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## **Corrosion Behavior of Ternary Zr-**25Ti-5Sn Alloy Doped with Ge as **Biomaterials Implant in Simulation Body Fluid Solution**

Corrosion research of metal alloys of Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Geas biomaterials has been carried out in fluid solution. Zr-25Ti-5Snalloy is a ternary metal alloy developed for hard tissue biomaterials. Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge alloys is melted in electric arc furnace. After being melted Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge were characterized by optical microscopy. Hardness testing was carried out by the hardness microvickers method to determine the effect of germanium addition on Zr-25Ti-5Sn alloys. Corrosion testing of ternary metal alloys Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge was carried out by the Tafel polarization method using three electrode systems. From the results of microstructure examination with optical microscope, the microstructure found in the ternary metal alloy Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge are parallel plates and dendritic. The hardness test results show that the addition of germanium to the Zr-25Ti-5Sn ternary alloy increased the hardness of the alloy. Corrosion test results on ternary alloy Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Geindicated that corrosion resistance of Zr-25Ti-5Snincreased when no addition of Germanium to Zr-25Ti-5Snalloy.

Keywords: Corrosion, Biomaterial, Metal Alloy, Terner Zr-25Ti-5Sn

## 1. INTRODUCTION

Biomaterial as any substance or combination of substancescan be used for any period of time to augments or replaces partially or totally any tissue, organ or function of the body. Biomaterials are widely used in order to maintain or improve the quality of life of the individual. Such definition, however, does not include materials such as orthodontic brackets and surgical instruments[1].

Biotoleran materials should not be rejected by the human body when implanted in bone tissue. The implanted material will be create a response from the body and vice versa. Bioinert material can be movedon bonesurface, which leads toosteogenesis formation. In this conditionbody tissue is not permitted in the presence of implant material, it becomes materialit can be accepted by biological tissue andOsseointegration processes can occur. Bioactive materialsallowsnew bone formationon the surface of the material due to ions movementwith the host network. Then it forms chemical bonds of osteogenesis. Collagenand bone minerals will bind directly tosurface happenedbone implants contact.Bioinert and Bioactive ingredients act as osteoconductive, a scaffold to form bone growth on the surface of the implant[2,10].

Metallic materials are being increasingly used in medical applications as implants to restore lost functions or release organ functions below acceptable levels. Currently, the biomedical metals widely used for orthopedic implants mainly consist of titanium-based alloys, 316L stainless steel and cobalt-chromium-based alloys. Despite the considerable clinical success in service, current implant alloys still encounter complexities including wear debris or particles and release of toxic metallic ions, which are usually generated by tribocorrosion when implant alloys serving under physiological environment. Zirconium alloys are among the most used metallic biomaterials, particularly for orthopedic load-bearing components of the human body, including the hip, knee, finger joints and dental roots. Due to their excellent combination of high mechanical

strengthapplications. They possess a set of suitable properties for these applications such as low specific weight, high corrosion resistance, and biocompatibility. The introduction should present the paper theme, justifying the aim based on literature. It should also present the paper objectives. [3,4]

BIODYNAMIC ACTIVITIES	CHEMICAL COMPOSITION			
	METAL	CERAMICS	POLYMER	
Biotoleran	Gold		Polyethylene	
	cobalt chromiumalloys		Polyamide	
	Stainless steel		Polymethylmetacrylate	
	Zirconium		Polytetrafluoroethline	
	Neobium		Polyurethane	
	Tantalum			
Bioinert	Pure titanium	Alumunium oxide		
	Ti-6Al-4V	Zirkonium oxide		
Bioaktif		Hidroksiapatit		
		Trikalsium fosfat		
		Brushite Carbon-vitrous-		
		Pyrolitic Carbon-silicon- Bioglass		

