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Enhanced ductility in an Al–Mg2Si *in situ* composite processed by ECAP using a modified B_C route

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ABSTRACT

A modified B_C route, noted as $B_{C\text{-}m}$, was designed and employed to process an Al–Mg_2Si composite in an effort to improve the ductility of the composite. The modification was implemented to improve the effectiveness of particle redistribution of the conventional B_C route while maintaining its high grain refinement efficiency. The experimental results demonstrated that route $B_{C\text{-}m}$ was strongly effective in redistributing particles while maintaining a comparable microstructural refinement efficiency relative to route B_C, which led to an increase in ultimate tensile strength and much higher ductility in the composite. Additionally, the use of route B_C resulted in higher yield strength because of its stronger work-hardening effect compared to route $B_{C\text{-}m}$.

1. Introduction

In situ Al–Mg_2Si composites, as a new class of ultra-light materials for aerospace and other advanced engineering applications, are attracting the attention of increasingly more material scientists and design engineers [1]. However, practical engineering applications of these composites as structural components have been greatly limited owing to their low ductility at room temperature. The large Mg_2Si particle size, the brittle Al–Mg_2Si eutectic matrix [2,3] and the inhomogeneous distribution of Mg_2Si in the composites produced by the ingot metallurgy (I/M) process are mainly responsible for this low ductility and low strength of these composites. Therefore, research efforts have been focused on refining primary and eutectic Mg_2Si crystal grains in Mg_2Si reinforced composites and modifying their morphology and distribution, in an effort to improve the properties of these composites. Other than advanced processing techniques, such as rapid solidification processing [4] and mechanical alloying [5], many efforts have been focused on modifying the Mg_2Si structure by alloying the material with elements, such as P [6] and Li [7], and compounds, such as TiB_2 or AlP/TiB_2 [8], and with an extra Si content [9]. Yet the elongation to failure is still disappointingly low, generally around 1–3%. So far, the highest elongation to failure reported in the literature is 8%, achieved in the Al–15% Mg_2Si–8% Si composite by the die casting technique [9].

Equal channel angular pressing (ECAP) is a severe plastic deformation process that has shown great potential in refining the microstructure [10] and homogenizing the distribution of particles in cast metal–matrix composites [11]. It has been shown that the processing route has a significant influence on the efficiency of ECAP processing in microstructural refinement as well as in the fragmentation and the distribution modification of the reinforcing particles. Among the four fundamental ECAP processing routes B_C, A, B_A and C, route B_C is recognized as the most effective in microstructural refinement when a die with $\Phi = 90^\circ$ is used [12] but as the least effective in particle redistribution [13]. In the present study, a modified B_C route was designed to improve the effectiveness of route B_C in particle redistribution while maintaining a high efficiency in microstructural refinement. To clarify this effectiveness, the modified route B_C was employed to process a hypoeutectic Al–Mg_2Si cast *in situ* composite to refine the structure and modify the distribution of eutectic Mg_2Si, in an effort to improve the ductility of the composites. Four passes, as a complete processing cycle for route BC [14], were performed to investigate the effectiveness of this modification. The microstructure and tensile mechanical properties of the composites processed by two routes were investigated.

2. Experimental procedures

Commercially pure Al, Mg and an Al–24% Si master alloy were used as starting materials to prepare a hypoeutectic Al–6.3 Mg–5.68 Si alloy composite corresponding to a composition of Al–10 Mg_2Si–2 Si. All compositions are given in weight percent unless otherwise stated. The alloys were prepared in air using an

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electrical resistance furnace. 0.8% Sb wrapped with aluminum foil was added into the alloy melt at 740 °C to modify the eutectic Mg_2Si structure. The melt was poured into a permanent mold of 40 mm in diameter and 120 mm in height at 700 °C.

ECAP samples were machined into 10 mm × 10 mm × 55 mm billets along the longitudinal direction of the ingots. A modified die ($\Phi = 90°$, $\Psi = 16°$) with function of back pressure [15] was used in this experiment. The samples were processed at 250 °C with a pressing speed of 0.5 mm/s in both the B_C route and the modified B_C route, denoted as $B_{C\text{-}m}$. The $B_{C\text{-}m}$ route followed route B_C in that the sample was rotated 90° between consecutive pressings; the only difference was that the billet was placed upside down before the fourth passage. The pressed samples were lubricated with a mixture of vaseline and graphite prior to ECAP processing. Four ECAP passes were performed to obtain an equivalent total strain of ~4 [16].

A scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) was used to examine general microstructures, and specially to study the morphology, size and distribution of eutectic Mg_2Si as a function of processing routes. EDS analysis of the constituent phases was carried out using the polished samples without etching. These same specimens were then deeply etched by using 5% HNO_3 and 95% methanol solution at −30 °C and 35 mA to partially remove the Al matrix and were examined by SEM to study the structure details of eutectic Mg_2Si. SEM observations were performed on the flow plane (*Y*) and the transverse plane (*X*) of the samples [17] that underwent the ECAP process to give representative information on the distribution and particle size of Mg_2Si. The changes in the Mg_2Si particle distribution caused by routes B_C and $B_{C\text{-}m}$ were quantitatively analyzed further by quadrat method. This method was found to be the most effective in characterizing the reinforcement distribution in metal–matrix composites (MMCs) with highly clustered particles [18]. Each image was divided into 225 rectangular quadrats of 2.8 μm × 2.11 μm according to the optimum quadrant size, which was twice the size of the mean area per particle. Five SEM images per route at 3000× magnification were analyzed, and a frequency histogram of the number of Mg_2Si particles per quadrat Nq was plotted.

