Abstract: Mg-3Zn-1Al (AZ31) alloy is a popular wrought alloy, and its mechanical properties could be further enhanced by the addition of calcium (Ca). The formation of stable secondary phase \((\text{Mg,Al})_2\text{Ca}\) enhances the creep resistance at the expense of formability and, therefore, necessitates the establishment of safe working window(s) for producing wrought products. In this study, AZ31-3Ca alloy has been prepared by the disintegrated melt deposition (DMD) processing route, and its hot deformation mechanisms have been evaluated, and compared with similarly processed AZ31, AZ31-1Ca and AZ31-2Ca magnesium alloys. DMD processing has refined the grain size to 2–3 \(\mu\)m. A processing map has been developed for the temperature range \(300–450^\circ\text{C}\) and strain rate range \(0.0003–10\ \text{s}^{-1}\). Three working domains are established in which dynamic recrystallization (DRX) readily occurs, although the underlying mechanisms of DRX differ from each other. The alloy exhibits flow instability at lower temperatures and higher strain rates, which manifests as adiabatic shear bands. A comparison of the processing maps of these alloys revealed that the hot deformation mechanisms have not changed significantly by the increase of Ca addition.

Keywords: thermomechanical processing; magnesium alloy; calcium addition; disintegrated melt deposition; processing map; formability

1. Introduction

In recent years, magnesium alloys have become popular for use as lightweight structural components in automobile, aerospace and biomedical applications [1,2]. AZ31 is the most popular wrought magnesium alloy that has been investigated extensively [1,3]. However, the alloy faces limitations of creep strength and corrosion resistance. Its creep strength may be enhanced by the addition of alloying additions like rare-earth or alkaline-earth elements. The addition of Ca to magnesium alloys has been studied extensively in view of its cost advantage over the rare-earth elements [4–6] and its effect on strength and hot workability has been reviewed recently [6]. The room temperature formability of AZ31 is improved by the addition of 0.5% Ca [7,8] since it reduces the basal texture promoting prismatic slip. Sakai et al. [9] reported that addition of 1%Ca enhances the room temperature strength of AZ31 significantly although the ductility improves only at temperatures higher than about 150 \(^\circ\text{C}\). Ca addition to an extent of 0.7% enhances the corrosion resistance of AZ31 alloy [10] although higher additions are not particularly beneficial. The ultimate compressive strength of AZ31 alloy at its service temperature (about 150 \(^\circ\text{C}\)) increases from 134 MPa to 235 MPa with 2% Ca
addition [6]. Further additions of Ca to AZ31 hold a potential in improving its higher temperature strength and in this study, it is proposed to explore this possibility by enhancing the Ca content to 3%.

Complex intermetallic phases of \((\text{Mg},\text{Al})_2\text{Ca}\) and \(\text{Ca}_2\text{Mg}_6\text{Zn}_3\) are formed when alloying elements of Al, Zn, and Ca are added to magnesium, and they become dispersed in the microstructure with different morphologies depending on the melting and casting processes adopted [4,11,12]. However, a technique called disintegrated melt deposition (DMD) has been developed to inherit the combined benefits of stir-casting and spray-processing techniques [13,14]. Similar to stir casting, this method involves the vortex mixing of alloying elements/reinforcement in the molten matrix and the resulting slurry is deposited onto a metallic substrate after disintegration by jets of inert gases. Unlike conventional spray processes, this method employs lower impinging velocity of gas jets with almost 100% recovery of poured material as the formation of overspray powders is avoided [13]. Therefore, the DMD technique offers the features of: (i) simplicity and cost effectiveness of conventional stir cast foundry process and (ii) fine grain and homogeneous microstructure associated with spray deposition process.

DMD technique of processing involves atomization of liquid metal alloy which helps in the production and deposition of fine metal droplets to form fine grains and a uniform distribution of intermetallic phases in the microstructure. The collected metal droplets form a billet of low density and can be further consolidated by extruding at desired temperature to produce rods/bars of desired size [15,16]. The so-called DMD rods of AZ31, AZ31–1Ca and AZ31-2Ca alloys prepared by DMD route have been studied earlier as regards their microstructure and hot working mechanisms [17–19].

