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Dynamic Ageing of Two Al-Mg-Si Alloys Using Equal Channel Angular Extrusion

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Abstract

Dynamic ageing of two Al-Mg-Si alloys, alloy 6061 (Al-1.34% Mg_2Si) and alloy 6069 (Al-2.25% Mg_2Si), using equal channel angular extrusion (ECAE) has been studied. Compared to the nominal ~1000 minutes for conventional static peak-ageing treatment at 170°C, the time scale for dynamic ageing using ECAE reduces to ~10 minutes. It has been found that there is a notable further increase in ultimate tensile strength (UTS) of both alloys using dynamic ageing than static peak-ageing: over 40 MPa for the 6061 alloy and 100 MPa for the 6069 alloy. Ductility of dynamically aged alloys is found to be comparable to that of the statically peak-aged samples. The microstructures of dynamically aged alloys were characterized by transmission electron microscopy (TEM).

1. Introduction

Aluminium alloys containing Mg and Si as the major solutes are strengthened by precipitation of the metastable precursors (β'') of the equilibrium β (Mg_2Si) phase. In practice, artificial ageing following solution treatment (T6) is a universally accepted method to strengthen this series alloys, however T6 treatment is a thermally-activated diffusion process that depends on time and temperature applied [1]. Dynamic ageing (occasionally referred to as forming ageing) is an integrated process combining thermo-mechanical processing (TMP) and ageing treatment. Rather than temporal discretion in nucleation and growth of precipitates in conventional static ageing, the precipitation reaction occurs simultaneously during the dynamic ageing process [2].

Dynamic strain ageing in Al alloys was initially reported by Morris [3]; in this paper, he claimed that the change in strain-rate sensitivity with temperature is due to the dynamic strain ageing and that the UTS of materials can be enhanced in this way. Later Mulford and Kocks [4] proposed a model based on dynamic strain ageing to interpret the jerky flow recorded during tensile deformation.

They left two questions unanswered: (A) no direct microstructural evidence of dynamic ageing was provided and (B) tensile deformation changes the overall geometry of the sample and therefore limits the potential application of dynamic ageing.

Equal channel angular extrusion (ECAE) is an efficient TMP method, which was invented by V.M. Segal in 1977 [5]. An outstanding advantage of ECAE over other TMP is that a competitive high strain can be gained with little change in the sample geometry. The impact of ECAE processing on the static ageing of Al alloys has been investigated to some extent. It is reported that the ageing responses in some Al-Mg-Si alloys were accelerated by using ECAE [6] and that pre-ECAE followed by ageing in a commercial 6061 alloy contributed to a higher strengthening effect than post-ECAE after ageing [7]. But unfortunately these two processes were never integrated. In this work, we report the practice of applying ECAE into dynamic ageing, the mechanism of the dynamic ageing and possible market potential of this novel technique.

2. Experimental materials and procedures

Two Al-Mg-Si alloys with contrasting contents of intermetallic Mg_2Si phase (a dilute alloy, 6061 with 1.34% Mg_2Si, and a concentrated alloy, 6069 with 2.25% Mg_2Si) were supplied by Alcan Ltd. in the form of ~10cm diameter ingots with average grain size of ~110 μm diameter [8].

ECAE samples of $\phi 15\times100$ mm were machined from the ingots along longitudinal axis. Samples were initially solution treated at 560°C for 3 hours and water-quenched to room temperature. Static ageing was performed in an oil bath of 170°C ± 2°C and dynamic ageing was conducted through ECAE die having a die angle of 120° at the speed of 30 mm/min. During dynamic ageing specimens were lubricated with PTFE tape and 3 ECAE passes were performed to gain a total effective strain of 2.0: one ECAE pass at 100°C followed by 2 ECAE passes at 170°C. During ECAE all samples were extruded at the same orientation between each pass. Samples for TEM and Vickers hardness number (VHN) measurements were cut from near the centre of the ECAE rod along the longitudinal direction.

Thin foil TEM specimens were prepared by electro-polishing in a solution of 700 ml analar methanol + 300 ml 69% nitric acid at a temperature of –30°C ± 2°C and 12 V DC. The thin foils were examined in a Philips CM20 electron microscope operated at 200 kV.

Hardness data were determined using a 10 Kg load from the average of four hardness readings from each sample. Tensile specimens with a gauge diameter of 6.30 mm and a length of 33.60 mm were machined from the rod along the longitudinal axis. Tensile testing was carried out at room temperature using an Instron 1185 machine operating at a constant crosshead speed of 1.0 mm/min.

