Study on Microhardness and Texture of AlFeMn Alloy Sheets During Cold Rolling

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Abstract: The microhardness and texture of cold rolled AlFeMn alloy sheets were investigated by means of hardness tester, transmission electron microscopy(TEM), and X-ray diffraction(XRD). It was found that the hardness of the rolled AlFeMn alloy sheet increased with the increase of cold rolling reduction, reaching the peak at 82% cold rolling reduction, and then decreasing. Simultaneously, the dislocation density of the rolled sheets decreased obviously, accompanied by the polygonization of sub-structures. It showed that there was a transition from work hardening to work softening during the cold rolling of AlFeMn alloy. At low and medium strains (≤78% cold rolling reduction), the content of copper and S orientation increased slightly with the increase of cold rolling reduction, while the content of Brass orientation remained almost unchanged. When the cold rolling reduction reached 82%, the content of copper, S and Brass increased sharply, and then decreased sharply when the cold rolling reduction exceeded 84%. The content of cube orientation decreased during the work hardening and increased slightly at the end of work softening. The results showed that the occurrence of work softening was accompanied by changes in grain orientation.

Key words: AlFeMn alloy; cold rolling; microhardness; microstructure; texture

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0 Introduction

With higher strength than 1XXX aluminum alloys, as well as good formability and corrosion resistance, AlFe alloys are widely utilized.

Over the years, a large amount of work has been conducted to study the microstructure and properties of AlFe alloys. The effect of precipitation on the evolution of recrystallized texture in AA8011 aluminum alloy sheet was studied by Ryu[1]. The evolution of recrystallization microstructure and mechanical properties during the annealing process of AA8011 alloy was studied[2]. The microstructure and texture changes of AA8011 aluminum alloy during deformation and recrystallization were studied[3].

Some scholars have studied the processing behavior of AlFe alloys. It has been reported[4-5] that different processes affected the processing behavior of AlFe alloy prepared by high-purity aluminum. Refs.[6-7] investigated the processing behavior of AlFe alloys prepared from aluminum ingots with different purity, and found that the purity of aluminum ingots and the content of Fe element in Al-Fe alloys affected the processing behavior.

However, there are few studies on the processing behavior of AlFeMn alloys, especially the microstructure evolution during cold rolling. It is not clear how the hardness and grain orientation of the AlFeMn alloy change during the cold rolling process. In this paper, the microhardness, microstructure and texture orientation of the AlFeMn alloy obtained under different cold rolling reductions were characterized by means of a microhardness tester, transmission electron microscope (TEM) and X-ray diffraction (XRD), so as to provide some insight in-

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to the processing behavior of the AlFeMn alloy, helping to better understand and utilize the processing characteristics of the AlFeMn alloy.

1 Materials and Methods

The ingot of AlFeMn alloy was produced using 99.7% purity aluminum metal and master alloys with certain proportion by direct chill casting, with the chemical compositions given in Table 1.

Table 1 Chemical compositions of AlFeMn alloy (in mass) %

Fe	Si	Mn	Ti	Al
1.40	0.09	0.21	0.015	Bal.

The ingot was homogenized at 590 ℃ for 3 h and at 480 ℃ for 3 h. After homogenizing, the ingot was hot rolled at 480 ℃ from 50 mm to 6.5 mm in multiple passes. Then the hot rolled sheet was cold rolled to a thickness of 1.5 mm in 6 passes. The diameter of the working rolls was 300 mm and the rolling speed was 8 m/min. Next, the cold rolled sheet was heated from room temperature to 420 ℃ held for 4 h, and then cooled in air to room temperature. The annealed sheet was subsequently cold rolled to different thicknesses in multiple passes with different cold rolling reductions ranging from 0 to 98%.

After grinding with 1500# metallographic sandpaper, the conductivity of the sheet before and after annealing was examined at 25 ℃ by a SIGMATEST 2.069 eddy current conductivity meter, and the average value of three test points was taken as the final result. The microhardness of the final rolled sheets with different cold rolling reductions, performed on the rolling direction-normal direction plane of the sheets, was measured by a HVS-1000 Vickers hardness tester after grinding and mechanical polishing. For samples with a thickness of 0.4 mm and above, the test loading force is 0.49 N for 15 s, and for samples with a thickness of less than 0.4 mm, the test loading force is 0.098 N for 15 s. Each microhardness value was determined as the average of six indentations. The microstructures were observed using a Tecnai G2 20 TEM. TEM samples were prepared by double jet polishing with a solution (CH_3OH: $C_3H_5(OH)_3$: $HClO_4$ = 7:2:1) at −35 ℃ and 20 V. The crystallographic textures of the sheets were measured by an Empyrean XRD, and the orientation distribution functions (ODFs) were calculated from the incomplete (111), (200), and (220) pole figures.

