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ARTICLE

Room Temperature Recovery Behavior of Cold-Rolled Aluminum Foil Under Severe Plastic Deformation

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Abstract: Pure aluminum foil (dilute Al-Fe-Si alloy series) with Cu or Mn addition was prepared by severe cold-rolling deformation, and the effect of room temperature storage or low temperature annealing on the tensile properties and microstructure was investigated through tensile tests, optical microscope, scanning electron microscope, electron back scatter diffraction, and atom probe microscope. Results show that the ultimate tensile strength and elongation simultaneously decrease after room temperature storage. The recovery mechanism of substructure, such as subgrain coalescence, leads to the decrease in tensile properties. The decrease in plasticity is more significant for the Mn-containing alloy due to the more significantly increased subgrain size. The atom cluster strengthening can compensate for the strength loss to some extent, whereas the effect of the secondary phases is negligible.

Key words: aluminum foil; tensile properties; severe plastic deformation; recovery

With the fast development of electrical vehicles and portable electronic devices, the demand for aluminum foils, which are mainly made from 1XXX aluminum alloys, as the positive current collector of Li ion batteries is rapidly increased. High compaction density can increase the energy density of Li ion batteries. As a result, the thinner and stronger aluminum foil is required. The thickness of aluminum foil for positive current collector can be reduced to ⩽13 μm, and the typical tensile strength of over 200 MPa can be achieved with elongation of over 4%, which greatly exceed the standard for 1060-H18 temper (tensile strength⩾110.32 MPa, elongation⩾ 1%, ASTM B209-2014).

Such high strength of aluminum foil can only be achieved by applying severe plastic deformation (SPD) through repetitive cold rolling with total strain of ⩾5.5. SPD techniques, such as equal channel angular press, high pressure torsion, and accumulative roll bonding, have been widely researched. However, the microstructure and properties of aluminum foil produced by industrial-scale rolling mills are rarely investigated. Different microstructures are formed when different processing techniques are applied. During SPD by cold rolling, the deformed grains with lamellar structure appear[1]. During the equal channel angular pressing or high-pressure torsion, the ultra-fine equiaxed grains are formed under the large shear strain[2–3]. Different microstructures result in different properties and plastic behavior of materials. Besides, for aluminum foil with thin thickness, the effect of specimen dimension can also influence the foil properties.

During the production of severely deformed aluminum foil, both the tensile strength and elongation are decreased at room temperature, and the microstructure evolution mechanism of this phenomenon is still obscure. The effect of room temperature annealing on severely deformed metals has been widely researched. Yu et al[4] found that for cold-rolled AA1050 alloy, after deformation with true strain of 5.5, the annealing treatment at 5 – 100 °C can decrease the alloy hardness. However, the plasticity change has not been

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investigated[1,4–6]. Commonly, the annealing of deformed aluminum alloy will increase the plasticity and decrease the strength. The simultaneous decrease in plasticity and strength hardly occurs. Thus, the microstructure of aluminum foil produced by cold rolling through industrial-scale rolling mills and the effect on tensile behavior and microstructure evolution mechanism should be investigated.

In this research, trace Cu or Mn was added into the 1XXX alloy to improve the work hardening ability[7]. Two typical alloys were chosen for the investigation. This research provided guidance for the design of aluminum foil with high performance.

1 Experiment

The aluminum foil was produced from the base metal of dilute Al-Fe-Si alloy, whose composition was similar to that of the commercial AA1050 alloy. Cu or Mn with different contents was added into the base metal. The ingots were produced by the direct chill casting method. The Al-5Ti-1B master alloy was used as the grain refiner for casting. The AA1050 alloys after Cu and Mn addition were named as 1050-Cu and 1050-Mn alloys, respectively, and their nominal chemical composition is shown in Table 1.

The ingots of about 8 tons after direct chill casting were homogenized at 600 °C for 10 h and cooled to the initial rolling temperature (480 °C). Then, they were hot-rolled into plates of 6 mm in thickness. The hot-rolled plates were heated at 380 °C for 3 h (intermediate annealing) to achieve the foil with 2 mm in thickness, and subsequently cold-rolled to the foil with thickness of 13 μm. All the rolling processes were performed by the industrial-scale rolling mills. In order to observe the decreased tensile properties, the aluminum foil was kept at 20 °C (room temperature) for 60 d.

