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Cyclic testing of thin Ni films on a pre-tensile compliant substrate

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ABSTRACT

A novel experimental approach to study the cyclic plastic deformation of thin metallic films is presented. 300 nm thick Ni films are deposited on both sides of a pre-tensile soft substrate which allows to deform the films alternately in tension and compression (approximately from $+2.7$ GPa down to -2 GPa) relative to the as-deposited residual stress state. Nanocrystalline thin films' intrinsic elastic strains (or stresses) and true strains have been measured step by step during two loading/unloading cycles thanks to the X-ray diffraction (XRD) and digital image correlation (DIC) techniques respectively. From the first cyclic deformation, a significant Bauschinger effect is evidenced in the films, however, little or no cyclic hardening is observed during the two cyclic tests.

1. Introduction

Nanometric thin films are ubiquitous in numerous modern application fields, such as micro or nanoelectromechanical systems (MEMS/NEMS) and flexible electronics [1–3]. The performance, reliability and lifetime of such systems are strongly dependent on the mechanical properties of thin films, since they are subject to complex and multiple cyclic stresses in service. Residual stress is also an important factor which may arise due to the presence of impurities and partial grain growth in the films, or thermo-mechanical differences between the film and the substrate. An excessive compressive residual stress can produce film buckling, while excessive tensile stress can cause cracks. It is well known that the mechanical behavior of a metallic material depends not only on its residual stress state but also on its deformation history. The plastic deformation in one direction can affect subsequent plastic response in the reverse direction. One consequence is the decrease of the yield strength of a metal when the direction of strain is changed. This specific mechanical response evidenced in bulk polycrystalline materials is known as the Bauschinger effect [4]. The basic mechanisms are generally ascribed to either long-range effects or to short-range effects, such as the dislocation pile-ups and tangles at grain boundaries [5]. However, due to the extremely small size at least in one dimension, thin films can behave differently compared to their bulk counterparts [6–9]. Consequently, it is imperative to study the cyclic deformation behavior of thin films, such as the strain hardening and the Bauschinger effect, and specific characterization techniques are required.

For bulk materials, the cyclic testing in tension and compression are well established to study various mechanical properties especially the plasticity. However, this is much more difficult for thin freestanding films or film-substrate composites owing to the large lateral dimension/thickness ratio which can cause buckling instability during the compression. Furthermore, tension and compression must be applied in the same experiment.

In the past, several dedicated methods have been proposed. For freestanding thin films, uniaxial microtensile load/unload testing is the most direct and interpretable way. Rajagopalan et al. [10,11] performed the cyclic tests in a series of steps (applied strain at each step: 0.05–0.1%) using a displacement controlled specimen straining holder in a transmission electron microscope. After recording the stress-strain data, a substantial Bauschinger effect in gold and aluminum films was observed and studied.

For thin films coated on substrates, Xiang and Vlassak [5,12] experimentally investigated the Bauschinger effect in passivated thin films. Some theories and simulations were proposed to describe this experimental phenomenon. For instance, a three-dimensional (3D) dislocation dynamics simulation was used by Zhou and LeSar [13]. They found in an excellent agreement that the passivated films exhibited a significant Bauschinger effect, and either an increased prestrain or a smaller film aspect ratio could promote a stronger Bauschinger effect. Their analyses showed that the reverse motion of dislocation pile-ups and the collapse of misfit dislocations were responsible for this effect. It should be noted that the films tested by Xiang and Vlassak [5,12] and Rajagopalan et al. [10,11] are different in terms of material, film thickness, microstructural size and heterogeneity. In the former case, the average grain size is 0.3–1.5 μm and the Cu film thickness is 0.3–4.2 μm, whereas in the latter case, the mean grain

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size is 70–140 nm of 150–250 nm thick Al films.

Hommel et al. [14] proposed a powerful technique combining X-ray diffraction and tension test to study the cyclic deformation of 700 nm thick and magnetron sputtered Cu films on Kapton® substrates. The elastic strain in the film was measured by X-ray diffraction and the total strain in the substrate was measured thanks to a strain gage on the substrate's backside. With the elastic strain as a function of the total strain, they found that the applied strain in the substrate was fully transferred to the film up to ~0.1% (linear relationship with a slope equals 1). A deviation from this linear behavior was attributed to the appearance of plastic deformation. After knowing the elastic constants of films, the yield strength, cyclic plastic deformation and strain hardening behavior can be well investigated [15].

Substrate curvature [16] is also a notable technique to investigate the cyclic deformation of thin films on substrates, and insightful research on the Bauschinger effect has been conducted. Moreover, cyclic tests were carried out by Sim et al. [17] using a fatigue tester with in situ electrical resistance monitoring. In order to avoid buckling, the Ag film-substrate specimen was pre-stretched to given strains prior to fatigue testing. With the normalized resistance, the number of cycles, applied strain range and the effects of pre-straining (severe plastic deformation has already occurred) on the fatigue behavior were explored. Nonetheless, all these approaches associated with a tester have the same limitation: compressive strain cannot be applied when regarding the initial state of as-deposited thin films as a reference, i.e. the tensile/compressive deformation occurs in only one direction compared to the unloaded state. For thermal cycles, the total strain is limited by the thermal expansion coefficient mismatch and the imposed temperature range. Undesirably, the applied strain and the temperature change cannot be decoupled so that other deformation mechanism may act at a certain temperature.

