

Article

The Microstructure and Deformation Behavior of Al-Fe-Mn Alloys with Different Fe Contents during Cold Rolling

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Abstract: The microstructure transformations and deformation behavior of Al-Fe-Mn alloys with different Fe contents during their cold rolling process were investigated by means of hardness testing, conductivity testing, and transmission electron microscopy. It was observed that the hardness of the two alloys increased initially along with the levels of cold rolling reduction, then reduced when levels of cold rolling reduction increased further. Two kinds of deformation behaviors, work hardening and work softening, were observed during cold rolling for both Al-Fe-Mn alloys with different Fe contents. The critical level of cold rolling reduction that led to the change from work hardening to work softening was different in both alloys and the critical level of cold rolling reduction of the alloy with high Fe content was significantly lower than that of the alloy with low Fe content. During the work hardening process, the number of dislocations in the alloys increased continuously as the level of cold rolling reduction increased and they were accompanied by the formation of substructures. After the occurrence of work softening, the dislocation density in the alloys was significantly reduced. The sub-grain structures polygonized and ultimately transformed into equiaxed sub-grains.

Keywords: Al-Fe-Mn alloy; Fe content; cold rolling; work softening

1. Introduction

Al-Fe alloys have excellent mechanical properties, corrosion resistance, and formability. Furthermore, with addition of Mn, the strength of the Al-Fe alloys can be further enhanced without adverse effects on other properties. Therefore, Al-Fe and Al-Fe-Mn alloys have been widely used in packaging, air-conditioning, vehicles, and architectural fields [1–7].

In several previous studies [1,8–13], it was reported that Al-Fe alloys had the characteristic of work softening, which was influenced by the purity of their aluminum matrix, iron content, and processing method.

Work softening of the Al-0.1/0.2/0.5/1.0/2.5Fe alloys prepared from 99.99% purity aluminum metal occurred during their cold rolling process. However, no softening of the Al-1.0Fe alloy occurred during its cold rolling process after solution treatment [8].

It was observed that work softening occurred with the Al-1.9Fe alloy prepared from 99.997% purity aluminum metal when it was heat treated to produce precipitates or hot rolled to produce dispersions of intermetallic compounds. It only showed work hardening without treatment [9].

Reference [10] showed that the phenomenon of work softening only occurred during the cold rolling process of the Al-2.0Fe alloy prepared from 99.96% purity aluminum metal, while it did not occur for the Al-0.2/0.7/1.2Fe alloys.

It was also observed that work softening occurred during the cold rolling process of the Al-2.0Fe alloys prepared from 99.96% and 99.996% purity aluminum metal, but that no work softening occurred for the Al-2Fe alloy prepared from 99.6% purity aluminum metal [11,12].

In the present study, work softening occurred during the cold rolling of the Al-1.4Fe-0.2Mn alloy prepared from 99.7% purity aluminum metal [13]. Such a phenomenon has also been observed with the Al-0.9Fe-0.2Mn alloy prepared with 99.7% aluminum metal during cold rolling. However, the critical level of cold rolling reduction which caused work hardening to change to work softening was different for the two Al-Fe-Mn alloys. This paper investigates the deformation behavior and microstructure transformations of the two Al-Fe-Mn alloys with different Fe contents during the cold rolling process.

2. Materials and Methods

The chemical composition of the alloys used in the present study is given in Table 1. The ingots of Al-Fe-Mn alloys were prepared by direct chill casting using 99.7% purity aluminum metal, Al-20Fe, Al-10Mn, and Al-20Si master alloys in specific proportions. They were hot rolled after homogenization. The 6-mm gauge hot sheets were cold rolled to a thickness of 2 mm and annealed at a temperature of 360 °C for 1 h. They were subsequently cold rolled into final sheets and foils with different cold rolling reductions (0~96%).

Table 1. Chemical composition of the alloys used in the present study (mass%).

Alloy	Fe	Si	Mn	Ti	Al
N1	0.9	0.09	0.24	0.016	Bal.
N2	1.4	0.09	0.21	0.015	Bal.

