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# Investigation of the effect of various Ca content on microstructure and mechanical properties of as-cast ZK60 Magnesium alloys

# *Farklı oranlarda Ca katkısının döküm ZK60 Mg alaşımlarının mikroyapı ve mekanik özellikler üzerindeki etkisinin incelenmesi*

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# Investigation of the Effect of Various Ca Content on Microstructure and Mechanical Properties of As-cast ZK60 Magnesium Alloys

## *Highlights*

- *%wt. Ca addition to ZK60 Magnesium Alloy*
- *Atmosphere Controlled Low-Pressure Die Casting (LPDC) with Argon and $SF_6+CO_2$ gas mixtures*
- *Hot Tensile Testing at 345°C*
- *Mechanical Properties (Yield Strength, Ultimate Tensile Strength, Elongation, Wear Rate, Hardness)*
- *Microstructure Characterization*

## *Graphical Abstract*

*In this study, Ca added ZK60 Mg alloys were subjected to hot tensile testing for various Ca contents. Experimental results were analyzed and microstructural characterization was performed.*

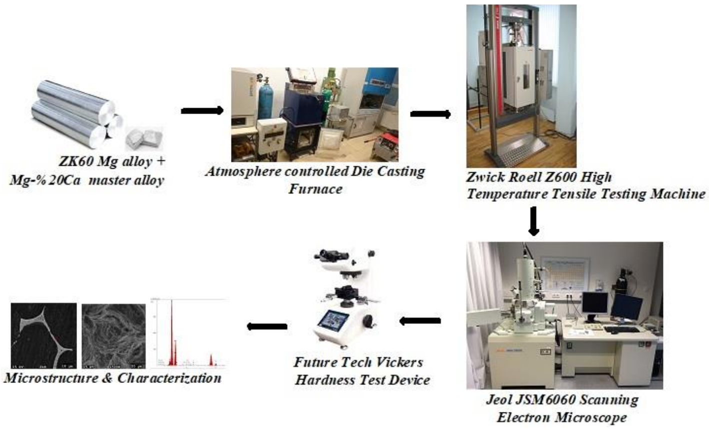

**Figure.** Experimental flow chart

## *Aim*

*The study aims to investigate the effect of various Ca (wt%) content on mechanical properties and microstructural features of ZK60 alloys at high-temperature tensile testing without applying a secondary thermomechanical process*

## *Design & Methodology*

*Hot tensile tests applied to five different %wt.Ca added ZK60 Mg alloys as-cast condition, their mechanical and wear properties were investigated.*

## *Originality*

*Unlike the literature, it is the first time hot tensile tests were applied to Ca-reinforced ZK60 Mg alloy as-cast condition without secondary TMT to characterize grain size effect on microstructure and mechanical properties.*

## *Findings*

*The yield and tensile strength have slightly changed but elongation increases to 1.26 at 345°C when wt.% Ca is 1.6 as the grain size gradually decreases.*

## *Conclusion*

*Ca added to ZK60 Mg alloys affect the microstructure and mechanical properties in such a way to increase ductility at high-temperature tensile tests contrary to decrease both yield and tensile strength in the absence of secondary TMT for further retarded dynamically recrystallized small grains.*

## *Declaration of Ethical Standards*

*The author(s) of this article declares that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.*

# Investigation of the Effect of Various Ca Content on Microstructure and Mechanical Properties of As-cast ZK60 Magnesium Alloys

***Araştırma Makalesi / Research Article***

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## ABSTRACT

The effect of various Ca content on microstructure and mechanical properties of as-cast Mg-6Zn-0.5Zr (wt.%) Mg alloys with hot tensile testing at 345°C were investigated. The alloy was produced with the LPDC method [1]. Microstructural characterization as-cast alloy demonstrated that wt.% Ca content increase, grain size decreases till wt.% 0.8Ca, the further increase affects gradual change in grain size to ~60 µm at 345°C. Therefore, increase in Ca ratio from 0.4 wt% forming to strip-like Mg-Zn intermetallics at grain boundaries, Ca-Mg-Zn ternary phase at grain boundary corners. Dramatical decrease in yield and tensile strength at wt% 1.6Ca addition is 37.6 MPa and 39.7 MPa respectively which is relatively low at 345°C due to the absence of retarded DRX'ed small particles prohibited the grain growth after the TMT process. Conversely, elongation-to-fracture increases were observed as 1.26 at 345°C showing the temperature-dependent texture softening results in improvement of mechanical properties. Wear rate is also affected from wt.% Ca addition so the gradual increase up to 1.2 wt% Ca is 3.70 x $10^{-3}$ $mm^3/m$. Similarly, a gradual increase in hardness values was obtained with Ca addition in ZK60 alloy. In this study, the effect of temperature-dependent Ca addition (wt %) on gradual enhancement in mechanical properties was observed at as-cast magnesium alloys.

