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Mastering the biaxial stress state in nanometric thin films on flexible substrates

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ABSTRACT

Biaxial stress state of thin films deposited on flexible substrate can be mastered thanks to a new biaxial device. This tensile machine allows applying in-plane loads F_x and F_y in the two principal directions x and y of a cruciform-shaped polymer substrate. The transmission of the deformation at film/substrate interface allows controlling the stress and strain field in the thin films. We show in this paper a few illustrations dealing with strain measurements in polycrystalline thin films deposited on flexible substrate. The potentialities of the biaxial device located at Soleil synchrotron are also discussed.

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1. Introduction

Nanometric thin films are generally submitted to complex stress conditions when they are used in manufacturing applications [1]. In order to better understand the mechanical behavior of thin films, it is of utmost importance to assess the material mechanical behavior under complex combined multiaxial stress conditions. A few experiments have been developed to investigate the mechanical behavior of thin films under biaxial conditions such as Bulge test [2–5] and the ring-on-ring test [6]; however, these mechanical tests are limited to equi-biaxial loading. On the other hand, equi-biaxial stress states can also be applied to the film by annealing the thin film/substrate samples at elevated temperatures [7–9] profiting from the thermal expansion mismatch between film and substrate.

Up to now, mechanical test of thin films supported by flexible substrates have only been carried out for single strain path (uniaxial tensile or compressive tests on the film-substrate composite [10–16]). Recently, we have developed a tensile testing device that allows mastering biaxial loading on thin films deposited onto cruciform compliant substrates. The biaxial device is available at DiffAbs beamline of the French synchrotron radiation facility (SOLEIL, Saint-Aubin) [17,18]. A cruciform specimen is used for applying a longitudinal and a transverse loading simultaneously. By varying the ratio of these loadings, we are able to vary the biaxial stress state in the thin film in a controlled manner.

In the present paper, we make a short overview of the experimental approach based on the synchrotron biaxial tensile device for studying the mechanical behavior of films under various loadings. The studied systems are W/Cu nanocomposite thin films and ultra-thin Au films deposited on polyimide cruciform substrate. Synchrotron X-ray diffraction (XRD) is used to measure strains in the film and digital image correlation (DIC) is used to determine strains in the substrate. This method provides important information regarding thin film behavior, the stress and strain field occurring in the film and in the compliant substrate. Especially, the initiation of plasticity and/or damaging in thin films can be scrutinized, thanks to combined measurements of very small strains in both film and substrate with high accuracy [19].

2. Experimental methodology

We have employed the DIFFABS-SOLEIL biaxial tensile device working in the synchrotron environment for *in situ* diffraction characterization of thin polycrystalline films mechanical response [17]. The setup is shown in Fig. 1 in the DiffAbs experimental station at Soleil (Saint-Aubin, France). The biaxial tester is shown

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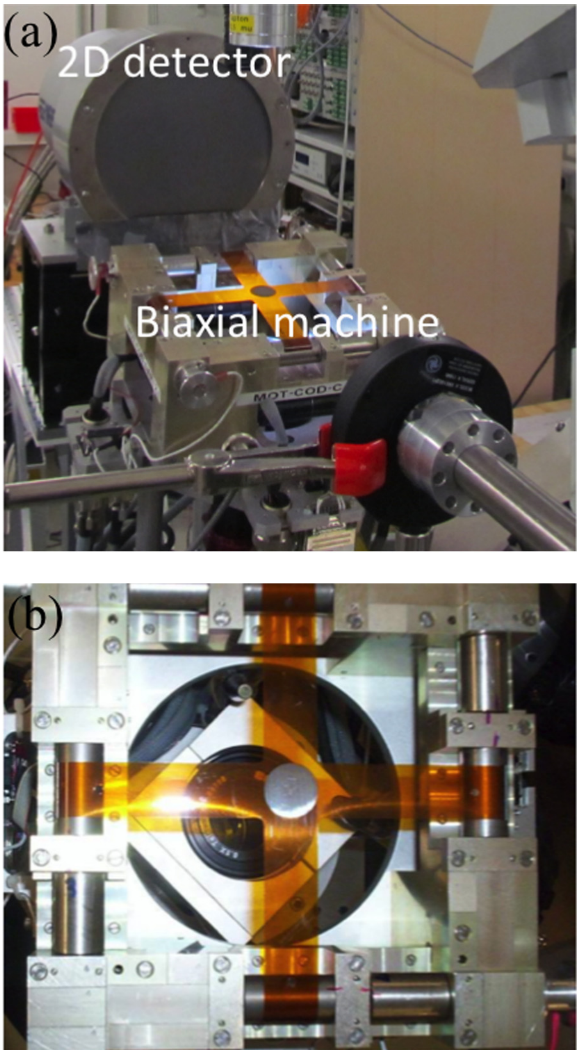