In these investigations, it is found that addition of Ca to AZ31 has enhanced its compressive strength without restricting the hot workability. The aim of the investigation is to examine the effect of further addition of Ca to an extent of 3% on the hot workability of AZ31. In order to introduce a higher Ca content in the alloy and distribute it homogeneously, the advantage of the DMD processing route has been exploited. As in the case of earlier investigations of hot workability of DMD AZ31 alloys, the approach of processing maps has been adopted in this study also.

2. Methodology

The principles behind the development of processing maps have been extensively documented in the literature [20–22] following a dynamic material modeling approach. In simple terms, the model differentiates the way the applied energy to deform a material is utilized or dissipated. The relative proportion of energy dissipated for the generation of heat and microstructural changes determines the efficiency of power dissipation. In the latter case, the energy dissipation may result in increase of dislocations, dynamic recovery and recrystallization, etc. A simple parameter that can provide an estimate of power partition between heat and microstructural changes in the strain rate sensitivity \((m)\) of flow stress under a given set of deforming conditions of temperature and strain rate. The efficiency of power dissipation \((\eta)\), responsible for bringing the microstructural changes during deformation can be calculated using:

\[ \eta = 2m/(m + 1) \]  

(1)

It is convenient to present the dissipation efficiency in the form of a map consisting of iso-efficiency contours over the chosen ranges of temperature and strain rate to distinguish their effects.

Another important consideration is whether the material flow is stable during deformation under the chosen forming conditions. In simple terms, the material flow rate should not exceed the applied velocity. For example, fracture or wedge cracking may propagate at a faster rate than the moving tool that imparts energy, which is not desirable in forming processes. A stable flow in general enables material integrity after hot deformation and results in better product. The transition from stable to unstable flow can be estimated using the extremum principles of irreversible thermodynamics as applied to continuum mechanics of large plastic flow [23]. The criterion for the onset of flow instability is:

\[ \xi(\dot{\varepsilon}) = \frac{\partial \ln [m/(m + 1)]}{\partial \ln \dot{\varepsilon}} + m \leq 0 \]  

(2)
where \( \xi \) is the instability parameter for a given strain rate \( (\dot{\varepsilon}) \). The flow becomes localized and causes flow instability for deformation conditions where \( \xi (\dot{\varepsilon}) \leq 0 \).

A processing map integrates both the important considerations mentioned above, namely, dissipation efficiency and flow stability, by superimposing their contour maps to provide a comprehensive understanding of material response under applied process conditions of temperature and strain rate. High dissipation efficiency domains that form hills in contour or 3-dimensional maps with stable flow can be identified easily where the workability of a material is optimal due to the occurrence of favorable mechanisms, such as dynamic recrystallization (DRX). Similarly, conditions of flow instability, even though dissipation efficiency may be high, can be earmarked as failure regimes that should be avoided for hot deformation of the material. It is possible that several desirable domains and undesirable regimes may be found over the chosen ranges of temperature and strain rate due to specific deformation mechanisms that are operative.

The commonly used kinetic rate equation relating the flow stress \( (\sigma) \) to strain rate \( (\dot{\varepsilon}) \) and temperature \( (T) \) of hot deformation is given by Jonas et al., [24]:

\[
\dot{\varepsilon} = A\sigma^n \exp\left[-\frac{Q}{RT}\right]
\]

(3)

where \( A \) is a constant, \( n \) is a stress exponent, \( Q \) is the activation energy, and \( R \) is the gas constant. This kinetic rate equation is obeyed within a domain where single deformation mechanism dominates and, therefore, can be applied to evaluate the associated apparent activation energy. The activation parameters \( n \) and \( Q \) facilitate the identification of rate-controlling deformation mechanisms in corresponding domains.

