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The effect of annealing temperature on the recrystallization and mechanical properties of severe plastic deformed commercial pure aluminium during ultra-fast annealing

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Abstract

In the research a commercial pure aluminium was cold rolled by 98% accumulative severe plastic deformation and then ultra-fast annealed at 350 °C–520 °C with the 1000 °C/s heating rate and 1.0 s holding time. The microstructure evolution and the mechanisms of the recovery and recrystallization for the above ultra-fast annealed pure aluminium were analyzed by Gleeble 3500 thermal simulation system, electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). For the ultra-fast annealing pure aluminium, the grain size increases from 2.05 μm to 17.10 μm with the increase of annealing temperature from 410 °C to 520 °C; the tensile strength of the annealed pure aluminium decreases from 116.48 MPa to 53.43 MPa, and the uniform elongation increases from 1.20% to 39.78%. When annealing at 410 °C, the storage energy was transformed into the driving force for grain nucleation, which greatly refined the grains. When annealing at 380 °C–410 °C, the pure aluminium is in the stage of recrystallization, and the average grain size refined to 2.05 μm. When annealing at 435 °C, the number of small-angle grain boundaries (<15°) was significantly reduced and the number of large-angle grain boundaries increased. When the annealing temperature increased to 470 °C or 520 °C, the crystalline grains had merged each other, leading to the grain size grow to 17.10 μm. The annealing temperature of ultra-fast annealing should be in the range of 410 °C to 435 °C for optimizing the mechanical performances of commercial pure aluminum.

1. Introduction

Pure aluminium and aluminium alloys are nowadays used in a variety of fields, including aircraft [1], ship construction [2], and packaging [3], mainly due to their low material densities, good elongation performances, and high corrosion resistance. Commercial pure aluminium shows such good process ability that it can be transformed into double zero foils, which have been widely used in the field of renewable energy, such as capacitors [4], lithium-ion batteries [5], and photocatalysis, and so on [6].

Severe plastic deformations (SPD) can improve both the strength and plasticity of aluminium and aluminium alloys. There are various SPD techniques, mainly including equal-channel angular pressing (ECAP) [7–9], high-pressure torsion (HPT) [10], accumulative roll-bonding (ARB) [11, 12], cyclic extrusion-compression (CEC) [13–15], multi-directional forging (MDF) [16, 17] and rolling, etc. ECAP and HPT techniques are limited by the sample size. The diameter of the prepared sample is generally not larger than 20mm, which decreases the possibility for industrialization. CEC technology deforms in a closed cavity, with dual functions of extrusion and upsetting. The length of sample should not be too long due to the limitation of die mechanism, which also limits its industrial production. ARB can obtain relatively large reduction in theory, break through the limit of traditional rolling reduction, and can continuously prepare sheet ultra-fine grained
metallic materials. MDF and rolling originate from traditional forging and rolling, respectively. They are mature, efficient and the preparation technologies are easy to learn, comparing with ARB. The use of existing forging or rolling equipments can produce large-sized block or sheet metals of fine or ultra-fine grain, which are most suitable for industrialization.

In terms of the industrialization annealing process, the annealing heating rate is generally controlled at 30–50 °C/h [18], and the rate of keep continuous heating is in the range 5–10 °C/s [19] (sometimes reaching 25 °C/s [20]). The low annealing heating rate has seriously restricted the production efficiency. In order to improve the efficiency, the ways of faster heating rate, such as resistance heating, induction heating [21, 22], and cold plasma discharge heating [23], has attracted wide attention. When the heating rate increases up to several thousand degrees Celsius per second, the ultra-high rate heat treatment is referred to as ‘ultra-fast annealing’ [24]. The advanced technology not only greatly reduces the heating and cooling time, but also affects the recovery, recrystallisation, and grain growth mechanism of the metal [25, 26].

