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Ultramicroscopy

journal homepage: <www.elsevier.com/locate/ultramic>

Assessing strain mapping by electron backscatter diffraction and confocal Raman microscopy using wedge-indented Si

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article info

Article history: Received 11 September 2015 Received in revised form 5 February 2016 Accepted 14 February 2016 Available online 17 February 2016

Keywords: EBSD AFM Confocal Raman Microscopy FEA Indentation Strain

abstract

The accuracy of electron backscatter diffraction (EBSD) and confocal Raman microscopy (CRM) for smallscale strain mapping are assessed using the multi-axial strain field surrounding a wedge indentation in Si as a test vehicle. The strain field is modeled using finite element analysis (FEA) that is adapted to the near-indentation surface profile measured by atomic force microscopy (AFM). The assessment consists of (1) direct experimental comparisons of strain and deformation and (2) comparisons in which the modeled strain field is used as an intermediate step. Direct experimental methods (1) consist of comparisons of surface elevation and gradient measured by AFM and EBSD and of Raman shifts measured and predicted by CRM and EBSD, respectively. Comparisons that utilize the combined FEA–AFM model (2) consist of predictions of distortion, strain, and rotation for comparison with EBSD measurements and predictions of Raman shift for comparison with CRM measurements. For both EBSD and CRM, convolution of measurements in depth-varying strain fields is considered. The interconnected comparisons suggest that EBSD was able to provide an accurate assessment of the wedge indentation deformation field to within the precision of the measurements, approximately 2×10^{-4} in strain. CRM was similarly precise, but was limited in accuracy to several times this value.

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1. Introduction

1.1. Technical motivation

Many advanced technologies depend on control of deformation or strain at micro- or nano-scales in order to enhance device performance. For example, strain engineering of conducting channels in semiconducting structures increases the mobility of carriers via piezoresistive effects, thereby improving the perfor-mance of microelectronic devices [\[1\].](#page--1-0) Strain engineering of bandgaps in optical materials determines photon absorption and emission wavelengths, thereby controlling the performance of optoelectronic devices [\[2\].](#page--1-0) Strain engineering of membranes and other components in microelectromechanical systems (MEMS) determines device sensitivities to pressure or electric fields, thereby affecting the ability of MEMS devices to perform as sensors or actuators [\[3\]](#page--1-0). Conversely, lack of strain control can lead to thermomechanically-induced or direct mechanical failure, particularly in cases in which disparate materials are brought into contact, for example in microelectronic devices $[4,5]$, or in which

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<http://dx.doi.org/10.1016/j.ultramic.2016.02.001> 0304-3991/Published by Elsevier B.V.

the deformations can be large, for example in MEMS devices $[6]$.

In all cases, strain control depends on the ability to measure and map strain at the micro- or nano-scales—that is, perform quantitative strain microscopy at very small length scales. Two techniques have emerged over the past few decades capable of strain microscopy in small-scale structures formed from silicon (Si), a material pervasive in the technologies highlighted above: high resolution electron backscatter diffraction (EBSD) [\[7](#page--1-0)-[10\]](#page--1-0) and confocal Raman microscopy (CRM) [\[11](#page--1-0)–[16\].](#page--1-0) These techniques are complementary, and both offer fine spatial resolution and great strain sensitivity. EBSD is a high-vacuum scanning electron microscope (SEM)-based technique that determines strain by crosscorrelation of high resolution electron backscatter diffraction patterns (EBSPs) formed from elastically backscattered electrons. Lateral spatial resolutions of approximately 50 nm and strain resolutions of less than 10^{-4} are possible and experimental scan rates of about one pixel/s are common $[8-10]$ $[8-10]$. CRM is an ambient atmosphere Raman spectroscopy-based technique that determines strain by measuring shifts in the frequency of photons inelastically scattered by lattice phonons. Meaningful pixel spacing of approximately 70 nm and strain resolutions of 10^{-4} are possible and experimental scan rates are also about one pixel/s [\[15](#page--1-0)–[19\]](#page--1-0). In both cases, the strain maps are internally calibrated relative to a reference location of known strain, usually taken to be strain free. A

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key requirement for advancing these microscopy techniques for strain mapping of small-scale structures is that specifications be provided for method accuracy (how closely strain values estimate the true values) and precision (how closely repeated measurements distribute about the mean strain value). Such specifications will enable comparison of measurements performed using different techniques, comparison of experimental measurements and modeling results, and predictions of device performance.

