HARDNESS AND CORROSION RESISTANCE Zr-2Ag ALLOY DOPED WITH CO ELEMENT FOR BIOMATERIAL SCREW DENTAL IMPLANT APPLICATION

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*ABSTRACT*

Implant material is one solution to overcome damage or replace the function of human organs. The many needs regarding implants or biomaterials make researchers continue to carry out various studies. One of the criteria as an implant material is to have good corrosion resistance so that it can prevent rejection from within the body. This study aims to determine the corrosion behavior of Zr-Ag-Co implant material in lactated Ringer's solution. Zr-Ag-Co implant material using 5% and 7.5% Co content variation was synthesized by melting process using Single Arc Melting Furnace with argon gas atmosphere. The results of the smelting process were characterized using an optical microscope and Vickers hardness tester. Corrosion behavior of Zr-Ag-Co implant material was tested using a potentiostat with the Tafel polarization method in Ringer's lactate solution.

From the results of characterization by optical microscopy and XRD, it shows phases such as -Zr and -Zr as well as intermetallic compounds Zr2Co and Zr2Ag. The results of the hardness test showed that an increase in Co content could increase the hardness value of the Zr-Ag-Co alloy so that it had a hardness value ranging from 496.87 to 510.44 HV.

Keywords: Implant Material, Corrosion Resistance, Zr-Ag-Co, Tafel Polarization, Ringer's Lactate Soluti

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**PRELIMINARY**

**Background**

Based on information from the Data and Information Center of the Indonesian Ministry of Health, dental and oral health problems, especially dental caries, are a disease that affects almost half of the world's population. [1] Dental caries can cause tooth loss. [2].

Lost teeth should ideally be restored immediately in order to achieve a good normal condition, so as to improve normal contours, comfort, aesthetics, speech function, and prevent caries. One solution to overcome cases of tooth loss is to use dental implants.[3].

One type of dental implant is a dental screw material that serves to support dentures. [4] Dental implants will come into direct contact with body tissues, where the tissue can respond to a foreign object's resistance. [3] For this reason, dental implant materials should ideally have biocompatible properties, be strong to withstand chewing loads, be corrosion resistant, and resistant to fracture. [5] The dental implant material that is widely used is Ti-6Al-4V because it has excellent biocompatibility, is not prone to allergies and has good corrosion resistance [3,6,7,8,9]. However, Ti-6Al-4V has a fairly high economic value, as an alternative it is necessary to research dental implant materials which have lower economic value, but have the same characteristics or even better than Ti-6Al-4V dental implant materials. One of the dental implant materials that is expected to meet these requirements is a Zirconium-based dental implant material.

Zirconium dental implants have excellent biomechanical properties and biocompatibility, and are corrosion resistant. [5] In this study the author will combine Zirconium material with Cobalt and Argentum. Cobalt was chosen because based on research, Cobalt alloys are known for their excellent corrosion resistance and wear resistance, so they are widely used for dental implants and artificial joints.[10,11] While Argentum is used because it has good anti-bacterial properties.[12,13].

This research is expected to study the properties of Zr-Ag-Co alloy, as a new material to replace Ti-6Al-4V for Screw Dental Implants, using the casting method and material characterization with visual tests, hardness, microstructure, X-Ray Diffraction, Scanning Electron Microscope. ( SEM ) , and corrosion rate ( Tafel polarization method ) dissolved in Simulated Body Fluid ( SBF ) solution.

**Research Problems**

How to make alloys for Screw Dental Implant materials using Cobalt-dopped Zr-Ag compounds in order to obtain good microstructure, hardness and corrosion resistance?

**Research purposes**

The purpose of this research is as follows:

1. Designing and making Zr-Ag-Co alloys.

2. Analyze the influence of Co variation on the hardness of Zr-Ag alloy

3. Analyze the influence of Co variation on the microstructure of the Zr-Ag alloy.

4. Analyzing the corrosion resistance of Zr-Ag-Co alloy on Simulated Body Fluid solution.

**Scope of problem**

In this study, it is stated that there is a problem limitation with the intention that this research focuses on the research objectives that have been described above while the problem limits include:

1. Research is only limited to making alloys, not implant products that are ready to use.
2. Materials used are Zirconium balance, 2% Argentum and Cobalt (0%; 5%; 7.5%).
3. The alloy has good corrosion resistance as evidenced by the results of the polarization test (Tafel method)

**RESEARCH METHODOLOGY**

**Research Process Parameters**

Table 0.1 Material balance and its tests

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | Zr | Ag | Co | Struktur Mikro | XRD | SEM/EDS | Hardness |
| 1 | 98 | Balance | 0 | ✓ | ✓ | ✓ | ✓ |
| 2 | 93 | 5 |
| 3 | 90,5 | 7,5 |

## **DATA AND DISCUSSION**

## **Research Result Data**

## Based on the research process conducted on "Corrosion Resistance of Zr-Ag-Co Terner Alloys for Dental Implant Biomaterial Applications in Ringer's Lactate Solution" data obtained in the form of images, graphs and numbers from visual inspection, metallographic testing, hardness testing, phase or compound testing (XRD ), measurement of solution pH, corrosion testing, Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) testing as well as bacterial testing from the results of the tests that have been carried out.

