J Mater Sci (2007) 42:3969–3976
DOI 10.1007/s10853-006-1301-3

Influence of texture on the mechanical properties of commercially pure magnesium prepared by powder metallurgy

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Received: 19 May 2005 / Accepted: 23 January 2006 / Published online: 31 January 2007

Abstract In the present work the influence of texture on the mechanical properties up to 500 °C of commercially pure magnesium prepared by PM was determined. Extrusion of magnesium powders was carried out between 250 and 450 °C. All extruded materials exhibited an intense fibre texture with the basal planes parallel to the extrusion direction whose intensity increased in line with the extrusion temperature. The microstructure consisted of highly elongated magnesium powder particles. All the materials presented a heterogeneous grain size resulting from the size distribution of the original magnesium powder particles. In addition, small MgO particles were found mainly decorating the original powder boundaries. The best mechanical properties corresponded to the materials extruded at 400 and 450 °C. This behaviour was associated particularly with the intense fibre texture of these materials.

Introduction

Most research on magnesium alloys has been focused on the development of cast products in view of their low production costs [1]. The microstructural heterogeneity and coarse grain size of the materials obtained with by melting and casting techniques results in materials of low strength. Subsequent improvements in mechanical properties may be achieved by decreasing the grain size through the appropriate thermomechanical processing of the cast billets.

Powder metallurgy (PM) processes offer an alternative route for the production of high strength magnesium alloys. A major drawback impeding the commercial production of PM magnesium alloys has traditionally been the high price of powder production as well as the high reactivity of magnesium powders. These are probably the main reasons of the very few studies published on pure PM magnesium [2–5]. However, advances in powder processing techniques, stimulated by the large amounts of reactive powders required for pyrotechnic applications, have considerably lowered the cost of producing fine magnesium powders. The advantages of magnesium alloys prepared by PM stem from the fine grain size, the increase in solid solubility of alloying elements and oxide dispersion strengthening. The two formers assure good mechanical properties at low temperatures, while the nanometric dispersion of MgO particles results in high creep strength, even with fine-grained magnesium [2, 3]. Whereas, the strengthening effect of MgO dispersion has been well established for PM materials, no specific studies about the influence of the extrusion temperature have been carried out.

The present work deals with the effect of changes in the texture, through changes in the extrusion temperature, on the mechanical properties up to 500 °C of commercially pure PM magnesium. The experimental data have been compared with those reported in the literature for the creep of pure magnesium. This research has been carried out in pure magnesium to simplify the study by avoiding effects associated with solid solution of alloying elements and/or second phase presence.

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Experimental

Commercially pure magnesium powders with a particle size of less than 45 μm and grain sizes ranging between 1 and 8 μm, were cold-pressed at 310 MPa pressure level, leading to a densification of around 95%. The compacts were hot-extruded into rods at 250, 325, 400 and 450 °C using an extrusion ratio of 18:1.

Microstructural characterisation was carried out by optical microscopy (OM), scanning (SEM) and transmission electron microscopy (TEM). The X-ray diffraction technique (XRD) was used for texture measurements. Texture analysis was carried out by the Schulz reflection method using a SIEMENS TM Kristalloflex D5000 diffractometer equipped with a close Eulerian cradle. The X-radiation used was β-filtered CuKα. The orientation distribution functions (ODFs) and the inverse pole figures were computed from the measurement of (0002), $(10\bar{1}0)$, $(10\bar{1}1)$, $(10\bar{1}2)$, $(10\bar{1}3)$ and $(11\bar{2}0)$ pole figures by the series expansion method. The reference system was selected with the Z-axis parallel to the extrusion direction.

Mechanical properties were evaluated using cylindrical specimens (radius 2.5 mm and gauge length 10 mm) machined from the extruded bars with the longer dimension parallel to the extrusion direction. Tensile tests were performed in a universal tensile machine under a constant cross-head speed at an initial strain rate of 10^{-4} s^{-1} in the 25–500 °C temperature range. Jump strain rate tests were also conducted at temperatures above room temperature in order to determine the stress exponent. The specimens were initially deformed at a strain rate of 10^{-4} s^{-1} until the steady state was reached, at which point the strain rate was reduced to 10^{-5} s^{-1} until the steady state was again attained. Successive jumps, increasing the strain rate, were performed following this procedure.