**Tabel 1:** Classification of implant material

For the human body, assistive devices This bone connector is a foreign body that must adapt to its environment consisting of fluid. It has been owned by blood containing 355 -376 mg chloride per 100 ml of blood fluid. Research to get new materials with high performance is done by many people, especially in industrial countries such as America, Europe and Japan. One of the new materials currently being developed is an amorphous metal material that has high tenacity, and is often called metallic glasses. In glass structures the atoms are arranged periodically, but not over a long period. The structure of metallic glass was first discovered by P. Duwez for the Au-Si alloy in the 1960s, High corrosion resistance is one of the requirements that must be owned by a biomaterial. Corrosion is a form of degradation of material quality due to chemical reactions with the environment. Improvement in material performance related to its properties can be done in several ways, one of which is to vary the composition by changing the concentration or adding a combining element, This study aims to determine the value of zirconium-based metallic glass material for implant material corrosion rate, and determine the potential of zirconium-based metallic glass material for implant material. [8,7]

Currently, with the increase of the population of over 65 years old persons, in countries such as Japan, United States, Germany etc, the need for the use of artificial implants is also increasing, making necessary more thorough studies on materials that may be used for such an end. Some Zr and Ti alloys present, among other qualities, excellent mechanical properties, very good corrosion resistance, biocompatibility, and good durability, they become interesting and promising materials for use in implants.[4,9]

Biomaterials used to make implants and devices surgical implants and devices that replace parts or functions of the body's organs safely and economically. Biomaterials cover all types of materials ranging from metals, ceramics, to polymers. Table 1 summarizes the materials commonly used as biomaterials. [4,17]

MATERIAL	EXCELLENCE	WEAKNESS	APPLICATION
Metal: stainless steel, titanium alloy, alloy cobalt-chrome, etc.	Strong, tough, resilient	Non-bioactive	Orthopedic implants, dental implants, artificial joints, heart rings (stents), etc.
Ceramics: zirconia, alumina, bioglass, hydroxyapatite, etc.	Bioactive, inert	brittle	Orthopedic implants and teeth
Polymers: nylon,polyylactide,polyethyle ne, polyester, etc.	Bioactive, elastic	Not strong enough	Vascular grafts, sewing threads, artificial joint sockets, etc.
Composite: amalgam, fiber-reinforced bone cement, etc	Specially made	Relatively difficult to make	Bone cement, dental resin, etc.

Tabel 2 : Types of biomaterials and their applications

As a biomaterial, metal is used to come into contact with living tissue in the body so it must be biocompatible. Other important properties include good mechanical and physical properties, such as tensile strength, stiffness, fatigue resistance, and mass density.[7,11]

Tabel 3 : Environmental conditions of the body where metal implants are exposed

PARAMETER	SCORE	CONSEQUENCE
Body temperature	37°C	Chemical reactions run faster than at room temperature.
Ph :	7 15 - 7 35	Although hody fluid is a huffered solution the ph
<ul><li>b. Inter-cell fluid</li><li>c. Cell</li></ul>	7,0 6,8	can sometimes drop to 5.2 around the implant.
Dissolved oxygen: a. Arterial blood b. Venous blood c. Matrix between cells	100 mmhg 40 mmhg 2 ~ 40 mmhg	Corrosive environment
Chloride ions: a. Serum b.Intermediate fluid	113 meq/l 117 meq/l	Corrosive environment
Mechanical load: a. Kancelus bones b. Cortical bone c. Arterial walls d. Myocardium e. Muscle (max) f. Tendons (max)	0 – 4 mpa 0 ~ 40 mpa 0,2 – 1 mpa 0 – 0,02 mpa 40 mpa 400 mpa	Can cause stress corrosion and stress (stress corrosion cracking)

Dynamic load: a. myocardial contractions b.Finger joint movement c. ambulation	5 x 106 – 4 x 107 /year 105 – 106 /year 2 x 106 /year	Can cause fatigue and wear and tear
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Compared with other biomaterials such as ceramics and poylmers, the Metal biomaterials have extraordinary properties because they are capable resistance to tensile stress, which in the case of alloys, may be very high and also dynamic. This is the reason why alloys, for example which has adequate flexural flexural strength, is widely used as structural material for frame reconstruction when workload is high expected to occur. A typical example for implants that are very much loaded are hip and knee endoprostheses, plates, screws, nails, dental implants, etc.[13,14]

