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A review of micro-scale focused ion beam milling and digital image correlation analysis for residual stress evaluation and error estimation

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ABSTRACT

In the past decade several versions of micro-scale residual stress analysis techniques have been developed based on Focused Ion Beam (FIB) milling at sample surface followed by Digital Image Correlation (DIC) for the determination of the resulting strain relief. Reliable and precise estimation of the error bounds on these measures is critical in determining the usefulness and accuracy of residual stress evaluation. Here we present an overview of the steps necessary for effective outlier removal, error propagation and estimation in order to provide reliable confidence limits for the stress value obtained.

Error propagation analysis begins with DIC marker tracking errors that depend on imaging contrast and magnification, and can be improved with sub-pixel tracking and marker shift averaging. We demonstrate how the outliers and poorly tracked markers ought to be removed from the data set using correlation coefficient thresholding and/or correlation peak confidence intervals. Markers showing large displacements relative to their neighbours can also be identified as aberrant, and removed.

By performing careful error propagation throughout the analysis chain we quantify the displacement and strain fields, and qualify them with the associated confidence intervals. These values, in combination with the elastic modulus confidence limits, are then used to provide the final confidence intervals for the determined residual stress values. The generic nature of the methodology presented ensures its suitability for all residual stress analysis techniques based on FIB milling and image correlation analysis. An example of implementation is presented for the micro-scale ring-core FIB–DIC approach.

Contents

Abbreviations: DIC, Digital Image Correlation; FIB, Focused Ion Beam; FE, Finite Element; SEM, Scanning Electron Microscope; EBSD, Electron Back Scattered Diffraction.

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1. Introduction

With the on-going refinement of capabilities for material structural characterisation, modelling and manipulation, the need for stress measurement at higher resolution (down to the micro-to-nano scale) has become apparent over the last decade. The design of miniature devices such as RF MEMS [1], micro-cantilever sensors [2], micro-switches [3] and nanowire arrays [4] requires reliable quantification of the internal stresses that are unavoidably incorporated in the structure during fabrication, and exert a strong influence on its performance in terms of function (e.g. switching) and durability (e.g. fatigue life). Optimal design and fabrication can only be achieved through effective understanding of the nature of residual stresses, and their evolution in service.

Interestingly, the need for finely spatially resolved stress measurement also arises in applications where the principal function of component is at the macroscopic scale, e.g. in polycrystalline structural alloys used in aerospace applications [5], or “thick” tribological coatings (from a few microns to fractions of a millimetre) [6] and multilayers [7]. For example, the quantification intragranular stress at the micron scale can provide insight into the origins of tensile grains and their role in early onset failure [8].

The arsenal of methods available for high spatial resolution residual stress evaluation has expanded greatly in the past decade. These techniques have recently been reviewed in detail [9,10]. Here we provide a brief summary of the key insights.

Unlike strain, that can be expressed simply as measured displacement divided by a reference length, residual stress is never measured directly. According to its very definition, residual stress is determined from a thought experiment that involves imaginary sectioning, and is calculated as the ratio of the force required to maintain equilibrium to the cross-sectional area through which it is transmitted. The first important lesson is: by the very definition, the concept of stress contains *averaging*, since internal variation of the force within the section considered is deliberately ignored, and stress is computed as the *total* force divided by the *total* area. The nature of averaging is such that finer scale internal force oscillations within the chosen sampling volume must be disregarded to comply with the definition. The second lesson is: the definition of stress immediately introduces the concept of length scale, by association with the size of the area considered. Hence e.g. if the linear dimension of the section is millimetres, the stress can be thought of as macroscopic, whilst if it is microns, then microscopic stress is probed; and so on, down to the nano-scale. The applicability of the concept of continuum stress to sub-nanometric volumes is the subject of ongoing research and presents interesting challenges that, however, will not be tackled in the present paper.

The next important step is the nature of the experimental approach that can be *destructive*, i.e. associated with material removal, or *non-destructive*, i.e. probing some physical property of material that can be correlated closely and precisely with stress. Accordingly all stress evaluation techniques can be classified: examples of the former method include curvature measurement [11], slitting [12] and hole drilling [13], whilst the latter includes crystal lattice diffraction techniques (e.g. electrons [14], X-rays [15] and neutrons [16]) and spectroscopy (e.g. Raman [17]).

Most methods from both classes can be miniaturised down to the micron scale, provided suitably fine probes can be prepared. Thus, Focused Ion Beam (FIB) milling and Digital Image Correlation (DIC) based techniques can be used to measure stresses in gauge volumes smaller than one micron [18], whilst micro- or nano-focused electron and X-ray beams can be used to carry out high resolution analysis non-destructively [9]. The main advantages of FIB milling and DIC methods is the improved gauge volume definition (compared to non-destructive through thickness averaging, typically over length scales >50 μm [19]). Also, the absolute residual stress determination is obtained, i.e. FIB milling and DIC analysis does not require relative comparison with an unstrained lattice parameter [20], as discussed in more detail in Section 2.1.

These advantages, in combination with the increasing accessibility of combined Scanning Electron Microscope (SEM) and FIB systems, as well as improvements in DIC coding, has resulted in the increased use and progressive refinement of FIB milling and DIC based residual stress analysis techniques. In the last decade, a wide range of these methods have been developed, as summarised in Section 2.3.

A key aspect of the FIB milling and DIC techniques is their interpretative nature, giving rise to the need for careful error analysis and uncertainty quantification, since the final use of the results in design practise relies crucially on the reliability, repeatability and accuracy of stress evaluation. In the present review we focus our attention on the identification and quantification of the generic uncertainties that arise in the context FIB milling and DIC based techniques [9,10], with particular emphasis on the error estimation and propagation involved in strain quantification and stress evaluation. Where appropriate, specific examples have been provided based on the micro-scale ring-core version of the FIB milling and DIC approach [8].

2. Residual stress analysis using FIB milling and DIC techniques

The underlying principle of FIB milling for residual stress evaluation is the introduction of micro-scale traction-free surfaces at the location of interest. A range of experimental techniques have been proposed, differing in terms of the FIB milling geometries designed to measure a particular aspect of the stress state (Section 2.3).

The introduction of traction-free surfaces results in the re-equilibration of the stress state in the region of interest. This can be observed as a strain change in the regions neighbouring the milling location and can be recorded using high resolution SEM imaging.

In order to quantify the resulting strain relief, DIC analysis is performed on the sequence of SEM images of the sample surface. Comparisons between the displacement fields observed and the results of analytical or FE models are drawn, typically using optimisation to fit a parametric description of the strain relief observed. Back-calculation of the residual stresses originally present in the surface can then be performed using these relief estimates.

2.1. The benefits of FIB milling and DIC residual stress evaluation

One of the key requirements of micro-scale residual stress analysis is the precise determination of the gauge volume position and size. This requirement is very important in spatially resolved residual stress analysis, which has previously been shown to be critical in understanding failure in a wide range of samples [9]. The nano-scale precision associated with FIB milling and SEM imaging means that the gauge volume can be quantified to sub-micron accuracy and therefore techniques based on these technologies have the capabilities of providing insight at the scale required.