**Keywords: ZK60 alloy, Ca addition, microstructure, hot tensile testing, mechanical properties.**

# Farklı Oranlarda Ca Katkısının Döküm ZK60 Mg Alaşımlarının Mikroyapı ve Mekanik Özellikler Üzerindeki Etkisinin İncelenmesi

## ÖZ

Ağırlıkça farklı Ca (%) oranlarında katılarak elde edilen olarak bilinen döküm Mg-6Zn-0.5Zr (ağ. %) ZK60 Mg alaşımının 345°C'de sıcak çekme testleriyle mikroyapı ve mekanik özellikleri incelenmiştir. Deneysel bu çalışmada, alaşım düşük basınçlı kokil döküm (LPDC) yöntemiyle üretilmiştir. Döküm alaşımının mikroyapı karakterizasyonunda ağ.% 0.8Ca oranına kadar tane boyutu küçülmüş, Ca oranı yavaş yavaş arttırıldıkça tane boyutu da 345°C sıcaklıkta yaklaşık ~60 µm boyutuna ulaştığı gözlemlenmiştir. Sonuç olarak, ağ.% 0.4Ca oranına bağlı olarak tane sınırlarında şerit benzeri Mg-Zn intermetalik yapıyı tane köşelerinde ise kabalaşmış üçlü Ca-Mg-Zn intermetalik yapısını oluştururlar. ağ.% 1.6Ca ilavesinde akma ve çekme mukavemetleri mukayese edilebilecek şekilde 345°C sıcaklıkta sırasıyla 37.6 Mpa ve 39.7 MPa değerlerine düştüğü görülmüştür. Kopma uzaması ise, bunların tam tersine sıcaklığa bağlı tekstür yumuşaması neticesi mekanik özelliklerdeki artışına bağlı olarak, ağ.% 1.6Ca oranında 1.26 % olarak ölçülmüştür. Aşınma oranı ağ.% Ca oranından etkilenmiştir ağ.% 1.2Ca oranında aşınma hızı 3.70 x$10^{-3}$ $mm^3/m$ olarak ölçülmüştür. Benzer şekilde ZK60 Magnezyum alaşımı sertlik değerlerinde de Ca içeriğine bağlı artışlar grafiklerde gösterilmiştir. Bu çalışmada döküm ZK60 magnezyum alaşımlarına % ağ. Ca katkısıyla sıcaklığa bağlı etkisinin mekanik özellikleri geliştirdiği gözlemlenmiştir.

**Anahtar Kelimeler: ZK60 alaşımı, Ca ilavesi, mikroyapı, sıcak çekme testi, mekanik özellikler.**

## 1. INTRODUCTION

Magnesium and magnesium alloys being known as the lightest materials are attracting much interest by automotive, aerospace, aviation, and defense industries because of their unique mechanical properties like being the lightest material with high specific strength and thermal conductivity together with their high dimensional stability and vibration damping, good machinability, and castability, high corrosion resistance at ambient temperature [2–6]. Besides, Mg alloys with their anisotropic hexagonal close-packed crystal (HCP) structure do not form at room temperature because of an inadequate number of independent slip systems resulting in low ductility [7]. Research showed that these eligible features of Mg alloys can not be maintained further at higher temperatures so the material undergoes mechanically creep deformation [8,9]. The addition of alloying elements like alkaline earth (Ca and Sr), rare earth (RE), and semi-metal (Si and Sb) aims to enhance the mechanical features [9–11]. Contradictory to the effect of alloying elements in developing mechanical properties, a decrease in ductility was observed. To

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overcome this dramatic drop in ductility while developing the mechanical features of the material in as-cast, it is necessary to refine grain size simultaneously [10,12–14]. By refining the grain size, it homogenizes the microstructure leading to prevention of segregation and constitution of porous structure resisting corrosion [9,10,15].

In Al-free Ca added Mg alloys, due to its grain refining effect of Zr attributes to constitute fine and densely dispersed $MgZn_2$ grains [15–17]. On the other hand, by addition of Ca into the Mg alloy with its refining effect [16] together with high heat resistance [17] and reduced flammability [18] and suitably applied thermomechanical processes the strength can be increased [19–29]. So the Ca addition to the Mg alloy results in the dynamic recrystallization and the occurrence of related grain growth to be inhibited. Therefore, the formation of finely dispersed precipitates observed at extruded material due to their pinning effect results in highly dense dislocations together with strengthening the basal texture [30]. For this reason, metallic Ca was used at Mg-Zn [26] and AM60 [32], AZ31 [37], Mg-Mn [29,38], Mg-Al [24], Mg-Sn [27] Mg alloys systems. Potentially Mg-Zn dual Mg alloy systems, with their gained high wrought strength magnesium due to solution [39] and precipitation [31] hardening mechanisms activated. Although numerous studies have worked on the effect of Ca addition on these alloy systems for investigating the properties of magnesium alloys with secondary thermomechanical treatments (TMT) so far, limited studies have focused on the effect of properties of magnesium alloy as-cast condition. To the best of the authors' knowledge, the temperature effects on grain size related to microstructures and on mechanical properties by hot tensile testing of Ca added as-cast ZK60 Mg alloys without applying secondary thermomechanical treatments have been studied for the first time in the literature. In this study, we focused on the contribution of various Ca (wt.%) content to the mechanical properties and microstructural features of ZK60 alloys at high-temperature tensile testing.

