International Journal of Modern Manufacturing Technologies
ISSN 2067–3604, Vol. XV, No. 2 / 2023
https://doi.org/ 10.54684/ijmmt.2023.15.2.63

STUDY OF THE TRIBOLOGICAL AND MECHANICAL PROPERTIES OF SOME BIODEGRADABLE Mg-Ca-Zn ALLOYS

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Abstract: Magnesium-based biodegradable alloys used in medical applications have been intensively researched throughout the years. As is well known, the biodegradability of medical alloys is improved by alloying Mg with biocompatible and non-toxic metals. According to studies, Mg, Zn, and Ca are abundant in the human body and help to the regeneration of bone tissue efficiently and effectively. Due to their biodegradability, magnesium alloys, particularly Mg–Ca–Zn alloys used to manufacture implants, reduce patient stress by eliminating the need for a second surgery. Due to magnesium alloys' similar Young's modulus (41-45 GPa) to that of natural bone, the stress shielding effect can be significantly reduced. The objective of this research is to examine the tribological and mechanical properties of a novel experimental biodegradable alloy composed of magnesium (Mg) and 0.5 wt. % Ca, with variable amounts of zinc (Zn) at 0.5, 1.5, and 3.0 wt. %, with the aim of improve the mechanical characteristics. The microstructure and mechanical characteristics were determined using light microscopy (LM), a CETR UMT-2 Tribometer, and an Instron 34SC-5 instrument. A magnesium solid solution as well as a lamellar Mg_2Ca and a $MgZn_2$ intermetallic compound were discovered as a result of this research. The tribological parameters exhibit a decrease in coefficient of friction (COF) with increasing Zn concentration and a little increase in hardness and Young modulus up to 3 wt.%.

Key words: Mg-Ca-Zn biodegradable alloys, light microscopy, tribological properties, mechanical properties

1. INTRODUCTION

In biomedical applications such as bone tissue regeneration medicine, metallic materials are increasingly utilized due to their exceptional mechanical strength properties, which are somewhat missing in other biomaterials such as ceramics and polymers [1,2]. At the beginning of the 19th century, new biocompatible metallic orthopaedic devices with greater ductility and fatigue resistance were manufactured and studied [3-5].
Currently, the metallic materials used in the clinic for the improvement, repair or replacement of a damaged or destroyed bone tissue are represented by stainless steel (316L), cobalt-chrome based alloys (Co-Cr), and titanium (Ti), which are manufactured by several methods [6,7]. However, despite the favorable biocompatibility, satisfactory mechanical properties, stability and relatively low immunogenicity, traditional metal implants face a series of shortcomings that limit their use in hard tissue applications [8,9].
First of all, the specific integration process of an implantable device largely depends on the reactions it can induce at the local site level, reactions that can influence the biocompatibility of the material. Thus, when the biomaterial is exposed to a physiological environment, in contact with biological fluids, over time, it can degrade leading to the release of toxic particles for the tissue surrounding [10,11].
Thus, traditional metallic biomaterials, following corrosion and wear processes, release toxic particles and metal ions such as Cr, Co, nickel (Ni), which can lead to the appearance of a persistent chronic inflammatory process that affects the biocompatibility of the implant and causes tissue damage its proximity [12]. Second, implantable materials can lose their stability when there is a mechanical incompatibility between bone and implant due to an inadequate Young's modulus. That is why it is desirable that the biomaterial used has a modulus of elasticity equivalent to that of the surrounding bone tissue. The modulus of elasticity of the bone tissue differs according to age, thus in adults the compact bone shows a low elasticity and plasticity and an increased hardness compared to the bone tissue of children. Compared to the Young's modulus of natural bone tissue (4–30 GPa), non-degradable

