Influence of strain rate on microstructures and mechanical properties of 2524Al alloy fabricated by a novel large strain rolling

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Abstract

2524Al alloy sheets with excellent strength–ductility balance were successfully fabricated by large strain rolling in a strain rate range from 5 s\(^{-1}\) to 20 s\(^{-1}\) at 400 °C. The microstructural evolution, mechanical properties and fracture mechanism of the as-rolled sheets were investigated. The results show that there are numerous coarse precipitates of Al\(_2\)CuMg and Al\(_2\)Cu along the dendrite grain boundaries for as-cast 2524Al alloy. Most of the precipitates disappear after homogenizing annealing treatment. Grain refining plays a key role in the as-rolled microstructural evolution and the mechanical properties. The dendrite grain were broken and become streamline structure after rolled. The sheet fabricated at 10 s\(^{-1}\) exhibits a higher strength and excellent ductility, with the ultimate tensile strength and elongation of 611 MPa and 10.5%, respectively.

1. Introduction

Al–Cu–Mg alloy is a commercial alloy widely used in aerospace and automotive industries principally due to its advantages of excellent heat-resistance, high specific strength, good fatigue resistance and fracture toughness [1–3]. A generation of 2524 T3 sheets have been used as the skin sheets in Boeing and Airbus aircraft since the 1990s [4]. With the development of aerospace industry, we should research novel plastic deformation technology to improve the mechanical properties of Al–Cu–Mg alloy.

It is well known that grain refinement is one of the most effective methods to achieve both high strength and good ductility. Various methods for severe plastic deformation, such as accumulative roll-bonding (ARB) [5], cyclic extrusion compression (CEC) [6], equal-channel angular pressing (ECAP) [7–9], and large strain rolling [10, 11], have been used to fabricate ultra-fine grained aluminum alloy. Among these methods, large strain rolling, which involves rolling the metallic materials with a reduction rate above 50% in a single pass, is an attractive and cost-effective method to improve the mechanical properties of aluminum alloy. So far, large strain rolling has been widely used for obtaining magnesium alloy. The typical work is reported by Yan and Zhu et al. [12–14], they develop a novel high strain-rate rolling technology and investigate some series magnesium alloy, such as Mg–Al–Zn–Mn [12] and Mg–Zn–Mn [14] alloy. Their investigations have mainly focused on the effect of high strain-rate rolling on grain refinement and subsequent mechanical properties. However, compared with magnesium, little work has been done on high strain-rate rolling in aluminum alloy. Mohammad Reza Jandaghi et al. studied the severe plastic deformation has a great influence on the microstructure and properties of Mg–Mn–Si alloy. Severe strain has a highly effectiveness on particles refinement, and fine particles may get chance to be involved in dimple formation, severe strain also lead to the occurrence of recrystallization, reduce the grain size and the dislocation density, these factors make ductile fracture easier to occur [15]. They also found that severe plastic deformation can habit pitting because of random distribution of intermetallic precipitates beside their fragmentation under heavy imposed strains and impressive they were insufficient size to be impress as cathodic sites. At the same time, the occurrence of dynamic recrystallization results in lattice expansion (increased d-spacing). The increase in d-spacing improves the conductivity of Al–Mn–Si alloy [16, 17]. Zhao
Shiteng et al. found that severe plastic deformation can be attributed to larger mobile and forest dislocation densities as well as an obviously fined grain size, these factors intensified the dynamic effect aging [18]. Zhang peng et al. compared the effects of traditional rolling and high-strain rolling on 5A12, and found that when the total hot rolling deformation is 70%, obvious dynamic recrystallization can occur in the large strain rolled sheet, and the traditional multi-pass hot-rolled sheet still maintains elongated fibrous structure. At the same time, they also found that aging softening phenomenon can be effectively inhibited by large rolling process [19]. Therefore, it can be obtained that severe strain has more advantages than traditional rolling.

