The Effect of Solution Treatment on Mechanical Properties and Micro Structure of Zr-10Ti-Sn Alloy for Screw Dental Implant Application

Deva Ayu Utami

Metallurgical Engineering
University of Jenderal Achmad Yani
Terusan Gatot Subroto, Bandung,
Jawa Barat, INDONESIA
devaayu17@gmail.com

R.Henny Mulyani

Metallurgical Engineering
University of Jenderal Achmad Yani
Terusan Gatot Subroto, Bandung,
Jawa Barat, INDONESIA
Henny.mulyani@lecture.unjani.ac.id

Djoko Hadi Prajitno

National Nuclear Energy Agency of
Indonesia (BATAN)
Jalan Tamansari No 71, Bandung,
Jawa Barat, INDONESIA
djokohp71@yahoo.co.id

The research was to investigate the effect of solution treatment on the mechanical properties and microstructure of Zr-10Ti-Sn alloy for dental implant biomaterials. The addition of titanium as a material has good corrosion resistance and stannum as a material has a low modulus young and can increase hardness of the alloy in accordance with the material criteria as a dental implant, which must have good corrosion resistance and good mechanical properties. Zr -10Ti-xSn alloy ($x = 0, 2$, and 4% wt) were subjected to solution treatment with temperature variations of 900°C, 1000°C and 1100°C with water quenching. The results is increase in stannum content can also increase the hardness because stannum can inhibit the enlargement of grain boundaries and the microstructure is more homogeneous and tends to be smaller with an uniaxial shape. Stannum as alpha stabilizer will form a hard alpha phase. Micro Vickers hardness test with the highest hardness value of 601,438 HV alloy Zr-10Ti-2Sn with 900°C solution treatment, phases formed were α -Zr and β -Zr and intermetallic Zr_4Sn and $SnTi_3$ Zr-10Ti-4Sn alloys which were identified using X-Ray Diffraction (XRD).

Keywords: Implant Material, Solution Treatment, Zr-Ti-Sn Alloy, Alfa Stabilizer.

1. INTRODUCTION

The results of basic health research from the Ministry of Health of the Republic of Indonesia in 2018 showed 57.6% of the prevalence of dental health in Indonesia is at a bad level [1]. Generally, dental caries and tooth decay occur due to old age and accidents. So, do not be surprised if the permanent tooth loss is increasing. This not only disturbs the appearance but also makes the chewing function not optimal.[2]

For a long time, this problem was treated with the use of dentures. However, their simple methods often make use less convenient and more complicated. Starting from the frequent removal of dentures to be cleaned or removed and then swallowed. Another solution, nowadays medical technology is developing rapidly by implanting teeth (dental implants), which is an ideal natural tooth substitute by implanting the roots of the denture with a material made of metal, having properties and shapes resembling natural teeth.[3]

Another solution, nowadays medical technology is developing rapidly by implanting teeth (dental implants), which is an ideal natural tooth substitute by implanting the roots of the denture with a material made of metal, having properties and shapes resembling natural teeth. According to Neo Tee Khin, a dental implant expert at the Henry Lee Dental Surgery clinic at Singapore's Mount Elizabeth Medical Center.

Dental implants are implantation of dentures made of pure titanium metal which has biocompatible properties in the sense that the implants can be accepted and integrated into the human body without complications or rejection reactions from human tissues [2]. However, there are still many disadvantages of installing these dentures. Among them are easy to detach, break easily, wear easily, need

special care, and if used for a long time will cause discoloration of the dentures. Dental implants are one of the most popular tooth replacement methods.

Because dental implants are able to replace teeth function completely and permanently. Dental implants consist of several components, namely screws (threads or implant bodies), healing cups, abutments and crowns [4-7]. Dental screws (implants) are usually made of metal material. The aim of this research is to develop zirconium (Zr) alloy material for dental implant applications because in recent years zirconium is very attractive to be used as a new material for dental implants. Addition of the element titanium (Ti) which is corrosion resistant and stannum (Sn) which is an alpha stabilizer which can increase hardness [6]. Solution treatment is one method used to homogenize the existing phase of the alloy by heating the specimen at a certain temperature for a certain time and then let cool on a cooling medium such as water, oil, air [12].