1. **Visual Inspection Result Data**

Specimens that have been made through the smelting process using a Single Arc Melting Furnace are visually examined by documenting their surface morphology using a digital camera. This examination is carried out to determine the surface image of the specimen.

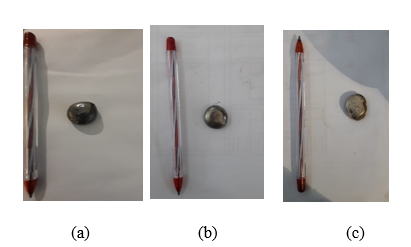


Figure 4.1 Results of Visual Examination After Smelting Process (a) Specimen 1 (Zr-2Ag), (b)

Specimen II (Zr-2Ag-5Co), and (c) Specimen III (Zr-2Ag-7.5Co)

The first specimen was dark gray and matte, the second specimen was bluish and glossy, while the third specimen was golden and glossy. Differences in color may occur due to differences in the cleanliness conditions of the tip of the electrode used during the smelting process

A picture containing text

Description automatically generatedPrior to the characterization process, the specimen was shaped into a cylinder with a diameter of 15 mm with a thickness of ± 4 mm to facilitate the characterization process.

Figure 4.2 Visual Examination Results Before Characterization Process (a) Specimen I (Zr-2Ag), (b)

A picture containing text

Description automatically generatedSpecimen II (Zr-2Ag-5Co), and (c) Specimen 111 (Zr-2Ag-7.5Co)

Figure 4.3 Visual Examination Results After Characterization Process (a) Specimen 1 (Zr-2Ag). (b)

Specimen II (Zr-2Ag-5Co). and (c) Specimen III (Zr-2Ag-7,5Co)

Specimens before characterization were gray and not shiny. Then the specimen undergoes a sanding process from 80 mesh to 2000 mesh until the color changes to a more glossy finish before finally going through the process of metallographic inspection, hardness testing, and corrosion testing. After the corrosion test was carried out, the part of the specimen of ± 10 mm2 which was in direct contact with the test media solution changed color to become not shiny while the part that was retained by the holder remained shiny as before.

1. **Metallographic Test Results Data**

2.1 **Qualitative Metallography**

In the process of this research, the first test was metallographic testing which was carried out at the Metal Laboratory, Jenderal Achmad Yani University, Bandung. This metallographic test aims to determine the microstructure of the specimen using an optical microscope.

a) Magnification 20 x

|  |  |
| --- | --- |
|  | A picture containing text  Description automatically generated |
| (a) | (b) |
|  | |
| (c) | |

Figure 4.4 Metallographic Examination Results with 20 m Magnification (a) Specimen 1 (Zr-2Ag), (b)

Specimen II (Zr-2Ag-5Co), and (c) Specimen III (Zr-2Ag-7.5Co)

b) Magnification 40 x

|  |  |
| --- | --- |
| A picture containing text, outdoor  Description automatically generated |  |
| (a) | (b) |
| A pile of small rocks  Description automatically generated with low confidence | |
| (c) | |

Figure 4.5 Metallographic Examination Basil with 40 m Magnification (a) Specimen I (Zr-2Ag), (b)

Specimen 11 (Zr-2Ag-5Co), and (c) Specimen 111 (Zr-2Ag-7.5Co)

c) Magnification 200 x

|  |  |
| --- | --- |
| A picture containing outdoor  Description automatically generated | A picture containing rock  Description automatically generated |
| (a) | (b) |
| A picture containing rock  Description automatically generated | |
| © | |

Figure 4.6 Results of Metallographic Examination with a Magnification of 200 m (a) Specimen 1 (Zr-2Ag), (b) Specimen FI (Zr-2Ag-5Co), and (c) Specimen (II (Zr-2Ag-7.5Co)

Based on the metallographic examination in Figure 4.7, the microstructure obtained is dendritic in shape which shows the results of the casting process. In the microstructure produced from the Zr-2Ag alloy, the -Zr phase is found as a matrix and the -Ti phase is marked by the bright part above the needle-shaped matrix. Meanwhile, in the Zr-2Ag-5Co and Zr-2Ag-7.5Co alloys, intermetallic compounds such as TiiCu and ZriCu were found which were characterized by white spots on the matrix and blackish color at the grain boundaries.

