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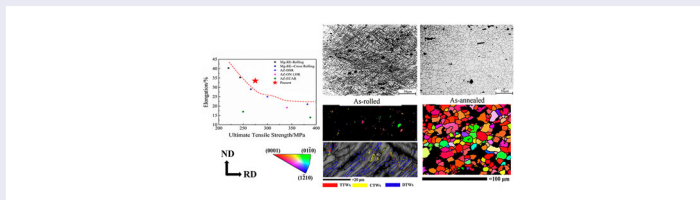
Multiple-twinning induced recrystallization and texture optimization in a differential-temperature-rolled AZ31B magnesium alloy with excellent ductility

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ABSTRACT

The effect of differential roller temperature (DTR) (low and high roller temperature difference: 60 and 180 K) on the microstructure evolution, deformation mechanism, and mechanical properties were investigated in a commercial AZ31B alloy sheet. Compared with the LDTR sample, a considerable shear strain (>0.4) in the HDTR sample led to a multiple-twinning system, especially the formation of contraction twins (CTWs) and double twins (DTWs). After annealing, the HDTR-annealing (HDTR-A) sample achieved an excellent ductility ($\sim 33.6\%$) as a result of a higher recrystallization fraction ($\sim 71.75\%$) and more optimized texture component (max basal texture intensity decrease from 36.13–8.06).



IMPACT STATEMENT

A new method, differential temperature rolling, was used in AZ31B Mg alloy to induce multiple-twinning systems, which strongly influences the recrystallization mechanism and texture evolution, leading to a high ductility.

ARTICLE HISTORY

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KEYWORDS

Magnesium alloys; microstructures; twinning; deformation mechanisms; recrystallization

1. Introduction

Magnesium (Mg) and its alloys are attractive as green engineering materials, especially in vehicles, due to their low density, high strength to weight ratio, and electromagnetic shielding capacity [1–5]. Unfortunately, the application of Mg alloy sheets in the industry remains limited predominantly due to its low ductility and strong mechanical anisotropy, which is caused by insufficient independent slip systems and strong basal texture at room temperature. Thus, the primary structure strategy aims to act more deformation modes and weaken or randomize the detrimental basal texture, which is crucial to manufacturing Mg alloy with a good combination of high specific strength, good ductility, and excellent formability [1, 6].

Numerous works have been performed to introduce and control dynamic recrystallization (DRX) for texture optimization. Generally, the DRX mechanisms can be

specially divided into continuous DRX (CDRX) and discontinuous DRX (DDRDX) based on the nucleation and growth mechanisms [7–10]. The newly-recrystallized grains formed by rearranging dislocations in the CDRX process inherit parent orientations [8,11–13]. However, the DDRDXed grains, which show distinct rotational orientations, mainly nucleate at serrated high angle grain boundaries (HAGBs) by bulging and growing through grain boundary migration [8,14–16], and thus, it is beneficial for the randomization of texture. Regrettably, the activation of the DDRDX mechanism is difficult [10,17,18]. Moreover, deformation twinning [19,20] is crucial to accommodate the plastic strain along the c-axis of Mg alloys for generalized plastic deformation. It has been widely reported that the promoted twins can influence the recrystallization behavior and texture development, referred to as twin-induced recrystallization (TRX) [21–27]. Generally, the $\{10\bar{1}1\}$ contraction twins (CTWs)

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and $\{10\bar{1}1\} - \{10\bar{1}2\}$ double twins (DTWs) [23,24,28,29] are thought as effective nucleation sites for recrystallization since its high internal stored energy for the subsequent recrystallization and growth [22,23,26,27,30–32]. In S.M.Fatemi, et al. recent study [27], the twin-related DRX can promote texture rotating along the normal direction at 250 °C. Moreover, Guan D, et al. reported that twin–twin and twin–grain boundary (GB) intersections could provide effective recrystallization sites and weaken basal texture [24,25,27]. Therefore, it is meaningful to study how to activate more twinning systems and their interactions for the favorable recrystallization of Mg alloys, provided that premature failure does not happen..

