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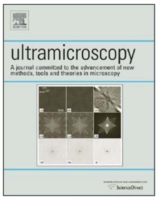

Broad ion beam serial section tomography

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ABSTRACT

Here we examine the potential of serial Broad Ion Beam (BIB) Ar$^+$ ion polishing as an advanced serial section tomography (SST) technique for destructive 3D material characterisation for collecting data from volumes with lateral dimensions significantly greater than 100 μm and potentially over millimetre sized areas. Further, the associated low level of damage introduced makes BIB milling very well suited to 3D EBSD acquisition with very high indexing rates. Block face serial sectioning data registration schemes usually assume that the data comprises a series of parallel, planar slices. We quantify the variations in slice thickness and parallelity which can arise when using BIB systems comparing Gatan PECS and Ilion BIB systems for large volume serial sectioning and 3D-EBSD data acquisition. As a test case we obtain 3D morphologies and grain orientations for both phases of a WC-11%wt. Co hardmetal. In our case we have carried out the data acquisition through the manual transfer of the sample between SEM and BIB which is a very slow process (1–2 slice per day), however forthcoming automated procedures will markedly speed up the process. We show that irrespective of the sectioning method raw large area 2D-EBSD maps are affected by distortions and artefacts which affect 3D-EBSD such that quantitative analyses and visualisation can give misleading and erroneous results. Addressing and correcting these issues will offer real benefits when large area (millimetre sized) automated serial section BIBS is developed.

1. Introduction

Accurate reconstruction and analysis of volumetric data are critical to understanding material microstructure in three dimensions (3D) and for improving the accuracy and efficiency of image-based 3D modelling [1]. Given the range of length scales for which 3D information is required a suite of methods have been developed including mechanical serial sectioning [2–5], microtomy [6], SEM ultra-microtomy [7–9], laser sectioning [10], Xe$^+$ Plasma Focused Ion Beam (PFIB) sectioning [11], Focused Ga$^+$ Ion Beam (FIB) sectioning [12–14], 3D Transmission Electron Microscopy (3D TEM) [15], and 3D Atom Probe techniques [16]. Taken together they cover a very wide range of scales and they can be integrated into what has recently been termed correlative tomography schemes [17]. Here we examine the potential of serial Broad Ion Beam (BIB) Ar$^+$ ion polishing as an advanced serial section tomography (SST) technique for destructive 3D material characterisation that holds particular promise with regard to collecting data from volumes with lateral dimensions significantly greater than 100 μm and potentially millimetre sized areas.

Conventionally when applying serial section tomography it is assumed that the slices are planar and parallel, that the individual image slices in the X-Y plane are not distorted by the image acquisition system, and that the slice thickness in the Z-direction is the same for all slices. Post-processing and image segmentation of the data set usually only takes into account rigid body motions, i.e. X-Y alignment and rotation of individual slice images caused by the any stage movements between milling and imaging operations [2–4,12–14]. More advanced data analysis can allow for shearing and stretching to compensate for drifts arising during acquisition in scanning probe based imaging. For well-defined microstructures, advanced analysis use mutual information from optical micrograph-EBSD image pairs with a high level of similarity and Nelder-Mead based optimisation algorithms [1].

In practice slice thickness and orientation can vary from slice to slice [18,19]. This variation can arise from: offset errors in slice thickness calibration, variations in slice thicknesses associated with moving between milling and imaging operations, and interaction between the material surface and the 'cutting tool', e.g. surface charging effects [20], beam heating and fluctuations in the ion beam source. In addition, the surface of the slice may display some deviation from flatness. This depends on the sectioning method used; for

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example mechanical polishing often produces convex surfaces with dimples in the softer phases [21]. PFIB top-down cross-sectioning [22] and FIB based SST may generate pronounced topographical effects, e.g. curtaining and shadowing [23]. Finally, the 'cutting' process may induce changes in the material being imaged, e.g. mechanical polishing may transform cubic Co into the hexagonal phase in WC-Co hardmetal [14]. In particular, the FIB-irradiation process can lead to both geometrical changes (e.g., material redeposition or swelling), but also to changes in the intrinsic physical properties (e.g., crystallinity, elasticity, conductivity, electrostatic charge and residual stress) and chemical characteristics (e.g., surface composition and hydrophilicity) of the surface layer [24,25].

Serial sectioning by argon broad ion beam (BIB) milling [26] for 3D-EBSD data acquisition and visualisation [27] allows crystallographic information to be obtained at SEM resolutions from much larger volumes of material (250×250×100 μm^3 or larger) than is feasible with FIB based SST. BIB polishing produces clean undamaged surfaces yielding high quality electron backscattered patterns (EBSPs) even for difficult materials comprising hard and soft phases such as hardmetals [21], as will be shown later in Section 3.1. However, sections may be non-planar with pronounced topographical effects, e.g. curtaining and shadowing [26,28], which locally distort the EBSD maps. Irrespective of the polishing method, the long acquisition times and high e-beam incidence angle[2] associated with EBSD mapping can additionally introduce image drift (time-varying distortions) and long-range image distortion (spatial distortions), e.g. trapezoidal and rhomboidal distortions [29]. As a result, the EBSD reconstructed microstructure can misrepresent the true structure, leading to inaccurate measurement of microstructural features both in 2D and 3D. By contrast, SEM images can be acquired at normal angle to the sample surface (minimising spatial distortions) within seconds (reducing time-varying distortions), giving image distortion below a pixel. A very efficient way of reducing time-varying distortions in the SEM is the acquisition of SEM images by integration over several frames (6–10) collected at short dwell times of about 3 µs per point. This reduces time-varying distortions to < 0.02 pixels [30].

