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Focused ion beam four-slot milling for Poisson's ratio and residual stress evaluation at the micron scale

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ABSTRACT

A novel method is presented for the assessment of the Poisson's ratio and residual stress on the micron scale, based on focused ion beam (FIB) two-step four-slot micro-milling and in-situ digital image correlation (DIC) analysis of the induced relaxation strains at the specimen's surface.

The methodology has been fully validated through modelling and experiments on three different materials, namely, physical vapour deposition (PVD) chromium nitride (CrN), and as-deposited and annealed Cu thin films. The cases of non-equibiaxial stress state and non-isotropic elastic behaviour are discussed in detail, and a quantitative evaluation of the accuracy, error sensitivity and the effects of microstructure and anisotropy is presented. This method represents a substantial improvement over the existing state-of-the-art. It is suitable for application on amorphous and nanostructured materials, and provides a breakthrough in micro-scale Poisson's ratio analysis of thin films and small structures and components.

1. Introduction

Detailed knowledge of residual stress and local mechanical properties, such as elastic modulus, Poisson's ratio, hardness, is an essential requirement for nanostructured materials, thin films and small scale structures, as those are crucial design parameters for the development of reliable micro-devices with improved lifetime.

The measurement of residual stress is the principal issue for reliability assessment of thin films [1–3] and micro/nano-electro-mechanical systems (MEMS/NEMS [4]). Residual stresses directly affect the adhesion and fracture toughness of the layers [1,5] load bearing capacity [5], functional properties of low-k nano-porous films [3], and resonance frequency of MEMS [4]. Nonetheless, the assessment of the residual stress in (sub)micron layers is still a challenging task, especially in case of strongly fibre textured, complex crystalline or amorphous materials.

At the same time, Poisson's ratio directly affects some of the critical mechanical and thermo-mechanical parameters, such as indentation resistance [6], shear modulus [7] and thermal shock resistance [6]. The development of innovative materials with controlled, or even negative, Poisson's ratio [8] is therefore a relevant issue for thin film and MEMS technologies (e.g. in view of the design of innovative micro-actuators) and innovative thin film materials (e.g. ultra-low-k films). The issue of Poisson's ratio experimental assessment is extremely critical in the case of thin films (e.g. in case of telephone-cord buckling [9]), where there exists a clear lack of reproducible and accurate measurement protocols. In the case of ultra-low-k dielectric films [10,11] a substantial reduction of elastic properties is associated with increased porosity. Therefore, the assessment of Young's modulus and Poisson's ratio on a (sub)micrometre scale enables a sophisticated reliability analysis of such systems, as they experience severe thermal stresses during service.

Several techniques have been proposed for the measurement of Poisson's ratio at the micron scale, including X-ray diffraction (XRD) coupled with wafer curvature analysis [12], bulge testing [13], XRD in-situ tensile testing in a synchrotron [14], orientation dependence of surface acoustic waves analysis [10], in situ uniaxial tension tests coupled with atomic force microscopy (AFM) and digital image correlation [15], Brillouin light scattering measurements [3], nanoindentation combined with acoustical methods [16], and bidirectional thermal expansion measurements [11].

The analysis of the literature clearly shows that most of the currently available methodologies for the analysis of Poisson's ratio on a micron-scale are subject to major limitations. Their experimental implementation is not straightforward, as they often require either a complex experimental setup like a synchrotron, are indirect techniques or depend on a concerted combination of different techniques. In addition, either the knowledge or assumptions about other key properties (e.g. elastic

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modulus or thermal expansion coefficient) are sometimes necessary [11–13].

Consequently, no reliable methodology currently exists that allows the direct measurement of Poisson's ratio at the micron scale. Fundamental research efforts are still needed to find straightforward ways for the evaluation of this property in thin films and small scale devices.

In this paper we present an innovative methodology for the simultaneous and direct measurement of Poisson's ratio and residual stress at specimen surface with micrometre lateral resolution. The method uses a focused ion beam (FIB) to remove material in a stepwise, depth-controlled fashion [2,17–21]. The introduced free edges thus allow local stress relaxation, which is directly measured by digital image correlation (DIC). By using an original geometry of milled trenches, four-slot milling, an anisotropic stress relaxation is induced. It is shown that the measured relaxation strains can be uniquely, reliably and accurately correlated to the Poisson's ratio and residual stress by a simple equation, obtained via a purely analytical derivation. Additionally, results of finite element analysis (FEA) are presented to validate the obtained theoretical equation, to evaluate the geometrical conditions (range for size and depth of trenches, with respect to film thickness) where the principal equation is applicable, and to investigate quantitatively the edge/interface effects and other possible experimental artefacts.

The experimental implementation was performed on three different thin films: a cathodic arc evaporation (CAE) PVD chromium nitride (CrN) on steel substrate, and two magnetron sputtering (MS) PVD copper (Cu) on silicon substrate.

2. Materials and methods

2.1. Main idea and analytical modelling

The starting point for the development of the newly proposed method is the assumption that a residual stress state is present at the surface of an elastic isotropic material. This situation is frequently encountered in coatings and thin films on substrates [1–5] and can also occur in bulk materials (e.g. shot peened metals, bulk metallic glasses [20] and at machined or polished surfaces). In the following, the details of the process will be described and an analytical solution will be derived.

