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Thermal stability of ultrafine grained AA8090 Al–Li alloy processed by repetitive corrugation and straightening

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ABSTRACT

The thermal stability of an ultrafine-grained (UFG) AA8090 Al-Li alloy with an average grain size of 2 μm is reported. The UFG structure was obtained by repetitive corrugation and straightening (RCS) process after 8 passes using optimized processing parameter conditions identified in the previous research, i.e., processing temperature 300 °C; ram velocity 1.5 mm/s using V-Grooved corrugating die profile (pitch 20 mm, corrugating angle 30° and curve radius 2 mm) and straightening by a flat die. The grain size distribution ranged from 200 nm to 8 μm. The average hardness of the RCS processed specimen had increased to a mean value of 104 HV from an average value of 75 HV in the parent material. The RCS processed specimens were annealed at different temperatures (7) for varying time periods (t) to investigate their thermal stability using hardness and microstructure changes as the quantitative measures. The microstructure analysis of the samples was done using electron backscattered diffraction (EBSD) analysis. The study was based on a full factorial design of experiments (DOE) and the results were analyzed using a TOPSIS optimization tool. The results prove that thermal stability is more influenced by temperature than the time of annealing, a known result based on earlier qualitative studies on conventional materials. The study also demonstrates that the material is fairly stable up to 300 °C. The activation energy for grain growth is found to be 76 kJ/mol in a range where the annealing time is 2–6 h and the temperature range is 200–400 °C.

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1. Introduction

Utilization of aluminium-lithium alloys has been seen in recent commercial jetliner EH101 helicopter airframes [1]. Development of aluminium-lithium has replaced many of the existing aluminium alloys in reducing the weight of aircraft and aerospace structures. Some of the most important commercial alloys in this class include 2090, 2091, 8090, and Weldalite 049 that were introduced in the 1980s. Al–Li Alloy 8090 has been replacing longest serving commercial aluminium alloys, namely 2014 and 2024. AA8090 is known for 10% lower density and 11% higher modulus than these conventional counterparts. AA8090 also exhibits superior mechanical properties at cryogenic temperatures.

There are several techniques employed for improving the strength of metallic materials which are solid solution...
strengthening [2], work hardening [3] dispersion strengthening [4], and grain refinement [5]. Solid solution strengthening is governed in alloy systems where solute and solvent are soluble in each other to some extent [6, 7]. Particle reinforced metal matrix composites (PRMMCs) are strengthened by dispersion strengthening mechanism [8–10]. However, the strengthening effects due to the above mechanisms are limited. Severe plastic deformation (SPD) technique is an effective way to produce ultrafine grain (UFG) and nanostructures (NS) in bulk metallic materials, compared to the grain size reduction by precipitation hardening [11]. Different SPD techniques have been developed and UFG structures have been produced in different materials, e.g. aluminium alloys [12], magnesium alloys [13], copper alloys [14]. Other than the structural application due to its improvement in mechanical properties, NS/UFG materials also have the scope in the field of electromagnetic waves absorption [15]. Among the different SPD techniques, Equal Channel Angular Pressing (ECAP) is capable of producing UFG/NS [16, 17]. Repetitive Corrugation and Straightening (RCS) has been recently used to produce UFG in AA5083 and Al–Mg alloy sheets [18, 19]. In recent work, the present authors achieved homogeneous grain refinement in AA8090 Al–Li alloy by repetitive corrugation and straightening using ‘V’ grooved dies [20]. Improvement in strength and hardness due to grain refinement was reported. The RCS produces a microstructure with a combination of high angle grain boundaries (HAGB) and many low-angle grain boundaries (LAGB) [21]. Therefore, the stability of the UFG materials at elevated temperatures needs to be assessed.

Hoseini et al. [22] investigated the thermal stability of UFG pure titanium prepared by ECAP. The specimens were subjected to annealing at different temperatures from 450 °C to 600 °C and for the time duration from 10 min to 6 h. The EBSD analysis of the annealed deformed specimen and the associated grain growth activation energy reported that the samples are thermally stable up to 450 °C for 6 h. The UFG/NS materials tend to exhibit superplasticity at high temperatures, while they are stable only at the lower temperatures [23], due to the instability of sub-grains [24]. Sub-grains with LAGB’s readily grow at higher temperatures and lead to a drastic reduction in properties [25].