metals possess a much higher elastic modulus (316L - 190-210 GPa; Co-Cr alloy - 210-253 GPa; Ti – 105- 110 GPa), thus leading to the emergence of the so-called "stress shielding" phenomenon, a term that refers to the inhomogeneous transfer of stress between the implant and the bone [13].
This clinical phenomenon can result in bone refracture, tissue atrophy, and weakening of the implantable device-bone interaction, thus necessitating a second surgical revision of the device. Finally, with the end of the natural healing process, an additional intervention to remove the inert metal implants is absolutely necessary. In this context, biodegradable metallic materials with mechanical properties similar to human bone tissue (increased hardness and low modulus of elasticity) have begun to attract the attention of researchers in the field, and among the potential candidates, alloys based on magnesium (Mg), iron (Fe) and zinc (Zn) have become the subject of a vast number of studies aimed at evaluating corrosion resistance and biological behaviour both in vitro and in vivo [14-18].
Mg and its alloys are of particular interest in the field, being used as biodegradable metallic materials that enter into the design of orthopedic, dental implants and spinal fixation devices [19-24]. Mg alloys have a major advantage represented by their mechanical properties suitable and compatible with human bone, having good biocompatibility and a corrosion rate in correlation with the healing rate of bone tissue. These aspects rank Mg-based alloys at the forefront of the attention of researchers in the field [25-27].
Recently, magnesium alloys is proposed for biomedical applications, such as: Mg–Ca, Mg–Zn, Mg–2.0Zn–0.2Mn, Mg–1.2Mn–1.0Zn, Mg–Ca–Zn, and Mg–Zn–x (x = Ca, Mn, Si) [5,15,17]. According to Mao et al. [27], when the Zn concentration increases, the grain size is refined and the mechanical properties may be increased. Rosalbino et al. [11] say that Mg-Zn-x (x = Ca, Mn, Si) alloys may satisfy the mechanical strength and corrosion resistance requirements for hard tissue implants [28-30], however neither publication has investigated the cytotoxic effects. For their applications, it is crucial to adapt the mechanical characteristics, corrosion resistance, and biocompatibility of Mg-Zn-Ca systems through alloy composition optimization [31-32].
Incesu et al. [33] demonstrate that raising the number of alloying elements (Zn and Ca) in magnesium decreases grain structure and increases strength. Although Horky et al. [34] certify that decreasing the melting temperature significantly reduces the size of the grains of the initial bimodal pattern of grain size, raising the level of deformation (increasing the number of D-ECAP passes) almost exclusively decreases the area fraction of the larger grains. Sun et al. [35] reported that the incorporation of Zn and Ca into a magnesium alloy results in an increase in its corrosion potential. Furthermore, the in vitro degradation rate of the Mg-4.0Zn-0.2Ca alloy in SBF [36] was found to be similar to that of high-purity magnesium.
The purpose of this investigation is to examine the microstructural, tribological, and mechanical characteristics of a novel experimental Mg-based biodegradable alloy — Mg–0.5wt.% Ca with controlled additions of Zn as follows: 0.5, 1.5, and 3.0 wt.%. Light microscopy, microindentation test, scratch analysis, and tensile testing were used to examine alloys that were produced in a controlled atmosphere electric resistive melting furnace. This research describes the structure and mechanical characteristics of novel experimental biodegradable Mg-based alloys, while figure 1 presents a schematic summary of the article.

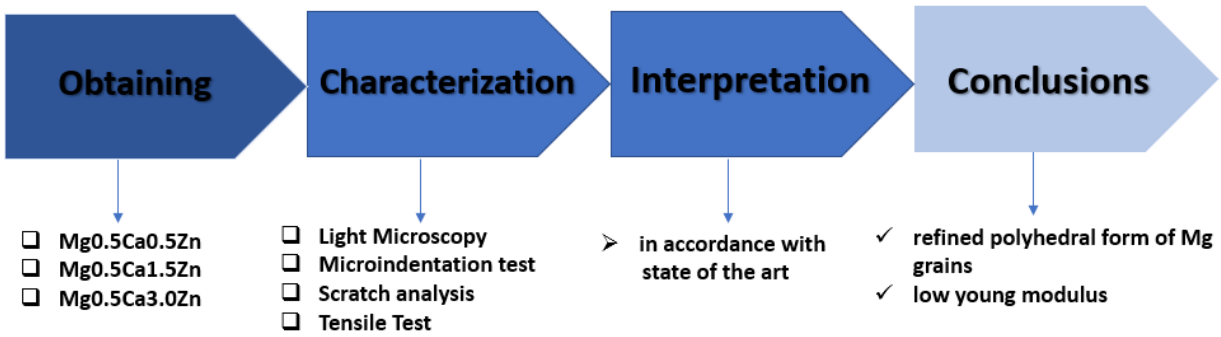

Fig. 1. Schematic overview of the paper.

