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ARTICLE

Effect of Cryogenic Treatment Prior to Rolling on Microstructure and Mechanical Properties of AZ31 Magnesium Alloy

Huang Zhiquan[1], Wei Jianchun[2], Huang Qingxue[1], Ma Lifeng[1], Gao Xiangyu[1], Yue Zhaohan[1]

[1] Heavy Machinery Engineering Research Center of the Ministry of Education, Taiyuan University of Science and Technology, Taiyuan 030024 China; [2] Guangxi Liuzhou Iron and Steel Group Company Limited, Liuzhou 545002, China

Abstract: The effects of cryogenic treatment prior to hot rolling on the strength, toughness and ductility of as-cast AZ31 magnesium alloy plates were investigated. The cryogenic treatment conditions were −60 °C/12 h, −120 °C/12 h, −180 °C/12 h, −60 °C/2 h, and −180 °C/2 h. Experimental results show that appropriate cryogenic treatment conditions can significantly refine the grain size and the improve strength, toughness and ductility. A large number of twins appear in both kinds of samples cryogenically treated for 2 h. The sample experienced cryogenic treatment for 12 h at −60 °C followed by rolling process shows the finest and most uniform and increase in yield strength by 25.8%. Meanwhile, a four-time increase in extension rate (3.06% to 12.31%) is attains. The tensile fracture present a mixed-mode fracture, which is characteristic of brittle and ductile fracture mechanisms.

Key words: AZ31 magnesium alloy; cryogenic treatment; second phase particles; fracture morphology

AZ31 magnesium alloy is one of the most widely used metal materials. However, magnesium alloy has some disadvantages, such as lower strength and toughness. Deep processing is easy to cause cracks, which seriously restrict its promotion and application. Therefore, developing new products of magnesium alloy with higher strength and toughness will become the popular trends in the coming period of time. As one of the most important processing techniques to elevate the property of materials in industry field, cryogenic treatment technique was widely used in the production of metal parts and components to further improve the hardness, wear resistance and other special properties[1-3]. A large number of researchers have studied the effect and mechanism of cryogenic treatment on tool steel, die steel and high speed steel [4-6]. Many of them found that deep cryogenic treatment can reduce the number of residual austenite and modify the carbide morphology, which directly result in a significant increase in wear resistance and hardness of steel [7-9].

In recent years, cryogenic treatment has gradually expanded from ferrous metals to nonferrous metals with the development of cryogenic technology [10-12]. Magnesium alloy has some advantages of light weight, good thermal conductivity and excellent damping properties, which make it to be the best substitute for the traditional alloys[13,14]. But some weakness, such as cracking resistance and wear resistance behavior, hinder the large-scale production of magnesium alloy and a wider range of applications. Cryogenic treatment has advantages of being simpler and less costly compared with the optimization of alloy composition, so that researchers have the power to make a large number of experiments on cryogenic treatment of magnesium alloys. Yong Liu et al.[15] studied the influence of cryogenic treatment on the microstructure and

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Corresponding author: Huang Qingxue, Professor, Heavy Machinery Engineering Research Center of the Ministry of Education, Taiyuan University of Science and Technology, Taiyuan 030024, P. R. China, Tel: 0086-351-6010290, E-mail: hqx4350@163.com

properties of 1.5Zn-0.15Gd-Mg magnesium alloy and found that the volume fraction of the precipitated W phase increased with the cryogenic time increasing from 1 min to 24 h. The cryogenic treatment might be effective only when the formation of precipitates in the atomic density matrix was much lower. The wear behavior of AZ91 magnesium alloy during quenching and cryogenic treatment environment was studied by Kamran Amini et al[16]. It is believed that hardness and wear resistance were improved and aluminum atoms jumped to near the defect to provide preferential nucleation sites for $Mg_{17}Al_{12}$ phase. Asl Kaveh Meshinchi et al.[17] proposed that deep cryogenic treatment changed the distribution of the precipitated phases and almost all the tiny particles melt into the microstructure, while coarse divorced eutectic phase was infiltrated into the matrix. The mechanical properties of the alloy have been improved remarkably as the result of this change of microstructure. According to Junwei Liu et al.[18], the appearance of ordered solid solution in AZ91 magnesium alloy after cryogenic treatment made its peak stress be greatly improved.

