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ORIGINAL ARTICLE

Effect of semi-solid forming temperature and heat treatment on mechanical properties and microstructure of Mg-Al-Zn Alloy (AZ91D) for automotive light application

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ABSTRACT – Magnesium alloy usage in manufacturing engineering components resulting in weight reduction and as a consequence, reduction in fuel and energy consumption. Magnesium has a relatively low density and roughly 30% lighter than aluminum. However, magnesium is considered to be difficult to deform because of the HCP structure. In this present work, the effect of semi-solid forming temperature and heat treatment on mechanical properties of Mg-Al-Zn were investigated. Mg-Al-Zn ingot was machined into a billet and formed with three different temperatures and underwent T4 heat-treatment process. To determine the mechanical properties and microstructure of the magnesium alloy, tensile and hardness test were performed and the result indicates that the highest average maximum tensile stress was achieved at 209 MPa at 530ºC after forming with T4 heat treatment and highest hardness value was at 21.44 HRB at 560ºC. On the other hand, effect of the forming temperature gives impact to the evolution of the microstructure from large grain size (as-cast) to the smaller grains size (0.00797mm^2) forming at 560°C. This relate to the extensive dynamic recrystallization (DRX) occurs during forming and Mg-Al-Zn was sensitive with heat either direct or indirect heating method.

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INTRODUCTION

Usage of Magnesium in the automobile industry is significantly increasing [1]. Magnesium is one of the most widely distributed elements in nature, ranks eighth, accounting for about 2.25% of the mass of the earth's crust. Although, magnesium alloy have comprehensive performance such as low density, high specific strength and stiffness, super damping and thermal conduction [2]. However, magnesium alloy has poor plastic forming ability because of the hexagonal close-packed (HCP) crystal structure [3, 4].

The semi solid forming process composites of the advantages of the solidification and plasticity processing, and has the advantages of the lower processing temperature than the fluid processing, lower deformation resistance than solid processing, length of the die life and less shrinkage of solidification, and also can be formed to the net shape [5]. The forming mechanism of preparing the semi-solid slurry and billets is the most important mechanism of the study for the semi-solid metal forming process, whose key and foundation are how to produce the semi-solid material with tiny spheroidization or non-dendritic microstructure [5]. Present forming technologies for magnesium alloy is mostly from casting and forging. However, mechanical properties from casting processes have a drawback to the finish goods and for forging, the shape required to make a complex shape product can't be achieved. Recently, considerable interest has been generated in the semi-solid forming because it offers numerous advantages over conventional casting and forging, such as reduction of macrosegregation, reduction of porosity and low forging efforts [6].

On the previous works, back pressure thixoextrusion of AZ80 magnesium alloy by partial remelting at 560°C isothermal holding temperature for 15-25 min and the result of the ultimate tensile strength (UTS), yield strength (YS) and elongation are 293MPa, 248MPa, and 4.1%, respectively [7]. The microstructure and mechanical properties of Mg-Zn-RE-Zr alloy after thixoforming and T6 heat treatment for 6 hours at 500°C and obtain 92µm grain size and precipitate within the α(Mg) grains of two kinds of phase (Mg_7Zn_3RE) and $RE(Mg,Zn)_{11}$ responsible for increasing the yield strength to 135MPa and compression strength to 383 MPa and hardness 73 HV [8]. Beside casting and forging, hot extrusion processes give a significant impact to the dynamic recrystallization (DRX) of the magnesium alloy and the deformation take place at the temperature between 220°C to 380°C resulting in the fine equiaxed grains with serrated grain boundaries [3].

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Z. Szklarz et al. [9], investigate the effect after thixocasting processed by the electrochemical behaviour of Mg-Zn-RE alloy after solution treatment and obtain significantly improved mechanical properties and simultaneously cause slight deterioration of corrosion resistance [9]. Furthermore, investigation of tensile anisotropy by equal channel angular pressing (ECAP) without solution treatment increased the yield strength (YS) to 220MPa in the transverse direction (TD) and significant refinement had occurred after a single pass by ECAP technique through dynamic recrystallization (DRX) [10, 11]. Silva et al. [12], investigate the effect of the rare earth element in ZK60 plus T4, T5 and T6 heat treatment. The result obtained was at 1.5% of the rare earth element and further T6 heat treatment increase the hardness up to 85HV [12]. Vesling et al. [13], investigate the effect of heat treatment on the superplasticity of AZ91, AE42, ZRE1, and QE22 magnesium alloy. Heat treatment temperature 470°C at 10 hours and determine a fine grain structure exhibit good plasticity characterization and increased the elongation. Meanwhile, Han et al. [14], research on the solution tretment and aging processes after the squeeze casting at the temperature of 413°C at 24h and 200°C at 2h to 8h. The result obtained the yield strength of aged squeeze-cast AZ91 alloy is approximately proportional to its micro-hardness, which can be used to predict the mechanical properties [14]. Zhang et al. [15], research on the AZ91-Ca for rheo-squeeze casting and further heat treatment at 410°C. Result obtain is the γ-($Mg_{17}Al_{12}$) dissolved and undissolved Al_2Ca phase change from connected network-like partially spheroidized. Yield strength of the AZ91-Ca alloy was decrease gradually while ultimate tensile strength and elongation rises continuously. The aims of this study are to evaluate the effect of semi-solid forming temperature on microstructure and mechanical properties of standard magnesium alloy (AZ91D) without any addition of the rare earth element.