The specimens were cut along the rolling direction for tensile tests. The dimension of rectangular uniaxial tensile specimens was set according to ASTM E8/E8M-16a standard: the gauge length was 50 mm and the rectangular gauge cross-section was used for observation, as shown in Fig. 1. All tensile tests were performed by an electronic tensile testing machine at room temperature under a constant crosshead speed of 10 mm/min. Each data point was tested three times.

Table 1 Nominal chemical composition of 1050-Cu and 1050-Mn alloys (wt%)

Alloy	Fe	Si	Cu	Mn	Ti	Al
1050-Cu	0.20	0.05	0.04	-	0.02	Bal.
1050-Mn	0.20	0.05	-	0.02	0.02	Bal.

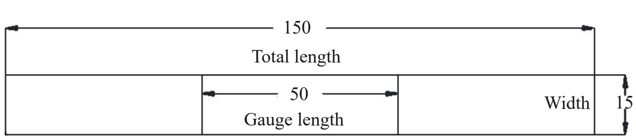

Fig.1 Schematic diagram of tensile specimen

Optical microscope (OM, Zeiss AX-10 metallographic microscope) and transmission electron microscope (TEM) were used for microstructure observation. Scanning electron microscope (SEM, TESCAN MIRA3 microscope) equipped with electron backscattered diffractometer (EBSD, OXFORD Nordlys Nano EBSD detector) was also used for micro-structure analysis. EBSD data was analyzed by Channel 5 software. The specimens were mechanically and electroly-tically polished before OM/SEM observation.

The specimens for atom probe microscope (APM) obser-vation were prepared by the standard two-step electro-polishing technique, and the sharp needle-like specimens were obtained[8]. APM experiments were performed on the LEAP 4000 HR instrument under the ultrahigh vacuum condition ($\leqslant 1.47\times10^{-8}$ Pa) at about 20 K. The pulse fraction was 20%, the pulse repetition rate was 200 kHz, and the detection rate was 0.01 atom per pulse. The detection efficiency (DE) and image compression factor (ICF) were adjusted to produce the authentic 3D reconstruction images. Fig. 2 shows TEM morphology of needle-like APM specimen. The experiment results were analyzed by IVAS 3.6.8 software. The distribution of Cu atoms was analyzed within the distance d_{max} =0.6 nm by the cluster analysis tools in IVAS software.

2 Results and Discussion

2.1 Decrease in tensile properties at room temperature

The tensile properties of different aluminum foils of 13 μm in thickness after room temperature storage for 60 d are shown in Fig.3. The total plastic strain of the aluminum foil is beyond 5.0.

It can be seen that the initial ultimate tensile strength of 1050-Cu foil is higher than that of 1050-Mn foil, while the initial elongation of 1050-Mn foil is higher than that of 1050-Cu foil. The higher strength of 1050-Cu foil can be attributed to the higher solute concentration and the increased strain hardening caused by Cu solutes[9]. Commonly, the high strength of Al-Cu alloy is attributed to the cluster of solute atoms during the natural aging[10]. However, the 3D APM results show that in dilute alloys, the cluster of Cu atoms is negligible.

