Semisolid forming of Al-10mass%Mg alloy by blending of elemental powders

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By semisolid processing of metals, not only complicated shapes, but also products with a quality close to forged materials can be produced. In this research the forming temperature range, formability and mechanical strength of Al-10mass%Mg alloy formed by elemental blended powders semisolid forming are investigated. The powders used were gas-atomized aluminum as the high melting point powder, and ball-milled Al-20mass%Mg and Al-30mass%Mg powders as the low melting point ones. Differential thermal analysis has been used to determine the forming ability range. Tensile strength of the product has been evaluated by tests performed on $\phi 10 \times 50$ mm specimens. The relation between tensile strength and heat treatment has been also studied. Furthermore, the formability was investigated by forming cup shaped samples (outer diameter ϕ 424× length 50 mm) with a thin wall (1 mm). © 2003 Kluwer Academic Publishers

1. Introduction

Elementally blended powders semisolid forming (EPSF) process utilizes two high and low melting point powders as the starting materials. The process was first proposed by Young et al. [1] in mid 1970s. In this process a slurry is prepared by melting only the low melting point material which is then cast in a metallic mold. Using this process, authors tried the forming of Ti-Al alloys or intermetallic compounds and showed that highly active and high melting point alloys such as Ti-Al alloys can be formed in the open atmosphere and in steel molds [2-4]. However, porosities were formed during the heat treatment of the formed products, necessitating further special contrivances. The porosities are formed due to the sudden alloying of the elements during the heat treatment process. Accordingly, it can be concluded that the porosities can be controlled if the powders are alloyed in advance, or the abrupt alloying is prevented by oxidizing the surface of the particles. However, there has been no research utilizing alloyed powders in EPSF processing hitherto.

In this research, using aluminum and aluminum magnesium alloy powders as the starting materials, Al-10%Mg alloy was formed by EPSF process and its forming conditions, structure, formability and tensile strength were basically investigated.

2. Process and the powders

In the EPSF process two metal powders, one with a high and the other with a low melting point, are used as starting materials. They are well blended at room temperature and then poured into a mold where an appropriate amount of pressure is applied and a preform is produced. The preform is then heated to a temperature where the low melting point metal is melted and a slurry mixture is obtained before being poured into a preheated mold for forming where an appropriate amount of pressure is applied to obtain a green compact (GC). The GC, being just a loose compaction of the powders, is again heat treated to improve the diffusion between the powders and form an alloy.

In this research, the high melting point metal was an aluminum powder produced by gas atomization $(-100 \ \mu\text{m})$ and the low melting point metal was an Al-20mass%Mg or Al-30mass%Mg alloy. The Al-30Mg alloy, being extremely brittle and easily pulverized, was powdered in a ball mill. Fig. 1 shows the SEM observation of the powders used. The shape of the aluminum particles is almost spherical. On the other hand, Al-20M and Al-30Mg particles are massive, coarse and oxidized on their surfaces. These powders were blended to reach a composition of Al-10mass%Mg. In the cases where either Al-20Mg or Al-30Mg alloy particles are used, the mixture is termed as Al-(Al-20Mg) or 2Al-(Al-30Mg), respectively.

3. Production of the slurry

As was stated earlier, this process is based on producing a slurry by heating the blended powder and pouring it into the mold for forming. However, since the slurry is thermodynamically unstable, depending on the alloys used intermetallic compounds may form, or alloying may take place as soon as the powder with low melting temperature is melted, making the production of the slurry impossible.

Fig. 2 shows the equilibrium phase diagram of Al-Mg alloy, where the eutectic temperature is 723 K, the liquidus temperatures for Al-30Mg is 760 K, and the



Figure 1 Elemental powders.

solidus and liquidus temperatures for Al-10Mg alloy are 788 K and 875 K, respectively. Accordingly, the solidus temperature of Al-10Mg is higher than the liquidus temperature of Al-30Mg alloy and therefore if the blended powder of 2Al-(Al-30Mg) is heated, the Al-30Mg powder will melt first and instantly react with the aluminum and solidify, before being able to form a slurry. For this reason, the temperatures where aluminum and Al-Mg alloys react to form an alloy were determined by differential thermal analysis. To be used as the specimen, 0.1 mg of the powder was compacted in a mold with an inner diameter of 5 mm under a pressure of 150 MPa. The incremental rate of temperature was 0.33 K/s in an argon atmosphere.

