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Microstructure Evaluation Study of Al5083 Alloy Using EBSD Technique after Processing with Different ECAP Processes and Temperatures

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Abstract: The temperature effects on the microstructural evolution of a coarse-grained Al5083 alloy during equal channel angular pressing (ECAP), were studied at ambient and high temperatures. The microstructural evaluation was done using an EBSD (electron backscattering diffraction) process. The grain refinement occurred as the number of passes increased, which had a positive effect on its strength. Additionally, increasing the pressing temperature leads to a decrease in the new grain's formation and an increase in the normal grain size in the third pass. This can be ascribed to the unwinding of strain similarity between the grains because of the continuous activity of dynamic recuperation and the grain limit sliding occurring at a higher temperature. The attainment of grain refinement is examined exhaustively in this study.

Keywords: ECAP; EBSD; Al5083 alloy; high temperature; grain refinement

1. Introduction

ECAP is the most appealing and conceivably the most helpful severe plastic deformation (SPD) technique. Through this technique, ultrafine fine-grained (UFG) samples or materials with grain estimates commonly in the scope of the range of 100–1000 nm can be created [1]. The preparing technique for ECAP incorporates squeezing or pressing a billet by utilizing a die comprising of two channels of equivalent cross-section converging at a specified angle (Φ). Since the cross-section of the channels is equal, the sample's cross-section remains the same with every pass of ECAP. Additionally, depending on the angle (Φ), the ECAP process results in critical refinement of the grains and the sample acquiring exceptional mechanical properties [2–6]. The forced strain accumulated on the sample fundamentally relies upon two boundaries: the inward crossing point of the channels (channel angle, Φ) and corner ebb and flow angle (curvature angle, ψ). It was observed that for a die with a converging channel point of 90° and a corner curve point of ~20°, the forced strain after each pass of the sample is around one [7].

The ECAP die parameters (the channel angle (Φ) and the curvature angle (Ψ) shown in Figure 1), and the processing parameters such as strain rate, pressing route (A, B_c, C), number of passes (N), and pressing temperature influence the properties and microstructure of the billets exposed to the ECAP process [1,5–7]. High strength aluminum alloys, such as Al-7075, can be promising candidates for use in aviation and aerospace applications as

well as automotive and marine industries due to their high strength and low density [8,9]. Al-7075 is a heat treatable alloy that is heat treatable and can be tailored considerably during the manufacturing process to obtain desirable properties [10]. Furthermore, the ECAP process can be combined with different aging conditions can be applied to Al-7075 alloy to further enhance its strength.

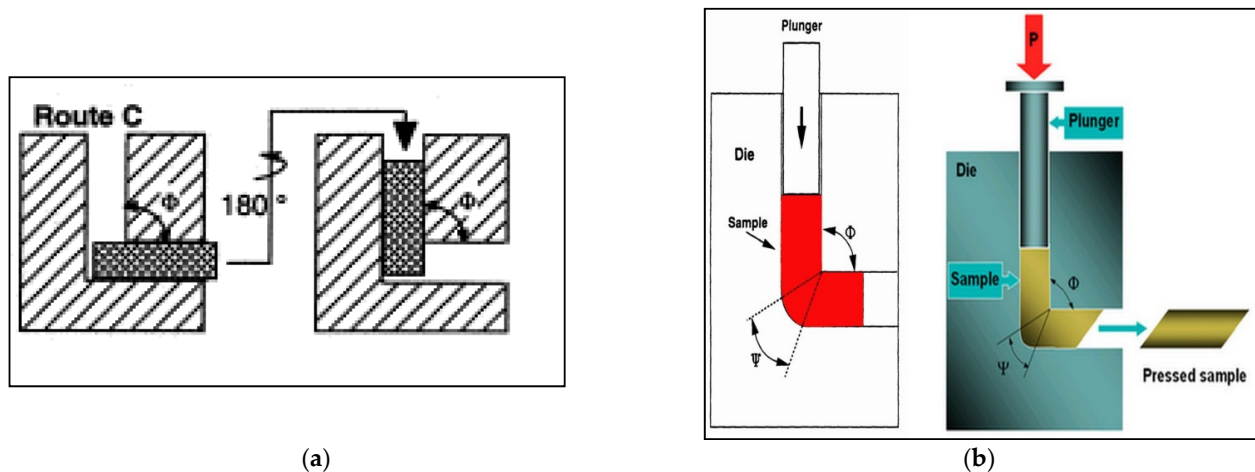


Figure 1. A schematic diagram of equal-channel angular pressing (ECAP) die: (a) route C path, (b) three-dimensional (3D) presentation of ECAP die.

During the ECAP of the hardenable Al alloys, strain hardening of the alloy takes place in addition to the grain size reduction along with precipitation solidifying [11,12]. In this way, the combination of ECAP and aging treatment may assist in achieving better properties due to the development of the ultra-fine-grained microstructure [13,14].

