Damage characterization via 2D and 3D X-ray refraction techniques

Itziar Serrano-Munoz[*1], Bernd R. Müller[†1], Andreas Kupsch[‡1], René Laquai[⁂1], and Giovanni Bruno[‡1]

[1]BAM, Bundesanstalt für Materialforschung und -Prüfung, Unter den Eichen 87, D-12205 Berlin, Germany

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Summary: We present two examples of the potential of synchrotron X-ray refraction techniques. First, we focus on the 3D imaging of hydrogen assisted cracks in an EN AW – 6060 aluminium alloy which are otherwise undetected by absorption-based CT. The second work is a quantitative analysis of the damage evolution in an Al/Al_2O_3 Metal Matrix Composite during interrupted in-situ tensile load.

1. INTRODUCTION

In this poster we report the use of a powerful, yet not so wide-spread, set of X-ray techniques based on refraction effects. Driven by partly insufficient performance of conventional techniques in the case of hidden interface microstructures of relevance for material properties, X-ray refraction techniques were introduced a couple of decades ago [1], and have been successfully applied to non-destructive characterization problems [2, 3]. Refraction occurs at all interfaces (cracks, pores, particles, phase boundaries) where the density of the material changes, being more sensitive to density changes than attenuation. Due to the short X-ray wavelength, X-ray refraction techniques are able to detect the scattering response of nano-structures without actually imaging the individual interfaces. Therefore, it is possible to detect smaller cracks otherwise undistinguishable with conventional X-ray techniques at comparable spatial resolution. Moreover, X-ray refraction techniques allows quantification of the relative damage accumulated within a sample by measuring the internal specific surface (i.e., surface per unit volume).

2. EXPERIMENTAL METHODS

Synchrotron X-ray refraction is an analyser-based imaging (ABI) technique. A Si (111) single crystal, is placed into the beam path between the sample and the detector. Refraction at interfaces within the specimen causes X-rays to propagate at different directions than the incident beam. According to Bragg's law, only incident X-rays within a narrow range around the Bragg-angle are diffracted from the analyser into the detector. Hence, the analyser acts as an angular filter for the transmitted beam. This filtering allows to turn the refraction and scattering of X-rays into image contrast. Both the analyser crystal and the detector are positioned according to Bragg's law of diffraction, i.e, the analyser crystal is inclined by the Bragg angle θ_B and the detector is positioned at an angle of $2\theta_B$ with respect to the incident beam. Note that the refraction effect has a high defect orientation dependency because the filtering of the X-rays only occurs in the scattering plane of the analyser crystal, which is perpendicular to the crystal surface.

For ABI radiography. (hereafter Synchrotron X-ray Refraction Radiography, SXRR), the angular position of the analyser is rocked across a narrow range (about ±0.002°) around the Bragg angle θ_B in order to record the so-called rocking curve. We apply a peak fitting approach to extract the relevant information from the rocking curve. Using an algorithm based on X-ray refraction topography [4], we compute the *refraction value* image using the peak height and peak integral images. This refraction value is directly proportional to the inner surface density of the sample. The image of the refraction value is then reconstructed using a filtered back projection algorithm [5]. To extract the pure refraction information from the measurement, a conventional radiograph must be taken as well, and the linear attenuation coefficient is computed according to Lambert-Beer's law.

However, the recording a complete rocking curve for each projection angle of a CT measurement proves to be impractical due to the immensely long measuring time. Therefore, when performing CT, the analyser crystal remains fixed at the position of the maximum of the intrinsic (i.e., without sample) rocking curve (this technique is referred as Synchrotron X-Ray Refraction Tomography, SXRCT). A second CT measurement without the analyser crystal in place is conducted under the exact same conditions. Because this method requires a highly parallel and monochromatic beam, only synchrotron radiation can be used. The experimental set-up is installed at BAMline (BESSY II, Berlin).

