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Effect of cerium addition on microstructure and mechanical properties of Al-Zn-Mg-Cu alloy

A.K. Chaubey[1], S. Mohapatra[1], K.Jayasankar[1], S.K. Pradhan[1], B. Satpati[1], S.S. Sahay[2], B.K. Mishra[1] and P.S. Mukherjee[1]

[1] *Institute of Minerals and Materials Technology (IMMT), Bhubaneswar-751013, India*
[2] *Tata Research Development and Design Centre, A division of Tata Consultancy Services, Pune -411013, India*

E-mail : anil_chaubey@yahoo.com

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Abstract
Effect of cerium addition on the microstructure and mechanical properties of an Al-Zn-Mg-Cu alloy have been investigated. In this study, aluminum alloys with up to 0.4% cerium content have been prepared by melting, metal mould casting followed by thermo-mechanical processing. The alloys were extensively characterized by optical and transmission electron microscopy, followed by mechanical property examination by tensile tests as well as nanoindentation tests. It was observed that cerium addition results in up to 5% grain refinement of the cast dendritic structure as well as up to 38% refinement of the heat treated microstructure. Transmission electron microscopy (TEM) has revealed the uniform distribution of fine GP zones and some semi coherent $\beta(MgZn_2)$ precipitates in the Al rich matrix. Further TEM results show that when the Ce content was changed from 0.1% to 0.4%, precipitate size increased from 5 to 50nm and the precipitate morphology changed from spherical to needle shape. Evaluation of mechanical properties through tensile and nano-indentation tests have exhibited that both Young's modulus and tensile strength increases with Ce addition up to 0.3% and subsequently decrease.

1. Introduction

In pursuit of developing high strength aluminum alloys, microalloying of rare earth elements have shown remarkable effect in terms of refining as cast structure, retarding recrystallisation and refining precipitate phases [1-5]. Rare earth Cerium forms various eutectics in Al-based multi-component system [6], which makes it a promising element for developing a new group of Al alloys instead of traditional casting alloys. The use of rare earth as micro-alloying element in Al has been studied for several years. It was reported that microalloying Ce decreases the as-cast grain size, increases the nucleation ratio, and decreases the growth speed of the precipitates during artificial aging [7].It has also been reported that Ce affected the grain size of a binary Al-Li alloy and the mechanical properties of Al-Li-Mg casting alloys containing impurity Fe. The number of grains per unit area in the binary alloy linearly increases with raising the Ce content up to 1.1% (mass fraction), and the mechanical properties of the Al-Li-Mg casting alloys were improved [8]. The addition of Ce was also reported to affect the ductility and fracture, and the negative effect of impurity Fe was controlled by Ce addition. The Ce addition can toughen 8090 alloy sheets rich in impurities of Fe, Si and alkali metals [9].

Zhihui Li et.al have investigated the microstructural evolution and mechanical properties of an Al-Zn-Mg-Cu alloy (pre-stretched) thick plate during two stage ageing treatment at 115ºC and 160ºC and have shown that the first stage ageing at 115ºC gives dominant formation of GPI and GPII zones, and promotes major increment in the tensile strength of the alloy and during second stage ageing, initial increase in strength gives rise to a peak after a relative short period which then decreases gradually [10]. But there is little information available for the effect of rare earth Ce addition on precipitation hardening and strengthening in advanced 7075 aluminium alloy. The present work was thus to examine such effect with a view of establishing the potential for enhancing the mechanical properties of these alloys.

2. Experimental

The alloys were prepared by liquid metal casting technique by melting high purity 99.7% aluminum ingot, industrially pure 99.9% Zn, 99% Cu, 99.9% Mg, and ultra-pure 99.99% Ce powder. Pit-type electrical resistance furnace was used to melt the alloys at 780ºC and the melt was cast in a 100 mm cube steel mould. The chemical composition of the cast alloys as determined by wet chemical method are shown in Table 1.

