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The Influence of Heat Treatments of Sustainable Zinc on Microstructure and Mechanical Properties

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Zinc is important in many applications due to its resistance to corrosion under different conditions; it is used in galvanization. This article presents a study of zinc from remelting waste zinc (waste collected from manufacturing scrap; galvanizing zinc) and recycled by sand casting, then heat treated under different conditions. Impact and hardness testing ascertained the mechanical properties of the zinc casting, while optical microscopy characterized its microstructure. The study found that the grain growth increased after natural aging and reached a maximum of approximately 41 % of the treatment at 350 °C, for 3 h followed by water quenching, which exhibited the largest grain size, indicating significant grain growth. In contrast, the treatment at 350 °C, for 5 h followed by water quenching refined the grain structure and reduced the precipitation fraction, suggesting a more stable microstructure. The as-cast sample exhibited the highest hardness (approximately 94 HV), whereas heat treatment initially reduced hardness because of microstructural contrast dissolution during solution treatment. Among the heat-treated specimens, the treatment at 350 °C, for 5 h followed by water quenching, produced the highest hardness (approximately 82 HV), indicating an effective balance between precipitation strengthening and grain refinement. Specimens showed intermediate hardness values in the heat treatment at 370 °C for 3 h and 5 h, then aged at 180 °C for 90 min, which are attributed to grain growth during aging. The results demonstrate that heat treatment significantly affects the precipitation behavior, grain evolution, and hardness of high-purity zinc, with the treatment at 370 °C, for 5 h followed by water quenching providing the most favorable combination of microstructural stability and mechanical performance. The impact strength was strongly influenced by the heat treatment condition, with the heat treatment at 370 °C for 5 h, then aged at 180 °C for 90 min achieving the highest value of 5 J/cm², comparable to commercial ZP3 (Zamak 3) and higher than ZP5 (Zamak 5).
Keywords: microstructures, water quenching, heat treatments, zinc, mechanical properties.

1. INTRODUCTION

Zinc and its alloys have numerous industrial uses, including the automotive, aerospace, and electronics industries. This is because of their exceptional qualities, which include strength, low density, and castability. These alloys can undergo additional optimization through heat treatment. Materials can be heat-treated to change their mechanical and microstructural characteristics by carefully heating and cooling them. By altering the size, shape, and distribution of the phases within the alloy during heat treatment, zinc-based alloys can have their mechanical properties improved. Mechanical and microstructural analyses of zinc-based alloys following heat treatment have been conducted. Heat treatment reduces the strength and hardness of ZA-27 alloys while increasing their elongation. The duration of solutionizing affects these changes. Castings benefit from heat treatment in terms of tribological properties. The tribological behavior of a heat-treated sample produced from a steel mold is better than that of a sand-cast sample. According to Yang et al., the heat treatment of the ZA27 alloy was examined using XRD, SEM, and mechanical characterization. The first sample's microstructure was made up of α that had broken down into various phases. The α and η phases disintegrated at 365 °C for an hour, leaving a primarily supersaturated β microstructure behind. Afterward, the phases decomposed [2].

Heat treatment of zinc-based alloys can be used to accomplish a number of goals. With an emphasis on this relationship, this study focuses on how differences in microstructure affect the mechanical properties of zinc.

Immersion and electrochemical methods are used to test the corrosion resistance of heat-treated samples at 37 °C. Zhou et al. (2010) studied the AZ91D magnesium alloy in order to create a biocompatible and biodegradable implant. T4 microstructure corrosion speeds up due to the dissolution of the Mg17Al12 phase. Intergranular corrosion is also promoted by fine precipitates of Mg17Al12 within T6, albeit at a lower rate than for the T4 microstructure as cast [3].

A Zn-based alloy has been developed by Dambatta et al. (2014) as a potential biodegradable implant material. Various defects are often associated with the casting process for this biomaterial. Hence, Zn-3Mg alloy was cast conventionally and homogenized for 10 hours at 370 °C to enhance microstructure uniformity. According to the microscopic analysis, the Zn-3Mg alloy exhibits a star-like dendritic structure with a eutectic mixture of Mg_2Zn_{11} phases. Heat treatment disperses this segregation, resulting in a Zn-3Mg alloy with a more uniform microstructure and fewer casting defects [4].