2. MATERIALS AND METHODS

This study uses materials consisting of Zirconium sponge with 99% purity (Aldrich Chemical), Titanium rod with 99.8% purity and Stannum sponge with 99.9% purity (M&B laboratory chemical).

The equipment used in this study consists of Mettler Toledo AL204 Analytical Scale to weigh the materials according to the composition in Table 1, single arc melting furnace of PSTNT BATAN to melt the materials into alloys, hardness micro vickers Zwick/Reoll to obtain the hardness value of the alloy.

Table 1: Composition of Alloy (in % Wt)

Name of Sample	Zr		Ti		Sn	
	%	g	%	g	%	g
Zr-10Ti	90	18	10	2	-	-
Zr-10Ti-2Sn	88	17,6	10	2	2	0,4
Zr-10Ti-4Sn	86	17,2	10	2	4	0,8

Optical microscope Eclipse LV150 to analyze the microstructure of alloy, X-Ray Diffraction (XRD) PANalytical X'Pert PRO PW3040/x0 to analyze compounds and phases in alloy, tube furnace VTF6060 Rodatherm (USA) for solution treatment process and Scanning Electron Microscopy (SEM) to analyze phases and composition in alloy.

The materials are weighed with the composition in Table 1 with a total weight of 20 grams per alloy, then the melting process is conducted using single arc melting furnace, the melting process uses Tungsten electrode (W) given an electric current with a voltage of 230 V and current of ± 110 A so as to produce an arc that will melt the materials in a copper crucible hearth which is flowed with water as a cooler, the process is carried out in the atmosphere of Argon UHP 99.99% gas with high purity which aims to protect the melted materials from oxidizing. The melting process of each alloy is carried out 4 times to get homogeneous alloy results, melting results such as buttons. Each alloy (as cast) is prepared to form a cylindrical shape of ± 15 mm diameter and ± 4 mm thick.

Each alloy undergoes initial characterization in the form of hardness test using hardness micro vickers referring to ASTM E 92 with indentation in the form of a diamond cone and load of 100 g. Metallographic examination is conducted at the upper alloy surface, each alloy is prepared first with the sanding process using 60-2000 mesh sandpaper, polishing process uses a velvet cloth and CrO_2 polishing powder as the abrasive particle and the etching process uses etching solution with the composition of 5% H_2SO_4 , 10% HF, 30% HNO_3 and 55% H_2O , the etching is conducted by. The initial XRD testing is conducted on the upper alloy surface, after obtaining initial characterization, each alloy is subjected to solution treatment with 3 different temperature 900°C, 1000°C and 1100°C at tube furnace with nitrogen gas vacuum environmental after solution treatment materials are carried out hardness micro vickers testing, XRD testing and metallography, to find out the difference of materials before and after solution treatment.

Figure 1: Graphic of solution treatment

3. RESULTS

3.1 Hardness

Based on the results of hardness testing using micro vickers method, the hardness test results data on each alloy are obtained.

Table 2: Result of hardness micro vickers test Zr-10Ti-Sn alloy as cast

Spesimen	Pengujian ke- (HV)					Rata-rata
	1	2	3	4	5	
Zr-10Ti	314.06	311.4	306.19	322.23	314.06	313.54
Zr-10Ti-2Sn	456.12	437.97	425.05	460.83	446.9	445.37
Zr-10Ti-4Sn	485.54	480.44	470.48	495.98	470.48	480.58

Table 3: Result of hardness micro vickers test Zr-10Ti-Sn alloy after solution treatment