Adamczyk et al. [26] studied thermal stability in Al alloys (0.7% Li and 1.6% Li) using differential scanning calorimetry (DSC) and annealed them at a constant homologous temperature. It was found that the stability of microstructure is higher in the alloy containing a higher amount of lithium. During heating, grain growth is restrained by precipitation and ordering of precipitates. To improve stability, uniform dispersion of the second phase is important. A large number of fine secondary particles pins the grain boundaries and retards boundary diffusion [27]. In another study, Pavel et al. [28] investigated the thermal stability of pure Ti and Ti–6Al–7Nb alloy in terms of microhardness values (0.5 kgf load and 10 s dwell time), microstructures, and electrical resistivity. As the pre-recovery stage could not be unambiguously established by the microstructure, electrical resistance at the recovery and recrystallisation stages was used as an alternative measure. Similarly, the thermal stability of ultrafine grained Cu–Cr–Zr alloy, produced by equal channel angular pressing (ECAP), was studied by Khadidja et al. [29]. They reported that a particle-stimulated nucleation mechanism is responsible for discontinuous recrystallisation in the alloy. It is clear that there are many influencing factors associated with the thermal stability of UFG/NS materials such as grain size/volume of grain boundaries, nature of grain boundary, dislocation density, latent heat (stored energy) associated with dislocations, elastic stress field, substitutional solutes, precipitates, segregation at grain boundaries, and many other. Notwithstanding this, thermal stability is a property, which can be assessed accurately to a large extent by its microstructural stability. The thermal stability can also be evaluated by measuring associated properties such as strength, hardness, electrical conductivity/resistivity, latent heat, etc. This paper examines the thermal stability of UFG AA8090 alloy produced by repetitive corrugation and straightening.

### Table 1 – Control factors and levels.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>200, 300, 400</td>
</tr>
<tr>
<td>Time duration (h)</td>
<td>2, 4, 6</td>
</tr>
</tbody>
</table>

### 2. Material and methods

An AA8090 alloy (Al – 94.46%, Li – 2.5%, Cu – 0.5%, Mg – 0.89%, Zr – 0.175%, Ni – 0.165%, Pb – 0.387%) was processed by the repetitive corrugation and straightening technique using a V-Groove die (pitch 20 mm, corrugating angle 30° and curve radius 2 mm) for the corrugation at a pressing velocity 1.5 mm/s. One corrugation followed by a straightening step represents one pass. The detailed procedure of RCS is described elsewhere [20]. Microhardness was measured using a Wilson Wolpert (Germany) hardness tester with a diamond indenter. A load of 9.8 N and an indenting speed of 5 mm/s was used. The microstructure study was carried out using electron backscatter diffraction (EBSD) analysis. The samples for EBSD analysis were prepared by fine polishing by SiC emery sheets of 300, 600, 1000, 1200 mesh grids followed by suspended alumina polishing and colloidal silica polishing.

The recrystallization temperature of the AA8090 alloy is above 300 °C [20]. The average grain size and an average hardness of the parent material are 65 ± 5.7 μm and 75 ± 1 HV0.5 (hardness calculated with 0.5 kg load; dwell time 10 s) respectively. The specimens were subjected to a maximum of 8 passes at a processing temperature of 300 °C and a pressing speed of 1.5 mm/s. The hardness of the alloy sheets after the 8th pass was 104 ± 2.9 HV0.5 and the average grain size 2 ± 1.5 μm, with the range of grain size being 200 nm to 8 μm.

