3D High Resolution X-ray Microscopy on Materials Characterization

Authors: Daniel Uliana, Fabrizzio Rodrigues Costa, Henrique Kahn

Affiliation: Lab. de Caracterização Tecnológica, Escola Politécnica da USP, São Paulo, Brazil.

The microscopy through high resolution X-ray tomography (or X-ray microscopy – XRM) provides a unique capability to characterize internal features like fractures, porosity, microstructures, defects in encapsulated parts, etc., by acquiring high resolution three-dimensional images (voxel size of about 1 μm³). Additionally, it provides advantages such as fast sample preparation, direct acquisition of volumetric data, quantitative analysis in 3D and less acquisition time [1].

The discovery of X-rays in 1895, by the German physicist Wilhelm Conrad Röntgen, provided a new way of imaging – radiography. The main advantage of X-rays is the ability to penetrate, nondestructively, in thick volumes of matter without changing it, recording the difference between its constituents, since they have natural contrast. In conventional radiography, the overlapping structures can create ambiguity as to their physical location; that problem can be solved by employing the tomographic technique [2].

The mathematical principle of computerized tomography (CT) was developed in the early 20th century by Radon [3], with unprecedented commercial application in 1973 from the first CT system developed by Godfrey Hounsfield in partnership with Allan Cormack, resulting in Nobel prize for Medicine in 1979 [4].

In CT, the radiation is transmitted along straight lines through the object to a detector and the reconstruction of projections produces a non-invasive evaluation of the structure from external analyzes. Thereby, the sample is rotated between the X-ray source and the detector to acquire projections in various positions. These projections are proportional to the amount of radiation reaching the detector, which varies according to the different coefficients of attenuation of X-rays of each material [2].

In recent decades, medicine is undoubtedly the field of application that most drove the research and development of X-ray tomography. Although the operating principle is the same used in materials science, mineral industry and other non-biological areas, the architecture of the equipment is different.

The laboratory microtomography equipment have a cone-beam design, which allows a quite small distance between the X-ray source and the sample (<1 mm). The image is normally obtained by converting X-rays into visible light by a scintillator and projected on a detector (charge coupled device - CCD). The resolution and field of view are controlled by the relative positions between source-sample and sample-detector, besides the X-ray source diameter and detector resolution.
The introduction of a set of lenses between the sample and the sub-micron resolution CCD detector, combined with a micrometer focus X-ray source, in 2007, boosted the high resolution tomography (<1 μm; Xradia-Zeiss patent), obtaining gain of at least one order of magnitude for these lens based systems.

The continued progress in improving the optics and instrumentation of X-ray techniques permitted achieving spatial resolutions below 25 nanometers, leading to its proliferation. Consequently, in recent years, the research emphasis has been changing from instrumentation to application, creating a demand for XRM systems simpler to operate as well as for automated processes for data acquisition and reconstruction of 3D images [5].

Cracks and fractures characterization in composites, steel porosity measuring and phase nucleation are examples applications in the field of materials science. In situ tests at varying conditions, such as tension, compression, oxidation, humidity and temperature variations are also possible with the use of specific accessories. Other examples are failure analysis for quality control and defects location in encapsulated materials in a nondestructive way.

Different problems of fluid transport in the pore scale can be analyzed by XRM 3D scanning of a porous structure coupled with computational flow simulation methods (4D) for determining the local flow from which the macro-flow properties can be obtained [6; 7], as in petroleum reservoir rocks characterization or acid leaching processes [8].

For biological samples, applications such as morphological characterization of bones, neural networks mapping and study of vascular systems are improved due to the high resolution and high contrast of current XRM systems.

Among many other advantages of XRM, there is the possibility of analyzing materials with little or no sample preparation, even for non-conductive materials and those impossible to analyze in vacuum, since the equipment remains at atmospheric pressure.

The main challenges today are the enhancement of image contrast for efficient differentiation between phases, especially for lower atomic number materials, besides the improvement of more robust applications for image processing.

References: