metals

MDPI

Article

Effect of Cryogenic Time on Microstructure and Properties of TRCed AZ31 Magnesium Alloy Sheets Rolled during Cryogenic Rolling

Chen-Chen Zhi [1], Pei-Jin Li [1], Zhi-Quan Huang [1], Peng-Tao Liu [1], Hai-Jie Xu [2], Wei-Tao Jia [1] and Li-Feng Ma [1,*]

1 School of Mechanical Engineering, Taiyuan University of Science and Technology, Taiyuan 030024, China
2 Part Rolling Key Laboratory of Zhejiang Province, Ningbo University, Ningbo 315211, China
* Correspondence: mlf_zgtyust@163.com; Tel.: +86-151-3515-0129

Abstract: Rolling experiments of TRCed AZ31 magnesium alloy with different cryogenic treatment time were carried out to study the evolution mechanism of its microstructure and mechanical properties. The experimental results showed that with the increase in cryogenic time, the grain size of the sheets after cryogenic rolling was significantly refined, and the dislocation density and texture strength were greatly weakened. The combined effect led to a significant increase in the elongation and tensile strength of the sheet after cryogenic rolling. The tensile strength, elongation and average hardness of the sheet increased from 282.6 MPa, 8.2%, and 54.6 HV to 305.4 MPa, 16.3%, and 62.8 HV, respectively. Therefore, when the cryogenic treatment time was 60 s, the performance of the rolled sheet was the best. At the same time, the appearance of dimples after cryogenic rolling led to a change of the fracture mechanism, which was also the key to the improvement of the sheet elongation.

Keywords: AZ31 magnesium alloy; cryogenic time; microstructure; mechanical properties

check for updates

Citation: Zhi, C.-C.; Li, P.-J.; Huang, Z.-Q.; Liu, P.-T.; Xu, H.-J.; Jia, W.-T.; Ma, L.-F. Effect of Cryogenic Time on Microstructure and Properties of TRCed AZ31 Magnesium Alloy Sheets Rolled during Cryogenic Rolling. *Metals* **2023**, *13*, 870. https://doi.org/10.3390/met13050870

Academic Editor: Massimo Pellizzari

Received: 31 March 2023
Revised: 26 April 2023
Accepted: 27 April 2023
Published: 29 April 2023

1. Introduction

As a light and high-strength material, a magnesium alloy has a very extensive research significance in modern industrial applications. In previous studies, magnesium alloys were processed mainly by extrusion, forging, and traditional rolling to obtain good properties. However, the phenomenon of strong plastic mismatch often occurs in magnesium alloys produced by traditional rolling, forging, or extrusion processing. Xu et al. [1] studied and compared the extrusion parameters and produced a Mg-7.5Gd-2.5Y-3.5Zn-0.9Ca-0.4Zr (wt%) alloy with a strength of up to 400 MPa, but its plasticity was only 8.8%. Zhang et al. [2] studied the properties of AZ31 by hot ring forging and obtained that the fracture elongation of the alloy was as high as 28%, but its strength was only 238 MPa. Liu et al. [3] produced a sheet with a maximum tensile strength of 332 MPa by hot-roller-cold-material rolling, but its fracture elongation was only 5%.

Because a magnesium alloy is often used to manufacture spacecraft, automobile parts, medical instruments and other products with high requirements, its practical application needs to withstand complex and variable loads and cannot tolerate any mechanical failure or quality problems in these fields [4]. If magnesium alloy materials with unmatched strength and plasticity are used, safety hazards from product failure are likely to occur, causing great damage to the environment and human life. Therefore, a magnesium alloy with matched strength and toughness can improve product safety, make it more capable of bearing pressure and challenges under various extreme working conditions, and adapt to the needs of the industrial market.

The strength and elongation of metal materials are challenging to improve simultaneously, and the improvement of one side often accompanies the weakening of the other. In recent years, studies have shown that the cryogenic rolling process under ultralow-temperature deformation can improve the strength and elongation of the alloy at the same time. Cryogenic rolling is a rolling process with liquid nitrogen as the cooling medium, which cools the rolled sheet after cryogenic treatment to liquid nitrogen temperature and then plastically deforms the material. It mainly inhibits the recovery and recrystallization of the alloy during deformation at a low temperature so as to improve the strength and elongation of the alloy effectively.

The ultrafine grain structure of an Al 7075 alloy was obtained by cryogenic rolling at liquid nitrogen temperature by Das et al. [5]. The yield strength and impact toughness of the material after cryogenic rolling were increased by 108% and 60%, respectively, mainly due to refined grain strengthening. Yu et al. [6]. Obtained a nanostructured 1050 aluminum alloy by cryogenic asynchronous rolling and improved the strength and elongation of aluminum alloy sheets. By applying cryogenic rolling in copper alloy, Yue Lu et al. [7] carried out large plastic deformation of a Cu-Ag alloy by a cryogenic treatment of liquid nitrogen. They explored its texture evolution and recrystallization behavior. The results showed that the dynamic recovery was inhibited during cryogenic rolling, resulting in a high deformation energy storage in the alloy. Zhu et al. [8–10] found that the cold-rolling process was more conducive to generating ultrafine grains and twins. Due to the low stacking-fault energy of the alloy, this behavior could improve the strength and elongation of the alloy at the same time. Li et al. [11] found that a cryogenic treatment could significantly improve the hardness and strength of stainless steel without a negative impact on plasticity. Therefore, cryogenic rolling substantially enhances the strength and elongation of aluminum alloy, copper alloy, steel, etc.

In recent years, due to the unique contribution and advantages of magnesium alloys in the production of lightweight vehicles, more and more researchers have paid attention to the research and application of magnesium alloys' cryogenic treatment [12]. Huang et al. [13] and Preciado Mónica [14] found that appropriate cryogenic treatment conditions could significantly refine the grain size of magnesium alloys and improve their strength, elongation, and elasticity. Hu et al. [15] found that the secondary phase generated and increased with the different action times, and many twins were observed in the grains after cryogenic treatment. Jiang et al. [16] found that cryogenic treatment could change the grain orientation and improve the tensile strength and hardness of AZ31 magnesium alloy by cryogenic treatment. Liu et al. [17] analyzed the microstructure, mechanical properties, and physical characteristics of an AZ91 magnesium alloy before and after cryogenic treatment. The results showed that the compressive strength and elongation of the sample were significantly improved after cryogenic treatment. At the same time, the resistance and crystal lattice constant of the alloy was reduced considerably.

Although cryogenic treatment has been used to improve the properties of magnesium alloys, many factors affect the microstructure and properties of the alloys during cryogenic rolling, such as the cryogenic time, rolling speed, pass reduction, and initial temperature. Therefore, in this paper, the effect of the cryogenic time on the microstructure and properties of a magnesium alloy was studied by the cryogenic rolling experiment of an AZ31 magnesium alloy intermediate sheet after a cryogenic treatment, combined with an electron backscattered diffraction (EBSD) study and scanning electron microscopy (SEM) of the rolled magnesium alloy sheet, an analysis of the room temperature tensile properties, and a hardness test to better control the cryogenic time of cryogenic rolling and improve the cryogenic treatment process.

