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**Meeting-report**

# Improved Sample Preparation Technique for Transmission Kikuchi Diffraction (TKD) Analyses Allows Large Area Data Acquisition

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Using an electron backscatter diffraction (EBSD) system and a scanning electron microscope (SEM) to analyze an electron-transparent sample is the basis of the transmission Kikuchi diffraction (TKD) technique [1, 2]. This technique offers crystallographic orientation mapping of electron-transparent samples, as well as bright field and dark field electron imaging possibilities, and a resolution of ~3 to 10 nm.

To achieve optimal TKD results, managing the sample thickness and uniformity are critical:

- If the sample is too thick, the SEM electron beam will scatter more broadly, which will cause a significant loss of resolution [2, 5], thus reducing the effective number of electrons that traverse the full sample thickness and produce Kikuchi patterns. This can cause an attenuation (or inversion) of Kikuchi pattern contrast [6, 7].
- If the sample is too thin, sample provides lower total scattering signal, i.e. there will be insufficient number of scattered electrons to enable formation of Kikuchi patterns [8].
- If the sample does not have an area of uniform thickness, accurate and fast TKD measurements may not be possible.

The focused ion beam (FIB) technique is a popular method for preparing electron-transparent samples [3, 4]. However, FIB sample preparation can introduce potential obstacles to optimal TKD analysis, such as structural damage and amorphization of the sample surface due to Ga or Xe ion interactions [9, 10]. This damage can cause complete attenuation of the diffraction signal.

The geometry of a standard FIB lamella also presents a challenge for preparing a sample for TKD analysis. A standard FIB lamella has dimension of 10×5 µm and typically has a wedge shape. Within that sample area, a thinner window is created. Only an approximate 3×1 µm area near the top of the lamella is thin enough for quality data acquisition [11]. This is relatively small area; a larger area is preferable, especially for TKD microstructural mapping.

We present a post-FIB sample preparation technique using a concentrated ion beam (CIB) milling system that overcomes the sample thickness, uniformity, and large area requirements for optimal TKD analysis. In addition, the resulting samples are free of structural damage and amorphization.

Figure 1 compares TKD measurements from a sample (cold-rolled Ni alloy) prepared by a Ga FIB system at 5 keV [Scios DualBeam, Thermo Fisher Scientific] and the same sample that was milled post-FIB preparation using an Ar CIB system at 500 eV [Model 1040 NanoMill® TEM specimen preparation system, Fischione Instruments]. The sample was milled from the backside using the CIB milling system, with the sample at a 15 to 20° angle relative to the Ar ion source. The TKD pattern of the FIB-milled sample (Fig. 1a, inset) has poor contrast and a 36% indexing rate, which revealed no microstructural details. After backside CIB milling, the same sample is much thinner and has a uniform thickness (Fig. 1b). The TKD patterns are sharp with very strong contrast (Fig. 1b, inset); the indexing rate is 97% and reveals microstructural details of the deformation.

The proposed sample preparation technique allows removal of FIB milling damage from the entire lamella and results in a uniform thickness over large area, as shown on Fig. 2. The TKD kernel average misoreintation (KAM) map shows a deformed Ni alloy microstructure after cold rolling (total sheet thickness was reduced by 95%).

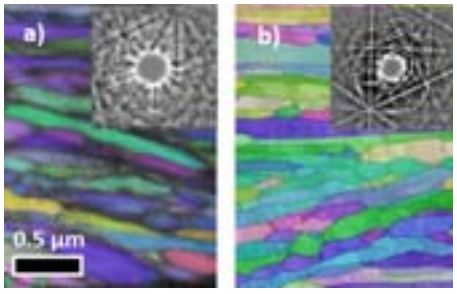


**Fig. 1.** TKD patterns and inverse pole figure maps collected from: a) a sample prepared by a Ga FIB system at 5 keV, b) the same sample prepared by an Ar CIB mill at 500 eV.

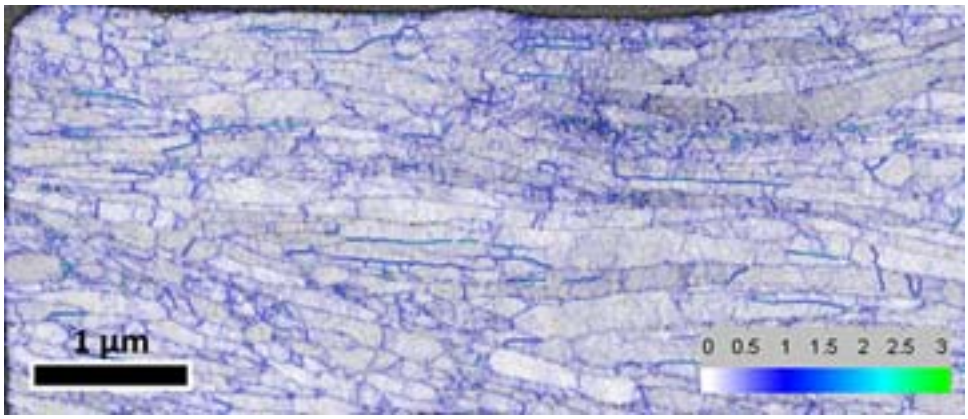


**Fig. 2.** KAM map shows a uniform sample thickness across a large area, which is ideal for accurate TKD measurements.

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