2 Results

2.1 Microhardness

Fig.1 shows the change in microhardness of the AlFeMn alloy rolled sheet as a function of cold rolling reduction. During cold rolling, the microhardness of the rolled sheet initially increases and then decreases.

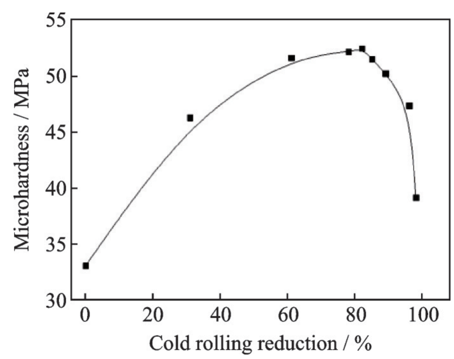


Fig.1 Microhardness change curve of rolled sheets with different cold rolling reductions

In the inter-annealed state (0% cold rolling reduction), the microhardness of the sheet is 33.05 MPa. The microhardness increases significantly with the increase of the cold rolling reduction from 0% to 60%, and reaches the value of 51.5 MPa at about 60% reduction. When the cold rolling reduction exceeds 60%, the microhardness continues to increase slowly until it reaches a peak value of 52.4 MPa at about 82% reduction. Subsequently, the microhardness decreases slightly when the reduction increases to 85%. When the reduction exceeds 90%, the microhardness decreases sharply. At 98% reduction, the microhardness decreases by 25% from its peak value of 52.4 MPa at 82% reduction to 39.1 MPa.

2.2 Texture characteristics

Figs.2—6 show the $\varphi_2 = 0°$, 45° and 65° constant section of ODFs of the rolled sheet with 0%, 31%, 78%, 82%, and 98% reduction, respectively. Both recrystallization textures consisting of cube {100} <001>, R {124} <211>, and deformation textures consisting of brass {110} <112>, copper {112} <111>, S {123} <623> can be found in the sheet with 0% reduction (annealed sheet). For the cold rolled sheets, only the texture components of copper, S and R are shown in the ODFs.

Based on the ODFs results in Figs.2—6, the statistical results of the orientation content of sheets with different cold rolling reductions are calculated, as shown in Fig.7. In fact, the samples with different reductions have the same components, including cube, brass, copper, R, S, and Goss {011} <100>.

It can be found that the contents of cube and Goss orientation first decrease with the increase of the cold rolling reduction (as shown in Fig.7(b)), then the content of cube increases at 98% reduction, while the Goss increases slightly but not obviously when the cold rolling reduction exceeds 78%.

