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Residual stress evaluation at the micrometer scale: Analysis of thin coatings by FIB milling and digital image correlation

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ABSTRACT

In this report, an optimised method for residual stress determination at the microscopic scale is presented. The newly proposed approach involves incremental Focused Ion Beam (FIB) milling of annular trenches at material surface, combined with high resolution SEM imaging of a previously deposited marker pattern. Digital image correlation (DIC) analysis of the relative displacements between markers with respect to the undisturbed state provides a measure of strain relief. Results of finite element modeling show that the proposed configuration gives complete strain relief when the annular trench depth becomes comparable with the diameter of the remaining stub, thus allowing analytical calculation of the average residual stress from measured strain components. Basing on results of modeling, the experimental methodology has been developed and optimised for residual stress analysis in thin coatings. In order to cover a wide range of material properties and residual stress states, two different materials have been selected: TiN CAE-PVD coating (hard and stiff, with compressive residual stress) on WC–Co substrate, and also an Au MS-PVD coating (soft and compliant, with tensile residual stress). The procedure for the optimization of FIB milling parameters is reported. Results are validated by comparison with residual stress evaluation by X-ray diffraction and curvature measurement on the two different specifically selected PVD coatings.

1. Introduction

Residual stresses play a crucial role in determining the deformation behaviour and performance of engineering components and materials, from bulk alloys and composites used in construction and manufacturing industries down to micro-mechanical MEMS/NEMS systems and the stresses within individual grains of polycrystalline aggregates, thin films and coatings. Residual stresses exist across the scales, from macro- to nano-, and their evaluation must necessarily be performed using appropriately sized probes. The traditional methods of residual stress evaluation are limited in their spatial resolution to fractions of a millimetre, making them ill-suited to the study of e.g. intragranular stresses in polycrystalline systems with the grain size of a few micrometers.

On the other hand, the rapid development of nano-science and nano-technology in recent decades calls for the development of appropriate nano-scale (or at least sub-micron) analysis tools for residual stress evaluation [1–5]. The applications where (sub)micron-scale residual stress measurement is in demand include nano-structures, nano-devices and nano-structured materials. A portable and accessible residual stress methodology should be applicable not only in research context, but preferably be reducible to procedures that would allow routine use in industry, including in the context of production and quality control.

Residual stress evaluation techniques can be classified into non-destructive techniques, such as X-ray and neutron diffraction, and destructive and semi-destructive methods that involve material removal and the measurement of consequent surface strain relief.

The spatial resolution of the classical methods of residual stress evaluation by X-ray diffraction using laboratory sources is limited to a fraction of a millimetre. In most cases this is insufficient for the study of intragranular stresses. X-ray beams that are many orders of magnitude better in terms of flux and parallelism are produced at synchrotrons.

In recent years the development of micro-focus synchrotron X-ray beams has opened the way for stress evaluation at the micron and sub-micron scales. The key to the possibility of using this approach is the availability of high intensity, high brightness, micro-focus polychromatic X-ray beams only produced at third generation synchrotron sources. X-ray beams generated by a bending magnet or an insertion device such as a wiggler or wavelength shifter are focused on the sample by the use of Kirkpatrick–Baez (KB) achromatic mirrors, allowing the creation of beam spot on the sample less than 100 nm in diameter. The wide bandwidth of the incident beam ensures that the interaction between the incident beam and the

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crystalline gauge volume leads to the creation of a Laue diffraction pattern on a 2D detector. Data collection can be conducted in both the reflection and transmission geometries. The analysis of the diffraction pattern allows simultaneous determination of the orientation and lattice parameters of the crystal(s) present within the gauge volume. Further details of this technique and its implementation can be found in the literature [1,2].

One of the limitations of synchrotron-based micro-beam Laue technique for the measurement of residual stresses in (sub)micrometer-sized volumes is the restricted access to synchrotron instruments, making the use of this technique difficult for routine industrially-motivated analysis.

The purpose of the present study was to consider the possibility of developing a portable and flexible semi-destructive method that would be applicable down to the microscopic scale, and would allow routine determination of residual stresses in a variety of single and poly-crystals using the combination of focused ion beam (FIB) milling, SEM imaging, digital image correlation (DIC) analysis, and finite element modeling (FEM).

In the context of microscopic residual stress evaluation, focused ion beam (FIB) provides a natural choice of the machining tool. Similarly, scanning electron microscopy (SEM) offers excellent imaging resolution for input into digital image correlation analysis. Based on this reasoning, dual beam FIB-SEM system was chosen as the instrumental base for the implementation of the new method.

