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Mechanical Characteristics of Hot Extruded Non-Combustible Magnesium Alloy using Water Atomized Powder[†]

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Abstract

SWAP (Spinning Water Atomization Process), is one of the rapid solidification processes which promises to produce coarse non-combustible magnesium alloy powder with $1 \sim 4mm$ length, having fine α -Mg grains and Al_2Ca intermetallic compounds. AMX602 (Mg-6%Al-0.5%Mn-2%Ca) powders were compacted at room temperature. Their green compacts with a relative density of about 85% were heated at 573-673 K for 300s in Ar gas atmosphere, and immediately consolidated by hot extrusion. Microstructure observation and evaluation of mechanical properties of the extruded AMX602 alloys were carried out. The uniform and fine microstructures with grains less than 0.45~0.8 µm from dynamic recrystallization during hot extrusion were observed, and were much smaller compared to the extruded AMX602 alloy fabricated by using cast ingots. The extremely fine intermetallic compounds 200~500 nm diameter were uniformly distributed in the matrix of powder metallurgy (P/M) extruded alloys. These microstructures produced excellent mechanical properties in the wrought alloys. For example, in the case of AMX602 alloys extruded at 573K, the tensile strength (TS) of 447 MPa, yield stress (YS) of 425 MPa and 9.6% elongation were obtained.

KEY WORDS: (Rapidly solidification) (Magnesium alloy powder) (Al₂Ca) (Hot extrusion) (Water atomization)

1. Introduction

Magnesium has a low density of 1.8 kg/m³, and is the lightest of the industrial metals. It has a higher specific tensile strength than other metals such as aluminum, iron and titanium alloys¹⁾. Therefore, it promises to save energy consumption and reduce air pollutions such as CO_2 , SO_x and NO_x by a remarkable weight reduction when applying it to structural components used in automobiles. motorcycles and airplanes. In general, the strengthening of materials is useful for the weight reduction of their components. Grain refinement is one of the effective strengthening processes. For example, the Hall-Petch equation is well known to quantitatively express the effect of the grain size on the yield stress of the materials²⁾. In this equation, a larger Hall-Petch constant, corresponding to the gradient of the equation, is more effective for increasing the strengthening effect. When the constant of magnesium and comparing aluminum, the former is about twice as large as the latter³⁾. Accordingly, the process design by grain

refinement is often employed to improve the mechanical properties of magnesium alloys. For example, hot extrusion, rolling, forging, high pressure torsion (HPT), accumulative roll bonding (ARB) and roll-compaction (RCP), which are typical severe plastic working processes, are all useful to refine grains of the matrix via dynamic recrystallization⁴⁻⁸). The strong texture, however, is formed in the matrix at the same time when these plastic working processes are applied to the magnesium alloys. It is concluded that the severe plastic deformation is available to increase the tensile property by the grain refinement, but causes its anisotropy of the magnesium alloys⁹. On the other hand, a rapid solidification process is useful to prepare ultra-fine microstructures of metal powders such as iron, aluminum, magnesium, copper and allovs¹⁰⁻¹³). their Non-equilibrium phases, amorphous and metallic glass structures can be also built up in the rapidly solidified metal powders¹⁴⁻¹⁶. They also promise to improve the mechanical and physical properties of their consolidated materials.

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In general, microstructure refinement by the rapid solidification process strongly depends on the particle size. For example, the finer powders are significantly effective to form a smaller grain size. It is, however, obvious that finer powders of active metals such as aluminum, magnesium, and titanium, are very dangerous in handling. It is also difficult to consolidate them via the solid-state sintering process because of the thermally stable oxide films covering the powder surfaces. Therefore, from the above two points of view, it is necessary to handle and consolidate the fine magnesium powders, ribbons and flakes under a vacuum condition. In this study, the microstructure observation and structural analysis were carried out on coarse magnesium powders with 1~4 mm length prepared by Spinning Water Atomization Process (SWAP). The non-combustible magnesium alloy¹⁷⁾, AMX602 (Mg-6%Al-0.5%Mn-2%Ca), was selected. When consolidating AMX602 powders by hot extrusion, the effect of the temperature in the pre-heating process before extrusion on the microstructural and mechanical properties of the extruded AMX602 powder metallurgy (P/M) alloys was investigated in detail. The extruded material fabricated using AMX602 cast ingot was employed as the reference. P/M AMX602 alloys showed extremely fine grains with a diameter of 0.45~0.8 μ m, and a good balance of tensile strength of 422 MPa and 14.2% elongation at room temperature.

