

Experiment Brief

K3 IS Camera and STEMx System

Title

Virtual (BF/DF) imaging reveals the position and concentration of precipitates in a Ni-W alloy

Gatan Instrument Used

The K3™ IS camera delivers simultaneous low-dose imaging via real-time electron counting, fast continuous data capture, and a large field of view (FOV). In a 4D STEM experiment, the STEMx™ system precisely synchronizes the speed of the scanning probe to the camera frame rate to enable high-speed data acquisition and eliminates the potential for data loss.

Background

Ni-W based alloys have potential applications in the nuclear industry and national defense due to their superior corrosion resistance, mechanical properties, and higher density. The performance of these alloys can be tailored by the addition of a variety of precipitates at different compositions. Traditionally, dark-field (DF) TEM images are acquired to locate the precipitates in a thin film TEM specimen. If one is interested in covering a broad range in reciprocal space, then the objective aperture would be moved manually, and several images would have to be captured. This is a laborious and time-consuming task on the microscope.

Materials and Methods

A Gatan K3 IS camera, and a STEMx system were used to capture 4D STEM diffraction datasets at 4.8 ms/pixel rate in electron counting mode on a JEOL ARM 300F. The full dataset consists of an array of 342 x 213 pixel positions with a 1048 x 1048 pixel diffraction pattern acquired at each pixel position. The dataset took approximately 350 s to acquire. The dataset was binned 4x in the reciprocal space to result in a manageable size of ~4 GB. DigitalMicrograph® software was then used on an offline computer to process the data and generate the DF images to find the positions and concentration of the precipitates.

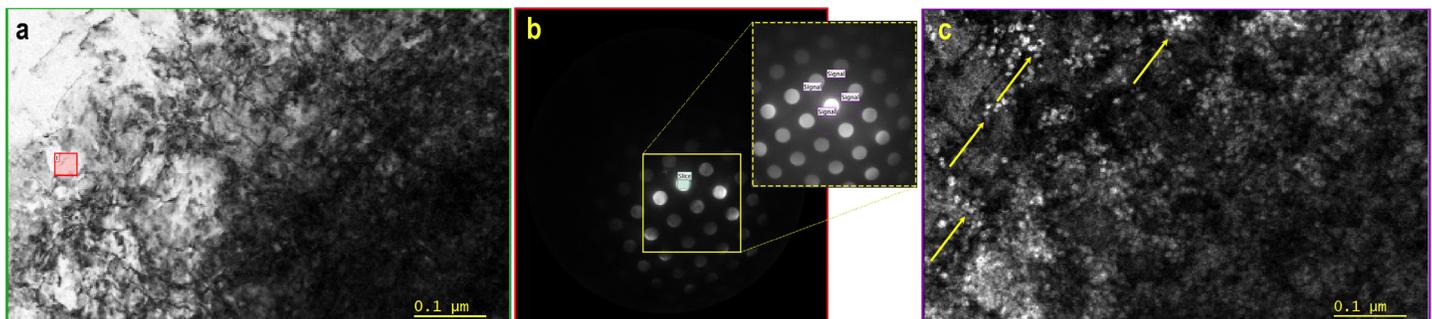


Figure 1. Virtual imaging of Ni-W based alloys. a) Virtual bright-field (BF) image generated from a virtual SAED aperture (green box in (b)). b) Virtual SAED image (sum of diffraction images from pixels in the red box in (a)). c) Virtual DF image highlighting the position of precipitates in the Ni-W based alloy (yellow arrows). The inset in (b) shows the four virtual objective apertures (purple) used to generate image (c).

Summary

Gatan K3 IS with STEMx enables hardware synchronized 4D STEM diffraction experiments at very high speed (>3500 fps at 256 x 256 pixel resolution) and at the highest possible signal to noise ratio (using electron counting). Unlike conventional DF imaging, 4D STEM gives access to a broad range of angles in reciprocal space after the specimen is removed from the microscope, and the TEM session is over. This not only saves valuable microscope time but also allows the user to design and optimize an unlimited number and combination of apertures (e.g., objective, SAED, annular, segmented) to generate virtual images tailored to highlight the feature of interest in their specimen. This can all be done on an offline PC during the post-processing of the data.

Credit(s)

A special thanks to Northwestern University, including Xiaobing Hu, Roberto dos Reis, and Vinayak Dravid

Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

This work made use of the EPIC, Keck-II, and SPID facilities of Northwestern University's NUANCE Center, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-1542205); the MRSEC program (NSF DMR-1720319) at the Materials Research Center; the International Institute for Nanotechnology (IIN); the Keck Foundation; and the State of Illinois, through the IIN.