METHODS AND MATERIALS

An ingot AZ91D magnesium alloy was used as experimental material in this work. AZ91D contains 8.6% of Al, 0.85% Zn, 0.002% Fe, 0.03% Mn and the balance is Mg, which is a commercial use for the die casting process. The ingot was cut and machined into a billet dimension Ø20 x 100mm as in Figure 1(a) and Figure 1(b). The as-form billet, as shown in Figure 1(d) and T4 heat treatment, involves 20 hours of process time at 415ºC (Figure 1(e)). The sample labelled as A1 and A2 is a reference billet and T4 heat-treated, respectively. Sample B1, B2, and B3 have undergone a forming temperature of 510ºC, 530ºC and 560ºC by high-frequency induction heating as in Figure 1(c) with forming speed is 0.7m/s. The k-type thermocouple was used to determine the forming temperature. The summary of the experimental parameter as in Table 1. All the samples were formed and undergo the heat treatment process. Samples were cut into a tensile test dimension by wire electrical discharge machining (WEDM) according to ASTM 557M-10 with a thickness of 6mm and conducted tensile strength test and hardness test. For the Rockwell hardness test, the 100kgf load with indenter of 1/16 inch steel ball was used to determine the hardness of parameter in Table 1. Next, for the microstructure observation, there is five grade of sandpaper consists of 320, 400, 600, 800, 1200 grit was used to obtain the semi mirror, mirror finish and was completed by polishing using HSB polish cloth and 3µm diamond solution. Later, the etching process was conducted using 2% of Nital acid and rest distilled water. After that, the specimen was cleaned by spraying the specimens with distilled water thoroughly and drying under the hot blower.

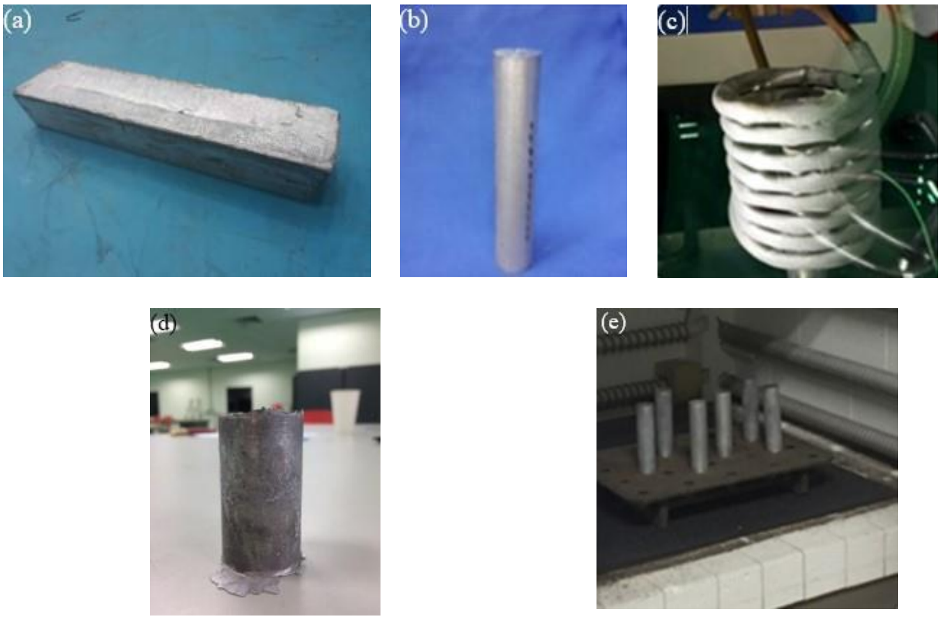

Figure 1. Semi solid processing flow used in present study: (a) AZ91D ingot, (b) as-machined billet, (c) induction heating and forming, (d) as-form and (e) T4 heat treatment

Table 1. Experimental process parameter

Samples	Process Parameter	Forming Temperature
A1	Reference Billet	-
A2	T4 Heat Treatment	-
B1	T4 Heat Treatment	510ºC
B2	Induction heating + Forming	530ºC
B3	Induction heating + Forming	560ºC
C1	Induction heating + Forming + T4 HT	510ºC
C2	Induction heating + Forming + T4 HT	530ºC
C3	Induction heating + Forming + T4 HT	560ºC