2. Experimental

In SWAP powder preparation, the non-combustible AMX602 magnesium alloy ingots were melted at 1053K in the ceramic crucible under the protection of inert gas, and their molten metals were directly streamed into the spinning water from the crucible nozzle. Table 1 shows chemical compositions of AMX602 alloy powders prepared by SWAP. The calcium content of 2.09% is satisfied with the requirement. The impurity content of Fe and Cu is successfully controlled less than 0.005% because they are corrosive elements against magnesium alloys. As shown in Fig.1, a length of the coarse AMX602 powders prepared by SWAP is 1~4mm, and they have an irregular shape. The cast ingot with the same compositions was also prepared as a reference input material.

Table 1Chemical compositions of non-combustivemagnesium alloy powders

AÌ	Zn	Mn	Fe	Si	Cu	Ca	Mg
6.01	0.007	0.26	0.002	0.038	0.004	2.09	Bal.



Fig. 1 Morphology of coarse non-combustible magnesium alloy powder (AMX602; Mg-6%Al-0.3%Mn-2%Ca) prepared by SWAP.

The columnar compact and cast ingot were heated at $573 \sim 673$ K for 180 s in argon gas atmosphere, and immediately consolidated to full density by hot extrusion. The extrusion ratio of 37 and 1m/s extrusion speed were used in this study. To evaluate the thermal stability of the microstructures of raw powders, differential thermal analysis (DTA, Shimadzu DTG-60) and x-ray diffraction (XRD, Shimadzu XRD-6100) analysis were carried out. Optical microscope (Olympus, BX-51P) and JOEL scanning electron microscope (SEM, JSM-6500F) with energy dispersed X-ray spectrometer (EDS, JOEL EX-64175JMU) were used to investigate the grain size and intermetallic compounds of the raw powders and their extruded alloys. The image scanning soft ware (Image Pro-Plus II) was applied to the photos to estimate the grain size distribution. The micro-Vicker's hardness of powders and wrought materials was measured by using the hardness tester (Shimadzu, HMV-2T). The tensile test specimens were machined from the extruded alloys, and evaluated at room temperature under a strain rate of $5*10^{-4}$ /s. The fractured surfaces of the tensile test specimens were observed by SEM to investigate the fracturing mechanism of the magnesium alloys.

3. Results and Discussion

Figure 2 indicates optical microstructures of AMX602 cast ingot (a), as-received fine powders less than 0.5mm (b) and coarse powders over 1mm (c) prepared by SWAP. The cast ingot material consists of coarse α -Mg grains of 60~150µm diameter, and some intermetallic compounds are observed at their grain boundaries. As shown in (b), fine powders reveal small dendrite structures formed during the rapid solidification of molten Mg alloy droplets after atomization.



Fig. 2 Optical microstructures of input raw materials; as-cast ingot billet (a), as-received fine SWAP powder (b) and coarse powder (c).

The dendrite arm spacing (DAS) is about $1 \sim 3 \mu$ m. The coarse powders shown in (c) indicate large grains of $4 \sim 8 \mu$ m diameter caused by a smaller solidification ration during atomization. In both powders, any intermetallic compounds are not detected obviously at the grain boundaries. Figure 3 shows DTA profiles of the atomized coarse powders and cast ingot.



Fig.3 Differential thermal analysis profiles of AMX602 cast ingot billet and as-received coarse SWAP powder.

