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Hai-liang YU

University of Wollongong, hailiang@uow.edu.au

A Kiet Tieu

University of Wollongong, ktieu@uow.edu.au

Cheng Lu

University of Wollongong, chenglu@uow.edu.au

Xiang-hua Liu

Northeastern University China

Ajit Godbole

University of Wollongong, agodbole@uow.edu.au

See next page for additional authors

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Mechanical properties of Al-Mg-Si alloy sheets produced using asymmetric cryorolling and ageing treatment

Abstract

An asymmetric cryorolling technique was used to reduce the thickness of an Al-Mg-Si alloy sheet from 1.5 mm to 0.19 mm. The samples were subsequently aged for 48 h at 100 degrees celcius. The hardness and tensile strength of both rolled and aged sheets increased with the number of passes up to the sixth pass, but the tensile stress decreased after the seventh pass. Investigation of the microstructure of the sheets showed that the grain size after seven passes was about 235 nm and revealed the presence of Fe-Cr-Mn-Si particles in the samples. The deformation of Fe-Cr-Mn-Si particles and sheet thickness affects the ductility when the sheet thickness is less than 0.4 mm, and the strength when the thickness is less than 0.2 mm.

Keywords

si, produced, mg, sheets, al, properties, alloy, mechanical, asymmetric, cryorolling, ageing, treatment

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Authors

Hai-liang YU, A Kiet Tieu, Cheng Lu, Xiang-hua Liu, Ajit Godbole, and Charlie Kong

Mechanical properties of Al–Mg–Si alloy sheets produced using asymmetric cryorolling and ageing treatment

Hai-liang Yu^{a,b,*}, A. Kiet Tieu^a, Cheng Lu^a, Xiang-hua Liu^c, Ajit Godbole^a, Charlie Kong^d

^a School of Mechanical, Materials & Mechatronic Engineering, University of Wollongong, NSW 2500, Australia

^b School of Mechanical Engineering, Shenyang University, Shenyang 110044, China

^c State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110004, China

^d Electron Microscope Unit, University of New South Wales, Sydney, NSW 2250, Australia

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ABSTRACT

An asymmetric cryorolling technique was used to reduce the thickness of an Al–Mg–Si alloy sheet from 1.5 mm to 0.19 mm. The samples were subsequently aged for 48 h at 100 °C. The hardness and tensile strength of both rolled and aged sheets increased with the number of passes up to the sixth pass, but the tensile stress decreased after the seventh pass. Investigation of the microstructure of the sheets showed that the grain size after seven passes was about 235 nm and revealed the presence of Fe–Cr–Mn–Si particles in the samples. The deformation of Fe–Cr–Mn–Si particles and sheet thickness affects the ductility when the sheet thickness is less than 0.4 mm, and the strength when the thickness is less than 0.2 mm.

1. Introduction

Aluminum-based alloys, such as Al 6061, have been widely used as structural components in the automobile and aerospace industries because of their desirable attributes, such as high strength-to-mass ratio, easy formability, good weldability, high corrosion resistance and low cost compared to other materials. The properties of this type of material could be further improved by precipitation hardening with heat treatment, in which Mg and Si are the major solutes which increase the strength of the alloy by precipitation hardening.

In recent years, the production of materials with nano-sized grains by Severe Plastic Deformation (SPD) techniques has attracted much attention. This is due to improved physical and mechanical properties inherent to nano-structure materials. SPD techniques such as Accumulative Roll Bonding (ARB) [1–3], Dissimilar Channel Angular Pressing (DCAP) [4], High-Pressure Torsion (HPT) [5–7], Equal Channel Angular Pressing (ECAP)/Equal Channel Angular Extrusion (ECAE) [8–11], etc have been developed to fabricate bulk nano-structure or ultrafine grain samples from different metals. Among the currently available SPD techniques, only the ARB technique is appropriate for

production of continuous nanocrystalline and ultrafine grain strips and plates.

