Non-Destructive Micro Crack Detection in Modern Materials

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Abstract. For the purpose of nondestructive crack detection X-ray refraction is applied. Micro cracks are essential indicators for fatigue and degradation. The physics of refractive crack detection is based on the scattering of x-rays at the phase boundaries of different materials or gaps. The sensitivity for cracks is nearly independent of the crack width and it is independent from the spatial (scanning) resolution of the system, because the integral scattering intensity is a measure of the total crack density in a certain material. Due to topographic measurements (i.e. two-directional scanning of the sample) and high resolution x-ray refraction tomography, the spatial distribution of cracks is also detectable. In addition, the measurement of crack orientation can be performed with high accuracy. Even the correlation between crack density and mechanical parameters is possible. Mainly materials of low absorption like polymers, ceramics or composites are investigated.

1. Introduction

Major tasks within the field of non-destructive characterization of materials are related to the finding and application of structure/property relations like correlations between atomic, nano and micro structures with macroscopic properties. X-ray refraction topography contributes to this, as the structural selectivity covers a range of five orders of magnitude, from 0,1 mm to 1 nm, based on different scattering effects. The spatial resolution up to 10 µm is achieved by scanning techniques under pre-selected scattering conditions. The simultaneous detection of single defects covers classical NDT tasks as well.
The mechanical properties of materials are significantly determined by interface topology, micro cracks and pores. X-ray topography can easily contribute to reveal such correlations and exploit them for materials development, for quality assurance or micro damage analysis.

2. Principles of X-ray Topography

Small Angle X-ray Scattering (SAXS) is a classical tool of colloid, polymer and biological research. Particle dimensions smaller than 50 nm are determined by ‘Guinier’ and ‘Porod’ analysis (diffraction) [1], larger ones by refractive scattering which is well appropriate for crack detection [2, 3].
X-Ray Refraction Topography makes use of the well known optical effect of refraction at interfaces according to Snell’s law which for X-rays occurs at small scattering angles of a few minutes of arc. In a modified commercial Kratky camera a very narrow monochromatic X-ray beam of 18 keV Mo-K-radiation crosses a sample, which is scanned in the surface plane and scattering intensities are taken at all positions. The technique provides nearly linear contrast for specific surfaces and interfaces. A standard PC controls the X-ray count-

ers and the micro drives. The high quantum efficiency of scintillation counters permits measurements at reasonable speed, e.g. 1 s /step.
The refraction instrumentation with X-ray fine structure tube and Kratky camera is relatively straight forward. The camera needs an additional scattering foil for the primary beam attenuation measurement and a micro manipulation device for the sample (Fig. 1). For practical measurements the refraction detector remains at a fixed scattering angle increment above 2 minutes of arc and the relative surface density C (volume related specific surface) of the specimen can be measured according to [4]:

$$C = \frac{1}{d} \cdot \left(\frac{I_R \cdot I_{A0}}{I_{R0} \cdot I_A} - 1 \right) \qquad (1)$$

with intensities IR and IR0 from the refraction detector with and without sample respectively, IA an IA0 from the absorption detector and d the wall thickness of a sample. Apart from the choice of materials the relative surface density C depends merely on the scattering angle and the radiation wavelength. The absolute surface density is determined by comparison with a known calibration standard at retained boundary conditions (wavelength and scattering angle).

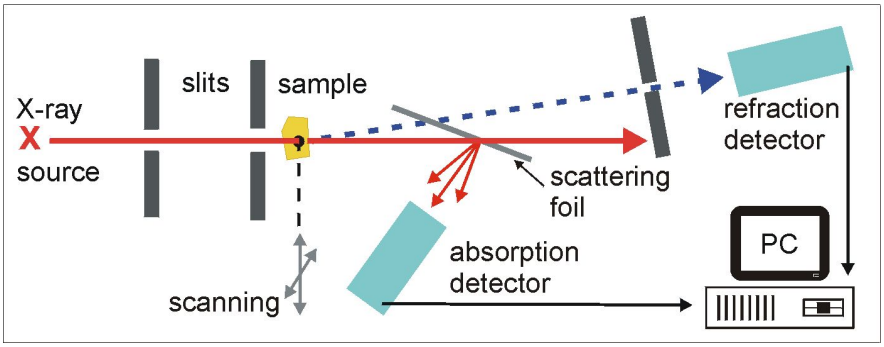

Fig. 1. Scheme of a X-ray refraction instrumentation with collimated X-ray beam, sample manipulator, a refraction detector for refracted intensity IR with sample or IR0 without sample and an absorption detector for the attenuation intensity IA or IA0 of the primary beam.

