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Microstructural characterization of an extruded Mg–Ni–Y–RE alloy processed by equal channel angular extrusion

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Abstract: Equal channel angular extrusion (ECAE) was performed in a conventionally extruded Mg–Ni–Y–RE material to develop fine-grained microstructures. The microstructure of the extruded alloy consisted of agglomerated phase particles zones (APZs) aligned along the extrusion direction and phase particles free zones (PFZs) containing recrystallized magnesium grains smaller than 5 μm in size. Some PFZs contained a banded unrecrystallized structure. The global texture was very weak and described by a (1 0 0) fibre which is common in extruded magnesium alloys. PFZs grain size was reduced to less than 1 μm after ECAE deformation at temperatures of 275, 310 and 375 °C, although some PFZs remained unrecrystallized. The mechanical properties of ECAE deformed materials were correlated with grain size and texture.

Keywords: Magnesium alloys, Severe plastic deformation, Equal channel angular extrusion, Texture.

1. Introduction

Severe plastic deformation (SPD) has been introduced in materials processing to produce ultra-fine grained microstructures (<100 nm) [1–3] that improve their properties at low and high temperatures [4–6]. At low temperature, generally, a small grain size enhances ductility and increases the yield strength, albeit a high strength is sometimes accompanied by low ductility and vice versa. At high temperature, a fine-grained microstructure usually exhibits superplastic behaviour under appropriate conditions of temperature and strain rate. A typical SPD technique is equal channel angular extrusion (ECAE), which consists of subjecting the material to intense plastic straining without changing its original cross-section. This intense straining is achieved by simple shear pressing the material through a die composed by two channels with the same cross-section, as illustrated in Fig. 1 [7–14].

Mg–Ni and Mg–Ni–Y appear to be promising systems to develop new alloys with some functional applications such as tunable mirrors or hydrogen storage medium. These new alloys could substitute conventional Mg alloys in numerous applications. Alloys belonging to Mg–Ni system, on the other hand, have high hydrogen retention and lower specific weight than other hydrogen storage alloys [15,16]. Recent studies have shown that multi-pass ECAE processing of eutectic lamellar Mg–Ni alloy at elevated temperatures

results in a homogenous microstructure with sub-micrometric grains of Mg and Mg₂Ni. Moreover, it appears that ECAE can increase the hydrogen retention capability of Mg–Ni alloys [17]. Similar results have been also obtained in ZK60 alloys [18].

Bars of Mg–Ni–Y–RE alloy were prepared by casting and subsequent hot extrusion at 400 °C. The microstructure consisted of small grains of about 4–5 μm in size embedding a high volume fraction of second-phase particles [19]. This microstructure exhibited high strength at temperatures below 250 °C and superplastic behaviour above 300 °C [19]. These results suggest that further grain refinement in the extruded material could improve strength at room temperature as well as superplastic properties at high temperatures.

ECAE experiments have been performed on a conventionally extruded Mg–Ni–Y–RE alloy containing a high volume fraction, around 30%, of coarse second-phase particles. The effect of ECAE temperature on microstructure and mechanical properties was investigated. The present work is part of a comprehensive study that deals with microstructural changes, texture and mechanical properties induced in this alloy by ECAE.

2. Experimental techniques

The Mg₉₆Ni₂Y₁CeMM₁ (at.%) alloy was prepared from binary Mg–Y, Mg–Ce mischmetal (CeMM) and Mg–Ni master alloys, with a composition close to the eutectic composition. The starting materials were pure Mg, Ni, Y and CeMM (53 wt.% Ce, 26 wt.% La, 16 wt.% Nd, 5 wt.% Pr). The melt was poured into bars, 42 mm in diameter, which were machined up to 40 mm and then subjected to conventional extrusion at 400 °C using an extrusion ratio of 18:1. Samples of 90 mm long were cut from the as-extruded bars to carry out ECAE experiments.

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Fig. 1. Processing steps of $Mg_{96}Ni_2Y_1CeMM_1$ alloy: (a) as-cast ingot, (b) conventional extrusion at $400\text{ }^{\circ}\text{C}$ and (c) ECAE.