Prior attempts at the development of such techniques involved the use of FIB milling as the micro-scale machining tool; and various methods of strain measurement, notably Moiré interferometry [5] and digital image correlation (DIC) [6]. Sabate et al. [6] used FIB to machine straight shallow or deep trenches within the sample surface. Analytical solutions for the displacement fields around a crack taken from linear elastic fracture mechanics were used to estimate the residual stress component in the direction perpendicular to the trench extension. Results have been presented for the cases of silicon wafers and coated systems [7,8].

Massl et al. [9] proposed a cantilever method for the determination of residual stress depth profiles in thin films by measuring the deflection of a FIB-fabricated micro-cantilever, as a function of the gradually reduced film thickness. The main limitation of this approach is that the micro-cantilever must be necessarily fabricated in the proximity of the specimen edge, or near a previously fractured surface, making it unsuitable for micro-scale residual stress mapping, e.g. for intragranular stress analysis in polycrystalline materials, or stress measurement in thin films on metallic substrate.

However, strong limitations are still present for these adopted geometries, in particular:

- None of the selected geometries allows to do in situ testing with sufficient spatial resolution (i.e. lower than 1 μm);
- In the case of hole and slot geometries, strain gradients are always present in the proximity of the milled region. Furthermore, the maximum amount of strain relief always takes place in the vicinity of the FIB-damaged area;
- In the case of the slot-milling and blind hole drilling geometries, FEM modeling is required for residual stress calculation after relaxation strain measurement;
- The micro-cantilever method can be realized only at the sample edge, and only in the case of coatings on brittle substrates;
- In the case of slot-milling and the micro-cantilever method, only one stress component can be evaluated.

In this work, we propose an innovative geometry for the FIB milling experiment, which involves incremental focused ion beam (FIB) milling of annular trenches at material surface (Fig. 1). This method is intended to give complete information on the in-plane residual strain components when applied in combination with high resolution SEM imaging of a previously deposited marker pattern and Digital Image Correlation (DIC) analysis of the relative displacements between markers with respect to the undisturbed state.

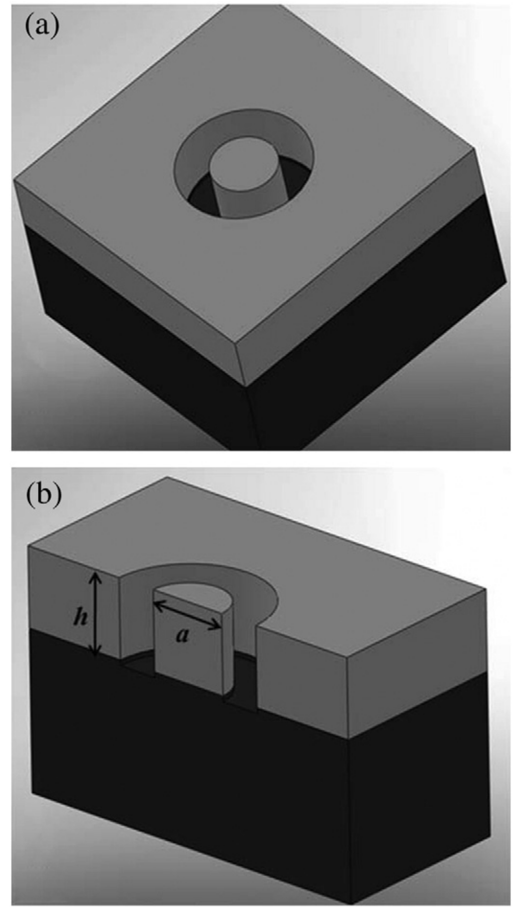

Fig. 1. Illustration of the principle of ring drilling, and the idealised geometry of the remaining "stub".

There exists an important additional advantage of the proposed configuration. The surface of the "stub" is not subjected to machining operation, and under careful cutting should remain substantially undisturbed. This situation lends itself naturally to strain measurement using digital image correlation. If a pattern of at least three non-collinear markers is deposited on the sample surface prior to ring drilling, then three independent components of strain can be determined, and the complete two-dimensional characterisation of the strain relief be obtained.

The proposed approach shares many common features with macroscopic incremental hole drilling, and admits calibration using substantially similar approaches using finite element modeling for influence coefficient evaluation [10]. Analytical solutions for the eigenstrain influence functions have also been derived [11,12].

It is important to note that, in contrast with the hole drilling situation, where strain relief around the drilled hole is always partial and position-dependent, in the present configuration the relieved strain state at material surface can be assumed to be to a good approximation uniform; and complete strain relief can be assumed to take place once a certain drilling depth is reached. This ensures maximum sensitivity of the newly proposed method to pre-existing residual stress.

In this work, finite element modeling (FEM) has been adopted to investigate the relaxation strain profile during incremental ring-core