## 2. MATERIAL AND METHOD

### 2.1. Material Preparation

In this experimental study commercially supplied ZK60 Mg alloy has a composition of in weight as Mg%93.5-%6Zn-%0.45Zr. (Mg-Ca) a master alloy containing 20 wt.% metallic Ca was added proportionally with varying weight ratios, except for one as pure ZK60 for comparison donated to letters from A to E prepared for six specimens. The constituent compositions of efficient metallic wt.% Ca are given in Table 1.

**Tablo 1.** X-ray Dispersive Spectral analysis of mixed materials in the as-cast condition. Letters A to F denotes chemical compositions of ZK60 – *x*Ca (wt%) respectively.

| | Mg (wt.%) | Zn (wt.%) | Zr (wt.%) | Ca (wt.%) |
|---|---|---|---|---|
| *As-cast* | | | | |
| A | 93.00-93.50 | 5.50-6.50 | 0.40-0.50 | 0.00-0.00 |
| B | 91.50-93.50 | 6.00-7.00 | 0.30-0.40 | 0.30-0.40 |
| C | 91.50-92.50 | 6.50-7.50 | 0.40-0.50 | 0.60-0.80 |
| D | 91.00-92.00 | 6.50-7.00 | 0.50-0.60 | 1.00-1.20 |
| E | 90.00-91.50 | 6.50-7.50 | 1.30-1.40 | 1.40-1.60 |
| F | 90.00-91.50 | 6.00-7.00 | 1.50-1.60 | 1.60-1.80 |

Ingot casting was held under the controlled gas atmosphere of $SF_6$+$CO_2$ and Argon gas mixtures after the heating to 750°C then waited for 30 minutes before pouring into a steel mold. The tensile test specimens machined with wire erosion method into rectangular bars with 50 mm gauge length and 4 mm thickness were then subjected to high-temperature tensile testing according to EN ISO 6892-1 [32] standards. The specimens were subjected to 345°C at a strain rate of $10^{-4}$ $s^{-1}$ and 2 MPa pre-loaded for the high-temperature tensile test performed at Zwick Roell Z600 Hot Tensile Test Machine respectively. The stoichiometry of the prepared samples after casting is given in Table 2.

**Table 2.** The grain size of wt.% Ca added ZK60 Mg alloy samples before and after high-temperature tensile tests (*345°C*)

| | Grain Size ((µm) | |
|---|---|---|
| | Before | After |
| | *25°C* | *345°C* |
| *As-cast* | | |
| A | 98 | 136 |
| B | 88 | 79 |
| C | 78 | 61 |
| D | 95 | 81 |
| E | 67 | 64 |
| F | 74 | 60 |

The casted parts before applying high-temperature tensile tests, the metallographic studies were held with acidic picral for etching before mechanical polishing. After the completion of the process, the OM examination was carried out using Olympus BX41M-LED for grain size determination. The XRD pattern for intermetallics was obtained using Rigaku with 40 kV, 20 mA, between 20°- 90° $2\theta$ with 2° step size. After the tests, surfaces and fractured sections of the samples were characterized with Jeol JSM 6060 Scanning Electron Microscope (SEM) and the energy dispersive X-ray spectrum for detailed at.% concentration in microstructures was obtained by using EDS analysis equipped. Their hardnesses were measured on Futura Tech Vickers Hardness (HV) test machine. The wear tests were performed for as-cast hot tensile tested samples at a reciprocating wear test machine. The sliding distance proposed was 250 m in dry sliding conditions under the 5N applied load for 150 rpm sliding speed. DIN 100Cr6 steel ball was used as counterface grinding material. To compare the grain size of the Ca added samples, as-cast conditions to the machined counterparts exposed to high-temperature tensile tests were then tabulated in Table 2. The grain sizes were determined by the linear intercept method under the optical microscope. The strain rate was kept constant for all selected temperatures to observe the effect of grain size on mechanical properties. In this experiment, Yield strength ($Rp_{0,2}$), Tensile Elongation ($A_g$) in Mpa, Elongation (%), Wear Rate ($mm^3/m$), Vickers Hardnesses (HV), Grain Sizes (µm) were comparably examined.

Figure 2 illustrates the XRD profiles of the as-cast ZK-*x*Ca alloys. ZK60 alloy profiles show α-Mg and Mg-Zn binary phase systems. Whereas the maximum solid solubility of Zn in Mg solid solution to form an Mg-Zn binary compound is 6.2 wt.%, according to the Mg-Zn phase diagrams, 50 wt.% of solid constituent occurs at 615°C for 6 wt.% Zn [34] due to inconsistency in solidification during casting.

XRD pattern of the produced from ZK-*x*Ca alloys reveals at all the solidified Mg-Zn alloys have changed their XRD pattern gradually decrease in Zn percentage with their corresponding intensities changed in depending on reversibly increase in Ca content.

