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Some Studies on Hot Extrusion of Rapidly Solidified Mg Alloys

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Rapidly solidified magnesium alloys show great potential for application in automotive and aerospace industries. In this study, Mg-Al-Zn alloys (AZ91) were rapidly solidified by a melt-spinning process to form ribbons. Pulverized ribbons were cold-compacted and then hot-extruded to form rods. During extrusion, a specially designed die with constant strain rate profile was used and found to be advantageous. By properly establishing the complete process, extruded rods of rapidly solidified AZ91 alloys exhibiting good combination of room temperature strength and ductility were produced. Microstructural investigations were carried out on melt-spun ribbons and extruded rods. Effects of extrusion die shape, extrusion ratio, and extrusion temperature on mechanical properties of the extruded rods were also investigated.

Keywords extrusion die profile, hot extrusion, magnesium alloy, rapid solidification

1. Introduction

Among the light metal alloys, magnesium is the lightest structure material except for beryllium, and therefore, magnesium alloys are quite attractive for structural use in the aerospace and automotive industry. However, these alloys have not seen extensive use due to the high chemical reactivity and excessive oxidation tendency of magnesium during melting and casting by conventional methods. Further, lower mechanical strength and poor corrosion resistance of conventionally cast Mg alloys compared with aluminium alloys have also limited their use. Rapid solidification technology offers a possible solution to these problems and therefore has received increasing attention in recent years (Ref 1-6).

Rapid solidification is a process of freezing liquid metal at cooling rates greater than 10^4 K/s during liquid-to-solid transition. The total technology consists of planar flow or jet casting into ribbons, pulverization of ribbons to powder, and consolidation of powder into bulk shapes. This has significant potential due to the high degree of homogeneity of chemistry, extension of solid solubility to many orders of magnitude, and extremely refined microstructure/amorphous phases. As a result, some of the rapidly solidified Mg alloys exhibit an excellent combination of room temperature strength and ductility, which are superior to existing commercial alloys (Ref 1). However, to maintain unique properties of rapidly solidified materials during subsequent consolidation processing (compaction, extrusion, forging, rolling), these processes must be inherently optimized with regard to temperature, strain rate, strain, and friction.

Hot extrusion is one effective consolidation processing method of rapidly solidified Mg alloys. In this study, Mg-Al-Zn alloys (AZ91) with the normal composition Mg-9%Al-1%Zn (wt.%) were rapidly solidified by a melt-spinning process to form ribbons. Pulverized ribbons were cold-compacted and then hot-extruded to form rods. This paper discusses design aspects of extrusion die profile selection and experimental investigations on hot extrusion behavior of these rapidly solidified alloys.

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2. Experimental Study

2.1 Melt Spinning and Cold Compaction

One of the methods of rapid solidification is melt spinning, which involves melting the metal/alloy in inert atmosphere in a crucible with a tiny bottom orifice and ejecting the molten metal through the orifice onto a copper wheel rotating at specified high speed. In the present work, AZ91 alloys were melt-spun in argon atmosphere into thin ribbons (25-50 μm thicknesses) with a width of 2 mm from as-cast rods of 25-30 mm diameter. These as-cast Mg alloy rods were produced by Vikram Sarabhai Space Center, Thiruvananthapuram, India, using die-casting techniques and supplied to National Metallurgical Laboratory, Jamshedpur, India. The pilot melt-spinning unit used to produce ribbons is shown in Fig. 1, and significant features of the unit are listed in Table 1. The spun Mg alloy ribbons and cast rod are shown in Fig. 2. The ribbons were directly cut into flakes of average length 1.5-2 mm. These pulverized ribbons were cold-compacted to form 75 mm diameter cylindrical billets with an average length of 100-150 mm in a 5 MN vertical hydraulic press. During compaction, die walls were lubricated with graphite powder. A compaction pressure of 250-300 MPa was used, which resulted in billets with a density about 75-85% of theoretical value.