1. **Vickers Hardness Test Results Data**

The next test is the hardness test using the Vickers method which is carried out at the Metallurgy & Bonding Composite Laboratory of PT. Indonesian Aerospace. This hardness test aims to determine the hardness value of each specimen.

Table 4.2 Vickers Hardness Test Results

|  |  |  |  |
| --- | --- | --- | --- |
|  | Hardness Vickers (HV) | | |
|  | Alloy | Alloy | Alloy |
|  | Zr-2Ag | Zr-2Ag-5Co | Zr-2Ag-7,5Co |
| 1st Test Point | 477,40 | 494,50 | 522,68 |
| 2nd Test Point | 460,25 | 513,07 | 508,17 |
| 3rd Test Point | 468,62 | 486,32 | 504,93 |
| 4th Test Point | 476,34 | 489,67 | 503,68 |
| 5th Test Point | 485,25 | 500,81 | 512,73 |
| Nilai Rata-Rata | 473,57 | 496,87 | 510,44 |

The average value data that has been obtained is then converted into a graph to show the effect of the addition of Cobalt on the hardness of the Zr-2Agi alloy. The following is a graph of the hardness value data from the results of the tests that have been carried out:

Chart, bar chart

Description automatically generated

Figure 4.11 Effect of Co on the Hardness Value of Zr-2Ag . Alloy

Based on the data from the hardness test results using the Vickers method in Table 4.2 the test specimen with the composition Zr-2Ag-7.5Co has the highest average hardness value and the lowest average hardness value is owned by the Zr-2Ag specimen. This can indicate that the addition of Cobalt (Co) in the Zr-2Ag alloy has an effect on the hardness value.

1. **Discussion and Processing of X-Ray Diffraction (XRD) Testing Data**

**Zr98Ag2**

Figure 1 shows the diffraction of a sample of 98Zr, which was analyzed by Rietveld using the GSASS II software, showing peaks corresponding to the peak patterns of alpha Zr and Beta Zr using the CIF database 9008559 for Beta Zr and 9008523 for Alpha Zr.

Chart, histogram

Description automatically generated

Figure 1 XRD results from a sample of 98%Zr that has been analyzed by Rietveld using GSAS II software.

Chart, histogram

Description automatically generatedThe data from the Rietveld analysis were then processed into 3D images of the Alpha Zr and Beta Zr structures using VESTA software. For this image, Alpha Zr forms a hexagonal structure with a P63/mmc space group, while the Beta Zr phase forms a cubic structure with an Im-3m space group. In addition, it appears that the first sample formed another phase besides Alpha Zr, namely Beta Zr which is indicated by the orange line. While the model used has a value of Goodness of Fit (GOF) = 1.25 and wR = 5.666. The results of the calculation of the weight fraction of the first sample using GSAS-II are shown in table 1 as follows:

Table 1 ZrAg . sample weight fraction

|  |  |
| --- | --- |
| Phase | % Fraction |
| Alpa Zr | 80.3% |
| Beta Zr | 19.7% |

**ZrAg Doping Co**

In this section, we will discuss 2 samples consisting of two types of variations, namely variations in the addition of Co doping to the ZrAg sample. The first sample is Zr92%Ag3%Co5% sample which is synthesized with stoichiometric ratiowith the casting process at a temperature of 2000℃ for 10 minutes with cooling for 15 minutes. The aim is to add Co elements so that more secondary phases are formed during the sintering process. Rietveld refinement results using GSAS-II for this first sample can be seen as follows:

Figure 2 XRD results of the 5%Co sample which has been analyzed by Rietveld using GSAS II software.

Figure 2 XRD results of the 5%Co sample which has been analyzed by Rietveld using GSAS II software.

From Figure 2, it can be seen that the first sample is almost obtained with ZrAgMo samples, but it is still followed by two phases, namely alpha Zr which is indicated by a dark blue line and beta Zr phase which is indicated by a red line. The results of this first sample refinement show that the model used has a Goodnes of Fit (GOF) = 1.48 and wR = 5.464. Meanwhile, the results of the weight fraction calculation are shown in table 2 as follows:

Table 2 Sample weight fraction Zr92%Ag3%Co5%

|  |  |
| --- | --- |
| Phase | % Fraction |
| Alpa Zr | 52.6% |
| Beta Zr | 47.4% |

Furthermore, the second sample, namely Zr90.5%Ag2%Co7.5%, is the same as the first sample, the second sample is processed by casting at a temperature of 2000℃ for 10 minutes with cooling for 15 minutes. The results of rietveld refinement using GSAS-II for this first sample can be seen as follows:

Chart, histogram

Description automatically generated



**: α Zr :ꞵZr**

Figure 3 XRD results of the 7.5%Co sample that has been analyzed by Rietveld using GSAS II software.

