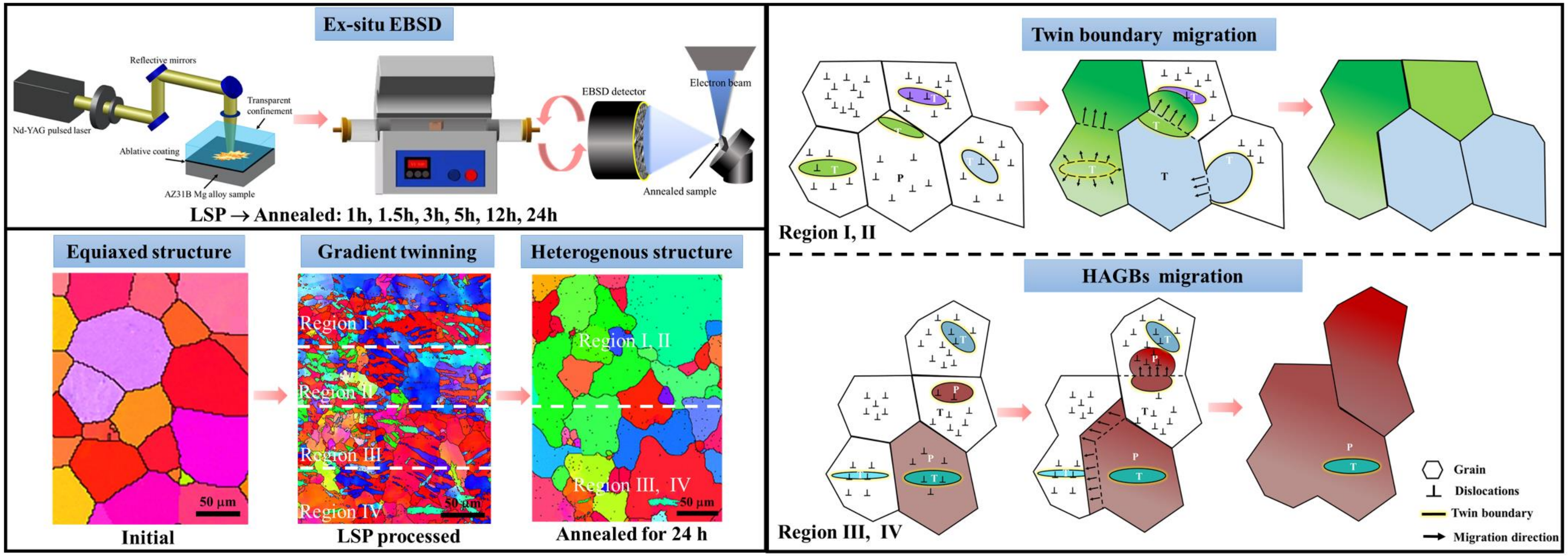
Manuscript File

Graphical abstract



Highlights

- Quasi-in-situ electron backscatter diffraction (EBSD) characterization was carried out to study the thermal stability of the gradient twinning microstructure in AZ31 Mg alloy processed by LSP.
- {10-12} tension twins have the capability to engulf non-corresponding adjacent parent grains, changing the basal texture to the twined texture.
- The isolated {10-12} tension twins within the parent grains cannot consume their grains, demonstrating excellent thermal stability.
- Combination of LSP and subsequent annealing allows for the design of texture-heterogeneous Mg alloys by changing the microstructure from gradient twinning to heterogeneous structure.

Quasi-in-situ EBSD study of the thermal stability of gradient twinning microstructure of an AZ31B magnesium alloy processed by laser shock peening

Qian Liu [a] ǂ, Wanting Sun [b] ǂ, Shuangjie Chu [a], Yuqian Wang [c], Yafei Wang [a], Xing Zhang [d] Bohao Zhou [a], Hao Wang [e], and Bo Mao [a*]

a) School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai, 200240, China

b) School of Engineering, Lancaster University, Lancaster, LA1 4YW, UK

c) Department of Mechanical Engineering, University of Nevada-Reno, Reno, 89557, USA

d) School of Mechanical and Electrical Engineering, Soochow University, Suzhou, Jiangsu, 215100, China

e) Shanghai Engineering Research Center of Laser Intelligent Manufacturing, Shanghai Institute of Laser Technology Co.,Ltd., Shanghai 200233, China

* Author to whom correspondence should be addressed. Electronic mail: Bo Mao (bmao@sjtu.edu.cn)

ǂ These authors contributed equally to this work

Abstract

In this study, the thermal stability of gradient microstructure in the commercial AZ31B magnesium (Mg) alloy processed by laser shock peening (LSP) is systematically explored using quasi-in-situ electron backscatter diffraction (EBSD) measurement. The mechanisms of grain growth and twinning evolution of LSP-processed AZ31B Mg alloy during the annealing treatment at 300°C are revealed. The experimental results demonstrate that there is a significant transformation of a gradient twinning microstructure into an almost twin-free microstructure, and the trend of grain growth is associated with LSP-induced strain energy storage. From the topmost surface to the sublayer of LSP-processed sample, the grain growth is mainly driven by the migrations of twin boundaries (TBs) and high-angle grain boundaries (HAGBs), respectively, which is attributed to the reduced accumulated strain energy along the LSP direction. It was demonstrated that the $\{10\bar{1}2\}$ tension twins possess the capability to engulf non-corresponding parent phases, which transcends the conventional understanding that twins interact solely with their corresponding parent grains. The twins not only interact with the parent grain but also have the capacity to engulf twins of adjacent parent grains. In contrast, the isolated twins within the parent grains struggle to grow during annealing, indicating that $\{10\bar{1}2\}$ tension twins have excellent thermal stability. The findings of this work can contribute to an in-depth understanding of the thermal stability and the grain growth mechanisms of LSP-induced gradient twinning microstructure in Mg alloys and provide the potential for the microstructure optimization to improve the comprehensive mechanical properties.

Keywords: Magnesium alloy; Laser shock peening; Gradient microstructure; Deformation twinning; Thermal stability.

1. Introduction

Due to their high specific strength, excellent machinability, and superior bio-compatibility, magnesium (Mg) and its alloys have shown great potency in the applications of automotive, aerospace, and biomedical industries [1, 2]. However, the high-temperature performance of Mg alloys is limited by challenges such as insufficient creep resistance, susceptibility to corrosion, high costs associated with rare earth elements, issues with thermal stability, and demanding processing techniques, which have spurred ongoing research in appropriate surface treatments and design optimizations to enhance their mechanical properties.

A variety of processing-oriented novel strategies have been developed and explored in recent decades for enhancing the mechanical performance of Mg alloys, including equal-channel angular processing [3], high speed rolling [4], ultrasonic treatment [5] and shot peening [6]. It should be noted that laser shock peening (LSP) is an advanced surface treatment technique, which can bring about the improved performance and longevity of metal components [7]. It extends service life significantly by introducing deep compressive residual stresses, so that the occurrence of cracking is inhibited, and the damage tolerance is enhanced. Besides, as a kind of cold working process, the heat-related issues can be avoided by LSP as compared to traditional thermomechanical processing, making it an ideal choice for industries where component reliability and performance are paramount [8, 9]. During LSP, a short-pulse high-power laser interacts with the material surface, causing the absorption layer to vaporize rapidly and form a plasma, which can expand violently and create a high intensity laser-induced shock wave [7]. The dynamic plastic deformation of material occurs as the pressure of plasma surpasses the elastic limit. The plastic deformation induced by LSP not only results in work hardening but also refines the grain structure through various mechanisms such as dislocation movement, twinning and phase transformation [10]. Eventually, the gradient microstructure with residual compressive stress is generated on the surface layer of material, and better surface integrity and superior performance can be achieved without altering the property of bulk material. Our recent studies have demonstrated that LSP is capable of improving engineering performance of Mg alloys by enhancing the surface hardness [11], wear resistance [12], corrosion resistance [13], and even stretch-formability [14]. These findings lead to extensive works focused on establishing the relationship between LSP processing parameters, the resulting microstructure evolution, and the consequent property improvements in Mg alloys.