3. Materials and Methods

The AZ31–3 wt% calcium (AZ31-3Ca) alloy was prepared by the disintegrated melt deposition (DMD) processing technique developed by Gupta and coworkers [15,16]. Briefly, the procedure consisted of the following two steps: (1) Primary processing: Rectangular pieces of AZ31 magnesium alloy were cut from ingots. Holes of 12 mm diameter and 30 mm depth were drilled in these rectangular pieces and were filled with the required amount of calcium. They were melted in a crucible that has bottom pouring arrangement and heated to reach a temperature of 750 °C. The molten alloy was stirred for 5 min at 450 rev min\(^{-1}\) using a twin blade (pitch 45°) mild steel impeller to facilitate chemical homogenization. The impeller was coated with Zirtex 25 (86% ZrO\(_2\), 8.8% Y\(_2\)O\(_3\), 3.6% SiO\(_2\), 1.2% K\(_2\)O and Na\(_2\)O, and 0.3% trace inorganic) to avoid iron contamination. The melt was then released through a 10 mm diameter orifice at the base of the crucible and the droplets of the melt were disintegrated by two jets of argon gas orientated normal to the melt stream. The argon gas flow rate was maintained at 25 L min\(^{-1}\). The disintegrated melt was deposited onto stainless steel substrate to obtain a preform billet of 40 mm diameter. (2) Secondary processing: The preformed billet was machined to 35 mm diameter, heated to 400 °C and kept for 1 h before directly extruding through a preheated die at 350 °C with colloidal graphite used as a lubricant. Rods of 9.8 mm diameter were obtained using an extrusion ratio of about 13 adopting this direct extrusion procedure.

The experimental procedure for hot compression testing was described in detail earlier [25]. Briefly, cylindrical specimens of 9.8 mm in diameter and 15 mm in height were machined from the extruded rods for uniaxial compression along the direction of extrusion. A hole of 1 mm diameter was machined at mid-height to reach the center of the specimen. A thermocouple was inserted in the hole to monitor the specimen temperature and also to measure the instantaneous temperature rise during deformation. The adiabatic temperature rise was measurable for tests conducted at strain rates of 0.1 s\(^{-1}\) and higher. Compression tests were carried out at strain rates in the range of 0.0003–10 s\(^{-1}\) and temperatures in the range of 300–450 °C. The selected constant true strain rate during a compression test was obtained through an exponential decay in the actuator speed of the machine. Graphite powder mixed with grease was applied as the lubricant at the specimen-die interfaces in all the
experiments. The specimens were deformed up to a true strain of about 1 and quickly cooled in water. The load-stroke data were converted into true stress-true strain values using standard equations. The true stress values were plotted as a function of measured temperature at different strain levels and were fitted by smooth curves. The corrected flow stress was obtained from these fitted curves at any selected temperature for calculating the strain rate sensitivity of flow stress. The processing map was developed at different strains from the variations of strain rate sensitivity of flow stress with strain rate at different temperatures using Equations (1) and (2) and the procedure described earlier [20].

The deformed specimens were sectioned in the center, parallel to the compression axis, and were mounted, polished and etched with acetic picral solution for metallographic examination.

4. Results and Discussion

The starting microstructure of DMD processed AZ31-3Ca alloy is shown in Figure 1a,b as viewed in an optical microscope and in scanning electron microscope, respectively. The grain size is fine with the average grain diameter in the range 2–3 µm, as determined using linear intercept method. The microstructure has a large volume fraction of (Mg,Al)2Ca intermetallic particles distributed uniformly in the microstructure along with a random distribution of a small volume fraction of Al8Mn5 particles.

![Image](a)  
(a) Optical, (b) scanning electron microscope (SEM) micrograph, and (c,d) energy dispersive spectroscopy (EDS) spectra at locations of intermetallic particles.

The elemental distribution of Al, Ca, Zn and Mn as obtained by energy dispersive spectroscopy (EDS) mapping is shown in Figure 2 which confirms the uniformity of their distribution. The formation of the above two phases is according to the predictions of thermodynamic model [26,27], and comparable to those reported in the literature [6,12,28]. The elemental composition at the selected spots on the
The compressive strength of the AZ31-3Ca alloy measured at a temperature 150 °C, typical heat resistant limit for magnesium alloys, along with the strengths of AZ31 alloy, AZ31-1Ca, and AZ31-2Ca are shown in Figure 3. It can be seen that the ultimate compressive strength (UCS) has significantly increased with the addition of Ca, with UCS of AZ31-3Ca nearly double that of base AZ31 alloy. However, AZ31-3Ca exhibited highest brittleness as well that makes forming most difficult, vindicating the need to establish precise thermomechanical processing options.
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The true stress–true strain curves obtained in compression of DMD processed AZ31-3Ca alloy at 300 °C and 400 °C and at different strain rates are shown in Figure 4a,b, respectively. The curves at 300 °C exhibited flow softening at strain rates higher than about 0.01 s\(^{-1}\) and are of steady-state type at lower strain rates. However, at higher temperatures the steady-state flow occurred at higher strain rates. The shapes of deformed specimens are shown in Figure 5 and the alloy exhibited homogeneous flow at all temperatures and strain rates.

