Visualization of delamination in composite materials utilizing advanced X-ray imaging techniques

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ABSTRACT: This work is focused on the development of instrumental radiographic methods for detection of delaminations in layered carbon fibre reinforced plastic composites used in the aerospace industry. The main limitation of current visualisation techniques is a very limited possibility to image so-called closed delaminations in which delaminated layers are in contact practically with no physical gap. In this contribution we report the development of innovative methods for closed delamination detection using an X-ray phase contrast technique for which the distance between delamination surfaces is not relevant. The approach is based on the energetic sensitivity of phase-enhanced radiography. Based on the applied methodology, we can distinguish both closed and open delamination. Further we have demonstrated the possibility to visualise open delaminations characterised by a physical gap between delaminated layers. This delamination type was successfully identified and visualized utilizing a high resolution and computed tomography table-top technique based on proper beam-hardening effect correction.

KEYWORDS: Inspection with x-rays; Detection of defects

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1 Introduction

The Carbon Fibre Reinforced Plastic (CFRP) layered composite material is widely used in high performance aerospace structures thanks to its excellent mechanical properties and relatively low weight. Utilization of CFRP is however limited by the ability to reliably detect possible defects occurring during manufacturing process and during aircraft operation. One of such defect is disconnection of CFRP layers, so called delamination.

The difficulty in the visualization of delaminations is given by the fact that changes between the proper and damaged sample are often below the spatial resolution and the sensitivity of available conventional imaging methods. When the layers of delamination are in contact (so-called closed delamination), projection X-ray radiography unfortunately often does not contribute at all to the image formation by attenuation. In the case of open delamination (a physical gap exists between the layers), visualization of these defect can be carried out using X-ray computed tomography (CT). Nevertheless, this technique has limited applicability for practical non-destructive testing of larger components. A technique that would enable reliable detection of delaminations ideally just using a single projection with a reasonable large field-of-view is therefore desired.

In this contribution we report the application of innovative methods for closed delamination detection using an X-ray phase contrast technique for which the distance between delamination surfaces is not relevant. The approach is based on a table-top setup enabling phase-enhanced radiography utilizing a micro-focus X-ray tube together with photon counting energy sensitive hybrid semiconductor pixel detector and a relatively large sample-to-detector distance.
2 Proposed approach

A general issue in phase-enhanced radiography is the major complexity of measured images formed simultaneously by attenuation and phase effects. Consequently, using a single radiography without application of further optical elements such as gratings, the separation of the absorption and phase information is essentially ambiguous. This problem can be solved using the energy sensitivity of the single photon counting pixel detector Timepix [1]. It is well known that absorption depends on energy as $1/E^4$ while refraction as $1/E^2$. Even though absorption contrast is apparently strongly energy sensitive\(^1\) the contribution of both effects can be resolved performing two measurements with two different energy thresholds. In this approach, the signal measured for each energy threshold is converted into its equivalent thicknesses, as described in detail in ref. [2]. As investigated composite materials do not change its composition during measurement both transformed images should be identical considering just absorption effects. After subtraction of both images, consequently, differences between images indicate the presence of phase contrast effect caused by delaminations (and other material interfaces as specimen boundaries for instance). Using this proposed approach, we demonstrate that we can distinguish both closed and open delamination. The reliability of the proposed method was confirmed using a single grating phase contrast technique [3].

2.1 Visualization of open delaminations

In some cases, so-called open delamination is presented in CFRP components due to its blocking by the permanent deformation. This delamination type can be identified and visualized utilizing high resolution computed tomography techniques. The experimental challenge of X-ray tomography for this task is, however, that the volume of the open delamination is still relatively small and the local density of the CFRP varies in the range of several hundreds of percent. The desired tomography contrast and spatial resolution have to be therefore very high. In our case, these issues are solved by application of a high-resolution table-top setup based on a micro-focus X-ray tube and an appropriate beam-hardening calibration. As a result, an open delamination shape was successfully identified and visualized even though closed delaminations are not visible in the tomographic reconstruction.