Based on the reaction of the tissue to the biomaterial, these are classified into three distinct categories, Biotolerant Materials: which are separated from bone tissue by a layer of fibrous tissue. Bioactive materials: which have the property of establishing chemical bonds with bone tissue, known as osseointegration. The collagen and mineral phase of the adjacent bone is deposited directly on the implant surface. Bioinert Materials: in this class it is possible, under certain conditions, to havedirect contact with the adjacent bonetissue. No chemical reactions shall occur between the implant and the tissue.[12,6]

The purpose of this study is to analyze the effect germanium substitutional elements on the structure, microstructure, and electrochemical measurements to reveal the corrosion performance to evaluate biocompatibility of the Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge wt% systems alloys.

## 2. MATERIALS AND METHODS

Synthesis Zr-25Ti-5Sn with and without germanium (Ge) byusing arc melting furnace under ultra high purity argon atmosphere, and ignited with a voltage40 V and an ampere of 100 A. Smelting is done by stike a tungsten electrode to a crucible made of pure Cu to generate arc containing raw materials. Ultra high purity jetting as a protective gas. Smelting is done 4 times, each material is 15 grams of Zr-25Ti-5Sn without germanium (Ge) doping and Zr-25Ti-3Sn-2Gegermanium (Ge) doping [8].

Stages of metallographic testing, carried out before the surface preparation of the sanding installation is completed with sandpaper paper 1000 mesh, 1500 mesh and 2000 mesh to the polishing installation using velvet cloth and zirconia powder (ZrO<sub>2</sub>) as an abrasive material to be able to use scratches on the surface as desired pattern planning interpretation of microstructure [8]. The etching stage to clarify the microstructure by corroding grain boundaries. The etching solution used was a mixture of 10 mL of fluoride acid solution (HF), 5 mL of HNO<sub>3</sub>, and 100 mL of aquadest with an immersion method for 20seconds[8]. Observation of the microstructure was carried out using an Olympus BX60M optical microscope with a magnification of 100x magnification [8].

Test specimens (as cast) were tested rigorously by the indentation method using the hardness microvickers with a holding time of 10 seconds. The number of tests was carried out by 5 points.

Corrosion testing is carried out using the polarization method on the as cast. By using electrochemical testing methods with polarization technique ASTM G59 in simulation body fluid solution media using DC 105 software [8]. The output of this test is a tafel polarization curve and corrosion potential curve which is then observed for changes in the polarization curve.[8]

REAGEN	COMPOSITION (g/mL)
Sodium Lactate (C <sub>3</sub> H <sub>5</sub> NaO <sub>3</sub> )	1.55 g/500 mL
Sodium Chloride (NaCl)	3.0 g/500 mL
Potassium Chloride (KCl)	0.15 g/500 mL
Calcium Chloride (CaCl <sub>2</sub> .2H <sub>2</sub> O)	0.1 g/500 mL
Water for injection	500 mL

Tabel 4: The content of simulation body fluid solution

 Tabel 5: Electrolyteconcentration of simulation body fluid solution

ELECTROLYTE CONCENTRATION			
KATIO [mEq/I]		ANION [mEq/I]	
Na <sup>+</sup>	130,3	Cl	109,4
$K^+$	4	Ca <sup>2+</sup>	2,7
Osmolaritas	274	Laktat <sup>-</sup>	27,7

## 3. RESULT

## 3.1 Micro structure

The picture below is a microstructure of Zr-25Ti-5Sn without germanium (Ge) doping and Zr-25Ti-3Sn-2Ge germanium (Ge) doping. It can be seen in figure 1 and 2.



**Figure 1:** A) Figure microstructure of ternary Zr-25Ti-5Sn alloys parallel plates magnification 20X. B) Figure microstructure of ternary Zr-25Ti-5Sn alloys parallel plates magnification 50X.



**Figure 2:** C) Figure microstructure of ternary Zr-25Ti-3Sn-2Gealloys dendritic magnification 20X. D) Figure microstructure of ternary Zr-25Ti-3Sn-2Gealloys densritic magnification 50X.