2.2 Hot Extrusion

Axisymmetric hot extrusion of these compacted cylindrical billets was carried out in a 5 MN horizontal extrusion press to produce rods of two different diameters (25 and 15 mm) with extrusion ratios of 9:1 and 25:1, respectively. Before extrusion, the billets were preheated in an electric furnace at either 300 or

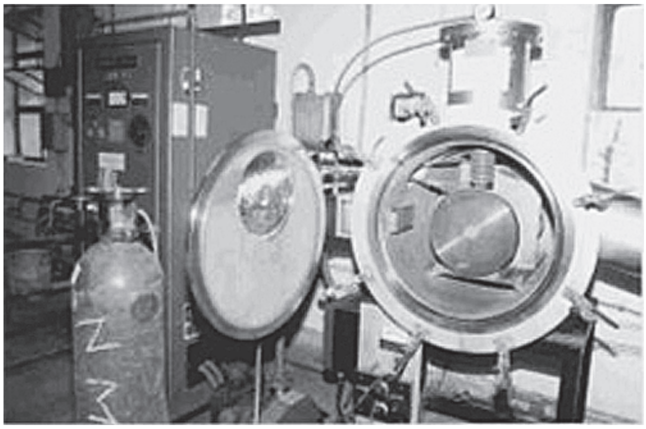


Fig. 1 Pilot melt spinning unit

Table 1 Experimental condition during melt spinning

Diameter of copper wheel	300 mm
Wheel peripheral speed	20-30 m s^{-1}
Diameter of crucible	30 mm
Diameter of orifice	0.3 mm
Cooling rate	more than 10^4 K s^{-1}
Atmosphere	Argon

350 °C for 2 h. To prevent atmospheric contamination during preheating, the billets were wrapped with aluminium foil. After the homogenization period was over, aluminium foils were removed and the billets were transferred rapidly to the extrusion press container maintained at either 300 or 350 °C. The speed of the press ram was in the range of 1-2 mm s^{-1}. During the extrusion, a graphite-containing spray was used to lubricate the die. No lubrication was provided between the bore of the container liner and the outer surface of the billets so that friction prevented the undesirable skin of the billet from sliding and going into the extrudates.

It is well known that a strong correlation exists among the die geometry, extrusion pressure, and the deformation of porous billets during the extrusion. Some preliminary experiments were first carried out to select a suitable die profile for extrusion. A flat-faced dies resulted in poor quality of the extrudates, which had fir-tree cracking on the surface. For the same extrusion speed, a conical die with 45° semidie angle resulted in some improvement in surface quality of the product, possibly due to the more homogeneous flow of metal as compared with flat-faced die. The extrusion pressure for the lubricated conical die was also considerably lower than that for the flat-faced die. However, further improvement in surface quality and mechanical properties of the extrudates was observed with a die having a constant strain rate throughout its deformation zone. The constant strain rate (CSR) die profile for extrusion of porous billet was calculated as described in the foregoing discussion. The compacted billet of 75 mm diameter and extruded rods of 15 mm diameter extruded using CSR die are shown in Fig. 3.

3. Design of Extrusion Die

The shape of the die surface was chosen to satisfy the reduction requirement and to have nearly a constant strain rate

