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Analysis of Mechanical Properties of Zn-0.5Fe-0.5Ag Alloy for Body Absorbed Implant Applications

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Abstract

Implants that can be absorbed by the body may be developed using alloy materials based on zinc (Zn), iron (Fe), copper (Cu), and silver (Ag). Zn-based alloys are known for their faster biodegradation rates, making them particularly suitable for biodegradable implant applications. The aim of this study is to determine the corrosion rate and hardness of Zn-0.5Fe-0.5Ag alloy specimens, as well as to examine the effects of heat treatment and the addition of Fe and Ag elements. The research utilizes a direct experimental observation method to analyze the mechanical properties of the Zn-0.5Fe-0.5Ag alloy specimens. In pure Zn, the largest grain size is observed in specimens without annealing treatment, while the smallest grain size is found in specimens annealed at 350°C. For the Zn-0.5Fe-0.5Ag alloy, the smallest grain size is observed in specimens annealed at 400°C, while the largest grain size appears in those annealed at 350°C. The addition of Fe and Ag to pure Zn significantly increases the hardness, with the hardness value rising from 33.77 HV (pure Zn) to 61.64 HV. In terms of corrosion, the highest corrosion rate in pure Zn was found in specimens without annealing. In contrast, the highest corrosion rate in the Zn-0.5Fe-0.5Ag alloy was observed in specimens annealed at 400°C. In conclusion, the addition of Fe and Ag elements, along with heat treatment, significantly affects the mechanical properties of the Zn-0.5Fe-0.5Ag alloy, improving both its hardness and influencing its corrosion behavior.

Keywords: Mechanical Properties, Zn-0.5Fe-0.5 Ag Alloy, Implants, Corrosion Rate, Hardness.

1. Introduction

Over the past four decades, significant advancements in biomaterials and medical technology have garnered considerable attention due to their potential to enhance the quality of human life. These innovations aim to replace or repair both soft and hard tissues, including bones, cartilage, blood vessels, and even entire organs of the human body [1], [2]. Metals, in particular, have played a vital role in this progress and have been widely utilized as orthopedic implants, cardiovascular intervention devices, and scaffolds in tissue engineering due to their excellent mechanical strength and durability [3], [4]. Traditional metallic biomaterials such as titanium alloys, stainless steel, and cobalt-chromium alloys are known for their superior corrosion resistance and have been commonly employed as permanent implants in clinical practice [5].

However, implants that are permanently present can potentially cause negative impacts on the organs of the body. For example, metal ions can detach from the implant due to defects in the oxide layer of its surface, which can eventually cause the implant to fracture. In other situations, the chronic inflammatory response to the implant can impair its therapeutic function. In these circumstances, additional surgery is often required to remove the implant, which can result in injury and additional costs [6], [7].

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Technological developments in the world of implants, the field of orthopedics has developed pen materials that can decompose in the body after reassembly, so there is no need to perform a second surgery to remove the implant. One of the biomaterials that is often used in the health field is metals. Biodegradable metals have several advantages, as the porous structure can allow the growth of new bone tissue and the implant itself to gradually decompose over its lifetime, with decomposition products absorbed or removed from the human body [8]. At the same time, new bone tissue grows and replaces the implant, thus achieving the goal of complete treatment. The use of biodegradable metals can also eliminate the need for follow-up surgery to remove the implant once the bone tissue has healed properly [8].

Implants that are absorbed in the body can be developed with zinc (Zn), iron (Fe), copper (Cu), silver (Ag) based alloy materials. Zn-based alloys have faster biodegradation rates, making them suitable for biodegradable implant applications [9]. Zn is an important element in the human body and plays several bio functional roles in human physiology, such as participation in nucleic acid metabolism, transmission signals, and the reaction of many organic ligands [10], [11]. Zn has significant advantages in biodegradable vascular scaffolding applications due to its effective anti-atherosclerotic properties [12]. In addition, Zn has good biocompatibility and contributes to bone healing [13]. Therefore, Zn-based alloys are currently being intensively explored for vascular implant and stent applications.

Zn alloys also show poor mechanical properties. However, if Zn is alloyed with iron (Fe) and silver (Ag) alloy elements, it will produce an alloy that has good mechanical properties and can be used as an absorbable implant [14]–[16].

