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Mechanical alloying of Mg-Zn-Ca-Er alloy

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Abstract. Magnesium-based materials are promising alternatives for medical applications, due to their characteristics, such as good mechanical and biological properties. Which opens many possibilities for biodegradable materials to be used as less-invasive options for treatment. The degradation is prompted by its chemical composition and microstructure. Both those aspects can be finely adjusted by proper manufacturing process, such as mechanical alloying (MA). Furthermore, MA allows for alloying of elements, that normally would be really hard to mix due to their very different properties. Magnesium usually needs various alloying elements, which can further increase its. Alloying magnesium with rare earth elements is considered to greatly improve the aforementioned properties. Due to that fact erbium was used as one of the alloying elements, alongside zinc and calcium to obtain $Mg_{64}Zn_{30}Ca_4Er_1$ alloy via mechanical alloying. The alloy was milled in the SPEX 8000 Dual Mixer/Mill high energy mill under an argon atmosphere for 8, 13, and 20 hours. It was assessed by X-ray diffraction, energy dispersive spectroscopy, granulometric analysis, and hardness. The hardness values reached 232, 250, and 302 HV respectively, which is closely related to their particle size. The average particle sizes were 15, 16, and 17 μm respectively.

Keywords: magnesium; mechanical alloying; erbium; rare earth elements

1. INTRODUCTION

Scientific research has always been continuously directed towards the improvement of used materials and their performance [1]–[3]. Improved properties have been obtained by many different processing techniques, such as chemical, mechanical, laser treatment and thermomechanical methods [3]–[5]. Because of this, ever-increasing demands for improvement have resulted in the development of both advanced materials and methods of their production [6].

Mechanical alloying (MA) is one of the abovementioned methods, which uses the mechanisms of repeated welding, fracturing, and re-welding of powdered particles. It allows for the production of homogeneous materials from a blend of powdered mixtures [1], [7]. It fits very well into the definition of advanced materials, as these are ones where the first consideration is given to the systematic synthesis and control of the structure of the material to provide a precisely tailored set of properties for demanding applications [6], [8].

MA allows for “precisely tailored properties” due to the many parameters which can be optimized during the milling process, such as the milling time, the milling medium, the ball-to-powder ratio, the milling atmosphere, and more. This method usually consists of a dry (although it can be wet as well) high-energy ball milling. As MA is one of the solid-state processing methods [9], it allows for the formation of different materials classes such as amorphous alloys, which usually are difficult to obtain by classic methods (i.e. casting) [10], [11]. Furthermore, it is possible to achieve a finer microstructure in crystalline, as well as nano-crystalline and amorphous alloys [12]–[20].

Thanks to these qualities it is possible to obtain specific

materials which normally would be very hard or high impossible.

That being said the magnesium alloys are one of the most important alloys nowadays. They are lighter while holding their strength. Moreover, they exhibit many beneficial properties such as good mechanical properties and corrosion resistance. The interest in magnesium can be found between many fields of science and industry, such as automotive or aerospace, and recently they are being considered as potential material for medical application as well. Magnesium alloy based implants, for instance, could be potentially better than their titanium or stainless steel counterparts. Mechanical strength apart, which is comparable to the bone tissue, they offer really good corrosion resistance. As it is known the human body is a very specific and very corrosive type of environment. Due to that fact, not many materials can be used as implants. Yet when coupled with the MA process it is possible to prepare highly specialized alloy for any purpose. It is faster, cheaper, and more specialized as compared to more traditional methods [21], [22].

Moreover, magnesium is a very specific material that by itself is not so strong and very corrosive. However, when prepared as an alloy with adequate materials it is possible to achieve the best possible characteristics. In this work Zn and Ca additions are used to improve both mechanical strength and corrosion resistance [23]–[28]. To further improve its properties rare earth elements (REE) can be used [20]. Although they exhibit low toxicity, they can be tolerated to a certain extent. However, they considerably improve the alloy mechanical properties and microstructure in certain cases [29], [30]. Many REEs can be used, as the majority of them increases the mechanical properties, and have a higher

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resistance to in vivo degradation [31], [32]. Erbium, as one of the REE is interesting because it was not widely researched in terms of possible uses in biomaterials. The effects of trace erbium additions were investigated in titanium and aluminum alloys. In the case of Mg-Al alloys, erbium addition has improved the purity of the alloy, refined the grains, and exhibited more uniform grain distribution. Overall, mechanical properties were improved [33], [34]. Mg-Er alloys were investigated as well, although in larger quantities in terms of atomic composition [32].