On the other hand, pre-stretching technique is wildly popular in compressing thin films on compliant substrates and in the controlled formation of 3D functional structures [18–21]. The main principle is to bond 2D micro/nanostructures to the adhesion sites of uniaxially or biaxially pre-strained elastomer substrates. Compressive stresses induced by relaxing the pre-strain in the substrate lead to the buckling and the formation of 3D structures. Nevertheless, the fabrication procedure is quite complicated which includes photolithography and etching, and unfortunately, how to maintain the pre-strain in the processing steps is not presented.

Recently, we demonstrated that both compressive and tensile stresses can be applied to the films with a uniaxial tensile tester when using thin films deposited on pre-stretched compliant substrates [22]. By virtue of this technique, Faurie et al. [23] studied the X-ray elastic strain of a strongly {111} fiber-textured Au film during the compression test. However, this technique is harmful to both the tensile tester and the deposition machine, especially the electrical parts even with an extra protection. Furthermore, only one specimen can be obtained with the film deposited on one side of the substrate.

In this study, a novel pre-stretching technique is developed which allows the film deposition on both sides of a pre-tensile substrate, and the testing in tension and compression relative to the original as-deposited state. Combined with XRD stress/strain and DIC strain measurements of thin Ni films, we investigate their cyclic deformation including the yield strength and the Bauschinger effect.

2. Materials and methods

2.1. Development of pre-stretching technique and film deposition

The complete process of our new pre-stretching method is illustrated in Fig. 1(a). The procedure starts with the installation of an uncoated polyimide substrate and the custom-designed grips on the Deben MICROTEST. The dogbone-shaped substrate is Kapton®, 125 μm thick with a nominal in-plane gage section dimensions of $15 \times 6\ mm^2$. Noticeably, the substrate has already been cleaned with acetone and ethanol ultrasonically and the two grips are independent or not connected initially. Then, the substrate is pre-stretched up to 50 N corresponding to a prestrain of ~2%. By virtue of four standard screws and cyanoacrylate glues, the grips are fixed together to be a rigid body. After removing from the tester, the grips with the pre-strained substrate can be taken into the deposition chamber. Thin metal films are deposited alternately on each side of the substrate. Eventually, we reinstall the setup on the Deben MICROTEST and separate the grips for further cyclic tensile/compressive testing. Herein, the force drops markedly from 50 N to 31 N due to the stress relaxation of Kapton® substrate.

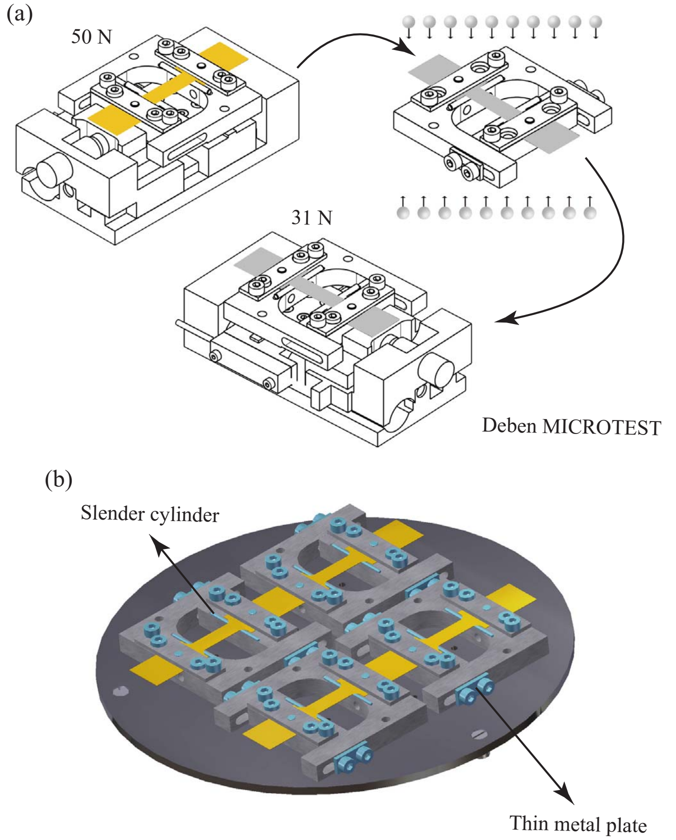

Fig. 1. Schematic illustration. (a) Pre-stretching technique: firstly, mounting and elastically stretching of a Kapton® substrate (50 N, prestrain: ~2%); then depositing films on both sides of the substrate thanks to the new grips removed from the Deben MICROTEST (maintaining the substrate's prestrain); ultimately, grips' reinstalling and separating on the tensile tester for further cyclic testing (31 N). (b) Film elaboration: four pre-tensile specimens adapted to the circular holder in the deposition chamber. Cyanoacrylate glues are added to both sides of the thin metal plates to help keeping the prestrain. With the support of slender cylinders, the central zone of the specimen is higher than the surface level of other parts ensuring the penetration of X-rays in the specimen center.

A detailed illustration of the film deposition on pre-tensile specimens is shown in Fig. 1(b). A machine named NORDIKO (ion-beam sputtering method) was used considering the fact that its pumping system is very powerful which allows to get a good vacuum even with many pre-stretching setups and Kapton® substrates. Furthermore, the high energy of the ion beam sputtering process results in extremely uniform, high density films with excellent adhesion to the substrate despite the presence of high compressive residual stresses [24,25]. This translates into high environmental stability and mechanical durability with low surface roughness.

It should be noted that four pre-tensile substrates having the same prestrain were installed on the carriage plate, and can be sputtered simultaneously in one deposition. This ensures the specimen repeat-