The research on ultra-fast annealing mainly focuses on iron and steel and generally focuses on the influence of the heating rate on the recrystallization. Muljono [27] showed that the recrystallization temperature of low carbon steel, and ultra-low carbon steel, will increase with the increase of the heating rate. Kesten [19] showed that the recrystallization temperature of IF steel-containing Ti will increase with the increase of the heating rate. It was also shown that the grain refinement will not occur when the heating rate is larger than 1000 °C/s. Hou Ziyong et al. [21] found that the micro-hardness of the Nb-IF steel will decrease continuously with the increase of the annealing temperature and within 500 °C ~ 700 °C, the recrystallization trends for low and high heating rates were identical. However when the heating rate was about 1500 °C/s, Petrov Roumen et al. [28] found the hardness first increase, and then decrease, with the increase of the annealing temperature for a cold-rolled high-strength steel containing 0.11% C and 2.07% Mn. Moreover, with the heating rate increasing from 140 °C/s to 1500 °C/s, the grain refines from 5 μm to 1 μm in diameter, and the final grain size depends on the reheating temperature and rate. Ferry [22] showed that ultra-fast annealing can reduce the recrystallization temperature of Al-Si alloys and shorten the recrystallization temperature range. In addition, the recrystallization grains will get coarser, which was called recrystallization softening. Atallah [29] indicated that an increased heating rate will decrease the recrystallization temperature of Al-Mg alloys. Therefore, ultra-fast annealing has the capability to both refine the recrystallization grain and change the recrystallization temperature. However, there are still disputes concerning the effects of deformation and critical heating rate on recrystallization and grain refinement, especially the mechanism of grain refinement for ultra-fast annealing is uncertain for aluminium and aluminium alloys.

In this research, the commercial pure aluminium sheets with 98% deformation have been obtained by using traditional rolling technique to storage enough deformation energy. After the above severe plastic deformed pure aluminium is annealed at 350 °C to 520 °C with 1000 °C/s ultra-fast heating rate, the accumulated deformation energy can be transformed into the driving force of recrystallization. Then the effect of the ultra-fast annealing temperature (from 350 °C to 520 °C) on the grain refinement and mechanical properties of pure aluminium with a 98% cold rolling deformation can be obtained. Also the mechanisms of recovery and recrystallization can be systematically analyzed without considering ultra-fast heating rate, deformation and alloying elements in the present study.

2. Materials and methods

As-cast commercial pure aluminium of 49 mm thickness was annealed in a muffle furnace at 380 °C for 2 h to transform into the O state. The chemical composition was listed in table 1. Then the samples were multiple passes cold-rolled to sheets of 1.05 mm thickness, with the total cold-rolling of 98%. The cold-rolled aluminium sheet was wire-cut into strip of 150 × 20 × 1.05 mm³ (rolling direction RD × transverse direction TD × normal direction ND). The microstructure and sampling direction of the sheet were shown in figure 1.

The ultra-fast annealing processes are shown in figure 2. The annealing parameters are as follows: annealing temperatures = 350 °C, 380 °C, 410 °C, 435 °C, 470 °C, and 520 °C; heating rate = 1000 °C/s; holding time at the annealing temperature = 1.0s, and cooling rate = 40 °C/s (air-cooling). Considering the effect of temperature overshoot under 1000 °C/s heating rate, the programmed temperature is 8 ~ 10 °C lower than the experimental temperature by program setting of Gleeble-3500 system. The annealing was carried out using a

| Table 1. Chemical composition of commercial pure aluminium (wt. %). |
|---------------------|-----|-----|-----|-----|-----|-----|-----|-----|
| Al             | Si  | Fe  | Mn  | Zn  | V   | Ti  | Cu  | Mg  |
| 99.50          | 0.25| 0.4 | 0.03| 0.05| 0.05| 0.03| 0.05| 0.03|
Gleeble-3500 thermo-mechanical simulator. The free span was set to 50 mm and copper grips were used to obtain 25 ~ 30 mm uniform temperature zones to meet the tensile tests. The temperature difference within the gauge length did not exceed ±1°.

The cold-rolled samples were cut through RD, mounted, mechanically polished, and electropolished using an electrolyte containing 10% perchloric acid + 90% ethanol solution, operating at a voltage of 25V. The anode and cathode were an aluminium alloy and a stainless steel sheet, respectively. The microstructure was characterized using electron backscatter diffraction (EBSD) on a JEOL JSM-7001F field emission gun scanning electron microscope (FEG SEM), equipped with an HKL EBSD detector. The accelerating voltage and canning step were 15KV and 0.5 μm, respectively. The samples for JEM-2010 transmission electron microscope (TEM) characterization were cut perpendicular to the cylinder axis at the central cross-section and 3 mm TEM discs were prepared by an MTP-A twin-jet electro-polishing in a solution of 10% perchloric acid in ethanol. TEM was operated at a voltage of 200 kV. The grain size was calculated based on the standard ASTM E112.

The tensile samples (with a gauge length of 25 mm and thickness of 1.05 mm) were cut from the ultra-fast annealed samples. The uniaxial tensile testing was carried out using INSTRON-8801 at a constant cross-head speed of 2mm min⁻¹.