The novelty of this paper is a new approach to the issue of potential materials for medical applications. Among other materials known in the literature, magnesium-based materials are promising alternatives for medical applications, due to their characteristics, such as good mechanical and biological properties, corrosion resistance and the reactive ability to interact with other elements. In fact, there are not many advanced studies on the influence of Er on the properties of cast magnesium-based alloys in the literature [32], [33], [34]. However, no single study to date reports an influence of Er on the effect of Er on the properties of magnesium-based alloys produced by mechanical alloying. Hence the idea was born to conduct the research starting from the basic elements, which is the influence of the milling time on the phase composition and microstructure of the material. In this paper, the quaternary Mg-Zn-Ca-Er alloy was investigated as a part of a preliminary study to ascertain the effect of the high energy milling times on the $Mg_{65}Zn_{30}Ca_4Er_1$ alloy, as well as its microstructure and properties. For better clarification, the samples will be represented as Er₁8, Er₁13 and Er₁20, where 8, 13 and 20 is the milling time in hour.

2. MATERIALS AND METHODS

The powders were processed by mechanical alloying. The nominal composition of the alloy was $Mg_{65}Zn_{30}Ca_4Er_1$. The initial mixture consisted of a mixture of pure powders Mg (99.99% wt.%), Zn (99.99% wt.%), Er (99.99% wt.%), and Ca pellets (99.99% wt.%). The mixture of powders with stainless steel milling balls was closed in stainless steel cylinders under high-purity argon (99.99% wt.%) atmosphere. The ball-to-ratio powder was 10:1.

The processes were carried out on the SPEX 8000D high-energy shaker ball mill at room temperature. The milling consisted of 30 min steps every 1 hour in shaking mode. The samples were alloyed at a constant frequency with varying milling times of 8, 13, and 20 hours.

The X-ray diffraction (XRD) patterns of studied alloys were measured by using the Empyrean Diffractometer with Cu radiation ($\lambda_{K\alpha_1} = 1.5418 \text{ \AA}$) and PIXcell detector. Phase analyses of substrates and milling products were performed with a High Score Plus PANalytical software integrated with the ICDD PDF4+ 2016 data base and structural studies were done using the Rietveld refinement method integrated with the PANalytical High Score Plus software.

Zeiss 35 scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS) was used to assess the morphology of the obtained powders. The level of accuracy for EDS of major elements is better than $\pm 2\%$.

The particle size distribution of the alloy powders were measured using the Fritsch Analyssette 22 MicroTec+ in ethyl alcohol.

The hardness test was performed on a Future-Tech FM700 Vickers hardness tester with 15 seconds dwell time and 50 grams of force (HV0.05). The samples were mounted in carbon-based conducting resin, ground and polished. The powder grains were then viewed under microscope in order to select an appropriate grain with sufficient surface area to conduct the microhardness testing.

3. RESULTS AND DISCUSSION

A. Phase composition.

The comparison of XRD scans obtained for 8, 13 and 20 hours of milling is presented in **Fig. 1**. The XRD patterns mainly showed the presence of a hexagonal close-packed (HCP) Mg structure (solid state solution) and $MgZn_2$. Moreover, in all milled samples (**Fig. 1**) the presence of unreacted erbium (Fm3m, cubic structure) has been identified. The structural analysis showed well defined erbium nanocrystallites with values above 500 Å. Crystallite size and changes of unit cell parameters of the Mg-based solid state solution and intermetallic $MgZn_2$ phase are presented in **TABLE 1**.

TABLE 1. Crystallite size and changes of unit cell parameters of the Mg-based solid state solution and intermetallic $MgZn_2$ phase - main two phases present in alloys after 8, 13 and 20 hours of milling.

