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EFFECT OF COLD ROLLING AND ANNEALING TEMPERATURE ON THE RECRYSTALLIZATION AND MECHANICAL PROPERTIES OF Al-4.7Zn-1.8Mg (wt. %) ALLOY FABRICATED BY SQUEEZE CASTING

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ABSTRACT

Aluminium alloys are developed as airplane body due to their lighter weight compared to steel and good formability. Aluminium 7XXX series with Zn and Mg alloying elements is commonly used because its mechanical properties can be improved through a deformation process. A deformation process such as cold rolling may increase the hardness of an alloy through strain hardening. An annealing process following the deformation process will recover ductility through stress relief, recrystallization, and grain growth mechanisms. This research aimed to discover the effect of cold rolling and annealing temperature on the recrystallization and mechanical properties of Al-4.7Zn-1.8Mg (wt. %) alloy. The alloy was produced by a squeeze casting process. Homogenization was conducted at 400^{o}C for 4 hours followed by cold rolling with degrees of deformation of 5%, 10%, and 20%. The samples with 20% deformation were then annealed at 300^{o}C, 400^{o}C, and 500^{o}C for 2 h. The Vickers hardness test was performed on the cold-rolled and annealed samples to reveal the strain hardening effect and subsequent recrystallization process. The microstructure was observed using an optical microscope and a Scanning Electron Microscope (SEM). The results showed that the higher the deformation, the more elongated the grains. Deformation of 5, 10 and 20% led to grain shape ratios of 2.19, 3.19 and 4.59, respectively and increase in the hardness of the alloy from 69.5 VHN to 95.3, 100.1 and 105.4 VHN, respectively. Slip bands and cross slips were found only in the 20% deformed samples. The annealing process resulted in recovery at 300^{o}C, followed by recrystallization at 400^{o}C (d_{grain} ~290 μm) and grain growth at 500^{o}C (d_{grain} ~434 μm). Annealing temperatures of 300^{o}C, 400^{o}C and 500^{o}C decreased the hardness of the alloy from 105.4 VHN to 71.5, 96.8 and 95.3 VHN, respectively.

Keywords: Al-Zn-Mg alloy; Annealing; Cold rolling; Grain growth; Recrystallization

1. INTRODUCTION

Aluminium 7XXX series with zinc and magnesium as alloying elements is commonly used in the airplane industry because its mechanical properties can be improved through a deformation process. The high solubility of zinc and magnesium in aluminium, 83.1 and 17.4 wt.% respectively, yields significant effect on the mechanical properties and characteristic of the alloys (Zolotorevsky et al., 2007). The zinc in aluminium 7XXX series strengthens the alloy by forming a solid solution. Sofyan et al. (2012) found in silicon carbide reinforced Al-xZn-6Mg composites, that the increase in zinc refines the dendrite structure and, together with magnesium, forms solid solutions.

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Agrawal et al. (2012) also found that the addition of magnesium to Al-4Zn (wt.%) increased the mechanical properties by solid solution strengthening.

Cold rolling and subsequent annealing are common processes for aluminium alloys. Deformation during cold rolling changes the microstructure and mechanical properties. For aluminium that has an FCC crystal structure, the deformation process is interesting because of its dependency on Stacking Fault Energy (SFE). Aluminium alloys have a higher SFE than other FCC metals such as brass and copper, which results in cross slip as a common deformation mechanism (Wang et al., 2015). Mohamed et al. (2016) found that a deformation rate of Al 7075 was sensitive to maturation time periods when conducted at 45^o to the rolling direction. An annealing process after cold rolling will lead to recovery, recrystallization, and grain growth.

An increase in the deformation degree was found to refine the grain size after the annealing process (Lee et al., 2014). This was due to the higher deformation increasing the nucleation rate during recrystallization so that finer grains were produced. Wang et al. (2015) studied the mechanical properties and behavior of AA502 after cold rolling. Their results indicated that the strength of the alloy increased with greater rolling reduction. This was due to an increase in dislocation density and the formation of a fiber microstructure that interfered with the dislocation movement. Ying et al. (2013) found that the addition of Sc and Zr to Al-5Zn-2Mg (wt.%), combined with a 75% rolling reduction, increased the recrystallization temperature. The thermal stability of the minor alloying elements increased the driving force for recrystallization to occur.