It is worth noting that the aluminum foils of different alloys

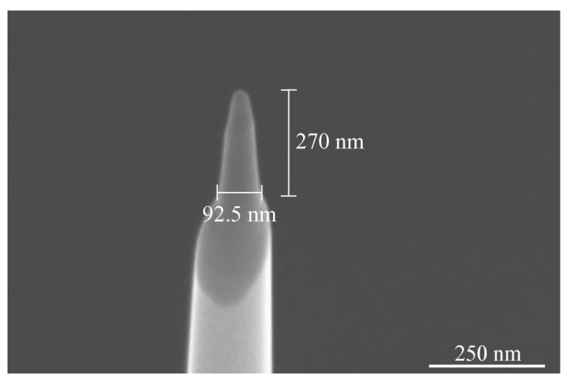

Fig.2 TEM morphology of needle-like APM specimen

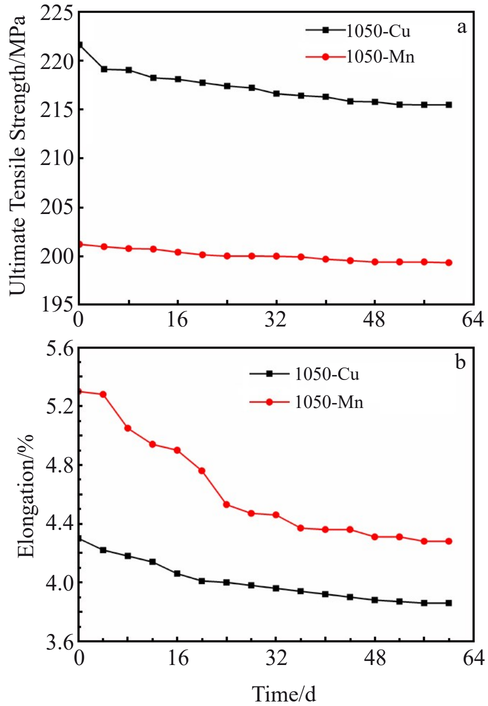

Fig.3 Tensile properties of different aluminum foils during room temperature storage for 60 d: (a) ultimate tensile strength and (b) elongation

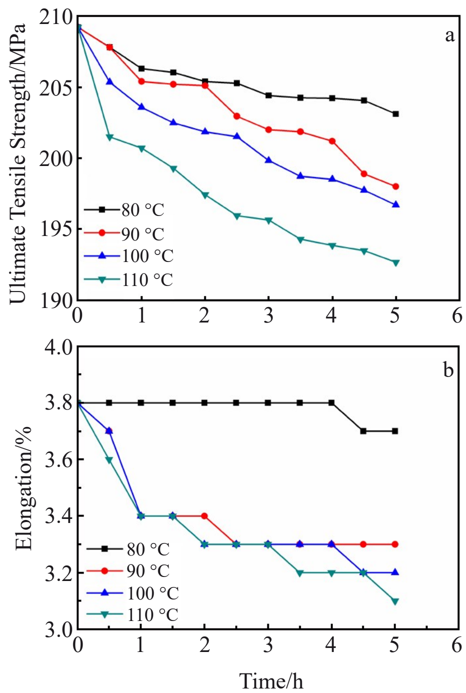

Fig.4 Tensile properties of 1050-Cu foils after annealing at different temperatures: (a) ultimate tensile strength and (b) elongation

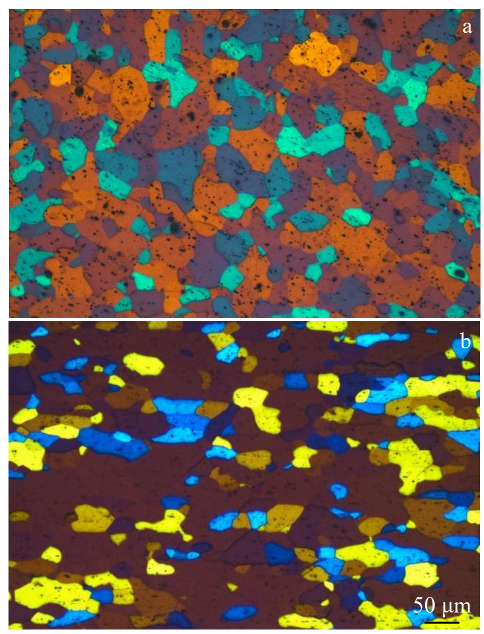

Fig.5 Grain microstructures of 1050-Cu (a) and 1050-Mn (b) foils after intermediate annealing treatment

both suffer a slight decrease in ultimate tensile strength and a significant decrease in elongation. Moreover, the decrement of elongation of 1050-Mn foil is greater than that of 1050-Mn foil.

2.2 Temperature effect

The effect of temperature on recovery and tensile properties of 1050-Cu aluminum foil was investigated through annealing at different temperatures (80–110 °C with temperature interval of 10 °C), as shown in Fig.4.

