Mechanical Properties and Microstructure of Mg-1.6Gd Alloys Reduction 80% Hot Rolling as Implant Materials

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Abstract. Mg-Gd alloys is of great interest for many potential application especially in implant material. Higher mechanical properties is always the goal pursued by researchers for the materials which generally have relatively still low strength, low ductile and low hardness. From this aspect, the present paper reviews the recent report of a kind of Mg-Gd rolled which have been able to achieve strength compared with Mg-Gd casting alloys. The aim of this paper is to investigate the mechanical properties and microstructure for Mg-Gd alloys that are rolled at high tempetaure with severe reduction. Mg-Gd rolled with 80% reduction will be applied by unidirectional and cross direction with purpose to find varian characteristic. The Temperature range are 400, 450, 500 and 550 °C. The results of this experiment show that the highest hardness number obtained by single rolling specimen as 64.86 VHN at 550 °C. The highest ultimate tensile strength numbers obtained by cross rolling with 163.2 MPa at 550 °C. For the grain size, single rolling specimen has the smallest number with 46.33 µm at 500 °C. Overall, temperature 500-550 °C give a big impact to improve Mg-1.6Gd alloys.

1. Introduction
Performance of Mg alloys is very significant to develop as biomaterial application. Moreover, it well known that the rare earth (RE) are known to offer excellent much properties. Magnesium-rare earth alloys have been a great developed that the alloys could significantly improve the strength and corrosion resistance. Recently, it has been reported that addition Gd is effective for improving mechanical properties by solid solution strengthening and precipitate strengthening. Mg5(Gd, Y) alloys have observed at temperature of 480 °C for 4 hours shows the only some Mg3(Gd 1.26 Y 0.74) particles [1]. Cizek et al also reported that formation of the metastable β’ phase of Mg-15 alloys causes a strong hardening and the peak hardening occur at remarkable lower temperature. This research also obtained that the number of precipitation on the grain caused by various thickness reduction [2]. Janacek et al have investigated on the Mg-22 alloys that the precipitation of stable Mg5Gd phase and the phase have higher hardness than magnesium matrix. The Mg-22Gd also result the grain refinement and lot of twinning after the alloys was processed by higher pressure torsion (HPT) a room temperature and the pressure of 2 Gpa [3]. Mg-20Gd sample by casting technique resulted as-cast ingot mainly contain α-Mg solid solution and Mg5Gd phase as represented by Peng et al. Tensile strength of Mg-20Gd at room temperature 254 Mpa and elongation at less than 2 %. Meanwhile, the fracture surface of the as-cast alloys is typical cleavage fracture [4, 5]. Kubasek et al explained the structures of the as-cast Mg-5Gd alloys were mostly by fine α-Mg dendrites and the formation of Mg5Gd phase. The Mg-Gd alloys was significantly harmfull effect on corrosion resistance [6]. It well known that Gd has a wide range of alloy composition and heat treatment by its large solubility 0f 23.49 wt% at eutectic temperature for changing of mechanical properties. Following that, Hort et al observed that
the different addition of Gd and heat treatment resulted also various mechanical properties and corrosion behaviour which can be used for many applications. Beside the addition of Gd in magnesium, furthermore, thermo-mechanical process can also enhance mechanical properties and corrosion resistance. They reported that the addition of Gd above 10 wt% can actually improve strength but another side, the much more Gd elements in magnesium can decrease the elongation. Therefore, some researchers have recommended by the small addition of gadolinium on magnesium by thermo-mechanical process which it exhibited excellent mechanical properties. Susanti et al reported that the hot extrusion of Mg-1.6Gd alloys revealed fine grain size after hot extrusion on temperature recrystallization. The increase of mechanical properties on the Mg-1.6Gd alloys compared as-cast condition. Although, the strength and hardness of the alloys decreased as the extrusion temperature increased [7]. Another thermo-mechanical process also carried out on Mg-1.6Gd alloys by hot rolling unidirectional rolling (UR) and cross rolling (CR) with 95 % reduction. The mechanical properties greatly exhibited by refinement grain size [8, 9].

It is the purpose of this paper, another severe hot rolling processing in term of UR and CR with a total reduction of 80 % is connected to Kg-1.6Gd ( wt %) alloys. The mechanical properties of the different direction of the alloys will be investigated and discussed.

2. Materials and methods

Samples of hot rolling were prepared on Mg-1.6Gd alloys ingot with two methods : Unidirectional (UR) and Cross rolling (CR) with dimension of 50 mm x 25 mm x 10 mm as reported by paper before [8]. Before experiment, the alloys ingots were homogenized for 5 hours at a temperature of 560 °C. The temperature of the hot rolled sample was measured and monitored with a thermocouple. Hot rolling was carried out in a temperature range from 400 to 550 °C and held for 15 min before pass rolling. A total reduction of 80 % at 40% per pass (for two times) with speed of 10 mm min\(^{-1}\) which resulted in final rolled sheet with at least 3 mm in thickness. The hardness of rolling specimens were prepared with 10 mm x10 mm size and determined from 10 individual measurements, each using a Vickers Hardness testers with 300 g load for a dwellingtime of 15 s. Flat tensile specimens were machined from the hot rolled material in accordance with the ASTM E8-04 standard. The tensile specimens having a gauge length of 24 mm n the plate were cut from the sheet with tensile axes lying parallel to the direction. Tensile tests were carried out with an uniaxial tensile testing machine (Gotech AI-7000 LA 10) at room temperature. Microstructure test will be examined by standard metallography procedure. An etching solution for microstructure test was 4 mL HNO\(_3\) + 100 mL ethanol. Furthermore, the fracture surface morphology of the rolling samples were observed by scanning electron microscopy (SEM).