As metallic materials with a hexagonal close-packed (HCP) crystal structure, Mg and its alloys exhibit distinct and complex deformation mechanisms compared with steels or aluminum alloys with a cubic crystal structure, as processed by LSP. Mg alloys have limited independent slip systems at room temperature and the most commonly observed $\{0001\}\langle 11\bar{2}0\rangle$ basal and $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ prismatic slip systems are only capable to accommodate the plastic strain along $\langle a\rangle$ axis [15, 16]. Therefore, deformation twinning, particularly the $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ tension twin (TTW), plays a significant role during LSP of Mg due to its low critical resolved shear stress (CRSS) and invasive growth capability [17, 18]. Our previous research results showed that a gradient twinning microstructure and complex twin-twin interactions were introduced in an AZ31B Mg alloy processed by LSP [19]. Abnormal twin-twin interactions were mediated by atomic shuffling rather than twinning dislocations, and different $\{10\bar{1}2\}$ twin variants were activated within individual parent grains, leading to the formation of isolated or disconnected twin islands with the same crystallographic orientation [19]. As the twin volume fraction increases from 0 (unprocessed samples) to 38%, the micro-hardness of the Mg alloy increased from 60 to 72 VHN, leading to a significant reduction of the wear rates by 58% [12]. Moreover, by carefully tailoring the distribution and amount of TTWs, the stretch-formability of Mg alloys could be enhanced by 63% by twinning induced crystallographic texture change [14].

Thermal stability of the processed metallic materials after exposure at high temperatures is an important concern for the practical application of LSP and thus draws extensive attention. For example, Luo et al. [20] found that the average grain size of the surface nano-crystallized layer of a K417 superalloy increased from 140 nm for the LSP sample, to about 220 nm after annealing at 900^{o}C for 10 hours. Yang et al. [21] showed that significant grain coarsening effect was identified in a TC17 Ti alloy processed by LSP followed by annealing at 400^{o}C for 1 hour, while no obvious change was found when annealing at 300^{o}C for the same time. However, little efforts have been paid to investigate the thermal stability of Mg alloys processed by LSP. Research carried out by Ren et al. [22] showed that high density dislocations in a AZ91D Mg alloy induced by LSP prevent the grain growth and further grain refinement was identified due to recrystallization after annealing below 300^{o}C. Nevertheless, all the experimental results and analysis mentioned above focused on dislocation dynamics and the resulting recrystallization and grain growth behavior. The evolution of TTWs and related microstructure changes in Mg alloys processed by LSP during post-annealing has never been addressed. In general, the evolution of deformation twins in Mg alloy during annealing is a complex process involving the interaction of multiple factors, including twin types, annealing conditions, and the changes in microstructure and texture [23]. By precisely controlling these factors, the performance of magnesium alloys can be optimized to meet specific application requirements [24, 25]. However, the thermal stability of twins produced under ultra-high strain rates of LSP deformation has not been extensively explored during post annealing. It is still required to provide insights into how twins behave under such extreme conditions, which is crucial for understanding the performance of alloys in rapid deformation scenarios.

In present work, a comprehensive investigation into the thermal stability of gradient twinning microstructure in LSP-processed AZ31 Mg alloy is conducted. The in-situ EBSD observations of twin boundary and grain boundary migration are performed to enhance the in-depth understanding of microstructural evolution and the mechanisms governing twinning and grain growth in HCP metals. It is expected that the exploration of gradient twin structures in the LSP-processed AZ31 Mg alloy reveals not only their thermal stability characteristics but also the potential for precise control of microstructural evolution, paving the way for innovative material design with enhanced performance in a variety of engineering applications.

2. Experimental procedures

2.1. Sample preparation

Rolled AZ31B Mg alloy plate with a chemical composition of Mg–96 wt%, Al–3.0 wt%, Zn–1.0 wt% was purchased from Metalmart.com for experiments. Sample blocks were cut from the plate with dimensions of 15 mm × 15 mm × 5 mm. Prior to LSP, the surface of the samples was grinded by SiC sand papers with different grades of roughness (from #180 to #1200), followed polishing with 1 μm diamond suspension. Ultrasonic cleaner filled with ethanol was utilized to clean the polished samples.

2.2. Laser shock processing and post-annealing

Fig. 1(a) shows a schematic diagram of the LSP configuration. A Q-switched Nd-YAG laser with a wavelength of 1064 nm and a pulse duration of 5 ns was used to deliver laser energy. A black tape with a thickness of 130 μm was used as ablative coating to absorb the laser. A transparent BK7 glass was laid on the top of the black tape and utilized as confinement. As the pulsed laser transmits through the BK7 glass, it interacts with the ablative coating which subsequently evaporates into plasma at high temperature. The expansion of the plasma is confined by the BK7 glass while the shockwave is generated and propagates into the Mg alloy target. In this study, LSP was performed along the transverse direction (TD) of the as-received rolling specimen. Laser intensity of 5 GW/cm^2 and a laser beam diameter of 2 mm were adjusted and utilized.

After LSP, the sample was carefully cut along the TD of the sample by passing the center of the indentation induced by LSP using a low-speed diamond cutter, as shown in Fig. 1(b-c). Detailed cross-sectional metallographic preparation method can be referred to our previous work [11]. Static annealing experiments were performed by a tubular furnace filled with argon gas at

300°C with a soaking time of 1,1.5, 3, 5, 12, and 24 hours (Fig. 1d). The sample was removed from the furnace and cooled by air for further microstructure characterization.

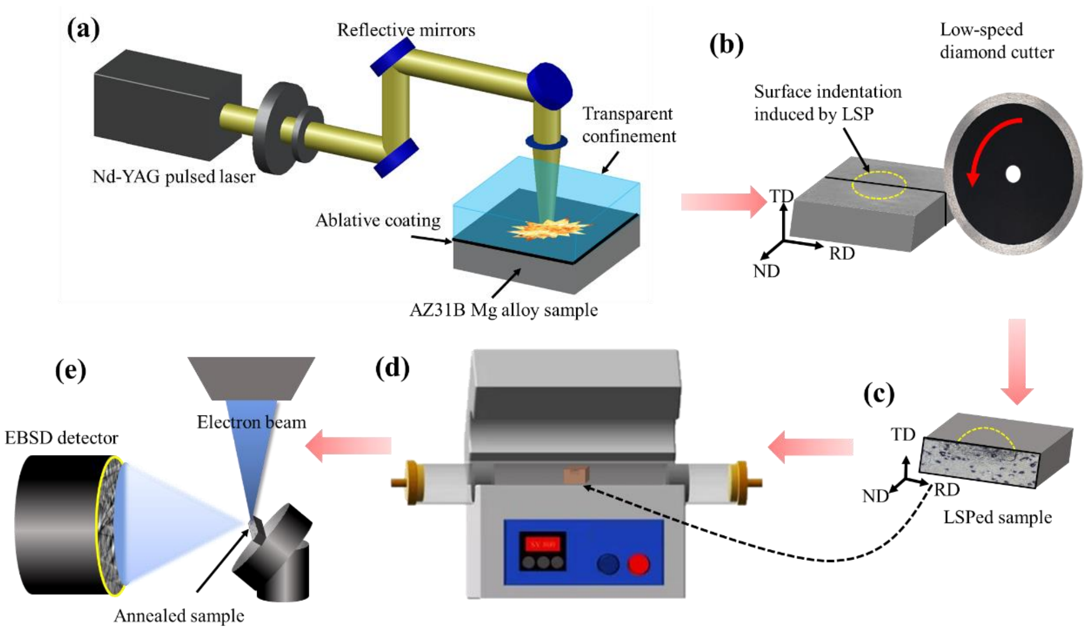


Fig. 1. Schematic illustration of the experimental process: (a) LSP set up, (b) sectioning process performed by a low-speed diamond cutter to reveal the cross-section microstructure, (c) LSP-treated AZ31B Mg alloy sample for annealing treatment, (d) annealing process was performed in a vacuum furnace filled with argon gas, and (e) EBSD observations of the LSP-treated samples after annealing for different periods of time.

2.3. Microstructure characterization

Microstructure of the samples before and after LSP were characterized using a Leica DM2700 optical microscope (OM) and a SM-7100FT field emission scanning electron microscope (FESEM) equipped with a Nordlys Max2 electron backscattered diffraction (EBSD) detector. Sample just after LSP for OM characterization was prepared by sectioning, mounting, polishing, and etching with the acetic picral solution (10 ml acetic acid +4.2 g picric acid +10ml distilled water +70ml ethanol). EBSD characterization was carried out on the unprocessed sample and the LSP processed one before and after annealing. Sample for EBSD characterization was grinded and vibrationally polished by a 0.5 μm diamond suspension followed by etching with 4% nitric acid alcohol solution. It should be noted that EBSD was performed on the same regions just beneath the LSP processed surface of the sample before and after annealing for different times. Since the surface of the sample was prevented from oxidation in argon environment during annealing, no further polishing treatment was required to conduct EBSD on the specimen with different annealing times. Different from the in-situ characterization process, this quasi in-situ EBSD characterization process was also adopted by other researchers to investigate the microstructure evolution of Mg alloys subjected to thermal annealing [26]. The microstructure evolution of Mg alloy samples during thermal annealing gained from EBSD data was analyzed by Channel 5 software.

