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ARTICLE

Effect of Warm Rolling on Microstructure and Mechanical Properties of 6061 Aluminum Alloy Cast-Rolled Sheet

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Abstract: The effect of different warm rolling temperatures and cumulative reductions on microstructure and mechanical properties of 6061 aluminum alloy sheet rolled by twin-roll casting was studied. Multi-scale characterization techniques were employed to study the microstructures of cast-rolled and warm-rolled sheets. The mechanical properties such as hardness, strength and elongation were also measured. The results show that the cast-rolled 6061 alloy mainly contains heat-resistant phase $Al_{0.7}Fe_3Si_{0.3}$, $Al_9Fe_{0.84}Mn_{2.16}Si$, and a small number of strengthening phase Mg_2Si. With increasing the rolling passes, the shape of second phases gradually changes from a mesh and a sheet shape to a line shape along the rolling direction, and finally becomes a fine granular shape. After warm rolling, the amount of new precipitates $Al_{0.5}Fe_3Si_{0.5}$ and Mg_2Si increases. Additionally, the hardness linearly increases with increasing the reduction, and the maximum slope (2.421 14) of hardness curves is found at the warm rolling temperature of 370 °C. At this temperature, fine AlFeSi precipitates and Mg_2Si phases are uniformly dispersed in the alloy, the highest hardness of ~842 MPa is obtained for plate, and the tensile strength, yield strength and elongation are 209.34 MPa, 79.09 MPa and 20.11% respectively.

Key words: 6061 aluminum alloy; twin-roll casting; warm rolling; microstructure; mechanical properties

The use of durable light structures in transportation, machinery, construction and other industries can significantly reduce the weight of vehicles, hence reducing fuel consumption and harmful pollution[1]. 6XXX series aluminum alloy possesses the characteristics of light weight, good formability and high specific strength. As a consequence, the application of this sort of alloy sheet broadens in many fields[2].

In the production of aluminum alloy sheet, rolling, as an irreplaceable procedure, is one of the important factors affecting the mechanical properties of the sheet. Currently, there are many studies focusing on the production of high strength Al-Mg-Si alloy sheets by cryo-roll (CR) [3-5] or high-ratio differential speed rolling (HRDSR) [6] techniques. Huang et al [7] found ultra-fine grain structure in the cold-rolled AA6061 aluminum alloy, which accelerates the dispersion of fine second phase particles in the aluminum matrix, and the strength is then improved using aging treatment. Kim et al [6] prepared ultra-fine grained Al-Mg-Si alloy sheet using HRDSR, and low temperature aging was employed to obtain ultra-high strength following SPD treatment. Twin-roll casting[8] is also one significant technique used to manufacture sheet metal, which allows direct production of sheet metal from melt. Compared to the traditional plate production processes, it has some obvious advantages, such as short production chain, and low energy consumption. Ref.[9-11] introduced the twin-roll casting of Al-Mg-Si alloy (6111, 6061 and 6082) sheet. However, it is found that 6000 series aluminum alloy twin-roll cast-rolled sheets are prone to segregation during solidification with poor mechanical properties. Liu et al [12] reported that there are obvious segre-

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gation zones in the twin-roll cast-rolled 6061 aluminum alloy sheets and the distribution of precipitated phase is considerably uneven. Kim et al [13] reported that a large number of central cracks appear on the plate surface under the conditions of wider crystallization interval and smaller separation force of the initial roll when twin-roll casting the 6022 aluminum alloy. Zuhailawati et al [14] reported that the twin-roll casted Al-Mg-Si alloy microstructure has poor uniformity and lower plate hardness. Some studies [15-18] reported that the microstructure of the Al alloy sheet can be effectively improved by warm rolling, as well as the mechanical properties. Tanaka et al [15] found that the average *r*-value of warm-rolled 7475 aluminum sheets is higher than that of the cold sheets, so the warm-rolled sheets have better deep-drawability in O-temper. Rao[16] found that warm rolling can improve the ultimate tensile strength and elongation of 6061 aluminum alloy after cold rolling. Miki et al [17] reported that asymmetric warm rolling can increase the Lankford value and improve the formability of the Al-Mg-Si aluminum alloy sheets. Kang et al [18] reported that the ultrafine-grain Al 6061 alloy with high strength can be fabricated by combining cryogenic rolling with warm rolling. However, there is few reports about the effect of warm rolling on the microstructure and mechanical properties of cast-rolled 6XXX aluminum alloy sheet. Based on this, the effect of warm rolling process on the microstructure and mechanical properties of 6061 aluminum alloy sheet prepared by twin-roll casting was studied. The purpose of this study is to reduce the segregation tendency, refine the grain size, ameliorate the morphology and distribution of precipitated phases, and significantly improve the strength and plasticity of the sheet.

1 Experiment

In this experiment, a 6061 aluminum alloy with a Mg/Si ratio of 1.404 was used as base material. The chemical composition of the alloy is shown in Table 1. According to Ref.[19], the main strengthening phase of 6061 aluminum alloy is Mg_2Si. When Mg/Si>1.73, excess Mg will reduce the solubility of Mg_2Si phase in solid aluminum. However, excess silicon in the case of Mg/Si<1.73 shows no obvious effect on the solubility of Mg_2Si in solid aluminum. The base material was placed in a SX2-10-12 box-type resistance furnace and heated to 710 °C. The alloy was stirred uniformly after melting, and kept at 710 °C for 10 min. After the temperature dropped to 690 °C, the cast-rolling test was carried out to obtain 6061 aluminum alloy cast-rolled sheet. A *Φ*130 twin-roll mill was employed for warm rolling test of four 6061 cast-rolled sheet samples. The plates were warm-rolled at different temperatures, and samples were taken before each pass for observing microstructure and testing mechanical properties. Five passes of rolling at each warm rolling temperature were performed, and samples were held for 1 h before the first pass and 0.5 h before each subsequent pass. The initial thickness of the sheet was 2 mm, and the final rolling was 0.4 mm. The warm rolling process parameters used in this study are shown in Table 2.