3. Results and Discussions

3.1 Ageing Behavior

Figure 1 shows the hardness variation in the dynamically aged alloys undergoing a further static ageing process at 170°C, typical hardness curves of the as-solutionized 6061 and 6069 alloys during static ageing at the same temperature are also included as references. While the hardness of the solutionized alloy increases to a peak hardness after ~1000 minutes of ageing and decreases sharply after that at 170°C, the hardness of the dynamically aged sample remains almost constant for ~1 hour and slowly decreases with time.

Compared to the gradual increase in the hardness of the solution treated samples during conventional static ageing, it is evident from Figure 1 that there is no further hardening effect apparent in either of the dynamically aged samples. Therefore it can be concluded that there is not sufficient solute content remaining in solid solution following dynamic ageing to give any further ageing response.

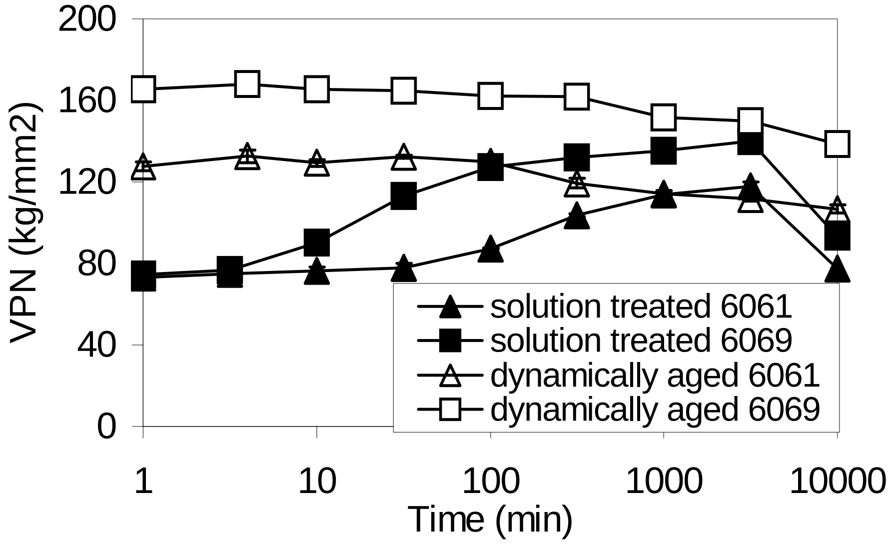


Figure 1: A comparison of ageing characteristics of the dynamically aged and the as-solutionized samples at 170°C.

3.2 Tensile Properties at Room Temperature

Mechanical properties of the dynamically aged and conventionally peak-aged alloys at room temperature were assessed from the strain-stress curves and the results are summarised in Figure 2.

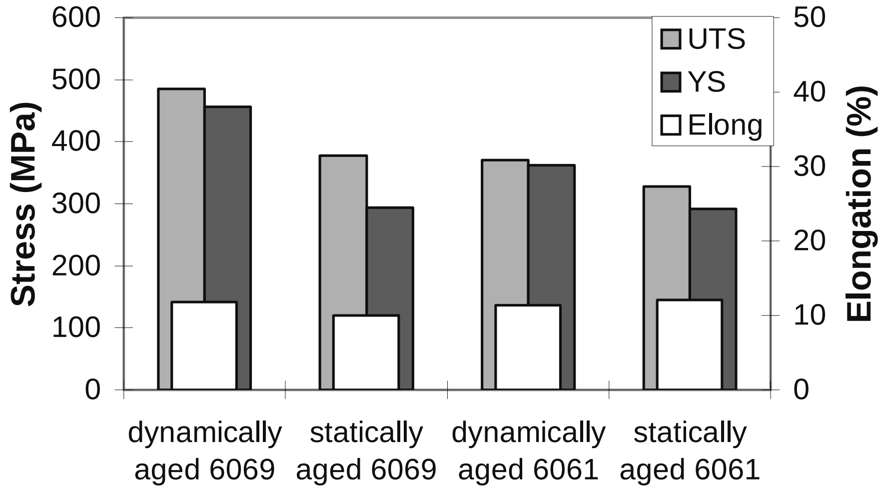


Figure 2: Tensile properties of the 6069 and 6061 alloys after dynamic ageing and static peak-ageing at 170°C.

It is evident from Figure 2 that dynamic ageing using ECAE leads to a significant improvement in strength compared with conventional peak-ageing, and with no cost of ductility In particular, the statically peak-aged 6061 at 170°C has a UTS of 328 MPa with an elongation of ~12% while the UTS of the dynamically aged alloy is 370 MPa with an elongation of ~11%. For the 6069 alloy the UTS after statically ageing to peak hardness is ~377 Mpa and the elongation is only 10% while the UTS of the dynamically aged 6069 alloy is ~484 MPa with an elongation of 12%..