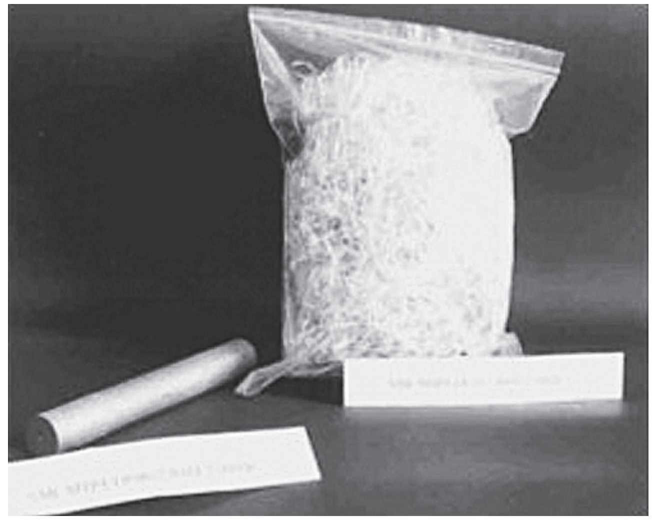


Fig. 2 As-cast rods and melt-spun ribbons of AZ91 alloy

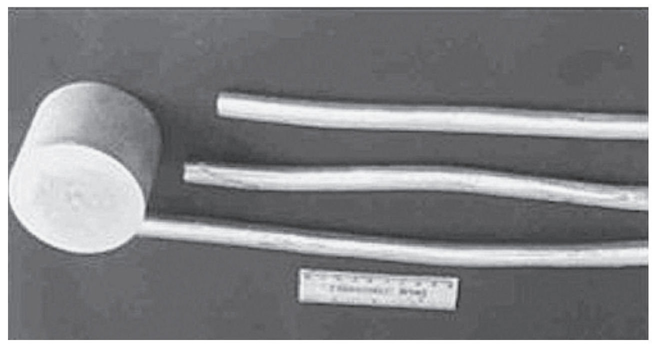


Fig. 3 Compacted billet and extruded rods of AZ91 alloy

throughout the extrusion. To calculate the die profile for constant strain rate, simplified slab (disc) analysis was used here. One major simplification of this method is that metal deforms uniformly in each cross section of the deformation zone. This method is quite justifiable also for porous billets used here because the billets have already been cold-precompacted and exhibit a large initial density (75-85% of theoretical density). Further, high temperature during soaking (preheating) provides a partial sintering of flakes, which prevents the grains from sliding.

3.1. Constant Strain Rate Die

Consider an axisymmetric extrusion process in which compacted material of initial density ρ_p undergoes a strain rate variation $\bar{\dot{\varepsilon}}$ as it flows through the die (Fig. 4). Here, we use the slab (disc) method and assume a disc of material of infinitesimal width l at a distance z from the die entrance. Assuming velocity to be uniform throughout the cross section, the time taken by the strip to move a distance dz is given by:

$$dt = \frac{dz}{v} \quad \text{(Eq 1)}$$

where v is the velocity of the material in the z direction at a distance z and can be determined using constancy of mass as:

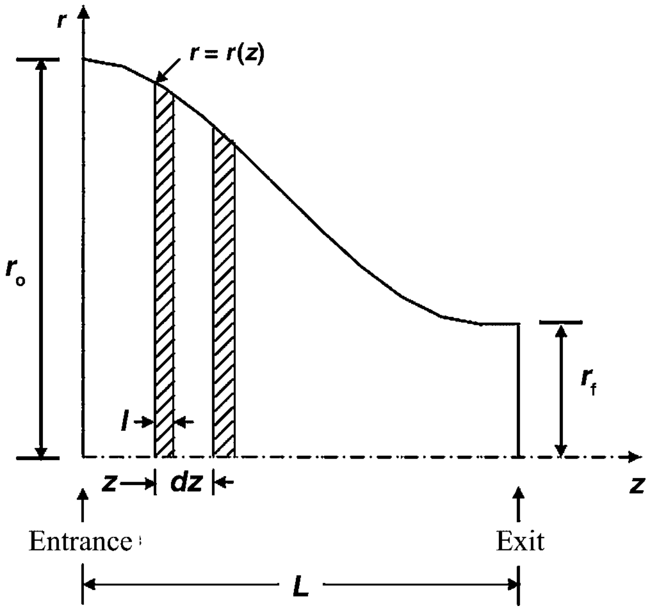


Fig. 4 Calculation of CSR die profile using the slab method

$$v = \frac{\rho_p v_o r_o^2}{\rho r^2} \quad \text{(Eq 2)}$$

where v_o is the entrance velocity of the material and r_o is the initial billet radius. ρ is the density of material at distance z.