There are a large amount of defects for as-cast Al–Cu–Mg alloy, such as pores and oxide, which will affect its deformation behavior. Crack is the common problem during hot rolling, especially in high strain-rate rolling. In our previous investigations, we amend the rolling process by applying a pre-plastic deformation, together with intermediate solution, aging treatment and cold rolling, namely large strain rolling [20]. As well known, microstructural change during plastic deformation can be controlled by deformation mode, strain pass, temperature, strain rate, etc.

In this study, a novel large strain rolling technique was carried out preparing 2524 aluminum alloy plate to improve its strength, the influence of strain rate on microstructures and ambient mechanical properties of 2524 aluminum alloy was investigated systematically.

2. Materials and experimental

The 2524Al alloy with the chemical composition of Al-4.5Cu-1.5Mg-0.6Mn-0.2Ti (mass fraction, %) was prepared by casting process in this study. The ingots were homogenized at 485 °C for 36 h, then rolled into plates from 14 mm to 2 mm by three passes in a strain rate range from 5 s\(^{-1}\) to 20 s\(^{-1}\), and the total reduction rate of about 85.7%. During hot rolling, the alloy was firstly rolled at 400 °C from 14 mm to 10 mm, then from 10 mm to ~4 mm, accompanied by solution at 495 °C for 1 h and pre-aging at 190 °C for 6 h. Finally, the plate with an initial thickness of 4 mm was cold-rolled with reduction rate of 50% (to 2 mm) and finally aged at 150 °C for 4 h.

Corrosion of 2524Al alloy with mixed acid solution (1% HF + 1.5% HCl + 2.5% HNO\(_3\) + 95% H\(_2\)O) and Metallographic analysis was performed using a DMI3000M optical microscope. Disks of 3 mm diameter were punched from ~80 μm thick sheet and thin foil specimens for TEM by electropolishing on MTP-1 twin-jet electropolishing device, the parameters are as follows that the voltage is 20 V, the temperature is controlled between −30 °C and −25 °C, and the electrolyte is 30% HNO\(_3\) + 70% CH\(_3\)OH solution, then the characteristics of the as-rolled precipitates were observed on a TecnaiG2 F20 transmission electron microscopy (TEM). The flow behaviors were measured by an ETM105D electronic universal testing machine in the rolling plane along the rolling direction with a strain rate of 2 mm min\(^{-1}\) at room temperature. The tensile fracture morphology was examined with SIGMA SEM.

3. Results and discussion

3.1. Microstructures of as-homogenization

Figure 1 shows the microstructures of as-cast and as-homogenized 2524Al alloy. It can be seen from figures 1(a) and (c) that the irregular dendrite grain size of as-cast 2524Al alloy is coarse, and there are numerous massive precipitates which mainly distribute continuously along the grain boundaries. From the results of EDS (figure 1(d) and) and XRD pattern (figure 2) we can confirm that these massive secondary phase are Al\(_2\)CuMg and Al\(_3\)Cu. After the homogenization treatment 485 °C for 36 h, the grains grow a little up to 102 μm and nearly all small precipitates dissolve into α-Al matrix. Thus, we can observe a lot of small pits in the as-homogenized 2524Al alloy (figure 1(b)). However, there are still some large precipitates exist.