The thermal stability test was conducted on the RCS processed samples by subjecting them to annealing at different temperatures and time durations. Full factorial experimental design and the Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) method are used to study the input parameters of the annealing experiment which have multiple output responses. Table 1 presents the different factors (temperature (°T), and time duration (t)) and the levels of the factors considered. Three temperature levels are considered for this
present study. The lower temperature (200 °C) was fixed below the recrystallization temperature, a temperature (300 °C) near the recrystallization temperature, and a temperature (400 °C) above the recrystallization temperature. Also, the temperature above 400 °C will be very close to the melting point of the material, and the microstructural stability is known to be highly negligible. Hence, the maximum temperature level is selected as 400 °C.

3. Results and discussion

3.1. Microstructure studies of parent and RCS processed samples

The microstructure and grain size distribution in the unprocessed and the RCS processed alloy are shown in Fig. 1. Fig. 1(a) shows the TEM image of the unprocessed parent material, shows the presence of disordered metastable δ′ (Al3Li) in the Al–Li alloy and this is identified by the XRD analysis shown in Fig. 1(b). The EBSD-IPF image of RCS processed Al–Li alloy after the 8th pass, shown in Fig. 1(c) reveals the presence of dense dislocation and fine grain structures. Fig. 1(d) is the TEM image of the RCS processed material which reveals the boundaries of refined grains pinned by ordered δ′ precipitates and T1 (Al2CuLi) and S′ (Al2CuMg) phases [30], which are confirmed by the XRD peaks in Fig. 1(e). The grain size distribution present in the RCS processed alloy is shown by Fig. 1(f).

Material strengthening is due to the combined effect of dislocations multiplication, secondary phases and the development of finer grains when the density of dislocations reaches a critical value [31]. The SPD process converts the disordered metastable δ′ phases (Al3Li) to ordered δ′ phases (Al3Li) and also facilitates the precipitation of S′ (AlCuMg) and T1 (Al2CuLi) particles, all of which pin the grain boundaries and triple junctions, see Fig. 1(d). From Fig. 1(a), it is clear that the parent material has less amount of metastable δ′ (Al3Li). Evidently, the precipitation of the S′ and the T1 phases, as well as the transformation of disordered metastable δ′ (Al3Li) precipitates to ordered δ′ (Al3Li) are the consequences of SPD processing [32].

3.2. Hardness and grain size with TOPSIS analysis

The average hardness of the RCS processed material after the 8th pass had increased to 104 ± 2.9 HV0.5 from a value of 75 ± 1 HV0.5, and the grain size had gone down to 2 ± 1.5 μm from 65 ± 5.7 μm. The hardness and average grain size were measured after the annealing experiment as per the design of experiments (DOE) at different temperatures and time durations (DOE is the method used to find out the cause and effect relationship). The results are reported in Table 2.

Table 2 shows that as the temperature and time increase, the hardness decreases and the grain size increases. At a higher annealing temperature, the grain growth (due to accelerated diffusion and dislocation annihilation) is more rapid. Therefore, the hardness decreases with increasing temperature (T) (inverse Hall-Petch/creep effect). The results show that increased time duration (t) also leads to a decrease in the microhardness value. It has been suggested that severe plastic deformation introduces micro-defects such as dislocations and vacancies that act as precipitation nucleation sites rather than precipitation kinetics enhancing agents [33,34]. The change in hardness due to different annealing temperatures indicates that at the lowest annealing temperature (200 °C) the microstructure is stable. The presence of Li in solid solution has ensured stability up to this temperature, notwithstanding the high density of dislocations caused by SPD. The output responses to the T and t of annealing are
Table 2 – DOE and output response for thermal annealing study.

<table>
<thead>
<tr>
<th>Run</th>
<th>Annealing condition</th>
<th>Responses</th>
<th>Closeness co-efficient values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature (°C)</td>
<td>Hour</td>
<td>Microhardness (HV&lt;sub&gt;0.5&lt;/sub&gt;)</td>
</tr>
<tr>
<td>1</td>
<td>200</td>
<td>2</td>
<td>96.2 ± 2</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>4</td>
<td>94.94 ± 2</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>6</td>
<td>93.54 ± 2</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>2</td>
<td>92.27 ± 2</td>
</tr>
<tr>
<td>5</td>
<td>300</td>
<td>4</td>
<td>91.27 ± 1</td>
</tr>
<tr>
<td>6</td>
<td>300</td>
<td>6</td>
<td>90.04 ± 2</td>
</tr>
<tr>
<td>7</td>
<td>400</td>
<td>2</td>
<td>88.36 ± 1</td>
</tr>
<tr>
<td>8</td>
<td>400</td>
<td>4</td>
<td>86.64 ± 1</td>
</tr>
<tr>
<td>9</td>
<td>400</td>
<td>6</td>
<td>84.32 ± 1</td>
</tr>
</tbody>
</table>

