

Activation energies of grain growth mechanisms in aluminum coatings

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Abstract

To produce a specific grain size in metallic coatings requires precise control of the time at temperature during the deposition process. Aluminum coatings are deposited using electron-beam evaporation onto heated substrate surfaces of both mica and lithium fluoride. The grain size of the coating is determined upon examination of the microstructure in plan view and cross-section. Ideal grain growth is observed over the entire experimental range of temperature examined from 413 to 843 K. A transition in the activation energy for grain growth from 0.87 to 2.04 eV atom⁻¹ is observed as the temperature increases from <526 K to >588 K. The transition is indicative of the dominant mechanism for grain growth shifting with increasing temperature from grain boundary to lattice diffusion.

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1. Introduction

The synthesis of fully dense coatings to several hundred microns in thickness in the form of free standing foils and as coated substrates is of growing interest for material behavior studies under dynamic loading conditions [1–3]. To pursue the controlled growth of specimens with grain sizes that range from 0.1 μm to 1 cm presents a challenge for preparation by physical vapor deposition technology. The use of electron-beam evaporation [4] is an established and appropriate process to systematically deposit such thick coatings as deposition high rates in excess of 0.1 μm·s⁻¹ can be routinely achieved. The classic zone model(s) for growth of vapor deposits [5–11] provide an excellent starting point for selecting the process conditions required to produce dense metal coatings. For the case of evaporative deposits [11], it's primarily the surface and bulk diffusion processes that progressively affect grain size growth with increasing temperature from dense columnar-type microstructures to equiaxed polycrystalline solids. Within this context, we

investigate the electron-beam evaporation conditions relevant to the formation of aluminum polycrystalline deposits.

The time at temperature affects the coating grain size. In order to quantify the kinetics of grain growth, the coating temperature during the deposition process should be nearly isothermal. Typical investigations of high-rate evaporation processes [6,7] have a focus on a narrow range of substrate temperatures relative to the melt point but do not document the time at temperature. The qualitative variation in microstructure and grain size observed for aluminum coatings [12], evaporated over a wide range (383–793 K) of substrate temperatures confirm the basic morphologies of the zone model. Although a quantitative analysis of the growth kinetics at temperature above 537 K was recently reported [13], a detailed study of growth kinetics at lower temperatures remains incomplete. New experimental results are presented for the time evolution of grain size from the micron-to-millimeter scale for the electron-beam deposition of aluminum coatings up to 100 μm thickness. The kinetics is evaluated with respect to the grain growth law. Analysis of the activation energy and the mechanism for grain growth are made for both temperature ranges above and below half the melt point (T_m).

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2. Experimental methods

The electron-beam evaporation method is used to produce a large range of grain sizes (d_g). The vacuum chamber is cryogenically pumped to a base pressure less than 1.3×10^{-5} Pa. A 0.9999 pure aluminum target is melted in a 40 cm³ pocket-crucible using an electron-beam operated at 10 kV with a discharge current range of 200–600 mA. The source-to-substrate distance range of 10–15 cm facilitates higher deposition rates for the synthesis of thick coatings. A tantalum substrate platen is resistively heated using a boron–nitride heating element. The 3 mm² substrates used are 50–75 μ m thick mica sheets and 125–500 μ m thick lithium fluoride (LiF) crystals. The substrates are fastened to the platen using a hard mask. The deposition rate is monitored using a calibrated 6 MHz gold-coated oscillating quartz crystal. The final coating thickness is measured using a contact profilometer.

The baseline temperature of the substrate platen is feedback-control regulated. However, it's the actual temperature of the coating (T_c) that needs to be measured for determination of the grain growth kinetics. In these experiments, thermocouples are placed in firm contact with substrate surface as well as platen. As the deposition process proceeds, the substrate surface thermocouples are embedded into the coating. This measurement provides the actual coating temperature during the deposition process. Although in equilibrium and numerically equivalent prior to the deposition process, the coating temperature may not equal the substrate temperature (T_s) during deposition. Independent measurements of the coating and substrate [13] have revealed a significant temperature gradient that results during the electron-beam deposition process. This method of temperature measurement [13,14] is especially important, for example, when the substrate is a thermal insulator as well as for the high-rate deposition of aluminum. A coating temperature range of 413–843 K is used in this study, noting a T_m for aluminum of 933 K, in order to ensure fully dense coatings [5,9–12].