To begin, let us consider the typical biaxial stress state for a thin film attached to a substrate. The equibiaxial residual stress state $\boldsymbol{\sigma^0}_{\boldsymbol{xx}} = \boldsymbol{\sigma^0}_{\boldsymbol{yy}} = \boldsymbol{\sigma_R}$, with the Cartesian axes x and y defined in the plane of the sample surface is shown in Fig. 1.

The approach to Poisson's ratio evaluation consists of considering surface strain relief as a consequence of two sequential material removal steps, as illustrated in Fig. 1. Stage I corresponds to the milling (e.g. by FIB) of two long parallel slots, leading to uniaxial relaxation strain $\boldsymbol{\Delta\varepsilon_{xx}}^{(\boldsymbol{I})}$ that occurs in the central squared area along the x-direction normal to the extent of the slots (blue arrows in Fig. 1a). Stage II consists of milling two additional slots along a direction perpendicular to the previous one (Fig. 1b), so as to induce additional relaxation of the strain components (red arrows in Fig. 1b) and leading to full biaxial stress relief in the central squared island ($\boldsymbol{\Delta\varepsilon_{xx}}^{(\boldsymbol{II})} = \boldsymbol{\Delta\varepsilon_{yy}}^{(\boldsymbol{II})}$).

In both cases, the final depth of milling is larger than the characteristic distance between the slots. In the case of coatings, the distance between the slots is also lower than the film thickness, in order to reduce the effects of the interfacial stresses, which cannot be released by the milling procedure, as the coating will be still attached to the substrate.

Based on those assumptions, the key hypothesis is that the ratio between the two strain relief components (with respect to the initial state) in the x-direction ($\boldsymbol{\Delta\varepsilon_{xx}}^{(\boldsymbol{II})}/\boldsymbol{\Delta\varepsilon_{xx}}^{(\boldsymbol{I})}$) measured after step-I and step-II, respectively, is a unique function of the Poisson's ratio of the sample volume.

To develop the main equations that form the basis of the method, let us consider the central squared island outlined by the dashed lines in the Fig. 1a.

Due to the thinness of coating and the proximity of the free surface, the out-of-plane stresses can be assumed to be equal to zero. Therefore, we use the plane stress approximation throughout:

$$\sigma_{xx} = \frac{E}{(1-\nu^2)}\left[\varepsilon_{xx} + \nu\varepsilon_{yy}\right] \tag{1}$$

$$\sigma_{yy} = \frac{E}{(1-\nu^2)}\left[\varepsilon_{yy} + \nu\varepsilon_{xx}\right] \tag{2}$$

Let the initial residual deformation state of the coating that arises due to its attachment to the substrate be described by an equibiaxial elastic strain $\boldsymbol{e}$. According to the above equation, this corresponds to the residual stress state:

$$\sigma^0_{xx} = \frac{E}{(1-\nu^2)}[e + \nu e] = \frac{Ee}{(1-\nu)} \tag{3}$$

Here $\boldsymbol{\sigma^0}_{\boldsymbol{xx}} = \boldsymbol{\sigma^0}_{\boldsymbol{yy}} = \boldsymbol{\sigma_R}$ is the initial equibiaxial state of elastic residual stress.

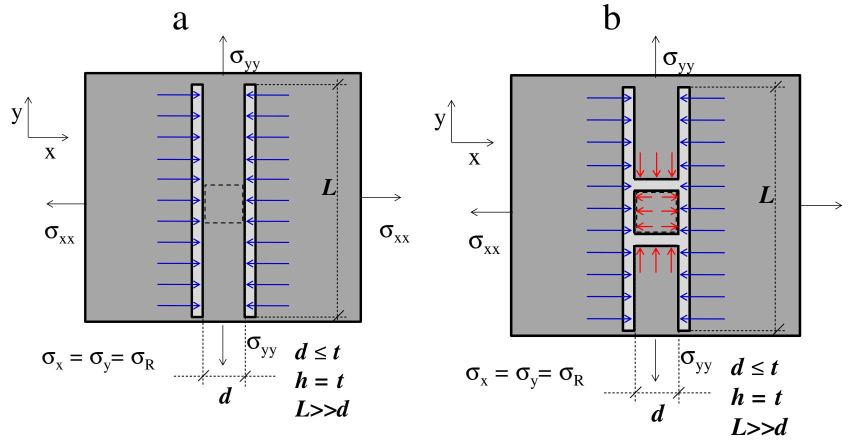

Fig. 1. Two-step method for Poisson's ratio evaluation. (a) Step-I milling of two parallel slots induces uniaxial stress relief $\boldsymbol{\Delta\varepsilon^{(I)}_{xx}}$ (blue arrows). (b) Step-II milling of two additional perpendicular slots induces further relaxation strains that lead to a final biaxial full stress relief $\boldsymbol{\Delta\varepsilon_{xx}}^{(\boldsymbol{II})} = \boldsymbol{\Delta\varepsilon_{yy}}^{(\boldsymbol{II})}$ (additional strain components highlighted by red arrows). The comparison of the two situations (that are experimentally realised in sequence during the same test) allows for the quantitative evaluation of the Poisson's ratio. Here $\boldsymbol{d}$ is the distance between slots, $\boldsymbol{L}$ is the length of slots, $\boldsymbol{h}$ is the depth of the trenches and $\boldsymbol{t}$ is the film thickness.