In the practical field, the combination of the ECAP process with age hardenable alloys usually leads to a decrease in the formability of the processed alloy due to strain hardening. Thus, breaking or cracking of the material is quite normal during the ECAP process at room temperature. By using the high temperature and utilizing back pressure, the formability issues described above can be minimized to a greater extent [15–18]. Back pressure is particularly significant in reducing the cracking or breaking of metals and alloys that have low ductility. Backpressure results in applying shear strain on the sample by means of hydrostatic pressing, thereby reducing the probability of sample cracking during the ECAP process [15–18]. In the ECAP process, pressing at high temperatures is helpful for materials that do not have good formability since the extreme shear strain happens in the midst of every pass of ECAP that may become the reason for breaking in these materials. The capability of materials gradually improves due to the high working temperature and thus stalls the age of shear breaks [19,20]. So, expanding the temperature of ECAP regularly prompts the decrease in the strength of the material, for which doing ECAP becomes quite easier. With these benefits, ECAP performed at high temperatures could be an extremely encouraging methodology with an extraordinary business potential [21]. Furthermore, extreme plastic deformation at raised temperatures significantly affects the grain structure (grain size, grain shape, and grain limit misorientation point) of hardenable Al compounds [20,22,23]. In spite of the great benefits of ECAP at higher temperatures, sample expulsion temperature is the most un-comprehended handling boundary among the previously mentioned boundaries.

In Al and their alloys, the changes in microstructure during ECAP at different temperatures have been found in many revealed works [1,20,24–32]. It was observed that the grain size and the negligible portion of developed low angle boundaries (LABs) are overall expanded with the expanding temperature of ECAP. The development of new grains in Al-based composites has been proposed because of the change in LABs shaped at the beginning phases of deformity into high angle boundaries (HABs) joined by powerful

recuperation during hot pressing. This mechanism is like in situ or nonstop powerful recrystallization [1,21,27,30–35]. It is recommended in [20,24–30] that the progress of LABs into HABs can be constrained by a recovery rate, which is sped up with expanding temperature. The negligible portion of HABs, just like the normal misorientation point, increments quicker in Al alloys at lower ECAP temperature [1,20,24,26–32]. The change of twisting converts LABs into HABs and happens at elevated temperatures. It has been pointed out in [30] that the attributes of newly formed grains at high strains are practically independent of squeezing temperature. On the other hand, the occurrence of continuous dynamic recrystallization during severe plastic deformation at high temperature has also been recognized as the geometric dynamic recrystallization, which results in an equiaxed grain structure with a small grain size comparable to the subgrain size [31,36,37].

In this present investigation, commercially available aluminum alloy (Al-5083) has been exposed to ECAP through route C [38] at room temperature and high temperature to improve its strength. Further examinations have been done on microstructure through the EBSD process to study the mechanical properties changes of this alloy in as-received and ECAP conditions.

2. Materials and Methods

2.1. ECAP Process of Al-5083 Alloy

The specimens were pressed up to 3 passes (one, two, and three) with the help of a hydraulic press (200 tons). This process was done at ambient and at high temperatures (250 °C). In the present study, route C was employed for pressing (Figure 1), and a 0.6 mm/s pressing speed was maintained during this process.

2.2. EBSD Process

On the RD (rolling direction)-TD (transverse direction) sections, the microstructures and crystallographic texture of the specimen were studied. A PANalytical X'Pert-PRO MRD goniometer with a Cu tube running at 40 kV was used to test the bulk texture. The specimen was machined and further polished mechanically to produce scratch-free and smooth surfaces before texture measurements. The texture test was conducted on the rolling samples' 1010 mm² surfaces. In an FEI Quanta FEG 250 FESEM, the microstructures of the specimen were analyzed using electron backscattering diffraction (EBSD). The sample for EBSD was electrolytically cleaned using 30 vol. percent HNO₃ (nitric acid) methanol solution at −30°C with a voltage of 14 V. Electropolishing in an HClO₄ + 900 mL CH₃OH solution was used to polish the sample for EBSD analysis. A FEG-SEM Zeiss Supra 55 VP with an orientation imaging microscopy OIMTM device mounted on it was used for EBSD measurement. The mapping phase size and scan area were set to 100 nm and 100 × 100 m², respectively.

3. Results and Discussions

3.1. EBSD Micrograph

As opposed to the normal micrograph-based strategies, e.g., (ASTM International), the EBSD estimations permit an accurate assurance of the size of the grain and its subgrain. The qualification regarding whether grain or subgrain sizes are estimated is totally free from the EBSD information record yet just relies upon client characterized misorientation edge esteems. A 15° misorientation is usually used to characterize HAGBs (high point grain limit) that separate individual grains in aluminum composites [39]. In this work, LAGBs somewhere in the range of 3°–15° are distinguished as grain limits isolating individual subgrains. The lower furthest reaches of 3° are picked to guarantee a specific distance to the estimation mistake of the EBSD method that stays in the scope of 1°–2° and relies upon the specimen piece and position of the engaged electron bar inside the estimation region. As per Humphreys [40], the extra rakish mistake because of the mutilation of the centered electron shaft in the pre-owned quick bar examining mode can reach up to 1°. The meaning of the base misorientation point treated as LAGB (low angle grain limit) between

subgrain fluctuates in various distributions however is normally thought to be under 5° . Here, the misorientation between nearby information focuses is assessed, permitting a restricted variety of directions inside individual distinguished grains, which is normal for the vast majority of the noticed micrographs. Figure 2 shows a re-established and cleaned direction map with over-layered HAGB $\geq 15^\circ$ and $3^\circ \leq \text{LAGB} \leq 15^\circ$. Figure 2a,b shows the distinguished subgrains and grains which are shaded relating to their grain ID.

