Stress and Deformation Analysis at the Micro-Scale

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Abstract

Improving the structural integrity of microstructured materials relies on the combination of advancements in modeling capability and spatially resolved experimental characterization of deformation and damage. We report the recent advances in the use of synchrotron X-ray diffraction techniques, as well as in the methods utilizing micro-scale material removal in combination with scanning electron microscopy and digital image correlation for relief strain measurement and stress evaluation.

Keywords: Diffraction, X-ray, Focused Ion Beam, electron microscopy, residual stress, Digital Image Correlation.

1. Introduction

With the contemporaneous advancement of the power of computers and of the sophistication of algorithms available for the simulation of material deformation, damage and fracture, it has become possible to refine the simulations to extents inaccessible previously. For example, it is not at all uncommon now to use sub-micron-sized elements (i.e. a few hundred nm in size) in finite element simulations to mesh the regions of stress concentration such as crack tips or sharp notches, edges of contacts, inclusions etc. At this level of refinement the use of constitutive equations that assume isotropic continuum behaviour that derives from averaging over many randomly oriented grains becomes questionable. Firstly, it fails to capture the crystallographically induced anisotropy of the mechanical properties of small volumes of material. Secondly, local gradients of stress, plastic deformation and consequently of such history-dependent material properties like hardening (flow stress) can only be taken into account correctly provided the underlying mechanisms responsible for their creation are captured in the model. Finally, in order to develop numerical models that can be used with any degree of confidence it is important to obtain their validation not only macroscopically, but also at the appropriate length scales smaller than a micrometre. This, in turn, requires the development, application and refinement of highly spatially resolved methods for the analysis of material structure and strain (stress) at the (sub)micron scales.

It is important to mention, in addition, that progressive miniaturisation not only of electronics components, but also of the components and assemblies that perform mechanical functions means that the variety and sophistication of micro-manufacturing technologies (micro-machining and micro-deformation) continues to increase. In this context, reliable process control and optimisation lead to similar requirements for both modelling and characterisation approaches.

The agenda of linked refinement in modelling and characterisation techniques can be applied with equal success to all types of structural materials such as metallic alloys (polycrystalline as well as single crystal), ceramics and coatings, polymers, composites involving materials of the above classes, as well as natural hierarchical materials, such as e.g. seashell nacre or human dental tissues. In the present discussion the focus is placed on polycrystalline engineering alloys of the type used in the aerospace, transport and energy sectors.

Some of the approaches suitable for deformation modelling and analysis in (sub)micron material volumes in these systems are listed and discussed. The following section is devoted to diffraction-based techniques for the combined characterisation of structure, defects and deformation in sub-micron volumes. Next, the use of electron microscopy based digital image correlation methods for studying deformation and residual stress is described. The combination of the two approaches for the precise evaluation of strain-free lattice parameter needed for reliable residual stress evaluation is also presented and discussed. Finally, the outlook is discussed in terms of challenges, opportunities and likely developments over the next few years.
2. Micro-deformation modelling approaches

Given the typical grain sizes in conventional polycrystalline metallic alloys that range from a few microns to a few tens of microns, the local volumes of dimensions of a few tens of nanometres (even following some stress relieving annealing treatment) should be considered as deformed crystals containing significant deviations from perfection: defects, distortion and damage. Three significant approaches have emerged as potentially suitable for modelling the deformation of sub-micron volumes: crystal plasticity, discrete dislocation dynamics and molecular dynamics.

2.1. Crystal plasticity

Crystal plasticity simulation framework builds on the knowledge that the physical mechanism of deformation is represented by crystal slip [1, 2]. This formulation allows the model to capture the sensitivity of micro-volume deformation response to its orientation with respect to the loading direction, and can incorporate the effect of elastic anisotropy by using appropriate crystal stiffness moduli in the simulation. Length-scale effects in plasticity [3-5] arise due to the presence of gradients of plastic strain associated with Geometrically Necessary Dislocations (GNDs) [5-7]. This relationship was put forward by Ashby [7] and presented in a form that lends itself to implementation within the finite element framework by Busso [8].

