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Effect of Uphill Quenching on Microstructure and Residual Stress Reduction of AZ31B Magnesium Alloy Plate

Pengfei Ji [1], Jin Zhang [1,*], Jinghan Yang [1], Yongle Zhao [1], Yong Lian [1], Xiaomin Yuan [1], Chaoyang Sun [2] and Shitao Dou [3]

[1] Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China
[2] School of Mechanical Engineering, University of Science and Technology Beijing, Beijing 100083, China
[3] Southwest Technology and Engineering Research Institute, Chongqing 400039, China
* Correspondence: zhangjin@ustb.edu.cn; Tel.: +86-10-82377393

Abstract: Residual stress may be generated during the deformation process; cold and hot treatments on magnesium alloy, causing deformation; cracking; and other effects. Reducing the residual stress of magnesium alloys is of great significance for its size stability and quality. In this paper, the residual stress in the AZ31B plate was compared with different uphill quenching processes: no uphill quenching (NUQ), liquid nitrogen–boiling water (100 °C) (LNB), liquid nitrogen–hot air (160 °C) (LNHA) and liquid nitrogen–water (25 °C) (LNR). Residual stresses with and without treatment were measured by X-ray diffraction. The effect of uphill quenching on hardness was discussed. The microstructure and diffraction pattern of the samples treated with different uphill quenching processes was investigated by EBSD and XRD. The results showed that the microstructure of magnesium alloy rolling plate was refined by the uphill quenching treatment, which can reduce the residual stress without decreasing the mechanical properties. The largest residual stress reduction rate was obtained by the liquid nitrogen–boiling water process. This treatment process can not only reduce the residual stress of the magnesium alloy rolling plates by 56% but also increase the hardness by 29%.

Keywords: AZ31B magnesium alloy; uphill quenching; residual stress reduction; microstructure; mechanical property

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1. Introduction

The residual stress generated by the plastic processing of magnesium alloys, such as extrusion, rolling and forging [1,2], can cause distortion failure, deformation, stress corrosion cracking or fatigue of work pieces [3,4]. More attention has been paid to the problems caused by the residual stress of magnesium alloy products, with the increasing application of magnesium alloy in transportation and electronics industries [5]. However, the research on residual stress and the methods of stress reduction for magnesium alloy products is still insufficient. There are unsolved questions in the traditional residual stress reduction methods applying to magnesium alloy products.

The traditional method of reducing residual stress is thermal aging. The key to the thermal aging method is the contradiction of at the same time reducing residual stresses and preserving mechanical property. Although the thermal aging method has a marked effect on reducing residual stress, it will reduce the mechanical properties of materials [6,7]. Vibration aging is a method used to eliminate residual stress by mechanical vibration, which has a shorter process time and saves energy compared with heat treatment, but it requires special and complex equipment [8]. Prestretching is a commonly used residual stress reduction process for aluminum alloy rolling or extruding sheets. The residual stress of the rolled or extruded aluminum sheets is released by additional plastic deformation after prestretching. However, it is difficult for magnesium alloy sheets to re-form at room temperature because of the strong c-axis//ND basal texture generated during the primary forming of the conventional rolling or extruding magnesium alloy sheets, which makes it difficult to coordinate the arbitrary deformation in 3D space in the process of secondary forming [9,10]. It is difficult to reduce the residual stress of magnesium alloy by prestretching. Therefore, it is necessary to find an effective method to reduce the residual stress in the work pieces.

The uphill quenching process is used in reducing the residual stress of ferrous alloys and aluminum alloys in the high-precision field and other fields [11–13]. It is the reverse process of conventional quenching [14]. The process is to treat the sample in a specific and controllable cryogenic environment (usually with liquid nitrogen as refrigerant), with an increase in temperature to reduce residual stress [15,16]. Research results have shown that magnesium alloy plates treated by cryogenic treatment during uphill quenching can be improved in microstructure, wear resistance, hardness and service life [17–20], which may solve the problem of the preserving mechanical property in thermal aging. However, the mechanism for the residual stress reduction of magnesium alloy by the uphill quenching treatment and the effect of different uphill quenching processes on residual stress reduction of magnesium alloy are unclear and need to be further studied.

In this paper, three uphill quenching processes were studied. The residual stresses were measured by the nondestructive X-ray diffraction method, and the residual stress reduction effect was evaluated by the relief rate. The hardness of the material was tested. Electron back-scattered diffraction (EBSD) and X-ray diffraction (XRD) were used to investigate the structure and phase of the materials [21]. According to the testing results, a better uphill quenching process was obtained, and the related residual stress reduction mechanism was discussed.

