UDK 669.721.5:536.483
Original scientific article/Izvirni znanstveni članek

ISSN 1580-2949
MTAEC9, 56(5)571(2022)

EFFECT OF INITIAL TEMPERATURE ON THE MICROSTRUCTURE AND PROPERTIES OF CRYOGENIC ROLLED AZ31 MAGNESIUM ALLOY

VPLIV ZAČETNE TEMPERATURE NA MIKROSTRUKTURO IN LASTNOSTI KRIOGENSKO VALJANE MAGNEZIJEVE ZLITINE VRSTE AZ31

Zhenyu Wu[1], Chenchen Zhi[1,2*], Lifeng Ma[1,2], Zebang Zheng[3], Yanchun Zhu[1,2], Weitao Jia[1,2]

[1]College of Mechanical Engineering, Taiyuan University of Science and Technology, Taiyuan, China
[2]Heavy Machinery Engineering Research Center of the Ministry of Education, Taiyuan University of Science and Technology, Taiyuan, China
[3]State Key Laboratory of Solidification Processing, Shaanxi Key Laboratory of High-Performance Precision Forming Technology and Equipment, School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an, China

Prejem rokopisa – received: 2022-03-08; sprejem za objavo – accepted for publication: 2022-09-05

doi:10.17222/mit.2022.443

By characterizing the properties of a twin cast-rolled AZ31 sheet after cryogenic rolling, the effects of the initial temperature on the microstructure and mechanical properties of the sheet obtained by cryogenic rolling were studied. The results indicate that the hardness of the alloy was a maximum due to the high-density dislocations in the microstructure when the initial temperature was room temperature. The highest yield strength and elongation appeared at the initial temperature of 200 °C, and the microstructure was mainly fine equiaxed grains. It was observed that fine grains increased, and the yield strength and elongation gradually decreased with the increase of the initial temperature. The overall trend of yield strength and elongation increased first and then decreased. Based on the results of different initial temperature treatments, the optimal microstructure and properties of AZ31 magnesium alloy were acquired when the initial temperature was 200 °C.

Keywords : cast-rolled AZ31 sheet; cryogenic rolling; microstructure; EBSD

Avtorji članka so preučili lastnosti pločevine iz magnezijeve zlitine vrste AZ3, ki je bila izvaljana neposredno iz taline na duo valjarniškem ogrodju. Določili so vpliv začetne temperature na mikrostrukturo in mehanske lastnosti izbrane zlitine po njenem nadaljnjem izotermalnem in kriogenskem valjanju. Rezultati študije so pokazali, da ima zlitina maksimalno trdoto zaradi nastanka visoke gostote dislokacij v mikrostrukturi, ko je začetna temperatura pločevine enaka sobni temperaturi (20 °C). Najvišjo natezno trdnost in največji raztezek pa ima zlitina, ko je začetna temperatura 200 °C. in so v mikrostrukturi v glavnem fina enakoosna kristalna zrna. Ugotovili so, da se z rastjo finih zrn in naraščajočo začetno temperaturo postopoma znižujeta meja plastičnosti in raztezek. V splošnem pa se trend meje tečenja in raztezka spreminja. Sprva narašča, nato pada. Na osnovi preizkusov pri različnih začetnih temperaturah ugotavljajo, da ima magnezijeva zlitina vrste AZ31 optimalno mikrostrukturo in najboljše mehanske lastnosti pri izbrani začetni temperaturi 200 °C.

Ključne besede: magnezijeva zlitina vrste AZ31, kriogensko valjanje, mikrostruktura, EBSD

1 INTRODUCTION

Material strength and plasticity are difficult to improve at the same time: an improvement of one property is often accompanied by the other property weakening.[1–3] Many techniques, such as severe plastic deformation (SPD)[4] and advanced thermo-mechanical processes, have been used to obtain alloys with high strength and good ductility at the same time, but cryogenic rolling has been identified as a promising path to produce ultra-fine-grain structures with small strain and continuous products.[5,6]

Cryogenic rolling technology is a kind of rolling technology in which the rolled piece is cooled to liquid-nitrogen temperature for plastic deformation of the material.[7] At present, cryogenic rolling technology has been successfully applied in the deformation processing of aluminum alloy,[8,9] copper alloy,[10,11] and steel,[12] and has a significant effect on improving the strength and toughness of the material. Das et al. obtained an ultra-fine-grain structure of Al 7075 alloy after cryogenic rolling at liquid nitrogen temperature, and the yield strength and impact toughness of the material after grain refinement were increased by 108 % and 60 %, respectively.[13] The mechanical property improvement caused by cryogenic rolling is also suitable for LM6 aluminum alloy.[14] After comparing AA6061 aluminum alloy samples at room and low temperatures, it proved that asymmetric rolling at low temperature could improve the uniform elongation and reduce the texture strength.[15] Shi et al. analyzed the microstructure and mechanical properties of 5052 aluminum alloy rolled at room and low temperatures. They found that the cryogenic rolling process can significantly enhance dislocation and boundary strength to improve the tensile strength and yield

