

IN SITU FRAGMENTATION ANALYSIS OF ALD-PVD MULTILAYERS ON FLEXIBLE SUBSTRATES

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Key Words: ALD, PVD, fragmentation, thin film, polymer

The deformation behavior of flexible thin film multilayer structures is usually found to be dominated by the most brittle component. Cracks in the brittle layers act as stress concentration points, causing the ductile layers to fracture at very low strains, thereby endangering the mechanical integrity of the multilayer [1]. The extent of the embrittlement often depends on the modulation period ($t_{\text{brittle}} + t_{\text{ductile}}$) as well as on the modulation ratio ($t_{\text{brittle}}/t_{\text{ductile}}$) [2]. In this work, fundamental deformation mechanisms of brittle/ductile multilayers on flexible polymer substrates are investigated using multilayers of Al / Al₂O₃ / Al...., produced by a unique combination of atomic layer (ALD, Al₂O₃) and physical vapor deposition (PVD, Al). Using this ALD/PVD combination, individual film thicknesses can easily differ by one order of magnitude or more. In particular, the ability of operating the ALD/PVD process without breaking vacuum opens up a wide range of otherwise unachievable modulation and thickness ratios. Thickness control with precision down to 0.1 nm can be achieved for the buried Al₂O₃ layers, well below the native oxide thickness of pure Al films. Figure 1a shows a TEM cross-section of an Al/Al₂O₃/Al....multilayer stack (Al 250nm, Al₂O₃ 1-10 nm). For flexible multilayer systems the Al layer thickness is reduced to 50 nm to minimize residual stresses. The Al₂O₃ layer thickness is varied across the multilayer cross-section (0.1 nm – 10 nm) to study crack onset and propagation as a function of oxide layer thickness. Single layered Al and Al₂O₃ films are used as reference materials. Ex situ and in situ uniaxial tensile experiments are performed to evaluate the deformation behavior of the flexible thin films using digital image correlation (DIC) to measure strains on the thin film surface. Lateral cracking (Figure 1b) is investigated with scanning electron microscopy (SEM), carefully avoiding changes in the deformation behavior by interaction of the electron beam with the polymer substrate. To study cross-sectional crack initiation and growth, focused ion beam (FIB) cross-sections are cut into the film under tension (Figure 1c). The in situ approach avoids crack closure due to relaxation of the polymer substrate after unloading. Primary results show that the multilayer structure has good adhesion between individual layers as well as to the polymer substrate. Grain growth of Al is limited by the Al₂O₃ layers (Figure 1a), allowing for easy discrimination of individual Al layers necessary for locating the crack onset, and for cross-sectional fragmentation analysis. The Al₂O₃ layers show increasing stretchability with decreasing film thickness, as a result of being extremely well defined and practically defect free. This study will improve the understanding of the deformation mechanisms in flexible thin film structures and can give useful guidelines for damage tolerant design of flexible thin films systems.

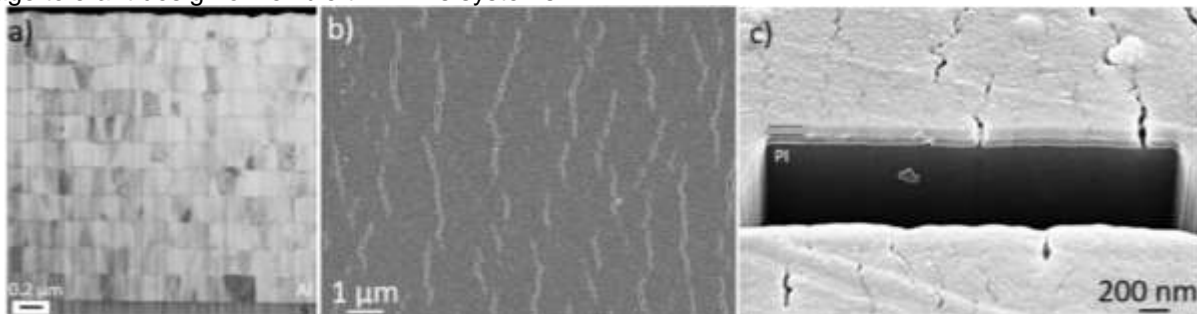


Figure 1 – a) Sequence of Al/Al₂O₃/Al multilayers produced by combined ALD/PVD process at EMPA. b) SEM image of the lateral cracking in Al/Al₂O₃/Al multilayer systems after straining to $\epsilon = 18\%$. c) Cross-sectional fragmentation analysis in an Al/Al₂O₃/Al tri-layer system using local FIB cuts.

References:

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