2. Materials and Methods

The AZ31B extruded plates (with an extruding temperature of 390 °C and an extruding speed of 1 m/min) was shaped into 100 mm (ED) × 110 mm (TD) × 9 mm (ND) and quenched in water at room temperature after being heated at 350 °C for 2 h. The material used in this work was magnesium alloy AZ31B, produced by Western Titanium Technology Co., Ltd. (Xi'an, China). The compositions of the main alloying elements are shown in Table 1.

Table 1. The compositions of the main alloying elements of AZ31B.

Element	Al	Zn	Mn	Si	Fe	Cu	Ni	Ca	Mg
Composition	2.50~3.50	0.60~1.40	0.20~1.00	≤0.08	≤0.003	≤0.01	≤0.001	≤0.04	Bal.

As shown in Figure 1, the quenched AZ31B plates were treated in three uphill quenching processes, separately, as follows:

(a) First deep cryogenic treated in liquid nitrogen (LN) for 0.5 h, then uphill quenched in room temperature water (LNR) of 25 °C for 0.5 h and cooled in air until room temperature at last;
(b) First deep cryogenic treated in liquid nitrogen (LN) for 0.5 h, then uphill quenched in boiling water (LNB) of 100 °C for 0.5 h and cooled in air until room temperature at last;
(c) First deep cryogenic treated in liquid nitrogen (LN) for 0.5 h, then uphill quenched in hot air (LNHA) of 160 °C for 0.5 h and cooled in air until room temperature at last.

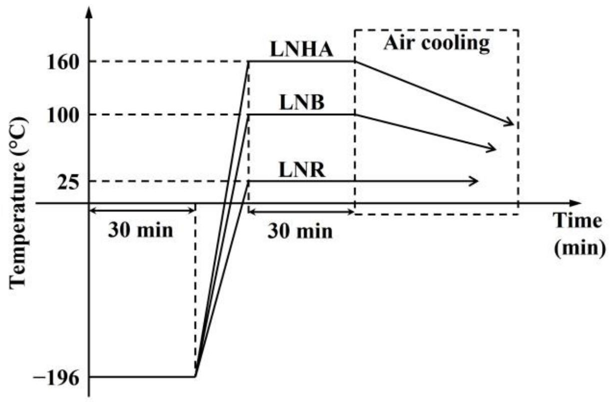

Figure 1. Three uphill quenching processes.

The testing specimens were cut into 10 mm × 10 mm × 9 mm blocks by electrical discharge machining. The progress of precipitation hardening was monitored by using Vickers hardness equipment calibrated with a standard test block to the requirements of ASTM E92–92. The testing force was 50 gf, and the maintaining time was 15 s [22].

Surface residual stress measurement using a cosα method was performed by a μ-X360 portable X-ray diffractometer(Pulstec Industrial Co.,Ltd. Hamamatsu, Japan) using Cr Kα radiation. The diffraction geometry of residual stress determination was shown in Figure 2a. Debye rings were determined for residual stress calculation. The position of the magnesium $\{20\bar{2}2\}$ peak was measured to calculate the residual stress. The distributions of measurement points on the AZ31B plate surface are shown in Figure 2b. To ensure the accuracy of experimental results, the testing points were chemically polished by nitric acid solution (20% vol) for 15 s before residual stress determination.

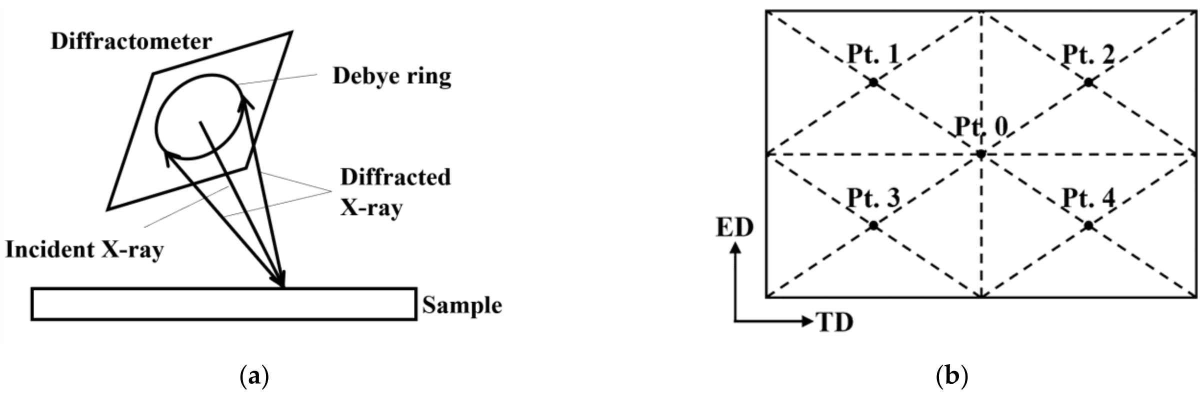

Figure 2. (**a**) Diffraction geometry of residual stress determination by X-ray diffraction and (**b**) distribution of the residual stress testing points of the AZ31B magnesium alloy plates.

