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# Measuring electro-mechanical properties of thin films on polymer substrates

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ABSTRACT

In order to advance flexible electronic technologies it is important to study the electrical properties of thin metal films on polymer substrates under mechanical load. At the same time, the observation of film deformation and fracture as well as the stresses that are present in the films during straining are also crucial to investigate. To address both the electromechanical and deformation behavior of metal films supported by polymer substrates, in-situ 4 point probe resistance measurements were performed with in-situ atomic force microscopy imaging of the film surface during straining. The 4 point probe resistance measurements allow for the examination of the changes in resistance with strain, while the surface imaging permits the visualization of localized thinning and crack formation. Furthermore, in-situ synchrotron tensile tests provide information about the stresses in the film and show the yield stress where the deformation initiates and the relaxation of the film during imaging. A thin 200 nm Cu film on 23 μm thick PET substrate will be used to illustrate the combined techniques. The combination of electrical measurements, surface imaging, and stress measurements allow for a better understanding of electromechanical behavior needed for the improvement and future success of flexible electronic devices.



## 1. Introduction

Advances in fabrication and design are making flexible electronic devices and sensors more available. From flexible solar cells [1–2], wearable sensors [3–5], and foldable displays [6,7], these devices will soon become a reality. The special combination of stiff films and islands connected by deformable metal lines and supported by a compliant polymer substrate is important for flexible device design and reliability. It is essential that these material systems have consistent electrical and mechanical behavior over a wide range of loading conditions (stretching, bending, rolling, twisting, etc.). However, measuring the combined electro-mechanical properties and investigating the failure mechanisms is often difficult, requiring the development or advancement of new techniques.

In order to study the mechanical behavior of metal films on compliant polymer substrates, fragmentation testing is often employed [8–12]. During fragmentation testing, the film-substrate couple is strained under uni-axial tension and observed with light microscopy (LM) or scanning electron microscopy (SEM). Brittle metals or ceramic films fracture, forming through thickness cracks (channel cracks) at low strain perpendicular to the straining direction. On the other hand, ductile metal films will first deform locally in the form of necks at low strains (Fig. 1a) and with increased strain through thickness cracks (TTC) can evolve (Fig. 1b). Fragmentation testing is best performed in-situ with LM or SEM so that the strain when the first crack forms can be observed. The initial fracture strain of the film, also known as the crack onset strain, can then be used to determine the interfacial fracture shear stress with knowledge of the crack spacing at saturation, $\lambda$, film thickness, $h$, and the fracture stress, $\sigma_f = E_{film}\varepsilon_f$, where $\varepsilon_f$ is the fracture strain, using the shear lag model [8,13,14]. In-situ fragmentation testing with LM or SEM allows for the crack spacing evolution to be observed as a function of applied strain (Fig. 1c). Under tensile straining conditions, a brittle film will initially fracture at very low strains (<1%) and then with further strain continue to form cracks until the saturation crack spacing is reached. After the saturation spacing has been reached, cracks can no longer form between existing crack fragments and the film could delaminate via buckling.

LMs and SEMs are very good imaging techniques for brittle films, however, the same methods are more difficult to use on ductile films like Cu and Au because the SEM is unsuited to resolve

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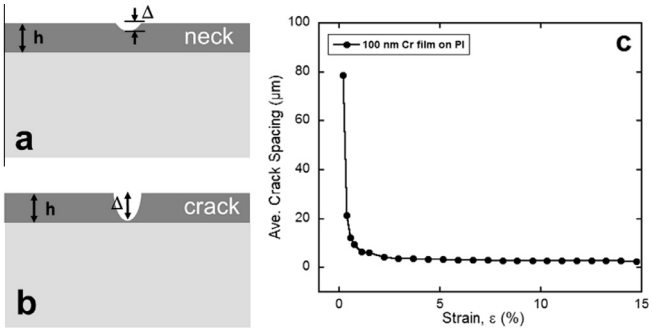


**Fig. 1.** Schematic diagram of (a) local necking and (b) through thickness channel cracks found during uni-axial tensile straining of ductile films. (c) Example of the crack spacing evolution as a function of applied tensile strain for a 100 nm Cr film on PI.

small changes in surface height, especially at large working distances required to accommodate in-situ straining stages. For fragmentation testing of ductile films, atomic force microscopy (AFM) is an approach that allows for the recognition of TTC and localized necking at the film surface [15–18]. With the ability to distinguish between a TTC and a neck, the spacing of TTC and necks can be quantified as a function of strain. In-situ AFM fragmentation testing has also been utilized on brittle films to reveal bending the crack fragments under strain [14,19] which could not be detected without the height resolution of the AFM.