2. MATERIALS AND METHODS

2.1. Microstructural analysis

In the Mg-Ca-Zn alloying process, Mg-98.5 wt.%, Mg-Zn (80% - 20% wt.%), and Mg-Ca (85% - 15% wt.%) were used as high-purity components and master alloys. For melting Mg-Ca-Zn alloys, a SY0002-2000W-1Kg electric resistive melting furnace with a controlled atmosphere was used [37]. Using an electronic weighing balance, the raw materials were dosed for each batch and prepared for casting. The melting technique included the following steps: melting by heating at 720 °C for 5 minutes for homogeneity; purging the graphite crucible with inert gas (Ar 4.8) while melting; and homogenization using a stainless-steel rod by continuous mixing of the

load.
The optical microscopy analyses were carried out on cylindrical samples, approximately 20 mm in diameter and 2 mm thick, prepared in advance by grinding, polishing and attack with specific chemical reagents. A LEICA 5000DMI Optical Microscope (Leica, Wetzlar, Germany) was used for optical microscopic analysis. The samples were sanded with SiC abrasive paper of different sizes of 180, 360, 500, 600, 800, 1200 and 1500 and polished until a metallic luster was obtained on the surface of the samples. The alumina slurry used for polishing was 0.3 and 1 µm. The microstructure is obtained by metallographic chemical attack, it was made with a mixture that has in its composition 2 ml HF (48%), 2 ml HNO_3 (concentrate) and 96 ml H_2 O.

2.2. Microindentation test & scratch analysis

For the micro-indentation test, specimens with a 20 mm diameter and 2 mm thickness were used. A Rockwell diamond-tipped indenter with a 120° opening angle at its edge was utilized. For greater accuracy, three indentation measurements were performed on the surface of each sample. The analysis consisted of an increase in indentation force from 0 to 5 N, followed by a return to the starting position. CETR-UMT Test Viewer was the program for micro-indentation and microscratch testing. For the micro scratch test, the same half-disc samples were used, and a micro lamella was moved across a distance of 4 mm with a constant force of 5 N. Each sample's apparent coefficient of friction (COF) was calculated. Using an FEI Quanta 200 3D microscope equipped with scanning electron microscopy (SEM), the scratch morphology was analysed.

2.3. Tensile Test

The mechanical properties were evaluated by conducting a tensile test on the experimental specimens. The present investigation aimed to assess the mechanical properties of the Mg-Ca-Zn specimens. To this end, the specimens were subjected to testing using an INSTRON 34SC-5 universal testing machine, which has a maximum load capacity of 5 KN. The experimental protocol involved presenting each specimen to a strain rate of 0.5 mm/min until failure. The mechanical properties under consideration, including Yield Tensile Strength and Ultimate Tensile Strength, were evaluated via tensile testing conducted in accordance with ASTM B557-15 [38,39].

3. RESULTS AND DISCUSSION

3.1. Light Microscopy

Figure 2 (a-c) shows the microstructure of the experimental alloys by optical microscopy. In the structure of the magnesium alloy, the existence of α-Mg type magnesium grains, the separation at the boundary of the magnesium grains and the lamellar-shaped distribution of the Mg_2Ca phase are observed. A higher percentage of zinc led to the formation of a structure with globular grains (Figure 2c). These microstructural studies confirm the SEM images performed in [37], where the Mg grain refinement is significantly visible.