Fig. 1. (a) Biaxial tensile device mounted on the goniometer of DiffAbs beamline (SOLEIL Synchrotron). A 2D detector allows capturing the Debye–Scherrer diffraction rings. (b) Photography of a cruciform-shaped sample (the metallic thin film is deposited at the center of the polyimide substrate) set on the device.

mounted on the goniometer (Fig. 1a) and allows X-ray diffraction in reflection mode at glancing angles (no shadowing edges). Two couples of motors and force sensors are fixed to the device frame. The four motors can be actuated separately in order to keep the studied area at a fixed position in the goniometer. The cruciform compliant substrates are coated by a thin film in their central area of 20 mm in diameter only and gripped by a cam rotating in a cylindrical fixation (Fig. 1b). As shown in Fig. 2, this tensile device allows applying simple or complex loadings in a controlled manner.

Strain measurements can be performed for different loading paths using both X-ray diffraction and DIC techniques:

(i) Synchrotron XRD is used to measure lattice strains within the thin film over coherent diffraction domains. Classically, changes in interplanar spacing d_{hkl} can be used with Bragg's law $\lambda = 2d_{hkl}\sin\theta_{hkl}$ to determine the elastic strain $\varepsilon_{\varphi\psi}$ through the knowledge of the incident wavelength λ and the change in the Bragg scattering angle. Employing an X-ray area detector, grain selective strain measurements can be monitored for several directions of the diffraction vector $k = k_d - k_i$ during straining. This vector gives the direction of the strain measurement and can be differently oriented during a same experiment either by placing a 2D X-ray detector close to the sample in order to acquire an important part of Debye–Scherrer rings (Fig. 3), or by rotating the sample and the detector (0D or 2D) using the 7-Circle goniometer of the DiffAbs beamline.

(ii) DIC is used to assess the strain in the polyimide cruciform substrate. An image of the bottom of the substrate (the uncoated side) is captured with a CCD camera. Image correlation is used to determine displacement and strain fields on the surface of an object by capturing images of the surface at different states [20]. One state is recorded before loading, *i.e.* the reference image, and the other states are subsequent images of the deformed object. DIC uses random patterns of gray levels of the sample surface to measure the displacement *via* the correlation of a pair of digital images.

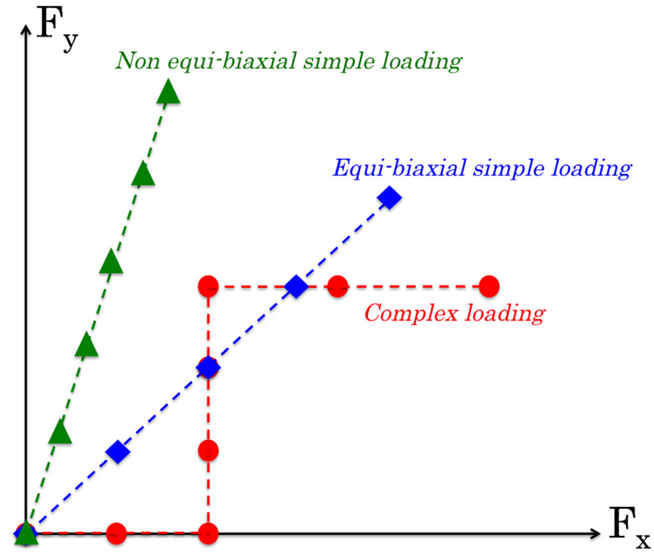

Fig. 2. Graph showing the potentialities of the biaxial tensile device in term of loading paths.

Knowing the macroscopic strains ε_{xx} and ε_{yy} in the substrate, and assuming a complete strain transfer at the film/substrate interface, the in-plane stresses σ_{xx} and σ_{yy} can be deduced using Hooke's law. One interesting way is to compare the measured strain and stress field in both film and substrate in order to study the strain transmission during elastic deformation [18] and to find signature of plastic deformation and/or damaging when it occurs (separation of the two curves) [21].

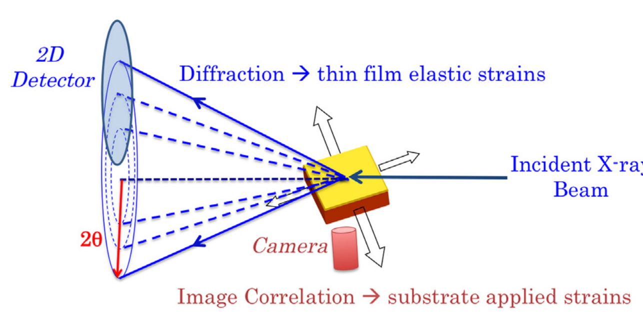

Fig. 3. Sketch of the experimental set-up developed at DiffAbs beamline. The thin film elastic strains are measured by X-ray diffraction while the substrate macroscopic strains are measured by digital image correlation.

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