The ECAE procedure is depicted in Fig. 1. The die is composed by two channels, with circular cross-sections, intersecting at an angle of 120° between the entrance and exit channel. The test bars were machined to fit tightly within the channel. They were lubricated with graphite paste, inserted into the die, held until they reached the processing temperature, and then pressed at 20 mm/min . Elevated temperature ECAE processing at 275 , 310 and $375\text{ }^{\circ}\text{C}$ was accomplished by heating the whole die to the desired temperature using built-in cartridge heaters.

Specimens were cut from longitudinal and transversal sections of as-extruded and ECAE deformed materials to examine the microstructure. The specimens were successively grounded, polished by alumina and swabbed for a few seconds using a fresh Nital solution. Microstructural analyses were made using field emission gun scanning electron microscope (FEGSEM) and transmission electron microscope (TEM). Phase identification was performed by X-ray diffraction (XRD) and energy dispersive X-ray microanalysis (EDX).

Texture measurements were carried out using a Siemens diffractometer equipped with a D5000 goniometer. Incomplete (100) , (002) pole figures (PFs) were measured over a range of azimuthal angles $0^{\circ} \leq \chi \leq 70^{\circ}$ in steps of $\Delta\chi = \Delta\Phi = 5^{\circ}$ using a Siemens software. A textureless standard sample of pure Mg was used for defocusing correction of the polar coordinates χ and Φ .

For mechanical characterization, cylindrical specimens with a radius of 3 mm and a gauge length of 10 mm were machined from the ECAE deformed bars with the tensile axis parallel to the extrusion direction (ED) as shown in Fig. 1c. Tensile tests were conducted at room temperature at an initial strain rate of 10^{-4} s^{-1} .

It is relevant to know the degree of deformation damage in extruded and ECAE processed materials. Several techniques for damage characterization are available such as X-ray microtomography [20–22] and high resolution transmission electron microscopy (HRTEM) [23,24]. These techniques are limited for detecting the early formation of small solute or vacancy clusters. However, coincidence Doppler broadening (CDB) measurements of positron annihilation radiation and positron lifetime spectroscopy (PLS) have been revealed as successful techniques to detect small clusters of solute atoms and impurity vacancy complexes in alloys [25,26].

PLS and CDB measurements were done at room temperature on ECAE samples. Positron annihilation experiments were performed on pairs of high grade polished samples enclosing a ^{22}Na positron source sealed inside thin kapton foils. CDB experiments were carried out using two HP-Ge detectors in timing coincidence with equal calibration ($\sim 81.1\text{ eV/channel}$) and a shaping time of $2\text{ }\mu\text{s}$. The analysed spectra were obtained by the addition of 20 measured spectra, each with $\sim 10^6$ and the centroid positioned at the centre of the measured 512×512 coincidence energy matrix E_1, E_2 of the two e^-e^+ annihilation γ -ray, respectively. CDB ratio profiles were obtained by taking into account the annihilation events within the strip $2m_0c^2 - 1.6\text{ keV} < E_1 + E_2 < 2m_0c^2 + 1.6\text{ keV}$; where m_0 denotes the rest electron mass and c the light speed. To enhance the difference between the CDB spectra of the different samples, the named ratio spectrum is obtained by dividing the counts at a given γ energy by the corresponding for pure Mg sample. Previously, the areas of the spectra are normalized to unity and smoothed. PALS experiments were carried out in a conventional fast-fast coincidence spectrometer with a resolution of 240 ps full-width at half-maximum. The lifetime spectra were collected up to acquire a count total of $\geq 10^6$.

3. Results and discussion

3.1. As-extruded material

The starting material for this study was obtained via casting and conventional extrusion at $400\text{ }^{\circ}\text{C}$ [19]. Fig. 2 shows the microstructure of the extruded alloy. Two different type of zones could be clearly differentiated; agglomerated particles zones (APZs) aligned along the ED (Fig. 2a) and particles free zones (PFZs) (Fig. 2b and c). The total volume fraction of APZs is estimated to be about 30%, which is higher than those frequently observed in most of magnesium alloys, often below 10%. PFZs are $50\text{--}150\text{ }\mu\text{m}$ length and $15\text{--}30\text{ }\mu\text{m}$ width. The grain size of the magnesium matrix is about $4\text{--}5\text{ }\mu\text{m}$, although finer grains are found in very thin PFZs. Straight boundaries and triple junction point angles of about 120° evidence dynamic recrystallization during extrusion, as shown in Fig. 2d.