This paper mainly focuses on the effect of cryogenic treatment prior to rolling on the microstructure evolution of as-cast AZ31 magnesium alloy. At the same time, analysis of the second phase distribution after cryogenic treatment and mechanical properties were tested after rolling. The purpose of this study was to provide an important foundation for the new strategies to produce magnesium alloy sheet.

1 Experiment

The experimental material was as-cast AZ31 magnesium alloy. The alloy's main chemical composition and content used in the experiment is shown in Table 1. Samples of 100 mm in length, 100 mm in width, and 10 mm in thickness were cut from the as-cast alloy and then directly subjected to cryogenic treatment. The cryogenic treatment experimental process was accomplished by placing the specimens in a closed chamber containing the cryogenic medium liquid nitrogen. After cryogenic treatment, the samples were frozen to room temperature in the cryogenic chamber. The experimental scheme is shown in Table 2. After cryogenic treatment, the specimens were then rolled by an experimental two-high laboratory mill (320 mm diameter and 340 mm length), and the rolling speed was 0.2 m/s. All the specimens were rolled to 5.5 mm thickness with the same reduction.

The tensile specimen and metallographic sample of cryogenic treatment and rolling experiment were cut by the electric spark wire-cutting machine. The mechanical properties of the rectangular tensile specimens of rolled AZ31 magnesium alloy were studied by a universal tensile test machine. Given that the deformation of the AZ31 magnesium alloy tensile sample may be relatively small, we adopted an extensometer to measure strain and obtain a more precise stress-strain curve. The precipitate distribution after cold treatment, the micro-structural changes, and the fracture morphology of the tensile

Table 1 Chemical composition of as-cast AZ31 magnesium alloy (wt%)

Al	Zn	Mn	Si	Fe	Cu	Ni	Mg
3.35	0.96	0.4	0.1	0.005	0.05	0.005	Bal.

Table 2 Experimental schemes of deep cryogenic treatment

Sample	Cryogenic temperature/°C	Cryogenic time/h	Cooling rate/°C·min^{-1}
DCT1	−60	12	1
DCT2	−120	12	1
DCT3	−180	12	1
DCT4	−60	2	2
DCT5	−120	2	2

specimens of rolled samples were analyzed by field emission scanning electron microscopy (SEM). The composition and content of the precipitated phase after cold treatment were measured by X-ray energy dispersive spectroscopy (EDS).

2 Results and Discussion

2.1 Precipitation phase characteristics after cryogenic treatment

Fig. 1 shows the microstructures of as-cast AZ31 magnesium alloy after different cryogenic treatments. Before cryogenic treatment, the volume of second-phase is relatively small and it shows a single form, as see in Fig.1a. A large number of second-phase particles precipitate in all the cryogenically treated samples, and the morphology of the particles are significantly altered. After cryogenic treatment for 12 h at −60 °C, the homogeneous distribution of the precipitated phase in the matrix is obtained and the microstructure of AZ31 magnesium alloy consists of α-Mg matrix and a substantial amount of precipitated second phase. The second phase in the microstructure mainly presents in two forms. The first form is larger in size with massive morphology (eutectic morphology), and the second form involves fine particulate or tiny laminar-shaped matter surrounding the first form (chiefly large precipitates). To characterize these precipitates, energy spectrum analysis (EDS) was carried out. The chemical composition of the precipitated phase obtained is displayed in Table 3. These compounds are confirmed mainly by the decreased solubility of aluminum atoms in the Mg-matrix with the decrease in cryogenic temperature. This behavior induces the Mg-Al or Al-Mn alloy compound to form and precipitate.

