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Characterizing deformed ultrafine-grained and nanocrystalline materials using transmission Kikuchi diffraction in a scanning electron microscope

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Abstract

The recent development of transmission Kikuchi diffraction (TKD) in a scanning electron microscope enables fast, automated orientation mapping of electron transparent samples using standard electron backscatter diffraction (EBSD) hardware. TKD in a scanning electron microscope has significantly better spatial resolution than conventional EBSD, enabling routine characterization of nanocrystalline materials and allowing effective measurement of samples that have undergone severe plastic deformation. Combining TKD with energy dispersive X-ray spectroscopy (EDS) provides complementary chemical information, while a standard forescatter detector system below the EBSD detector can be used to generate dark field and oriented dark field images. Here we illustrate the application of this exciting new approach to a range of deformed, ultrafine grained and nanocrystalline samples, including duplex stainless steel, nanocrystalline copper and highly deformed titanium and nickel–cobalt. The results show that TKD combined with EDS is a highly effective and widely accessible tool for measuring key microstructural parameters at resolutions that are inaccessible using conventional EBSD.

Keywords: Nanocrystalline; Ultrafine grained; Severe plastic deformation; Transmission Kikuchi diffraction; Electron backscatter diffraction

1. Introduction

Nanocrystalline, nanostructured and ultrafine-grained (UFG) materials offer properties that are vastly different from and often superior to those of the conventional microcrystalline materials [1,2]. Improvements include, but are not limited to, higher strength and hardness [3–6], enhanced fatigue resistance (B.L. Boyce, unpublished research; see also Ref. [7]), greater diffusivity, superior magnetic properties [8,9], and self-healing of radiation-induced damage through the absorption and recombination of point defects [10]. Nanocrystalline thin films, membranes, laminates, and coatings are becoming ubiquitous in micro- and nanoscale structures and devices. In contrast, processing limitations have slowed the widespread introduction of bulk structural materials, but grain size refinement by severe plastic deformation (SPD), primarily

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through equal channel angular pressing (ECAP) and high-pressure torsion (HPT) [11–14], have received much attention and hold considerable promise. Grain size refinement by SPD has the extra benefit of introducing additional nanoscale structures into the material, including dislocation substructures, nanotwins, and nanoscale precipitates, all of which can further improve the material's mechanical strength [15,16].

Detailed understanding of the processing–structure–property relations in these UFG and nanocrystalline materials requires quantitative characterization on the grain and subgrain scale, which poses a significant challenge. Transmission electron microscopy (TEM) is the most widely applied technique for studying nanocrystalline materials. TEM has the necessary spatial resolution, and electron diffraction analysis enables the measurement of crystallographic orientations on the nanometre scale, and recent developments in automated electron diffraction systems utilizing precession techniques [17,18] show much promise in enabling rapid collection of orientation maps on truly nanocrystalline materials. However, TEM analyses require significant technical expertise and are relatively difficult to perform. Moreover, the physical requirements of TEM make in situ testing significantly harder than they are by scanning electron microscopy (SEM). Many published analyses of nanocrystalline materials are based on interpretations of bright and dark field (DF) TEM images [19,20]; whilst clearly showing the dislocation structures, it is very difficult to determine effectively true grain sizes on the basis of such images because many estimations of grain size are all too often representations of intragranular cell structures with relatively low lattice misorientations.

For many fine grained and UFG materials SEM-based orientation mapping with electron backscatter diffraction (EBSD) has become the characterization technique of choice. EBSD enables the rapid measurement of phase and crystallographic orientations from polished surfaces of bulk materials, with sub-micrometre spatial resolution [21–23]. EBSD can also be used simultaneously with energy dispersive X-ray spectroscopy (EDS) to characterize the chemistry of a sample, albeit with poorer spatial resolution (typically of the order of 1–5 μm). SEM is also a versatile imaging platform, and the high sample tilt required for EBSD analyses is ideal for acquiring channelling (or orientation) contrast images using forescatter detectors mounted below the EBSD detector phosphor screen [24]. However, the spatial resolution of the EBSD technique is limited by the acceleration voltage of the incident electron beam and the atomic number of the sample: the best absolute resolution figures recorded for EBSD analyses on Cu are of the order of 30 nm, and for Al 100 nm [21,25,26], with significantly worse resolution down the tilted sample surface. These figures are mostly achieved using lower accelerating voltages in order to reduce the diffraction pattern source volume: however, reducing the accelerating voltage makes diffraction pattern collection slower and increases the influence of sample contamination and drift. It is clear that conventional EBSD is not an ideal technique for characterizing truly nanocrystalline materials. Even coarser grained, severely deformed samples are challenging to measure using EBSD, as the high dislocation density results in blurred or non-existent diffraction patterns and very low indexing rates.

In the last 1–2 years there has been significant interest in and development of an alternative electron diffraction technique using SEM, namely transmission Kikuchi diffraction (TKD) [27,28], sometimes referred to as transmission EBSD (t-EBSD), although technically it does not utilize backscatter diffraction. TKD involves the analysis of electron transparent samples, similar to those prepared for TEM work, and the collection of Kikuchi patterns projected from the underside of the sample using a conventional EBSD detector. The advantage of the TKD technique over conventional EBSD is the significant improvement in spatial resolution, shown to be in the range 2–10 nm for a range of materials. Trimby [28] demonstrated the potential of TKD in the SEM (SEM-TKD) for the routine automated analysis of nanostructured samples, and a number of recent publications have shown applications of this new technique [29–32]. However, many of these data sets were relatively small, or showed significant problems with the data quality, including numerous unindexed points, indexing errors or sample drift.

In this paper we demonstrate the power of SEM-TKD for characterizing microstructures of UFG and nanocrystalline materials that have undergone deformation, including several that have been severely plastically deformed, and discuss the impact that this accessible technique will have on our understanding of material deformation on the nanoscale.

2. Materials and methodology

2.1. Materials

Four different samples were chosen to illustrate the efficacy of characterizing nanoscaled materials with TKD. These materials cover a range of materials, microstructures and processing histories. The details of each sample are described below.

2.1.1. Nanocrystalline copper

A bimodal nanocrystalline Cu sample was produced using electron beam evaporation, creating a 200 nm thick Cu film on a photoresist substrate. The substrate was then etched away using acetone. The mean grain size of the nanocrystalline matrix produced using this technique was measured (by TEM image analysis) as 39 nm, with 26.1% of the area accounted for by coarser grains that had a mean grain size of 361 nm. The film was then strained at room temperature using a high-accuracy load cell to a final strain of 2.5% at a strain rate of $5 \times 10^{-6}\ s^{-1}$. Subsequent TEM analysis indicated a slight coarsening of the grain size in the nanocrystalline matrix to 45 nm.