Results

Characterisation of extruded materials

After the extrusion stage, the original magnesium particles, whose perimeter was perfectly outlined by the presence of a fine dispersion of magnesium oxide particles of less than 1 μm, were highly elongated in the extrusion direction, as shown in Fig. 1. MgO particles were not always found at the original powder boundaries, and some of them were encountered inside the grain in regions close to the particle boundary. The grain size was highly heterogeneous and was determined by the original size of the individual powders. It was observed that the larger the diameter of the original magnesium powder particle, the larger the grain size inside that particle.

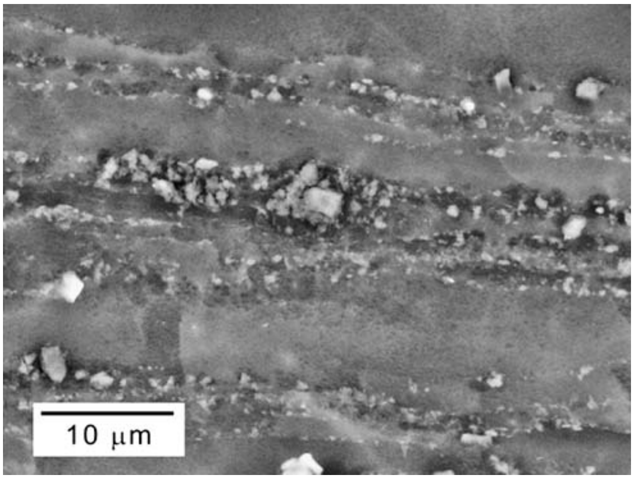

Fig. 1 Dispersion of MgO particles at the original boundaries of magnesium particles

The influence of the extrusion temperature was only reflected in some grain growth, especially in coarse magnesium particles, as can be seen in Fig. 2. Elongated magnesium particles usually consisted of a column of grains traversing their thickness, especially as the extrusion temperature increased. At 250 °C, however, isolated coarse magnesium particles with a fine equiaxed grain structure were found. TEM observations revealed grains with a high dislocation density forming a subgrain microstructure in some regions, as shown in Fig. 3. This substructure was absent at extrusion temperatures above 325 °C.

Texture analysis was carried out to determine the influence of extrusion temperature on the microstructure. Figure 4 shows the influence of the extrusion temperature on the inverse pole figures of magnesium prepared by powder metallurgy. All the extruded materials exhibited a fibre texture with the basal plane parallel to the extrusion direction; a typical extrusion texture in magnesium alloys, but differences in intensity were found. Figure 4 shows the intensity of the $\{10\bar{1}0\}$ and {0002} pole figures as a function of the polar angle for magnesium. The fibre intensity decreases in line with the extrusion temperature, although it was practically the same for the materials extruded at 400 and 450 °C. Additionally, the basal planes can be deviated up to 20 °C from the extrusion direction, and the number of such basal planes was found to be higher in the materials extruded at the lowest temperatures.

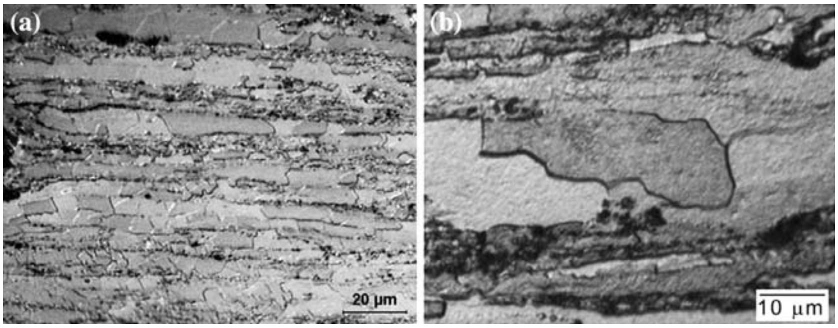

Fig. 2 Microstructure of consolidated material after extrusion at 250 °C (**a**) and 450 °C (**b**)