From Figure 3 it can be seen that the second sample is better than the first sample because more and more beta Zr phases are formed. The results of this second sample refinement show that the model used has a Goodness of Fit (GOF) = 1.67 and wR = 5.941. While the results of the calculation of the weight fraction are shown in table 3 as follows:

Table 3 Sample weight fraction Zr90.5%Ag2%Co7.5%

|  |  |
| --- | --- |
| Phase | % Fraction |
| Alpa Zr | 37.2% |
| Beta Zr | 62.8% |

Based on the results of the refinement and calculation of the weight fraction, it can be concluded that the synthesis of the ZrAg phase was reproducible. For a clearer comparison of the three samples can be seen in the image below:

Figure 4 XRD comparison of the three ZrAg . samples

|  |  |  |
| --- | --- | --- |
| Parameter | 97%ZrAg | |
| Alpha Zr | Beta Zr |
| Struktur Kristal | Hexagonal | Cubic |
| *Space Group* | P 63/m m c | I – m 4 3 m |
| *Statistic Parameter* | | |
| Weighted R profile | 5.666 | 5.666 |
| Goodness of Fit (χ2) | 1.15 | 1.15 |
| *Lattice Parameter* | | |
| a (Å) | 3.2523 | 3.63468 |
| b (Å) | 3.2523 | 3.63468 |
| c (Å) | 5.1869 | 3.63468 |
| alpha [°] | 90 | 90 |
| beta [°] | 90 | 90 |
| gamma [°] | 120 | 90 |
| *Volume* [Å3] | 46.5929 | 47.486 |
| *Density* [g/cm3] | 6.392 | 6.405 |
| % Fraction | 80.3% | 19.7% |

All three samples have Alpha Zr forming a hexagonal structure with a P63/mmc space group, while the Beta Zr phase forms a cubic structure with an Im-3m space group with a general crystal structure as shown below:

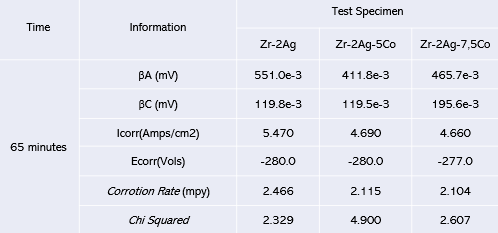
|  |
| --- |
| Chart, bubble chart  Description automatically generated    Figure 5a. Alpha Zr Structure  Chart, bubble chart  Description automatically generated |
| Figure 5b. Alpha Zr Structure |
|  |

1. **Qr code

   Description automatically generatedSEM Test**

Figure 6 Result of SEM Test

1. **Polarization Test**

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**CONCLUSIONS AND RECOMMENDATIONS**

**Conclusion**

1. Based on the results of the study, it was found that a new alloy as an alternative biomaterial for screw dental implants Zr-Ag-Co needs further research
2. The addition of Cobalt (Co) elements can increase the hardness of the Zr-2Ag alloy from 473.57 HV to 496.87 HV in the Zr-2Ag-5Co alloy (5% increase) and 510.44 HV in the Zr-2Ag-7.5Co alloy ( increase of 3%).
3. Based on XRD and SEM test results, the phases formed from the Zr-Ag-Co alloy are -Zr, and -Zr accompanied by intermetallic compounds Zr2Co, Zr3Co and Zr2Ag.
4. The addition of Co increases the phase thereby increasing the corrosion resistance (Co phase stabilizer ).
5. The addition of Cobalt (Co) element can increase corrosion resistance by reducing the corrosion rate of Zr-2Ag alloy from 2.466 mpy to 2.115 mpy in Zr-2Ag-5Co alloy and 2.104 mpy in Zr-2Ag-7.5Co alloy in Simulated Body Fluid solution. Of the three alloys, Zr-2Ag-7.5Co has the best corrosion resistance.

**Suggestion**

1. Perform antibacterial testing using harmful bacteria in the oral cavity
2. Further research is needed on Zr-Ag-Co alloys with different percent variations of Co and Zr.
3. Need to be tested in the real environment
4. Further research is needed to develop a manufacturing process for the manufacture of Zr-2Ag-Co screw dental implant alloys.

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