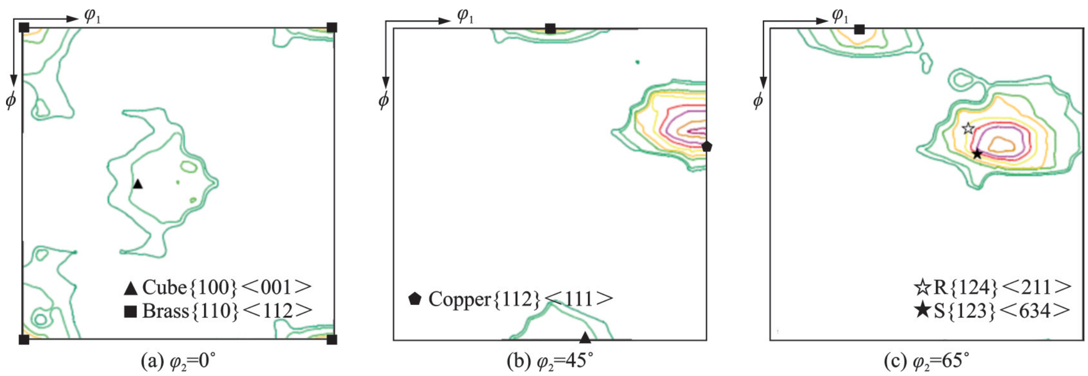


Fig.2 ODFs of the rolled sheet with 0% reduction

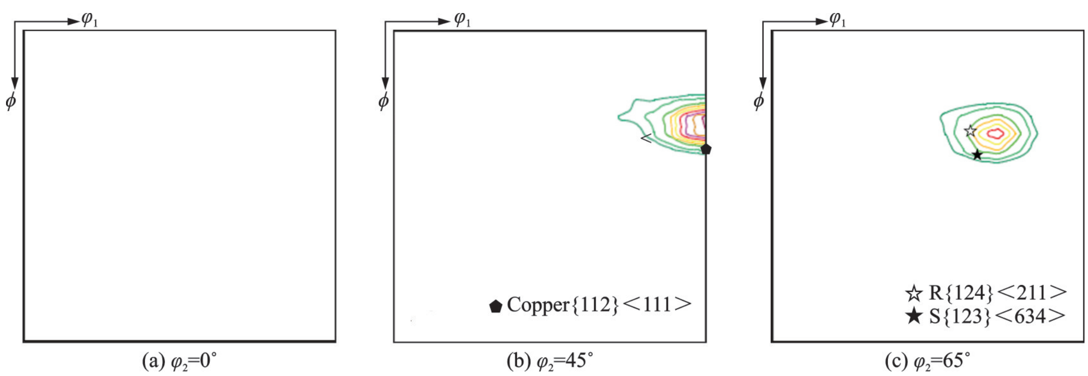


Fig.3 ODFs of the rolled sheet with 31% reduction

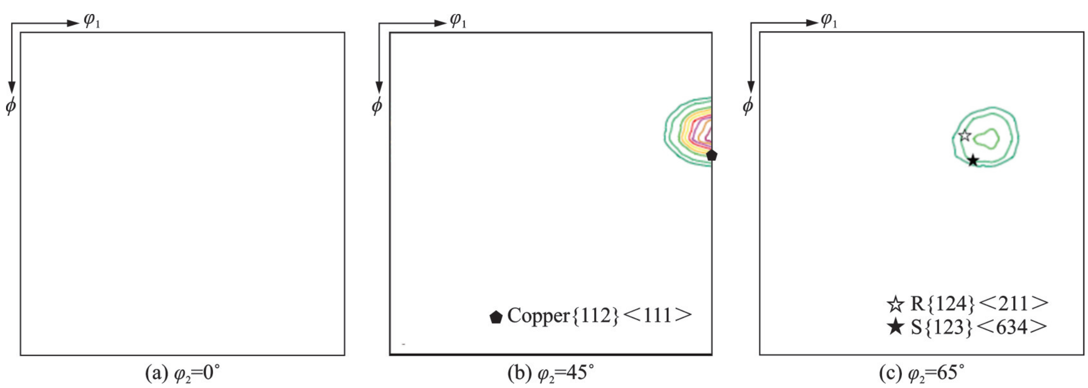


Fig.4 ODFs of the rolled sheet with 78% reduction

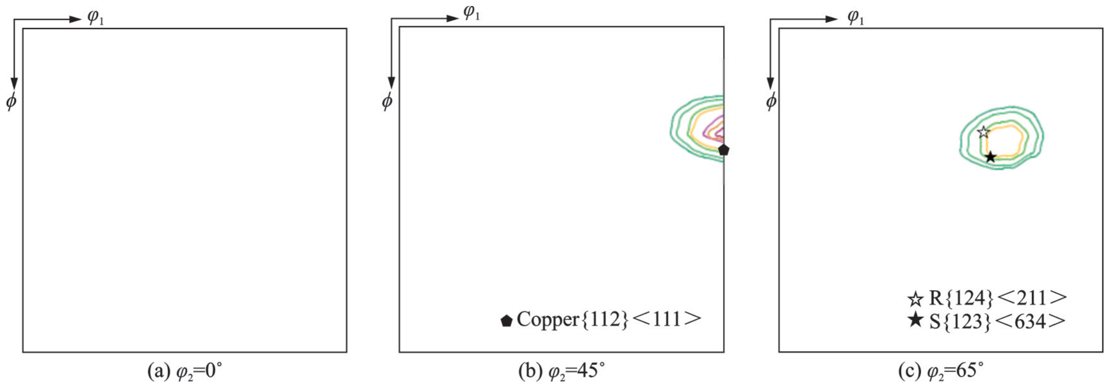


Fig.5 ODFs of the rolled sheet with 82% reduction

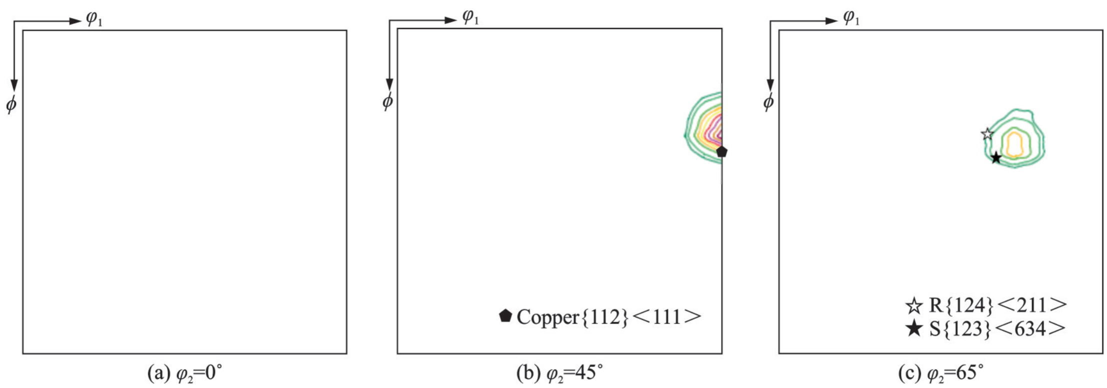


Fig.6 ODFs of the rolled sheet with 98% reduction

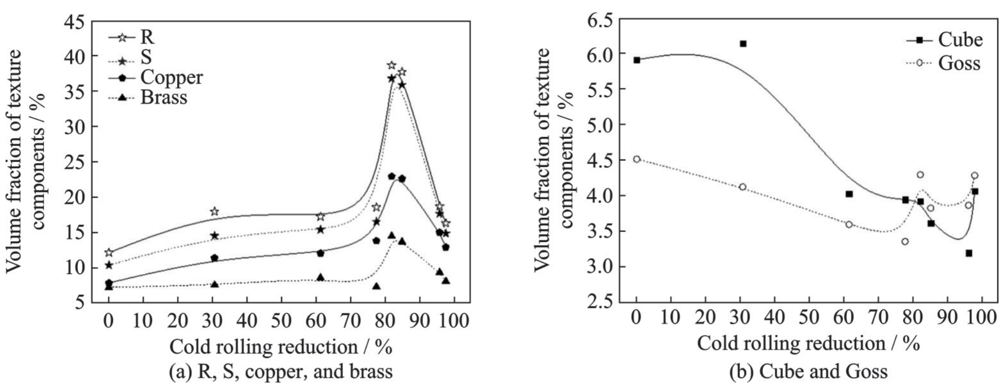


Fig.7 Statistical results of orientation content of sheets with different reductions

During cold rolling, R, S, and copper components increase significantly with the increase of the cold rolling reduction from 0% to 31%, and increase slightly with the reduction increasing from 31% to 78%, while the content of brass component remains almost unchanged during this process. When the cold rolling reduction reaches 82%, the contents of R, S, copper, and Goss quickly reach their peak values, and then decrease rapidly when the reduction exceeds 84%. At 98% reduction, the content of each orientation basically returns to the level of 30% reduction.

