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Effect of Different La Additions on the Microstructure and Mechanical Properties of Hot Extruded SWAP Mg-Al-Zn-Ca Powder

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Abstract

The microstructure and mechanical properties of hot extruded Mg-Al-Zn-Ca alloys with different Lanthanum additions were investigated. Both rapidly solidified powders, produced via Spinning Water Atomization Process, and cast billets were extruded at 573, 623 and 673 K to optimize the processing conditions for obtaining better mechanical response. Powders were consolidated to prepare the extrusion billets using both cold compaction and Spark Plasma Sintering at 473, 573 and 623 K. The tensile properties of the extruded alloys were then evaluated and correlated to the observed microstructure. The results show that the use of rapidly solidified Mg-Al-Zn-Ca powder with La additions could lead to effective grain refinement, which in turn resulted in the improved mechanical response, especially compared to the extruded conventional cast material. The La content of 1.5% has shown superior tensile properties compared to that of 1.3%, and to other Mg alloys like AMX602, especially the elongation, as shown in Fig. 1. This may help extend the applications of Mg alloys to higher load carrying parts while maintaining the excellent advantage of light weight parts.

KEY WORDS: (Rapid solidification) (Magnesium alloy powder) (La) (Hot extrusion)

1. Introduction

Recently, magnesium alloys have received much attention for applications requiring light weight materials due to their low density and high specific strength. Applications of Mg include the use of both cast and wrought alloys. It was shown to be important to use wrought alloys in applications requiring higher strength$^{1)}$. However, the applications of wrought Mg alloys are still limited by their low room temperature ductility and anisotropy as a result of the hexagonal close packed crystal structure, which is characterized by its limited number of active slip systems at room temperatures$^{2\text{-}6)}$. Hence, the improvement of the mechanical properties of Mg alloys is still needed to extend their applications to high load-carrying parts. Grain refinement is a very powerful tool for improving the strength of crystalline materials, especially Mg$^{7)}$, as it has high value of the strengthening factor, according to Hall-Petch equation$^{8\text{-}10)}$.

Grain refinement could be obtained in metals by rapid solidification$^{11,12)}$. It was shown that rapid solidification can contribute to the realization of high strength of non-combustible magnesium alloys containing both Al-Ca and Zn-RE combinations$^{9\text{-}10)}$. Spinning Water Atomization Process (SWAP) has proven its ability to produce rapidly solidified powder particles with ultra fine grained microstructure having grain size less than 0.3 microns$^{11)}$. In this process, gas atomization is combined with water atomization in SWAP, which in turn increases the cooling rate to about 10^6 K/s, while it ranges from 10^2 to 10^5 K/s for the conventional atomization processes. The objective of this study is to investigate the effect of La additions combined with the use of rapidly solidified powder metallurgy for the realization of improved mechanical properties of the used Mg-alloy. La addition has many advantages including increasing both the heat resistance and corrosion resistance. However, the effect of La addition on the tensile properties of extruded Mg alloy was only dealt with in this study.

2. Experimental

Mg-7Al-1Zn-1Ca-xLa magnesium alloy powder, with x equals 1.5 and 3.3%, produced using Spinning Water Atomization Process (SWAP) was used in this study. For the purpose of comparison, extruded cast material was also used to confirm the effect of rapid solidification of SWAP powder on the final properties of the extruded material. Microstructure analysis of both the SWAP powders and cast billets was performed using an optical microscope, while for the extruded material, Scanning Electron Microscope (SEM, JEOL: JSM-6500F) was used as the very fine grains of the extruded materials

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could not be analyzed by the highest magnification of optical microscope at 1000x. Energy Dispersive X-ray Spectroscopy (EDS, JEOL: EX-64175JMU) attached to SEM was used to analyze the compounds present. Differential Thermal Analysis (DTA, Shimadzu: DTG-60) was used to investigate the metallurgical transformations of both SWAP powder and cast material during thermal processing. The analysis was performed while heating the sample of about 18 mg of powder with reference to 30 mg of Alumina as reference material in an Ar atmosphere. X-ray Diffraction (XRD) analysis was used to investigate the present phases in both SWAP powder and cast material using an X-ray diffractometer (Shimadzu: XRD 6100) over a range of 2θ of 20 to 80°.