Microstructure of the alloy sample is shown in Fig.1. An Axio Vert.A1 Zeiss optical microscope and a ΣIGMA HD scanning electron microscopy were used to observe the microstructures of the samples. Before observation, the samples were mechanically polished, and then etched using a solution composed of 95 mL distilled water, 2.5 mL HNO_3, 1.5 mL HCl and 1.0 mL HF. The phase composition of the samples was analyzed using EDS. The composition of precipitated phases was determined by XRD (PanAnalytical X'Pert Powder, Netherlands). Tensile tests were conducted on UTM5305 universal testing machine. Hardness test was carried out on Austrian Q10M micro Vickers hardness tester. The average of three samples was obtained.

2 Results and Discussion

2.1 Microstructure of 6061 aluminum alloy cast-rolled sheet

Fig.2 shows the XRD pattern of 6061 aluminum alloy sheet. Five distinct diffraction peaks can be seen from the pattern. Heat-resistant phases $Al_{0.7}Fe_3Si_{0.3}$ and $Al_9Fe_{0.84}Mn_{2.16}Si$ and strengthening phase Mg_2Si are observed in the samples.

Table 1 Composition of 6061 aluminum alloy (wt%)

Mg	Si	Cu	Cr	Fe	Mn	Zn	Ti	Al
0.834	0.594	0.194	0.051	0.182	0.028	0.023	0.016	Bal.

Table 2 Specific parameters of warm rolling process (mm)

Temperatures/°C (thickness/mm)	First time (1 h)	Second times (0.5 h)	Third times (0.5 h)	Fourth times (0.5 h)	Fifth times (0.5 h)
320 (1.96)	1.568	1.274	0.980	0.686	0.392
345 (2.01)	1.608	1.307	1.005	0.704	0.402
370 (2.03)	1.624	1.320	1.015	0.705	0.406
395 (1.95)	1.584	1.287	0.990	0.693	0.396

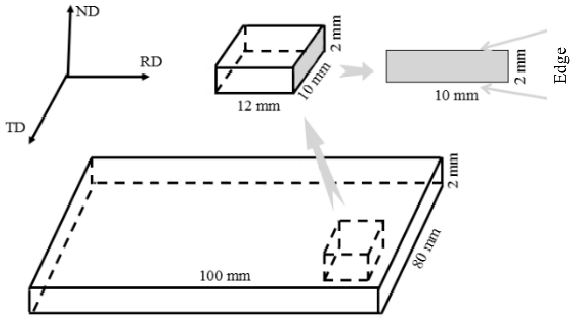

Fig.1 Samples of sheet alloys

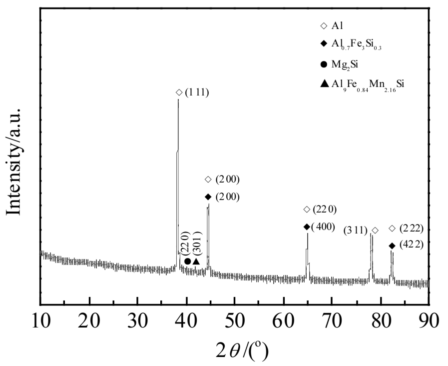

Fig.2 XRD pattern of 6061 aluminum alloy cast-rolled sheet

Fig.3 shows the SEM images of 6061 aluminum alloy cast-rolled sheet, and Fig.4 shows the EDS results of edge of Fig.3. It is clear from EDS and XRD analysis that the precipitates shown in Fig.3 are $Al_{0.7}Fe_3Si_{0.3}$ and Mg_2Si. It can be seen from the XRD pattern that the $Al_{0.7}Fe_3Si_{0.3}$ phases are grid-like and sheet-like precipitates. It can be assumed from Ref.[20] that the $Al_{0.7}Fe_3Si_{0.3}$ phases are β-AlFeSi precipitates. Reticulated β-AlFeSi tends to initiate local cracks during plastic deformation, which reduces the strength and plasticity of the alloy, and also damages the surface quality of the alloy. This is consistent with the results described in Ref. [21]. Mg_2Si precipitates mainly exist as long rods in the cast-rolled sheet. The long rod-like Mg_2Si precipitates cannot strengthen the matrix significantly. However, fine Mg_2Si particles with uniform distribution in the matrix can enormously improve the strength and plasticity of the alloy. During twin-roll casting, the number of precipitated phases in the edge structure of the alloy is larger than in the core due to the rapid decrease in temperature and the high stress at the edge. Fig.3a shows the distribution of the second phase at the edge of 6061 aluminum alloy cast-rolled sheet. A large number of grid-like light gray precipitated phases and a small number of black precipitated phases with lines can be seen in Fig.3.