3.3 Transmission Electron Microscopy

Figure 3 and Figure 4 show bright field (BF) and dark field (DF) (for DF micrographs the diffraction spot used is indicated by a circle in the diffraction pattern) TEM micrographs of the dynamically aged 6061 and 6069 alloy respectively, resolving the fine precipitates and large amount of dislocations formed during dynamic ageing. The presence of a large number of tiny dots in the BF images represents the β'' needles viewed end-on; in the DF micrograph, the set of precipitates that are parallel to the electron beam are visible and they appear with a circular cross section. The distortion in the micrographs and diffraction patterns is due to the existences of dislocations, residual stress fields and the submicron subgrain structure produced during deformation. Figure 3c and Figure 4c contain the diffraction patterns of the alloy with the electron beam in the [100] direction showing faint symmetrical streaks in the diffraction spots of Al matrix, which generated from the precipitates parallel to the beam direction. The diffraction patterns look similar to those of the statically peak-aged aluminium alloys [9-12].

The nucleation mechanism of precipitates during dynamic ageing is proved to be a dislocation-assisted process, which is clearly suggested by the regularly aligned precipitates along dislocations in the DF image of the 6061 alloy after dynamic ageing (Figure 3b). The same principle can be applied to the dynamic ageing of the 6069 alloy under the circumstances that the same processing was applied and fine precipitates were observed within ~10 minutes of ageing.

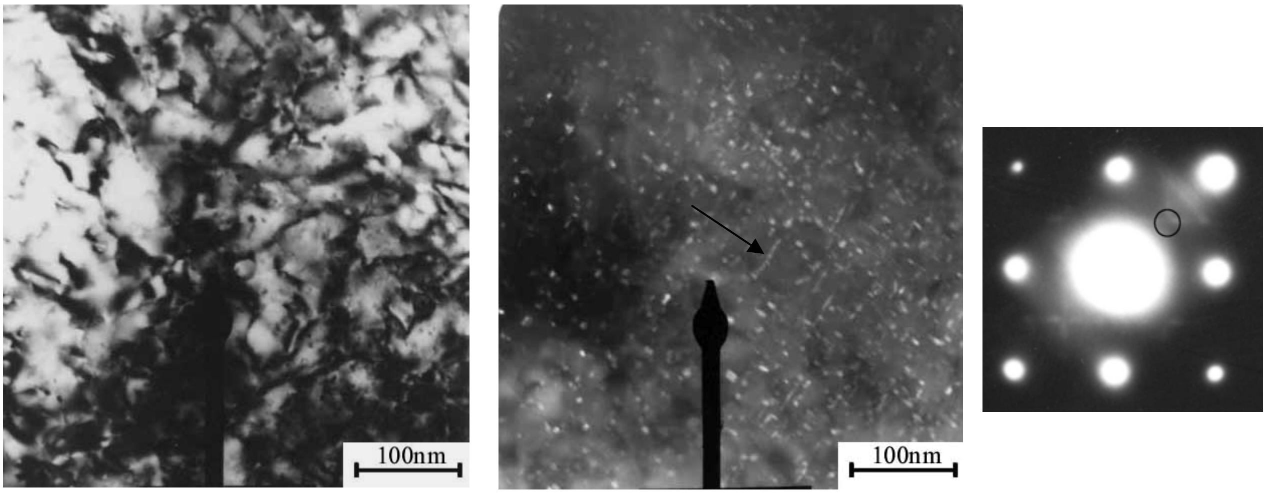
The morphology of the precipitates in the dynamically aged alloys can be determined from BF and DF micrographs coupling the diffraction patterns: needle-shaped, but it is difficult in determining the length from the BF micrographs (Figure 3a and Figure 4a). This is due to a high density of dislocations and the associated strain fields. However, it is also possible that the needle-shaped precipitates were fragmented during dynamic ageing and therefore the length along the *c*-axis was significantly shortened [6].

Compared to the microstructure of the same alloy after static peak-aging at the same temperature, precipitate refinement in the 6069 alloy and coarsening of the precipitates in the 6061 alloy were found [13-14]. Refinement is due to a high density of dislocations produced from the first ECAE pass at 100°C, which may then act to provide a high density of heterogeneous nucleation sites for precipitation during subsequent dynamic ageing at 170°C; whereas coarsening may be due to dislocations acting as short-circuit diffusion path for solute during dynamic ageing [13-16]. Precipitate coarsening during dynamic ageing can be controlled either by increasing ECAE speed or by decreasing the ageing temperature.