As-received SWAP powders were consolidated using two techniques, namely cold compaction and Spark Plasma Sintering (SPS, Sumitomo Coal Mining: SPS-1030). In cold compaction, 60 gm of powder was compacted in a 42 mm diameter mold at room temperature using a pressure of 600 MPa. On the other hand, SPS was carried out on the same amount of powder and same size of mold at 350 ℃ in vacuum under the pressure of 30 MPa. Heating was done at a rate of 10 ℃/min followed by holding at the sintering temperature for 30 min. Both cast billets and consolidated powder billets were extruded using the 2000 KN hydraulic press machine at temperatures of 300, 350 and 400 ℃. Extrusion was performed using die that produces extrusion rods of 7 mm, which is equivalent to an extrusion ratio of about 37. The preheating of billets was done just before extrusion using a heating rate of 1 ℃/sec. The billets were held at the extrusion temperature in the furnace for 5 min prior to extrusion to ensure homogenous temperature distribution. Both the extrusion container and the die were also preheated to each extrusion temperature to keep the temperature of the billet constant during the extrusion process. Both hardness and tensile tests were performed on the extruded materials to evaluate their mechanical response and to optimize the extrusion conditions. Hardness tests were carried out using a Micro Vickers tester (Shimadzu: HMV-2T) with a test load of 0.491 N. Tensile tests were performed using (ORIENTEC: RTC-1310A) at room temperature. The tensile specimens, having the diameter of 3 mm and the gage length of 10 mm, were evaluated using a strain rate of $5x10^{-4}$ /sec.

3. Results and Discussion

Figure 1 shows the optical microstructures of SWAP powder, as well as that of cast Mg-Al-Zn-Ca-La alloy. For cast material, flakes of precipitated compounds distributed in the matrix of α-Mg matrix are observed in **Fig. 1 (a)**. These compounds were then analyzed using XRD analysis in which it was revealed that both Al_2Ca and $Al_{11}La_3$ compounds exist in the cast material, as shown in **Fig. 2**. The SWAP powder shows directional solidification in terms of dendrite structure with ultra fine size along with some amount of equi-axed grains in **Fig. 1 (b)**. The dendrite size greatly depends on the powder particle size through the effect of cooling rate. The secondary dendrite arm spacing (DAS) showed the value of 0.91 microns for particle sizes below 100 microns, while it showed 2.03 microns for particle sizes over 1 mm. According to the relation between DAS (λ, μm) and the cooling rate (R, K/s):

$$\lambda = 35.5R^{-0.31}$$

These values of DAS correspond to the values of cooling rates of $1.36x10^5$ and $1.02x10^4$ K/s, respectively[17]. No precipitation of compounds can be observed in SWAP powders, as could be also confirmed by **Fig. 2** in which the XRD pattern of SWAP powders show only the peaks of α-Mg. That is due to the super-saturation of alloying elements in the matrix as a result of the use of super high cooling rates during the production of powders using SWAP process.

The DTA profiles for SWAP powder as well as for cast Mg-Al-Zn-Ca-La alloy, as shown in **Fig. 3** showed an endothermic reaction due to the melting of Al_2Ca phase at about 530 ℃. Another endothermic reaction was shown at about 580 ℃ due to the melting of $Al_{11}La_3$ phase. This means that thermal processing of both SWAP powder and cast material at temperatures below 530 ℃ has no effect on compound formation or melting. The last peak observed is due to melting of α-Mg.

