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Nanoscale residual stress depth profiling by Focused Ion Beam milling and eigenstrain analysis

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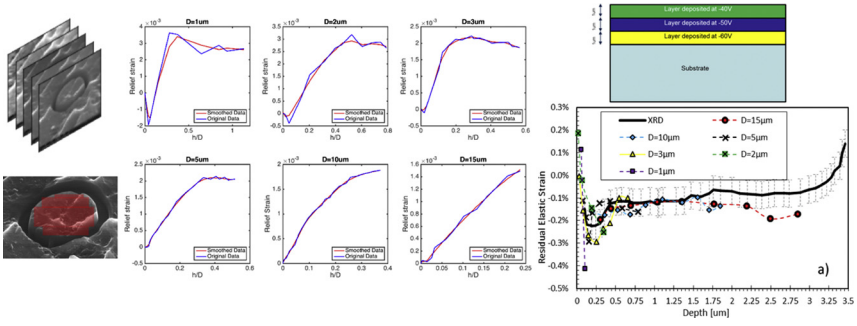
HIGHLIGHTS

- The use of multiple micro-ring-core FIB-DIC analysis for residual stress profiling is proposed.
- Eigenstrain analysis is used to reconstruct residual stress variation at resolution of 50 nm.
- Results are validated against nanofocus synchrotron X-ray diffraction data.

GRAPHICAL ABSTRACT



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ABSTRACT

Residual stresses play a crucial role in determining material properties and behaviour, in terms of structural integrity under monotonic and cyclic loading, and for functional performance, in terms of capacitance, conductivity, band gap, and other characteristics. The methods for experimental residual stress analysis at the macro- and micro-scales are well established, but residual stress evaluation at the nanoscale faces major challenges, e.g. the need for sample sectioning to prepare thin lamellae, by its very nature introducing major modifications to the quantity being evaluated.

Residual stress analysis by micro-ring core Focused Ion Beam milling directly at sample surface offers lateral resolution better than 1 μm, and encodes information about residual stress depth variation. We report a new method for residual stress depth profiling at the resolution better than 50 nm by the application of a mathematically straightforward and robust approach based on the concept of eigenstrain. The results are validated by direct comparison with measurements by nano-focus synchrotron X-ray diffraction.

1. Introduction

The presence of residual stress in mechanical components has a profound effect on their performance from the point of view of structural integrity and reliability [1,2]. Tensile residual stresses promote crack

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nucleation and propagation from surface notches and internal flaws, while compressive residual stresses tend to improve the strength and fatigue resistance of components. However, stress is a scale-dependent concept that displays intricate interaction with the micro- and nano-structure of materials. By the early 1980's the classification of residual stresses became widely accepted into Type I, II and III stresses that can loosely be associated with the macro-, micro- and nano-scale [3–5]. The significance of Type II and III stresses became apparent in the context of the analysis of cracks, notches, surface machining, coating and thin film deposition, heat treatment, phase transformation, grain structure evolution, grain boundary fracture, creep damage and cavitation, corrosion and environmentally assisted cracking, etc. Several decades of modelling effort and careful reasoning pointed at the importance of incorporating the knowledge of micro-scale residual stresses into predictive modelling for reliable design. The principal difficulty in implementing this agenda lies primarily not in implementing numerical simulations that have been developing apace, but in the absence of reliable, universal, flexible and generic reference-free methods for the evaluation of residual stress across the scales, down to the smallest relevant structural dimensions of a few tens of nanometres.

Accurate evaluation of the residual stress is of crucial importance for rational design. For instance, the analysis of thin (multi)layers is of paramount importance for coated systems in which correct prediction of failure conditions must take into account the residual stress distribution through the film thickness. Several techniques are available for the experimental assessment of residual stress depth profile at different length scales. One of the most common non-destructive techniques is X-ray diffraction [6–9]. The recent developments in the generation of nano-focused X-ray beams have allowed probing the stress variation at the nanometre scale [10,11]. A related class of techniques is associated with spectroscopic methods, such as Fourier transform infrared spectroscopy (FTIR) [12] and Raman [13,14]. A further technique that is important to mention, not belonging to the X-ray family, is the well-established method used residual stress in films based on surface curvature analysis using the Stoney equation [15].

