

Microstructure and Mechanical Behavior of as Cast and Hot Extruded AlZnMgCu Alloy with Rare Earth Erbium Additions

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Abstract: The effects of rare earth element, erbium (Er) additions on the microstructure and mechanical properties of Al-15Zn-2.5Mg-2.5Cu alloy have been investigated. This new high strength alloy with erbium additions (0.5, 1.0, 1.5 and 2.0 wt%) was synthesized by liquid metallurgy route followed by hot extrusion. Microstructural characterization was performed using scanning electron microscope and electron probe microanalysis. Significant amount of grain refinement was observed with erbium addition in the hot extruded and heat treated alloy. Tensile test was performed to investigate the effect rare earth on mechanical behavior of alloy in as cast and hot extruded condition before and after T6 heat treatment. The combined effect of erbium addition, hot extrusion and heat treatment significantly enhanced the tensile strength of alloy (602 MPa) when compared to the as cast alloy without erbium addition (225 MPa). The strengthening of the alloy was attributed to grain refinement caused by erbium along with hot extrusion and formation of precipitates after T6 heat treatment. Fractographic investigations revealed that the hot extruded alloy with erbium addition after heat treatment showed uniformly distributed deep dimples exhibiting ductile behavior.

Keywords: Casting, Extrusion, Rare earth, Microstructure, Mechanical Properties.

1. INTRODUCTION

It is well known that Al 7xxx series alloys are very popular for structural applications due to their low density, high strength, weldability and good workability. In particular the extruded profiles are used for making crash boxes in cars, fuselage stringers and frames for airframe structures [1,2]. The high strength in this alloy is mainly due to the presence of various alloying elements and formation of fine precipitates whose precipitation sequence is given as: Supersaturated solid solution → Guinier-Preston zones (GP) → Intermediate Phase (η') → Stable phase (η - MgZn_2). The intermediate or metastable η' phase is mainly responsible for peak hardening while coarsening of η phase can be detrimental for the strength. High strength is generally obtained by uniform dispersion of GP zones and metastable η' phase which is achieved by proper combination of heat treatment. The artificial aging is usually done in two steps in

which annealing is carried out as first step in order to nucleate the GP zones followed by second step in which GP zones are allowed to grow to facilitate the nucleation of η' phase [3-5]. Suh et al [6] reported the microstructural evolution of AlZnMgCu alloy during hot extrusion and post heat treatment process. The microstructure of extruded alloy showed elongated grains while recrystallization was observed during heat treatment. It was found that the similar alloy with higher zinc and copper content showed recovery process during extrusion with abnormal grain growth after heat treatment. Paulisch et al. [7] studied the mechanical properties of two aluminium alloys Al 7175 and Al 7108 after hot extrusion and artificial peak ageing process. The Al 7175 alloy showed homogeneous dispersion of GP zones after artificial ageing with an ultimate tensile strength value up to 636 MPa. On the other hand Al 7108 showed mechanical properties were mainly influenced by the ageing process with ductility in the range of 16% to 17.5%. Overall the

work displayed that a combination of hot extrusion and artificial ageing process can result in significant enhancement in strength and ductility.

Owing to their alloying additions, Al 7xxx series possess very high strength but at the expense of ductility which limits its application in certain areas. In order to address such issues the physical metallurgy of these alloys can be tailored by addition to grain refiners based on rare earth elements, following right combination of heat treatments or subjecting them to secondary processing techniques like hot extrusion or hot forging [8,9]. Grain refinement plays an important role in improving the various characteristics of both as cast and wrought Al 7xxx alloys. In particular the grain refinement in Al alloys is mainly by heterogeneous nucleation and by grain growth. Several studies have shown the importance of addition of various grain refiners like Al-10Ti, Al-5Ti-1B, Al-8B to aluminium alloys has influenced the microstructure, mechanical and tribological properties [10-13]. Especially the combination of new processing technique like strain induced melt activation process and grain refiners have enhanced the mechanical properties of Al 7xxx series [14-16]. On the other hand the rare earth elements like La, Sc, Ce and Er are also used for refining grain sizes and modifying the eutectic microstructures. Fang et al. [17] studied the effect of rare earth element like Er on the mechanical and corrosion characteristics of AlZnMgCu alloy. The secondary precipitate like Al_3Er with size of 15-25 nm was found to inhibit the recrystallization contributing in high resistance to stress corrosion and improved fracture toughness. In their work, Liu et al. [18] studied the effect of Sc and Zr addition on hardness and tensile properties of AlZnMgCu alloy sheets fabricated by a combination of hot and cold rolling. With the increase in Sc and Zr addition, the alloy found to exhibit resistance to recrystallization by inhibiting dislocation mobility. Hardness and tensile strength was found to be enhanced due to presence of large volume fraction of precipitates of Al_3Sc and Al_3Zr .