2. Material and Methods

2.1. Materials

A 7.0 mm thick cast-rolled AZ31 magnesium alloy sheet was selected, and a 6.8 mm thickness sheet was obtained after grinding and cleaning the surface. Three 300 °C isothermal rolling experiments were carried out on the 6.8 mm sheet on the isothermal twin-roll reversible rolling mill. The reduction of each pass was 35%, and the cumulative reduction of the three passes was 72%. Finally, the 40 mm × 140 mm × 1.9 mm twin-roll-casted

(TRCed) AZ31 sheets were obtained as the initial sheet, and the subsequent cryogenic rolling was carried out. The process flow is shown in Figure 1.

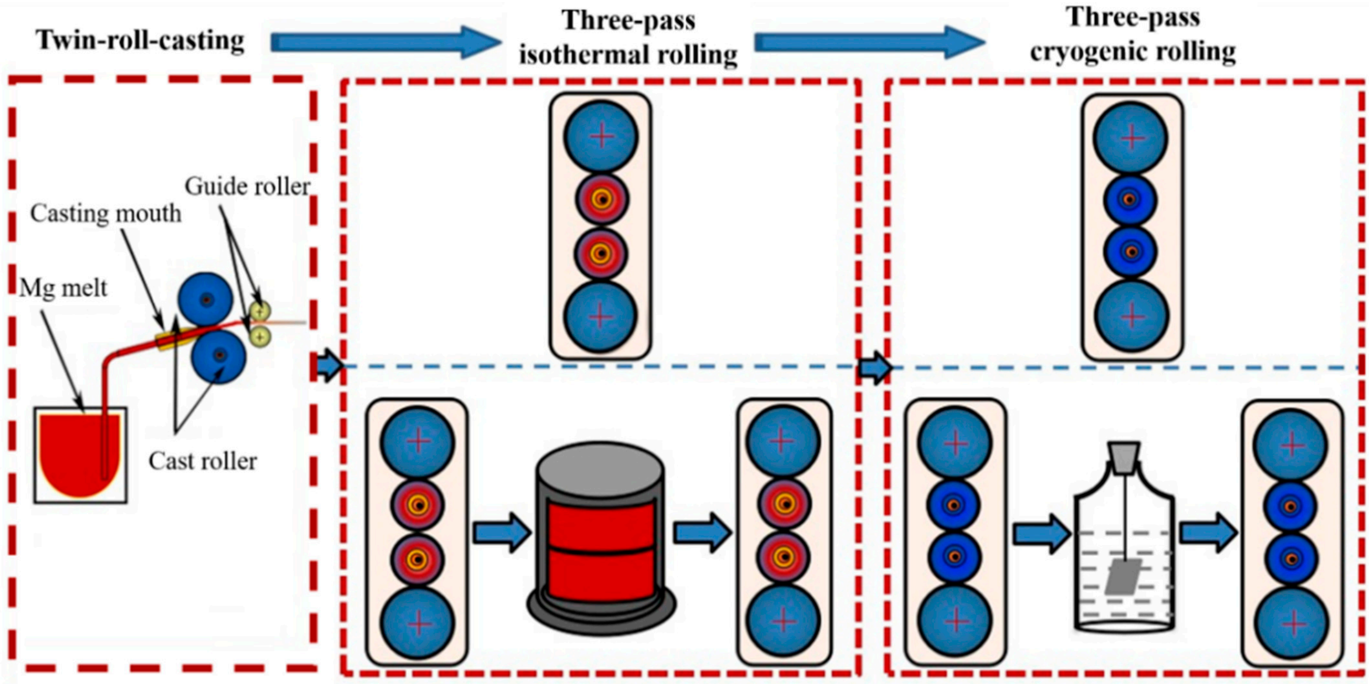

Figure 1. Process flow of TRCed AZ31 sheets at cryogenic rolled.

2.2. Cryogenic Rolling

The cryogenic rolling experiment was carried out on a two-roll mill with a roll diameter of 380 mm, a roll length of 300 mm, and a maximum rolling force of 1200 kN. The intermediate sheet was subjected to three-pass cryogenic rolling according to the process parameters.

Specific experimental scheme: a setting rolling speed of 0.2 m/s, a single-pass reduction of 5%, three-cumulative-pass reduction of 14.2%, and the sheet was rolled from 1.9 mm to 1.6 mm. Before starting cryogenic rolling, the rolled sheet was wholly immersed in the liquid nitrogen tank for cryogenic treatment. For cryogenic treatment, the plates needed to be completely immersed in the liquid nitrogen tank, and the two-high mill was used for rolling immediately after removal from the liquid nitrogen tank. Cryogenic treatment was performed before each rolling, and the cryogenic treatment time was the same each time. In this paper, three kinds of low-temperature rolling processes were designed, and the low-temperature times were 0 s, 30 s and 60 s, respectively. The experimental samples were directly immersed in liquid nitrogen for rapid cooling and cryogenic treatment, and the cryogenic equipment was a double-layer insulation liquid nitrogen tank.

2.3. Methods for the Characterization of the Microstructure

The samples with the cryogenic treatment (CT) time of 0 s, 30 s, and 60 s were tested by EBSD and SEM and for tensile properties and hardness at room temperature. The microstructures were characterized by ZEISS Sigma-300 electron backscattered diffraction, and their parameters were tested by a 2 kV accelerated voltage, a 22 mm working distance, a 70° tilt, and 0.2~0.5 µm scanning steps depending on the magnification. We drew the processing diagram of a tensile sample according to the GB/T 16865-2013 standard and to scale. The sample was cut along the rolling direction (RD) of the sheet by an electric discharge cutting machine. Then, the tensile property was tested by an IN-STRON 5969 universal material testing machine with a tensile rate of 1 mm/min. Three samples were measured for each process to ensure data accuracy. An HVS-1000A digital display Vickers hardness tester was used for the hardness test. The loading load was 0.98 kN and the loading time was 10 s. The three samples were recorded as CT−0 s, CT−30 s, and CT−60 s, respectively.

3. Results and Discussion

3.1. Microstructure Characteristics after Rolling

The inverse pole figure and grain statistical figure of the RD-TD plane of the AZ31 magnesium alloy with a thickness of 1.6 mm after cryogenic rolling are shown in Figure 2. Figure 2a shows the microstructure of a cast-rolled AZ31 magnesium alloy sheet directly placed in the air without cooling in liquid nitrogen after rolling, presenting the typical microstructure. It was mainly composed of coarse grains whose part was as wide as 18 μm and a large number of refined grains whose part was as narrow as 2.8 μm, with large grains elongated along the rolling direction. The microstructure of the sheet cooled in liquid nitrogen after cryogenic rolling had apparent changes, as shown in Figure 2c,e. The grain size was relatively uniform, and the grain was significantly refined. The average grain size of the sheet without cryogenic treatment was about 3.98 μm. After cryogenic treatment before rolling for 30 s, the average grain size decreased to 3.39 μm. After cryogenic treatment before rolling for 60 s, the average grain size decreased to 2.53 μm. The average grain size decreased with the increase in cryogenic time. It can be seen from the grain statistical diagram that the proportion of grains above 10 μm was significantly reduced in the CT−30 s sample compared with the sheet without cryogenic treatment. Compared with the CT−30 s sample, the proportion of grains smaller than 4 μm in the CT−60 s sample increased significantly, reaching about 80% of the total grains. Therefore, with the increase in the cryogenic time, it can be seen that the grain size was refined substantially, the microstructure uniformity was improved dramatically, and the average grain size was gradually reduced.

