China Steel Technical Report, No. 24, pp. 56－63, (2011)

A Study of the Microstructure, Texture and Sputtering Properties of 5N High-purity Aluminum Target

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This investigation studies the effects of the microstructure and texture on the sputtering properties of a high purity aluminum target. In this study, the purity of aluminum target is as high as 99.999% (5N). The 5N aluminum ingot first underwent hot-rolling and heat treatment, and was then cut into different kinds of test specimens. A series of microstructural observations, texture analyses and sputtering experiments were conducted to study the properties of the 5N aluminum target. The research results show that the 5N aluminum target experiences dynamic recovery during hot-rolling due to its high stacking fault energy. Heat treatment at the proper temperature could help the 5N aluminum target complete recrystallization, enabling the target to possess a fine grain and cube texture. In addition, since the purity of the aluminum target is as high as 99.999%, any slight temperature deviation during hot-rolling and heat treatment would affect the microstructure and texture of the aluminum target. The results of the sputtering experiments show that any undesirable microstructure or texture of the aluminum target would influence its sputtering rate and the properties of the aluminum thin film after sputtering. A fine grain and cube texture are beneficial to the 5N aluminum target by permitting an increased sputtering rate, and allowing the aluminum thin film to possess a low electrical resistance after sputtering. Moreover, hillocks would appear on those aluminum thin films produced by an aluminum target with low sputtering rate at lower temperature, causing short circuits in the LCD panel. Therefore, to improve the sputtering rate of 5N aluminum target in order to obtain aluminum thin film with a low electrical resistance and excellent anti-hillock properties, the temperature of the hot-rolling and heat treatment must be controlled precisely to ensure that the aluminum target possesses a fine grain and cube texture.

Keywords: 5N aluminum target, Sputtering rate, Texture, Hillock

1. INTRODUCTION

The Liquid Crystal Display (LCD) was developed in the late 1960s and early 1970s. With continuous progress and improvements, the applications of LCD involve cell phones, PDAs, navigators, laptops and LCD panels. The LCD has become a common product for daily use in our modern life.

5N aluminum has been used as the main material for conducting lines within the LCD panel due to its low electrical resistance. 5N aluminum target is sputtered onto glass to form aluminum thin film. Via a series of exposure, developing and etching, the aluminum thin film is manufactured into conducting line, playing a crucial role in transmitting signals within the LCD panel. If the electrical resistance of the aluminum thin film is too high, the transmission of electrical signals is delayed, causing a motion blur problem as films containing high speed motion are played. The electric resistance of the aluminum thin film is closely related to the sputtering rate of the aluminum target. Under the same sputtering conditions, aluminum target with a low sputtering rate produces thinner aluminum thin film. In general, the electric resistance of the thin aluminum thin film is higher than that of the thick one, affecting the transmission of the electrical signal[1-2].

It has been known that the sputtering rate of the target mainly depends on its microstructure and texture. Undesirable microstructure and texture deteriorate the sputtering rate of the target[3-4]. However, until now, there have been limited studies on the effect of microstructure and texture on the sputtering properties of a 5N aluminum target, and further study is certainly necessary and helpful for a better understanding of the mechanism leading to a 5N aluminum target possessing a high sputtering rate. Therefore, this work investigates the effects of the microstructure and texture of a 5N aluminum target on its sputtering properties to obtain the optimal parameters for making high quality 5N aluminum targets.

2. EXPERIMENTAL METHOD

2.1 Material Preparation

An aluminum target, whose purity is as high as 99.999%(5N), was investigated in this study. The composition of the 5N aluminum target is listed in Table 1. The high purity aluminum ingot was heated at 500°C for 2 hours and then hot-rolled with total reduction rate of 80%. After hot-rolling, the aluminum plates were heat-treated at 100, 200, 300, 400 and 500°C for 1 hour respectively. The heat-treated aluminum plates were prepared for microstructural investigation, texture analysis, and experimental sputtering.

