



# Grain growth in thin Al films during deposition from partially ionized vapor



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## ARTICLE INFO

### Article history:

Received 10 January 2016

Accepted 24 April 2016

Available online 3 May 2016

### Keywords:

Aluminium  
Thin films  
Grain growth  
Metal plasma  
Self-ion bombardment

## ABSTRACT

Grain growth in thin Al films during deposition from partially ionized vapor flux with simultaneous self-ion bombardment was studied in this work. The films were deposited at constant ion energy of 940 eV and total specific power of 0.4 W/cm<sup>2</sup> while the deposition time  $t$  of 6 s to 246 s and the resulting substrate temperature ( $T_s/T_m$  of 0.35–0.96) were varied. Thin continuous Al films exhibited normal grain growth through the entire experimental range of deposition time without limitation of grain growth by the film thickness effect. Three kinetic stages of the grain growth were observed within 100 s of deposition time: the first one exhibits very slow grain growth, accelerated grain growth occurs in the second stage and then it rapidly changes to a retardation and stagnation mode in the third stage. Large average grain sizes  $D_g$  up to 11.3  $\mu\text{m}$  at film thickness of 1.4  $\mu\text{m}$  and integral grain growth rates up to 0.16  $\mu\text{m/s}$  were observed in this study. The experimental results were evaluated against various mechanisms of inhibition of grain growth. An estimate of the effective activation energy of the grain growth yields a value of 0.27 eV which is lower than that of the bulk Al and much higher than the activation energy of surface self-diffusion on (111)Al monocrystal. The power law  $D_g = (kt)^{0.5}$  gives good match with experimental results in the initial deposition phase preceding the grain growth retardation, while another model that is based on the grain size dependent pinning force adequately explains the entire grain size dependence on time. It is deemed both ion enhanced film/surface interaction and impurities on one side and thermal grooves on another side contribute to the rapid retardation of the grain grooves commencing the second growth stage.

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## 1. Introduction

Kinetics of grain boundary (GB) motion and grain growth in course of isothermal annealing of bulk metals [1–3] and as-deposited thin films [4–7] is well studied experimentally and theoretically. The microstructure evolution during thin film formation in course of physical vapor deposition (PVD) was also extensively studied [8–12], including nucleation, coalescence and subsequent grain coarsening processes. There are some basic regularities of grain growth during uninterrupted deposition that are usually observed in continuous films of f.c.c. metals with high grain boundary mobility. The regularities include normal grain growth accomplished by the motion of grain boundaries that is primarily driven by grain boundary capillary forces and exhibits log-normal distribution of grain size [9,10,13–15]. Continued grain growth occurs

during film thickening [13,16] that is scaled with film thickness due to the “thickness effect” and the thermal grooving drag [10,17]. An  $\exp[-E_a/RT]$ -like and  $t^n$ - type of dependencies of grain size on substrate temperature  $T$  [14] and deposition time  $t$  [13,14], respectively, are usually observed where  $E_a$  is the activation energy of grain growth. Such regularities are qualitatively similar to those at isothermal annealing of as-deposited films [5,9,15].

Effects of low-energy ion bombardment (IB) during deposition of thin films is also extensively studied within the energy range of  $\sim 10^1$  eV through  $\sim 10^3$  eV [11,18–20]. In the context of grain growth, the ion irradiation is usually known to decrease the average grain size in continuous films due to much higher initial nucleation density and hence smaller grain size at completion of coalescence [11,18,21]. The influence of self-ion bombardment (S-IB) on the nucleation density and formation of (111) fiber texture in Al and Ag films was studied in [21,22]. It is suggested ion-enhanced adatom diffusivity and re-nucleation processes at the surface play an important role in formation of film microstructure.

At the same time, the grain growth during the ion assisted PVD of continuous thin films where the IB may also directly or

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indirectly influence the grain growth was not studied in details. It is therefore the main purpose of this paper to represent and discuss the grain growth in thin Al films deposited from partially ionized vapor flux with simultaneous S-IB. The deposition was performed at well defined and constant energetic parameters (ion energy and power density) while the deposition time  $t$  and the resulting substrate/film temperature  $T_s$  were the only variables. The entire substrate/film temperature range from a temperature close to R. T. up to almost Al melting point was covered in one experimental series. Aluminium was selected as a good model material because it is well studied both in a bulk and thin film states that provides many reference points to compare with.

