Hot Extrusion Enhanced Homogenization of Microstructure in a Spray Deposition Aluminum Alloy

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Abstract: Homogeneous heat treatment serves the significant roles in eliminating the segregation and tuning the microstructure of alloy ingot. It usually cost tremendous power and time to achieve a homogeneous microstructure for aluminum alloys. In this article, the hot extrusion was directly introduced on the spray deposited aluminum alloy 7055 ingot before performing heat treatment to explore the newly feasible homogeneous routine. Equiaxed grains without dendrites or PPB were obtained in our current parameters of spray deposition, which allowed the as-deposited alloy to be deformed without being subjected to pre-homogeneous heat treatment. Significant amount of stored energy was produced during hot extrusion at 420 °C with area reduction ratio of 6.25, which effectively promoted the homogeneity of microstructure and reduced significantly the heat treatment time. A newly feasible short routine, heat treatment at 450 °C/6 h + 470 °C/1 h following the hot extrusion, proved capable of obtaining a homogeneous microstructure for the spray deposited aluminum alloy 7055.

Keywords: aluminum alloy 7055; spray deposition; hot extrusion; heat treatment; microstructure homogenization

1. Introduction

The rapid development of the aerospace industry required aluminum alloys to possess increasingly excellent combinations of mechanical properties and corrosion resistance. Among the active-use aluminum alloys, 7055 alloy has been widely applied in numerous components owing to its ability to sustain the in-service high stress and hostile corrosion environment [1–3]. However, the macro segregation formed in the as-cast 7055 ingot made this alloy more inclined to suffer cracking during the processes of casting and the subsequent hot deformation [4]. Previous reports showed that the
macro segregation could be improved by long-time homogenization heat treatment after casting, but it took tremendous power and time to achieve a uniform distribution of elements. It was well accepted that the diffusion of atoms would be accelerated under the combined effects of heat and stress [5]. However, the cast billet cannot be directly deformed due to the existence of macro segregation and casting defects, i.e., voids, concentration of elements, which would lead to the occurrence of cracks if not controlled properly. Thus, the as-cast aluminum alloy 7055 was usually subjected to homogeneous heat treatment at 46–47 °C for 24 h before deformation [6].

Fortunately, spray deposition, a rapid solidification process, was proposed in the 1960s and was firstly applied in fabrication of super-high strength aluminum alloy in the 1990s [7,8]. With the assistance of spray deposition method, the aluminum alloy with high alloying could be produced with segregation being controlled at the micro-scale level. In this situation, the as-deposited alloy could be deformed under controllable parameters without being subjected to pre-homogenization heat treatment [9]. Recently, significant efforts have been made to study the hot deformation behaviors [10–15], quenching sensitivity [16–19], corrosion resistance [20–23] and weldability [24–29] of 7055. However, few reports were concerned with the improvement of homogenization in microstructure with less energy or short processes. In this article, hot extrusion was introduced to directly extrude the billet after spray deposition, wherein the microstructure evolution and corresponding mechanism during hot deformation and extrusion were uncovered. The subsequent heat treatment was employed to evaluate the effects of parameters on microstructure evolution of the hot-extruded alloy. Significant amount of stored energy was produced during hot extrusion at 420 °C with the area reduction ratio of 6.25, which effectively promoted the homogeneity of microstructure and reduced significantly the heat treatment time. A newly feasible short routine, heat treatment of 450 °C/6 h + 470 °C/1 h following the hot extrusion, proved capable of obtaining a homogeneous microstructure for the spray deposited aluminum alloy 7055. Our results provide a new perspective for promoting the uniformity of microstructure in aluminum alloys and will trigger broad interest in other alloy fields.