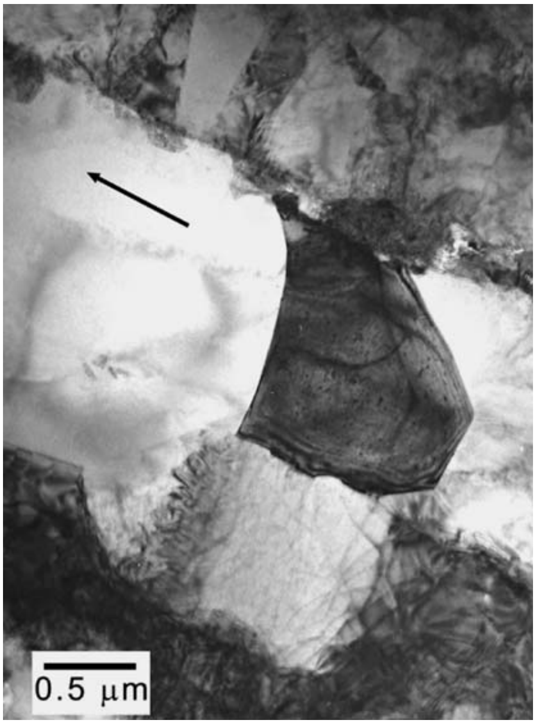

Fig. 3 TEM micrograph showing the substructure developed in magnesium after extrusion at 250 °C. The arrow indicates the extrusion direction

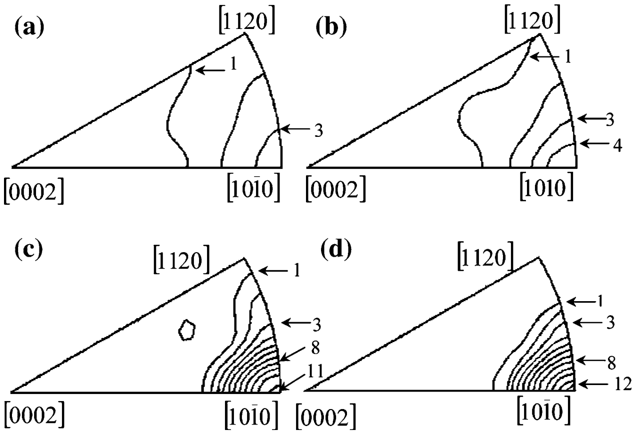

Fig. 4 Inverse pole figures of magnesium extruded at 250 °C (**a**), 325 °C (**b**), 400 °C (**c**) and 450 °C (**d**).Pole figure intensity of the {10$\bar{1}$0} (solid symbols) and {0002} (open symbols) planes as a function of the polar angle (Θ)

Mechanical properties

The tensile behaviour in the 25–500 °C temperature range of the materials extruded at 250 °C and 450 °C is shown in Fig. 5. The extrusion temperature clearly influenced the mechanical properties of magnesium. The materials extruded at the highest extrusion temperatures always exhibited the highest yield stress and ultimate tensile strength (UTS) values. Table I summarises the mechanical properties of the different materials as a function of the extrusion temperature. The yield stress and UTS values were very high at room temperature but tended to decrease in line with the extrusion temperature. Maximum UTS values of around 260 MPa were found at room temperature for the materials extruded at 400 and 450 °C while values of around 190 MPa were obtained for the material extruded at 250 °C. These values decreased substantially as the testing temperature increased, especially at temperatures above 100 °C. This behaviour is usually associated with the activation of non-basal slip by the increase in temperature [6]. It is interesting to note that the tensile curves at 400 and 500 °C were practically overlapped in the case of the materials extruded at 250 and 325 °C. This behaviour is probably due to microstructural changes occurring in the material as result of its exposure to a much higher temperature, 500 °C, than that used in the extrusion stage. As may be observed in Fig. 6, appreciable grain growth took place. The elongation-to-failure values ranged between 2 and 8% in the entire temperature regime, reaching maximum values for the material extruded at 250 °C. Although the variation in elongation-to-failure with the testing temperature did not follow a general rule, minimum values of around 2–3% were usually found at 300 and 400 °C. Maximum values were found at 100 °C.