2.2 Microstructral Investigation

Metallographical investigations were carried out using optical microscopy (OM), transmission electron microscopy (TEM) and electron back-scattered diffraction (EBSD). The specimens were mechanically polished with wet SiC paper of 2000 grit and then finely polished with 0.05um Al_2O_3 powder to enable optical observation to be made. Finally, an etching solution consisting of 5ml of HBF_4 and 95ml of H_2O was used to electrically etch the specimens for 10 minutes. The microstructure of the high purity aluminum targets were examined by OM. The morphology of the subgrain and of the dislocation existing in the heat-treated aluminum targets were observed by TEM. The high angle and low angle grain boundaries of the heat-treated aluminum targets were investigated by EBSD.

2.3 Texture Investigation

After hot-rolling and heat treatment, the aluminum targets were manufactured into square specimens of 15mm in length and width. D500X was used to measure the (111), (200) and (220) pole figure and to calculate the orientation distribution function (ODF), in order to clarify the effect of heat treatment on the orientation of the aluminum grain.

2.4 Sputtering Experimental

To investigate the effects of microstructure and texture on the sputtering rate of the aluminum target and on the properties of the aluminum thin film, the hot-rolled plates were manufactured into circular targets with 21.2mm diameter and 6mm thickness, and then bonded with a Cu back plate to conduct the experimental sputtering. The aluminum targets were sputtered on the glass substrate with different sputtering parameters to form aluminum thin films. The working power was set for 500W, and the sputtering time was set for 120, 240, and 360seconds respectively. The α-step was used to measure the thickness of each aluminum thin film. The thickness of each aluminum thin film was divided by its own sputtering time to obtain the individual sputtering rates. Finally, three sputtering rates from different aluminum thin films were averaged to obtain the sputtering rate for each aluminum target.

3. RESULTS AND DISCUSSION

3.1 Effect of Hot-rolling and Heat Treatment on the Microstructure of a 5N Aluminum Target

OM images of 5N aluminum targets hot-rolled at different temperatures reveal that many strip-like deformation grains exist in the 5N aluminum target when the temperature of hot-rolling is 150°C, as shown in Fig.1(a). As the temperature of hot-rolling increases to 250°C, not only do the strip-like deformation grains disappear and the grain size decrease, but also the grain uniformity improves obviously, as shown in Fig.1(c). The grain size of a 5N aluminum target hot-rolled at 250°C is about 120um. When the temperature of hot-rolling increases to 300°C or higher, the grain of the aluminum target is larger than 500um. In this research, the purity of aluminum target was as high as 99.999% so that no impurity could drag the grain boundary. Therefore, any slight temperature deviation of hot-rolling would greatly affect the microstructure of the aluminum target. For a 5N aluminum target, the optimum microstructure could be obtained only when the temperature of hot-rolling is controlled at 250°C. An innapropriate temperature of hot-rolling would cause an undesirable microstructure to appear in the 5N aluminum target. Too high a temperature of hot-rolling would bring about coarse grain, whereas too low a temperature of hot-rolling would cause strip-like deformation in the grain.

Figure 2 shows the OM images of 5N aluminum targets hot-rolled at 250°C, and then heat-treated at various temperatures for 1hour. The OM images show that the grain size of the aluminum targets decreases initially and then increases with an increasing heat treatment temperature. This phenomenon closely relates with the high stacking fault energy of 5N aluminum target.

The stacking fault energy (SFE) is expressed as $\sigma_{SFE} = \dfrac{Gb^2}{2\pi d(1-\nu)}$, so stacking fault energy is inversely proportional to the space of stacking fault area (d) (5-6). The space of stacking fault area of 5N aluminum is quite small, because its stacking fault energy is as high as 200erg/cm^2. During hot-rolling, the partial dislocation in the 5N aluminum target would combine into the perfect dislocation to cross-slip to another slip plan due to its small stacking fault area. Therefore, a dynamic recovery of the 5N aluminum tends to occur during hot-rolling(7-9).