RESULTS AND DISCUSSION

Tensile Strength and Hardness

Comparison of Ultimate Tensile Strength (UTS) of all samples is shown in Figure 2. The UTS for as-cast (A1) a is 219 MPa, as-cast + T4 heat treatment (A2) is 255 MPa. Forming at 510ºC (B1) is 157 MPa, forming at 530ºC (B2) is 161 MPa, forming at 510ºC (B3) is 150 MPa, forming at 510ºC + T4 heat treatment (C1) is 182 MPa, forming at 530ºC + T4 heat treatment (C2) is 209 MPa, and lastly forming at 560ºC + T4 heat treatment (C3) is 174 MPa. The graph from Figure 2 showed forming at 560ºC (B3) obtained the lowers ultimate tensile stress (UTS) when compared to the average of the samples and highly differences from expected. Although the highest UTS was obtained by the sample as-cast + T4 heat treatment (A2), the samples are not yet formed compare with the sample forming at 530ºC + T4 heat treatment (C2). In the term of finish goods, samples that are already formed is more reliable compared to as-cast. The UTS trend from sample B1, B2, B3, C1, C2, and C3 was slightly increased at the forming temperature of 530ºC and decrease at the temperature 560ºC. It shows that the sensitivity to temperature gives a significant effect on the strength of the forming magnesium alloy (AZ91D).

At the as-cast stage, the strength of AZ91D is high compared to the at the forming stage. The forming sample in this study showed an inconsistent result where it's value does not exceed the as-cast UTS result. In the previous study on the effect of forming on A356 aluminum mechanical properties by cooling slope technique, the UTS can reach as high as 293 MPa and yield strength 234 MPa and elongation up to 15% [16]. This kind of result was to expected when tested on AZ91D forming sample. Although magnesium alloy and aluminum alloy is not is the same crystal structure, but both of the material is mainly for industrial and lightweight application. In term of flexibility strength, aluminum has some advantages compared with magnesium. On the other hand, went come to the lightweight, magnesium gives a better result compare with aluminum.

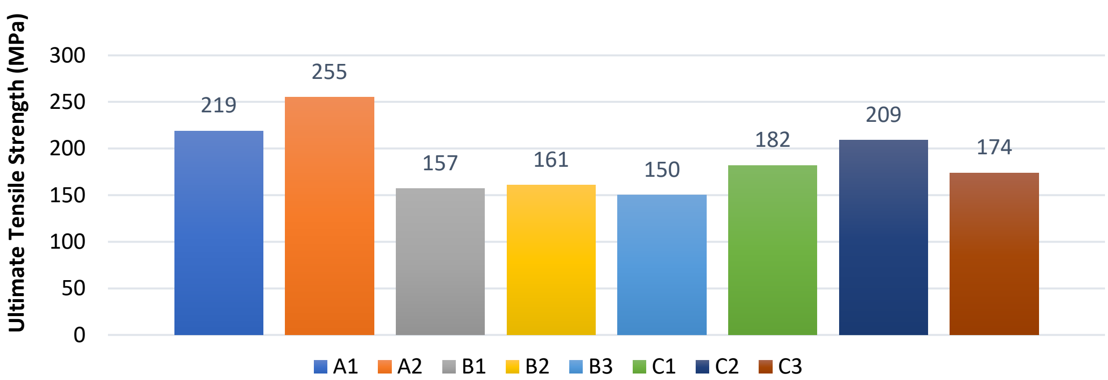

Figure 2. Ultimate tensile strength (MPa) versus samples

Comparison of the hardness test (HRB) of all samples is shown in Figure 3. The hardness test for as-cast (A1) is 13.41 HRB, as-cast + T4 heat treatment (A2) is 13.26HRB. Forming at 510ºC (B1) is 17.63HRB, forming at 530ºC (B2) is 17.52HRB, forming at 510ºC (B3) is 21.44HRB, forming at 510ºC + T4 heat treatment (C1) is 16.77HRB, forming at 530ºC + T4 heat treatment (C2) is 17.52HRB, and lastly forming at 560ºC + T4 heat treatment (C3) is 21.07HRB. The graph from Figure 3 showed as-cast +T4 (A2) obtained the lowest hardness value when compared to all average of the samples. The highest hardness value is forming at 560ºC and forming at 560ºC+T4 heat treatment where 21.44 HRB and

21.07HRB respectively. It shows that forming temperature at 560 ºC gives a significant effect on the hardness of AZ91D material but gives among the lowest ultimate tensile strength value. The trend of the hardness value was increasing at the forming levels and drop when applying the T4 heat treatment.