In Fig.1b, dispersed second-phase particles are well distributed across the matri x. However, a comparison among Fig.1a~1d reveal that the precipitation phase of the AZ31 magnesium alloy is enriched with the cryogenic temperature decreasing with the same cold treatment time. In this case, the uniformly distributed particulates agglomerate into larger blocks in the microstructure. This occurrence may primarily explain the reduction in tensile properties after cryogenic

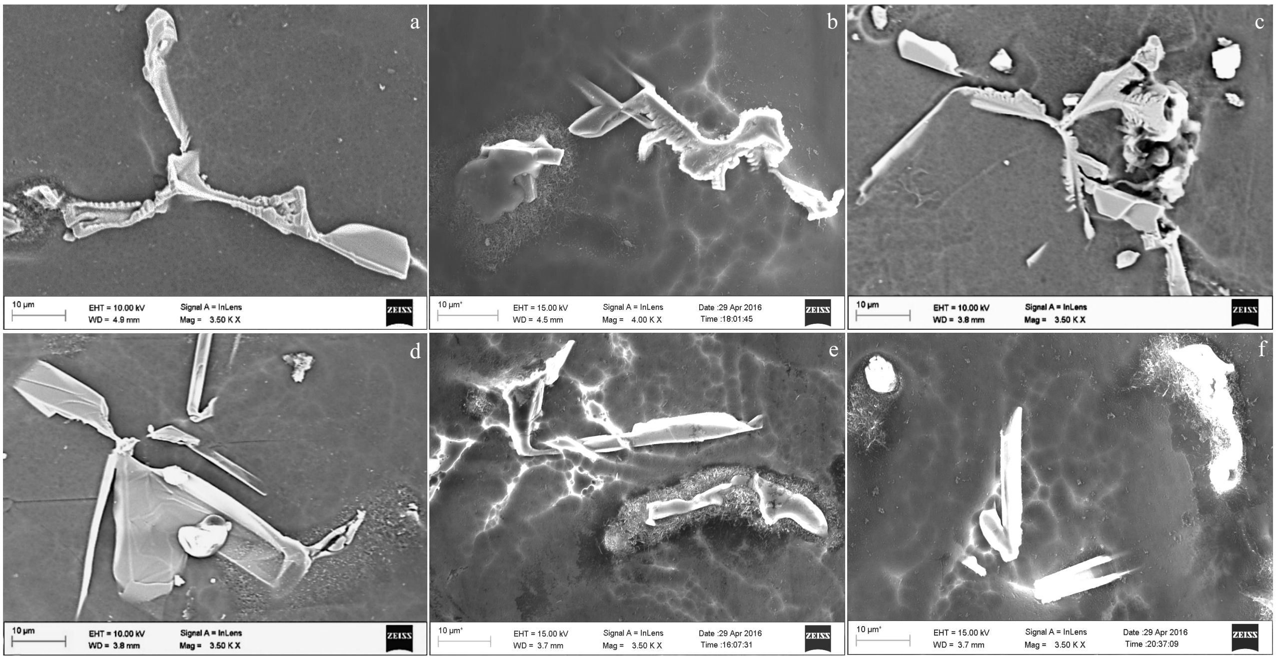

Fig.1 Transverse microstructures of as-cast AZ31 magnesium alloy under different cryogenic treatment conditions: (a) No DCT, (b) DCT1, (c) DCT2, (d) DCT3, (e) DCT4, and (f) DCT5

Table 3 EDS results of the precipitated phase (wt%)

Sample	Mg	Al	Mn	Zn	Si	C	O
No DCT	15.3	25.2	37.5	0.5	0.8	7.9	12.8
DCT1	18.8	30.1	43.1	0.7	0.8	4.4	2.1
DCT2	29.4	26.1	32.2	0.6	0.7	7.8	3.2
DCT3	42.3	22.6	20.2	1.3	0.6	8.7	4.3
DCT4	49.3	24.1	17.1	0.8	0.7	4.8	3.2
DCT5	55.0	17.1	17.6	1.1	0.6	5.5	3.1

treatment, indicating that the lower cryogenic temperature does not necessarily achieve better results. The precipitation phase distributions of samples DCT1, DCT4, DCT3, and DCT5 show that the large precipitated-phase particles are broken into smaller particles with increasing cryogenic time. This pattern is mainly due to the rapid acceleration of the precipitate nucleation and growth velocity with the prolongation of cryogenic time. When AZ31 magnesium alloy is processed from room temperature to cold treatment temperature by liquid nitrogen, additional space is generated by the sudden lattice contraction and provided space for the precipitation of the second phase. Simultaneously, the matrix inevitably produces compressive stress and deformation energy, and the distortion energy is unable to merely supplement the heat loss of the cold treatment process. Instead, the distortion energy is also converted to internal energy, which improves the metastable microstructural state[19]. The compressive stress will lead to a large dislocation during deformation.