The results of the analysis for Al-30Mg, 2Al-(Al-30Mg) and Al-(Al-20Mg) powders are shown in Fig. 3. It is shown that in all of the cases an endothermic reaction is observed at the eutectic temperature of 723 K. The heat released at about 770 K in the case of Al-30Mg is due to the oxidation of magnesium. However, in the case of 2Al-(Al-30Mg) this exothermic reaction is not observed, implying that in the case of the powder blended with aluminum no oxidation of magnesium is taking place and therefore the forming process can be conducted in the open atmosphere. Increasing the temperature to higher values, at about 850 K an endothermic reaction is observed, implying that a partial melting has occurred which is probably due to the alloying with aluminum, with the complete melting taking place at about 910 K. From this figure it can be found that alloying is started at about 850 K. In the case of Al-(Al-20Mg), in which the amount of eutectic is less compared with the other two powders, the heat absorbed due to the eutectic reaction is less, and the temperature at which melting of aluminum powder starts is higher compared with 2Al-(Al-30Mg), being about 860 K.

The blended powder of 2Al-(Al-30Mg) was formed into a $10 \times \phi$ 10 mm bar under a pressure of about 150 MPa with a thermocouple positioned at its center, heated up to a slurry state in an electric furnace and water quenched after being kept at that temperature for 120 s. Photomicrographs of the quenched specimens are shown in Fig. 4. Despite the fact that the Al-30Mg powder is supposed to be in the liquid state at the temperature of 843 K, the shape of particles are similar to those in the solid state shown in Fig. 1, and an evidence of alloying reaction with aluminum powder is not observed at the boundary of both particles. Al-30Mg particles change to a rounded shape at 863 K and an alloying reaction proceeds partially at some areas. White aluminum particles are not observed in the sample at 883 K, implying that the alloying reaction is completely finished. The starting temperature of alloying reaction was estimated to be about 860 K from this experiment. This result is in good agreement with the one obtained from the differential thermal analysis.



Figure 2 Al-Mg phase diagram.

As was stated above, the low melting point powder of Al-30Mg, that forms a liquid phase from the eutectic



Figure 3 Thermal differential analysis of preforms.



Figure 4 Structure of prefoms heated to slurry state and quenched.

temperature, is in a slurry state with solid aluminum in the range of its liquidus temperature, that is 760 K, and 850 K. This range can be used for producing a slurry from the blended powders. The densities of Al-20Mg and Al-30Mg alloys were examined and were found to be $\rho = 2.25$ and 2.44 g/cm², respectively. Based on these values and the phase diagram, the ratio of liquid for 2Al-(Al-30Mg) was estimated. In 2Al-(Al-30Mg) the weight and volume ratios of Al-30Mg powder are 33% and 37% respectively. Therefore, just after the eutectic temperature the ratio of liquid is about 25% that increases as the temperature is increased reaching a value of about 37% at 760 K, above the liquidus temperature of Al-30Mg. This ratio remains constant up to 850 K, at which aluminum starts to melt, and increases as the temperature is increased.

4. Tensile strength test

Clarifying the conditions for producing the slurry of blended powders as is stated above, cylindrical specimens were produced and after heat treatment were tested for tensile strength. The blended powders used were Al-(Al-20Mg) and 2Al-(Al-30Mg). The powders were preformed in a cylindrical mold with an inner diameter of 19 mm under a pressure of 300 MPa. The mold, which is splitable consisting of two ϕ 10 mm and ϕ 20 mm cylinders as is shown in Fig. 5, was preheated to 673 K, filled with a slurry of 833 K and was put under a force of 10 kN. The bars obtained in this way were then heat treated at 693 K for 86 ks and quenched. They were then machined to specimens for tensile strength test with a diameter of 7 mm and length of 14 mm at their straight section.

In addition to the above, Al-10Mg alloy was melted and cast in a mold with an inner diameter of 20 mm and tested for tensile strength and compared with the results obtained for specimens formed by EPSF.

The results of the tensile strength tests of different specimens are shown in Fig. 6. As is clearly shown in this figure, compared with the tensile strength of the cast specimen which is about 180 MPa, the strength of the one formed by EPSF is about 280 MPa at the state of GC, showing even a further slight increase when heat treated. Regarding the elongation, compared with a value of 5% obtained for the cast specimen and those formed by EPSF at the GC state, the result obtained

for the heat treated GC is about 10%. Moreover, the results obtained for strength and elongation of the cast specimens show a large deviation compared with the ones obtained for ESPF, implying the existence of casting defects in the cast specimens due to bad fluid flow



Figure 5 Metal mold for tensile strength specimens.



Figure 6 Tensile strength results of the specimens.



As cast

Green compact

Heat-treated $100 \,\mu$ m

Figure 7 Microscopic structure of the tensile test specimens.

and the susceptibility to gas absorption. Of course, the alloying of Al and Al-Mg alloy powders is almost complete at the forming state and therefore porosities will not be formed even if heat treated.

