Computed Tomography Experiments on a Laboratory Multipurpose Diffractometer

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Abstract
Over the past hundred years a large number of analytical techniques based on X-rays has been developed. Nowadays X-ray diffraction is a well established method of solid materials’ characterization. Hereby the X-ray diffractometer is mainly used for phase identification and structure determination. The multipurpose diffractometer Empyrean allows to extend the capabilities of a standard laboratory X-ray diffractometer by other analytical methods. By simply exchanging few modules one can switch from classical powder diffraction to computed tomography, small-angle X-ray scattering or use X-ray scattering to perform pair distribution function analysis. In this contribution we present the application of soft (Co and Cu X-ray tubes) and hard (Mo and Ag X-ray tubes) radiation in CT experiments on various types of samples. A thorough qualitative as well as quantitative analysis of porous, oriented or multicomponent materials is demonstrated.

Keywords: computed tomography, x-ray diffraction, composites, fibers, porosity.

1 Introduction
Computed Tomography (CT) has been a well-established technology in medical diagnostics for decades. In the past few years, industrial CT has become a very powerful nondestructive analytical method. It can provide detailed information about the object of interest, e.g. its structure, composition, defect/pore sizes and their distribution. In this contribution we present CT measurements performed on the Empyrean platform – a powder X-ray diffraction (XRD) instrument that allows combination of conventional XRD applications (Powder XRD, Stress & Texture, μ-XRD etc.) with a CT experiment on one multipurpose platform. Due to different PANalytical’s area detectors, the CT capability can be extended from metal or heavy element containing objects (typical for industrial CT) to light materials (pharmaceutical products, polymer composites etc.).

2 Experimental
Experiments were performed on a PANalytical multipurpose Empyrean diffractometer equipped with Co (6.93 keV) and Ag (22.16 keV) X-ray tubes. The size of the analyzed samples varied depending on the dimensions of the used area detector. Soft radiation measurements have been performed using silicon chip technology detector PIXcel3D (14x14 mm²). Experiments with hard radiation have been carried out with help of CdTe photon counting detector GaliPIX3D (24 x 30 mm²). The measurement time varied between 15 minutes and 1.5 hours per sample depending on the used detector. Each measurement resulted in nine hundred 2D images, which were used to create a 3D reconstruction of the measured sample. 3D reconstruction as well as the analysis of the reconstructed objects was performed using VGStudio MAX 3.0 software [5].

3 Results and Discussion
3.1 Soft Radiation
A composite material, consisting of a polymer matrix and oriented carbon fibers was measured with Co radiation.

Figure 1: 2D radiograph (a), 3D reconstruction (b) and a plane projection (plane normal [0 0 1]) of a composite material reinforced with carbon fibers.

In Figure 1 the 2D radiograph (a) as well as 3D reconstruction (b) of a composite material reinforced with carbon fibers (a piece of an archery arrow) is presented. For this sample the Fiber Composite Material Analysis [5] has been performed. In order to estimate the fiber orientation, various plane projections have been evaluated (an example shown in Figure 1 c). To quantify fibers’ orientation, projection angle distributions have been plotted (see Figure 2).
3.2 Hard Radiation

In order to estimate the structural changes occurring upon discharging of an alkaline battery, a commercially available cylindrical battery (AAAA type) has been measured using Ag radiation in the original (1.5 V) and discharged (0.9 V) state. Upon discharging the outer layer consisting of MnO$_2$ is swelling by 0.2 mm (from ~1.4 mm to ~1.6 mm), which can be clearly seen both from 2D projections (Figure 3 a-b) as well as from the Wall Thickness Analysis [5] histograms (see Figure 3 c-d). This swelling leads to removal of the electrolyte (the gap between the outer layer and the central part becomes narrower). These observations are in agreement with results obtained on the same type of samples at the synchrotron facility [6].

At the same time changes in the material packing in the central part of the battery have been observed. Zn that is filling the central part of the battery transforms to ZnO, which is followed by increase of the packing density and, as a consequence porosity decrease of this material by 10 % is observed (discharged: 10 % vs. initial: 20 %).

4 Conclusion

Various CT experiments of good quality can be performed on both organic and inorganic samples by applying soft radiation, like Co and Cu, or hard radiation, like Mo and Ag. Once the 3D reconstruction is completed, different sample parameters can be extracted, such as porosity, orientation of certain components, distribution of the materials, size of inclusions, wall thickness or other features of the measured objects.

References


