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In situ TEM straining of single crystal Au films on polyimide: Change of deformation mechanisms at the nanoscale

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Abstract

In situ transmission electron microscopy straining experiments were performed on 40, 60, 80 and 160 nm thick single crystalline Au films on polyimide substrates. A transition in deformation mechanisms was observed with decreasing film thickness: the 160 nm thick film deforms predominantly by perfect dislocations while thinner films deform mainly by partial dislocations separated by stacking faults. In contrast to the 160 nm thick film, interfacial dislocation segments are rarely laid down by threading dislocations for the thinner films. At the late stages of deformation in the thicker Au films prior to fracture, dislocations start to glide on the (0 0 1) planes (cube-glide) near the interface with the polymer substrate. The impact of size-dependent dislocation mechanisms on thin film plasticity is addressed.

Keywords: Thin films; Transmission electron microscopy (TEM); Dislocation dynamics; Plastic deformation; Twinning

1. Introduction

Applications in flexible electronics require that thin metal films grown on elastomer substrates can undergo significant deformation. For particular applications such as paper-like displays or flexible circuits that can be folded or rolled [1], the mechanical properties of metal films on polymer substrates are thus of prime concern [2,3]. With the ongoing trend of miniaturization in thin film technology, size-dependent deformation mechanisms of metal films on polymer substrates, such as film thickness dependent dislocation plasticity [4–6], need to be explored. The deformation mechanisms of metal/polymer laminates can be studied directly by in situ transmission electron microscopy (TEM) straining experimentation owing to the benefit of polymer substrates [7]. The electron beam easily penetrates polymer substrates as thick as ~2 μm at an acceleration voltage of 200 kV. During straining the polymer substrates may deform elastically all the way to the failure of the metal film [3].

In situ TEM straining has proven itself to be an indispensable technique for observing deformation processes in a variety of materials in real time. This technique becomes even more powerful for nanocrystalline materials and thin films since the TEM sample preparation does not alter their original dimensions significantly, allowing the underlying deformation mechanisms to be revealed explicitly. Concerning metal thin films on polymer substrates, a recently proposed focused ion beam (FIB) design allowed the simplified production of tensile testing samples [7]. More importantly, this method eliminates the stress localization problem usually encountered in conventional TEM samples with a rough perforation, which often has made in situ TEM staining experimentation a tedious task to perform.

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Motivated by previous quantitative tensile testing using X-ray diffraction (XRD) techniques [8], we implemented in situ TEM experiments on 40, 60, 80, and 160 nm thick (0 0 1)-oriented single crystalline Au films on polyimide substrates. The in situ XRD stress–strain curve measurements performed on a thickness series of the Au/polyimide samples showed that the flow stress increases with decreasing film thickness and reaches a stress plateau (450 ± 50 MPa) for further decrease from 80 to 20 nm [8] (see Fig. 1). The present study aims to unveil the deformation mechanisms of single crystal Au films in order to understand their mechanical behavior as a function of film thickness. While the main emphasis is placed on in situ TEM straining experiments, microstructural differences of the various film thicknesses are also analyzed. TEM observations are discussed in connection with recent theoretical and experimental studies on thin film plasticity.

2. Experimental details

2.1. Deposition of Au films

Epitaxial Au films with thicknesses of 40, 60, 80, and 160 nm were grown on (0 0 1)-oriented NaCl single crystal substrates (30×30 mm^2) at 300 °C by magnetron sputtering in a deposition system with a base pressure better than 10^{-8} mbar. Subsequently, the epitaxial Au films on NaCl substrates were spin-coated with an ~8 μm thick layer of polyimide using conventional precursors (PI2611, HD Microsystems). The sample was cured for 1 h at 350 °C in a glass oven under N_2 atmosphere. After curing, the sample was exposed to deionized water to remove the NaCl substrate. The tensile testing samples of Au/polymide were prepared for in situ TEM straining experiments as outlined in the next section.