This research studied the change in microstructures and hardness of Al-4.7Zn-1.8Mg (wt.%) alloy after various degrees of cold rolling followed by an annealing process at different temperatures. The aim was to learn how the cold rolling reduction affected the deformation mechanism in aluminium alloy and how the annealing temperature affected the recrystallization process to improve its mechanical properties.

2. EXPERIMENTAL METHOD

Pure Al, Zn, and Mg ingots were used as the starting materials. The ingots were melted in an electric furnace at 850^{o}C and degassed with argon at a flow of 5 l/min for 2 min. The molten alloy was then poured into a 300^{o}C preheated metal mold and squeezed at a pressure of 76 MPa for 10 min. The nominal composition of the alloy is listed in Table 1. The as-cast alloy was homogenized at 400^{o}C for 4 h and then cold-rolled with degrees of deformation of 5%, 10%, and 20% in multiple passes. The samples with 20% deformation were then annealed at 300^{o}C, 400^{o}C, and 500^{o}C for 2 h in a muffle furnace.

Table 1 The chemical composition of the Al-4.7Zn-1.8 Mg (wt.%) alloy

Zn	Mg	Fe	Si	Cr	Cu	Mn	Ni	Ti	Al
4.737	1.790	0.150	0.043	0.020	0.004	0.004	0.009	0.005	Bal.

The microstructures were observed using an optical microscope and a Scanning Electron Microscope (SEM) with a secondary detector and a working distance of 9.5–10 mm. Standard metallographic preparation was conducted with a Keller's etching of 2.5 ml HNO_3 + 1 ml HCl + 1.5 ml HF + 95 ml distilled water. A Vickers hardness test was performed according to ASTM E384. Five indentations were made for each measurement.

3. RESULTS AND DISCUSSION

3.1. Deformation Process

The microstructures of the as-cast and as-homogenized alloy are shown in Figure 1. The as-cast microstructure has typical dendritic structures with its α(Al) matrix and interdendritic areas filled with Mg3Zn3Al2 and Mg5Al8 intermetallic phases (Raghavan et al., 2010). The homogenization process led to diffusion of the interdendritic phase into the aluminium matrix, which led to the dendritic structures becoming more globular. This was followed by an increase in secondary dendrite arm spacing (SDAS) from 31.08 µm in the as-cast condition to 35.06 µm in the as-homogenized condition. It also decreased the hardness of the alloy from 94.5 to 69.5 VHN (Figure 2).