The as-cast ingots were homogenized at 450ºC for 4 hour, followed by hot forging on 3.15 tonne hammer in the temperature range of 350 – 410ºC. The hot working ratio was

Table 1 : Chemical composition of alloys (mass fraction, %)

Alloy No.	*Cu*	*Mg*	*Zn*	*Fe*	*Ce*	*Al Balance*
1	2.35	2.1	8.25	0.14	0.10	
2	2.34	2.4	8.18	0.13	0.20	
3	2.45	2.45	8.15	0.10	0.30	
4	2.25	2.18	8.3	0.12	0.40	

kept around 9.00. The forged ingots were solution treated at 470^{o}C for 2 hour followed by double aging at 107^{o}C (6 hour) and 177^{o}C (6 hour) and subsequently air cooling. The samples for metallographic examination were polished and observed in under an optical microscope (NIKON- LV-150). The TEM investigations were carried out on samples prepared by mechanical thinning and followed with Ar-ion milling. The thinned samples were observed under FEI microscope (TECNAI G^2 20, TWIN) operating at 200 kV, equipped with a GATAN CCD camera as well as an EDS attachment for Elemental analysis. Phase analysis of alloys was carried out on DMAX3C rotary-target X-ray diffractometer. Nanoindentation tests were performed in a UMIS system (Fisher-Cripps, Australia) with a diamond Berkovich indenter at 10 mN load using the Oliver-Pharr method [11]. The load-depth data were analyzed to derive the Young's modulus and hardness values. For calculation purpose, the Poisson's ratio of aluminum was taken as 0.33. Tensile testing was carried out on 40T Universal Testing Machine (Instron).

3. Results and discussion

3.1 Optical microscopy

Figure 1 shows the as cast optical microstructure of the alloys. The average grain size of the alloy 1, 2, 3 and 4 are about 24, 15, 10 and 8μm respectively. It can be seen that addition of cerium obviously refines the grain size of the alloys. Segregation along the grain boundary increases with cerium addition and segregated phase is rich in cerium has been found by EDX analysis. Figure 2 shows the microstructure of the homogenized and age-hardened alloys. Segregation along the grain boundary breaks after forging and subsequent aging operation and the grain size further reduced with Ce addition and the minimum grain size was observed for 0.4% Ce alloy with an average grain size of 6.8μm. These observations are consistent with the earlier work of Song Min *et al* [12].

3.2 Transmission electron microscopy

The present study shows that the addition of Ce affected the microstructure development in the Al-Zn-Mg–Cu alloys in various ways. Bright field TEM micrographs of the alloys after forging and double aging at 107^{o}C (6 hour) and 177^{o}C (6 hour) is shown in Fig. 3. These micrographs show that the alloy has small, closely-spaced grain-boundary precipitates (η, $MgZn_2$) and a matrix consisting primary of very fine GP zones and semi-coherent η' ($MgZn_2$) precipitates (20 to 50 nm) distributed homogeneously. The work of Park *et al.* [13]