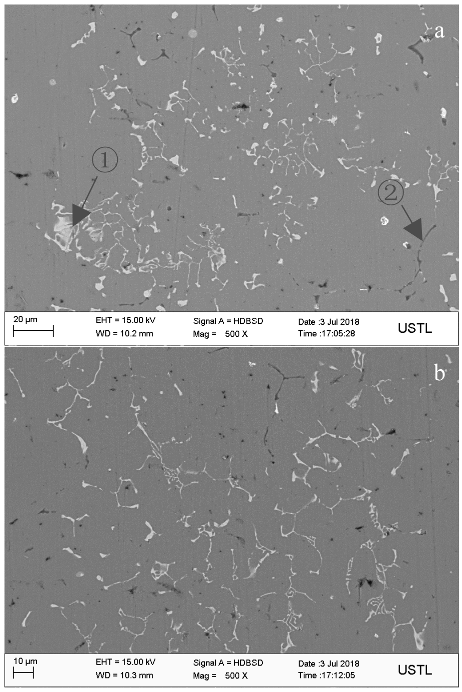

Fig.3 SEM images of second-phase in 6061 aluminum alloy cast-rolled sheet: (a) edge of sheet and (b) core of sheet

2.2 Microstructure of warm-rolled 6061 aluminum alloy cast-rolled sheet

Fig.5 shows the edge microstructures of 6061 aluminum alloy cast-rolled sheet at different warm rolling temperatures and reductions. It can be seen that most grain structures at the edge of the alloy are equiaxed and uniformly distributed, while the shape is nearly spherical. In addition, there are many eutectic phases with low melting point. Simultaneously, casting-rolling structure is obvious at the edge of the alloy. The grain shape begins to change significantly with warm rolling. For instance, elongated grains are obtained at 35% cumulative reduction (Fig.5e~5h) and 50% cumulative reduction (Fig.5i~5l). Strip shape grain distribution is found along the rolling direction. The grain shape changes from sphere to ellipse, while the equiaxed grains almost disappear. When the cumulative reduction is 50%, the as-cast structure is completely transformed into rolling structure. The grains are further flattened and elongated in the rolling direction, except for a small amount of grains broken and others remain unchanged (Fig.5m~5p). When the cumulative reduction reaches 80%, the grains are further flattened, almost completely broken and evenly distributed at the edge of the alloy (Fig.5q~5t).

The microstructure of the edge of the alloy sheet varies with respect to the warm rolling temperature. When the warm rolling temperature is 320 °C, the morphology of grains changes from approximately circular to final fibrous shape. Most grains are compact with the exception of distances between individual grains. No obvious difference in the grain

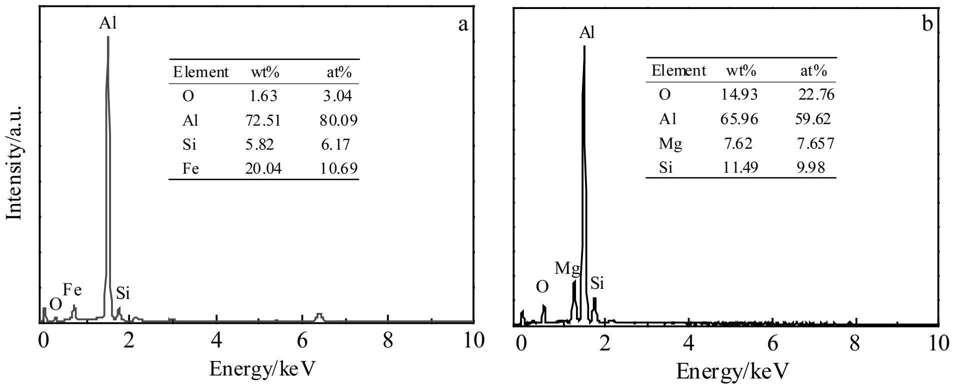

Fig.4 EDS results of the edge ① (a) and ② (b) marked in Fig.3a for 6061 aluminum alloy cast-rolled sheet