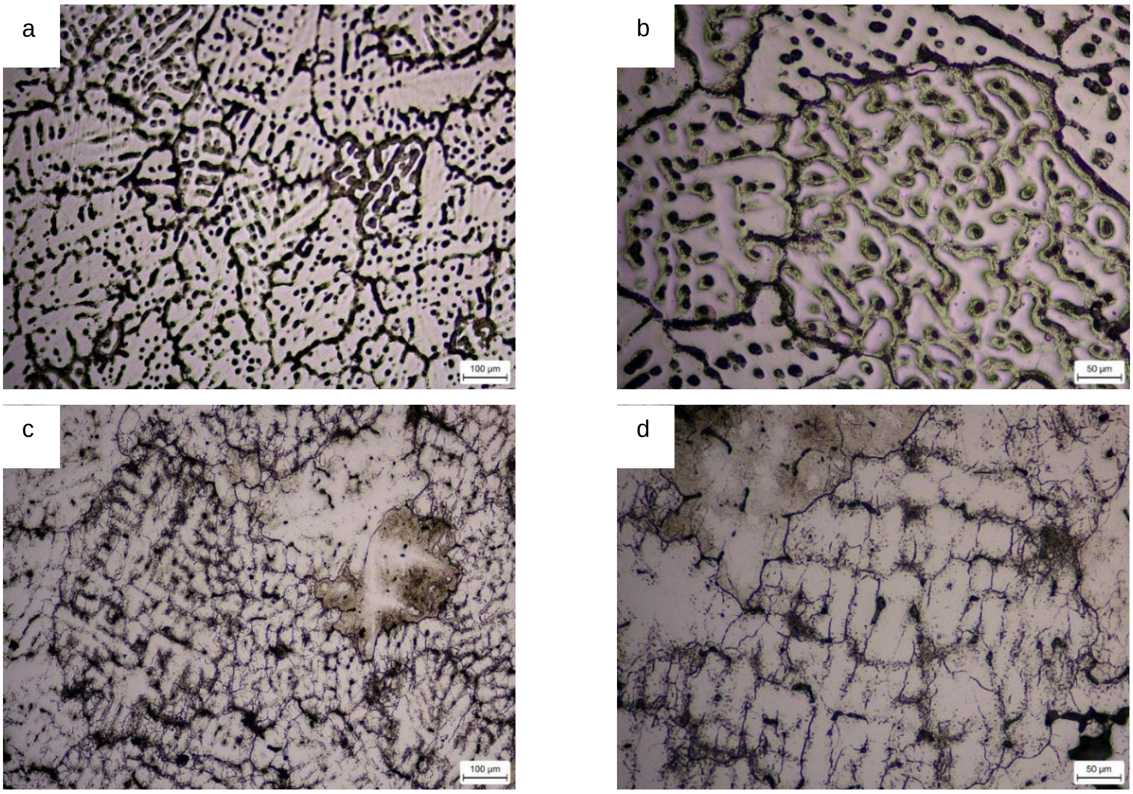

Figure 1 Microstructures of Al-4.7Zn-1.8Mg (wt.%) in (a-b) as-cast, and (c-d) as-homogenized condition

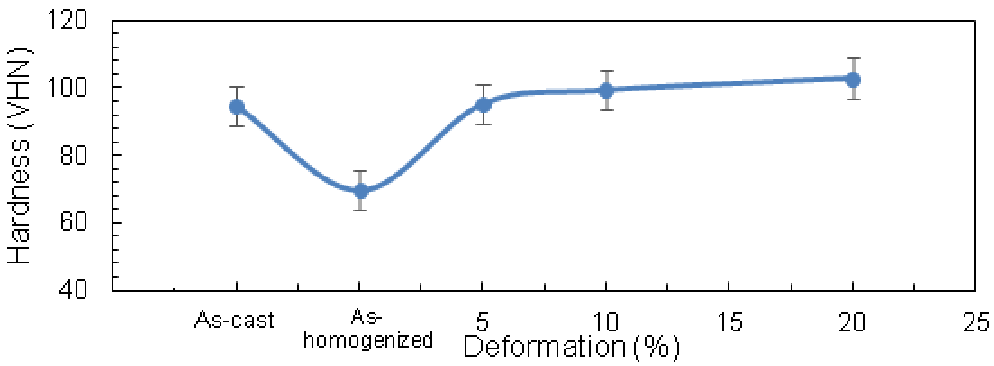

Figure 2 Changes in the hardness of Al-4.7Zn-1.8Mg (wt. %) alloy after the homogenization and deformation process

Figure 3 presents the microstructures after different degrees of cold rolling. The structure remained dendritic, but typical strained structures were observed with grains elongated along the rolling directions. It is clear that the higher the deformation level, the more elongated the grains. The grain deformation ratios were measured at 2.19, 3.19, and 4.59 for rolling reductions of 5%, 10%, and 20%, respectively. It is noteworthy that the microstructures after cold rolling of 5% and 10% (Figures 3b and 3c) do not show any slip bands, while the 20% deformed sample (Figure 3d) shows slip bands and a small number of cross slips (as indicated by the arrows). This implies that the amount of dislocation movement in the slip system after the 5% and 10% cold rolling is not dense enough for slip bands to occur. Only after the 20% deformation is the dislocation dense enough to form slip bands and cross slips (Rao et al., 2014).