3. Results

3.1. Influence of ultra-fast annealing temperature on the microstructure

Figure 3 shows the distribution of EBSD crystal orientation for the pure aluminium annealing at different temperature. At 350 °C, there was just a small change in the microstructure and the elongated fibers that were
formed during the cold-rolling were still maintained. At 410 °C, the pure aluminium still retained its fibrous microstructure, while new and finer recrystallization grains had appeared in large quantities. They positioned at the edges of, or inside, the deformed tissue, and some recrystallization grains had even begun to grow. At 435 °C, the fibrous microstructure had disappeared completely and was replaced by equiaxial grains. Therefore the critical annealing temperature for full recrystallization should be between 410 °C - 435 °C. When the annealing temperatures are 470 °C and 520 °C, the new grains had gradually merged and also continued to grow in a lateral direction. Figure 3(f) lists the corresponding grain sizes. When the annealing temperature increases form 410 °C to 520 °C, the average grain size increases from 2.05 μm to 17.10 μm.

Figures 4 and 5 show the TEM microstructure of the pure aluminium after ultra-fast annealing at different temperatures. After the pure aluminium had undergone severe plastic deformed, but prior to the annealing, a large number of dislocations accumulated in the local areas, and entangle each other. Some of these dislocations also lined up to form many small-sized cellular substructures. When the pure aluminium was annealed at 410 °C, this low temperature, in addition to the extremely short recovery time, was not enough to decrease the dislocation density (which was still high). Only a small part of the dislocations (as movements, rearrangements, and combinations) has moved to the grain boundaries. The dislocation walls (DW) were also observed to become separated from the dislocation pile-up (which transforms the polygonal sub-cristalline and deforms the cell structure that was initially formed by cold-rolling into a typical sub-cristalline microstructure). Furthermore, a large number of dislocations were still accumulated in the grain boundaries. Figure 5 shows that, for annealing at 470 °C, the movable dislocations will quickly become annihilated in the high-angle grain boundaries, forming dislocation traps. The dislocation densities at the grain boundaries are, thereby, significantly reduced. In this case, the annealing temperature is relatively high, and the ultra-fast annealing process makes the recovery period very short. These circumstances promote the growth of large-angular grain boundaries within recrystallized crystal nuclei, and also provide larger deformation storage energy for grain growth.

Figure 3. Diagrams of EBSD orientations and grain size distributions for pure aluminium annealed at different temperatures: (a) 350 °C; (b) 410 °C; (c) 435 °C; (d) 470 °C; (e) 520 °C; (f) average grain sizes.
3.2. Influence of ultra-fast annealing temperature on the mechanical properties

Figure 6 shows the change in trends for the mechanical properties of the pure aluminium that were analyzed with EBSD and TEM (see section 3.1). When the annealing temperature increased from 350 °C to 520 °C, the tensile strengths of the pure aluminium were observed to decrease from 116.48 MPa to 53.43 MPa. In addition, the yield strengths decreased from 112.45 MPa to 23.14 MPa, while the elongations increased from 1.20% to 39.78%. Without any heat treatment of pure aluminium, both of the tensile and yield strengths became very high (127.57 MPa and 116.95 MPa, respectively). However, the plasticity was extremely poor with an elongation of only 5.36%. For an increase in annealing temperature from 350 °C to 380 °C, the material strength and plasticity did only decrease to minor extents. For an annealing temperature in the range 410 °C to 470 °C, the strength of pure aluminium was observed to markedly decrease, while the plasticity increased to a large extent. The tensile strength decreased from 103.29 MPa to 56.52 MPa (about 45%), and the elongation increased from 23.9% to 39.45% when increasing the temperature from 410 °C to 470 °C. When annealing at temperatures in the range from 470 °C to 520 °C, the tensile strength (and elongation) values decreased (and increased) slower. The final values of the tensile strength and elongation became 24 MPa and 40%, respectively.

4. Discussion

4.1. Relationship between microstructure and properties for ultra-fast annealed pure aluminium

As can be seen in figure 3, for the annealing temperature of 350 °C, the fibrous microstructure after cold-rolled still maintained. As a result of annealing at a somewhat higher temperature (410 °C), the amount of fine grains in the fibrous tissue increased sharply, and the work hardening that was caused by the large plastic deformations did gradually decrease. However, there was still a dominance of grain sizes less than 10 μm. When annealing at 435 °C, the dominance of the smaller grain sizes had decreased to about 23.97%. However, the sizes of these grains were a bit larger; 10 μm to 20 μm. This is an increase of about 57.78%, which indicates that as the
annealing temperature increased, the original small-sized grains began to merge and grow. When increasing the annealing temperature even more (to 470 °C), the grain sizes were in the range 31.5 μm to 43.5 μm. For an even larger annealing temperature (520 °C), the large-sized grains became evenly distributed over the whole matrix, and a certain amount of small sub-grains had begun to appear. As a result of the cold-rolled of pure aluminium, the overall grain size did continuously increase with an increase in the annealing temperature.