Spesimen	Solution treatment temperature	Pengujian ke- (HV)					Rata-rata
		1	2	3	4	5	
Zr-10Ti	900°C	607,27	628,12	607,27	597,23	607,27	609,432
	1000°C	424,08	427,06	430,07	436,19	421,13	427,706
	1100°C	513,13	525,18	501,49	501,49	525,18	513,294
Zr-10Ti-2Sn	900°C	505,33	505,33	490,24	499,7	497,7	601,438
	1000°C	505,33	490,24	501,49	517,1	497,7	502,372
	1100°C	529,29	537,66	531,93	529,29	531,93	532,02
Zr-10Ti-4Sn	900°C	533,45	533,45	529,29	525,18	529,29	530,132
	1000°C	462,03	479,37	436,19	458,68	445,62	456,378
	1100°C	472,32	468,85	465,43	479,37	472,32	471,658

Based on the data obtained from the tables, it can be known about those Zr-10Ti-4Sn alloy as cast has the highest average value of 480,58 HV and Zr-10Ti alloy after solution treatment at 900°C has the highest average value of 609,432 HV.

3.2 Micro Structure

The results of metallographic examination of Zr-10Ti-xSn as cast and after solution treatment with 500x magnification can be seen below:

Figure 2: a) Microstructure of ternary Zr-10Ti alloys as cast. b) Microstructure of ternary Zr-10Ti after solution treatment at 900°C. c) Microstructure of ternary Zr-10Ti alloys after solution treatment at 1000°C. d) Microstructure of ternary Zr-10Ti alloys after solution treatment at 1100°C .

Figure 3: a) Microstructure of ternary Zr-10Ti-2Sn alloys as cast. b) Microstructure of ternary Zr-10Ti-2Sn after solution treatment at 900°C c) Microstructure of ternary Zr-10Ti-2Sn alloys after solution treatment at 1000°C. d) Microstructure of ternary Zr-10Ti-2Sn alloys after solution treatment at 1100°C .

Figure 4: a) Microstructure of ternary Zr-10Ti-4Sn alloys as cast. b) Microstructure of ternary Zr-10Ti-4Sn after solution treatment at 900°C c) Microstructure of ternary Zr-10Ti-4Sn alloys after solution treatment at 1000°C. d) Microstructure of ternary Zr-10Ti-4Sn alloys after solution treatment at 1100°C .

Based on previous research, it can be seen that the phases formed in Zr-10Ti-Sn alloys are smooth α -Zr phases with white equiaxial structures as matrices and β -Zr phases along the grain boundaries with black [13]. Addition of stannum causes a reduction in grain size in generally the alloy has an α and β phase structure which is flat and shaping webbing connected to each other, or the commonly known basket weave structure.[14]

3.3 X-Ray Diffraction

Figure 5: XRD result of ZrTi alloy as cast

Figure 6: XRD result of ZrTi-2Sn alloy as cast

Figure 7: XRD result of ZrTi alloy after solution treatment

Figure 8: XRD result of ZrTi-2Sn alloy after solution treatment

Based on the XRD test results, the most dominant phase of the three alloys is the α -Zr phase. In the XRD results of the alloys with added Sn elements, it was found that the intermetallic compounds Zr Sn and Sn_1Ti_3 were found. however, in the alloys subjected to the solution treatment process, there is a decrease in the α -Zr phase and an increase in the β -Zr phase.

4. DISCUSSIONS

The results of this hardness test indicate that the addition of Sn to the alloy can increase the hardness of the Zr -Ti alloy . The higher the Sn composition added, the higher the hardness value of the alloy. This is because the addition of Sn material to Zr alloys or Ti alloys can cause the formation of Zr_4Sn and SnTi_5 compounds which are formed as precipitates so that they can increase the hardness and even the mechanical properties of the alloy.