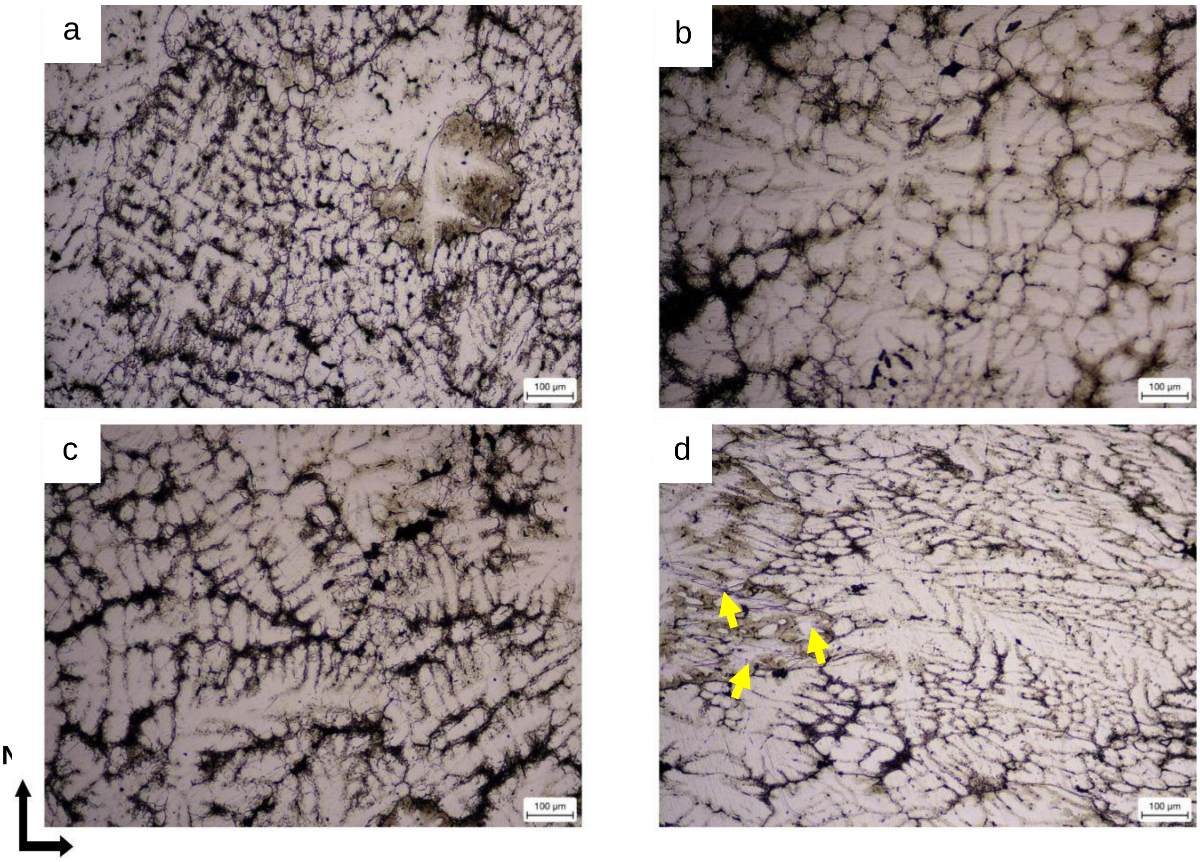

Figure 3 Microstructure of Al-4.7Zn-1.8Mg (wt.%) alloy in an (a) as-homogenized condition and after cold rolling with reductions of (b) 5%; (c) 10%; and (d) 20%

The hardness of the alloy increased rapidly in line with the increases in deformation degree (Figure 2), indicating the occurrence of strain hardening. The hardness changed significantly from 69 VHN to 95.3 VHN when the deformation was applied from 0% (as-homogenized) to 5%. However, further deformation to 10% only slightly changed the hardness to 99.4 VHN. This minimal change in hardness may have been caused by the inhomogenized distribution of the deformation. When 20% deformation was applied, the hardness increased to 102.9 VHN. The high hardness related to significant strain hardening, as shown by the heavily deformed grains, slip bands, and cross slip in the microstructure (Yvind et al., 2006).