The change in microhardness of these rolled sheets and foils was measured with an HVS-1000 Vickers hardness tester (Shanghai Wanheng Precision Instruments Co., Ltd, Shanghai, China). Each microhardness value was determined as the average of six indentations. The electrical conductivity of the sheets was measured with a SIGMATEST 2.069 tester (Foerster Instruments Incorporated, Pittsburgh, USA) and the sample was tested at three points on both sides. The electrical resistivity of the foils was measured with a QJ36 single and double bridge (Shanghai Precision Instrument Co., Ltd, Shanghai, China). The foil samples were cut along the rolling direction to a length of 250 mm and a width of 25 mm and three samples were tested for each result. Their electrical conductivity and resistivity values were then converted into the value of their specific conductivity. The microstructures were observed using a Tecnai G2 20 transmission electron microscope (TEM) (FEI Company, Hillsboro, USA).

3. Results

3.1. Microhardness and Specific Conductivity of Alloys

Figure 1 shows the change of microhardness throughout cold rolling reductions of two Al-Fe-Mn alloys.

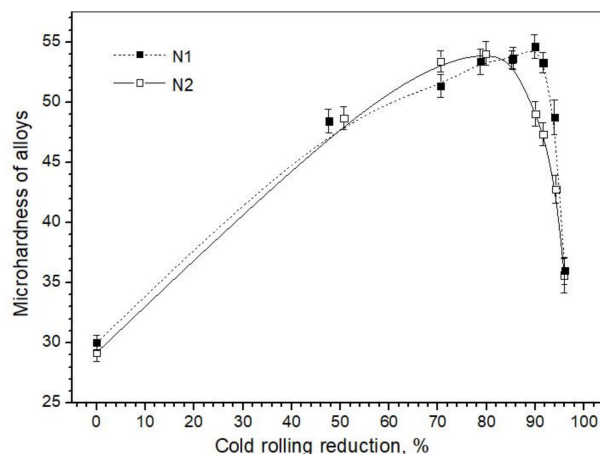


Figure 1. Change in microhardness.

At first, the microhardness of both alloys significantly increases as the level of cold rolling reduction increases. It then reaches a peak. As the level of cold rolling reduction increases further, the microhardness decreases drastically. The microhardness of alloy N1 reaches its peak at a level of cold rolling reduction of about 90%. The corresponding level for alloy N2 is about 80%.

Before the microhardness peak is reached, the increase in the alloys' microhardness as the level of cold rolling reduction increases is known as work hardening. When the level of cold rolling reduction exceeds this peak, or critical point, the decrease of the alloys' microhardness as the level of cold rolling reduction increases further is the behavior of work softening.

Figure 2 shows the change in specific conductivity throughout the cold rolling reduction of two Al-Fe-Mn alloys. Both alloys exhibit similar variations. The conductivity of alloy N1 is lower than that of alloy N2 throughout each cold rolling reduction. At a level of cold rolling reduction of 0%, the conductivity of alloy N1 is especially lower than that of alloy N2.

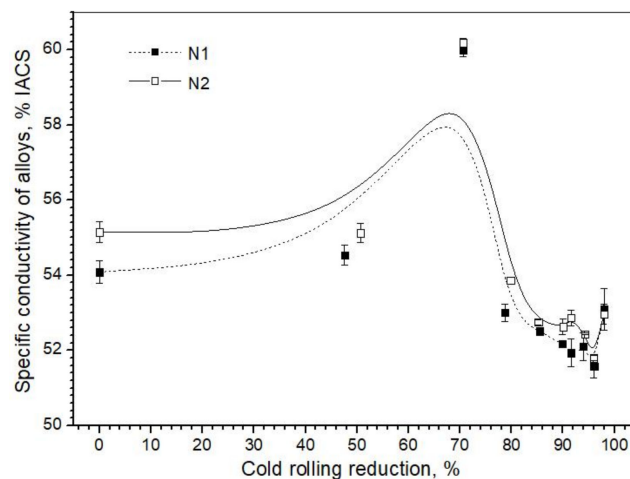


Figure 2. Change in specific conductivity.

3.2. Microstructure of Alloys

TEM images of alloys N1 and N2 at 50% cold rolling reduction are shown in Figures 3 and 4, respectively. It was found that the dislocations were mainly concentrated along the grain/sub-grain boundaries. There were few dislocations inside the grains, although many sub-grain structures formed inside them. During the cold rolling process, dislocations formed and gradually slipped to concentrate around the grain boundaries and the second phase particles. As a result, the substructures formed in positions in which dislocation is difficult to cross-slip and climb [14]. Sub-grain structures can impede

the movement of dislocations in the same way grain boundaries can. This contributes to an overall increase in the alloys' strength.

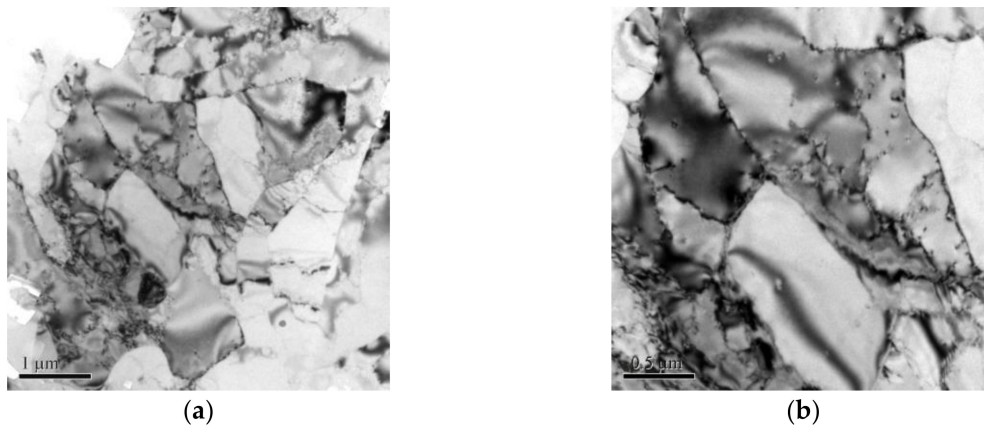


Figure 3. TEM images of alloy N1 at 50% cold rolling reduction. (a) with 2550 times magnification and (b) with 5000 times magnification.

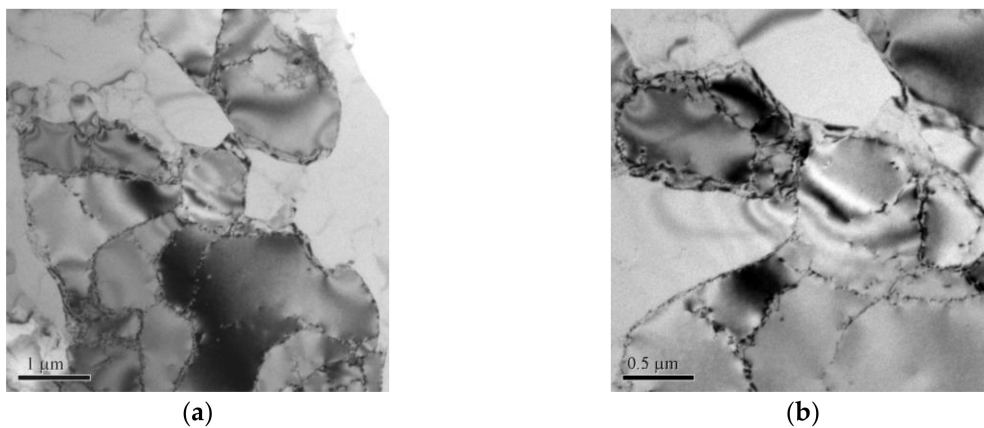


Figure 4. TEM images of alloy N2 at 50% cold rolling reduction. (a) with 2550 times magnification and (b) with 5000 times magnification.

The microstructures of alloy N1 at 85% cold rolling reduction are shown in Figure 5. The microstructures of alloy N2 at 80% cold rolling reduction, corresponding to the hardness peak point at which the microhardness of alloy N2 is close to that of alloy N1, are shown in Figure 6.

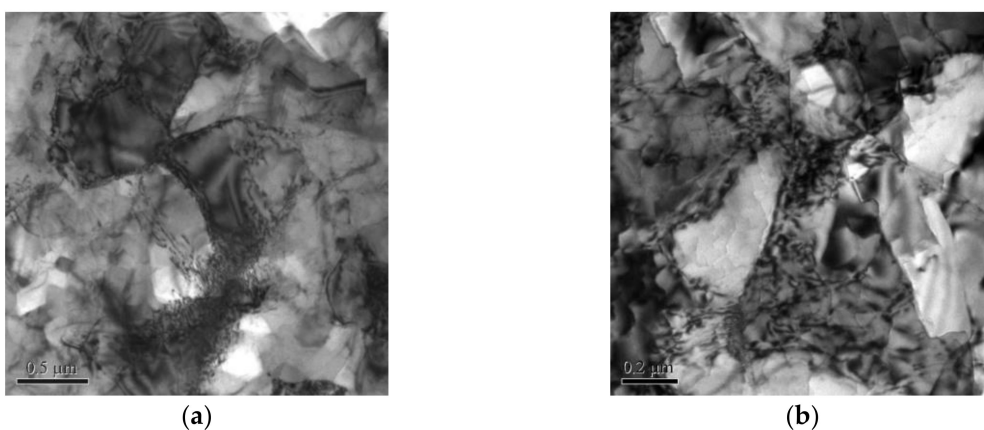


Figure 5. TEM images of alloy N1 at 85% cold rolling reduction. (a) with 5000 times magnification and (b) with 9900 times magnification.

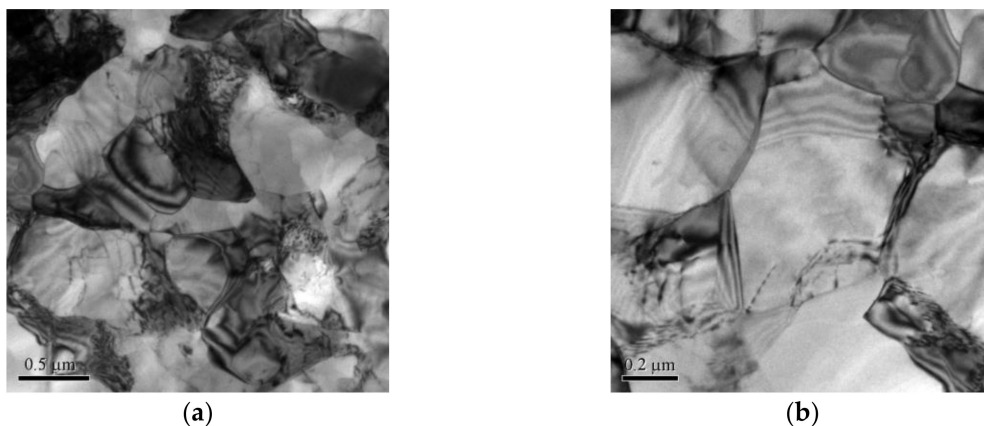


Figure 6. TEM images of alloy N2 at 80% cold rolling reduction. (a) with 5000 times magnification and (b) with 9900 times magnification.

The dislocations can be observed both along the grain boundaries and inside the grains, and the density of dislocations is significantly higher than that of the samples subjected to lower levels of reduction. A large number of dislocations became intertwined, appearing as black cluster structures and cellular structures formed by the dislocation lines. Meanwhile, there was an obvious increase in the number of sub-grain structures, which further hindered dislocations. As a result, the alloy shows further work hardening.

It can be observed that a small number of sub-grains is equiaxed in the shape, as shown in Figure 6, while none are in Figure 5. This phenomenon is related to the critical point of transition from work hardening to softening. At 80% cold rolling reduction, alloy N2 is in a critical state, while alloy N1 is still in a state of work hardening at a cold rolling reduction level of 85%.

TEM images of alloys N1 and N2 at a cold rolling reduction level of 96% are shown in Figures 7 and 8, respectively. Compared with Figures 5 and 6, the number of dislocations located at grain boundaries and inside grains is greatly reduced. Moreover, the tangle extent of dislocations is significantly decreased. Therefore, the recovery process occurred during cold rolling. Furthermore, it may be observed that many sub-grain structures polygonized and transformed into equiaxed sub-grains.