First, the EBSD samples were mechanically polished. Then electrolytical polishing was employed to remove the stress layer on the surface to ensure the accuracy of the testing results.

3. Results

3.1. Residual Stress

To evaluate the relief effect of uphill quenching, the residual stresses before and after uphill quenching were measured. The formula used to calculate the relief rate was as follows:

$$\eta = \frac{|\sigma_1| - |\sigma_2|}{|\sigma_1|}, \tag{1}$$

where σ_1 and σ_2 is the residual stress of the specimens before reduction and after reduction, respectively, and η is the relief rate. Figure 3 shows the residual stress relief rates at different points treated by the liquid nitrogen–boiling water method. The highest relief rate in the test points is the center point (point 0) in the extruding direction and reached 90%. The relief rate at point 4 is the lowest, with a relief rate of 27%. The average relief rate is 56% for the whole piece of plate.

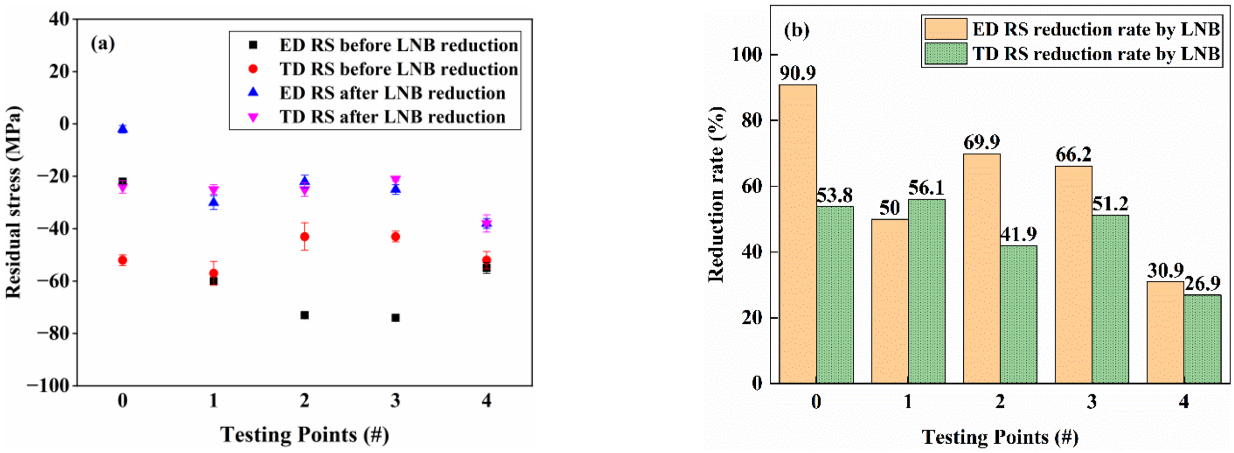

Figure 3. (**a**) ED and TD residual stress and (**b**) relief rate at different points treated by LNB.

The residual stress relief rates at different points treated by the liquid nitrogen–boiling water method are shown in Figures 3 and 4. The maximum relief rate is 74% at point 1 of extrusion, and the minimum relief rate is 31% at point 0 at the center of the plate. The average relief rate of point 5 is 52%. The reason can be illustrated as follows: the lower heat exchange coefficient of air to liquid leads to the slow drop of surface temperature of magnesium alloy in the liquid nitrogen–hot air process, resulting in a smaller temperature difference between the inside and the surface of the magnesium alloy plate, while the lower temperature increase cannot provide larger energy for the microdeformation of the plate. As a result, the relief effect is not as obvious as that of the liquid nitrogen–boiling water process.

