Influence of Uniaxial Deformation on Texture Evolution and Anisotropy of 3104 Al Sheet with Different Initial Microstructure

Sofia Papadopoulou 1,2,*, Vasilis Loukadakis 2, Zisimos Zacharopoulos 2 and Spyros Papaefthymiou 2

Citation: Papadopoulou, S.; Loukadakis, V.; Zacharopoulos, Z.; Papaefthymiou, S. Influence of Uniaxial Deformation on Texture Evolution and Anisotropy of 3104 Al Sheet with Different Initial Microstructure. Metals 2021, 11, 1729. https://doi.org/10.3390/met11111729

Abstract: Optimum mechanical behavior is achieved by means of controlling microstructural anisotropy. The latter is directly related to the crystallographic texture and is considerably affected by thermal and mechanical processes. Therefore, understanding the underlying mechanisms relating to its evolution during thermomechanical processing is of major importance. Towards that direction, an attempt to identify possible correlations among significant microstructural parameters relating to texture response during deformation was made. For this purpose, a 3104 aluminum alloy sheet sample (0.5 mm) was examined in the following states: (a) cold rolled (with 90% reduction), (b) recovered and (c) fully recrystallized. Texture, anisotropy as well as the mechanical properties of the samples from each condition were examined. Afterwards, samples were subjected to uniaxial loading (tensile testing) while the most deformed yet representative areas near the fractured surfaces were selected for further texture analysis. Electron backscatter diffraction (EBSD) scans and respective measurements were conducted in all three tensile test directions (0°, 45° and 90° towards rolling direction (RD)) by means of which the evolution of the texture components, their correlation with the three selected directions as well as the resulting anisotropy were highlighted. In the case of the cold-rolled and the recovered sample, the total count of S2 and S3 components did not change prior to and after tensile testing at 0° towards RD; however, the S2 and S3 sum mostly consisted of S3 components after tensile testing whereas it mostly consisted of S2 components prior to tensile testing. In addition, the aforementioned state was accompanied by a strong brass component. The preservation of an increased amount of S components, and the presence of strain-free elongated grains along with the coexistence of a complex and resistant-to-crack-propagation substructure consisting of both high-angle grain boundaries (HAGBs) and subgrain boundaries (SGBs) led into an optimal combination of $\Delta r$ and $r_m$ parameters.

Keywords: 3104 alloy; crystallographic texture; cold rolling; tensile testing

1. Introduction

Beverage cans and food packaging are manufactured by use of 3xxx alloy series such as 3004 and 3104 due to their high strength and formability [1]. After many years of research and optimization of the relevant thermomechanical processes, the industry heavily relies on the use of those alloys [2]. Although these alloy series cover current market demands, the existence of failures along with a relatively high percentage of rejected material, are frequently observed during the forming processes in which those products are subjected [2,3]. This phenomenon is attributed to sheet anisotropy provoking the formation of “ears” of varying extent and orientation. Earing is a dominant defect induced during the deep drawing process and defined as the creation of waviness on
the uppermost portion of the resulting cup. Rolling texture components favor earing principally at 45°/135° with respect to the RD [4,5]. With regards to earing, a low ear ratio can be achieved through an optimal combination of crystals with an orientation that results in both 45°/135° and 0°/90° ears [6–9]. The crystallographic components which are formed during recrystallization depend on the stacking fault energy (SFE), the previous deformation state, the annealing temperature as well as the precipitation state. Metals with high SFE (like Al) mainly exhibit a deformation substructure consisting of dislocation tangles and cells instead of banded, linear arrays of dislocations [10]. The anisotropy is, essentially, a result of the texture obtained throughout the thermomechanical processes.

A thorough understanding of the microstructural evolution, may result into the effective control of the resulting texture after each thermomechanical process. This essentially relates to the response to deformation with the aim of minimizing rejections, increasing the product’s life cycle, and, consequently, improving the Al industry’s economic and environmental footprint.

Moreover, study of the texture components which are present at the most susceptible to fracture areas can provide fruitful information in relation to the underlying failure mechanisms. Grain orientation may strongly affect the deformed microstructures as metals, in general, are plastically deformed due to crystal lattice shifts in planes where more space is available. Furthermore, areas within a grain can be separated from each other due to the glide of dislocations, thus, plastic distortions are imported in the crystal lattice [11]. Various studies [12–15] have highlighted a strong correlation between the texture of aluminum alloys, e.g., β-fiber percentage, and their mechanical properties, formability as well as strain rate sensitivity. In particular, the presence of a cube texture tends to worsen the mechanical properties of the material, whereas mixed textures may improve them [16].

The present article aims to underline the effect of uniaxial tensile loading on texture for several thermomechanical states of a 3104 Al cold-rolled sheet (deformed, recovered, and recrystallized). In addition, the response of the microstructural characteristics such as the misorientation grain boundaries is studied to examine whether a correlation among the abovementioned characteristics and anisotropy exists. The effect of different initial microstructures, and, in particular the strong effect of the uniaxial loading is also addressed. Study of the behavior of the different texture components and subsequent substructures under uniaxial loading through the proposed experimental approach is critical and innovative since it could lead an enhanced understanding of the texture evolution. This information could then be used for the induction of the desirable amount of texture components to be able to produce Al sheet with optimum response towards drawing. Towards that direction the texture near the “neck” area of all tested tensile specimens was examined. The obtained results were reviewed and discussed regarding the microstructural characteristics such as the misorientation grain boundaries and r-values. The correlation among the texture evolution and the resulting anisotropy is crucial since studies which combine texture and plastic deformation are of great practical relevance.