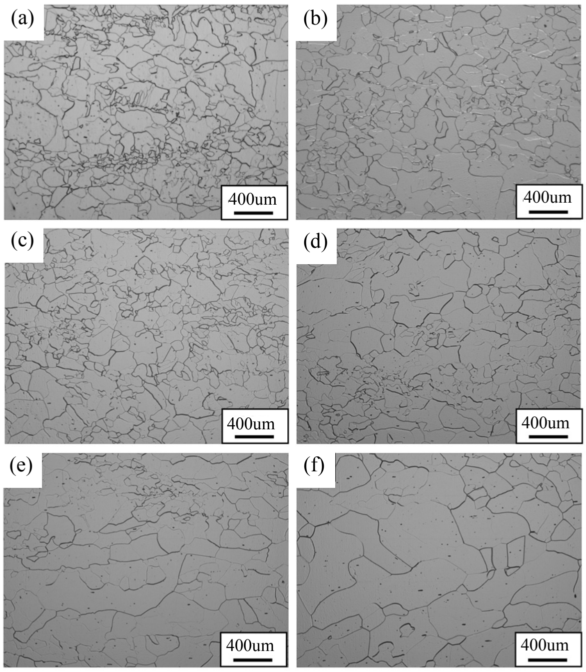

Fig.1. OM images of 5N high purity aluminum hot-rolled at various temperature. (a) 150°C, (b) 200°C, (c) 250°C, (d) 300°C, (e) 325°C, (f) 350°C.

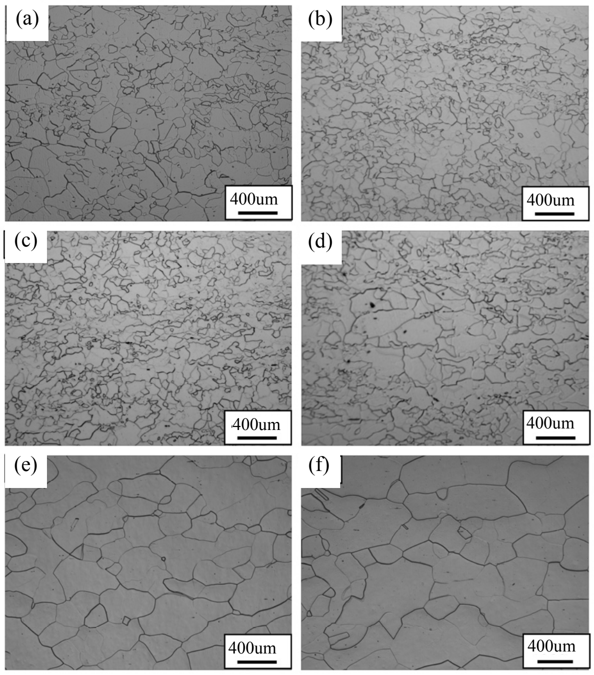

Fig.2. OM images of 5N high purity aluminum hot-rolled at 250°C, and then heat-treated at various temperature for 1 hour. (a) as-rolled, (b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, (f) 500°C.

The driving force for both the recovery and recrystallization is the release of the strain energy stored in the plastic material. Recovery and recrystallization thus compete with each other because their driving forces are the same(10). As recovery happens during hot-rolling, a part of the strain energy is released leading to the recrystallization that could not previously occur(5-7,10). The heat-treatment after hot-rolling can release the residual strain energy existing in the aluminum target, prompting the 5N aluminum target to complete the recrystallization. Therefore, the grain size of the hot-rolled aluminum target decreases after subjecting the target to a 100°C or 200°C heat-treatment for 1 hour. However, significant coarse grain was observed in the 5N aluminum target once the temperature of heat-treatment exceeded 300°C because grain growth occurs in the 5N aluminum target.

The EBSD images of heat-treated aluminum targets, as shown in Fig.3, reveal that the amount of high angle grain boundary decreases and the amount of low