The reason for the above situation was that after the AZ31 magnesium alloy at about 200 °C was immersed in liquid nitrogen for cryogenic treatment, the temperature of the sheet decreased rapidly leading to the shrinkage of the volume and lattice of the material. At this time, a large amount of internal stress was generated inside the material, resulting in an internal stress concentration. After rolling, residual stresses in the alloy were applied to the vacancy defects and larger grains, forming dislocation walls and further transforming into grain boundaries, which then formed new grains. [18] At the same time, due to the influence of the low temperature, the rapid grain nucleation resulted in an insufficient time for recrystallization grain growth [19].

The extension of the cryogenic time from 30 s to 60 s resulted in changes in the lattice sequence of the matrix and a gradual thinning of the matrix grains. On the other hand, it led to the accumulation of strain energy, which provided the power and time for the dislocation to climb so that the dislocation with the same sign was rearranged and the dislocations with different signs cancelled each other out. The dislocation wall of the same dislocation arrangement was transformed into a grain boundary forming a subcrystal, and the grain size of the alloy was refined, which resulted in the decrease of the average grain size with the extension of the cryogenic time.

The (0001) pole figure of the sheet after different cryogenic rolling processes is shown in Figure 2. Most of the grains of the sheet after rolling had crystal planes parallel to the rolling direction, so they all produced a firm matrix texture on the (0001) plane. In Figure 2a, the sheet without cryogenic treatment was the most obvious, and its maximum texture strength reached 17.86. In comparison, the sheet with cryogenic treatment before rolling presented a weak (0001) texture in Figure 2b,c because with the increase in cryogenic time, the growth of grain produced in the rolling deformation process can be stopped as soon as possible to complete the recrystallization process. This caused the decrease in average grain size, which led to a sustained weakening of the substrate texture [20]. The maximum texture strength drops to 12.11 for the CT−30 s sample and 11.91 for the CT−60 s sample.

Compared with the texture without cryogenic treatment, the strength of the texture after cryogenic treatment was significantly reduced by more than 30%. However, with the increase in cryocooling time, the strength of the texture fluctuates slightly. Meanwhile, the study of Wang et al. [21] showed that the weakening of the texture strength is conducive to enhancing the ductility of the magnesium alloy. Therefore, with the increase in cryogenic time, the texture strength of the sheet gradually decreased and reached the lowest point when the cryogenic time was 60 s.

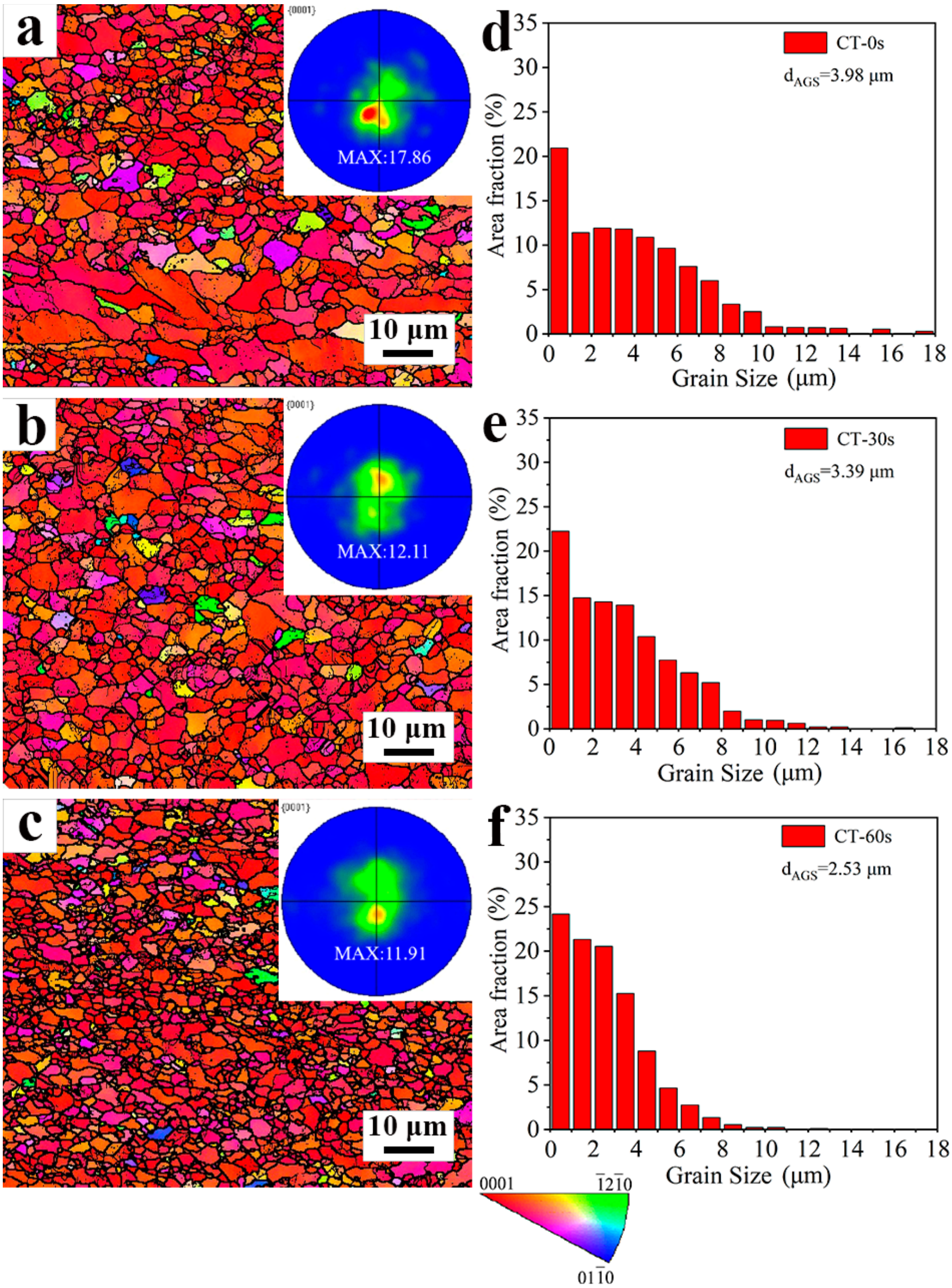

Figure 2. Inverse pole figure, (0001) pole figure: (**a**) CT−0 s, (**c**) CT−30 s, (**e**) CT−60 s, and grain size statistics (**b**) CT−0 s, (**d**) CT−30 s, (**f**) CT−60 s of TRCed AZ31 alloy after different cryogenic treatments.