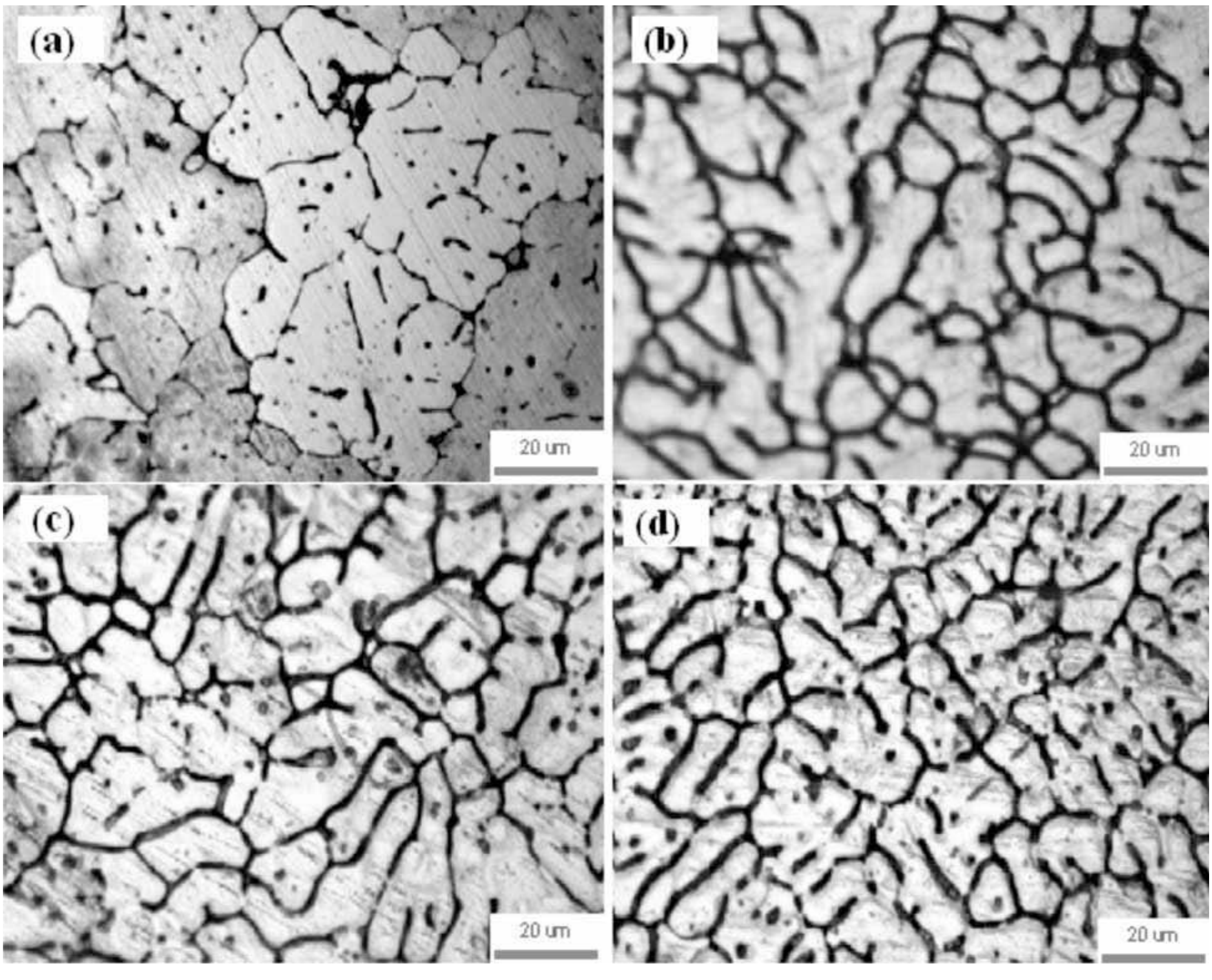

Fig. 1 : Optical Micrograph of the as cast alloys (a) alloy1 (b) alloy 2 (c) alloy 3 (d) alloy 4.