3. Results and discussions

3.1. Gradient twinning microstructure

Fig. 2 shows the EBSD analysis of the as-received AZ31B Mg alloy. From the 3-D inverse pole figure (IPF) map in Fig. 2(a), it can be seen that the microstructure of as-received Mg alloy is composed of equiaxed grains with an average grain size of around 30 μm, but no deformation twinning can be detected. The pole figure in Fig. 2(b) shows that the as-received AZ31 alloy has a strong basal plane texture, which is typical for rolled Mg alloy [27]. The maximum texture intensity value of ~56 indicates a considerable degree of preferred orientation along a particular direction, and most of the basal planes are parallel to the RD-TD planes.

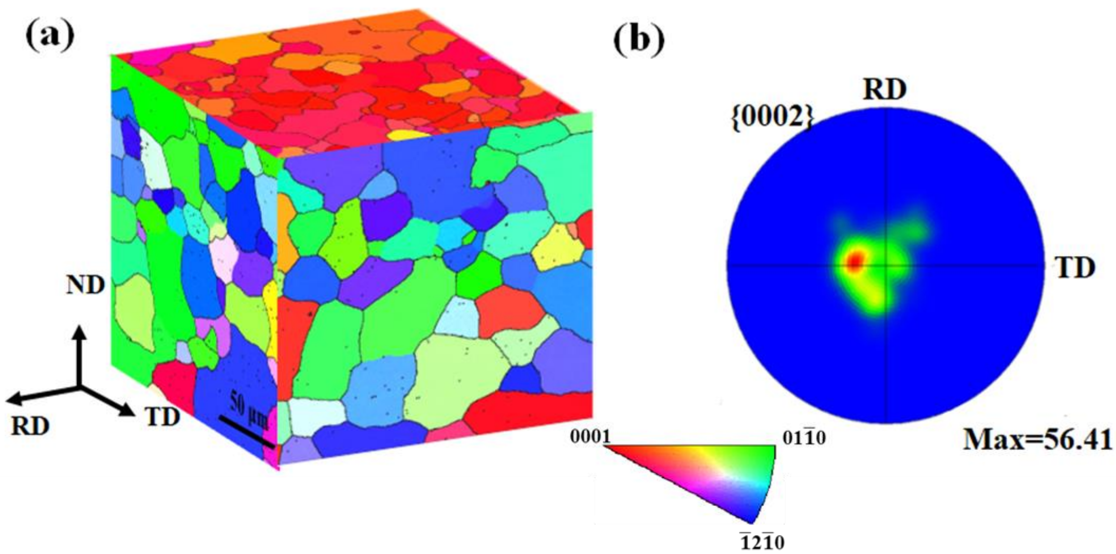


Fig. 2. Microstructure of the as-received AZ31B Mg alloy sample: (a) 3-D reconstructed IPF maps and (b) {0002} pole figure.

Fig. 3 shows the microstructure characteristics of the RD-TD cross-section of Mg alloy processed by single pulse LSP with a laser intensity of 5 GW/cm^2. As depicted in the cross-sectional OM image in Fig. 3(a), the LSP-processed AZ31B Mg alloy exhibits a gradient twinning microstructure. This gradient feature suggests a variation in twin density from the surface to the interior of the material, which is attributed to the strain gradient induced by the LSP process. The depth of the LSP affected zone could reach around 600 μm. As shown in Fig. 3(b) and (c), the IPF map and an image quality map provide detailed insights into the crystallographic orientation of grains and twins within the designated area as marked by dash rectangle in the OM image. It is observed that after LSP deformation, the volume fraction of twins is decreased with the increase of depth, and the nearly fully twined microstructure can be achieved on the top surface. To characterize the nature of interfaces, different types of grain boundaries are presented with different colors in the image quality maps (Fig. 3c): the blue lines represent $\{10\bar{1}2\}$ TTW boundaries; the yellow lines represent $\{10\bar{1}1)$ compression twin (CTW) boundaries; the red lines represent boundaries resulted from the TTW-TTW interactions; the green lines represent $\{10\bar{1}1\}$-$\{10\bar{1}2\})$ double twin (DTW) boundaries. Low angle grain boundaries (LAGB) were also marked by blackish green color. The observation of the high density of blue boundaries reveals that $\{10\bar{1}2\}$ TTW dominates the plastic deformation of the Mg alloy processed by LSP. As shown in Fig. 3(d), the intensity of {0002} basal texture is reduced and $\{10\bar{1}2\}$ twin variants are activated (the discrete spots around the RD on the {0002} pole figure. The weakening of the basal plane texture in rolled AZ31 magnesium alloy following LSP can be attributed to the specific microstructure evolution. High strain rates induced by LSP can disrupt the preferred grain orientations, potentially randomizing the crystallographic structure. Additionally, the generation of dense dislocation network and the activation of non-basal slip and twinning systems can alter grain orientations, leading to a more random texture [28].

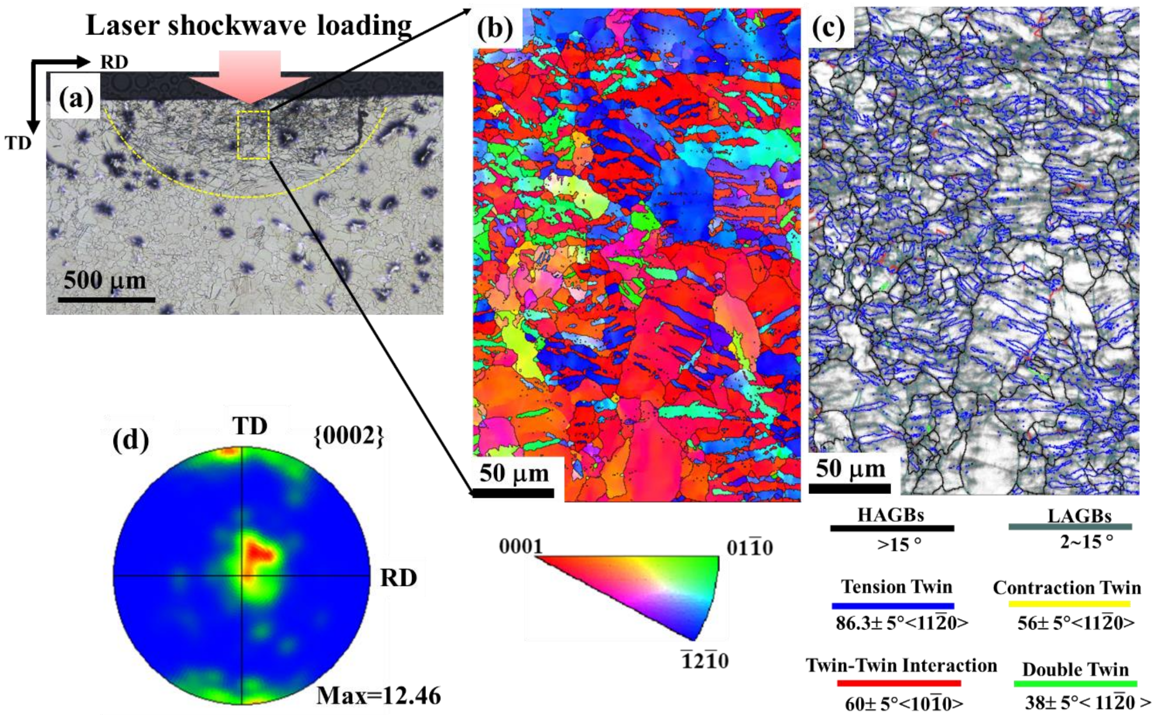


Fig. 3. Microstructure analysis of the AZ31B Mg alloy sample processed by LSP with a laser intensity of 5 GW/cm^2: (a) Cross sectional OM image showing the gradient twinning microstructure, (b) IPF map and (c) image quality map of the area marked by the dash rectangle in (a). (d) {0002} pole figure of the area.