The extremely large exothermic peaks detected at 723 K in both profiles are caused by changing the heating rate from 10 to 5 K/min. The large endothermic peaks are obviously detected at 798~800 K in both, and correspond to a melting point of Al₂Ca intermetallics¹⁷⁾. The endothermic peak at about 880 K is due to the appearance of a liquid phase of this alloy. According to the above results, the extrusion temperature of the atomized powders and cast ingot should be controlled less than melting point of Al₂Ca intermetallics at 798 K. In this study, the pre-heating temperature of the green compact and cast ingot material is set at 573~673 K. Figure 4 shows XRD patterns of the input raw material and its extruded alloys at 573 K and 673 K. In the case of SWAP powders shown in (a), a magnesium oxide (MgO) peak is detected at 2 $\theta = 43^{\circ}$, which is caused by oxidation of molten Mg alloy droplets during water atomization. No Al₂Ca intermetallic peak is shown in the profile of the raw powder, but it is detected in the extruded alloys. This means that Al and Ca are in solid-solution in the matrix of the as-received raw powder by rapid solidification, and Al₂Ca compounds are precipitated after hot extrusion. The peak intensity of Al₂Ca gradually increases with increase in the extrusion temperature. That is, the of precipitated Al₂Ca intermetallics amount increases by a larger thermal history in pre-heating. On the other hand, as shown in (b), the cast ingot material before extrusion originally contains coarse Al₂Ca intermetallics crystallized during casting. There is no remarkable difference in the profile of the wrought alloys extruded at between 573K and 673K.



Fig.4 X-ray diffraction patterns of input raw material and AXM602 alloys extruded at 573K and 673K using SWAP powder compact (a) and cast ingot billet (b).

Figure 5 reveals SEM observation results on the extruded AMX602 alloys by using the powder compact (a) and cast ingot billet (b). The extrusion temperature is 573K and 673K. The powder extruded materials show extremely fine grains of $0.3 \sim 1.1 \,\mu$ m in diameter, compared to the cast ingot extruded ones shown in (b). With regard to the effect of the extrusion temperature on the grain size of the powder extruded material, the mean grain size of the wrought alloy at 573K calculated by the image scanning soft ware was $0.45 \,\mu$ m, and that of the extruded alloy at 673K was $0.8 \,\mu$ m. The larger thermal history during pre-heating in the latter at 673K causes a small grain growth after dynamic recrystallization in hot extrusion.



Fig.5 Scanning electron microscope observation on AMX602 alloys extruded 573K and 673K using SWAP powder compact (a) and cast ingot billet (b).



Fig.6 SEM-EDS analysis on intermetallic compounds of AMX602 alloy extruded at 573K using cast ingot billet.

The amount of precipitated Al₂Ca intermetallic compounds, which correspond to white fine particles of 100~300 nm distributed in the matrix, of the extruded alloy at 673K is larger than that of the wrought alloy at 573K. On the other hand, Fig. 5 (b-1) indicates that the AMX602 wrought alloy extruded at 573K using the cast ingot billet consists of fine α -Mg grains of 1~3µm via dynamic recrystallization. Some of large grains of 5~10µm are observed in the matrix. The mean grain size of the extruded materials at 573 K is 1.96um. However, when employing the higher pre-heating temperature at 673K, a lot of coarse grains over 10µm exist in the matrix as shown in (b-2) and a few fine grains less than 3µm are observed. The mean grain size is 3.29µm. Compared to the microstructure shown in (b-1), the grain growth and coarsening of (b-2) certainly occurred during extrusion at higher temperature of 673K after dynamic recrystallization. Both wrought alloys using cast ingots contain coarse intermetallic compounds with irregular morphologies. Compared to AMX602 cast ingot material, some small compounds are observed because the severe plastic deformation during hot extrusion caused the fragmentation of coarse and brittle intermetallics distributed in the matrix.



Fig.7 Dependence of micro-Vicker's hardness on grain size of extruded AMX602 alloys.

As shown in **Fig.6**, SEM-EDS analysis results on the specimen extruded at 573K shown in **Fig.5 (b-1)** indicate the intermetallic dispersoids are mainly Al_2Ca compounds, and exist at grain boundaries. A very few small compounds with spherical shapes correspond to Al-Mn intermetallics. Both intermetallics are typical of the conventional AMX602 cast ingot ¹⁷.