3.2. Annealing Process

Figure 4 shows the microstructures of 20% rolled samples after annealing at different temperatures. After annealing at 300^{o}C (Figure 4b), there are no significant changes in the microstructure; the elongated dendrites still exist, but the secondary arms are eliminated due to the diffusion of the intermetallic phase into the α matrix. The recovery stage may thus take place at this annealing temperature. Increasing the annealing temperature to 400°C resulted in recrystallization, at which point the grains are now equiaxed with an average size of 290 μm (Figure 4c). It is interesting to note that new stress-free grains were mainly formed around the dendrite boundaries and slip bands. Grain growth was observed at 500°C with an average grain size of 434 μm. These results are related to those of a previous study by Ying et al. (2013), whereby recrystallizations of the Al 7XXX series were observed after cold rolling and annealing at 350–450°C.

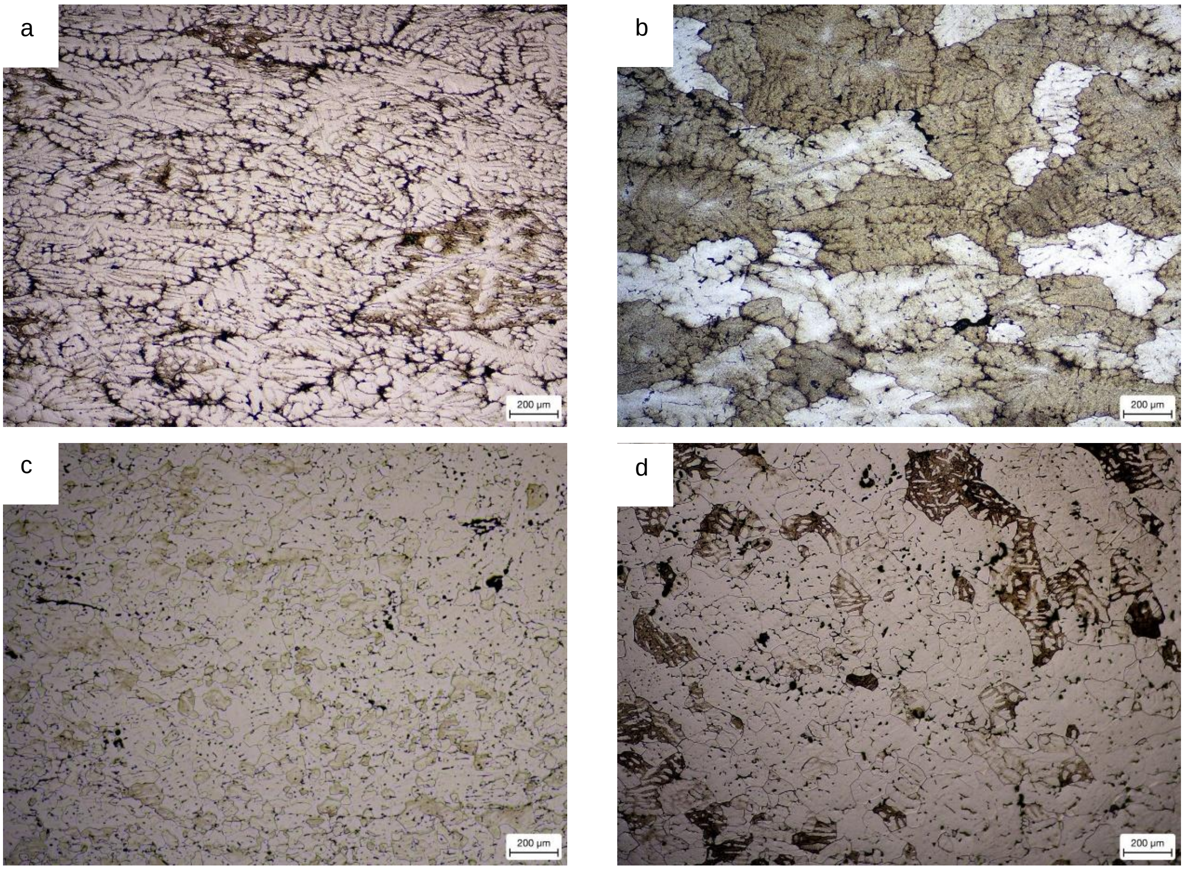

Figure 4 Microstructures of Al-4.7Zn-1.8Mg (wt.%) alloy after: (a) 20% cold rolling; and subsequent annealing at (b) 300°C; (c) 400°C; and (d) 500°C for 2 h

Figure 5 shows the SEM images of as-rolled Al-4.7n-1.8Mg (wt.%) alloy with 20% reductions and subjected to different annealing temperatures. As can be seen from Figure 5, the slip bands and cross slips no longer exist after annealing at 300°C. Annealing at 400°C resulted in new stress-free equiaxed grains, and higher annealing at 500°C increased the grain size. It can be deduced that recrystallization occurs at 400°C. It is noteworthy from the SEM images that the new stress-free grains comprise cube-like grains (as indicated by the arrows). Ying et al. (2013) also found the same cube-like grains in the newly recrystallized grains of Al-5.6Zn-1.9Mg (wt.%) alloy.

