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The Preparation of Large Area Transmission Kikuchi Diffraction Samples From Bulk Material Without Requiring Lift Out

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Transmission Kikuchi diffraction (TKD) is an increasingly popular technique in the scanning electron microscope (SEM) for the characterization of nanoscale structures in wide range of materials [1-3]. The convenience of being able to utilize a conventional commercial electron backscatter diffraction (EBSD) system in a field emission SEM or focused ion beam (FIB) SEM has made TKD a particularly attractive alternative to orientation mapping techniques in the transmission electron microscope (TEM). However, as with TEM, there exist significant challenges in the preparation of suitable electron-transparent samples for TKD analyses and, in particular, for samples with sufficient surface area to be fully representative of the microstructure of the bulk material.

For the analysis of metals and alloys, sample preparation for TKD often utilizes standard electropolishing preparation techniques, with 3 mm TEM discs providing large electron-transparent regions adjacent to the central perforation. The use of standard lift-out techniques in FIB-SEMs are more appropriate for applications where site-specific electron-transparent samples are required or for samples where electropolishing is not an option. However, this is a process that is both time consuming and susceptible to high failure rates with the additional drawback that it will typically result in relatively small sample areas (e.g. a maximum of 30 x 10 um) and necessitates low energy clean-up milling steps to eliminate unwanted ion-beam induced damage.

Recently, a new workflow for in-situ TKD sample preparation has been developed [4]. This approach, involving milling of a sloping trench on the side of the sample, does not require any lift-out and can also utilize a pre-polished or as-deposited sample surface, negating the need for any subsequent low energy ion polishing steps. Following the preparation, the sample can be further tilted and then analyzed directly using a standard off-axis electron backscatter diffraction (EBSD) detector. For a conventional sized sample (e.g. a few 10s um field of view) the whole process, including sample preparation and TKD analysis, can be completed in less than 1 hour using a Ga-source FIB-SEM.

The latest generation of plasma-FIB (PFIB) instruments significantly speeds up the sample preparation workflow: for the same area of sample, the process can be completed in just a few minutes or, alternatively, significantly larger electron-transparent areas can be prepared in the same time. However, in this paper we consider the potential of in-situ large area (LA)-TKD sample preparation using a PFIB-SEM equipped with a femtosecond laser. The further increase in ablation rates now enables the rapid preparation of areas many 100s um across. The femtosecond laser has been shown to cause minimal thermal damage to the sample structure, making it possible for direct EBSD analyses on the laser ablated surface [5]. However, in most cases a brief glancing ion-beam milling step will further improve the surface quality.

A greater concern for the use of femtosecond lasers for LA-TKD sample preparation is the topography associated with light induced periodic surface structures (LIPSS); although these can be polished using the PFIB, care needs to be taken to balance between speed and quality when choosing PFIB conditions to avoid other types of topography, such as the corrugated structures seen clearly in figure 1. Here a Ti-6Al-4V alloy has been prepared using the in-situ LA-TKD sample preparation technique, using a TriBeam system with a Xe-plasma FIB and a femtosecond laser. The rapid PFIB milling, with low beam overlap, used to polish such large areas, have led to perforations in a number of locations. Even so, the LA-TKD orientation map (figure 1b) highlights the alpha-Ti laths across an area that would be challenging to prepare using conventional lift-out methods.

We will explore the limits of this new approach to LA-TKD sample preparation, with particular emphasis on the requirement to finish the process with a plasma-FIB clean up step to achieve more uniform sample thicknesses and subsequently improved TKD results.

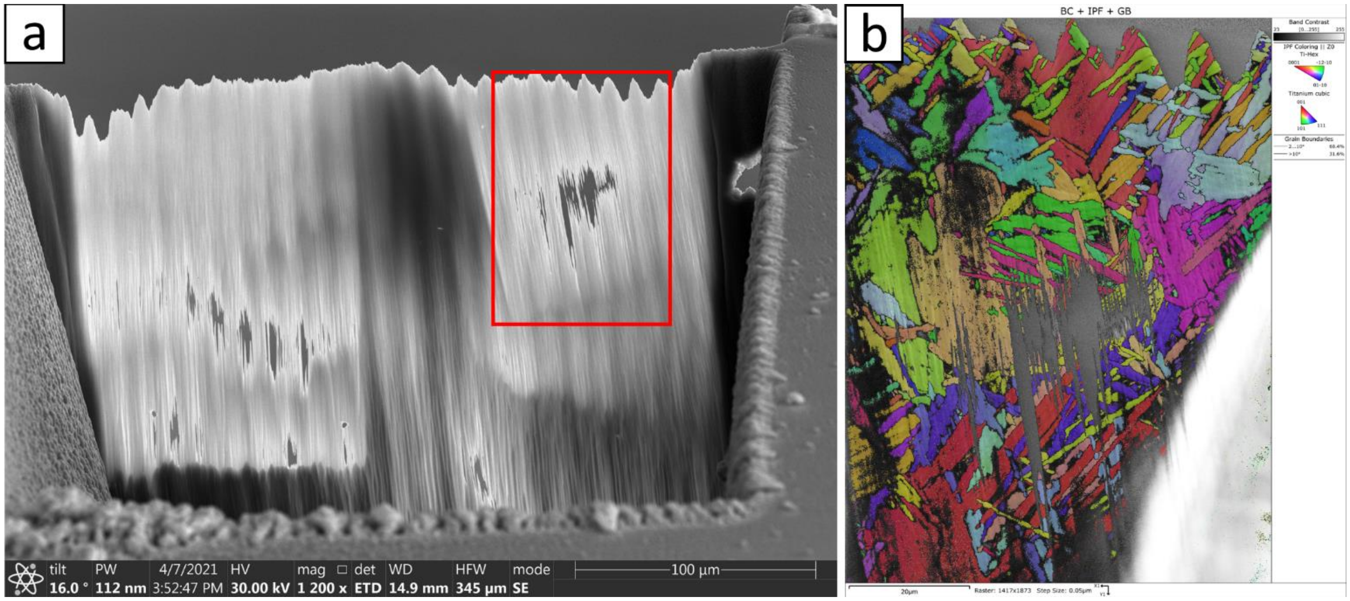

Figure 1. (a) Secondary electron image showing a 250-300 um wide electron transparent region in a Ti64 sample prepared using a fs-laser and PFIB. The red box marks the area analyzed by TKD. (b) Corresponding TKD orientation map (inverse pole figure z-direction) of a 70 x 95 um area. Note that shadowing onto the EBSD detector prevented robust data collection in the lower right area.

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