doi:10.1017/S1431927615009113 Paper No. 0832
Microsc. Microanal. 21 (Suppl 3), 2015

Semi-Automated DigitalMicrograph Routine for Real-Time Phase Identification

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Individual nanocrystallite composition can be determined by comparing high resolution transmission electron microscopy (HRTEM) measurements of lattice spacings to the known lattice spacings of materials [1]. Without the aid of expensive software, however, this process can be overwhelmingly time consuming for even a single image containing multiple crystals [2]. Here, we describe a semi-automated routine that enables the user to rapidly identify the phases present in HRTEM images using the native acquisition software on most commercially available TEMs.

The algorithm, described below, has two main functions. The first is to overlay rings corresponding to some or all known lattice spacings of a chosen material in an electron diffractogram. The second is to filter the electron diffractogram at the selected spacings and then show in real space where each phase is located. The routine produces an intuitive graphical user interface (GUI) directly in DigitalMicrograph, which allows image processing without data type conversion or third-party software processing [3].

When first running the routine, the user inputs a text file containing the lattice spacings and corresponding hkl values for each possible phase present. The routine parses output text files from a standard X-ray diffraction database; adaptation to other formats is straightforward. As shown in Figure 1, the program loads a GUI containing lists of several different phases and their corresponding hkl values. The example shown in Figure 2 is a post-catalysis mixture of zinc and copper nanoparticles, where the exact composition is unknown. Here, we have chosen to examine the possible presence of zinc oxide, metallic copper, copper oxides, copper hydride, brass, metallic zinc, and zinc stearate.

Pressing ‘Generate FFT’ in the GUI computes the fast-Fourier-transform of the HRTEM image and displays the resulting electron diffractogram. Selecting a material in the GUI then overlays rings on the electron diffractogram at each material’s known lattice spacings. As shown in Figure 2c, rings at individual spacings are displayed by selecting only those corresponding hkl values. In this manner, determining which phases are present is done rapidly by inspection; in this case both zinc oxide and metallic copper are identified.

Once the user identifies all present phases, the contribution of each to the original HRTEM image is highlighted. Selecting ‘IFFT on selected’ in the GUI places ring-shaped masks of adjustable width and Gaussian tail length on the electron diffractogram at each selected spacing, generating an additional electron diffractogram for each masked region. The inverse-fast-Fourier-transform is then computed for each new electron diffractogram. As shown in Figure 2d-e, this routine produces real space images localizing each phase. The contribution from each hkl selection can be displayed separately to indicate specific lattice orientations if desired (not shown). Finally, the resultant real space images are colored and overlaid on the original HRTEM image. Figure 2f shows such an image, in this case the HRTEM image contains a cluster of ZnO nanoparticles surrounding a metallic Cu nanoparticle. The ability to quickly identify regions containing an interface between catalyst species should lead to a better understanding of the active reaction sites in nanoparticle catalytic systems [4].

References:
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[4] The authors acknowledge funding from EPSRC grant number EP/K035274/1. Dr. Bernhard Schaffer is thanked for his advice with scripting DigitalMicrograph.