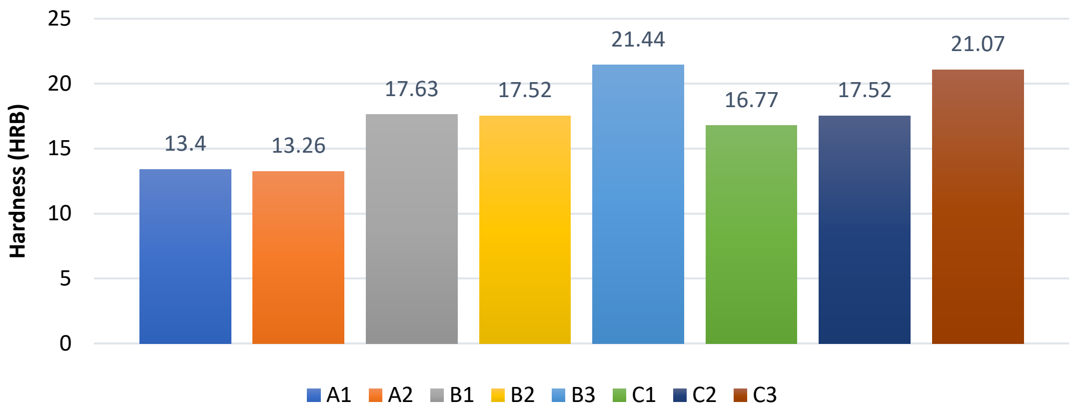

Figure 3. Hardness (HRB) versus samples

Comparison of the current work to the previous work as shows in Table 2. Most of the researchers conclude the intermetallic phases play an important role in the heat treatment process either strengthen alloy besides increase the hardness value. Many researchers [13–15] agree that the γ-($Mg_{17}Al_{12}$) disappeared when the heating temperature is rising and prolong the heat treatment time. Meanwhile, forming in liquid and semi solid process such as squeeze cast and diecasting in a permanent moulds helps the solidification and formation of the said phases.

Table 2. Comparison of current work to the previous work

No.	Processing Type	Material	Microstructural observation	Parameters	Hardness and Tensile strength
Yuan et al. [17]	Extrusion	AZ91	Homogenous equiaxial and fine grains with size of 10 µm	Extrusion temperature 380°C	-
Chen et al. [18]	Heat Treatment + aging	ZW21	Mg2Sn phases in the alloy are dissolved into the matrix	Solution treatment at 420°C	167MPa and elongation 4.2%
Huang et al. [19]	Heat treatment	AZ61+ SiC	Reinforce particle leads to microvoids and major cause to form a large crack	410°C for 24 hours	Hardness increase from 65Hv to 75Hv
Swetha Chowdary V. et al. [20]	Heat Treatment + machining	AZ91	Many of α-mg bounfries as free from the γ-($Mg_{17}Al_{12}$)	410°C at 6 hours	Increase hardness up to 172 Hv
X.J. Wang et al. [21]	HPDC + Heat treatment	AZ91	Most of the γ-($Mg_{17}Al_{12}$) dissolved in the α-Mg	413°C at 20 hours	241 MPa (HPDC Vacuum)
Han et al. [14]	Solution treatment and aging	AZ91	γ-($Mg_{17}Al_{12}$) morphology characteristic of the precipitate consists of three kinds of faces.	413°C at 24 hours	Yield strength - 116 MPa and hardness – 84 Hv
Current work	Forming + Heat treatment	AZ91D	γ-($Mg_{17}Al_{12}$) dissolve into the matrix	Forming Temperature at 360°C	Hardness 21.44HRB

As-cast Microstructure

The as-cast samples, as well as the forming samples, were analyzed and the effect forming temperature and T4 heat treatment on microstructure morphology was observed by an optical microscope model Nikon LV100 with difference magnifications to obtain the best image. For the analysis on the microstructure, i-solution software was used to identify the area, grain count and others. Microstructure of an as-cast (AZ91D) magnesium alloy with micro shrinkage at almost of the sample as in Figure 4(a). The microstructure of the as-cast AZ91D sample consists of γ-intermetallic phase, known as $Mg_{17}Al_{12}$, precipitates in the α-magnesium matrix [22]. Original image of the as-cast microstructure with 50x magnification as in Figure 4(a). The α (Mg) phases that are in blue color and green color show the γ-($Mg_{17}Al_{12}$) phase or eutectic phase same as [1] as shown in Figure 4(b). Shrinkage porosity was found in the as-cast samples in black color, as in Figure 4(a). The total area of the α (Mg) is 0.725mm^2 and the total area of the γ ($Mg_{17}Al_{12}$) including shrinkage porosity is 0.148mm^2. The as-cast microstructure is a reference to monitor the dynamic recrystallization (DRX) during forming and the heat sensitivity of the phases. The average grain size receives from the as-cast is about 100µm (Figure 4(a) almost similar to yuan et al. [17].