The kernel average misorientation (KAM) maps on the corresponding EBSD results are shown in Figure 3. Figure 3a shows the microstructure without cryogenic treatment. Some areas with a high geometrically necessary dislocation (GND) density are green, and the overall performance is blue, indicating that the microstructure of this sample was mainly recrystallized grains, and the internal orientation difference was slight. In Figure 3b, the green area increased significantly, showing green and blue crisscross, indicating that the intragranular orientation difference increased significantly after the cryogenic treatment, and most of these green areas existed in refined grains without recrystallization growth, so the internal orientation difference was significant, and the dislocation density was high. Due to many dynamic recrystallizations in the material rolled after the CT−60 s, many dislocations were consumed, significantly reducing the dislocation density. Therefore, the green area of Figure 3c decreased compared with that of Figure 3b, and the dislocation density in the material increased first and then decreased with the increase in cryogenic time.

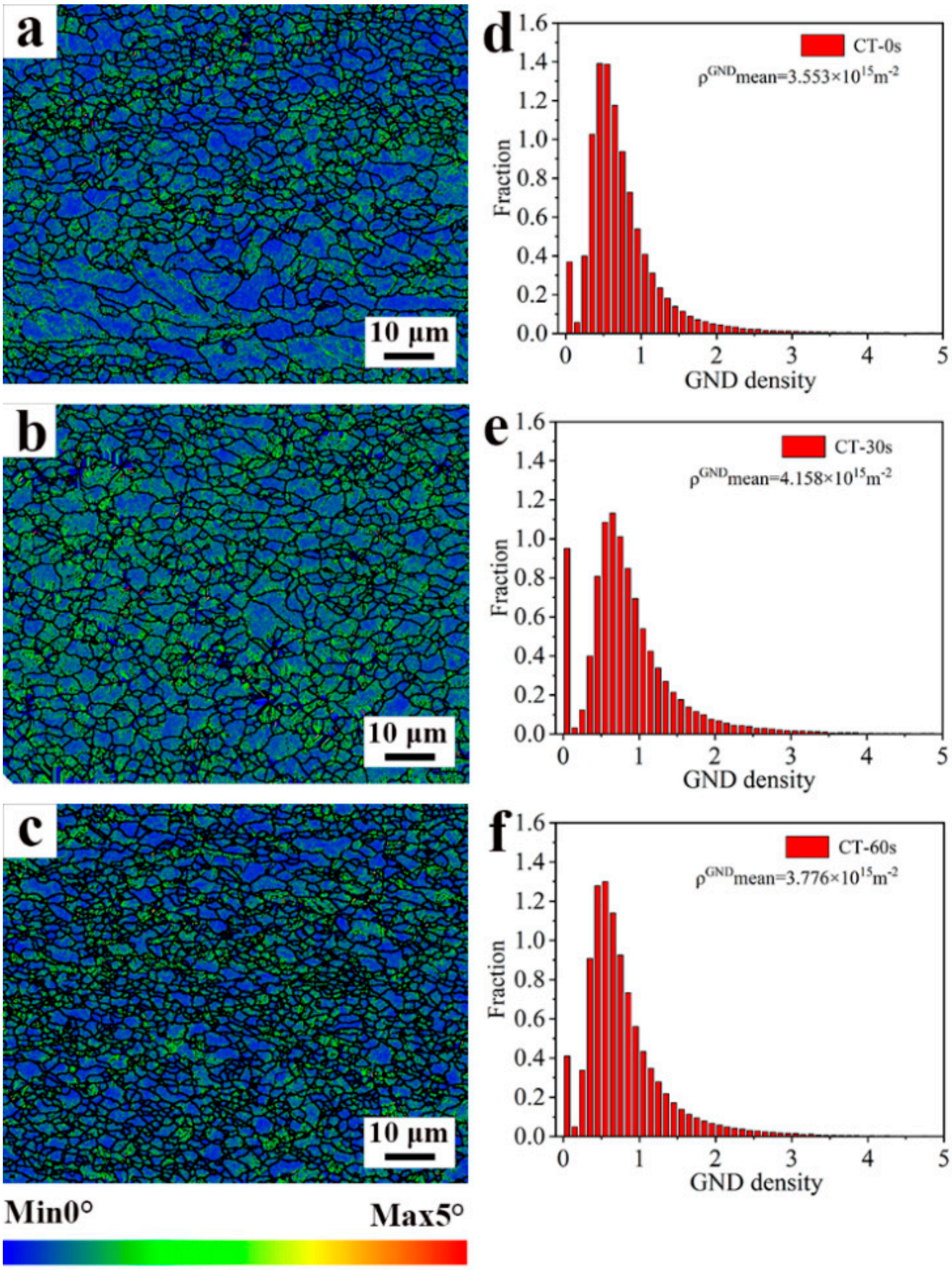

Figure 3. Kernel average misorientation (KAM) maps on corresponding EBSD result in Figure 2 of (**a**) CT−0 s, (**b**) CT−30 s, (**c**) CT−60 s and GND density statistics (**d**–**f**).

In order to intuitively explain the KAM values of the three samples, the dislocation density of the alloy was calculated by Zhu et al. [22]:

$$\rho^{GND} = \frac{2\theta}{\mu b},$$

where θ represents the local directional dislocation derived from KAM results, b is the Burgers vector (the value of Mg is 3.21×10^{-10} m), and μ is the step size of the EBSD test (0.26 μm). According to Figure 3d–f, the ρ^{GND} values under different conditions were obtained. According to Wang et al. [23] and Ma et al. [24], the KAM value positively correlates with the GND density. The GND density of the CT−30 s sample was 4.158×10^{15} m^{-2}, which was significantly higher than that of the other two samples, so the dislocation density of the CT−30 s sample was the largest.

In general, changes in dislocation density affect both the strength and toughness of the material. [25] The larger the dislocation density, the stronger the material and the worse the elongation. However, it can be seen from the KAM diagram that with the increase in cryogenic time, the dislocation density in the material first increased and then decreased, and at the same time, the grain refinement and texture strength decreased, as shown in Figure 2. In order to confirm the influence mechanism of the comprehensive factors on the properties of the materials, we carried out uniaxial tensile experiments on the materials.

3.2. Analysis of Mechanical Properties

The effect of cryogenic time on the mechanical properties of the AZ31 magnesium alloy before rolling was analyzed by tensile tests at room temperature. The engineering stress–strain curves are shown in Figure 4. It can be seen that the ultimate tensile strength, yield strength, and elongation of the alloy at room temperature increased with the prolonged cryogenic time. The ultimate tensile strength, yield strength, and elongation of the sheet without cryogenic treatment were 282.6 MPa, 211.4 MPa, and 8.2%, respectively. The change of ultimate tensile strength of the sheet after a 30 s treatment was relatively small, reaching 298.4 MPa, but the yield strength and elongation increased significantly, reaching 251.2 MPa and 13.3%, respectively. The performance of the CT−60 s sample was the best, slightly higher than the CT−30 s sample. The ultimate tensile strength, yield strength, and elongation reached 305.4 MPa, 261.14 MPa, and 16.3%, respectively. At the same time, the study of Miao et al. [26] on multipass cryogenic rolling of an AZ31 magnesium alloy and the study of Wu et al. [27] on cryocooled sheets at different initial temperatures obtained sheets with good performance and balanced tensile strength and elongation.