2. Materials and Methods

The AA 3104 alloy sheet samples (Table 1) that were examined were produced through direct chill (DC) casting and originated from a 600 mm thickness plate. Additionally, the sheet samples were subjected to hot and cold rolling as well as to thermal treatments. As such, the final samples that were extracted were representative of both the recovered and the fully recrystallized states. Regarding sample designation, a four-letter system was used for the nomenclature as follows: The two first letters indicate the sheet sample state where c.r. stands for cold rolled, rc. stands for recovered, rx. for recrystallization, AR for as-received state, RD for samples subjected to tensile parallel to the rolling direction (RD), 45 for 45° towards RD, TR for transverse to RD. Various annealing temperatures were applied through the range of 200 °C to 340 °C with a 10 h ramp and a 2 h soaking time similar to common industrial practice. Through this process indicative samples were selected for the two aforementioned states. It is noted that sample selection was
based on the use of grain orientation spread (GOS) maps, through which the states under examination were detected.

Table 1. Samples description.

| State                          | Condition                                      | Sample ID |
|-------------------------------|-----------------------------------------------|-----------|
| Cold rolling 90% reduction (deformed) | As received state                             | c.r.AR    |
|                               | After tensile testing of //RD sample          | c.r.RD    |
|                               | After tensile testing of 45° towards RD sample | c.r.45    |
|                               | After tensile testing of ⊥RD sample           | c.r.TR    |
| Recovery annealing (recovered) | As received state                             | rc.AR     |
|                               | After tensile testing of //RD sample          | rc.RD     |
|                               | After tensile testing of 45° towards RD sample | rc.45    |
|                               | After tensile testing of ⊥RD sample           | rc.TR     |
| Recrystallization annealing (fully recrystallized) | As received state                             | rx.AR     |
|                               | After tensile testing of //RD sample          | rx.RD     |
|                               | After tensile testing of 45° towards RD sample | rx.45    |
|                               | After tensile testing of ⊥RD sample           | rx.TR     |

2.1. Tensile Testing

The materials were subjected to uniaxial tensile strength, as per the requirements of the ISO 6892-1 testing standard. In particular, the \( R_p \) (yield strength—YS), \( R_m \) (ultimate tensile strength—UTS) and \( A_g \) (elongation) parameters were measured. The tensile strength specimens were cut, in orientations of 0°, 45° and 90° relevant to the RD. The planar anisotropy parameter \( \Delta r \) (1) and the normal anisotropy parameter \( r_m \) or \( r \)-bar (2) were measured according to [17]:

\[
\Delta r = \frac{r_0 + \frac{r_90 - 2r_{45}}{2}}{2}
\]

\[
r_m = \frac{r_0 + \frac{r_90 + 2r_{45}}{4}}{4}
\]

where \( r_0 \), \( r_{45} \), and \( r_{90} \) are the r-values in orientations of 0°, 45° and 90° relevant to the RDn, respectively.

2.2. Hardness and Conductivity Measurements

The Vickers method was used for hardness testing. A load of 0.2 kgf was selected for hardness measurements whereas for the electrical conductivity measurements, a frequency of 240 kHz was used. The mean values presented originate from the average of 10 measurements.

2.3. Optical Microscopy

Samples were sectioned parallel to the RD and were cold mounted to avoid any non-desirable annealing effects. Metallographic preparation was conducted through successive grinding and polishing steps, whereas the grain structure was revealed through the use of Barker’s electrolytic etching according to references [3,18]. Examination of the grain structure was performed with polarized light and by use of a Nikon Epiphoto 300 inverted metallographic microscope (Nikon, Minato City, Tokyo, Japan). Optical metallographic examination was conducted at a magnification range of 500×–1000× after Barkers etching.
2.4. Metallographic Examination—Texture

Microscopic analysis was performed using a JEOL IT-800 HL Scanning Electron Microscope (SEM, JEOL Ltd., Akishima, Tokyo, Japan) under a 20 kV accelerating voltage, coupled with an EDAX Apollo XF, equivalent to octane super EDS, silicon drift detector (SDD, EDAX, Mahwah, NJ, USA) in conjunction with TEAM (EDAX, Mahwah, NJ, USA) and SMILE VIEW™ Map software (JEOL Ltd., Akishima, Tokyo, Japan) and Digital Surf, Besançon, France). EBSD analysis was carried out using an EDAX Hikari XP EBSD, high-speed camera (EDAX camera, EDEN Instruments SAS, Valence Cedex, France). When a polycrystalline metallic material, is subjected to thermomechanical processing, a dominant orientation, namely texture is observed. The texture could be formed through the deformation, annealing, or as a result of both processes. Texture can be categorized into several components, represented by \( \{hkl\}<uvw> \). The latter are further divided into two groups, consisting of deformation and annealing texture components.