It is well known that conventional rolling (the isothermal and constant speed conditions) inevitably causes a robust basal texture of Mg at room temperature. Differential speed rolling (DSR) can enhance the mechanical properties of Mg by introducing an additional amount of strain [28,33,34]. Recently, a new method, differential temperature rolling (DTR), is successfully used to improve the mechanical anisotropy and optimize the texture of 3xx aluminum [35]. However, the report about DTR is limited, especially in Mg. In this work, the DTR method has been adopted in this studied AZ31B Mg alloys to introduce the shear strain for manipulation of twinning, recrystallization, and randomization of texture. The influence of roller temperature difference (low and high) on the microstructure characteristics, including the deformed substructure, recrystallization behavior, and texture evolution, was analyzed by electron backscatter diffraction (EBSD). Especially, this work focused on the role of multiple-twinning systems on the recrystallization mechanism and its contribution to the mechanical properties. Finally, the relationship between differential temperature rolling procedure, microstructure, and mechanical properties was investigated in detail to provide a new guide for designing Mg alloys sheets with good ductility.

2. Materials and methods

The materials used in this study were commercial AZ31B Mg alloy sheets with a chemical composition of 3.1Al-0.9Zn-0.32Mn-0.012Si-0.0021Fe-Mg (wt. %). The sheets were machined into samples with a dimension of 50 mm in the rolling direction (RD), 30 mm in the transverse direction (TD), and 5 mm in the normal direction (ND). The temperature of the upper roller was held on 513 K, and the lower roller was set as two different temperatures: 453 K (the difference of the rollers' temperature was 60 K, named as low differential temperature rolling (LDTR)) and 333 K (the difference of the rollers' temperature was 180 K, named as high

differential temperature rolling (HDTR)), respectively. Before rolling, the sheets were preheated at 673 K for 30 min. After the first rolling step, the sheets were reduced from 5 mm to 3 mm and then were heated at 673 K for 5 min. Subsequently, the TR rolling route was used [33] to perform the second step rolling (the sheets were reduced from 3 mm to 2 mm) to avoid the microstructure difference between the top and bottom half part of the rolling samples and get homogeneous microstructure. Finally, the rolled-samples were annealed at 573 K for 10 min, and the as-rolled and annealed samples were marked as LDTR, HDTR, LDTR-A and HDTR-A, respectively. Particularly, parallel marks were carved on the RD-ND surface to semi-quantify and evaluate the shear stress component.

A room temperature tensile test was employed using a SANS test machine with a strain rate of $3.33 \times 10^{-3} \text{ s}^{-1}$, and an extensometer was used during the tensile strains. The uniaxial tensile specimens with a gauge of $10 \pm 0.1 \text{ mm}$ and a width of $14 \pm 0.1 \text{ mm}$ were cut along the RD. The scanning areas were the middle part of samples since the top and bottom half part of the rolling samples did not show a noticeable difference. The optical microscope (OM) observation, X-ray diffractometer (XRD), and EBSD were used to investigate the overall microstructure morphology and grain orientation. The cross-sectional OM samples were mechanically polished and etched with a solution of 1 g oxalic acid, 1 ml nitric acid, 1 ml acetic acid, and 150 ml deionized water. The XRD samples were progressively smoothed using 600–2000-grit silicon carbide paper and mechanical polished. The macro-texture of the samples was determined using an X-ray diffractometer (D/max 2200PC) with Cu $K\alpha$ radiation at 40 kV and 30 mA and analyzed by LABO-TEX Analysis software. The EBSD samples were prepared by ion etching and then observed within an FEI Quanta200 FEG scanning electron microscope equipped with Oxford Instruments Nordlys Nano EBSD system (acceleration voltage 20 kV, step size $0.07 \mu\text{m}$). Textures, grain sizes, and other related microstructural features were acquired from EBSD data using HKL CHANNEL5 analysis software. The recrystallized grains were distinguished by the internal average misorientation angle within the grain [24,36]. The kernel average misorientation (KAM) values were used to indicate geometrically necessary dislocations (GNDs) [37,38].