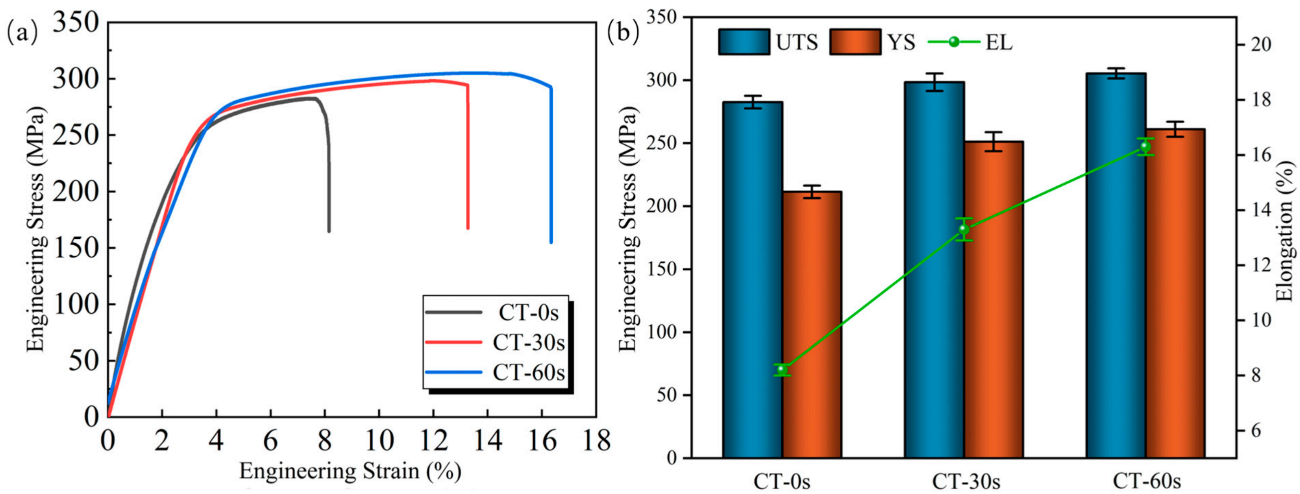

Figure 4. (**a**) Engineering stress–strain curve of AZ31 magnesium alloy after cryogenic treatment; (**b**) histograms of tensile properties of the alloys at room temperature.

However, conventional production methods cannot simultaneously improve the strength and toughness. For example, in powder metallurgy rolling studied by Wang et al. [28], the tensile strength of the AZ31 magnesium alloy sheet was significantly increased, from 244.37 MPa to 296.56 MPa, but the elongation was significantly decreased, from 22.97% to 12.22%. Vignesh et al.'s [29] study on cross rolling only obtained sheets with the maximum tensile strength and optimal elongation of 254 MPa and 6%, respectively. Ren et al.'s [30] study on the asymmetric rolling of an AZ31B magnesium alloy produced sheets with only a 250.3 MPa tensile strength and a 12.5% elongation. The results showed that it was feasible to manufacture magnesium alloys with excellent properties by regulating the process of cryogenic rolling.

The tensile samples of the AZ31 magnesium alloy after cryogenic rolling at different cryogenic times are shown in Figure 5, where a–c are the macroscopic fracture surfaces after cryogenic treatment for 0 s, 30 s, and 60 s, respectively, and d–f are the local characteristic magnification images of a–c, respectively. The macroscopic fracture morphology of the samples before cryogenic rolling in Figure 5a,b showed prominent characteristics of river pattern, marked in Figure 5d, and cleavage step, marked in Figure 5e, which was a typical cleavage fracture belonging to a brittle fracture. Compared with the samples without cryogenic treatment before rolling, the samples after cryogenic treatment in Figure 5b,c had dimples, marked in Figure 5f, and a large number of tear edges, which indicated that the samples had a certain degree of plastic deformation during the fracture process, presenting a mixed fracture mechanism of brittle fracture and ductile fracture. The results also confirmed that the cryogenic treatment before rolling could improve the elongation of the AZ31 magnesium alloy.

With the increase in cryogenic time, the number of dimples in the macro fracture morphology increased significantly, which led to a significant increase in the proportion of ductile fracture in the ductile–brittle mixed fracture of the specimen. Moreover, due to the dislocation entanglement of the CT−30 s sample, it was easy to produce microcracks, leading to the expansion of the specimen along the cleavage plane and thus affecting the plasticity of the material. Therefore, the elongation of the CT−60 s samples was slightly improved compared with that of the CT−30 s samples.

After a cryogenic treatment for 30 s, the performance of the sheet after cryogenic rolling was greatly improved. The main reason was that after cryogenic treatment, the ultralow-temperature environment of liquid nitrogen made the internal lattice of the material shrink, resulting in the accumulation of a large number of dislocations in the alloy after cryogenic rolling, and the dislocation movement was easy, resulting in a dislocation entanglement. On the other hand, the texture of the sheet was weakened, and the grains were relatively uniform and small compared with the sheet without cryogenic treatment before rolling, which ultimately led to a substantial increase in material performance [31]. However, for the sheet after the 60 s cryogenic treatment, with the occurrence of a dynamic recrystallization of the sheets during cryogenic rolling, the dislocation density was slightly lower than that of the sheet after the 30 s cryogenic treatment, but the grains were more refined. The further weakening of its texture and the further refined grains caused a slight increase in the elongation and yield strength of the sheet; Liu et al. [32] also found a similar trend in the texture study of AZ31.

Moreover, the sudden drop in temperature led to the sharp contraction of the crystal structure of the alloy. The degree of different crystal planes of the magnesium alloy was different, resulting in a lattice distortion and deformation energy [33]. The energy generated by the lattice distortion could hinder the slip deformation of the dislocation and finally, the strength of the material was slightly improved.

3.3. Microhardness Analysis

Figure 6 is the mean value, deviation, and sampling method of the microhardness of cryogenic rolled sheets at different times; each sample was tested at 15 points of uniform distribution according to the sampling method shown in the figure. When the cryogenic time was 0 s, the hardness value of the alloy varied significantly at different positions due to the alloy's uneven microstructure, as can be seen from the width of the deviation bar in Figure 6. The irregular grain size of the sheet after rolling caused an uneven hardness distribution. Under the two cryogenic conditions, the hardness value of the alloy at different positions fluctuated. Still, the overall distribution was relatively uniform, which was mainly caused by the elimination of the internal stress of the sheet before each pass of cryogenic treatment so that the grain distribution of the sheet was uniform [34]. When the cryogenic time was 0 s, the average hardness of the alloy was 54.6 HV. When the AZ31 magnesium alloy was cryogenically cooled for the CT−30 s sample, the average hardness increased to 57.2 HV; when the cryogenic time was 60 s, the average hardness of the alloy increased to 62.8 HV.