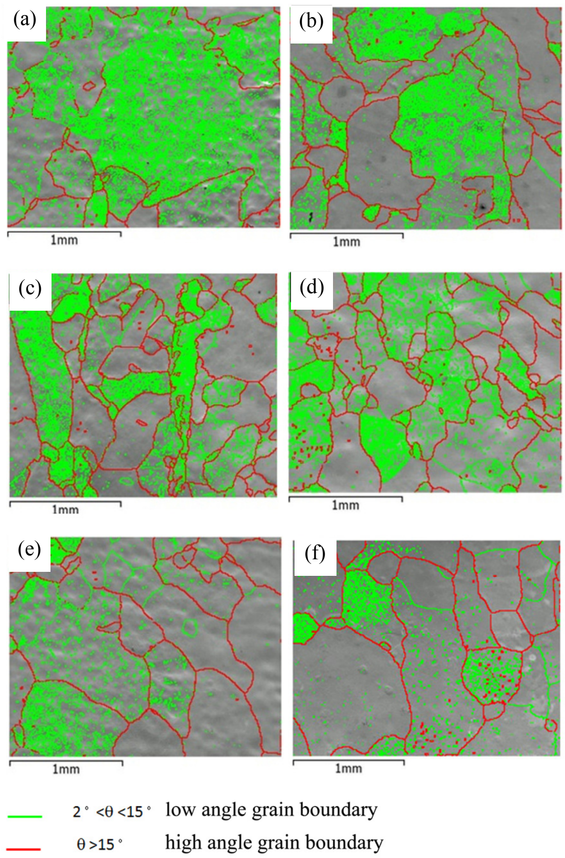

Fig.3. EBSD images of 5N high purity aluminum hot-rolled at 250°C, and then heat-treated at various temperature for 1 hour. (a) as-rolled, (b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, (f) 500°C.

angle grain boundary increases with an increasing temperature of heat treatment, demonstrating that the grain refining of the aluminum target after heat treatment is due to recrystallization. This recovery of the grain refining occurs as dislocation and rearranges the grains into a low energy dislocation structure (LEDs) by slip, climb and cross slip(5). With the ongoing process of recrystallization, the subgrain with low angle grain boundaries combine with each other by the rotation of subgrains or the migration of grain boundaries to form a subgrain with high angle grain boundaries(5).

Moreover, brass and copper textures are the deformation textures often appearing in hot-rolled or cold-rolled aluminum(11-12). Cube texture is the only texture appearing in the 5N aluminum as the 5N aluminum completes recrystallization(13-15). Therefore, for 5N aluminum, a cube texture can be regarded as the signal of full recrystallization. The ODFs (Φ_2=45°) of heat-treated 5N aluminum targets, as shown in Fig.4, reveal that with an increasing heat treatment temperature, the intensity of the cube texture is strengthened, whereas the intensity of brass and copper texture is weakened.

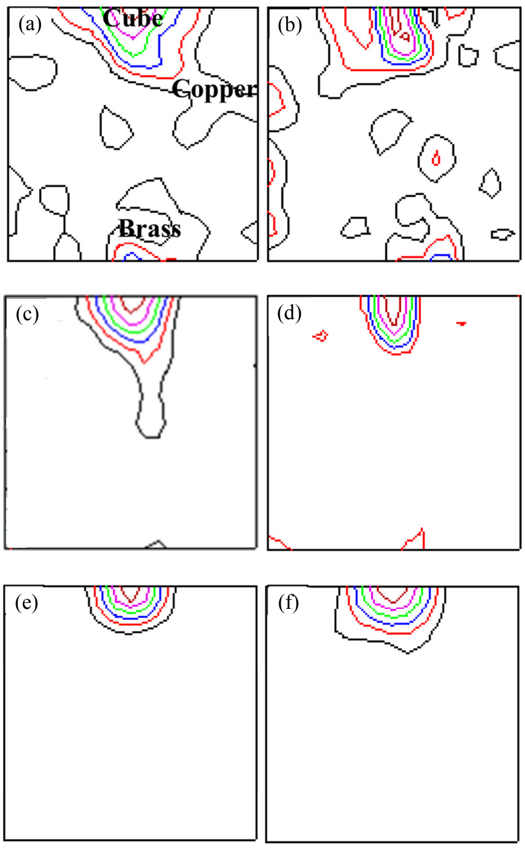

Fig.4. The ODFs (Φ_2=45°) of 5N high purity aluminum hot rolled at 250°C, and then heat-treated at various temperature for 1 hour. (a) as-rolled,(b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, (f) 500°C.

The above result indicates that a dynamic recovery takes place in the 5N aluminum target during hot-rolling. Even during hot-rolling at high temperature, no recrystallization occurs. This is due to the high stacking fault energy of 5N aluminum which facilitates the recovery process, leading to a decrease in the driving force for recrystallization(6, 8-9). As a result, brass and copper deformation textures exist in the aluminum target because full recrystallization does not occur. The heat treatment can release the residual strain existing in the 5N aluminum target, prompting the 5N aluminum target to complete the recrystallization process.