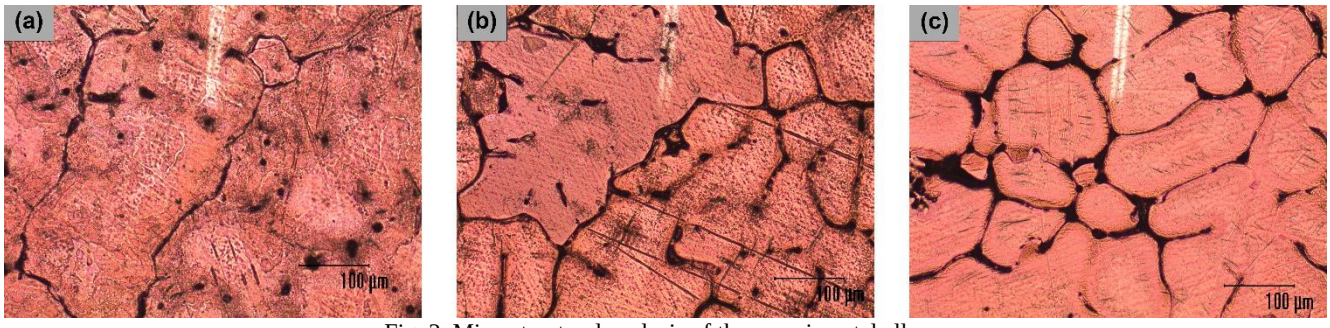

Fig. 2. Microstructural analysis of the experimental alloys.

Zinc is an important alloying element with solubility in Mg and the mechanical properties of the alloy are given by the strength of the formed solid solution. From the point of view of the studies done, the fracture resistance and elongation of cast Mg-Ca-Zn alloys increase significantly with the introduction of up to 3% of Zn. Literature studies [30, 34] on Mg-based biodegradable alloys with a Zn concentration between 1 wt.% and 3 wt.% indicate that Zn seems to have a high influence on grain size and shape.

3.2. Microindentation test & Scratch analysis

To determine the Young's modulus, a set of three microindentation tests were conducted on the surface of every alloy sample. The results of the measurements of the alloy's modulus of elasticity, hardness, and stiffness are displayed in Figure 3. Following examination of the mechanical properties, an increase of approximately 3% in Young's modulus was detected as the Zn content increased from 0.5wt.% to 3wt.%. In the same consideration,

an increase in hardness by 6% is identified. These characteristics are inversely proportional to the stiffness which has a decreasing character from 4.053 N/µm to 3.856 N/µm (Figure 3 a,b,c). These results may be due to the formation of globular segregation-like compounds and microstructural refinement due to increasing Zn concentration.