The inner surface of pores and particles can be determined as well as interfaces and cracks like fibre de-bonding in composites. A model composite has been made in order to demonstrate the refraction behaviour of a bonded and a de-bonded 140 µm sapphire fibre in a polymer matrix (Fig. 2, left). Fig. 2, middle shows the resulting intensity distribution of a two-dimensional refraction scan of the model composite. The upper ray crosses the bonded fibre-matrix interface causing a small amount of deflected intensity. At the de-bonded fibre and at the matrix surfaces (lower ray) much more X-rays are deflected, as of the larger difference of the refractive index against air. The polymer channel is clearly separated from the fibre surface.
For comparison a mapping of IA yields the transmission topograph containing only the absorption information of the projected densities like in conventional radiography (Fig. 2, right). In case of a real composite material the much thinner fibres are not spatially resolved, but the higher refraction signal of de-bonded fibres reveals quantitatively the percentage of de-bonded fibres.

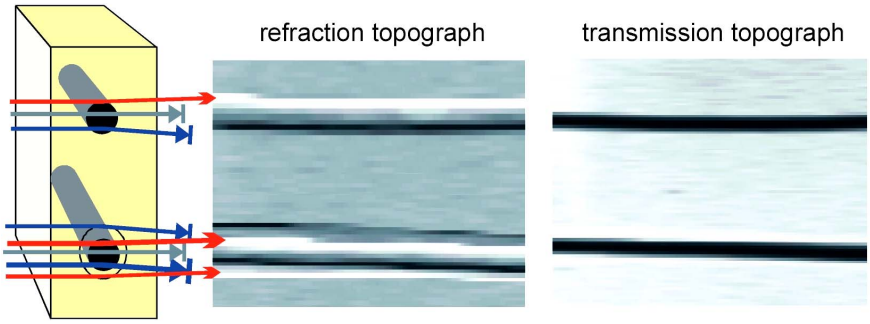

Fig. 2. X-ray scanning topography; left: model composite of polymer matrix with embedded bonded (top) and de-bonded (bottom) 140 µm sapphire fibre; middle: X-ray refraction topograph of C-values resolving debonding spatially after (1), free from absorption effects; right: scan of transmission intensity due to absorption

3. Crack investigations

In order to study the scattering of oriented cracks as a reference for more realistic micro cracks of engineering materials a densely packed stack of polymer foils is investigated. Two mm wide stripes are stacked with 120 µm periodicity (Fig. 3, top left) and scanned through the primary beam of the Kratky camera in 25 µm steps while the refraction detector registers the scattering intensity. This procedure is repeated after inclination of the sample by 0.1 degree steps within an angular range of + 5 deg. about the symmetry direction. The resulting rotation topograph shows the angular intensity distribution at the linear scan positions with a gap in the center (Fig.3, top right).
Considering that the scattering origins from X-ray total reflection and refraction at the foil surfaces (Fig.3, bottom, left) the gap is obiously due to the fact that the projections of the slits have their smallest width at zero inclination angle. At rising inclination angles the two symmetric intensity peaks decay due to continuous reduction of the scattering angle according to Snell's law below the limiting angle of the detector slit.

As the scattering intensity is mainly determined by the width of the slit projection under inclination it is independent of the slit width if this is smaller than the projection. An estimation of the (linear) total scattering cross section of the two mm long cracks gives Q = 8 µm. This explains the high scattering intensities. A comparison of this reference intensity with unknown crack surfaces permits the quantitative measurement of crack surfaces down to a few nano meters as the applied wavelength is still 100 times smaller.
In order to investigate the crack behaviour of a PE-PP-polymer blend a specimen is scanned after surface impact, which is a standard treatment for testing material toughness. The crack micro structure of such a damage is generally unknown and hardly detectable by other techniques, especially the crack orientation distribution. Fig. 4 indicates the sample scanning area inside of 1 cm x 2 cm. The transmission scanning radiograph reveals no density variation of the impact area. The refraction topographs A and B represent the inner surface density at orthogonal orientations. The top row of Fig 4. shows the deduced crack orientations. It is interesting to note that the crack directions are not symmetric about the (normal) impact direction. This becomes more evident by the intensity plots under inclination starting at orientation A and B. One plot shows isotropic crack distribution the other one a crack orientation within + 20 deg. about the surface normal. The investigation reveals a complete characterization of the three dimensional spatial and angular crack density distribution.