*Corresponding author's e-mail:
zhicc0629@163.com (Chenchen Zhi)

strength of the 5052 alloy effectively. High-density dislocation of cryogenic rolling could further accelerate the speed of dynamic recrystallisation and grain refinement of the alloy.[16] Regarding the application of cryogenic rolling in copper alloy, Yue et al. carried out large plastic deformation on a Cu-Ag alloy at the temperature of liquid nitrogen to explore its texture evolution and recrystallisation behavior.[17] The results showed that the dynamic recovery was inhibited during the cryogenic rolling process, which led to the accumulation of high deformation energy storage in the alloy. Because of the low lamination fault energy of copper alloy, cryogenic rolling is more conducive to the generation of ultra-fine grains and twin crystals, and can simultaneously improve the strength and plasticity of the alloy.[18–21]

Due to the significant improvement of the microstructure and properties of materials brought by the cryogenic rolling process, many researchers began to apply this process to magnesium alloys. Preciado et al. found that through the cryogenic treatment of AZ91 magnesium alloy, the grain size was obviously refined, and the elongation of the material was significantly improved.[22] By carrying out cryogenic multi-pass rolling deformation on the ZK60 magnesium alloy sheet, the alloy underwent dynamic recrystallization (DRX), the grain size was significantly refined, and the microstructure uniformity was improved considerably.[23] Discontinuous dynamic recrystallization and twin dynamic recrystallization were the main mechanisms of microstructure evolution for the Mg-14Li-1Al (La141) alloy processed by large deformation cryogenic rolling. Through these two plastic deformation mechanisms, more submicron dynamic recrystallized grains were produced, which significantly improved the mechanical properties of the alloy.[24] Cryogenic rolling has been extensively used in enhancing the properties of magnesium alloys, while the influencing factors for the cryogenic rolling of magnesium alloys have not formed a complete processing system, which cannot have enough impact on production and life.

In this paper, the AZ31 magnesium alloy intermediates underwent cryogenic rolling and characterization after treatment. The influence of the initial temperature on the microstructure and properties of the magnesium alloy was studied. Compared with the microstructure and properties of magnesium alloy materials at different initial temperatures, the optimal initial temperature parameters to improve the strength and toughness of AZ31 magnesium alloy were obtained.

2 EXPERIMENTAL PART

The experimental work was carried out on twin cast-rolled AZ31 magnesium alloy sheets. The thickness of the plates changed from the original 7 mm to 6.8 mm after grinding and cleaning, and sample sizes were cut into dimensions of (60 × 45 × 6.8) mm for the subsequent rolling. After heating in preservation at 400 °C for 2 h, the samples were isothermally rolled by a double-roll mill with a roller diameter of 380 mm, and the temperature of the rollers was controlled at (70 ± 5) °C. The plates were rolled from 6.8 mm to 1.9 mm after five passes.

The intermediate plates were air-cooled to room temperature or placed in a furnace for supplementary temperature for 15 min to a different initial temperature required by the experiment. To find the optimal initial temperature of cryogenic rolling, four initial temperatures were designed as room temperature, (200, 250, and 300) °C respectively (marked as AR, A200, A250, and A300 respectively below). Cryogenic rolling was carried out by dipping the samples in liquid nitrogen for 1 minute and rolling for three passes. After each pass, samples were immersed into the liquid-nitrogen tank for cooling. These samples were rolled from 1.9-mm-thick to 1.6-mm-thick after cryogenic rolling. **Figure 1** is the schematic of the cryogenic equipment. The flow chart of this experiment is shown in **Figure 2**.

3 RESULTS AND DISCUSSION

3.1 Influence of initial temperature on microstructure

Figure 3 gives the inverse pole figure (IPF) maps of samples that were held at different initial temperatures. When the initial temperature is room temperature, the microstructure of AR is shown in **Figure 3a**. Most grains are elongated parallel to the rolling direction and a small