*e-mail: itziar.serrano-munoz@bam.de
†e-mail: bernd.mueller@bam.de
‡e-mail: andreas.kupsch@bam.de
⁂e-mail: rene.laquai@bam.de
‡e-mail: giovanni.bruno@bam.de

3. CASE STUDIES

• **SXRCT** was used to investigate hydrogen assisted cracking (HAC) in an aluminium alloy weld (EN AW – 6060). It is well established that the welding process can induce significant hydrogen intake, although the mechanisms of HAC are not completely understood yet. Hydrogen-induced cracks are very small, especially during their first stages of formation, which makes their non-destructive detection difficult even by common synchrotron CT techniques. Because SXRCT technique enables the detection of cracks smaller than the effective resolution of the imaging system, it is possible to observe that the weld contains areas of high microcrack density (indicated in red, see Fig. 1a). The length and width of this area has been measured to be ~1 mm and ~0.5 mm, respectively [6].

• **SXRR** was combined with a tensile load rig (originally designed and built for tests in scanning electron microscopes but modified to fit to the refraction setup) to perform an interrupted tensile test where a dog bone-shaped specimen was imaged (~1 mm thickness using Al alloy (6061)/10 vol%Al_2O_3 MMC). The investigated area is 1.7×1.3 mm^2, the pixel size $(3.5 \, \mu m)^2$, and the energy of the monochromatic parallel beam is $E = 22$ keV. SXRR technique allowed to evaluate the 2D distribution of the specific surface C_m/μ at six different tensile load states. Here, the C_m/μ values are interpreted as the amount of damage caused by the tensile load. At loads below 550 N no change in the specific surface is observable. Above 600 N, the specific surface increases dramatically with increasing load and at 680 N, C_m/μ is increased by nearly 25% with respect to the initial undamaged state. Note that in conventional absorption-based imaging, only the last loading stage shows some indication of damage within the sample [7].

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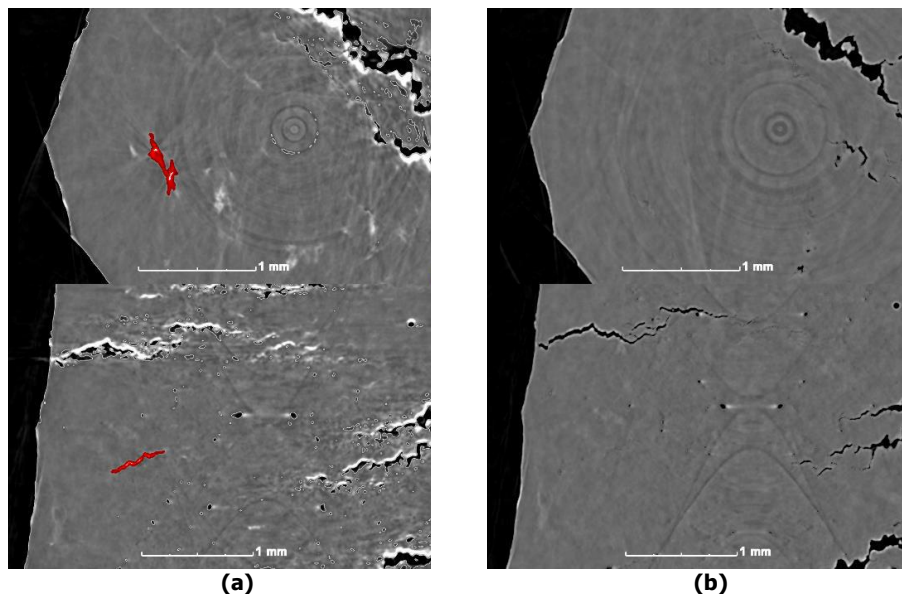
Figure 1: Selected tomograms through the reconstructed volume for: (a) SXRCT measurement where a 3D array of microcracks is highlighted in red and, (b) absorption-based SCT measurement where the microcracks are indistinguishable. The voxel size is (3.5 μm)^3 and E = 25 KeV [6].