Rare Metal Materials and Engineering
Volume 51, Issue 5, May 2022
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Fang Xiaoyang, Ren Jieke, Chen Dengbin, et al. Effect of Alloying Elements and Processing Parameters on Microstructure and Properties of 1XXX Aluminium Alloys[J]. Rare Metal Materials and Engineering, 2022, 51(05): 1565-1571.

ARTICLE

Effect of Alloying Elements and Processing Parameters on Microstructure and Properties of 1XXX Aluminium Alloys

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Abstract: Direct chill casting (DCC) and twin roll casting (TRC) methods were applied to produce 1XXX series aluminium blanks with different contents of Fe, Si, Cu, and Mn elements. Different treatments, including homogenization, hot rolling, cold rolling, and annealing, were subsequently applied to produce the aluminium foil of 13 μm in thickness. Results show that the casting process barely affects the tensile strength of the aluminium foil, while the fine particles of secondary phase introduced by twin roll casting has a positive effect on the elongation of aluminium foil. The coarse grain boundary intermetallic in ingots after DCC can be refined at homogenization temperature above 580 °C, and subsequently be broken into small particles through the following hot rolling process. The addition of Cu is better than that of Fe, Si, or Mn elements, as Cu solute atoms can increase the strain hardening rate. The foil thickness greatly affects the tensile properties during intermediate annealing process, which is related to the ratio of thickness to grain size. Room temperature storage causes deterioration of tensile properties of thin aluminium foil due to the recovery mechanism after application of very large strain.

Key words: aluminium alloy; tensile properties; secondary phases; grain structure

The aluminium foil has been extensively applied in packaging, heat exchangers, condensers, and Li ion batteries. With the fast development of electrical vehicles and portable electronic devices, the demand for aluminium foil as the positive current collector of Li ion batteries, which is mainly produced from 1XXX aluminium alloys, is rapidly increasing. Fe and Si usually exist in commercial 1XXX aluminium alloys, as alloying elements or impurities. Cu, Mn, Mg, V, and Zn are also the typical alloying or impurity elements in 1XXX aluminium alloys[1,2], and the Al-Ti-B master alloy is commonly used as an industrial grain refiner[3].

Great efforts have been made to increase the energy density and structural stability of Li ion batteries, leading to requirements for thinner thickness and better properties of aluminium foil. The thickness of aluminium foil for positive current collector already reduces from 15~20 μm to 9~10 μm. The optimal properties of strength and plasticity of the aluminium foil are also required for the production of Li ion batteries. The typical tensile strength can reach 220 MPa or even higher value with elongation of over 3%, which greatly exceeds the properties of standard 1100-H18 alloys (tensile strength≥151.7 MPa with elongation≥1% according to ASTM B209-2014). It is very difficult to achieve such a high strength with good plasticity under the condition of high strain for thin aluminium foil.

Most researches about the positive current collector focus on the surface treatment or electrochemical properties of aluminium foil[4,5], and the properties and microstructure of aluminium blanks for foil production[6,7]. Few researches investigate the mechanical properties of aluminium foil[8], because conventionally the thin aluminium foil can only be produced by industrial-scale high-speed roller mills. Therefore, in this research, the effect of alloying elements and processing parameters on the microstructure and tensile properties of thin aluminium foil of 1XXX series aluminium alloys was investigated. Direct chill casting (DCC) and twin roll casting (TRC) methods were used to prepare the aluminium foil. The ingots and slabs were heat-treated and

Received date: May 11, 2021
Foundation item: Fundamental Research Funds for the Central Universities (2018XZZX001-05)
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rolled repeatedly to produce thin foil with a thickness of 10~15 μm. This research provided a guidance for design and manufacture of high-performance aluminium foil.

1 Experiment

Both DCC and TRC methods were applied to produce the 1XXX aluminium alloys, and the measured chemical composition of alloys after DCC and TRC treatments is shown in Table 1 and Table 2, respectively. The Fe and Mn additions were in the form of pure metal powders. The Si and Cu additions were in the form of Al-12wt% Si and Al-50wt% Cu master alloys, respectively. The Ti addition was in the form of Al-5Ti-1B master alloy as grain refiner. The overall components of the alloys were determined by the ARL 3460 Advantage Spectrometer.