The observations of TEM images, as depicted in Fig.5, show that the amount of subgrain and dislocation in aluminum targets decrease with the heat treatment temperature. This result also demonstrates that heat treatment can make hot-rolled 5N aluminum targets complete the recrystallization process.

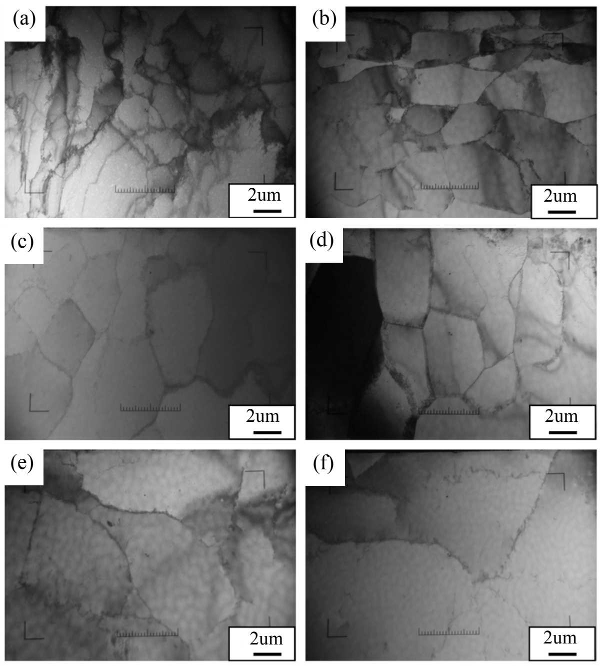

Fig.5. TEM images of 5N high purity aluminum hot-rolled at 250°C, and then heat-treated at various temperature for 1 hour. (a) as-rolled,(b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, (f) 500°C.

3.2 Effect of Microstructure and Texture on Sputtering Properties of High-purity Aluminum Target

The LCD panel suffers from a motion blur problem when the electrical resistance of the aluminum thin film is too high to transmit the electrical signal at sufficient speed. The electrical resistance of an aluminum thin film is closely related to the sputtering rate of the aluminum target. It is known that the electrical resistance of a thinner aluminum thin film is higher than that of a thick one(1-2). Therefore, if the sputtering rate of the aluminum target is too low, the thickness of the aluminum thin film would be inadequate after sputtering under the same sputtering conditions, leading to the aluminum thin film possessing a high electrical resistance. To improve the sputtering properties of a target through controlling microstructure and texture, sputtering experiments are used to investigate the effect of the microstructure and texture of a target on its sputtering properties.

Figure 6 shows the relationship between the sputtering rate and the hot-rolling temperature of 5N aluminum targets. The sputtering rate of a 5N aluminum target hot-rolled at 150°C is just 27.57Å/sec. In such a target, strip-like deformation grains and a deformation texture were observed, and the difference between each grain was quite large. As the temperature of hot-rolling increases to 250°C, the deformation grains disappear and are replaced by a fine grain, and its sputtering rate increases to 29.75Å/sec. However, when the hot-rolling temperature increases to 325°C or higher, the grain size of the aluminum target is over 500μm due to grain growth leading to the sputtering rate significantly decreasing to 28.50Å/sec or lower.

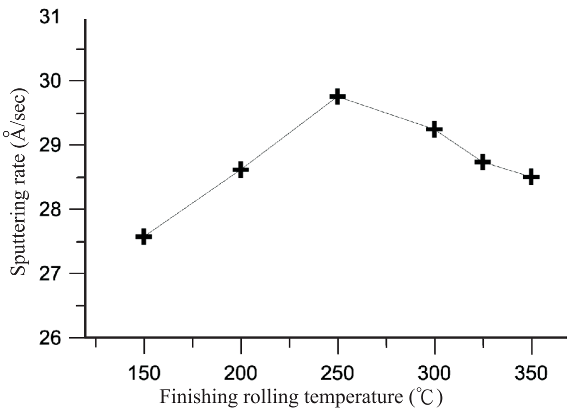

Fig.6. Correlation diagram between sputtering rate and finishing rolling temperature of 5N high purity aluminum.