The prediction of closeness co-efficient values was determined using TOPSIS for multi-response ANOVA analysis.

Fig. 2 – Output responses to the annealing study: (a) micro-hardness, (b) grain size.

illustrated in Fig. 2 for ease of understanding. Evidently, the microhardness does not change significantly at 200 °C up to 2 h time duration (very slow kinetics of grain growth) (Fig. 1). As the annealing temperature increases to 300 °C and 400 °C, the microhardness values decrease considerably. As is well known, the effect of temperature in decreasing the microhardness is more profound than that of time and this is borne out in these experiments also in a quantitative manner. At 400 °C, the hardness value decreases even after the shortest time of annealing for 2 h. The decrease in HV<sub>0.5</sub> after four hours of annealing is by about 15.4%, which decreases further to 19% after 6 h of annealing (Fig. 2a).

The grain size of the RCS processed alloy as a function of annealing temperature and time is shown in Fig. 2(b). The differences in hardness and grain size of the annealed specimens can be explained by the differences in the stored deformation energy that provides the driving force for rebuilding the microstructure [35]. The UFG metals produced by SPD are mostly unstable and at elevated temperatures undergo recovery and recrystallisation prior to grain growth [36].

3.3. ANOVA

The ANOVA (Analysis of variance) was performed to predict the statistical significance of the process parameters by performing an F-test (F test is the one-way analysis of variance).

The output response has been analyzed and the percentile contribution of the input parameters has been given in Table 3. From the analysis conducted at 95% confidence and 5% significant level, the temperature has the highest contribution towards the response values obtained. The percentage of contribution to grain growth by temperature is 63.03% and by time is 35.87% with an error of 1.1%. F value greater than 4 determines the input parameters having a significant effect on the quality of the characteristics. From the ANOVA it is found that both temperature and time are quality characteristics to be considered as meaningful experimental variables. The proportion of significant value shows that temperature is the more significant and time is the significant parameters. (Proportion of significant value less than 0.05 is declared as significant.)

3.4. Microstructure stability

Fig. 3 shows the microstructure of AA8090 Al–Li alloy after annealing for different ’T’ and ’t’ values. Lithium solutes and secondary phase (’’<i>t</i>’’, ’’<i>s</i>’’, ’’<i>t</i>’’) particles have a strong influence on the deformation structure and annealing behaviour [37]. Generally, the ’’<i>t</i>’’ (Al<sub>12</sub>Li) secondary phases are predominant in the Al–Li alloy system. However, the XRD analysis also traces ’’<i>s</i>’’ (Al<sub>12</sub>CuMg) and T1 (Al<sub>12</sub>CuLi) phases in the AA8090 alloy system in the RCS processed samples those are not seen in the unprocessed samples. These compounds are considered to be precipitated out during RCS processing, which has a
Table 3 – ANOVA results of the contribution of different parameters for thermal stability of RCS processed AA8090 Al–Li alloy.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P</th>
<th>% of contribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>2</td>
<td>0.5085</td>
<td>0.2543</td>
<td>115.68</td>
<td>0.000(^a)</td>
<td>63.03</td>
</tr>
<tr>
<td>Time</td>
<td>2</td>
<td>0.2894</td>
<td>0.1447</td>
<td>65.84</td>
<td>0.001(^a)</td>
<td>35.87</td>
</tr>
<tr>
<td>Error</td>
<td>4</td>
<td>0.0088</td>
<td>0.0022</td>
<td></td>
<td></td>
<td>1.10</td>
</tr>
<tr>
<td>Total</td>
<td>8</td>
<td>0.8067</td>
<td></td>
<td></td>
<td></td>
<td>100</td>
</tr>
</tbody>
</table>

\(^a\) Most significant, DF, degree of freedom; SS, sum of squares; MS, mean sum of square; F, Fisher value, P, proportion of significant value.