Calculation of the various texture components, resulting from the applied manufacturing process, was performed according to [19]:

- **Rolling components**: Copper \( \{112\}<111> \), S2\{213\}\(-1-42\), S3 (or S) \( \{123\}<634> \), Taylor\{441\}\(<1118> \), Brass \( \{011\}<211> \)

- **Recrystallization components**: Cube \( \{001\}<100> \), P\{011\}<111>, Q\{013\}<2-31>, R\{124\}<211>, Goss\{011\}<100>

EBSD scans were collected through a hexagonal grid. For the EBSD analysis, samples were examined in the as polished condition with a tilt of 70° relative to the electron beam. Voltage was set at 20 kV, while the step size was set at 0.1 µm to 1 µm depending on the material condition and the magnification used (100×–2000×) (examined areas 30×30 µm²–150×150 µm²). Regarding the tensile samples, the areas near the “neck” were further investigated metallographically after the tests. To characterize the fractured samples, EBSD scans at low magnification were also performed. Through this examination, the area exhibiting the higher kernel average misorientation (KAM) values and the higher amount of subgrain boundaries was selected for further analysis at higher magnification. The black points observed in the inverse pole figure (IPF) maps are points with low confidence index and were therefore excluded from the measurements. Scanning data were then further analyzed by means of a post-processing software package (OIM) for the various texture components to be identified and calculated. The misorientation angles were determined according to 2°–5° (subgrain angle boundaries), 5°–15° (low-angle boundaries) and 15°–62.8° (HAGBs). It is noted that misorientation angles less than 0.5° corresponding to dense dislocation, are not considered reliable for evaluation purposes due to the angular resolution of the EBSD [20]. Finally, post-processing was conducted to clean up the data (grain dilation).

3. Results
3.1. Mechanical Behavior (Tensile Testing, Anisotropy)

The lower \( \Delta r (-0.20) \) and highest \( rm \) values (1.44) were recorded in the case of the rc.AR sample (rc.RD, rc.45 and rc.TR). In Figure 1 the effect of thermal treatment to ultimate tensile strength (UTS) is presented. As expected, the specimens that were tempered in a higher temperature appear to have inferior strength, due to their recrystallized microstructure. Likewise, these samples, are more ductile than the c.r.AR sample which exhibited elongation values lower than 5%. An increase of the electrical conductivity (EC) was evident and could be attributed to the precipitation of dispersoids.
The planar anisotropy parameter (Δr) is an indicator of the ability of a material to demonstrate a “non-earing” behavior (lower values are preferable) while r_m, is an excellent indicator of the ability of a material to be subjected to deep drawing (higher is better), (Table 2). The maximum load before fracture was recorded first samples transverse to the RD direction (c.r.TR and rc.TR). After recrystallization, the maximum load values were recorded in the parallel to RD direction (1102 N). A similar behavior was observed for R_m, while the Rp_0.2 values at 90° relative to RD, were the highest among all measurements (Table 2, Figures 1 and 2). Elongation did not exhibit any noteworthy correlation with the direction examined; however, it was observed that after each thermal treatment (recovery and recrystallization annealing) the ductility almost doubled from ≈4% to ≈7% and, eventually, increased to 18%. An inversely proportional correlation was observed in hardness and conductivity measurements, however (Figure 3).

![Image of graph showing tensile strength of samples depending on their relative angle to RD.](image)

**Figure 1.** Tensile strength of the samples depending on their relative angle to RD.

The recovered sample exhibited an isotropic behavior with a maximum deviation of the mechanical properties for each sample close to 10% (Figure 4). With regards to orientations 0°, 45° and 90°, it is noted that the most pronounced shift of mechanical properties during recrystallization occurred in the 90° orientation (YS: from 234 MPa to 75 MPa and UTS: from 259 MPa to 181 MPa) and therefore can be characterized as a more sensitive direction, with a UTS reduction of the order of 76 MPa. In the 0° direction (YS: from 210 MPa to 76 MPa and UTS: from 247 MPa to 188 MPa) a decrease of UTS by 60 MPa is noted, whereas in the 45° direction (YS: from 222 MPa to 75 MPa and UTS: from 243 MPa at 185 MPa) a decrease in UTS of 58 MPa is recorded. Bases on the aforementioned results,
it is noted that as the angle increases from 0° to 45° stress decreases whereas in 90° it increases again to reach its maximum value.

Figure 2. Yield strength of the samples depending on their relative angle to RD.

Figure 3. Conductivity vs. Hardness diagram of the three states examined (cold-rolled, recovered, and recrystallized).

Figure 4. Maximum deviation of the mechanical properties for each sample.
Regarding the planar anisotropy parameters, it is noted that the predominant deformation texture in the case of the rc.AR sample is equal to 1.5 which is the highest among the examined annealing temperatures (Figure 5). It is also mentioned that in the case of rc.AR sample, the calculated $\Delta r$ is equal to $-0.2$. As such a weaker “earing” phenomenon is expected.

![Figure 5](image.png)

**Figure 5.** (a) Plastic strain ratio ($r$ value) of examined sheet samples, (b) in-plane anisotropy in a yield strength, Rp0.2.

The cold-rolled sample exhibited the maximum recorded Rp0.2, Rm, and hardness values compared to the other samples. After the recovery annealing process, all measured parameters decreased except for elongation which almost doubled compared to the previous state. At the fully recrystallized state, the sheet sample exhibited the lowest measured Rp0.2 and Rm values as well as the maximum elongation. The measured $r$-values led to the conclusion that the optimal combination of the resulting properties along with a low possibility of failure during drawing was exhibited by the recovered sample exhibiting $\Delta r = 0.20$ and $r_m = 1.44$. The hardness value decreased during annealing due to the underlying strain relief. At the same time, electrical conductivity exhibited a slight increase indicating a “cleaner” matrix.

3.2. Microstructural Analysis and Fractography

Microstructural evolution of the c.r.AR sample is presented in Figure 6a. It starts with a strongly deformed microstructure (fibrous), and retains its morphology after recovery/annealing. The recrystallization annealing lead to a fully recrystallized microstructure, consisting of coarse equiaxed grains throughout its thickness (Figure 6c).

