Neutron diffraction and the residual stress distribution of magnesium processed by equal channel angular pressing

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Abstract. Neutron diffraction and the residual stress distribution of magnesium processes by equal channel angular pressing was investigated in term of the grain refinement process. Magnesium is one of the metallic material for a biomedical implant due to the biodegradable properties. The structure and strength of biodegradable metallic material are quite essential to discuss for biomedical implant purposes. The ultrafine-grained structure of magnesium was prepared by equal channel angular pressing until four passes by route Bc at 523 K. The specimen was characterized by electron backscattering diffraction for the structure and neutron diffraction for residual stress. The grain refinement happened during the equal channel angular pressing on magnesium. The texture after four passes of ECAP shows a random orientation compare to coarse grain. The result shows that the strength of the ultrafine grain structure is lower than the coarse grain structure due to the crystal structure of magnesium as hexagonal. The other reason is that the distribution of residual stress on ultrafine grain bulk specimens, which measured by neutron scattering facilities.

1. Introduction

The implant material requirements, in general, must have biocompatible, bioactive properties, excellent mechanical properties within a specified period until tissue healing occurs, and have biodegradation rates that are in accordance with the speed of tissue healing [1], [2], [3], [4], [5], [6]. Metal implants that are commonly used are stainless steel [7], titanium [8], cobalt alloys [7], and polymer-based resorbable materials. Each of these materials has advantages and disadvantages as an implant material [6 – 14]. In metal materials such as stainless steel, titanium, and cobalt alloys have excellent mechanical properties and corrosion resistance. However, after the healing process is complete, the implant is no longer needed, and the implant will continue to be in the body so that secondary surgery is needed for the removal of the implant. While the bioresorbable polymer material is able to be absorbed by body tissues in the time needed when it is no longer needed, but its mechanical properties are less satisfactory.

One metal material that is being developed for use as an implant material is magnesium and its alloys. That is because magnesium has several advantages, namely it has biodegradable properties (can decompose without causing toxins to the body) so that when the implant is no longer needed the material can decay naturally, bioresorbable (able to be absorbed by body tissue), is a lightweight
metal, has a density which is closer to the bone, has a higher fracture toughness compared to ceramic biomaterials, and has a modulus of elasticity that approaches the bone so as to prevent the effects of stress shielding.

Magnesium also has several disadvantages as an implant material, including the rapid rate of corrosion or degradation, which causes the magnesium implant and its alloy to lose its mechanical strength before the hard tissue undergoes a healing process [1, 4, 11, 15 – 17]. Therefore, we need a way to control the speed of biodegradation so that it can be adjusted to the speed of healing of body tissues. Some research that has been done to overcome these deficiencies is by the grain refinement method. One method of grain refinement that gets a lot of attention is equal channel angular pressing (ECAP).

ECAP is a simple and inexpensive method of reducing grain size, resulting in an increase in the mechanical strength of magnesium [18 – 25]. The ECAP process is a method of pressing material through a die in an angular channel, and because of its fixed cross-sectional dimensions when pressed through the die, then repetitive pressing will produce a specimen with a very high voltage.

The characterization of bio-implant materials by nuclear techniques, such as the neutron scattering technique, which is a complement to other similar techniques has several advantages because of the neutron characteristics when interacting with the atoms of the material. This technique has the ability to reveal the properties of advanced materials. Neutron scattering can be used to investigate material properties such as bulk nanostructured metal.

2. Experimental procedure

Pure magnesium 99.9% was processed by equal channel angular pressing (ECAP) at 523 K. ECAP die has \( \phi = 110^\circ \) and \( \psi = 20^\circ \). Pressing speed was used at 5 mm/min by route Bc ECAP. The structure was characterized by FE-SEM (JEOL 7001) with accompanying the electron backscatter diffraction. Tensile experiments were conducted in a Universal testing machine (Autograph AGS-10k ND, SHIMAZU), under displacement control. The specimen for the tensile test was 3 mm in gage length, 1 mm in gage width and 1 mm in thickness. In neutron diffraction, the residual stress is obtained from the strain measurement results on the axis, therefore ideally the diffraction angle in the sample is made close to 90 \(^\circ\), and the shape of the slit beam comes and the diffraction beam is rectangular.

3. Result and discussion

![Figure 1](image_url)

**Figure 1.** Orientation image by EBSD of as received with (a) normal plane, (b) rolling plane, (c) transversal plan, and four passes ECAP (d) normal plane, (e) rolling plane and (f) transversal plane.
Biomaterials are all materials used to replace or improve the function of body tissues, either continuously or in contact with bodily fluids [1], [3], [6], [26], [27]. Biomaterials are widely used in the medical field both in medicine and dentistry. Biomaterials can come from natural or synthetic. The purpose of using this biomaterial is to improve the quality of a person's life so as to achieve a better level of health.