Results of testing the hardness of the *solution treatment* can be increased due to the addition of Sn element that can hinder magnifying an item so that the mechanical properties can be improved. In addition, Sn is an *alpha stabilizer* that can multiply the alpha phase, where the alpha phase is hard

Based on metallographic examination, the phase formed is α -Zr which is shown by the light colored part and the β -Zr phase which is shown by the part which is dark colored. The addition of Sn in the form of the microstructure becomes *equiaxial* and causes the appearance of Zr_4Sn and SnTi_3 compounds based on the Zr-Sn phase diagram and Ti-Sn phase diagram. After the *solution treatment of the shape of the microstructure changes* , the Zr-10Ti alloy obtained an α -Zr phase and a β -Zr phase. The higher the temperature, the greater the grain size growth so that its mechanical properties decrease.

Unlike the case with alloy Zr-10Ti-2Sn and Zr-10Ti-4Sn at a temperature of 900°C phase formed is α -Zr phase, β -phase Zr, Zr compound $_4\text{Sn}$ and Sn_3Ti_5 . The shape of the microstructure of the results of *solution treatment more equiaxial* forms, but at a temperature of 1000°C there is an enlargement of the grain due to the higher temperature. At temperatures of 1100°C occurred great diminution item back as caused by the addition of Sn element can enlargement grains, so that the form of the microstructure of *equiaxial* more homogeneous and tends to be small [11].

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The results of the hardness test increased due to the addition of the element Sn which can inhibit grain enlargement so that the mechanical properties increase. In addition, Sn is an alpha stabilizer that can multiply the alpha phase, where the alpha phase is hard.

XRD results of the Zr-10Ti as cast alloy contained alpha Zr and beta Zr phases with 83% alpha phase and 17% beta phase respectively. After the solution treatment was carried out the Zr-10Ti alloy with a heating temperature of 1100°C containing 57% alpha phase and 53% beta phase. It can be concluded that the solution treatment process can increase the beta phase, because the function of the heating process is to get the beta phase which has good corrosion resistance.

The XRD results of Zr-10Ti-4Sn as cast alloy contained an alpha phase, a beta phase, and formed intermetallic compounds, namely Zr_4Sn and $SnTi_3$ with 54.6% alpha Zr, 9% beta Zr, 27% Zr_4Sn and 9% $SnTi_3$ percentages respectively. After the solution treatment was carried out at a temperature of 1100°C for the Zr-10Ti-4Sn alloy, the XRD results showed that there was an alpha phase, a beta phase, and an intermetallic compound was formed, namely $SnTi_3$ with 40% alpha Zr, 40% beta Zr and 20% $SnTi_3$ respectively. From the comparison of the XRD results above, it shows that the higher the temperature of the solution treatment, the less intermetallic compounds formed, causing the hardness value to be lower than the as-cast alloy.

Zirconium alloy has phases α and phases β where the Ti content contained in this combination has a role as α stabilizer and Sn as β 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.[15]

5. CONCLUSION

The addition of titanium element can increase the hardness due to the formation of intermetallic compounds, XRD results show the formation of $SnTi_3$ compounds in the Zr-10Ti-4Sn alloy resulting from the solution treatment process at 1100°C. The addition of 2% and 4% stannum elements can increase the hardness of the Zr-10Ti alloy because stannum is an alpha stabilizer which can increase the hard alpha phase. Alloy Zr-10Ti-4Sn as cast with the highest hardness value that is 480.58 HV. The solution treatment process is carried out at 3 different temperature variations, namely 900°C, 1000°C and 1100°C for 7 hours which can cause changes in hardness values and changes in average grain size. The hardness value of the Zr-10Ti alloy after solution treatment has decreased due to the higher the temperature, the larger the grain size is so that the hardness value decreases. The alloy hardness value of Zr-10Ti-2Sn and Zr-10Ti-4Sn after solution treatment has decreased at 1000°C due to the higher temperature there is an enlargement of the grain size and the formation of the beta phase so that the hardness value decreases. The hardness value of Zr-10Ti-2Sn and Zr alloys -10Ti-4Sn after solution treatment has increased at 1100°C due to the role of Sn as an alpha stabilizer which can increase the alpha phase and inhibit the enlargement of grain size so that the hardness value increases.

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