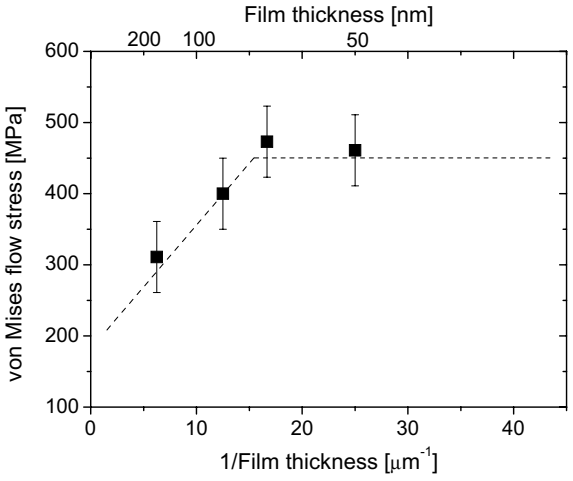

Fig. 1. Flow stresses of single crystal Au films on polyimide substrates plotted as a function of inverse film thickness. While only four data points are presented in this paper and are analyzed in detail by in situ TEM, the dotted lines delineate the overall trend of a full set of data points obtained by the XRD experiments.

2.2. FIB design of tensile testing samples

We used the FIB design proposed by Dehm et al. [7]. (A schematic drawing of the sample design is presented in Fig. 6a). A rectangular Cu support was used as the carrier for a metal/polyimide stripe. The support is 3 mm wide, 6 mm long and about 0.1 mm thick, and has 1 mm diameter holes at both ends to fix it with screws to the straining stage. The center of the support contains a 1×2 mm^2 rectangular hole over which the actual TEM sample – in this study a Au/polyimide stripe cut ~3 mm in length and less than ~1 mm wide – was glued with the region of interest positioned above the hole in the support. The stripes were cut along the ⟨1 0 0⟩ directions using a razor blade. A conventional superglue was used to fix the sample on the Cu support. Final milling of the sample was done in a FIB (Leo XB 1540) after carbon coating the sample to avoid charging.

The FIB design started with cutting the stripe in a "dog-bone" shape. The side grooves were cut for stress concentration. For this purpose, a Ga^+ ion beam current of $\leqslant$10 nA at an accelerating voltage of 30 kV was used. In the center region a 50×50 μm^2 square was milled down to a remaining thickness of polyimide less than ~3 μm using a beam current of $\leqslant$10 nA at an accelerating voltage of 30 kV in order to obtain an electron transparent area of constant thickness without perforation. The Ga^+ ion milling time was calibrated for the applied parameters by cutting test structures in a polyimide reference sample. The milled area was checked for electron transparency by TEM. If the sample was not sufficiently milled, additional thinning was performed by Ar^+ ion milling (Gatan Duomill). During the Ar^+ ion milling the sample was cooled with liquid N_2. In order to prevent redeposition of the removed material on the Au film surface, a glass disk was placed on the bottom surface of the sample holder.

Additionally, cross-sectional TEM lamellae were made from the 60 and 160 nm thick Au films using a FIB (Seiko SII 3050 SE) in order to study the film morphology. The cross-sectional samples were made with the foil normal parallel to the Au ⟨1 1 0⟩ direction. The TEM lamellae were fixed on a half circle Cu grid with a comb structure.

2.3. TEM

Tensile deformation was carried out inside the TEM (Jeol, JEM-2010) at 200 kV using a displacement control, single tilt, straining specimen holder. Since the tensile axis was parallel to the [1 0 0] direction of the (0 0 1)-oriented Au films, all glide {1 1 1} planes were inclined and had a constant Schmid factor of 0.408. The in situ experiments were recorded on DV-Cam using a real time TV-rate camera (25 frames per second). The cross-sectional TEM study was performed by using a field emission TEM (Jeol, JEM 2010F) operating at 200 kV.