3. Results

3.1. OM and mechanical properties

The overall optical microstructure and corresponding mechanical properties of LDTR, LDTR-A, HDTR, and HDTR-A samples are shown in Figure 1. Table 1 shows

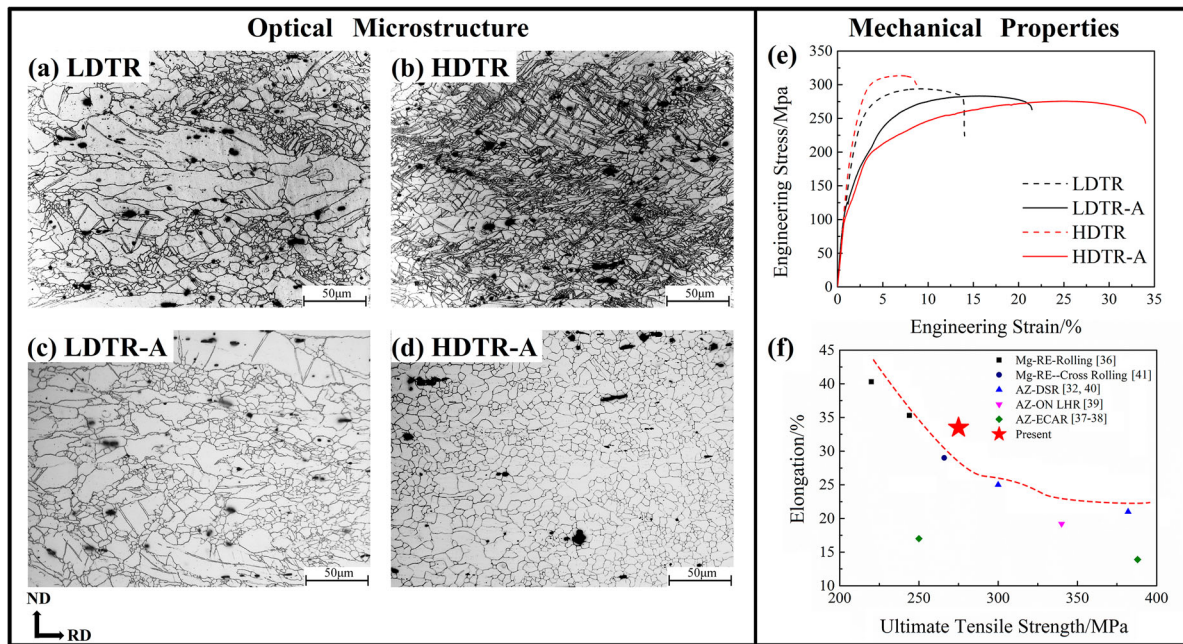


Figure 1. The OM graphs of the (a) LDTR, (b) HDTR, (c) LDTR-A and (d) HDTR-A samples; (e) corresponding mechanical properties and (f) comparison of ultimate tensile strength versus ductility for processed Mg alloys [33,39–44].

Table 1. The engineering mechanical properties of the different samples.

Sample	Yield Strength/MPa	Ultimate Tensile Strength/MPa	Total Elongation/%
LDTR	173.3 ± 1.0	293.0 ± 1.2	13.4 ± 0.2
LDTR-A	131.3 ± 4.0	280.0 ± 2.4	20.4 ± 0.4
HDTR	201.7 ± 2.2	314.3 ± 2.8	8.1 ± 0.4
HDTR-A	119.7 ± 4.3	275.0 ± 2.4	33.6 ± 0.3

the statistic tensile properties, including the YS, UTS, and TEL values of the as-rolled and annealed samples after different DTR procedures. Both the LDTR and HDTR samples show high yield strengths (YS) but low total elongations (TEL) values (Figure 1e). But after annealing, the HDTR-A sample exhibits a more significant ductility enhancement (from 8.1% to 33.6%). For overall optical microstructure, the LDTR and HDTR samples show inhomogeneous microstructure features, which consist of coarse grains, newly-recrystallized grains, and deformation twins (Figure 1a–b). However, the HDTR sample exhibits significantly larger amounts of twin boundaries with different directions and finer twin spacing than the LDTR sample. Moreover, some twin-twin intersections are observed in the HDTR samples, which is hardly found in the LDTR sample. After annealing, the overall morphology of the LDTR-A sample remains inhomogeneous, and there exist a high volume fraction of twin boundaries inside the coarse grains (Figure 1c) than that of the HDTR-A sample. A more homogeneous microstructure

and finer recrystallized microstructure are obtained in the HDTR-A sample (Figure 1d).