The ingots for DCC treatment were homogenized at 600 °C for 10 h and cooled down to the initial rolling temperature of 480 °C, and then hot-rolled into plates of 6 mm in thickness. The intermediate annealing thickness was 0.4, 1.5, and 6.0 mm, and only the specimens with intermediate annealing thickness of 1.5 mm were used for main research. Then the hot-rolled plates were cold-rolled into the foil with thickness of 13 μm. The ingots which were 7 mm in thickness for TRC treatments, were then intermediate-annealed into plates of 0.4, 1.5, and 4.0 mm in thickness, and finally cold-rolled into the foil with thickness of 13 μm. Only the specimens with intermediate annealing thickness of 1.5 mm were used for main research. The practical annealing temperature curves of large coils were measured by thermocouples, as shown in Fig.1. The average heating rate was about 0.1 °C/s.

The tensile properties of aluminium foil were assessed using the tensile specimens manufactured along the rolling direction. The dimension of uniaxial tensile specimens has a gauge length of 50 mm and a rectangular gauge cross-section. All the tensile tests were performed using an electronic tensile

Table 1 Chemical composition of DCC 1XXX aluminium alloy ingots (wt%)

Alloy	Fe	Si	Cu	Mn	Ti	Al
1100-0.04Cu	0.69	0.06	0.04	-	0.01	Bal.
1100-0.08Cu	0.52	0.05	0.08	-	0.01	Bal.
1100-0.12Mn	0.36	0.21	0.06	0.12	0.01	Bal.
1060-without Cu	0.20	0.08	-	-	0.01	Bal.
1060-0.04Cu	0.17	0.08	0.04	-	0.01	Bal.
1060-0.08Cu	0.17	0.08	0.08	-	0.01	Bal.

Table 2 Chemical composition of TRC 1XXX aluminium alloy ingots (wt%)

Alloy	Fe	Si	Cu	Mn	Ti	Al
1100-0.08Cu	0.68	0.06	0.08	-	0.014	Bal.
1100-0.26Si	0.54	0.26	0.08	-	0.020	Bal.
1100-0.20Mn	0.51	0.19	-	0.20	0.019	Bal.
1060-0.04Cu	0.19	0.07	0.04	-	0.018	Bal.

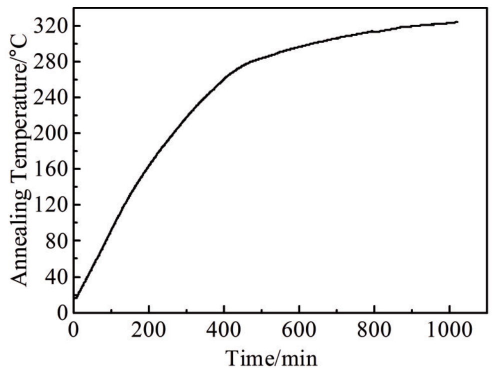

Fig.1 Typical annealing temperature curve of large coils

testing machine at room temperature under a constant crosshead speed of 10 mm/min. The mean value of results of three specimens was used, and the magnitude of the error bar represented the standard deviation around the mean value.

The Zeiss AX-10 optical microscope (OM) was used. The FEI Quanta FEG 650 scanning electron microscope (SEM) equipped with an EDAX Octane SDD energy dispersive spectroscopy (EDS) was also used. EDS electron beam diameter was 1 μm and the scanning duration was 60 s for each analysis point. The specimens for OM/SEM observation were polished mechanically and electrolytically through conventional techniques.

2 Results

The tensile properties of different aluminium alloys after DCC or TRC treatments are shown in Table 3.

It can be concluded that both the strength and elongation of 1100 series aluminium alloys are slightly higher than those of 1060 series aluminium alloys with the same Cu content. For alloys with the similar element contents, the casting method has little effect on the strength of aluminium alloys. However, the elongation of 1100 series aluminium alloys after TRC is slightly higher than that after DCC. The effect of more addition of Fe, Si, or Mn on the tensile strength of aluminium alloys is also negligible, while the higher Cu content can increase the tensile strength at the cost of elongation reduction. The Cu addition can obviously increase the strain hardening rate, as shown in Fig.2. The true strain is defined as $\ln(1+\Delta l/l_0)$, where Δl is the extension of the gauge length and l_0 is the original gauge length.