This paper looks at the feasibility of obtaining accurate 3D reconstructions by serial section BIB polishing looking particularly at 3D EBSD acquisition through the consideration of characterising a hardmetal. These materials are widely used in machining, cutting, mining and drilling tools for example in the oil and gas industries. Hardmetals contain a high fraction of a ceramic phase (here WC) held together by a ductile metallic phase (here Co) and are typically formed by liquid-phase sintering. The mechanical properties of cemented carbides strongly depend on the size of the WC grains [31,32]. During sintering the average grain size coarsens, sometimes leading to a bimodal grain size distribution where some grains are significantly larger than the average through abnormal grain growth (AGG) [33–35]. Until recently the grain size distribution and the coarsening have been studied using planar images [34,36] along with methods that transform the 2D size distribution to 3D (the inverse Saltykov method) [14,33]. Recently, the 3D grain size distribution has been studied using 3D-EBSD in the FIB-SEM [14], however this has been limited to fine grained materials because of the small volumes (< 50 µm in size) that can be examined in this way.

Here we explore the use of two different Broad Ion Beam systems: one with a single wide Ar^+ ion beam (a Gatan PECS system), and one with two much narrower beams (a Gatan Ilion system) for large volume SST. We have identified and quantified uncertainties and artefacts arising from sample preparation and the data acquisition process and propose methods for their correction using WC-11%wt. Co hardmetal as a case study. While these proof of concept studies have involved manual transfer between the BIB system and the electron microscope and are thus time consuming (1–2 slice per day), future automated sample transfer methods [37] are likely to significantly extend the efficacy of the method for large volume, low damage serial sectioning for 3D imaging.

2. Experimental procedure and 3D reconstruction methodology

The workflow for large volume broad ion beam 3D-EBSD serial section tomography is conceptually the same as for SST using mechanical, FIB or PFIB sectioning. In essence a series of sections (layers) are successively revealed. After each material removal step in the BIB system the sample is placed in an SEM and crystallographic information is collected by EBSD mapping, followed by secondary electron (SE) image acquisition of the mapped area. EBSD maps of band contrast (BC) and orientation (with inverse pole figure colouring) (IPF) are used to form EBSD/SEM image pairs in the subsequent analysis to correct distortions of the EBSD maps. Typically, the co-registration of successive images is aided by markers introduced on the specimen surface, e.g. microhardness indents [1] or in our case using FIB milled markers (renewed after 6–12 slices). For FIB or PFIB serial section tomography the experimental procedure can be fully automated and performed within a dual ion and electron beam microscope [14,22,38]. By contrast, for large area mechanical serial sectioning material is successively removed outside the SEM [1,5]. An attempt has been made to incorporate a BIB system inside the SEM chamber [26], however in our study the sample is manually passed between the BIB system and the FIB-SEM (see Fig. 1). Subsequently, the datasets are post-processed/corrected, registered, segmented and the microstructure is reconstructed using dedicated 3D visualisation and analysis software. Considerable potential exists for employing automated workflows in the future.

2.1. Samples

The material studied in this investigation is a coarse grained (≈5 µm mean grain size) WC-Co hardmetal (11E) containing 11 wt% Co. The alloy was produced by Marshalls Hard Metals Ltd using conventional powder metallurgical methods. Two samples, one for the PECS and one for the Ilion broad ion beam system, were cut to 1×1×3 mm^3 and 1×3×4 mm^3 respectively using a diamond wheel. One side (1×3 mm^3) of each specimen was metallographically prepared by mechanical polishing using 9 and 1 µm diamond suspension in consecutive steps prior to BIB serial sectioning.

2.2. Broad ion beam sectioning

In order to determine the 3D microstructure of WC and Co grains, closely spaced serial sections were taken for each specimen using either the Gatan PECS or an experimental version of the Gatan Ilion broad ion beam systems (see Fig. 1). In essence, both systems use Ar^+ ions guns to mill/etch the material surface. The PECS system has one (5 mm full width at half maximum (FWHM)) gun whereas the Illion uses two (1 mm FWHM), etching/sectioning guns. In each case a mask/shield is used to selectively expose a layer of material (Fig. 2). In the Illion BIB the sample was mounted on the back of a titanium shield using 'silver dag' and aligned flat with the top of the back flat edge of the shield. During the milling the front edge of the shield is exposed to the BIB and gradually milled away with the sample top surface. After milling dozens of layers the top edge of the Ti shield becomes concave requiring replacement of the shield. A custom designed sample holder with variable tilt and specimen rotation for PECS and FEI xT Nova NanoLab 600i Ga^+ FIB-SEM (see Fig. 2 the top row) was used. The PECS system uses a molybdenum shield conferring a longer lifetime.

Prior to manual serial sectioning, a series of tests were performed to establish the milling conditions giving undamaged sample surfaces

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[2] Angle between surface normal and e-beam path, typically 70°.