Even so, the generation of precipitated phase is conducive to the appearance of dislocations in the plastic deformation process. Thus, the strengthening phase precipitate inevitably along the dislocation line and grain boundaries in the microstructure. The improved mechanical properties after the cryogenic treatment of the AZ31 magnesium alloy can be explained by the microstructural evolution. Such evolution of properties is consistent with the changes in microstructure.

2.2 Microstructural characteristics after rolling

Fig.2 displays the rolled microstructures of as-cast AZ31 magnesium alloy after different cryogenic treatments, in which the white particles are the precipitated second phase. The microstructure is obviously inhomogeneous in the rolling

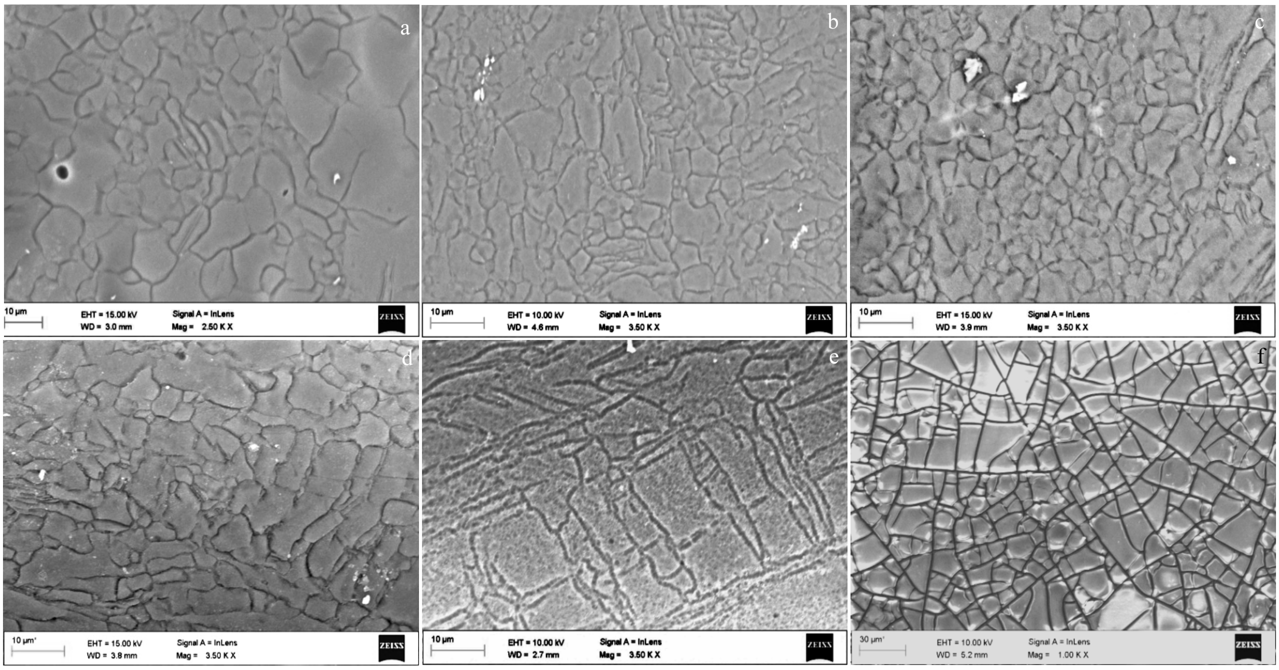

Fig.2 Evaluation of the rolled microstructures of the AZ31 alloy under different cryogenic treatment conditions: (a) No DCT, (b) DCT1, (c) DCT2, (d) DCT3, (e) DCT4, and (f) DCT5