2.2. Discrete dislocation dynamics

Inelastic deformation of metallic polycrystals at moderate temperatures occurs mostly by dislocation glide. It is logical to seek to simulate this process by monitoring the movement (dynamics) of the agents mediating the deformation, i.e. dislocations. Dislocation motion is caused by the action of the Peach-Koehler force related to the local lattice stress, and governed by mobility that reflects the effects of lattice orientation, solute elements, hydrostatic stress etc. Kubin et al. [9] developed a 3D lattice-based dislocation dynamics model and used it e.g. to investigate the strengthening effect of dislocation interactions during stage I, II and III hardening of single crystals. An alternative 3D dislocation dynamics framework proposed by Bulatov and co-workers [10] considers dislocations as line defects discretised into segments that propagate in the elastic continuum. In view of the very significant computing resources are required for 3D DDD, it may be attractive to consider an alternative 2D approach proposed by Needleman et al. [11] who considered a simulation plane in which dislocations glide along three pre-defined slip systems offset by 60° from each other. The boundary value problem was solved by a finite element model, which provides a natural means of connecting simulation scales. The attractiveness of the 2D model lies in the fact that, whilst being comparatively straightforward to implement and computationally inexpensive, it neglects the complexities of 3D dislocation interactions, yet manages to capture successfully many of the underlying trends of material behaviour. Gaucherin et al. [12] proposed an extension that takes into account a more realistic distribution of slip systems based on the projection of 3D crystal slip systems for an FCC metal onto the simulation plane. The model could be referred to as “2.5D”, as it manages to capture some of the 3D orientation effects.

2.3. Molecular dynamics

The understanding of the crystal lattice rearrangement, rotation, distortion and deformation at the finer level can be aided by the use of an atomistic modelling approach such as Molecular Dynamics [13]. In face-centred cubic metals such as copper, homogeneous nucleation of dislocations, their motion, interaction and pinning that control hardening have been studied using molecular dynamics (MD) for over a decade, in parallel with X-ray diffraction experiments with in situ shock compression of copper single crystals to pressures of up to 100 GPa. In the case of laser driven shock compression, where the length and time scales are compatible with MD, both qualitative and quantitative agreement has been achieved, paving the way towards ever larger and longer simulations in the future, and towards the application of these techniques to other systems, e.g. body-centred metals such as iron and steels [13].

3. Micro-focus synchrotron X-ray diffraction

The flux and parallelism of the X-ray beams generated by the insertion devices at 3rd generation synchrotron facilities allows subsequent manipulation of these beams to achieve sub-micron focusing suitable for highly spatially resolved characterisation of structure and strain in samples of polycrystalline metallic alloys.

The use of tightly focused polychromatic beams (Laue mode) for micro-scale analysis offers significant advantages over the more widespread monochromatic setup. Firstly, the use of white beam eliminates the need for sample rotation in order to find a reflection: this operation becomes particularly challenging when using sub-micron beam size. Analysis of multiple Laue reflections allows simultaneous determination of crystal orientation, and its deformation state. Whilst the diffraction pattern contains information about the deviatoric strain components, the identification of the energy of individual reflections e.g. by using monochromator crystal filtering also allows assessing the hydrostatic component. The spreading out (“streaking”) of individual Laue reflections provides information about lattice mis-orientation within the scattering volume, giving insight into the plastic deformation occurring at yet finer scales, and providing experimental data for model validation.
Song et al. [14] used strain gradient crystal plasticity as the predictive framework in order to simulate the accumulation of lattice distortion (curvature) within a polycrystalline foil of commercially pure nickel. Physically based dislocation density finite element simulation was carried out using the formulation derived from Busso et al. [8], and a special post-processing procedure was developed in order to predict the “streaking” observed on the diffraction detector.

The setup used in the experiments is illustrated in Fig.1 [14]. Pinhole collimation allowed the beam spot on the sample to be reduced to a few microns. Fig.2 shows an example of the Laue diffraction pattern obtained in the experiment [14]. There is apparent “streaking” of some reflections observed, i.e. the change in the shape of reflections from approximately equi-axed Gaussian peak towards something that appears as a band of increased intensity elongated in a particular direction. The consideration of the underlying distortion of the crystal lattice caused by dislocation-mediated elastic-plastic deformation reveals that it reflects the increase in the lattice curvature due to crystal slip on a particular slip system. Multiple slip system activity causes multi-directional broadening of reflections. Quantitative matching of numerical simulations to these observations allows the underlying strain gradient plasticity model to be validated and refined.