![Figure 6](image.png)

**Figure 6.** Optical micrographs after Barkers electrolytic etching showing the microstructure of (a) c.r AR, (b) rc.AR and (c) rx.AR.

Metallographic and fractographic inspection of all samples after tensile testing was also performed. An indicative micrograph of sample rx.TR is shown in Figure 7. The directivity of the equiaxed grains due to uniaxial loading, is noted and indicated by the arrows.
The fracture surfaces of the broken tensile specimens were examined by use of SEM. The numerous deep and large dimples observed highlight the ductile nature of the tested material (Figure 8a). An indicative 3D reconstruction of the observed surface is presented below in Figure 8b to provide a quantitative perspective of the observed dimples. All samples exhibited similar fracture features.

3.3. EBSD Scans and Analysis

After the metallographic examination of the samples, EBSD scans were conducted on the fractured samples near the “neck” area. It is noted that due to tensile loading many voids were formed near this area. At the same time, the deformation led to the distortion of the lattice, thus many areas cannot be evaluated with regards to texture. Those points were excluded from post-processing examination due to their low confidence index (CI) value and are marked with black color in all maps (see Figures 9–12). An indicative EBSD scan, which was used to detect the most appropriate area is presented in Figure 9. The fracture
surface is shown in Figure 9a where the prevalence of the (001), (111) and (112) orientations is evident in the IPF map. In Figure 9b the (white) arrows indicate the areas where the highest KAM values were observed, which is also the area where the maximum density of SGBs (Figure 9c) was measured. In the final map of this figure (Figure 9d) the coexistence of both rolling and recrystallization texture components is underlined in the selected area. For the main part of the EBSD data, a comparative map of the samples in the as-received and after-tensile-testing conditions is constructed for all examined orientations. GOS maps were used for the selection of the most indicative samples from all three examined states (deformed, recovered, and recrystallized), (Table 3). The IPF intensity did not exhibit a high variance indicating a non-strong overall texture.

Table 3. Results of misorientation angles, IPF intensities, and GOS percentages.

| Sample ID | Condition   | IPF Intensity | 0°–1° | 1°–10° | GOS  |
|-----------|-------------|---------------|-------|--------|------|
| c.r.A     | Deformed    | 2.7           | 3.7   | 95.1   |      |
| rc.A      | Recovered   | 2.4           | 18.7  | 66.6   |      |
| rx.A      | Recrystallized | 2.0       | 73.3  | 26.1   |      |

As previously observed through the optical micrographs, the sample c.r.A.R exhibited a fibrous, highly deformed microstructure with elongated grains (Figure 10a). After tensile testing, voids were formed, and the resulting texture was significantly influenced. According to Table 4, KAM values increased from 0.3 in the initial state to ≈0.5 after tensile testing in all examined orientations, due to the augmentation of disorders within the grains. The highest KAM value was measured for the cr.TR sample. Regarding the misorientation angles, the highest value recorded was that of low-angle grain boundaries (LAGBs) for all states. After tensile testing, a significant amount of LAGBs was present (≈20%), as expected. The texture components of the cr.AR sample were dominated by rolling texture components. S2 accounted for ≈40% of the identifiable components whereas it was reduced to 10.6%, 1.2% and 0% for samples cr.RD, c.r.45 and c.r.TR respectively, after tensile testing. S3 (usually referred as S), in the cold-rolled state was 12.5% and after uniaxial loading a maximum value of 43.4% was obtained for the sample parallel to the RD whereas it was almost absent in all other orientations. The sum of S2 and S3 before and after tensile testing remained stable at approximately 50% of the overall texture (the brass component exhibited a strong presence and along with the S component, they reached about 60% of the overall texture. The behavior of P and Goss texture components is noteworthy, in the sense that they could barely be detected prior to tensile testing whereas in sample c.r.45 they exhibited an increase of 15.1% and 64.2% respectively, after testing. Augmentation of the P component was detected in the case of sample c.r.TR also, reaching 44.4% of the overall texture. The presence of the R component is also pronounced, and increased in the orientation parallel to the RD whereas it could not be detected in all other orientations. An opposing behavior was observed with regards to Goss components, which are barely detected = in all states except for the c.r.45 sample. The differences between orientations can be explained through an alteration of the IPF plot at the bottom of Figure 9 where the initial state at (101) rotating towards (111) and (112) orientations can be seen. The intensity of the IPFs did not exhibit any significant differences. In the case of the c.r.AR sample the predominant recrystallization texture component is the R at 10.4%, while the predominant crystallographic deformation texture component is the S accounting for more than 40% of the overall texture (Table 4).
Figure 9. Indicative scan of sample rx.RD which was accomplished at lower magnification to detect the areas of interest (indicated by the black arrow). (a) IPF map, (b) KAM map, (c) misorientation angle map and (d) map illustrating the crystallographic components (light blue: rolling components and dark blue: recrystallization components).
Figure 10. EBSD scans and respective maps for sample (a) column CRAR, (b) CRPAR, (c) CR45 and (d) CRTRA.

