

Original Article

# Effect of Squeeze casting on Mechanical and Microstructure Properties of Magnesium Alloy AZ31

Tri C Wahyudi<sup>1,2</sup>, Tarkono<sup>2,\*</sup>, Irza Sukmana<sup>2</sup>, Fauzi Ibrahim<sup>3</sup>, Y Burhanuddin<sup>2</sup>, and Fethma M Nor<sup>4</sup>

<sup>1</sup> Department of Mechanical Engineering, University of Muhammadiyah Metro, Jl. Ki Hajar Dwantara 38 Banjarrejo, Metro 34124, Indonesia

<sup>2</sup> Department of Mechanical Engineering, Faculty of Engineering, Universitas Lampung, Gedung H – 2<sup>nd</sup> floor, Jl. Prof. Soemantri Brojonegoro No. 1, Bandar Lampung 35143, Indonesia

<sup>3</sup> Department of Mechanical Engineering, Faculty of Engineering, Universitas Malahayati, Jl. Pramuka No. 27, Bandar Lampung 35153, Indonesia

<sup>4</sup> School of Design, Universiti Teknologi Brunei, Gadong BE1410, Brunei Darussalam

\* Correspondence: tarkono70@gmail.com

Received: 18 March 2022; Accepted: 12 May 2022; Published: 30 June 2022

**Abstract.** The use of Magnesium and its alloys for industrial and medical devices has been increasing significantly in recent years. In the medical application, it grows along with the increasing cases of fractures that occur in bones, both accidents and other events, require a natural or artificial material that can interact with the human body's system to repair, restore and replacing damaged tissue or as a liaison for body tissues. Magnesium, as a biodegradable stent material, is an essential structural tissue in the human body's organs, has good mechanical properties, and can survive during the implantation process without showing failure. The sample in this study has been carried out with a squeeze casting process with pressure variations of 250 MPa, 350 MPa, 500 MPa, and 550 MPa at a temperature of 4000C for a pressing time of 1 minute, with a holding time of 5 minutes and argon gas pressure of 1 bar. The results showed that pressure variations significantly affect the hardness results; it can be seen that the higher the pressure, the greater the hardness value. The highest value is found at a pressure of 550 MPa at 51 HRV, and the highest maximum stress value is 128.26 MPa. This value is close to the tensile strength of the mechanical properties of the cortical and cancellous bone.

**Keywords:** *Lightweight materials, squeeze Casting, Magnesium AZ31, Semi-Solid.*

## 1. Introduction

The amount of damage in various cases of bone fractures, accidents and other events increases the need for implant materials to replace damaged functions and tissues. Due to a large number of cases, materials are needed, which are natural or artificial materials that can interact with the body system to repair, restore and replace damaged tissues or as a liaison for body tissues.

The material used for manufacturing until now is non-degradable, although this stainless material has advantages because it can provide maximum stability [1]. However, several shortcomings were found clinically, such as difficulties taking X-rays and MRI (magnetic resonance imaging) images and doing a second operation to remove implanted lightweight structural materials for aerospace. So that the use of stainless materials can also disturb and traumatize the patient again, and there are holes in lightweight structural materials for aerospace that can cause or have the potential for further bone fractures.

Magnesium as a biodegradable stent material is also based on a fixed structure network, an essential element in human organs. Magnesium is a substantial inter cation involved in more than 300 biological reactions of cells. Magnesium is also considered a non-carcinogenic element. The results of the implantation of stent material show that the material's mechanical properties can survive during the implantation process without showing failure [2].

The previous study [3] wrote that the mechanical properties of magnesium material are closer to the type of human cortical bone and can reduce stress levels. Mechanical properties of cortical bone at tensile strength between 50-150 MPa [4]. Many methods are used to manufacture bone bolts, including roller metal forming techniques, die casting, and squeeze casting. The squeeze casting process on aluminium can increase the surface hardness by 22%. The process temperature in pouring, time pressure and force influence the result of surface hardness. The material's microstructure matures as the pressure increases [5].