3.2. Microstructure evolution during static annealing

The quasi-in-situ EBSD analysis as shown in Fig. 4 illustrates the progressive microstructure evolution and texture changes of the LPS-processed AZ31 Mg alloy sample upon static annealing at 300°C. Initially, the LSP-processed AZ31 Mg alloy exhibits a microstructure characterized by a high dislocation density, prevalent deformation twins, and refined grains, which is derived from the severe plastic deformation induced by LSP. The microstructure of LSP-processed AZ31 Mg alloy can be divided into four regions along the depth (Fig. 4a): Region I totally twinning; Region II, twin interaction; Region III a small amount of needle-like twins with a low density; Region IV coarse parent grains without twins. Besides, when the LSP-processed AZ31 Mg alloy is subjected to the annealing treatment, the initially refined grains and deformation twins begin to coarsen, which is driven by the reduction in stored energy and facilitated by thermal activation. As shown in Fig. 4(a)-(h), the IPF map and image quality map can highlight the different regions of varying dislocation density that influence the rate and pattern of grain growth [29].

During the annealing, the LSP-processed AZ31 Mg alloy undergoes recovery, where dislocations rearrange and annihilate, forming subgrains that serve as nuclei for grain growth. Recrystallization occurs with new strain-free grains nucleating and growing, particularly in regions of higher stored energy. When the annealing duration is extended to 12 h, the $\{10\bar{1}2\}$ tensile twins almost completely disappear, and thus the microstructure in the Region I and Region II near the surface are fully consisted of coarse grains twin structure, as evidenced in Fig. 4(d). While the grain size in the Region III and Region IV far away the surface is also increased, indicating that the deformed grains exhibit more stable structure and lower internal strain energy than the grains within the surface layer. The reduction of grain boundary energy is favorable for the coalescence of smaller grains into larger stable grains induced by the stress relief. As shown in Fig. 4(i)-(l), the {0002} pole figures exhibit a dramatic crystallographic texture change, reflecting the response of LSP-induced gradient twin structure to the annealing process. As the annealing time is increased, due to the activated recovery mechanism, a reduction in dislocation density and the reorientation of grains cause a weakening of the initial strong basal texture typically associated with deformation processing. As a result, a heterogenous texture structure consisting of non-basal texture near the surface and basal texture in the center area of the sample was formed by LSP and post-annealing. Accordingly, the grain size evolution in the LSP-processed AZ31B Mg alloy following annealing is a complex interplay of thermally activated processes, including recovery, recrystallization, grain boundary migration influenced by the local dislocation, twin densities, as well as the stored energy.

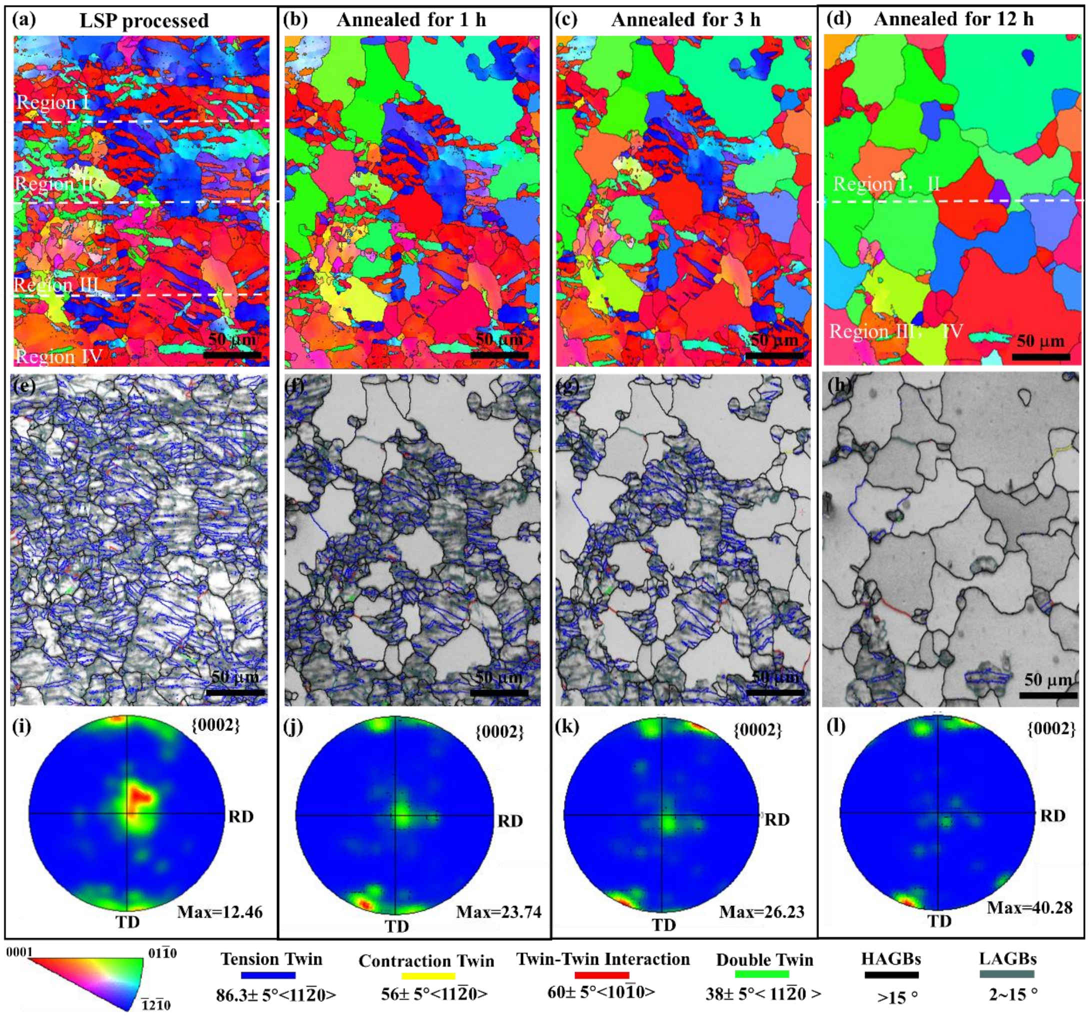


Fig. 4. Quasi-in-situ EBSD IPF map and image quality map of the LPS deformed Mg alloy sample after static annealing at 300 ℃ for different times: (a) LSP processed; (b)1h; (c) 3h; (d) 12h.

3.3. Thermal stability of parent and twin variants

3.3.1. The growth of the twin variants

To gain detailed insights into the microstructural evolution of LSP-treated Mg alloys during annealing, Fig. 5 shows the growth of twin variants in the topmost surface layer (region I). In the LSP-processed AZ31 Mg alloy, the microstructural evolution on the topmost surface during annealing is characterized by a complex interplay of twinning and grain boundary migration.

Initially, two deformed grains G1 and G2 can be observed, and G1 is fully twinned by $\{10\bar{1}2\}$ tension twin (T1) and retaining only a few parent grains (P1). In contrast, G2 is partially twinned, which contains rod-shaped $(10\bar{1}2)$ tension twin (T2) and parent grain (P2). As the annealing progresses, the deformed grains are gradually consumed by the persistent growth of T2, which is associated with a reduction in stored energy within the enlarged grains. The direction of boundary movement is described in the IQ map and kernel average misorientation (KAM) map, with yellow arrows indicating the migration path. During the annealing for 1h, T2 initiates its consumption with parent grain P2 and rapidly occupies half of G2. The deformed grains within the LSP-treated sample are also consumed by the advancing twin boundaries of T2. The driving force then redirects the twin boundary of T2 towards grain G4, which possesses higher internal energy, bypassing grains G1 and G3. As the annealing duration is increased, the high-energy segments of G1 are incrementally absorbed by T2, while the low-energy portions of G1 (T1) persist after annealing for 12h. This endurance of the low-energy T1 is attributed to the minimal difference in stored strain energy between T1 and T2, leading to a sluggish rate of twin boundary migration.

Throughout the annealing progress, the orientation of T2 remains consistent, expanding in size as it absorbs surrounding grains. The different energy stored along the twin boundaries serves as the driven force for twin boundary migration (TBM), propelling T2 from regions of lower stored strain energy towards those of higher stored strain energy, following the sequence of P2→G4 → G1. It indicates that the driving forces behind the twin evolution during annealing are likely the reduction of stored strain energy. Due to thermal activation, the migration of grain boundaries towards regions of higher internal stored energy during the annealing treatment. The continuous growth of T2 and the consumption of parent and neighboring grains suggest that the twins are dynamic and responsive to the distribution of stored strain energy within the material, which is influenced by LSP deformation.