| Table 4. KAM values, misorientation, and texture components of the cold-rolled sheet sample. |
|---------------------------------|--------|--------|--------|--------|
|                                 | c.r.AR | c.r.RD | c.r.45 | c.r.TR |
| KAM values                      | 0.3    | 0.5    | 0.4    | 0.6    |
| Misorientation angles           |        |        |        |        |
| 2–5°                            | 69.8   | 70.6   | 77.7   | 70.0   |
| 5–15°                           | 9.2    | 23.5   | 15.9   | 23.3   |
| 15–62.5°                        | 21.0   | 5.9    | 6.4    | 6.7    |
| Rolling components              |        |        |        |        |
| Copper (90, 35, 45)             | 1.1    | 12.2   | 0.4    | 0.5    |
| S2 (47, 37, 63)                 | 40.4   | 10.6   | 1.2    | 0      |
| S3 (59, 37, 63)                 | 12.5   | 43.4   | 0.2    | 0.2    |
| Taylor/Dillamore (90, 27, 45)   | 0.3    | 1.8    | 0.5    | 0.1    |
| Brass (35, 45, 90)              | 3.4    | 21.7   | 0.1    | 0.1    |
| Recrystallization components    |        |        |        |        |
| Cube (0, 0, 0)                  | 1.9    | 0.5    | 0      | 0      |
| P (70, 45, 0)                   | 2.3    | 0.9    | 15.1   | 44.4   |
| Q (58, 18, 0)                   | 4.2    | 2.0    | 12.1   | 6.1    |
| R (57, 29, 63)                  | 10.4   | 5.8    | 1.2    |        |
| Goss (0, 45, 0)                 | 1.6    | 2.6    | 64.2   | 1.4    |
The cold-rolled sample exhibited a high amount of SGBs accompanied by a high percentage of S2, S3, and R components. After tensile testing, the SGBs preserved their high percentage in the overall texture whereas the previously mentioned crystallographic components (S2, S3 and R) were accompanied by a quite noticeable increase of brass and copper components for the sample parallel to the RD. At 45° relative to the RD, P, Q, and mainly Goss components dominated the observed texture, reaching altogether approximately 92% of the overall texture. The P component exhibited an increase of 45% for the sample transverse to RD.

The sheet sample (after being recovered) did not exhibit any noteworthy differences with regard to grain morphology whereas it exhibited a higher variation of KAM values compared to the c.r.AR sample in all examined orientations (Figure 11 and Table 5). The maximum KAM value after tensile testing was measured in the orientation of 90° (1.0), and exhibited behavior similar to the sample c.r.AR. As for the misorientation grain boundaries, the recovered sample at the as-received state exhibited a higher amount of HAGBs (57%) compared to the cold-rolled condition, along with a significant amount of SGBs l (35.9%). After tensile testing, an increase of the subgrain boundaries (SGBs) and a decrease of the HAGBs is observed, with a maximum of ≈73% being recorded for the rc.TR sample. In the case of sample c.r.AR, the S2 component exhibited a maximum intensity in the as-received condition and was succeeded by S3 after tensile testing in 0° relative to the RD. Both S2 and S3 amounted for 50% of the overall texture. After recovery, the opposite behavior is observed, where the S3 component prevails at as-received state. Again, the sum of these two rolling components, amounts for 50% of the overall texture in the as-received state as well as after tensile testing in 0°. The behavior of the brass and cube components
is also noteworthy since, brass decreases from 30% to 12% in sample rc.RD, while cube increases from 1.5% to 5.6%. An analogous, to the previous sample, presence of P and Goss components, as well as Q is detected. P and Goss increased in sample rc.45 (from 0.3% and 0.5% to 9.4% and 5.7% respectively) whereas Q increased in the case of sample rc.TR (from 1.1% to 19.4%). The R component continued to be the dominant in the final recrystallization texture components, accounting for 10.4% of the overall texture for the rc.TR sample and an even higher percentage of 33.4% after tensile testing in the rc.RD sample whereas in the other direction, it was vanished. The dominant (101) orientations were retained after the annealing process whereas after the deformation process that tendency altered since the grains were more prone to rotate towards the (112) and (001) orientations. The (101) orientation of the crystal is parallel to the assigned sample direction while after tensile testing a double fiber is provoked, namely the (001) and (112).

For the final examined state, the recrystallized, rx.AR sample, exhibited a low KAM value (Table 6). HAGBs dominated the rx.AR samples since it mainly consisted of well-formed equiaxed recrystallized strain-free grains, a condition that was not preserved after tensile testing. In 0° and 90° textures were dominated by SGBs whereas in 45° HAGBs were mostly evident. In addition, the copper component decreased, whereas S2, S3, Taylor, and brass increased. P increased significantly in 90° while Q increased in all directions with the maximum being observed in the 45° orientation. The amount of Goss was reduced from \(\approx24\%\) to \(\leq6\%\) for all orientations. Finally, the initial state exhibited a dominance of
(112) and (313) orientations whereas after the deformation process the (001), (112), and (111) were the most prominent orientations that were observed. A weaker texture according to IPFs was observed in the as-received condition whereas a 50% increase was observed after tensile testing.