3. Results and discussion

The grain size number showed in figure 1. Generally, the grain size of unidirectional rolling has smaller grain size than cross rolling specimen. The smallest grain size of UR specimen is about 46.33 µm at 500 °C while for cross rolling is about 54.27 µm at 500 °C. Meanwhile, the largest grain size for UR specimen is about 65.63 µm at 400 °C and 65.63 µm for CR specimen at 450 °C. As shown in Figure 1, the grain size decline significantly at 450 °C for UR specimen and 500 °C for CR specimen. At those temperature, the hot rolling process give a big impact for grain size as refinement microstructure. The grains size increase at temperature 550 °C which the UR specimen has 56.31 µm and the CR specimen has 52.47 µm.
Figure 1. The grain of the hot rolled Mg-1.6Gd alloys for Ur and CR.

Figure 2 and figure 3 show the microstructure of the hot rolled Mg-1.6Gd alloys for UR and CR specimen. The UR specimen has the shape of long grain while CR specimen has an equiaxed grain. Precipitates occur on the hot rolled specimen. However, it were only found on CR specimen. The there are precipitates in grain where it was also explained by Hort et al. (2010) as Mg5Gd. Both of grain size and shape are very important which can result the good mechanical properties for Mg-1.6Gd alloys.

Figure 2. Microstructure of the hot rolled Mg-1.6Gd Alloys at various temperature with reduction of 80%: a) 400°C, b) 450°C, c) 500°C, d) 550°C of Cross Rolling (CR)
Figure 3. Microstructure of hot rolled Mg-1.6Gd Alloys at various temperature with reduction of 80% : a) 400°C, b) 450°C, c) 500°C, d) 550°C of Unidirectional Rolling (UR)

Figure 4(a) showed the hardness number for both of UR and CR specimen. The hardness increases as temperature increases where the highest hardness of UR is about of 58.02 VHN and 64.86 VHN for CR at temperature 550 °C. UR has bigger number than CR because UR specimen has smaller grain size compared to CR specimen. It well known that the grains size are contributed to mechanical properties of material. Both of the UR and CR specimen are better than pure magnesium that it has the hardness of about 30 VHN. Figure 4(b) shows the ultimate strength for both of UR and CR specimen. The highest strength for UR is 159 MPa at temperature of 550 °C and the lowest is 121 at temperature of 400 °C. In the other hand, CR has the highest strength about of 163 MPa and the lowest is 126 MPa at 550 °C and 400 °C, respectively. The ultimate strength increases as temperature increases of the hot rolled Mg-1.6Gd alloys. CR is influenced by precipitates and the shape of grain, so that the strength values bigger than UR. Precipitates in the grain can hold the movement of atomic and then the dislocation is so hard to move. Figure 4(c) shows the yield strength for both of UR and CR specimen. As shown in Figure, the yield strength increases as rolling temperature increases. The yield strength of CR is bigger compared to the UR. The largest yield strength for CR is 138 MPa while the UR is 135 MPa. This result is related to previous discussion that CR has bigger ultimate strength than UR. It caused by load that can be received by CR specimen. The yield strength value also indicated the properties of material. In this case, CR specimen is more elastic than UR. Figure 4(d) shows the elongation for both of UR and CR specimen. Generally, it shows that the CR elongation is bigger than UR. CR has elongation of 9.8% and 5.4% as the lowest elongation value. Meanwhile, UR has the highest elongation of 6.7% and 2.9 % for the lowest one. This result affected by the grain size of material where CR specimen has the smallest grain size compared to UR. It proves that the grain size of material can affect the mechanical properties.
Figure 4. Various mechanical properties of Mg-1.6Gd alloys: a.) Hardness, b.) Ultimate Tensile Strength (UTS), c.) Yield Strength (YS) and d.) Elongation.

4. Conclusion
The mechanical properties of Mg-1.6Gd alloys can be exhibited by hot rolling at temperatures in the range of their crystallization temperature. The finemest microstructure is obtained at low temperature and the grain size increase at higher temperature. Formation of precipitates occur at cross rolling which are related with mechanical properties. The strength and yield strength of the CR has higher than unidirectional rolling of about 163 MPa and 138 MPa, respectively. Meanwhile, the hardness of UR specimens are generally higher than CR due to the small grain size. Overall, the addition of Gd on magnesium alloys could give the improvement of mechanical properties as material implant.

Acknowledgments
This study is supported by the Directorate of Research and Community Service, Universitas Andalas and through, Hibah Riset Fakultas Teknik 2020.

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