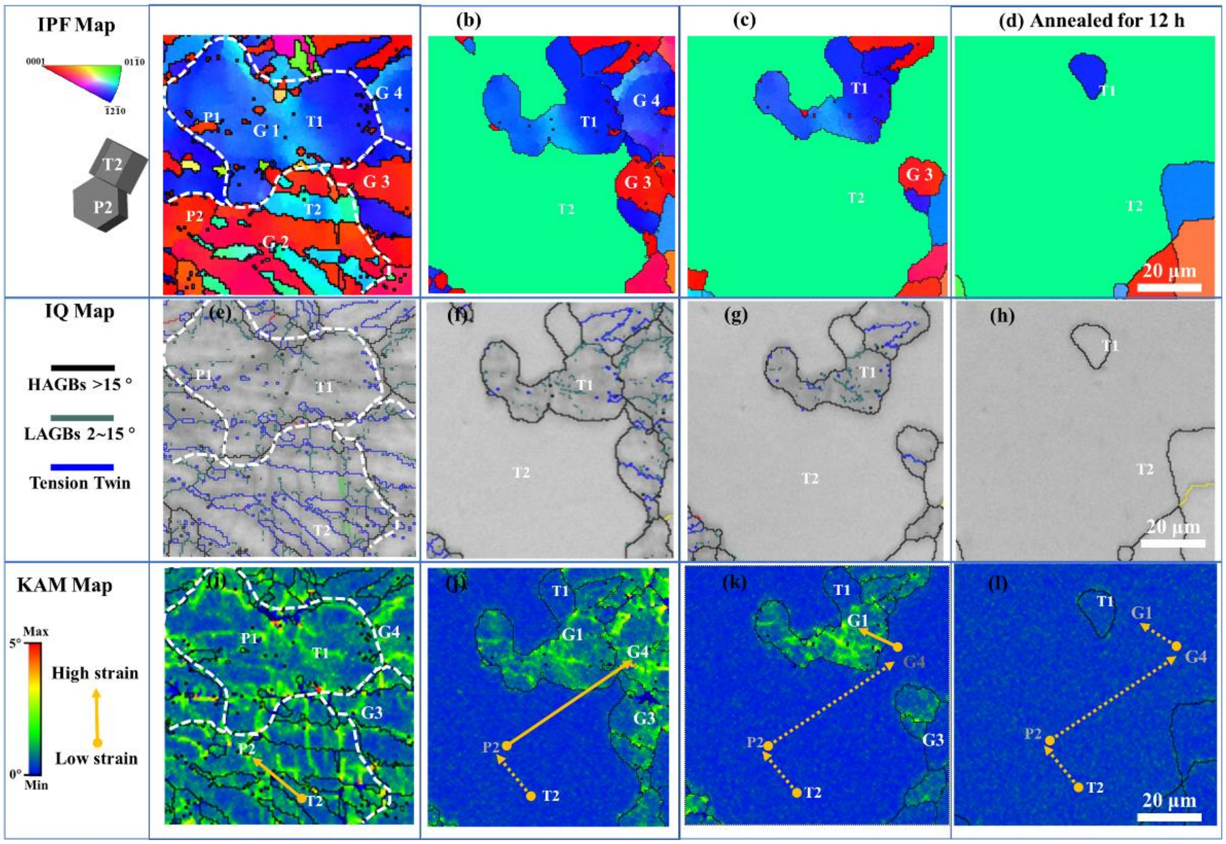


Fig. 5. Twins in topmost surface consumes their parent grains and neighbor grain after static annealing at 300 ℃ for different times: (a) LSP processed; (b)1h; (c)5 h; (d) 12 h.

Fig. 6 shows the thermal stability of the LSP-induced twins in the subsurface layer during annealing, revealing a series of intricate interactions between twins and their neighboring grains. Initially, the LSP-processed subsurface layer (referred to as Region II) exhibits a majority of partially twinned grains with distinct twins labeled T1-T4 and parent grains P1-P4. As shown in Fig. 6(a)-(d), the continuous growth of twin T3 can be observed throughout the annealing process, and the neighboring grains are consumed in a selective manner. Notably, the parent grain P3 of T3 is consumed by the growth of T4 instead of T3, indicating that the growth of twins in the subsurface region of the LSP-treated sample are unable to consume their parent grains. It reflects a resistance to self-consumption of parent grains by their respective twins in the LSP-treated subsurface layer. As shown in Fig. 6(g), after the annealing treatment for 5 h, the grain G1 still retains some low-angle grain boundaries (LAGBs) and strain energy that cannot be fully absorbed and replaced by T3. This phenomenon can be explained by the orientation pinning effect, and thus the migration of grain boundaries is hindered when encountering grains of similar orientation [30, 31]. As the annealing duration is extended to 12 h (Fig. 5h), the residual deformed grain and the twins are consumed by the growth of T3. The growth of twins presents a preference for consuming the grains with higher internal strain energy, such as G4, followed by G2 and then G1, as indicated by yellow arrows in Fig.6(i-1). This energy-directed growth pattern suggests a thermodynamic drive towards minimizing the overall energy. Therefore, the evolution of twins and grains during thermal annealing is influenced by a combination of selective growth, orientation pinning, and energy-directed consumption. The preservation of parent grains and the intricate interplay between energy minimization and grain boundary migration can make the contributions to the unique microstructure evolution observed in the subsurface layer of LPS-processed AZ31 Mg alloy.

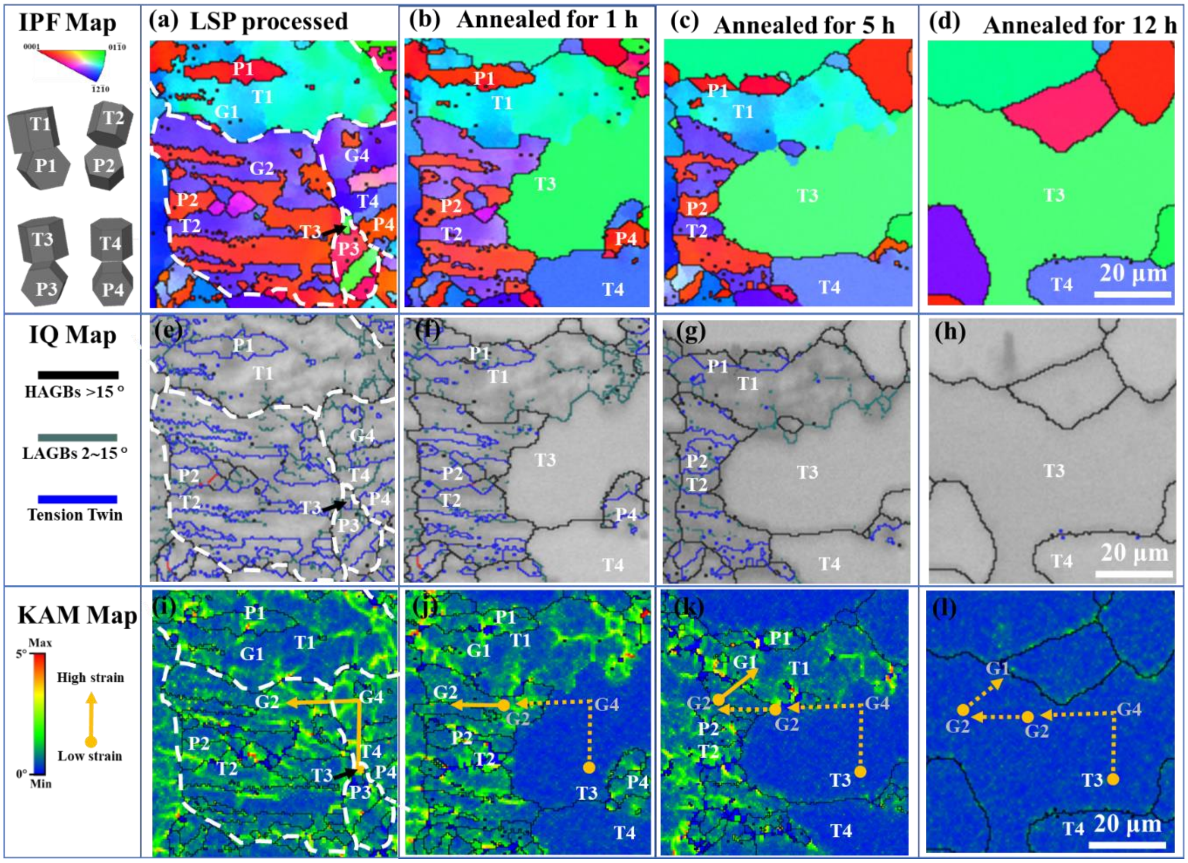


Fig. 6. The evolution of twins in subsurface layer through the consumption of their neighbor grain during the static annealing at 300 ℃ for different times: (a) LSP processed; (b)1h; (c)5 h; (d) 12 h.