Sample (milling time)	Mg(Zn, Er, Ca)				$MgZn_2$			
	Theoretical (ICDD PDF4+ card: 04-015-0486)	Refined (RR) a/c [Å]	Crystallite size D [Å]	Lattice strain η [%]	Theoretical (ICDD PDF4+ card: 04-008-7744)	Refined (RR) a/c [Å]	Crystallite size D [Å]	Lattice strain η [%]
8	a = 3.2110 c = 5.2130	3.2066(2) 5.2050(9)	370	0.26	a = 5.2210 c = 8.5670	5.2249(9) 8.5672(8)	300	0.09
13	Space Group: $P6_3/mmc$	3.2040(4) 5.1869(3)	300	0.17	Space Group: $P6_3/mmc$	5.3406(2) 8.7977(6)	150	0.37
20	Crystallographic System: hexagonal	3.1853(8) 5.1292(6)	270	0.22	Crystallographic System: hexagonal	5.4855(2) 8.8054(1)	110	0.46

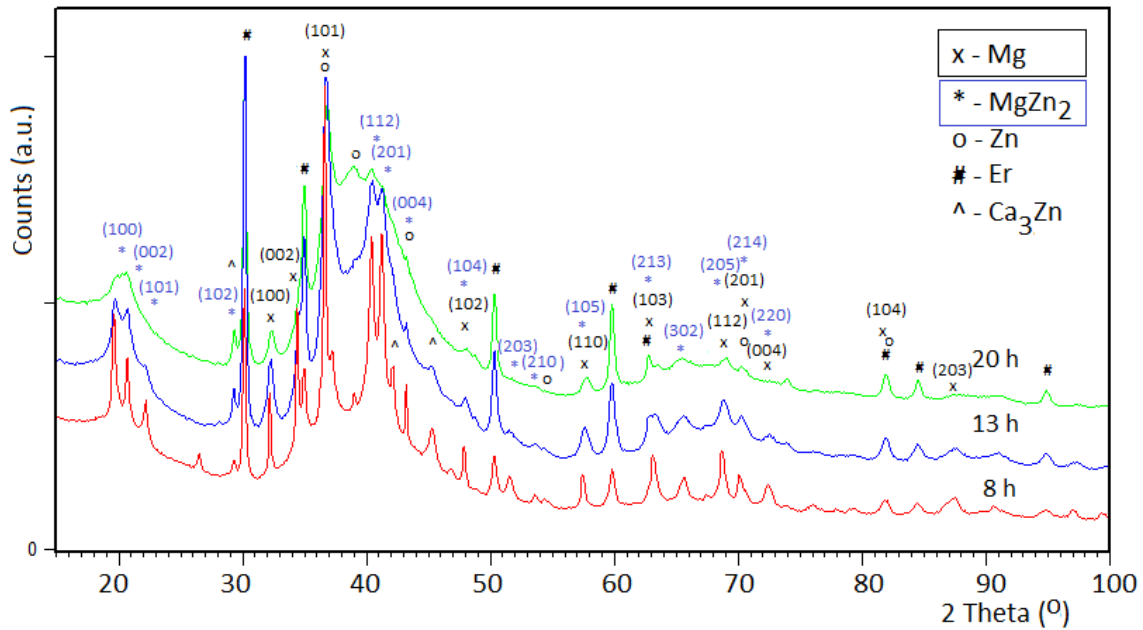


FIG. 1. X-ray diffraction patterns of the Mg-Zn-Ca-Er powders milled for 8, 13 and 20 h.

B. Granulometry and hardness.

The cumulative volume curve distributions and the general volume distribution histograms for Mg-based alloys with varying milling times are presented in FIG. 2 (a-c) for 8, 13, and 20 hours, respectively.