specimens not subjected to cryogenic treatment (Fig.2a). The microstructure displays an inhomogeneous state; the coarse grains are mixed with tiny dynamic recrystallized grains, and the tiny dynamic recrystallization grains are observed only in the local region (Fig.2a). Meanwhile, the microstructures of the samples treated at cryogenic temperatures −60 °C (DCT1) and −120 °C (DCT2) for 12 h are more uniform. Furthermore, equiaxed grains are generated in the local area (Fig. 2b, 2c). When the cryogenic temperature reaches −180 °C (DCT3), twins appear in the microstructure. Meanwhile, fractured twins resemble small linear-chained recrystallized grains. This observation may be attributed to the gradual growth of the fine recrystallization grains with the continuous decline in cryogenic temperature, which reduce the amount of twins. Twin grains are broken into smaller crystalline grains and result in twin-grain separation. For the samples with cryogenic time of 2 h (DCT4 and DCT5) (Fig.2e), numerous twin grains occur in DCT4, but the number of twins and distribution density are significantly greater in those samples than in DCT3. Although the cooling rate of DCT4 reaches 2 °C/min, which is twice higher than that of DCT3, the cryogenic time is only 2 h. Hence, the duration is insufficient to achieve dynamic recrystallization in the microstructure. The twins are not completely broken into fine recrystallized grains. Interestingly, the transition among all neighboring grains is sharp-edged in the microstructure rather than sharp-ellipsed.

Given their hexagonal close-packed crystal structures, magnesium alloys exhibit plastic deformability usually reliant on basal slip systems and the prismatic slip system are difficult to start at room temperature[20]. As a complementary mechanism for coordinated deformation, twinning plays an important role in the plastic deformation of magnesium alloys[21]. The effect of twinning appears to be more prominent for coordinated deformation in slip process; otherwise the slip is excessively delayed to carry out. The twinning effect in the AZ31 magnesium alloy is enhanced by cryogenic treatment followed by the plastic deformation process. The interaction between dislocations and twins or twin and dislocation can promote the nucleation of recrystallization grains. The recrystallization mechanism is known as twinning-induced dynamic recrystallization. This result is due to the internal stress effect generated in the specimen during the cryogenic treatment process. Besides, the phenomenon of stress concentration easily occurs at the deformation zone and enables the generation of sub-grains in the matrix. The interaction of these sub-grains with dislocation greatly enhances the stability of the structure.

2.3 Mechanical property analysis

Fig.3 shows the stress-strain curves of tensile samples under different cryogenic treatment conditions after rolling. The mechanical properties of rolled sample are shown in Table 4. The yield strength, the ultimate tensile strength, and elongation of the sample subjected to cold treatment at −180 °C for 2 h do not substantially change after rolling. Other cryogenic treatment conditions can significantly increase the peak stress of AZ31 magnesium alloy. The YS of sample

DCT1 is increased from 186 MPa to 234 MPa, which increases by 25.8% and the elongation is increased to 4 times from 3.06% to 12.31%. By careful observation, we note that the peak stress and elongation of the AZ31 magnesium alloy become smaller with increase in temperature, whereas the specimens are subjected to cryogenic treatment simultaneously (DCT1, DCT2, and DCT3). This result is mainly due to the decrease in cryogenic temperature, which further causes the more even distribution of the precipitated phase. Moreover, the precipitation of the brittle-phase particles inhibit the dislocation motion and generate dislocation pile up, which results in the smaller elongation of AZ31 magnesium alloy. However, for the samples with the same cryogenic temperature, the peak stress and elongation increase with the growth of cryogenic treatment time (DCT4 and DCT1, DCT5 and DCT3). This aspect was achieved mainly because the rise in deep cryogenic treatment time would produce more dislocations and sub-crystals in the microstructure, which is necessary for improving material strength.

2.4 Fracture morphology analysis

Fig.4 displays the macroscopic fracture morphologies of the tensile specimens of rolled AZ31 magnesium alloy under different cryogenic treatment conditions. The macroscopic fracture morphology of the specimen without cryogenic treatment before rolling is a typical brittle fracture, and