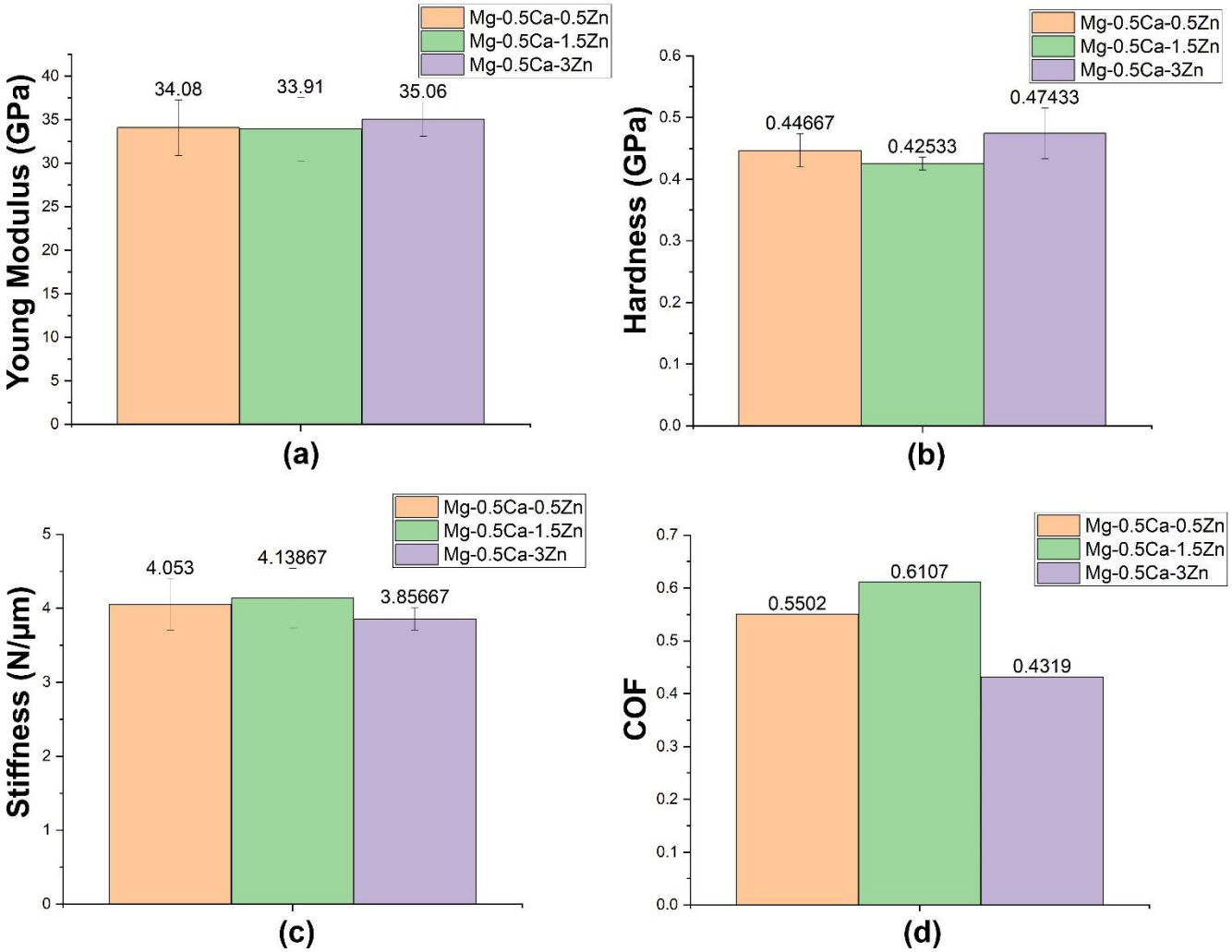

Fig. 3. Mechanical properties alloys resulting by microindentation: a) Young Modulus; b) Hardness; c) Stiffness; d) COF.