Taking a cue from various works conducted on enhancing the strength keeping ductility in mind we report the development of high strength AlZnMgCu by adding various weight percentage of rare earth element erbium. Attempt is made to achieve refined microstructure and high strength without losing much ductility. The alloy developed using combination of casting and hot

extrusion technique followed by subjecting it to T6 heat treatment conditions. The combination of addition of erbium, hot extrusion and heat treatment on mechanical properties of AlZnMgCu alloy is reported here.

2. EXPERIMENTAL PROCEDURE

In the current study aluminum alloy of chemical composition as mentioned in the Table 1 is prepared by melting pure Al (99.9%), Mg (99.9%), Zn (99.9%) and Cu (99.9%) ingots. These ingots with proper proportion were machined and placed in a graphite crucible for melting in electrical resistance furnace. Melting was carried out at 750°C with addition of Al-Er modifier with the composition of 0, 0.5, 1.0, 1.5 and 2.0 wt% Er respectively. In order to check the microstructure and mechanical properties of the alloy with and without Er in as cast conditions, the melt was poured in standard tensile test bars mold which was fabricated according to ASTM B557M-10 standard. Fig. 1 (a) shows the fabricated mold for making tensile test bars. The design of tensile test bar mold was done in such a way that during pouring of molten metal the turbulence was avoided and air entrapment was reduced. A neat sketch of tensile sample is shown in Fig 1 (b). Extrusion was carried out at 320°C with a ram speed of 1 mm/s using a hydraulic press by adopting an extrusion ratio of 6:1. A simple schematic of extrusion set up with extrusion die and plunger is shown in Fig. 1 (c). Extrusion process was carried out using graphite base oil between metal and dies in order avoid friction. Further heat treatment (T6 conditions) was carried out on as cast and hot extruded Al-15Zn-2.5Mg-2.5Cu alloy samples. The samples were initially heated to 460°C for 8 h followed by water quenching. Finally, the quenched samples were tempered at 120°C for 24 h and were furnace cooled. For microstructural studies, scanning electron microscope (SEM) (Make: Cam Scan MV2300) equipped with electron probe microanalysis was used. For grain size analysis the alloy samples were polished using SiC based abrasive papers and then etched by Keller's reagent (2 ml HF, 3 ml HCl, 5 ml HNO₃ and 190 ml H₂O) to reveal the microstructure. The grain size of the samples was measured in

Table 1. Chemical composition of AlZnMgCu alloy obtained after casting process (wt.%)

Al	Zn	Mg	Cu	Ti	B	Fe	Si
Rem	15.24	2.54	2.46	-	-	0.16	0.03

accordance with the ASTM: E112 standard. Tensile tests on all the samples were performed at room temperature using SANTAM universal testing machine at the strain rate of 1 mm/min. Four test bars were tested for each sample and the average value is reported here.

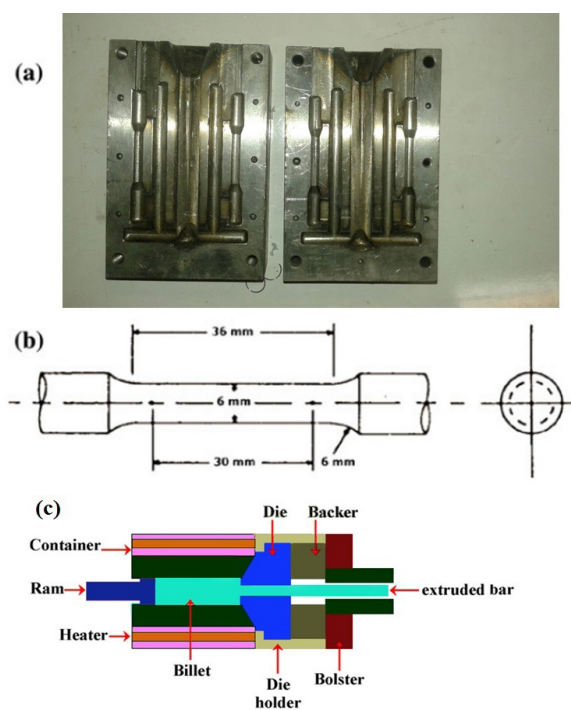


Fig. 1. (a) Cast iron mold for making tensile samples, (b) tensile sample dimensions and (c) schematic diagram of the extrusion set up.

