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Automated Acquisition of 3D Grain Contrast Maps of Austenitic-Ferritic Duplex Steel and other Materials

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For many years, focused ion beam (FIB) systems have enabled users to straightforwardly cut cross-sections into metals and alloys, and obtain extremely valuable microstructural surface information by imaging the section using FIB-induced secondary electrons [1]. Thanks to the channeling-dependent penetration of the ions, outstanding grain orientation contrast is achieved. More recently, DualBeam systems, combining SEM and FIB, have demonstrated the unique ability to automatically and accurately cut a series of cross-sections with the FIB and sequentially collect information from the section surface, based on SEM images [2], EBSD [3] or EDS maps [4].

In this work, we are investigating how state-of-the-art DualBeam automated FIB sectioning and data collection can be expanded to provide 3D microstructural information, based on FIB-SE imaging. The principle used for sequential FIB sectioning and FIB-SE imaging is presented in figure 1. As the FIB polishing requires the beam to be parallel to the surface of each section (fig. 1a), a sample tilt and rotation step was introduced to allow for FIB-SE imaging at an angle of 52 degrees (fig 1b). Using this technique, we examined an austenic-ferritic duplex steel sample. A volume of $15x20x5\mu m^3$ consisting of a series of 120 slices, each spaced by 40 nanometers, was automatically processed in approximately 3 ½ hours (fig. 2) on an FEI Helios NanoLab 600i DualBeam.

The 3D FIB-SE data acquisition technique presents several benefits and challenges that are reviewed in the light of this use-case. Following our initial results, additional 3D datasets were acquired using an automation that allowed collecting both FIB-SE and FIB-secondary ions (SI) images from each slice, as well as some SEM SE/BSE images, in order to better understand how automated 3D FIB-SE and -SI differentiate from the more established FIB sectioning and SEM imaging techniques.

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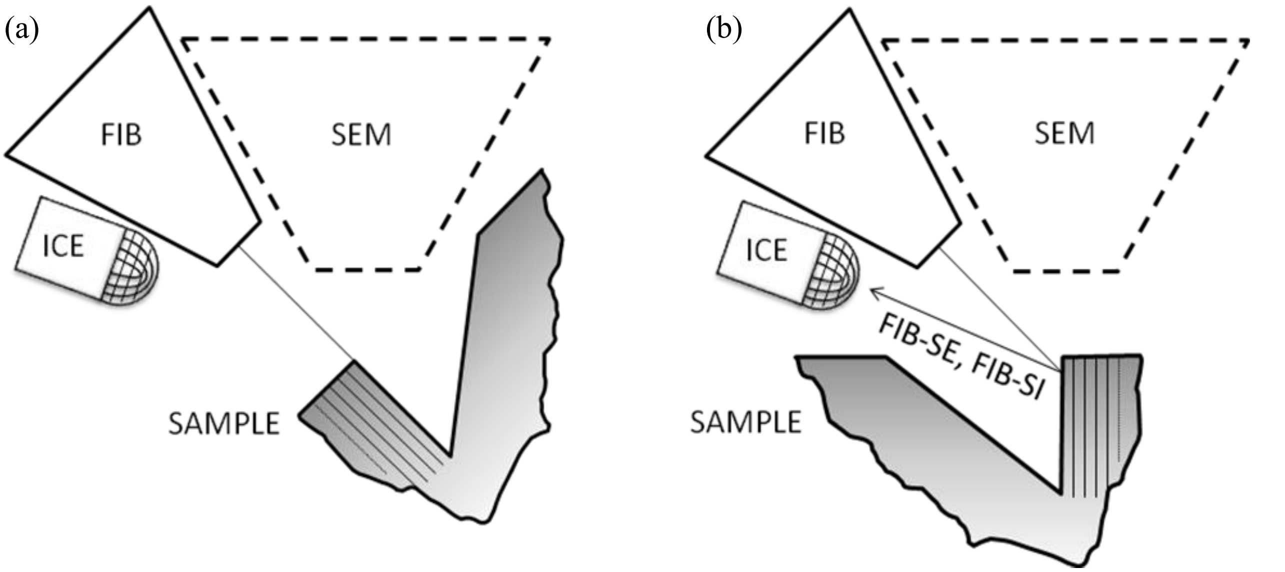

FIG. 1. Principle of sequentially (a) slicing with the FIB and (b) imaging each slice with FIB induced secondary electrons (FIB-SE) or secondary ions (FIB-SI) in a DualBeam. In this work, a dedicated FIB-SE and -SI detector (ICE) was used.

