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# FORMABILITY ENHANCEMENT IN HOT EXTRUDED MAGNESIUM ALLOYS

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## Abstract

The effects of cerium (Ce), aluminum (Al) and manganese (Mn) additions on the microstructure and mechanical properties of hot extruded magnesium alloys have been investigated. Seven different compositions of Mg alloys - Pure Mg, Mg-0.2Ce, Mg-0.5Ce, AM30, AM50, Mg-3Al-0.2Ce and Mg-5Al-0.2Ce were hot extruded under optimized process conditions. Minor addition of Ce to Mg was found to enhance its ductility from <10% to >30% and 3 - 5% Al addition resulted in ~40% and 50% increase in strength respectively, compared to pure Mg. A combination of 5% Al and 0.2% Ce addition resulted in concurrent improvement in strength and ductility as a result of concurrent grain size refinement, texture randomization and solute strengthening.

## Introduction

Magnesium (Mg) is emerging as a potential material for automotive applications, primarily because of its light-weight and cost competitiveness. When alloyed, Mg has higher strength-to-weight ratio compared to other structural metals and alloys. Mg alloys possess good damping characteristics and machinability. However, limited ductility and poor room temperature formability are major obstacles for wider use of wrought Mg. Higher temperature is required to allow activation of prismatic {1010}<11 2 0> and pyramidal {1011}<11 2 0> slip systems, in addition to basal (0001)<11 2 0> slip systems [1-2] to enable Mg alloys to be formed to any shape. The demand for lightweight, energy efficient materials provides a strong impetus to develop magnesium alloys with high strength and improved ductility, formable at room temperature.

Ductility in Mg can be increased by several methods. Chapman et al [3] have used grain refinement to improve ductility and strength. Menzen et al [4] in 1940 showed that alloying of Mg alloys with Ca, Th, Mn and certain rare earth (RE) elements improved cold rolling behavior at ambient temperature [5]. More recently, Mohri et al [6] have reported a combination of high strength and high ductility in hot extruded Mg-Y-RE (rare earth) alloy. Mukai et al [7] have found that grain refinement in WE43 Mg alloy results in enhanced ductility even at a dynamic strain rate of $\sim 2\times10^3$ $s^{-1}$. Barnett et al [8] have studied cold rolling behavior of pure Mg, AZ31 and Mg-0.2% Ce alloys and reported that, unlike pure Mg and AZ31 alloys, Mg-0.2% Ce alloy could be cold rolled to 90% reduction without failure. This enhanced cold rollability has been ascribed to modification of texture sharpness and shear banding. Ma et al [9] have reported grain refinement in extruded ZK60 alloy where higher RE content leads to increased tensile strength but reduced ductility. Mukai et al [10] have reported grain size refinement and texture modification in AZ31 by ECAP method with enhanced room temperature ductility.

There still continues to be a lack of data on the effect of processing parameters on rare earth alloyed Mg produced by single-pass extrusions. In the first part of this paper, the results of a systematic study of the effect of small Ce addition on the strength and ductility of Mg extruded at different temperatures and at different extrusion ratios are presented. The second part of the paper presents the effect of individual and combined addition of Ce and Al on hot extruded rods under optimized extrusion conditions to identify an alloy with combined high strength and high ductility. Microstructure and texture studies to establish the underlying mechanisms for the enhancement of strength and ductility are reported.

## Experimental

The chemical composition of the billets extruded in this work is shown in Table 1. Hot extrusion experiments were conducted using a 500-ton Wellman Enefco vertical hydraulic press from 75 mm x 200 mm size billets. The effects of extrusion ratio (ER) and billet temperature on microstructure and properties of the extruded rods were investigated and optimized for Mg-0.2Ce billets for a constant extrusion speed of 10 mm (billet)/s, which is within the acceptable range for magnesium extrusions [11]. Extrusion ratios (ER) of 9:1, 25:1 and 36:1 and billet temperatures of 350°C, 400°C and 450°C were used. The effect of alloy chemistry was investigated employing the optimized processing parameters listed in Table 2 on the rest of the billets.