It can be observed that at the strain below 2.5, the strain hardening rate of 1060 series aluminium alloys with and without Cu addition is similar; however, when the strain exceeds 2.5, the strain hardening rate of 1060 series aluminium alloys without Cu significantly decreases, while that with Cu addition nearly remains unchanged.

The tensile properties of 1060-0.04Cu aluminium foil after DCC with different intermediate thicknesses are shown in Table 4. The annealed aluminium plates were subsequently rolled into the foil with the final thickness of 13 μm.

The tensile properties of 1060-0.04Cu aluminium foil are

Table 3 Tensile properties of 1XXX aluminium alloy foil

Treatment	Alloy	Yield strength/MPa	Ultimate tensile strength/MPa	Elongation/%
DCC	1100-0.04Cu	185±0.9	221±4.1	4.2±0.1
	1100-0.08Cu	211±1.6	238±2.5	3.7±0.1
	1100-0.12Mn	202±2.7	232±3.1	2.4±0.3
	1060-without Cu	169±2.5	193±2.2	4.3±0.3
	1060-0.04Cu	183±0.8	215±2.0	3.2±0.1
	1060-0.08Cu	200±1.3	222±3.7	2.4±0.2
TRC	1100-0.08Cu	209±0.8	235±1.3	4.4±0.2
	1100-0.26Si	210±4.1	237±2.1	4.1±0.1
	1100-0.20Mn	181±2.7	204±0.9	3.2±0.3
	1060-0.04Cu	184±1.9	214±0.7	3.6±0.1

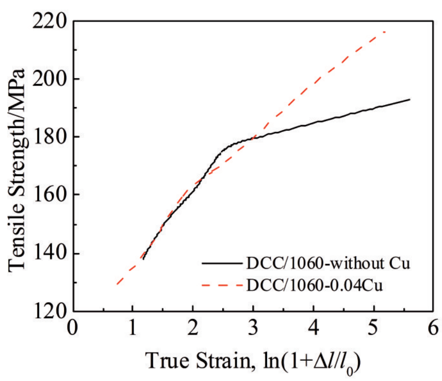

Fig.2 Strain hardening rate curves of 1060 series aluminium alloy foil with different Cu contents

significantly affected by the intermediate annealing thickness: the higher the intermediate annealing thickness, the better the tensile strength and elongation.

A decrease in tensile strength and elongation of aluminium foil after large strain deformation can be observed after storage at ambient temperature for several days, as shown in Table 5. The deterioration rate is decreased with time.

3 Discussion

3.1 Microstructure

The microstructure of as-cast aluminium alloys after DCC or TRC was discussed in Ref. [9-11]. TRC results in finer secondary phase particles (if macro segregation is inhibited), while DCC leads to a more uniform grain structure. The recrystallized grain sizes of 3003, 3105, 5052, and 5182 alloys after DCC or TRC show different conclusions, which can be related to different alloying element contents, solidification conditions, and heat treatment parameters. In this research, the recrystallized grain sizes of the alloys with similar components are similar, regardless of the casting methods. The grains of 1060 series aluminium alloys are coarser than those of 1100 series aluminium alloys, as shown in Table 6.

Homogenization of TRC alloys usually leads to the coarsening of grain and secondary phase particles, while the microstructure of DCC alloys is only slightly affected by the homogenization. The microstructure of 1100-0.08Cu alloys after homogenization at different temperatures for 10 h is shown in Fig.3. Homogenization at 550~570 °C barely affects the microstructure, so the as-cast microstructure basically

Table 4 Tensile properties of 1060-0.04Cu aluminium foil with different intermediate annealing thicknesses

Intermediate annealing thickness/mm	Yield strength/MPa	Ultimate tensile strength/MPa	Elongation/%
0.5	172±1.0	186±4.0	2.5±0.3
1.5	183±0.8	215±2.0	3.2±0.1
4.0	196±2.1	223±2.6	4.2±0.2

Table 5 Tensile properties of aluminium foil after storage at ambient temperature

