy, see Fig. 4c.

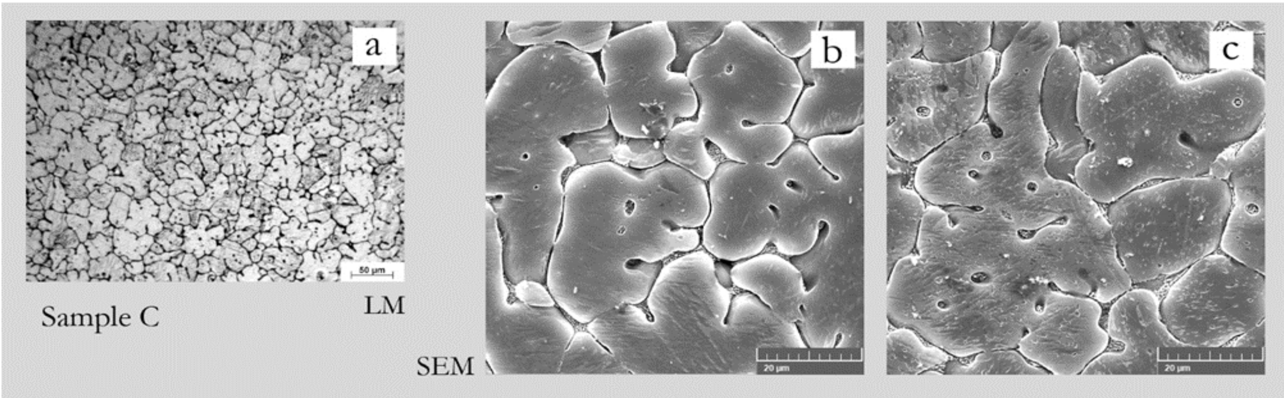

Fig. 5 *Microstructure of sample C, a) and b) as-cast, c) after heat treatment (250°C/128h)*

The microstructures of C-F alloys are similar to the microstructure of B alloy. They are formed by dendrites of a solid magnesium solution and phases formed in the interdendritic regions. They differ from each other mainly in the volume of phases in the interdendritic regions, see Fig. 5-8.

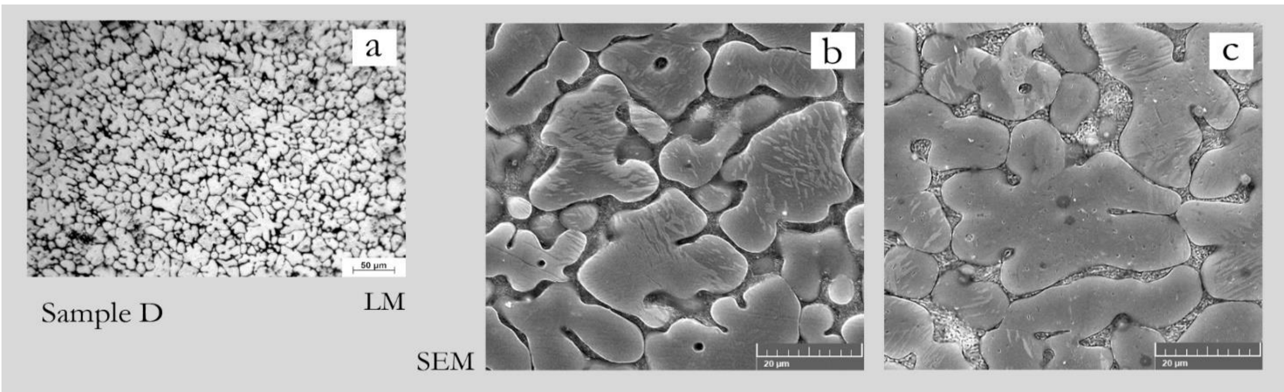

Fig. 6 *Microstructure of sample D, a) and b) as-cast, c) after heat treatment (250°C/128h)*

The microstructures show a very significant influence of the zirconium content on the morphology of the dendritic phase.