2. Materials and Methods

2.1. Material

The aluminum alloy 7055 (ϕ500 mm × 1800 mm) used in this work was fabricated by a spray-deposited machine (SFZD-5000, Jiangsu Haoran Inc. Zhenjiang, China) following the parameters in Table 1. The composition of the spray-deposited billet was examined by ICP-AES (SPECTRO BLUE SOP, SPECTRO Analytical Instruments GmbH & Co.KG, Kleve, Germany) and listed in Table 2. As can be seen, the total amount of major elements Zn, Mg, Cu reached as high as 12.8 wt. %, wherein the content of Zn was up to 8.31 wt. %, and the ratio of Zn/Mg was 4.0 wt. %. The contents of Fe and Si impurities were effectively controlled as 0.08 wt. % and 0.06 wt. %, respectively. After spray deposition, the surface layer was mechanically removed about 20 mm for the following use.

| Table 1. Key parameters used in spray deposition of aluminum alloy 7055. |
|-----------------------------|------------------|
| Items                       | Parameters       |
| Pour Temperature            | 780–950 °C       |
| Atomization Gas Rate        | 20–35 m³/min     |
| Metal Flow Rate             | 8–55 kg/min      |
| Gas to Metal Ratio          | 0.5–2            |
| Atomization Gas Pressure    | 0.5–1 MPa        |
| Spray Angle                 | 25–35°           |
| Deposition Distance         | 150–290 mm       |
| Substrate Rotation Rate     | 100–200 r/min    |
2.2. Thermal Simulation and Hot Extrusion

Cylinders with dimensions of 8 mm in diameter and 12 mm in height were cut out from the spray-deposited billet in the same concentric circles to ensure identical microstructure using electrical discharge machining (EDM). The hot compression tests were conducted on the dynamic thermal Gleeble 3800 test system (Dynamic System Inc., New York, NY, USA) under parameters of temperature ranging from 340 °C to 460 °C with interval of 40 °C and strain rate of 0.001 and 1 s⁻¹. All the specimens were deformed to a height reduction of 50%, after which the deformed specimens were subjected to water quench within 3 s to evaluate the workability and microstructure evolution. Based on the results of thermal simulation, the hot extrusion was performed under the extrusion ratio of 6.25 and temperature of 420 °C. Following that, the bar was fast cooled to ambient temperature within 15–20 s for subsequent microstructure examination and heat treatment study.

2.3. Heat Treatment Study

After the hot extruded bar was cooled down to ambient temperature, samples were cut out from the bar for heat treatment study. Temperatures of 450 °C /470 °C were selected as the target temperature of solution treatment and the samples were soaked for 2, 4, 6 and 8 h to uncover effects of dwell time on microstructure evolution. After soaking time was reached, all the solution heat treated samples were subjected to water quench for microstructure examination.

2.4. Microstructure and Phase Characterization

The compressed samples were cut in half along the compression axis using EDM, and the central area was used for microstructure study. For the extruded bar and heat-treated samples, the examined areas for microstructure detection were the sections perpendicular to extrusion. All the samples for microstructure examination were prepared following the standard metallographic procedures in the ASTM E3-11 standard. The surface was mechanically grinded to remove the scratch gradually using SiC paper with different grits of 80, 200, 400, 800, 1000 and 2000, after which vibration polishing with 8 h was carried out to remove the surface stress. Microstructure observations were carried out on the field emission scanning electron microscope (FEI Quanta 650), equipped with an EBSD (Electron backscatter diffraction) detector (Oxford Instrument plc, NordlysMax2, Abingdon, UK). For EBSD detection, the binning mode of charge-coupled device was selected as 320 × 240, and the spot size was chosen as 5.0 in order to balance the processing speed and data accuracy [30]. The step size was set as 0.5–2.0 µm to guarantee sufficient data and resolution in each grain [31,32]. The collected data was post processed using software OIM 7.5 (TexSEM Laboratories, Inc, Provo, UT, USA) / Channel 5 (Oxford Instruments, Abingdon, UK), and the average grain size was determined under the guideline of ASTM E2627-13 standard. Local misorientation was obtained by calculating average misorientation between every pixel and its surrounding pixels and assigning the mean value to that pixel. The filter size in this component was set as 5 × 5 and ‘subgrain angle’ was 5° to balance the resolution and accuracy [33,34].