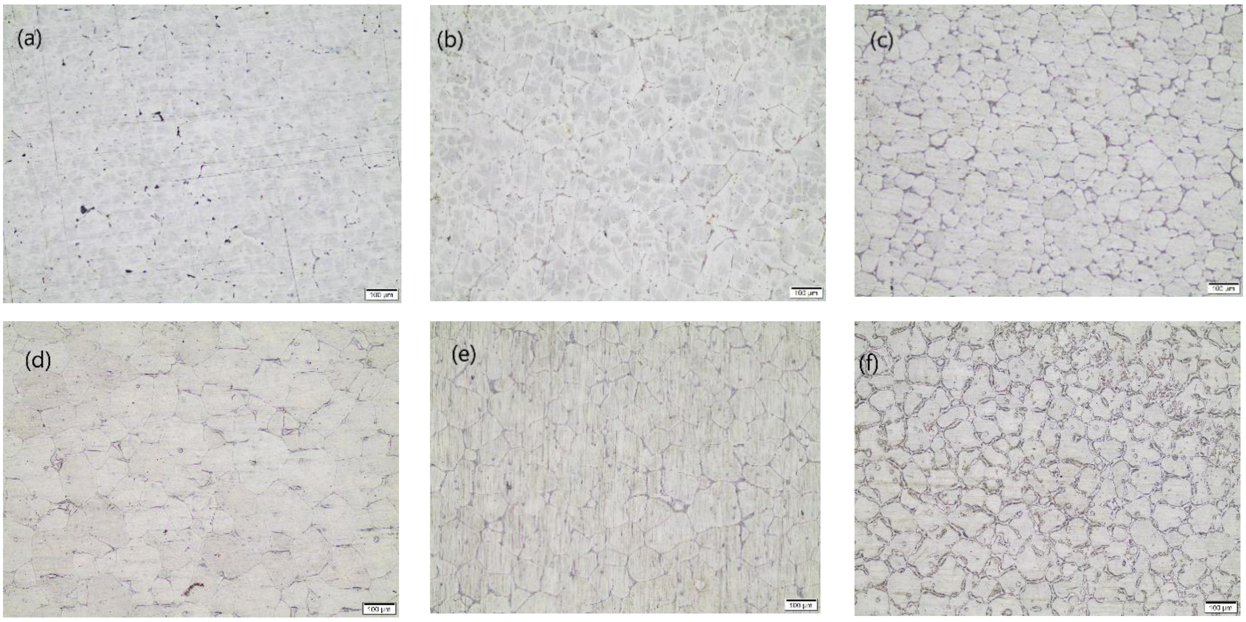

**Figure 1** Letters A-F represent grain sizes of the wt.% Ca added ZK60 magnesium as-cast shown in **Table 1** respectively.

## 3. RESULTS AND DISCUSSION

### 3.1. Microstructure of Cast Alloys

The measured as-cast Ca free ZK60 alloy (Figure 1-a) the grains with equiaxed shape relatively coarse about ~98 µm in size. And the grain size decreases parallel to increases in Ca wt.% till the maximum solute solubility of Ca at (Figure 1-d) and coarsens further depending on the Ca ratio. Figure 2. illustrates the XRD profiles of the as-cast ZK60 - *x*Ca alloys. ZK60 alloy profiles show α-Mg and Mg-Zn binary phase systems. Whereas the maximum solid solubility of Zn in Mg solid solution to form an Mg-Zn binary compound is at 6.2 wt.% [33], according to the Mg-Zn phase diagrams, 50 wt.% of solid constituents occurs at 615°C for 6 wt.% Zn. Corresponding XRD profiles of the Ca added magnesium alloys are demonstrated in Figure 3. The graphs verify that all the solidified Mg-Zn alloys have drawn the same XRD profiles, even though, their corresponding peaks vary with the Zn content.

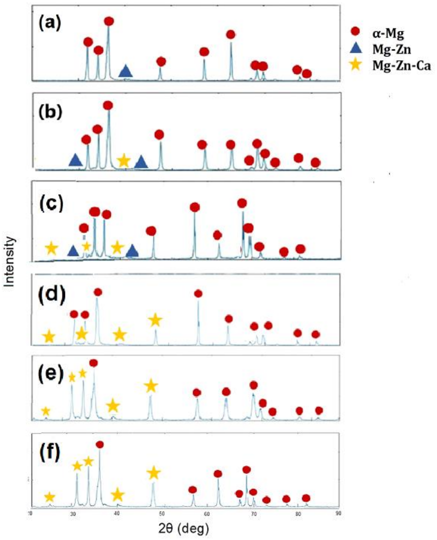

**Figure 2** XRD patterns of the ZK60 –*x*Ca alloys: (a) ZK60, (b) ZK60-0.4Ca, (c) ZK60-0.8Ca, (d) ZK60-1.0Ca, (e) ZK60-1.2Ca and (f) ZK60 -1.6Ca.