Figure 3. (a,b) Stress-strain curves obtained in compression for AZ31, AZ31-1Ca, AZ31-2Ca, and AZ31-3Ca alloys. The ultimate compression strengths of the alloys are also shown for comparison.

The true stress–true strain curves obtained in compression of DMD processed AZ31-3Ca alloy at 300 °C and 400 °C and at different strain rates are shown in Figure 4a,b, respectively. The curves at 300 °C exhibited flow softening at strain rates higher than about 0.01 s\(^{-1}\) and are of steady-state type at lower strain rates. However, at higher temperatures the steady-state flow occurred at higher strain rates. The shapes of deformed specimens are shown in Figure 5 and the alloy exhibited homogeneous flow at all temperatures and strain rates.

Figure 4. True stress–true strain curves obtained in compression on DMD processed AZ31-3Ca alloy at different strain rates at temperatures of (a) 300 °C, and (b) 400 °C.
Figure 4. True stress–true strain curves obtained in compression on DMD processed AZ31-3Ca alloy at different strain rates at temperatures of (a) 300 °C, and (b) 400 °C.

Figure 5. Shapes of deformed cylindrical specimens of DMD processed AZ31-3Ca alloy compressed at different temperatures and strain rates.

The processing map obtained at a true strain of 0.7 is shown in Figure 6. The numbers assigned to the contours represent efficiency of power dissipation expressed in percent. The maps at other strains are not significantly different after steady-state flow has occurred. The map exhibits three domains in the temperature and strain rate ranges given below.

Domain (1): 300–375 °C and 0.0003–0.003 s$^{-1}$ with a peak efficiency of about 53% at 375 °C/0.0003 s$^{-1}$.

Domain (2): 375–450 °C and 0.0003–0.003 s$^{-1}$ with a peak efficiency of about 58% at 405 °C/0.0003 s$^{-1}$.

Domain (3): 375–450 °C and 0.1–10 s$^{-1}$ with a peak efficiency of about 35% at 425 °C/0.1 s$^{-1}$.

Figure 6. Processing map for DMD processed AZ31-3Ca alloy at a strain of 0.7. The numbers on the contours represent efficiency of power dissipation in percent.
Domains (1) and (2) have merged at about 375 °C in view of the higher efficiency range exhibited by Domain (2).

The microstructure obtained on a specimen deformed at 300 °C/0.0003 s\(^{-1}\) in Domain (1) is shown in Figure 7a. It exhibits fine grain size suggesting that dynamic recrystallization occurs in this domain. At temperatures corresponding to this domain, basal slip along with prismatic slip occurs. In view of the slower strain rates, the recovery mechanism that nucleates DRX will be climb of edge dislocations involving lattice self-diffusion, although limited amount of cross-slip may be associated with prismatic slip. Kinetic analysis of the temperature and strain rate dependence of flow stress has been conducted using Equation (3). The variation of normalized flow stress with logarithms of strain rate is shown in Figure 8a and the Arrhenius plot showing the variation of normalized flow stress with inverse of absolute temperature is shown in Figure 8b. It may be noted that the kinetic rate equation is obeyed within the deterministic domains. The stress exponent obtained in Domain (1) is 4.29 and the apparent activation energy is 158 kJ/mole. For the mechanism of climb, which is controlled by lattice self-diffusion, the activation energy is expected to be 135 kJ/mole [29]. The estimated value is higher than that for lattice self-diffusion, suggesting that the high-volume fraction of Ca-containing intermetallic particles causes considerable back stress enhancing the apparent activation energy.

![Figure 7](image)

**Figure 7.** Microstructures obtained on DMD processed AZ31-3Ca alloy deformed at (a) 350 °C/0.0003 s\(^{-1}\) (Domain 1) (b) 400 °C/0.0003 s\(^{-1}\) (Domain 2) (c) 450 °C/10 s\(^{-1}\) (Domain 3), and (d) 300 °C/10 s\(^{-1}\) (instability regime). The compression axis is vertical.
The apparent activation energy estimated in this domain (Figure 8) is 163 kJ/mole, which is higher than the activation energy for grain boundary self-diffusion in Mg (95 kJ/mole) [30]. The apparently higher value may be attributed to the back-stress caused by the large volume fraction of Ca-containing intermetallic phase (Mg,Al)₂Ca increases and the grain size decreases to about 2–3 μm.