3. RESULTS AND DISCUSSION

3.1. Microstructural Characterization

Fig. 2 shows the SEM micrographs of as cast microstructures of Al-15Zn-2.5Mg-2.5Cu alloy with varying weight percentage of Er (0, 0.5, 1.0, 1.5 and 2.0 wt%). The micrographs clearly show that there is a significant change in dendrite morphology of the Al-15Zn-2.5Mg-2.5Cu alloy after adding Er. The microstructures of alloy revealed a rosette-like microstructure of primary α -Al grains solid solution surrounded by interdendritic secondary phases as shown in Fig. 2 (a). Alloy without Er

addition displayed showed coarser morphology. It is observed from SEM micrographs shown in Fig. 2 (b) to (d) that after adding Er to the alloy, grain boundary volume is found to increase which also promotes a more homogeneous distribution of intermetallic precipitates. The effect of Er is such that there is a change in the shape and size of the eutectic phase. The SEM micrographs shown in Fig. 2 (b) to (d) explain that the nucleation of α -Al grains help enriching the growth front with the Er element. Due to this local compositional undercooling takes place which further stimulate the nucleation of α -Al grains leading to grain refinement of the alloy. However the grain refinement in the alloy after addition of 1, 1.5 and 2 wt% Er was found to be same which is mainly attributed to attaining the saturation point of enrichment of Er to the growth front. This is due to the aggregation of Al-Er phases which causes the weakening in enrichment of Er in the growth fronts [19,20].

Fig. 3 shows SEM micrograph and electron probe microanalysis of 1 wt.% Er refined Al-15Zn-2.5Mg-2.5Cu as cast alloy. The various phases were clearly seen from the micrographs. In addition to this, the electron probe microanalysis carried out on this alloy showed the presence of Al_3Er intermetallic which indicates that Al and Er contents are higher than the average level of chemical composition in the alloy. Fig. 4 shows SEM micrograph of 0.5 wt.% Er, 1 wt.% Er and 2 wt.% Er refined Al-15Zn-2.5Mg-2.5Cu as cast alloy after T6 heat treatment. It is obvious that the addition of Er changes the shape and size of the precipitates. In order to achieve a desirable combination of microstructure and mechanical properties, heat treatments are widely applied to 7xxx series alloy. As is well known, the precipitation sequence for 7xxx alloys is as follows: supersaturated solid solution (α) to GP zone to metastable η' to stable η ($MgZn_2$) phases. In general, GP zone is atom clustering zone and is coherent with α -Al matrix. The η' phase is a metastable hexagonal phase and is semi-coherent with α -Al matrix. The η phase is equilibrium phase and is non-coherent with α -Al matrix. In these three types of precipitates, the GP zones and metastable η'

(MgZn₂) phase are the main aging-hardening phase in commercial Al–Zn–Mg–Cu alloys.

Fig. 5 shows the influence of various amounts of Er on the grain size of hot extruded Al–15Zn–2.5Mg–2.5Cu alloy after T6 heat treatment. The average grain size of Al–15Zn–2.5Mg–2.5Cu alloy without Er addition was found to be 550 μm. The alloy with Er addition was found to be significantly refined by changing the coarse columnar primary α-Al grains to fine equi-axed α-Al grains of average size of 65 μm. The main reason is that Al₃Er particles act as nucleating agents during the solidification of α-Al grains. It can be clearly seen from Fig. 2 that the microstructure of alloy with Er addition shows the presence of Al₃Er intermetallic phase. The Al₃Er phase is uniformly distributed in the α-Al matrix. Several mechanisms have been proposed for the grain refining process. In some mechanisms the presence of some particles like Al₃Er are known to be effective for grain refinement procedure. The alloying elements such as Mg and Cu are known to improve the efficiency of Al–30Er master alloy. With increasing content of Er wt.%, these particles pin the grain boundaries, increase the grain refinement and cause increase of strength. When wt.% of Er is more than 1 wt.%, the grain size is found out to be constant and effect of grain refinement can be said to have reached saturation point.