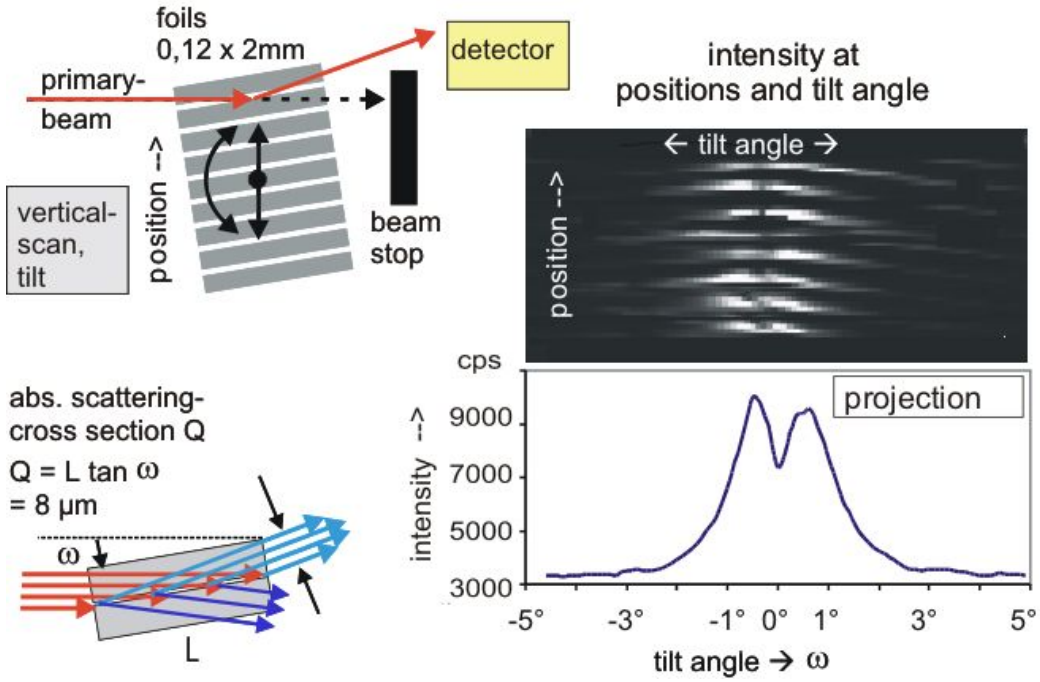

Fig.3. X-ray refraction of densely packed stack of 120 µm polymer foils reveals interfacial reference cracks. Multiple linear scans under inclination steps of 0.1 deg. reveal rotation topograph (top, right) and its projection. The scattering cross section is identical to the relative projection width in the primary beam.

