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# Thin Solid Films



# Effects of deposition conditions of the Al film in Al/glass specimens and annealing conditions on internal stresses and hillock formations

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#### ARTICLE INFO

Article history: Received 3 March 2011 Received in revised form 7 December 2011 Accepted 11 December 2011 Available online 16 December 2011

*Keywords:* Hillock Stress Microstructure Aluminum film

#### ABSTRACT

In the present study, 25 kinds of specimen with five Al-film thicknesses were prepared to investigate the relation between the internal stress formed during the annealing process and the hillocks. In the preparation of specimens, the governing factors including deposition conditions, annealing temperature, and annealing time, were arranged following the orthogonal table of five-level and six-factorial ( $L_{25}(5^6)$ ) design. Stoney's formula is applied to describe the internal stresses before and after annealing ( $\sigma_0$  and  $\sigma_f$ ), respectively. The internal stress arising during the annealing process ( $\sigma_{an}$ ) is evaluated using the model developed by Flinn et al. [1]. Then, the response surface methodology (RSM) is used to express the three stress parameters in terms of influential factors. The incipient  $\sigma_{an}$  value for hillocks appearing in the specimens was found to be between -28.7 MPa and -32 MPa in a compressive form. The annealing temperature, time, and Al-film thickness are the three major factors, affecting internal stress  $\sigma_{an}$ . An increase in the annealing time reduces the tensile stress or increases the compressive stress, or both. The tensile stress and the compressive stress increases during the annealing process with increasing Al film thickness and annealing temperature. The number of hillocks formed in a unit of area is linearly proportional to both  $\sigma_{an}$  and ( $\sigma_f - \sigma_{an}$ ). (© 2012 Elsevier B.V. All rights reserved.)

### 1. Introduction

Bottom-gate thin-film transistors (TFTs) have been widely investigated for applications in active-matrix liquid crystal displays. Aluminum (Al) is a nearly ideal material for gate bus lines due to its low resistivity, low cost, high adhesion, and superior patternability. However, at high annealing temperatures, aluminum experiences stressmigration, which can cause defects (e.g., hillocks) as a result of short-circuits among adjacent metal lines. Al-based alloys have been studied to minimize hillock formation, with Al-rare-earth alloys showing high hillock resistance [2-5]. Hillock formation in thin films during the annealing process has been known [6,7]. Hillocks form preferentially on grain boundaries or triple points due to fusion creep [8-10], and are thus affected by grain size and crystallographic orientation [11,12]. In the study of Arai et al. [13], an Al-Nd alloy was used for the gate bus lines of LCDs. The results indicated that adding 2 at.% Nd to Al effectively prevented the Al film from forming hillocks and whiskers. Hillock size increased with annealing temperature and time duration [14]. No hillocks or whiskers were found in polycrystalline pure Al film exposed to a thermal stress of 300 °C [15]. Cao Martin et al. [16] evaluated the performance of a variety of aluminum

alloys. For all film compositions, the deposition temperature had the most significant effect on the hillock density. The number of hillocks increased rapidly with elevated temperature [17]. A systematic study of the effect of the sputter deposition conditions on the hillock formations and the Al thin films employed as gate metallization for a-Si:H thin film transistors was carried out by Nathan et al. [18].

The microstructure of thermal hillocks on blanket Al films was studied by Kim et al. [19] using several techniques. Hillock growth kinetics and size distribution were investigated by Zaborowski and Dumania [20]. It is known that the compressive stress relaxation of Al thin films happens via various deformation mechanisms, including hillock formation, microstructural changes, and creep. The stress relaxations of Al thin film during isothermal annealing were investigated using wafer curvature measurements and in-situ hillock observations [21]. The dependence of hillock distribution on film thickness and annealing temperature was studied by Hwang et al. [22] for pure aluminum films. As the film thickness increased, the hillock density decreased and the average diameter increased. The total hillock volume increased linearly with both film thickness and annealing temperature [23].

The study of Iwamura et al. [24] presented the results of an in-situ scanning electron microscope (SEM) observation of hillock and whisker growth on Al–Ta alloy films. The dependence of hillock/whisker formation temperature on Ta content in films and the composition of hillocks were also reported. The mechanism and characteristics of aluminum whisker generation were studied by Takatsuija et al. [25]





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<sup>0040-6090/\$ –</sup> see front matter 0 2012 Elsevier B.V. All rights reserved. doi:10.1016/j.tsf.2011.12.027

using pure-aluminum thin film sputter-deposited on a glass substrate. The experiments revealed that whisker generation is accelerated at thermal stress temperatures above 300 °C since the exposure time at the maximum temperature becomes longer. Al films deposited in a high vacuum onto SiO<sub>2</sub>/Si substrates at various substrate temperature and deposition rates were examined in the study of Chang and Vook [26]. Hillock formation occurred only on films deposited at low substrate temperatures and low deposition rates.

The morphology of Al–2.0 at.% Ta alloy films before and after annealing was investigated for applications of interconnections for liquid crystal displays in the study of Onishi et al. [27]. It was found that the microstructures strongly influence the morphology and the grain orientation of Al alloy films.

In the study of Smith et al. [28], three fundamental local stress relaxation phenomena were observed. Compressive film stresses were, at elevated temperature, relaxed by hillock formation. In-situ SEM observations provided information on the growth mechanism of hillocks. In the study of Flinn et al. [1], the changes in stress observed during thermal cycles were interpreted quantitatively in terms of a simple model of elastic and plastic strains in the metal. The effects of changes in deposition conditions, film composition, and film structure were discussed.