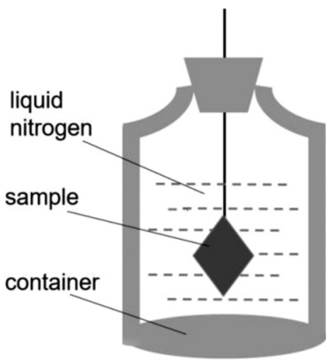

Figure 1: Schematic diagram of liquid nitrogen cooling equipment

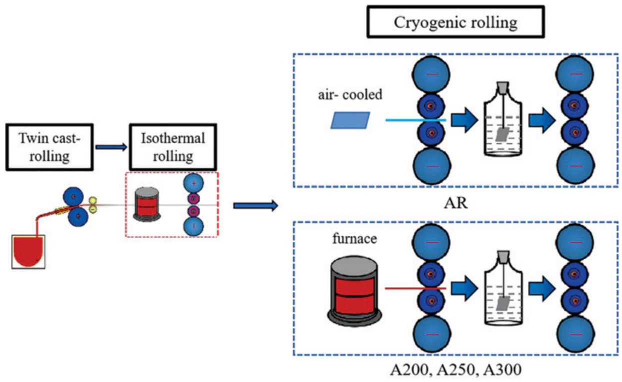

Figure 2: Flow chart of the process

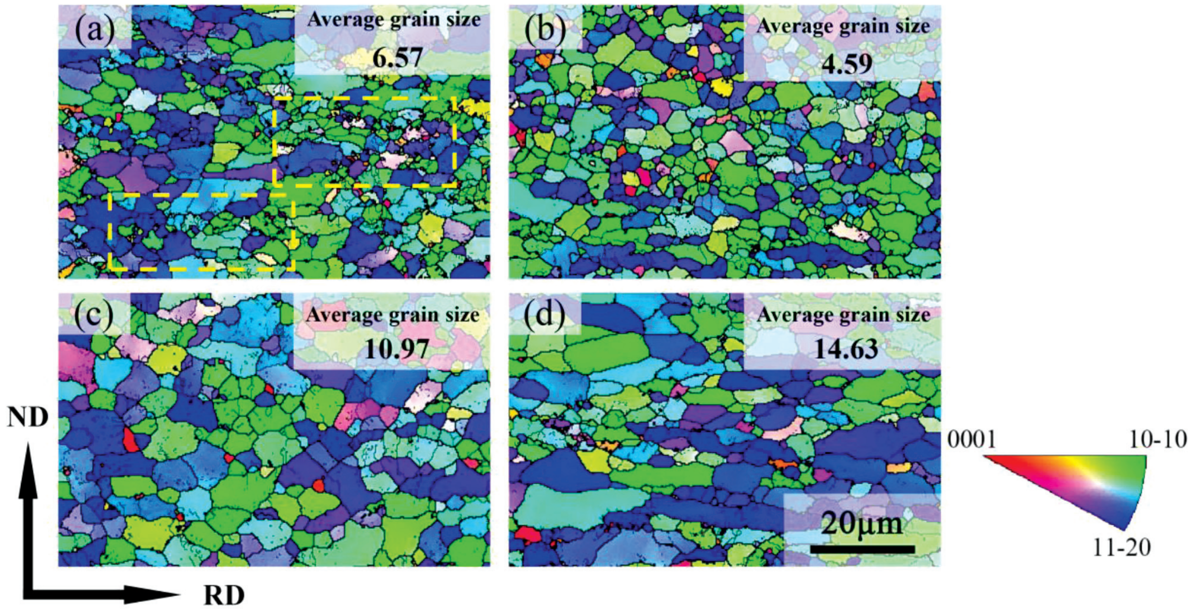

Figure 3: IPF maps of samples at different initial temperatures: a) AR, b) A200, c) A250, d) A300

number of equiaxed submicron grains exist. Fine grains formed because many dislocations accumulated in the intermediate plate after three passes of the isothermal rolling process. The dislocations do not recrystallize or recover at room temperature, so a large number of dislocations are intertwined during the rolling deformation. In addition, the intermediate plates enter the cryogenic treatment in liquid nitrogen after low-temperature rolling, and the plastic deformation at low temperature promotes the formation and retention of high-density dislocations, as shown in **Figure 4**, inhibiting the dynamic recovery effectively.[25] The formation of high-density dislocations will increase the energy near the grain boundary, which not only leads to the formation of new grains[26] but also reduces the average grain size of the alloy.[27] Therefore, fine grains formed near the grain boundary in the AR, and the average grain size is much lower than A250 and A300. A similar conclusion obtains after the cryogenic rolling of industrial pure zirconium plates.[28]

When the initial temperature of the intermediate plate was set at 200 °C, the sample's microstructure shows in **Figure 3b**. The fine grains almost disappeared, the microstructure showed fine equiaxed grains, and the average grain size was 4.59 μm. The reason is that the intermediate plate accumulates many dislocations after un-

