

High strength-ductility of thin nanocrystalline palladium films with nanoscale twins: On-chip testing and grain aggregate model

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Abstract

The mechanical behaviour of thin nanocrystalline palladium films with an ~ 30 nm in plane grain size has been characterized on chip under uniaxial tension. The films exhibit a large strain hardening capacity and a significant increase in the strength with decreasing thickness. Transmission electron microscopy has revealed the presence of a moderate density of growth nanotwins interacting with dislocations. A semi-analytical grain aggregate model is proposed to investigate the impact of different contributions to the flow behaviour, involving the effect of twins, of grain size and of the presence of a thin surface layer. This model provides guidelines to optimizing the strength/ductility ratio of the films.

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1. Introduction

Thin palladium membranes constitute an enabling material in hydrogen storage [1], sensing [2], purification and separation technologies [3]. These membranes must be as thin as possible to ensure high hydrogen permeance while remaining mechanically stable [3]. The lack of ductility of thin Pd films [4], as observed for a majority of thin metallic films, is a key issue for these applications [5]. From a more fundamental perspective, the mechanical behavior of nanocrystalline Pd has received attention in recent literature dealing with several interesting scientific questions related to deformation mechanisms and the connection to the

microstructure (see Grever et al. [6], Ivanisenko et al. [7] and Rösner et al. [8] for experimental studies, and Bachurin and Gumbsch [9] and Stukowski et al. [10] for numerical modelling). The lack of ductility of thin metallic films is related to a poor strain hardening capacity due to the small grain size, similar to bulk nanocrystalline systems. The presence of nanotwins constitutes an attractive way to enhance the ductility of nanocrystalline metals without altering the high strength, as demonstrated for Cu [11–15]. Kulkarni and Asaro theoretically studied the deformation mechanisms in nanotwinned fcc metals like Pd, Cu, Al and Ag, addressing the problem from a more general perspective [16]. These authors found, for instance, a transition in the deformation mechanism which is a function of the twin lamella spacing, except for Pd. There are thus multiple motivations to investigate the behaviour of thin Pd films as a system combining both nano-grain sizes and nanotwins.

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The Pd films studied in Idrissi et al. [17] exhibit high strength owing to the small grain size (~ 30 nm), a high strain hardening capacity and a moderate ductility of between 3% and 6%. The high strain hardening capacity of the films was understood to possibly result from dislocation/twin interactions based on detailed transmission electron microscopy (TEM) characterization of the individual deformation mechanisms and defect interactions, although other contributions were invoked, such as kinematic hardening. The freestanding films were deformed on chip by a microtensile testing technique [18–20]. In this paper the mechanical response of Pd films is analysed in detail and a semi-analytical model is developed in order to improve our understanding of the relationship between the heterogeneous nanostructure and the overall flow behaviour. The model is based on the collective response of an aggregate of grains with different twin densities. This model confirms the potentially large impact of the twins on the high strain hardening capacity, but also accounts for a long elasto-plastic transition and highlights the effect of a residual surface layer on one side of the test structures.

The outline of the paper is as follows. The experimental conditions and the on-chip tensile testing method are presented first. Then the stress–strain curves of the Pd films are analysed and the main TEM observations are summarized. The elementary grain aggregate model is described next, followed by a parameter identification, validation and parametric study.

2. Experimental study

2.1. Materials and methods

The principles of the on-chip tensile testing method used to measure the uniaxial stress–strain curve of thin free-standing films have been described in detail elsewhere [18–20]. The technique relies on relaxation of the internal stress present in one beam material to deform another beam attached to it. This technique allows a large number of tests to be performed on very thin films without the difficulties related to the manipulation of samples and to the measurement of extremely small loads, and without the need for external actuation. In order to fabricate the Pd test structures investigated in the present study the method involves the deposition of four different materials on top of a silicon wafer (see Fig. 1a).

1. A silicon dioxide (SiO_2) layer is deposited by plasma enhanced chemical vapour deposition (PECVD). This layer is 1 μm thick and acts as a sacrificial layer.
2. A silicon nitride (Si_3N_4) layer is deposited at 800 $^\circ\text{C}$ by low pressure chemical vapour deposition (LPCVD). This layer involves a high level of internal tensile stress (~ 1 GPa). The large internal stress arises from the thermal expansion coefficient mismatch between the silicon substrate and the silicon nitride layer.

3. A 5 nm thick Cr layer is deposited by e-beam evaporation. This layer ensures adhesion between the $\text{SiO}_2/\text{Si}_3\text{N}_4$ layers and the Pd layer to be deposited next.
4. Pure Pd films are deposited by e-beam evaporation with thicknesses of 80, 160 and 310 nm. The internal stress level in the Pd films is systematically measured using the Stoney curvature measurement technique.

The silicon nitride layer is patterned by classical photolithography followed by plasma dry etching with SF_6 . The Pd films are patterned by lift-off photolithography to produce a dog bone shaped beam. Lift-off photolithography is a method to pattern thin films. In contrast to classical photolithography the photoresist is first deposited on the substrate and patterned. The target material is then deposited over the resist. The target material lying on the photoresist is lifted off by dissolving the resist with a solvent, e.g. acetone, while the material deposited between the resist pattern remains on the substrate [21]. Concentrated hydrofluoric acid (HF) (73%) is used to etch away the sacrificial SiO_2 layer in order to release the test structures from the Si substrate. Cr being resistant to HF, this layer remains present after the release step. Once the sacrificial layer is etched away the Pd beams are deformed under uniaxial stress until load equilibrium is attained between the specimen and the Si_3N_4 beam. The Si_3N_4 beam used to impose a displacement on the sample is called the actuator. Fig. 1 presents a schematic view of a typical test structure before (Fig. 1a) and after (Fig. 1b) the release step. Several samples of varying lengths are needed to generate a full stress–strain curve (Fig. 1c). A simple mechanical analysis allows the extraction of the stress and the strain level in each test specimen, based on measurement of the displacement imposed on the test specimen [20] and of the elastic strain (also called the mismatch strain) in the actuator material using dedicated structures [22–24]. The displacement u imposed on the test specimen is measured by scanning electron microscopy (SEM) as the distance between moving and fixed cursors (Fig. 1b). SEM is also used to measure the specimen and actuator dimensions after release. The total strain in the specimen is the sum of the mechanical strain $\varepsilon^{\text{mech}}$ and of the mismatch strain ε^{mis} (which is equal to minus the elastic strain stored in the test specimen upon deposition):

$$\varepsilon^{\text{total}} = \varepsilon^{\text{mech}} + \varepsilon^{\text{mis}} = \ln \left(\frac{L_0 + u}{L_0} \right), \quad (1)$$

where L_0 is the initial length of the test beam. The stress is obtained by imposing force equilibrium between the actuator and the test specimen as:

$$F = \sigma_a S_a = \sigma S, \quad (2)$$

where σ_a and S_a are the stress and the cross-sectional area of the actuator beam, respectively, and σ and S are the stress and the cross-sectional area of the specimen, respectively. As the actuator material (Si_3N_4 here) deforms elastically, the stress in the sample is:

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