Assessments of the accuracy and precision of strain measurements performed by EBSD and CRM have been made by comparing measurements from both techniques on the same structure and by comparison with measurements or predictions from additional methods: Strain variation around a wedge indentation in a Si surface was measured by EBSD and CRM and the agreement between the two techniques shown to be very good [\[17\],](#page--1-0) especially when the CRM excitation wavelength was small, leading to surface-localized CRM measurements, similar to those of EBSD. Surface deformation around a similar wedge indentation was measured using atomic force microscopy (AFM) and compared with the deformation inferred from EBSD and predicted by a simple indentation model; the two measurements and model were in good agreement [\[18\].](#page--1-0) (In some earlier studies, AFM topography measurements were correlated with CRM measurements adjacent to surface scratches and Vickers indentations in Si, but in a qualitative manner $[20,21]$.) CRM shifts adjacent to an imbedded tungsten (W) structure in Si were compared with shifts predicted from an opto-mechanical extension of finite element analysis (FEA) of the strain field arising from the W deposition process and thermal expansion mismatch with the Si; the measurements were in very good agreement with the predictions [\[19\].](#page--1-0) In a recent detailed study [\[22\],](#page--1-0) EBSD strain measurements of silicon-germanium (SiGe) thin-film structures heteroepitaxially deposited on a Si substrate were performed. The measurements were compared with predictions from independent composition and X-ray diffraction measurements of the strain arising from the SiGe and Si lattice mismatch. For films that were coherent with the Si substrate, the EBSD strain measurements were in agreement with the predictions to within 2×10^{-4} , similar to earlier studies [\[7,8](#page--1-0),[10\].](#page--1-0)

Here we extend the above comparisons, applying all four of EBSD, CRM, AFM, and FEA to a single test vehicle, a wedge indentation in a Si surface similar to those considered previously [\[17,18\]](#page--1-0), Fig. 1(a). Application of all four techniques further refines assessments of the accuracy and precision of EBSD and CRM strain microscopy. In addition, many other extensions to the previous works are made here, including: (a) the use of a FEA model that incorporates the elastic anisotropy of Si and a semi-elliptical indentation deformation zone that is more realistic $[23]$ than the rectangular zone $[24]$ used previously $[18]$; (b) self-consistent comparison of the strain fields determined from EBSD and CRM with that of the model, using AFM to adapt the FEA model parameters; (c) greater surface localization of the CRM measurements using a smaller excitation wavelength; and, (d) explicit consideration of the effects of depth convolution on EBSD and CRM outputs. The four techniques provide different levels of information regarding deformation and strain states, and of course all four have very different input requirements in order to generate a strain map. The following section considers the input and output quantities for each technique, detailing the quantitative points of comparison, and provides a framework for the experimental and analytical results to follow.

1.2. Comparison of deformation and strain measurement techniques

Wedge indentation of a Si surface generates a residual contact impression associated with a localized sub-surface irreversible

Fig. 1. (a) SEM image of the analyzed wedge indentation and adjacent deformation field. The image is foreshortened in the vertical direction along the indentation long axis by a factor of 2.5. (b) AFM-obtained three-dimensional rendering of the residual deformation profile of the central section of the wedge indentation. The $x_1-x_2-x_3$ coordinate system used is indicated. (c) The load-displacement behavior observed during indentation.

deformation zone [\[17,25\]](#page--1-0) that includes plastically deformed and phase transformed material $[26,27]$. The residual irreversible deformation zone is in a state of compression. In response to the strain mismatch between this zone and the surrounding matrix, a distributed elastic strain field [\[28\]](#page--1-0), including a surface uplift field $[18]$, is generated in the matrix. As the contact impression is long (here 20 μm) relative to the impression width and associated irreversible deformation zone ($<$ 2 μ m), the state of deformation is approximately plane strain in the x_1-x_3 plane perpendicular to the impression long axis, $Fig. 1(b)$, with negligible deformation in the x_2 direction parallel to the long axis.

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