In addition, a D/max 2500 type X-Ray diffraction (XRD) machine was employed to characterize the phases formed in the process of spray deposition using the following parameters: Cu-Kα, tube voltage of 40 kV, tube current of 250 mA and scanning speed of 8°/min. The collected data was post-analyzed using MDI Jade 5.0 software (MDI, Livermore, CA, USA).

| Elements | Al | Zn | Mg | Cu | Zr | Fe | Si |
|----------|----|----|----|----|----|----|----|
| wt. %    | Bal. | 8.3 | 2.1 | 2.5 | 0.1 | <0.1 | <0.1 |

Table 2. Composition of spray-deposition 7055 aluminum alloys.
3. Results

3.1. Microstructure Characterization of Spray Deposited 7055 Ingot

Figure 1 shows the inverse polar figure (IPF) of spray deposited 7055 ingot. As demonstrated in Figure 1b, equiaxed grains with sizes ranging mostly from 30–70 \( \mu \text{m} \) were formed during process of spray deposition. The average grain size was determined as 42 \( \pm 1.2 \) \( \mu \text{m} \) with maximum distribution frequency from 40 to 70 \( \mu \text{m} \) based on the output of EBSD data. In addition, no obvious preferential orientation was found in the spray deposited ingot, indicating the direction of recrystallization during the spray deposition is random, which is consistent with previous reports. The orientation distribution density shown by the polar figure (insert in Figure 1a) discloses a weak texture was detected in the as-cast alloy.

To further investigate the phases that formed in the spray deposited alloy, XRD was used and the result of which is shown in Figure 2. As can be seen in Figure 2, two unambigous sets of peaks were found in the spray deposited alloy 7055 based on the analysis of MDI Jade 5.0 software. The primary summit was calibrated as \( \alpha \)-Al (marked by ‘☆’), and the other one was \( \eta \)-MgZn\(_2\) (marked by ‘Δ’). No other phases were detected mainly due to the amount of phases under detection limitation of XRD. Figure 3 presents the images obtained by SEM using back scatter electron (BSE), which shows the microstructure of as-deposited alloy 7055. The ‘white’ particles were distributed randomly and uniformly inside the grains, while some particles with larger size (>20 \( \mu \text{m} \)) were generated on the grain boundaries, which is similar to the as-cast one. However, no dendrites (usually formed in the conventional cast process) or prior particle boundaries (usually formed in the powder metallurgy process) can be apparently found in the as-deposited alloy 7055, indicating that the alloy fabricated by spray deposition can overcome some metallurgical defects generated in the above techniques.

**Figure 1.** The EBSD (Electron backscatter diffraction) image of spray-deposition 7055 aluminum alloy ingot: (a) inverse pole figure with grain boundary and polar figure (inserted); (b) distribution of grain size.

**Figure 2.** The XRD (X-ray diffraction) of spray-deposition 7055 aluminum alloy showing the existed phases [9] (Metals, 2017).

**Figure 3.** The microstructure of as-deposited alloy 7055.
To further uncover the composition of particles that formed in the boundary and the distribution of elements in the spray deposited alloy 7055, energy dispersive spectroscopy (EDS) was employed and the mappings of elements are presented in Figure 4.

It was found that the element Al was distributed evenly in the matrix and absent in most of the particles for both inside the grain and on the grain boundary, while the elements Zn, Mg and Cu were simultaneously concentrated in most particles with size of 1–10 µm inside the grains (as shown in Figures 3 and 4a obtained by SEM). These particles with various elements were formed into different sizes in the process of solidification owing to the different melting point and cooling rate for parts of particles with size of 10–20 µm consisting of elements Zn, Mg and Cu. According to the previous works, these kinds of particles that simultaneously contained elements Zn, Mg and Cu were usually considered as η-MgZn2 and/or S-Al2CuMg [35]. Particles with size larger than 25 µm were found to primarily contain the elements Cu and Fe, which were believed to be Al2Cu2Fe according to previous works [6]. It is reasonable to conclude that the particles with large size on the grain boundary were consisted of more than two kinds of phases, i.e., η-MgZn2, S-Al2CuMg and Al2Cu2Fe.

Figure 2. The XRD (X-ray diffraction) of spray-deposition 7055 aluminum alloy showing the existed phases [9] (Metals, 2017).