3.2. Texture evolution

Figure 2(a–d) exhibits the inverse pole figure (IPF) and pole figure (PF) maps of LDTR, LDTR-A, HDTR, and HDTR-A samples. For the LDTR sample, the occurrence of multi-orientations can demonstrate the existence of dynamic recrystallization (DRX, ~9.77%) after rolling (Figure 2a). However, many coarse grains (about 13 ~ 34 μm) still exist inheriting the parent orientation even after annealing (Figure 2c). For the HDTR sample, a higher number density of twins and a lower number fraction of DRX (4.03%) are observed in the HDTR sample after rolling. Its texture component tilts toward RD and TD (Figure 2b) with maximum intensity (MI) of 36.13. However, it seems that the deformation twins in the HDTR-A sample have disappeared after annealing, and instead, it is found that plenty of homogeneous recrystallized grains with an average grain size of ~5.25 μm exhibit random orientations. This contributes to the lowest basal texture intensity of 8.06 (Figure 2d). In addition, the macro-texture of LDTR-A and HDTR-A samples, including (0001), (10 $\bar{1}$ 0), and (10 $\bar{1}$ 1) pole figure (PF) is tested by XRD (Figure 2 e–f). The formation of the double-peak pole figure within (0001) PF indicates that there exist partly twins in the LDTR-A sample (Figure 2e) [29]. Differently, in the HDTR-A sample, some extra