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Zn-15 wt.%Al alloys subjected to heat treatment were examined by Movahedi et al. 2019. An annealing temperature below 275 °C results in a homogeneous distribution of granular particles. As the annealing temperature increases, the strength of the alloys increases as well. However, when the alloys are annealed above 275 °C and then water-quenched, their strength initially decreases with aging time and then stabilizes. The γ' phase gradually decomposes into an equiaxed microstructure during aging, causing a reduction in strength. Transmission electron microscopy (TEM) studies suggest grain boundary sliding and rotation dominate deformation in water-quenched alloys after severe plastic deformation. On the other hand, dislocation tangles are common in the lamellae of air-cooled alloys, suggesting that dislocation slip, along with grain boundary sliding and rotation, contributes to deformation. Using phase transformation dynamics and the law of mixtures, a strength model is developed for water-quenched alloys annealed above 275 °C. Moreover, the number of α particles in the γ'phase is considered a key parameter for understanding the evolution of mechanical properties during heat treatments of Zn-15 wt.%Al alloys. When the density of particles is higher in the 'phase, the alloys' plastic deformation is easier, their strength is lower, and their elongation is greater [5].

A study by Silva et al. 2021 examined the microstructural evolution of Zn-1 wt.% Mg-(0.5 wt.% Ca, 0.5 wt.% Mn) alloys, which exhibit common intermetallic compounds including $Zn_{11}Mg_2$ and Zn_2Mg in a eutectic alloy. In comparison, Zn 1 wt.% Mg alloy microstructure is finer than Ca and Mn alloys. Hardness tests demonstrate increased hardness due to microstructural refinement and specific IMCs due to correlations between cooling rate and dendritic or eutectic spacing. Biodegradable Zn-based alloys could be improved by understanding alloy formation and improving mechanical properties [6].

Maree 2021 examined the microstructure and damping behavior of ZA-27 alloy using a dynamic mechanical analyzer. Solid solution treatment of ZA-27 alloys at 120 °C, 240 °C, and 360 °C for an hour was carried out on the fabricated alloys. According to their findings, every phase in the as-cast microstructure of ZA-27 alloy decomposes at 360 ºC. In a temperature range from room temperature to 300 °C, the dynamic mechanical properties of the as-cast alloy and heat-treated alloy were studied. The specific damping capacity and loss modulus of heat-treated alloys were higher than those of as-cast alloys. Furthermore, as temperature increased, the storage modulus decreased [7]. The purpose of this work is to examine how heat treatment affects the microstructure of zinc, assess how heat treatment affects the mechanical characteristics of hardness and impact of zinc, and determine the ideal heat treatment parameters for producing a desired microstructure in zinc.

Zinc alloys have attracted growing scientific interest over the past decade due to their unique combination of microstructural characteristics and mechanical behavior. The properties of zinc alloys are highly dependent on manufacturing conditions, such an alloying additions and thermal effects [8]. The heat treatment in particular, T4 and T6 treatments have been widely reviewed in zinc and its alloys because they can considerably effect on phase, hardness and strength [9]. In biodegradable zinc alloys, the effect of secondary phases is critical, as they strongly affect degradation behavior and corrosion rate [10, 11], zinc alloy suitable biocompatibility for medical applications [12]. In orthopedic applications, zinc alloy fixation screws have demonstrated antibacterial effects and reduced osteolytic activity, making them attractive for bone repair strategies [13]. However, several challenges remain. Controlling degradation rate and ensuring long-term mechanical reliability are still major issues that limit clinical translation [14, 15].

Surface modification processes have been broadly examined as a solution to these limitations. Different coatings and treatments have been observed to improve both biological behavior and corrosion resistance of biodegradable zinc alloys [16 – 18]. Interestingly, even coarse-grained zinc alloys have the ability to achieve improved mechanical characteristic [19].