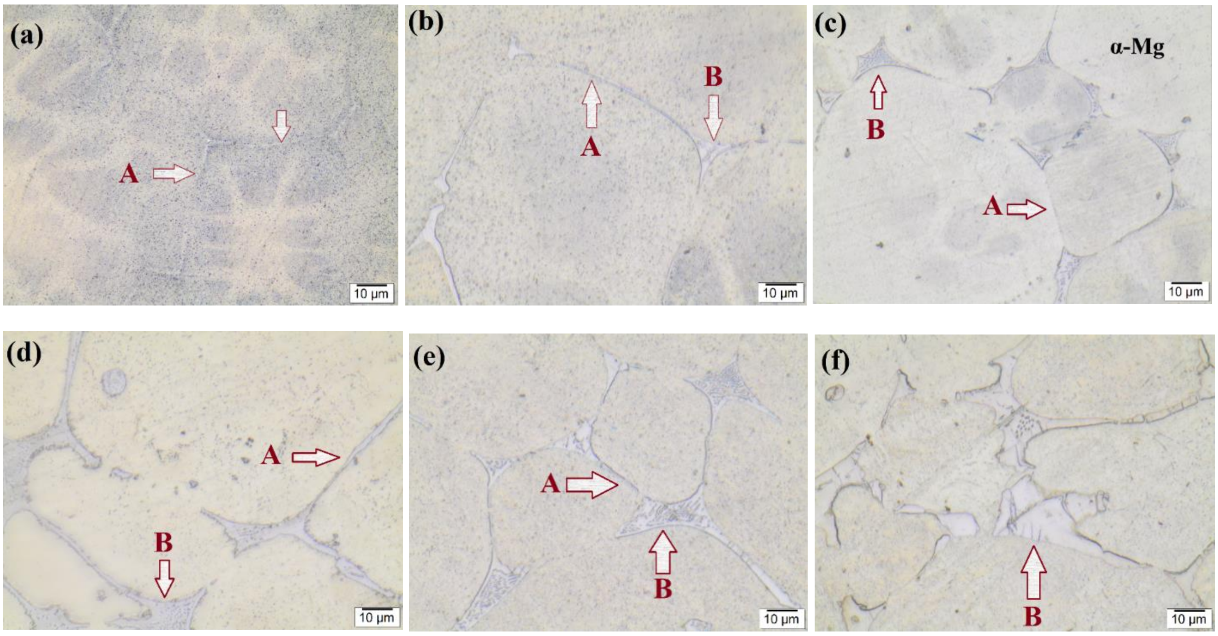

**Figure 3** OM microstructures of as-cast ZK60-*x*Ca alloy at 345°C where A denotes binary Mg-Zn and B for Mg-Zn-Ca ternary intermetallic phases at TD.

The emerging peaks measured in between 25° and 48° after Ca addition assigning to Mg-Zn-Ca ternary alloys given in (Figure 2. b-f). The growing peaks show that the increasing amount of Ca addition has developed to a considerable amount of Mg-Zn-Ca ternary phases. To understand the microstructural characteristics of the ZK60-*x*Ca alloy, OM and SEM studies were performed for this alloy.

Figure 3 demonstrates light microscope pictures of the ZK60-*x*Ca alloys at the tensile direction (TD) at 345°C. It is proved that α-Mg grain size gradually decreases concerning an increase in wt.% Ca addition due to the effect of Ca atom enrichment at the solid-liquid interface during solidification. Depending on the rise up in Ca content of the alloy deteriorates the binary Mg-Zn intermetallics at grain boundaries denoted as A Figure 3 (a-f) reversibly concentrate the ternary Mg-Zn-Ca phase at grain corners denoted as B. As it can be understood the effect of Ca addition given in Figure 4 demonstrates that the grain sizes decrease as the wt.% Ca content increases as-cast ZK60-*x*Ca, Mg weight in the alloy contrary to the increase in volume ratio in ternary intermetallic phases in the microstructure. Binary Mg-Zn strip-like interphases diminish and eventually interrupt between grain boundaries meanwhile increasing in concentrate at grain corners, that is, irregular globular-like in shape in Figure 3. The EDS analyses Figure 5 (a-b) for wt.% 1.6 Ca clearly show that the as-cast ZK60 alloy composition for intermetallic phases at 2kX magnification is mainly from a high concentration of Zn and Ca and irreversibly dropping concentration of Mg due to preferential formation of ternary intermetallic phases which is also given in Table 3 the values in at. %.

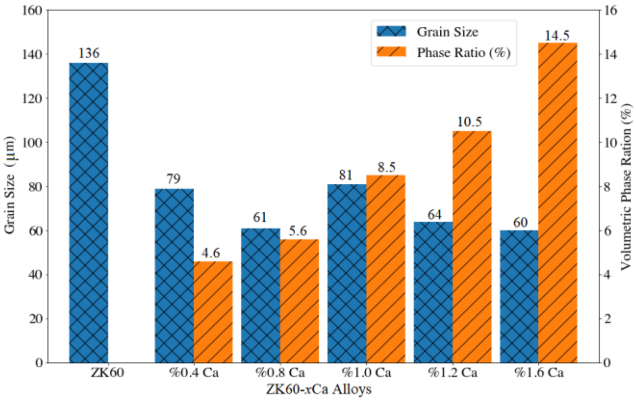

**Figure 4** Grain Size (μm) and Volumetric Phase Ratio (%) of as-cast ZK60-*x*Ca alloy at 345°C

On the other hand, in absence of the homogenization process, Zr in Mg-Zr rich region during the solidification process affects thermal stability enhanced by the presence of Zr even after solidification.