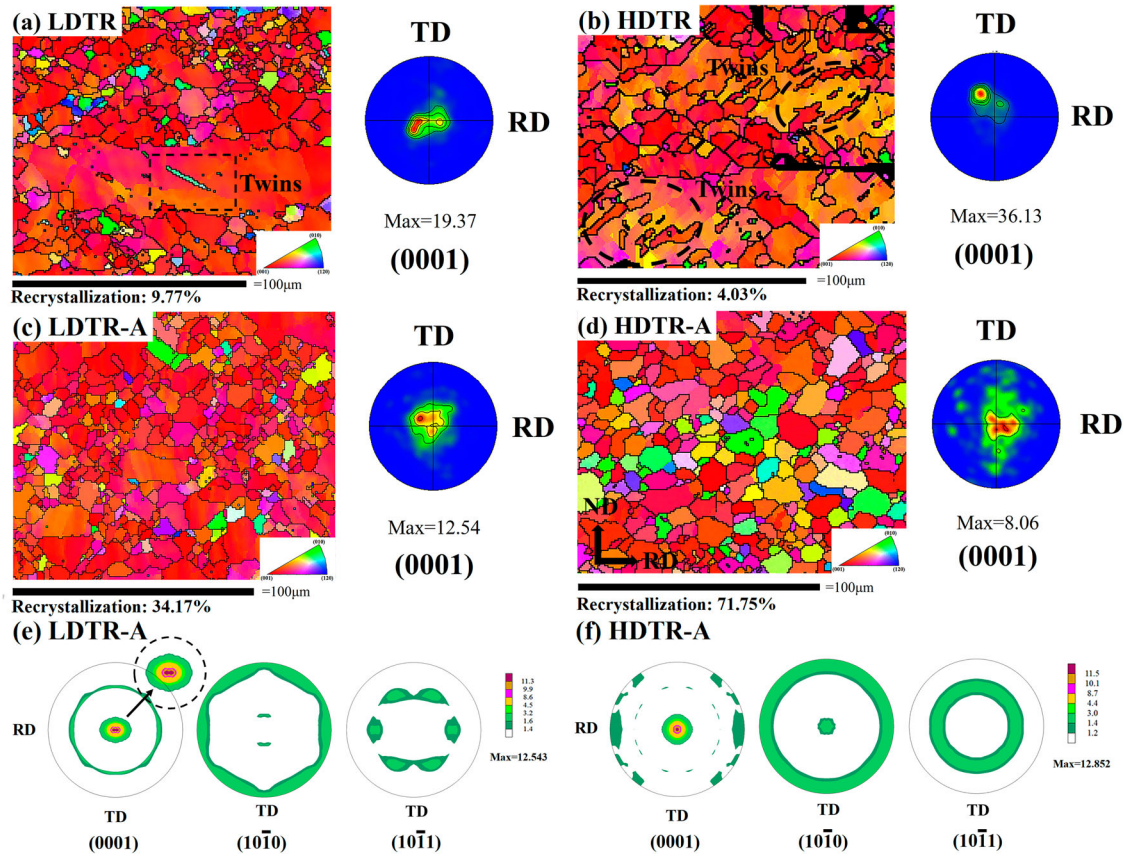


Figure 2. The EBSD maps including inverse pole figure (IPF) map and pole figure of (a) LDTR, (b) HDTR. (c) LDTR-A and (d) HDTR-A; and the XRD maps of (e) LDTR-A and (f) HDTR-A samples.

non-basal texture can be found apart from the typical (0001) basal texture (Figure 2f).

4. Discussion

4.1. The role of shear strain on the deformation mechanism during the DTR process

In our present work [35,45], it has been demonstrated that the DTR can generate temperature gradient and shear stress. According to elastic-plastic mechanics [46,47], the equivalent strain (ε_{eq}) and shear strain (γ) was calculated to quantitatively analyze the shear strain under different DTR processes after first pass. The detailed data is shown in Table 2. The tilted angle of the HDTR sample is higher than that of the LDTR sample. A large part of the overall equivalent strain in the HDTR sample, while the compression strain dominates the deformation of the LDTR process. Clearly, increasing

the temperature gradient, the HDTR process can introduce a higher shear strain ($0.40 \sim 0.57$) than that of the LDTR process ($0.05 \sim 0.15$).

For the LDTR process, some non-basal slip, shear bands (Figure 1a), and twinning modes (especially TTWs) can be motivated. The narrow shear bands provide areas for DRX. Moreover, the limited extra shear strain, high temperature, and large driving force leading to a higher degree of DRX. Two specific regions, R1 and R2 of the LDTR sample, are selected to investigate the DRX mechanism, as shown in Figure 3a. The typical CDRX mechanism and DDRX mechanism can be observed in R1 and R2, respectively. The orientation of new CDRXed grains (Figure 3g) is similar to the parent grains. However, the new DDRXed grains exhibits random orientations, which agree with Zhang et al. reports [7,8,10,48]. The CDRX and DDRX coexist in the LDTR sample. According to numerous studies reported [13,49–52], traditional Mg alloys are thought

Table 2. The shear strain and equivalent strain of LDTR and HDTR processes.

Process	d_0/mm	d/mm	Reduction/%	Tilted angle/ $^\circ$	γ	ε_{eq}
LDTR	5	2	40	5 ~ 15	0.05 ~ 0.15	0.59 ~ 0.60
HDTR	5	2	40	35 ~ 45	0.40 ~ 0.57	0.63 ~ 0.68