Figure 7 shows the micro hardness dependence on the grain size of the extruded AMX602 alloys fabricated by using SWAP powder compacts and cast ingot billets. Micro-Vicker's hardness of each material shown in Fig.5 is as follows; 113 Hy (a-1), 94.3 Hv (a-2), 77.0 Hv (b-1), and 69.9 Hv (b-2). The hardness of wrought alloys using atomized powder compacts is larger than that using the cast ingot. This is due to the extremely fine grains and very small intermetallic compounds of the former materials as mentioned in Fig.5. It reveals that the micro hardness is proportional to $d^{-0.5}$ (d; α -Mg grain size), and Hall-Petch behavior is shown in these data. Figure 8 indicates a dependence of tensile strength (TS) and yield stress (YS) of the extruded AMX602 alloys on the pre-heating temperature. TS and YS of the wrought alloys using the cast ingot billets is 298~311 MPa and 204~251 MPa, respectively. It is reported that the content of Ca included AM60 alloys is effective for not only their non-combustibility but also mechanical properties because coarse intermetallics of Al₂Ca with irregular shapes shown in Fig.5 (b) cause the decrease of the tensile properties¹⁷⁾. Therefore, when using the cast ingot billet, the maximum content of Ca is 2 mass%. On the other hand, the alloys using the SWAP powder compacts show an extremely high strength of 391~452 MPa TS and 358~428 MPa YS.



Fig.8 Tensile strength, yield stress and elongation dependence on pre-heating temperature.

This is due to the very fine α -Mg grains and Al₂Ca intermetallics shown in Fig.5 (a). In particular, small Al₂Ca precipitated compounds formed by rapid solidification are significantly useful for the dispersion strengthening effect of these magnesium alloys. Concerning their dependence on the temperature, both materials reveal a decrease of TS and YS with increase in the pre-heating temperature due to the grain growth and coarsening as shown in Fig.5. In particular, the decrement of the strength of SWAP powder extruded alloys is larger than that of extruded materials. cast ingot The fine microstructures via dynamic recrystallization of the former are very sensitive to the temperature, and the grain growth by solid-diffusion easily occurs when a higher pre-heating temperature is applied. As shown in Fig.8 (b), the elongation increases with increasing the temperature. The SWAP powder extruded alloy at 623K shows a good balance of 422 MPa TS and 14.2% elongation. As shown in Fig.9 (a), fine dimple fractured patterns, which mean typical fractures inside α -Mg grains, are observed, and no fracture at the primary particle boundaries is revealed by SEM observation.



Fig.9 SEM observation on fractured surface of tensile test specimen of extruded at 623K using powder compact (a) and cast ingot (b).

The fragmentation of intermetallic compounds at the fractured surface is not observed. Therefore, this material shows a high strength and good ductility. On the other hand, **Fig.9 (b)** indicates the fractured surface of the cast ingot extruded alloy at 623K. It also shows dimple fractured patterns. However, the coarse brittle intermetallic compounds include some cracks marked with white arrows, and they correspond to the initiation and propagation of the fracture.

4. Conclusions

The non-combustible AMX602 magnesium alloys were fabricated by extruding the green compacts of rapidly solidified coarse powders with 1~4 mm via SWAP. They showed extremely fine α -Mg grains of 0.3~1.1µm in diameter from dynamic recrystallization. Fine Al₂Ca compounds with a particle sizes of 100~300 nm were precipitated during hot extrusion, and uniformly distributed in the matrix. Compared to the AMX602 extruded alloys using the cast ingot billets, TS and YS of the powder extruded materials showed a significant increment of 30~45%. The optimization of the pre-heating temperature before hot extrusion was effective in forming very fine recrystallized α -Mg grains and intermetallic dispersoids. For example, a good balance of 422 MPa TS and 14.2% elongation was obtained when employing the pre-heating temperature at 623 K.

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