The microstructure obtained on a specimen deformed at 400 °C and 0.0003 s⁻¹ near the peak efficiency in Domain (2) is shown in Figure 7b which exhibits equiaxed dynamically recrystallized structure. In addition, intercrystalline cracking has occurred during deformation. Firstly, in view of the higher temperature range in which this domain occurs, second-order pyramidal slip occurs readily. The availability of large number of intersecting slip systems and the higher stacking fault energy (172 mJ/m²) [30] promote the occurrence of extensive cross-slip which nucleates DRX. Secondly, once the fine-grained structure is produced by DRX, grain boundary sliding occurs since the strain rates are slow resulting in wedge cracking and cracking along the grain boundaries. The high efficiency in this domain (58%) and the steep increase in efficiency with decreasing strain rate (appearing as closely spaced contours representing steep efficiency hill) are also indicative of such a grain boundary sliding mechanism. Thus, the end result of deformation in this domain is grain boundary cracking, which is not desirable.

The microstructure of a specimen deformed at 450 °C/10 s⁻¹ (Domain 3) is shown in Figure 7c and exhibits DRX. Within the temperature range in this domain (375–450 °C), basal, prismatic as well as second-order pyramidal slip systems can cause deformation. However, since this domain occurs at higher strain rates (0.1–10 s⁻¹), the recovery mechanism is likely to be grain boundary self-diffusion since lattice self-diffusion is a slower process and higher strain rates slow down recovery by cross-slip. The apparent activation energy estimated in this domain (Figure 8) is 163 kJ/mole, which is higher than the activation energy for grain boundary self-diffusion in Mg (95 kJ/mole) [30]. The apparently higher value may be attributed to the back-stress caused by the large volume fraction of Ca-containing intermetallic particles in the microstructure.

The processing map exhibits a regime of flow instability in the temperature range 300–310 °C at strain rates higher than about 0.1 s⁻¹. The microstructure of the specimen deformed at 300 °C/10 s⁻¹ is shown in Figure 7d which reveals that the flow instability manifestation is adiabatic shear band formation (marked by arrows). Cracking has occurred along the shear band due to high intensity of localization which is nearly at 45° with respect to the compression axis.

An interesting feature exhibited by the processing map is the occurrence of a bifurcation in the temperature range 325–425 °C and strain rate range 0.003–0.1 s⁻¹, where the efficiency has reached a
low value of 28%. This represents a region between the three domains identified above. In dynamic systems terminology [31], the bifurcation is akin to a dissipative energy hill and may be viewed as a saddle configuration. Processing under conditions of bifurcation will lead to microstructural instability since any small perturbation will trigger a different mechanism surrounding the bifurcation. The microstructures of the specimen deformed at 400 °C and at two strain rates (0.001 and 0.01 s⁻¹) near the bifurcation region are shown in Figure 9a,b. The microstructural instability is manifested in terms of a combination of large and small grains in Figure 9a and very fine grains in Figure 9b, revealing that microstructural control is difficult if processed around the bifurcation region.

Figure 9. Microstructures obtained on DMD processed AZ31-3Ca alloy specimens deformed at (a) 400 °C/0.001 s⁻¹ (b) 400 °C/0.01 s⁻¹ (near bifurcation region).

The processing maps for DMD processed AZ31 [17], AZ31-1Ca [18], AZ31-2Ca [19] are compared in Figure 10 with that obtained currently on AZ31-3Ca alloy at a strain of 0.7. The starting microstructure, the characteristics of the different domains exhibited by the maps and the interpretations are shown in Table 1. With increasing Ca content in AZ31, the volume fraction of Ca-containing intermetallic phase (Mg,Al)₂Ca increases and the grain size decreases to about 2–3 μm. The increasing volume fraction of the intermetallic phase has an effect on the hot working behavior of AZ31.

Figure 10. Cont.
Initial grain size, AZ31-1Ca, AZ31-2Ca, and AZ31-3Ca magnesium alloys.

