Optimum Load in Vickers Microhardness Testing of Treated Magnesium Alloy by Thermal Oxidation

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Abstract. Magnesium and its alloy have good properties as biomedical implant such as biodegradable material and excellent biocompatibility. Their hardness is an important mechanical property in using of these materials. Vickers microhardness is one of hardness testing methods in metal materials especially for thin layer on the metal. Mass of microhardness testing load also has important effect on result validity. The object of this research is to investigate optimum load in Vickers microhardness testing of magnesium alloy treated by thermal oxidation. Magnesium alloy AZ61 was treated by annealing at 150; 225; 300; 375; 450 °C of temperature. Vickers microhardness testing was carried out on this treated material using 5, 10, 25, 50, and 100 g of load. Five data for both each load and temperature, therefore there are 25 data for each load. Optimum load is determined the load which has lowest standard deviation in Vickers microhardness. The optimum load in this research was happened at 10 g in load of Vickers microhardness testing. The Vickers microhardness in this load was 36.0 g.mm⁻² and the standard deviation was 2.7 g.mm⁻².
1. Introduction

Development of biomaterial for fracture fixation or joint replacement has an important role in orthopedic surgery. Orthopedic biomaterial is implanted in body to heal of the fracture bone [1]. Magnesium and it alloy is one of the biomaterial which have some advantages as an implant material. Magnesium already existed more than one century [2] and has been improved until now. This material has density (1.738 g/cm³) [1] more closely to cortical bone density (1.75 – 2.1 g/cm³) [3] than other metal implants. Another advantage, its biocompatibility is good and increase the bone formation in the body [4]. Beside of their advantages, the corrosion resistance is one of the disadvantages of these materials.

Treatment of these materials have been developed to reduce their drawbacks. Surface treatments such as coating, shot peening, surface mechanical attrition treatment already were developed to minimize their weakness. Coating treatment can be done by thermal oxidation, ion implantation, etc. Ion reaction between substrate and its environment [5] or deposit the coating material on the substrate surface [6] are common method in coating treatment. Coating of magnesium by embedded in NaOH solution results in passive layer on the substrate surface [7]. Corrosion resistance of magnesium enhance by coating of calcium phosphate [8]. Coating by fluoride on magnesium formed MgF2 and also increased the corrosion resistance [9]. Biodegradable of coated magnesium by phosphate coating is lower than that in original material [10].

Thermal oxidation is a simple method in treatment of material [11]. Thermal oxidation of magnesium alloy AZ91 results in oxide layer composing pile of oxide grains covered entirely of magnesium surface [12]. Thermal oxidation treatment changes mechanical properties of materials such as tensile strength, hardness, toughness. The hardness change occurred on the top surface to the inner materials. One of the methods to measure hardness of treated material by thermal oxidation is Vickers microhardness. Both excessive and lack of the load in Vickers microhardness testing will effect in data validity. The aim of this investigation is to achieve optimum load in Vickers microhardness testing of magnesium alloy AZ61 treated by oxidation thermal.

2. Method and Parameters

Magnesium alloy AZ61 specimen was prepared in 20×20×6 mm³ of dimension. There are five specimens for five temperatures (150; 225; 300; 375; 450 °C) in thermal oxidation. Specimens were prepared by polishing with abrasive paper until 1500 mesh. Thermal oxidation used Nabertherm heat treatment furnace which has 1200 °C of maximum temperature. Each specimen was annealed in this furnace for 6 hours at each temperature. Vickers microhardness test used Vickers microhardness machine (FM-800, Future-Tech Corp.). Load parameters were arranged at five loads (5, 10, 25, 50, and 100 g) for each specimen and five repetitions for each parameter. Vickers Hardness Number (VHN) for each load was calculated by average of 25 data and its standard deviation also calculated from 25 data. The optimum load was chosen at the least standard deviation.

3. Result Analysis

Vickers hardness and its standard deviation for each load at each thermal oxidation temperature are shown in Table 1. The first row demonstrate the loads from 5 to 100 g and the first column shows the thermal oxidation temperature form 150 to 450 °C. At 150 °C, the Hardness Vickers (HV) is 34.5 for 5 g of load. The HV increases with the load and achieves maximum HV at 42.4 for 25 of load, after that the HV decreases with load increase. At 225 °C, the HV decreases from 37.1 to 35.8 for 5 g to 10 g of load and increase until 47.6 at 50 g of load. For 300 °C of thermal oxidation, the HV increases as load increase and achieve the maximum HV at 38.0 for 100 g of load. The HV increases from 22.5 to 36.2 for 5 – 50 g of load at the 375 °C of thermal oxidation and decreases to 27.2 at 100 g of load. For the higher temperature of thermal oxidation (450 °C), the HV is inconsistent result. It increases from 5 to 10 g of load, decreases for 25 g of load, increases at 50 g of load and decreases again at 100 g of load.
Table 1. Vickers Hardness of magnesium alloy treated by thermal oxidation

| Temperature (°C) | Load : 5 g | Load : 10 g | Load : 25 g | Load : 50 g | Load : 100 g |
|------------------|------------|-------------|-------------|-------------|--------------|
|                  | HV (g.mm\(^{-2}\)) STD EV | HV (g.mm\(^{-2}\)) STD EV | HV (g.mm\(^{-2}\)) STD EV | HV (g.mm\(^{-2}\)) STD EV | HV (g.mm\(^{-2}\)) STD EV |
| 150              | 34.5 2.5 | 36.3 2.4 | 42.4 11.9 | 39.7 7.2 | 34.2 1.6 |
| 225              | 37.1 4.0 | 35.8 1.8 | 43.4 5.0 | 47.6 4.6 | 39.2 1.8 |
| 300              | 27.3 1.9 | 34.2 2.4 | 35.3 1.5 | 35.9 0.9 | 38.0 3.1 |
| 375              | 22.5 1.5 | 29.6 1.8 | 31.8 1.9 | 36.2 3.7 | 27.2 6.5 |
| 450              | 39.8 8.2 | 44.0 5.3 | 37.8 6.1 | 50.1 6.5 | 46.4 2.2 |
| Average          | 32.2 3.6 | 36.0 2.7 | 38.1 5.3 | 41.9 4.6 | 37.0 3.1 |

Figure 1 represents the HV of magnesium alloy treated by thermal oxidation for load variation. The HV for 5; 10; 25; 50; 100 g of load are 32.2 ± 3.6; 36.0 ± 2.7; 38.1 ± 5.3; 41.9 ± 4.6; 37.0 ± 3.1 respectively. The HV increases from 32.2 to 41.9 for 5 to 50 g of load and decreases at 100 g of load. Deviation standard of 25 g load is 5.3 and it is a highest standard deviation in this research. The lowest standard deviation is 2.7 at 10 g of load. The average HV for all loads is 37.0 and the standard deviation is 3.9.

Figure 1. Vickers microhardness of magnesium alloy treated by thermal oxidation

Vickers microhardness of magnesium alloy has variety value due to the load of Vickers microhardness testing. The optimum load is chosen base on the deviation standard of results. The least of standard deviation indicates more consistent of the value. The load of 10 g has the least standard deviation, therefore the optimum load in this Vickers microhardness testing is 10 g.

4. Conclusions

One of the Vickers microhardness test parameter is the load in indentation. Optimum load should be chosen to get better result in Vickers microhardness. The optimum load in this research is 10 g because of the least standard deviation in the Vickers Hardness. Higher standard deviation makes the results have less accuracy.
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