Dynamic recrystallization is a typical high temperature deformation mechanism in Mg-based alloys [27]. However, no grain fragmentation is observed in some PFZs, designated in the following as unrecrystallized PFZs, which are characterized by the presence of traces of plastic flow produced during the extrusion, as shown in Fig. 2b. This behaviour could be attributed to the fact that the driving force for the onset of dynamic recrystallization in these zones was low during the extrusion.

The texture of the as-extruded material was measured on the transverse section (TS) and it is illustrated as measured (100) PF and calculated (100) and (002) PFs in Fig. 2e, f and g, respectively. Good agreement between measured and calculated (100) PF is found. Although the intensity is relatively low, a concentration of poles at the centre of the (100) PF is observed, indicating that texture can be interpreted as a (100) fibre texture. This texture is typical of extruded magnesium alloys, where $\{10\bar{1}0\}$ directions tend to be aligned along the ED, i.e. the (002) basal planes are parallel to the ED.

3.2. ECAE deformed materials

3.2.1. Grain structure

Fig. 3 shows the microstructures of the material ECAE deformed at 275 , 310 and $375\text{ }^{\circ}\text{C}$. As expected, the as-extruded material under-goes microstructural changes, especially grain refinement, when it is subjected to ECAE process. At $275\text{ }^{\circ}\text{C}$ the microstructure in Fig. 3a exhibits interesting features compared with the conventionally extruded material shown in Fig. 2c. New small-recrystallized grains inside the PFZs, with grain sizes smaller than $1\text{ }\mu\text{m}$, are observed. This phenomenon, however, proceeds only partially in some original unrecrystallized PFZs. This is revealed by the fact that plastic flow traces in PFZs, initially parallel to the ED, are altered by lattice rotation during ECAE. This fragmentation process, typical of dynamic recrystallization phenomenon, starts preferably at APZs neighbourhoods, as shown in Fig. 3a and b, and then it should progress into the unrecrystallized structure with increasing the ECAE temperature, as revealed by the continuous decrease in the volume fraction of unrecrystallized zones. Recrystallization in these zones may probably be deformation and orientation dependent. Lattice rotation resulting from recrystallization process would depend on the angular relationship between the Burgers vector of mobile dislocations in these zones and the shear direction [28]. In other words, lattice rotation should be small for low angles and large for angles close to 90° . This will be tried when discussing the ECAE deformation at $375\text{ }^{\circ}\text{C}$.

Apparently, such unrecrystallized zones make harder shearing during ECAE at $275\text{ }^{\circ}\text{C}$. Probably, the strain energy stored in unrecrystallized zones regions is minimized by dissolving some Y and Ni atoms into the Mg basal planes. The concentration of solute atoms

Fig. 2. Microstructure of as-extruded $Mg_{96}Ni_2Y_1CeMM_1$ on a plane parallel to the extrusion direction showing: (a) and (b) the phase particles distribution, and (c) and (d) the grain structure in the PFZs, (e) measured (100) PF, and calculated PFs for (f) (100) and (g) (002). The intensity levels are valid for the all PFs given in this study. TA and ED in (g) indicate the tension axis and extrusion direction, respectively. (For interpretation of the references to color in this artwork, the reader is referred to the web version of the article.)

in the basal planes would be higher than in the surrounding areas; so microscopic structural instabilities are expected to occur around the solute atom clusters. Consequently, the critical shear stress for dislocation breakaway from the solute atom atmosphere may be higher than that expected in absence of solute atoms. Breakaway stress could be also reduced by the activation of additional slip systems when the ECAE temperature is increased. This process should be assisted by the progressive segregation of such solute atoms.