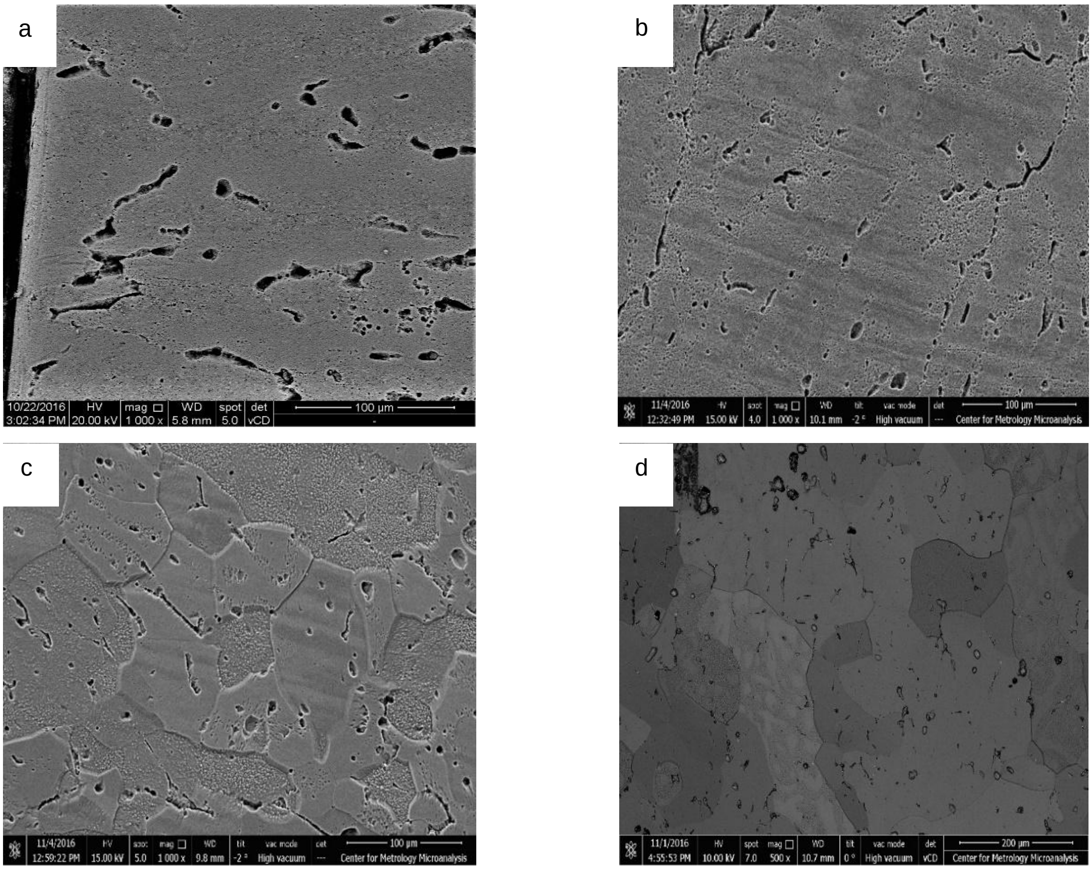

Figure 5 SEM images of Al-4.7Zn-1.8Mg (wt.%) alloy after: (a) 20% cold rolling; and subsequent annealing at (b) 300°C; (c) 400°C; and (d) 500°C for 2 h

The change in hardness after the annealing process is shown in Figure 6. Annealing at 300°C significantly decreased the hardness from 102.9 VHN to 95.7 VHN. This is due to stress relief and the removal of the secondary arms of the dendritic phase. Annealing at 400°C further decreased the hardness to 94.9 VHN. The formation of new stress-free grains as shown in Figure 4c contributed to the reduction in hardness. After annealing at 500°C for 2 h, an additional decrease in hardness to 94.1 VHN was observed. This was caused by the growth of stress-free grains. These results are in line with those of the previous study by Wang et al. (2015), whereby hardness declined as the annealing temperature increased. The decrease in hardness indicates that the strain hardening effect from cold rolling can be diminished by an annealing process, which is important for the formation of aluminium to meet the required final dimension.