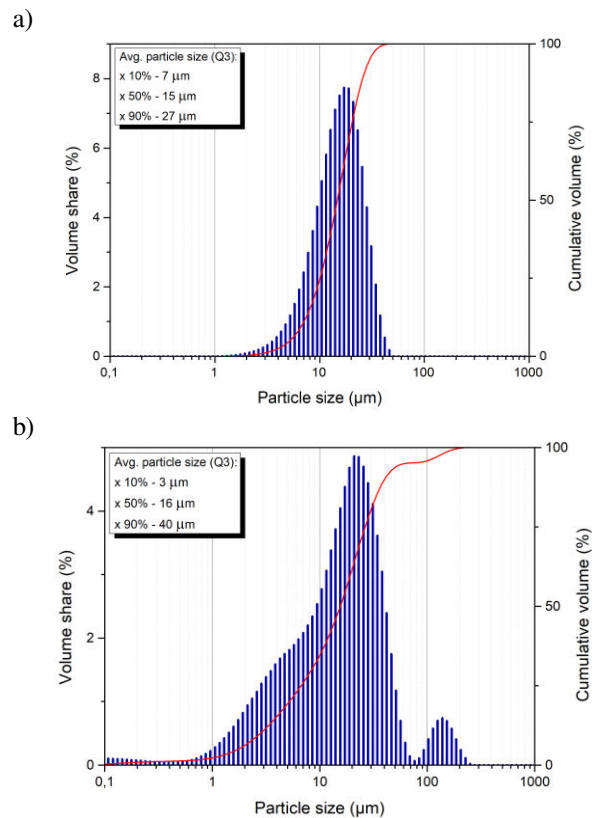
The median value (Q3x50%) for each result was 15, 16, and 17 μm for subsequent samples. The average particle sizes are presented in TABLE 2, for a clearer representation. It can be seen that the samples are uniformly distributed between ~10 and ~60 μm in all cases. In FIG. 2 (b) the smaller peak around 100 μm is caused by particle agglomeration. Although the particles decrease in size during milling, due to the finer particle share (Q3x10%) in FIG. 2 (b-c) being larger than after 8 hours in FIG. 2 (a).

TABLE 2. Hardness test results and average particle size for samples milled for 8, 13, and 20 hours.

Sample	Hardness (HV0.05)				Avg. particle size [μm]
	Exp.1	Exp.2	Exp.3	Avg.	
Er ₈	198	246	253	232 ± 24	15 ± 0.8
Er ₁₃	253	265	233	250 ± 13	16 ± 1.8
Er ₂₀	302	294	309	302 ± 6	17 ± 1

The constant size of the average particle is pointing to the powders being continuously refined, as a result of constant crushing and rewelding [35]. Those claims are supported by

the hardness results, which increase with the milling time. The average hardness is 232, 250, and 302 HV for samples after 8, 13, and 20 hours, respectively. More detailed results can be seen in TABLE 2. As the particles change to ultrafine particles their hardness increases [36].



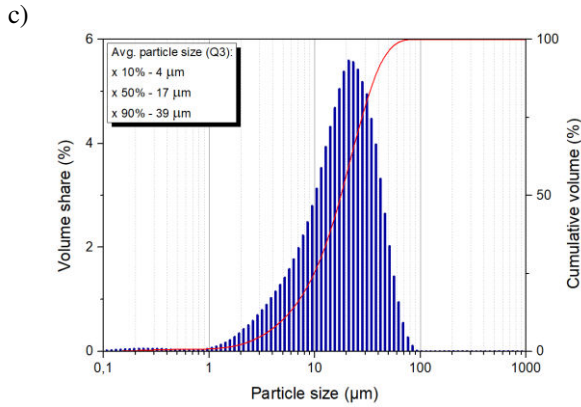


FIG. 2. Particle size volume share (histogram) and their cumulative distribution (curve) for $Mg_{65}Zn_{30}Ca_4Er_1$ alloy milled for a) 8, b) 13 and c) 20 hours.

C. Scanning Electron Microscopy – Morphology and qualitative chemical composition analysis.

The micrographs representing the powders share many similarities, as seen in **FIG. 3**.