The microstructure of the alloy after ECAE at $310^\circ C$ is very similar, but the total volume fraction of recrystallized PFZs increases. In some localized regions, shearing in unrecrystallized PFZs occurs, as shown in Fig. 3c. Small nuclei at the intersection of ECAE shear bands with the banded structure are emerged, as indicated by

arrows in Fig. 3c. Formation of these nuclei can be interpreted in terms of local lattice rotations induced by the activation of new slip systems. These new nuclei, however, are not clearly outlined. Furthermore, microstructural observations also prove that ECAE deformation does not introduce pores or cavities in the deformed material, indicative of the excellent cohesion at the particle/matrix interfaces. This is observed in the deformed areas adjacent to the particle in Fig. 3d. Plastic flow at the neighbourhoods of such particle suggests that such phase, i.e. a phase with a long period stacking ordered (LPSO phase) structure, exhibits a good plastic behaviour under ECAE deformation at high temperature. In general, highly deformed (or rotated) areas around the particles are not observed.

Fig. 3. LS microstructure after ECAE deformation at different temperatures: (a) recrystallized PFZ containing small grains and an unrecrystallized PFZ containing banded structure and (b) details close to LPSO phase particle at 275 °C. (c) Grain structure inside a PFZ and (d) microstructure in the adjacent area to LPSO particle at 310 °C. (e) Unrecrystallized PFZ showing the banded structure parallel to the ED and small new grains at the APZ and (f) details of grain nucleation at the intersection between the banded structure and sheared zones at 375 °C. The arrows indicate new grains close to APZs.

The recrystallization mechanisms described in the samples pressed at lower temperatures may be clearly distinguished in the few unrecrystallized PFZs remaining in the material pressed at 375 °C, as observed in Fig. 3e and f. Nucleation of new grains proceeds by two main processes. The first one takes place at APZ boundaries, as indicated by arrows in Fig. 3e and f. Then, recrystallization progresses toward the core of PFZs. The main driving force for nucleation is the stress concentration generated at particle neighbourhoods. The second nucleation process is manifested at the intersections between shear bands and original plastic flow traces, forming an angle of 57°, as seen in zones labelled 1, 2 and

3 of Fig. 3f. This suggests that shearing takes place on pyramidal (101) planes during the ECAE process. Thus, it follows that nuclei form by lattice rotation around an axis normal to ED, i.e. $\langle 1 \ 2 \ 1 \ 0 \rangle$ axis.

3.2.2. Texture

Texture of ECAE deformed samples is presented as (100) and (002) PFs in Fig. 4. It is important to note that the pole density at centre of (100) PF decreases at all temperatures compared with conventionally extruded material (Fig. 2e). This agrees with occurrence of dynamic recrystallization, which usually induces a

Fig. 4. Measured (100) and calculated (100) and (002) PFs: (a), (b) and (c) for 275 °C; (d), (e) and (f) for 310 °C; (g), (h) and (i) for 375 °C.

decrease in texture intensity [14,29]. A slight difference, however, emerges when the calculated (0 0 2) PF for ECAE deformed samples is compared with (0 0 2) PF for the as-extruded material. The pole density between the normal to the ED and an axis at $\chi = 60^\circ$ from the ED increases with increasing ECAE temperature. This finding seems to be compatible with the reorientation of (0 0 2) basal planes that accompanies nucleation induced by lattice rotation, according to the mechanism described above.

The present results suggest a correlation between texture and the volume fraction of unrecrystallized PFZs remaining in the deformed alloys. Dynamic recrystallization generally randomizes texture, especially in materials containing a considerable amount of precipitates and second phase particles. Thus, recrystallized PFZs shown in Fig. 2c should not contribute to texture. Therefore, texture depicted in Fig. 2e for the as-extruded material can be associated exclusively with the contribution of unrecrystallized PFZs, where (100) prismatic planes are normal to the ED.

3.3. Mechanical tests

Fig. 5 shows the true stress–true strain curves at room temperature for as-extruded and ECAE deformed materials. In all cases, a constant flow stress is attained and the elongation to failure is

about 5%. This value is still smaller than that commonly observed in conventional Mg-based alloys. This can be related to the deformed microstructure that after one pass does not reach a stable structural state. Tensile tests were not performed on the sample ECAE deformed at 275 °C because the material was delaminated along the ED. This damage behaviour is probably due to a combined effect of temperature and directionality of the plastic flow in the APZs along the ED. It is believed that a high accumulation of plastic strain may be generated in the APZs during ECAE, which can produce plastic incompatibilities at the grain/particle interfaces, resulting in material delamination.