1.1. Cryorolling

Cryorolling can produce continuous and long specimens. This is potentially useful for large-scale industrial production of nano-structure materials. Cryorolling is a simple low-temperature rolling process in which the low temperature is maintained by liquid nitrogen. The processing also requires a relatively lower load to induce severe strain [12–18]. Wang et al. [12] found that when pure copper was processed by cryorolling, the matrix grains imparted high strength to the samples. The inhomogeneous microstructure was seen to induce strain hardening mechanisms, leading to a high elongation upto 30%. Cryorolling has also been identified as one of the potential routes to produce bulk ultrafine grain Al alloys [13]. The microstructure and mechanical properties of a precipitation-hardened Al–Cu alloy subjected to cryorolling, low temperature annealing and ageing treatments were studied by Rangaraju et al. [15], who produced ultrafine grain microstructure with improved tensile strength and good ductility. Panigrahi et al. [16] discovered that both the tensile strength and yield strength of an Al–Mg–Si alloy were considerably increased due to the suppression of dynamic recovery during cryorolling. The microstructural evolution of the above precipitation-hardenable alloy subjected to cryorolling at different strain levels was studied by Panigrahi and Jayaganthan [18]. The formation of

* Corresponding author at: University of Wollongong, School of Mechanical, Materials and Mechatronic Engineering, Wollongong, NSW 2500, Australia. Tel.: +86 0425055006.

E-mail addresses: hailiang@uow.edu.au, yuhailiang1980@tom.com (H.-I. Yu).

ultrafine grains in cryorolled Al alloys was found to be due to a higher density of dislocations and an effective suppression of the dynamic recovery during cryorolling. The severe strain induced at very low temperature facilitates the formation and retention of defects, which act as a driving force for the formation of sub-structures, and subsequently, ultrafine grains.

1.2. Asymmetric rolling

Asymmetric rolling is also a suitable technique to produce long specimens with refined grain size [19–29]. In the asymmetric rolling process, sheets are passed between rolls that either have different diameters, or rotate at different angular speeds. Asymmetric rolling has a significant potential for industrial applications because it requires a lower rolling pressure and torque, and yields a better product shape. It is also possible to obtain a quasi-uniform shear strain distribution through the thickness of the material under certain rolling conditions [19]. After subsequent annealing significant alterations in the texture and microstructure may lead to an improvement in the mechanical properties of the as-rolled and annealed strips [20]. The material is subjected to enhanced shear deformation, while develops high-angle grain boundaries, and ultrafine grains are formed by continuous recrystallization on annealing [21]. Ji and Park [22] found that the grains of magnesium alloy AZ 31 sheets were recrystallized and could be reduced to 3 μm in diameter by the asymmetric rolling process. Kim et al. [23] found the asymmetrical rolling process effective in enhancing the strength of oxygen-free-copper. Zou et al. [25] obtained a 500 nm grain-size pure aluminum sheet by asymmetric rolling. Wronski et al. [27] studied the grain refinement of an Al 6061 alloy by asymmetric warm-rolling. They developed fine grains with an average size of 1 μm in asymmetrically rolled strips with a thickness reduction of 91.8% at 300 °C. Thus the complete strain state imposed on the strip during asymmetric rolling is a combination of plane strain deformation and an additional shear component, which could refine the grains.

1.3. Asymmetric cryorolling

Recent developments in both cryorolling and asymmetric rolling processes have led to a renewed interest in the improvement of grain refinement in materials. However, there has not been much research into a technique that integrates features of both these processes. Yu et al. [30] used the asymmetric cryorolling technique to produce nano-structure Al 1050. They found that both the tensile strength and the ductility increased with a higher roll speed ratios. Haga et al. [31] found that the Al–Mg–Si alloy 6061 could be used for automobile body sheets. However, to date, there are no reports on studies of Al–Mg–Si alloys subjected to the asymmetric cryorolling process.

This paper describes the use of the asymmetric cryorolling process to produce Al 6061 alloy sheets. The mechanical properties of the sheet after each pass are analyzed for both rolled and aged samples. Finally, the authors present a discussion on the microstructure and the influence of the sheet thickness and the deformation of the secondary particles on the material performance.

2. Experiment and FE simulation

Commercial Al 6061 alloy sheets of dimensions 200 mm (length) $\times$ 60 mm (width) $\times$ 1.5 mm (thickness) were used. Before rolling, the samples were well tempered. Asymmetric cryorolling was carried out on a multi-function rolling mill, as shown in Fig. 1. The maximum rolling force is of 50 kN, and the

Fig. 1. Asymmetric cryorolling of Al 6061 alloy sheet.

Table 1
Schedule of asymmetric cryorolling.

Rolling pass, number	0	1	2	3	4	5	6	7
Strip thickness, mm	1.5	1.3	1.0	0.82	0.58	0.4	0.26	0.19

rolls of 120 mm diameter are independently driven by two 5.5 kW motors. The rolling speed ratio between the upper and lower rolls was set as 1.1. The thickness of the sheet was reduced from 1.5 mm to 0.19 mm after seven passes. The rolling schedule was presented in Table 1. After rolling, the rolled samples were aged at 100 °C for 48 h to obtain the best mechanical properties, as suggested by Kim and Wang [8].