3.2. As-rolled microstructure evolution

Microstructures of the 2524Al alloy sheets rolled to 4 mm at different strain rates are shown in figure 3. It can be seen that the 2524Al alloy possesses a highly elongated band structure characteristic along the rolling direction, and there are a large amount of constituent phases distributed along the grain boundaries. When the strain rate is 5 s\(^{-1}\), the grain size is refined compared to as-homogenization (figure 3(a)), the grain size is about 95 μm. The initial coarse grains are elongated under the huge stored energy and with fuzzy and discontinuous grain boundaries. When the strain rate is up to 10 s\(^{-1}\), we can find that the grain size become finer, the grain size is reduced to approximately 86 μm and their shapes are inclined to equiaxed crystal and a few small grains nucleated at the original grain boundaries (figure 3(b)), which may be attributable to the dynamic recrystallization caused by the larger stored energy during large strain rolling. Further increasing the strain rate to 15 s\(^{-1}\) and 20 s\(^{-1}\), the deformation degree of grains increased and strip-like deformation bands occurred, but grain size did not change obviously, the grain size is about 87 μm and 86 μm, respectively. However, we observe a
large amount of fine precipitates along the grain boundary. As been reported [20, 21], dynamic precipitation can easily occur in aluminum alloy during severe plastic deformation. To determine the dynamic precipitates developed during large strain rolling process, the SEM images of the alloy for $\varepsilon = 10 \text{ s}^{-1}$ and $\varepsilon = 20 \text{ s}^{-1}$ are shown in figure 4. It can be seen that the size of the dynamic precipitates is much smaller than that of coarse undissolved second phases and mainly distributed at the deformation bands. The EDS result confirmed that this high-temperature precipitates contain Mg, Cu, Al elements (shown by the white arrow), and some investigations indicate that when the ratio of Cu/Mg is between 8 and 4, the main precipitate are $\theta(\text{Al}_{2}\text{Cu})$ and $\text{S(Al}_{2}\text{CuMg})$ phases. When the ratio of Cu/Mg ranges from 4 to 1.5, the main precipitate is $\text{S(Al}_{2}\text{CuMg})$ phase. For 2524
aluminum alloy, the contents of Cu and Mg are 4.5% and 1.5% (wt%), respectively. So, the ratio of Cu/Mg is about 3, and Combining with XRD pattern (figure 5), it can be concluded that the main precipitate is $\text{S(Al}_2\text{CuMg)}$ phase [22] during rolling and aging. Dynamic precipitates in aluminum alloy play a significant effect on aging precipitation of the alloy [21], so dynamic precipitation and heat treatment should be comprehensively considered.

The microstructural changes of 2524Al alloy processed by large strain rolling under various strain rates accompanied by solution at 495 °C for 1h and pre-aging at 190 °C for 6 h is investigated (as shown in figure 6). The increase of strain rate leads to the variation of the degree of recrystallization and grain size of the 2524 aluminum alloy. It can be seen from figures 6(a) and (b) that only a few recrystallized grains formed along the original grain boundaries when the strain rate is below 10 s$^{-1}$, which indicates the rolling structure is very stable. Further when the strain rate is increased to 15 s$^{-1}$ and 20 s$^{-1}$, the recrystallization is obvious and the grain size
increases. This difference in the microstructures is attributed to the higher internal energy caused by increasing strain rate. The stability of deformation structures will decrease with the increasing of internal energy. Thus, the recrystallized grains will grow during subsequent heat treatment, and when the rolling rate is increased from 5 s\(^{-1}\) to 20 s\(^{-1}\), the grain size is 72 \(\mu\)m, 47 \(\mu\)m, 40 \(\mu\)m, and 48 \(\mu\)m, respectively. These grain size are determined by the degree of recrystallization and grain growth. Comparing with figure 4, we can also find nearly all of the phases along the grain boundaries have disappeared, the phases dissolve into aluminum matrix during solution treatment. The \(\text{Al}_2\text{CuMg}\) precipitates will precipitate in the inner grains during the pre-aging at 190 °C for 6 h.

Figure 5. XRD pattern of 2524Al alloy processed by large strain rolling with strain rate of (a) \(\varepsilon = 10\) s\(^{-1}\), (b) \(\varepsilon = 20\) s\(^{-1}\).

Figure 6. Optical micrographs of 2524Al alloy processed by large strain rolling with various strain rate after 495 °C/1 h + 190 °C/6 h heat treatment: (a) \(\varepsilon = 5\) s\(^{-1}\), (b) \(\varepsilon = 10\) s\(^{-1}\), (c) \(\varepsilon = 15\) s\(^{-1}\), (d) \(\varepsilon = 20\) s\(^{-1}\).
Figures 7–9 show the microstructures of 2524Al alloy processed by cold rolling and consequent final aging at 150 °C for 4 h with various strain rates, respectively. It can be seen that streamline is more obvious after cold rolling, the grain size unchanged after ageing. The lamellar spacing of the elongation bands is narrower and more uniform when $\varepsilon = 10$ s$^{-1}$. We can also find a large amount of similar twinning structures for the three conditions (as shown in figures 7 and 8), as is never been reported by any researchers. We deduced the similar twinning structure is caused by the severe shear energy under the condition of cold rolling with a reduction rate of 50%. And these structures will not disappear during the sequent aging treatment and affect its mechanical properties significantly. Further observations by TEM show the equiaxid grains are ultra-fine within 1 μm, and there are numerous rod-like Al$_2$CuMg phases (figure 9(a)), together with a typical dislocation cell structure (figure 9(b)). These ultra-fine grains and dislocation may cause the substantial increase of mechanical properties.