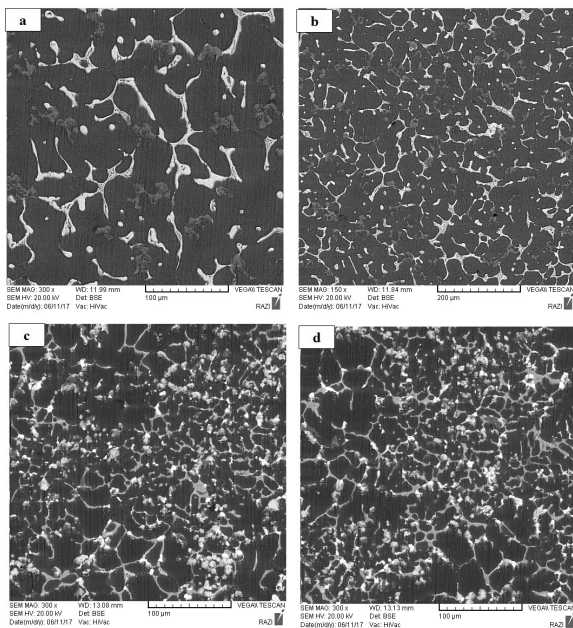


Fig. 2. SEM micrographs of as cast AlZnMgCu alloy with (a) 0.0 wt.%, (b) 0.5 wt.%, (c) 1 wt.% and (d) 2 wt.% Er.

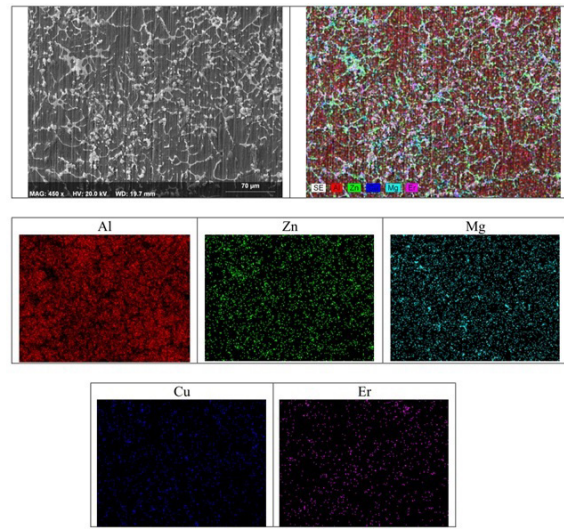


Fig. 3. Electron probe microanalysis of the AlZnMgCu alloy with 1% Er and distribution of the major elements.

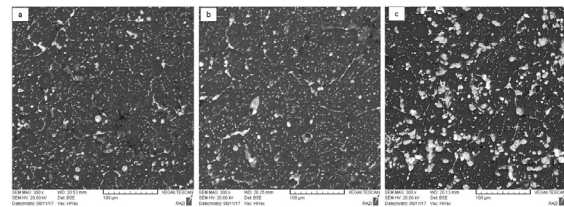


Fig. 4. SEM micrographs of AlZnMgCu alloys with (a) 0.5 wt.% Er, (b) 1 wt.% Er and (c) 2 wt.% Er after T6 heat treatment.

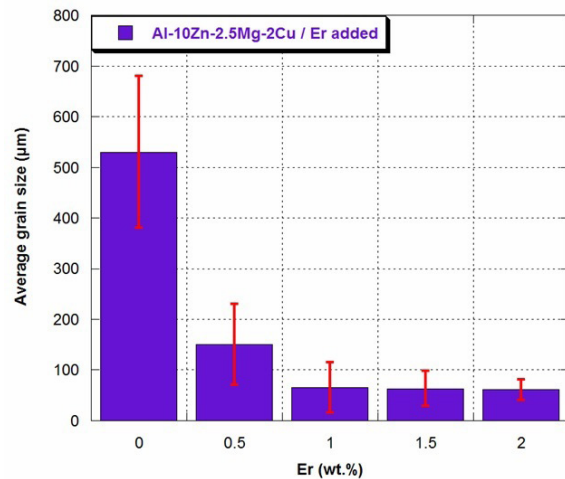


Fig. 5. Grain size of as hot extruded AlZnMgCu alloy with different weight percentage of Er after T6 heat treatment.