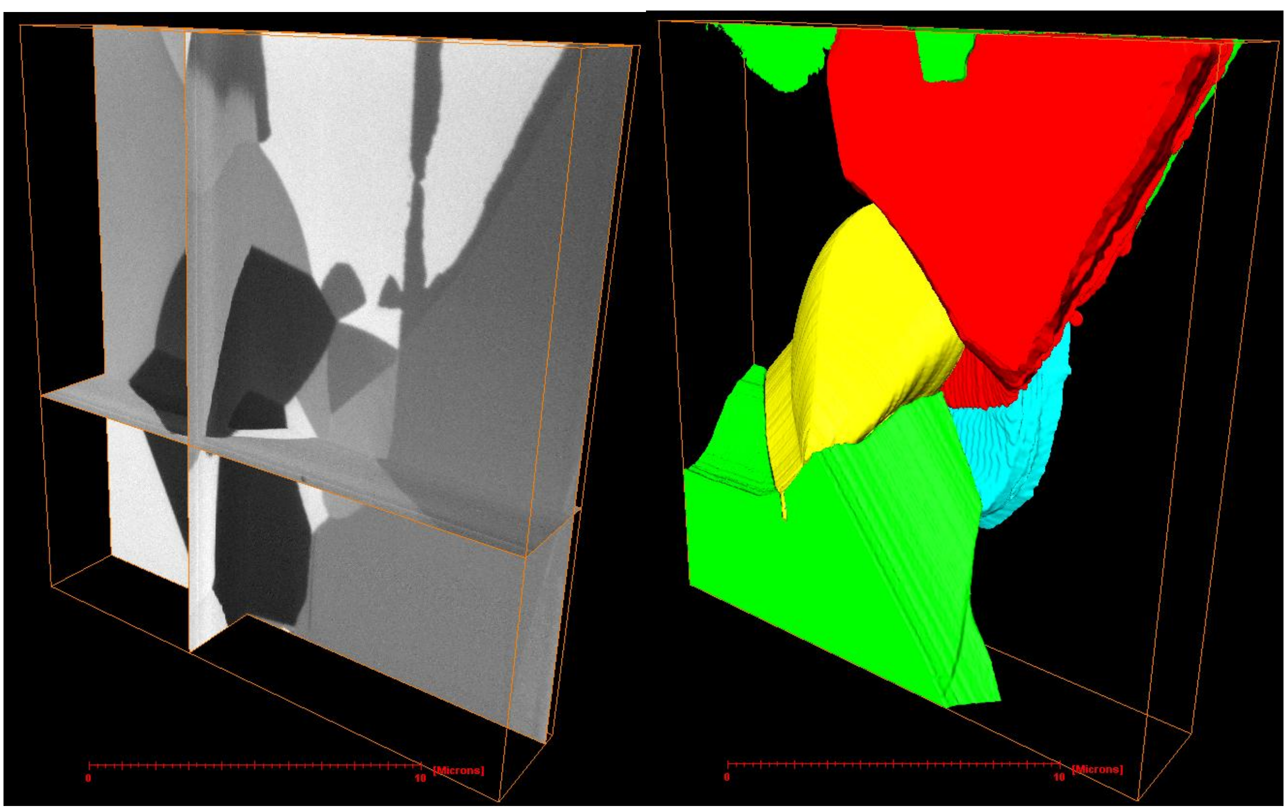

FIG. 2. 3D FIB-SE DualBeam characterization of austenic-ferritic duplex steel. The volume analyzed is 20 x 15 x 5 μm^3.

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