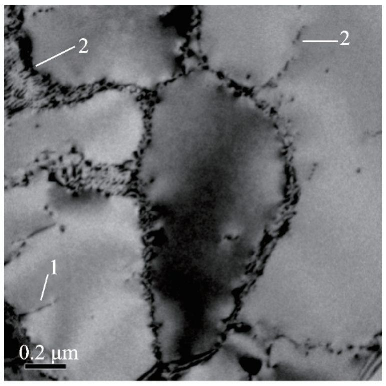
3 Discussion

3.1 Change of microhardness

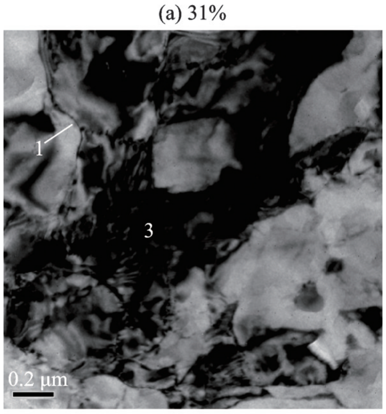
The microhardness results in Fig.1 indicate that the work softening behavior occurs in the studied AlFeMn alloy sheet during cold rolling. The microhardness of the studied AlFeMn alloy sheet first increases with the increase of the cold rolling reduction, and then decreases when the reduction exceeds a certain amount.

The microstructure of the cold rolled AlFeMn alloy sheet subject to a 31% reduction is shown in

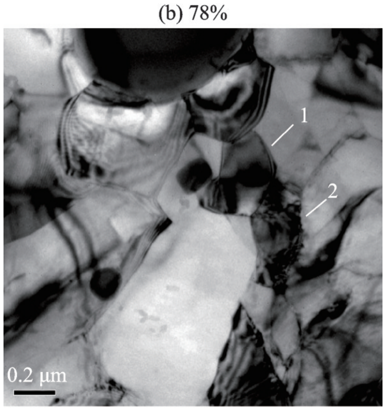
Fig.8(a). It is seen that there are some dislocations accumulating at the grain/subgrain boundaries, which hinders the movement of dislocations and causes an increase in hardness. However, due to the lower dislocation density at the grain/subgrain boundaries, the hardness increases to a lesser degree.



(a) 31%



(b) 78%



(c) 98%

1: Sub-structure, 2: Dislocation, 3: Cluster

Fig.8 TEM images of AlFeMn alloy sheet with different cold rolling reductions

When the reduction increases to 78%, as shown in Fig.8(b), the dislocation density increases significantly, and a large number of dislocations intertwine and develop into black cluster structures, leading to a sharp increase in microhardness.

In contrast with 31% and 78% reduction, a reduction of 98% results in noticeable decrease in dislocation density and degree of dislocation entanglement, as shown in Fig.8(c). It is found that a large number of the sub-structures exhibit a polygonal morphology. This indicates that the alloy has experienced obvious recovery during the cold rolling process at this stage, resulting in a decrease in hardness.

Fig.9 shows that the conductivity of the annealed AlFeMn sheet is obviously higher than that of the rolled sheet. Compared with work hardening, solid solubility of alloying elements has a greater effect on the conductivity of aluminum alloy[8]. It can thus be concluded that obvious precipitation occurs during the annealing process.

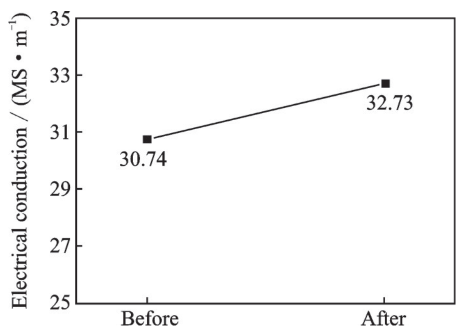


Fig.9 Electrical conductivity of AlFeMn alloy sheet before and after annealing

The precipitation during annealing leads to a reduced solid solubility of the solute atoms in the matrix and thus promotes the work softening of the AlFeMn alloy during the cold rolling process, which is consistent with the previous result[9].

According to Ref.[10], the relationship between the alloy conductivity σ and the solid solubility amount of alloying elements in the aluminum matrix (Unit: $10^{-6}\,\Omega\cdot$m) is $1/\sigma=0.026\,7+0.03\,3\mathrm{Mn_{ss}}+0.006\,8\mathrm{Si_{ss}}+0.032\mathrm{Fe_{ss}}+0.03\mathrm{Ti_{ss}}$, where $\mathrm{Mn_{ss}}$, $\mathrm{Fe_{ss}}$, $\mathrm{Si_{ss}}$, and $\mathrm{Ti_{ss}}$ are the solid solubility of Mn, Fe, Si, and Ti in the aluminum matrix, respectively.

The content of Si and Ti in the studied AlFeMn alloy is low, and their effect on the electrical conductivity is negligible.

