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Hollow-cone Dark Field (HCDF) Imaging for Nano-grained Mg: Experimental and Simulated Contrast

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The orientation of individual grains in polycrystalline materials has always been an interest to researchers, due to its close relationship with materials performance. The orientation determination of nanoscale grains has been a challenging issue over decades although a few possible techniques, such as conical dark field scanning or nano-beam diffractions [4], can be applied using transmission electron microscopy (TEM). Similar to the conical dark field technique where beam is rotated around the optical axis, hollow-cone dark field (HCDF) imaging is used in current study to provide orientation information of nanoscale grains with much higher spatial resolution [5]. The beam is tilted so that certain interested reflections (including close-packed planes such as {0002}, {10-10} and {10-11} of magnesium (Mg) in current study) are approximately at the optical axis center, then an objective aperture is applied to collect only information coming from the interested reflections. The contrast of the grain will then change as it rotates to different orientation, due to changing intensities of selected reflections. It is also worth mentioning that since only intensity information is being collected, the approach described here is inherently incapable of differentiating between zone axes belonging to the same family.

In current study, nano grain Mg-Gd (2.0 w.t.%) was prepared for experimental image contrast comparison by mechanical polishing using a MultiPrep™ polishing system manufactured by Allied High Tech company, the averaged grain size is determined to be ~170 nm. The experimental and simulation HCDF collecting angle (inner and outer radius of the objective aperture) is set to be between around 8.445-11.772 mrad.

In order to relate the experimental contrast to corresponding orientation, quantification of the contrast changing needs to be made. A multi-slicing algorithm developed by Kirkland [6] is adopted and modified to simulate the contrast of HCDF images normalized to incident beam of magnesium under different orientation. The simulation yields essentially high-resolution TEM (HRTEM) images, two examples of simulated HRTEM image viewed along $[11\bar{2}0]$, [0001] and their corresponding contrast oscillation against sample thickness are shown in Fig. 1. Given that the simulated HRTEM images are usually size ~a few nanometers in length, which is much smaller than one pixel in experimental HCDF images, the simulated intensity in HCDF images is calculated by averaging intensities of every pixels over each HRTEM images. It is worth mentioning that apart from influenced by orientation, the contrast of a given orientation oscillates with sample thickness too. However, this is proven to be less problematic since the simulation results suggest most simulated orientations, except few like $[11\bar{2}0]$, have similar oscillation period of ~40 nm. Thus if the sample thickness can be precisely measured, one can directly compare the contrast of experimental and simulated HCDF image normalized to incident beam, to find out the matching orientation. Contrast of a normalized experimental HCDF image taken from the nano-grained sample is shown in Fig.2 as an example, from which some grain with certain orientation clearly possess much higher normalized contrast. Further experiment on the thickness measurement needs to be made in order to relate it to the simulated contrast. It is also worth mentioning that some linear-like contrast can be seen inside some grains, which might be attributed to high density of stored dislocations, similar dislocation activity inside even smaller grains has been reported in ref [7]. A [0001] inversed pole figure of Mg is shown in Fig.3 for example, where the normalized contrast of various orientation simulated is plotted, the simulation thickness is held constant around 20 nm for every orientations. Clear contrast difference between orientations can be seen.

Such technique requires only single HCDF image to give the grain orientation information, thus can be used to track the grain orientation in real time. Further investigation regarding using such technique to quantitatively interpret experimental images is still undergoing.