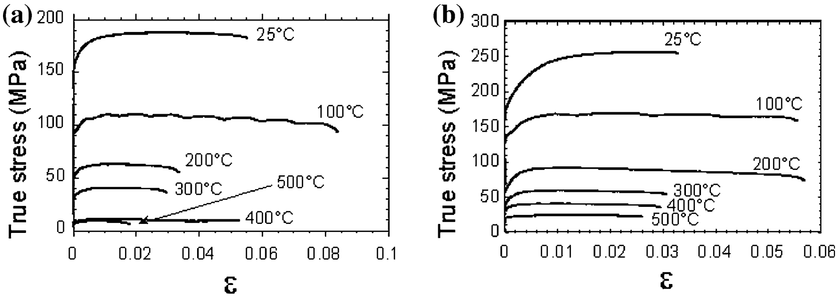

Fig. 5 True stress-true strain curves in the 25–500 °C temperature range for the material extruded at 250 °C (**a**) and 450 °C (**b**)

Table 1 Yield stress, maximum strength and elongation to failure from room temperature to 500 °C for the different extruded materials

Extrusion temperature (°C)	Test temperature (°C)	Yield stress	Maximum strength	Elongation to failure
250	25	170	188	5.5
	100	100	110	8.4
	200	58	63	3.3
	300	36	40	3
	400	9	11	5.2
	500	8	9.5	1.8
325	25	216	233	2.8
	100	132	149	2.7
	200	59	78	2.8
	300	45	49	2
	400	15.5	17	3
	500	11.5	13.5	1.7
400	25	232	259	4.3
	100	152	163	6.5
	200	89	92	3.2
	300	56.5	58	1.9
	400	35	38.5	2.5
	500	14	18	5
450	25	203	257	3.3
	100	148	170	5.5
	200	80	91	5.6
	300	53	59	3
	400	38	40	3
	500	22	24	2.6

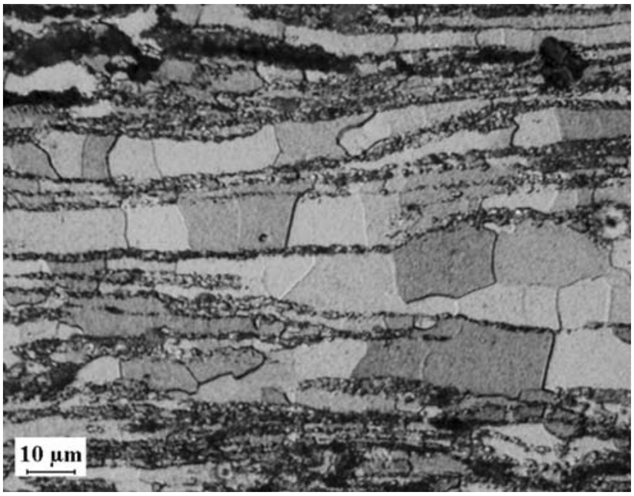

Fig. 6 Microstructure of the material extruded at 250 °C (head region) after testing at 500 °C

Figure 7 plots the strain rate versus stress in the steady state in a double logarithmic manner for all the extruded materials tested in the 100–500 °C temperature range according to the general-power law constitutive creep equation

$$\dot{\varepsilon} = k\sigma^{n_{\mathrm{app}}} \mathrm{e}^{\left(-Q_{\mathrm{app}}/RT\right)}$$

where k is the creep constant, n the apparent stress exponent, σ the stress in the steady state, Q_{app} the apparent activation energy, T the absolute temperature, and R the universal gas constant. As is shown in these plots, the stress exponent took very high values, 9–19, in the entire temperature interval. The stress exponent n reached a maximum value at 100–300 °C and then, slight decrease at higher temperatures. The process activation energy can be determined by plotting in a semi-logarithmic manner the stress versus the inverse of the absolute temperature at a constant strain rate value. Thus, the activation energy is given by $Q_{\mathrm{app}} = nRm$, where n is the stress exponent, R the universal gas constant, and m the slope of the σ–1/T curve. Since n was not constant through all the entire temperature interval, an average value was taken at the temperatures were n remained practically constant. Two kinds of Q_{app} values were found: at 100, 200 and 300 °C, Q_{app} ranged between 115 and 125 kJ/mol; at higher temperatures, Q_{app} was situated between 150 and 190 kJ/mol. Changes in Q_{app} are associated with a change in the mechanism controlling the deformation.