If the width of strip at $z + dz$ is $l + dl$, then using the constancy of mass, the corresponding equivalent strain increment can be expressed as:

$$d\bar{\varepsilon} = \frac{dl}{l} = -2\frac{dr}{r} - \frac{d\rho}{\rho} = -\left(\frac{2}{r}\frac{dr}{dz} + \frac{1}{\rho}\frac{d\rho}{dz}\right)dz \quad \text{(Eq 3)}$$

Now, using Eq 1, 2, and 3, the equivalent strain rate that the material experiences during this displacement is:

$$\dot{\bar{\varepsilon}} = \frac{d\bar{\varepsilon}}{dt} = -\frac{\rho_p v_o r_o^2}{\rho r^2}\left(\frac{2}{r}\frac{dr}{dz} + \frac{1}{\rho}\frac{d\rho}{dz}\right) \quad \text{(Eq 4)}$$

It is known that the relative density (ratio of apparent specific mass of porous body by the specific mass of fully dense material) increases during extrusion from the entrance to the exit of the die. In particular, the voids must be eliminated during extrusion so that a sound product with good metallurgical structure is obtained. Here we assume that at the exit of the extrusion, the material reaches its theoretical density ρ_{th} and the density variation is linear along the z direction, such that:

$$\rho = \rho_p + (\rho_{th} - \rho_p)\frac{z}{L} \quad \text{(Eq 5)}$$

Now, if a constant strain rate is imposed on the material being extruded, then Eq 4 in combination with Eq 5 can be integrated to yield the die shape as follows:

$$\frac{1}{r^2} = \frac{1}{\alpha_\rho r_o^2}\left[\alpha_\rho + (1 - \alpha_\rho)\frac{z}{L}\right]\left[1 + (\alpha_\rho \lambda - 1)\frac{z}{L}\right] \quad \text{(Eq 6)}$$

where λ is the extrusion ratio r_o^2/r_f^2 and α_ρ is the relative density (ρ_p/ρ_{th}).

The constant equivalent strain rate is given by:

$$\dot{\bar{\varepsilon}} = \frac{v_o}{L}(\alpha_\rho \lambda - 1) \quad \text{(Eq 7)}$$

It is worthwhile here to make a comparison of CSR die with two other dies (conical and third-degree polynomial) with regard to die surface and equivalent strain rate distribution.

3.2 Conical Die

This die profile is represented by:

$$r(z) = r_o - \frac{r_o - r_f}{L} z$$

In this case, the equivalent strain rate distribution in the z direction can be calculated using slab method to be:

$$\dot{\bar{\varepsilon}}(z) = \frac{2 v_o r_o^2 (r_o - r_f)}{L r(z)^3}$$

3.3 Third-Degree Polynomial Die

This die profile is calculated using third-degree polynomial equation by imposing smoothing condition of flow at the entrance and exit of the die:

$$r(z) = r_o + (r_o - r_f)\left(2\frac{z^3}{L^3} - 3\frac{z^2}{L^2}\right)$$

In this case, the equivalent strain rate distribution in the z direction can be calculated using slab method to be:

$$\dot{\bar{\varepsilon}}(z) = \frac{12 v_o r_o^2 (r_o - r_f) z (1 - z/L)}{L^2 r(z)^3}$$

The die lengths L for extruded rods of diameter 25 and 15 mm were taken to be 50 and 60 mm, respectively. The different die profiles have been plotted to scale in Fig. 5 with r_o = 75 mm, r_f = 25 mm, and die length L = 50 mm. The ratios of equivalent strain rate distribution and extrusion speed for the three die profiles with the above dimensions have also been plotted in Fig. 6.