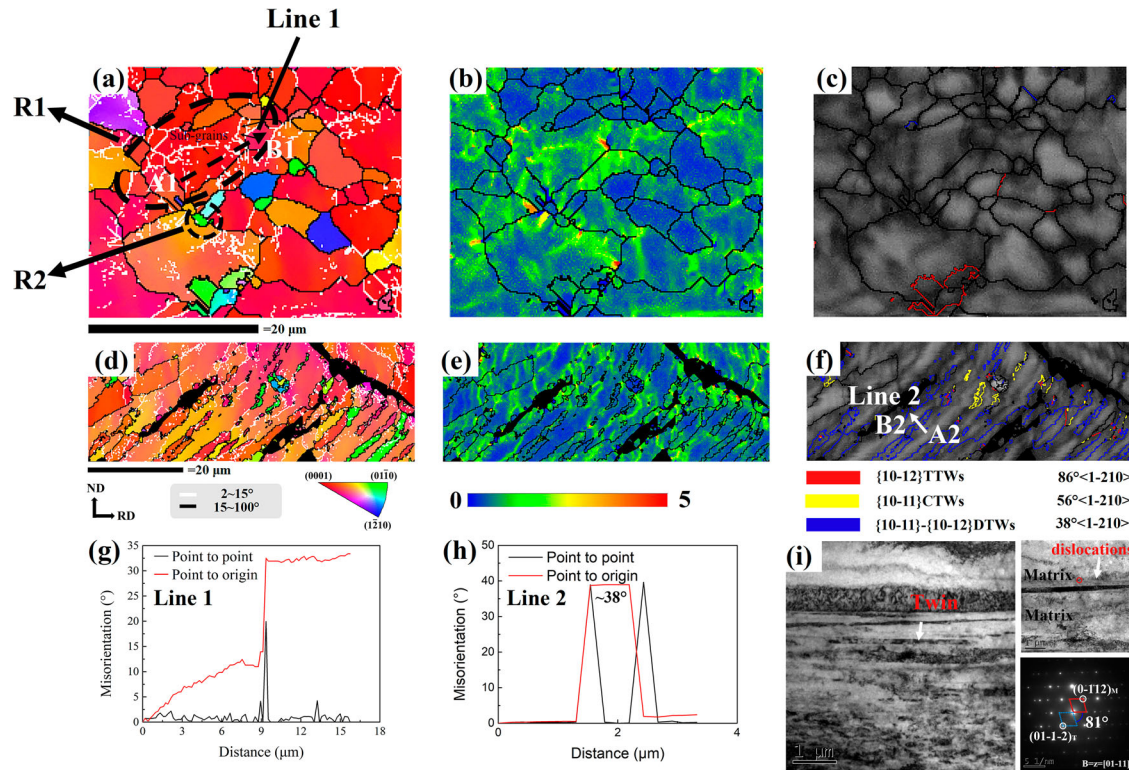


Figure 3. The EBSD maps of LDTR sample ((a) IPF, (b) KAM map, (c) BC) and HDTR sample ((d) IPF, (e) KAM map, (f) BC); and the corresponding line graph of the misorientation angle along line1 (g), line2 (h); and TEM image showing twins (i).

to recrystallize uniformly at high temperature (i.e. continuous dynamic recrystallization (CDRX)). Our results (Figure 2a and Figure 3a) show agreement with them as well. Differently, in the HDTR sample, a combination of various deformation modes, including shear bands and the multiple twinning system is activated under a more considerable shear strain ($0.40 \sim 0.57$), which can be only occur during the severe plastic deformation (SPD) process, such as ECAP and DSR [28,33,53], or under an enormous compression strain [54,55]. A more significant number of twins, including CTWs and DTWs, than that of shear bands, are observed (Figure 3f and i), while the DRX process is suppressed, which may be attributed to a low driving force due to a lower deformation temperature even if the stored strain energy is very high. The HDTR sample shows a meager recrystallization degree (4.03%) and a high average KAM value (1.724°). Although some shear band nucleation could be found in Figure 1b, almost total twins disappear after annealing indicating the occurrence of twin-introduce recrystallization and the effect of multiple twin systems on grain nucleation and texture modification. Moreover, according to S.M.Fatemi, et al. report [26], those triangle-shaped grains (Figure 3f) may be attributed at least partly to the occurrence of ‘twin-assisted’ recrystallization. The part of the twin in parent grain is suggested to be affected by extended dynamic recovery resulting

in either consuming the twinning texture or partly new grains. Those newly TDRXed grains have non-basal orientations, but the contribution to texture optimization is meager due to a limited amount. As the limited DRX process cannot consume enough dislocations and the extra dislocations are continuously piled up and accommodated by twins (Figure 3i), the average KAM value of the HDTR sample (1.724°) is significantly higher than that of the LDTR sample (1.528°). The HDTR process can provide sufficient stored energy for the subsequent SRX. Also, the (0001) basal texture of the HDTR sample is tilted away ND with a higher pole density (Figure 2b).