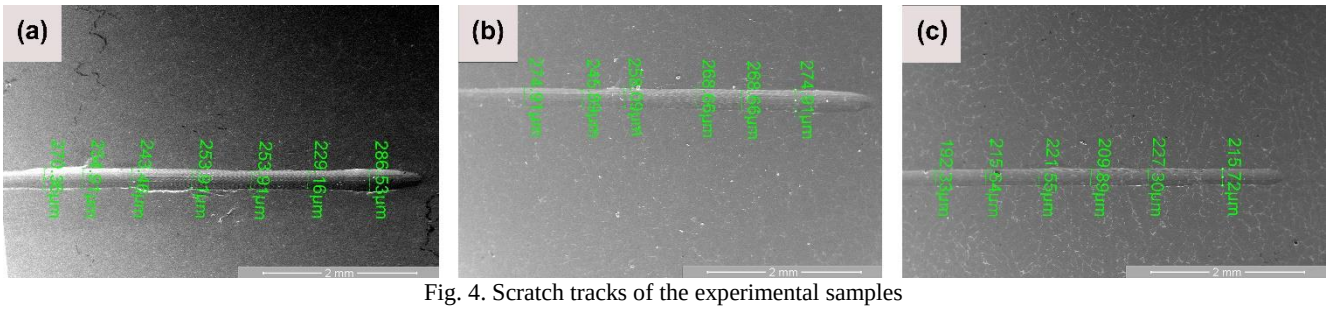

Fig. 4. Scratch tracks of the experimental samples

Figure 3d shows the values of the apparent coefficient of friction and Figure 4 shows the scratch test traces. The Mg-0.5Ca-1.5Zn alloy indicates a COF value of 0.6107, indicating a 10% and 30% increase in comparison to the COF values of Mg-0.5Ca-0.5Zn (0.5502) and Mg-0.5Ca-3Zn (0.4319), respectively. Furthermore, the mean width of the trace produced by the scratch indenter confirms this argument. The data presented in Figure 4a indicates that the average width trace of Mg-0.5Ca-0.5Zn is 253.03 µm. Similarly, Figure 4b shows that the average width trace of Mg-0.5Ca-1.5Zn is 265.18 µm, while Figure 4c indicates the average width trace of Mg-0.5Ca-3Zn is 213.72 µm.

3.3 Tensile Test

Figure 5 shows the macroscopic appearance of the fracture pattern after tensile testing. In the case of the first two specimens a relatively oblique fracture is observed, with a more pronounced aspect for the Mg-0.5Ca-0.5Zn specimen, while in the case of the Mg-0.5Ca-3Zn specimen a straight fracture is observed which evidences a more brittle behaviour. This diagonally cut cross-section stores high strain energy and this aspect highlights an elasto-plastic behavior.

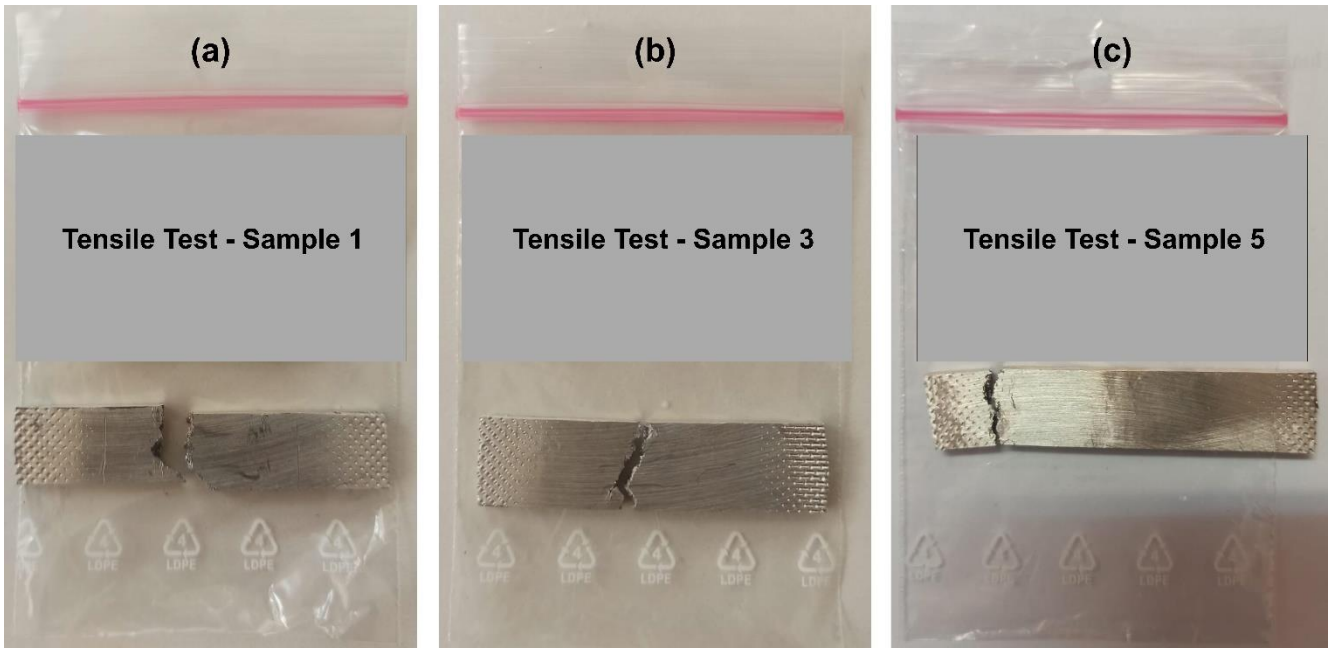

Fig. 5. The samples obtained after the tensile strength test.