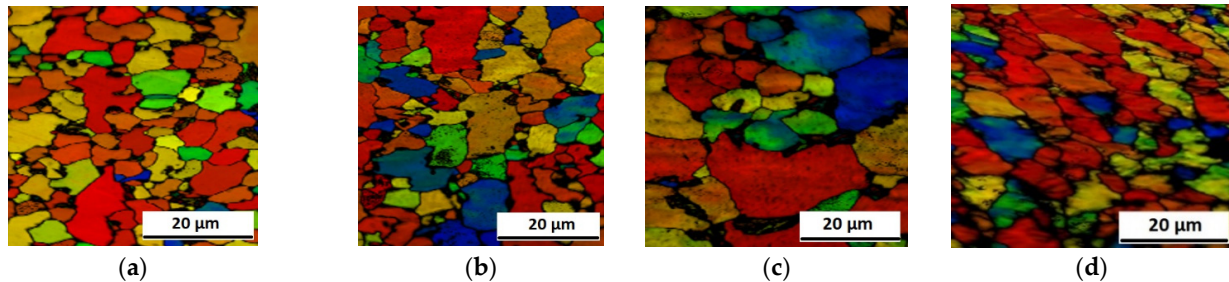


Figure 2. EBSD micrograph of Al5083 alloy after ECAP processing (a) no pass, (b) first pass, (c) second pass, (d) third pass at ambient temperature.

Due to high temperature, dynamic recrystallization occurs, or the high temperature accelerates the kinetics of recrystallization. Figure 3 exhibits the EBSD micrograph at a higher temperature. From Figure 3, it is clear that some inactive slip frameworks take place and introduce some latent slip systems and increase the interaction between dislocations on different slip systems, the probability of cross-slip and rearrangement of dislocations, and finally, recrystallization enhances. It should be noted that when the stored energy increases with further equivalent strain, the recrystallization temperature shifts to a lower value and the aggregate heat effect makes the recrystallization possible during or after ECAP processing [41].

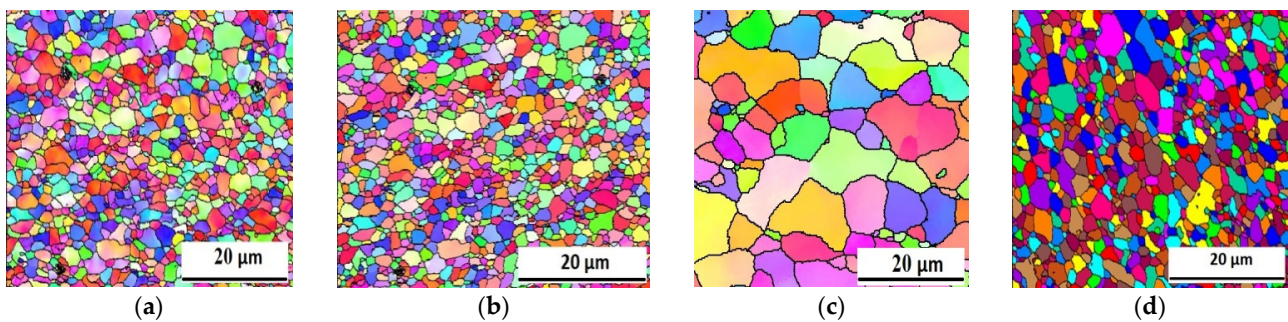


Figure 3. EBSD micrograph of Al 5083 alloy after ECAPed processing (a) no pass, (b) first pass, (c) second pass, (d) third pass at high temperature.

The microstructural improvement in an entire volume of the ECAPed Al composite is barely impacted by the squeezing temperature in the first and second pass when the development of the new structure, which consists of fine grain, is altogether influenced by the temperature from the third pass. It is presumed that strain-initiated grain arrangement results from the dynamic development of HAGBs in the first pass and second pass, trailed by continuous activity of dynamic recuperation in the HAGBs created in the third pass.

The measurable portrayal of the grain direction spread determined for all examples was introduced in Figure 4. This measurement might be utilized to portray inner grain heterogeneity. A higher spread proposes neighborhood nonuniformity among the grain or inside the grain. A higher spread was noticed for the underlying example, and the factor esteem diminishes with increasing distortion esteem, affirming that the more uniformity of the grains. A reasonable connection between the grain size and grain direction spread was noticed. Little grains are very homogeneous; in the meantime, the spread orientation

of these grains is higher. The geometrically necessary dislocations (GND) have been assessed utilizing EBSD investigation. Geometrically necessary dislocations are like-signed dislocations that are essential to incorporate the plastic bending inside a crystalline material. They are present whenever the plastic deformation in the material is accompanied by internal plastic strain gradients. Estimation of the disengagement thickness was done dependent on the nearby normal grain limit misorientations that appeared in Figure 5. The normal grain size increments with an expanding twisting worth. The normal GND thickness was expanded from $3.2 \times 10^{12} \text{ m}^{-2}$ in the underlying state to $7.4 \times 10^{12} \text{ m}^{-2}$ for the example disfigured in the third passes. The noticed patterns affirmed past perceptions, where a critical difference in the microstructure happens after the third passes. Figure 5 shows the histograms of the ECAPed sample at ambient temperature.