Table 5. KAM values, misorientation, and texture components of the recovered sheet sample.

|                  | rc.AR | rc.RD | rc.45 | rc.TR |
|------------------|-------|-------|-------|-------|
| KAM values       | 0.1   | 0.8   | 0.9   | 1.0   |
| Misorientation angles |
| 2–5°             | 35.9  | 51.9  | 43.8  | 72.6  |
| 5–15°            | 7.1   | 28.3  | 29.1  | 16.6  |
| 15–62.5°         | 57.0  | 19.8  | 27.1  | 10.7  |
| Rolling components |
| Copper (90, 35, 45) | 40.8  | 8.3   | 0.7   | 0.6   |
| S2 (47, 37, 63)   | 6.5   | 15.6  | 2.7   | 0.4   |
| S3(59, 37, 63)    | 45.2  | 31.6  | 1.3   | 0.2   |
| Taylor/Dillamore (90, 27, 45) | 2.6   | 2.4   | 0.7   | 0.2   |
| Brass (35, 45, 90) | 29.5  | 11.9  | 0.5   | 0.5   |
| Recrystallization components |
| Cube (0, 0, 0)    | 1.5   | 5.6   | 1.2   | 1.4   |
| P (70, 45, 0)     | 0.3   | 0.6   | 9.4   | 7.2   |
| Q (58, 18, 0)     | 1.1   | 3.9   | 2.9   | 19.4  |
| R (57, 29, 63)    | 5.6   | 6.1   | 0     | 0.4   |
| Goss (0, 45, 0)   | 0.5   | 2.3   | 5.7   | 0.1   |

Table 6. KAM values, misorientation, and texture components of the recrystallized sheet sample.

|                  | rx.AR | rx.RD | rx.45 | rx.TR |
|------------------|-------|-------|-------|-------|
| KAM values       | 0.1   | 1.0   | 0.8   | 1.0   |
| Misorientation angles |
| 2–5°             | 3.4   | 70.4  | 39.4  | 63.7  |
| 5–15°            | 4.6   | 8.1   | 11.5  | 8.3   |
| 15–62.5°         | 92.0  | 21.6  | 49.1  | 28.0  |
| Rolling components |
| Copper (90, 35, 45) | 6.4   | 3.6   | 5.1   | 5.8   |
| S2 (47, 37, 63)   | 22.0  | 3.0   | 11.3  | 2.9   |
| S3(59, 37, 63)    | 1.4   | 15.9  | 5.8   | 9.4   |
| Taylor/Dillamore (90, 27, 45) | 1.1   | 13.2  | 2.8   | 2.5   |
| Brass (35, 45, 90) | 8.5   | 12.1  | 0.5   | 2.8   |
| Recrystallization components |
| Cube (0, 0, 0)    | 0.1   | 1.3   | 0.4   | 9.1   |
| P (70, 45, 0)     | 2.6   | 0.8   | 3.7   | 10.8  |
| Q (58, 18, 0)     | 8.2   | 8.4   | 10.0  | 8.8   |
| R (57, 29, 63)    | 0.1   | 6.7   | 4.3   | 4.8   |
| Goss (0, 45, 0)   | 23.6  | 0.0   | 5.6   | 0.8   |
The highest tensile strength was measured in the case of the cold-rolled sample where a rotation from (101) to (112) and (111) is also noted. The recovered sample initiated with (101) as well and rotated mainly towards (112) and to a lesser extend to (001) orientations. The recrystallized sample exhibited (112) and (313) orientations which were partly retained (112), (001) and (111). The cold-rolled sample and the recovered sample, did not exhibit significant differences with regards to texture and the dominant orientations. A different behavior however was observed after tensile testing due to the lack of the effect of dislocations inside the recovered grains. The main orientations rotation observed was (101) to (112). This shift resulted into a higher stiffness of the examined sheet samples in compared to the recrystallized sample where no significant orientation shift was noted (the (112) orientation was present before and after tensile).

The texture components are qualitatively represented by the orientation distribution functions (ODF) maps below. Although IPFs did not exhibit a strong texture in some cases, ODFs highlight the presence of the crystallographic components and their solid presence for each sample (Figure 13). The black arrows indicate the positions of the components, with an intensity high enough to be measured and evaluated.

![Figure 13. ODFs of the examined samples.](image)

**4. Discussion**

In the present study the microstructure and texture properties of Al3104 sheet samples were evaluated by means of optical and scanning electron microscopy with EBSD in order study the correlation of the observed texture components resulting from the deformation and annealing processes with the measured r-values.
With regards to samples subjected to tensile testing, an improved behavior was reported for the recovered sample since it maintained its toughness at a significant degree and at the same time exhibited a significantly higher elongation value. This is further supported by the measured $\Delta r$ and $r_m$ values indicating an optimum response towards drawing among the samples examined. Hardness decreased due to the re-arrangement of dislocations induced during cold rolling. Electrical conductivity was elevated as the Al matrix increased through the annealing process. This was mainly due to the increased average free movement distance of the electrons and secondly due to the re-arrangement of the alloying elements within the matrix during the annealing process.

The EBSD measurements underlined the significant effect of the deformation process on different areas of the microstructure. A shift of the crystallographic orientation during thermomechanical processing is observed through the evolution of SGBs. This is the key parameter for the correlation between anisotropy, mechanical behavior, and grain orientation. The slip pattern was found to be the connecting factor of a microstructural characteristic such as the misorientation angle boundaries and the crystallographic orientation obtained during deformation [21]. The recovered state of the Al sheet was examined since dislocations and misorientation grain boundaries response in relation to tensile testing and subsequent fracture is obscure [22]. Different amounts of misorientation boundaries, differences in morphology, dislocation density, and stored energy all had a great impact on the material’s response towards recovery and recrystallization. Most of the dislocations concentrated at the boundaries are induced through active slip systems. The total shear can be separated towards different slip systems and according to Taylor [23], five slip systems represent the required number of slip systems for the shape changes in an undergoing deformation. However, inside the cell block, maybe less than five slip systems could be sufficient to induce dislocation jog formation [24]. Dislocation boundaries form a subdivision of the deformation microstructure, where various characteristics are provoked through different orientations of the initial deformed grain. This correlation is important since the microstructural characteristics had a great impact on the mechanical behavior of thermomechanically treated metals [22]. The dislocation boundaries which exhibit a correlation with the slip planes (111) and a 5° difference among the slip lines and the dislocation boundaries are characteristic of deformation from a crystallographic perspective. At the same time, structure morphology, misorientation angles and dislocation boundaries indicate the presence of variations with regards to dislocation density and stored energy [22].