Several studies have investigated the properties, degradation, and applications of zinc-based materials [20-22]. In biomedical applications, zinc alloys have also become evident in energy storage, mainly aqueous zinc-ion batteries, due to their safety and low cost. Some research focuses on developing adaptable zinc-alloy anodes capable of working under different current densities [23].

Electrodeposited zinc alloys have been proposed as an effective approach to enhance battery stability, suppress dendrite formation, and extend service life [24]. In addition, copper-zinc alloy layers have been stated to provide ultra-stable zinc metal anodes with improved electrochemical performance [25]. The modified zinc alloys show promising behavior, where spontaneous grain refinement notably improves cycling durability [26]. Zinc alloys use in both biomedical and industrial environments; their degradation mechanisms have been widely studied. Zinc and zinc-alloy coatings have demonstrated good corrosion resistance, particularly in marine conditions [27]. Finally, time-dependent corrosion behavior in simulated body fluid has been investigated using electrochemical techniques and surface characterization methods. These studies provide valuable insight into degradation mechanisms under physiological conditions [28 – 30]. Zinc poses some mechanical issues in the use of the metal for biomedical implants, leading to research on techniques like casting, plastic forming, and additive manufacturing. Mechanical processing is important in transforming the microstructure, which in turn affects the creep resistance and toughness of zinc [31 – 37].

2. EXPERIMENTAL

2.1. Materials

Waste zinc metal collected from manufacturing scrap that it was used in galvanization for corrosion protection.

2.2. Apparatus and instruments

Create a magnified image using an optical microscope for samples of zinc casting before and after heat treatment condition by grinding and polishing, then etching with an etchant solution for inspecting microstructure: 200 g CrO_3, 15 g Na_2SO_4, 1000 ml H_2O. Electric furnace with a maximum temperature 1000 °C.

2.3. Casting in sand mold

The mold was made for 6 samples of zinc by sand casting in these steps: making a suitable system of opening holes in the sand mold, in order to pour the melted zinc waste and allow the gases to escape, where the sand mold dimensions are 15 mm diameter and 150 mm height; melting the metal with a temperature (450 – 500 ºC) and then pouring the molten metal into the mold cavity; remove the sample from the mold; leave the metal to cool; break the sand mold and remove the cast metal samples.

2.4. The heat treatment of zinc casting

The solution treatment is the first step in the precipitation-hardening process, in which metal and alloy are heated above their solvus temperature and soaked there until a homogeneous solid solution is obtained. Natural aging at 150 ºC over 30 days at room temperature; the heat treatment at 370 °C for 5 h and 3 h, then the samples were aged at 180 °C for 90 min; the heat treatment at 370 °C for 5 h and 3 h, followed by water quenching for all samples.

2.5. Preparation of samples for microscopic inspection

Samples prepared for microscopic examination are generally mechanically prepared. Cutting, grinding, and polishing follow embedding. In order to remove the material, successively smaller abrasive particles are used. 180, 500, 800, and 1200 grit sandpaper is used to remove saw marks, level, and clean the surface of the specimen. Polishing removes the artifacts of grinding and removes very little material, and the velocity of the grinding machine is 321 – 421 rpm to grind the surface, alternative fine grinding by polishing using standard sized aluminum oxide powders and resilient cloths is used [38]. The etching solution was prepared using 15 g of Na_2SO_4 sodium sulfate, and 200 g of chromium oxide, CrO_3. It was prepared in the laboratory with as little contact with the materials as much as possible to avoid risks the materials solution.

3. RESULTS AND DISCUSSION

The chemical composition by spectro analyzer of zinc casting is shown in Table 1, after melting and pouring in a sand cast.

Table 1. Zinc casting components inspection by spectro, (Ametk, Materials spectrometer analyzer, Germany made)

Al, %	Cu,%	Fe, %	Pb, %	Ag, %	Cd, %	Zn, %
0.033	0.065	<0.0030	<0.0060	<0.0005	<0.0010	99.9
<0.0050	0.067	<0.0030	<0.0060	<0.0005	<0.0010	99.9

According to the measured chemical composition in Table 1, it can be noted that Zn content is approximately 99.9 wt.%, so the material can be classified as commercial high-grade zinc, which is close to the EN 1179 Zn99.9 grade and ASTM B6 high-grade zinc.