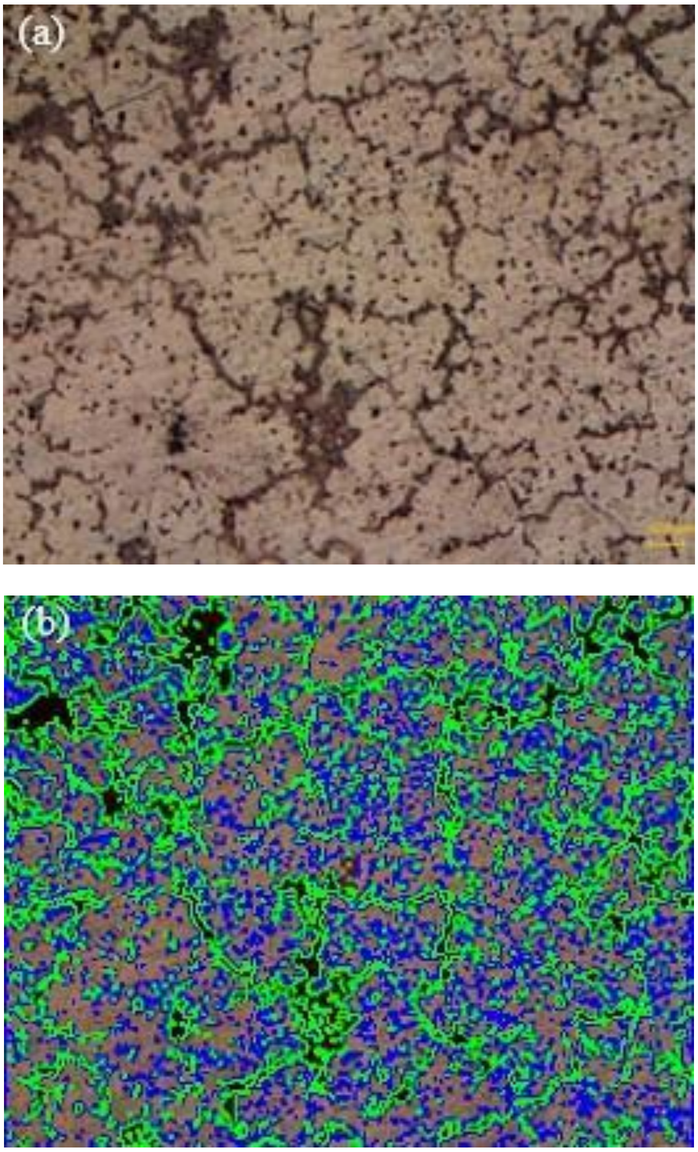

Figure 4. As-cast ingot used in the current experiment: (a) As-cast microstructure morphology, (b) 50x magnification with α(Mg) and γ (Mg17Al12) phase

Lamellar α (Mg) + γ ($Mg_{17}Al_{12}$) eutectic mixture can be found at the surrounds the precipitates of the as-cast and form a grain boundary consist of these 2 phases and the coarsening microstructure at the grain boundary with 1000x magnification as shown in Figure 5(a). The solidification starts from grains from the centre of the grain boundary to the end of the intermetallic compound. Recrystallization takes place during the solidification of the as-cast at the transition liquid to the solid phase. The mixing between α (Mg) + γ-$Mg_{17}Al_{12}$ and form grain boundary make the bonding between other grain stronger as a result in Figure 2 for the A1 sample show the ultimate tensile strength (UTS) for the as-cast sample is 219MPa. Nucleation of the α (Mg) + γ- $Mg_{17}Al_{12}$ start at the centre and form the lamellar intermetallic in the grain boundary and the average length of the intermetallic is 13.44µm as shown in Figure 5(b). The area of γ-$Mg_{17}Al_{12}$ at the grain boundary was 1754.88 µm, as shown in Figure 5(c). Length of intermetallic at the grain boundaries for Figure 5 (b) are shown in Table 3.