4. Experimental Results and Discussion

4.1 Microstructure

The melt-spun ribbons as well as the extruded rods of AZ91 alloys were studied under optical and scanning electron microscopes. Transverse sections of the melt spun ribbons were polished after mounting with araldite. Figure 7(a) shows the optical microstructure of AZ91 ribbon showing fine grain structure. A scanning electron micrograph of melt-spun ribbon is shown in Fig. 7(b) depicting grain-like features of size 1-2 μm. Aluminium-enriched regions toward the periphery of each grain of magnesium matrix were noticeable in the form of precipitates. To study the effect of heat treatment on micro-

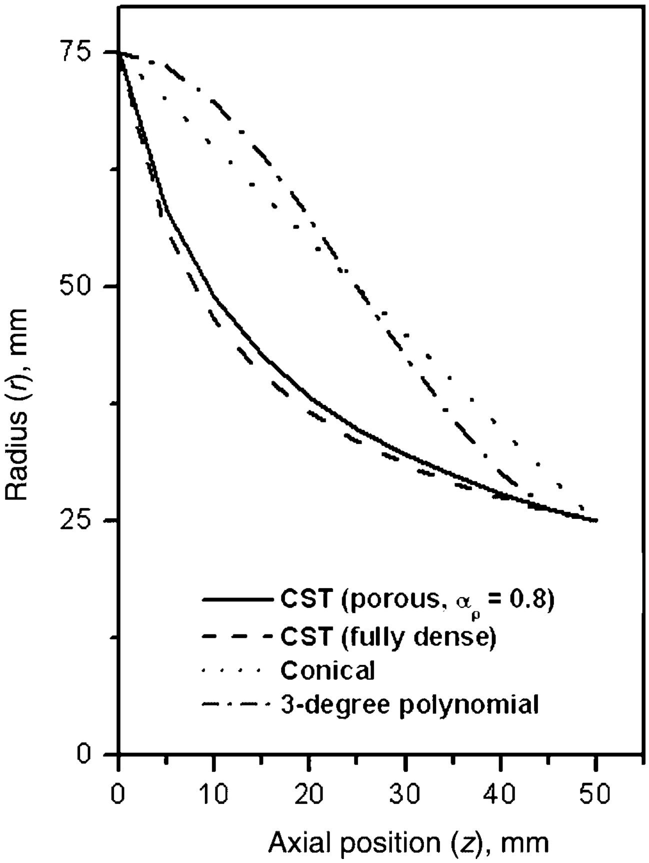


Fig. 5 Variation of die radius with axial position z for different die profiles

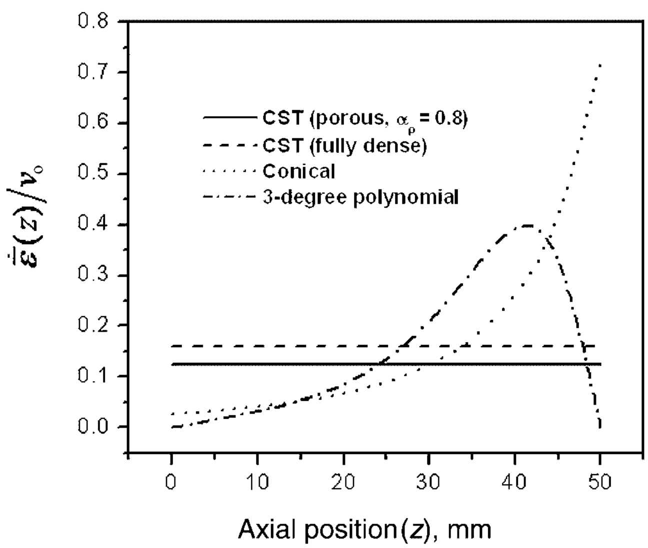


Fig. 6 Variation of ratio of equivalent strain rate and extrusion speed with axial position z for different die profiles

structure of ribbons during preheating of billets, melt-spun ribbons were heated at 300 °C in a temperature-controlled oven for 2 h and quenched immediately in water. Figure 8 depicts microstructure of heat-treated ribbon showing good amount of equilibrium γ precipitates ($Mg_{17}Al_{12}$). The precipitates formed preferentially along the grain boundary and were fine in size, typically of the order of 0.5 μm with rounded edges.