Tensile specimens with a gauge dimension of 15 mm × 3.5 mm × 2 mm were machined from the billets along the longitudinal axes for both the as-cast and the as-pressed samples. Tensile tests were performed at room temperature on an Instron universal testing machine at a constant crosshead speed of 0.5 mm/min.

3. Results and discussion

The typical microstructure of the as-cast composite is shown in Fig. 1. A large volume fraction of the grey phase was identified as primary α-Al dendrites with a grain size of 100–200 μm, and they were surrounded by the coarse pseudo eutectic constituents Al–Mg_2Si where Mg_2Si shows a dark contrast, as revealed in the polished sample (Fig. 1(a)). A few white phases were identified as Mg_3Sb_2 (Fig. 1(a)). The stereo configuration of the eutectic Mg_2Si was further revealed as clusters of coarse flakes after deep etching, as shown in Fig. 1(b).

Fig. 2 represents the microstructures of the composite processed by ECAP in routes B_C and $B_{C\text{-}m}$, respectively. It is evident that processing up to four passes by route B_C produces clusters of fine Mg_2Si particles together with large particle-free zones (Fig. 2(a) and (c)), whereas processing by the modified route $B_{C\text{-}m}$ induces large relative displacements between the fine Mg_2Si particles and redistributes the particles significantly, as shown in Fig. 2(b) and (d). Therefore, the modified route $B_{C\text{-}m}$ led to a more homogeneous and dispersed distribution of fine Mg_2Si particles in the α-Al matrix. Although a certain degree of particle clustering was also observed on the flow plane (FP) of the composite processed by route $B_{C\text{-}m}$ (Fig. 2(b)), the spacing between particles, even in highly clustered areas, was much larger than that of the composite processed by route B_C where the densely clustered Mg_2Si particles maintained their initial distributions as in the as-cast microstructure, as indicated in Fig. 3(a) and (b) at a higher magnification. The difference in particle distribution was further confirmed by the quadrat method shown in Fig. 4. It revealed a more homogeneous particle distribution in the composite processed by route $B_{C\text{-}m}$ than route B_C. In the case of route B_C, high frequencies of empty quadrats and quadrats containing four or more particles were present, as shown in Fig. 4(a). In contrast, frequencies of quadrats containing four or more particles were drastically reduced, and almost no quadrats contained more than six particles in the case of route $B_{C\text{-}m}$ (Fig. 4(b)). In addition, fewer empty quadrats were seen for composites processed by route $B_{C\text{-}m}$ compared to route B_C. Nevertheless, the particle refinement efficiency was similar between route $B_{C\text{-}m}$ and route B_C. The Mg_2Si particle sizes were ~1 μm and ~0.9 μm in the cases of route $B_{C\text{-}m}$ and route B_C, respectively, as shown in Figs. 2 and 3. Consequently, the modified route $B_{C\text{-}m}$ is more effective in particle redistribution while maintaining the rapid grain refinement efficiency compared to route B_C.

The differences in particle redistribution and particle refinement effectiveness between the two processing routes are attributed to the nature of strain provoked in routes B_C and $B_{C\text{-}m}$. Route B_C is a redundant strain process in that shear deformation occurs cyclically every four passes, and the strain is restored after 4*n* passes in route B_C [10,14]. Therefore, route B_C did not geometrically redistribute the eutectic Mg_2Si particles [13]. However, the shear system of the fourth pass in the modified route $B_{C\text{-}m}$ was changed because the sample was placed upside down before the fourth passage through the die; therefore, the strain after four passes was not restored, and route $B_{C\text{-}m}$ was no longer a redundant strain process. Thus, Mg_2Si particles were redistributed during the shear deformation processes of the four ECAP passes in route $B_{C\text{-}m}$. In route B_C, shear occurred on different planes with an angle of 54.7°, between the shear planes of the first and third pass as well as between the second and fourth pass. Furthermore, the shear directions were reversed between the first and third pass as well as between the second and fourth pass. This rapidly increased the dislocation density as dislocations intersected and tangled, which in turn promoted the formation of grain boundaries during a complete processing cycle [14]. That is why route B_C shows a higher efficiency in grain refinement. Thereby, route $B_{C\text{-}m}$ would be slightly less effective in grain refinement owing to the variation of shear plane and shear direction on the fourth pass.

When compared to route $B_{C\text{-}UD2}$, as proposed in Ref. [14], route $B_{C\text{-}m}$ produces a more homogenous microstructure similar to route B_C and maintains the high efficiency in particle redistribution that is characteristic of route $B_{C\text{-}UD2}$. This is because in route $B_{C\text{-}m}$, the sample is placed upside down once every three passes (just before the fourth pass pressing), making the process no longer a redundant strain process. However, the sample is placed upside down once every two passes in route $B_{C\text{-}UD2}$, similar to route B_A, which produces a pancake structure with precipitates aligned approximately along the longitudinal direction of the specimen [14].

Fig. 5 shows the room temperature tensile testing results of the as-cast sample and the processed ones. The ultimate tensile strength (UTS) of the processed samples increased from 167 MPa of the as-cast sample to 201 MPa for route B_C and 215 MPa for route $B_{C\text{-}m}$, which corresponded to an increase of 20% and 29%, respectively. The comparable UTS increase of the processed sam-