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Transmission Electron Diffraction Investigation of White Etching Areas in Bearing Steels: A Comparison Between TKD and TEM

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White etched areas (WEAs) are microstructural alternations in bearings induced by dynamic loading conditions. As indicated by the name, WEAs are more resistance to etching by chemicals and demonstrate "white" contrast under the optical microscope (Figure 1). The occurrence of WEAs can lead to the initiation of cracks, which can propagate and cause premature failures of bearing components. [1] Our previous investigations using transmission electron microscopy (TEM) [2] showed that the WEAs are very fine-grained (10-100 nm in diameter) severely plastically deformed zones. The grains in the WEAs are much finer than those in the original microstructure. Microcracks and voids were also observed in the WEAs, and pre-existing carbides are no longer visible. We emphasize that one of the major challenges working with WEAs is the high level of plastic deformation combined with a very fine-grained microstructure.

Recently, transmission electron backscattered diffraction (T-EBSD) or transmission Kikuchi diffraction (TKD) in the scanning electron microscope (SEM) has emerged as a very promising approach to characterize materials at the nano-scale. The spatial resolution provided by TKD can be below 10nm [3], which is a significant improvement over conventional EBSD. In most cases, this resolution enables characterization of such regions without the need for expensive TEMs or highly skilled operators. Together with a FIB-SEM system, this approach enables selection of a site-specific region, followed by preparation of a TEM thin foil and its characterization, all within a short time and all within a single instrument. This approach is proving to be a powerful failure analysis tool [3,4].

In this presentation, we will discuss the effect of FIB sample preparation conditions on data quality, and the advantages and limitations of this approach for samples containing WEAs (Figure 2). Data will be presented from samples prepared using FIB lift-out, as well as samples prepared using conventional TEM sample preparation methods, but recognizing the geometrical limitations imposed by TEM specimen holders. The TKD orientation maps and conventional bright field and dark field TEM data will also be compared with orientation mapping performed with a NanoMEGAS ASTAR system.

References:

[1] Evans M.H., *Material Science and Technology,* Vol 28, No 1, 2012, pp 3-22
[2] V. M. Bedekar, R. Scott Hyde and Rajiv Shivpuri, Microscopy and Microanalysis, **18,** Supplement S2, July (2012), p 1778.
[3] P.W. Trimby et al. Acta Materialia, 62 (2014) p, 69.
[4] A. Avishai, J. Carter, M. Z. Azar, B. Narayanan, and A. H. Heuer, Microscopy and Microanalysis, **19,** Supplement S2, Aug (2013), p 694.

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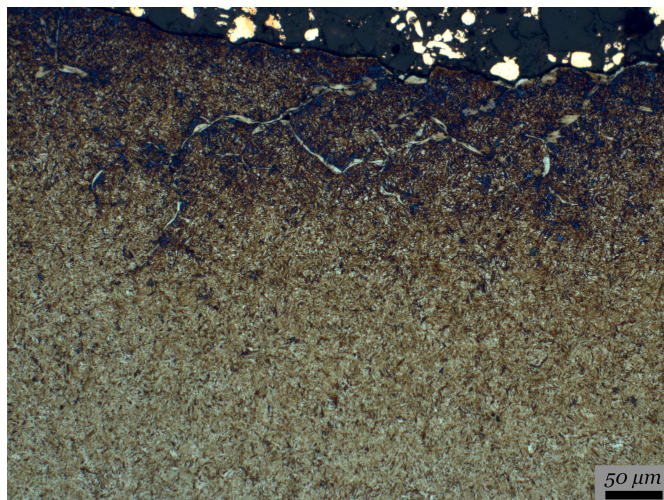

Figure 1. An example of white etching areas observed on a cross-sectioned sample using optical microscopy. The sample was mechanically polished and then etched with a 4% nital solution.

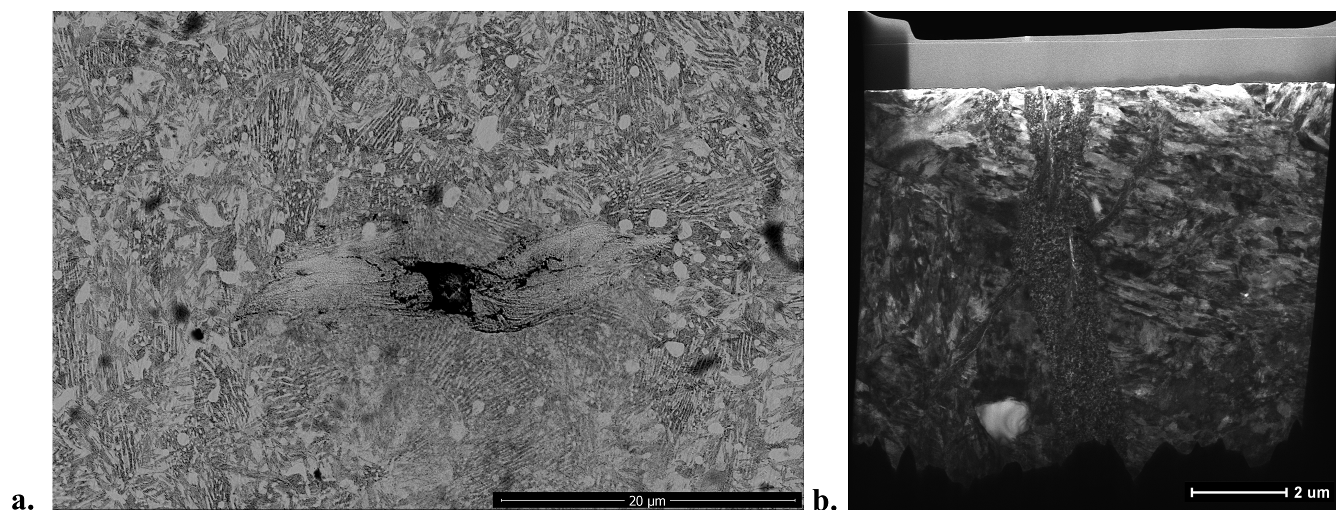

Figure 2. a). SEM image of the WEA region, b). a TEM lamella extraction from the WEA region showing the fine grain structure and microcrack at its center.

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