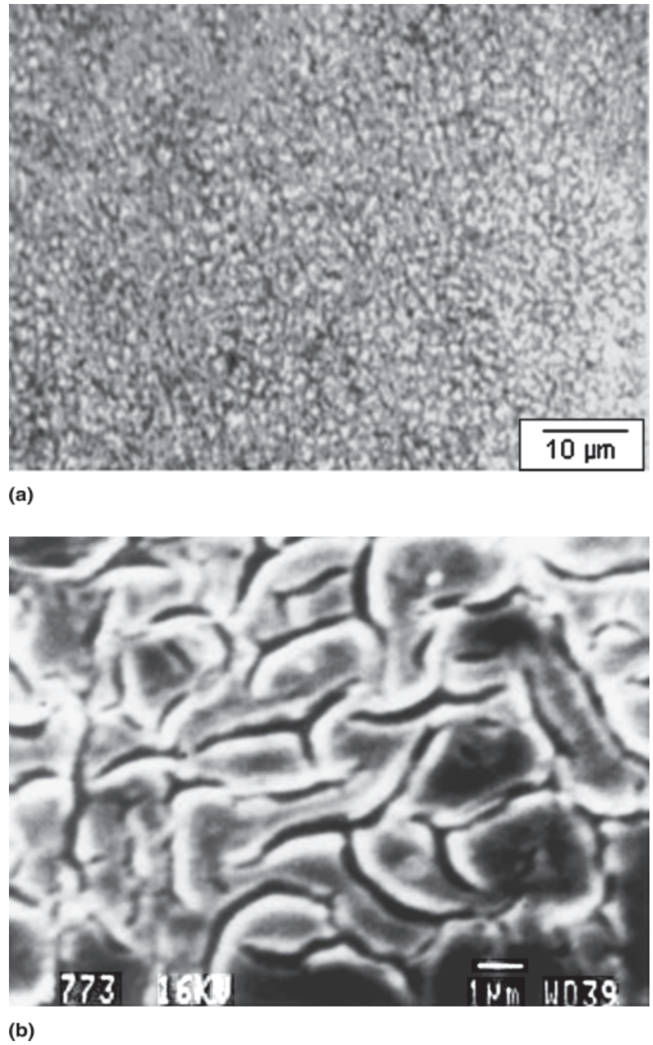


Fig. 7 (a) Optical and (b) scanning electron micrographs of as-melt-spun AZ91 alloy