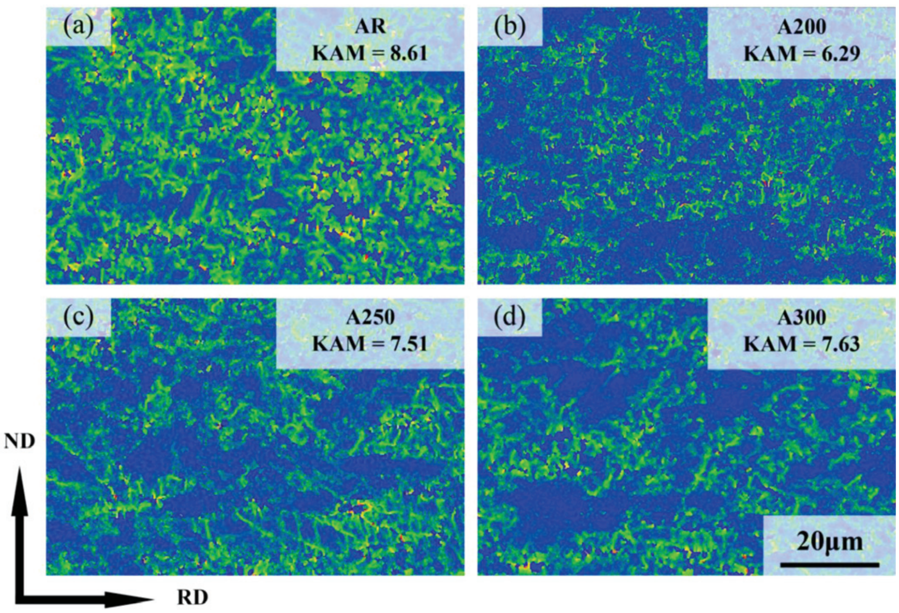

Figure 4: KAM maps of samples at different initial temperatures: a) AR, b) A200, c) A250, d) A300

dergoing multiple isothermal rolling processes. When the initial temperature was 200 °C, recrystallization and recovery occurred, which promoted grain refinement, shortened the displacement range and the dislocation accumulation, and reduced the stress concentration near the grain boundary.[29] Therefore, compared with AR, the KAM of A200 is significantly reduced, and the geometrically necessary dislocation is reduced, suggesting the recovery was activated at this initial temperature.[30] Due to atomic diffusion, dislocations are suppressed, and the microstructure remains recrystallized in subsequent cryogenic rolling. Some grains are elongated along the rolling direction to coordinate the deformation during dynamic recovery.[16]

The microstructures of A200 and A300 are still dominated by equiaxed grains, but the grains elongate along the rolling direction, and the average grain size is 10.97 μm and 14.63 μm, respectively (**Figure 3c** and **3d**). Studies have shown that the increase in annealing temperature significantly reduces the nucleation and growth time of recrystallization.[31] Therefore, the increase of the initial temperature makes the grains of A250 and A300 grow.

3.2 Influence of initial temperature on tensile properties

A universal material-testing machine was used to conduct the tensile tests on the samples obtained from different rolling process parameters at room temperature, and the true stress-true strain curves were obtained as shown in **Figure 5**.

As can be seen from the figure, the alloy has high strength and poor plasticity at the initial temperature of room temperature. This is because a large number of dislocations are accumulated during the isothermal rolling, as shown in **Figure 4**. When the initial temperature is room temperature, the necessary conditions for recrystallization and recovery cannot be satisfied. The decrease in temperature inhibited the dynamic recovery of the alloys, which increased the dislocation density and the degree of work hardening;[32] dislocations accumulate more and intertwine with each other after several passes. Panigrahi et al. studied the precipitation hardening process of an Al-Mg-Si alloy during cryogenic rolling and found that low-temperature conditions are conducive to the formation and retention of high-density dislocations.[33] Therefore, after cryogenic treatment in liquid nitrogen, the alloy retains this microstructure state and causes more dislocations because of the internal lattice of the material.

The specific data and the variation trend of the mechanical properties measured by tensile tests at room temperature are shown in **Figure 6**. The tensile strength of the AZ31 magnesium alloy decreases gradually with the increase of the initial temperature, from 311 MPa to 283 MPa at room temperature. The yield strength and elongation of the alloy have a similar trend, increasing first with an increase of the initial temperature and then gradually decreasing. Compared with AR, A200 and A250 show a typical trade-off relationship, the growth of initial temperature leading to the plastic enhancement and a slight decrease in strength.

