THE EFFECT OF AGING TREATMENT ON MECHANICAL PROPERTIES AND MICROSTRUCTURES OF Ti-12Cr IN ORTODONTIC APPLICATIONS

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Abstract. Many adults experience tooth loss due to accidents and lack of care. This causes various problems, one of which is a problem in chewing food. One solution that can be used is the use of dental implants. Dental implants that are currently made of titanium Ti-6Al-4V at a very expensive price. One of the newest types of titanium alloy material that can be used with relatively cheap prices is Ti-12Cr. As a new material, the effect of heat treatment processes on mechanical properties has not been investigated in more detail. So that research is needed on the effect of heat treatment with the aging treatment process on the mechanical properties and microstructure of the Ti-12Cr material as a substitute for cheaper titanium implants. In this research, a solution treatment (ST) heat treatment was carried out at 885°C and an aging treatment (AT) at 540°C. Aging treatment is carried out with variations in holding time of 30 ks and 60 ks. Then the microstructure inspection and mechanical properties testing are carried out through hardness and tensile tests. With the aging treatment of the microstructure of the material, Ti-12Cr experienced grain refinement and the hardness obtained was higher than that of Ti-6Al-4V. The hardness values of Ti-12Cr ST, Ti-12Cr AT 30 ks, Ti-12Cr AT 60 ks and Ti-6Al-4V are 457 VHN, 605 VHN, 772 VHN, and 349 VHN. However, the tensile strength of Ti-12Cr is lower than the tensile strength value of Ti-6Al-4V.

1. Introduction

Many people lose teeth (toothless teeth). This is caused by various factors, such as accidental damage and lack of maintenance. Statistics show that 70% of adults expelled from 35-44 years of age increase. One in four adults over 74 years has lost all their teeth.

Tooth loss cannot only reduced appearance but can cause various problems such as difficulties when chewing food, and difficulty speaking. Currently, various solutions are available to replace missing teeth, one of which is dental implants. Dental implants are part of orthodontics. The main components of orthodontics are dental implants, brackets, auxiliary, and orthodontic wire (archwire) [1]. Some of the options available are complete dentures made of acrylic which are usually used if all the teeth have been lost, partial dentures that can be used when only lose a few teeth. Besides, there is a dental bridge
that is a denture that is permanently placed and finally a dental implant whose use involves placing a small metal rod into the jawbone. This stem functions like a tooth root, when the implant is attached to the jawbone, the dental crown can be attached to it. Generally, metal implants that are often used and are quite popular are stainless steel and titanium. Both have high strength and good tenacity [2]. The stainless steel used is the type of SUS 316L (ASTM F138)[3]. Stainless steel is used because it has corrosion-resistant properties. This type of material still causes problems that are allergens to some consumers due to hypersensitivity reactions[4]. The main factor causing allergies is the element content of Nickel (Ni). Especially titanium alloy is a safe material for the body, because it has the best biocompatibility among metals[5], and has high strength, more corrosion resistance, and lower elastic modulus [6] [7]. Titanium used for dental implants today is a type alloy ($\alpha+\beta$) Ti-6Al-4V-ELI alloy (ASTM F136) [8] with aluminum and vanadium alloys. The mechanical properties of these metal biomaterials can be seen in Table 1. From the Table, it can be seen that compared to other types of biomaterials, Ti-6Al-4V alloys have the best tensile properties for biomaterials. Ti-6Al-4V has the highest yield and tensile strength even though the elasticity is slightly higher than Cp-Ti. Titanium has a very expensive price of around 15 million / tooth shaft [7]. To overcome the problem of relatively expensive prices, alternative materials are needed that are not too expensive but have high tensile strength and yield strength. The titanium that is being developed at the moment $\beta$-type titanium including TNTZ (Ti-29Nb-13Ta-4,6Zr) [5][9] and the latest Ti-12Cr with a few and inexpensive alloy elements, such as Cr [10]. Research on Ti-12Cr alloys was only carried out at the mechanical properties testing stage, namely bending test to determine the elastic modulus (Young's modulus) of the alloy [11]. The modulus of elasticity and strength of Ti-12Cr alloys are suitable for applications in backbone support [11]. It has been concluded that Ti-12Cr is more recommended for backbone applications [12]. From the description above, it can be seen that previous studies on the Ti-12Cr material were limited to bone implants. However, research on the effect of the heat treatment process on the mechanical properties of Ti-12Cr for dental implants is not yet clearly known. Therefore, this research will examine how the effect of aging treatment on Ti-12Cr material by looking at mechanical properties, especially tensile properties and microstructure in the hope that it can approach the properties of Ti-6Al-4V.