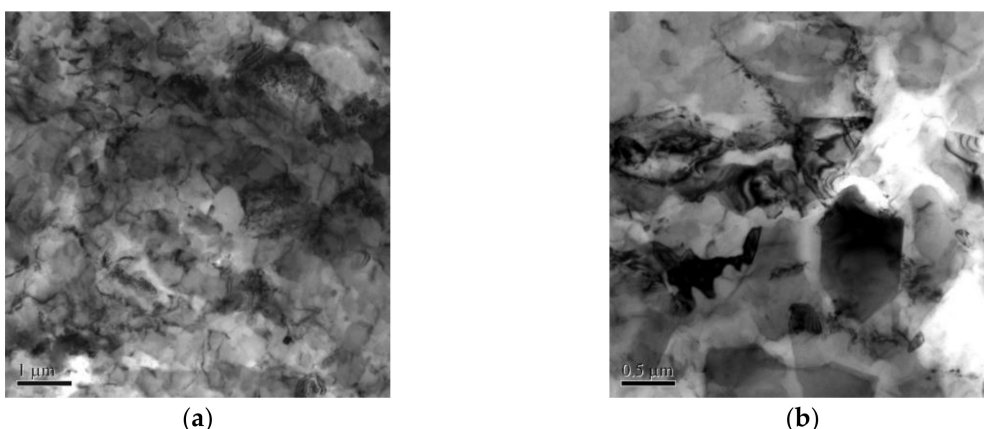


Figure 7. TEM images of alloy N1 at 96% cold rolling reduction. (a) with 2550 times magnification and (b) with 5000 times magnification.

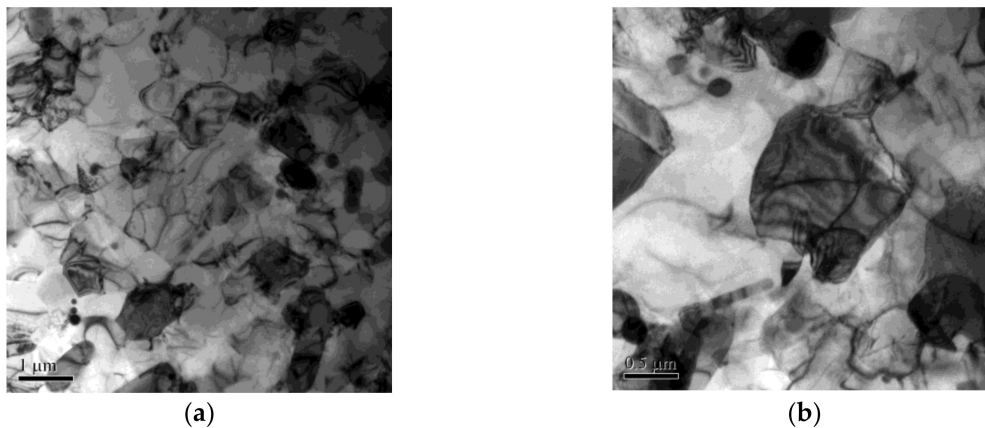


Figure 8. TEM images of alloy N2 at 96% cold rolling reduction. (a) with 2550 times magnification and (b) with 5000 times magnification.

4. Discussion

4.1. Change in Microhardness

As shown in Figure 1, it was found that there are some differences between the deformation behaviors of the two alloys. The softening behavior of alloy N2 with high Fe content occurs earlier and the transition from work hardening to work softening is gradual. However, the work softening behavior of alloy N1 with low Fe content appears at a higher level of cold rolling reduction and it abruptly changes from work hardening to work softening.

It may also be observed that the microhardness of the two alloys is very similar in the initial and final stages of the cold rolling after the intermediate annealing.

Since the solubility of iron in an aluminum matrix is very limited, the solubility of iron in two alloys can be deemed similar. Furthermore, the alloys are completely soft when they have been annealed at a temperature of 360 °C. Therefore, the hardness of the two alloys is similar, because of their very similar composition. The hardness of alloy N1 is slightly higher than that of alloy N2, owing to the greater solid solubility of Mn into alloy N1. This is in accordance with the results from the measurement of the alloys' specific conductivity, as shown in Figure 2.