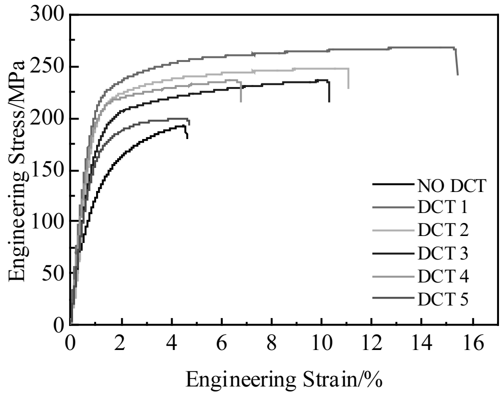

Fig.3 Stress-strain curves of rolled AZ31 magnesium alloy under different DCT conditions

Table 4 Mechanical properties of AZ31 magnesium alloy under different DCT conditions

Sample	DCT1	DCT2	DCT3	DCT4	DCT5	No DCT
Yield strength/MPa	234	229	216	228	196	186
Ultimate tensile strength/MPa	268	248	236	236	199	192
Elongation/%	12.31	7.59	7.06	3.87	2.93	3.06

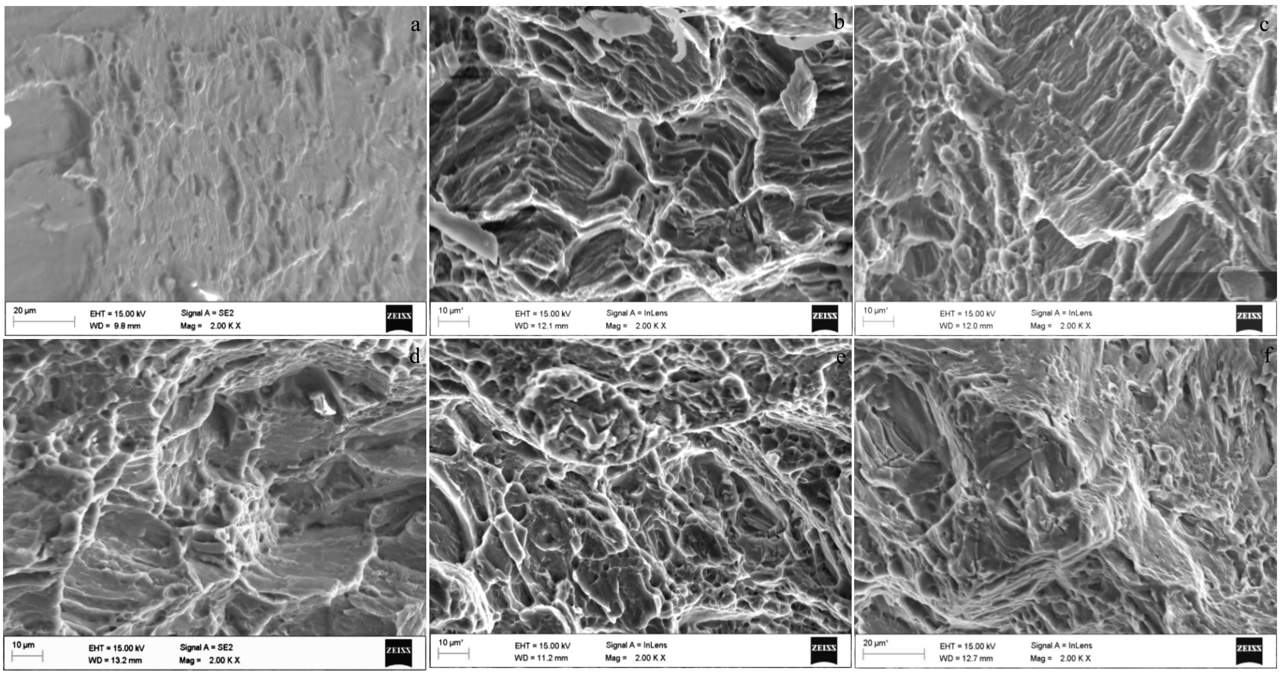

Fig.4 Fracture morphologies of rolled samples under different cryogenic treatment conditions: (a) No DCT, (b) DCT1, (c) DCT2, (d) DCT3, (e) DCT4, and (f) DCT5