Table 1. Details of (a,b) microstructural parameters, (c) temperature—strain rate ranges for domains, (d) peak efficiency (η) conditions, (e) apparent activation energy (Q<sub>app</sub>) and (f) proposed mechanisms (DRX: dynamic recrystallization) in the various domains of processing maps for DMD processed AZ31, AZ31-1Ca, AZ31-2Ca and AZ31-3Ca magnesium alloys.

| Parameter                          | AZ31 (Ref. 17) | AZ31-1Ca (Ref. 18) | AZ31-2Ca (Ref. 19) | AZ31-3Ca (current work) |
|------------------------------------|----------------|--------------------|--------------------|-------------------------|
| Initial grain size, µm             | (a) 9          | 3                  | 3                  | 2–3                     |
| Phases in the microstructure       | (b) Mg<sub>6</sub>Al<sub>12</sub> | (Mg,Al)<sub>2</sub>Ca Al<sub>3</sub>Mn<sub>5</sub> | (Mg,Al)<sub>2</sub>Ca Al<sub>3</sub>Mn<sub>5</sub> | (Mg,Al)<sub>2</sub>Ca Al<sub>3</sub>Mn<sub>5</sub> |
| Domain (1)                         |                |                    |                    |                         |
| (c) 300–350 °C/0.003–0.01 s<sup>-1</sup> | 300–375 °C/0.003–0.003 s<sup>-1</sup> | 300–375 °C/0.003–0.003 s<sup>-1</sup> | 300–375 °C/0.003–0.003 s<sup>-1</sup> |
| (d) 300 °C/0.0003 s<sup>-1</sup>; peak η: 39% | 300 °C/0.0003 s<sup>-1</sup>; peak η: 45% | 350 °C/0.0001 s<sup>-1</sup>; peak η: 37% | 375 °C/0.0003 s<sup>-1</sup>; peak η: 53% |
| (e) Q<sub>app</sub>: 126 kJ/mole | Q<sub>app</sub>: 112 kJ/mole | Q<sub>app</sub>: 133 kJ/mole | Q<sub>app</sub>: 158 kJ/mole |
| (f) DRX: Basal slip + climb by Lattice self-diffusion | DRX: Basal slip + climb by lattice self-diffusion | DRX: prismatic slip + climb by lattice self-diffusion | DRX: Basal slip + prismatic slip + climb by lattice self-diffusion |
| Domain (2)                         |                |                    |                    |                         |
| (c) 350–450 °C/0.003–0.01 s<sup>-1</sup> | 400 – 450 °C/0.0003–0.003 s<sup>-1</sup> | 375–450 °C/0.0003–0.003 s<sup>-1</sup> | 375–450 °C/0.0003–0.003 s<sup>-1</sup> |
| (d) 425 °C/0.0003 s<sup>-1</sup>; peak η: 46% | 450 °C/0.0003 s<sup>-1</sup>; peak η: 38% | 450 °C/0.0003 s<sup>-1</sup>; peak η: 48% | 405 °C/0.0003 s<sup>-1</sup>; peak η: 58% |
| (e) Q<sub>app</sub>: 144 kJ/mole | Q<sub>app</sub>: 166 kJ/mole | Q<sub>app</sub>: 192 kJ/mole | Q<sub>app</sub>: 215 kJ/mole |
| (f) Wedge cracking | Wedge cracking | Second-order pyramidal slip + cross slip | Grain boundary cracking |
| Domain (3)                         |                |                    |                    |                         |
| (c) 350–435 °C/1–10 s<sup>-1</sup> | 325–390 °C/0.3–10 s<sup>-1</sup> | 375–450 °C/0.3–10 s<sup>-1</sup> | 375–450 °C/0.1–10 s<sup>-1</sup> |
| (d) 400 °C/10 s<sup>-1</sup>; peak η: 26% | 350 °C/10 s<sup>-1</sup>; peak η: 32% | 400 °C/10 s<sup>-1</sup>; peak η: 38% | 425 °C/0.1 s<sup>-1</sup>; peak η: 35% |
| (e) Q<sub>app</sub>: 127 kJ/mole | Q<sub>app</sub>: 87 kJ/mole | Q<sub>app</sub>: 166 kJ/mole | Q<sub>app</sub>: 163 kJ/mole |
| (f) DRX: climb by grain boundary self-diffusion | DRX: climb by grain boundary self-diffusion | DRX: climb by grain boundary self-diffusion | DRX: climb by grain boundary self-diffusion |
The characteristics of Domain (1) did not change significantly although the temperature for peak efficiency and the apparent activation energy have increased with increasing Ca content. In this domain, DRX occurs by basal slip + prismatic slip and recovery primarily by climb of edge dislocations controlled by lattice self-diffusion. The higher back-stress generated by the presence of intermetallic particles increases the back-stress to dislocation generation as well as dynamic recovery process which is responsible for moving the domain to higher temperatures and for higher apparent activation energy.