Figure 3. The SEM (scanning electron microscope) images of spray-deposition 7055 at different magnification [9] (Metals, 2017): (a) 1000×; (b) 2000×.
without being subjected to pre-heat treatment [9]. In this article, we introduced isothermal compression samples, showing the microstructure evolution with variation of deformation parameters. For a general look, the fraction of particles was decreased with the increase in temperature and/or decrease in strain rate. With temperature increasing from 340 to 380 °C, the inside the grains remained the same with the one of ingot. While the particles on the grain boundaries were broken, and some of them were distributed randomly in the matrix. When the temperature increased to 420 °C, a large fraction of particles inside the grains started to dissolve into the matrix, accompanied with small amount of particles remaining on the grain boundary. It should be pointed out that decreasing the strain rate can facilitate the process of dissolution of particles into the matrix, as can be found by horizontal comparison in Figure 5a–h.

3.2. Microstructure Characterization of Spray-Deposition 7055 after Hot Compression and Extrusion

Temperatures ranging from 380 to 460 °C were proved to be the optimal deformation window according to our previous work, in which, the as-deposited alloy 7055 could be directly deformed without being subjected to pre-heat treatment [9]. In this article, we introduced isothermal compression to evaluate the effects of deformation temperatures (340 to 460 °C) and strain rates (0.001 and 1.0 s⁻¹) on the homogeneity of the microstructure. Figure 5 illustrates the SEM images of the deformed samples, showing the microstructure evolution with variation of deformation parameters. For a general look, the fraction of particles was decreased with the increase in temperature and/or decrease in strain rate. With temperature increasing from 340 to 380 °C, the

Figure 4. SEM image showing the interested area (a) and EDS (energy dispersive spectroscopy) mapping of different elements for spray-deposition 7055 aluminum alloy: (b) Al; (c) Zn; (d) Mg; (e) Cu; (f) Fe.

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general look, the fraction of particles was decreased with the increase in temperature and/or decrease in strain rate. With temperature increasing from 340 to 380 °C, the deformed samples presented similar microstructures, wherein the amount of undissolved particles inside the grains remained the same with the one of ingot. While the particles on the grain boundaries were broken, and some of them were distributed randomly in the matrix. When the temperature increased to 420 °C, a large fraction of particles inside the grains started to dissolve into the matrix and the size, as well as fraction, of particles on the grain boundary were dramatically reduced, as shown in Figure 5e. With temperature increasing to 460 °C, the particles inside the grains nearly dissolved into the matrix, accompanied with small amount of particles remaining on the grain boundary. It should be pointed out that decreasing the strain rate can facilitate the process of dissolution of particles into the matrix, as can be found by horizontal comparison in Figure 5a–h.

![Figure 5. Cont.](image-url)
Figure 5. Back-scattered electron images showing the microstructure of spray deposition alloy 7055 after being deformed at different parameters.

In addition, EDS was used to investigate the distribution of elements for deeply revealing the composition of the undissolved particles, results of the specimen deformed at 460 °C/0.001 s\(^{-1}\) were presented in Figure 6.

Figure 6. Cont.
It is noted that the elements Zn and Mg were still found in the particles that distributed randomly in the matrix, as shown in Figure 6c,d, while the elements Cu and Fe can be hardly observed in the remaining particles, as illustrated in Figure 6c,d. The above results indicated that the particles containing the elements Cu and Fe were nearly dissolved into the matrix during deformation at condition of 460 °C/0.001 s\(^{-1}\), mechanism of which will be discussed in the following sections.

Basing on our previous work, 420 °C was selected as the extrusion temperature in this work. The ingot was extruded at temperature of 420 °C with an extrusion ratio of 6.25 and the microstructure was examined by SEM and EBSD and shown in Figure 7. It was found that the particles with large size (>20 µm) decorated around the grains of the ingot were broken into small ones, which are presented as ‘stream-lined’ shapes and distributed along the direction of extrusion, as shown in Figure 7b. Figure 7b is the microstructure of section perpendicular to the direction of extrusion, in which the line analysis of elements is also embedded. Similarly, the elementary composition of these particles is consistent with the one found in the ingot, wherein elements Mg, Zn and Cu were found co-existed in one single particle. After hot extrusion, no obviously preferential orientation was detected according to the demonstration of IFP coloring, as demonstrated in Figure 7c. The inserted polar figure discloses that a weak texture with a maximum value of 9.16 m.u.d (multiples of uniform density, m.u.d.) was formed after extrusion. The average grain size was significantly reduced as small as 6.5 ± 0.4 µm based on the grain size distribution in Figure 7d.
were nearly dissolved into the matrix after soaking for 1 h at 470 °C. In this work, as-deposited and as-extruded alloy were both subjected to heat treatment, which was formed in the conventional cast process and would easily lead to cracking in subsequent hot working [6]. In this report [6], which remained undissolved at temperature as high as melting point.