Based on the standards it can be seen that the phases formed in the zirconium alloy Zr-25Ti-5Sn (as received) are  $\alpha$ -Zr phases which have a brightly smooth color with a white equaxial structure as a matrix and  $\beta$ -Zr phases along the grain boundaries with black. In the  $\beta$  colonies are formed  $\alpha$  and  $\beta$ , containing equaxial  $\alpha$  with the same crystallographic orientation. Overall the shape of the as cast structure is parallel plates figures 1 (a) and (b) are 20x and 50x optical magnification.

Based on previous research, it can be seen that the phases formed in Zr-25Ti-3Sn-2Ge alloys are smooth  $\alpha$ -Zr phases with white equaxial structures as matrices and  $\beta$ -Zr phases along the grain boundaries with black. This result is almost the same as the Zr-25Ti-5Sn microstructure based on ASM metals handbook vol. 9. This is because the germanium element replaces the Sn atom (substitution). Figures (c) and (d) are 100x optical magnification.

Based on research that has been done before, it can be seen that the microstructure of Zr-25Ti-5Sn and the micro structure of Zr-25Ti-3Sn-2Gewill form a passive layer, ZrO<sub>2</sub>, TiO<sub>2</sub>, and GeO<sub>2</sub>. This passive layer will be known its thickness by XRD and SEM testing.

## 3.2 Hardness

Based on the data obtained from the tables obtained, it can be known about those that are incompatible with germanium on zirconium alloy Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge (as cast) having the highest average added value of 430,08 HV and 441,98 HV.

Table 6 : Effect of Ge on the hardness microvickers of ternary Zr-25Ti-5Sn alloys

Alloy (as cast)	Value hardness microvickers (HV)
Zr-25Ti-5Sn	430,08
Zr-25Ti-3Sn-2Ge	441,98

### 3.3 Corrosion testing

Based on ASTM G59 corrosion testing of zirconium alloys Zr-25Ti-5Sn(as cast), and Zr-25Ti-3Sn-2Ge (as cast) in simulation body fluid solution media and variations in the immersion time of 15, 50 and 75 minutes and with the help of software DC 105 and Echem Analyst V.5.66, anodic and cathodic constants are obtained, corrosion potential and corrosion rate as shown in Table 7. Polarization curve data is shown in figures 3 and 4. Corrosion potential curve data is shown in figure 5 and 6.

Table 7 : Effect of Ge on the hardness microvickers of ternary Zr-25Ti-5Sn alloys

ALLOYS		IMMERSION TIME		
(AS CAST)	INFORMATION	15 MINUTES	50 MINUTES	75 MINUTES
Zr-25Ti-5Sn	βa (V/decade)	0,3903 V/decade	0,5066 V/decade	0,4397 V/decade
	βc (V/decade)	0,1644 V/decade	0,1460 V/decade	0,2606 V/decade
	Icorr (µA)	1,260 µA	1,160 µA	2,640 µA
	Ecorr (mV)	-289,0 mV	-262,0 mV	-235,0 mV
	Corrosion rate (mpy)	1,141 mpy	1,053 mpy	2,397 mpy
Zr-25Ti-3Sn-2Ge	βa (V/decade)	0,5083 V/decade	0,9696 V/decade	0,8749 V/decade
	βc (V/decade)	0,0886 V/decade	0,1285 V/decade	0,1011 V/decade
	Icorr (µA)	5,790 µA	7,780 µA	6,760 μA
	Ecorr (mV)	-288,0 mV	-294,0 mV	-299,0 mV
	Corrosion rate (mpy)	5,261 mpy	7,155 mpy	6,144 mpy

Processing the curve is known that Zr-25Ti-5Sn(as cast) when immersed with simulation body fluid solution for 15 minutes, 50 minutes, 75 minutes each has: Icorr of 1,260  $\mu$ A, 1,160  $\mu$ A and 2,640  $\mu$ A, Ecorr - 289,0 mV, -262.0 mV and -235.0 mV and corrosion rate of 1,141 mpy, 1,053 mpy, 2,397 mpy.