In manufacturing tools to support orthopaedic needs, knowing how the device can work correctly is necessary. Therefore, the mechanical properties of the material must be considered. The mechanical properties depend on several factors, such as the forces that will later act on them, the mechanical loads on these forces that will work internally in the material, and the ability of the material to withstand these loads over a long period. Owned by the implant. Magnesium is a light metal with a density of about 1.74 g/cm<sup>3</sup>, slightly lower than that of natural bone, which ranges from 1.8-2.1 g/cm<sup>3</sup>. The modulus of elasticity of pure Magnesium is 45 GPa. Human bone varies between 40 and 57 GPa. Due to the similarity in the elasticity of Magnesium applied to hard bone tissue, thereby reducing the possibility of surface tension, taking into account these mechanical properties, Magnesium is the best choice for biodegradable orthopaedic implants [6]. This study aims to determine the effect of manufacturing process parameters on the mechanical properties of Magnesium from squeeze casting.

## 2. Materials and Methods

### 2.1. Tools and materials

Tools and materials that will be used in this research include:

- The materials used are Magnesium AZ-31, and Aluminum AL-31, both resulting from casting. The primary alloy of AZ-31 is Aluminum (Al) 3% and Zinc 1%.
- The squeeze casting process is where the material that has been shaped is then put into a die, which has previously been heated, after approaching a semi-solid state and then pressed.
- Perform the tensile test, microhardness, microstructure, and SEM to see the test results.

### 4.2. Direct Squeeze Casting (DSC) Process

DSC is a process in which molten metal is cooled by applying direct pressure to prevent gas porosity and shrinkage in a material, as shown in Figure 1 below.

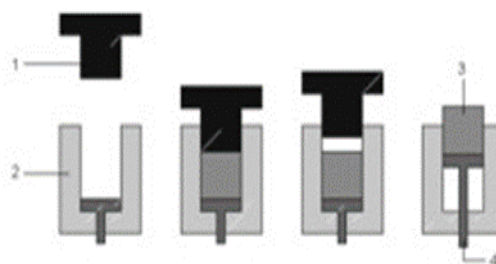


Figure 1. Mechanism of Direct Squeeze casting (DSC)

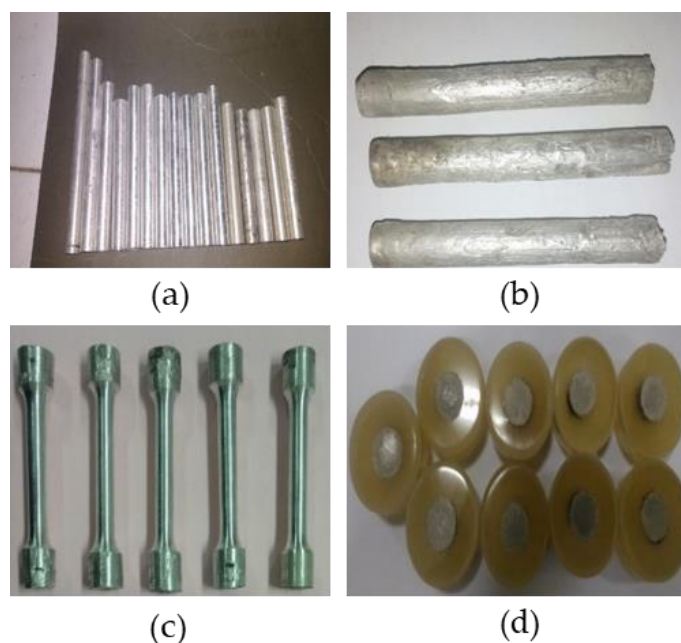
### 4.3. Squeeze Casting Parameters

The parameters used in this study, the sample used is a material that has been squeezed casting process with variations of each pressure, namely 250 MPa, 350 MPa, 500 MPa, and 550 MPa at a temperature of 4000C for a pressing time of 1 minute, with a holding time of 5 minutes and argon gas pressure of 1bar.

