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Author(s)	Elsayed, Ayman; Imai, Hisashi; Umeda, Junko et al.
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Effect of Consolidation and Extrusion Temperatures on Tensile Properties of Hot Extruded ZK61 Magnesium Alloy Gas Atomized Powders via Spark Plasma Sintering†

ELSAYED Ayman*, IMAI Hisashi**, UMEDA Junko** and KONDOH Katsuyoshi***

Abstract

The effects of powder consolidation and extrusion temperatures on the microstructure and the tensile properties of hot extruded ZK61 alloy powder were investigated. The powder was produced by the rapid solidification technique of spinning atomization. Consolidation of the powder was done using the spark plasma sintering process at 200, 300 and 400 ℃. Hot extrusion was then performed at 250, 300 and 400 ℃. The tensile properties were then evaluated and correlated to the Microstructural features, i.e. grain size. The results show that the use of rapidly solidified atomized powder could lead to improved mechanical response as a result of the refinement of both grain and precipitated compounds which are obtained through the use of powder metallurgy process. These results, along with other previously reported studies, prove that powder metallurgy technique can help to extend the applications of Mg-alloys in higher load carrying components.

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

1. Introduction

Magnesium alloys are now receiving 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. However, these applications are still limited due to the low ductility at room temperature [1]. Different attempts aiming to improve the mechanical properties of Mg alloys have been performed. ZK61 alloy has been extensively investigated and introduced to the industry. Zr addition is known to improve the grain refinement of Mg alloys [2,3]. It also has the strengthening effect of forming fine compounds that enhance the tensile properties of the alloy [4]. To further improve its properties, other alloying elements have been added to the alloy including rare earth elements Ce, Gd and more specifically Y [5-8]. The mechanism of grain refinement was also the prominent one associated with Y-additions. The effect of grain refinement on the tensile strength is well known through the Hall-Petch relationship in which the strengthening factor of Mg is higher than that of other materials [9]. Grain refinement could be obtained through different techniques including alloying with other elements, as mentioned above. However, the use of the powder metallurgy technique has provided promising improvements in the tensile properties of Mg-alloys [10,11]. The use of rapidly solidified alloy powder has resulted in extensive microstructure refinement in both the grains and precipitated compound particles due to its effect on the super saturation of the alloying elements. This improvement in the strength of Mg-alloys exceeds that obtained using conventional cast materials [12]. The aim of this study is to investigate the effect of using rapidly solidified powder metallurgy of ZK61 alloy prepared using different consolidation and extrusion temperatures on its tensile properties.

2. Experimental procedure

Rapidly solidified ZK61 alloy powder that contain 5.2 wt.% Zn and 0.34 wt.% Zr was used in this study. The powder, provided by an industrial company, was produced using spinning atomization in an argon gas atmosphere. This process resulted in powders with ultimately homogenous round morphology with an average particle diameter of about 110 microns. Such morphology improved the flow ability of powder while handling. It also decreased the surface area of the powder particles, which resulted in safer processing of the Mg powder. Microstructure analyses of both the atomized powder and extruded bars were performed using an optical microscope and scanning electron microscope

† Received on December 18, 2009

* Graduate Student
** Specially Appointed Researcher
*** Professor

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(SEM, JEOL: JSM-655F). Energy Dispersive X-ray Spectroscopy (EDS, JEOL: EX-64175JMU) attached to SEM was used to analyze the compounds present. X-ray Diffraction (XRD) analysis was used to investigate the phases present 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 powder was consolidated using Spark Plasma Sintering (SPS, Sumitomo Coal Mining: SPS-1030). SPS was carried out on 60 g of powder using a carbon die with the diameter of 42 mm at different temperatures in vacuum under the pressure of 30 MPa. The temperatures used were 200, 300, and 400 ℃. Heating was done at a rate of 10 ℃/min followed by holding at the sintering temperature for 30 min. Consolidated powder billets were then extruded using the 2000 KN hydraulic press machine at temperatures of 250, 300 and 400 ℃. Extrusion was performed using a die that produces extruded rods of 7 mm, which was 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. 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 a universal testing machine (Shimadzu: Autograph AG-X 50KN) 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

The powder used in this study was produced using rapid solidification through the process of spinning atomization. This process resulted in the formation of a super saturated matrix of α-Mg grains, as shown in **Fig. 1**. Along the boundaries of these grains a small amount of tiny networks of second phase exist, which was confirmed to be Mg_4Zn_7 compound using XRD analysis, as shown in **Fig.2**. This structure with the tiny morphology of compounds is expected to improve the properties of the extruded material as it improves the grain refinement.