Fig. 3 – EBSD orientation map of RCS samples processed by V-groove die at 1.5 mm/s and annealed at (a) 200 °C for 2 h, (b) 200 °C for 4 h, (c) 200 °C for 6 h, (d) 300 °C for 2 h, (e) 300 °C for 4 h, (f) 300 °C for 6 h, (g) 400 °C for 2 h, (h) 400 °C for 4 h, and (i) 400 °C for 6 h.

The EBSD-IPF microstructures (Fig. 3(a–i)) show that the grains retain their size and structure after the initial annealing period of 2 h. The \(\delta'\) phase of the Al–Li alloy does not show a significant difference in its EBSD-IPF microstructure appearance, which is due to the negligible mismatch of the lattice structure [26]. The annealing time is very important and the prolonged annealing time allows the dislocations to get annihilated. The importance of annealing time is reported by Caillard [33] through his in situ straining experiment done at 0–350 K, he ensures that the time asserts mobility of atoms at an elevated temperature and leads to neutralize the positive and negative dislocations, which is known as annihilation [38]. But, the temperature is a greater influencing factor than time, which is evident from the coarse grain microstructure of the sample annealed at 400 °C for 6 h.

Fig. 4 displays the grain size distribution plots for different annealing temperatures and time. From the grain size distribution, it is seen that the grain size plot moves towards the coarse grain size region on increasing the annealing temperature and time. For a clear understanding, Fig. 1(d) is referred to, which shows the initial grain size range of the RCS processed alloy to be from 200 nm to 8 \(\mu\) m. Fig. 4(a) shows the grain size distribution of a sample annealed at 200 °C for 2 h, which has a grain size distribution in the range of a few microns to a few hundred microns. Coarser grain sizes dominate in Fig. 4(i) (400 °C annealing for 6 h; grain size distribution in the range of a few hundred microns to several thousand microns) compared with the distribution present in Fig. 4(f), which corresponds to annealing at 300 °C for 6 h. The microstructure
observations reveal that the changes in the hardness value can be correlated with an increase in grain size. Speaking on annealing time, the annealing for 4 h and 6 h results in significant grain growth over 2 h annealing for each temperature segments. After annealing at 400 °C, the microstructure experiences a drastic grain growth. In contrast, annealing at 200 °C retains the grain size distribution (Fig. 4(a)) where a large proportion of grains have a fine size with an average grain size value of 6.4 μm.

Bacca et al. [39] have pointed out that most of the work during plastic deformation is released as heat, but a small amount of energy is associated with the dislocations generated by the deformation process. During dynamic recrystallisation, such stored energy is released generally by thermal activation and the dislocations get annihilated. These effects get accelerated by increased temperature and time of annealing. As mentioned earlier, the effect of temperature is greater than that of time.

The strengthening mechanism of the AA8090 alloy matrix by the effect of ordering the available secondary phase and precipitated inter-metallic compounds in accordance with the Orowan pinning mechanism [31] was well discussed by the authors elsewhere [32]. Hence, it is wished to study the influence of the ordered secondary phases β' (Al₃Li) and the precipitated inter-metallic compounds like S' (Al₃CuMg) and T₁ (Al₂CuLi) particles, all of which pin the grain boundaries and triple junctions. It is well known that at elevated temperatures, the material strength reduces due to the stress relaxation [40]. Hence, the formed sub-grain and dislocation start diffusing with respect to the increase in temperature. However, the precipitated intermetallic compounds neither decomposed and diffused in the matrix nor disordered. Fig. 5 shows the TEM micrograph of RCS processed AA8090 Al-Li alloy annealed for 2 h at 200 °C and 6 h at 400 °C. Fig. 5(a) shows evidence for the stability of the grain boundaries and Fig. 5(b) shows evidence to the diffused sub-grain boundaries and dislocations which leads to grain growth. It is also seen that arrays of precipitates left out in the matrix have not decomposed into the matrix again. This is due to the strengthening of precipitates by protective layer formation due to Debye temperature [41].