Alloy	Strain	Storage duration/d	Yield strength/MPa	Ultimate tensile strength/MPa	Elongation/%
DCC/1060-0.04Cu	5.7	0	196±2.1	223±2.6	4.2±0.2
		2	189±0.5	219±1.5	3.9±0.3
		4	186±0.1	217±1.0	3.8±0.3
TRC/1100-0.26Si	5.5	0	218±2.3	243±1.2	4.0±0.2
		2	216±0.9	239±0.1	3.8±0.1
		4	215±1.0	238±0.3	3.6±0.1
		8	213±0.9	236±2.8	3.4±0.2
		16	211±0.2	236±2.9	3.3±0.1

Table 6 Recrystallized grain sizes of 1060 and 1100 series aluminium alloys after TRC and DCC

Alloy	Treatment	Average grain size/μm
1060	DCC	33.4±1.5
	TRC	30.2±2.1
1100	DCC	21.3±1.3
	TRC	23.2±0.9

remains.

It can be observed that coarse intermetallic compounds are formed on grain boundaries of 1100-0.08Cu alloy ingots after DCC, and they are only slightly refined, even after homogenization at high temperatures of 580~590 °C. After homogenization at 600 °C, the coarse phases become dispersed particles of micron scale. These grain boundary phases are broken into much smaller particles with less than 10 μm in size during the subsequent hot rolling.

The OM microstructures of hot-rolled DCC 1XXX series aluminium alloys are shown in Fig.4. It can be observed that most grains are elongated into fibrous structure, which is similar to those of the cold-rolled alloys. Some small recrystallized grains can be observed on the grain boundaries

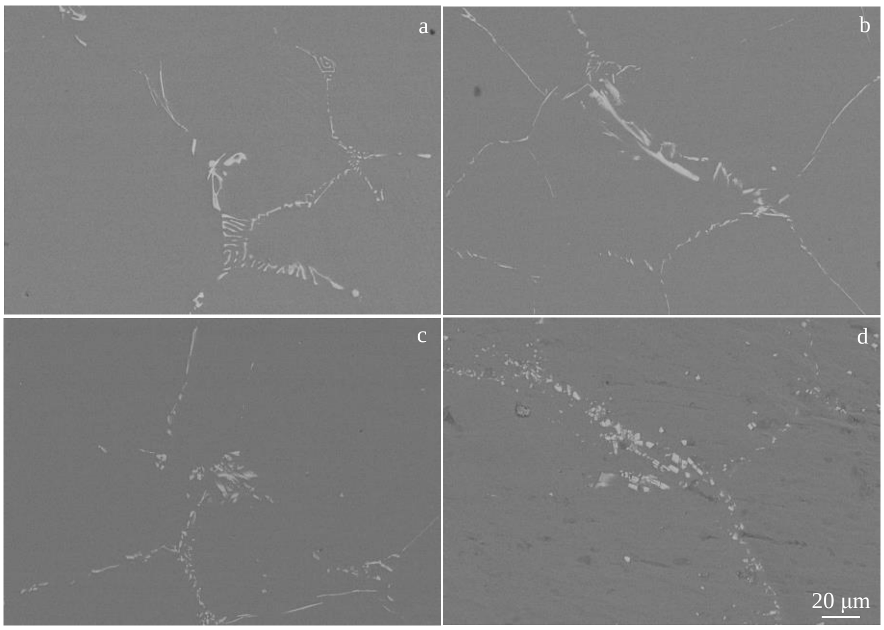

Fig.3 SEM microstructures of 1100-0.08Cu alloys after homogenization at different temperatures: (a) as-cast, (b) 580 °C, (c) 590 °C, and (d) 600 °C