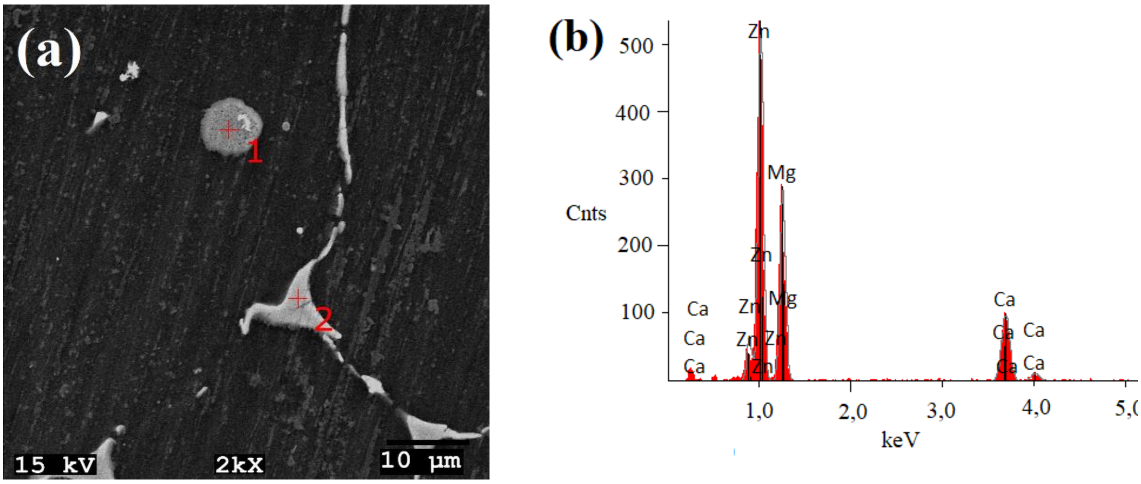

**Figure 5** SEM-EDS analysis of TD surface of ZK-1.6Ca alloy at 345°C

**Table 3** at.% values of respective elements forming intermetallic phases at SEM-EDS analysis

| Point | Mg<br>at.% | Ca<br>at.% | Zn<br>at.% |
|---|---|---|---|
| 1 | 71,60 | 11,50 | 16,90 |
| 2 | 54,40 | 16,35 | 29,25 |

This showed that the grain size as-cast condition decreased till to wt.% 0.8Ca which is measured as ~61 µm then further decreased prior to a slight increase from wt.% 1.0Ca till to wt.% 1.6Ca the grain size is ~60 µm irreversibly continuous increase in the second phase volume ratio to ~14.5. Therefore, without applying any secondary high-temperature Thermo Mechanical Treatments (TMT) processes like extrusion instead, increase in addition of wt.% Ca in ZK60 alloy by itself can not be adequate to drop the grain size although an increase in their volume ratios respectively.

Figure 6 shows tensile characteristics of as-cast ZK60 alloy at various wt.% Ca addition. Comparably, measured yield and tensile strength from unalloyed sample toward wt.% Ca alloyed samples demonstrated the slight decrease in strength values for example yield strength for an unalloyed sample is 45.7 MPa, it is only 37.6 MPa for wt.% 1.6Ca. İt is evident for cast magnesium alloy, further increase in Ca weight ratio does not bring any dramatic drop at both yield and ultimate tensile strength besides elongation, which increases parallel to increase in wt.% Ca content that is 0.43 for unalloyed ZK60, 1.26 for wt.% 1.6Ca respectively.

Therefore, as it is seen only slight change in yield and ultimate strength together with elongation only wt.% 0.4Ca addition to ZK60 alloy.

This phenomenon can be explained by Hall-Petch theory [12,35,36] where grain size decreases yield strength increase due to dislocation piles-up at grain boundaries.

In this study, the grains as-cast conditions after hot tensile testing, are relatively coarse compared to the exposure TMT process where the grain sizes are quite small. The small grains refer to many grain boundaries which hinder dislocation movement eventually causes yield strength increase. So the lack of a secondary TMT process in this regard prevents the occurrence of retarded DRX where the fine and the dense Mg-Zn-Ca intermetallics act as pins to the moving dislocations.