The hardness test results by Vickers hardness measure before and after heat treatment shown in Fig. 1, The heat treatment of zinc casting influences microstructure changes, decreasing Vickers hardness.

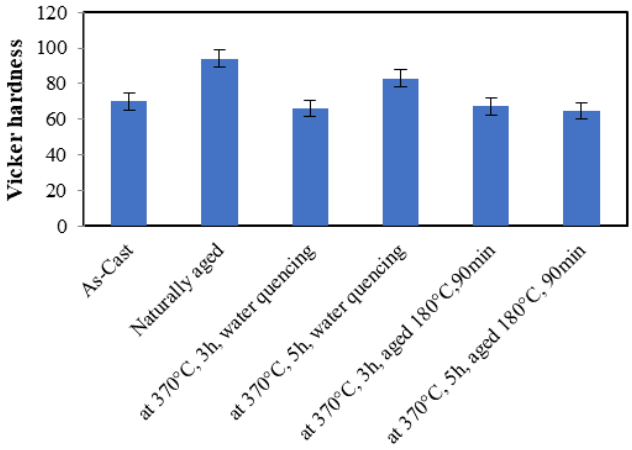


Fig. 1. Average Vickers hardness

The microstructure of the zinc casting changed from a dendritic structure as in Fig. 2 to beginning grain growth nature ageing and of treatment at 370 °C for 5 h and 3 h as shown in Fig. 3, Fig. 4 and Fig. 5.

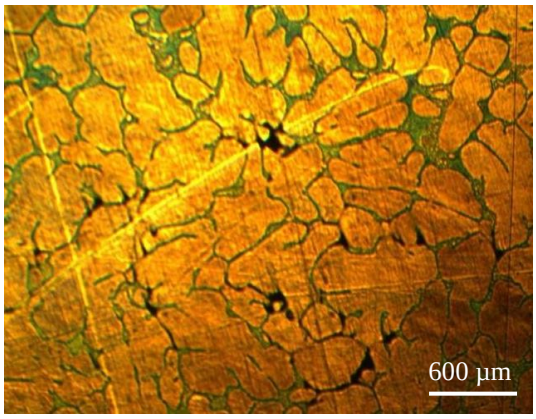


Fig. 2. The microstructure of zinc: as cast

The microstructure of the zinc casting changed from a dendritic structure as in Fig. 2 to a globular structure after the heat treatment of 370 °C at 3 h and 5 h, then aged at 180 °C for 90 min as shown in Fig. 6 and Fig. 7, heat treatment, resulting in improved ductility and toughness. The hardness and impact strength of the zinc casting also decreased after heat treatment due to the reduction in dislocation density and the refinement of the microstructure.

