X-RAY REFRACTION 2D AND 3D TECHNIQUES

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Summary: X-ray refraction techniques represent a very promising, yet not so wide-spread, set of X-ray techniques based on refraction effects. They allow determining internal specific surface (surface per unit volume) in a non-destructive fashion, position and orientation sensitive, and with nanometric detectability. While they are limited by the X-ray absorption of the material under investigation, we demonstrate showcases of ceramics and composite materials, where understanding of microstructural features could be achieved in a way unrivalled even by high-resolution techniques such as electron microscopy or computed tomography.

1. INTRODUCTION

Because of the low contrast, it is difficult to detect pores, cracks, or flaws in weakly absorbing or heterogeneous materials such as fibre reinforced composites and ceramics using standard X-ray absorption methods (X-ray radiology or CT). Analogous to visible light, also X-rays display refraction effects when interacting with matter. X-ray refraction follows Snell's law, with the peculiarity that the refraction index for matter is $n = 1 - \varepsilon < 1$, where $\varepsilon \sim \rho\times\lambda^2$ (ρ electron density, λ radiation wavelength). It is to notice that the refracted intensity and the scattering angle (always below the critical reflection angle, *i.e.*, between a few seconds and a few minutes of arc) they both depend on Δn between two neighbouring materials or between air and the material under investigation. Therefore, increasing the X-ray energy to better penetrate matter would be counterproductive to refraction effects.
Refraction effects occur at very low scattering angles (this is why they are difficult to detect), and are exactly the physical origin of what in the computed tomography community is called phase contrast. They can be used to detect cracks and interfaces in the bulk of materials, which would otherwise not be directly observable by absorption-based imaging techniques, because of the limited resolution.

2. EXPERIMENTAL METHOD

Several techniques were developed in the last decades, mostly at BAM:

[1]. ***X-ray refraction topography (XRRT)*** [1] is based on sample manipulation (scanning) of a (usually flat) sample through a pencil beam produced by a standard laboratory X-ray analytical source. It is therefore a radiographic technique, whereby the spatial resolution of the image is basically determined by the beam cross section. The latter is typically about 2000µm × 50µm (width × height). Those beam dimensions are determined by a compromise between intensity and spatial resolution.

[2]. ***X-ray refraction radiography (or imaging, XRRR)*** is based on direct 2D imaging of a sample (or a region of it). This technique is mostly implemented at a synchrotron source (synchrotron X-ray refraction radiography, SXRR), where a parallel beam with a typical cross section of about 10mm × 10mm (depending on the X-ray energy, see below) allows using a CCD camera, and imaging whole objects. This technique has been referred to as diffraction enhanced imaging (DEI) in the literature [2]. This technique uses an analyser crystal (placed between the sample and the detector) to select beam directions. In the symmetric position, the analyser crystals selects only the rays transmitted forward, in asymmetric positions, refraction contributions will be included and transmitted contributions excluded.

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[1]. ***X-ray refraction tomography (XRCT)*** utilizes the basic concept of XRRT extended by an additional rotation of the specimens in a typical absorption-based CT measurement. The set-up on a laboratory X-ray refraction equipment foresees a rotation around each of the axes perpendicular to the beam direction, and the acquisition of one topogram (*i.e.*, a full 2D map) for each rotation angle. Tomograms are then reconstructed by classic filtered back-projection. The set-up at a synchrotron (Synchrotron Refraction Computed Tomography, SXRCT) needs a sample rotation around an axis perpendicular to the incident beam. Since the number of rotation angles is generally large (typically 1800), it is not practicable to measure analyser crystal rocking curves for each sample rotation angle, and a good compromise between measurement time and imaging accuracy can be reached by setting the analyser crystal position to the symmetric one (Bragg condition) throughout the whole refraction CT scan.

3. CASE STUDIES

Typically, light materials can be excellently imaged by X-ray refraction techniques. Therefore, ceramics and composites yield excellent examples of the application of X-ray refraction techniques. In particular, structural details with size below the resolution of computed tomography (even synchrotron radiation) can be detected, such as fibre de-bonding in epoxy composites reinforced with ceramics.

In Fig.1a, the 3D reconstruction of tomographic data of a Ti-matrix composite reinforced with SiC long fibres is shown. The high resolution of the synchrotron data allows detecting the carbon core of the fibres, and the main crack in the matrix. However, X-ray refraction tomography allows detecting (see Fig.1b) also fibre de-bonding, and fully appreciating the main crack length.

X-ray refraction techniques have been successfully used to:

- Detect and evaluate porosity and pore orientation in ceramics [3];
- Non-destructively and position sensitively determine porosity and pore size in ceramics [4];
- Map fibre de-bonding and inclusions in glass and ceramic reinforced composites.

Last but not least, X-ray refraction techniques are increasingly used associated with *in situ* mechanical testing, thereby providing unique insight in the failure and damage mechanisms of light materials.

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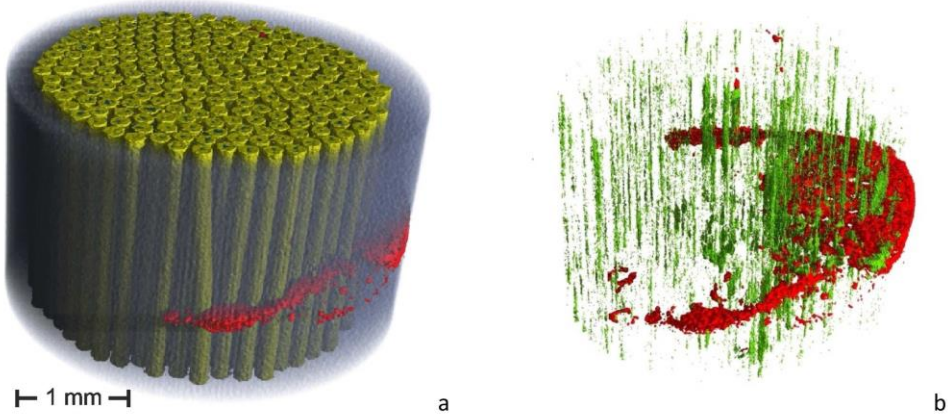

Figure 1: (a) 3D reconstruction of tomographic data of a SiC reinforced MMC tensile sample containing a main crack (red). (b) Fibre de-bonding (green) is made visible by reconstruction of 3D refraction data.