Table 1. Composition of billets.

| Material | Composition (wt%) | | | | |
|---|---|---|---|---|---|
| | Ce | Al | Mn | Zn | Mg |
| (a) Pure Mg | -- | -- | -- | -- | ~100 |
| (b) Mg-0.2Ce | 0.20 | 0.009 | 0.027 | <0.001 | 99.69 |
| (c) Mg-0.5Ce | 0.60 | 0.011 | 0.029 | <0.001 | 99.63 |
| (d) AM30 | <0.001 | 2.45 | 0.42 | <0.001 | 96.89 |
| (e) AM50 | <0.001 | 4.64 | 0.32 | <0.001 | 94.74 |
| (f) Mg-3Al-.2 Ce | 0.17 | 2.440 | 0.150 | 0.003 | 97.23 |
| (g) Mg-5Al-.2 Ce | 0.18 | 4.490 | 0.220 | 0.001 | 95.10 |

The metallographic samples for porosity, grain size and second phase studies were prepared by etching polished samples of longitudinal and transverse sections of extruded rods using a solution containing 20 ml acetic acid - 6 gm picric acid - 20 ml $H_2O$ - 50 ml ethanol. Microstructural evaluation as well as fractography was carried out using a Nikon Optical Microscope interfaced with a Leco Image Analyzer and a Leo 1450 scanning electron microscope (SEM) operating at 20 kV.

Table 2. Optimized hot extrusion processing parameters.

| | |
|---|---|
| Container dia (mm) | 75 |
| Billet temperature (°C) | 400 |
| Soaking time (hrs) | 2 |
| Extrusion ratio (ER) | 25:1 |
| Pressing speed (mm/s) | 10 |
| Lubricant | Boron Nitride |

Vickers Microhardness was measured using a Future Tech Microhardness Tester employing a load of 300g. Dumbbell shaped tensile specimens with 25 mm gauge length and 6.25 mm gauge diameter were used for tensile testing at room temperature at a strain rate of $0.66 \times 10^{-3}$/s using a Universal Testing Machine. Only the ultimate tensile strength (UTS) and % elongation were measured to assess and optimize strength and ductility values. Electron backscatter diffraction (EBSD) analysis of the samples were done using TSL camera and software in conjunction with a Leo 1455 SEM operating at an accelerating voltage of 20kV and a camera length of 18mm.

## Results

The average volume fraction of pores in the cast Mg-0.2Ce billet is measured to be 2.48%, which drops to a level of 0.54% for the rods extruded at 450$^0$C at ER of 9:1. At ERs of 25:1 and 36:1 the porosity content is found to be negligible (~0.02%). Optical micrographs of Mg-0.2Ce rods extruded at 450$^0$C in Figure 1 show that the grains are nearly equiaxed in the transverse section for all three ERs. Few elongated bands of fine grains are present in the longitudinal section. The fraction of fine-grained bands is higher at higher ER. Figure 2 shows the EBSD grain maps and texture maps from the transverse section. It is clear that the texture of the extruded rod depends on the extrusion ratio. The rod extruded at ER of 25:1 has the c-axes of the grains at ~45$^0$ to the extrusion axis while that at 9:1 and 36:1 have their c-axes nearly normal to the extrusion axis [12]. Average grain size and mechanical properties are shown in Figure 3. The grain size in Figure 3 progressively increases with ER in both the transverse and longitudinal directions. Maximum ductility of ~28% with an optimum strength of 171 MPa is obtained at ER=25:1. Extrusion ratio of 25:1 is chosen as the optimum ER for the rest of the study from this data.

Figure 4 shows optical micrographs of Mg-0.2Ce alloy, extruded at billet temperatures of 350, 400 and 450°C at an extrusion ratio of 25:1. Both longitudinal and transverse directions exhibit equiaxed grains at all extrusion temperatures and the grain size increases with temperature. Figure 5 shows the mechanical properties (UTS and % elongation) of extruded Mg-0.2Ce alloy at different billet temperatures. The average grain size in the longitudinal direction at the three temperatures of 350, 400 and 450°C is 26, 34, and 47 μm, respectively, consistent with data in the literature [11, 13]. It is observed that while the UTS does not change with extrusion temperature, an optimum combination of strength and ductility is obtained at a billet temperature of 400°C. This temperature is considered to be optimum for subsequent extrusion experiments.

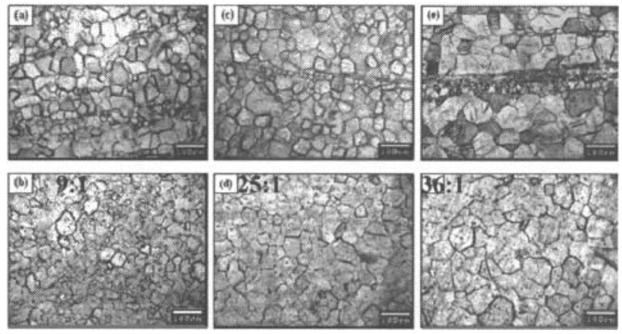



Figure 1. Optical micrographs from longitudinal (Top row, ED horozontal) and transverse (bottom, ED normal to image plane) sections of Mg-0.2Ce rods extruded at 450$^0$C.