The increment of strength increase of the 6069 alloy by using dynamic ageing is due to the contributions of refined precipitates and grain/subgrain size, and the high density of dislocations. Although the precipitates in the dynamically aged 6061 alloy were coarser than the conventionally aged, the alloy was further strengthened, which is related to the refined grain/subgrain structure and large dislocation density. The contributions of particles, grain size and dislocations to the overall strength of the alloy can be combined in an additive form as suggested by Equ. (1) [2, 14]:

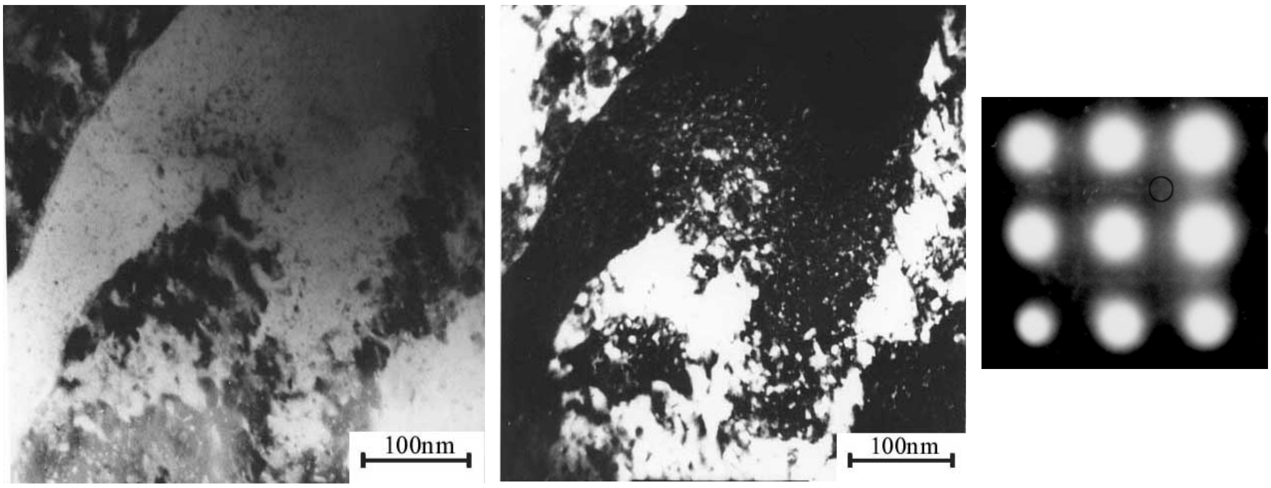
$$\sigma_y = \sigma_0 + k_1 \frac{\mu b}{r} + k_2 d^{-\alpha} + k_3 \mu b \sqrt{\rho}\,, \qquad (1)$$

where σ_y is the yield strength, σ_0 is the yield strength of the matrix, μ is the shear modulus, *b* is the Burger's vector, *r* is the mean radius of the precipitate, *d* is the mean grain/subgrain diameter, α is the stress/microstructure dependent parameter, ρ is the density of dislocations and k_i (*i*=1, 2 and 3) are the constants. The differences in precipitate size, dislocation density and grain structure determine the mechanical properties of statically and dynamically aged Al-Mg-Si alloys.



(a) BF TEM micrograph (b) DF TEM micrograph of (a) (c) Diffraction pattern. Beam direction [100]

Figure 3: TEM micrographs of the 6061 alloy after dynamic ageing.



(a) BF TEM micrograph (b) DF TEM micrograph of (a) (c) Diffraction pattern. Beam direction [100]

Figure 4: TEM micrographs of the 6069 alloy after dynamic ageing.

A discussion of the relative contributions of these effects is beyond the scope of the present work, but the reader is referred to [14] for further discussion on this topic.

This study shows that dynamic ageing of Al-Mg-Si using ECAE resulted in a shorter processing time and better combination of mechanical properties. It is reasonable to expect the application potential of this technique in industry once the ECAE technique is scaled-up for commercial applications.

4. Conclusions

(1) Dynamic ageing using the ECAE technique resulted in an increase in the UTS of both the 6061 and 6069 alloys when compared to conventional static peak-ageing treatment at the same temperature.
(2) Dislocation-assisted precipitation occurs during dynamic ageing of the 6061 and 6069 alloys at 170°C.
(3) The size of precipitate in the dynamically aged alloys is amendable by the temperature and deformation rate. The growth of precipitates during dynamic ageing is governed by the dislocation-assisted diffusion mechanism.
(4) Dynamic ageing using ECAE is superior to static ageing in producing both improved mechanical properties and reduced time to peak strength.

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