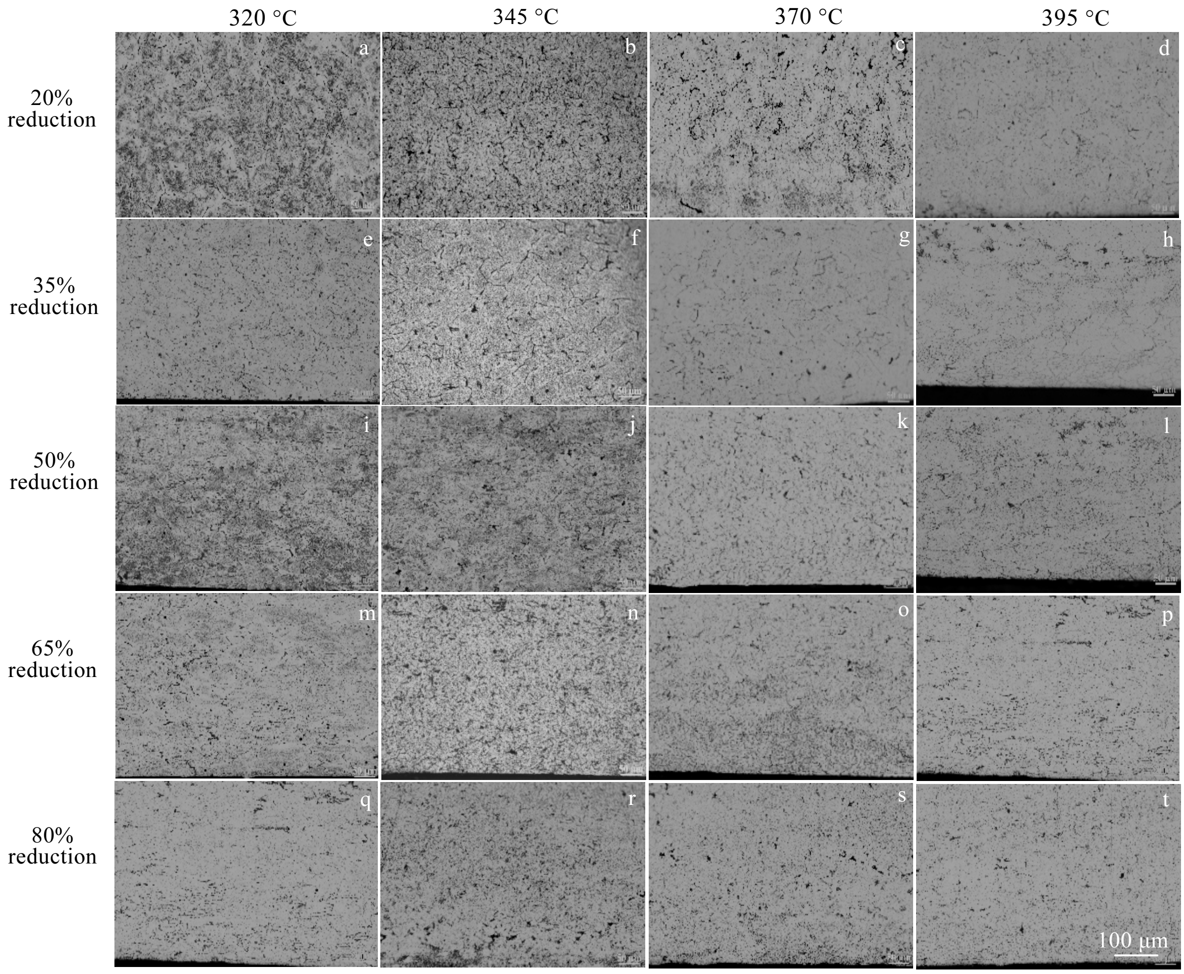

Fig.5 Edge microstructures of 6061 aluminum alloy cast-rolled plate at different warm rolling temperatures

evolution trend is observed between 345 and 320 °C. However, the grain size is noticed to increase at 345 °C. When the warm rolling temperature is 370 °C, the grain size remains uniform. When the cumulative reduction reaches 50%, the grains occlude with each other. With increasing the rolling passes, the grain changes from spherical to elongated shape and finally brakes. When the warm rolling temperature is 390 °C, the grain growth predominantly occurs due to the high temperature. The grain structure change will negatively affect the mechanical properties of the sheet, which is consistent with the results in Ref.[22].

Fig.6 shows the XRD analysis of 6061 aluminum alloy cast-rolled sheet at different warm rolling temperatures (320, 345, 370 and 395 °C) and reductions. The composition and

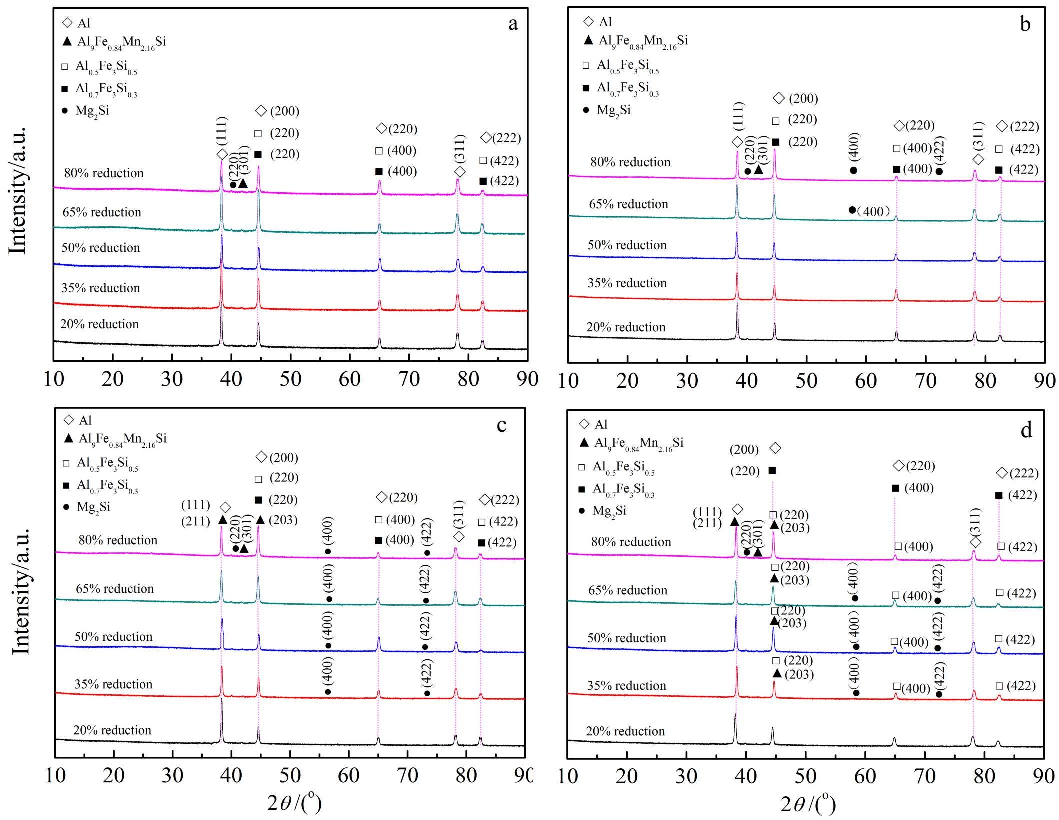

Fig.6 XRD patterns of 6061 aluminum alloy cast-rolled sheet at different warm rolling temperatures: (a) 320 °C, (b) 345 °C, (c) 370 °C, and (d) 395 °C

quantity of phases of the detection area of the alloy after warm rolling increase compared to XRD analysis of cast-rolled sheet. In addition to obvious (α) Al matrix, there are new precipitates $Al_{0.5}Fe_3Si_{0.5}$ in the alloy and the content of Mg_2Si phase increases. $Al_{0.5}Fe_3Si_{0.5}$ phases form in the first pass of warm rolling as rolling force changes the composition and distribution of $Al_{0.7}Fe_3Si_{0.3}$ present in the original cast-rolled sheet. In addition, with increasing the warm rolling passes, the original Mg_2Si precipitates in the upper and lower surface layers are crushed and extruded together, resulting in the increase of Mg_2Si precipitates in the unit layer.