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 by using a muffle furnace, due to localized heating during the consolidation process. However, only the microstructure of the extruded ZK61 alloy is discussed below.

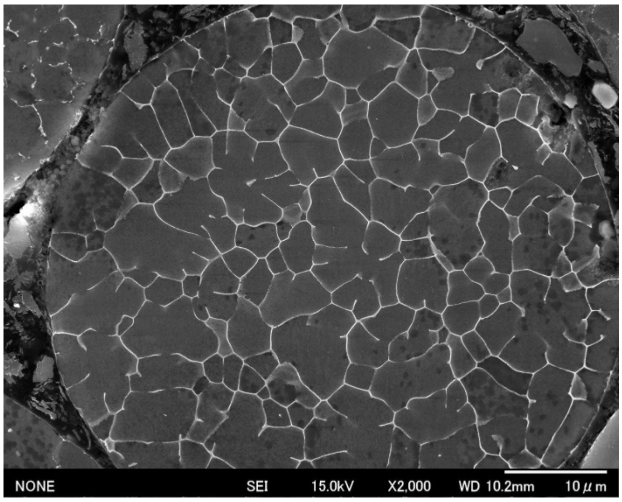

Fig. 1 Microstructure of atomized powder of ZK61 alloy.

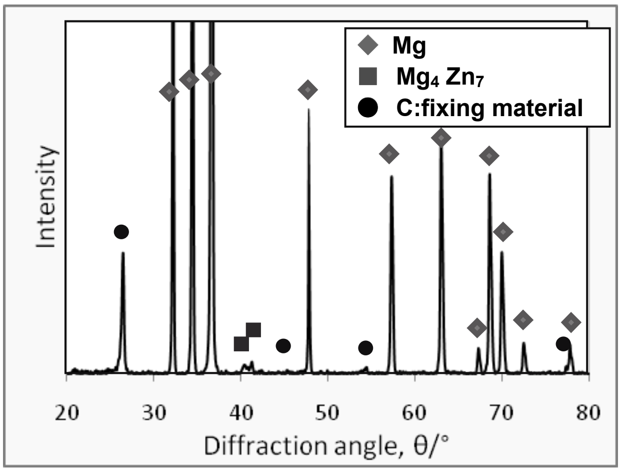

Fig. 2 XRD patterns of atomized powder of ZK61 alloy.

Figure 3 shows the observed optical microstructure of extruded ZK61 alloy. Only 4 examples of the used SPS and extrusion conditions were selected to show the effect of the preparation conditions on the microstructure of the extruded alloy. The grain size, which was calculated by the image analysis software, was shown to decrease as the extrusion temperature decreased. Generally, the extruded atomized powders resulted in very fine and uniform grain sizes in the order of 1 to 2 microns, while extruded cast billets of previously reported research on matching alloy resulted in coarser grains in the order of 10 microns [12)]. The microstructure, shown in **Fig. 3**, along with the results of XRD, not shown herein, confirms that extruded atomized powder materials indicate that Mg_4Zn_7 compound is well dispersed in the structure.

It can also be shown from **Fig. 3** that dynamic recrystallization has occurred homogenously during the extrusion process, which resulted in monotonic microstructure with homogenous grain sizes. In contrast, previously reported microstructure of extruded cast ZK61