7 Cu 32.1%, Fe 13.4% (in weight percent, average) based on the results of EDS (insert in Figure 9d), as shown in the embedded figure in Figure 9d. As can be seen, with increase of soaking time and temperature, the fraction of particles that dissolved into the matrix increased. However, the considerable fraction of \( \eta \)-MgZn\(_2\) macro segregations containing element of Fe with large size (as shown in the close-up view in Figure 8e,f) apparently remained after heat treatment, even that of 470 °C/24 h (Figure 8f), indicating the segregations cannot be fully eliminated by ordinary or simple heat input.

To further evaluate the microstructure responses of heat treated specimen after hot extrusion, the specimens were subjected to different soaking time at a temperature of 450 °C. Figure 9a demonstrates the microstructure of specimen soaked for 4h at a temperature of 450 °C, which reveals that a large fraction of particles was dissolved into a matrix. Figure 9b,c discloses that prolonged soaking time at this temperature can facilitate the dissolution of the remaining particles. However, the small fraction of particles with larger size of about 2–5 \( \mu \)m was still found after soaking for 8h at 450 °C. In order to enhance the dissolvability, we introduced a double solution heat treatment and raised the second-step solution temperature to 470 °C after treating it at 450 °C for 6 h. As can be seen in Figure 9d, the particles were nearly dissolved into the matrix after soaking for 1 h at 470 °C. The average composition of remaining particles (presented as ‘gray’ color, indicated by the arrows) was determined to be Al 54.5%, Cu 32.1%, Fe 13.4% (in weight percent, average) based on the results of EDS (insert in Figure 9d), as shown in the embedded figure in Figure 9d. This kind of particles proved to be \( \delta\)-Cu2Fe according the previous report [6], which remained undissolved at temperature as high as melting point.

3.3. Microstructure Responses of Heat Treated Specimen

Homogeneous heat treatment was usually adopted to reduce or eliminate the macro segregation, which was formed in the conventional cast process and would easily lead to cracking in subsequent hot working [6]. In this work, as-deposited and as-extruded alloy were both subjected to heat treatment for comparisons. Figure 8 shows the microstructures of as-deposited alloy experiencing different heat treatment parameters: soaking time from 1 to 24 h at 450 to 470 °C. As can be seen, with increase of soaking time and temperature, the fraction of particles that dissolved into the matrix increased. However, the considerable fraction of \( \eta \)-MgZn\(_2\) macro segregations containing element of Fe with large size (as shown in the close-up view in Figure 8e,f) apparently remained after heat treatment, even that of 470 °C/24 h (Figure 8f), indicating the segregations cannot be fully eliminated by ordinary or simple heat input.

Figure 7. Microstructure of as-extruded 7055 aluminum alloy: (a) SEM image showing the section parallel to direction of extrusion (b) line scans demonstrating the distribution of elements on the cross section; (c) inverse pole figure with grain boundary and polar figure (inserted); (d) distribution of grain size.

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Figure 8. Microstructure of as-deposited alloy experiencing different heat treatment parameters.