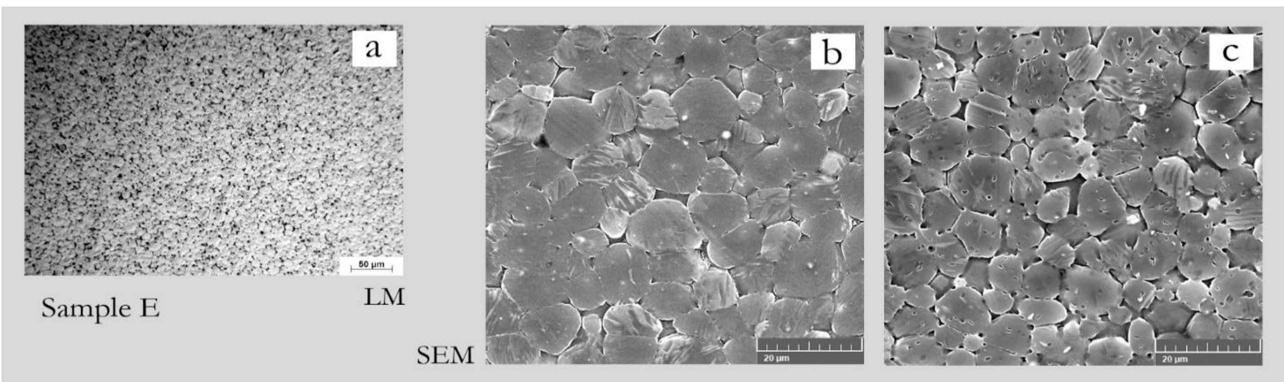

Fig. 7 *Microstructure of sample E, a) and b) as-cast, c) after heat treatment (250°C/128h)*

In the case of the studied alloys, the finest microstructure was achieved in the case of a Zr content of 0.60 wt.% in alloy E. This alloy is expected to achieve the best mechanical properties of all the studied alloys. In alloys with a lower Zr content (B-D), the microstructure is significantly coarser. A coarser microstructure was also surprisingly observed in alloy F with the highest Zr content (1.82 wt.%).

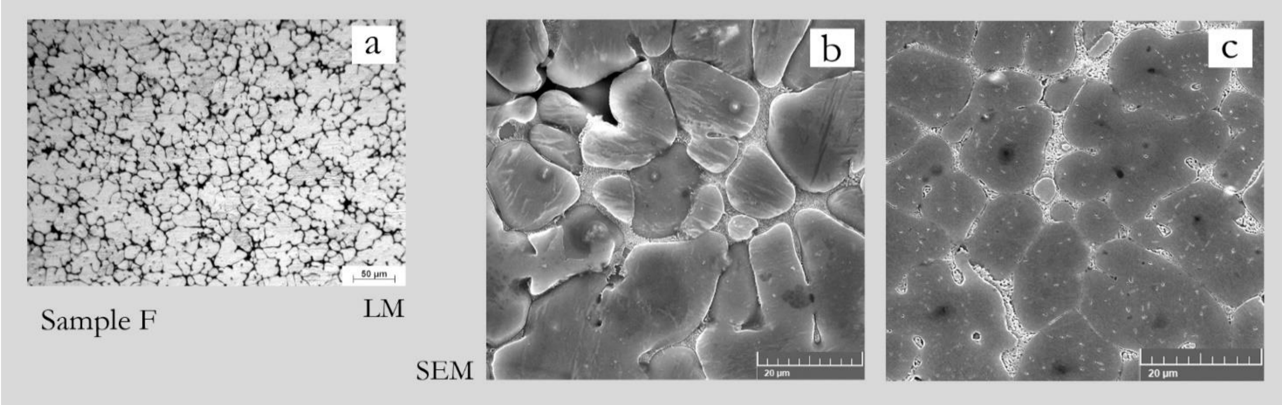

Fig. 8 *Microstructure of sample F, a) and b) as-cast, c) after heat treatment (250°C/128h)*

Fig. 9 shows an example of a map of the distribution of individual elements in the microstructure of the alloy (D) both in the cast state and after heat treatment. For the other alloys, the distribution of the elements present in the microstructure is similar.