The yield stress σ_y and the tensile strength σ_{sth} for the as-extruded material are 295 and 333 MPa, respectively. After one pass at 310 °C, σ_y and σ_{sth} increase to about 363 and 391 MPa, respectively. This increase in the mechanical properties is attributed to the grain refinement from a grain size of 4–5 μm to less than 1 μm produced by ECAE. In the case of the material pressed at 375 °C, σ_y and σ_{sth} are 340 and 368 MPa, respectively. These values are slightly lower than those obtained at 310 °C, and are mainly related to the grain size and dislocation density, even though texture can also contribute slightly in such strength decrease. Although SEM observations do not show significant microstructural differences between ECAE materials deformed at 310 and 375 °C, TEM

Fig. 5. True stress–true strain curves at room temperature for the as-extruded material and the ECAE deformed materials.

analyses reveal a significant change in grain size. Fig. 6 shows TEM micrographs of ECAE samples deformed at 310 and 370 °C. It is apparent that the grain size after ECAE at 375 °C is larger than that at 310 °C, 1.4 μm against 0.8 μm . This is attributed to dynamic recovery, i.e. grain boundary migration and dislocation annealing, which are expected to be faster at 375 than at 310 °C.

The Hall–Petch relationship between the yield stress and the grain size, d , is

$$\sigma_y = \sigma_0 + \frac{K}{\sqrt{d}} \quad (1)$$

where σ_0 is the yield stress of a single crystal and K is a constant whose value increases with the Taylor factor. Since the number of slip systems for hexagonal structures are very limited, i.e. the Taylor factor is large, magnesium strengthening is expected to be mainly influenced by the grain size. A K value of 110 $\text{MPa } \mu\text{m}^{1/2}$ was found. This value is slightly lower than those reported for conventionally processed pure Mg and Mg alloys but similar to that for ECAE deformed AZ91 alloy [30]. Since the phase particles distribution in the matrix is quite similar in all ECAE deformed samples, strengthening mechanisms at room temperature could be ascribed to grain boundary hardening. In the same way, it is well known that the distribution of basal planes in ECAE deformed materials plays an important role in deformation behaviour at room temperature, since strain in hexagonal systems is easily accommodated by basal slip. It should be noted that an increase in (0 0 2) pole density between $60^\circ < \chi < 75^\circ$ is observed for the ECAE deformed materials respect to the as-extruded material (Fig. 4). This is produced by the reorientation of the banded structure as discussed in Section 3.2.1, see Fig. 3f. Thus, the emerging new orientations could exhibit easy slip, contributing to the reduction of the tensile strength. However, the elongation to fracture in the tensile tested samples remains unchanged.

Regarding above, the effect of texture on the mechanical properties of Mg and its alloys has not been comprehensively studied, and contradictory results have been reported [31–33]. For example, it has been shown that 2–3 times improvement in the elongation of ECAE deformed AZ31 alloy is due to the randomisation of the strong texture induced by the initial extrusion [32]. On the contrary, other studies in AZ31 and AZ61 magnesium alloys have reported a ductility improvement and a strong texture increase induced by ECAE deformation [34,35]. Further extensive ECAE experiments using different temperature range, strain paths and number of passes are required to elucidate the relationship between the mechanical properties and the microstructural characteristics of the $\text{Mg}_{96}\text{Ni}_2\text{Y}_1\text{CeMM}_1$ alloy.

Fig. 6. TEM micrographs of ECAE deformed samples at: (a) 310 °C and (b) 370 °C. The arrows indicate the traces of LPSO phase.

Fig. 7. CDB ratio spectra for (a) as-extruded material and materials ECAE deformed at 275 and 375 °C and (b) pure yttrium and nickel.

3.4. Positron annihilation experiments

It was reported that SPD as ECAE process introduces drastic changes in the microstructure, inducing the formation of pores or cavities (damages), which may degrade subsequently the properties of the material. Appropriate post-treatments are therefore required to overcome such structural damage, such as annealing and/or deformation. These treatments become of great importance while the damages size is very small to such a point that it cannot be detected by microscopic observations. At first, FEGSEM and TEM microscope have shown that no damages were observed neither in as-extruded state nor after ECAE deformation. However, positron annihilation experiments, which provide deep information on defects, even vacancy clustering, have shown a difference in the magnitude of defect formation between as-extruded material (starting material) and ECAE deformed materials.

