COMBINING TOMOGRAPHY AND DIFFRACTION AT PSICHE BEAMLINE OF SYNCHROTRON SOLEIL

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Summary: Tomography typically reconstructs the distribution of attenuation coefficient to reveal the internal morphology of a sample. This scalar value does not uniquely identify the crystalline phase or composition of the material. We have developed a scheme for combining tomography with spatially resolved diffraction in order to add such information.

1. INTRODUCTION

Synchrotron X-ray tomography reconstructs the 3D internal morphology of a sample. The output is typically a 3D grey-scale volume, in which the voxel value is related to either the local attenuation coefficient (absorption contrast) or the electron density (phase contrast). Generally, the grey-level in the reconstruction can be interpreted as being proportional to local density. This information reveals a great deal about the structure, or the processes occurring inside a sample. In the ideal case the values reconstructed are quantitative. However, in the real world many factors may mean that this is not the case. For example, local tomography, polychromatic illumination, non-ideal detector response, the presence of x-ray absorption edges, and the difficulty in separating the phase and attenuation contributions to the projected image may all mean that the reconstruction is no longer truly quantitative. Furthermore, structures below the spatial resolution of the tomogram are not resolved, and instead an "average" value is reconstructed. Thus, in a sample of unknown, or incompletely known, composition, it can be impossible to relate grey-level to composition. Furthermore, two different crystalline phases may have very similar attenuation or electron density, and hence be impossible to distinguish in this way. In addition, we may want to know more about a sample than just the local composition. Elastic strain, the degree of plastic deformation, sub-resolution structure or porosity, and preferred crystalline orientation are all parameters that can help us to understand more about a given sample.

In this presentation we describe a scheme for combining synchrotron x-ray tomography with spatially resolved energy dispersive x-ray diffraction to add valuable complementary information. Both techniques can be performed at the same experimental station, and without un-mounting the sample. Thus, by aligning the two systems, the tomographic reconstruction can be used to guide the diffraction measurement. The ultimate aim is to produce an instrument analogous to a modern scanning electron microscope, in which the electron image can be complemented with electron backscattered diffraction (EBSD) or x-ray fluorescence signals.

2. EXPERIMENTAL METHOD

The PSICHE beam line of Synchrotron SOLEIL is a multipurpose beam line, which performs both diffraction, mainly for samples at extreme conditions, and tomography for materials science as well as diverse other applications [1]. The combined tomography and diffraction developments have focused on the white beam station of the beam line, which is used for fast parallel beam tomography using filtered white beam illumination, and energy dispersive x-ray diffraction (EDD). EDD is performed using the combined energy and angular-dispersive structural analysis and refinement (CAESAR) instrument [2]. EDD is suited to this experiment because the measurement is spatially resolved in 3D. The volume sampled is defined by the intersection of the incident and

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diffracted beams, referred to as the gauge volume. The detector is positioned on a unique high precision rotation stage, allowing the diffraction angle to be change while maintaining a precise alignment of the gauge volume within the sample. Figure 1(a) is a schematic showing the instrument.

For the combined measurement, the instrument is carefully aligned so that the axis of rotation of the tomography coincides with the gauge volume of the diffraction measurement. The sample is first scanned using the tomograph, and the tomogram reconstructed. A Python function is then used to visualise the reconstruction, and to select points for investigation using EDD. The function calculates the motor positions required to bring the desired point in the sample to the gauge volume. The tomography rotation axis provides an extra degree of freedom, which can be used to orient the sample, either to minimise the path length of the beam through the sample, or to align the diffraction scattering vector with a particular direction in the sample. It can also be used to oscillate the sample during the acquisition to improve crystallite-sampling statistics.

3. RESULTS

The proposed technique has been applied to a number of phantom samples in order to demonstrate the principle of the measurement (figure 1(b)). It has recently been used for an experiment with an external user group in order to identify and map crystallographic phases in archaeological samples [3]. The additional crystallographic information added assists in understand the corrosion and mineralisation processes occurring.

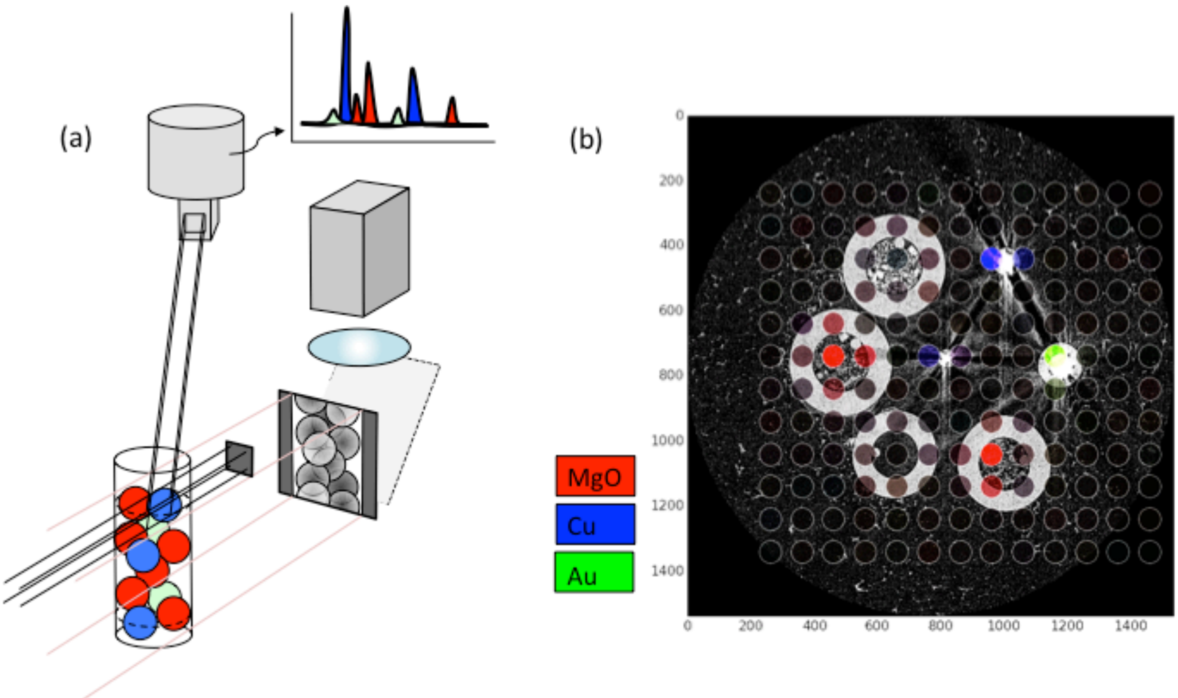

Figure 1 (a) Schematic showing the combined tomography and energy dispersive diffraction acquisition. (b) Combined tomographic reconstruction and diffraction mapping result for a phantom sample.

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