### Table 1. Comparison of some mechanical properties of several metals commonly used as biomaterials [13]

| Alloy Type       | Modulus of Elasticity | Yield Strength | Maximum Tensile Strength |
|------------------|-----------------------|----------------|--------------------------|
|                  | GPa 10⁶psi            | MPa            | MPa 10⁶psi               | MPa 10⁶psi               |
| Stainless steel  | 200                   | 29             | 150-170                  | 25-110                   | 465-950                  | 65-140                   |
| Co-Cr-Mo         | 200-230               | 29-33          | 275-1583                 | 40-230                   | 600-1795                 | 90-260                   |
| Commercial Pure Ti | 105                 | 15             | 692                     | 100                      | 785                      | 115                      |
| Ti-6Al-4V        | 110                   | 16             | 850-900                  | 120-130                  | 960-970                  | 140-141                  |

The expected goal of this study is to determine the effect of aging treatment on microstructure and mechanical properties, namely the value of hardness and tensile strength of Ti-12Cr as material for dental implants.

### 2. Methodology

#### a. Sample Preparation

The samples tested were $\beta$-type Ti-12Cr titanium in the form of plates with a thickness of 3 mm and each test of 3 samples. Then the cutting process is done using wire cutting with a size of 12X10 mm. Next, level the surface using sandpaper starting from the smallest 400, 800, 1200, 1500 mesh, the last mesh 2000 and polished to produce a shiny surface free from scratches.

#### b. Aging Treatment Process

The aging treatment process begins with heating using a Ney Ceramfires vacuum furnace. Before the aging treatment process, the solution treatment process is carried out by heating the specimen to a temperature of 885°C degree of temperature increase of 10°C / s, holding time for 1 hour. Next, do a
quick cooling by dipping the specimen in water (water quenching). After that, the heating process is re-heated at 540˚C withholding for 30 ks (about ± 8 hours) and variations in the heating process to 60 ks (about ± 16 hours) are also carried out. Then for each per variation carried out rapid cooling using water. Seen in Figure 1, the aging treatment process occurs.

![Figure 1. Scheme of the aging treatment process](image)

c. **Inspection of Micro Structure**
   Testing using an Olympus optical microscope to see the microstructure of titanium Ti-12Cr material that has been carried out the heat treatment process with three treatments namely solution treatment, aging treatment withholding time 30 ks, and aging treatment withholding time 60 ks. The magnification variation used starts from 20 times, 50 times and 100 times.

d. **Mechanical Testing**
   Mechanical testing is done by a hardness test and tensile test. A hardness measurement of Ti-12Cr refers to ASTM 384; Standard Test Methods for Knoop and Vickers Hardness Materials [14]. The indenter used in this test is a rectangular pyramid with a 136° angle and a loading value of 9.8 N with an indentation time of 10-15 seconds. Tensile testing with Universal Testing Machine (UTM).

3. **Result and Discussion**
   A. **Ti-12Cr Micro Structure following Aging Treatment Process.**
   The process carried out to see the results of the Ti-12Cr microstructure description is a metallographic process.

   1. **Solution Treatment**
   This water quenching treatment is carried out after giving a treatment solution that changes the β phase to the α phase on heating at 890˚C. The results of observing microstructure with solution treatment can be seen in Figure 2.
Figure 2. The results of the micro-structure Ti-12Cr treatment solution treatment (a) magnification 20 times, (b) magnification 50 times, and (c) magnification 100 times.

In Figure 2, it is seen that the results of the solution treatment process have a martensite phase (α phase) with a needle-shaped microstructure. At a magnification of 20 times, a phase that resembles very tight needles is formed. In Figure 2 (b) with a magnification of 50 times, the needling phase is not too tight. Whereas in Figure 2 (c) with a magnification of 100 times the shape of the needle is seen more clearly.

2. Aging Treatment 30 ks

This aging treatment occurs after the existence of solution treatment (water quenching). The aging treatment is carried out in the form of heating withholding for 30 ks (± 8 hours), then cooling again using water. The results of the 30 ks aging treatment microstructure can be seen in Figure 3. The phases formed in this treatment are precipitated α and β phase. Precipitation α is formed after the aging treatment. The longer the aging treatment time, the more smooth the precipitate phase occurs.

Figure 3. Ti-12Cr microstructure results of aging treatment 30 ks, (a) magnification 20 times, (b) 50 times magnification, and (c) magnification 100 times.