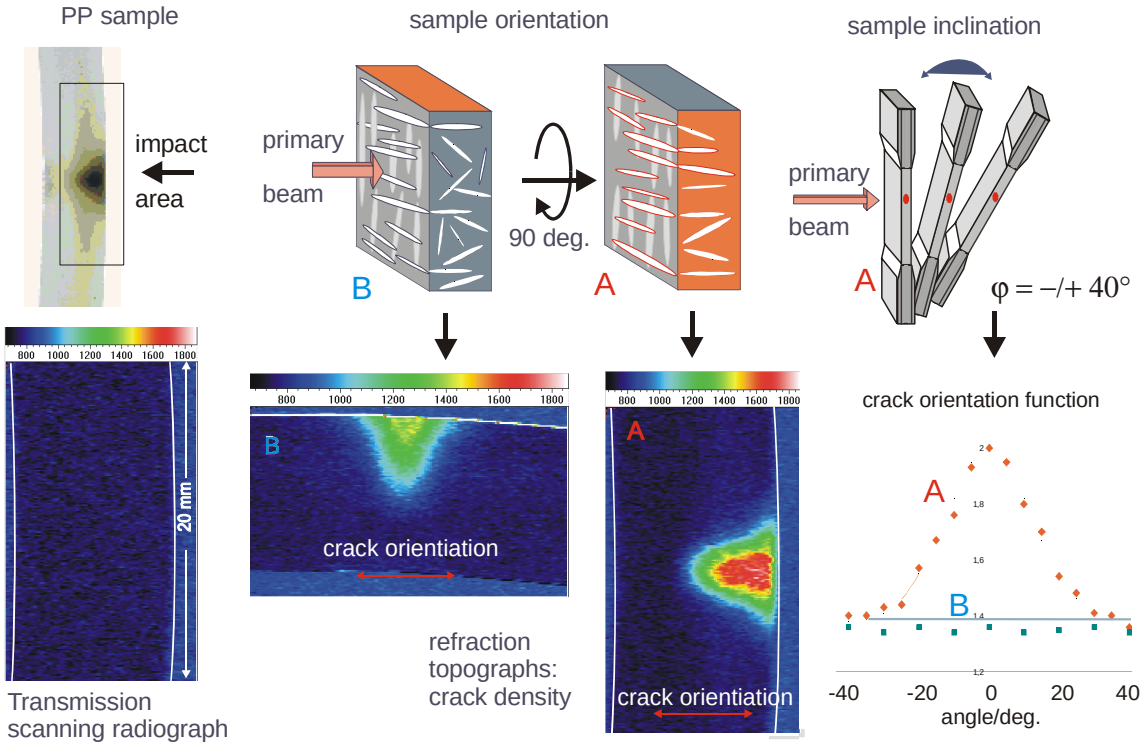

Fig. 4. Crack behaviour of a PE-PP-polymer blend after surface impact; micrograph of sample with indicated scan area; Sample and deduced crack orientations, transmission (scanning) radiograph without damage indication, different (normal incidence) refraction topographs at orientations A and B, isotropic and non-isotropic crack orientation distribution under different inclination directions.

4. X-Ray Refraction Computed Tomography

Refraction-Topography images the projection of the specific surface, but a transversal section image may be desired in order to reveal details. This is achieved by two-dimensional scanning computed tomography. A section of three stacked C/C ceramic matrix (CMC) composite is investigated in order to image the different crack patterns developing during pyrolysis at three different levels of fiber debonding.

For a classical (absorption) density map by Computed Tomography (CT) multiple linear scans of the sample are repeated at different rotation angles at zero scattering angle. The absorption signals are reconstructed by parallel beam filtered back projection as shown by Fig. 5, center. The resulting density tomograph reveals only major cracks.

The reconstruction of the refractive scattering intensity, reveals an interface image (Fig. 5, right) of much finer cracks. The average intensity levels reveal the quantitative crack density [4].

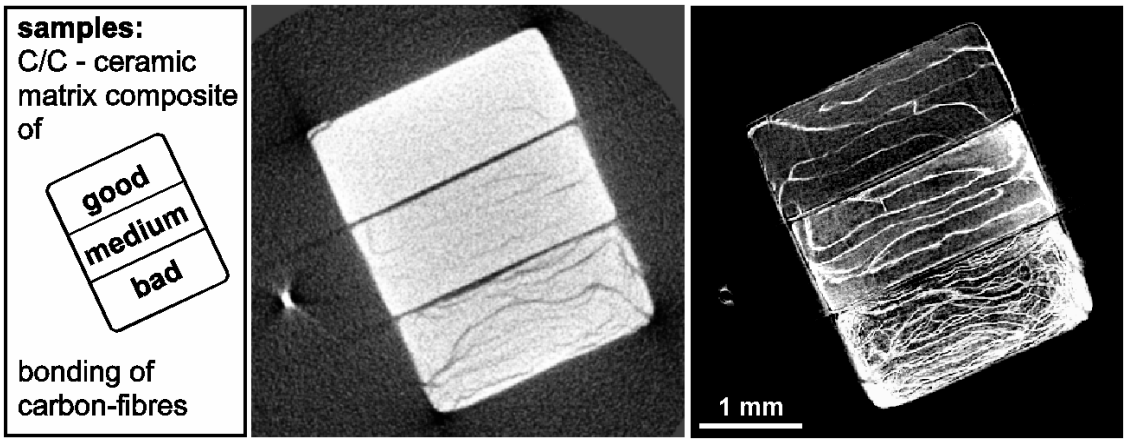

Fig. 5. X-ray Computed Tomography (CT) of Carbon/Carbon ceramic matrix composite (C/C-CMC); left: sample arrangement, middle: conventional (X-ray absorption) CT, right: cracks by Refraction Computed Tomography.

5. Further potential

The methods of X-Ray Topography request preferably the application of ‘soft’ X-rays below 20 keV. As their absorption in metals is high the restriction to light weight materials is recommend in most cases. The most frequent requests for topographic investigations come from industrial developments of polymer processing, plastic foams, composites, ceramics and special papers. Harder radiation of higher brilliance at synchrotron radiation facilities provides faster topographic measurements and higher resolution but more effort as well.

References

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