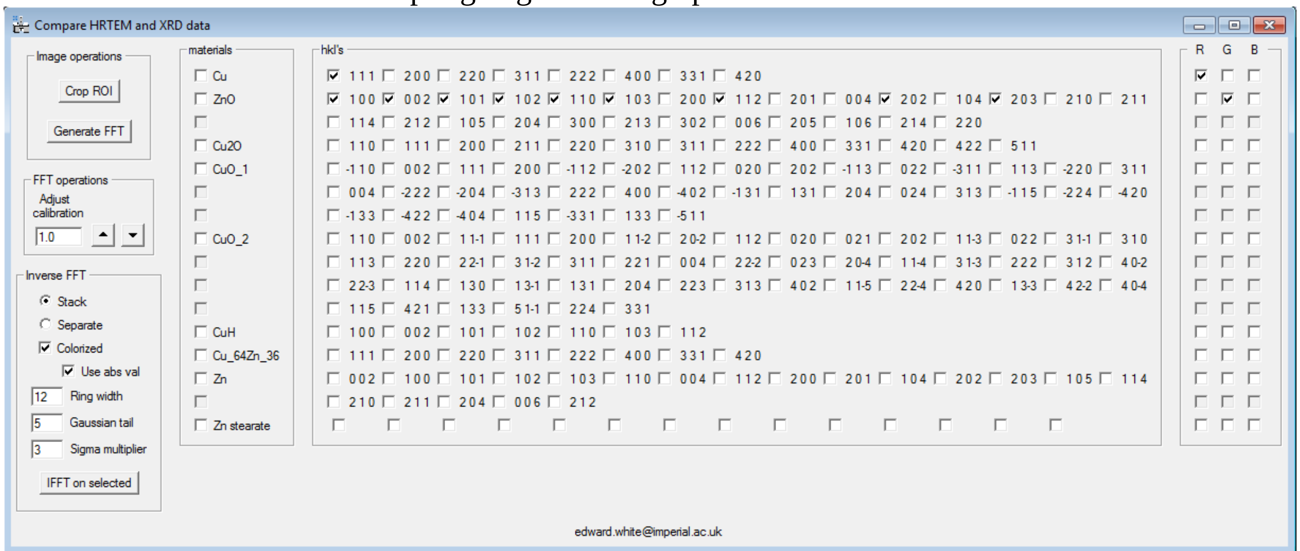

Figure 1. Graphical user interface for real-time phase identification DigitalMicrograph routine.

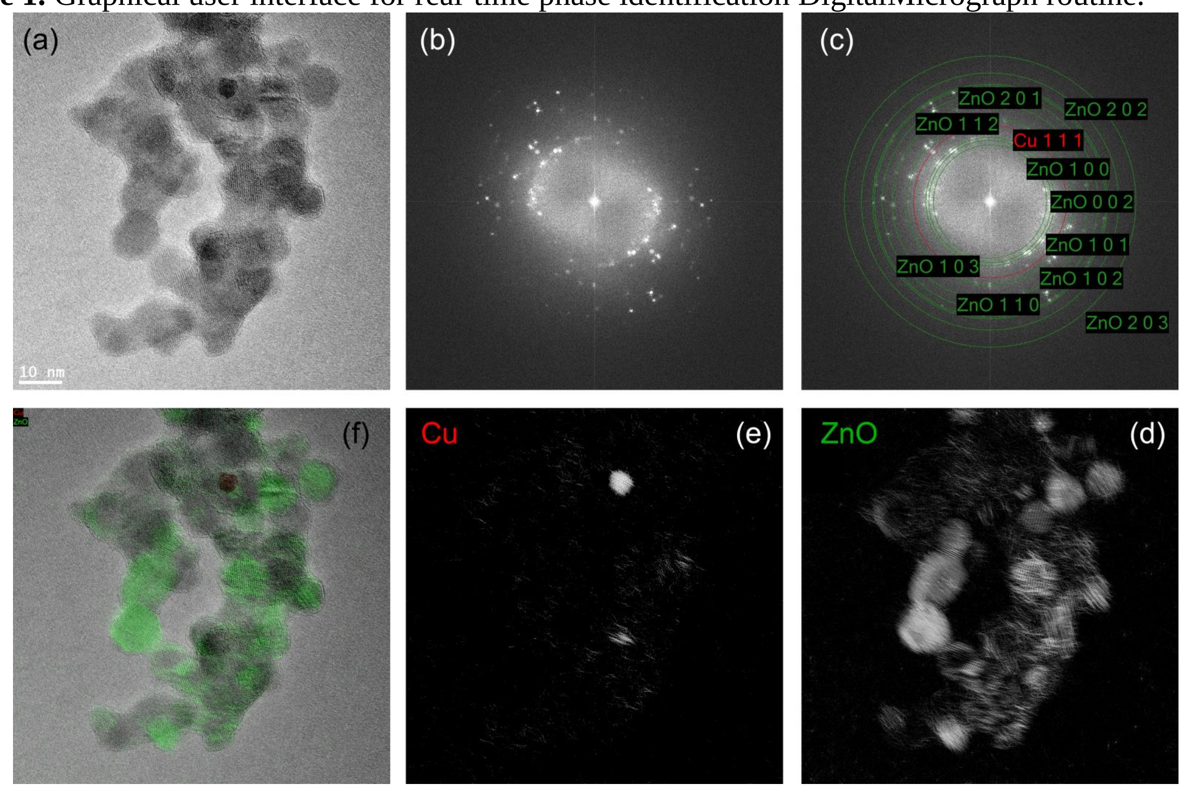

Figure 2. Example of routine in operation. (a) HRTEM image of nanoparticle cluster. (b) Electron diffractogram computed from (a). (c) Phase identification by selecting the hkl values shown in Figure 1. (d) Sum of all IFFTs with corresponding filters placed at ZnO spacings. (e) IFFT with a filter placed at the Cu(111) spacing. (f) Overlay of images (d) and (e) colored green and red respectively, on (a).

https://doi.org/10.1017/S1431927615009113 Published online by Cambridge University Press