Therefore, it can be concluded that the main factor affecting the conductivity of the alloy is the amount of Mn and Fe atoms in the solid solution. It can be calculated that about 0.06% Mn(in mass)+Fe is precipitated from the aluminum matrix after annealing.

3.2 Textures of annealed sheet

After annealing at 420 ℃, recrystallization occurs in the cold rolled AlFeMn alloy sheet, resulting in the formation of recrystallization texture consisting of cube and R orientations, but the content of cube is less (5.9% in volume fraction) than that of R (12.2% in volume fraction), as shown in Fig.2 and Fig.7. The annealed sheet also contains some deformation textures (S, copper, brass) with a total content of 25.5%.

Alloying elements have little influence on the deformation texture, but can strongly affect the recrystallization texture. The element Mn will prevent the development of cube orientation, whether in the form of solute atoms in the solid solution or in the precipitated phases[11]. During the annealing process, the precipitation of fine particles will hinder the nucleation and growth of recrystallized grains with cube orientation[2,12]. On the other hand, the presence of the great number of large-sized intermetallic particles in the AlFeMn alloy sheet (Fig.10) can suppress or weaken the formation of the cube texture due to random nucleation[3]. As the main alloying element of AlFeMn alloy, Fe element in solid solution tends to segregate at grain boundaries in cube-oriented grains, which inhibits the growth of cube orientation[13] and promotes the formation of R orientation in recrystallization texture[14-15]. Therefore, during annealing, the R orientation, which is related to the S orientation to 40° <111> orientation grows preferentially.

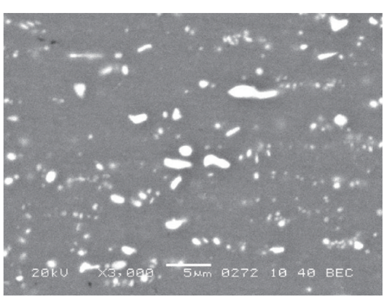


Fig.10 Large-sized intermetallic particles in AlFeMn alloy sheet

During annealing, R-oriented grains can nucleate within S-oriented grains at the grain boundaries between the deformed bands under the mechanism of the strain-induced grain boundary migration, and perform a subsequent growth selection as caused by orientation pinning[15].

Precipitation and recrystallization take place simultaneously during the current isothermal annealing process, and the fine precipitates inhibit the migration of grain boundaries during the recrystallization process, thus allowing part of the deformation textures to be retained[16]. Consequently, the annealed AlFeMn alloy sheet contains a certain amount of deformation textures in addition to recrystallization textures.

3.3 Texture changes during cold rolling

For face-centered cubic (FCC) metal, the common target orientation lines of grain orientation contain α-fiber orientation and β-fiber orientation. The main texture components of α-fiber orientation are Goss and brass orientation, while the main texture components of β-fiber orientation are brass, S, and copper orientation, and the R orientation is also near the β-fiber orientation[13].

The test results of textures (Figs. 2—7) indicate that the textures of cold rolled AlFeMn alloy sheets are dominated by S and copper orientations which increase with the increase of reduction. The content of brass orientation basically remains unchanged at the early stage of cold rolling, while the Goss orientation is weakened with the increase in the cold rolling reduction. This indicates that the grain orientation of the AlFeMn alloy sheet tends to gather along the β-fiber orientation during cold rolling.

Stacking fault energy plays a very important role in the development of deformation texture in FCC materials, and high stacking fault energy is conducive to the formation of more copper and S orientations[3,13]. Therefore, for the present AlFeMn alloy with high stacking fault energy, the content of copper and S orientations in the cold rolling process is significantly higher than that of brass orientation.

During the rolling process, the density of S orientation with a large Taylor factor and high deformation energy storage will be significantly enhanced, resulting in a higher content of S orientation in the studied AlFeMn alloy sheets[17].

The deformation of metals with high stacking fault energy is mainly completed by dislocation glide. When the deformation is carried out only by dislocation glide, the orientation of copper and brass is stable, and the grain orientation continuously rotates towards these two orientations during the rolling deformation[13]. However, the rotation of grains towards the brass orientation will cause large shear strain around the normal line, which is difficult to achieve under rolling geometric conditions. Accordingly, the grain rotation is more difficult to reach the brass orientation, but easier to the copper orientation[16]. Therefore, the content of copper orientation is greater than that of brass, and shows an obvious increase in the cold rolling process.