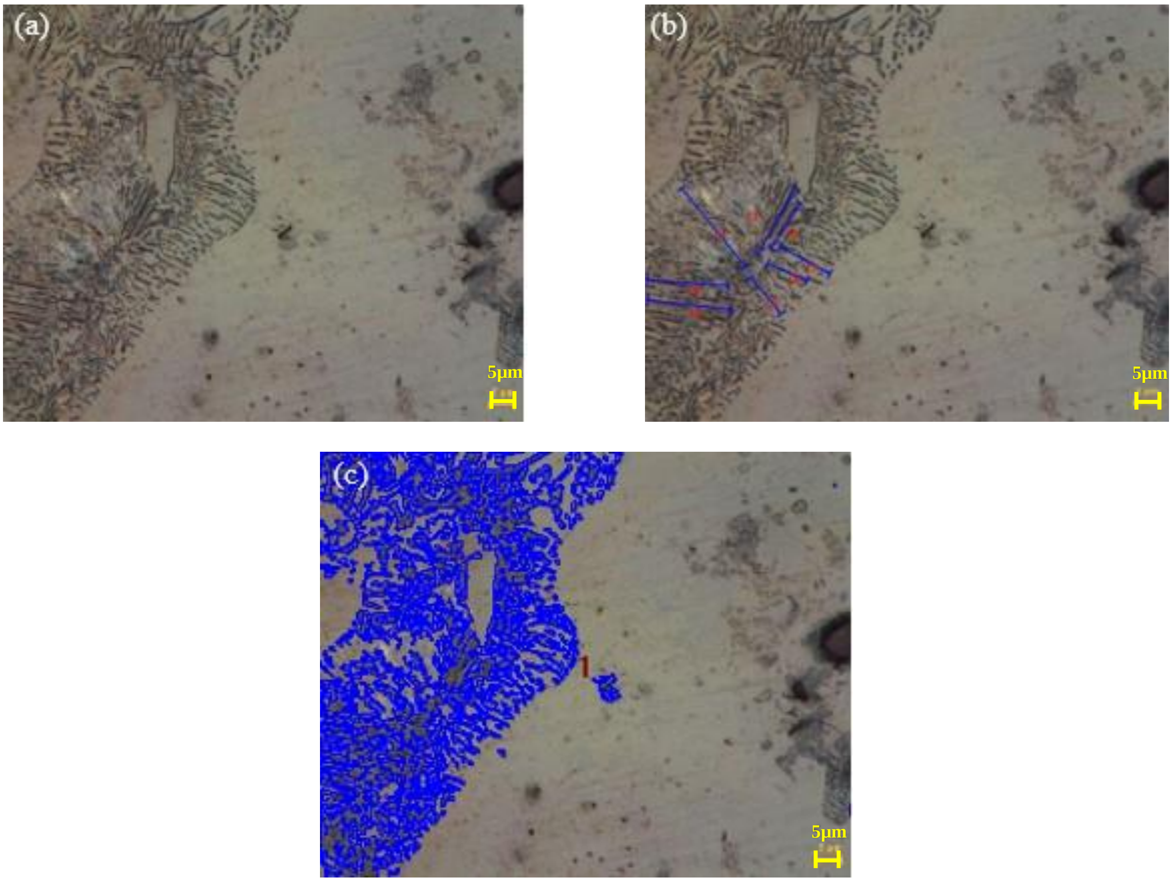

Figure 5. Intermetallic phases in the as-receive ingot: (a) α (Mg) + γ(Mg17Al12) with 1000x magnification, (b) length of the intermetallic phases and (c) area of the γ (Mg17Al12) phase

Table 3. Length of the intermetallic phases α (Mg) + γ (Mg17Al12)at the grain boundary as in Figure 4(b)

Number	Length Name	Length, (µm)
1	L1	15.14464
2	L3	20.07022
3	L4	15.93756
4	L5	9.48646
5	L6	16.72393
6	L7	11.17539
7	L8	8.44385
8	L9	10.5997

Effect of Semi-Solid Forming Temperature on the Microstructure Without Heat Treatment

Specimen from each type was observed. The microstructure of B1 sample, as shown in Figure 6(a) and higher magnification, is in Figure 6(b). It seems that the dendrites start to appear when forming and heating is applied. Forty-eight units of lengths of dendrites were capture in the B1 sample, as shown in Figure 6(c). The maximum length of dendrites is L22 that is 538.19µm and the minimum length of dendrites is L13 that is 28.90µm as in Figure 6(c). The average dendrites for the B1 sample were 210.39 µm. From the observation, α-mg was larger and the dendrites were gotten smaller when compared to the as-cast specimen. Porosity at the microstructure becomes smaller compared with the as-cast. This is because the dynamic recrystallization (DRX) during the forming process occurs [23]. The result shows the AZ91D was sensitive with heat, either direct or indirect heating method. In this case, induction heating was applied to the sample. Furthermore, by looking at the tensile strength result of B1, the strength among the lowers compare to other samples. This is because of the primary dendritic and secondary dendritic lead to the higher propagation of cracks up to the grain boundary. Larger length of dendritic decrease the strength of the material. Comparing with the C2 samples, as Figure 8, the dendritic is was completely disappear and leaves only a solid α-Mg and γ- $Mg_{17}Al_{12}$ phases.