For Al sheets produced through cold rolling, such as those examined in the present study, the Schmid factor analysis indicates that the augmented number of available slip planes leads to changes of the orientation of the boundaries. The orientations obtained non-crystallographic positions [25], from a state of being almost parallel to slip planes. Therefore, it could be reasonable for one to correlate the microstructural characteristics and the behavior of the sheet after plastic deformation. In all examined cases, the number of active slip systems is reduced during plastic deformation to ease the deformation locally. This behavior is expected when the deformation is inhomogeneous [26], a condition not relating to slip zones. After the uniaxial loading, which results to fracture, transformations of SGBs to HAGBs were evident, in the rx.45 sample, which exhibited a homogeneous distribution of the slip zones over the crystal volumes thus it shed light to the “fragmentation” as well as the “mosaic” of SGBs into the crystal [26]. The differences with regards to the evolution of the SGBs could be attributed to neighboring regions which are suppressed in different lattice rotations.

The main structural parameters considered are the following: (i) boundary morphology, (ii) crystallographic orientation, (iii) crystallographic texture, (iv) misorientation angle boundaries, which express the deformation of the material in different terms in comparison to the macroscopic plastic measurements in small to medium strains. In higher strains (>1) lamellar bands (LBs) are formed. When an Al sheet is subjected to deformation, a part of the mechanical energy is stored (as dislocations mostly). Dislocations are not
allocated inside the material randomly. They are concentrated at dislocation boundaries functioning as dividers of low dislocation density areas. This subdivision is common for the fcc alloys with medium to high SFE. Increased strains led to an average misorientation angle of $\geq 15^\circ$. Parts of the grain rotate differently towards various rotations inside the grain, and consist of the observed texture components [21]. This phenomenon is observed in highly deformed grains, and is visually represented in IPF maps as a chromatic difference in the interior of the grain. Such differences can be the result of localized shear [21,27]. From a microstructural point of view the pattern that is created due to rolling relates to the arrangement of dislocation boundaries planar cell blocks measured as geometrically necessary dislocations (GNDs)] [21]. The dislocations located at the boundaries are known as GNDs (GND Nye tensor). The formation of those dislocations relates to the lattice rotation required for the individual grains of the polycrystalline material to be plastically deformed, and convert into SGBs, [28]. Knowledge of the distribution of the misorientation boundaries could clarify the subsequent behavior observed during recrystallization, since the state of the deformed microstructure determines the process of nucleation and subsequent growth. The misorientation boundaries lead to an estimation of the stored energy [29]. Dislocations “jump over” through small SBGs easier than larger ones, leading to the assumption that smaller subgrains are harder [30,31]. This could also be understood by the fact that boundary conditions of higher order are expected from the lattice rotation of smaller SGBs. This could be related to the higher $R_m$ accompanied by a higher amount of SGBs in sample rx.RD.

With regards to texture components, the Dillamore component relates to the Cu through a rotation of $8^\circ$ relative to the transverse direction (TD). In the recovered state of the rc.AR sample, well-defined SGBs and thus dislocation-free grains were observed, indicating the high mobility of the dislocations as well as the fact that they are rearranged through cross slip and climb during deformation. Due to the existence of shear bands, a component Q $\{013\}$ $<231>$ forms, which appears in significantly lower texture intensities compared to cube texture. This explains the high amount of Q component observed in the cold-rolled sample. Concerning the particle stimulated nucleation (PSN) mechanism, it is important to note that the cube orientation tends to increase at the expense of the PSN orientations, such as P, and the precipitation of $\text{Mg}_2\text{Si}$ particles tends to impede PSN more strongly than the nucleation at the cube bands [7].