For commercial pure aluminium, since there is no strengthening element, or strengthening phase, in this material, its strength is mainly determined by the combined effect of grain boundaries and dislocations. Moreover, the dislocation gliding is the cause of plastic deformations. However, the entangled dislocation clusters, in addition to the grain boundaries, are expected to hinder these movements to a certain extent. The temperature annealing that takes place after the applied plastic deformations will recover the dislocations inside the material and, at the same time, decrease the dislocation densities. As the annealing temperature increases, the degree of activated dislocations will increase, which results in a gradual decrease in dislocation density. Also, the grain sizes become larger. According to the Hall-Patch relationship, the strength of the material will also decrease, which leads to the characteristics of material softening [30].

After the initial 98% cold-rolling, the crystal grains were observed to severely break down to pieces and a large number of dislocation entanglements were produced. Due to work hardening, this aluminium material received a high material strength. For a low annealing temperature, such as 350 °C or 380 °C, the pure aluminium experienced a very short low-temperature recovery, and the dislocation movements were also extremely limited. The original, cold-rolled microstructure was almost retained, with only a slight decrease in strength. Figure 4 shows that when the annealing temperature was 410 °C, the pure aluminium did undergo recrystallization, and the high-density dislocation entanglements remained at the grain boundaries. However, the atomic mobilities also became stronger. Although the recovery time was very short, it also promoted the dislocation mobilities, as well as the production of sub-grain boundaries and sub-grains. In addition, the sizes of the grains were small, which lead to the phenomenon of grain boundary strengthening. As a result, the pure aluminium experienced a relatively high strength and high plasticity. Figure 5 shows that as the annealing temperature increased to 470 °C, the dislocation mobility in pure aluminium also increased. The grain boundaries present the effect of dislocation traps, which made the movable dislocations in the crystal to disappear (i.e., when moving into the grain boundaries). Thus, the density of the movable intracrystalline dislocations had decreased. Since the thermal annealing process was extremely short (i.e., ultra-fast), the dislocations in the pure aluminium crystal grains could not be completely removed, which caused a part of the dislocation sources in the crystal to remain. With a further increase in annealing temperature, the grain sizes did also increase, and both factors lead to the decline in pure aluminium strength. That is, an annealed softening phenomenon occurred.

As can be seen in figure 6, when annealed at 350 °C and 380 °C, the pure aluminium was still in its recovery. It maintained most parts of the microstructure from the cold-rolling process, with a slight decrease in strength and a slight increase in plasticity. With an annealing temperature of 410 °C, the pure aluminium was in its recrystallization phase experiencing a higher yield strength and tensile strength. When annealing the pure aluminium at 435 °C, fine equiaxed crystals were formed, and the material showed relatively high strength and plasticity. When annealing the pure aluminium at 470 °C and 520 °C, the recrystallized grains were found to

Figure 6. Mechanical properties of cold-rolled aluminium after ultra-fast annealing at different annealing temperatures.
enter the growth stage. Also, the grains continued to merge into larger grains, which resulted in a continuous decrease in strength. In conclusion, the mechanical properties of pure aluminium are at their best when annealing in the temperature region of 410°C to 435°C.

4.2. Analysis of recrystallization mechanisms obtained during ultra-fast annealing of pure aluminium

Figure 7 shows the differences in grain boundary orientations at different annealing temperatures for the pure aluminium. When the pure aluminium was annealed at 350°C and 410°C, the orientations of the grains became almost identical to the small-angle grain boundaries (with angles between 2° and 15°). The presence of large-angle grain boundaries was just minor. When annealing at 435°C, the proportion of the large-angle grain boundaries (with angles larger than 15°) rapidly increased with an average misorientation angle of 28.57°. The pure aluminium was in its recrystallization stage. This means that the number of large-angular grain boundaries will rapidly increase with an increase in annealing temperature. For the higher annealing temperatures of 470°C and 520°C, the grain orientations were mainly in the region of small-angle boundaries (with angles in the 2°−15° region). Thus, the numbers of large-angle grain boundaries were sharply decreased and the grain boundaries were in high-energy unstable states, with entangled dislocations. Also the combined static recovery and recrystallization lead to an increasing of large-angle grain boundaries. The grains have grown by mutual assimilations for the high enough annealing temperature, which leads to the decrease in number of grain boundaries, with large angles. At the same time, many small-size sub-grains were produced at the grain boundaries, and the number of grain boundaries, with small angles, increased.