Briefly, the difference in the shear strain contributes to the change of deformation mechanisms from basal slip to multiple twinning. For the LDTR process, a high roller temperature (180°C and 240°C) contributes to dynamic recrystallization and dislocation recovery. In contrast, a larger roller temperature difference can lead to a higher shear strain due to a higher temperature gradient, which results in a more significant deformation incompatibility between the upper and lower surface. A higher shear strain not only leads to the activation of non-basal slip and twinning modes, which become the main nucleating site of static recrystallization for grains with random orientations, but also contributes to the inhibition of DRX and increasing of stored strain energy.

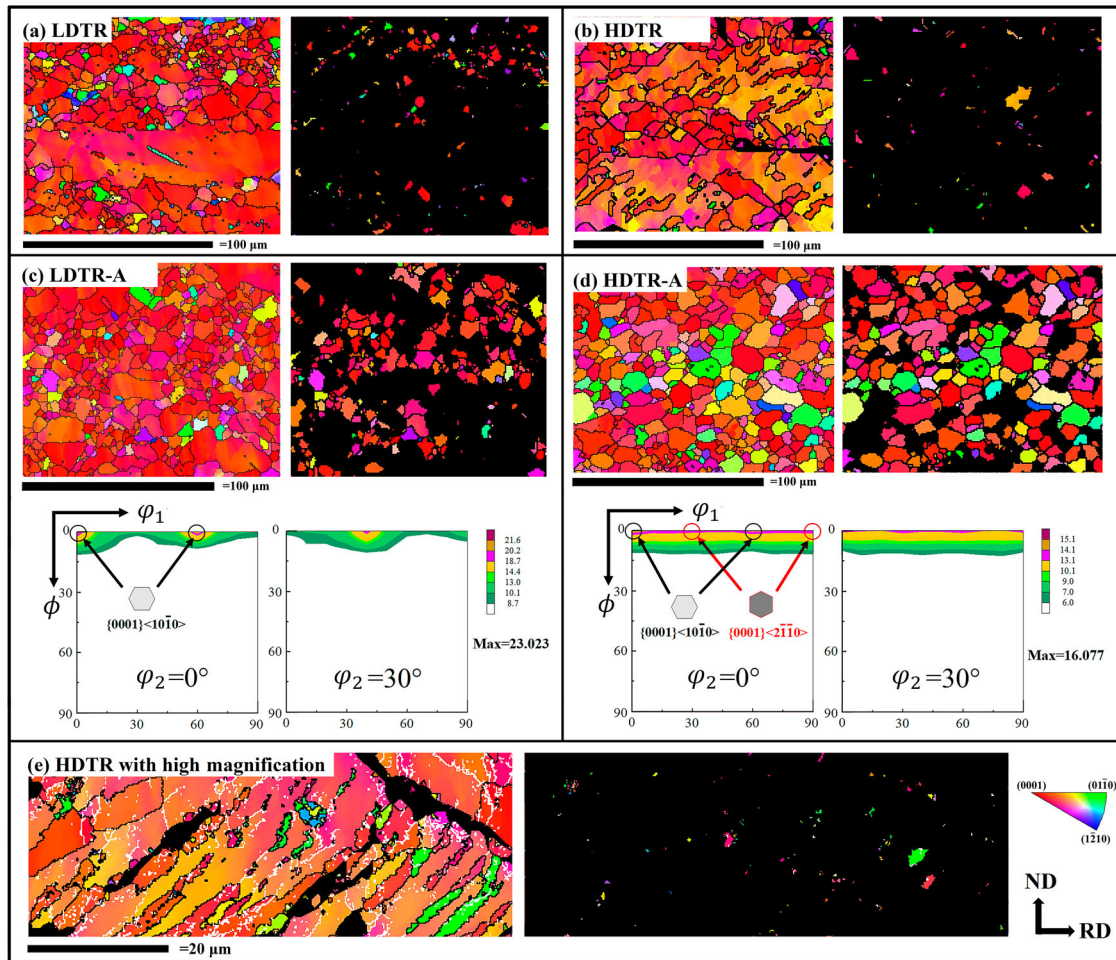


Figure 4. The IPF maps (left) and corresponding highlighted recrystallized grains in IPF maps (right) of (a) LDTR; (b) LDTR-A; (c) HDTR; (d) HDTR-A; (e) HDTR with high magnification.