## 3. Results

### 3.1. Microhardness Result Data

In the research process for microhardness testing, the material used consists of material that has been squeezed casting process with variations of each pressure variation, and one sample without treatment is tested to compare the results of the material that has been squeeze casting processed which serves to find out how much the influence of temperature and pressure on the results of the hardness of the material that the process has carried out.



**Figure 2.** The flow of specimen making: (a) Initial material (b) Squeeze Casting Results (c) Tensile Test Specimen (d) Hardness Test Specimen.

### 3.2. Microhardness Test Results

In the research process for microhardness testing, the material used consists of material that has been squeezed casting process with variations; each pressure variation is 250 MPa, 350 MPa, 500 MPa, and 550 MPa, and one sample without treatment is tested to compare the results of the material that has been carried out by the squeeze casting process which serves to determine how much influence the temperature and pressure have on the hardness results. Material that has been processed. The data after the test is in table 1. The results of the microhardness test on the AZ31 material in the squeeze casting process.

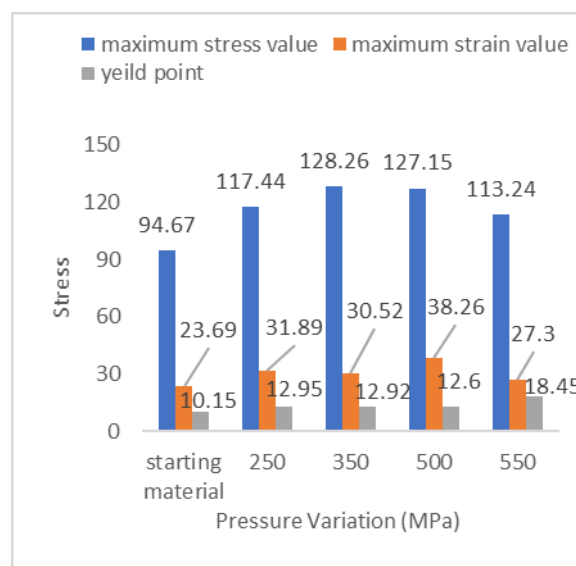
**Table 1.** Pressure variations on microhardness testing

Number of points	Pressure variation (MPa)				
	0	250	350	500	550
1	42	46	43	42	54
2	44	46	46	44	43
3	37	44	47	48	53
4	44	46	46	45	55
5	42	45	45	50	50
Average value	41,8	45,4	45,4	45,8	51

## 4. Discussions

### 4.1. Tensile Test Results on Pressure Variations

In the tensile test that has been carried out using AZ31 material which is processed by the squeeze casting method, varying the temperature with pressure, it produces data along with graphs, where in the process, the material is squeezed in semi-solid condition. The results will be compared with the material used. The same but without squeeze casting treatment, so that it can be seen how much influence occurs and obtain data that shows the quality of the material close to bone strength. The tensile test curve can be divided into three regions: region I, the area from the starting point to the yield point. It is the elastic region where the test material can return to its original shape when the tensile load acting on it is removed. Region II is the area from the yield limit to the maximum stress. The material undergoes homogeneous plastic deformation (universal deformation) and strain-hardening events in this area. And area III is the site from the point of ultimate tensile strength to the end of fracture. In this area, a localized deformation causes the specimen's necking (localized reduction). Results from a tensile test with variations in pressure where the test temperature is equalized, as in Fig. 3.



**Figure 3.** Graph of pressure variation tensile test results.

After testing and getting data according to the graph above, it can be seen that in the initial material, a tensile test was carried out with a maximum stress value of 94.63 MPa, this value is lower than the value for the pressure variation of 250 MPa of 117.44 MPa, and 127.15 MPa at a pressure of

500 MPa, after that at a pressure of 550 MPa there were 113.24 MPa. Still, at a pressure variation of 350 MPa, the resulting stress value was higher than other variations and materials without treatment, with 128.26 MPa.