3 Investigated CFRP sample

The angle bar CFRP for which delaminations were inspected was 3 mm thick and 25 mm wide with fatigue disbonding created by bending loading (specimen was prepared by EADS Deutschland GmbH). Delaminations are present in the specimen radius. Photography of the sample is depicted in figure 1.

4 Phase contrast imaging

Variations of the refractive index across the sample result in wave effects affecting the phase of the X-ray beam traversing the sample [4]. Consequently, X-rays refract on the object interfaces

\(^1\)In cases where the object is more or less homogenous in terms of elemental composition and only a defects need to be visualised within the sample.
Figure 1. The specimen QUI_EADS_17 used for identification of delaminations. It is 3 mm thick and 25 mm wide CFRP angle bar specimen with fatigue crack created by bending loading.

Figure 2. X-ray imaging of a wasp placed behind a 3 mm thick CRFP composite sample. a) Photography of the object; b) transmission radiograph (cropped); c) detail of wasp body boundary (pixel scale); d) signal in the column crossing boundary (blue line in the detail), where the phase contrast effect is clearly visible. Phase shift peaks have 25 µm pitch.

Similarly as optical light traveling through media with gradients of the index of refraction in the direction perpendicular to the beam path. Induced phase gradients produce changes in the direction of the beam [5] superposed on the projected image plane which can be detected.

Phase contrast imaging is a powerful imaging tool especially in cases when subtle objects exhibiting very small absorption contrast are to be visualized. The demonstration of the system capability to provide a sufficiently strong phase-contrast signal is shown on an X-ray radiograph of a wasp, which is well visible even through the 3 mm thick CFRP specimen (see figure 2). Note that even thin features such as the wasp wing (several µm thick) are recognizable. The image was taken with a large area pixel detector WidePix/Timepix [6], resulting 5 µm image pixel size, 1100 mm object to detector distance, X-ray tube voltage 90 kV, current 110 µA and 180 sec integration time.

The large contrast achieved is entirely due to phase shift effect across the subtle structure of the wasp body. It is remarkable that the phase contrast effect is clearly visible even for the relatively short distance between the object and the detector thanks to the high spatial resolution of the Widepix/Timepix detector used. For instance, observed peaks corresponding to the phase shift have pitch 25 µm only, see plot d) in figure 2.

As the delaminated interface between the CFRP layers differs significantly in the index of refraction (the air/vacuum — CFRP interface is present), this interface causes deflection or even
reflection in some cases of the X-ray beam. The deflected beam consequently causes a clear light and dark line pair in the radiogram.

### 4.1 Enhancement of the phase contrast effects in the image employing convolution

The measurement was carried out on the specimen shown in figure 1. A micro focus X-ray tube with 5 µm spot was operated at 70 kV and 140 µA. The single photon counting detector Timepix with 1 mm thick silicon sensor was utilized as imager. The focus to detector distance (FDD) was 700 mm. The curved part of the specimen was imaged with magnification 4.5×. The focus to object distance (FOD) was 155 mm achieving an image resolution of 12 µm.

The enhancement of the phase contrast effects employing the convolution of the analyzed radiogram with an appropriate kernel (the band with the maximum and minimum intensities) [7] is displayed in figure 3: the phase contrast effect is nicely revealed in the position of the delamination — labelled by the arrows. The red arrows indicate the closed delamination. The yellow arrows indicate the open delamination. The open delamination is expressed by a doubled curve, i.e. the phase contrast effect occurred on both surfaces of the delamination. It was estimated measuring doubled curve pitch, that distance between faces of the open delamination is ∼85 µm. It can be also estimated that distance between faces of the revealed closed delaminations is negligible, comparing phase shift peaks in figure 2 d) and phase shift peaks derivated from figure 3 d). Note, that accuracy of this method strongly depends on the appropriate choice of the kernel used. Nevertheless the signal from the phase contrast effect induced by the delamination may be similar to the signal caused by the inner layered structure; therefore this measurement has to be validated by another method.