The relationship between the sputtering rate, the heat treatment temperature, the grain size and the texture intensity, as exhibited in Fig.7, reveal that the sputtering rate initially increases and then decreases with any increase in the heat treatment temperature. After heat treatment at 200°C for 1 hour, not only does the grain size of the 5N aluminum target decrease, but also the brass deformation texture disappears and the cube texture intensity increases due to the full recrystallization of the 5N aluminum target, resulting in an increase of the sputtering rate of the 5N aluminum target from 29.75Å/sec to 30.82Å/sec. However, as the heat treatment temperature is increased to 300°C or higher, the sputtering rate of the 5N aluminum target decreases to 29.13Å/sec or lower due to the coarse grain resulting from the significant grain growth. These results demonstrate that “fine grain” and “cube texture” are beneficial to increasing the sputtering rate of 5N aluminum target.

In general, fine-grained targets provide a large grain boundary to grain volume ratio. It is believed that

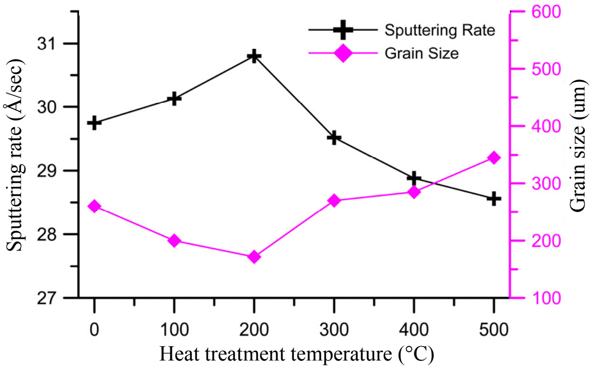

Fig.7. Correlation diagram between sputtering rate, heat-treatment temperature and grain size of 5N high purity aluminum hot-rolled at 250°C.

the atoms at the grain boundaries of the target material are more easily bombarded and ejected to form thin film on the substrate because of their weaker bonding force compared to the interior atoms of crystal lattices(3-4). As a result, fine-grained targets always possess high sputtering rates.

Moreover, from the standpoint of thermodynamics, the sputtering process is like the precipitation process. In both processes, atoms must overcome the attractive force from other surrounding atoms to precipitate a new phase or deposit on the substrate. Both processes are thermal activation, and follow the Arrhenius equation(5). Therefore, not only is the precipitation rate proportional to $\exp\frac{-G}{RT}$, but also to the sputtering rate.

During the sputtering process, in order to depart from the matrix phase, firstly, the atoms need to overcome the attractive force from the surrounding atoms to be deposited on the substrate, as shown in Figure 8. The activation energy atoms must overcome during this procedure can be expressed as $G_{migration}$. In addition, the strain field hinders atoms from migrating to the substrate during sputtering, as shown in Fig.8. Therefore, once atoms depart from the matrix and begin to migrate to the substrate, atoms need to overcome one more activation energy, G_{strain}. As a result, the sputtering rate of target is proportional to $\exp\left[\frac{-(G_{migration}+G_{strain})}{RT}\right]$.

The residual strain existing in the aluminum target increases the activation energy that atoms need to overcome to migrate to the substrate. When the G_{strain} increases, the sputtering rate of the aluminum target will decrease. Therefore, as the 5N aluminum target is hot-rolled at low temperature, leading to lots of deformation grains appearing in the target, the sputtering rate of the aluminum target will be quite low.