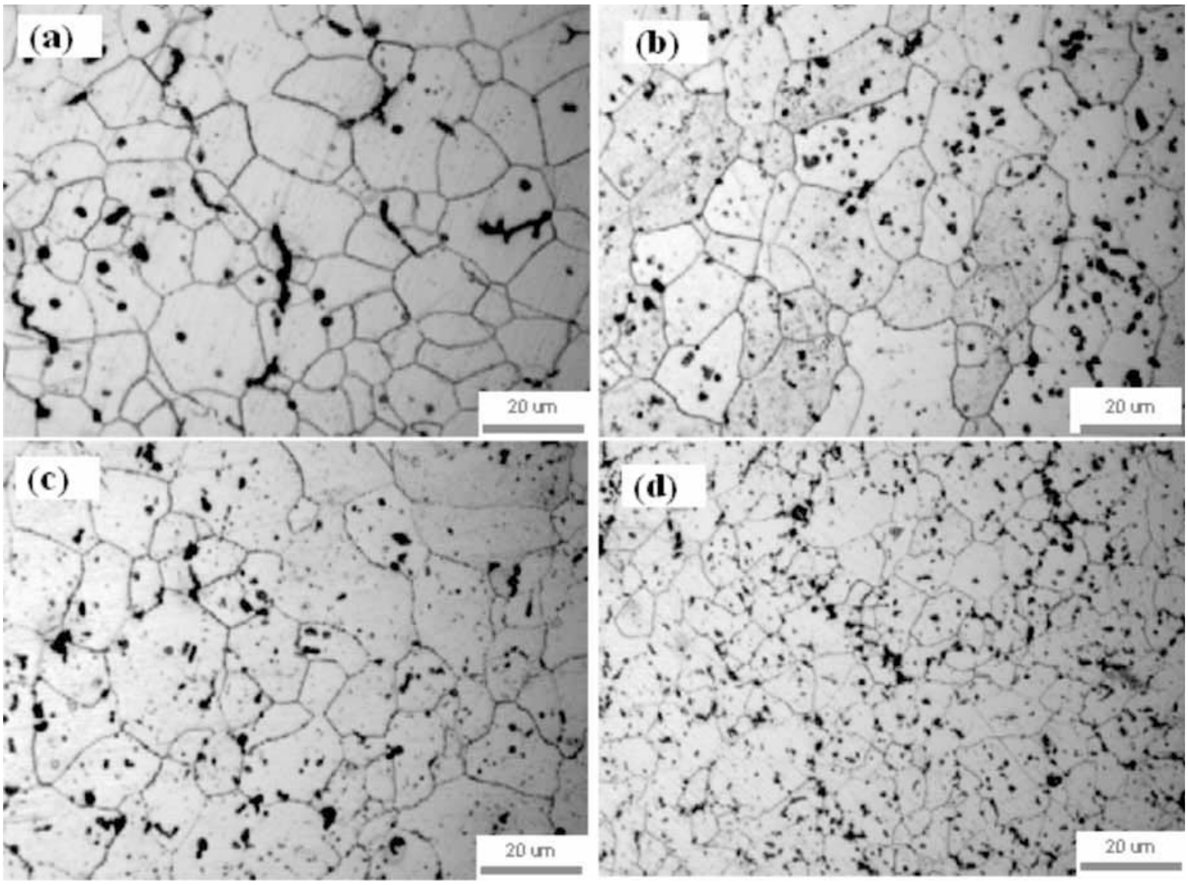

Fig. 2 : Optical Micrograph of the alloys after homogenization and age-hardening (a) alloy1 (b) alloy 2 (c) alloy 3 (d) alloy 4.