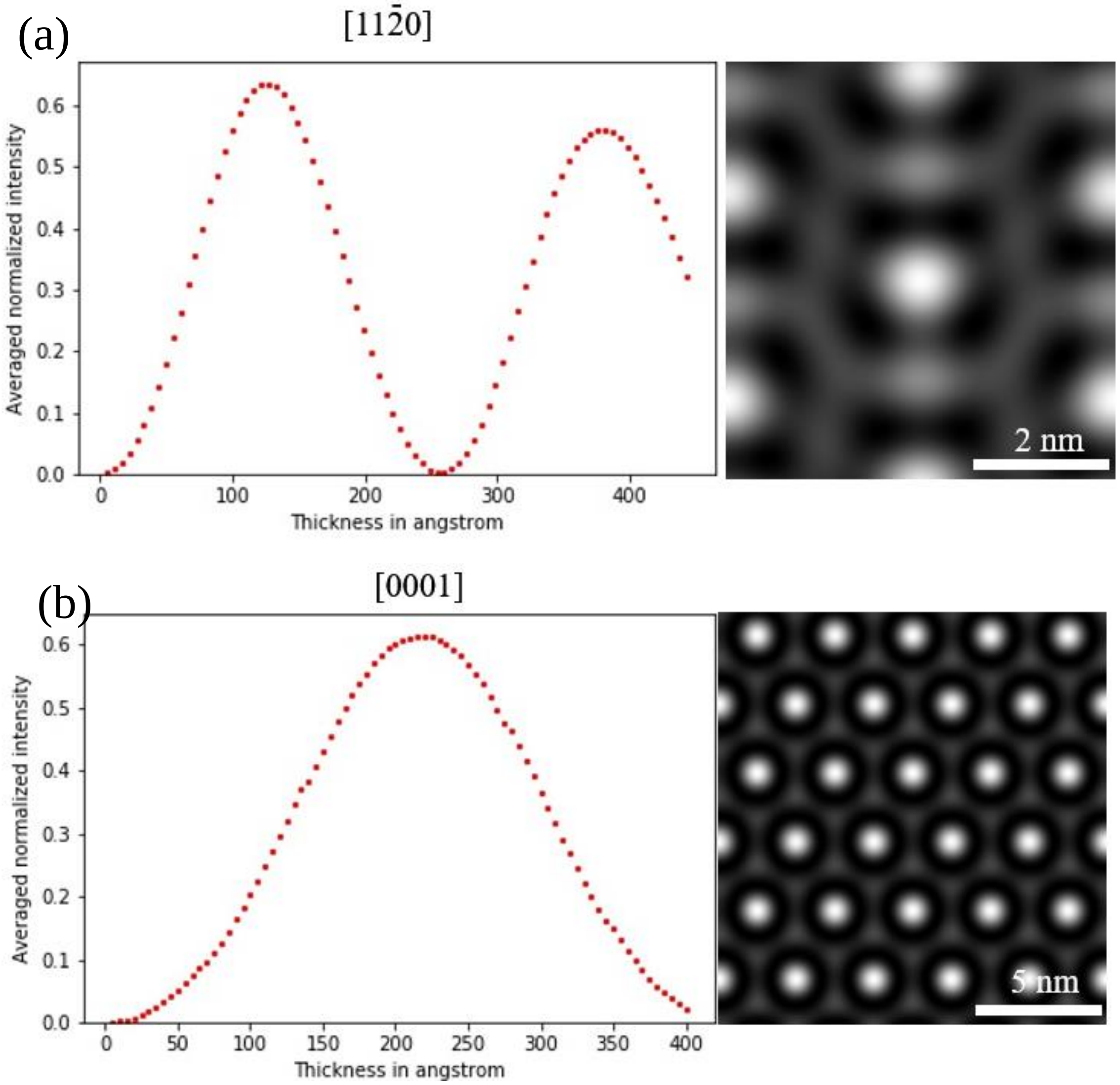

Figure 1. Simulated HRTEM image and corresponding contrast oscillation against thickness viewed along (a) $[11\bar{2}0]$ (b) [0001] using the modified multi-slicing algorithm.