Discussion

SEM and TEM observations showed that extrusion temperature influences the nature of the microstructural evolution. At the lowest temperature of 250 °C, grain growth was very slow and only a limited coarsening of the magnesium grains took place. A

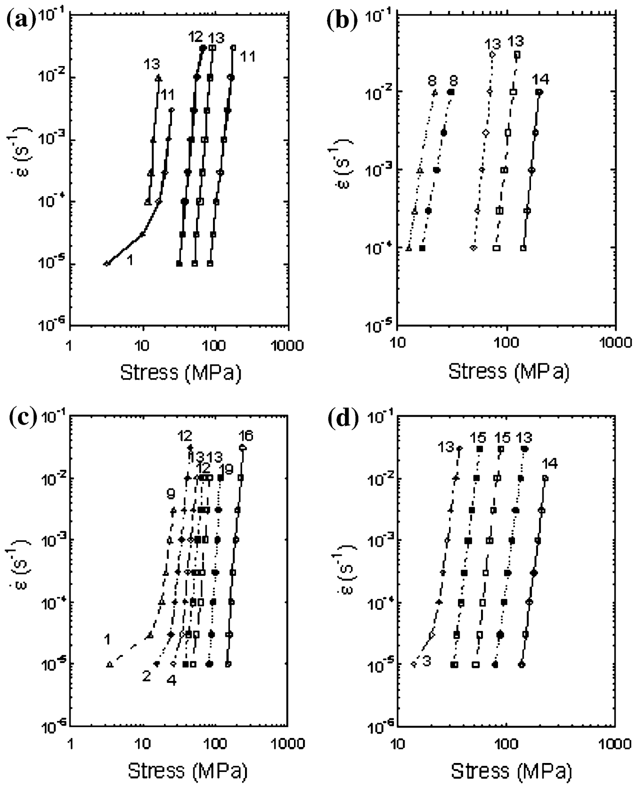

Fig. 7 Plot of strain rate-true stress curves in the steady state between 100 and 500 °C for the materials extruded at: (**a**) 250 °C, (**b**) 325 °C, (**c**) 400 °C (includes also data at 350 and 450 °C), (**d**) 450 °C

substructure containing a high density of dislocations, which were rearranged as sub-boundaries, was observed inside the grains. This indicates that dynamic recovery processes were operating during the extrusion process. The grain size increased progressively with increasing extrusion temperatures, while the dislocation density decreased in such a way that the microstructure of the materials extruded at temperatures above 400 °C consisted of practically dislocation-free grains. It is interesting to note that no clear evidence of recrystallisation was found in the materials extruded at 400 and 450 °C. This agrees with previous works on the processing of Al-SiC composites [7, 8], which reported that the recrystallisation process was slowed down by alumina particles, especially for unreinforced materials, resulting in a higher recrystallisation temperature. This behaviour was totally different to that observed in extruded or rolled coarse-grained cast materials [9, 10], where dynamic recrystallisation took place during the extrusion process, resulting in less textured materials.

During extrusion the basal planes were constrained to be arranged parallel to the extrusion direction. Since pure magnesium yields at very low stresses at temperatures above 350–400 °C, the plastic flow in the extrusion direction should be favoured by increasing the temperature. This can clearly be observed in the variation of the intensity as a function of the polar angle. At the highest extrusion temperatures most of the basal planes were practically parallel to the extrusion direction, whereas at the lowest temperatures, especially at 250 °C, only a small decrease in the intensity in a wider polar angle range was measured, indicating that many basal planes were not well aligned in the extrusion direction. Furthermore, the size distribution of magnesium powders, from nanometric up to 45 μm diameter particles, may also have affected the plastic flow during extrusion. It should be noted that the hardness of the individual particles depends on their size. Consequently, at low temperatures the plastic flow of softer particles could be disrupted by harder particles, contributing to reducing the intensity of the texture. Such an effect would be similar to the well-known decrease in the texture of metal matrix composites when the volume fraction of a hard reinforcement is increased [7, 11].

Mechanical properties at room temperature

The mechanical properties of the materials were strongly dependent on the extrusion temperature, i.e. on the microstructure. The materials extruded at 400 and 450 °C exhibited the highest yield stress and tensile strength values. This behaviour may be due to the contribution to the flow stress of different microstructural parameters such as grain size, initial dislocation density, MgO particle dispersion or texture.