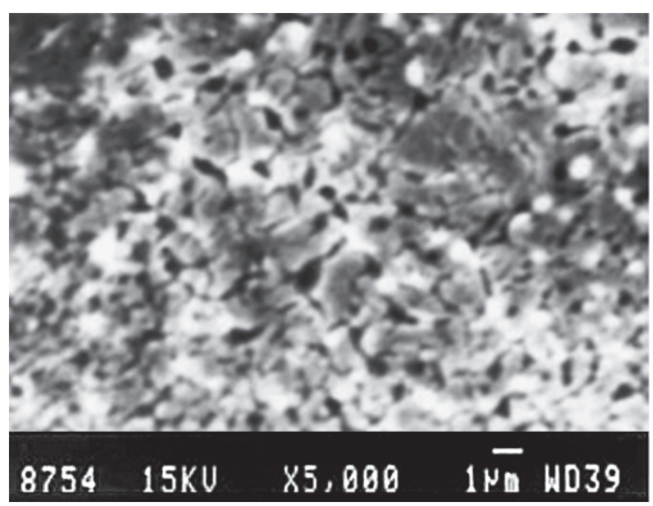
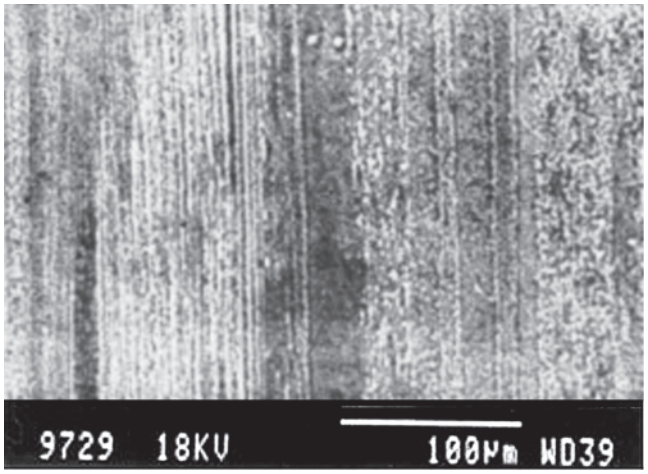
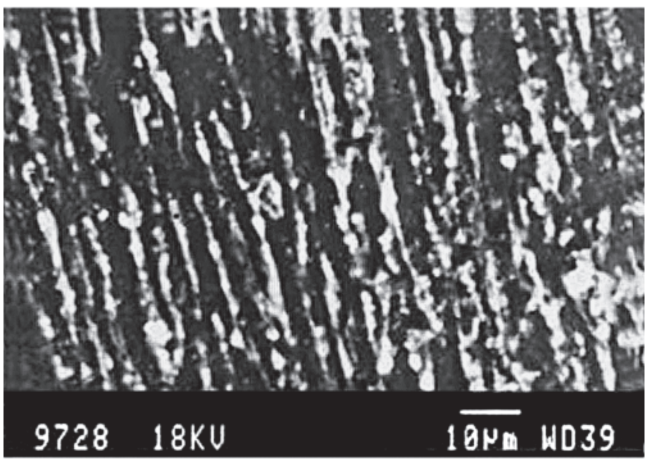


Fig. 8 Scanning electron micrograph of AZ91 melt-spun ribbon heat-treated at 300 °C for 2 h

The microstructure of rod extruded at 300 °C showed typical banded structure in the extrusion direction with the precipitates aligned along the bands, as shown in Fig. 9. However,



(a)



(b)

Fig. 9 Scanning electron micrographs of as-extruded rod of rapidly solidified AZ91 alloy extruded at 300 °C, at two magnifications (a and b)

when the extrusion temperature was increased to 350 °C, banded structure was not prevalent and improvement in microstructure was observed, as shown in Fig. 10. This can be attributed to the reason that dynamic recrystallization (DRX) occurred when extrusion temperature was increased to 350 °C and the microstructure was refined.

4.2 Mechanical Properties

Extruded rods of rapidly solidified AZ91 alloys were tested for hardness values and tensile properties. The test results given in Table 2 depict that rapidly solidified AZ91 alloy exhibits a good combination of room temperature strength and ductility. Effects of different extrusion conditions on the mechanical properties evaluated are also shown in Table 2. An increase in mechanical properties of rods extruded through CSR die over those extruded through conical die was observed that could be attributed to lower residual porosity and more homogeneous distribution of second phase particles in extrudates in case of CSR die. It is possible to expect that the die profile, which provides more uniform distribution of strain rate, also yields a more uniform distribution of microstructure. The increase in mechanical properties with increased extrusion ratio can be explained by the improvement of microstructure densification. As the extrusion ratio increases, the degree of workability also increases and the grains are further reduced in size in the transverse direction of extrusion. The effect of extrusion ratio on elongation can be seen to be more pronounced than that on tensile strength.