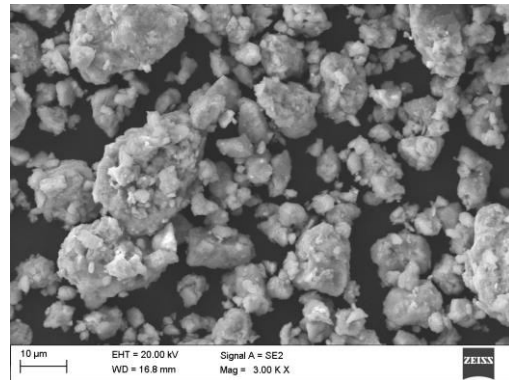
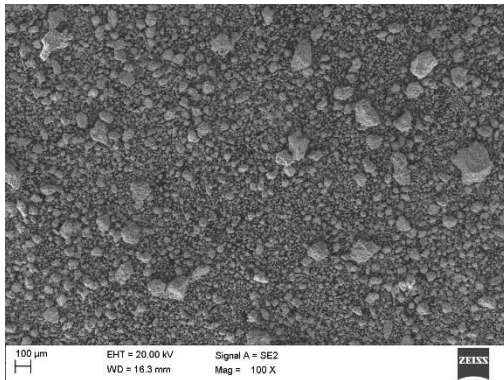
Visible irregular shapes can be seen and areas with finer powders. Those irregular shapes are agglomerations caused by the repetitiveness of the mechanical alloying process. As the powder particles constantly clash with each other, they are subjected to welding and fracturing, hence the agglomerations occur. The particles may seem bigger in **FIG. 3 (a)** than those in **FIG. 3 (b-c)** as opposed to the granulometry results in **FIG. 2**. But this results from agglomeration and re-

welding of the powders are later crushed and finish as a finer powder. Moreover in **FIG. 2 (b)** the volume share of finer particles is much larger than in **FIG. 2 (a)**. Hence the difference of sizes of the particles in **FIG. 3 (a)** and **(b)**, albeit having similar average particle size as reported in **FIG. 2**

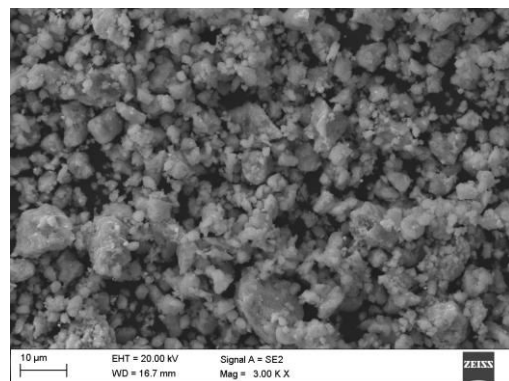
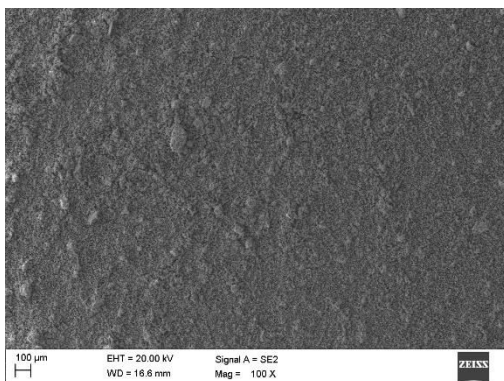
The results from the energy-dispersive spectroscopy are presented in **TABLE 3**. The chemical composition is stable after 8 hours of milling and does not vary much from the nominal composition of the powder mixture inserted before milling. This trend continues over 13 and 20 hours (**FIG. 4**), meaning the chemical composition is stable and the only changes occurring are of structural and mechanical nature.

The roundness and uniform distribution of the particles results from longer milling times as was studied by Dobrzański [37], [38]. This effect can be seen and compared between micrographs in **FIG. 3 (a-c)**. Moreover the analysis has confirmed the presence of unreacted, nanocrystalline erbium (~500 nm) which can be seen in **FIG. 4 (d)** and has been identified in the XRD analysis in **FIG. 1**. Maps analysis showed a very uniform distribution of unreacted particles in the Sample. It was revealed that the unreacted Er are firmly bound to the Mg matrix (**FIG. 3**, **FIG. 4**). Also can be observed areas with an increased atomic share of the remaining structural components of the powder, i.e. Zn (**FIG. 4 (b)**) and Ca (**FIG. 4 (c)**).