Another family of experimental techniques includes destructive methods that rely on material removal causing the modification of boundary conditions that leads to measurable changes in displacement or strain. Numerous variants have been developed for application at the macro- and down to micro- scale, e.g. the contour method. A subset of this family are techniques that are semi-destructive, in that they involve minimal modification of sample surface e.g. by 'blind' or through hole drilling [16–18], ring-core [19,20], cantilever deflection [21], slot cutting [22] slitting [23,24] and nanoindentation [25–27]. Material removal at the macroscopic scale can be performed by conventional tools (e.g. drilling, milling, electric discharge machining), and strain relief can be monitored by the application of strain gauge rosettes or using Digital Image Correlation (DIC) that allows full field displacement mapping and strain calculation. When the evaluation of residual stress is sought at the (sub) micron scale, the material removal process can no longer be conducted by conventional machining tools, and more refined means are required. Focused Ion Beam (FIB), coupled with Scanning Electron Microscope (SEM) allows the combination of material removal and displacement evaluation with the accuracy of the order of a few nanometres. The FIB-DIC micro-ring-core variant has become probably the most widespread technique for the evaluation of residual stress at the micro- to nano-scale, and has been shown to be capable of mapping Type II and III stresses [28,29,4,30], by incremental FIB milling of annular trenches and continuous image acquisition of the surface of the produced micro-pillar, with subsequent strain relief interpretation by DIC post-processing.

The structure of the present report is as follows. Section 2 introduces the materials and experimental techniques used in the study, including the samples and their fabrication, electron and ion microscopy procedures, and the basic interpretation procedures. Section 3 is devoted to the derivation and analysis of the novel approach to residual stress depth profiling using eigenstrain-based analysis of multiple diameter micro-ring-core FIB-DIC data. The key relationships are presented, quantified, and incorporated in the interpretation procedure. Section 4 is devoted to the application of the procedure, the newly developed approach is applied to the evaluation of residual stress variation with depth for the samples chosen for this study. The capability of the method to achieve ~50 nm resolution is demonstrated. To the best of the authors' knowledge, there is no source in the literature that reports residual stress depth-profiling by a means that obviates the need for complex and destructive sample preparation procedures, such as TEM lamellae milling, or the fabrication of micron-thin samples for synchrotron nano-diffraction – the method that is used for cross-validation of the new technique in the present report.

2. Materials and methods

2.1. Materials

The sample was composed of a 375 μm-thick Si wafer which was coated with a nanocrystalline TiN multi-layer thin film. Reactive pulsed DC magnetron sputtering was performed using industrial-scale CemeCon CC800/9 MLT which was equipped with four unbalanced magnetrons and Ti targets (99.97% purity) of dimension $500 \times 88 \times 10$ mm^3. The films were deposited at 550 °C on double-sided polished Si (100) substrates (of size 20×7 mm^2). Before deposition the wafers were ultrasonically cleaned in acetone and ethanol for 10 min, and were then sputter-etched in an argon plasma discharge. During deposition the magnetrons were operated in constant power mode at 7 kW (corresponding to 600 V) per target at 50 kHz. During deposition, the asymmetric bipolar pulsed DC bias voltage U_b at the substrate holder was varied in three stages corresponding to the values of −60 V, −50 V and −40 V at 350 kHz and 1.0 μs reversal time, corresponding to the duty cycle (fraction of time during each period when voltage was applied) of 65%. By considering the information provided in the literature, the appropriate values of Young's modulus and the Poisson ratio for the coating film were chosen as 400 GPa and 0.25, respectively [31].

The films consisted of three sub-layers of equal thickness ~1 μm deposited under different conditions, and therefore expected to contain different residual stress states (Fig. 1). A constant flow of Ar and N_2 was used to obtain a nitrogen partial pressure of $P_{N2} = 0.25$ Pa and a total pressure $P_{tot} = 0.6$ Pa (corresponding to $P_{N2}/P_{Ar} = 0.4$). The base pressure in the deposition chamber was $\leq 4 \cdot 10^{-3}$ Pa. After 165 min of deposition, in which there were two complete cycles of substrate rotation, a uniform film thickness of 3.2 ± 0.1 μm was obtained across the entire wafer.

2.2. XRD measurements

Transmission X-ray diffraction was performed at the nano-focus extension of the ID13 beamline at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. 100 μm-thick slices of the substrate and

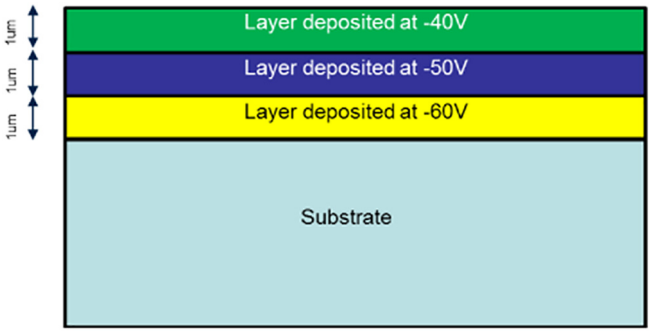

Fig. 1. Schematic representation of the coated sample structure.