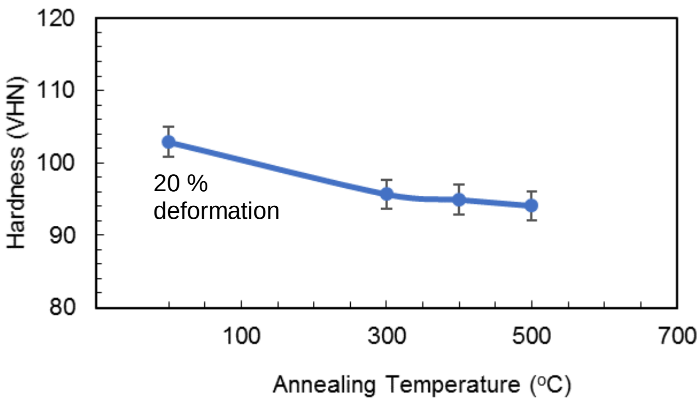

Figure 6 Effects of annealing temperature on the hardness of Al-4.7Zn-1.8Mg (wt.%) alloy

4. CONCLUSION

The results of the investigation on Al-4.7Zn-1.8Mg (wt.%) alloy revealed that the homogenization process of as-cast alloy leads to a diffusion of the interdendritic phases into the α matrix, followed by an increase in SDAS from 31.08 to 35.06 μm, more globular dendrites, and a decrease in hardness from 94.4 to 69.52 VHN. Deformations of 5%, 10%, and 20% led to grain shape ratios of 2.19, 3.19, and 4.59 and an increase in the hardness of the alloy from 69.5 VHN to 95.3, 99.4, and 102.9 VHN, respectively. Slip bands and cross slips were found only in the 20% deformed samples. The annealing process resulted in recovery at 300^{o}C, followed by recrystallization at 400^{o}C (d_{grain} ~290 μm) and grain growth at 500^{o}C (d_{grain} ~434 μm). Annealing temperatures of 300^{o}C, 400^{o}C, and 500^{o}C decrease the hardness of the alloy from 102.9 VHN to 95.7, 94.9, and 94.1 VHN, respectively.

5. ACKNOWLEDGEMENT

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