### 3.3. Mechanical properties and fracture mechanism

Figure 10 shows the tensile properties of 2524Al alloy processed by large strain rolling with various strain rate. As we can see that the mechanical properties of 2524Al alloy are substantially improved with the increasing of strain rate. The sheet fabricated at strain rate of 10 s$^{-1}$ exhibits a higher strength and excellent ductility, with the ultimate tensile strength and elongation of 611 MPa and 8.7%, respectively. Microstructural evolution of the composite during the pressing can be used to rationalise the results. In fact, the phases and grains are the two main interactional structural features for 2524Al alloy. From figures 1–8, we know rolling can substantially decrease the defects, and more importantly, refine the grains and obtain homogenous and fine fibrous structures. At the same time, numerous Al$_2$CuMg phases precipitate along the grain boundary and within the inner grains. All of these factors can increase ultimate tensile strength and elongation of 2524Al alloy. To understand the fracture behavior, figure 10 shows SEM of the fracture surfaces of 2524Al alloy fabricated at strain rate of 10 s$^{-1}$. The fracture surfaces bear evidence of ductile fracture and with a mass of fine dimples. A lot of fracture edges approximately parallel to each other can be observed (shown by red lines and arrows in figure 11(a)), as has been reported [23], these fracture edges can cause the tenacity of to be remarkably promoted and mainly caused by the motions of dislocation. Further observation, there are some broken Al$_2$CuMg phases with a smooth fracture surface at the bottom of fracture edges (shown by arrows in figures 11(b) and (c)).
size is $\sim 4$ $\mu$m. There still a little finer broken AlCuMn phases with the length of $\sim 1.8$ $\mu$m at the bottom of dimples (shown by arrows in figure 11(d)), which are insoluble phases and almost no effect on properties of aluminum alloy [24]. Another significant characteristic is that the Al$_2$CuMg phases will debond with aluminum matrix under tensile load (figure 11(c)), and this may be one of the factors leading to the fracture of 2524Al alloy.

4. Conclusions

(1) When the 2524Al alloy is rolled to 4 mm at different rates, the grain boundaries are elongated, and recrystallization occurs at some grain boundaries. At the same time, the rate has a greater impact on the microstructure. As the rate increases from $5$ s$^{-1}$ to $10$ s$^{-1}$, the grain size becomes smaller.
changes from 10 s\(^{-1}\) to 20 s\(^{-1}\), the grain size does not change significantly. At the rates of 15 s\(^{-1}\) and 20 s\(^{-1}\), a large amount of fine Al\(_2\)CuMg phase precipitated at the grain boundaries.

(2) When the 2524Al alloy after the solution and pre-aging of the 4 mm plate, the second phase becomes relatively diffuse, and at the same time, each rolling rate has a very large effect on the microstructure of the solution and pre-aging. As the rolling rate increases, the degree of recrystallization increases and accompanying growth of recrystallized grains.

Figure 10. Mechanical properties of 2524Al alloy.

Figure 11. Scanning electron micrograph of the fracture surface of 2524Al alloy fabricated at strain rate of 10 s\(^{-1}\).
(3) When the 2524Al alloy after cold rolling, the grains become streamlined and a large number of twin structure appear, and these structures will not disappear during the subsequent aging treatment. When the rate is 10 s⁻¹, the lamellar spacing of the elongated band is narrower and more uniform. The ultrafine equiaxed crystals within 1 μm, many rod-like Al2CuMg phases and typical dislocation unit structures are are observed by TEM, at the same time, combined with grain refinement and second phase dispersion distribution make The tensile strength of 10 s⁻¹ reaches 611 MPa.

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