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**Review Article** 

# Achieving dispersed fine soft Bi particles and grain refinement in a hypermonotectic Al–Bi alloy by severe plastic deformation and annealing



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#### ARTICLE INFO

Article history: Received 24 February 2018 Received in revised form 11 June 2018 Accepted 11 June 2018 Available online xxxx

Keywords: ECAP Particle dispersion Al-Bi alloys Microstructure Recrystallization

#### ABSTRACT

The present work revealed that equal-channel angular pressing (ECAP) deformation combined with appropriate annealing could represent as an efficient route to modify immiscible as-cast microstructure of a monotectic Al-8Bi alloy by a complete redistribution of immiscible Bi particles and grain refinement of alloy matrix. Especially, we found that the mean size of Bi particles in the 4-pass ECAPed Al–8Bi sample decreases after annealing at 200 °C for 8 h, due to inverse coarsening driven by the reduction of elastic energy and interfacial energy induced by morphology change and spatial distribution of Bi particles.

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Hypermonotectic Al–X (Pb/Bi/In) alloys consisting of soft particles have been considered as candidate bearing materials for a long time [1]. However, since phase separation occurred in the liquid state, and as large density differences between Al and soft phase X [2], it is difficult to obtain a homogeneous distribution of the minority phase by conventional casting methods [3–7].

During deformation, as-cast alloys containing heterogeneous distributions of particles may undergo redistribution of reinforcing particles along with microstructure refinement of the matrix [8]. Recently, modifying the distribution of constituent particles via shearing and/or fragmentation by severe plastic deformation (SPD) such as equal-channel angular pressing (ECAP) has received increased attention [9]. For example, ECAP has been demonstrated to affect significantly the morphology and distribution of hard coarse Si + Al<sub>5</sub>FeSi- [8], Al<sub>2</sub>O<sub>3</sub>- [10] particles and rod-like MgZn<sub>2</sub> precipitates [11] in Al-matrix composites. In addition, our previous work revealed that the size, shape and distribution of soft Bi particles could be substantially modified via shearing,

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fragmentation, coalescence and ripening of particles by room temperature ECAP [12].

Nevertheless, the major challenge in the application of Al-matrix composites and alloys containing non-deformable particles is their limited ductility and formability, as cracks and voids tend to form and grow around the matrix-particle interface, which will normally lead to decreased ductility and fracture toughness [13]. Note that almost all available literature has been concentrated on ductile Al matrix strengthened by either hard coarse particles or fine dispersoids. In contrast, to the best of the authors' knowledge, seldom has any effort been devoted into Al alloys containing soft particles upon deformation, although previous work [12, 14] revealed that soft Bi particles have a similar effect to that of the hard particles, e.g. Al<sub>13</sub>Fe<sub>4</sub>, in promoting grain refinement of Al matrix [15].

Furthermore, a normal coarsening process of constituent particles was often observed during heat treatment [16], which is driven by a decrease in particle-matrix interface area and a reduction in interface free energy [17, 18]. However, as reported by *Voorhees* et al. [19], inverse coarsening can be stimulated by elastic strain energy associated with misfitting particles, where small particles grow at the expanse of large ones. Currently, inverse coarsening in solid state has been only verified by computational simulation [19, 20], seldom any observations of inverse coarsening of particles in a metal matrix have been reported in literature.



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The present work is initiated to investigate the microstructure evolution of a hypermonotectic Al—8Bi alloy containing soft Bi particles subjected to room temperature ECAP and subsequent annealing. Especially, the main effort has been directed towards elucidating the combined influence of ECAP and annealing on the size and dispersion of Bi particles. The present work reveals that ECAP deformation at low to moderate strains (<~4) combined with an appropriate annealing at moderate temperatures (~200 °C) is a potential approach to prepare monotectic Al–X (Pb, Bi, In) alloys with well-dispersed fine soft second phase particles.

Materials used for the present work were produced by melting 99.999 wt% purity Al and Bi in a fiberfax coated clay-graphite crucible to a composition of Al–8Bi (wt%), and cast in an insulated, bottom-chilled Cu mold with a diameter of 30 mm. Samples were machined to bars with dimensions of  $19.5 \times 19.5 \times 50$  mm<sup>3</sup>. The bars were deformed in a 90° ECAP die giving a strain  $\varepsilon = 1$  for each pass, employing the B<sub>c</sub> route at ambient temperatures [21]. A thin layer of a graphite lubricant was covered in the samples to lower friction during pressing. Post-ECAP annealing was performed on the samples subjected to 4 passes ECAP using oil furnaces. The temperature was controlled within  $\pm 5$  °C in the furnaces. Immediately after annealing, the samples were water-quenched to room temperature.

Samples for hardness measurement and microstructure observations were cut from longitudinal sections in centre regions of the ECAP bars, and prepared by standard metallographic techniques followed by electro polishing and ion milling, employing established procedures as given in ref. [12, 22]. Microstructural studies were carried out in a Zeiss 55VP FEG-SEM equipped with a Nordif electron backscatter diffraction (EBSD) detector. EBSD characterization was performed with 20 kV acceleration voltage, 20 mm working distance, 70° tilt, and with 0.05–0.2  $\mu$ m scan steps. TSL OIM software [23] was used for the analysis of the EBSD images. Diameters of Bi particles are measured using the Image J software [24] on 4 BSE images at 500× magnification for each sample.

Transmission electron microscopy (TEM) observations were carried out using a JEOL 2100F operating at 200 kV. The specimens for TEM investigation were cut from the center of ECAPed billets in ED-ND section. TEM foils were prepared by twin-jet electropolishing in a solution of 33% nitric acid in methanol at -30 °C.

Vickers micro hardness measurements were performed under a 500 g load applied for 15 s. At least six separate measurements were conducted for each condition.

The as-cast Al-8Bi alloy has a coarse elongated heterogeneous microstructure with grain size of 100-500 µm and the second phase Bi particles distribute quite inhomogeneously (Fig. S1(a) and (b)). After 1 pass ECAP, the original grains become elongated and split into volumes and deformation bands (DBs) of various orientations (Fig. 1(a)), where sub boundaries are frequently observed. In addition, some discontinuous short HABs segments distributed inhomogeneously among sub-boundaries together with a few equiaxed sub grains formed locally (Fig. 1(b)), which indicates a grain refinement mechanism associated with the operation of continuous dynamic recrystallization (CDRX). After 2 passes ECAP, extensive microshear bands formed in the coarser DBs further divide the original coarse grains (Fig. 1(c)). Also, more new HABs are seen located around coarser Bi particles (Fig. 1(c)), which become more visible in the 4 pass sample where more equiaxed (sub) grains become predominated (Fig. 1(d)).



**Fig. 1.** EBSD maps taken from the longitudinal section of (a) 1 pass and (b) is the high magnification of (a); (c) 2 passes and (d) 4 passes Al–8Bi alloy, where narrow gray and coarse black lines depict differences between neighboring grid points  $2^{\circ} < \theta < 15^{\circ}$  and  $15^{\circ} < \theta < 180^{\circ}$ , respectively. Inserted in (d) is the color code for the orientation maps.

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