3.2. Tensile Properties

Fig. 6 and 7 shows the mechanical properties such as tensile strength and ductility of Al–15Zn–2.5Mg–2.5Cu alloy processed under different

conditions such as cast, extruded and their respective heat treated counterpart samples. As it is shown in Fig. 6, the average ultimate tensile strength (UTS) of the specimens before and after extrusion processes and T6 heat treatment increases from 225 ± 12 MPa to about 602 ± 10 MPa. It is well known that the tensile strength of the Al-15Zn-2.5Mg-2.5Cu-xEr alloy mainly depend on the shape, size and size distribution of the α -Al grains, eutectic structure and the distribution of the intermetallics in the interdendritic or grains [21]. Ultimate strength of as-cast alloys has a low value which is due to the presence of shrinkage porosities inside the grains and boundaries. After subjecting the cast alloy to extrusion and T6 heat treatment process, the tensile strength values of alloys were found to be significantly enhanced than those of as cast and without heat treatment. The main reason for improvement in strength of extruded and heat treated alloys is reduced grain size. It is well known that according to Hall-Petch theory the finer the grains, the higher the strength [22]. But, due to the several mechanisms engaging in strengthening of Al 7xxx alloys, especially precipitation hardening, the dependence of the strength to grain size was unclear. Intermetallic compounds are brittle and considered as important crack initiating sites during loading. The second strengthening mechanism for increment in strength can be attributed to Orowan looping. Several reports indicate that metastable coherent or semicoherent η precipitates are formed during aging treatment [23, 24]. The presence of second phase particles often causes lattice distortions. These lattice distortions result when the precipitate particles differ in size and crystallographic structure from the host atoms. Smaller precipitate particles in a host lattice leads to a tensile stress, whereas larger precipitate particles leads to a compressive stress. Dislocation defects also create a stress field. Above the dislocation there is a compressive stress and below there is a tensile stress. Consequently, there is negative interaction energy between a dislocation and precipitates that each respectively causes compressive and tensile stress or vice versa. In other words, the dislocation will be attracted to the precipitate. In addition, there is positive interaction energy

between a dislocation and a precipitate that have the same type of stress field. This means that the dislocation will be repulsed by the precipitate. In Fig. 4 (a) precipitate particles are smaller and are finely distributed in α -Al matrix. When the precipitate size is small, the required shear stress $\{\displaystyle \tau\}$ is proportional to the precipitate size $\{\displaystyle r^{1/2}\}$. However, for a fixed particle volume fraction, this stress may decrease at larger values of owing to an increase in particle spacing. The overall level of the curve is raised by increases in either inherent particle strength or particle volume fraction. The dislocation can also bow around a precipitate particle through so-called Orowan mechanism. On the other hand Al-15Zn-2.5Mg-2.5Cu alloy is strengthened by precipitation of Al_3Er particles after addition of Al-30 wt.% Er master alloy. The micro or nano size Al_3Er particles play a crucial role in the strengthening mechanism. Therefore, the ultimate tensile strength of alloys increased significantly with addition of Er. Overall the increment in strength cannot be restricted to anyone strengthening mechanism but it is combination grain strengthening, precipitation strengthening and dispersion strengthening.

Fig. 7 shows the elongation of AlZnMgCu alloy processed under different conditions. The elongation before fracture has the maximum value for alloys which have undergone both after extrusion and heat treatment processes. For instance the alloy without Er was found to have highest elongation when compared to that of alloys with Er addition. However the drop in the elongation value after Er addition is not significant as the elongation is about $\sim 8\%$ which is very close that of as cast alloy.

3. 3. Fractography Of Tensile Specimens

Fig. 8 presents the fracture surfaces of the AlZnMgCu alloys processed in different conditions. It can be clearly seen that extensive irregular cleavage planes and some tearing ridges are apparent on the entire fracture surface of the unmodified alloy. It indicates that the fracture characteristics exhibit quasi-cleavage fracture, resulting in low strength value of the specimen.

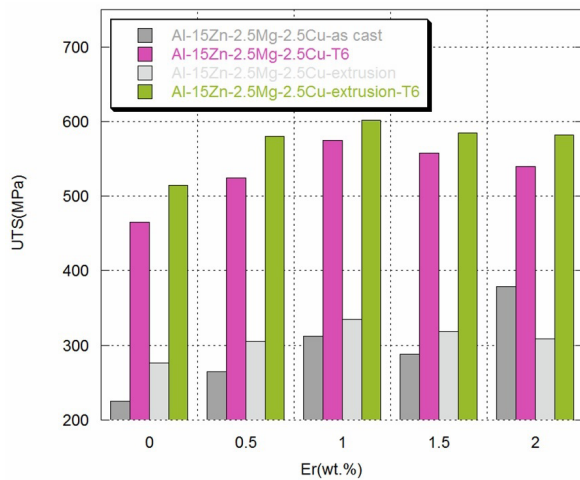


Fig. 6. Ultimate tensile strength of AlZnMgCu alloy with and without Er addition at the different processing conditions.