In this study, antibacterial metals such as Zn, Fe, and Ag will be combined to produce a new biodegradable alloy when viewed from the mechanical properties, biodegradability, biocompatibility and antibacterial properties. In this study, the microstructure, hardness and corrosion rate of the alloy will be reviewed. The metal alloy that will become an implant absorbed by the body is expected to solve the problem of infection that often occurs during and after implantation.

2. Method

The research method used is a method of direct experimental observation. This research method was used to analyze the characteristics of the mechanical properties of the alloy Zn-0.5Fe-0.5Ag, namely to determine whether there is a difference in mechanical properties due to the addition of Fe and Ag to the alloy. The trick is to test the mechanical properties of Zn-0.5Fe-0.5Ag alloys using corrosion rate testing and hardness testing with the microvickers method on both alloys. Then the microstructure observation of the alloy was carried out using an optical microscope (Figure 1).



Figure 1. a) Micro Vicker Hardness Tester, b) Nabertherm Furnace, c) Metrohm I Stat 8,400

2.1 Testing Procedure

2.1.1 Sample Preparations

The procedure for conducting research on Zn-0.5Fe-0.5Ag alloy for the application of bodyabsorbed implants is to prepare research tools and materials. Prepare the specimen used, which is an alloy of Zn-0.5Fe-0.5Ag. The specimen which is in the form of as-cast alloy Zn-0.5Fe-0.5Ag is cut into diameter sizes of 20 mm and thickness of 5 mm. Annealing measured by temperature variations of 350°C and 400°C with a holding time of 8 hours. The annealing process uses a nabertherm furnace. Specimens are cooled in a heating furnace [17]. Specimen preparation is carried out using tools nabertherm furnace, microstructure sampling using optical microscope, hardness testing using microvickers and corrosion rate using metrohm I stat 8,400 devices.

2.1.2 Process Annealing

Cut the specimen that was originally in the form of bars and rods into a predetermined shape and size, then clean the sample from the remains of the cut using a grinding machine with sandpaper roughness from 320 to 1500 sizes. Make sure the surface is completely flat and smooth, then place the sample in a special container to be put into the heating furnace. Set the temperature on the heating furnace, in this test the temperature is used 350°C and 400°C, then adjust the flow of argon gas that will be flowed into the heating furnace, then set it to the automatic setting of the argon gas valve opener at the temperature of 350°C and 400°C and hold it for 8 hours, after that lock the argon gas valve let the specimen cool itself in the furnace. Once cooled, carefully remove the sample using tongs and gloves and place the sample in the place provided.

2.1.3 Optical Microscope Testing

Prepare the specimen that has been annealed, then the specimen is placed on the resin mold that has been prepared, then the resin liquid that has been mixed with the hardener is poured into the mold in which the specimen is located, leave until the resin adheres and hardens to the specimen aforementioned. After it is confirmed that the resin has hardened, then the specimen is removed from the mold. Then the top and sides are leveled using a grinding machine, then flattened on the surface of the specimen that will be observed for microstructure using a sandpaper grid of size 320 to 1500 with the help of water during the grinding process. Furthermore, the specimen is cleaned using running water and alcohol, then dry the specimen using a hair dryer, after drying it continues the polishing process using polishing paper and given additional auto sol. After the polishing process is considered sufficient, the specimen is cleaned again using running water and alcohol and then dried again using a hair dryer. Prepare the etching solution (5% HNO₃/Alcohol solution) in the container that has been prepared. Then apply the etching solution using cotton buds to the specimen (in accordance with the ASTM E 407-99 standard). After you see a change in color from the surface of the specimen, rinse with running water and clean it with a clean cloth and then clean again with alcohol evenly. Next, the specimen is dried again using a hair dryer. Once dry, place the specimen under an optical microscope. Observe on the monitor screen and change the microscope magnification lens brightness to get the desired image result. After obtaining the desired particle image, the image will be processed using software on an optical microscope so that the required information is obtained, namely the grain size of the particle.