The variation trend of a diffraction peak in Fig.6 indicates that the phase composition of 6061 aluminum alloy cast-rolled sheet is not changed during warm rolling at 320 °C. However, the intensity and half-peak width of the diffraction peak obviously change. With increasing the rolling passes, the height of Mg_2Si diffraction peak at 40.041° increases which is related to the increase of plate deformation caused by the progressive rolling passes.

It is clear from Fig.6a and Fig. 6b that the phase composition of the alloy has no obvious change. When the hot rolling temperature is 345 °C and the cumulative deformation reaches 65%, the diffraction peak of Mg_2Si appears at 57.96°. However, a diffraction peak with low intensity at 72.42° corresponding to Mg_2Si begins to appear at the cumulative deformation of 80%. It can be seen that the diffraction peaks at 57.96° and 72.42° corresponding to Mg_2Si appear when the cumulative deformation of the alloy sheet ranges from 35% to 80% at the rolling temperature of 370 °C (Fig.6c). With increasing the warm rolling temperature, $Al_9Fe_{0.84}Mn_{2.16}Si$ phase appears at 38.38° and 44.61°. With increasing the holding temperature during warm rolling, Mn element replaces Fe element partially in AlFeSi phase due to the rolling force, thus forming $Al_9Fe_{0.84}Mn_{2.16}Si$.

The $Al_{0.5}Fe_3Si_{0.5}$ phase begins to appear at the warm rolling temperature of 395 °C and the cumulative deformation of 35% (Fig.6c and Fig.6d). This can be attributed to high holding temperature of the plate which is close to hot rolling, and a smaller deformation has little effect on it. $Al_9Fe_{0.84}Mn_{2.16}Si$ phase appears at 38.38° at the deformation of 35%. The Mg_2Si phase at 57.96° and 72.42° disappears as Mg_2Si phase is dissolved in the matrix at high holding temperature as the accumulated deformation reaches 80%.

The SEM images of the edge of 6061 aluminum alloy cast-rolled sheet rolled at different temperatures (320, 345, 370 and 395 °C) are shown in Fig.7. At a constant rolling temperature, the amount of Mg_2Si phase increases with increasing the deformation. Furthermore, the shape of precipitated phase at the edge of the alloy gradually changes from dendritic and flaky to linear with increasing the reduction amount. Finally, the linear precipitated phase breaks into fine granule at 80% reduction. The coarse second phase in aluminum alloy seriously affects mechanical properties, while after warm rolling, the coarse second phase breaks into fine particles, and such fine dispersed granular precipitates can greatly improve the hardness and strength of the alloy, which is consistent with the results described in Ref.[23].

Table 3 shows the microstructural characteristics of 6061 cast-rolled sheet with 80% cumulative reduction at different temperatures. The analysis of Table 3 and Fig.7 shows that at a constant reduction, the coarse precipitates in the alloy decrease and gradually change into linear phase when the warm rolling temperature is higher as the second phase more easily breaks due to the rolling force during rolling deformation. When the cumulative reduction reaches 80%, the second phase in the alloy is obviously refined and evenly distributed in the edge matrix of the alloy (Fig.7e~7t). The overall analysis of SEM images at different rolling temperatures indicates that with increasing the temperature, the second phase Mg_2Si obviously increases, and is eventually uniformly distributed in the alloy as the rolling passes increase.