a)



b)



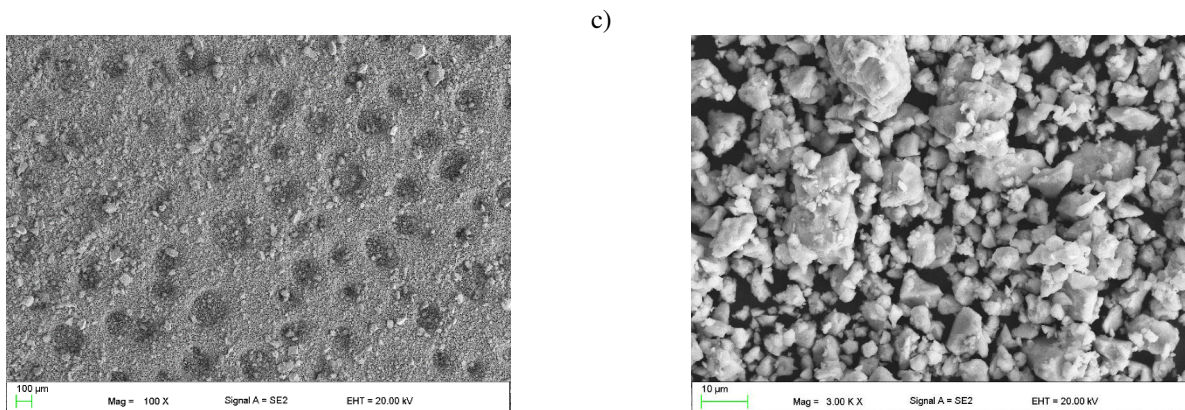


FIG. 3. Selected SEM micrographs showcasing the $Mg_{65}Zn_{30}Ca_4Er_1$ alloy after a) 8, b) 13 and c) 20 hours.

TABLE 3. EDS results for samples milled for 8, 13, and 20 hours. The level of accuracy for EDS of major elements is better than $\pm 2\%$.

Sample	Chemical composition (at.%)			
	Mg	Ca	Zn	Er
Er ₁ 8	65.2	4.8	29.2	0.8
Er ₁ 13	64.2	4.0	30.6	1.2
Er ₁ 20	67.1	3.8	28.1	1.0