Domain (2) occurs in similar temperature range in all the alloys although the temperature at which peak efficiency occurs increases in AZ31-1Ca and AZ31-2Ca alloys and decreases in AZ31-3Ca alloy. The peak efficiency exhibits a high value in AZ31-3Ca alloy and the apparent activation energy is higher than that for lattice self-diffusion in all the alloys. In view of the higher temperature in which this occurs, DRX occurs by second-order pyramidal slip and recovery by cross-slip. The fine-grained structure produced by this DRX process promotes grain boundary sliding during large deformation, which results in wedge cracking and intercrystalline fracture. However, in AZ31-2Ca alloy, wedge cracking did not occur since the grain boundary sliding process is restricted by Ca-containing intermetallic particles. When the Ca content is further increased to 3%, the grain boundaries are weakened by a large volume content of the particles resulting in intercrystalline cracking. Thus, processing in Domain (2) which occurs at higher temperatures and lower strain rates, is not desirable since it results in intercrystalline cracking except in AZ31-2Ca alloy.

Domain (3) moves to higher temperatures with increasing Ca content in AZ31. In this domain, non-basal slip systems operate extensively to cause DRX. The recovery mechanisms of climb by lattice diffusion and cross-slip to nucleate DRX are restricted in the high strain rate range at which this domain occurs. Instead, recovery occurs by grain boundary self-diffusion. The apparent activation energy values are higher than that expected for grain boundary self-diffusion (95 kJ/mole) [29] which may be attributed to the higher back stress caused by the Ca-containing intermetallic particles. It may be noted that much slower rates are required for recovery in AZ31-3Ca since a larger volume content of Ca-containing intermetallic particles will be present at the grain boundaries slowing the rate of recovery by grain boundary self-diffusion. In all the Ca-containing AZ31 DMD processed alloys, hot working is probably best done in this domain.

5. Conclusions

The hot deformation mechanisms in DMD processed AZ31-3Ca alloy have been evaluated using processing maps, and the behavior has been compared with that of base AZ31, AZ31-1Ca and AZ31-2Ca alloys with a view to bringing out the effect of Ca addition. The following conclusions are drawn from this investigation.

(1) Addition of 3% Ca to AZ31 refines the grain size and increases the volume fraction of (Mg,Al)\textsubscript{2}Ca intermetallic phase, which is distributed uniformly in the microstructure.

(2) The processing map for AZ31-3Ca alloy exhibited three domains in the temperature strain rate ranges: (1) 300–375 °C and 0.0003–0.003 s\textsuperscript{-1}, (2) 375–450 °C and 0.0003–0.003 s\textsuperscript{-1}, and (3) 375–450 °C and 0.1–10 s\textsuperscript{-1}.

(3) In Domain (1), DRX occurs when basal + prismatic slip associated with dynamic recovery by climb controlled by lattice self-diffusion. In Domain (2), the plastic flow results in intercrystalline cracking promoted by grain boundary sliding initiating wedge cracks. In Domain (3) that occurs at higher strain rates, DRX occurs by non-basal slip with recovery by climb controlled by grain boundary self-diffusion. This recovery is promoted by fine grain size in the alloy.

(4) The alloy exhibits some flow instability at lower temperatures and higher strain rates, and this manifests as adiabatic shear bands.

(5) A comparison of processing maps on the Ca containing AZ31 alloys revealed that the hot deformation mechanisms have not significantly changed by increasing Ca addition except in
AZ31-2Ca alloy where wedge cracking is avoided in Domain (2) due to the reduction of grain boundary sliding that is attributed to pinning by intermetallic particles.

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