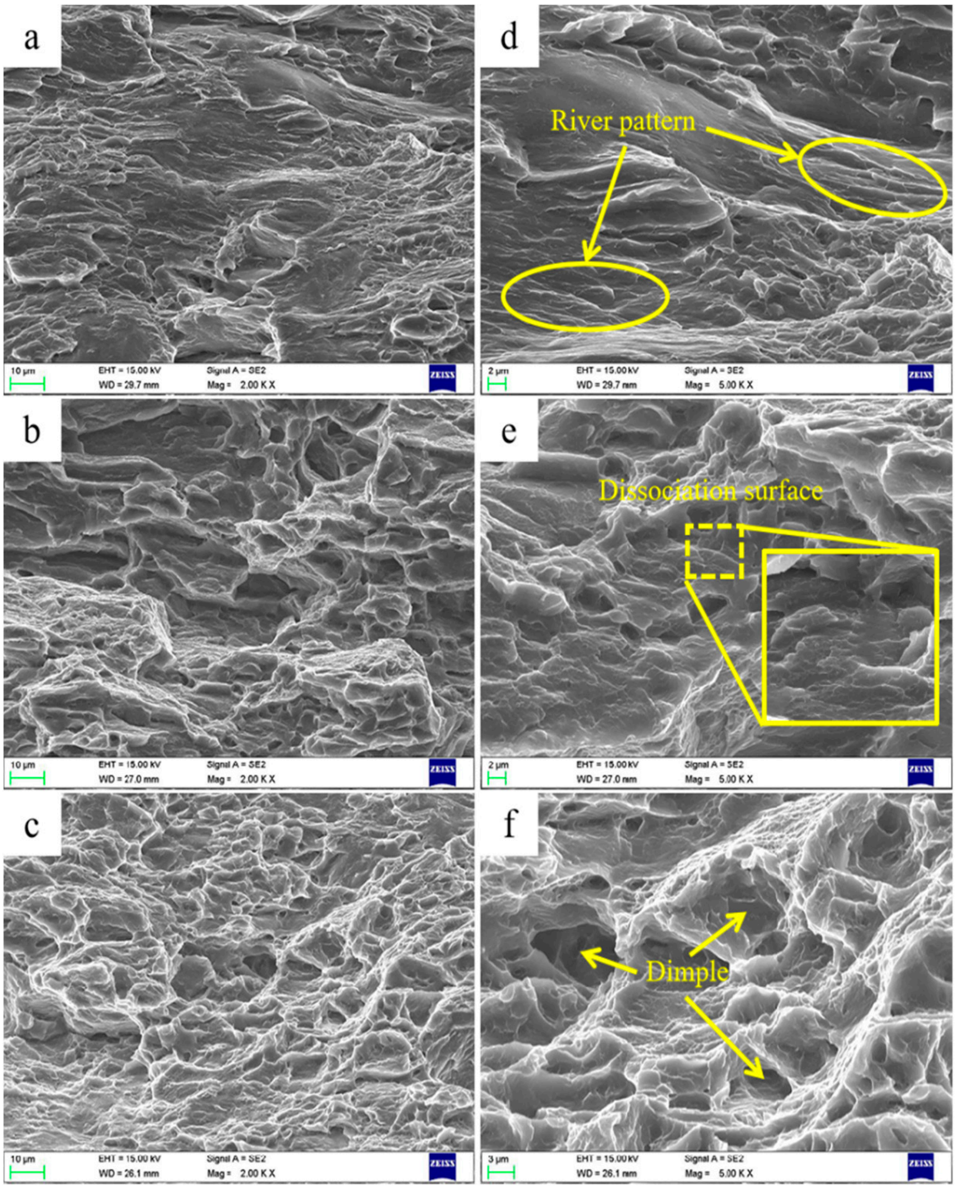

Figure 5. Fracture morphologies of rolled samples under different cryogenic treatment conditions: (**a**) CT−0 s; (**b**) CT−30 s; (**c**) CT−60 s; (**d**–**f**) are the local enlargements graphs of (**a**–**c**), respectively.

It can be seen from the average hardness value of the alloy that it increased gradually with the increase in cryogenic time. In this experiment, the microhardness of the alloy reached its peak value with 60 s of cryogenic time. From the papers published by Yu et al. and Yadollahpour et al., we learned the influence mechanism of a cryogenic treatment on a titanium alloy, aluminum alloy, and copper alloy [35,36]. A cryogenic treatment can affect the residual stress on the material's surface and increase the concentration of atomic arrangement vacancy and dislocation density. From the microstructure point of view, the ultralow-temperature environment of a cryogenic treatment can promote the precipitation of the dispersed phase and significantly refine the grain [37]. During a cryogenic treatment, various influencing factors interact and finally show the uniformity and improvement of the overall hardness of the sheet.

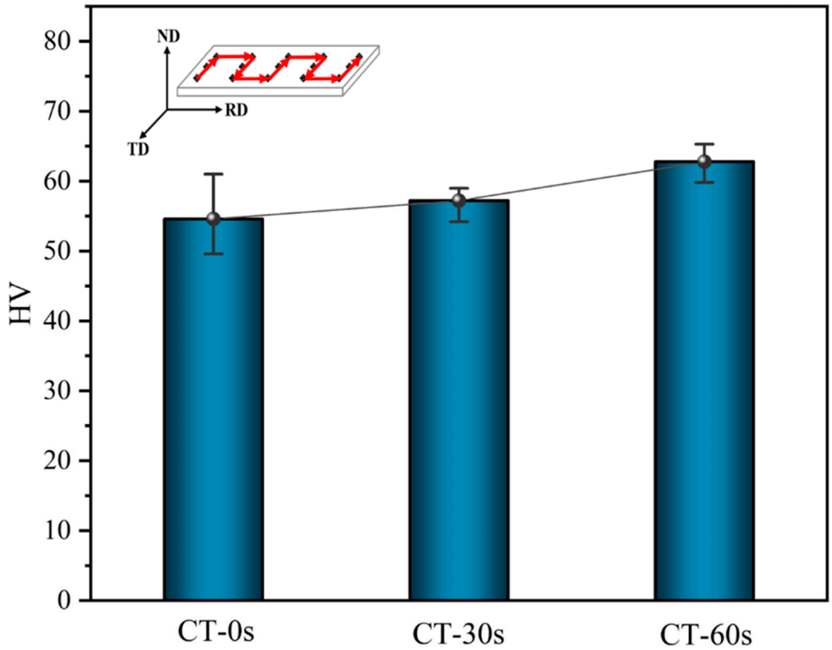

Figure 6. The mean value, deviation, and sampling method of the microhardness of cryogenic rolled sheets at different times.