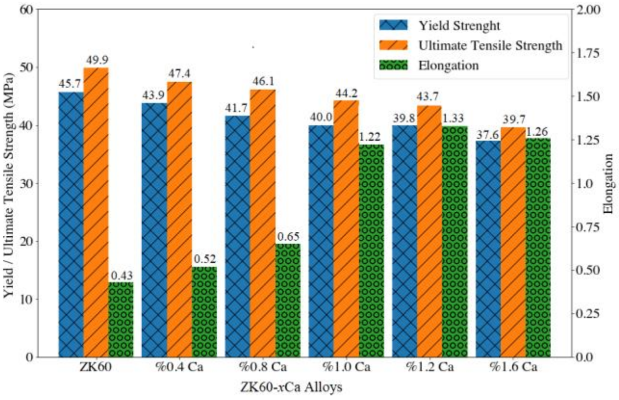

**Figure 6** Tensile characteristics of as-cast ZK60-*x*Ca alloys at 345°C.

Referring to Figure 6 after a wt.% 1.0Ca content the elongation drastically increased to double although the slight change in yield and tensile strength values. Coarse grains as-cast state affect the microstructure of the alloy showing elongation-to-fracture where the increase in % Ca addition, the grains slightly get smaller and grain boundaries disappear which means secondary phases stick on fractured grain surfaces given in Figure 7 SEM micrographs at 345°C. Therefore, depending on the increased Ca content among the stuck grains extends Figure 7(a-c) the fracture-to-elongation retards so the cracked surface of the grains topographically more sharpen and leaving residual second phase on these cracked surfaces Figure 7(d-f). Coarsened grains with second phases might have propagated around the particles affecting to decrease not only in yield strength but also in tensile strength while increasing the elongation because of the positive effect of Ca on ductility enhancement. It is also seen Cleavages and sharp edges on the fractured surface as-cast magnesium alloys refer to low ductility. It is evident that the higher amount of broken second phase particles indicates the premature fracture during hot tensile testing where the plastic deformation is predominant thus, irregularly shaped dimples together with localized but sharp edges are proof at Figure 7 (a-d) ZK60-*x*Ca alloy fractographs. Figure 8 demonstrates the Vickers Hardness (HV) values for respective unalloyed and alloyed ZK60 alloys as-cast after hot tensile testing at 345°C for average figures of 9 measurements for each samples shows Ca addition does not bring understandably effect on hardness values.

Because the Ca in microstructure refines the matrix and increase the ductility, therefore, the hardnesses at all Ca concentration are almost the same only, slight increases are observed depending on the increase in Ca content.