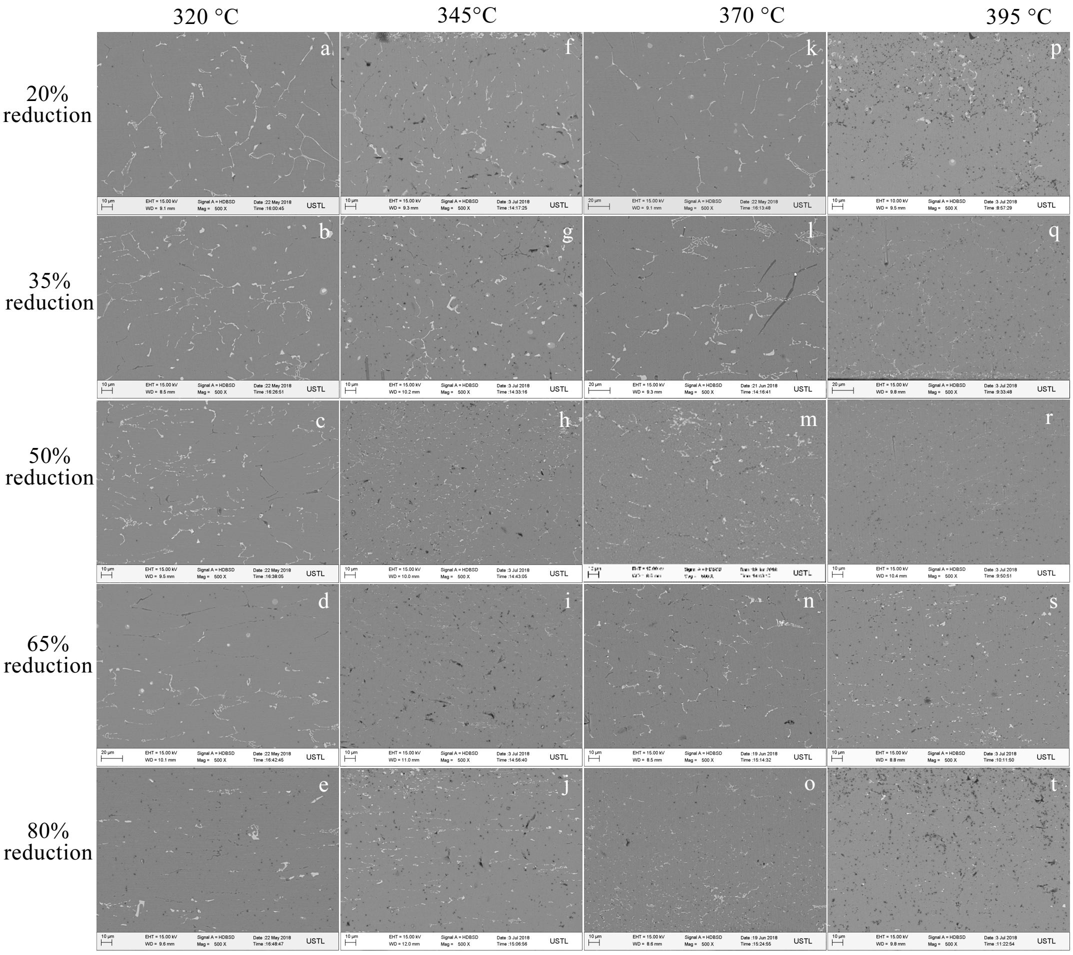

Fig.7 SEM images of edge of 6061 aluminum alloy cast-rolled sheet at different rolling temperatures

Table 3 Microstructural characteristics of cast-rolled 6061 sheet with 80% cumulative reduction at different temperatures

Temperature/°C	Average length of AlFeSi phase/μm	Maximum length of AlFeSi phase/μm	Mg_2Si phase morphology
320	6.3	15.2	Rod-like and granular
345	4.1	8.5	Granular
370	3.4	9.1	Granular
395	2.2	4.7	Granular

2.3 Mechanical properties of warm-rolled 6061 aluminum alloy cast-rolled sheet

Fig.8 shows the hardness of 6061 aluminum alloy cast-rolled sheet at warm rolling temperatures of 320, 345, 370 and 395 °C. The hardness of 6061 aluminum alloy cast-rolled sheet varies linearly (Fig.8). This trend can be described by linear fitting equation. The hardness of the sheet increases linearly with increasing the deformation reduction. As mentioned earlier, a large number of grid-like and sheet-like AlFeSi precipitates and linear Mg_2Si precipitates are formed in 6061 aluminum alloy cast-rolled sheet (Fig.3a). These precipitates will significantly reduce the grain boundary bonding strength, fracture toughness and hardness of the material. With increasing the deformation amount, the precipitated phases at the edge of the alloy gradually break up into fine grains and disperse into the matrix (Fig.7). Such dispersed second phase particles have strong pinning effect on dislocations and sub-grain boundaries, increase the movement resistance of dislocations, and effectively hinder the dislocation movement, thus improving the strength of the alloy.

At the studied warm rolling temperatures, the slopes of the fitted equations are 1.28, 1.71, 2.42 and 1.08. The largest slope of hardness curve is obtained at the rolling temperature of 370 °C, which indicates that the hardness increase is the fastest at this time. At the cumulative deformation of 80%, the deformation energy stored in the grain reaches the maximum. Simultaneously, the external pressure of the alloy becomes the largest, and the hardness of the alloy sheet is ~842 MPa. During warm rolling at 395 °C, a small amount of the second phase, such as Mg_2Si, solidifies in the matrix during heat preservation. At higher temperatures, heat preservation will promote grain growth, resulting in a decrease in grain density per unit area, thus reducing the hardness of the alloy.

Fig.9 show the effect of rolling deformation on the tensile properties of the alloy at different temperatures (320, 345, 370 and 395 °C). It is clear that the rolling deformation has a significant effect on the tensile properties of alloy. With the progress of warm rolling, the yield strength and ultimate tensile strength of the sheet are improved due to fine grains obtained (Fig.5). According to Hall-Petch relationship, the smaller the grain size, the greater the resistance to dislocation and the higher the yield strength of the material. During the plastic deformation of the alloy, dislocation slip and entanglement occur, resulting in a significant decrease in elongation. However, when the amount of deformation reaches 80%, the elongation of the sheet increases suddenly as the grains in the alloy are compactly extruded and the grain density per unit area is higher at the cumulative deformation of 80%, which helps to improve the elongation of the sheet.