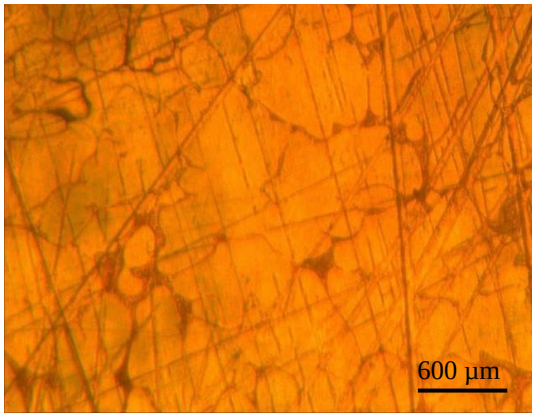


Fig. 3. The microstructure of zinc: naturally aged

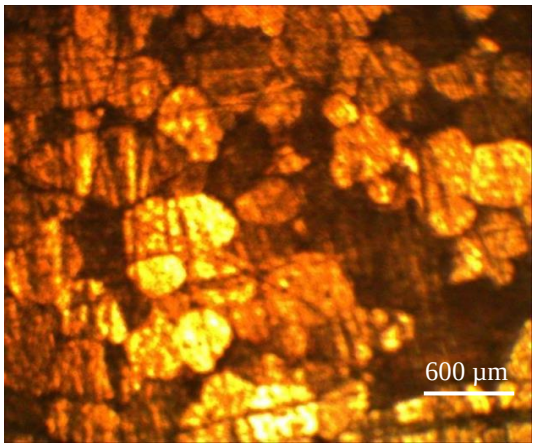


Fig. 4. The microstructure of zinc after the treatment at 370 °C, for 5 h followed by water quenching

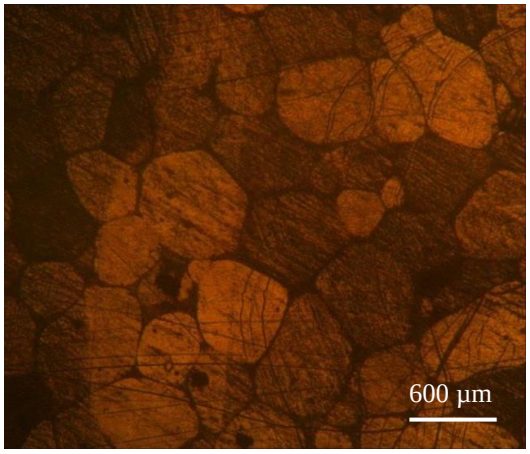


Fig. 5. The microstructure of zinc after the treatment at 370 °C, for 3 h followed by water quenching

The MATLAB R2018a analysis in Fig. 8 a shows the effects of heat treatment on the microstructure contrast area and mean grain size. The area increases from approximately 36 % in the as-cast condition to a maximum of about 41 % after the treatment at 350 °C, for 3 h followed by water quenching, then decreases with prolonged aging, reaching its lowest value (~32 %) for the treated at 370 °C, 5 h followed by water quenching. The MATLAB R2018a analysis in Fig. 8 b, shows the mean grain area development with different heat treatments.

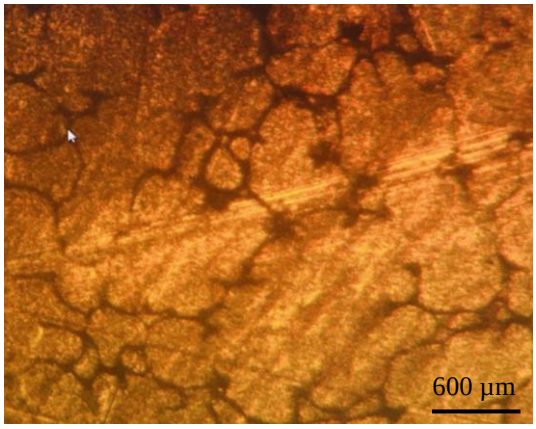


Fig. 6. The microstructure of zinc after the treatment at 370 °C for 5 h, then aged at 180 °C for 90 min

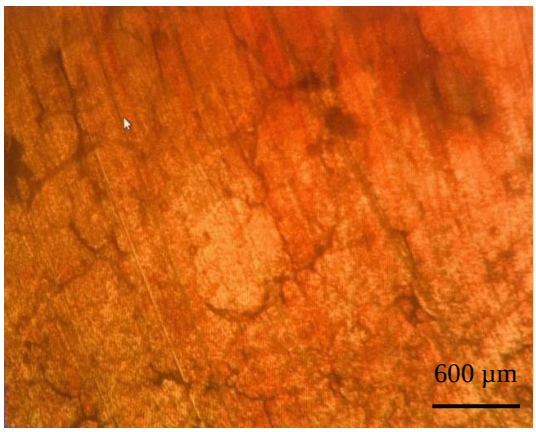


Fig. 7. The microstructure of zinc after the treatment at 370 °C for 3 h, then aged at 180 °C for 90 min