It can be observed that both the tensile strength and elongation are decreased during the annealing. However, the decreasing rate is much higher, compared with that caused by the variation in foil composition. In addition, the decreasing rate is increased with increasing the annealing temperature. Ref. [11 – 12] report that the recrystallization temperature of 1050 aluminum alloy is usually over 200 ° C. Thus, the annealing temperature is below the recrystallization temperature, and only the crystal recovery occurs. The conventional recovery of the aluminum alloy increases plasticity at the cost of strength. Therefore, the results in this research indicate that the foil thickness has a significant effect on the tensile properties. It can also be inferred that the microstructure evolution during the annealing treatment is similar to that during the room temperature storage. Thus, the tensile properties are decreased after annealing treatment with an even faster decreasing rate.

2.3 Grain microstructures and the secondary phases

The grain microstructures of 1050-Cu and 1050-Mn foils after intermediate annealing are shown in Fig. 5a and 5b, respectively. It can be observed that the full recrystallization occurs, and therefore the strain is negligible after annealing. The grain sizes of the two alloy foils are similar, indicating that the trace addition of Cu or Mn has similar effect on the recrystallization of the alloy foil.

SEM morphologies of the secondary phases in 1050-Cu and 1050-Mn foils are shown in Fig.6a and Fig.6b, respectively. It

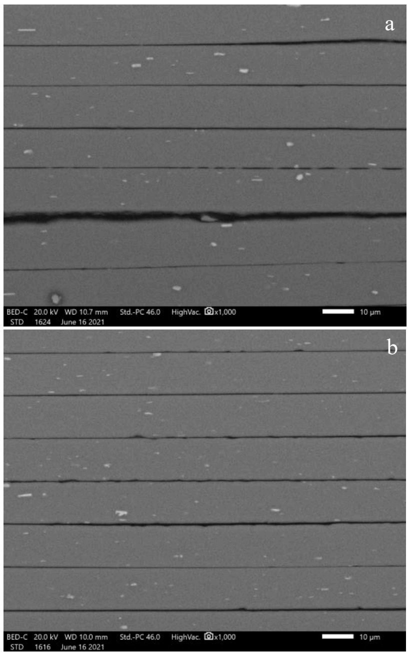

Fig.6 SEM morphologies of the secondary phases in 1050-Cu (a) and 1050-Mn (b) foils

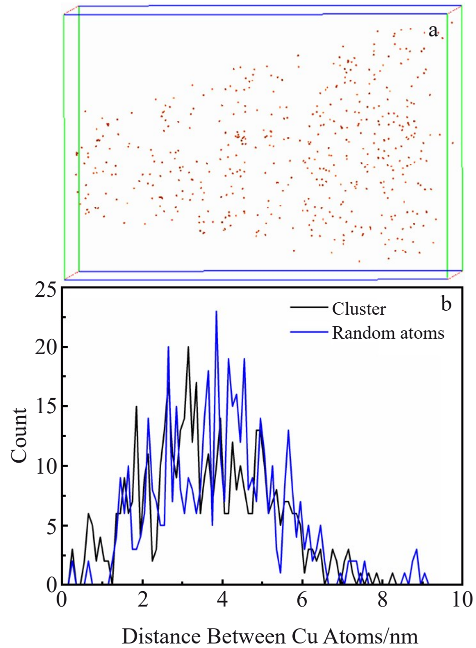

Fig.7 Spatial distribution (a) and N-N distributions of Cu solute atoms (b) in 1050-Cu foil after room temperature storage for 60 d

can be observed that for the two alloys, the size, morphology, and distribution of the secondary phase particles are similar. EDS results show that the composition of the secondary phases is mostly Al and Fe, with a minor concentration of Si. As a result, it can be inferred that a trace amount of Cu or Mn addition has no significant effect on the intermetallic phases.

2.4 Segregation of solute atoms

APM was applied to investigate the solute atom segregation of the 1050-Cu and 1050-Mn foils. For the 1050-Cu foil after room temperature storage for 60 d, the spatial distribution and solute-solute nearest-neighbor (N-N) distances of the Cu atoms are shown in Fig.7.