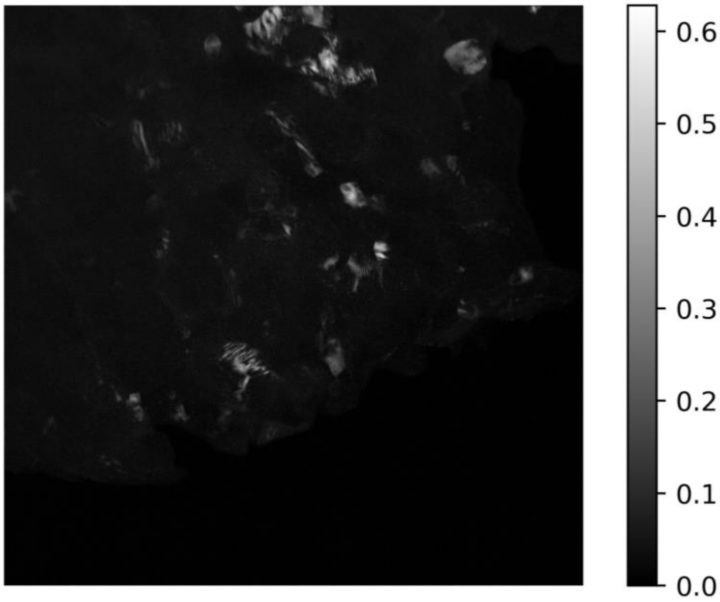

Figure 2. TEM HCDF image normalized to incident beam, grains rotated to certain orientation show clearly brighter contrast

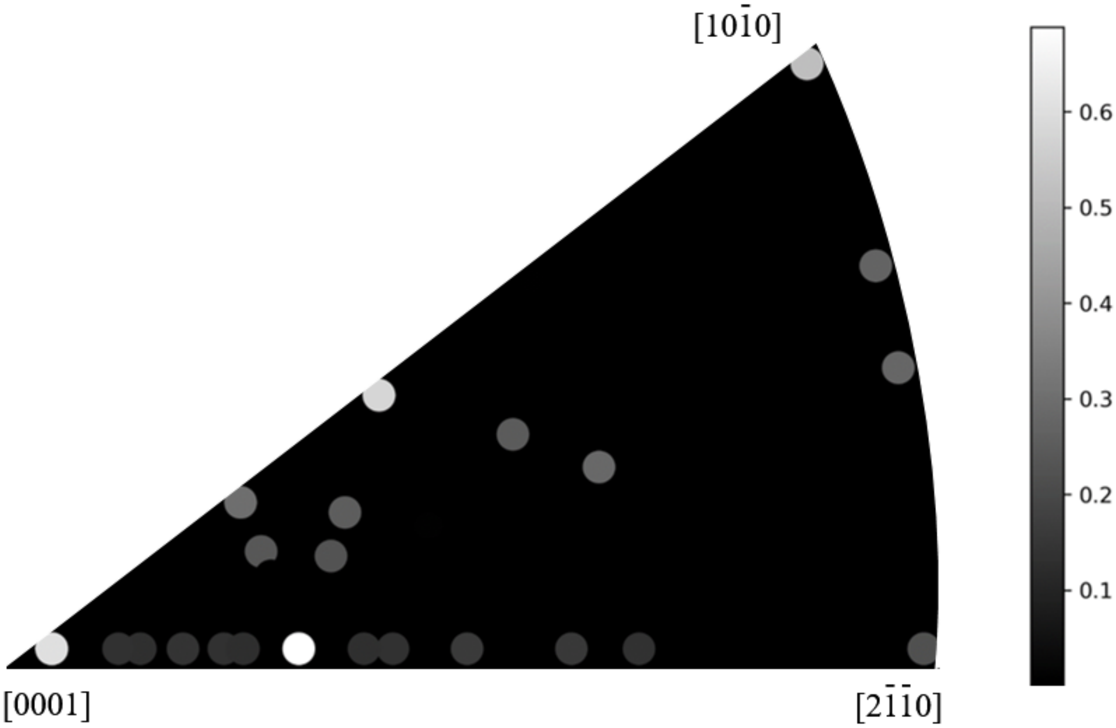

Figure 3. [0001] IPF of Mg showing normalized contrast of simulated orientations, simulation box thickness is ~20 nm.

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