2.1.4 Corrosion Testing

Corrosion Rate Testing on Zn-0.5Fe-0.5Ag alloy by potentiodynamic method. Polarization testing was performed in the Hanks balanced saline solution media. The stat potency uses a three-electrode system that can adjust its potential so that the relationship curve between the potential (E) and the current (V) is obtained, namely the table curve. After the curve is obtained, the I_{corr} and E_{corr} values are searched to be included in the calculation to find the corrosion rate according to (ASTM vol 03.02.G02) with the corrosion rate formula:

$$CPR = \frac{I_{corr}KE_{W}}{A\rho}$$
(1)

Where *CPR* is the Corrosion rate (mm/yr), I_{corr} is the corrosion current (amp), E_w is the equivalent weight (gr), ρ is the density (gr/cm³), A is the area (cm²), K is the constant (3272/(amp.cm.yr).

2.1.5 Hardness Testing

Testing of the hardness value of Zn-0.5Fe-0.5Ag alloy test specimen using a material hardness test tool using the microvickers hardness test method (Future-tech FM800, Japan). This hardness test was performed at 3 different location points on each specimen with a load of 500gf and a containment time of 15 seconds in accordance with ASTM E384-17 [18] guidelines which are also in accordance with the research of [19]. The average value of hardness is obtained using the following equation:

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$$HV = \frac{(1.8544)P}{d^2}$$
(2)

Where P is the indenteror load (kg) and d is the diagonal length of the compressive trace load (mm).

3. Result and Discussion

From the series of experiments that have been carried out, the Zn-0.5Fe-0.5Ag alloy specimens are annealed with the temperature used, namely at 350°C and 400°C. In this annealing process, a hold time of 8 hours is applied. From the specimens that have been prepared, several tests were carried out and several data were obtained, and the data in question include: grain size of Zn-0.5Fe-0.5Ag alloy particles observed through an optical microscope, followed by hardness testing using the hardness microvickers test, and corrosion rate testing using the potentiodynamic method using the method I stat 8400 tool using HBSS (Hanks Balanced Salt Solution) as a medium for simulating body fluid.

3.1 Results of Microstructure Tests with Optical Microscope

Figure 2 shows the composition of the Zn-0.5Fe-0.5Ag alloy. Based on the test results, the results are shown in Table 1.



Figure 2. Alloy Composition Zn-0.5Fe-0.5Ag

Before microstructure observation, the Zn-0.5Fe-0.5Ag sample was first observed in its composition which can be seen in Table 1. This test aims to determine the composition of the content contained in the Zn-0.5Fe-0.5Ag alloy which aims to ensure whether the alloy casting process is successful or not.

Table 1. Al	loy Comp	osition Zn-0	.5Fe-0.5Ag
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Element	Mass concentration %
Oxygen	5.57
Zinc	89.12
Iron	1.93
Silver	0.60

In this test, prior to observation of Zn-0.5Fe-0.5Ag alloys, the specimen must be prepared according to the procedure described. This observation uses a Zeiss microscope with the use of a 100x magnification lens. In Figure 3, you can see the difference in the microstructure of pure Zn alloy with Zn-0.5Fe-0.5Ag alloy. This can be caused because in figure b there is the addition of Fe and Ag elements to the alloy.

In this observation, Figure 3 parts a, c and e are pure Zn specimens and in parts b, d, and f are Zn-0.5Fe-0.5Ag alloy specimens. It can be seen from the image that there is a microstructural difference

between pure Zn specimens and Zn-0.5Fe-0.5Ag alloy specimens. This can be caused by the addition of Fe and Ag elements to pure Zn specimens. In addition, changes in the shape of the microstructure can be caused by the annealing effect given to each specimen, both pure Zn specimens and Zn-0.5Fe-0.5Ag alloys. In this study, the annealing temperature used was 350°C and 400°C with a holding time of 8 hours.