After the cold rolling process with intermediate annealing, the dislocations in the alloys proliferated continuously by cold deformation, thus increasing the hardness of the alloys. At lower levels of cold rolling reduction, the dislocation density of the two alloys was nearly the same, as shown in Figures 3 and 4. Thus, the hardness of the two alloys was roughly the same.

It has been reported that dynamic recovery and static recovery can easily take place in Al-Fe alloys [15]. At a cold rolling reduction level of 96%, recovery happened in both alloys, obviously leading to a significant decrease in the number of dislocations in the alloys. Thus, the hardness of the alloys is determined by the solid solution of the matrix and the small number of residual dislocations in the alloys. This explains why the two alloys have almost the same microhardness at the end of the cold rolling process.

4.2. Change in Specific Conductivity

At a cold rolling reduction level of 0%, when the alloys are in a completely annealed state, their conductivity is mainly determined by the type and content of the elements dissolved in the matrix [16]. Owing to the low solubility of iron into an aluminum matrix (much lower than 0.002 mass% at room temperature), the effect of iron on the specific conductivity of an aluminum alloy is negligible [17,18]. The solubility of Mn in an aluminum matrix is much higher than that of Fe: 1.82 mass% at 658 °C and 0.2~0.3 mass% at room temperature [17,18]. The content levels of Si, Ti, and other elements (except Fe and Mn) of the two alloys are almost the same, thus the conductivity of the two alloys is predominately

controlled by the solubility of Mn in the matrix [16]. As the Mn content of alloy N2 is lower than that of alloy N1, the Mn content of the solid solution in the matrix of alloy N2 is lower than that of alloy N1 under the same processing conditions. Therefore, the conductivity of alloy N2 is higher than that of alloy N1.

For both alloys, there are three peaks along the conductivity curve. The first noticeable peak occurs at a cold rolling reduction level of around 70%. The second peak appears at 94% (alloy N1) and 91% (alloy N2), respectively, and the change is not obvious. The third peak occurs at the end of cold rolling, when the cold rolling reduction reaches its maximum.

During the cold rolling process, dislocations continued to proliferate in the alloys, generally resulting in a decrease in conductivity. However, as the level of cold rolling reduction increased from 50% to 70%, the conductivity of both alloys increased without any decrease, as shown in Figure 2. This phenomenon is very interesting and may be related to precipitation.

Precipitation is a thermal activation process that involves nucleation and growth. The nucleation rate depends on the nucleation activation energy and the diffusion activation energy of the solute atoms. The dislocations in alloys interconnect with one another to form a network of dislocations. Diffusion atoms diffuse along the dislocations, which leads to the amount of diffusion activation energy being reduced by half. Furthermore, increasing the dislocation density can accelerate the diffusion process and decrease the nucleation activation energy, which greatly shortens the incubation period of the precipitation and increases the nucleation rate [19,20].

The redistribution of the precipitate-forming elements between the dislocation cell walls and the cell interiors during deformation may result in an increase of local supersaturation, which will affect the thermodynamics of precipitation. Both the nucleation site density and diffusivity of precipitate-forming elements in the material increase with that of the dislocation density by deformation. This helps to promote precipitation. In addition, the segregation of Mn at dislocations during cold rolling can cause a decrease in the Mn concentration in the matrix, leading to an increase in conductivity [20].

Microhardness is not sensitive to the precipitation process [20]. Therefore, in the process of increasing the level of cold rolling reduction from 50% to 70%, the microhardness curves (as shown in Figure 1) of the alloys do not fluctuate.

In order to prove that the above deduction is correct, further research work is needed to investigate the phenomenon of precipitation.

The second peak occurs in the process of work softening, which may be related to the decrease of dislocations and the occurrence of recovery. The second peak of alloy N2 is brought forward to that of alloy N1, which corresponds to the process of work softening in the two alloys.

The appearance of the third peak is a result of the complete softening of the alloys. During this process, the number of dislocations decreases notably and the residual stress is obviously released due to polygonization.