Zr-25Ti-3Sn-2Ge(as cast) when immersed with simulation body fluid solution for 15 minutes, 50 minutes, 75 minutes each has Icorr at 5,790  $\mu$ A, 7,780  $\mu$ A and 6,760  $\mu$ A. Ecorr from -288.0 mV, -294.0 mV, and -299,0mV. Corrosion rate of 5,261 mpy, 7,155 mpy, 6,144 mpy for 15 minutes, 50 minutes, and 75 minutes immersion.



**Figure 3:** Figure tafel scan polarization curve of ternary Zr-25Ti-5Sn alloys with imertion time 15 minutes, 50 minutes and 75 minutes.



Figure 4: Figure tafel scan polarization curve of ternary Zr-25Ti-3Sn-2Ge alloys with imertion time 10 minutes, 50 minutes and 75 minutes.



Figure 5: Figure corrosion potential curve of ternary Zr-25Ti-5Sn alloys with imertion time 10 minutes, 50 minutes and 75 minutes.



Figure 6: Figure corrosion potential curve of ternary Zr-25Ti-3Sn-2Ge alloys with imertion time 10 minutes, 50 minutes and 75 minutes.

#### 4. DISCUSSION

From the discussion of the result, it can be concluded as follows that this zirconium alloy has phases  $\alpha$  and phases  $\beta$  where the Ti content contained in this combination has a role as  $\alpha$  stabilizer and Sn as  $\beta$  stabilizer. The strength of Zr alloys for biomedical applications is influenced by the type of alloy and its microstructure. Zirconium is known as an element that has very good toxic resistance so it is suitable to be used as a combination in the manufacture of implant material whose application is in the body of living things.[13]

Based on the standards it can be seen that the phases formed in the zirconium alloy Zr-25Ti-5Sn (as received) are  $\alpha$ -Zr phases which have a brightly smooth color with a white equaxial structure as a matrix and  $\beta$ -Zr phases along the grain boundaries with black. In the  $\beta$  colonies are formed  $\alpha$  and  $\beta$ , containing equaxial  $\alpha$  with the same crystallographic orientation. Overall the shape of the as cast structure is parallel plates figures 1 (a) and (b) are 20x and 50x optical magnification.[4]

Based on previous research, it can be seen that the phases formed in Zr-25Ti-3Sn-2Ge alloys are smooth

 $\alpha$ -Zr phases with white equaxial structures as matrices and  $\beta$ -Zr phases along the grain boundaries with black. This result is differ from the Zr-25Ti-5Sn microstructure based on ASM metals handbook vol. 9. This is because the germanium element replaces the Sn atom (substitution). Figures 2 (c) and (d) are 20x and 50x optical magnification.[9]

Based on research that has been done before, it can be seen that the microstructure of Zr-25Ti-5Sn and the micro structure of Zr-25Ti-3Sn-2Ge will form a passive layer, ZrO<sub>2</sub>, TiO<sub>2</sub>, and GeO<sub>2</sub>. This passive layer will be known its thickness by XRD and SEM testing.[7]

Based on the data obtained from the tables obtained, it can be known about those that are incompatible with germanium on zirconium alloy Zr-25Ti-5Sn and Zr-25Ti-3Sn-2Ge (as cast) having the highest average added value of 441,98 HV [2,11]. This increase in violence is caused by the replacement without zirconium which has the value of violence. The germanium atom will substitute for the atom so that in the  $\beta$  grains a stable  $\beta$ -phase will occur.[5]