(a) 450 °C × 1 h
(b) 450 °C × 2 h
(c) 450 °C × 4 h
(d) 450 °C × 12 h
(e) 460 °C × 24 h
(f) 470 °C × 24 h

Figure 9. Cont.
In this work, the spray deposited 7055 alloy ingot presents microstructure with equiaxed grains and free of dendrites. It has been well proved that the process of spray deposition was co-multivariable controlled by several factors, as listed in Table 1. It was well documented that the solidification rate was primarily controlled by the atomization gas rate, metal flow rate, atomization gas pressure, substrate rotation rate, etc., which can be described as follows [36]:

$$Q_{g} = h S (T_{d} - T_{g}) + \sigma \varepsilon S (T_{d}^{4} - T_{w}^{4}) = m C_{d} \Delta T / \Delta t$$

wherein $Q_{g}$ is the rate of radiant heat loss, $S$ is the surface area of droplet, $\sigma$ is the Stefan constant, $\varepsilon$ is the radiation rate, $T_{d}$ is the droplet temperature, $T_{g}$ is the gas temperature, $T_{w}$ is the temperature of furnace wall and $h$ is the convective heat transfer coefficient, which was given as [36]:

$$h = K_{g} \left( 2 + 0.6 R_{e}^{0.7} P_{r}^{0.33} \right) \left( \frac{C_{g(avg)}}{C_{g}} \right) ^{0.26} / d$$

wherein $K_{g}$ is the gas heat conductivity, $P_{r}$ is Prandtl number, $C_{g}$ is the gas specific heat capacity, $C_{g(avg)}$ is average gas specific heat capacity when the temperature is equal to $(T_{d} + T_{g})/2$.

In this work, the solidification rate was estimated as $10^{3}$–$10^{4}$ °C/s based on the parameters listed in Table 1, which provided sufficient cooling rate to solidify the melt within one second. The extreme fast cooling rate forced the crystallization of partial melt to occur directly from the liquid state without generating dendrites; similar result was found in previous reports [4,5,9]. As a result, equiaxed grains with average size $42 \pm 1.2 \mu m$ were obtained after the spray deposition, as illustrated in Figure 1.

Based on the above results, the grain size was dramatically refined as fine as $6.5 \pm 0.4 \mu m$, after the ingot was hot extruded with an extrusion ratio of 6.25 at temperature of $420 \degree C$, as indicated in Figure 7. To further reveal the mechanism of refinement in grain size, we examined the recrystallization and grain boundary behavior of the as-extruded alloy. Figure 10 is the EBSD image demonstrating the recrystallized, substructured and deformed grains after hot extrusion. Herein, misorientation between two grains that larger than 15° was used to separate recrystallized grains from the others, and misorientation larger than 2° was defined as substructured grains. The renaming ones other than recrystallized and substructured were considered as deformed grains. It was found that a large fraction (>72%) of grains has completed the recrystallization process, as indicated by ‘blue’ color in Figure 10a. It should be noted that grains with fractions of 19.3% appeared as substructure and only a small fraction
(8.6%) of grains were presented as deformed ones. During hot extrusion, severe plastic deformation occurred, producing large density of dislocation around the grain boundary and inside the grains. The dislocation network was further consumed by dynamic recovery and/or dynamic recrystallization with continuous deformation. In this work, the misorientation of grain boundaries was calculated and presented in Figure 10b. It was well accepted that the boundaries with misorientations (10° < θ < 15°) were treated as the transition zone from LAGB (low angle grain boundary) to HAGB (high angle grain boundary) [37,38]. As can be seen, the fractions of misorientation under 10° and over 15° both exceed 45%, while the fraction of misorientation between 10° and 15° (defined as middle angle grain boundary, MAGB) was only 3%. The small fraction of MAGB indicated the rapid transition of LAGB to HAGB. It should be pointed out that the misorientations under 3° were apparently found around the deformed grains by comparing the Figure 10a,b. With deformation progressing, the LAGBs were rapidly transformed to HAGBs by rotation, which indicated that new recrystallized grains were formed from deformed grains. Generation of new recrystallized grains by rotation of grains without forming the nuclei was considered as the characteristic of continuous dynamic recrystallization; similar results can be found in previous works [39–43].