It can be observed that some Cu atoms form the solute atom clusters after the natural aging, and the measured N-N distances of the Cu solute atoms are slightly smaller than those of atoms with random distribution[13]. It can be inferred that due to the low concentration of solute atoms, the solute atom cluster only accounts for a minor proportion of Cu atoms during the room temperature storage. Therefore, this phenomenon cannot result in the decrease in tensile properties. Thus, the microstructure evolution mechanism for the decrease in tensile properties requires further investigation.

2.5 Substructure evolution

The distribution of grain boundary misorientation angle in 1050-Cu and 1050-Mn foils before and after room temperature storage is shown in Fig.8.

A large number of low angle grain boundaries (LAGBs) can be observed in both alloy foils, which are normally introduced by plastic strains[14]. The distributions of grain boundary misorientation angle in two alloys are similar. Thus, it can be concluded that the effect of different solute atoms on the misorientation angle is negligible. The area fractions of LAGBs are slightly increased after room temperature storage, which probably results from the transformation from subgrain to LAGB after assimilating the dislocations[15–16]. The transformation of subgrains is also in agreement with the subgrain size evolution.

The subgrain boundary distributions of 1050-Cu and 1050-Mn foils before and after room temperature storage are shown in Fig. 9, and their subgrain diameter distributions are shown in Fig.10.

Ref. [1] and Ref. [5] report that the recovery mechanisms, such as triple junction motion, can cause uniform coarsening of deformed lamellar microstructures, which leads to a more equiaxed morphology. The coarsening of lamellar subgrains is supported by the aspect ratios of the subgrains: the aspect ratio of 1050-Mn foil is decreased from 2.0 to 1.8 after room temperature storage for 60 d. This phenomenon may result in the decrease in the ratio of thickness to (sub)grain size, which may be detrimental to the plasticity of aluminum foils[17]. Thus, it can be concluded that the greater the subgrain size, the greater the decrease in elongation and the better the recovery. Besides, the increase in subgrain size is more significant in the 1050-Mn foil, compared with that in 1050-Cu foil, which leads to greater elongation loss.

The decrease in the tensile strength is less significant than that in elongation, because the cluster of solute atoms will result in solute cluster strengthening effect[18], which can compensate for the loss in tensile strength during the recovery. However, the effect of the solute cluster strengthening is very limited, due to the low Cu/Mn concentration in the alloy foil. As a result, the recovery of substructures, such as subgrain coalescence, occurs during the room temperature storage, resulting in the decrease in tensile properties.

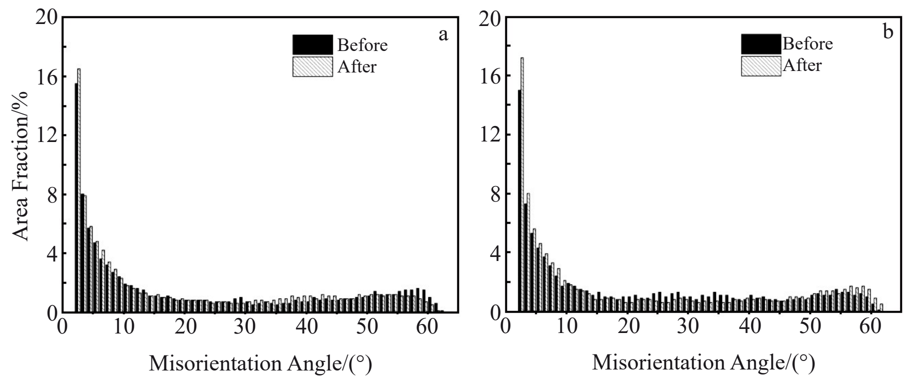

Fig.8 Distributions of grain boundary misorientation angle in 1050-Cu (a) and 1050-Mn (b) foils before and after room temperature storage