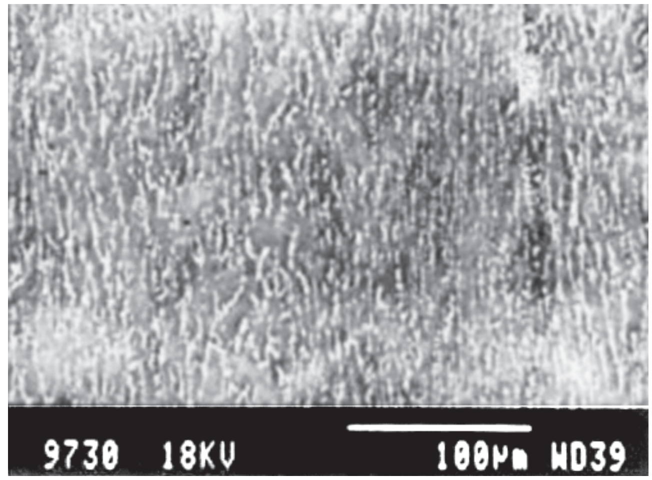


Fig. 10 Scanning electron micrograph of as-extruded rod of rapidly solidified AZ91 alloy extruded at 350 °C

Extrusion temperature had an obvious effect on mechanical properties. It is seen that hardness and both yield strength and tensile strength decreased when extrusion temperature was increased from 300-350 °C. As mentioned earlier, DRX occurred when extrusion temperature increased to 350 °C. These results indicate that DRX during extrusion has a negative effect on mechanical properties of the extruded rods except for providing a marginal improvement in elongation value. This is mainly due to softening of the matrix as a result of DRX. Furthermore, during DRX partial dissolution of precipitates in the matrix at increased extrusion temperature can also possibly occur diminishing the precipitation hardening effect in the alloy.

5. Conclusions

Mg-Al-Zn alloys (AZ91) were rapidly solidified by the melt spinning technique to form ribbons. Pulverized ribbons were cold-compacted to form 75 mm diameter billets, and preheated billets were hot extruded to form rods of diameters 25 and 15 mm. During extrusion, a specially designed die with constant strain rate (CSR) profile calculated using the slab method was used. By properly establishing the process parameters, extruded rods of rapidly solidified AZ91 alloys exhibiting a good combination of room temperature strength and ductility were produced.

A fine grain (1-2 µm) structure was observed in rapidly solidified AZ91 alloy ribbons due to a very high cooling rate. During heat treatment of ribbons for 2 h at 300 °C, a good amount of fine γ precipitates ($Mg_{17}Al_{12}$) of the order of 0.5 µm in size formed preferentially along the grain boundary.

During extrusion, the CSR die was found to be advantageous as compared with conical die and led to better mechanical properties of the extrudates by lowering residual porosity and increasing the uniformity of microstructure in the extrudates. However, to achieve uniformity of grain size and better

Table 2 Mechanical properties of extruded rods of rapidly solidified AZ91 alloys

Extrusion condition			Yield strength, MPa	Ultimate tensile strength, MPa	Elongation, %	Hardness, VPN
Temperature, °C	Extrusion ratio	Die profile				
300	9	Conical	315	370	3.8	102
300	9	CSR	330	390	4.0	105
300	25	CSR	340	410	5.0	110
350	9	CSR	295	360	4.5	95

mechanical properties, control of other process parameters such as extrusion speed, temperature, and extrusion ratio is equally important. By maintaining extrusion speed low in the range of 1-2 mm/s, and keeping it the same in all cases, effects of extrusion ratio and extrusion temperature on mechanical properties of the extruded rods were also investigated. An increase in mechanical properties with increased extrusion ratio was observed, which can be explained by the improvement of microstructure densification as a result of increased workability. Similarly, an increase in extrusion temperature from 300-350 °C resulted in refinement of microstructure caused by dynamic recrystallization. However, mechanical properties (yield strength and tensile strength) of the extruded rods decreased due to dynamic recrystallization.

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