eCHORD crystalline orientation maps: channeling contrast at interfaces

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Background incl. aims
Every signal acquired by a scanning electron microscope (SEM) originates from a certain interaction volume below the surface, making localization of the received information challenging. For orientation maps obtained by EBSD, the Kikuchi patterns acquired at the interface between two grains contain contributions from both crystals. This impacts the precise localization of the interfaces on the generated map and thus, influences the spatial resolution when dealing with objects of a size comparable to the interaction volume [1]. For EBSD, this interaction volume can be hardly reduced, as Kikuchi patterns significantly weaken at accelerating voltages of 5kV and lower.

The same “blending” effects are expected in eCHORD orientation maps obtained using SEM since the channeling contrast also arises from a certain interaction volume beneath the sample surface [2, 3]. However, at such low accelerating voltages, electron channeling contrast can still be accomplished, hence orientation maps with a reduced interaction volume could potentially be obtained via the eCHORD approach.

Methods
When considering the orientation maps obtained using the eCHORD approach [2, 3], the raw data comprises of an image series captured by rotating the region of interest (ROI) within the SEM, with the sample being tilted at approximately 10-15°. Such an image series constitutes a datacube from which intensity profiles can be extracted, one for each beam position on the ROI, representing the variation of the backscattered electron (BSE) signal as the sample rotates. Similar to Kikuchi patterns, these intensity profiles are also influenced by the respective contributions of two neighbouring crystals.

To evaluate how this effect affects the eCHORD orientation maps, acquisitions were carried out on a copper thin film of 3 µm thickness exhibiting submicronic twins with 80 nm in thickness. The acquisition conditions are outlined as follows: 120 images (2048*1536) were captured using a frame time of 20.3s per image, at an accelerating voltage of 5kV, with a sample tilt of 15°, and utilizing a below-the-lens BSE detector. It is worth noting that an accelerating voltage of 5kV is now a routine condition for eCHORD experiments.

Results

Experimental evidences of a hybridisation effect are presented, showing that the intensity profile at a given interface is a blend between the intensity profiles of the two neighbouring grains. In addition to the interaction volume, various other factors explaining these hybridisation effects are presented and discussed: CHORD geometrical setup, image alignment, denoising, and indexing by pattern matching. Numerical simulations were also performed to quantitatively assess the effect of this hybridisation on the results of indexing intensity profiles, leading to a proposed adjustment in the indexing algorithm for the eCHORD method. This adjustment involves deriving the experimental as well as the theoretical profiles of the CHORD database prior to indexation, aiming to limit the effect of hybridisation on the final orientation maps. We demonstrate that this new algorithm has beneficial effects on indexation quality in general, compensating for differences between simulated and experimental profiles.

Conclusion

An effect of hybridization at interfaces has been observed in eCHORD orientation maps, which is limited by the adjustment of the indexing algorithm, thus demonstrating its robustness against degraded data.
This work represents an intermediary step in a more comprehensive approach aiming to enhance the spatial resolution in orientation maps obtained by eCHORD. This is achieved through the reduction of the accelerating voltage, improving the acquisition procedure, and by enhancing the post-treatment operations.

Graphic:

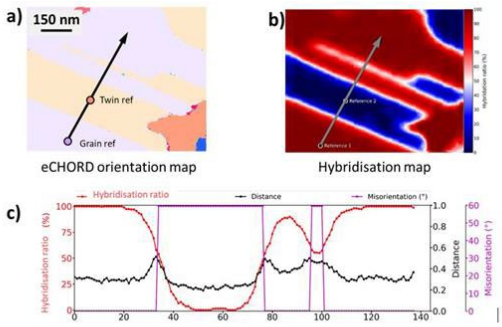

Figure 1: a) Detail of an eCHORD orientation map at 5kV, tilt 15°kV. b) Hybridisation map from references in the grain and in the twin, c) Evolution of the hybridisation ratio (red), pattern matching quality ("Distance", black), and misorientation compared to grain orientation (purple).

Keywords:

eCHORD, orientation mapping, interfaces, SpatialResolution,

Reference:

[1] Brodu E, Winkelmann A, Seefeldt M. (2022) A Pattern Processing Method to Map Nanoscale Phases by EBSD. Microscopy and Microanalysis 28(3) 716-722. doi:10.1017/S1431927622000526

[2] Lafond C., Douillard T., Cazottes S., Steyer P. and Langlois C. (2018) Electron CHanneling ORientation Determination (eCHORD): An original approach to crystalline orientation mapping. Ultramicroscopy, 186, 146-149. doi:10.1016/j.ultramic.2017.12.019

[3] Lafond C., Douillard T., Saad H., Deville S. Meille S., Steyer Ph., Cazottes S., Langlois C. (2021), eCHORD orientation mapping of bio-inspired alumina down to 1 kV, Materialia 20 101207. doi: 10.1016/j.mtla.2021.101207