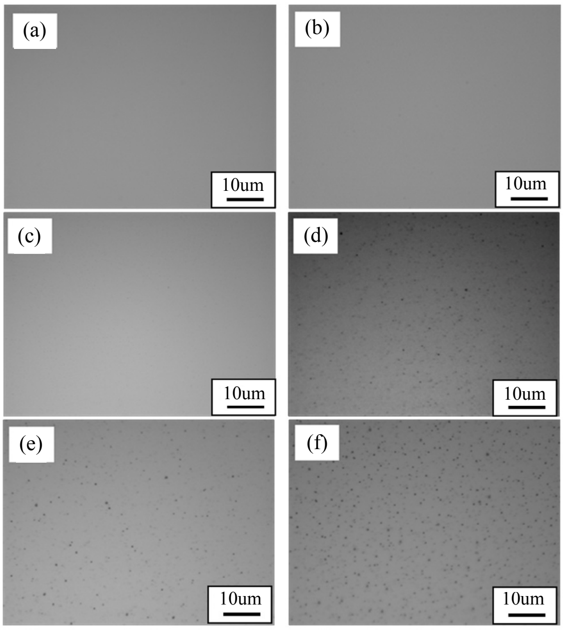

Fig.8. OM images of the thin films, sputtered by the 5N high purity aluminum target with sputtering rate 29.75Å/sec, were heat treated at various temperature for 1 minute. (a) without heat-treatment, (b) 150°C, (c) 200°C, (d) 250°C, (e) 300°C, (f) 400°C.

Cube texture is the sole texture appearing in the full recrystallization of 5N aluminum[3,13,17,19]. For the 5N aluminum target, the appearance of cube texture and the disappearance of any brass or copper texture can be regarded as the 5N aluminum target having completed the recrystallization process and turned into a strain-free structure, resulting in a G_{strain} decrease. Without the impediment of the strain field, atoms can more easily migrate to the substrate during sputtering, leading to an aluminum target that possesses a high sputtering rate. Accordingly, cube texture is beneficial to increasing the sputtering rate of the 5N aluminum target.

3.3 Effect of Sputtering Rate of 5N Aluminum Target on Properties of Aluminum Thin Film

During the LCD fabrication process, the metallic layers are subjected to thermal cycles. The differences in the coefficients of thermal expansion between the metallic layers and substrates give rise to large thermal stresses in the thin film layer. These stresses may result in the formation of hillocks which can cause reliability problems such as dielectric cracks or short circuits to LCD panel. 5N aluminum thin film is qualified for use as conducting line in an LCD panel due to its low electrical resistance. However, the yield strength of 5N aluminum thin film is so low that aluminum thin film forms hillocks more easily than other kinds of thin film alloys when the aluminum thin film is subject to thermal stress.

Microstructure observations of different aluminum thin films heat-treated at various temperatures reveal that hillocks appear on the aluminum thin film sputtered by the aluminum target with a sputtering rate of 29.75Å/sec at 250°C, as shown in Fig.8. As the aluminum target was sputtered with a sputtering rate of 27.57Å/sec and 16.93Å/sec to produce the aluminum thin film, the temperature of the hillocks appearing on the surface of aluminum thin film decreases to 180°C and 150°C respectively, as shown in Fig.9. The above results show that the lower the sputtering rate the aluminum target possesses, the easier it is for hillocks to appear on the aluminum thin film after sputtering. In general, when the sputtering target with a low sputtering rate is used to sputter the thin film, it takes a longer time to obtain a thin film with adequate thickness leading to more Ar ions permeating into the aluminum thin film during sputtering, thereby increasing the stress in the aluminum thin film[20]. To release the stress, the aluminum thin film tends to form hillocks at high temperature. Therefore, the sputtering rate of aluminum target not only affects the thickness of the aluminum thin film after sputtering, but also affects the stability of the aluminum thin film at high temperature.

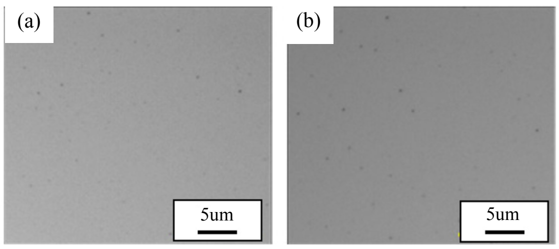

Fig.9. OM images of the thin films, sputtered by the 5N high purity aluminum target with sputtering rate 27.57Å/sec, were heat treated at various temperature for 1 minute.(a) 150°C, (b) 180°C.

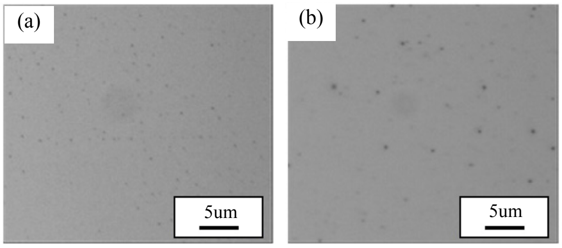

Fig.10. OM images of the thin films, sputtered by the 5N high purity aluminum target with sputtering rate 16.93Å/sec, were heat treated at various temperature for 1 minute.(a) 100°C, (b) 150°C.