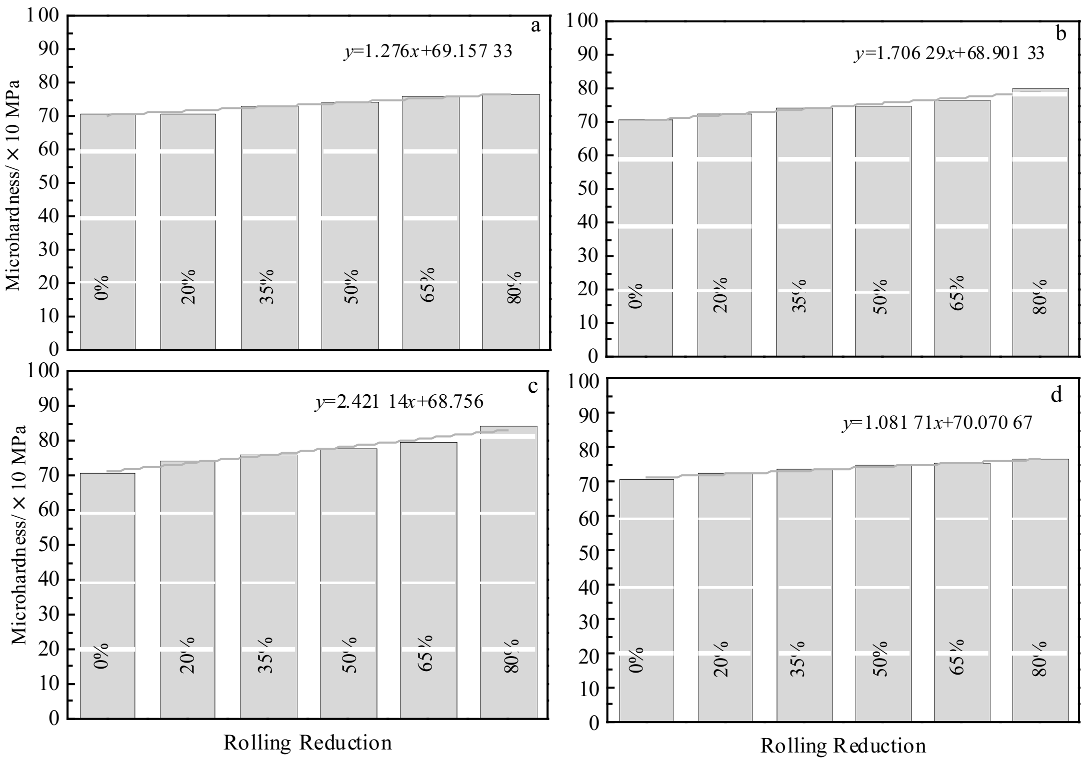

Fig.8 Hardness of 6061 aluminum alloy sheet cast-rolled at different temperatures: (a) 320 °C, (b) 345 °C, (c) 370 °C, and (d) 395 °C

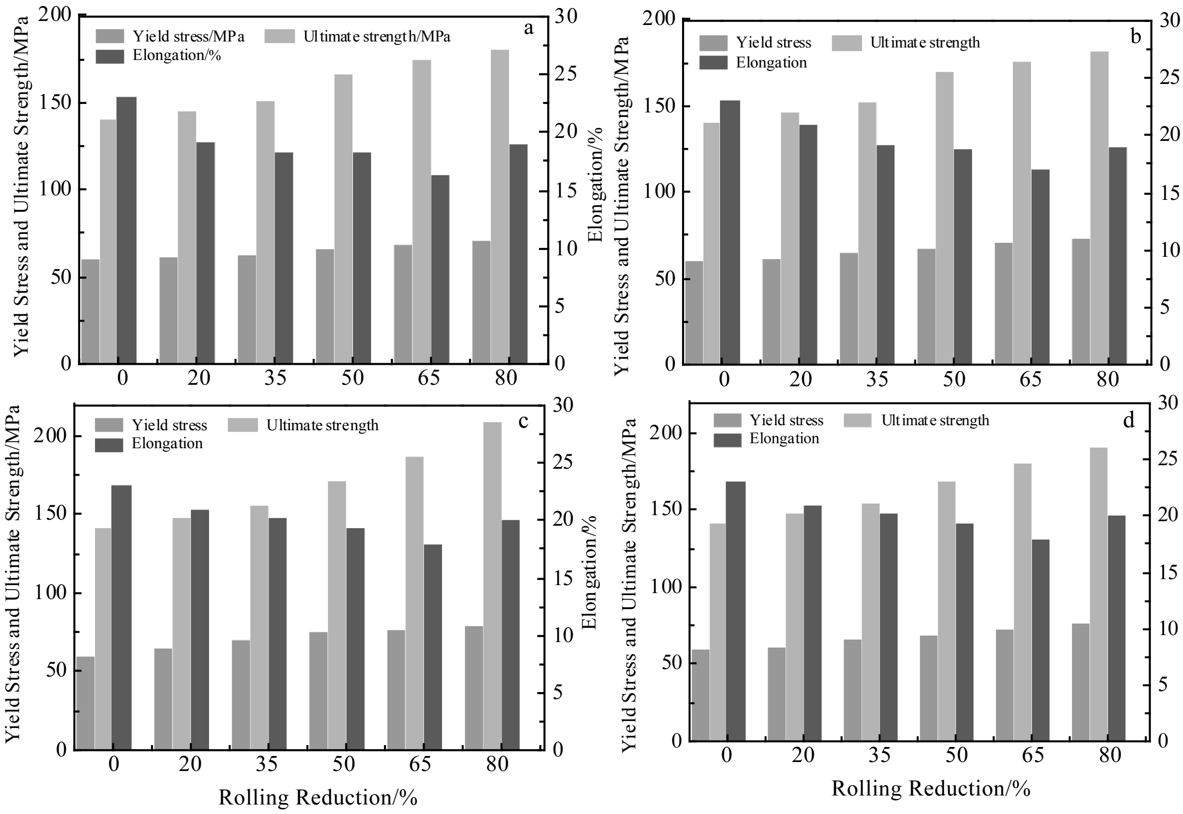

Fig.9 Tensile properties of 6061 aluminum alloy cast-rolled sheet during warm rolling at different temperatures: (a) 320 °C, (b) 345 °C, (c) 370 °C, and (d) 395 °C

Both yield strength and ultimate tensile strength of the alloy increase with increasing the rolling reduction at 320 °C (Fig.9a). The ultimate tensile strength increases from 145.29 MPa to 180.97 MPa, while the elongation decreases from 19.23% to 16.43%, but ultimately reaches 19.01% under 80% cumulative deformation.