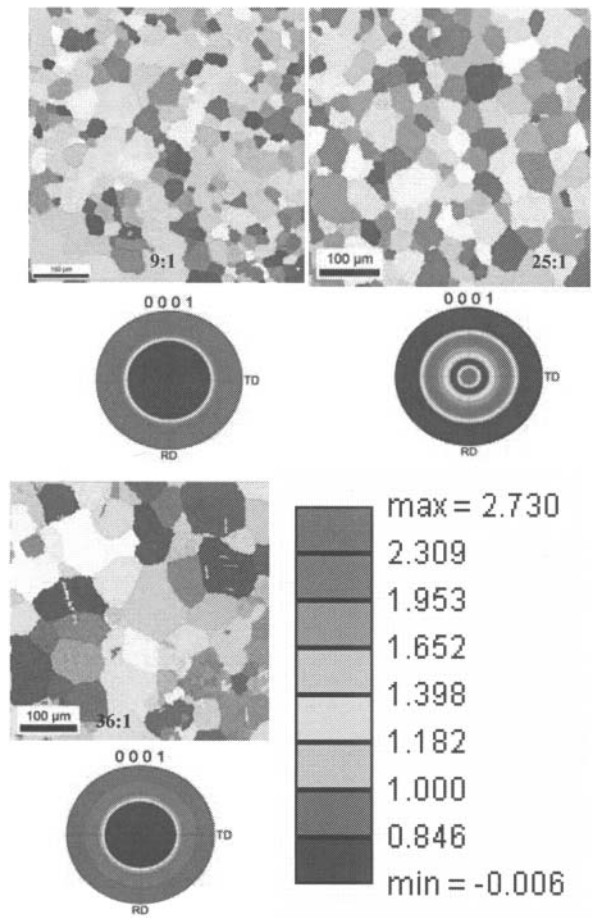



Figure 2. EBSD inverse pole figure maps and corresponding (0001) axial (symmetry) texture plots from transverse sections of the rods.

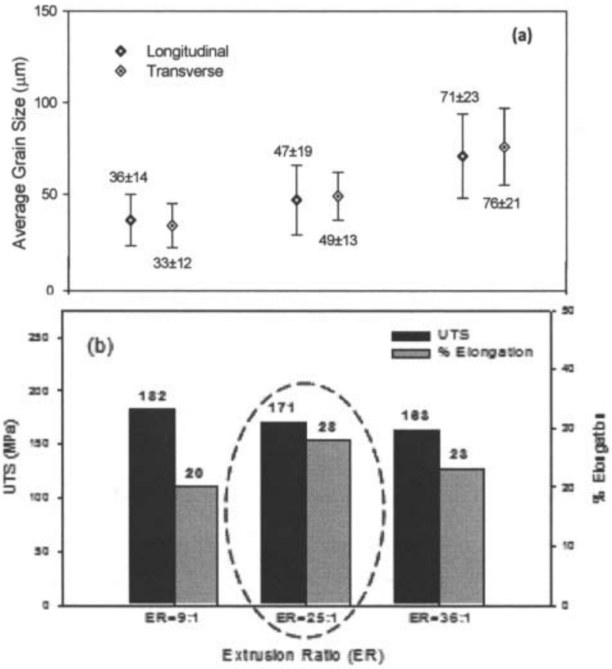



Figure 3. Effect of extrusion ratio (ER) on (a) average grain size, (b) tensile properties, of Mg-0.2Ce alloy [billet temperature: 450°C, pressing speed: 10 mm/s].

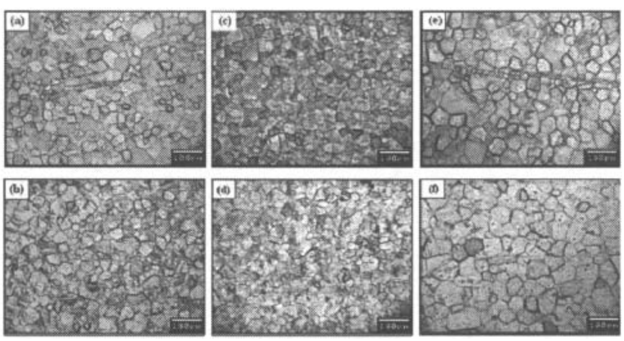


Figure 4. Microstructure of extruded Mg-0.2Ce alloy showing the effect of billet temperature (350 let, 400 center and 450 right) in longitudinal (top) and transverse (bottom) direction (ER = 25;1).