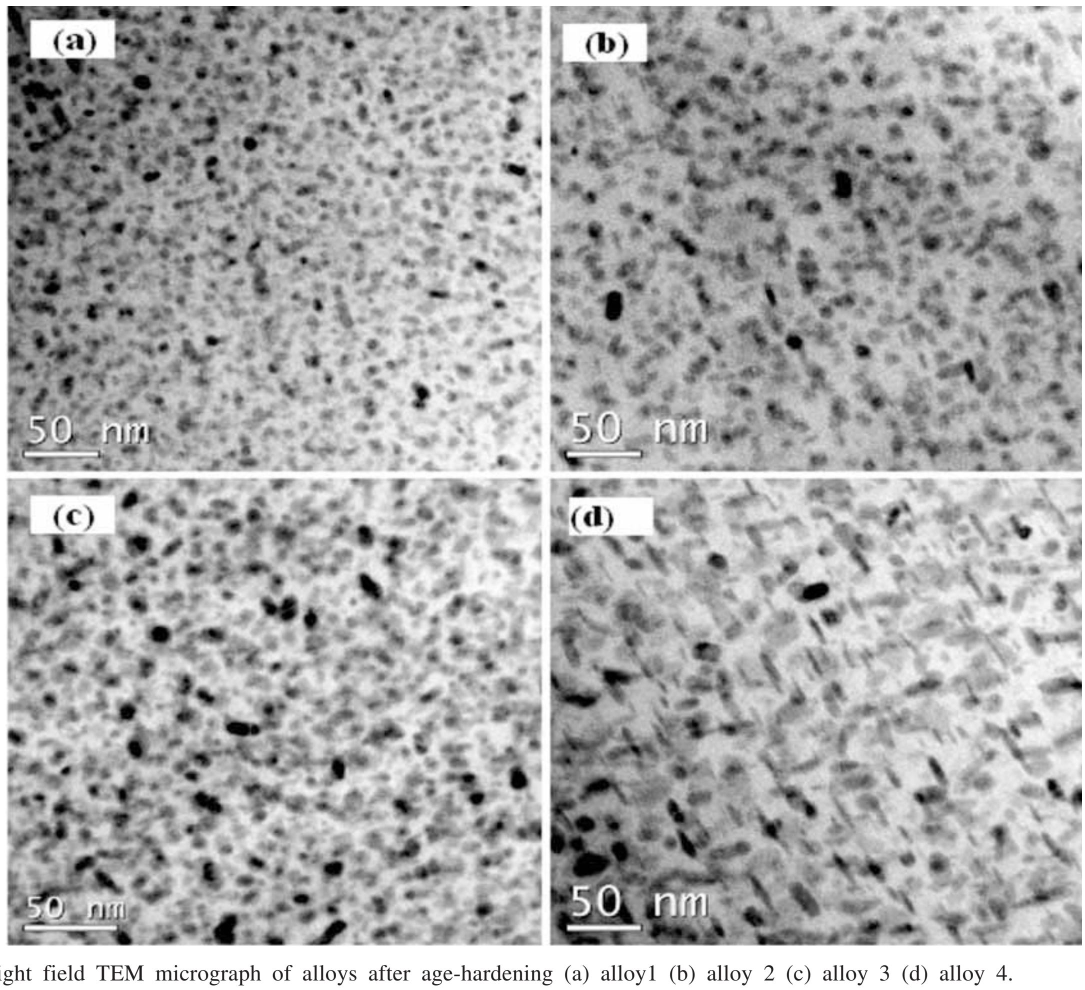

Fig. 3 : Bright field TEM micrograph of alloys after age-hardening (a) alloy1 (b) alloy 2 (c) alloy 3 (d) alloy 4.

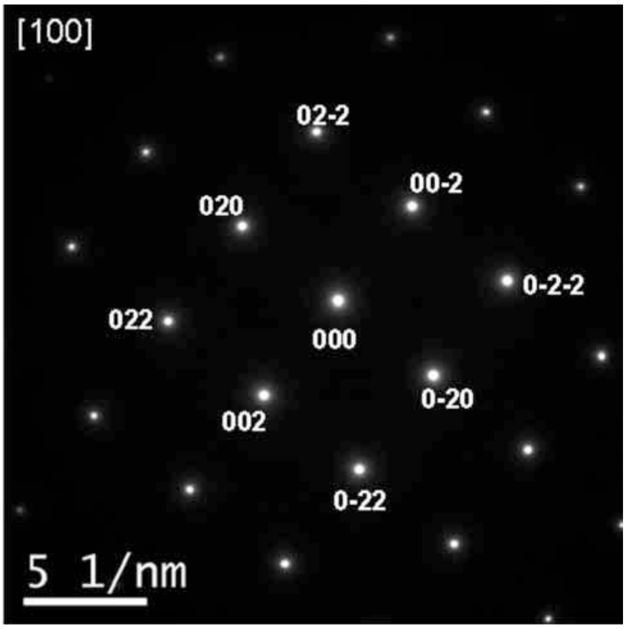

Fig. 4 : SAED pattern of the image shown in fig.4 (d).

shows that η′ and η phases have a platelet or rod-like shape, their size are 5-15 nm and 15-30 nm respectively. As shown in Fig.3 (a), the 0.1% Ce alloy comprises very fine (5-10 nm) closely spaced and nearly round precipitates, which increases to 10-20 nm size and reduced number density, when the Ce content is increased to 0.2% (Fig.3 (b)). The morphology of these precipitates changes to thick needle shape, when the Ce content is added to 0.3% (Fig.3(c)) and 0.4% (Fig.3 (d) 40-50-nm). This can be attributed to the retardation of GP zone and enhancement of precipitate formation by Ce addition. The SAED pattern shown in the Fig.4 taken from the sample with 0.4% Ce shows that needle like precipitates are mostly oriented in (200) direction. In Fig. 5(a) and 5(b) the alloy having 0.1% Ce and 0.2% Ce, the grain boundary exhibit very fine and almost continuously distributed precipitates, without any clear precipitation free zone (PFZ). But when the Ce content is increased to 0.3% (Fig. 5(c)) and 0.4% (Fig. 5(d)) formation of a narrow PFZ in regions adjacent to grain boundaries and local precipitation of small grain boundary particles are visible along with coarse (100–200 nm) $MgZn_2$ precipitates. In case of specimen with 0.4% Ce, low density elongated grain boundary precipitate can also be observed.