In Figure 3 you can see the dark-colored precipitate α and the light-colored β phase. In Figure 3 (a) it is seen that the α-shaped precipitate specimen is small in magnification 20 times. In Figure 3 (b) the precipitate α appears to be greater than 20 times magnification, whereas in Figure 3 (c) the specimen has a greater precipitate α compared to 50 times magnification. Precipitate α is formed after aging treatment. The microstructure is smooth compared to water quenching treatment.
3. **Aging Treatment 60 ks**

The 60 ks aging treatment is the same as the 30 ks aging treatment. The difference occupies in the length of detention, which is at 60 ks (± 16 hours). The results of observing the microstructure of the 60 ks aging treatment can be seen in Figure 4. The phases formed in this treatment are precipitated α and β phase. The precipitate α that is formed in the 60 ks aging treatment is finer than the 30 ks aging treatment. The finer the precipitate formed, the harder the material [15].

![Figure 4](image_url)

**Figure 4.** The results of the Ti-12Cr microstructure aging treatment treatment 60 ks (a) magnification 20 times, (b) magnification 50 times, and (c) magnification 100 times.

In Figure 4 there is a dark-colored precipitation α and a light-colored β phase. In Figure 4 (a) the specimen looks precipitate α, small in size with a magnification of 20 times. In Figure 4 (b) precipitation α looks greater than a magnification of 20 times. In Figure 4 (c) the specimen has a precipitate α greater than 50 times magnification. The precipitation α formed in Figure 4 is finer than the precipitation α found in the results of the 30 ks aging treatment test.

### B. Hardness Ti-12Cr

Hardness value was obtained from 3 tests, namely testing the average hardness value of the Ti-12Cr titanium alloy was carried out at 5 points for each test. As shown in Table 2 the value of hardness shown is the average price of the hardness of alloys that have been treated with solution treatment and aging treatment. Based on the data summarized in Table 2, a comparison graph is obtained from the average hardness of the aging treatment (Figure 5).

| No | Treatment                  | Hardness value | Mean |
|----|----------------------------|----------------|------|
| 1  | Solution treatment         | 404            | 457  |
|    |                            | 463            |      |
|    |                            | 488            |      |
|    |                            | 499            |      |
|    |                            | 430            |      |
| 2  | Aging treatment 30 ks      | 553            | 605  |
|    |                            | 631            |      |
|    |                            | 625            |      |
|    |                            | 688            |      |
|    |                            | 528            |      |
| 3  | Aging treatment 60 ks      | 665            | 772  |
|    |                            | 708            |      |
|    |                            | 745            |      |
|    |                            | 898            |      |
|    |                            | 843            |      |
In Table 2 and Figure 5, it can be seen that the Ti-12Cr hardness value obtained is quite significant after undergoing the aging treatment process. The average value of violence increased from 457 HVN in the treatment solution to 605 HVN in the 30 ks aging treatment as well as for aging treatment 60 ks in the average hardness to 772 HVN. The highest hardness value obtained from aging treatment 60 ks. This is in line with previous research as shown in Table 3, the value of violence increased after aging treatment [16]. Based on Table 2 and Table 3, the hardness value of Ti-12Cr after experiencing aging treatment is higher than Ti-6Al-4V.

![Graph of hardness testing of Ti-12Cr](image)

**Figure 5.** Graph of hardness testing of Ti-12Cr

### Table 3. Hardness of Ti-6Al-4V with solution treatment and aging treatment [14].

| Titanium alloy Ti6Al4V | Solution treatment | 1050°C/1h/water | 1050°C/1h/air | 950°C/1h/water | 950°C/1h/air | 800°C/1h/water | 800°C/1h/air |
|------------------------|--------------------|-----------------|--------------|----------------|--------------|----------------|--------------|
| Hardness in as-cast condition 312 HV 10 | Hardness | 405 | 320 | 395 | 311 | 344 | 319 |

| Treatment + aging | 550°C/4h | 550°C/4h | 550°C/4h | 550°C/4h |
|-------------------|---------|---------|---------|---------|
| Solution treatment | 326 | 332 | 340 | 342 |
| Aging treatment 30 ks | 320 | 326 | 332 | 338 |
| Aging treatment 60 ks | 316 | 322 | 328 | 334 |

C. Ti-12Cr Tensile Strength Test

The test was carried out using a UTM machine with a scale A load and a total load of 3 tons. This test uses support for supporting the specimen because of its small size. The following is the attachment of the tensile strength test data using the UTM machine as shown in Table 4.

### Table 4. Tensile strength value Ti-12Cr

| No. | Treatment | Test | Tensile strength (MPa) |
|-----|-----------|------|------------------------|
| 1   | Solution treatment | 1    | 925                    |
|     |           | 2    | 905                    |
|     |           | 3    | 900                    |
| 2   | Aging treatment 30 ks | 1    | 310                    |
|     |           | 2    | 450                    |
|     |           | 3    | 435                    |
| 3   | Aging treatment 60 ks | 1    | 120                    |
|     |           | 2    | 110                    |
|     |           | 3    | 105                    |
Based on Table 4, it can be seen that the strength value of the specimens that experienced higher treatment treatments is around 900 MPa, followed by 30 ks aging treatment test and the lowest value is 60 aging ks treatment. Noticeable differences occur for the results obtained between strength testing and hardness testing. According to his theory violence and strength are directly proportional [13]. Whereas based on the results of the hardness test, the highest hardness was obtained in the 60 ks aging treatment test, followed by the 30 ks aging treatment, and the lowest hardness in the solution treatment test.