Although it is well reported that magnesium verifies the Hall-Petch relationship at temperatures up to 250 °C [12, 13], in the present work the yield stress increases with increasing the grain size, i.e. with increasing the extrusion temperature. Consequently, grain size strengthening strengthening seems not to be main contribution to the high strength of materials extruded at 400 or 450 °C.

A high dislocation density should increase the yield stress of the material. In the present work, however, the decrease in the dislocation density observed as the extrusion temperature increased was not accompanied by a decrease in the yield stress. Therefore, other microstructural parameters must account to the additional hardening of the materials extruded at the highest temperatures.

The dispersion of fine MgO particles, resulting from the rupture of the oxide film covering the magnesium powders during the extrusion process, could reinforce the material through the Orowan mechanism. Since the loads applied during extrusion at low temperatures are much higher than those needed at higher temperatures, i.e., higher shearing forces acting on the oxide film, a finer dispersion of the MgO particles could be expected at the lowest extrusion temperature. But these fine oxide particles will only be effective if they are inside the grains, so that they represent an obstacle to dislocation motion. However, most of these oxide particles were located at the original powder particle boundaries in such a way that no significant strengthening associated with dislocation bypass could be produced. Extrusion at high temperatures favoured some grain growth beyond the original powder particle boundaries so that some oxide particles were located close to the particle boundaries. In this case, MgO particles should provide additional hardening to the material.

Texture contribution to the yield stress may be significant in an anisotropic material like magnesium. Different authors have verified that the compression yield stress of magnesium single crystals is determined by the crystal orientation [14, 15]. According to these authors, single crystals oriented for non-basal slip, i.e., basal planes lying parallel to the compression direction, exhibit a maximum strength, while the strength is minimal for the single crystals in which basal glide or twinning is easily activated. Analogous behaviour has been reported for polycrystalline magnesium and other magnesium alloys [16–19]. In the present study, the number of grains with basal planes parallel to the extrusion direction, unfavourably oriented for basal slip, increased as the extrusion temperature rose as shown in Fig. 4. Therefore, the sharper the texture intensity, the higher the stress necessary for basal slip activation.

From the preceding discussion it follows that the main contribution to the high yield strength of pure PM magnesium extruded at high temperatures comes from the texture of the alloy. Other mechanisms associated with grain size strengthening, hardening induced by fine oxide particles or dislocation density, only shift the strength in different directions depending on the extrusion temperature. This agrees with previous studies reporting the prevalence of texture intensity over a decrease in the grain size of the alloy in the strengthening of AZ31 alloy [20].

Mechanical properties at high temperatures

The stress exponent n calculated for the different materials ranged between 9 and 19 in the entire interval of testing temperatures. Only at strain rates lower than 10^{-4} s^{-1} at test temperatures above 400 °C did magnesium yield at very low stresses, with n taking values of about one. Under such conditions, a diffusional flow mechanism controlled the plastic deformation of pure PM magnesium. This agrees with previous studies in coarse-grained polycrystalline pure magnesium, although a diffusional flow mechanism was reported at temperatures above 500 °C and lower strain rates [21]. This different behaviour may be attributed to grain boundary diffusion creep, which could probably be favoured by the smaller grain size of PM magnesium. The influence of grain size is clearly manifested for the material extruded at 450 °C tested at 500 °C, at which certain grain growth took place, by a higher n value ($n \approx 3$), indicating that transition towards the diffusional flow mechanism should occur at higher temperatures.