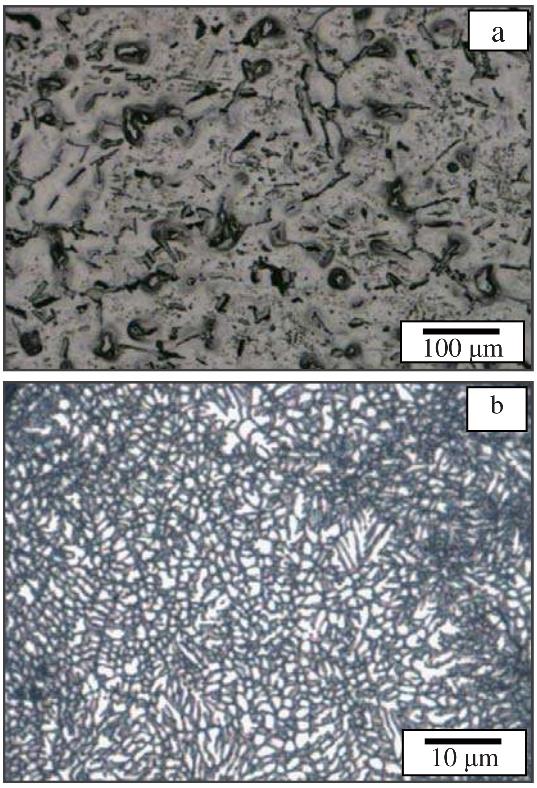

Fig. 1 Microstructure of cast material (a) and SWAP powder (b).

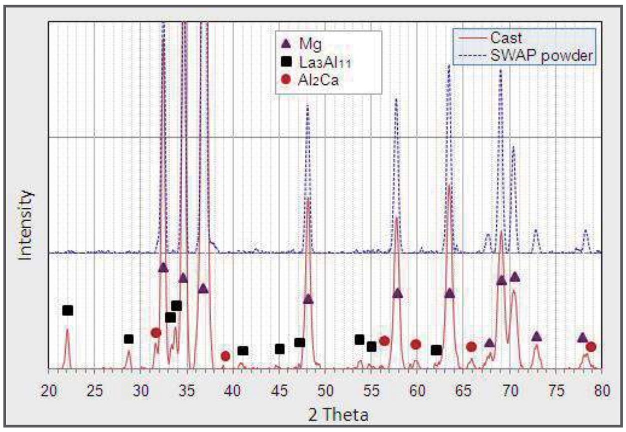

Fig. 2 XRD patterns of both SWAP powder and cast billet of ZAXE1711 alloy.

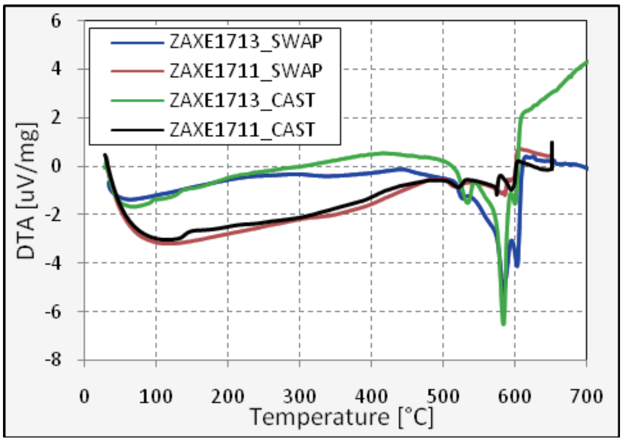

Fig. 3 DTA patterns of SWAP powder and cast billet of ZAXE1711 and ZAXE1713 alloys.

As for the consolidated billets before extrusion, SPS results in the improved bonding between powder particles along with the uniform sintering and reduced internal stresses due to evenly dispersed spark plasma energy between their particles. Another very important benefit of SPS is the minimized grain growth, compared to that of conventional sintering, due to localized heating during the consolidation process. However, only the microstructure of the extruded Mg-Al-Zn-Ca-La alloy is discussed below. **Figure 4** shows the observed SEM images of extruded Mg-Al-Zn-Ca-La alloy. As examples, SWAP powders containing 1.5 and 3.3% La consolidated via cold compaction and extruded at 300 and 400 ℃ only are shown. The grain size, which was calculated by the image analysis software, was shown to decrease as the extrusion temperature decreased. The same behavior was also observed for the case of the size of precipitated compounds. Generally, the extruded SWAP powders resulted in very fine and uniform grain sizes in the order of 0.4 to 0.5 microns, while extruded cast billets resulted in coarser grains in the order of 5 microns. Extruded SWAP powder materials indicate that both Al_2Ca and $Al_{11}La_3$ compounds are dispersed in the structure as could