The contents of S, copper, and brass reach their peak (Fig.7(a)) at 82% reduction which is consistent with the cold rolling reduction at peak hardness (Fig.1). When the reduction exceeds 85%, the contents of S, copper, and brass decrease sharply. In the subsequent cold rolling process after the reduction exceeds 85%, recovery occurs and dislocation density is significantly reduced due to the rearrangement and cancellation of dislocations by cross slip and climb, resulting in the grain orientation deviating from the β-fiber orientation.

During the cold rolling process, the cube orientation is unstable, and the grain orientation deviates from the cube orientation and rotates toward the deformation texture orientation[3], resulting in the decrease of cube orientation with the increase of reduction. After work softening, some of the sub-grain orientations tend to rotate toward the cube orientation, which leads to a slight increase in the cube orientation.

The Goss orientation is mainly nucleated in the shear bands[16]. No obvious shear bands are found in the rolled sheet in the present study, coupled with the influence of particle stimulated nucleation (PSN) promoting the formation of random recrystallization textures, thus the Goss orientation in the annealed sheet is weak with a content of only 4.5%. In the cold deformation process, the orientation density of the textures with small Taylor factor and low deformation energy storage do not change significantly[17]. Therefore, the content of Goss orientation fluctuates very little (3.4%—4.5%) during the whole cold rolling process.

4 Conclusions

The following conclusions can be drawn based on the present investigation:

(1) After annealing at 420 ℃ for 4 h, the microhardness of the AlFeMn alloy sheet first increases and then decreases during the subsequent cold rolling process, and the cold rolling reduction corresponding to the peak hardness point is 82%. The dislocation density keeps increasing in the work hardened sheet. After the work softening, the dislocation density decreases obviously and approximately equiaxed subgrains appear.

(2) In the annealed AlFeMn alloy sheet, the recrystallization texture mainly consists of R orientation (12.2% in volume fraction), a small amount of cube (5.9% in volume fraction), and Goss (4.5% in volume fraction) orientations as well as the deformation texture of S, copper, and brass (10.4%, 7.9%, 7.2%, respectively).

(3) During the cold rolling process, the grain orientation of the AlFeMn alloy tends to gather along the β-fiber orientation, the content of S and copper orientations increases with the increase of cold rolling reduction. In contrast, the content of brass basically remains unchanged, while the content of cube and Goss decreases. After work softening, the deformation texture decreases sharply and cube orientation increases slightly.

(4) In this study, the cold rolling reduction at the inflection point of the change in S, copper, and brass is consistent with that at the inflection point of the change in hardness.

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Author contributions Mr. **PAN Yanfeng** designed the study, conducted the analysis, interpreted the results and wrote the manuscript. Prof. **SHEN Yifu** contributed to the discussion and background of the study. Dr. **YUAN Xini** and Dr. **CAO Lingyong** contributed to the discussion. All authors commented on the manuscript draft and approved the submission.

Competing interests The authors declare no competing interests.

(Production Editor: XU Chengting)

AlFeMn合金冷轧过程中显微硬度与织构研究

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摘要：采用硬度测试仪、透射电镜（Transmission electron microscopy，TEM）、X射线衍射仪（X-ray diffraction，XRD）等手段研究了冷轧过程中AlFeMn合金板材的硬度、微观组织、织构变化。AlFeMn合金的显微硬度先随冷轧率增加而增加，在冷轧率为82%时，硬度达到峰值；然后硬度随冷轧率增加而下降。与此同时，合金中的位错密度明显降低，出现多边化亚晶。结果表明，冷轧过程中，AlFeMn合金存在由加工硬化转变为加工软化的行为。加工软化在中低应变量（≤78%冷轧率）时，copper、S取向含量随冷轧率增加略有增加，而brass取向含量基本保持不变；在达到82%冷轧率时，copper、S、brass取向含量明显急剧增加，在冷轧率超过84%后含量又急剧降低。Cube取向含量在加工硬化期间一直降低，在加工软化后期含量略有增加。结果表明，加工软化的发生伴随有晶粒取向的变化。

关键词：AlFeMn合金；冷轧；显微硬度；微观组织；织构