At temperatures below 400 °C and/or strain rates larger than 10^{-4} s^{-1}, n values were much higher than those usually reported for polycrystalline magnesium prepared by conventional methods. Generally, PM magnesium presented higher strength and quite different creep behaviour to magnesium prepared by conventional methods. A survey in the literature of different data on magnesium creep [21, 22] shows great scatter in stress dependence, with n ranging from 3 to 18. Only limited data reporting n values comparable to those calculated in the present study can be found in the literature [2, 4], which correspond to PM materials. Crossland and Jones attributed the different behaviour of PM magnesium to the presence of a fine dispersion of MgO particles [22]. On the other hand, Vickers and Greenfield [3] calculated n value. In addition, lower n values have been reported for extruded-cast fine-grained Mg-30(% vol.)Y_2O_3 composite [23]. Between 200 and 400 °C n takes relatively small values, about 9, not as high as could be expected for a material with a high volume fraction of small yttria particles (0.3 μm). Only at temperatures above 425 °C n increases to 15. This behaviour is opposite to that found in the present work, in which maximum n values around 13–19 are achieved for the materials tested at between 100 and 300 °C. Therefore, it is apparent that oxide dispersion is not enough to raise n values in our materials in the temperature range 100–300 °C. Other factors such as grain size and/or texture may also have contributed to these high values.

Regarding the effect of grain size it must be considered that creep studies on conventionally processed materials have been carried out with coarse-grained materials with grain sizes between 80 and 625 μm [21, 22]. Only a few data on the creep of fine-grained magnesium can be found in the literature [3, 24]. The analysis of these studies suggests that a significant contribution of grain size may be dismissed since $n \approx 8$ has been reported for a fine-grained Mg-1%vol. MgO [3] and $n \approx 10$ for a Mg-3(%vol.)SiC nanocomposite [24].

Although crystal orientation influences the mechanism controlling deformation during the creep of single crystals [25–27], texture has not been taken into account during creep of polycrystalline magnesium. Most studies do not calculate this parameter, although a weak texture could be expected for the magnesium used in these studies because most of the microstructures were obtained after homogenisation treatments at temperatures above 500 °C. The stress exponent of these non-textured pure magnesium takes values around 4–6 in the temperature range 200–450 °C, which increased to eight at higher temperatures [21]. Similar variation of *n* with the temperature has been reported for extruded-cast Mg-30 (% vol.)Y_2O_3 composite [23]. This behaviour, however, is opposite to that observed in all materials studied in the current work, in which *n* decreases at temperatures above 200 °C or 300 °C depending on the extrusion temperature. It is interesting to note that high *n* values have been also reported for extruded PM magnesium whose grains are well elongated in the extrusion direction [2, 4]. This behaviour suggests the existence of a certain correlation between *n* and texture whereas, prismatic or pyramidal slip cannot be activated. The influence of texture on *n* value in magnesium extruded at 400 °C is manifested by the fact that the highest *n* values were attained by this material at test temperatures below 200 °C. That is because of highest number of basal planes unfavourably oriented for their slipping and the impossibility of non-basal slip. The last is occurs because the critical resolved shear stress (CRSS) for basal slip at temperatures below 200 °C is significantly smaller than that corresponding to pyramidal or prismatic slip [15, 17]. The smaller effect of the texture in the case of the material extruded at 450 °C could be related to its coarser grain size, which should provide a higher creep resistance.

The preceding analysis indicates that orientation, i.e. texture, may result in variations in the stress exponent values in such a way that the mechanism controlling the creep of strongly textured magnesium may differ from that controlling non-textured magnesium, as has been observed in the literature.

Conclusions

From the present study the following conclusions may be drawn regarding the influence of extrusion temperature on the mechanical properties of pure PM magnesium:

1. All extruded materials exhibited a fibre texture with basal planes parallel to the extrusion direction The absence of recrystallisation during the hot extrusion of cold-pressed powders has resulted in textured materials. The higher the extrusion temperature, the stronger the intensity of the fibre texture.
2. Higher strength values (around 260MPa) have been achieved with the materials extruded at the highest extrusion temperatures, 400 and 450 °C, at room temperature.
3. The texture of the alloy contributes significantly to increase the tensile strength of magnesium prepared by powder metallurgy.
4. High stress exponent values, ranging between 9 and 19, have been obtained in all extruded materials in the 100–500 ºC test temperature range, attaining maximum values between 100 and 300 ºC. MgO dispersion as well as texture intensity are responsible for high stress exponent values of PM-processed pure magnesium. The material extruded at 400 ºC exhibited the highest *n* value at 100 and 200 ºC. This may be related to the higher number of grains with their basal planes unfavourably oriented for slipping.

Acknowledgements This work was supported by CAM under CAM 07N 0075-2002 project. P.P is indebted to MCYT for its financial support within the Ramón y Cajal Programme.

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