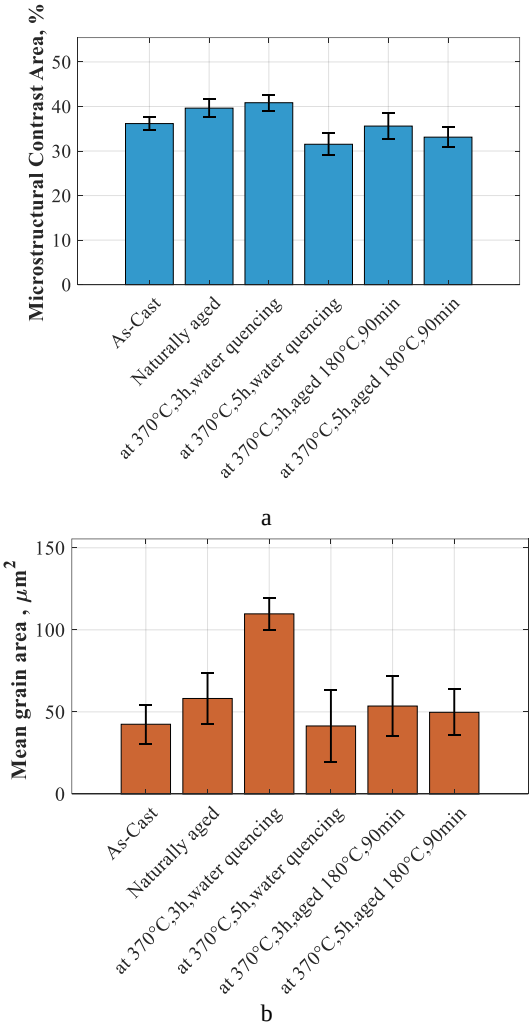


Fig. 8. Analysis of microstructures results: a – quantitative contrast area fraction; b – evaluation of mean grain size evolution across different heat treatments

Microstructure analysis shows specific stages of grain growth, refining and stabilization according to different processing conditions. In the as-cast condition, the baseline mean grain area is found to be equal to approximately 42 µm^2. The increase of mean grain area is seen after natural aging to about 58 µm^2 because of solute atom agglomeration in the early stage of the natural aging process. Grain growth becomes more significant during annealing of the material

at 370 °C for 3 h; when the mean grain area achieves the maximum value of 110 μm^2. It is believed that such a high value is caused by grain growth due to thermal activation, which results in higher grain boundary mobility at 370 °C and reduces the total grain boundary energy. Prolonging of annealing at 370 °C up to 5 h causes a sharp decrease in mean grain area to 41 μm^2. This means that grain refinement in these conditions is caused by dynamic or static recrystallization, when new, strain-free grains are nucleated and replace old coarsened grains. Subsequently, aging at 180 °C for 90 min stabilizes the microstructure. Aging causes the mean grain area to refine and stabilize at a value.

Typical Charpy impact test specimens measure 55 mm × 10 mm × 10 mm, with a notch as shown in Fig. 9. An impact load is applied to a standard notched specimen to measure its energy absorption. When the specimen is held securely at each end, it is struck with a hammer on a pendulum arm. Nocks are struck opposite the hammer. By measuring the decrease in motion of the pendulum arm, you can determine how much energy is absorbed by a specimen. Stress concentrators such as the collapse of a material and high strain rates affect its toughness [39].

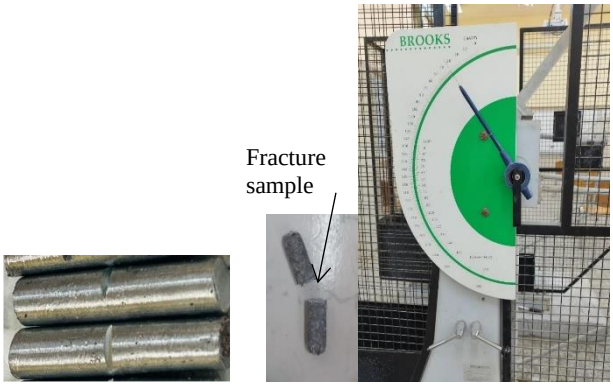


Fig. 9. Charpy impact test: a – Charpy impact sample; b – impact devise

In the impact test the samples were broken, and the impact energy was determined for each sample at different heat treatments as shown in Table 2. The T6 heat treatment for 5 h provided the highest impact strength (5 J/cm²) among the tested heat treatment conditions, indicating that prolonged artificial aging improves the alloy's ability to absorb impact energy.