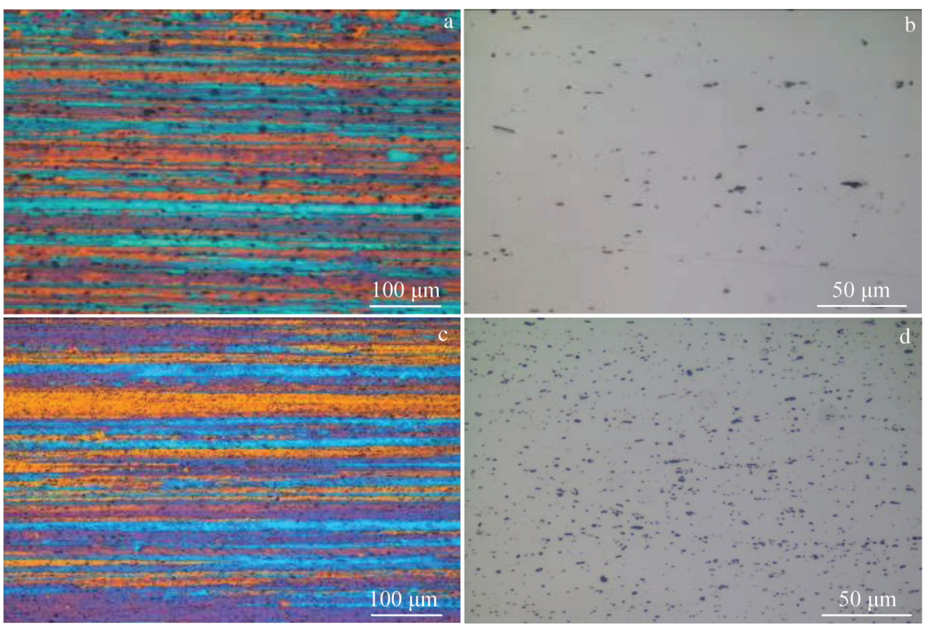

Fig.4 OM microstructures of grains (a, c) and intermetallics (b, d) of hot-rolled DCC 1060-0.04Cu (a, b) and 1100-0.04Cu (c, d) aluminium alloys

between the deformed grains in Fig. 4. However, under the industrial hot rolling conditions, the applied large strain is over 4.4 and the temperature decreases by over 130 °C (from 480 °C to below 350 °C), resulting in the fact that the hot-rolled microstructure of 1XXX series aluminium alloys is different from the typical dynamically recrystallized microstructure. A much higher density of intermetallic can be observed in Fig. 4d due to the increased Fe content in 1100 series aluminium alloys. The solubility of Fe is extremely low in Al matrix (0.05wt% at the eutectic temperature, negligible at room temperature), so Fe atoms can form the secondary phases, such as θ-$Al_{13}Fe_4$[1].

The recrystallized grain structures of cold-rolled plates are mainly equiaxed grains after intermediate annealing. The equiaxed grains are then drastically elongated after repeated rolling until achieving the final thickness, forming a lamellar structure, as shown in Fig.5.

It is noteworthy that the secondary phase particles are not further broken during the subsequent cold rolling, even after the application of large strains. The intermetallic particles (less than 3 μm in diameter) in aluminium foil after TRC are generally finer than those after DCC.

3.2 Factors affecting the strength

As the age hardening is not considered in Al-Fe-Si alloys, the precipitation strengthening is not available in these alloys. The primary strengthening mechanisms are solid-solution strengthening, dislocation strengthening, dispersoid strengthening, and (sub)grain-boundary strengthening. The solid-solution strengthening effect of different solute atoms is summarised in Fig.6[12].

However, the results in this research are not in agreement with Ref. [12]. Only the increase in Cu content can dramatically improve the tensile strength of the alloys, while

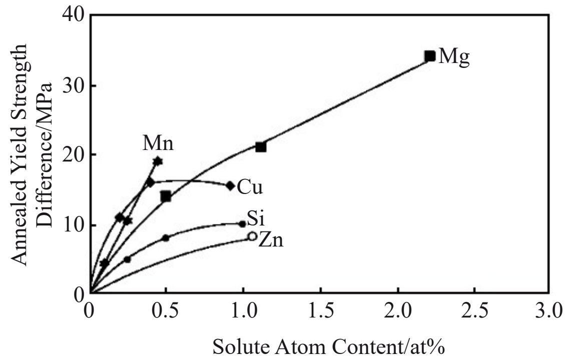

Fig.6 Solid-solution strengthening effect of solute atoms in aluminium alloys[12]

the increase in Si and Mn contents does not have an obvious effect on the tensile strength. This can be explained by the fact that Si or Mn addition results in grain boundary phases containing Si or Mn, as shown in Fig. 7. As a result, only a small number of Fe, Si, or Mn atoms remain in the matrix as solute atoms. Besides, little Cu can be observed in the intermetallic. Thus, it can be concluded that most Cu atoms are dissolved in the matrix.