As presentations of our results above, the segregations can be nearly eliminated by the heat treatment of 450 °C/6 h + 470 °C/1 h after hot extrusion at 420 °C with extrusion ratio of 6.25. This indicates that the hot extrusion process can promote the elimination of macro segregation. It was well proved that the residual stored energy produced by strain or lattice distortion in the alloy would be activated and released during heat treatment, which would provide additional driving force to facilitate the microstructure evolution [44]. In EBSD, local misorientation could be used to locate deformed regions and was considered as an indication of magnitude of stored energy [45,46]. Figure 11 demonstrates the local misorientation profiles corresponding to as-deposited and as-extruded alloy, showing the relative frequency with different local misorientation. Generally, the larger the value of local misorientation, the more severe the local deformation that the alloy suffered. As can be seen in Figure 11a, no obvious concentration of local misorientation with larger angle (>2°) was observed, as indicated by the color profile. Further statistic calculation disclosed that a considerable fraction of local misorientation with angle smaller than 2° was detected, as shown in Figure 11b, which reveals that the low stored energy remained in the as-deposited alloy. However, after hot extrusion, a significant

Figure 10. EBSD image showing the distribution of the recrystallized (blue), substructured (yellow) and deformed grains (red) (a) and distribution of grain boundary (b) of as-extruded alloy.

4.2. Mechanism of Hot Extrusion Enhanced Homogenization of Microstructure

As presentations of our results above, the segregations can be nearly eliminated by the heat treatment of 450 °C/6 h + 470 °C/1 h after hot extrusion at 420 °C with extrusion ratio of 6.25. This indicates that the hot extrusion process can promote the elimination of macro segregation. It was well proved that the residual stored energy produced by strain or lattice distortion in the alloy would be activated and released during heat treatment, which would provide additional driving force to facilitate the microstructure evolution [44]. In EBSD, local misorientation could be used to locate deformed regions and was considered as an indication of magnitude of stored energy [45,46]. Figure 11 demonstrates the local misorientation profiles corresponding to as-deposited and as-extruded alloy, showing the relative frequency with different local misorientation. Generally, the larger the value of local misorientation, the more severe the local deformation that the alloy suffered. As can be seen in Figure 11a, no obvious concentration of local misorientation with larger angle (>2°) was observed, as indicated by the color profile. Further statistic calculation disclosed that a considerable fraction of local misorientation with angle smaller than 2° was detected, as shown in Figure 11b, which reveals that the low stored energy remained in the as-deposited alloy. However, after hot extrusion, a significant
amount of local misorientation with angle over 1° (present as ‘green color’) was apparently found in Figure 11c, which is further confirmed by the distribution curve of local misorientation in Figure 11d. The high fraction of local misorientation with the value over 2° indicated the high stored energy was produced after hot extrusion at 420 °C with extrusion ratio of 6.25. The stored energy would be activated and released when sufficient heat was input, providing additional driving force to promote dissolution of the segregations or remaining η-MgZn₂ phase, as shown in Figure 9. With increase in soaking time, more and more stored energy was consumed, and the promoting effect on dissolution of particles decreased. As a result, dissolution of the segregations or remaining η-MgZn₂ phase reached a limitation after soaked for 6 h at 450 °C, as indicated by Figure 9b,c. To further eliminate the remaining segregations, additional heat treatment at the temperature of 470 °C was introduced for 1 h, and the segregations were found to nearly dissolve into the matrix. The above analysis proved that the hot extrusion can effectively promote the homogeneity of microstructure and reduce significantly the heat treatment time.

Figure 11. EBSD images showing the local misorientation profiles and corresponding distribution: (a) and (b) represent the as-deposited alloy; (c) and (d) correspond to as-extruded alloy.
5. Conclusions

This article focused on an investigation of the homogeneity of microstructure in a spray deposited aluminum alloy 7055. The hot extrusion was introduced before performing heat treatment on the as-deposited ingot to explore the newly feasible homogeneous routine. Equiaxed grains free of dendrites or PPB were obtained in our current parameters of spray deposition, which allowed the as-deposited alloy to be deformed without being subjected to pre-homogeneous heat treatment. The significant amount of stored energy was produced during hot extrusion at 420 °C with area reduction ratio of 6.25, which effectively promoted the homogeneity of the microstructure and reduced significantly the heat treatment time. As a result, a newly feasible short routine, heat treatment of 450 °C/6 h + 470 °C/1 h following the hot extrusion, proved capable of obtaining a homogeneous microstructure for the spray deposited aluminum alloy 7055.

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