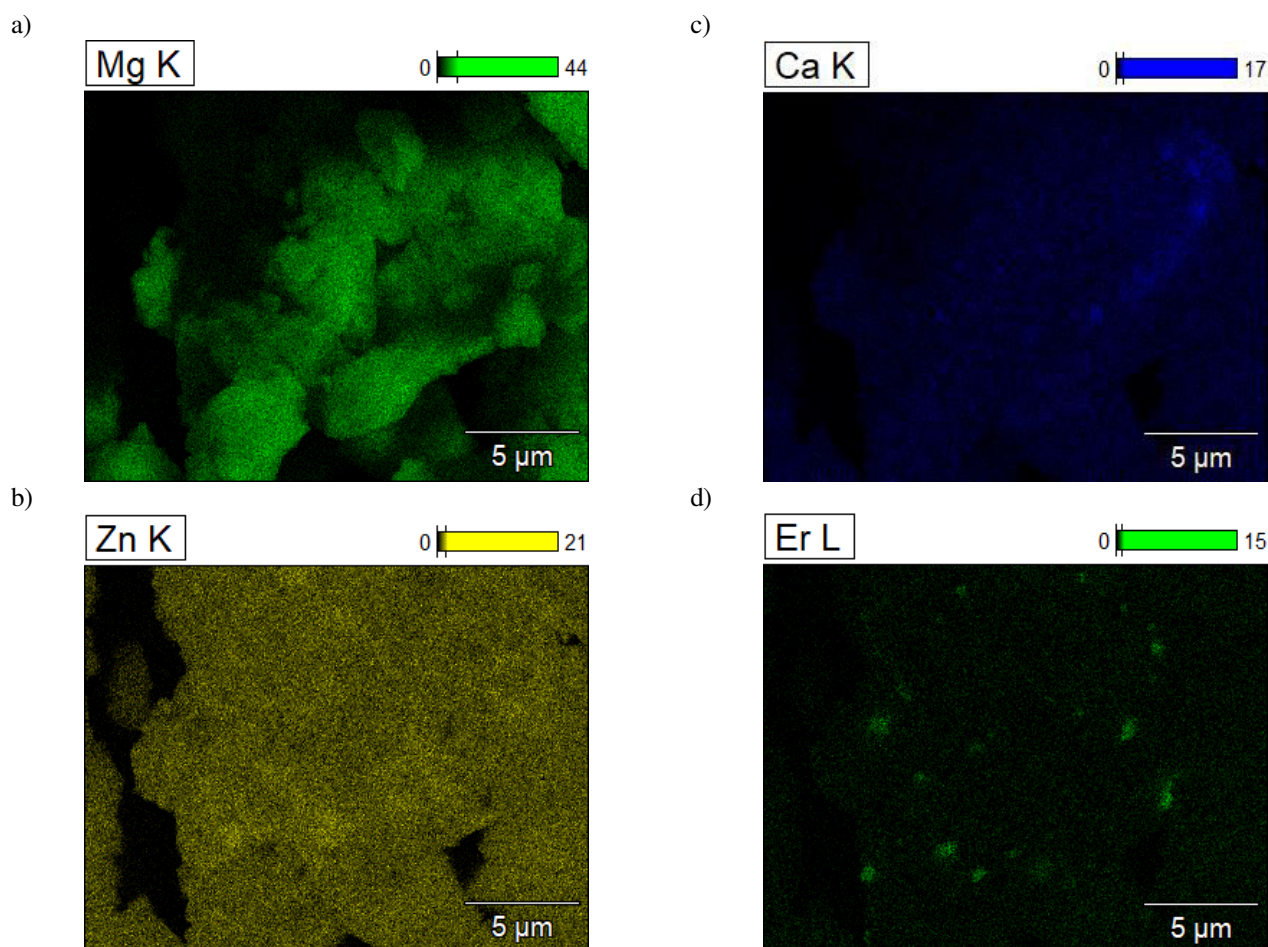


FIG. 4. EDS elemental mapping for $Mg_{65}Zn_{30}Ca_4Er_1$ milled for 20 h visible in Mg (a), Zn (b), Ca (c) and featuring an unreacted Er (d).

4. CONCLUSIONS

In order to fully examine how the erbium reacts with magnesium alloys during the MA process, it is important to consider the process parameters such as the milling time and its influence on the phase composition and microstructure. Those aspects are not commonly discussed in various articles, books and journals that were reviewed in order to gather the data backing up this research. Thus, in order to characterize both physical and chemical properties of the designed material the following tests were used: X-ray diffraction method, SEM microscopy, particle size distribution by use granulometric analysis, chemical composition by use energy dispersive spectroscopy and Vickers hardness (HV). Results of those tests were taken into the consideration and presented below:

The $Mg_{65}Zn_{30}Ca_4Er_1$ was prepared and milled for 8, 13, and 20 hours. As follows, the effect of milling times on its morphology, hardness, chemical, and phase composition of $Mg_{65}Zn_{30}Ca_4Er_1$ alloy were investigated.

The particles of the milled powders are characterized by size between the ranges of 10 to 60 μm , with 15, 16, and 17 μm of average values for 8, 13, and 20 hours of milling time. Although different the average values are in the error range, hence it can be said that they are statistically similar. Basing on this statement, it can be concluded that the powders reached the moment of further refining instead of decreasing in size, although the finer particle share increases with the milling time.

The Vickers hardness tests yielded results of 232, 250, and 302 HV showing that the hardness increases with the milling time. The EDS studies revealed the stability of the chemical composition and SEM micrographs show a uniform distribution of fine particles. XRD analysis revealed an unreacted erbium (Fm3m, cubic structure) in powder after 20 hours of milling.

As this was a part of a preliminary study, the $Mg_{65}Zn_{30}Ca_4Er_1$ alloy powder will be taken into future consideration for sintering materials.

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