## 2. Experimental details

A detailed description of the deposition method, experimental set-up, operation modes, and basic physical parameters were reported elsewhere [23]. Therefore the only particular experimental conditions are presented in this paper along with a brief description of the deposition method. The plasma deposition unit was located in the main vacuum chamber which was evacuated down to  $6 \times 10^{-5}$  Pa by a cryogenic pump. Aluminium was evaporated from a water cooled crucible by a 10 kW e-beam evaporator. Partial ionization of the aluminium vapour was realized in the r.f. inductive metal plasma without supporting gas. The metal plasma was generated by a water-cooled copper inductor located above the evaporator and supplied with the r.f. power of a 1 kW/13.56 MHz generator connected via matching device. The substrate holder was fed with the r.f. power of a separate 1 kW/13.56 MHz generator in order to create the bias voltage  $V_b$  on the surface of nonconducting substrates. As a result, the condensation process was accompanied by simultaneous S-IB in a high vacuum environment. A load-lock unit provided an automatic loading and replacement of substrates in the main chamber. It was separately pumped down to  $1.3 \times 10^{-3}$  Pa by a turbomolecular pump. The substrate holder was located in a special rotating system. This system rotated substrates in their own plane ( $\omega = 120$  rpm) and tilted them with respect to the evaporator so the angle between the substrate normal and the imaginary line connecting the center of the substrate and the center of the ingot (evaporation spot) was equal to  $44^\circ$ . Location of substrate holder and the tilt angle were optimized for uniformity of film thickness at  $\pm 2\%$  within the substrate diameter and step coverage on shallow surface relief elements. The substrates used in this study were polished 100 mm Si(100) wafers with a thermally grown  $\text{SiO}_2$  layer of about  $0.13 \mu\text{m}$  thickness. The substrates were not pre-heated prior

to deposition. A crucible for evaporation was loaded with the ingot of high purity (99,999%) commercial aluminium. All deposition runs were performed in one vacuum cycle. The physical deposition parameters shown in Table 1 were sustained constant with the exception the deposition time  $t$  which was varied from 6 s to 246 s. As a result, a series of  $0.05\text{--}2.05 \mu\text{m}$  continuous aluminium films was deposited.

Powerful IB during condensation results in a substantial heating of the substrate and the Al film [23] so substrate/film temperature  $T_s$  is strongly dependent on deposition time  $t$ . The S-IB specific power  $W_i$  in our experiments was kept constant at  $0.29 \text{ W/cm}^2$ . Beside the ion induced heating there are some other heating sources and mechanisms in the plasma deposition system [23]. They are the radiation heat transfer from the evaporator, specific heat of vapor condensation, electron-ion recombination of the film surface, and kinetic energy of neutral atoms. They total to  $0.11 \text{ W/cm}^2$  so the total energy flux density being dissipated on the film/substrate surface was approximating  $W = 0.4 \text{ W/cm}^2$  for all deposition runs. The IB specific power  $W_i = 0.29 \text{ W/cm}^2$  and corresponding total specific power  $W$  of  $0.4 \text{ W/cm}^2$  was chosen for this study to ensure the whole temperature range from a near-to-room temperature at  $t = 0$  up to almost a near-to-melting

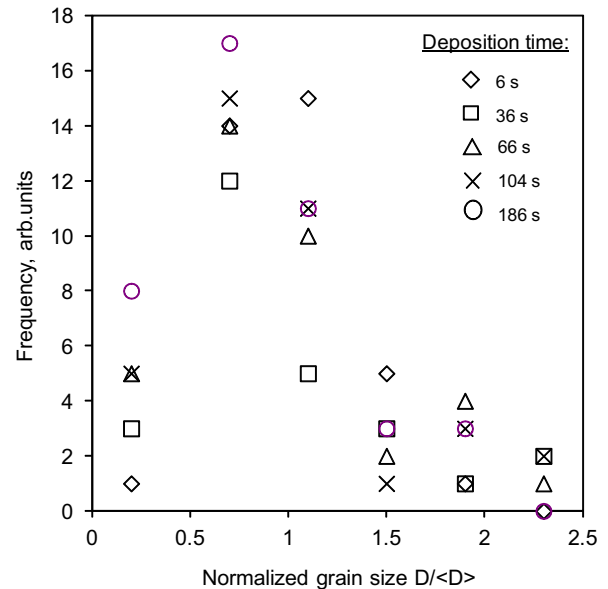


Fig. 1. Distribution of normalized grain size in thin Al films deposited from ionized vapor flux.

Table 1  
Basic parameters of Al thin film deposition.

Parameter	Value/range
Residual gas pressure during condensation, $p_{\text{cond}}$	$(1\text{--}3) \times 10^{-4}$ Pa
Effective deposition rate, $v_{\text{eff}}$	8.3–9.1 nm/s
Effective atom flux, $n_{\text{eff}}$	$(5.0\text{--}5.5) \times 10^{16} \text{ cm}^{-2} \text{ s}^{-1}$
Substrate bias, $V_b$	–900 V
Plasma potential	+40 V
Ion energy, $E_i$	940 eV
Al self-sputtering yield $Y$ ( $E_i = 940$ eV), calculation per Ref. [24]	1.25
Ion current density, $j_i$	0.31 mA/cm <sup>2</sup>
Ion flux density, $n_i$	$2.0 \times 10^{15} \text{ cm}^{-2} \text{ s}^{-1}$
Atom flux, $n_a$	$(5.04\text{--}5.54) \times 10^{16} \text{ cm}^{-2} \text{ s}^{-1}$
Ratio $n_i/n_a$	0.036–0.04 (ave. –3.8%)
Ratio $n_i/(n_a + n_i)$	0.034–0.038 (ave. –3.6%)
Added (normalized) energy $E_i \times n_i/(n_a + n_i)$	33.8 eV
Specific power of ion bombardment, $W_i$	0.29 W/cm <sup>2</sup>
Total specific power dissipated at substrate, $W$	0.40 W/cm <sup>2</sup>
Total ion dose, $D_i$	$1.2 \times 10^{16}\text{--}4.9 \times 10^{17} \text{ cm}^{-2}$

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