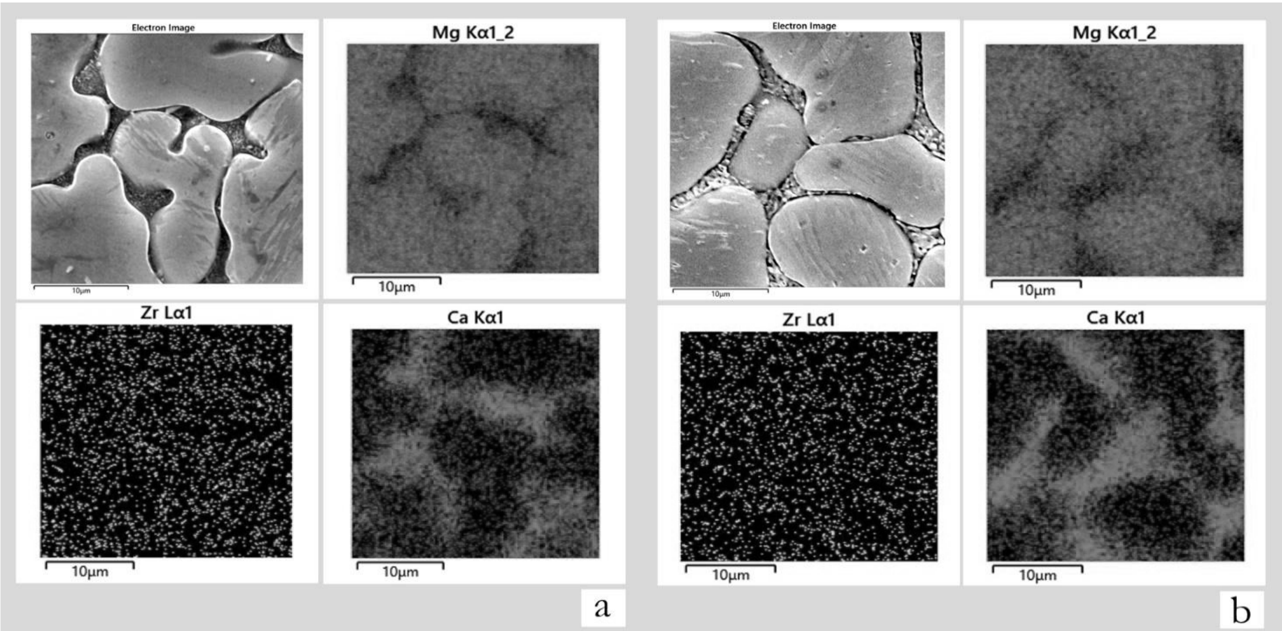

Fig. 9 *Distribution of elements in microstructure of D alloy, a) as-cast, b) after heat treatment (250°C/128h), SEM-EDS*

Both magnesium and zirconium are distributed uniformly in the microstructure. Calcium, on the other hand, forms the already mentioned phases (probably Mg + Mg_2Ca eutectic phase) in the interdendritic regions. The character of the microstructures of the alloys in the cast state and after heat treatment did not differ significantly, which shows the effectiveness of the used alloying elements for stabilizing the microstructure at elevated temperatures.

3.2 Mechanical properties

All mechanical properties of alloys are directly related to their microstructure. It is therefore not surprising that the alloy with the finest microstructure (E) has the highest mechanical properties. This is shown in tab. 4, where the hardness of alloys in the cast state are summarized. Alloy E achieved the highest hardness of all studied alloys. This is due to both the finest microstructure and the overall highest content of alloying elements.

Tab. 4 *Brinell hardnes of alloys, as-cast*

Alloy	A	B	C	D	E	F
HBW 2.5/31.25	36	43	51	59	61	53
Standard deviation	4	1	2	1	1	2

All studied alloys were further exposed at elevated temperatures (150, 200 and 250°C) as part of the thermal stability study and their hardness was continuously measured. The results are summarized in Fig. 10-12.

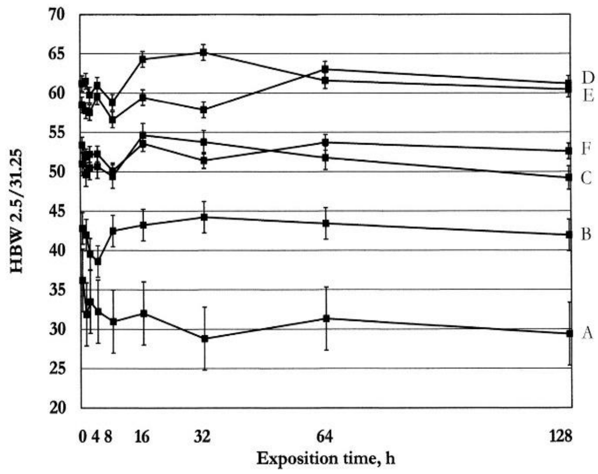

Fig. 10 *Thermal stability of alloys at the temperature of 150°C*

The character of the time dependences of hardness is similar for all alloys. In the first hours of annealing, phenomena the non-equilibrium casting microstructure bringing closer to thermodynamic equilibrium probably occur in the alloys. This is manifested in the determined curves by significant fluctuations in the hardness values. After that, the hardness stabilizes and is followed by a very slight decrease in the hardness values. For all studied alloys, their hardness decreases when the temperature increases.

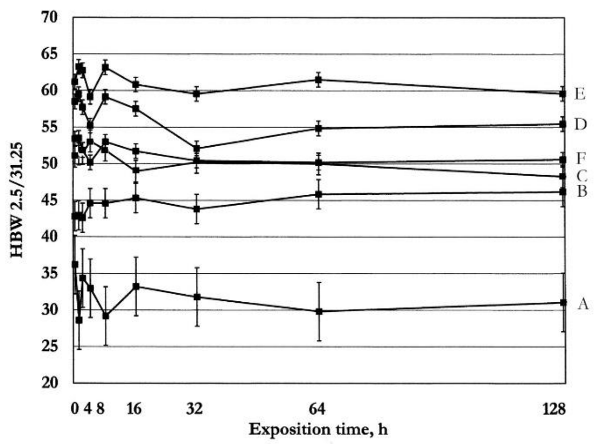

Fig. 11 *Thermal stability of alloys at the temperature of 200°C*

Calcium and zirconium have been proven to stabilize the microstructure of alloys at higher temperatures. The higher the amount of alloying elements in the alloy, the higher the mechanical properties of the alloy.