Figure 6 shows optical micrographs from longitudinal section of rods of different Mg alloys listed in Table 1. The average grain size and Vickers microhardness of each of the alloy is also shown in the inserted table. The maximum refinement of microstructure is obtained for Mg alloyed with Al and 0.2% Ce. Microhardness value increases marginally with Ce addition but considerably with Al addition. SEM micrographs taken in back scattered mode and their corresponding EDS patterns show the microstructure of pure Mg to be single-phase α-Mg. Presence of Mg-Ce intermetallics in Mg-Ce alloys and Mg-Ce-Al or Mg-Al-Mn intermetallics in Mg-Ce-Al and AM series alloys are seen. The fraction and size of intermetallics increased with alloying content. The EBSD texture data from the longitudinal sections of the rods for Mg, Mg-0.2Ce, Mg-0.5Ce and Mg-3Al-0.2Ce, shown in Figure 7, confirm that the c-axes in Mg-0.2Ce and Mg-0.5Ce are oriented at an angle to the extrusion axis while those in pure Mg and Mg-Al-Ce are nearly normal to the extrusion axis.

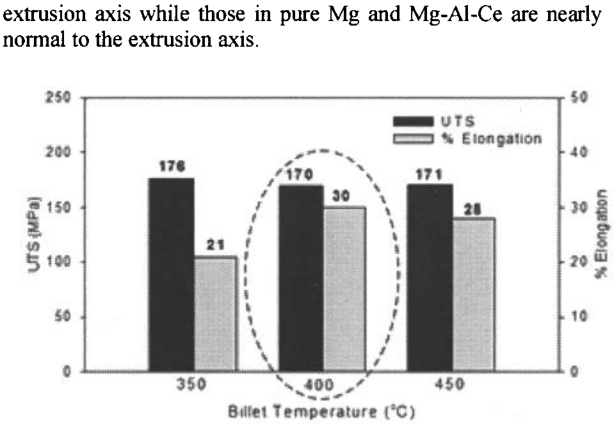
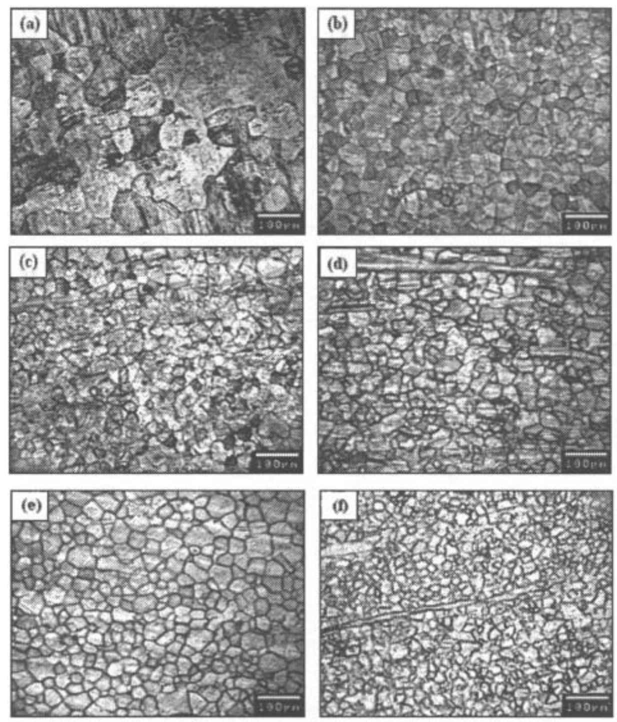
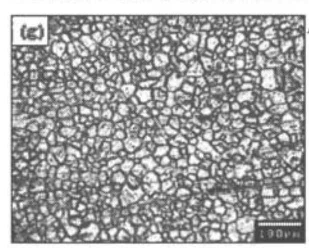



Figure 5. Effect of billet temperature on tensile properties of Mg-0.2Ce alloy [ER=25:1, pressing speed: 10 mm/s].





| Alloy | Grain Size (mm) | Microhardness HV300 |
|---|---|---|
| a | 61 | 32 |
| b | 34 | 35 |
| c | 25 | 39 |
| d | 30 | 47 |
| e | 31 | 51 |
| f | 27 | 50 |
| g | 26 | 54 |

Figure 6. Optical metallographs from seven extrusions with their microhardness and grain sizes in the insert table. Alloy compositions in Table 1.