Table 2. Impact strength (Charpy notched at room temperature 28 °C)

Condition of heat treatments	Impact strength, J/cm^2
Naturally aged	3
The heat treatment at 370°C for 3 h, then aged at 180°C for 90 min	3
The heat treatment at 370°C for 5 h, then aged at 180°C for 90 min	5
The treatment at 3 h, 370°C	4
The treatment at 5 h, 370°C	3
ZP3Alloy 3, Zamak3, ZP0400, ZnAL4 [40]	5

Natural aging and T6 treatment for 3 h both exhibited the lowest impact strength (3 J/cm²), suggesting that insufficient aging time does not effectively enhance toughness. The treatment at 370 °C, for 5 h followed by water quenching achieved a moderate improvement (4 J/cm²), while extending the treatment to 5 h reduced the impact strength back to 3 J/cm², indicating that excessive aging under the treatment of 370 °C conditions may reduce toughness. Compared with commercial zinc alloys, the heat treatment at 370 °C for 5 h, then aged at 180 °C for 90 min treated alloy exhibited an impact strength equal to ZP3 (Zamak 3) (5 J/cm²) and higher than ZP5 (Zamak 5) (4 J/cm²).

This heat treatment for 5 h is the optimum condition among those investigated, providing the best balance of impact resistance and performance relative to standard commercial Zamak alloys.

According to Table 3, heat treatment reduces zinc casting impact strength depending on its process and parameters. It is possible for brittle phases to form during the treatment or for large, coarse grains to form, thus reducing the material's impact resistance. To determine the specific impact strength changes, zinc casting must undergo experiments and be analyzed for its microstructure and mechanical properties. These methods include impact testing, microscopy, and other relevant characterization techniques.

4. CONCLUSIONS

Zinc casting microstructure and mechanical properties (hardness and impact strength) are significantly impacted by heat treatment in this study, the heat treatment conditions significantly influenced on the grain size of the zinc alloy. The treatment of 370 °C treatment for 3 h produced the highest microstructure contrast area fraction (≈ 41 %), indicating distinct microstructural contrast evolution during artificial aging. However, this condition also resulted in the largest mean grain size, suggesting considerable grain growth. In contrast, the treatment of 370 °C treatment for 5 h reduced both the precipitation area fraction and grain size, indicating that prolonged aging promoted microstructural stabilization and suppressed excessive grain growth. The T6 treatments exhibited intermediate behavior, with moderate precipitation fractions and grain sizes, the treatment at 370 °C, for 5 h condition provided the finest grain structure while maintaining an acceptable grain boundary.

Heat treatment had a pronounced effect on the hardness of the investigated zinc. The as-cast condition exhibited the highest hardness due to the presence of the original solidification microstructure. The treatment at 370 °C, for 3 h caused a marked reduction in hardness, while extending the treatment to 5 h partially recovered the hardness through precipitation hardening. In contrast, the T6-treated samples exhibited lower hardness than the treatment at 370 °C, for 5 h condition, indicating that prolonged aging promoted grain growth and reduced the strengthening effect. Among the heat-treated samples, this provided the highest hardness (≈ 82 HV), representing the optimum balance between precipitation strengthening and microstructural evolution.

The impact strength was significantly affected by the heat treatment condition. The highest value (5 J/cm²) was obtained after T6 heat treatment for 5 h, matching the impact strength of ZP3 (Zamak 3) and exceeding that of ZP5

(Zamak 5). In contrast, natural aging, T6-3 h), and the treatment at 370 °C, for 5 h exhibited the lowest impact strength (3 J/cm²). These results indicate that T6-5 h provides the best impact performance among the investigated conditions.

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