Figure 3. a) Pure Zn Alloy, b) Zn-0.5Fe-0.5Ag As-Cast Alloy, c) Zn Alloy Pure Annealing 350°C, d) Zn-0.5Fe-0.5Ag Alloy Annealing 350°C, e) Zn Alloy Pure Annealing 400°C, f) Zn-0.5Fe-0.5Ag Alloy Annealing 400°C

In the Table 2, it can be seen in the pure Zn section, the size of the grain size pure Zn particles without treatment annealing has a size grain size largest while the size of the grain size the smallest is located in the specimen pure Zn treatment annealing 350°C. In the Zn-0.5Fe-0.5Ag alloy, it can be seen

Table 2. Grain Size Data			
Sample	Annealing Temperature (°C)	Item size (µm)	
	-	33.6	
Pure Zn	350	3.5	
	400	14.1	
As cast Zn-0,5Fe-0,5Ag	-	16.8	
	350	33.6	
	400	5.0	

from the table that the size grain size the smallest particles present in the treatment annealing at 400°C and grain size large particles found in the annealing at 350°C.

3.2 Hardness Test Results using the Vickers Hardness Method

This test used a load suppression of 500 gram-force and a holding time of 15 seconds. In the hardness result data, it can be seen that the annealing temperature treatment has a great impact on the hardness of the specimen. This can be due to changes in the shape and size of particles contained in the specimen resulting from annealing treatment at 350°C and 400°C which is hold for 8 hours. Figure 4 shows a graph of the average hardness values of pure Zn and the Zn-0.5Fe-0.5Ag alloy. The left-hand graph shows the average hardness values of pure Zn. The right-hand graph shows the average hardness values of the Zn-0.5Fe-0.5Ag alloy.



Figure 4. Average Hardness Value of Pure Zn and Zn-0.5Fe-0.5Ag Alloy

In this study, pure Zn was used as a reference for the hardness value of the specimen before adding elements Fe and Ag. The addition of Fe and Ag elements to pure Zn alloys has a significant impact on the hardness price of specimens that initially pure Zn had a hardness of 33.77 HV to 61.64 HV. This indicates that the addition of Fe and Ag elements can improve the mechanical properties of pure Zn alloys.

In another study, it was explained that the hardness of pure Zn without the addition of other elements had a value of 33HV. The results of this study can be used as a reference for values as a benchmark for Zn values in ongoing research. In another study, adding the element Ag to the Zn alloy can increase the mechanical strength which was initially 36.6 HV increased to 78.6 HV. In another study, the addition of Fe elements with different compositions increased the hardness value from 41 HV to 53 HV [14]. In another study, adding 1.4% of the element Fe made the hardness of the Zn alloy to 56 HV [20].

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In another study using Zn-0.5Mg-0.2Fe samples with treatment heat treatment at 350°C there was an increase in hardness to 156 HV and in the same study as Zn-0.5Mg-0.2Ag samples with the treatment heat treatment at the same temperature is applied an increase in hardness to 110 HV [21].

From several tests that have been presented as a reference in this test, the addition of Fe and Ag elements can improve the mechanical properties of Zn alloys so that this study can be said to be in line with the various references used as a reference in this study.

3.3 Corrosion Rate Testing

Corrosion test is a measuring device to test a metal object with the aim of finding out certain characteristics of a substance that can affect the resistance of the metal itself from the consequences of corrosion indications.



Figure 5. Potentiodynamic Polarizion Curve Pure Zn

Figure 5 is the result of the potentiodynamic polarization curves of pure Zn at normal temperature (green), pure Zn at 350°C (red) and pure Zn at 400°C (black). In the curve of the table above, it can be seen that the green curve indicates that pure Zn at normal temperature has a corrosion rate value of 0.02061 mm/y, and in the red curve which indicates pure Zn that has gone through the annealing stage at a temperature of 350°C has a corrosion rate of 0.00874 mm/y and the black curve which indicates that pure Zn that has gone through the annealing stage at a temperature of 400°C has a corrosion rate of 0.00267 mm/y.



Figure 6. Potentiodynamic Polarization Curves Alloy Zn-0.5Fe-0.5Ag

Figure 6 shows the results of the potentiodynamic polarization curves of as-cast Zn-0.5Fe-0.5Ag (red), Zn-0.5Fe-0.5Ag annealing 350°C (green) and Zn-0.5Fe-0.5Ag annealing 400°C (blue). In the

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curve above, the as-cast potentiodynamic results were obtained which were marked by a red curve with a velocity of 0.04597 mm/y. In Zn-0.5Fe-0.5Ag alloy annealing at a temperature of 350°C marked with a green curve, the corrosion rate was 0.03916 mm/y and Zn-0.5Fe-0.5Ag annealing alloy 400°C marked with a blue curve obtained a corrosion rate of 0.09237 mm/y.