Before rolling, the sheet was dipped into liquid nitrogen (−196 °C) for at least 8 min [13], and the temperature of the workpiece is required to be lower than −100 °C after each pass [12]. To estimate the temperature rise in the sheet during asymmetric cryorolling, a three-dimensional thermal-mechanical Finite Element (FE) model was set up in LS-DYNA. A range of rolling reduction ratios were used in the model [32]. In the FE models, the sheet and roll were meshed with eight-node hexahedral elements. There were 28,860 nodes and 18,576 elements in the model. During the rolling process, 90% of the mechanical work (plastic work due to deformation of the workpiece and friction work caused by friction between the roll and the workpiece) was converted into heat [33]. The initial temperatures of the sheet and the roll were respectively set at −196 °C and 20 °C. Fig. 2 shows the calculated temperature rise as a function of rolling reduction ratio. When the reduction ratio is less than 40%, the temperature rise caused by plastic and friction work is less than 50 °C. Therefore, the sheet temperature after rolling is expected to be lower than −100 °C with the rolling schedule in Table 1.

The tensile curve and the micro-hardness of samples were determined separately. For the tensile tests, both the rolled and aged samples were machined into ASTM specimens with 25 mm gauge length. Uniaxial tensile tests were conducted with an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ on an INSTRON machine operating at a constant speed. All tests were repeated three times. Subsequently, an FEI xT Nova Nanolab 200 Dualbeam workstation was used to prepare thin-foil specimens from the Al–Mg–Si alloy sheets for further TEM observation. The specimens were then placed on a standard carbon film Cu grid using an ex-situ lift-out method. A Philips CM200 Field Emission Gun Transmission Electron Microscope (FEG/TEM) equipped with a Bruker Energy Dispersive X-ray (EDAX) Spectroscopy system operating at an accelerating voltage of 200 kV was used to investigate the details of the microstructure.

3. Results

Fig. 3(a) shows the tensile curve of the rolled samples after each pass. The strength of the samples increases with the number of passes until the sixth pass, and decreases sharply after the seventh pass. It can be observed from Fig. 3(b) that the aged samples show similar trends in strength after each pass. The tensile stress of the samples is shown in Fig. 3(c), where it can be seen that the tensile stress of the aged samples is higher than that of rolled samples. The tensile stress of the rolled samples after the

first pass is 140 MPa, and it increases to 235 MPa after the sixth pass. The tensile stress of the aged samples after the sixth pass reaches 247 MPa. However, the tensile stress of both rolled and aged samples decreases after the seventh pass compared to that after the sixth pass. Fig. 3(d) shows the elongation of the samples after each pass. The ductility of the samples reduces with the number of passes. The ductility of the aged samples is better than that of the rolled samples. It is also observed that the increase in elongation reaches a maximum after the fourth pass and decreases for the fifth, sixth, and seventh passes.

The change in micro-hardness as a function of the number of passes is shown in Fig. 4. Before rolling, the initial micro-hardness is 35.3 kgf/mm². The hardness of the rolled samples increases with each pass. After the first pass, the hardness is 50.9 kgf/mm², and increases to 66.9 kgf/mm² after the fourth pass. The rate of increment becomes more gradual after subsequent passes. The micro-hardness of the samples after the seventh pass is 71.5 kgf/mm². The hardness of the aged samples is higher than that of the rolled samples. This phenomenon is also observed in Al-Mg-Si alloys subjected to the ECAP technique [8]. The ageing treatment is seen to improved the hardness of the rolled samples by 1.4, 4.4, and 6.2 kgf/mm² after the first, fourth, and seventh passes respectively.

Fig. 5 shows TEM images of the aged samples. The gradual emergence of the subgrain structure and grain refinement after subsequent passes is evident. After the seven pass, the mean grain size reaches 235 nm. Also seen are some secondary phase particles. After the first pass, the secondary phase particles are mainly square-shaped, which are elongated along the rolling direction in subsequent passes.

Fig. 2. Temperature rise in sample caused by plastic and friction work.

Fig. 3. Tensile test of rolled and aged samples: (a) tensile curve for rolled samples; (b) tensile curve for aged samples; (c) comparison of tensile stress between rolled and aged samples; (d) comparison of elongation between rolled and aged samples.

4. Discussion

4.1. Microstructure of sheets

Fig. 6 shows the grain size of Al 6061 alloy resulting from a number of SPD processes. After five cycles of the ARB technique (equivalent strain 4), the mean size of the grains was seen to be about 230–240 nm [2,3]. When using the DCAP technique, the majority of subgrains or dislocation cells were observed in the interiors with the range size of 50–300 nm after five passes (equivalent strain 3.0) [4].