The graph above shows that the higher the pressure applied in the squeeze casting process, the lower the stress value. That is because the more significant the pressing process, the lower the strength value of the maximum stress. Come out directly because the specimen is attached to the die wall, so it takes a long time, resulting in the material inside experiencing excessive temperatures; this affects the quality of the test results and can also be in the less precise lathe process so that when performing tensile testing the data that can be obtained is not optimal. However, the lowest value of the variation is still above the maximum stress value of the untreated material specimen.

It shows that there is an influence from the squeeze casting process for semi-solid material if it is carried out with pressure variations; from the data above the pressure variation, the highest maximum stress value can be obtained, namely, at a pressure variation of 350 MPa at a temperature of 4000C with a value of 128.26 MPa, this is the best value of some other variation values. After processing, the material from Magnesium has a higher compressive strength, and the strain value increases with the starting material even though the final value is different. It is the best value of several other variation values. After processing, the material from Magnesium has a higher compressive strength, and the strain value increases with the starting material even though the final value is different. It is the best value of several other variation values. After processing, the material from Magnesium has a higher compressive strength, and the strain value increases with the starting material even though the final value is different [7].

One of the strengths that are usually known from a tensile test result is the yield point. The yield point is the point at which the point's value changes from elastic deformation to plastic deformation. Most materials experience a gradual change in properties from elastic to plastic and the point at which plastic deformation begins. At a pressure of 250 MPa, the yield value is 12.95 MPa, and at a force of 350 MPa is 12.92 MPa. At a force of 500 MPa is 12.6 MPa. This value is higher than the yield value in the initial material sample, which is 10.15, but at a pressure of 550 MPa, the highest value is 18.45.

At pressure variations, the value of the yield point has a range of values that is not too far away and tends to be close to the same both from the initial material sample or the sample being varied; this is because the yield or ductility itself is the ability of a material to withstand a load at the time of penetration and will return to its original shape or without causing plastic deformation. This measurement is carried out to show the results that a material can be deformed without fracture in a process during formation.

Tensile test results for pressure variations where the strain results show fluctuating values, such as at a pressure of 550 MPa having a strain value of 0.2730%, the value is greater than the initial material sample value of 0.2361%. Still, at a pressure of 500 MPa, the strain value is higher, equal to 0.3055% and 0.3197% at a pressure of 250 MPa. From the pressure variation, the most considerable weight is found at a pressure of 500 MPa, 0.3820%. This fluctuating value change is seen because the strain is an extension, so the strain changes in length and cross-section during the test.

In general, the squeeze casting process that has been carried out shows good improvement results when compared to the initial material sample because it can be seen if the comparison value is that the highest stress value is at 350 MPa pressure of 128.26 MPa, this value is close to the tensile strength at thickness. 2, namely the mechanical properties of the cortical and cancellous bones [4], and the yield point value of the best value at a pressure of 500 MPa, which is 38.26. And the value at the yield point of pressure variation of 550 MPa is the highest value of all variations and initial materials. at 18.45 Mpa.

It is clear that the values obtained in the tensile test by varying this pressure have a decreased value, and the value that reduces occurs at high pressure. It may be caused by limited tools, the tools used during the testing process, as well as the lack of control processes or inadequate solution accidents (poor treatment solutions) so that the values obtained do not show results that are not

optimal for each variation. In general, the value of these variations increases compared to starting material specimen.

Based on the data obtained from the tensile test results, the stress and strain curves obtained from the initial Magnesium and magnesium material have been carried out in a semi-solid process with temperature variations of 250 MPa, 350 MPa, 500 MPa, and 550 MPa with a holding time of 5 minutes and pressing time 1 minute. From the equation of the specimen results shown in Figure 4, the maximum stress value of magnesium AZ31 After the semi-solid treatment decreased at a pressure of 550 MPa, the heating temperature and pressure and longer isothermal holding time resulted in the AZ31 specimen containing a eutectic mixture with a more significant volume fraction. AZ31 samples were processed to semi-solid with more eutectic points [8].