From the results of this study, it is known that violence increases while strength decreases. This shows that there was an error in data retrieval that allegedly occurred during the tensile testing. In the tensile test, the speed used is too large while the specimen is small so that the specimen experiences a decrease in strength and causes agility. Then the data obtained is not a tensile test data but impact test data because the shape of the fracture that occurs in the form of brittle and there is no cross-section at all (can be seen in Figure 4.10, Figure 4.11, and Figure 4.12). In comparison to the results obtained from the study of Zhao, et al. (2012) [9] can be seen in Figure 6.

**Figure 6.** Tensile strength of Ti-xCr alloy undergoing treatment and cold rolling [9].

In Figure 6, it can be seen that the strength of the treatment solution ranges from 700-800 MPa. The value of tensile strength obtained in this study is close to the value of tensile strength carried out by Zhao, et al. (2012) [9].

The tensile test carried out obtained a curve as shown in Figure 7. The highest strength value is obtained in the treatment solution, while the lowest is in the aging treatment test.
Figure 7. Graph of the tensile test results of Ti-12Cr (a) solution treatment 1, (b) solution treatment 2, (3) solution treatment 3, (d) aging treatment 30 ks 1, (e) aging treatment 30 ks 2, (f) aging treatment 30 ks 3, (g) aging treatment 60 ks 1, (h) aging treatment 60 ks 2, dan (i) aging treatment 60 ks 3

The location of the fracture in the treatment solution can be seen in Figure 8.

Figure 8. The area of fracture in the treatment solution

Figure 8 shows that the fault occurred in the reduced cross-section. The area is the test area to be tested tensile. In this test, it can also be seen that the cross-section has been reduced before experiencing a break. The location of the fracture in the 30 ks aging treatment can be seen in Figure 9.

Figure 9. Areas of fracture treatment aging treatment 30 ks.

In Figure 9 there is a fault that occurred in the test area near the cross-section of the exemplar. In
this test, it can also be seen that the specimen did not experience the slightest reduction in cross-section before the occurrence of fracture. The area of fracture with 60 ks aging treatment can be seen in Figure 10.

![specimen (1) specimen (2) specimen (3)](image)

**Figure 10.** Fractional area of aging treatment 60 ks

In Figure 10 it appears that the fault happened in the area that should be, but for exemplar 3 the difference in fracture occurred in the area near the cross-section of the specimen. In the tests carried out did not experience the slightest reduction in cross-section before the occurrence of a fracture.

In testing the strength of the Ti-12Cr, the results obtained for the treatment of fracture treatment as shown in Figure 11.

![specimen 1, specimen 2, specimen 3](image)

**Figure 11.** The shape of the fracture in the treatment solution (a) specimen 1, (b) specimen 2, and (c) specimen 3

In Figure 11, it can be seen that the fracture that occurs in the solution treatment is ductile, the texture is fibrous and gray. Based on these results it can be seen that the material resulting from this treatment has a ductile fracture. The results of fractures that occur in the 30 ks aging treatment can be seen in Figure 12.

![specimen 1, specimen 2, specimen 3](image)

**Figure 12.** Form of fractures in the treatment of 30 ks, (a) specimen 1, (b) specimen 2, and (c) specimen 3
In Figure 12 it can be seen that the fracture has shiny features with a relatively flat shape. Based on the features seen, it can be said that the fracture in the 30 ks aging treatment has a brittle fracture. The fracture that occurred did not experience the slightest cross-section reduction. For results of fractures in the 60 ks aging treatment can be seen in Figure 13.

![Fracture Images](a) (b) (c)

**Figure 13** Form of fracture in aging treatment 60 ks, (a) specimen 1, (b) specimen 2, and (c) specimen 3

Figure 13 shows that the shape of the fault is almost the same as the 30 ks aging treatment because the shape of the fracture is flat and also looks shiny.

4. Conclusion

Aging treatment causes changes in the microstructure of Ti-12Cr, where the grain obtained is getting smoother.

Aging treatment increases the hardness value of the Ti-12Cr material. A significant increase from 457 VHN in the treatment solution to 605 VHN in the 30 ks aging treatment test and finally 772 VHN in the 60 ks aging treatment.

Aging treatment causes the tensile strength of titanium to decrease due to high loading speeds so it does not qualify as static loading.

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