3.3.2. The growth of the parent grains

As the distance from the surface increases, the deformed grains G1 and G2 are partially twinned within Region III, which is composed of rod-shaped $\{10\bar{1}2\}$ tension twins (T1/T2) and parent grains (P2/P2). As the annealing progresses, there is a noticeable increase in the size of parent grain P1, which extends into the deformed grain region as indicated by the red color in the IPF maps of Fig. 7(a)- 7(d). As reported, the orientation pinning effect can prevent P1 from completely consuming G6 [30, 31], leading to a transition of HAGBs to LAGBs, thereby diminishing the internal strain energy within G6. As shown in Fig. 7(i)-(1), the yellow arrows in the KAM map illustrate the movement direction of P1 grain boundary during the annealing process. After annealing for 1h (Fig.7(j)), the growth of P1 nearly obliterates the adjacent grain G2, leaving only minor remnants of grain T2 and its twin T1. With further annealing, the grown grains P1 are tending to consume G5 and surrounding G6 and G7 (Fig. 7k, l). The HAGBs migration of P1 causes a reduction in the accumulated strain energy within the deformed grains. The dissipation of stored strain energy is a result of a decrease in the dislocation density through a recovery process involving dislocation climb and cross-slip during annealing [32]. These results proved that parent grains in subsurface tend to first consume the neighbor grains and then its twins. It can be deduced that during the annealing process, the factors that drive the expansion of parent grains in the subsurface layer include the reduction of internal strain energy which stimulates grain growth, the limiting effects of orientation pinning on their growth, the microstructural relaxation marked by the transformation of grain boundaries, and the selective absorption of neighboring grains over twins, demonstrating a preference for lower energy configurations in terms of kinetics.

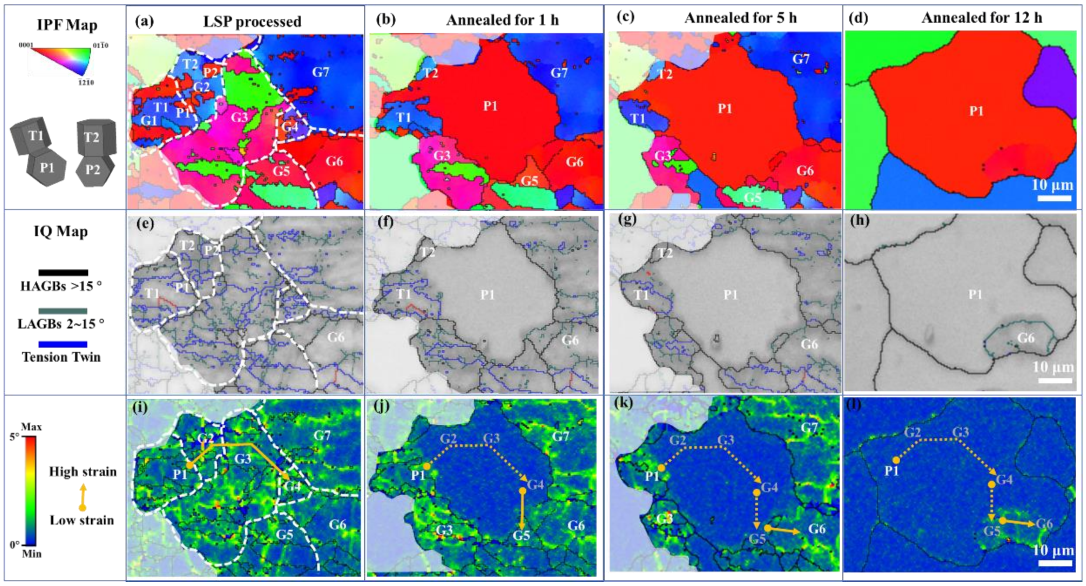


Fig.7. The evolution of parent grains in the subsurface layer the consumption of their twins and neighbor grains during the static annealing at 300 ℃ for different times: (a) LSP processed; (b)1 h; (c)5 h; (d) 12 h.

3.3.3. The survival of twin variants

Fig. 8 shows the evolution of the parent grains in Region IV far from the LSP-treated topmost surface during annealing. In Region IV, deformed grain G1 can be identified, which contains a parent grain (P1) and a twin band (T1). Upon annealing at 300°C for varying durations, the parent grain P1 can rapidly consume its neighboring grains G2-G4, which includes the segments of twin bands. However, even after annealing for 24 h, the twin band T1 are still maintained rather than completely dissolved. The survival of T1 is attributed to the tip of twin connection with HAGBs, where the junction experiences significant local stress and strain conditions, leading to a reduced migration rate of the twin boundary [33]. Furthermore, the twins far from the surface show minimal change, and it is probably due to the lower internal strain energy in all the deformed grains of Region IV as compared to those near the surface or subsurface. The limited energy difference and weaker migration of twin boundaries or HAGBs restrict the consumption of twins. These findings indicate that even after 24 h of annealing at 300°C, the parent grain P1 far from the LSP-treated surface cannot consume its own twin T1. This suggests that the evolution of twin variants during annealing is closely related to the local stress and strain conditions, which are influenced by the grain boundary characteristics and the distribution of strain energy within the grains.

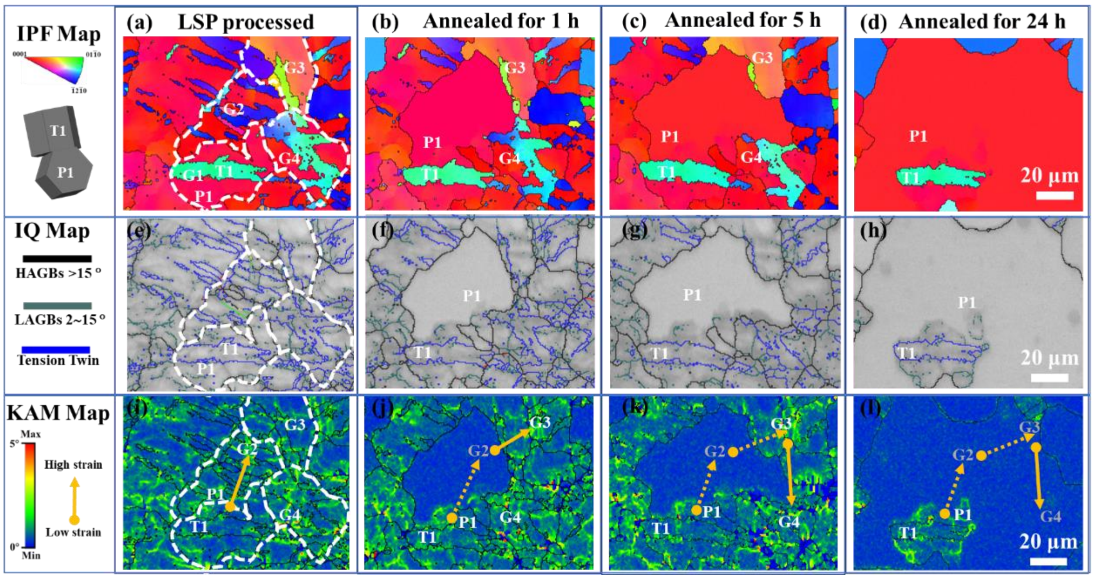


Fig. 8. The microstructure evolution of Region IV during the static annealing at 300 ℃ for different times: (a) LSP processed; (b)1h; (c)5 h; (d) 24 h.

