AN X-RAY INVESTIGATION OF VACUUM-EVAPORATED AL-FILMS IN RELATION TO SOME PREPARATION PARAMETERS

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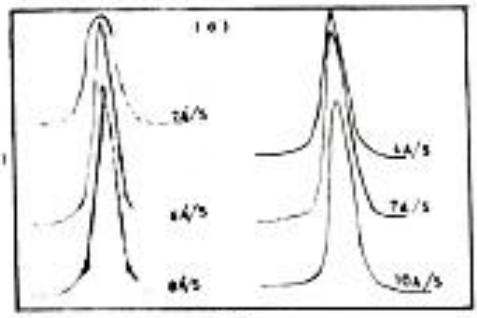
Abstract:

Al-films with thicknesses between 200 and 2000 Å were vacuum deposited with different deposition rates between 2 and 10 Å/S. The particle size and strain were measured by X-ray diffraction profile analysis. A preferential orientation of (111) planes parallel to the film surface was observed. Finer grain size was obtained as the deposition rate increases and film thickness decreases. The microstrain was found to decrease with increasing the deposition rate and film thickness.

Introduction

Using X-ray diffraction profile analysis, the important features of the microstructure including the crystallographic preferred orientation, the crystallite size and the residual microstrain of thin films at different preparation parameters could be studied. Depending upon preparation conditions, polycrystalline films obtained by electrodeposition or vapour deposition may contain large amount of faulting and high internal stresses which broaden the diffraction peaks and change their positions. It is usually assumed that the broadening of the peak profile is produced by a reduction in the size of the coherently diffracting domains (crystallite size), by faulting on certain (hkl) planes and by microstrains within the coherently diffracting domains. The broadening produced by small crystallite sizes and faulting is independent of the order of reflection, while the broadening due to strain depends on the order of reflection. The earlier X-ray investigators were usually content to use simple quantities such as peak breadths. However, many of the important and interesting features of the diffraction patterns were completely missed by working only with beak breadths instead of analysis of the entire line shape (1). In addition to the broadening or peak shift produced by the condition of the samples, the geometry of the instrument also modify the peak shape. Jones (2) proved that this modification can be expressed in terms of a convolution theorem. Using the profile of a well annealed sample free from broadening effects, Stokes (3) corrects for the instrumental factors by Fourier treatment and arrived at the pure diffraction profile. From the real component of Fourier coefficients, Warren and Averbach(1,4) have calculated the strain and grain size. This method has been widely used as one of the effective methods to analys the XRD profiles but it needs measuring



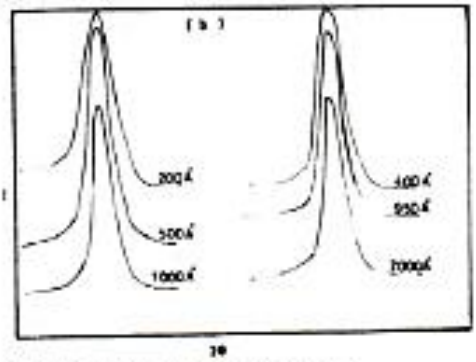


Fig.(1): The diffraction Patterns for Al-Films
a) 1000Å Thickness at different deposition rate
b) 17Å/s deposition rate at different thickness

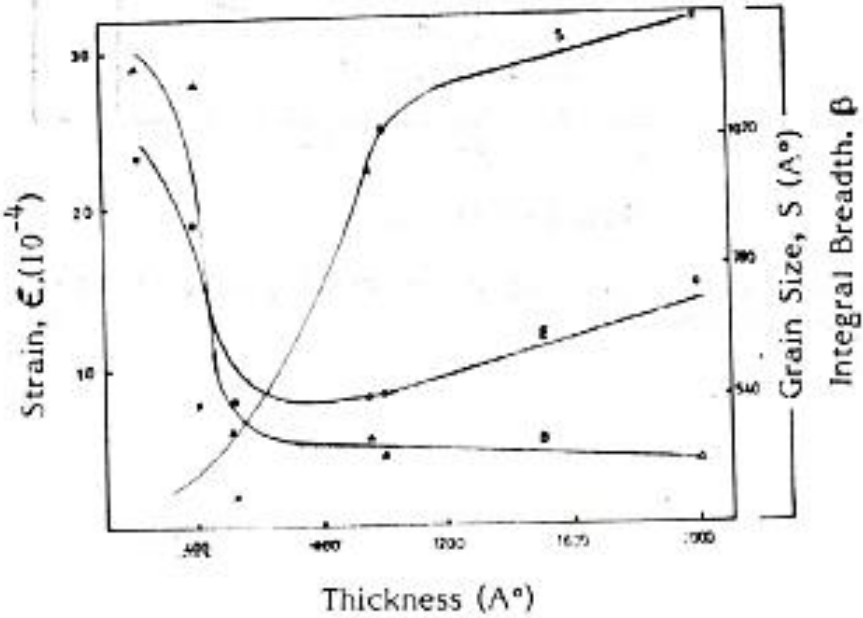


Fig.(2): Plot of microstrain (ε), integrated breadth (B) and grain size (S) vs deposition rate for Al-films with thickness 1000 Å

It was observed that the broadening decreased as the film-thickness increased. Broad diffraction patterns indicate fine grain and/or presence of residual strain.

For more quantitative information about the microstructure changes which occur in Al-films as a result of the preparation process, Fourier line-shape-analysis was required. It was found experimentally that the higher order reflections could not be measured precisely and only the first-order reflections were observed. Therefore, in this work Pines and Sirenko method (7) which deals with the case of single reflection has been used. From the normalized Stokes corrected Fourier coefficient A_n and by applying Pines and Sirenko method the effective particle size, D_e and the root mean square (rms) microstrain $\langle \epsilon^2_{[} \rangle^{1/2}$ were calculated. Fig. (2) shows the corrected integrated breadth, particle size and rms microstrain for Al-films of 1000 Å thickness as a function of deposition rate. The line breadth decreases with increasing the deposition rate as stated before. It was found that finer grain were obtained and rms microstrain was decreased as the deposition rate increased. This structural behaviour can be interpretted according to the increase of the velocity and intensity of the vapour atoms during rapid film formation. It seems, firstly, that more nuclei are formed and from which fine grains grow. Secondly, reaction with the residual gases during condensation is less likely to occur and more pure or less strained Al-film is formed.

In case of fixed deposition rate of 7 Å/S, as the film thickness increases, the integrated breadth and rms microstrain decrease while the grain size increase as shown in Fig. (3). For Al-films of thickness up to about 1000 Å, severe structural changes were observed. Films of larger thicknesses appeared to be stable in their structural features. Eichermuller and Philipsborn(10) were found for polycrystalline Si-layers prepared by low pressure chemical vapour deposition that, the grain size is dependent on layer thickness. Holder et al (11) did not found for Cu-film any systematic dependence of the effective domain size upon the thickness in case of oblique incident vapour and in case of normal one the systematic dependence was difficult to observe. However, in case of Ag-film a small dependence was found (5,12). Direct electron microscopic study (9) showed a continuous increase of the average grain diameter of Al-films with thickness. They (11) also found that the systematic dependence of the rms strain on the thickness cannot be observed but the average value is larger than that of the highly deformed bulk copper. The increase of average crystallite size and decrease of the lattice disorder with film thickness observed in this work is in agreement with literatures where a systematic changes were observed. However, the absence of systematic dependence of either crystallite size or strain in some literature may be attributed to the range of film-thickness and deposition rate used. As it was shown in this work, there is a film thickness range at certain deposition rate where no change in structure feature can be observed.