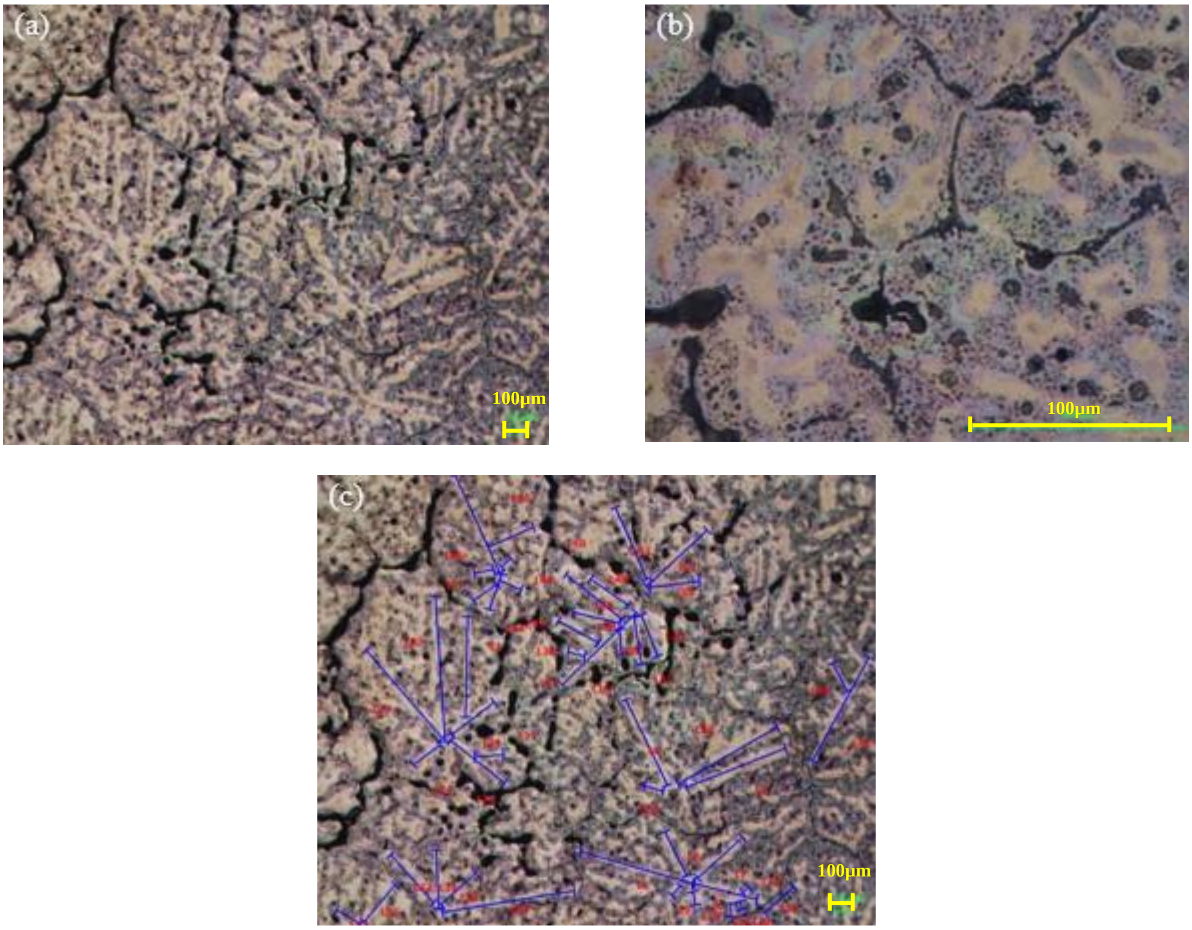

Figure 6. Microstructure after forming without heat treatment: (a) Dendrites at the B1 samples, (b) 200x magnification of B1 sample and (c) dendrites length measurement

On the other hand, The dendrites also change to rosette shape (B3) after as-cast specimen underwent the semi-solid process at 560°C as shown in Figure 7(a). From the observation, the length of dendrites became shorter compare to the B1 samples. The maximum grain size was $0.1339mm^2$ and the minimum size of the grains was $0.00013mm^2$. The average grain size of the B3 sample is $0.00797mm^{2,}$ as in Figure 7(b). The microstructure from Figure 7(a) is almost similar to the Qiang Chen et al. [24] after equal channel angular extrusion (ECAE).

Effect of Semi-Solid Forming Temperature on the Microstructure with T4 Heat Treatment

The solution heat treatment (T4) applied on the semi-solid processing C2 specimen revealed that the dendrite shape is no longer visible and leaving a big area of α-mg along with a thin grain boundary with the magnification of 200x as shown in Figure 8(a) and 500x (Figure 8(b)) respectively. The thin grain boundary created a greater bonding between each grain and increased the ultimate tensile strength (UTS) as shown in the Figure 2 label as C2 at 209MPa compare with C3 microstructure as in Figure 8(c) and higher magnification of C3 as in Figure 8(d) was only at 174Mpa. Although the grain size at the C2 was more than 100µm compare to the as-cast, but the strength receives more than 200 MPa. By closer gab of γ-$Mg_{17}Al_{12}$ phase will increase ductility. Furthermore, in C2 specimen, the phase γ-($Mg_{17}Al_{12}$) is decreased when rising the forming temperature at the semi solid level follow with the heat treatment of T4. According to yuan et al. [17], most of the aluminium atoms dissolved in the α-Mg matrix and became supersaturated at the room temperature. At this stage, the average grain size is at 100µm and further coarsening is extend the hours of heat treatment. Higher magnification of C3 samples (Figure 8(d)) and compare with Figure 5(a) the as-cast consist of α-mg + γ- $Mg_{17}Al_{12}$ phases dissolved in the α-mg matrix. This proves that α-mg + γ- $Mg_{17}Al_{12}$ phases were sensitive to heat. Further T5 or T6 were going to modify the precipitate to the fines microstructure. In this case, the ductility of the alloy will be increased and suitable for medium load bearing application in the automotive such as timing chain cover and transmission case.