In the study of Lin et al. [29], a technique for evaluating the twodimensional residual stresses in a coating film was developed using a stress expression developed using the Stoney formula (for local point only) and the measurements of the specimen topography. This technique in combination with the stress model developed by Flinn et al. [1] is applied in the present study to evaluate the residual stress arising during the annealing process.

In the present study, 25 kinds of specimen with five Al-film thicknesses were prepared to investigate the relation between the internal stress formed during the annealing process and the hillocks. In the preparation of specimens, the governing factors including deposition conditions, annealing temperature, and annealing time, were arranged following the orthogonal table in terms of five-level and sixfactorial ( $L_{25}(5^6)$ ) design. Stoney's formula is applied to achieve the internal stresses before and after annealing ( $\sigma_0$  and  $\sigma_f$ ), respectively. The internal stress arising during the annealing process ( $\sigma_{an}$ ) is, evaluated using the model developed by Flinn et al. [1]. The incipient  $\sigma_{an}$ value for hillocks appearing in the specimens is identified. The effects of the influential factors on hillock formation are also evaluated. The relationship between the number of hillocks formed in a unit of area and either  $\sigma_{an}$  or  $\sigma_f - \sigma_{an}$  is established in the present study.

#### 2. Experimental procedure

#### 2.1. Specimen preparation

In the present study, a substrate made of E2000-type glass (Corning, USA) was used. 25 kinds of Al/glass specimen with five Al-film thicknesses (500, 1000, 1500, 2000, and 2500 Å, respectively) were prepared and controlled by arranging the deposition conditions and the annealing conditions as shown in Table 1. The Al film was deposited on the glass substrate (1300 mm × 1100 mm). When the deposition process of the Al film on the glass substrate had finished, all specimens were annealed at temperature and time in accordance with the annealing conditions. A scanning electron microscope (SEM) was used to investigate the surfaces of the 25 specimens after annealing its operating voltage was 5K V and its operating current was  $10.5 \,\mu$ A. Surface roughness of the specimens was obtained from an industrial profilometry instrument (ET4000 Kosaka, Japan) with a z resolution of 20 nm.

The coating parameters including the deposition pressure, deposition power and deposition temperature, as shown in Table 1, were set to have 5 levels for each of the six influential factors. The 25 specimens shown in Table 2 were prepared in accordance with the arrangements of Al-film thickness, deposition conditions, annealing temperature, and annealing time following the orthogonal table of five-level and six-factorial design  $(L_{25}(5^6))$ . The deposition process of the Al layer was conducted on a magnetron DC sputtering physical vapor deposition system (ULVAC, Japan). In the magnetron DC sputtering process, gas ions were generated by a high-energy DC power source inside the plasma chamber. The ions collided with the target surface (the Al material) set in the negative surface, resulting in the sputtering of the target material and deposition on E2000-type glass. Argon gas flow was maintained at 150 sccm in the Al-film deposition process. When the deposition process of the Al film on the glass substrate had finished, all specimens were annealed at the corresponding temperature shown in Table 1. In Table 2, hillock formation is indicated by "yes", and absence of hillocks is indicated by "no".

# 2.2. Theoretical models for three internal stresses and the response surface methodology

In the present study, the possibility of hillock formation is governed by the specimen's stress during the annealing process and the specimen's stress some time after the annealing process,  $\sigma_0$  is defined as the internal stress of the specimen before the annealing process,  $\sigma_{an}$ as the stress of the specimen formed during the annealing process, and  $\sigma_f$  as the stress after the annealing process. The two stress parameters,  $\sigma_0$  and  $\sigma_f$  are determined as the mean values of these stresses distributed over an area of  $A = 2 \text{ cm}^2$ . Before obtaining their average values, the area A is divided into many element areas. If the radius of curvature of an element is available, Stoney's formula is applied to determine the mean residual stress of the element. Then, the mean value of all element stresses in the study is used to represent the specimen's stresses before ( $\sigma_0$ ) and after ( $\sigma_f$ ) the annealing process. The method was repeated to obtain the stress values of  $\sigma_0$  and  $\sigma_f$  for the 25 specimens listed in Table 2.

The  $\sigma_{an}$  parameter is difficult to determine because the radius of curvature during the annealing process is not available. An approach for obtaining the stress created during a thermal cycle like annealing was developed in the study of Flinn et al. [1]. The stress,  $\sigma_{an}$ , during the annealing process is expressed as a function of time (*t*):

$$\sigma_{an} = B\{ \ln(B/R) - \ln[t + (B/R) \cdot \exp(-\sigma_0/B)] \}$$
(1)

Table <sup>†</sup>	1
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The orthogonal table for  $L_{25}(5^6)$  design.

C C	, ,						
Factor co	ode	X1	X2	X3	X4	X5	X6
Factor		AL film thickness (Angstrom)	Deposition pressure (Pa)	Deposition DC power (kW)	Annealing temperature (°C)	Annealing time (s)	Deposition temperature (°C)
Level	1	500	0.40	80	340	300	280
	2	1000	0.35	100	320	250	250
	3	1500	0.30	120	300	200	220
	4	2000	0.25	140	280	150	190
	5	2500	0.20	160	260	100	160

Note: Argon gas flow maintained 150 sccm in the Al film deposition.

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