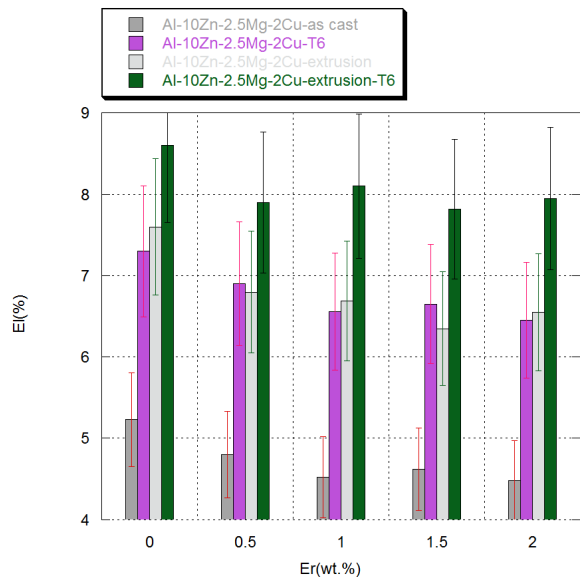


Fig. 7. Ductility of AlZnMgCu alloy with and without Er addition at the different processing conditions.

Due to the modification of Er on the eutectic, the area of the cleavage planes decreases and the number of dimples increases as shown in SEM micrograph in Fig. 8 (b). Moreover, the modification efficiency enhances with the addition of 1 wt.% Er in the alloy, as a result this alloy exhibits more dimples and higher strength, comparing with that of other Er modified alloy. From Fig. 8 (d) it can be clearly seen that the alloy after hot extrusion and subsequent heat treatment displayed fine dimples which are uniformly distributed over entire fracture surface. These fine dimples formed on the surface

of grain facets can be formed due to slip bands or intermetallic constituent particles. This clearly indicates that the failure in this alloy is mainly ductile in nature.

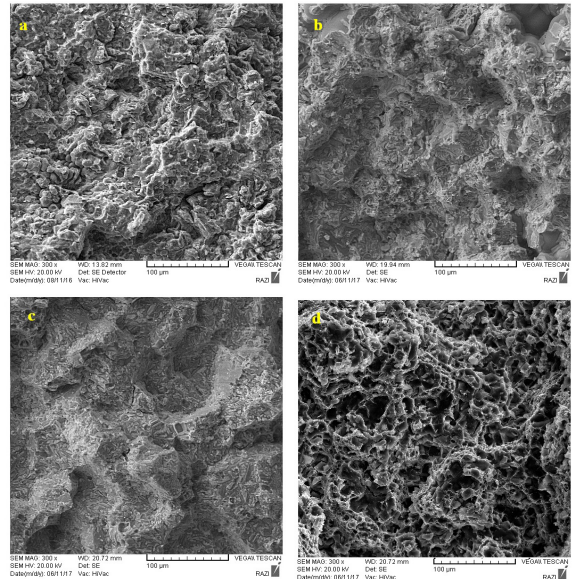


Fig. 8. Fractographs of the tensile samples from alloys: (a) unmodified-as cast, (b) 1 wt.% Er, (c) 1 wt.% Er after T6 heat treatment and (d) 1 wt.% Er after T6 heat treatment and extrusion process.

4. CONCLUSIONS

The following conclusions can be drawn from this study.

1. A new alloy was developed by adding different weight percentage of Er to AlZnMgCu by following liquid metallurgy and hot extrusion process followed by T6 heat treatment.
2. Developed alloys displayed significant changes in the microstructure after addition of Er. The microstructure after Er addition showed grain refinement due to the formation of nucleation of α -Al facilitated by Er enrichment at the growth front.
3. Ultimate tensile strength was found to be maximum in the hot extruded and heat treated alloy with 1 wt.% Er addition while the lowest strength was observed for as cast alloy without Er and heat treatment.
4. Elongation was found to have relatively similar values for all alloys with and without Er addition in extruded and heat treated conditions.

5. Fracture surface of hot extruded alloy with erbium addition after heat treatment showed uniformly distributed deep dimples exhibiting ductile behavior.

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