With the increase of the initial temperature, the sample microstructures transferred from deformed grains to fine and uniform equiaxed grains. When the initial temperature changes from room temperature to 200 °C, the elongation increases from 8.5 % to 14 %, i.e., by 64.7 %. When the plastic deformation of the materials with tiny and uniform grains occurs, each grain shares a certain amount of deformation so that the distribution of deformation is more uniform. The amount of dislocation packing and the stress concentration at the grain is remarkably excluded, the material has less tendency to crack, and the plasticity of the alloy is improved. According to the Hall-Petch formula shown in Equation (1), the strength was inversely proportional to the square root of its grain size,[34] but the dislocations remain in the cryo-

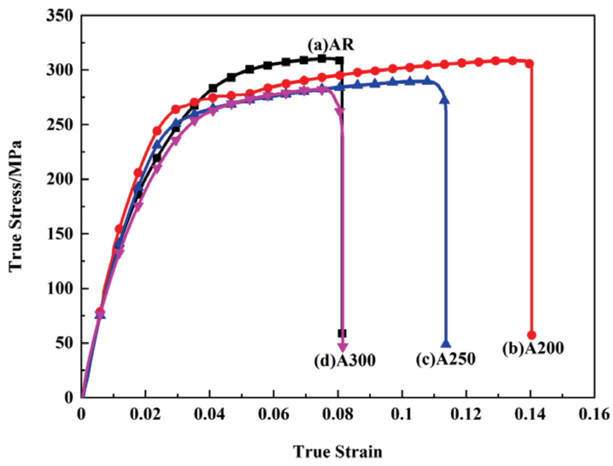

Figure 5: True stress-true strain curves of samples at different initial temperatures: a) AR, b) A200, c) A250, d) A300

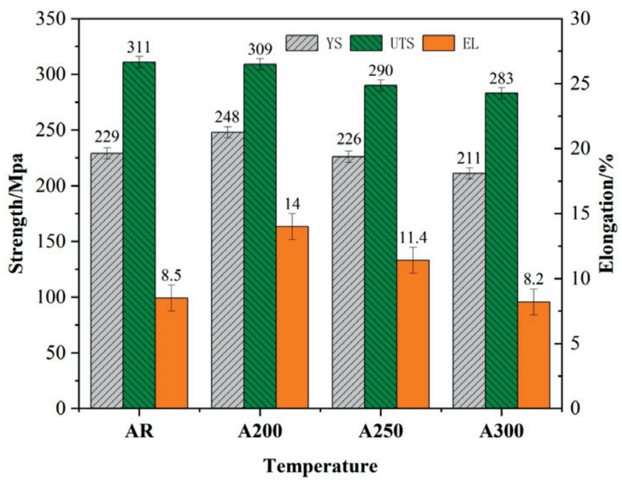

Figure 6: Tensile properties of samples at different initial temperatures

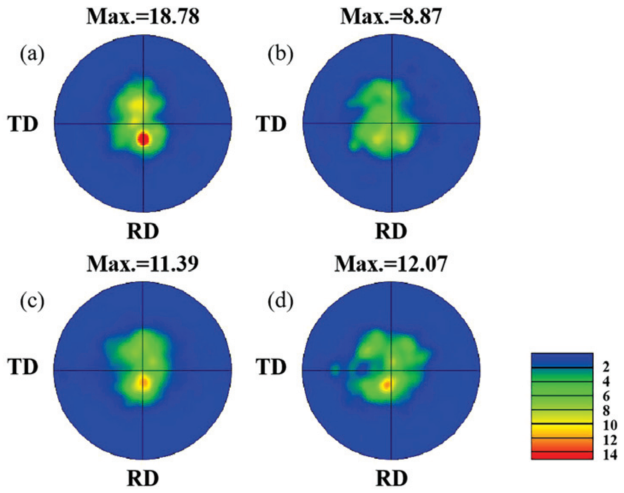

Figure 7: {0001} pole figures of samples at different initial temperatures: a) AR, b) A200, c) A250, d) A300

genic treatment process; thus, the ultimate tensile strength (UTS) is slightly reduced.

$$\sigma_y = \sigma_0 + kd^{-1/2} \quad (1)$$

Figure 7 shows the basal textures of the cryogenic rolled samples. The initial temperature of the intermediate plate is different, but the development tendency of the basal textures is similar. The base poles were named from ND to RD, and the base plane remained parallel to RD. When the initial temperature is 200 °C, the texture strength is 8.87 mrd (multiple random distributed). The texture strength increases with the initial temperature increase to 11.39 mrd and 12.07 mrd, respectively. The prominent {0001} basal texture reduces the ductility of the AZ31 alloy.[35] The stable texture generated at 250 °C and 300 °C reduced the ductility of the AZ31 alloy. In conclusion, the alloy elongation at 200 °C obtained better mechanical properties.