The corrosion rate decreased in the Zr-25Ti-5Snbioimplant material is supported by the length of immersion time in the simulation of simulation body fluid solutions that the Zr-25Ti-5Snmaterial in zirconium alloy will form a very thin pasiv layer so that it inhibits ion rate shift. But the corrosion rate increase in the ternaryZr-25Ti-3Sn-2Ge alloys bioimplant material, This is due toby the length of immersion time in the simulation of a simulated body fluid solution so that the Zr-25Ti-3Sn-2Ge material in the zirconium alloy will form a thin film layer of passive oxides  $ZrO_2$ , TiO<sub>2</sub>, and GeO<sub>2</sub> which reacts with the ionic rate in the simulation of body fluid solution, and the passive layer is peeled off with immersion time.the passive GeO<sub>2</sub> layer is unstable in low temperatures, but the passive GeO<sub>2</sub> layer will be stable at high temperatures.[3]

Test data shows changes based on the effect of the combining elements with and without germanium and immersion time on the corrosion rate of Zr-25Ti-5Sn zirconium alloys in simulated body fluid solution media, the greater the addition of germanium, the corrosion rate increase and makes decrease resistance corrosion. Without germanium the corrosion rate of Zr-25Ti-5Sn zirconium alloys in simulated body fluid solution media will increase and makes decrease resistance corrosion, so the corrosion rate is very small and this material is safe to use as biocompactible material. This is because the element of titanium and tin will replace the zirconium atom in the  $\alpha$ Zr and  $\beta$ Zr phase. So that passive layer TiO<sub>2</sub> stable in ternary Zr-25Ti-5Sn alloys under passive layer ZrO<sub>2</sub>, passive layer TiO<sub>2</sub>will not easily react with ions that make the passive layer of TiO2 peel offwhich will harm in the body. After being plotted in the pourbaix diagram it will be in the passivation area where they will form a very thin layer of passiv and inhibit even stop the ongoing corrosion rate. Passivation formed can be seen in XRD and SEM-EDS.Combining elements Tin in the ternary Zr-25Ti-5Sn alloys makes modulus elasticity increase, zirconium alloys become ductile. This is because the element of tin will replace the zirconium atom in the  $\beta$ Zr phase and making the zirconium alloy boundary become small and stable with an increase in temperature in simulated body fluid simulation.[8]

## 5. CONCLUSIONS

From the results of the discussion, it can be concluded as follows, addition of germanium (Ge = 2%) in ternary Zr-25Ti-5Sn alloy increase hardness, Zr-25Ti-5Sn alloy has hardness of 430,08 HV, Zr-25Ti-3Sn-2Ge alloy has hardness of 441,98 HV, the addition of germanium has produce microstructure dendritic in ternary Zr-25Ti-3Sn-2Ge alloys, and microstructure of ternary Zr-25Ti-5Sn alloys has produce parallel plates, the addition of germanium increase the corrosion rate and decrease corrosion resistance, compared to without doped germanium in ternary Zr-25Ti-5Sn, corrosion rate of Zr-25Ti-5Sn alloy is 2,397mpy, Zr-25Ti-3Sn-2Ge alloy is 6,144mpy. Increased immersion time of 15 minute, 50 minutes and 75 minutes increases corrosion resistance and decreases corrosion rate in Zr-25Ti-5Sn alloy, this is due to the formation of more stable  $ZrO_2$  and  $TiO_2$  passive layers. The results of corrosion testing with the polarization method on Zr-25Ti-5Sn alloy have a corrosion rate in the range of 1-3 mpy, the corrosion rate of ternary alloy Zr-25Ti-5Sn alloy enters the outstandingcategory with a range of corrosion rate values of 1-5 mpy. The results of corrosion rate of ternary alloy Zr-25Ti-3Sn-2Ge alloy have a corrosion rate in the range of 2r-25Ti-3Sn-2Ge alloy have a corrosion rate in the range of 2r-25Ti-3Sn-2Ge alloy have a corrosion rate in the range of 2r-25Ti-3Sn-2Ge alloy have a corrosion rate in the range of 2r-25Ti-3Sn-2Ge alloy have a corrosion rate in the range of 5-7 mpy, the corrosion rate of ternary alloy Zr-25Ti-3Sn-2Ge alloy enters the excellent category with a range of corrosion rate values of 5-10 mpy. The formation of ZrO2 and NbO2 passive layers increases corrosion resistance in alloys.

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