Figure 8 shows the UTS and elongation data for all seven extrusions. The data can be grouped into four groups. The maximum strength of 286 MPa was observed for Mg-5Al-0.2Ce alloy. It can be concluded that pure Mg has low strength and low ductility, Mg-0.2Ce and Mg-0.5Ce have high ductility but poor strength; AM30 and AM50 fall under the category of low ductility with high strength and Mg-3Al-0.2Ce and Mg-5Al-0.2Ce alloys are high strength and high ductility alloys.

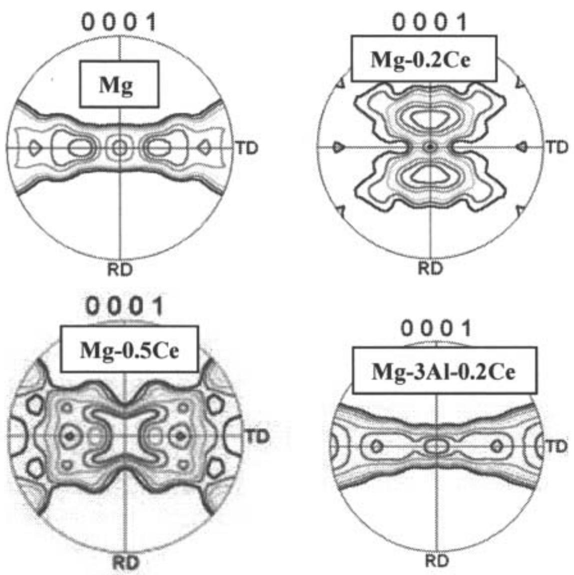



Figure 7. EBSD (0001) texture plots from longitudinal sections of pure Mg, Mg-.2Ce, Mg-.5Ce and Mg-3Al-.2Ce extrusions. RD parallel to extrusion direction. Pole figures are calculated with orthotropic symmetry for this orientation of the sample

## Discussion

The reduction of strength in case in Mg-Ce alloys is believed to be due to "texture softening" seen clearly in Figure 7. Ce addition to Mg modifies the texture to favor basal slip (the Schmid factor of the grains oriented at 45 degrees to extrusion axis is high), enhancing ductility (% elongation). The increase in strength with Al addition is from solute strengthening and through the formation of dispersions of Al-Ce-Mn intermetallics [14]. Equiaxed grains in samples up to ER 25:1 form due to dynamic recrystallization. Bands of extremely fine grains observed at ER 36:1 form mainly due to non-uniform dynamic recrystallization. The increase in average grain size with ER is opposite of previously reported results [15], a result of grain growth due to high extrusion temperature employed in the present investigation (450°C) and the additional adiabatic heating for higher ERs. Addition of Al and Mn in AM30 and AM50 alloys led to substantial grain refinement while only minor addition of Ce (0.2%) resulted in considerable grain refinement. The grain size further decreased with increasing Ce to 0.5%. Minimum grain size of 25±9 µm was observed in Mg-0.5Ce and nearly comparable grain sizes were observed with alloying additions of Al and Ce. In all these cases, substantial number of intermetallic particles are present. The addition of Ce and Al as alloying elements seems to have provided additional nucleation sites for dynamic recrystallization and stronger pinning sites for grain boundaries to prevent grain growth. Increasing Al content from 3% to 5% and keeping Ce addition to 0.2% didn't have much effect on the grain size, most likely because all the Ce was tied up with 3 % Al already and thus the number of these intermetallics did not increase as a result.