Fig. 4. Micro-hardness of samples before rolling, after rolling, and after ageing.

Loucif [5,6] and Moreno-Valle et al. [7] used the HPT technique to fabricate ultrafine grains of Al 6061 alloy. The grain size was about 500 nm at room temperature (equivalent strain 4) [6], and reached 170 nm at cryogenic temperature [7]. When using the ECAP/ECAE technique, the grain size is in the range of 200–500 nm after four passes (equivalent strain 3.9) when the angle of intersection (Φ) between the two parts of the channel is 100° [9], and the grain size reduces from 80 μm to 710 nm after eight passes (equivalent strain 8) when the angle Φ is 90° [10,11]. The HPT, DCAP, and ECAP/ECAE techniques can bring about more pronounced grain size refinement.

Fig. 6. Grain size of Al 6061 alloy after different SPD techniques.

Fig. 5. TEM image of aged samples after the (a) first, (b) third, (c) fifth, and (d) seventh pass.

Fig. 7. Precipitation in aged sample after the seventh pass.

The ECAP/ECAE and DECAP techniques are capable of producing relatively large bulk samples, while the HPT technique is suitable for small disk-shape samples. However, these three techniques are not suitable for continuous sheet production. Samples produced using ARB and asymmetric cryorolling have similar grain size as the above techniques. However, the ARB process has some limitations. The surface finish of the product and edge crack issues are more difficult to control in the ARB technique although it is suitable for continuous sheet production [2]. In addition, the nitrogen employed in asymmetric cryorolling is a natural gas that will not result in any environmental pollution. Thus, asymmetric cryorolling appears to combine the advantages of asymmetric rolling and cryorolling in one integrated process.

4.2. Strength of as-rolled and aged sheets

The dislocation density in Al sheets increases with a larger deformation due to an effective suppression of dynamic recovery at cryogenic temperatures. Therefore, during asymmetric cryorolling, the hardness (Fig. 4) and tensile strength increase significantly [18]. During ageing, annihilation of dislocations and formation of strain-free grains reduces the strength, while the formation of nanosized precipitates increases the strength [34–37]. The cumulative effect is that all aged samples have better properties compared to as-rolled samples, as shown in Figs. 3 and 4. This is similar to the results by Kim and Wang [8]. Fig. 7 shows the precipitation in the aged sample after the seventh pass. The precipitation sequence is seen to change in the presence of dislocations, with most precipitates in the deformed material being of the post- β'' type [37].

The hardness and the strength of samples are linearly related and thus the hardness is expected to show a similar trend as the tensile strength. As seen in Fig. 4, the sample hardness increases with the number of passes. However, Fig. 3(c) shows that the sample strength increases after each successive pass until the sixth pass, and they reduce after the seventh pass. After the seventh pass, the tensile strength of both as-rolled and aged samples decreases compared to that after the sixth pass. This anomalous result is important for material design from the point of view of the optimization of material deformation. Many secondary phase particles in the samples are observed in Fig. 5. The chemical composition of the secondary phase particle appears to be Fe–Cr–Mn–Si in Fig. 8. Maisonnelle et al. [40] also found a similar size of intermetallics in Al–Mg–Si alloys. Although these intermetallics do not contribute to the hardening of the alloy during the elongation of the material, the particles can be broken into smaller parts during the plastic deformation process [41]. Either because of

an intrinsic brittleness or because of a lack of coherence with the matrix, these particles will result in a damage initiation owing to the nucleation of small internal voids, which develop into cracks when exposed to stresses [38,39]. The plate-like particles can lead to a crack initiation and induce surface defects in the deformed material [42]. In these sheets, it is possible that the deformation of Fe–Cr–Mn–Si particles results in some micro-defects in sheets with increasing the number of passes, as shown in Fig. 9, which results in a reduction in the sample strength. The size of secondary phase particles in Fig. 9(b) is much smaller than that in Fig. 9(a). During rolling, the void initially occurs around larger-size secondary phase particles. At a higher number of passes, the voids occur around smaller size secondary phase particles. In Fig. 9(a) and (c), voids of much larger size are seen around similar size particles after the seventh pass compared to that after the sixth pass. It is thus possible to conclude that the void size should also increase with the number of passes.

With a reduction in the sheet thickness, the grains on the free surface are less constrained and more easily deformed at a substantially lower flow stress than in the case of the bulk state [43,44]. Kals et al. [45] expressed the volume fraction α of grains having a free surface, as shown in Eq. (1).

$$\alpha = 1 - \frac{(w-2d)(t-2d)}{wt} \quad (1)$$

where w – specimen width, t – specimen thickness, d – average grain size.