#### 4.2. Results Of Microhardness From The Effect Of Pressure Variations

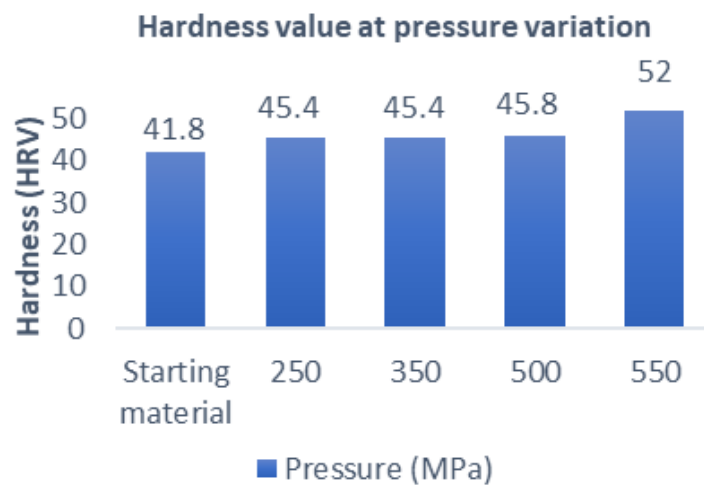


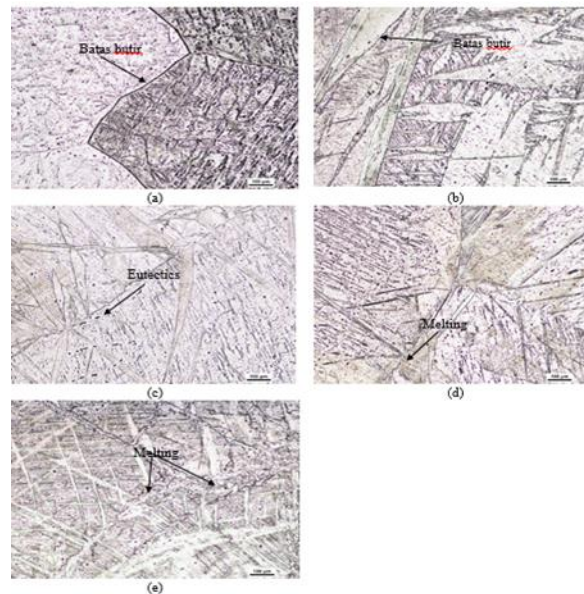
Figure 4. Graph of microhardness test results at 4000C temperature with pressure variations.

From the graph, it can be seen that with the pressure with the hardness of the sample, it can be seen that the greater the force applied, the higher the hardness value. Also, it can be seen that the hardness of the material sample that was tested for microhardness with variations in pressure showed an increase in hardness value compared to the material without treatment, with an average of 41.8 HRV increasing to 45.5 HRV at a pressure of 350 MPa and 500 MPa, then at a pressure of 500 MPa it increases but is not too significant; namely 45.8 HRV and the maximum point in this test is at a pressure of 550 MPa, namely with a hardness value of 51 HRV, it is possible that the higher the force used, the greater the hardness value. When associated with the wear mechanism, the higher the hardness value of a material, the more resistant the material is to the wear mechanism. Besides being determined by the hardness value, the selection of wear-resistant materials is also determined by the chemical composition, microstructure, and other variables. Likewise, when it is associated with the strength of the material, the hardness value is related to the strength of the material.

The hardness value is affected during pouring, temperature, pressure, and force [5]. This means the higher the hardness value of a material, the higher the material has high strength. When related to tensile strength, tensile stress and hardness can indicate a material's resistance to plastic deformation. The more complex the material, the greater the tensile strength. However, the more complex material is, the greater the tendency for that material to become brittle. It is because the movement of dislocations is minimal when loading is carried out on the material, so the plastic deformation is minimal. In contrast, the material tends to produce ductile values at low hardness values.