The microstructures of tensile test specimens are shown in Fig. 7. As is shown in these photos, compared with the EPSF specimens the cast specimens contain large grains of α phase with the β phase segregated in the grain boundary, which is the reason for the lower strength of cast specimens. Furthermore, in the EPSF specimens at the GC state the two powders are already alloyed and the β phase is present in the grain boundary. In experiments conducted on Ti-36mass%Al alloy by the authors [4], in the GC state the two powders were just in blended state and required heat treatment. Moreover, during the alloying heat treatment process porosities were formed, necessitating a second heat treatment. However, in the case of Al-10Mg the two powders are already sufficiently alloyed in the GC state and therefore, as is shown in Fig. 6, a high strength is obtained even without the final heat treatment.

As is shown in Fig. 4, only blending the powders and heating up to 850 K will not result in alloying of the powders. This is probably due to the fact that the Al-30Mg particles when melted are covered by an oxide layer, which acts as a barrier. However, when pressure is applied during the forming stage, the oxide layer is torn and aluminum particles can rapidly react with the melt to form an alloy. In other words, even though the two powders are highly reactive with respect to each other, the oxide layer keeps the two in a slurry state up to high temperatures.

Fig. 7 shows the microstructure of a GC specimen kept at 693 K for 86 ks and quenched. The β phase observed in the grain boundary is decreased compared with the one without heat treatment, implying the dissolution into the α phase. The shape of the remaining β phase is also changed from granular to a flaky shape. The α phase also shows some grain enlargement. However since the β phase does eventually exist in the grain boundary the tensile strength does not show an increase by heat treatment, while the elongation greatly improves. This means that even though in the present alloy system heat treatment of the GC is not necessary, it can improve the elongation.

5. Formability

One of the problems in semisolid forming is the lower formability compared with ordinary die cast methods due to the fact that the slurry contains solid particles. As was stated above, the slurry of 2Al-(Al-30Mg) alloy contains 63% of particles in volume fraction, being a problem during forming. Therefore, by using a metal mold as shown in Fig. 8a, cap shaped specimens were made for formability testing. The size of the cap was ϕ 24 mm in the outer diameter, 50 mm in length and an average thickness of 1 mm.

Eighteen grams of blended elemental powder (2Al-(Al-30Mg)) was formed into a ϕ 20 preform bar under a pressure of 150 MPa and heated to bring into a slurry state. The slurry was then poured into a metal mold for formability testing and GC's were formed under a load of 20 kN. An example of successfully formed sample is shown in Fig. 8b. Depending on the forming conditions, there are some cases in which the slurry could not fill up to the tip of the cavity.

Fig. 9 shows the border line of perfect and imperfect fillings by plotting the temperature of the slurry as the ordinate and the temperature of the metal mold as the abscissa. The symbol Δ shows the case of filling of the cavity with some porosities at the tip of the cap. Even when the slurry temperature is low, if the mold temperature is sufficiently high a perfect filling was obtained and the boundary of the two areas could be indecated by a straight line as shown in Fig. 9. The tensile test specimens mentioned above could be sufficiently formed at the metal mold and slurry temperatures of 673 K and 833 K, respectively. However a complicated shape such as a cap needed higher metal mold and slurry temperatures. Because of the low speed (0.002 m/s) hydraulic pressure apparatus used in these experiments, from the throwing of GC into the mold until the loading of pressure a time period of 15 s was needed. Therefore, there was a temperature drop of the slurry during this period, which was observed to be 20 to 30 K by practical measurements. Since an oil hydraulic press with a low ram speed (0.002 m/s) was used as the loading apparatus, the thixotropic behavior of the slurry cannot be expected in these experiments. If a high ram speed is adopted for forming, the border line in Fig. 9 would shift to lower temperatures.



Figure 8 Metal mold for formability testing (a) and formed sample (b).



Figure 9 Effects of mold and slurry temperature on the formability.

6. Conclusions

Using aluminum and Al-20Mg or Al-30Mg powders as the high melting point and low melting point powders, respectively, the semisolid forming and slurry formation of Al-10mass%Mg alloy was investigated. Tensile strength test specimens were produced by semisolid forming and tested. The results obtained are as follows.

1. Even though Al-20Mg and Al-30Mg powders are rapidly oxidized after melting, when blended with aluminum powder they do not oxidize and can be processed in open atmosphere. 2. For the blended powder, even at temperatures higher than the liquidus temperature of the low melting point powder, there is a temperature range where solid aluminum and liquid Al-Mg alloy can coexist.

3. It was found by differential thermal analysis that the temperature at which aluminum and Al-Mg alloy start to react is 850 K and 860 K for Al-20Mg and Al-30Mg, respectively.

4. The tensile strength of the specimen produced by semisolid forming was 280 MPa, being much higher than that of the cast specimen, that is 180 MPa.

5. In the case of Al-10mass%Mg alloy even though there is no need for diffusion heat treatment, it may improve elongation.

6. For forming complicated shapes, a higher mold or slurry temperature is required.

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