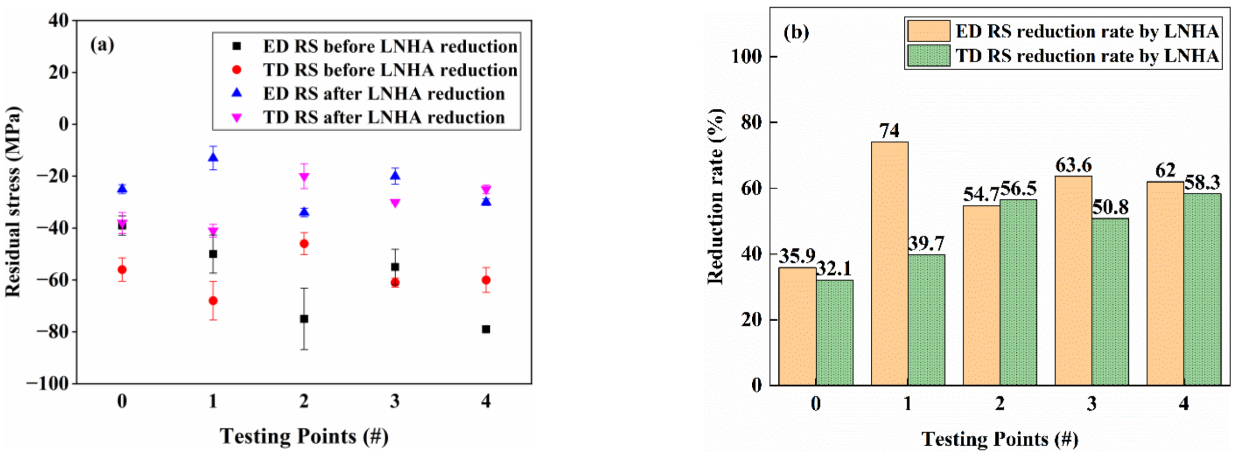

Figure 4. (**a**) ED and TD residual stress and (**b**) relief rate at different points treated by LNHA.

Figure 5 shows the residual stress relief rates at different points treated by the liquid nitrogen–room temperature water method. The residual stress relief rates of point 0, point 2 and point 4 are increased, point 2 and point 3 are reduced, and the relief rate is low, which indicates that the liquid nitrogen–room temperature water process has a weak effect on reducing residual stress and is not as sufficient as the others.

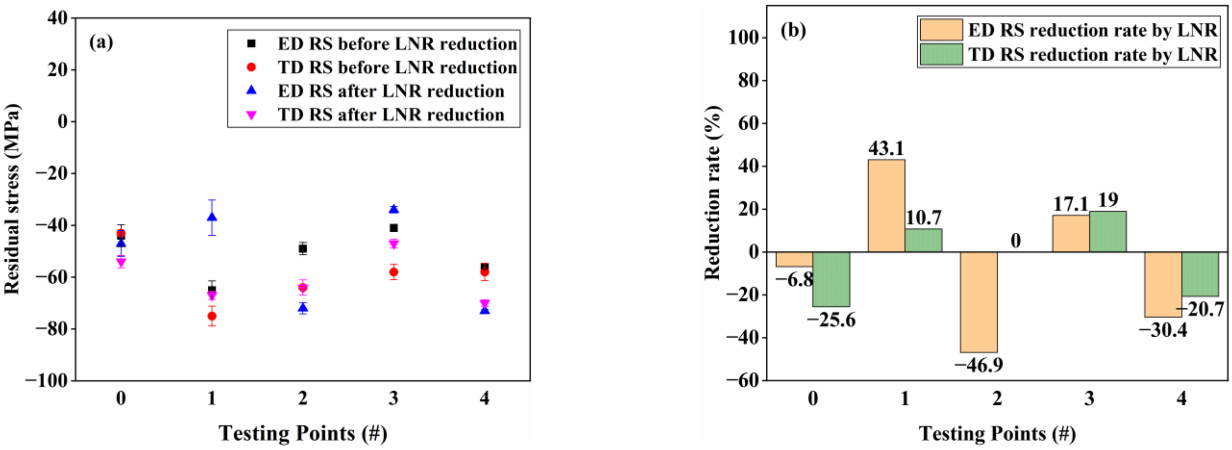

Figure 5. (**a**) ED and TD residual stress and (**b**) relief rate at different points treated by LNR.

A comparison of the above three methods reveals that the liquid nitrogen–boiling water method has the best effect on reducing residual stress, as shown in Figure 6. The effect of the uphill quenching process to reduce residual stress is related mainly to temperature variation. The temperature increased by 300 °C, 360 °C and 200 °C for the three processes, respectively, and the biggest temperature difference was obtained for the liquid nitrogen–hot air process. However, the best reducing effect is obtained by the liquid nitrogen–boiling water process because the liquid nitrogen–hot air heat exchange coefficient is lower than that in water, and it cannot produce ample energy in a short time to cause microdeformation in the plate. The liquid nitrogen–room temperature water process has the lowest effect on reducing the residual stress because there is not enough temperature difference and the energy generated is lower than the yield strength of the plate and it has little effect on the residual stress relief.