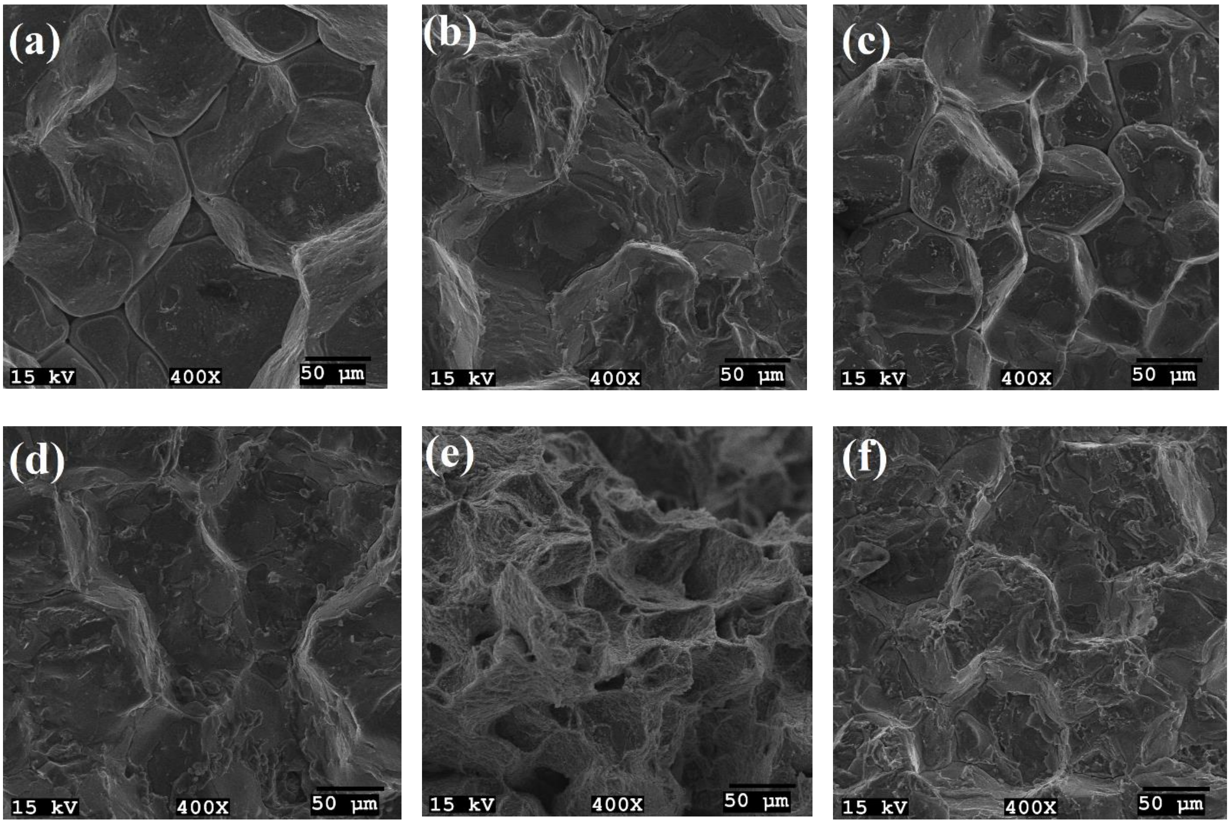

**Figure 7** SEM microstructure of fractured surface as-cast ZK60-*x*Ca alloy from (a) to (e) at 345°C.

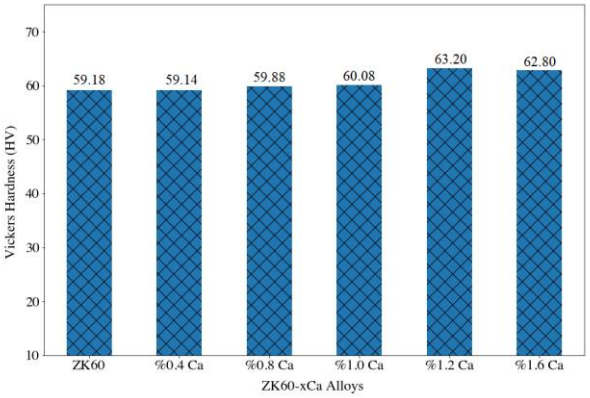

**Figure 8** Vicker Hardness Values of as-cast ZK60-*x*Ca Alloy at 345°C.

The wear rates applied as-cast samples after hot tensile testing at 345°C are demonstrated in Figure 9 under 5 N load as per sliding distance of 250 m for ZK60-*x*Ca alloy. The graphs show that an increase in Ca concentration for respective samples leads to a gradual increase in wear rate. This is attributed to the presence of second phase particles which leads to Ca-containing ZK60 alloys being prohibited to dislocation movement. From the graph further increase in Ca to %1.6 dropped to wear rate ağain. From here, it can be deduced that Ca added to the material primarily make it soft and ductile but the presence of second phase particles increases wear rate to some extent. Secondary TMT process, if applied, it is expected to wear surfaces with grooves, stratch prints, and sticking materials through sliding direction at SEM microstructure characterization. Besides existing worn surfaces due to adhesion of material caused by material and applied load. It can also be seen adhesion and oxidations due to the repeated cycle of frictional load on the materials.

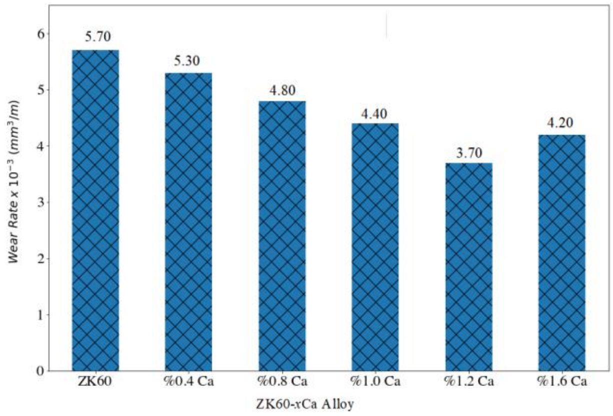

**Figure 9** Wear rate values of as-cast ZK60-*x*Ca alloys at 345°C.

## 4. CONCLUSION

In this study, ZK60 magnesium alloy with different wt.% Ca addition was cast with the Low Pressure Die Casting method under the controlled gas atmosphere condition. Differing from the literature, alloys were subjected to hot tensile testing without applying any further secondary TMT process. The results of microstructure and texture, tensile, hardness, and wear properties were tabulated for as-cast Ca added ZK60 alloys. The following conclusions can be deduced:

- Different ratios Ca addition resulted in the formation of strip-like secondary Mg-Zn intermetallics between grain boundaries, globular, ternary Mg-Zn-Ca phase formation at grain corners.

- As the Ca content increased, secondary strip-like Mg-Zn phase slightly disappeared and divorced to ternary Mg-Zn-Ca phases at grain corners.
- Grain size drastically decreased after wt% 0.4Ca addition and further Ca addition slightly changes the grain size irreversibly increase in volume ratio to 14.5% because of the presence of second phase ternary Mg-Zn-Ca intermetallics.

- Tensile properties ZK60 alloy were enhanced by increasing the Ca ratio in as-cast state, %21 for yield strength, %18 for ultimate tensile strength respectively. On the contrary, elongation-to-fracture was almost three times increased to wt.% 1.6Ca added ZK60 alloy.

- Coarsened grains with second phases might have propagated around the particles affecting to decrease not in yield strength but also in tensile strength while increasing the elongation because of the positive effect of Ca on ductility enhancement.

- Wear rate results of as-cast ZK60-*x*Ca alloys at 345°C indicate that increasing addition of Ca up to 1.2 wt.% affects positively so the wear rate increase but further addition oppositely deteriorates the wear rate.

- Vickers Hardness (HV) values for all Ca added ZK60 Mg alloys after hot tensile testing at 345°C demonstrates that depending on the increase in Ca content up to 1.2 wt.% so the hardnesses increase, over this point, exceeding to 1.6 wt.% Ca content, then the alloy showed the adverse effect on hardness.

- For the future direction, the weldability and corrosion properties of Ca added ZK60 Magnesium alloys can be investigated.

## ACKNOWLEDGEMENT

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## DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission

## AUTHORS' CONTRIBUTIONS

**Aykan AKBAŞ:** Performed the experiments and analyzed the results. Conducted the analysis and evaluated the results. Also, wrote the manuscript.

**Muzaffer ZEREN:** Performed the experiments and analyzed the results. Conducted the analysis and evaluated the results.

## CONFLICT OF INTEREST

There is no conflict of interest in this study.

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