The above results are the corrosion test output of pure Zn alloy and Zn-0.5Fe-0.5Ag alloy which are annealed at normal temperatures, 350°C and 400°C, respectively. All specimens were soaked in HBSS solution for 15 minutes until the system reached stability. After the steady current is polarized, polarization is carried out by scanning 0.035 v/s. Electrochemical Impedance Spectroscopy (EIS) analysis with a voltage of -0.8V to 0.8V. The results were analyzed with metrohm dropview 8400 software and obtained a corrosion rate of pure Zn as-cast of 0.02061 mm/y, 350°C pure annealing Zn of 0.00874 mm/y and 400°C pure annealing Zn of 0.00267 mm/y. In as-cast alloys Zn-0.5Fe-0.5Ag of 0.04567 mm/y, Zn-0.5Fe-0.5Ag annealing 350°C of 0.03916 mm/y and Zn-0.5Fe-0.5Ag of 0.09237 mm/y. So in this test, 2 conclusions were obtained, namely in pure Zn alloys, the greatest corrosion rate is located in pure Zn specimens that are given annealing at 400°C. Another conclusion is that the addition of Fe and Ag elements to pure Zn has the effect, namely accelerating the corrosion rate in the alloy.

In the corrosion rate test in this study, pure Zn samples were used as a reference for initial values before the addition of Fe and Ag elements by the potentiodynamic method. In this test, a voltage of -0.8 V to 0.8V with a scan rate of 0.035v/s was used. It can be seen from the Table 3 data that pure Zn alloy has a significant difference in its corrosion rate value compared to Zn alloys that have been added with Fe and Ag elements.

In the study conducted by Yilmazer with pure Zn alloy, the corrosion rate was obtained as 0.054 mm/y. heat treatment with a temperature of 350°C. The results of this study can be used as a comparison of the value of the corrosion rate of Zn alloys that have been added to the composition of Fe and Ag [22].

In another study conducted by Riaz et al. [21], which used sample variations, namely Zn-0.5Mg-0.2Fe and Zn-0.5Mg-0.2Ag with the treatment of heat treatment at a temperature of 350°C, the results of the corrosion rate were obtained of 0.0114 mm/y and 0.0113 mm/y, respectively. In this study, it also presented a Zn-0.5Mg sample as a reference value before adding Fe and Ag elements and obtained a lower result of 0.0049 mm/y [21].

Sample	Temperature (°C)	$E_{corr}\left(\mathrm{V}\right)$	I_{corr} (μ A)	Corrosion Rate (mmpy)
	-	-1.273	8.993	0.02061
Pure Zn	350	-1.265	5.447	0.00874
	400	-1.259	1.666	0.00267
	-	-1.441	22.500	0.04597
As cast Zn-0.5Fe-0.5Ag	350	-1.354	19.166	0.03916
	400	-1.366	45.206	0.09237

Table 3. Corrosion Rate Data

4. Conclusion

Based on the results of data analysis and discussion, it can be concluded that the conclusions obtained from this study are by adding Fe and Ag elements and annealing treatment on alloys that have just passed the casting stage (as-cast) to cause changes in almost every aspect of microstructure, hardness value and corrosion rate. In hardness testing with the addition of Fe and Ag elements to pure Zn can significantly increase the corrosion price and in corrosion rate testing carried out by adding Fe and Ag elements, heat treatment can also result in changes in all aspects. In this study, a variety of heat treatment annealing methods were used with normal temperatures, 350°C and 400°C. In this study, the optimal recommended temperature is at 350°C because annealing at this temperature provides good data results and is close to the various references used. And it can be concluded that the As cast alloy Zn-0.5Fe-0.5Ag can be used

as an initial reference as a biodegradable implant material. However, it is necessary to carry out other tests that are needed to get more data such as performing tensile, extrusion, rolling and other tests.

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