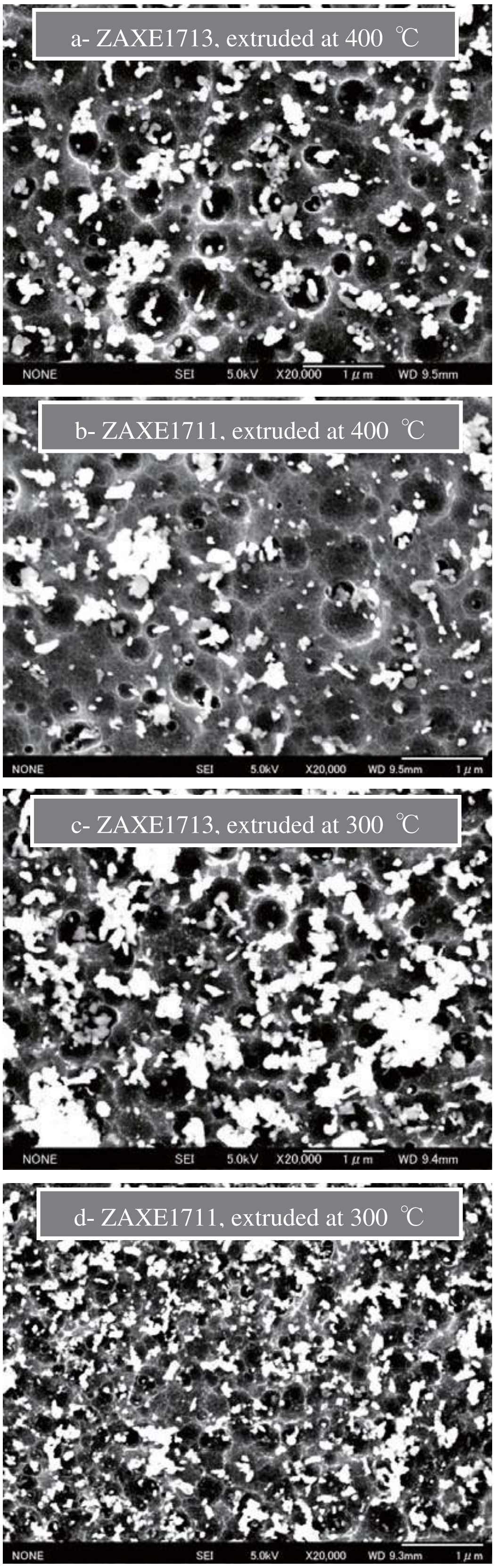

Fig. 4 Microstructures of cold-compacted and extruded ZAXE SWAP powders observed by SEM.

be confirmed using XRD analysis. The amount and size of these precipitated compounds was shown to depend on the La content. With increasing the content of La addition, coarser precipitated compounds particles could be observed. The use of SPS process has resulted in the formation of more and coarser precipitated compounds than that of cold compacted samples. That is because of the higher temperature used for SPS process, which was 350 ℃.

Figure 5 shows the dependence of tensile properties on the extrusion temperatures. The higher the extrusion temperature, the lower the yield, and tensile strengths of extruded Mg-Al-Zn-Ca-La alloy. That can be attributed to the strain hardening that occurs during plastic deformation of the extrusion process, which is increased in the case of extrusion at lower temperatures. It is also shown from the figure that the extruded SWAP powder could show tensile properties superior to that of cast billets, as the tensile strength for extruded cast materials had an average of 290 MPa and the yield strength had an average of 195 MPa for both alloys. This is due to the finer size of both grains and precipitated compounds of extruded SWAP powders compared to that of cast billets. The extruded SWAP powders containing 1.5% La have shown tensile properties that are much improved over that of SWAP powders containing 3.3% La. That could be attributed to the coarsening of precipitated compounds for the latter, which has overcome the grain refinement effect produced by La addition. It can also be shown from Fig. 5 that the use of rapidly solidified SWAP Mg powders alloyed with La additions could lead to improved levels of tensile strength while maintaining promising values of elongation in the range of 13 to 20%. The mechanical response of consolidated billets produced via SPS without extrusion is expected to be improved due to the effect of the better bonding between powder particles as well as minimized grain growth compared to conventional sintering. However, the effect of SPS on the mechanical response of the extruded materials in this study is minor as a result of the dominant re-crystallization effect of the hot extrusion process. This means that yield strength is exactly expressed by the Hall-Petch equation.