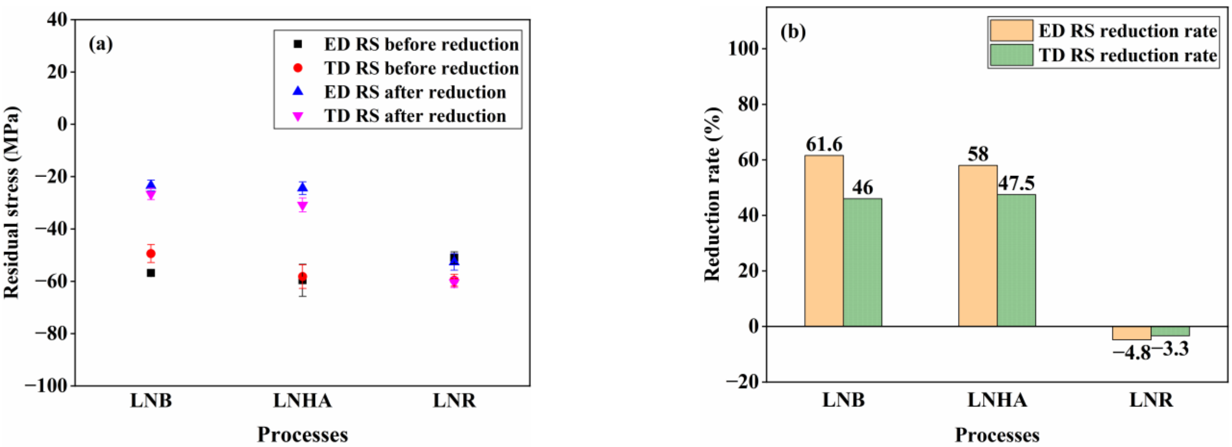

Figure 6. Comparison of average (**a**) ED and TD residual stress and (**b**) relief rate treated by three uphill quenching processes.

3.2. Microhardness

The result of microhardness in different positions of AZ31B magnesium alloy plates is shown in Figure 7. AZ31B magnesium alloy without being treated by the liquid nitrogen–boiling water process has an average Vickers hardness of 49 HV, while it reached 63 HV after treatment by the liquid nitrogen–boiling water process. AZ31B magnesium alloy undergoes a 29% increase in hardness after liquid nitrogen–boiling water treatment.

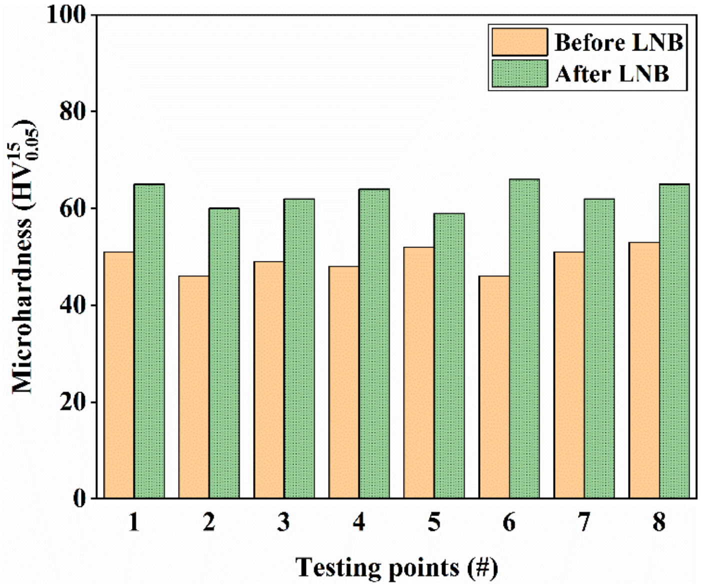

Figure 7. Microhardness of AZ31B before and after LNB.

Ample deformation energy and compressive stress inside the crystal has been generated during the uphill quenching process. The deformation can increase the internal energy of the matrix, and it is in the metastable state, which is beneficial to the change of crystal structure.

3.3. Diffraction Analysis

Figure 8 shows the XRD patterns of samples treated by different uphill quenching processes. The diffraction peak crystal faces are {0002}, {10$\bar{1}$1}, {10$\bar{1}$2}, {10$\bar{1}$3} and {0004}. The {0002} peak is the highest, instead of {10$\bar{1}$1}, which is the strongest peak of the standard pattern of Mg powder diffraction.