The increase in Fe, Si, and Mn contents can generate more secondary phase particles. A higher density of the secondary phase particles can slightly increase the tensile strength of 1060 and 1100 series aluminium alloys with the same Cu content. The dispersoid hinders the dislocation movement as it cannot be sheared by dislocations, according to Orowan mechanism. In dilute aluminium alloys, the dispersoid strengthening is relatively weak, as the dispersoid is generally over 1 μm in size, as shown in Fig.5.

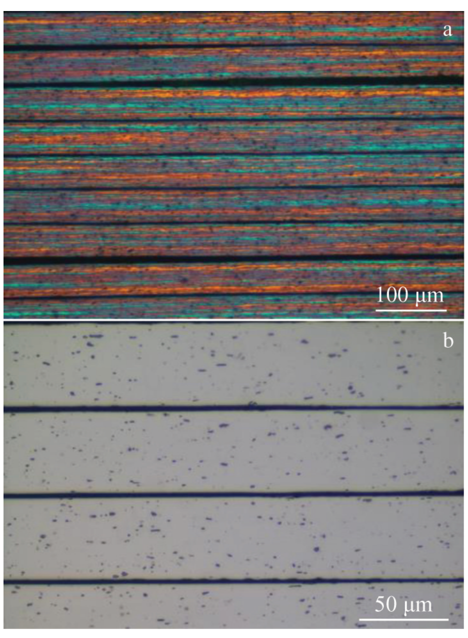

Fig.5 Grain (a) and intermetallic (b) microstructures of cold-rolled 1100-0.08Cu alloys after DCC with 48 μm in thickness

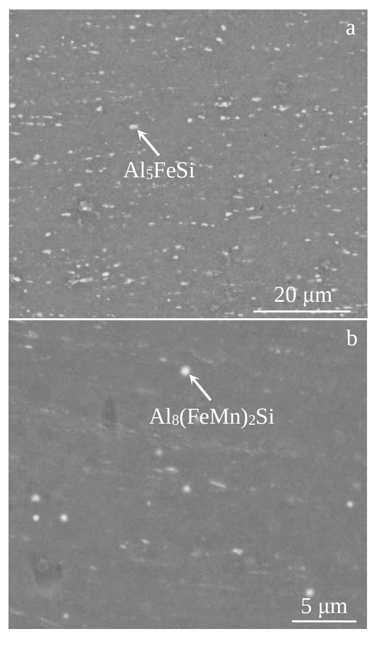

Fig.7 Morphologies of secondary phase particles with Fe and Si addition in 1100-0.26Si alloy (a) and Fe, Mn, and Si addition in 1100-0.20Mn alloy (b) after TRC

The changes of strain hardening rate are in agreement with existing theories that the strain hardening rate is mainly controlled by the dislocation mechanisms[13]. The dislocation density is firstly increased with increasing the strain until it reaches saturation and the multiplication of dislocations is in balance with the annihilation of dislocations[13]. In addition, after application of large strains, the mechanically assisted triple junction motion in the lamellar morphology is an important contributor to dynamic recovery, leading to an almost steady state[14].

The decrease in tensile strength of aluminium foil after storage at room temperature is similar to the results in Ref. [15]: a lamellar structure is formed in a dilute AA1050 aluminium alloy after application of large strain of 5.5, and the alloy hardness is decreased from 504 MPa to 456 MPa at room temperature for over 1000 h. The corresponding microstructure change mechanism is attributed to the operation of different recovery mechanisms at low temperature, including annihilation of dislocations, sub-grain growth by coalescence, boundary migration, and the triple junction motion[15,16]. The microstructure investigation for the room temperature softening of the aluminium foil should be further discussed.

3.3 Elongation

Generally, the stress or strain concentration may result in reduced plasticity, such as strain hardening or coarse intermetallic particles. The increase in Cu content decreases the elongation, because the Cu solute atoms facilitate the strain hardening of the alloy. This can also explain the high elongation of 1100 series aluminium alloys after TRC, whose intermetallic particles are obviously finer (<3 μm in diameter) than those after DCC (8~10 μm), as shown in Fig.5. However, in the aluminium foil with larger intermediate annealing thickness, the greater the strain hardening, the larger the elongation, which is contrary to the fact that larger strain is harmful to tensile plasticity of the alloys. The effect of intermediate annealing thickness on the elongation can be attributed to the increase in the ratio of thickness to grain diameter (T/D) after intermediate annealing, as the recrystallized grain sizes barely change under different strains, as shown in Table 7.