3.4. Proposed mechanism

As mentioned above, the annealing process of LSP-deformed AZ31 Mg alloy involves a complex evolution of twins and grain growth, and Fig. 9 illustrates the schematic diagram of the gradient microstructure evolution during annealing. Based on the quasi in-situ EBSD observations, the microstructure undergoes four types of distinct changes across different regions. (i) In Region I of the topmost surface, the twin variants grow despite the low thermal activation migration rate of the $\{10\bar{1}2\}$ twin boundary, which is not a preferred site for grain growth [34, 35]. Twins become thicker as they are significantly larger than their parent grains, and their boundaries continuously sweep through the parent grains and adjacent grains, dissipating strain energy through dislocation climb and cross-slip recovery processes [36]. (ii) In Region II of the subsurface layer, the deformation twins grow under the control of strain-induced twin boundary migration, consuming neighboring twins but not the parent grains. The effective driven force is decreased with depth, resulting in lower internal strain energy in the deformed grains below the subsurface compared to the topmost surface. (iii) In Region (III) below the subsurface layer, the parent grains grow by migrating and consuming surrounding grains. The migration of HAGBs is driven by the difference in strain energy, leading to a reduction in the high dislocation density in neighboring areas. After prolonged annealing, the twins can engulf the surrounding parent grains. (iv) In Region IV far from the topmost surface, the parent grains undergo HAGB migration to reduce the total strain energy of adjacent grains, leading to the formation of twin-free grains. However, the twins cannot consumed by the parent grains due to limited internal strain energy, and the strong local stress at the $\{10\bar{1}2\}$ twin tip against the grain boundary may prevent twin boundary migration [33], suggesting that the $\{10\bar{1}2\}$ twin has an excellent thermal stability. Similar results was also revealed in the research carried out by Sabat et al. [26], in which they found that growth mechanism of extension twin during annealing was different from the normal grain growth mechanism.

Microstructural evolution is a time-dependent process. Notably, when the annealing time is short ($\leqslant$1 h), the migration of HAGBs and twin boundaries is faster due to the reduction in dislocation density, and the storage strain energy in area of HAGBs and twin boundaries is reduced [37]. As the annealing time extends, the twins can engulf adjacent parent grains that are not their corresponding parent. Besides, when a twin is isolated within its own parent, it has difficulty growing. In fact, there is no new grain nucleation or growth during the annealing process, and the tensile twins exhibit excellent thermal stability with never engulfing their own parent grains.

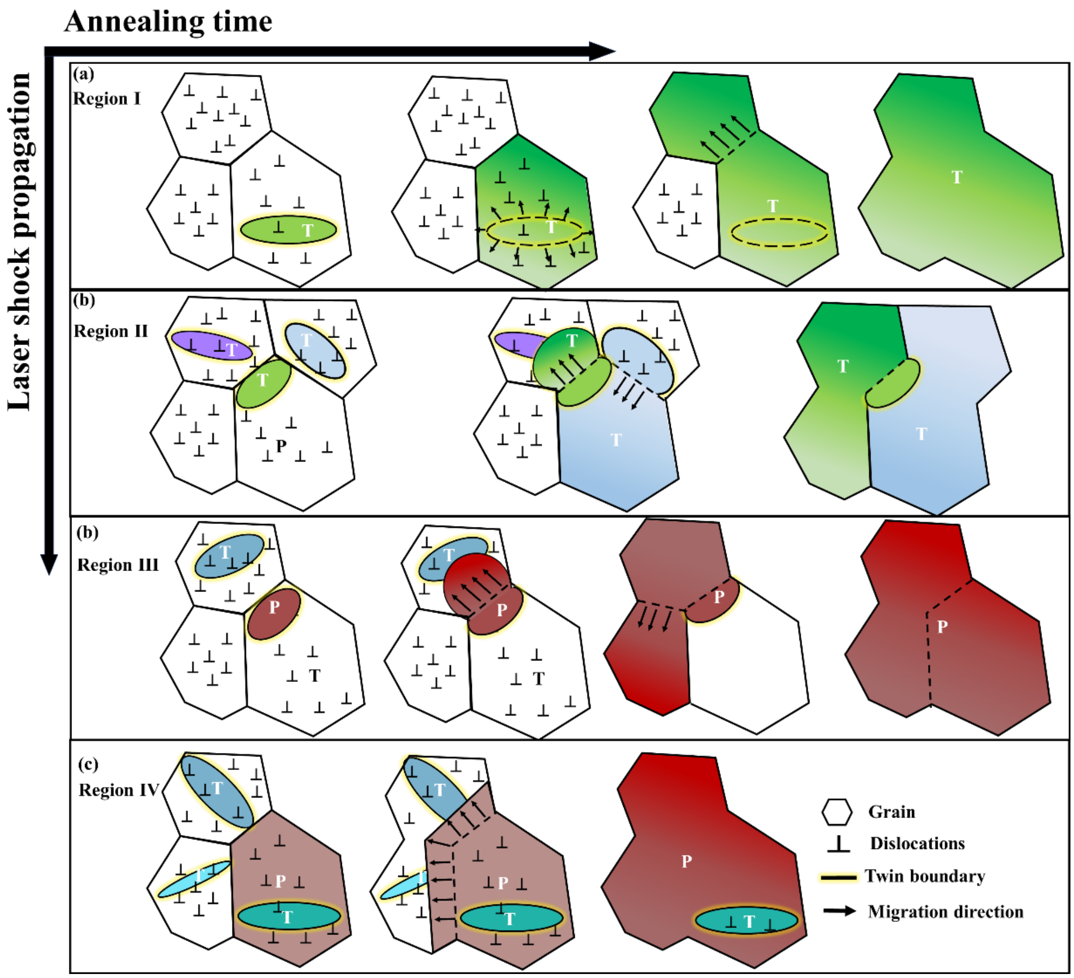


Fig.9. Schematic diagram of growth of the twin variants (a) in the region I, (b) in the region II. Growth of the parent grains (c) in the region III (d) in the region IV.

4. Conclusions

In this paper, the microstructure evolution of gradient twin structure in the LSP-deformed AZ31 Mg alloy during the post-annealing treatment was systematically investigated by quasi in-situ EBSD observations. The main conclusions can be drawn as follows:

(1) During the annealing processing of LSP-deformed AZ31 Mg alloy, the gradient $\{10\bar{1}2\}$ twins and residual matrix grains along the depth are gradually transformed into untwined grains. This process involves the migration of high angle grain boundaries (HAGBs) and twin boundary

migration (TBM), which is driven by the strain energy difference between adjacent grains. After the annealing treatment, the intensity of basal texture of LSP-processed AZ31 Mg alloy is reduced, indicating that the combination of LPS and subsequent annealing allows for the creation of texture-heterogeneous materials.

(2) The grain growth mechanisms are closely related to the gradient twin structure induced by LSP. On the topmost surface, deformation twin growth mediated by TBM consumes the matrix grains and neighboring grains. In the subsurface layer, due to lower internal energy, the growth of deformation twins mainly occurs by consuming neighboring grains. As the distance from the surface is increased, the migration of HAGBs becomes dominant, and the growth of matrix grains with lower internal energy is promoted, thereby consuming neighboring grains and twins. Additionally, no new grain nucleation or growth phenomena can be observed, indicating that the grain size and distribution remain relatively stable under the annealing conditions.

(3) It is found that the tensile twins possess the capability to engulf non-corresponding parent phases, which transcends the conventional understanding that twins interact solely with their corresponding parent grains. The tensile twins not only interact with the parent grain but also have the capacity to engulf twins of adjacent parent grains. In contrast, the isolated twins within the parent grains struggle to grow during annealing, indicating that $\{10\bar{1}2\}$ deformation twins have excellent thermal stability. The high thermal stability and microstructure optimization achieved by LSP treatment of AZ31 magnesium alloy, through precise control of microstructural evolution, provide a scientific basis for designing materials with gradient microstructures. It is expected that this work can provide a deep understanding of twinning phenomena in hexagonal close-packed (HCP) lightweight structural materials, offering new perspectives for the development of advanced alloy systems.

Acknowledgement

Bo Mao acknowledges the funding supports from the National Key R&D Plan of China (No. 2022YFB3705600), the National Natural Science Foundation of China (No. 52101046), the Excellent Youth Overseas Project of National Science and Natural Foundation of China, the Baowu Special Metallurgy Cooperation Limited (No. 22H010101336), and the Medicine-Engineering Interdisciplinary Project of Shanghai Jiao Tong University (No. YG2022QN076).

References

[1] D.A. Yukiko Ogawa, Yuji Sutou, Junichi Koike, A lightweight shape-memory magnesium alloy, Science, 353 (2016) 368-370.

[2] T.M. Pollock, Weight loss with magnesium alloys, Science, 328 (2010) 986–987.