4.2. The role of recrystallization on texture optimization and mechanical properties

After the rolling procedure, subsequent annealing is adopted for stress relief and dislocation recovery. Especially, static recrystallization (SRX) occurs during the annealing process, which strongly influences the final grain size, orientation distribution, and texture component. The orientations distribution of identified recrystallized grains of LDTR, LDTR-A, HDTR, and HDTR-A samples is shown in Figure 4. For the LDTR sample, only some DRXed grains exhibit random orientations, indicating DDRX can be activated under the shear stress; however, its number fraction is meager. Although the recrystallization fraction has increased a lot after annealing, the new recrystallized grains formed inside parent grains by rearranging dislocations always inherit the parent grains' orientations and exhibit few random distributions (Figure 4c). A higher density of $\{0001\} \langle 10\bar{1}0 \rangle$ basal texture is achieved and reveals that most basal planes lie parallel to the RD with their $\langle 10\bar{1}0 \rangle$ directions aligned parallel to the RD.

Moreover, the double peak $\{0001\}$ basal PF (Figure 2c) is related to the remained twins [29]. In contrast, although the DRX is hardly observed during the HDTR process, the grain orientation of new recrystallized grains formed at twin-twin intersections [24,25,27] and DTWs boundaries is not limited to the parent grains' orientations. Thus, after annealing, the HDTR-A sample achieves a more homogeneous microstructure with random orientations, and most basal planes lie parallel to the RD but spread along the RD, leading to the optimized and weakened basal texture. (Figure 4d).

Compared to the mechanical properties of as-rolled and as-annealed samples, the as-rolled sample exhibits higher YS and UTS but lower ductility. For the HDTR sample, due to the impeding of dislocation gliding for twin boundary strengthening effects, a large number of multiple twins leads to a higher YS than that of the LDTR sample. However, a high strain hardening rate cannot be sustained because the dislocation would be quickly saturated due to the limited space for moving,

thus leading to the lowest ductility. While multiple twin systems can provide practical nucleated areas for subsequent annealing. Thus, after annealing, a homogeneous fully-recrystallized microstructure can avoid the local stress concentration and premature failure is achieved in the HDTR-A sample. Most of those new recrystallized grains formed at DTWs and CTWs boundaries and twin-twin intersection with random orientations contribute to weakening basal texture and the activation of additional non-basal slip modes during deformation. Moreover, a smaller grain size can be beneficial for the activation of non-basal dislocations, which can accommodate strain along the c-axis and improve the uniform elongation [56]. However, some dislocations and twins are retained in the LDTR-A sample due to the insufficient recrystallization [57], contributing to a combination of dislocation strengthening, twin and grain boundary strengthening. Thus, the YS of the LDTR-A is higher than that of the HDTR-A sample, while the HDTR-A sample exhibits a higher ductility (compared with the LDTR-A sample and [33,39–44]) and a sustainable high work hardening rate [58].

5. Conclusion

- (1) There is a competition between DRX and the evolution of deformation substructure during the DTR process. One hand, a low shear strain (<0.15) in the LDTR process leads to the accumulation of dislocation and extension twins (TTWs), thus resulting in a high CDRX degree. In contrast, a considerable shear strain (>0.4) in the HDTR sample contributes to a multiple-twinning system including double twins (DTWs), contraction twins (CTWs), and the intersection of twin-twin boundaries while the DRX was hardly observed.
- (2) After annealing, the recrystallization fraction has increased in the LDTR-A sample, but the orientations of recrystallized grains always inherit the parent grains and exhibit few random orientations. However, a more homogeneous microstructure with a higher recrystallization fraction ($\sim 71.75\%$) was observed in the HDTR-A sample. The DTWs boundaries and twin-twin intersections can provide potential nucleation sites for recrystallization due to their high local stored energy, at where the new recrystallized grains exhibited random orientations.
- (3) Compared with the as-rolled samples, the annealed samples exhibit a similar UTS but higher total elongation values. The significant ductility enhancement from $\sim 8.1\%$ to $\sim 33.6\%$ of the HDTR-A sample is mainly correlated with the homogeneous

fully-recrystallized microstructure, weakened basal texture, and the activation of non-basal slip.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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