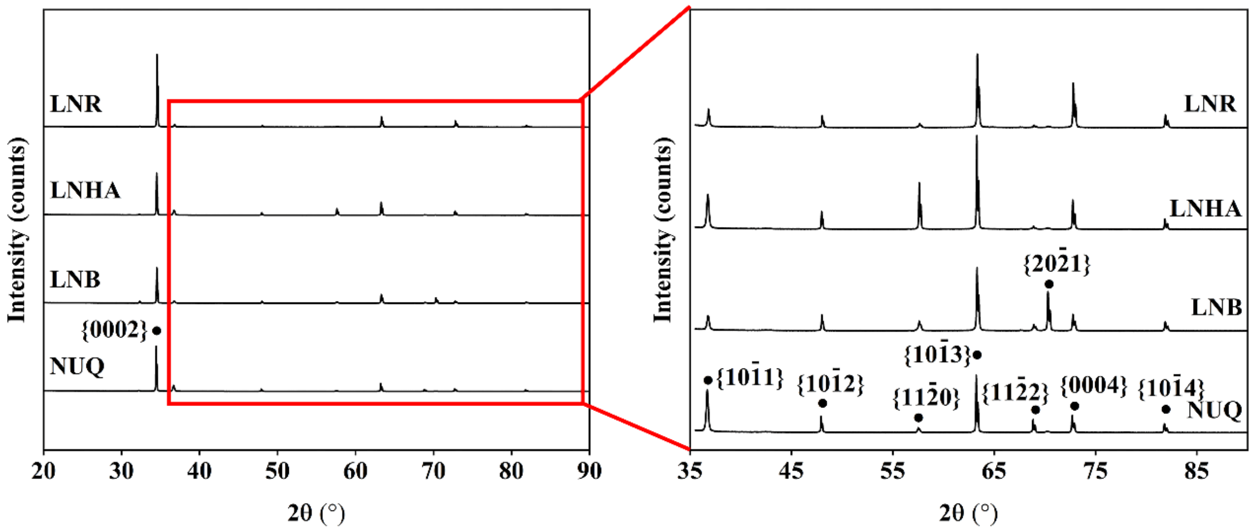

Figure 8. XRD patterns of AZ31B plates processed with different uphill quenching processes.

According to Figure 8, the diffraction peak intensity of {0002} is reduced, whereas the {10$\bar{1}$2}, {10$\bar{1}$3} and {0004} peaks increased. The residual stress relief of AZ31B magnesium alloy is the process of transferring {0002} to other crystal planes. Magnesium alloy produces a strong {0002} peak during processing. The uphill quenching process provides energy for the recovery and recrystallization inside the material, which is beneficial for generating dislocations and subgrains; reducing the orientation of {0002} during the generation of a large number of dislocations and subgrains and adding the texture of the crystal faces provides the possibility of reducing residual stress.

Figure 9a,b shows the Debye rings of the samples before and after undergoing the liquid nitrogen–boiling water process. The color of the Debye ring corresponds to the peak intensity, and the position of the ring corresponds to the 2θ angle. The intensity near the red diffraction peak is higher, and the intensity near the blue diffraction peak is lower. For isotropic materials, the diffraction intensity at each position of Debye ring should be similar. The texture of the rolled plates results in the high diffraction intensity points on the Debye ring. The texture is responsible for the points of higher diffraction intensity indicated by the arrows in Figure 9a,b. The decrease of intensity and number of those points shows that the strength of the texture decreases after LNB treatment. The position indicated by the red arrow in Figure 9b is closer to the blue than the position indicated in Figure 9a indicating that the strength of the texture of the AZ31B plate becomes weaker after LNB. It is proved that the method of uphill quenching (liquid nitrogen–boiling water method) can weaken the texture while reducing the residual stress.

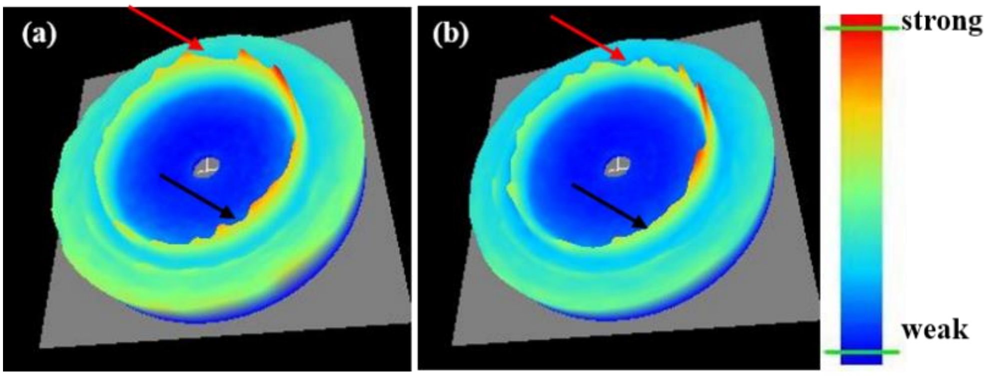

Figure 9. Change of Debye ring (**a**) before and (**b**) after LNB.