The differences in grain boundary orientation have also been found to be closely related to the grain boundary migration rates. As these differences increase, the energies of the grain boundaries have been found to also increase. In addition, the grain boundary stabilities decreased, which lead to an increase in grain boundary migration rates. Due to the low energy, and relatively stable microstructure, of a low-angle boundary, the possibilities for migration and sliding are extremely low [31]. At 350°C, the deformations were in the recovery stage. As can be seen in figure 7(a), the proportion of small-angle grain boundaries was relatively large after
annealing and the microstructure is relatively stable, as well as the fibrous microstructure after cold-rolled is still maintained. At 410 °C, the deformations were in the recrystallization stage. At this stage, the cold deformation storage energy has been transformed into the driving force for grain nucleation. As can be seen in figure 7(b), the proportion of small-angle grain boundaries (for the recrystallized grains) was relatively large after annealing (it is less than 350 °C). Since the grain boundary migration rate was low during the annealing process, many fine grains appeared in the recrystallized microstructure. When the pure aluminium was annealed at 435 °C, the large-angular grain boundaries were observed to occupy a larger proportion of the recrystallized microstructure, which lead to a high migration rate of the grain boundaries (see figure 7(c)). The grains were, hence, continuously coarsened with an increase in annealing temperature. When the ultra-fast annealing took place at a temperature between 470 °C and 520 °C, the pure aluminium was completely recrystallized. If the temperature continues to rise, the crystal grains would merge and continue to grow laterally. The phenomenon of abnormal grain growth occurred in a pure alumina matrix. The coarse grains were composed of several sub-grains with slightly different orientations. In addition, many sub-boundaries, with identical orientation, appeared in the grains (as shown in figure 7(d)-(e)), and the proportion of the small-angle grain boundaries increased. Furthermore, a large number of small-angle grain boundaries inside a grain can be regarded as a grain that is composed of a series of dislocations. It has, hence, been shown that some dislocations are still retained in the recrystallized grains as a result of the annealing at 470 °C and 520 °C. This is consistent with the conclusion presented in figure 7. In summary, the pure aluminium grain sizes will be minimized when performing ultra-fast annealing at a temperature within the range 410 °C to 435 °C.

The recovery and recrystallization are competitive processes. As compared with ordinary annealing, ultra-fast annealing will induce a shorter recovery time. There is either not enough time for the cold-rolled microstructure to recover, or the recovery process is not fully performed. Moreover, the storage energy of the cold deformation does not drop significantly, and the lattice distortions caused by the cold rolling is preserved. The driving force of recrystallization is, thereby, increased, with a lot of energies that can be used for both nucleation and recrystallization. High nucleation rates will induce a larger number of crystallites per unit volume, a smaller space for each crystal grain to grow, and a finer growth of crystal grains.

5. Conclusions

(1) When the annealing temperature is 410 °C, many new and finer recrystallization grains had appeared in large quantities, and the average grain size is 2.05 μm. When the annealing temperature is 435 °C, the fibrous microstructure had disappeared completely and was replaced by equiaxial grains, and the average grain size is 3.88 μm. When the annealing temperature increases to 470 °C and 520 °C, the new grains had merged and continued to grow to 9.46 μm and 17.10 μm. In addition, the tensile strength decreased from 116.48 MPa to 53.43 MPa and the elongation increased from 1.20% to 39.78% with the increase in annealing temperature (from 350 °C to 520 °C).

(2) When annealing at 380 °C ~ 410 °C, the pure aluminium is in the stage of recrystallization, and the average grain size reached 2.05 μm. When annealing at a higher temperature (435 °C), the number of small-angle grain boundaries (< 15°) was significantly reduced. The migration rates of the grain boundaries were thereby increased, leading to the continuous grain coarsening with the increase of ultra-fast annealing temperature. When the annealing temperature increased to 470 °C or 520 °C, the crystalline grains had merged and continued to grow.

(3) The annealing temperature of ultra-fast annealing should be in the range of 410 °C to 435 °C for optimizing the mechanical performances of the commercial pure aluminium.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
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