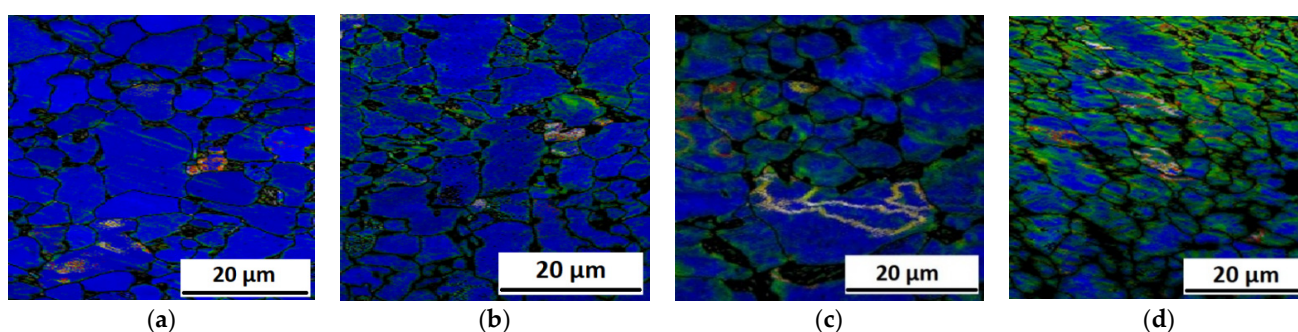


Figure 4. EBSD color map of Al 5083 alloy after ECAPed processing. (a) No pass, (b) first pass, (c) second pass, (d) third pass at ambient temperature.

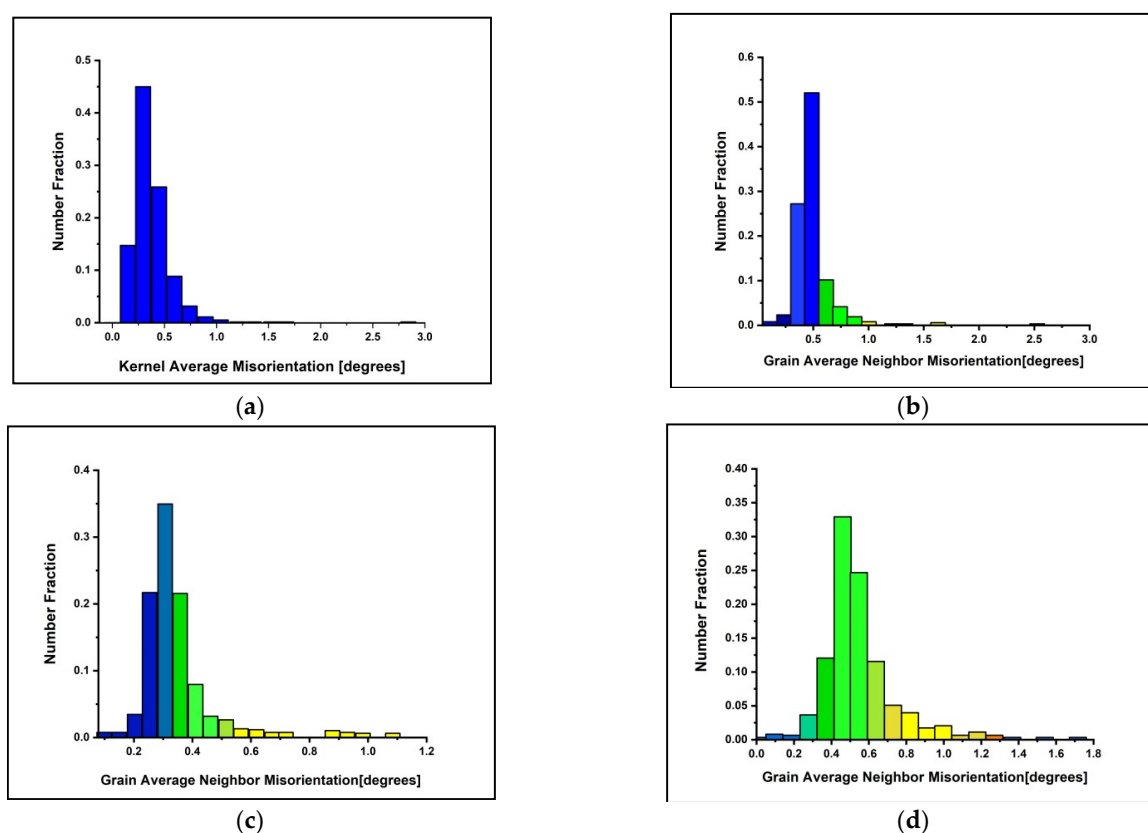


Figure 5. Frequency histograms of grain boundary misorientation of the ECAPed sample after (a) no pass, (b) first pass, (c) second pass, (d) third pass at ambient temperature.