Recent experiments conducted on the Test beamline B16 at Diamond Light Source make use of Kirkpatrick-Baez mirrors in order to produce sub-micron focused beams with FWHM of ~300nm. The use of such small beams allows finer features of the underlying dislocation structure to be probed. It is important to note that such effects as “streaking” of Laue reflections (Fig. 2) are closely associated with the ratio of the beam size to the characteristic linear dimensions of the underlying dislocation structure.

By way of illustration, consider the density of geometrically necessary dislocations (GND) in a deformed nickel sample to be \( p_c = 10 \, \mu \text{m}^{-2} \). Assuming the Burgers vector magnitude \( b = 3 \times 10^{-8} \mu \text{m} \), the lattice curvature caused by the GNDs can be estimated as \( p_c b = 3 \times 10^{-4} \, \mu \text{m}^{-1} \). In a diffraction experiment with an X-ray beam of spot size \( s = 3 \mu \text{m} \) obtained using a pinhole this corresponds to the change in the scattering angle of ~0.1° that causes appreciable “streaking”. However, if focusing is used to reduce the beam spot size down to \( s = 3 \times 10^{-3} \, \mu \text{m} \), then this corresponds to the change in the scattering angle of ~0.01°.

Our previous attempts to use micro-beam Laue diffraction to study plastically deformed soft metals such as single crystal copper were frustrated by the fact that lattice distortions caused complete smearing of X-ray diffraction spots making the patterns uninterpretable. The simple estimation above suggests that continuing improvements in X-ray beam focusing are likely to lead to a situation when the beam spot size becomes suitably small in order to overcome this challenge. A further improvement in the pattern interpretation can be achieved by introducing some detector energy resolution or beam energy filtering, e.g. by the insertion of a monochromating crystal in the transmission configuration.
4. Focused Ion Beam milling and Digital Image Correlation

Focused Ion Beam milling offers a method of material removal that can be accomplished with minimal disturbance (and hence only negligible introduction or modification of residual stress). This fact served as the starting point for the development of a minimally destructive method for residual stress evaluation at the (sub)micron scale [16],[17]. The central postulate of the approach consists of the statement that when an approximately equi-axed “island” (a circle or a square) is machined at the materials surface by Focused Ion Beam milling of a “trench” around it (see Fig.3), then its surface becomes fully strain-relieved, provided the depth of the trench exceeds the island’s in plane dimension (i.e. circle diameter or diagonal of the square). It follows that the comparison of surface strain before and after the ion beam milling operation should provide quantitative basis for the evaluation of residual stress at the (sub)micron scale.

The fundamental assumption of complete stress relief provided by sufficiently deep trench milling has been verified by direct experimental measurement using synchrotron X-ray diffraction [18]. The X-ray instrument used for the study was DIFFABS diffractometer at synchrotron Soleil, France. The intention was to utilize a monochromatic X-ray beam collimated to the approximate spot size of 30×30 μm². A nano-multilayer of Cu and W was deposited on Kapton substrate and cut into a dog-bone sample shape suitable for in situ testing (Fig. 3). FIB-milling was used to create an array of rectangular islands each measuring ~1 μm diagonally, the entire pattern spanning the area of about 50 μm. This was done to ensure that appropriately positioned X-ray beam would only illuminate coating material that lies within the patterned area and is therefore fully strain-relieved. During in situ sample stretching experiment, the elastic lattice strain in the as-received coating was monitored, and compared with that deduced from diffraction data obtained from the patterned region. It was shown that substrate deformation had no effect on the lattice parameter of material within the islands, providing unequivocal proof of the full strain relief assumption.

4. Conclusions

The development of the methods for the evaluation of deformation and stress at the (sub)micron scales is being driven by the needs of modern micro-fabrication and micro-machining. Two kinds of approaches can be identified: non-destructive, based on the use of X-ray beams; and destructive (associated with material removal), based on the use of ion beams. It is worth noting the recent advent of another prominent candidate for the role of material-removing beam, namely, the femto-second laser beam [19].

Given the growing technological demand, in the next few years it is reasonable to anticipate active development of different flavors of these methodologies for residual and “live” stress and strain evaluation at the micro-scale. Furthermore, it is likely that efforts will be directed at the standardization of these approaches, combined with rigorous error analysis, round-robin testing activities, refinement of the experimental techniques and the associated data interpretation approaches, the assessment of measurement errors and reliability of results, cross-validation between techniques, etc.
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References