#### 4.3. Analysis of Metallographic Test Results Microstructure Testing and SEM

Microstructure describes a collection of phases that can be observed through metallographic techniques. The microstructure of a metal can be seen using a microscope. Microstructure testing using the micro-hardness tester with 100 m and 500 m magnification. The results of the tests that have been carried out from the sample testing of magnesium squeeze casting AZ31 with variations in temperature and pressure, while samples without treatment were tested for microstructure. To find out and compare the microstructure of the interpretation, whether the results have similarities or are their other elements. The test results are seen in Fig. 5.



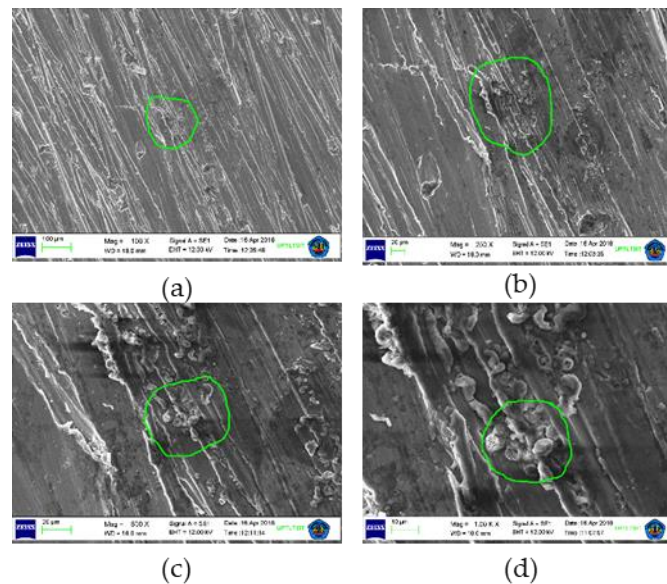
**Figure 5.** Microstructure of temperature 4000C with pressure variations, (a) starting material, (b) 250 MPa, (c) 350 MPa, (d) 500 MPa, (e) 550 MPa.

In the treatment in which the sample is in a solid state, the microstructure condition is not too much different from the microstructure at a pressure of 250 MPa, but there is a slight difference; at a pressure of 250 MPa in Figure 5 b, the grain boundaries have begun to decompose although this is not perfect. It can be caused by temperature and pressure, different in Figure 5 c, at a pressure of 350 MPa. The microstructure shows the eutectic phase, which occurs due to temperature treatment against stress so that the elements in the sample change to look neater and finely different from the previous one [8]. There are eutectic points, which are assumed to be Al and Mn elements. This variation is the pressure with the highest value because this condition is in a temperature close to semi-solid.

The sample's microstructure changes at a pressure condition of 500 MPa in Figure 5d. It is related to the increasing pressure, so it can be seen in some parts that experience a remelting phase at the grain boundaries. Although the mixed elements have not been thoroughly mixed, the changes in the features are visible in the grain boundaries. The pressure of 550 MPa is shown in Figure 5e, where the pressure process is the maximum pressure. This study shows that the microstructure undergoes a melting phase at the grain boundaries, and a more significant change in pressure causes a change in shape. Variations in pressure and temperature significantly affect the results of the microstructure in this test [9].

Holding time in the squeeze casting process also has an essential influence on the results of microstructures; when the holding time process is carried out quickly, it will produce a smooth melting point. The long or fast holding time affects the microstructure of the squeeze casting process [9]. The higher the temperature and pressure, the more melting the sample, as shown in Fig. 6. This may be because the high temperature has entered the melting phase, where the phase changes its

microstructure according to the magnesium phase diagram. In this study also, semi-solid treatment was able to increase the strain value on magnesium material but reduce the stress value of Magnesium [9,10].



**Figure 6.** SEM results at 4000C at a pressure of 550 Mpa, (a) Magnification of 100, (b) Magnification of 250, (c) Magnification of 500, and (d) Magnification of 1000.