Figure 6 shows the EBSD color map of the as-received (no pass) and ECAPed Al sample. The misorientation found in Figure 7a–c, that advanced at $\varepsilon = 3$ (ε denotes pass), shows a single peak type ranging mainly below 10° , while they shift a little to lower angles with increasing temperature. It is likewise noted here that the misorientation appropriations estimated in an entire volume and in fine-grained locales are generally comparable at $\varepsilon < 3$. The small part of HABs with misorientations above 15° reductions and that of LABs, on the other hand, increments even with expansion in temperature. It is worth taking note that the misorientation dispersions created at $\varepsilon = 3$ in an entire volume (strong line) and in the grain, which is fine in structure, are plainly unique at a higher temperature.

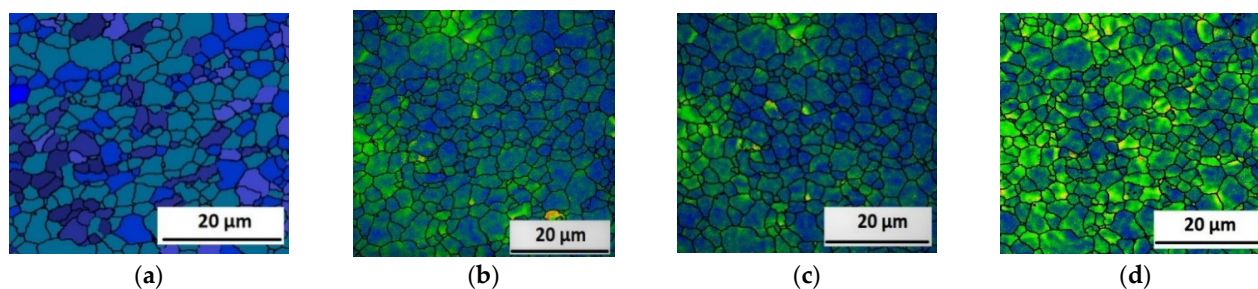


Figure 6. EBSD color map of high-temperature ECAPed Aluminum after (a) no pass, (b) first pass, (c) second pass, (d) third pass.

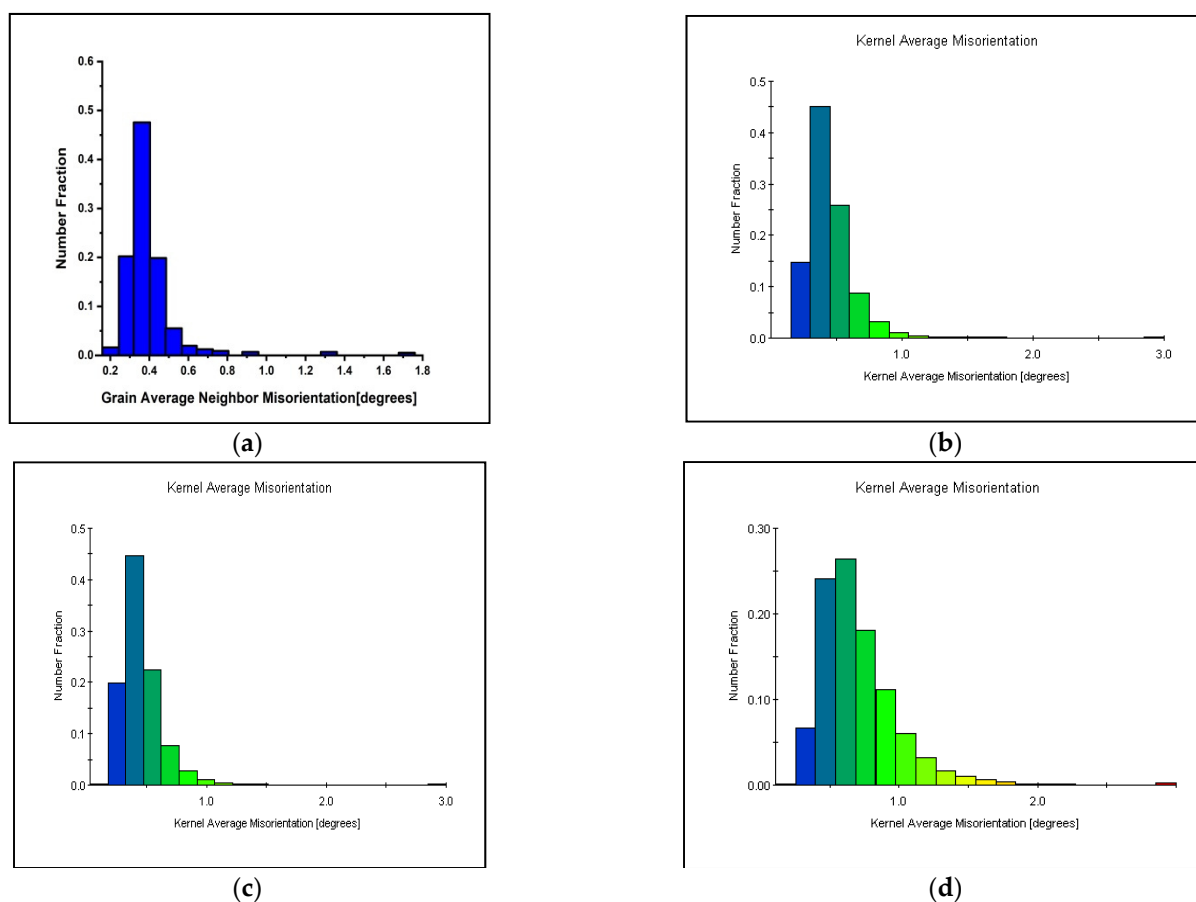


Figure 7. Frequency histograms of grain boundary misorientation of the high temperature ECAPed sample after (a) no pass, (b) first pass, (c) second pass, (d) third pass.