3.3 Influence of initial temperature on the hardness

The hardness of the AZ31 magnesium alloy varies with the initial temperature, as shown in **Figure 8**. When the initial temperature is room temperature, the hardness of the alloy is up to 69 HV. This phenomenon can be attributed to the accumulated deformation of cast-rolled AZ31 magnesium alloy during isothermal rolling. During cryogenic rolling, recrystallization is not activated, and cold hardening and grain slip occur. A lot of dislocation entanglement (as shown in **Figure 4**) and a mass of residual stress are generated inside the alloy. However, with the increase of the initial temperature, the hardness value of the alloy decreases significantly. When the initial temperature is 200 °C, the alloy hardness is 57.5 HV, which is 19.6 % lower than at room temperature. On the one hand, the change of internal stress has a substantial effect on the overall hardness of the sheets; the initial temperature increase releases the internal stress of the plate. On the other hand, the KAM value of the alloy decreases significantly (as shown in **Figure 4**), which means that the geometrically necessary dislocation density decreases, and the plate actively restores in the process of plastic deformation,[36] so the sheet's hardness decreases.

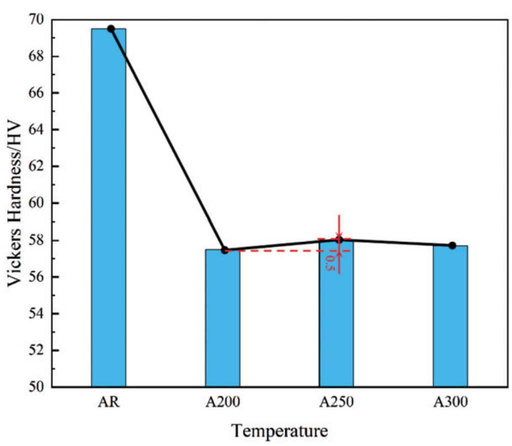

Figure 8: Hardness value of samples at different initial temperatures

The hardness values of the alloy at 250 °C and 300 °C are 58.2 HV and 57.7 HV, respectively. When the initial temperature increases, the hardness value of the alloy does not change remarkably, and the value fluctuates within 1 HV. This is because only the grain size of the magnesium alloy is changed when the alloy is held in preservation for a short time, and no structural change has taken place. Therefore, the average grain size grows with the increase of the initial temperature, but the effect on the hardness of the alloy is not distinct at the macro level.

4 CONCLUSIONS

The initial temperature has a significant effect on the microstructure and mechanical properties of the AZ31 magnesium alloy. When the initial temperature is room temperature, fine submicron grains appear at the grain boundaries due to the high-density dislocation accumulation caused by multi-pass isothermal rolling and cryogenic treatment. When the initial temperature increases, the fine equiaxed grains are the primary structures, and with the increase in temperature, the grains begin to grow and elongate along the rolling direction. Therefore, an appropriate initial temperature of 200 °C can provide a good matrix structure for the subsequent deformation treatment of the material.

The tensile strength of AZ31 magnesium alloy decreases with the initial temperature increase, but the decrease is not evident. The yield strength and elongation first increased and then weakened with the enhancement of the initial temperature. When the initial temperature was 200 °C, the yield strength and elongation reached a maximum. The comprehensive mechanical properties of AZ31 magnesium alloy are the best when the initial temperature is 200 °C after cryogenic rolling.

When the initial temperature is room temperature, the hardness of the AZ31 magnesium alloy is very high and exceeds 60 HV. When the initial temperature increases, the hardness value of the alloy decreases significantly. However, when the temperature continues to rise, the change of grain size has little effect on the macroscopic effect of the alloy, so the hardness range of the alloy is not distinct, not more than 1 HV. Therefore, the initial temperature of 200 °C is the most suitable for the cryogenic rolling of the AZ31 magnesium alloy.

Acknowledgment

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

5 REFERENCES

[1] G. He, J. Eckert, W. Lser, L. Schultz, Novel Ti-base nanostructure-dendrite composite with enhanced plasticity, Nature Materials, 2 (**2003**), 33–37, doi:10.1038/nmat792

[2] Y. H. Zhao, Y. T. Zhu, X. Z. Liao, Z. Horita, T. G. Langdon, Tailoring stacking fualt energy for high ductility and high strength in ultra-fine grained Cu and Its alloy, Applied Physics Letters, 89 (**2006**), 887, doi:10.1063/1.2356310

[3] Y. H. Zhao, X. Z. Liao, S. Cheng, E. Ma, Y. T. Zhu, Simultaneously Increasing the Ductility and Strength of Nanostructured Alloys, Advanced Materials, 18 (**2006**), 2280–2283, doi:10.1002/adma.200600310

[4] M. Kavyani, G .R. Ebrahimi, H. R. Ezatpour, M. Jahazi, Microstructure refinement, mechanical and biocorrosion properties of Mg–Zn–Ca–Mn alloy improved by a new severe plastic deformation process, Journal of Magnesium and Alloys, 10 (**2022**) 6, 1640–1662, doi:10.1016/j.jma.2020.11.013

[5] K. Gopala Krishna, K. Sivaprasad, K. Venkateswarlu, K. C. Hari Kumar, Microstructural evolution and aging behavior of cryorolled Al–4Zn–2Mg alloy, Materials Science & Engineering A, 535 (**2012**), 129–135, doi:10.1016/j.msea.2011.12.052