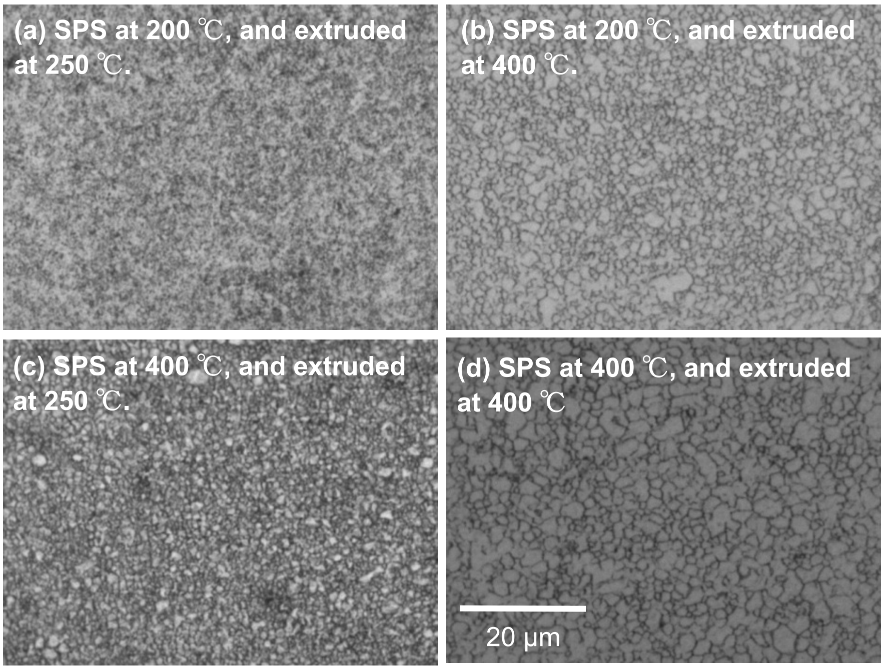

Fig. 3 Optical microstructures of extruded ZK61alloy powder

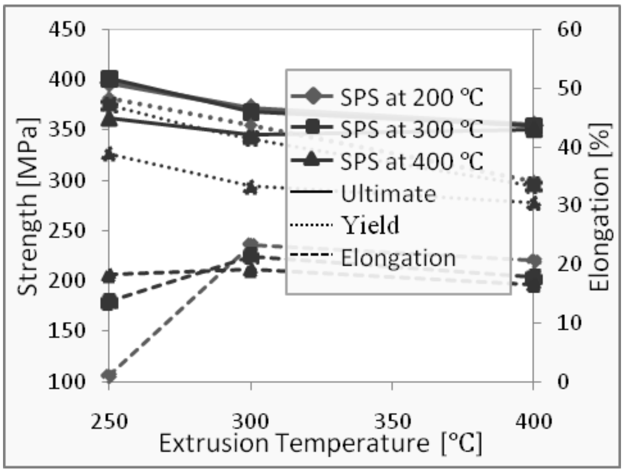

Fig. 4 Dependence of tensile properties of extruded ZK61 alloy powder on consolidation conditions.

alloy was characterized with bimodal grain sizes as a result of the non-homogenous recrystallization behavior associated with coarse grained cast microstructures.

Figure 4 shows the dependence of tensile properties on the extrusion temperatures. The higher extrusion temperature causes the lower yield and tensile strengths of extruded ZK61 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. This leads to the formation of finer microstructures that finally lead to improved mechanical response. It can also be shown that specimens consolidated at lower temperatures showed better tensile properties compared with those consolidated at higher temperatures in SPS. This can be attributed to the limited grain growth associated with SPS at higher temperatures. It can also be shown from **Fig. 4** that the use of rapidly solidified atomized Mg powders could lead to improved levels of tensile strength while maintaining promising values of elongation in the range of 10 to 22 %, except for the specimen consolidated at 200 ℃ and extruded at 250 ℃. This result shows that the consolidation conditions used for this specimen were not enough to produce good bonding between powder particles after extrusion. 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 the conventional sintering.

The hardness [Hv] values of the extruded ZK61 alloy powder showed a decrease in the hardness as the extrusion temperature increased a behavior that is consistent with that of tensile properties, as shown in **Fig.**

5. It can also be shown that the effect of the SPS temperature on the hardness is almost the same as that of the extrusion temperature.

By plotting the yield strength against the inverse of the root of the grain size, as shown in **Fig. 6**, the effect of grain refinement becomes clear on the tensile properties of the extruded ZK61 alloy, according to Hall-Petch relationship. The strengthening factor in this case has the value of about 0.16 $MPam^{-0.5}$, which is slightly lower than previously reported 0.17 to 0.22 MPa $m^{-0.5}$, but still shows the promising effect of grain refinement on the tensile response of Mg-alloys [9)].

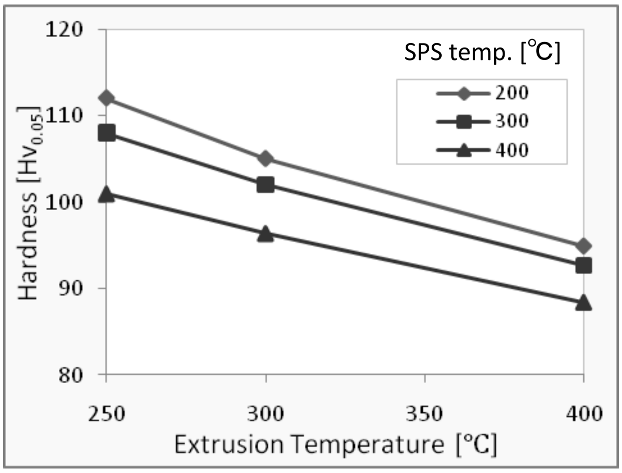

Fig. 5 Hardness of extruded ZK61 alloy powder for various temperatures of SPS and extrusion.

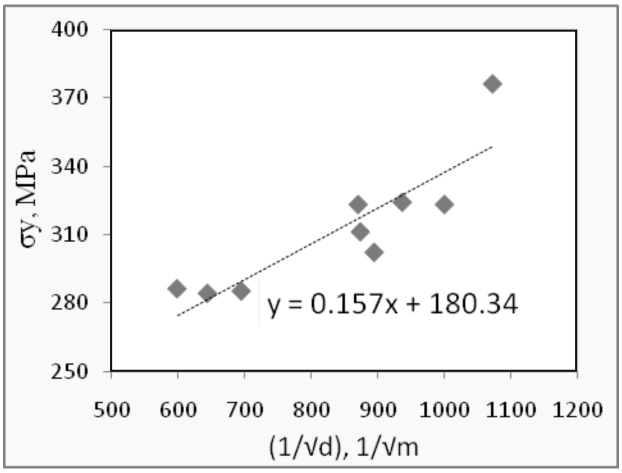

Fig. 6 Hall-Petch relationship of extruded ZK61 alloy.

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, as shown in **Fig. 7**. Generally, the fracture surface of all the fractured specimens showed dimpled pattern as an indication of ductile fracture. However, the size of dimples varied slightly among different consolidation and extrusion conditions of atomized powders. The tensile results of the extruded ZK61 alloy powder suggest that the strength of magnesium alloys can be effectively improved through grain refinement. Only a slight decrease within extruded atomized powder in the grain size by extrusion at lower temperature could lead to a drastic increase in the strength.

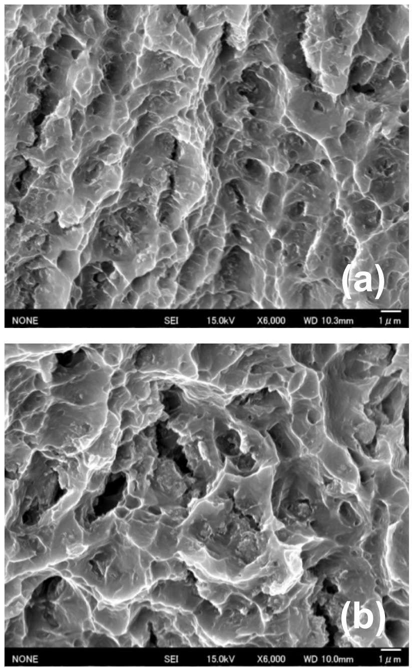

Fig. 7 Fractography of tensile test samples, SPS at 200 ℃ and extruded at 250 ℃ (a), SPS at 400 ℃ and extruded at 400 ℃ (b).

4. Conclusions

The use of rapidly atomized Mg alloy powder could lead to an effective improvement in the mechanical response of extruded ZK61 alloy by refining microstructure, compared to that of previously investigated extruded cast material. Extrusion of atomized powder at lower temperatures could lead to the extreme improvement in the strength of extruded ZK61 alloy. The tensile strength value of up to 405 MPa 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, as shown through the Hall-Petch relationship in which the strengthening factor is in fair conformance to previously reported values.

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