3.2. Kernel Average Misorientation (KAM)

The neighborhood misorientation in the samples can be portrayed by the KAM approach. In a given point, the KAM of that point concerning all its closest neighbors is determined with the arrangement that misorientations with surpassing resistance esteems (typically set to 15°) and related with grain limits are avoided from the averaging strategy. It was at that point shown [42,43] that this KAM boundary is for sure a decent device to decide the level of distortion and is significantly more effective in deciding the recrystallization degree during the intruded strengthening treatments. Whenever it has been determined for each point inside a guide, it tends to arrive at the midpoint of all focuses from a given stage.

KAM is typically used to address normal misorientation under 5° between a given point and its closest neighbors, which have a place with a similar grain. In this way, the KAM guide can be utilized to survey neighborhood plastic strain and, along these lines, reflect, somewhat, the thickness of separations. In order to find the stored energy in the material the most appropriate quantity is Kernels average misorientation (KAM) [44]. In the portion maps, the level of misorientation connected to the adjoining information focuses are demonstrated by a shading plan. A bit map and the related shadings appear in Figures 8a–d and 8e, giving the misorientation in degrees. Red tones demonstrate a serious level of misorientation contrasted with the encompassing information focuses; blue tones show a low misorientation and low strains. From Figure 8b,c, it may be seen that the second pass and third pass have not caused the material to recrystallize completely. Still, the separation content has stayed in the material. The fine grains which are shaped after the third pass were the consolidated impact of static recuperation and recrystallization.

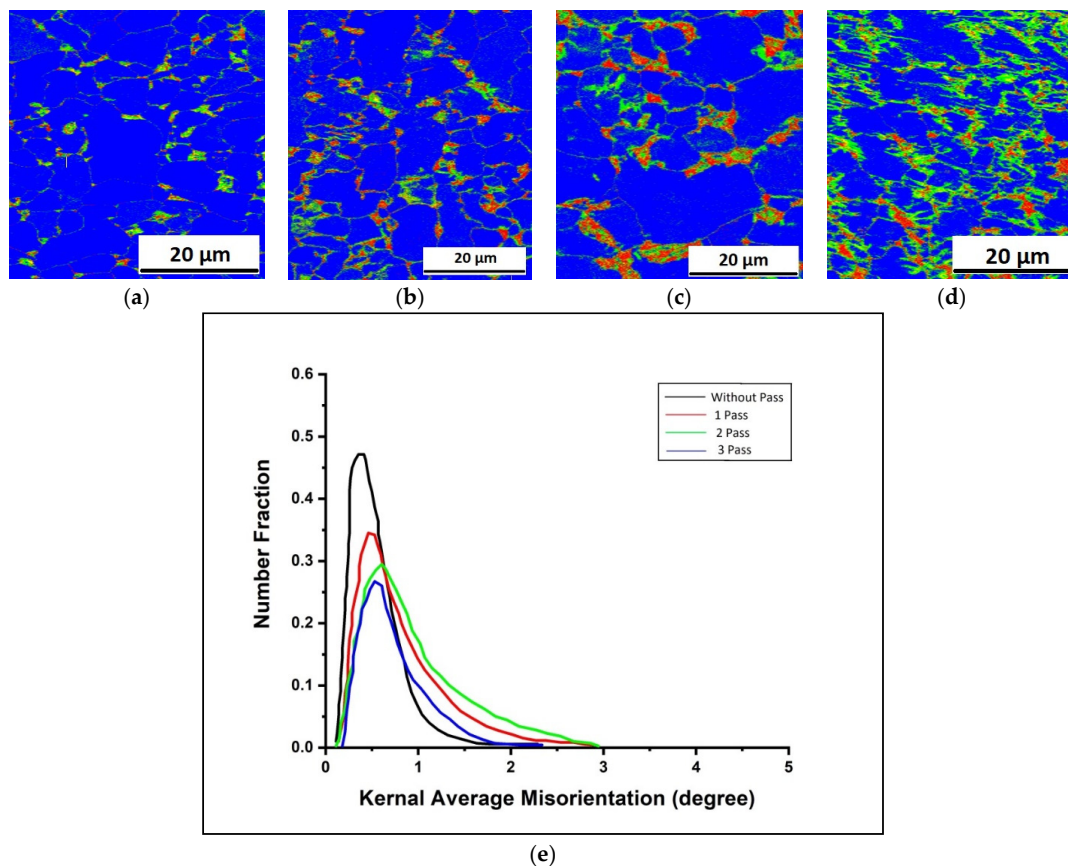


Figure 8. Kernel Average misorientation micrographs of (a) As received and ECAPed after (b) first pass, (c) second pass, (d) third pass. (e) Kernels average misorientation (KAM) plots at ambient temperature.