[6] Q. Du, C. Li , X. Cui, C. Kong, H. Yu, Fabrication of ultrafine-grained AA1060 sheets via accumulative roll bonding with subsequent cryorolling, Transactions of Nonferrous Metals Society of China, 31 (**2021**), 3370–3379, doi:10.1016/S1003-6326(21)65735-7

[7] S. K. Panigrahi, R. Jayaganthan, Development of ultrafine grained Al–Mg–Si alloy with enhanced strength and ductility, Journal of Alloys & Compounds, 470 (**2009**), 285–288, doi:10.1016/j.jallcom.2008.02.028

[8] F. Guo, H. Dong, W. Huang, X. Yang, L. Jiang. Nanocrystalline structure fabricated by cryogenic temperature rolling of AA 2099 aluminum alloy, Journal of Alloy & Compounds, (**2021**), 158293, doi:10.1016/j.jallcom.2020.158293

[9] Q. Y. Yang, Y. L. Zhou, Y. B. Tan, S. Xiang, F. Zhao, Effects of microstructure, texture evolution and strengthening mechanisms on mechanical properties of 3003 aluminum alloy during cryogenic rolling, Journal of Alloy & Compounds, 884 (**2021**), 161135, doi:10.1016/j.jallcom.2021.161135

[10] T. Konkova, S. Mironov, A. Korznikov, G. Korznikova, M. M. Myshlyaev, S. L. Semiatin, Effect of cryogenic temperature and change of strain path on grain refinement during rolling of Cu-30Zn brass, Materials and Design, 86 (**2015**), 913–921, doi:10.1016/j.matdes.2015.06.146

[11] P. Wang, J. Jie, C. Liu, L. Guo, T. Li, An effective method to obtain Cu-35Zn alloy with a good combination of strength and ductility through cryogenic rolling, Materials Science & Engineering, A. Structural Materials: Properties, Microstructure and Processing, 715 (**2018**), 236–242, doi:10.1016/j.msea.2018.01.014

[12] H. Zhan, G. Zhang, H. Zhou, Z. Liu, B. Xu, L. Hao, M. Sun, D. Li, Influence of cooling rate during cryogenic treatment on the hierarchical microstructure and mechanical properties of M54 secondary hardening steel, Materials Science and Engineering: A, 851 (**2022**), 143659, doi:10.1016/j.msea.2022.143659

[13] P. Das, R. Jayaganthan, I. V. Singh, Tensile and impact-toughness behaviour of cryorolled Al 7075 alloy, Materials & Design, 32 (**2011**), 1298–1305, doi:10.1016/j.matdes.2010.09.026

[14] S. Rawat, B. S. Rawat, A. Gupta, M. K. Pathak, P. K. Pant, EBSD studies on cryorolled LM6 Al alloy, Materials Today: Proceedings, 44 (**2021**), doi:10.1016/j.matpr.2020.12.103

[15] D. C. Camilo Magalhães, A. Madeira, K. M. Ferrante, V. L. Sordi, Asymmetric cryorolling of AA6061 Al alloy: Strain distribution, texture and age hardening behavior, Materials Science and Engineering A, 736 (**2018**), 53–60, doi:10.1016/j.msea.2018.08.075

[16] J. T. Shi, L. G. Hou, J. R. Zuo, L. Z. Zhuang, J. S. Zhang, Cryogenic rolling-enhanced mechanical properties and microstructural evolution of 5052 Al-Mg alloy, Materials Science & Engineering A, 701 (**2017**), 274–284, doi:10.1016/j.msea.2017.06.087

[17] L. U. Yue, M. A. Ru, Y. N. Wang, Texture evolution and recrystallization behaviors of Cu-Ag alloys subjected to cryogenic rolling, Transactions of Nonferrous Metals Society of China, 25 (**2015**) 9, 2948–2997, doi:10.1016/S1003-6326(15)63921-8

[18] X. Y. San, X. G. Liang, Lp. Chen, Z. L. Xia, X. K. Zhu, Influence of stacking fault energy on the mechanical properties in cold-rolling Cu and Cu–Ge alloys, Materials Science & Engineering A, 35 (**2012**), 480–483, doi:10.1016/j.matdes.2011.08.032

[19] X. Y. San, X. G. Liang, Lp. Chen, Z. L. Xia, X. K. Zhu, Influence of stacking fault energy on the mechanical properties in cold-rolling Cu and Cu-Ge alloys, Materials Science & Engineering A, 528 (**2011**), 7867–7870, doi:10.1016/j.msea.2011.07.023