Another in-situ fragmentation test measures the resistance of the film using the 4 point probe (4PP) geometry [20–23]. The advantage of this in-situ technique is that the exact fracture strain can be determined for brittle films. The fracture strain of the film can be determined when the resistance ratio deviates from the theoretical resistance ratio (Fig. 2). To date, in-situ 4PP fragmentation testing has been utilized on both brittle and ductile films on polymer substrates, with more advanced work on ductile films. These advances with in-situ 4PP have occurred on ductile films because the resistance gradually increases with applied strain rather than abruptly increasing to infinity. The theoretical background was also developed using ductile Cu films on polyimide [20,22,24]. For the ideal situation of a homogeneous, uniform and stress-free film the initial resistance, $R_o$, before straining is described as

$$R_0 = \rho \frac{L_0}{A_0} \tag{1}$$

where $\rho$ is the resistivity, $L_o$ is the initial distance between the contacts, and $A_o$ is the initial cross-section of the film. It should be noted that $L_o$ is assumed to be equal to the initial gauge length. During straining the instantaneous resistance is described by

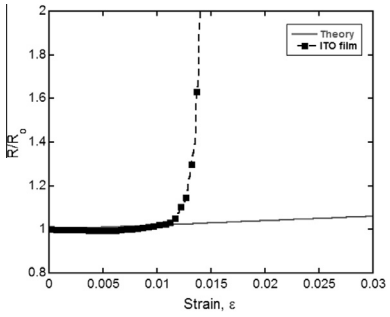


**Fig. 2.** When indium tin oxide (ITO) is strained in tension with in-situ 4PP resistance measurements, the film fractures at low strains and the relative resistance ratio ($R/R_o$) dramatically increases away from the predicted theory (straight line).

$$R = \rho \frac{L}{A} \tag{2}$$

where $L = L_o + \Delta L$ is the instantaneous gauge length with the strain is defined as $\varepsilon = \Delta L/L_o$ and $A$ is the instantaneous cross-section of the film. As the film is deformed plastically under tension, it is assumed that the volume of the film remains constant which gives rise to the very simple relation between the relative resistance ($R/R_o$) and the relative elongation ($L/L_o$)

$$\frac{R}{R_0} = \left(\frac{L}{L_0}\right) \equiv (1+\epsilon)^2 \tag{3}$$

When the film displays significant structural modification through the formation of cracks, Eq. (3) is no longer valid and the resistance is difficult to describe by an analytical formula. It should also be noted that although many researchers utilize in-situ 4PP fragmentation testing, the direct correspondence between the crack spacing and resistance for ductile films has not been demonstrated.

In this study, in-situ 4PP and in-situ AFM fragmentation methods are combined to investigate the role of film mechanical deformation on the electrical behavior. Localized necking of the film is the first sign of yield but it is unknown how the initial deformation affects the electrical behavior. By combining the two methods, a direct measurement and correlation of the induced mechanical deformation and the electrical properties as a consequence of the deformation can be measured. From the experiments, new insights into electromechanical behavior of metal films on polymer substrates can be uncovered.

## 2. Materials and methods

Ductile copper films were sputter deposited onto 23 μm thick polyethylene terephthalate (PET) substrates using an industrial method. In order to improve the adhesion of the 200 nm Cu to the PET substrates, a 5 nm Cr layer was deposited before the Cu film. The Cu films had an average grain size of about 100 nm which was measured using ion channeling contrast images.

Uni-axial tensile straining was performed on rectangular samples with approximate dimensions of 8 mm × 35 mm. For the combined electro-mechanical behavior, the samples were strained under a Dimension 3100 AFM utilizing a custom built small-scale straining stage which is screw-driven [15,16]. The AFM was used to image the Cu surface and to quantify the amount of mechanical damage as a function of strain. It should be noted that the custom built straining stage can only be used in displacement control, thus only the strain was determined. At every straining step, a 30 μm × 30 μm image was obtained of the same area using tapping mode. The AFM stage was adapted so that the straining stage could be fastened tightly to it. This adaption made it easy to always find the same area on the sample and no additional markers on the sample were needed. In order to capture the electrical behavior, four point probes were incorporated into the grips of the straining stage to measure the resistance of the film during straining [24]. The electrical resistance was measured with a Keithley 2400 source meter and the data recorded using a PYTHON script. The measurement combination of the electrical resistance and mechanically induced damage as a function of strain allowed for a direct comparison of both behaviors at once.

An experiment is performed in the following manner. A sample is loaded and the original gauge length and resistance and an image in the unstrained position are recorded. Then the sample is strained in small increments (approximately 1.5–3% steps) and the procedure repeated (gauge length and resistance measured, AFM image made) until the desired maximum strain. Strain increments of 1.5% were used in the early stages of straining in order to