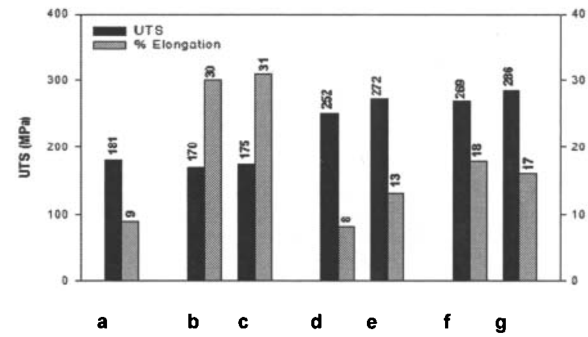



Figure 8. UTS and Elongation values of extruded rods

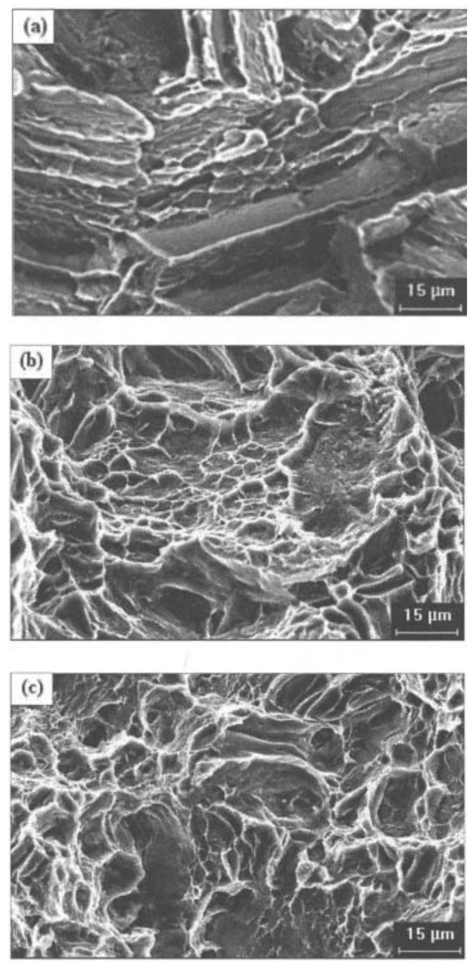



Figure 9. SEM images of fractured tensile bars from optimized (a) pure Mg, (b) Mg-0.5Ce and (c) Mg-3Al-0.2Ce rods.

The ductility of Mg-0.2Ce alloy is ~30%, which, in absolute terms, amounts to an enhancement of ~ 233% over that of pure Mg with a marginal decrease in the UTS value. It is believed that texture sharpness and severity of shear banding are responsible for the enhanced ductility of hot extruded Mg alloys with minor addition of Ce [12]. Though Al serves as solid solution strengthening element as observed in the case of AM30 and AM50 alloys, strengthening effect is more prominent in Mg-Al-Ce alloys due to the combined effect of solid solution effect, formation of complex Al-Ce-Mn intermetallics as well as the accompanying changes in grain size and texture.

The data suggests that addition of Ce to Mg-Al alloys does not contribute to ductility enhancement because Ce is tied up by Al-Ce intermetallics and is not available to alter deformation and recrystallization behavior that ultimately contributes to texture modification. In the absence of Al, there is a finite amount of Ce still dissolved in Mg that alters the deformation and recrystallization behavior. In a separate paper in this Proceeding [16], the influence of dynamic strain ageing in Mg-Ce alloys at high temperature and specific strain rates has been shown to influence the RE texture formation and this is believed to be behind the presence of RE texture in the sample extruded at 25:1 extrusion ratio. Since dynamic strain ageing occurs in a specific temperature/strain rate window, the observations strongly suggest this to be the governing physical mechanism for the texture modification in extruded Mg-Ce alloys.

Substantial improvement in ductility without much change in strength has been obtained with minor Ce addition to Mg. Mg-Al-Ce alloys have high strength coupled with high ductility. The fractured tensile specimens of Mg and Mg-0.5Ce were examined under SEM to correlate the fracture behavior with tensile properties [12]. SEM micrograph of the fractured surfaces of Pure Mg in Figure 9 shows quasi-cleavage fracture mode, while Mg-0.5Ce and Mg-Al-Ce alloy shows nearly spherical dimples from ductile failure. This fracture behavior contributes to improved ductility of Mg-0.5Ce and Mg-Al-Ce alloy.

## Conclusions

Minor addition of cerium (0.2% Ce) to magnesium resulted in texture softening and grain refinement under specific extrusion conditions. Increasing Ce content from 0.2% to 0.5% did not improve ductility further. SEM and EDAX investigations revealed the presence of uniform distribution of fine intermetallic particles. Fractured samples of Mg-0.5Ce showed near spherical dimples. The enhanced ductility in extruded Mg-Ce alloys is attributed to less texture sharpness and grain size reduction with accompanying changes in the deformation mechanisms. Addition of 3% and 5% Al to Mg (AM30 and AM50) resulted in strength improvement without significant improvement in ductility. Addition of Al in combination with 0.2% Ce enhanced both strength as well as ductility. Extruded Mg-5Al-0.2Ce rods had the best strength and ductility combinations.

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