Fractography of the fractured tensile test specimens showed that sufficient bonding between powder particles could be obtained at the extrusion temperatures used, as no primary particle boundary was observed at the fracture surface. Generally, the fracture surface of all the fractured specimens showed dimpled pattern as an indication for ductile fracture. However, the size of dimples varied slightly among different consolidation and extrusion conditions of SWAP powders.

The tensile results of the extruded SWAP Mg-Al-Zn-Ca-La powder suggest that the strength of magnesium alloys can be effectively improved through grain refinement. Only a slight decrease within extruded SWAP powder in the grain size by extrusion at lower temperature could lead to a drastic increase in the strength. The same behavior could also be observed when comparing extruded SWAP powder with extruded cast billet specimens.

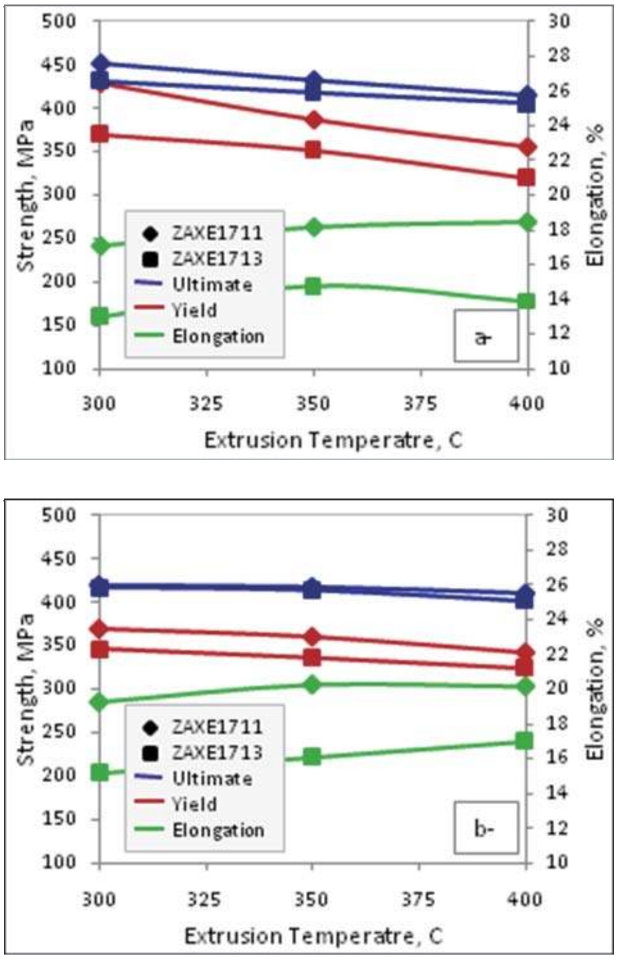

Fig. 5 Tensile properties of extruded SWAP powders consolidated using cold compaction (a) and SPS (b).

4. Conclusions

The use of SWAP powder could lead to an effective improvement in the mechanical response of extruded Mg-Al-Zn-Ca-La alloy, compared to that of extruded cast billets. Extrusion of both SWAP powders and cast billets at lower temperatures could lead to improvement in the strength of extruded Mg-Al-Zn-Ca-La alloy. The tensile strength value of up to 450 MPa for cold compacted Mg alloy powder extruded at 300 ℃ is very promising for the improvement of mechanical properties of Mg alloys. Grain refinement is a very powerful tool for the improvement of the mechanical response of Mg alloys. The grain refinement mechanism is more effective for the improvement of the strength of Mg-Al-Zn-Ca-La alloys than the mechanism of the compound precipitation. A La content of 1.5% could lead to improved mechanical properties compared to that of 3.3% as a result of the good balance between its effects on grain refinement and precipitated compound formation.

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