[3] R.B. Figueiredo, T.G. Langdon, Principles of grain refinement in magnesium alloys processed by equal-channel angular pressing, Journal of materials science, 44 (2009) 4758-4762.

[4] J. Su, M. Sanjari, A. Kabir, I.-H. Jung, J. Jonas, S. Yue, H. Utsunomiya, Characteristics of magnesium AZ31 alloys subjected to high speed rolling, Materials Science and Engineering: A, 636 (2015) 582-592.

[5] M.K. Aghayani, B. Niroumand, Effects of ultrasonic treatment on microstructure and tensile strength of AZ91 magnesium alloy, J. Alloys Compd., 509 (2011) 114-122.

[6] C. Liu, H. Zheng, X. Gu, B. Jiang, J. Liang, Effect of severe shot peening on corrosion behavior of AZ31 and AZ91 magnesium alloys, J. Alloys Compd., 770 (2019) 500-506.

[7] Q. Liu, S. Chu, X. Zhang, Y. Wang, H. Zhao, B. Zhou, H. Wang, G. Wu, B. Mao, Laser shock processing of titanium alloys: a critical review on the microstructure evolution and enhanced engineering performance, Journal of Materials Science & Technology, (2024).

[8] W. Jia, H. Zhao, Q. Hong, L. Li, X. Mao, Research on the thermal stability of a near α titanium alloy before and after laser shock peening, Mater. Charact., 117 (2016) 30-34.

[9] Z. Zhou, S. Bhamare, G. Ramakrishnan, S.R. Mannava, K. Langer, Y. Wen, D. Qian, V.K. Vasudevan, Thermal relaxation of residual stress in laser shock peened Ti–6Al–4V alloy, Surf. Coat. Technol., 206 (2012) 4619-4627.

[10] J. Lu, K. Luo, Y. Zhang, G. Sun, Y. Gu, J. Zhou, X. Ren, X. Zhang, L. Zhang, K. Chen, Grain refinement mechanism of multiple laser shock processing impacts on ANSI 304 stainless steel, Acta Mater., 58 (2010) 5354-5362.

[11] B. Mao, Y. Liao, B. Li, Gradient twinning microstructure generated by laser shock peening in an AZ31B magnesium alloy, Appl. Surf. Sci., 457 (2018) 342-351.

[12] B. Mao, A. Siddaiah, X. Zhang, B. Li, P.L. Menezes, Y. Liao, The influence of surface pre-twinning on the friction and wear performance of an AZ31B Mg alloy, Appl. Surf. Sci., 480 (2019) 998-1007.

[13] A. Siddaiah, B. Mao, Y. Liao, P.L. Menezes, Effect of laser shock peening on the wear-corrosion synergistic behavior of AZ31B magnesium alloys, J. Tribol., (2019) 1-22.

[14] B. Mao, B. Li, D. Lin, Y. Liao, Enhanced room temperature stretch formability of AZ31B magnesium alloy sheet by laser shock peening, Materials Science and Engineering: A, 756 (2019) 219-225.

[15] A. Akhtar, E. Teghtsoonian, Solid solution strengthening of magnesium single crystals—I alloying behaviour in basal slip, Acta Metall., 17 (1969) 1339-1349.

[16] R.E. Reed-Hill, W.D. Robertson, Deformation of magnesium single crystals by nonbasal slip, JOM, 9 (1957) 496-502.

[17] H. El Kadiri, C.D. Barrett, J. Wang, C.N. Tomé, Why are twins profuse in magnesium?, Acta Mater., 85 (2015) 354-361.

[18] A. Khosravani, D. Fullwood, B. Adams, T. Rampton, M. Miles, R. Mishra, Nucleation and propagation of twins in AZ31 magnesium alloy, Acta Mater., 100 (2015) 202-214.

[19] B. Mao, Y. Liao, B. Li, Abnormal twin-twin interaction in an Mg-3Al-1Zn magnesium alloy processed by laser shock peening, Scripta Mater., 165 (2019) 89-93.

[20] L. Sihai, N. Xiangfan, Z. Liucheng, Y. Xi, H. Weifeng, L. Yinghong, Thermal stability of surface nanostructure produced by laser shock peening in a Ni-based superalloy, Surf. Coat. Technol., 311 (2017) 337-343.

[21] Y. Yang, K. Zhou, H. Zhang, H. Hu, H. Qiao, Thermal stability of microstructures induced by laser shock peening in TC17 titanium alloy, J. Alloys Compd., 767 (2018) 253-258.

[22] X. Ren, X. Yang, W. Zhou, J. Huang, Y. Ren, C. Wang, Y. Ye, L. Li, Thermal stability of surface nano-crystallization layer in AZ91D magnesium alloy induced by laser shock peening, Surf. Coat. Technol., 334 (2018) 182-188.

[23] M. Panchal, L. Kaushik, M.-S. Kim, R.K. Renganayagalu, S.-H. Choi, J. Singh, Effect of pre-twinning and heat treatment on formability of AZX311 Mg alloy, Journal of Magnesium and Alloys, 12 (2024) 1154-1169.

[24] S. Yi, I. Schestakow, S. Zaefferer, Twinning-related microstructural evolution during hot rolling and subsequent annealing of pure magnesium, Materials Science and Engineering: A, 516 (2009) 58-64.

[25] A. Jäger, P. Lukáč, V. Gärtnerová, J. Haloda, M. Dopita, Influence of annealing on the microstructure of commercial Mg alloy AZ31 after mechanical forming, Materials Science and Engineering: A, 432 (2006) 20-25.

[26] R. Sabat, D. Panda, S. Sahoo, Growth mechanism of extension twin variants during annealing of pure magnesium: An 'ex situ'electron backscattered diffraction investigation, Mater. Charact., 126 (2017) 10-16.

[27] D. Guan, W.M. Rainforth, L. Ma, B. Wynne, J. Gao, Twin recrystallization mechanisms and exceptional contribution to texture evolution during annealing in a magnesium alloy, Acta Mater., 126 (2017) 132-144.

[28] M. Yoo, S. Agnew, J. Morris, K. Ho, Non-basal slip systems in HCP metals and alloys: source mechanisms, Materials Science and Engineering: A, 319 (2001) 87-92.

[29] Y. Feng, L. Qian, C. Sun, S. Xu, N. Zhu, C. Wang, Y. Liu, Twinning, dynamic recrystallization, and texture evolution in as-solution AZ80 Mg alloy during hot compression, Journal of Materials Research and Technology, 25 (2023) 5159-5173.

[30] R. Doherty, D. Hughes, F. Humphreys, J.J. Jonas, D.J. Jensen, M. Kassner, W. King, T. McNelley, H. McQueen, A. Rollett, Current issues in recrystallization: a review, Materials Science and Engineering: A, 238 (1997) 219-274.

[31] Y.J. Kim, H.J. Kim, S.H. Park, Deformation mechanisms and mechanical properties of rolled AZ31 alloy subjected to precompression and subsequent annealing: Effect of annealing temperature, Mater. Sci. Eng., A, 899 (2024).

[32] Y.J. Kim, J.U. Lee, Y.M. Kim, S.H. Park, Microstructural evolution and grain growth mechanism of pre-twinned magnesium alloy during annealing, Journal of Magnesium and Alloys, 9 (2021) 1233-1245.

[33] M. Arul Kumar, I.J. Beyerlein, C.N. Tomé, Grain size constraints on twin expansion in hexagonal close packed crystals, J. Appl. Phys., 120 (2016).

[34] L. Wu, S.R. Agnew, D.W. Brown, G.M. Stoica, B. Clausen, A. Jain, D.E. Fielden, P.K. Liaw, Internal stress relaxation and load redistribution during the twinning–detwinning-dominated cyclic deformation of a wrought magnesium alloy, ZK60A, Acta Mater., 56 (2008) 3699-3707.

[35] X. Ye, Z. Suo, Z. Heng, B. Chen, Q. Wei, J. Umeda, K. Kondoh, J. Shen, An in-situ study of static recrystallization in Mg using high temperature EBSD, Journal of Magnesium and Alloys, (2023).

[36] Y. Xin, H. Zhou, G. Wu, H. Yu, A. Chapuis, Q. Liu, A twin size effect on thermally activated twin boundary migration in a Mg–3Al–1Zn alloy, Mater. Sci. Eng., A, 639 (2015) 534-539.

[37] R. Kirchheim, Simple geometrical aspects of grain growth in the framework of Herring's analysis and a disclination model, Acta Mater., 173 (2019) 327-331.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: