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Synthesis and microstructure control of Mg alloy powder composites by multi-extrusion

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A B S T R A C T

Control of the microstructure of Mg-alloy powder composites was successfully conducted using the powder extrusion process. The resultant microstructure was analyzed as a function of the number of extrusions and area reduction ratio. The grain size (r_n) was proportionally reduced as the number (n) of extrusions and the area reduction ratio (R) increased. The variation in grain size agreed well with the value estimated using an equation of $r_n = r_{n-1}/R^{1/2}$. The effect of the microstructure control on the mechanical properties will also be discussed. This investigation identified that the process was not only useful for controlling the microstructure, but also abbreviating the de-canning step among the alloy powder consolidation frequently used.

1. Introduction

Microstructure is a key factor controlling the properties of materials, since the strength usually increases as the microstructure becomes finer. Among various approaches of microstructure control, plastic deformation [1,2] and rapid solidification [3,4] processes have been frequently used. The latter is used to control the microstructure during the solidification from liquid phase, whereas the former is conducted with solidified type. As an approach of plastic deformation, repeated extrusion was reported to be useful to refine especially the microstructure of powder type materials [5,6]. Easy anticipation of microstructural variation in size is an advantage of the process, since it changes dependent on the extrusion ratio and the number of extrusions. Applying the principle to powder metals can easily produce uniform two phase structures (composites), since the powders are usually extruded after canning. Again, one of those phases is from the powder and the other from the can containing the powder. Thus, the repeated extrusion as a powder metallurgy (PM) process has another advantage in making composite structures with various compositions.

Microstructure control can be optimized by a combination of both rapid solidification and repeated extrusion, since the process controls the liquid and solid phases concurrently.

In this investigation, fabricating two phase structures and controlling the microstructure were conducted by repeated extrusion using rapidly solidified Mg–Zn–Y alloy powders and Cu cans. Mg-alloys have received much attention due to their ultra-light weight [7,8]. However, it is necessary to improve their low mechanical properties to expand their application. Here, Mg–Zn–Y alloy powders were produced using a gas atomization process, which is a well known process of rapid solidification to refine the microstructure. The powders were canned in a Cu can, which presents an excellent plastic flow during extrusion, and extruded repeatedly. Examination of microstructural changes and changes in material properties was conducted as a function of the number of extrusions.

2. Experimental

$Mg_{97}Zn_1Y_2$ powders were prepared using a high-pressure gas atomization process by re-melting the master alloy at 200 K above the melting point. The Ar gas pressure used provided during the atomization was about 2 MPa, and the melt flow rate, as estimated from the operating time and the mass of atomized melt, was about 4–5 kg/min. The particles as atomized are almost spherical in shape, irrespective of the particle size.

The powders as atomized were shielded using a copper can of 30 mm in outer diameter and 1.5 mm in thickness, degassed at the temperature of 573 K for 20 min using a conventional rotary pump down to 10^{-3} Torr, and finally extruded at 673 K with an area reduction ratio of 10:1. The diameter of the extruded bar was approximately 8.8 mm. In order to perform the second extrusion, the first extruded bar was cut horizontally into 5 pieces with the same diameter, then reloaded in a new copper can and extruded again at the same temperature and reduction ratio. The

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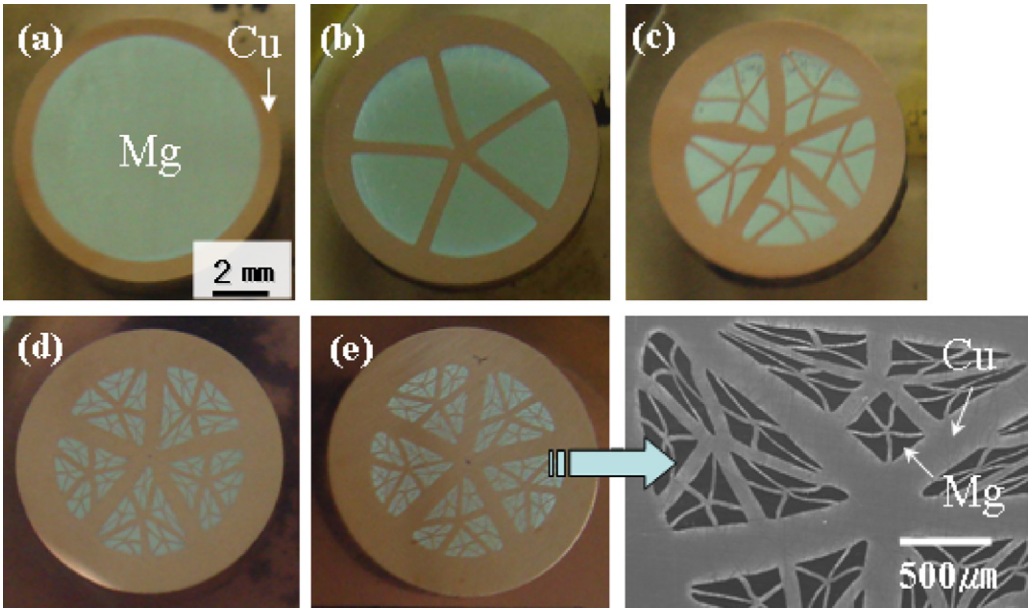

Fig. 1. Photos taken from and SEM micrographs of the $Mg_{97}Zn_1Y_2$/Cu composite at transverse direction of extrusion: (a) first-, (b) second-, (c) third-, (d) fourth- and (e) fifth-pass extruded bars.

3rd extrusion was also conducted using the 5 pieces of 2nd extruded bars, as the 2nd extrusion did. This process was repeated until the fifth extrusion. To understand the microstructural changes that occurred during the repeated extrusion, the bars were examined by scanning electron microscopy (SEM; JSM 5410). The mechanical properties of samples were measured at room temperature under tensile loading, where the dimension of the round test specimens was 8 mm in diameter and 40 mm in length.

3. Results and discussion

Fig. 1(a)–(e) shows the SEM photos taken from the $Mg_{97}Zn_1Y_2$/Cu composites fabricated using the multi-extrusion process, in which Fig. 1(a), (b), (c), (d) and (e) are the plain view of each bar obtained after the first-, second-, third-, fourth- and fifth-pass extrusion, respectively. In the longitudinal direction, each array is in the shape of filament. In the figure, the composite presents the uniform distribution of $Mg_{97}Zn_1Y_2$ (inner part) and copper phases (rim part). After the first extrusion, the $Mg_{97}Zn_1Y_2$ fibers (indicated as Mg in the figure) of about 8.21 mm in diameter were encapsulated by the copper of about 1.0 mm in thickness. As the number of extrusion increased, both the phases became fine, finally being 110 μm for the Mg phase after the fifth extrusion.

Table 1 lists the size variation of Mg fibers with the extrusion number. For comparison, the data of size variation calculated were given. The anticipation was conducted using Eq. (1) given below:

$$r_n = \frac{r_{n-1}}{R^{1/2}} \qquad (1)$$

where R is reduction ratio and r_n is the resultant radius of the extruded bar after n pass [6].

The equation is useful to anticipate the change of microstructure happened during the times of extrusion, since the extrusion is a well known working processes for making the products dense, hard and homogeneous. In addition, the microstructural variation during extrusion corresponds to the number of repetitions of extrusion and the area reduction ratio. Again, such a repeated extrusion makes the resultant microstructure fine as much as the reduction ratio allows.

Table 1
Resultant diameter of extruded bar calculated and measured with the number of extrusion.

Pass of extrusion	1	2	3	4	5
Calculated (mm)	8.54	2.70	0.85	0.27	0.09
Measured (mm)	8.21	2.91	0.99	0.44	0.11

According to the equation, the diameter of the extruded rod varies from 8.54 mm after the first extrusion to 2.70 mm after the second, 0.85 mm after the third, 0.27 mm after the fourth, and 0.09 mm after the fifth extrusion. The calculated data agreed well with the experimental results within a discrepancy of less than 5%. The mismatch may be due to factors as follows: (1) the difference of plastic flow rate between the first extruded bar and copper and (2) spaces produced between the cylindrical bars after the first extrusion. Thus, the differences can be modified by controlling these factors.

Calculating the size of a filament in accordance with Eq. (1) suggested that the size became finer to 30 μm after the 6th and 0.9 μm after the 7th extrusion as shown in Fig. 2. The degree of

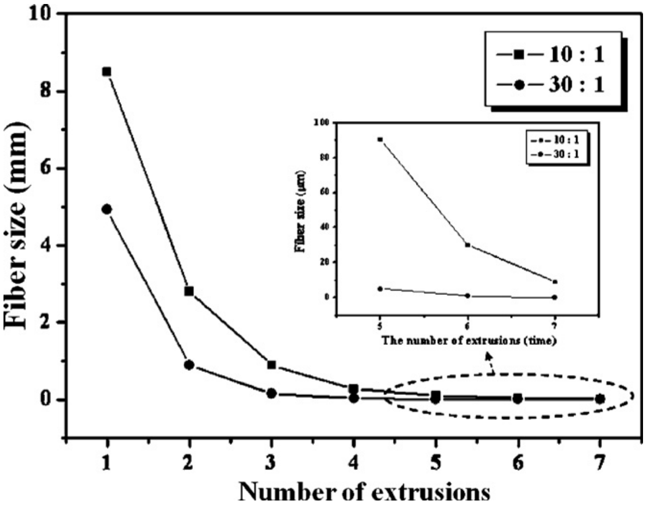

Fig. 2. Calculation of the fiber size as a function of the extrusion number and extrusion ratio.

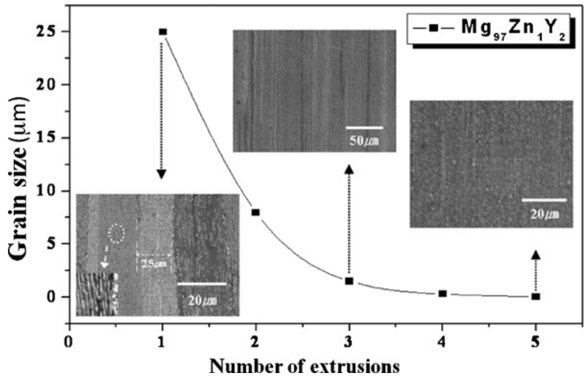

Fig. 3. Variation of grain size with the number of extrusion.

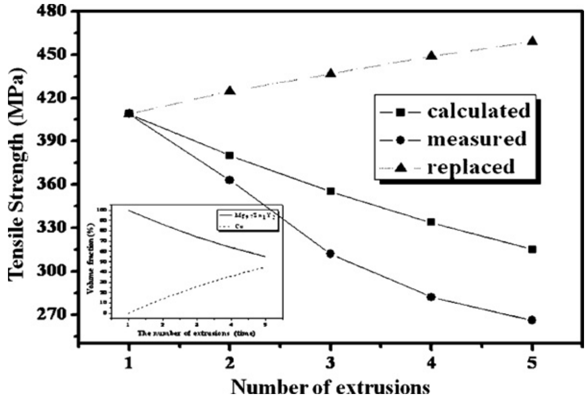

Fig. 4. The comparison of tensile strengths of $Mg_{97}Zn_1Y_2$ composites between the values calculated for a Cu can, measured for a Cu can, and calculated for an Al7075 can as a function of the number of extrusion.

reduction became larger as the area reduction ratio increased to 30:1. After the same 7th extrusion, the fiber size of 9 for $R = 10{:}1$ turned to 100 nm for $R = 30{:}1$. Therefore, the microstructure could be controlled using both the extrusion ratio and the repeated extrusion.

Fig. 3 shows the microstructural change of $Mg_{97}Zn_1Y_2$ as a function of passes of extrusion. The micrograph was taken longitudinally to the extrusion direction. As the extrusion repeated up to 5 times, the grain size became fine from 25 μm to 8, 1.5, 0.3 and 0.05 μm, respectively. The micrographs also indicate refinement with additional extrusions. Corresponding to this result and Eq. (1), the grain refinement, even to nano-size, was successfully achieved by controlling the number extrusion as well as the extrusion ratio.

Fig. 4 presents the tensile strengths of extruded bars obtained by the calculation and the measurement as a function of the number of extrusions. The calculated strength was obtained using the rule of mixture, in which the initial strength of Mg alloy and Cu were of 409 MPa and 200 MPa, respectively. Both the values were simultaneously decreased as the number of extrusion increased. The calculated value varied from 409 to 315 MPa, while the measured value varied from 409 to 266 MPa. The decrease in strength corresponded to the increases in the volume fraction of weaker phase (Cu) from 0%, 14%, 25%, 36%, and 45% with the extrusion number (inset of Fig. 4).

The lower strength in the experimental compared to the calculated might be due to the significant increase of the weak Cu phases. As the volume of weak phase increased, the resultant strength would decrease much more than the value expected by rule of mixture. However, this process could strengthen the composite if the copper was replaced by a stronger material (higher tensile strength). For example, if it was substituted by Al7075, the calculated strength would become much higher than that with the copper (indicated as calculated/Al7075′ in Fig. 4).

4. Conclusion

Microstructure control was achieved simultaneously with preparation of $Mg_{97}Zn_1Y_2$/Cu composites by multi-pass extrusion. The diameter and thickness of $Mg_{97}Zn_1Y_2$ fibers were reduced depending on the extrusion ratio and the number of extrusions. When the number of extrusions was increased from one to five, $Mg_{97}Zn_1Y_2$ fibers were refined from 8.2 mm to 0.11 mm (about 70%). The extruded bar consisted of a uniform dispersion of $Mg_{97}Zn_1Y_2$ fibers in the copper.

Such a variation of microstructure can be predicted within about 5% using an equation, $r_n = r_{n-1}/R^{1/2}$. The calculated microstructure change is close to that obtained by extrusion. The calculation indicated that the multi-pass extrusion would be obtained into very fine structure. The measured and estimated mechanical properties are known to vary depending on the volume fraction. The tensile strength of the composite decreased from 409 to 266 MPa from the first to fifth extrusion, in agreement with the predictions. Substitution of Cu with a stronger material, e.g., Al7075 could lead to a strengthening effect.

Therefore, increment of extrusion pass will refine the microstructure to the nano-size only using the plastic deformation process.

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