3.4. Microstructure

Microstructural evolution was studied by using electron back-scattered diffraction of AZ31B magnesium alloy after different ascending quenching processes. Figure 10a shows the sample without being treated by an uphill quenching process, Figure 10b treated by a liquid nitrogen–boiling water process, Figure 10c treated by a liquid nitrogen–hot air process and Figure 10d treated by a liquid nitrogen–room temperature water process. The magnesium alloy plate is recrystallized and regenerated, and fine grains are interposed in the middle of the large grains. Here, large grains are broken in severe temperature changes, and the grains are refined and uniform. In Figure 10c,d, parts of the grains are refined because only a small part of the energy from the temperature change provides power for grain refinement, resulting in incomplete grain refinement. During the uphill quenching process, the thermal expansion and contraction produces a large amount of strain energy and squeezing force. The strain energy can improve the internal energy of the AZ31B magnesium alloy plate. When it is in a metastable state, it is active, which is conducive to the initiation of dislocations. Granular arrangement is denser. Figure 10b–d shows that a large amount of fine subgrain appeared in the microstructure after the uphill quenching process, which is caused by large grain breakage during rapid cooling and heating.

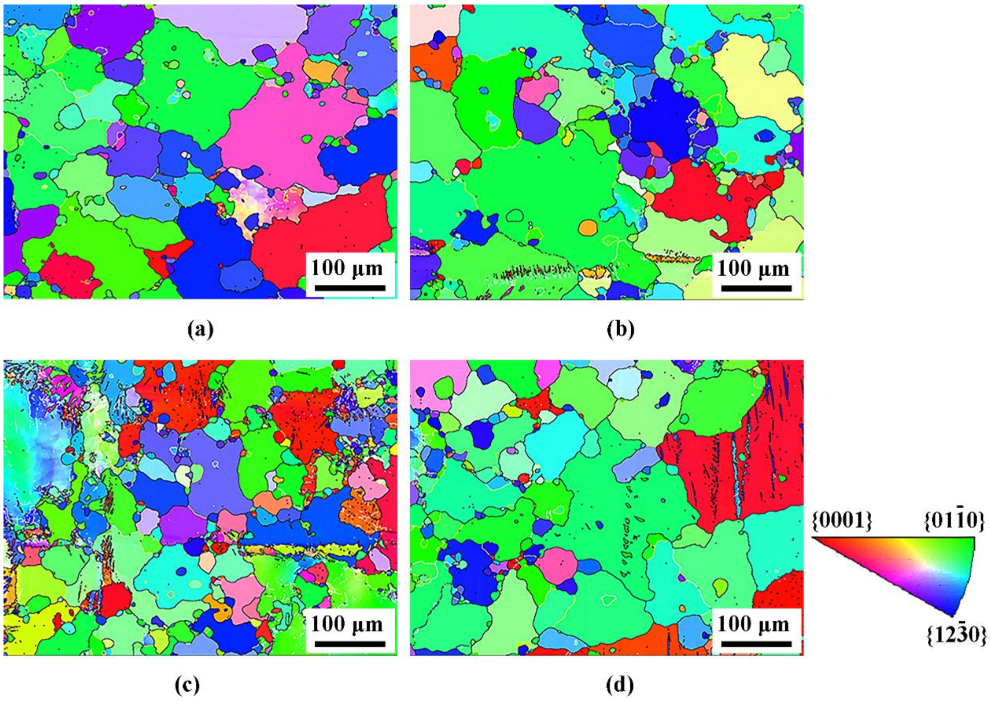

Figure 10. EBSD of AZ31B plates treated by (**a**) NUQ, (**b**) LNB, (**c**) LNHA and (**d**) LNR uphill quenching processes.