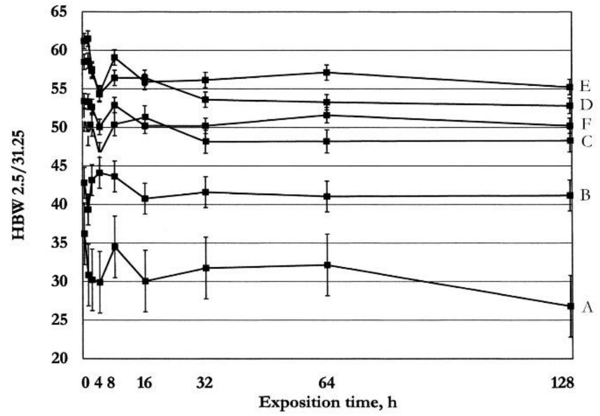

Fig. 12 *Thermal stability of alloys at the temperature of 250°C*

All studied alloys were also subjected to a uniaxial pressure test at normal temperatures as well as at elevated temperatures (20, 150, 200 and 250°C). The compressive yield strength and compressive strength were evaluated. The results are shown in Fig. 13 and 14. In the case of pure magnesium, it was not possible to calculate the compressive strength limit values at temperatures of 150°C and 150°C due to its plasticity. Due to the gravity casting technology used for the preparation of the alloys, the potential influence of foundry defects cannot be excluded. For most alloys, there was a slight decrease in mechanical properties with increasing temperature.

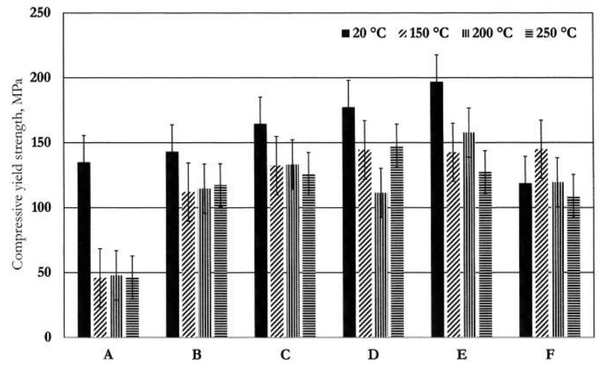

Fig. 13 *Compressive yield strength of alloys*

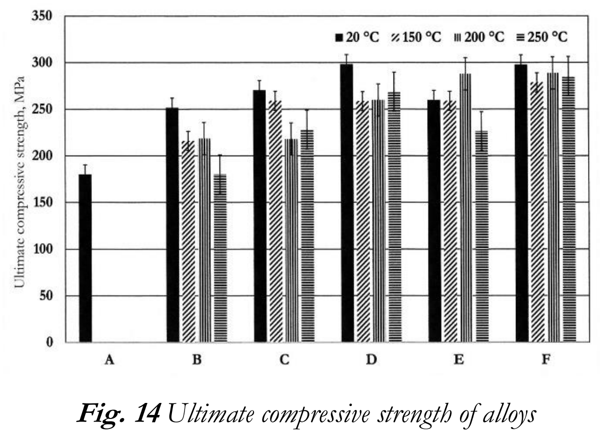

Fig. 14 *Ultimate compressive strength of alloys*

4 Conclusion

The microstructure of all studied Mg-Ca-Zr alloys is formed by dendrites of magnesium solid solution. Phases containing calcium (eutectic Mg + Mg_2Ca) are excluded in the interdendritic regions. Zirconium is distributed uniformly in the microstructure of alloys. The most significant refinement of the microstructure was achieved in the case of alloy with a content of 0.6 wt.% Zr.

The alloying of magnesium with calcium and zirconium had a positive effect on the thermal stability of the alloys at all studied temperatures. The decrease in hardness with time during constant temperature annealing was very slight or negligible. In the initial phase of annealing, most alloys showed fluctuations in hardness values, which can be explained with the tendency of the cast microstructure to approach the thermodynamic equilibrium.

The mechanical properties of the alloys under pressure at constant temperature increased with increasing amounts of alloying elements. The mechanical properties of the alloys in compression decreased slightly with increasing temperature, which is important for the use of these alloys for applications at elevated temperatures.

The alloying of magnesium with calcium and zirconium appears very promising to produce magnesium alloys with increased resistance at elevated temperatures. As a cheap metal, calcium has the potential to fully replace deficient rare earth metals.

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