Further cold rolling will not significantly affect T/D ratio, as the aspect ratio of the grains will increase in accordance with the changes in the macroscopic dimension. It is reported that both the specimen size and grain diameter play important roles in the tensile behaviour of thin metal sheets and foil[8,17-19]. Lederer et al[8] found that the aluminium foil with higher T/D ratio demonstrates a higher ultimate tensile strength. Critical values of T/D were discussed in Ref.[17,18]: the elongation is increased with increasing the T/D value as long as it is below the critical T/D value. Yang et al[18] found that the critical T/D value depends not only on the specimen thickness, but also on the average grain size of specimen. When T/D is larger than the critical value, the specimen is sufficiently thick with abundant grain boundaries to trap the dislocation. It can be inferred that for aluminium foil with the thickness of 13 μm, a higher value of T/D is beneficial to the strength-plasticity combination. The 1100 series aluminium alloys have a better elongation than 1060 series aluminium alloys do, because the T/D value is increased from 45 to 70 due to grain refinement along the thickness direction. It is noteworthy that the grain refinement does not necessarily benefit the elongation[17,20].

Table 7 Average grain size and T/D value of 1060 series aluminium alloys after annealing at different thicknesses

Intermediate annealing thickness, T/mm	Average grain size, D/μm	T/D
0.5	31.2±2.6	16
1.5	33.4±1.5	45
4.0	31.3±1.9	128

The effect of ambient temperature storage on the plasticity of aluminium alloy is not mentioned in Ref. [14-16, 21]. However, in heavily deformed metals, the recovery mechanism, such as triple junction motion, can cause the uniform coarsening of deformed lamellar microstructures, leading to a more equiaxed morphology[21], resulting in the decrease in T/D value, which may be detrimental to the plasticity of the alloys.

4 Conclusions

1) The direct chill casting (DCC) and twin roll casting (TRC) have little effect on the tensile strength of 1XXX series aluminium foil. The elongation of aluminium foil after TRC is slightly higher than that after DCC, because of the finer intermetallic in aluminium foil caused by TRC.

2) Homogenization at temperatures above 580 °C can slightly refine the coarse intermetallic at the grain boundaries in alloy ingots after DCC. The grain boundary phases are broken into particles of ≤10 μm in size during hot rolling, and the further cold rolling barely reduces the particle size.

3) The increase in Fe, Si, and Mn contents is not effective in increasing the tensile strength of the aluminium foil since these elements mainly exist in the intermetallic. Cu addition can significantly increase the strain hardening at strain≥2.5, thus increasing the tensile strength of thin aluminium foil.

4) The ratio of thickness to grain diameter (T/D) is basically unchanged during the cold deformation after annealing. The larger the T/D ratio value, the larger the elongation of the thin aluminium foil, which is attributed to abundant grain boundaries along the thickness direction.

5) Thin aluminium foil suffers deterioration of tensile properties during storage at room temperature, which is attributed to the recovery mechanism at room temperature after application of large strains.

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合金元素及制备工艺对1XXX系铝合金组织及性能的影响

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摘　要：通过直接激冷铸造（DCC）与双辊铸轧（TRC）方法生产了具有不同Fe、Si、Cu和Mn含量的1XXX系铝合金坯料，随后通过均匀化、热轧、冷轧和退火等加工工艺制备了厚度为13 μm的铝箔。结果显示，铸造工艺对铝箔强度影响较小，双辊铸轧引入的较细第二相有利于铝箔塑性。在580 ℃以上进行均匀化处理可细化直接激冷铸造铸锭的粗大晶界金属间化合物，并使其在后续热轧中破碎为小颗粒。因Cu溶质原子可提高加工硬化速率，添加Cu元素的效果优于添加Fe、Si和Mn元素。中间退火时铝箔厚度对力学性能有显著影响，这和厚度与晶粒尺寸之间的比值有关。室温储存会导致较薄铝箔的力学性能下降，这与大应变后发生的回复机制有关。

关键词：铝合金；拉伸性能；第二相；晶粒组织

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