The material can be used as a biomaterial must have several requirements, the main and most important thing is that the biomaterial must be biocompatible, this biomaterial must not show an adverse response from the body, must be non-toxic and non-carcinogenic. This requirement eliminates the many technical materials that can be used. In addition, biomaterials must have sufficient physical and mechanical properties to function as substitutes or multipliers of body tissues.

This research was conducted to assess the biodegradation process of ECAP magnesium as a material that has the potential to be used as a biomaterial for bone implants in the field of maxillofacial oral surgery. By going through the ECAP process, the material will decrease in grain size so that its physical properties will increase, including corrosion resistance [28]. The ECAP process in magnesium material will produce a good and homogeneous grain structure with an average grain size of fewer than 100 μm. Magnesium that has been through the ECAP process will have a better corrosion resistance when compared with magnesium produced from other processing methods. That a mechanical process such as ECAP can reduce grain size significantly and is an effective way to reduce the extensive corrosion of magnesium as a biodegradable implant material. Another advantage of this method is that it increases the mechanical properties and fatigue resistance which is also needed for implant material.

Figure 2. The texture of (a) as-received and (b) four passes ECAP by EBSD.

Orientation image by EBSD of as received and four passes ECAP shown in Figure 1, regarding the grain refinement and orientation of the grain. Magnesium has hexagonal closed packed (hcp) with many preferred [0001] orientations in normal and transversal plane, as seen in Figure 1. However, the four passes ECAP also exhibited more preferred [001] orientation.

The texture of as-received and four passes ECAP was observed by EBSD in terms grain refinement, as seen in Figure 2. After the ECAP process in the sample, orientation exhibited a random structure compare to the coarse grain structure which is focused on the transversal plane. The intensity of misorientation becomes higher after the ECAP process due to severe deformation in the magnesium sample. The macrotexture also random along a transverse plane which is similar to texture as seen in Figure 3.

The nature of a metal material that will be used as an implant material needs to be well known so that the selection of the material is appropriate to the needs of its use. Tensile testing is the basis for testing and studying a material about the strength of the material because this test is easy to do,
resulting in a uniform stress on the cross-section, and most materials are easier to pull than a compressive test, so that in engineering, the strength test of a material is most often expressed by tensile test. The nominal stress-strain curve shows the ultimate tensile strength decrease and ductility decrease, as seen in Figure 4. The general explanation can be used for those phenomena, such as the crystal structure of the magnesium sample contributed to the difficulty for grain refinement and misorientation process. The ECAP process in the magnesium needs a higher temperature than other materials such as fcc or bcc metallic materials. The others reason may be related to residual stress generated during the ECAP process. Because of this reason, the residual stress measurement needs to conduct for inspecting the reason why the ultimate tensile strength decrease.

![Figure 3](image1.png)

**Figure 3.** The macrotexture of (a) as-received and (b) four passes ECAP by EBSD.

![Figure 4](image2.png)

**Figure 4.** The nominal stress-strain of as received and four passes ECAP.
Figure 5 shows two diffraction peaks with every 10° of omega angle. This result shows peak broadening and higher intensity. It means the results of diffraction depend on the deformation plane of the materials. By this explanation, it would be given the different values of residual stress. One technique to measure it using neutron scattering. Three direction measurements need to establish such as radial, axial and hoop. Residual stress in the center shows slightly low compared to in the edge area as seen in Figure 6. The result of radial, axial and hoop value by the intensity of diffraction is shown in Figure 6, can be converted into a residual stress distribution by the distance from the center. This experiment result is a preliminary study of how the neutron scattering measures the residual stress on the ECAP sample.

![Diffraction pattern of four passes ECAP sample by neutron scattering.](image1)

**Figure 5.** Diffraction pattern of four passes ECAP sample by neutron scattering.

![The value of radial, axial and hoop during residual stress by neutron scattering.](image2)

**Figure 6.** The value of radial, axial and hoop during residual stress by neutron scattering.

In this study, there are still many shortcomings that allow further research to produce a plate and screw material. This research is also still an initial study of the mechanical properties of magnesium.
ECAP based on tensile tests. The drawback of this study is that research is still being done in vitro, the research sample is still in the form of basic material and the mechanical properties obtained only describe the material properties in general.

4. Conclusion

Grain refinement is an effective method for improving the mechanical properties and corrosion resistance of magnesium. Previous studies have shown that grain size of the α-Mg matrix significantly influences the corrosion resistance of magnesium and that grain boundary to have higher energy and provide a favorable location for grain formation, which then benefits the formation of protective oxide films. The coarse grain exhibited higher strength than ultrafine-grained structure material, due to crystal structure and residual stress. Neutron scattering can be used identified perfectly the residual stress value without destructing the sample.

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