The microstructure of the aluminum coating surface is imaged in plan view using a scanning electron microscope (SEM). The method of X-ray diffraction (XRD) provides a measure of the crystalline orientation of the grains in the aluminum coatings. The coating surfaces are scanned in the $\theta/2\theta$ mode using Cu $K\alpha$ radiation. The grain size is quantified from the plan view images using the lineal intercept method [15]. A circular test figure of known perimeter (P) is randomly applied to the image of the coating surface at magnification (M). The number (N) of test-circle intersections are counted with grain boundaries. The average grain diameter (d_g) for cubic grains equals $2.25L$ where the average lineal intercept (L) equals $nP(MN)^{-1}$ for a total number (n) of applications. That is, the average grain diameter is

$$d_g = 2.25\{nP(MN)^{-1}\}. \quad (1)$$

The value for d_g determined in this way from Eq. (1) is equivalent to the lineal intercept value determined from cross-section views of the average column width [13].

3. Results and analysis

The coating temperature is different than the substrate platen temperature [13] during the electron beam deposition of the aluminum coating on thermally insulating substrates as mica and LiF. In general, the coating temperature (T_c) increases with the substrate temperature (T_s) as shown in Fig. 1. However, the T_c may vary at constant T_s as dependent on the heat radiated from the evaporation source under varying deposition rate conditions and at different source-to-substrate distances. The T_c values plotted in Fig. 1 are the time-averaged measurements during the deposition, i.e. when the substrate is exposed to the evaporation source. These values are referenced during the grain size analysis to follow.

The SEM images of the Al coatings deposited on (100) LiF reveal the basic features of the microstructure. The grain boundaries are well defined at the Al coating surface in the SEM plan view images which are then used for the measuring grain size. For example, a progressive increase in grain size with increasing temperature is seen in the images of Fig. 2a–c from 10.9 ± 0.7 μ m for a 607 ± 8 K (2 μ m thick) deposit (Fig. 2a.) to 46.5 ± 1.6 μ m for a 688 ± 17 K (59 μ m thick) deposit (Fig. 2b), to a 3 mm² single-crystal for a 813 ± 15 K (74 μ m thick) deposit (Fig. 2c) in which grain boundaries are not present.

The XRD scans of the Al coatings on both LiF and mica reveal an invariant (111) growth texture, as seen in the representative $\theta/2\theta$ scan for a 808 K deposit (Fig. 3). The only peaks observable are the (111) and (222) Bragg reflections at 38.33° and 82.25° (2θ), respectively. In comparison, heteroepitaxial conditions for the growth of (111) aluminum is achieved [16] using (0001) sapphire substrates.

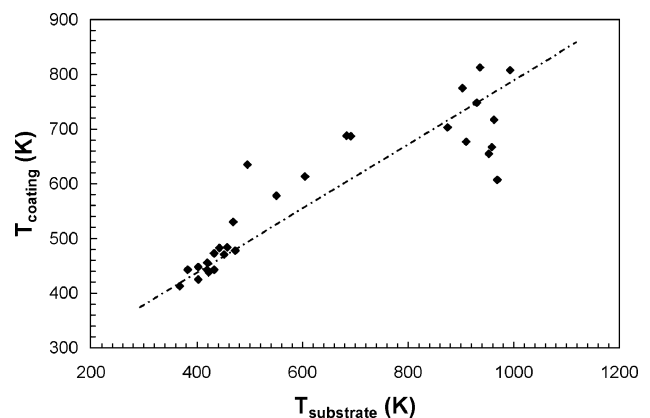


Fig. 1. The coating temperature (T_c) varies with the substrate temperature (T_s) during the electron-beam deposition of aluminum coatings at various deposition rates and at various source-to-substrate distances.

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