The results of observations in this test show that at a temperature of 4000C at a pressure of 550 MPa, as shown in Figure 6a with 100 times magnification, the grain is not visible, just like scratches, as well as seen in Figure 6b at 250 times magnification, different at 500 times magnification in Figure 6c. There is a difference in the shape change, which can happen because, during the process, it is in the near-melting stage. It is evident on the surface of the 6d image that the shape or structure of the material changes to be more homogeneous. Thus, it may contribute to future applications concerning corrosion and fatigue behaviours of the material [10,11].

## 6. Conclusions

In this study, it can be concluded that the pressure variation dramatically affects the hardness level results. In this test, it can be seen that the higher the pressure, the greater the hardness value. The highest value is found at 550 MPa pressure of 51 HRV, and the highest maximum stress value is 128.26 MPa. This finding opens more applications of Magnesium alloys in the future. The squeeze casting process may improve the mechanical and corrosion properties of the materials, mainly for lightweight applications, including aerospace engineering and other applied sciences. Further detailed testing of fatigue and corrosion behaviours of the squeeze-casted samples may be conducted.

## References

- [1] Witte, F. Abeln, I. Switzer, E. Kaese, V. Meyer, LA Windhagen, H. 2007. EvaluationOf The Skin Sensitizing Potential Of Biodegradable Magnesium Alloys. J BiomedMater. Res A 2008;86:1041–1047.
- [2] Moravej, M. Diego, M. 2011. Biodegradable Metals for Cardiovascular Stent Application: Interests and New Opportunities.International Journal of Molecular Sciences.12.4250-4270.
- [3] Farraro, K F. Kim, K E. Woo, S L V. Flowers, J R. McCullough, B. 2014. Revolutionizing Orthopedic Biomaterial The Potential Of Biodegradable And Bioresorbable Magnesium – Based Materials For



- Functional Tissue Engineering. *J Biomech* June. 27. 47(9):1979-1986.
- [4] Hermanto, A. 2016. Utilization of Magnesium Machining Waste Materials for Biodegradable Bone Screw Applications Using Powder Metallurgy Methodology. Lampung University.
- [5] Taufikurrahman. Nukman. Yanis, M. 2013. Effect Of The Squeeze Process On The Hardness And Micro Structure of Recycled Aluminum Materials. *Journal of Mechanical Science And Engineering*. Vol.1 no.1 October.
- [6] Tian, P. Xuang, L. 2015. Surface Modification Of Biodegradable Magnesium And Its Alloy Alloys For Biomedical Applications. *Regenerative Biomaterials*.135-15
- [7] Paraskevas, D. Dadbakhsh, S. Vleugels, J. Kim, V. Dewulf, W. Duflou JR 2016. Solid State Recycling of Pure Mg and AZ31 Mg Machining Chips Via Spark Plasma Sintering. *Materials and Design*. 109. 520–529.
- [8] Men, Y. Fukushima, S. Sugiyama, S. Yanagimoto, J. 2015. Cold Formability Of AZ31 Wrought Magnesium Alloy Undergoing Semi-solid Spheroidization Treatment, *Materials Science & Engineering A624* 148-156.
- [9] Kleiner, S. Beffort, O. Wahlen, A. Uggowitzzer, P J. 2002. Microstructure and mechanical properties of squeeze cast and semi-solid cast of Mg–Al alloys. *Journal of Light Metals* 2. 277–280.
- [10] Yusop, A H M. Alsakkaf, A. Kadir, M R A. Sukmana, I. Nur, H. 2021. Corrosion of porous Mg and Fe scaffolds: a review of mechanical and biocompatibility responses. *Corrosion Engineering Science and Technology*. 56(4), pp. 310–326.
- [11] Sukmana, I. Savetlana, S. Burhanudin, Y. Wicaksono, W A. and Nur, H. 2019. Fabricating and Testing of Porous Magnesium Through Powder Metallurgy Technique using TWSH (Titanium Wire Space Holder) for Biodegradable Bone Scaffold Material. *Journal of Engineering and Scientific Research*, vol. 1 issue 2, pp. 78-83.



This open-access article is distributed under the terms of the Creative Commons Attribution 4.0 International License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.