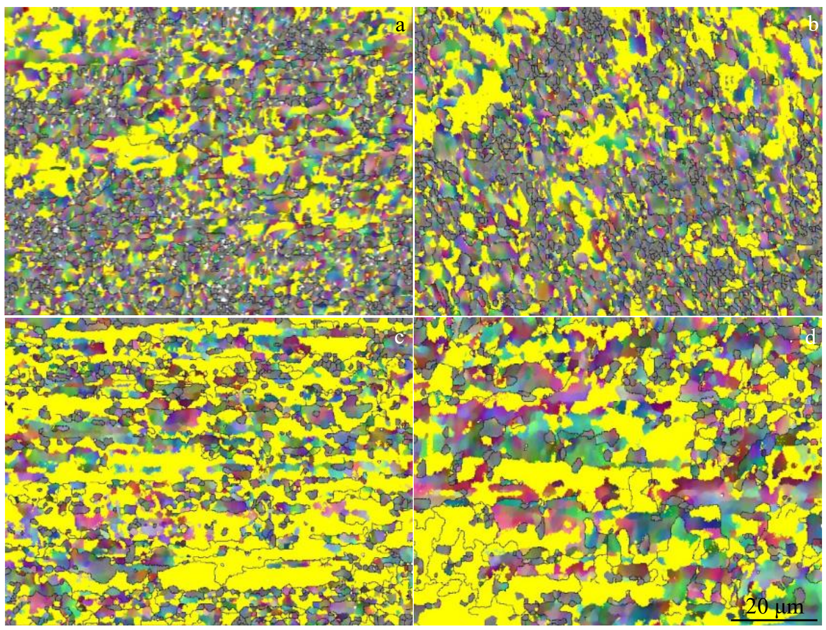

Fig.9 Subgrain boundary distributions of 1050-Cu (a, c) and 1050-Mn (b, d) foils before (a, b) and after (c, d) room temperature storage

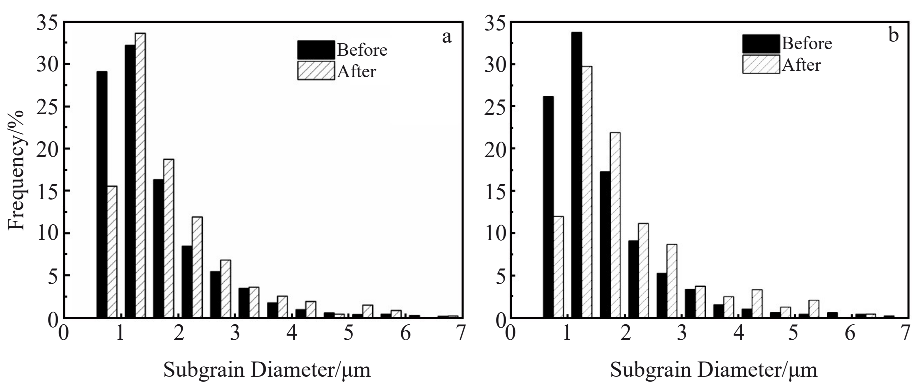

Fig.10 Subgrain diameter distributions of 1050-Cu (a) and 1050-Mn (b) foils before and after room temperature storage

3 Conclusions

1) Both the ultimate tensile strength and the elongation of the aluminum foils are decreased after room temperature storage for 60 d or after low temperature annealing for several hours. The loss of elongation is greater in the AA1050 alloy with Mn addition.

2) The solute atom clusters are formed, which can compensate for the strength loss through solute cluster strengthening effect to some extent. The effect of Cu/Mn

addition on the secondary phases is insignificant after room temperature storage.

3) The recovery of substructures, such as subgrain coalescence, occurs during the room temperature storage, resulting in the decrease in tensile properties. The increase in subgrain size is more significant in the AA1050 alloy with Mn addition, which leads to greater elongation loss.

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大塑性变形冷压铝箔的室温回复行为

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摘　要：通过大轧制变形制备了含铜或锰的工业纯铝箔（低合金铝铁硅系），通过拉伸试验、光学显微镜、扫描电镜、电子背散射衍射和三维原子探针研究了室温停放或低温退火对铝箔力学性能和组织的影响。结果表明，室温停放后铝箔的抗拉伸强度和延伸率同时下降。亚晶合并等亚结构的回复机制导致了力学性能下降。含Mn合金因其亚晶尺寸的增加更为显著，延伸率下降更为明显。原子团簇强化可以部分补偿强度下降，而第二相的影响可忽略不计。

关键词：铝箔；力学性能；大塑性变形；回复

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