Like 320 °C, both the yield strength and ultimate tensile strength of the alloy increase with increasing the deformation at 345 °C (Fig.9b). The ultimate tensile strength increases from 146.30 MPa to 182.00 MPa, and the ultimate elongation reaches 19.78% at 80% cumulative reduction.

The ultimate tensile strength of the alloy increases from 148.07 MPa to 209.34 MPa and the elongation increases to 20.11% with increasing the deformation at 370 °C (Fig.9c).

The ultimate tensile strength and elongation of the sheet reach 190.70 MPa and 19.78% at warm rolling temperature of 390 °C (Fig.9d), respectively.

Fig.10 shows the tensile properties of 6061 aluminum alloy cast-rolled sheet rolled at different temperatures with 80% cumulative reduction. It is clear that the maximum tensile strength (209.34 MPa) and yield strength (79.09 MPa) can be obtained through warm rolling at 370 °C. However, the elongation of the sheet is obtained to be 20.11% under this condition. This is because as the amount of reduction increases, the grains become denser and the work hardening phenomenon increases the strength of the sheet. Moreover, the amount of Mg_2Si in the edge of the alloy reaches the maximum at the rolling temperature of 370 °C, which plays an important role in improving the strength of the sheet. In addition, the appearance of $Al_9Fe_{0.84}Mn_{2.16}Si$ phase reduces the number of AlFeSi phase, which also affects the properties of the sheet.

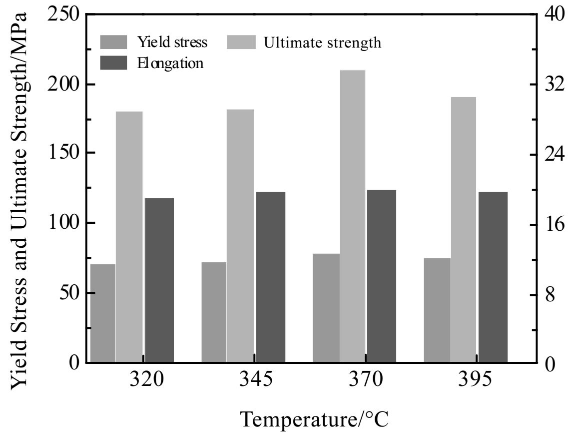

Fig.10 Tensile properties of 6061 aluminum alloy cast-rolled sheet under 80% cumulative reduction at different temperatures

From the above experimental results, it can be concluded that different warm rolling temperatures have different effects on the grain structure and the second phase particles of the alloy sheet. The grain refinement degree and the second phase amount are different at different warm rolling temperatures.

3 Conclusions

1) When the cumulative rolling reduction of the warm rolling reaches 50%, the as-cast microstructure in the original 6061 aluminum alloy cast-rolled sheet is transformed into a rolled structure.

2) $Al_{0.7}Fe_3Si_{0.3}$, $Al_9Fe_{0.84}Mn_{2.16}Si$ and Mg_2Si phases do exist in cast-rolled 6061 alloy. After warm rolling, a new precipitated phase $Al_{0.5}Fe_3Si_{0.5}$ and more Mg_2Si precipitated phase form.

3) The second phase in the alloy is gradually transformed from mesh and sheet shape to line shape and finally fine granular shape along rolling direction with increasing the rolling passes. When the warm rolling temperature is 370 °C, the distribution of the second phase particles is the most uniform.

4) The hardness of 6061 aluminum alloy cast-rolled sheet linearly increases with increasing the reduction amount, and the maximum slope of hardness curve is measured to be 2.421 14 at the warm rolling temperature of 370 °C. Under this condition, fine AlFeSi precipitates and Mg_2Si strengthening phases are uniformly dispersed in the alloy. The highest hardness of the plate is found to be ~842 MPa. The tensile strength, yield strength and elongation are measured to be 209.34 MPa, 79.09 MPa and 20.11% respectively.

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温轧对 6061 铝合金铸轧板材显微组织和力学性能的影响

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摘 要：采用温轧对双辊铸轧 6061 铝合金板材进行处理，观察不同温轧温度及累积压下量对铸轧板材的影响。采用光学显微镜（OM），扫描电镜（SEM），X 射线衍射仪（XRD），显微硬度仪和万能试验机等，表征了铸轧板材及温轧板材的显微组织，获得了材料的硬度、强度和延伸率等力学性能。研究表明，铸轧 6061 合金中主要含有耐热相 $Al_{0.7}Fe_3Si_{0.3}$、$Al_9Fe_{0.84}Mn_{2.16}Si$ 及少量强化相 Mg_2Si。合金中第二相随温轧道次的递增逐渐由网格状、片状转变为沿轧制方向的线条状，最终变为细小的颗粒状。经过温轧后，产生新的析出相 $Al_{0.5}Fe_3Si_{0.5}$ 且 Mg_2Si 析出相增多。铸轧板材温轧后，硬度随压下量的增大呈线性递增，且当温轧温度为 370 ℃时，硬度曲线斜率最大为 2.421 14。此时细小的 AlFeSi 类析出相及 Mg_2Si 强化相均匀弥散分布于合金中，板材的硬度最大，可达 842.8 MPa，抗拉强度、屈服强度和延伸率分别为 209.34 MPa、79.09 MPa 和 20.11%。

关键词：6061铝合金；双辊铸轧；温轧；XRD；力学性能

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