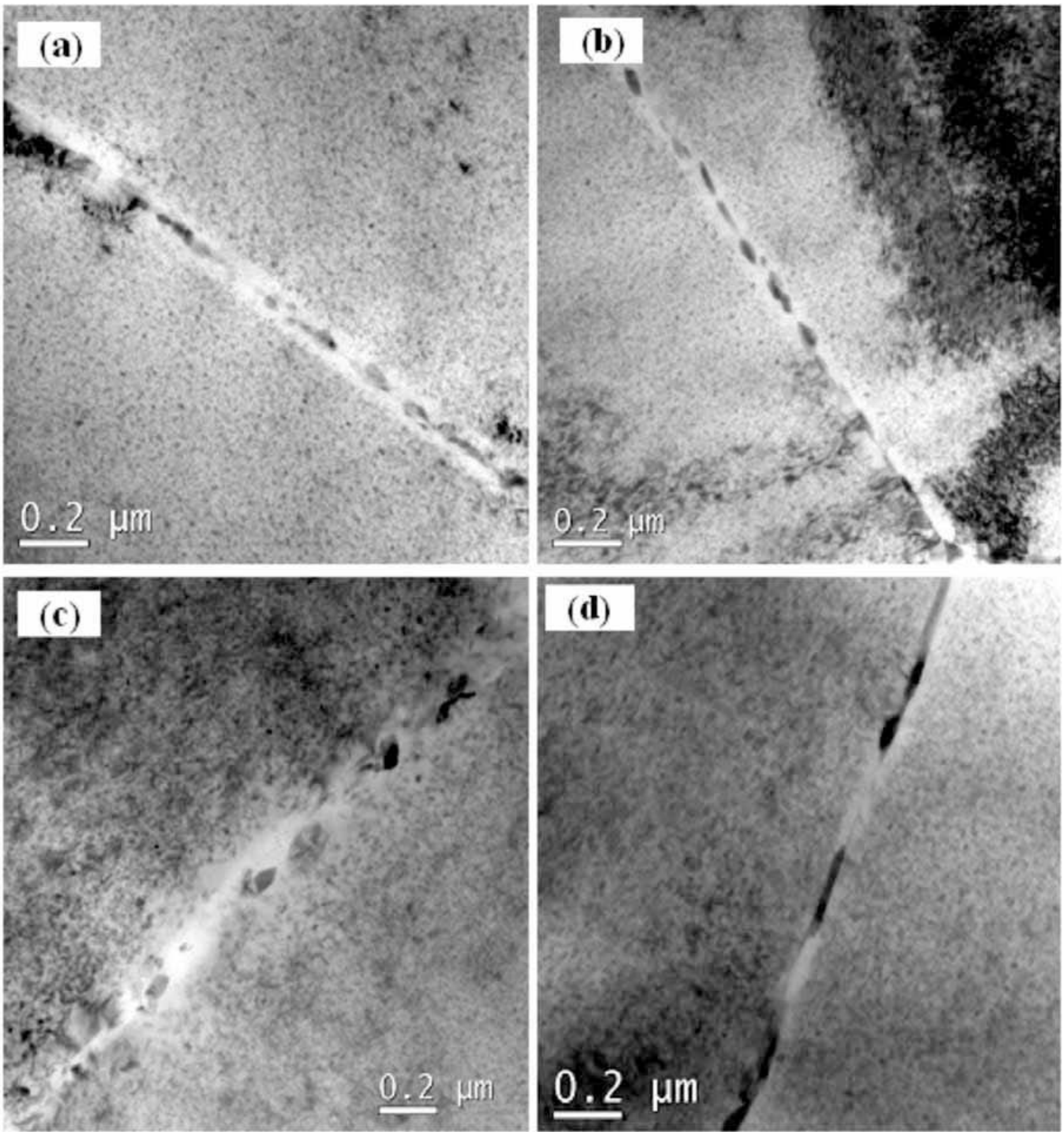

Fig. 5 : Bright field TEM micrograph showing grain boundary in the alloys (a) alloy1 (b) alloy 2 (c) alloy 3 (d) alloy 4.