Additionally, another recrystallization texture component appears in rolled and annealed aluminum sheets, namely the R $\{124\}$ $<211>$ component. It mainly appears due to the presence of Fe in the alloy and indicates either in-situ recrystallization or a strain-induced boundary migration nucleation process with subsequent growth. The R texture is usually interpreted as, a retained rolling texture S component although it exhibits distinct differences in its intensity and its exact positioning therefore their observed coexistence in the examined samples could be explained. With regards to the component’s growth, it appears that the R orientation emerges and grows substantially [7]. The R component is retained from rolling texture components in the cases with reduced stored dislocation energy (at recovery state), as it observed in the rc.AR sample. The high percentage of S components was also important as these grains, favor the nucleation of R components as well [32] between the deformed bands. The balance between deformation textures and annealing textures is crucial to controlling earing in beverage can stock [10,33]. The solute Mn plays an important role in decreasing the EC value of Al alloy [34]. Therefore, the lower hardness but higher EC value can possibly be correlated with a lower content of Mn in solid solution. It has been found that the higher the intensity of the deformation Cu component, the stronger the intensity of the subsequently produced cube component. Similarly, the intensity of the Goss component is directly related to the strength of the brass component which exhibited an intense presence in the cold rolled and recovered samples. Generally, brass and copper exhibit a greater tendency to recrystallize discontinuously, whereas at these cases S is retained from the previous cold rolling states. P and Q components originate in deformation inhomogeneities, i.e., particles or shear bands [13,35]. The SGBs cannot
“escape” from the particles and therefore the evolution of the substructure is controlled by subgrain growth which is directly correlated with the presence of second phase particles. The formation of a high percentage of HAGBs was observed at the rx.AR sample with a microstructure resembling a material subjected to discontinuous recrystallization. The driving force for the subgrain growth is directly proportional to the shape factor and misorientation dependent stored energy of the subgrain array and inversely proportional to the radius of the subgrains. Subgrains can grow through two mechanisms, i.e., boundary migration and coalescence of subgrains. In the case of boundary migration, subgrain coarsening occurs due to the migration of the low-angle boundaries. Large subgrains will grow at the expense of small subgrains and the exact mechanism will rely on the dislocation structure at the boundaries as well as the mobility of the triple junctions. Subgrains also grow through rotation assisted coalescence of subgrains. The recrystallized grains of the rx.AR sample exhibited quality patterns of higher quality and confidence index values from recovered grains. In addition, no orientation variation inside the grains was observed. Through the shift of the texture maximum observed at the ODF maps in the several \( \phi_2 \) sections, \( R \) oriented grains exhibited a competitive behavior towards brass, copper, and S. The SBGs formed near S orientations are more favorable to grow [32].

An optimal combination of a minimum \( \Delta r \) and a maximum \( r_m \) value was observed for the recovered state sample (rc.AR). The high amount of S components (S2 and S3), the strain-free elongated grains accompanied by an average amount of SGBs as well as the high amount of HAGBs in the as-received state, obstruct dislocations from overpassing them. The contribution of precipitates and dispersoid particles could be neglected if the precipitate spacing is greater than the mean free path of the electron, which is possibly the case as indicated by conductivity measurements. In the intermediate range of precipitate and dispersoid particle spacings commonly observed in commercial aluminum alloys, the contribution of precipitates to electrical resistivity is less clear [36–38]. A deformed material exhibiting high dislocation density initially forms cells and then exhibits annihilation of dislocations within the cells resulting into subgrain formation [30]. The increase of EC was evident in the rx.AR sample and could be attributed to the precipitation of dispersoids. The percentage of recrystallization components after each thermal treatment, increased slightly for the lower annealing temperature (rc.AR) and more dynamically for the higher ones (rx.AR). IPF intensities did not exhibit high variations (2.0 to 2.5) indicating a relatively weak overall texture. In the as-received condition, the most prominent component was S whereas after annealing, the most prominent components were R, Goss, and Q. Conventional or discontinuous dynamic recrystallization involves the nucleation of new, dislocation-free grains followed by their subsequent growth through the migration of HAGBs. Such nucleation occurs in areas where the strain and orientation gradients are high. As such, the process can be differentiated from recovery (where HAGBs do not migrate) and grain growth (where the driving force is the reduction in the boundary area). During plastic deformation, the work performed is the integral of the stress and strain in the plastic deformation regime. Although most of this work is converted to heat, some fraction (~1–5%) is retained in the material as defects—particularly dislocations.

5. Conclusions
The most significant conclusions of the presented work are summarized below:

- The optimal combination of \( \Delta r \) and \( r_m \) values was observed for the recovered sample.
- The S component exhibited a noteworthy behavior since, S2 and S3, namely similar components, retained the 50% of the texture before and after tensile testing in an orientation parallel to the RD.
- The texture components exhibited gradual orientation transitions over various layers of SGBs.
- The most prominent recrystallization texture component in the cold rolled and recovered states was R due to the previous intense presence of S components.
The recovered sample exhibited a recrystallization texture leading to better results with regards to the deep drawing behavior of the material, an assumption that was further supported through the measured r-values.

After uniaxial loading, the subgrain structure exhibited a gradual transformation to HAGBs in the recrystallized sample at 45° while a reinforcement of the SGBs was observed in all other examined cases.

The (101) rotation towards (112) led to satisfying results regarding the resulting mechanical response.

The combination of a high number of S-oriented grains, which were at the same time, strain-free due to recovery, as along with the presence of HAGBs and secondarily by SGBs, constituted the ideal substructure regarding the $\Delta r$ and $r_m$ values. The aforementioned state is expected to lead into better results with regards to the deep drawing behavior of the material.

Author Contributions: Conceptualization, S.P. (Sofia Papadopoulou); Methodology, S.P. (Sofia Papadopoulou), S.P. (Spyros Papaefthymioub); Software, S.P. (Sofia Papadopoulou); Validation, S.P. (Sofia Papadopoulou); Formal Analysis, S.P. (Sofia Papadopoulou); Investigation, S.P. (Spyros Papaefthymioub), Z.Z.; Resources, S.P. (Sofia Papadopoulou), Z.Z.; Data Curation, S.P. (Spyros Papaefthymioub); Writing—Original Draft Preparation, V.L.; Writing—Review and Editing, S.P. (Sofia Papadopoulou) and S.P. (Spyros Papaefthymioub); Visualization, S.P. (Spyros Papaefthymioub); Supervision, S.P. (Sofia Papadopoulou) and S.P. (Spyros Papaefthymioub); Project Administration, S.P. (Sofia Papadopoulou). All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Raw data are restored in the relevant electronic folders of the testing equipment.

Acknowledgments: The authors would like to express their gratitude to ELKEME S.A. and ELVAL S.A. managements for all the kind support.

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

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