Figure 9 shows the Kernel Average Misorientation (KAM) guides of the ECAPed Al-5083 alloy at a high temperature. It may be seen that the coarse grains, particularly their close by limits, had high KAM values in all passes, showing a high separation thickness in these locales. This gave additional proof that the coarse grains were disfigured. Consequently, the coarse grains were in the compound were the inward center spaces of the enormous beginning grains. Figure 9 shows the KAM esteem versus relative recurrence. It is evident that the overall recurrence of low KAM esteems was higher in the as-received sample, while high KAM esteems were more articulated in the ECAP sample, recommending that disengagement thickness in the as-received sample was lower than that in the ECAP sample. This showed that the new fine grains nucleated along previous coarse grain limits inferable from their high separation thickness during maturing, that is, the event of static recrystallization. Most importantly, the current outcome showed that deficient recrystallization along the previous coarse grain limits was the major justification in the development of the multimodal grain structure.

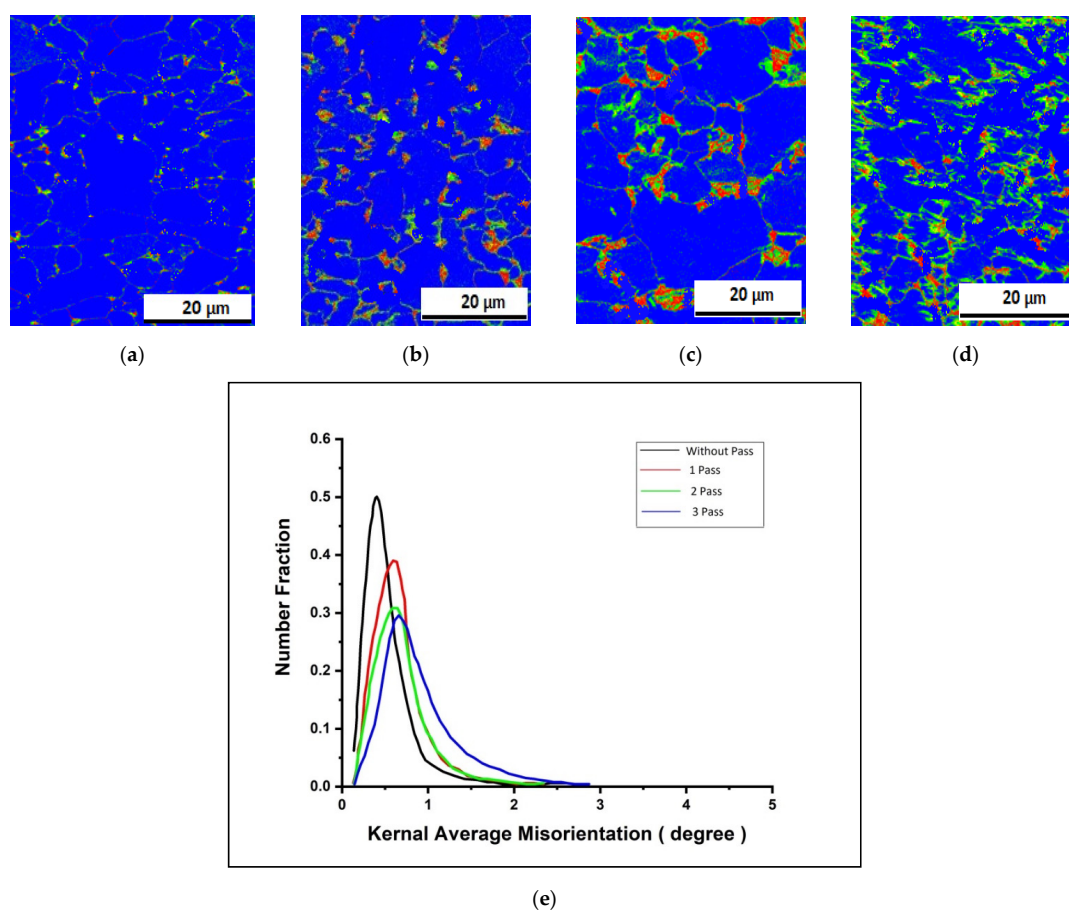


Figure 9. Kernel Average misorientation micrographs of (a) As received and ECAPed after (b) first pass, (c) second pass, (d) third pass. (e) Kernels average misorientation (KAM) plots at high temperature.

3.3. Texture

The Orientation Distribution Function (ODF) was determined for each sample from the direction information gathered by EBSD. To do this, a Gaussian peak was set around every deliberate direction (with a Gaussian spread set equivalent to 5°). Each pinnacle was then evolved utilizing the arrangement extension strategy [45], and the subsequent ODF was determined as the amount of every one of these pinnacles. Bunge [46] reported that the ODF is introduced in Euler space utilizing standard show. The volume parts of the surface segments of interest (see below) were determined by the method clarified in [47].

It has become apparent by different creators [48] that direction can be inferred by an expansion of ϕ_1 in the straightforward shear direction by half of the channel crossing point while the other two Euler points ϕ , ϕ_2 stay consistent. Orientation Distribution Functions (ODFs) were determined based on EBSD information from all inspected tests and are introduced in Figure 10 as far as ODF segments. From Figure 10, it is clear that the grains are exceptionally stretched and are longer than the estimated region (considering 15° limit). In any case, grains are heterogeneous inside. It implies that the assortment of precious stone directions is a lot bigger than the number of grains. This is the reason, for ODF estimations, that the huge enough region was chosen rather than the number of grains. The ODF was determined with triclinic example balance [49] and introduced up to the full reach with ϕ_1 from 0° to 90° .

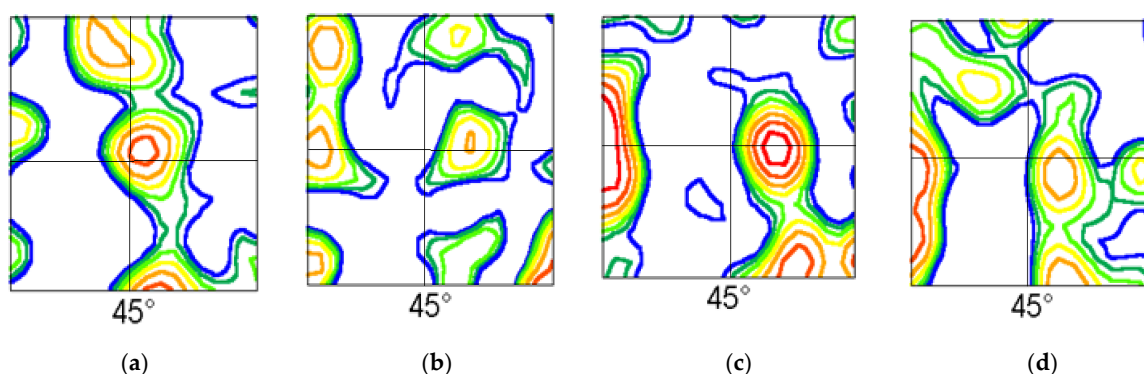


Figure 10. Typical ideal orientation observed after ECAP passes. (a) No pass, (b) first pass, (c) second pass, (d) third pass at ambient temperature.

The ODFs of the examples after one, two, and three ECAP passes at high temperature are introduced in Figure 11. It is accepted that this blended twisting mode that is a consequence of the fan-molded deformity zone and ECAP pass-on calculation is the justification for the slow surface part pivot. This outcome is matching with the findings of Vega et al. [50]. Additionally, notice that when the underlying surface is solid, surface development might be very unique, especially in the first few passes [51]. Then again, in light of the fact that the strain is restricted in one pass, it very well might be insufficient for the surface parts to be recovered close to the ideal surface segments. This can be one reason for the slants of the surface segments in the further number of passes as for their optimal positions. Another explanation can be ascribed to the presence of the subsequent stage and intermetallic segments in the Al-5083 alloy.

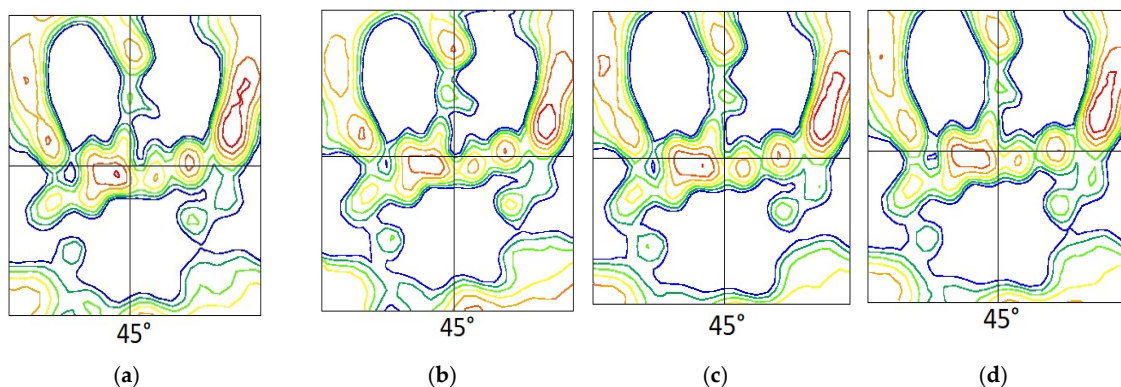


Figure 11. Typical ideal orientation observed after ECAP passes at high temperature. (a) No pass, (b) first pass, (c) second pass, (d) third pass.

4. Conclusions

1. Microstructural advancement in an entire volume of the Al alloy, which has undergone the ECAP process, is scarcely impacted by the pressing temperature in the first and second pass, while the arrangement of the new fine-grained structure is altogether influenced by the temperature from the third pass.
2. The normal GND thickness was expanded from $3.2 \times 10^{12} \text{ m}^{-2}$ in the underlying state to $7.4 \times 10^{12} \text{ m}^{-2}$ for the example distorted in the third passes.
3. Misorientation created a higher temperature after the third passes. The small portion of HABs with misorientations above 15° reductions and that of LABs alternately increased even with expansion in temperature.
4. It can be seen from both surrounding temperature and high temperature that the second pass and third pass have not caused to recrystallize the material fully. Still, the separation content has stayed in the material. The fine grains which are formed after 3rd pass were combined effect of static recovery and recrystallization.
5. In both ambient and higher temperature, the mixed deformation mode occurred due to the fan-shaped deformation zone and ECAP die geometry and constituted the reason for the gradual texture component rotation. The strain is restricted in one pass, and might very well be insufficient for the surface segments to be recovered close to the ideal surface parts. This can be one reason for the slants of the surface parts in the further number of passes concerning their optimal positions.

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Conflicts of Interest: The authors declare no conflict of interest.

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