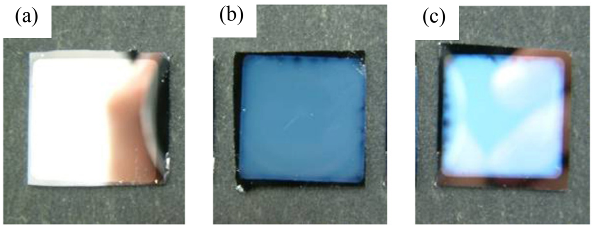

Fig.11. The appearance of aluminum thin film which were heat-treated at various temperature for 1 minute. (a) without heat-treatment, (b) 300°C, (c) 400°C.

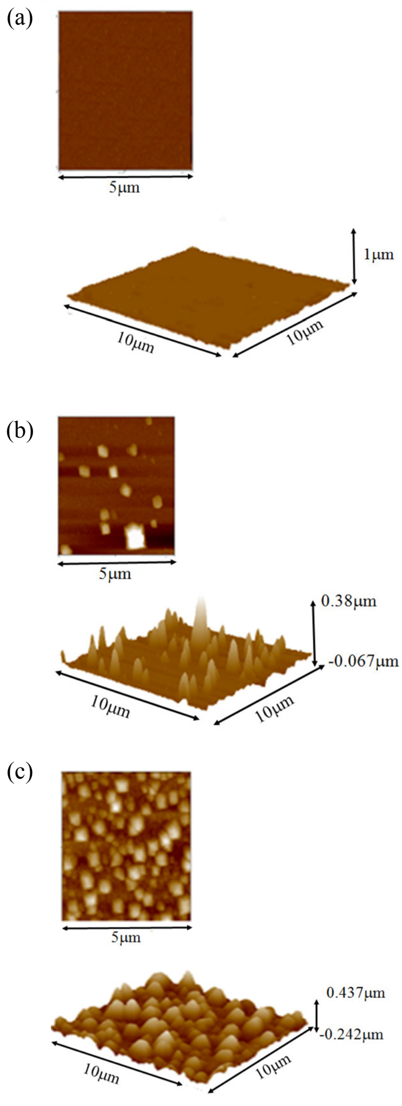

Fig.12. The AFM observations of aluminum thin film which were heat-treated at various temperature for 1 minute. (a) without heat-treatment, (b) 300°C, (c) 400°C.

Moreover, the aluminum thin film on the substrate is quite shiny before heat-treatment. After heat-treatment at 400°C, the shiny aluminum thin film turns into white-gray. The results of the AFM image show that the aluminum thin film is quite flat and no hillock can be observed before heat-treatment. The flat aluminum thin film can reflect the light effectively, so the aluminum thin film is shiny. However, with the increasing temperature of the heat treatment, the amount of hillocks on the surface of the aluminum thin film increases, leading to an increasing roughness of the aluminum thin film. The rough surface of the aluminum thin film makes the light scatter in a disorderly manner, resulting in the white-gray appearance of aluminum thin film.

4. CONCLUSIONS

(1)The purity of aluminum target is as high as 99.999% so that no impurity could drag the grain boundary. Even a small temperature deviation in the hot-rolling and heat-treatment processes would greatly affect the microstructure and texture of the aluminum target.

(2)Dynamic recovery takes place in the 5N aluminum target during hot-rolling due to its high stacking fault energy. Heat treatment can release the residual strain existing in the 5N aluminum target, prompting the 5N aluminum target to complete its recrystallization. As the 5N aluminum target completes the recrystallization process, the sputtering rate increases significantly.

(3)Any undesirable microstructure or texture of the aluminum target will influence its sputtering rate and the properties of the aluminum thin film after sputtering. Fine grain and cube texture are beneficial to increasing the sputtering rate of the 5N aluminum target.

(4)The sputtering rate of the aluminum target not only affects the thickness of the aluminum thin film after sputtering, but also affects the stability of the aluminum thin film at high temperature. The lower a sputtering rate an aluminum target possesses, the easier it is for hillocks to form on the aluminum thin film after sputtering.

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