4. Discussion

The reduction of residual stress by uphill treatment is caused by the process of the work piece's being chilled to a low temperature and then rapidly heated. Because of the different expansion coefficients between the phases in the structure, between the grains with different orientations and even between the directions inside the grains, the generated microstress is superimposed with the original residual stress. When the two stress directions are opposite, they cancel each other out; when the directions are the same, the addition of stresses is enough to exceed the yield strength of the material to cause local microscopic plastic deformation (but the macroscopic size remains unchanged or changes very little), and the residual stress relaxes. Figure 11 shows the change of stress field of AZ31B magnesium alloy plate during uphill quenching. Figure 11 shows the residual stress distribution after quenching. At the initial stage of quenching, the surface cooling rate is faster than that inside, resulting in a greater surface shrinkage than that inside. Tensile stress was formed on the surface and compressive stress inside. Afterward, the internal cooling rate is higher than the external cooling rate, resulting in compressive stress on the surface and tensile stress inside. Figure 11 shows the residual stress distribution after being subjected to the uphill quenching process. When the sample is placed in liquid nitrogen, the surface cooling rate is faster than the internal, tensile stress that was generated on the surface, and the internal compressive stress is opposite to the direction of the stress generated by the previous quenching, thus canceling each other out and the absolute value of the stress is reduced. As time increases, the temperature decreases; the internal cooling rate is greater than the surface; compressive stress was generated on the surface; and tensile stress is inside. When the sample is placed in the initial stage of boiling water, the surface temperature is higher than the internal heating rate, the surface is subjected to compressive stress, and the external is tensile stress. The superposition with the previous stress increases the residual stress. As the temperature increases, the internal temperature rises faster than the external heating rate, leading to the internal tensile stress of the surface tensile stress, which overlaps with the previous stress, and the stress decreases. The final plate stress distribution state is shown in Figure 11.

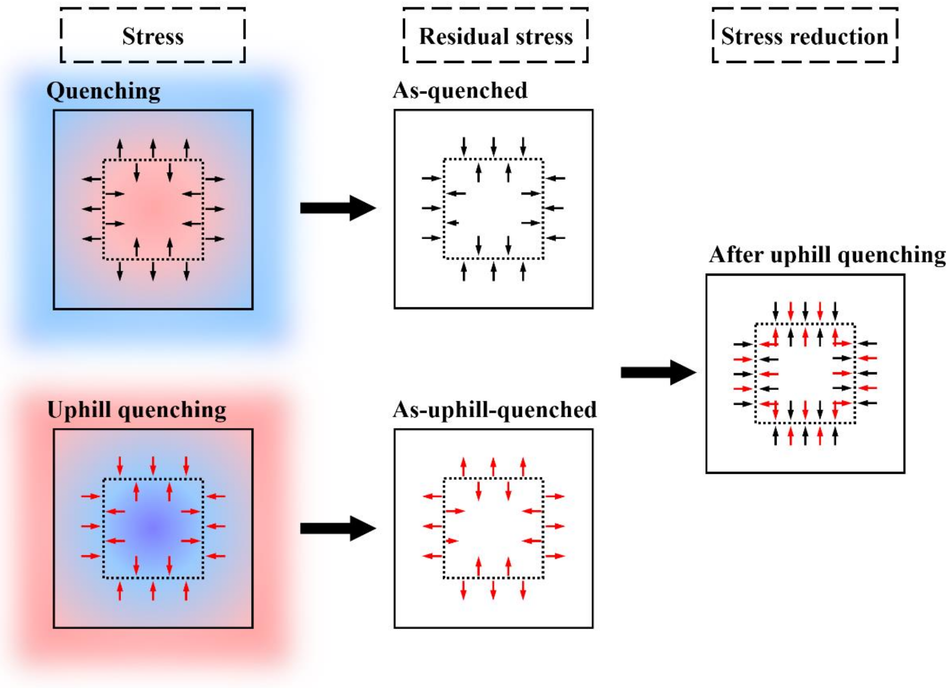

Figure 11. Stress field variation of a plate during the initial quenching process and the subsequent uphill quenching process.

5. Conclusions

(1) The residual stress of the magnesium alloy rolling plates was reduced during the three uphill quenching processes. The best effect of residual stress distribution with relief rate of 56% of AZ31B magnesium alloy was obtained by the liquid nitrogen–boiling water process, and the relief rate of the liquid nitrogen–room temperature water process was the lowest.

(2) Uphill quenching treatment was shown to be beneficial to improving the hardness of the material. The hardness of AZ31B magnesium alloy can be increased by about 29% after being treated by the uphill quenching process.

(3) The microstructure of the magnesium alloy rolling plate was refined by the uphill quenching treatment. The variation of crystal plane spacing and temperature field were the main factors to influence the residual stress of uphill quenching.

(4) There must be a certain temperature difference in the uphill quenching. The effect of reducing the temperature below a certain temperature difference was not obvious. The heat exchange coefficient should be large enough to beneficially reduce the residual stress. The liquid nitrogen–room temperature water process has a poor relief effect due to an insufficient temperature difference. The lower liquid nitrogen–hot air heat exchange coefficient resulted in a lower relief rate than that of the liquid nitrogen–boiling water process.

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