4. Conclusions

The effect of the cryogenic treatment time on the microstructure and properties of a TRCed AZ31 magnesium alloy was analyzed by characterizing the microstructure and testing the tensile properties and hardness. The main conclusions are as follows:

1. With the increase in cryogenic time, the texture strength of the sheets decreased continuously, and the microstructure uniformity of the sheets increased gradually. The average grain size decreased from 3.98 μm to 2.53 μm. This is mainly because the matrix lattice sequence changes with the extension of cryogenic treatment time, which leads to refining matrix grains. At the same time, the accumulation of strain energy also provides the power and time for dislocation climbing. This results in the formation of sub crystals which contribute to the reduction of average grain size. After the cryogenic treatment, the macrofracture mechanism of the sheet gradually changed from a brittle-fracture to a ductile–brittle mixed-fracture mechanism. These combined effects made the elongation and tensile strength of the cryogenic rolling improve constantly.
2. The comprehensive mechanical properties of the AZ31 magnesium alloy sheet were significantly improved with the increase in cryogenic treatment time. When the low-temperature time was 60 s, the performance of the sheet were best. The tensile strength was increased from 282.6 MPa to 305.4 MPa by 8.1%. The yield strength and elongation were increased from 211.4 MPa and 8.2% to 261.14 MPa and 16.3%, respectively, by 23.5% and 98.8%. The overall hardness was remarkably uniform, increasing from 54.6 HV to 62.8 HV. This was mainly because the grain refinement was often accompanied by an increase in strength and hardness, and a decrease in

texture strength often led to an increase in elongation. Therefore, compared with the conventional AZ31 preparation method, it is feasible to obtain a sheet with excellent properties by regulating the cryogenic treatment time.

Author Contributions: Conceptualization, C.-C.Z. and L.-F.M.; methodology, C.-C.Z.; software, C.-C.Z.; investigation, C.-C.Z., Z.-Q.H. and W.-T.J.; formal analysis, C.-C.Z.; writing—original draft preparation, C.-C.Z. and P.-J.L.; data curation, P.-T.L.; visualization, investigation, Z.-Q.H.; resources, P.-T.L. and L.-F.M.; supervision, P.-T.L. and L.-F.M.; visualization H.-J.X.; writing—review and editing, H.-J.X. and L.-F.M.; funding acquisition, L.-F.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China (U1910213, 52201047, and 52075357), Taiyuan University of Science and Technology Scientific Research Initial Funding (TYUSTSRIF) (20212007), and Award Grants for Outstanding Doctor Working in Shanxi (20212072).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: The authors express thanks to the National Natural Science Foundation of China (U1910213, 52201047, and 52075357), Taiyuan University of Science and Technology Scientific Research Initial Funding (TYUSTSRIF) (20212007), and Award Grants for Outstanding Doctor Working in Shanxi (20212072).

Conflicts of Interest: The authors declare that they have no known competing financial interest or personal relationships that could have appeared to influence the work reported in this paper.

References

1. Xu, C.; Nakata, T.; Qiao, X.G.; Jiang, H.S.; Sun, W.T.; Chi, Y.C.; Zheng, M.Y.; Kamado, S. Effect of extrusion parameters on microstructure and mechanical properties of Mg-7.5Gd-2.5Y-3.5Zn-0.9Ca-0.4Zr (wt.%) alloy. *Mater. Sci. Eng. A* **2017**, *685*, 159–167. [CrossRef]
2. Zhang, J.; Huang, H.; Yang, C.B. Effects of hot ring forging on microstructure, texture and mechanical properties of AZ31 magnesium alloy. *Mater. Sci. Eng. A* **2017**, *679*, 20–27. [CrossRef]
3. Liu, D.; Bian, M.Z.; Zhu, S.M.; Chen, W.Z.; Liu, Z.Y.; Wang, E.D.; Nie, J.F. Microstructure and tensile properties of Mg-3Al-1Zn sheets produced by hot roller-cold-material rolling. *Mater. Sci. Eng. A* **2017**, *706*, 304–310. [CrossRef]
4. Pant, R.; Singh, S.; Joshi, A.; Joshi, K.; Saxena, K. Mechanical performance and characteristics evaluation of material through Cryo rolling process: A review. *Mater. Today Proc.* **2022**, *62*, 3086–3096. [CrossRef]
5. Das, P.; Jayaganthan, R.; Singh, I.V. Tensile and impact-toughness behaviour of cryorolled Al 7075 alloy. *Mater. Des.* **2011**, *32*, 1298–1305. [CrossRef]
6. Yu, H.L.; Lu, C.; Tieu, K.; Liu, X.H.; Sun, Y.; Yu, Q.B.; Kong, C. Asymmetric cryorolling for fabrication of nanostructural aluminum sheets. *Sci. Rep.* **2012**, *2*, 772. [CrossRef]
7. Lu, Y.; Ma, R.; Wang, Y.N. Texture evolution and recrystallization behaviors of Cu–Ag alloys subjected to cryogenic rolling. *Trans. Nonferrous Met. Soc. China* **2015**, *25*, 2948–2957. [CrossRef]
8. San, X.Y.; Liang, X.G.; Chen, L.P.; Xia, Z.L.; Zhu, X.K. Influence of stacking fault energy on the mechanical properties in cold-rolling Cu and Cu–Ge alloys. *Mater. Sci. Eng. A* **2011**, *528*, 7867–7870. [CrossRef]
9. San, X.Y.; Liang, X.G.; Cheng, L.P.; Li, C.J.; Zhu, X.K. Temperature effect on mechanical properties of Cu and Cu alloys. *Mater. Des.* **2012**, *35*, 480–483. [CrossRef]
10. San, X.Y.; Liang, X.G.; Cheng, L.P.; Shen, L.; Zhu, X.K. Effect of stacking fault energy on mechanical properties of ultrafine-grain Cu and Cu–Al alloy processed by cold-rolling. *Trans. Nonferrous Met. Soc. China* **2012**, *22*, 819–824. [CrossRef]
11. Wang, L.; Dong, C.F.; Cao, Y.; Liang, J.X.; Li, X.G. Co-enhancing the Mechanical Property and Corrosion Resistance of Selective Laser Melted High-Strength Stainless Steel via Cryogenic Treatment. *J. Mater. Eng. Perform.* **2020**, *29*, 7052–7062. [CrossRef]
12. Ugur, K.; Husnu, K. An Experimental Investigation on Machinability of AZ31B Magnesium Alloy under Dry and Dipped Cryogenic Approaches. *J. Mater. Eng. Perform.* **2022**, *31*, 1285–1296.
13. Huang, Z.Q.; Wei, J.C.; Huang, Q.X.; Ma, L.F.; Yue, Z.H. Effect of Cryogenic Treatment Prior to Rolling on Microstructure and Mechanical Properties of AZ31 Magnesium Alloy. *Rare Met. Mater. Eng.* **2018**, *47*, 2942–2948.
14. Mónica, P.; Bravoa, P.M.; Cárdenas, D. Deep cryogenic treatment of HPDC AZ91 magnesium alloys prior to aging and its influence on alloy microstructure and mechanical properties. *J. Mater. Process. Technol.* **2017**, *239*, 297–302. [CrossRef]
15. Hu, Z.Q.; Guo, C.F.; Li, H.M. Influence of AZ31 sheet treated by cryogenic on punch shearing. *J. Cent. South Univ.* **2019**, *26*, 1582–1591. [CrossRef]