[20] X. Y. San, X. G. Liang, L. P. Cheng, S. Li, X. K. Zhu, Effect of stacking fault energy on mechanical properties of ultrafine-grain Cu and Cu-Al alloy processed by cold-rolling, Transactions of Nonferrous Metals Society of China, 22 (**2012**), 819–824, doi:10.1016/S1003-6326(11)61250-8

[21] L. P. Cheng, X. G. Liang, X. Y. San, Y. L. Gong, X. K. Zhu, Effect of Stacking Fault Energy on the Mechanical Properties of Cold-Rolling Cu and Cu-Al-Zn Alloys, Advanced Materials Research, 399–401 (**2012**), 708–712, doi:10.4028/www.scientific.net/AMR.399-401.708

[22] P. Mónica, P. M. Bravo, D. Cárdenas, Deep cryogenic treatment of HPDC AZ91 magnesium alloys prior to aging and its influence on alloy microstructure and mechanical properties, Journal of Materials Processing Technology, 239 (**2017**), 297–302, doi:10.1016/j.jmatprotec.2016.08.029

[23] W. Wang, L. Ma, S. Chai, W. Zhang, W. Chen, Y. Feng, G. Cui, Role of one direction strong texture in stretch formability for ZK60 magnesium alloy sheet, Materials Science and Engineering A, 730 (**2018**), 162–167, doi:10.1016/j.msea.2018.05.113

[24] H. Liu, Y. Wang, B. Li, X. Ma, R. Wu, Effect of cryogenic rolling process on microstructure and mechanical properties of Mg-14Li-1Al alloy – ScienceDirect, Materials Characterization, 157 (**2019**), 109903, doi:10.1016/j.matchar.2019.109903

[25] S. K. Panigrahi, R. Jayaganthan, V. Chawla, Effect of cryorolling on microstructure of Al–Mg–Si alloy, Mater .Lett., 62 (**2008**), 2626–2629, doi:10.1016/j.matlet.2008.01.003

[26] G. E. Dieter, Mechanical metallurgy, McGraw-Hill., 1989

[27] J. R. Newby, ASM Handbook volume 9: Metallography and microstructures, 1985

[28] D. Guo, L. Ming, Y. Shi, Z. Zhang, H. Zhang, X. Liu, B. Wei, X. Zhang, High strength and ductility in multimodal-structured Zr, Ma-

terials & Design, 34 (**2012**), 275–278, doi:10.1016/j.matdes. 2011.08.002

[29] W. L. Sang, S. H. Kim, S. H. Park, Microstructural characteristics of AZ31 alloys rolled at room and cryogenic temperatures and their variation during annealing, Journal of Magnesium and Alloys, 8 (**2020**), doi:10.1016/j.jma.2020.03.003

[30] T. Kong, B. J. Kwak, J. Kim, J. H. Lee, S. H. Park, J. H. Kim, Y. H. Moon, H. S. Yoon, T. Lee, Tailoring strength-ductility balance of caliber-rolled AZ31 Mg alloy through subsequent annealing, Journal of Magnesium and Alloys, 8 (**2020**):163–171, doi:10.1016/j.jma. 2019.11.005

[31] X. Z. Ding, T. M. Liu, J. Chen, Y. Zhang, L. W. Lu, Effect of twin boundary on static recrystallization of AZ31 magnesium alloy, The Chinese Journal of Nonferrous Metals, (2013), doi:CNKI:SUN: ZYXZ.0.2013-01-002

[32] Y. Yi, Cryogenic Deformation Behavior and Microstructural Characteristics of 2195 Alloy, Metals, 11 (**2021**), doi:10.3390/met11091406

[33] S. K. Panigrahi, R. Jayaganthan, V. Chawla, Effect of cryorolling on microstructure of Al–Mg–Si alloy, Materials Letters, 62 (**2008**), 2626–2629, doi:10.1016/j.matlet.2008.01.003

[34] L. Zhang, Q. Wang, W. Liao, W. Guo, B. Ye, W. Li, H. Jiang, W. Ding, Effects of cyclic extrusion and compression on the microstructure and mechanical properties of AZ91D magnesium composites reinforced by SiC nanoparticles, Materials Characterization, 126 (**2017**), 17–27, doi:10.1016/j.matchar.2017.01.008

[35] S. K. Wu, T. S. Chou, J. Y. Wang, The Deformation Textures in an AZ31B Magnesium Alloy, Materials Science Forum, 419–422 (**2003**), 527–532, doi:10.4028/www.scientific.net/MSF.419-422.527

[36] N. Peranio, Y. J. Li, F. Roters, D. Raabe, Microstructure and texture evolution in dual-phase steels: Competition between recovery, recrystallization, and phase transformation, Materials Science & Engineering A, 527 (**2010**), 4161–4168, doi:10.1016/j.msea.2010. 03.028