Advance in large volume, serial sectioning for 3D EBSD structural characterization

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Electron backscatter diffraction (EBSD) is an analytical scanning electron microscope (SEM) technique that is based on local crystallographic orientation measurements. Typically, EBSD is done in two-dimensional (2D) plan views that are cut through a crystalline sample. While 2D studies are accurate and yield good results, there are instances where a three-dimensional (3D) characterization of sample volume is required to understand the material's properties or structural behavior.

Examples include habit plane determination, crack propagation, statistical grain size and shape measurements. Only 3D volume EBSD with five-parameter grain boundary analysis can provide a full understanding of grain boundary roles and influences on a material's properties. The most common technique used for 3D volume serial sectioning is focused ion beam milling (FIB). This technique yields accurate results; however, it has limitations:

- the observed volume dimension is small: 50 x 50 x 50 μm^3 for Ga FIB [1] and 150 x 120 x 80 μm^3 for Xe plasma FIB [2]
- the resulting FIB material damage to the sample requires damage removal steps prior to EBSD acquisition [3]

Lin [4] achieved a 3D EBSD volume of 150 x 100 x 80 µm by combining mechanical polishing and electropolishing to obtain a surface quality suitable for EBSD acquisition.

The area size limitations are not problematic for micro and nano-scale materials characterization. However, in the case of materials with larger grain size (> 10 µm) it is necessary to use a technique that allows large-area volume serial sectioning - significantly larger than 100 µm.

In this work, we describe a recent development in large, 3D volume serial sectioning using argon broad ion beam (BIB) milling. The technique permits preparation of large volume sectioning of up to 500 x 400 x 100 μm^3. Figure 1 shows a 350 x 260 x 21 μm^3 volume obtained from multiphase material: tungsten beads in a nickel matrix. The volume was obtained from 35 slices with 0.6 x 0.6 x 0.6 μm^3 voxel size.

The advantage of BIB over FIB is not only the ability to prepare large sample areas, but also the ability to prepare a sample surface that is suitable for high-speed EBSD acquisition without the need for damage removal steps; this includes multiphase materials with significant differences in hardness and milling rates.

The advantages and disadvantages of BIB and FIB techniques will be discussed and examples of mono-and multiphase materials will be presented. We will show also that it is possible to control slice thickness during volume preparation using described technique.

Ar broad ion beam (BIB)

350 x 260 x 21 μm^3

Ga focused ion beam (FIB)

50 x 50 x 20 μm^3

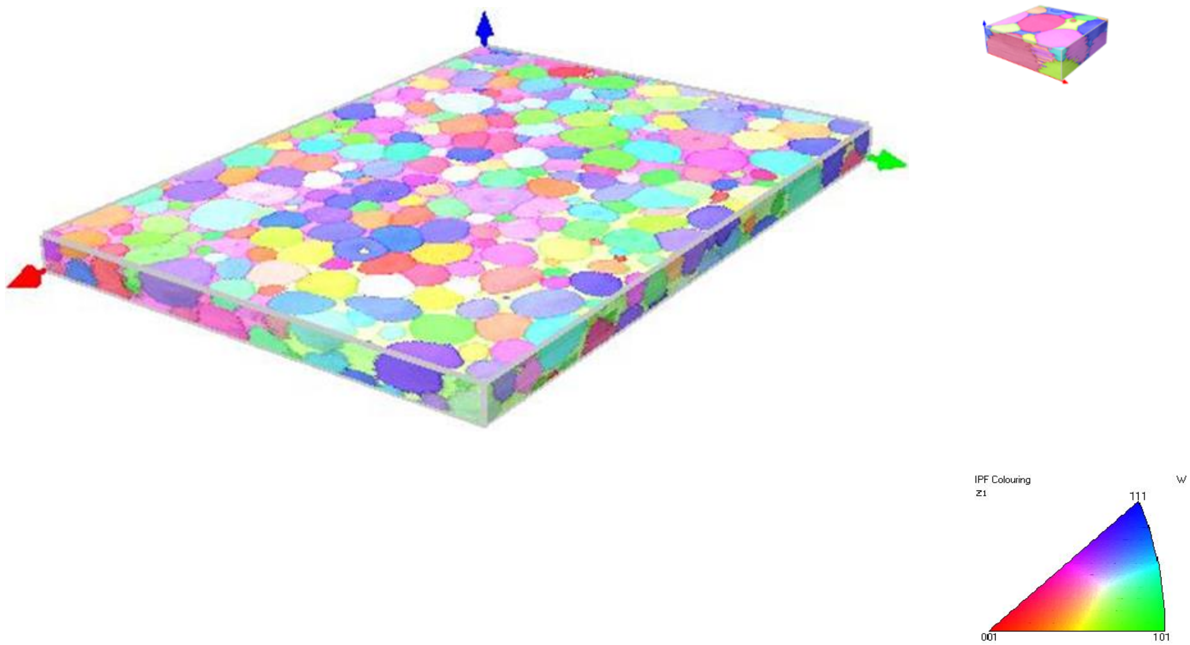

Figure 1. 3D EBSD volumes obtained from W-Ni composite materials using BIB and FIB.

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