4.3. Discussion on Work Softening

The width of the stacking fault of the metal with a high stacking fault energy is narrow, which makes the cross-slip easy to occur through bundling dislocations [12]. Owing to their face-centered cubic lattices, aluminum alloys contain many slip systems and a high stacking fault energy that could increase with reduced impurities in the matrix [12,14]. Thus, it is easy to form a cellular structure that can affect work softening in aluminum alloys thanks to the proliferation and interaction of dislocations during their deformation [8].

The subsequent deformation can provide sufficient power for recovery when the dislocation density rises to its maximum during cold rolling. The warming of rolled sheets during cold rolling can also accelerate recovery. Therefore, dislocation tangles in alloys can be broken and dislocations on the same slip plane annihilate one another by a combination of gliding and climbing mechanisms, which cause noticeable decreases in the dislocation density. Meanwhile, the sub-grain structures change due to polygonization. This is the typical phenomenon of work softening [8,10–14,21].

At the end of the first stage of recovery, there are excess dislocations left in the material. Upon further deformation, these excess dislocations are arranged in low angle grain boundaries or a low energy configuration in the form of regular arrays [21]. This mechanism is known as polygonization. Low angle grain boundaries are caused by a dislocation movement from the interior of cells toward their boundaries, leading to the formation of sub-grain structures [21].

The Fe element tends to combine with Si and Mn to form intermetallic compounds, which can decrease the amount of solid solution in the matrix. The composition of alloys N1 and N2 is almost the same except for their Fe content. Alloy N2 may have a higher stacking fault energy than that of alloy N1, because of its higher Fe content. Therefore, the critical point of work softening of alloy N2 occurs earlier than that of alloy N1.

It has been pointed out by Shinpei Maeda [22] that aluminum can recrystallize at room temperature through the addition of a small amount of calcium. The effects of Fe and Mn as they lower the recrystallization temperature of aluminum to room temperature are different. It is not beneficial to lower the recrystallization temperature, even with trace amounts of added Mn. However, the addition of Fe has almost no adverse effects. In the present study, due to the lower Mn content and the higher Fe content of alloy N2 compared with those of alloy N1, the recrystallization temperature of alloy N2 may have been lower than that of alloy N1, resulting in a greater susceptibility to softening.

Several studies [10–12] reported that work softening could not occur in Al-0.2/0.7/1.2Fe alloys prepared with 99.96% purity aluminum metal as well as in the Al-2Fe alloy prepared from 99.6% purity aluminum metal. They contradict the present work. Reasons for this may be the following: (1) the heat treatment parameters and particularly the cooling conditions may have been different, which may have affected the solid solubility of the matrix; (2) in the present study, the Mn content of the alloys is lower and mainly forms intermetallic compounds with Fe, leading to the limited solubility of the solution.

5. Conclusions

This study investigated the microstructure transformations and deformation behavior of Al-Fe-Mn alloys with different Fe contents during their cold rolling process. Based on our results, the primary conclusions of this work can be summarized as follows:

1. It was observed that there were two kinds of deformation behaviors during cold rolling in Al-Fe-Mn alloys with 0.9 and 1.4 mass% Fe content when prepared from 99.7% purity aluminum ingots: work hardening and work softening.
2. Once work softening occurred, the number of dislocations located along grain boundaries and inside grains greatly decreased. Many sub-grain structures polygonized and transformed into equiaxed sub-grains.
3. Compared with Al-0.9Fe-0.2Mn, the softening behavior of the Al-1.4Fe-0.2Mn alloy occurred at a lower level of cold rolling reduction. The critical levels of cold rolling reduction for the Al-1.4Fe-0.2Mn alloy and Al-0.9Fe-0.2Mn alloy at which work hardening transformed into work softening were 80% and 90%, respectively.
4. The conductivity of the Al-1.4Fe-0.2Mn alloy was higher than that of the Al-0.9Fe-0.2Mn alloy throughout each cold rolling reduction, which meant that the solid solution content of the matrix of the Al-1.4Fe-0.2Mn alloy was lower than that of the Al-0.9Fe-0.2Mn alloy. This was the main reason why the critical point at which work softening began for the Al-1.4Fe-0.2Mn alloy was earlier than that of the Al-0.9Fe-0.2Mn alloy.

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Conflicts of Interest: The authors declare no conflict of interest.

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