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ABSTRACT
A solutionized Al2024 alloy was subjected to rolling at liquid nitrogen temperature (cryorolling) resulting in an ultra-fine structure. The material was also subjected to recovery annealing at 160°C. The ultra-fine structured material demonstrated increased strength but very low ductility. The uniform elongation of the material after recovery annealing increased without any sacrifice of strength.

Keywords: cryorolling, ultra-fine structure, Al alloy

1. INTRODUCTION
There has been a strong desire to refine the grains or subgrains of metals into the submicrometer and nanocrystalline regimes as such metals have increased strength [1-3]. The increase of yield stress with reduction of grain size is commonly described in a phenomenological manner by the well-known Hall–Petch relationship. However, the mechanical properties of ultra-fine structured metals and alloys can be further improved via manipulation with dislocation density and precipitation through heat treatment.

In recent years, a number of methods have been developed to refine the microstructure (e.g. [1-3]). These include electro deposition, inert gas condensation, ball milling and different methods of severe plastic deformation. One of the most recent developed approaches is the rolling of metals at low homologous temperatures, i.e. cryorolling. This approach has been applied to pure Cu [4, 5], pure Ni [6] and Al alloys [7,8]. The current work focuses on the evolution of the microstructure after cryorolling in an Al2024 alloy and the effect of recovery annealing on the microstructure and structure-property relationship.

2. MATERIAL AND EXPERIMENTAL PROCEDURES
A commercial aluminium alloy 2024 was chosen for this investigation. The chemical composition of the material is given in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>Cu</th>
<th>Mg</th>
<th>Mn</th>
<th>Si</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4.1</td>
<td>1.45</td>
<td>0.8</td>
<td>0.1</td>
<td>0.03</td>
</tr>
</tbody>
</table>

The material was solutionized at 490°C for 10hr and quenched in water. This resulted in a homogeneous microstructure with average grain size of 38 μm. Billets 100x60x10 mm³ were rolled at liquid nitrogen temperature with a total reduction ratio of 78%. Some of the rolled material was subjected to recovery annealing at 160°C for 2hr.

The microstructure was studied using Transmission Electron Microscopy (TEM) on a Philips CM20 microscope operated at 200 kV. Samples were prepared by twin jet electropolishing in a solution of 30% of Nitric acid in methanol solution at –20°C at an operating voltage of 12V. Observations were made in both the bright and the dark field imaging modes, and selected area electron diffraction (SAED) patterns were
recorded from areas of interest using an aperture of 1.1 μm nominal diameter. Qualitative EDXS was performed using an Oxford link model MK6 ultra-thin window EDX detector with focused electron beam on Philips CM20 microscope. The nominal beam diameter was 2 nm. Images were taken from longitudinal sections of the billets. The microstructure was studied in different areas of at least three foils for each condition and the quantitative results obtained (cell size, particle size, etc.) were averaged.

The dislocation density was calculated by measuring the total dislocation line length in a unit volume of crystal giving a parameter in terms of length (m/m$^3$). So, the dislocation density ($\rho$), is given by [9]:

$$\rho = \frac{2N_t}{L_t}$$  \hspace{1cm} (1)

where $N_t$ is the number of intersections with dislocations, $L$ is the length of random lines and $t$ is the foil thickness.

Tensile specimens with a gauge length of 7.5 mm, a gauge width of 1.5 mm and a thickness of 1.2 mm were cut parallel to the rolling direction. Room temperature tensile tests were performed on an Instron type machine at a strain rate of $10^{-3}$ s$^{-1}$.

3. RESULTS

3.1. Microstructure of the Al2024 alloy after cryorolling and recovery annealing

The microstructure from the cryorolled condition is characterized by a high average dislocation density ($\rho = 10^{15}$ m$^{-2}$) with partial formation of cells through dislocation rearrangement with low angle boundary misorientations. Two types of cells were observed: (I) equiaxed cells having a size within the range of 200-500 nm and (II) elongated cells with the average length of 1±0.5 μm and an average width of

Figure 1. The microstructure of the cryorolled Al2024 alloy.

Figure 2. The microstructure of the cryorolled Al2024 alloy annealed at 160°C for 2hr.
The thickness of cell walls was estimated as their maximum projected width. Their average thickness was 70 nm. Shear bands containing a very high dislocation density formed during cryorolling were also observed. Annealing at 160°C for 2 hr resulted in recovery of the microstructure (Figure 2). The average dislocation density within the grains decreased to $\rho = 10^{14}$ m$^{-2}$. The formation of small nuclei with the average size of 0.4±0.1 μm and high angle boundary misorientations with the matrix was also found (Figure 2b). The tangled cell walls were transformed into more regular dislocation networks and the number of dislocations in the cell interiors diminished. The average cell size remained similar to that observed after deformation. Two types of particles were found in the microstructure of the annealed Al2024 alloy: coarse rod-like particles having a length of 0.3±0.02 μm and a thickness of 0.1 ± 0.04 μm (Figure 3a) and small round particles with an average size of 0.1±0.04 μm (Figure 4a). The coarse rod-like particles are most probably Al-Cu-Mn particles (Figure 3b) [10]. The EDX analysis and analysis of the diffraction patterns of small round particles show the formation of S orthorhombic Al$_2$CuMg precipitates (Figure 4b) [10].

Figure 3. a) The microstructure of the cryorolled Al2024 alloy; b) the results of EDX analysis of the marked particle.

Figure 4. a) The microstructure of the cryorolled Al2024 alloy; b) the results of EDX analysis of the marked dispersoid.
3.2. Mechanical properties of the Al2024 alloy after cryo-rolling and recovery annealing

Figure 5a shows the true stress – strain curves of the solutionized (coarse-grained) Al2024 in the naturally aged condition before the cryorolling. The yield strength of the material is $\sigma_y \approx 250$ MPa, the value of uniform elongation is $\delta \approx 21\%$. The material after cryo-rolling demonstrates a marked increase in yield strength, $\sigma_y \approx 550$ MPa, whereas the value of uniform elongation dramatically drops to $\delta \approx 2.2\%$. The recovery annealing does not affect the flow stress (Figure 5b). However, the uniform elongation increases compared to that of the coarse-grained (solutionized) material to $\delta \approx 8\%$. It can be assumed that this significant increase in ductility without a sacrifice in strength was caused by two microstructural changes that occurred during recovery annealing:

1) the decreased dislocation density;
2) the precipitation of second phase precipitates.

The lower dislocation density after aging provides more room for dislocation activity whereas the second phase precipitates compensate the loss of strength due to the decreased dislocation density after recovery annealing.

The results of this work show that cryo-rolling has the potential to be used at an industrial scale for fabrication of ultra-fine structured materials. The method allows production of sheets of metals and alloys with ultra-fine structure that have an increased strength. The ductility of these metals and alloys can be controlled via recovery annealing.

4. CONCLUSIONS

1) An Al2024 alloy with an ultra-fine structure was successfully produced via cryo-rolling. The material had an increased yield strength of 550 MPa, but a very low ductility, $\delta \approx 2.2\%$.
2) Recovery annealing of the ultra-fine structured Al2024 results in a significant decrease in dislocation density and the formation of second phase precipitates. This results in an increase of ductility to $\delta \approx 8\%$ without a sacrifice in strength.

ACKNOWLEDGEMENTS

The authors would like to acknowledge gratefully the Australian Research Council for the financial support through the Centre of Excellence for Design in Light Metals and the Federation Fellowship awarded to PH. IS and MB would like to thank Deakin University for the funding through the Central Research Grant Scheme.

LIST OF REFERENCES