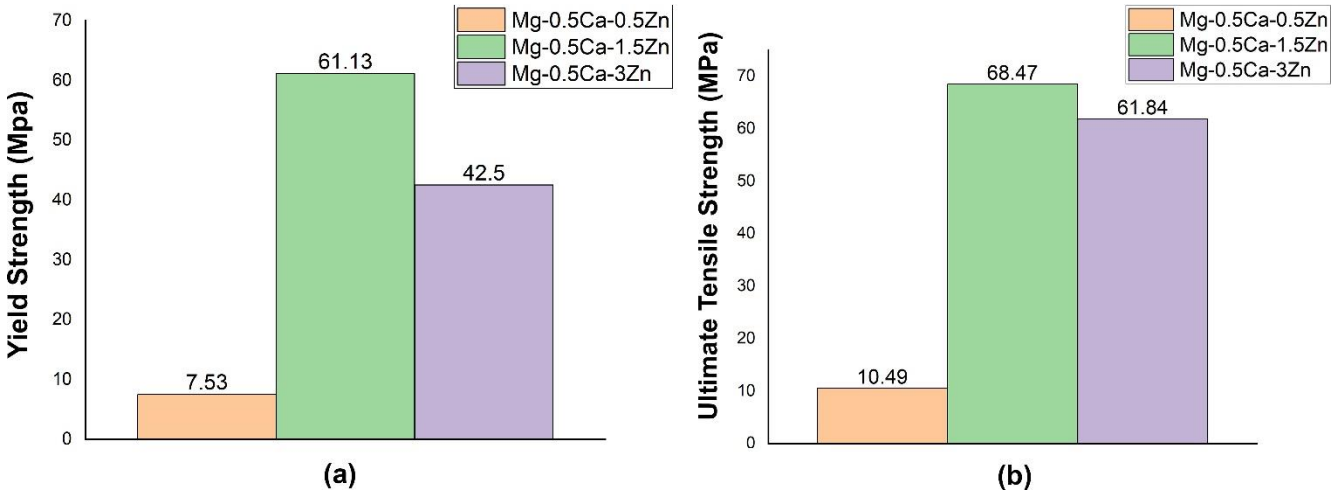

Fig. 6. Mechanical properties alloys resulting from tensile strength: a) Yield Strength; b) Ultimate Tensile Strength.

The aspect of breakage and the results obtained show a significant increase of the yield strength (YS) and ultimate tensile strength (UTS) from 0.5wt.% to 1.5wt.%, then a slight decrease for 3wt.%. The maximum values of the YS, respectively UTS are 61.13 MPa and 68.47 MPa for the alloy with 1.5wt.% Zn.

4. CONCLUSIONS

Magnesium-based alloys have numerous applications due to the properties they possess and are of high interest for the medical field due to their biodegradation characteristics. In terms of the microstructure, the Mg-Ca-Zn alloys present an α-Mg type magnesium grains, with separation at the boundary of the magnesium grains an Mg_2Ca eutectic compound and Mg-Zn globular phases. In terms of mechanical characteristics, the alloys show good mechanical resistance and the best result is the Mg-0.5Ca-1.5Zn alloy. The study revealed that the presence of Zinc significantly improves the mechanical strength of the alloy. The insertion of zinc results in an improvement of approximately 3% in the modulus of elasticity, with an increase in Zn concentration from 0.5 to 3 wt.%. In

addition, the highest YS and UTS values are 61.13 MPa and 68.47 MPa for the alloy containing 1.5% Zn. Future research will continue with studies regarding in vitro and in vivo analysis in terms of cellular viability, certifying that Mg–Ca–Zn alloys have enormous potential in the orthopedic domain.

Funding: This work was supported by a grant from the Ministry of Research, Innovation, and Digitization, CNCS—UEFISCDI, project number PN-III-P1-1.1-TE-2021-0702, within PNCDI III.

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Received: April 05, 2023 / Accepted: December 15, 2023 / Paper available online: December 20, 2023 © International Journal of Modern Manufacturing Technologies.