3.3 Mechanical properties

3.3.1 Tensile strength

The room temperature tensile tests were carried out on the forged and aged alloys to determine ultimate tensile and yield strengths as well as ductility. Fig.6 shows that tensile and yield strength increases with increase in Ce content. For example, the tensile strength increases from 506 to 559 MPa, when 0.3% Ce is added in the aluminum alloy. Further addition of Ce results in saturation of tensile and yield strengths. Earlier research has shown that Al-RE dispersions can effectively restrain the dislocation slipping and strengthen the alloy at high temperature [14]. As evident from TEM studies, in the present work the homogeneously distributed (η, $MgZn_2$) precipitate size increases with increase of cerium percentage and shape also gradually converted from spherical to needle. The coarsening of the precipitate strengthens the alloy by straining the matrix and these coarse precipitates block the dislocation movement and pinning sites are increased, resulting in increased tensile strength. In the present work, 0.3 % Ce seems to be the optimum content providing maximum strength in the investigated aluminum alloy. The yield strength exhibited similar behavior as the ultimate tensile stress, whereas marginal decrease in %elongation was observed with Ce content. The sharp deflection at 0.3% Ce was consistently observed in yield and tensile strength plots as well as in the % elongation behavior.

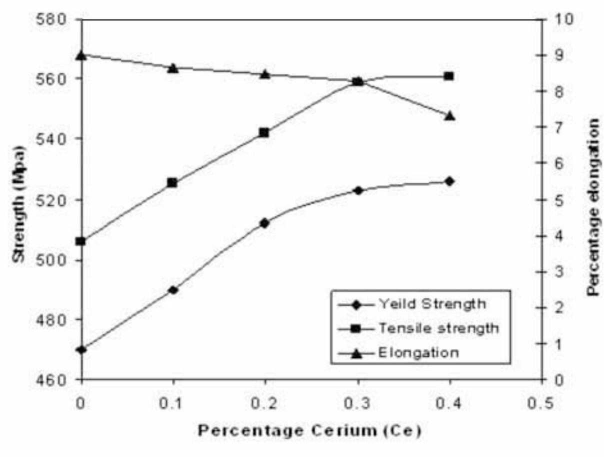

Fig. 6 : Mechanical property of the alloys with variation of Ce percentage.

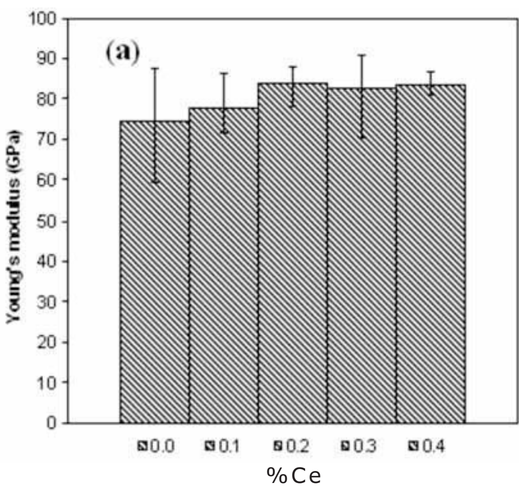

Fig. 7 : Variation of Young's Modulus with Ce addition obtained from nanoindentation tests.

3.3.2 Nanoindentation tests

Nanoindentation test for alloys with different Ce contents was carried out with a diamond Berkovich indenter at 10 mN load using the Oliver-Pharr method. The Young's modulus was calculated from the load–depth plot and presented in Fig. 7. As can be observed in Fig.7, the Young Modulus marginally increases with increase in Ce, reaches a maximum at 0.2% Ce and then marginally decreases. This behavior is similar to variation in tensile strength with very close optimum value (0.3% for tensile strength versus 0.2% for Young's modulus). It must be noted that grain size, dislocation and precipitates are not expected to influence the Young's modulus. The change in Young's modulus with Ce addition is expected to be affected by the stretching of atomic bonds of aluminum lattice. M. Zhang et. al [15] have reported that Ce addition decreases the tendency of reduction of the elastic modulus of 2090 alloys.

4. Conclusions

In the present work, the effect of Ce addition in Al-Zn-Mg-Cu alloy has been rigorously investigated through optical and transmission electron microscopy as well as mechanical properties evaluation through tensile and nano-indentation test. The effectiveness of minor addition of Ce in refining the dendritic cast structure has been demonstrated. In TEM study it was observed that matrix consists of closely spaced GP zone and fine grain boundary precipitates. The precipitates shape changes from spherical to needle and size (5-50nm) with increase of cerium (0.1 to 0.4%). Around 10% improvement in tensile strength was noted with 0.3% Ce addition, which saturated with further Ce addition. Similar behavior was observed for the Young's modulus measured through nanoindentation tests.

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