As the thickness decreases, the relative surface area per volume of a specimen increases. That is to say, when α decreases, the effect of the surface on the mechanical properties is enhanced. Suh et al. [46] found that the tensile strengths of Al 6K21-T4 sheets decreased almost linearly with thickness reduction when the thickness was reduced below a critical value.

The previous discussion on the effect of different parameters on the material strength, such as deformation of secondary phase particles, sheet thickness, precipitation and reduction, can be expressed in the form of Eq. (2), which considers all these factors together.

$$\Delta\sigma_y = \beta_1(\Delta\sigma_{ss} + \Delta\sigma_d + \Delta\sigma_p - \Delta\sigma_{dp}) \quad (2)$$

where $\Delta\sigma_y$ is the change of total yield strength, $\Delta\sigma_{ss}$ the strength increase due to solid solution, $\Delta\sigma_d$ due to strength hardening, $\Delta\sigma_p$ due to precipitation and $\Delta\sigma_{dp}$ the strength reduction due to the deformation of secondary phase particles; β_1 the coefficient describing the effect of thickness on strength.

Eq. (2) is based on Ref. [37], where the first three terms on the right hand side $\Delta\sigma_{ss}$, $\Delta\sigma_d$ and $\Delta\sigma_p$ have been proposed. The additional terms $\Delta\sigma_{dp}$ and the coefficient β_1 are introduced here to be compatible with the previous discussion.

4.3. Ductility of as-rolled and aged sheets

As shown in Fig. 3(d), the ductility of all rolled and aged samples reduces with the number of passes. The ductility of the aged samples is higher than the rolled samples. During the ageing of Al 6061 alloy samples, a significant number of dislocations are annihilated. The annihilation of dislocations resulting from the ageing treatment facilitates further accumulation of dislocations, resulting in a significant improvement in ductility. Nanosized precipitates are formed as a result of the ageing treatment, and the dislocations accumulate and surround the nanosized precipitates during a tensile straining. Due to a high density of precipitates, a higher number of dislocations is accumulated, which results in an enhanced ductility.

It is interesting to note that through the ageing treatment, the ductility improvement increases with the number of passes until

a

b

the fourth pass, and decreases gradually with subsequent passes. Generally, with more passes, the dislocation density will increase. At the same time, during ageing the number of annihilated dislocations will increase, which will result in a greater ductility improvement of the aged samples. The reduction in ductility improvement may be caused by micro-defects around the Fe–Cr–Mn–Si particles, as shown in Fig. 9. During the production of Al–Mg–Si alloy sheets, it is important to control the size and shape of Fe–Cr–Mn–Si particles. Previous research [47] has shown that micro-defects may appear around particles during cold rolling.

Similar to Eq. (2), Eq. (3) is proposed to express the failure strain (ε_F) in terms of its components,

$$\varepsilon_F = \beta_2(\varepsilon_{df} + \Delta\varepsilon_{ad} + \Delta\varepsilon_p - \Delta\varepsilon_{dp}) \quad (3)$$

Where ε_{df} is the strain due to rolling deformation, $\Delta\varepsilon_{ad}$ the increase of strain due to annihilation of dislocation, $\Delta\varepsilon_p$ due to precipitation, $\Delta\varepsilon_{dp}$ the strain reduction due to deformation of secondary phase particles, and the coefficient β_2 accounts for the effect of thickness on ductility.

5. Conclusions

In summary, the asymmetric cryorolling is proposed as an advanced manufacturing technique that can be used to produce long and continuous sheets.

- (1) Al 6061 alloy sheets were produced, with reduction in thickness from 1.5 mm to 0.19 mm using the asymmetric cryorolling technique through seven passes. After the seventh pass, the mean grain size was refined to 235 nm.
- (2) The tensile strength of both rolled and aged sheets was found to increase with the number of passes until the sixth pass, and to reduce sharply after the seventh pass. The strength of sheet could be expressed in terms of the total yield strength as in Eq. (2).
- (3) The increase in hardness and strength of samples as a function of the number of pass except for the seventh pass may be caused by the thickness effect and the micro-defects surrounding the secondary phase particles.
- (4) The ductility of both rolled and aged samples decreases with the number of passes. The ductility of aged samples is better than that of rolled samples. The effect of the sheet thickness and the deformation of second phase particles on the ductility of the samples become obvious when the sheet thickness is less than 0.4 mm.

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