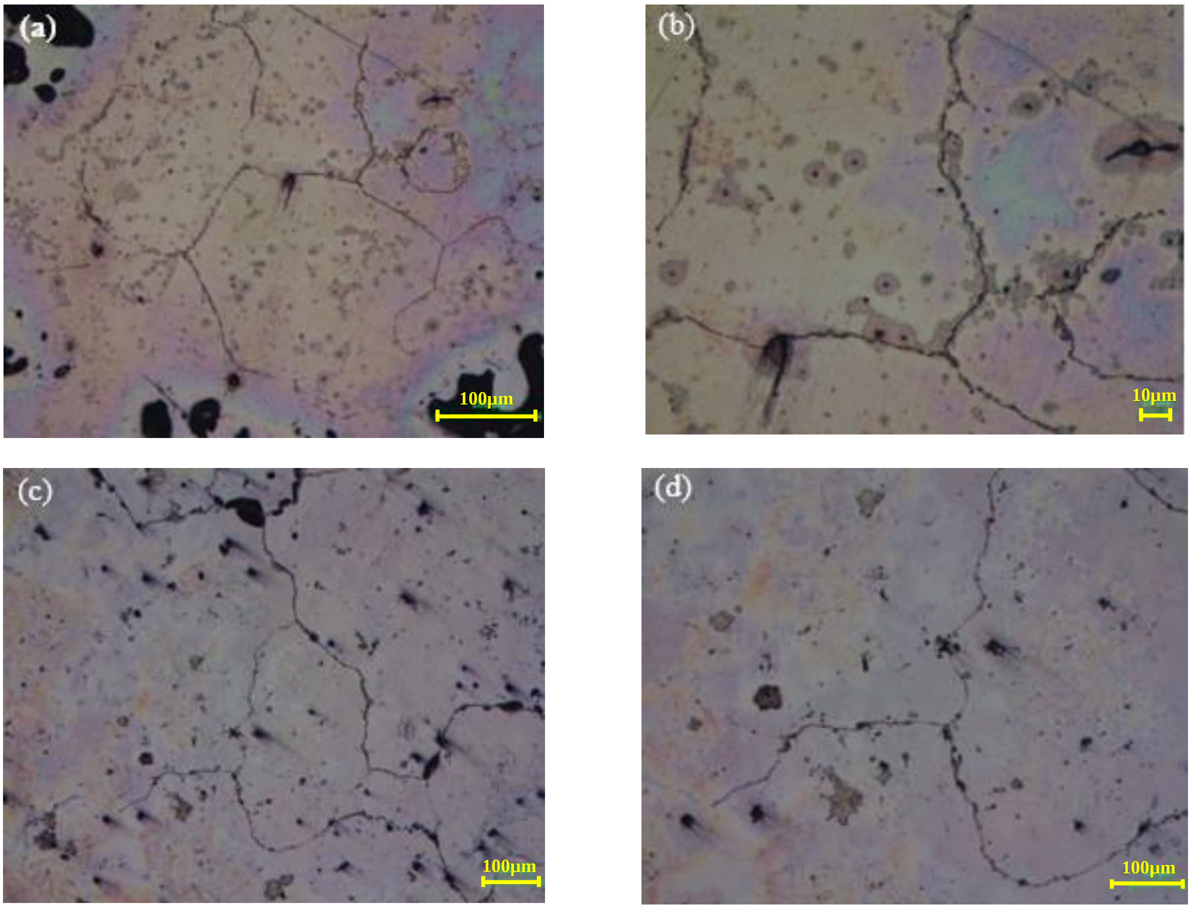

Figure 8. Microstructure after forming with heat treatment: (a) Sample C2 forming at 530°C +T4 (200x), (b) sample C2 forming at 530°C +T4 (500x), (c) sample C3 forming at 560°C +T4 (100x) and (d) sample C3 forming at 560°C +T4 (200x)

CONCLUSIONS

The mechanical properties covered in this study was tested using a tensile test where the ultimate tensile strength (UTS) was observed.

1. The result of the tensile test showed that the T4 without forming gives the highest value of UTS. Meanwhile the trend was increasing and reach the optimum at C2 samples before decreasing.
2. In the result of the hardness value in the Rockwell test showed that forming with 560°C gave out the highest value. Meanwhile, the hardness value of the forming at 560°C + T4 sample gives the second highest.
3. The dendrites also change to rosette shape (B3) after as-cast specimen underwent the semi-solid process at 560°C. From the experimental, it was proved that the strength of AZ91D can be improved by optimizing the forming temperature and T4 heat treatment, plus dynamic recrystallization (DRX) occurred during semisolid forming.
4. As-cast samples consist of α-mg + γ- $Mg_{17}Al_{12}$ phases was dissolve in the α-mg matrix when applying heat during the forming and further T4 heat treatment. This the reason of increasing the trend of ultimate tensile strength (UTS).

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