the fracture surface is smooth and flat. Compared with the sample No DCT, all the macroscopic fracture morphologies of the samples subjected to deep cryogenic treatment continued to exhibit brittle fracture, and the fracture surfaces shows an obvious predominant cleavage river pattern. However, a large number of dimples and tearing ridges appear in the local area. This result shows that a certain degree of plastic deformation behavior occurs prior to the specimen fracture, and the tensile fracture presents the mixed-mode fracture characteristics of brittle and ductile fracture mechanisms. The above-mentioned results further confirm that cryogenic treatment before the rolling process can improve the elongation and strength of AZ31 magnesium alloy. The second phase, which precipitates along a dislocation line and grain boundary exerts a blocking effect for dislocation motion. The existence of a dislocation pile up in this area will generate larger stress and micro-crack initiation. Hence, ductility decreases, which leads to surface fracture with brittle quasi-cleavage features. A comparison among Fig.4b, 4c, and 4d reveal that the fracture surface is distributed with a large number of dimple and teared edges in three kinds sample at different cryogenic temperatures for 12 h. The sizes of the dimples in the sample under cryogenic temperature 120 °C are relatively fine, whereas the dimple of the sample at cryogenic temperature −60 °C is deeper, and the local area is elongated. When the cryogenic temperature is reduced to −180 °C, the cleavage step becomes smoother, dimple is more shallow. The fracture surfaces of the two specimens with cryogenic time 2 h show a clear tear-edge phenomenon (Fig.4b and 4c). The distribution in the local region of the teared edges in the sample at 180 °C cryogenic temperature is more intensive, and most of the areas remain relatively smooth. The results also verify the tensile properties of the two specimens in 2 h cryogenic time are relatively poor compared with those under 12 h cryogenic time. Given the above analyses, we further verify that the cryogenic treatment process before rolling can significantly augment the plastic properties of the as-cast AZ31 magnesium alloy.

3 Conclusions

1) A large number of second-phase particles are precipitated in the as-cast AZ31 magnesium alloy after cryogenic treatment; the conduction of deep cryogenic treatment on samples changes the distribution of β-phase precipitates. In case of cryogenic temperature for 12 h, the homogeneous distribution of the precipitated phase is destroyed with the decrease in cryogenic temperatures; the precipitates in the matrix phase are diffused into grain boundaries.

2) Suitable cryogenic treatment before the rolling process can greatly improve the tensile strength, YS, and elongation of as-cast AZ31 magnesium alloy sheets. The YS of the sample cryogenically treated at −60 °C for 12 h is increased from 186 MPa to 234 MPa (25.8% rise), and the elongation is augmented by 4 times from 3.06% to 12.31%.

3) To a certain extent, cryogenic treatment before the rolling process can enhance the rolling formability of as-cast AZ31 magnesium alloy. The tensile fracture surface presents an mixed-mode fracture characteristics of brittle and ductile fracture mechanisms.

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轧前深冷处理对 AZ31 镁合金显微组织和力学性能的影响

黄志权[1]，韦建春[2]，黄庆学[1]，马立峰[1]，高翔宇[1]，岳照涵[1]

(1. 太原科技大学 重型机械教育部工程研究中心，山西 太原 030024)

(2. 广西柳州钢铁集团有限公司，广西 柳州 545002)

摘　要：通过对铸态 AZ31 镁合金经不同条件的深冷处理继而热轧，分析其强度、韧性、延展性效应及其规律。深冷实验条件分别为−60 ℃/12 h、−120 ℃/12 h、−180 ℃/12 h、−60 ℃/2 h 和−180 ℃/2 h。结果表明：AZ31 经适宜的深冷处理，晶粒得到细化，强度、韧性及延展性得到有效提高；与未经深冷处理试样相比，深冷处理时间为 2 h 的 2 种试样中都出现大量孪晶；经−60 ℃/12 h 深冷处理后再进行轧制试样的显微组织最为细小均匀，屈服强度提高了 25.8%，延伸率更是提高了 4 倍，由 3.06%提高到了 12.31%；深冷处理后的 AZ31 镁合金断口呈现出脆性断裂和塑性断裂集合的复合性断裂特征。

关键词：AZ31 镁合金；深冷处理；第二相析出；断口形貌

作者简介：黄志权，男，1981 年生，博士，副教授，太原科技大学重型机械教育部工程研究中心，山西 太原 030024，电话: 035-6100290, E-mail: hzq3991@163.com