16. Li, J.; Jiang, X.Q. Effect of Cryogenic Treatment on the Microstructure and Mechanical Properties of AZ31 Magnesium Alloy. *Mater. Sci. Forum* **2011**, *686*, 53–56. [CrossRef]
17. Liu, J.W.; Li, G.F.; Chen, D.; Chen, Z.H. Effect of Cryogenic Treatment on Deformation Behavior of As-cast AZ91 Mg Alloy. *Chin. J. Aeronaut.* **2012**, *25*, 931–936. [CrossRef]
18. Che, B.; Lu, L.W.; Zhang, J.L.; Zhang, J.H.; Ma, M.; Wang, L.F.; Qi, F.G. Effects of cryogenic treatment on microstructure and mechanical properties of AZ31 magnesium alloy rolled at different paths. *Mater. Sci. Eng. A* **2022**, *832*, 142475. [CrossRef]
19. Ren, X.W.; Huang, Y.C.; Liu, Y. Cryorolling-Induced Texture, Mechanical Properties and Fracture Behavior of Al-Mg-Si Alloys During Cold Deformation. *J. Mater. Eng. Perform.* **2018**, *27*, 3932–3939. [CrossRef]
20. Zhang, Y.; Ma, J.; Wang, Q.; Yang, Y.B.; Zhu, M.J.; Jia, P.L.; Duan, Y.H.; Zhang, Z.M. Study on anisotropic behavior of 7075 Al alloy after extrusion. *Mater. Res. Express* **2022**, *9*, 026508. [CrossRef]
21. Wang, S.H.; Ma, J.F.; Yang, J.L.; Zhang, W.C.; Sun, Y.P.; Pan, J.Q.; Wang, H.X.; Chen, W.Z. Improving the ductility of Mg-2.5Nd-0.5Zn-0.5Zr alloy by multi-pass hot rolling. *J. Mater. Res. Technol.* **2021**, *14*, 2124–2130. [CrossRef]
22. Zhu, C.; Harrington, T.; Livescu, V.; Gray, G.T.; Vecchio, K.S. Determination of geometrically necessary dislocations in large shear strain localization in aluminum. *Acta Mater.* **2016**, *118*, 383–394. [CrossRef]
23. Wang, P.Y.; Wang, B.Y.; Wang, C.; Wang, J.G.; Wang, H.Y. Design of multicomponent Mg–Al–Zn–Sn–Bi alloys with refined microstructure and enhanced tensile properties. *Mater. Sci. Eng. A* **2020**, *791*, 139696. [CrossRef]
24. Ma, C.Y.; Xia, N.; Wang, C.; Li, M.X.; Hua, Z.M.; Ren, M.W.; Wang, H.Y. A novel Mg-5Al-2Zn-2Sn alloy with high strength-ductility synergy fabricated via simple hot rolling and annealing treatment. *J. Alloys Compd.* **2021**, *869*, 159308. [CrossRef]
25. Ravi Kumar, N.V.; Blandin, J.J.; Desrayaud, C.; Montheillet, F.; Suéry, M. Grain refinement in AZ91 magnesium alloy during thermomechanical processing. *Mater. Sci. Eng. A* **2003**, *359*, 150–157. [CrossRef]
26. Miao, Q.; Zhu, L.T.; Wang, W.K.; Wang, Z.H.; Shao, B.; Chen, W.Z.; Yu, Y.; Zhang, W.C. Evolution of the Microstructure and Mechanical Properties of AZ31 Magnesium Alloy Sheets during Multi-Pass Lowered-Temperature Rolling. *Metals* **2022**, *12*, 1811. [CrossRef]
27. Wu, Z.; Zhi, C.; Ma, L.; Zheng, Z.; Zhu, Y.; Jia, W. Effect of Initial Temperature on the Microstructure and Properties of Cryogenic Rolled Az31 Magnesium Alloy. *Mater. Tehnol.* **2022**, *56*, 571–577. [CrossRef]
28. Wang, T.; Yang, L.; Tang, Z.F.; Wu, L.; Yan, H.Y.; Liu, C.; Ma, Y.Z.; Liu, W.S.; Yu, Z.J. Microstructure, mechanical properties and deformation mechanism of powder metallurgy AZ31 magnesium alloy during rolling. *Mater. Sci. Eng. A* **2022**, *844*, 143042. [CrossRef]
29. Vignesh, P.; Abraham, A.; Kumaran, S. Microstructural correlation of cross rolling temperature of AT55 magnesium alloy in mechanical and formability behaviour. *Micron* **2022**, *160*, 103305. [CrossRef]
30. Ren, X.W.; Huang, Y.C.; Zhang, X.Y.; Li, H.; Zhao, Y.X. Influence of shear deformation during asymmetric rolling on the microstructure, texture, and mechanical properties of the AZ31B magnesium alloy sheet. *Mater. Sci. Eng. A* **2021**, *800*, 140306. [CrossRef]
31. Wang, W.; Chen, W.; Zhang, W.; Cui, G.; Wang, E. Effect of deformation temperature on texture and mechanical properties of ZK60 magnesium alloy sheet rolled by multi-pass lowered-temperature rolling. *Mater. Sci. Eng. A* **2018**, *712*, 608–615. [CrossRef]
32. Liu, D.; Liu, Z.Y.; Wang, E.D. Evolution of twins and texture and its effects on mechanical properties of AZ31 magnesium alloy sheets under different rolling process parameters. *Trans. Nonferrous Met. Soc. China* **2015**, *25*, 3585–3594. [CrossRef]
33. Jiang, Y.; Chen, D.; Jiang, Q. Effect of cryogenic thermocycling treatment on the structure and properties of magnesium alloy AZ91. *Met. Sci. Heat Treat.* **2012**, *53*, 589–591.
34. Wu, C.B.; Kim, S.S.; Park, J.H.; Kim, T.H.; Seo, T.W.; Kim, J.W. Optimization of Design Variables for Minimization of Welding Residual Stress. *J. Korean Soc. Manuf. Technol. Eng.* **2017**, *26*, 565–569.
35. Yadollahpour, M.; Hosseini-Toudeshky, H.; Karimzadeh, F. Effect of Cryorolling and Aging on Fatigue Behavior of Ultrafine-grained Al6061. *JOM* **2016**, *68*, 1446–1455. [CrossRef]
36. Yu, H.; Yan, M.; Li, J.; Godbole, A.; Lu, C.; Tieu, K.; Li, H.; Kong, C. Mechanical properties and microstructure of a Ti-6Al-4V alloy subjected to cold rolling, asymmetric rolling and asymmetric cryorolling. *Mater. Sci. Eng. A* **2018**, *710*, 10–16. [CrossRef]
37. Luo, J.R.; Yan, Y.Q.; Zhang, J.S.; Zhuang, L.Z. Microstructure and mechanical properties of cryorolled AZ31 magnesium alloy sheets with different initial textures. *Int. J. Miner. Metall. Mater.* **2016**, *23*, 827–834. [CrossRef]