En effect, the as-extruded material exhibits a two-component positron lifetime spectrum, with lifetime values of $\tau_1 = 134$ ps and $\tau_2 = 252$ ps, with relative intensities of $I_1 = (13 \pm 1)\%$ and $I_2 = (87 \pm 2)\%$. The τ_2 agrees to the experimental positron lifetime for a single vacancy in Mg [36]. Moreover, the bulk positron lifetime τ_b calculated according to the standard two-state trapping model by the equation:

$$\tau_b = \frac{\tau_1 \tau_2}{I_2 \tau_1 + I_1 \tau_2} \quad (2)$$

is 226 ps, in agreement to the experimental value of 225 ps reported for pure Mg [36]. It is worth noting that single vacancies in pure Mg anneal out at room temperature [36].

After ECAE deformation the lifetime spectrum becomes single-component, with a lifetime value that decreases from 239 ps for the sample ECAE deformed at 275 °C to 224 ps for the sample

deformed at 375 °C. Then, the vacancy-type defects present in the as-extruded material are annealed out by the ECAE deformation at $T \geq 275$ °C. In particular, it appears that ECAE at 375 °C annealed out completely all the vacancy-like defects present in the as-extruded material.

The CDB ratio spectra for the as-extruded material and ECAE deformed materials are represented in Fig. 7a along with the spectra for samples of Y and Ni in Fig. 7b. The spectrum of the as-extruded material exhibits a wide peak centred at $11.3 \times 10^{-3} m_0 c$, which position and shape match with the characteristic peak found in the CDB ratio spectrum for the Y sample shown in Fig. 7b. This proves that a high fraction of positrons in the as-extruded material annihilate with electrons of Y atoms. ECAE deformation also produces a qualitative change in the features of CDB spectrum. The characteristic peak due to annihilation with electrons of Y atoms disappears at same time that the lifetime spectrum becomes single component. The features of the high-momentum region in the CDB ratio spectra have not been considered because the corresponding data appear to be subjected to high uncertainties.

The present positron annihilation results can be explained taking into account that the positron affinity of Mg is -6.18 eV against -5.31 and -4.46 eV for Y and Ni, respectively. This means that positrons mostly probe the Mg matrix and the Mg-rich phase particles. Therefore, the above results evidence that vacancy-like defects present in the as-extruded material are Mg vacancies attributed to Y atoms, i.e. Mg vacancy-Y pairs or complex vacancy-Y clusters, in the Mg matrix or in the $Mg_{12}RE$ phase. It should be mentioned that binding energy of the vacancy-Y pair in the Mg matrix is relatively high according to calculations [37].

4. Conclusions

1. After conventional extrusion at 400 °C the microstructure of the $Mg_{96}Ni_2Y_1CeMM_1$ alloy consisted of agglomerated phase particles zones, APZs, aligned with extrusion direction, and phase particles free zones, PFZs. This material had a structure of recrystallized grains, with an average size of 4–5 μm , and a weak (1 0 0) fibre texture. Some PFZs, however, exhibited an unrecrystallized banded structure.
2. After one ECAE pass in the temperature range of 270–375 °C the grain size was reduced to less than $\sim 1 \mu m$ whereas the initial texture vanished. In addition, the banded structure fragmented into small nuclei, and the amount of unrecrystallized PFZs decreased with increasing ECAE temperature.
3. ECAE deformed materials yielded at tensile strengths higher than as-extruded material but without any significant change in the elongation to failure. This is attributed to the fact that the microstructure did not reach a stable state. Subsequent passes are needed to produce further grain refinement and homogenous strain that stabilizes the final microstructure.
4. The positron annihilation experiments have revealed that the $Mg_{96}Ni_2Y_1CeMM_1$ alloy extruded at 400 °C retained a high concentration of vacancy-like defects associated with Y atoms in the Mg matrix, which very likely are Mg vacancy-Y pairs or Mg vacancy-Y clusters. One ECAE pass in the temperature range 275–375 °C annealed out these defects. No positron evidence for the formation of defects after ECAE process was found.

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