3.5. Activation energy for grain growth

The activation energy for grain growth is calculated by using Eq. (1) [22]

\[ \frac{D}{D_0} = \frac{D_0}{D_0} = tK_0 \exp \left( \frac{-Q}{RT} \right) \]

(1)

where D is the current grain size, D₀ is the initial grain size, n is the time exponent, t is the time, K₀ is the constant, Q is the activation energy, R is the gas constant and T is the absolute temperature. The activation energy for grain growth is estimated from the slope (−Q/R) of a plot between \( \ln(D/D_0) \) and 1/T (Fig. 6). The thermal studies were conducted at temperatures below, near, and above 0.5Tₘ, where Tₘ is the melting point of the material.
Table 4 – Time exponent n.

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>Time exponent, n</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>1.552</td>
</tr>
<tr>
<td>300</td>
<td>0.7652</td>
</tr>
<tr>
<td>400</td>
<td>0.549</td>
</tr>
</tbody>
</table>

The time exponents have been calculated and given in Table 4, with reference to the Ln(D^{1/2} - D_0^{1/2}) vs. 1/T plots given in Fig. 6 using Eq. (2) according to Beck et al. [42].

\[ D = Kt^n \]  (2)

where \( D \) is the current grain size, \( K \) is the constant, \( t \) is the time, \( n \) is the time exponent. This can be rewritten as Eq. (3)

\[ \ln D = \ln K + n \ln t \]  (3)

The time exponent ‘\( n \)’ is found as the slope of the \( \ln D \) vs \( \ln t \) plot (Fig. 7). The time exponent \( n \) could be expressed in terms of the driving force exponent, which is expressed as grain boundary mobility \( m \) and is equal to \((1/n) - 1\) [43]. Evidently, a lower \( n \) value leads to a higher \( m \) value, which, in turn, leads to faster grain coarsening. Evidently in our experiments, as is to be expected intuitively, the grain boundary mobility \( m \) increased with increasing annealing temperature.

In Arrhenius kinetics, the activation energy in a small temperature interval is assumed to be temperature independent. Using this assumption, the activation energy is found to be 76 kJ/mol, when the time interval is 2–4 h and the temperature interval is 200–400 °C. The mechanisms of grain growth represent a controversial topic. Based on the present macroscopic study, nothing concrete can be said about the grain growth mechanisms other than the microstructural stability.
Hence, there is a scope for a separate study on the grain growth mechanism of UFG/NS AA8090 alloy standstill.

4. Conclusions

Based on the observations made, the following conclusions could be drawn:

- The ordered $\delta'$ Al$_3$Li phase, and the formation of $T_1$ (Al$_2$CuLi) and $S'$ (Al$_2$CuMg) phases, formed during the RCS process only allow a weak softening of AA 8090 alloy up to 200 $^\circ$C.
- From the TOPSIS and ANOVA analysis, it is found that temperature has a greater effect (i.e. 63.03%) than the annealing time (i.e. 35.87%) in grain coarsening and thermal degradation of materials.
- A temperature higher than the recrystallisation temperature and prolonged annealing time significantly affect the thermal stability of the RCS processed AA8090 alloy. As the temperature and time period of annealing increase, the hardness decreases and the grain size increases.
- The time exponent $n = 1.5, 0.77, 0.55$ for 200 $^\circ$C, 300 $^\circ$C, and 400 $^\circ$C respectively. The activation energy for grain growth $Q = 76$ kJ/mol, for the RCS processed AA8090 alloy after annealing for 2–4 h in the temperature range of 200–400 $^\circ$C.

Data availability statement

The data provided in the EBSD and TEM analysis are raw data and all other data are reproduced data based on experimental readings.

Conflicts of interest

The authors declare no conflicts of interest.

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References