### 4.2 Identification of the phase contrast effect utilizing two energy thresholds

The identification of the phase contrast effect was also achieved utilizing two energy thresholds with the same experimental setup as above. The two subsequent acquisitions were performed with different energy thresholds 5 and 16 keV set by the Timepix detector.

The two specimen radiograms were converted into equivalent thickness using the appropriate calibration (the same beam hardening calibration method [2] as in the section above). If only absorption is present then both converted images should be identical. The difference between

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**Figure 3.** Enhancement of the phase contrast effects in the image employing convolution. a) Overall radiogram of the curved specimen; b) detail of the specimen where phase contrast effect is present; c) convolution kernel; d) enhanced phase contrast effect; e) detail of delamination.
Figure 4. Distinguishing the phase and absorption images using two energy thresholds. a) Overall radiogram; b) image acquired with energy threshold 5 kV; c) with energy threshold 16 kV; d) differential image enhancing phase contrast effect.

Figure 5. Experimental layout of the single grating method applied for the investigation of composite materials.

them indicates the presence of some other principle being involved such as material differences or phase effects. Since there is no reason for changes in the material properties during the X-ray measurement the only explanation is the phase effect. The images illustrating phase contrast identification are shown in figure 4. The phase contrast effect is nicely revealed in the position of the delamination — labeled by the arrows. The red arrows indicate the closed delamination and the yellow arrows indicate the open delamination. It is clearly visible, that delaminations were found in the same places as above. It was proven from this reason, that a quite simple convolution with appropriate kernel gives appropriate results. The method utilizing two energy thresholds gives more noise which is due to lower statistics resulting from the image differentiation.

4.3 Single shot measurement of absorption, phase and dark-field images

The single grating phase contrast method [3] pioneered by the SYRMEP group of the synchrotron Elettra in Trieste [8] was utilized as another method for identification of delaminations. This method is much more demanding in comparison with previously introduced techniques. The experimental arrangement of the single grating phase contrast method is shown in figure 5. The grating is aligned with the pixels of the detector so that each micro-beam hits the border between two detector columns. The period of projected pattern is exactly three pixels. Subsequently, in each period two pixels share the signal of the single micro-beam while a third pixel is completely shielded. When the sample is inserted, the micro-beams can be bent which leads to a change of the
Figure 6. Investigation of the composite material sample by means of three-modal X-ray micro-radiography. The attenuation image (left); the phase-gradient image (middle); and the dark-field image (right) representing the small angle scattering.

signal ratio between two irradiated pixels indicating the phase effect. The total signal registered by these pixels corresponds to absorption. The signal recorded by the third fully shielded pixel (so called dark field) corresponds to small angle scattering [9].

The technique was tested with a micro-focus X-ray tube at 70 keV and 20 µA; exposure time was 200 s; physical field of view 14 × 14 mm² and spatial resolution 15 µm (given by the geometrical magnification used). The result of this measurement with the same specimen as above is shown in figure 6.

The single grating method provides the attenuation image, the phase-gradient image and the so-called dark-field image showing locally the small angle scattering of the sample; see figure 6. One can see that delamination is best visible in the dark-field image. The detected delaminations are marked by arrows similarly as above. The positions of the delamination are slightly different than in the images in figure 3 and figure 4 as the specimen was irradiated under different angle; however the detected delamination shape is the same. Note that delamination is visible in the direction tangential or nearly tangential to the delamination surface, as mentioned above.

5 Open delamination detection utilizing computed tomography reconstruction

The specimen was tomographycally inspected with the following parameters: microfocus tube at 60 kV and 164 µA; 800 projections and 2 sec integration time. Perkin Elmer flat panel detector with pixel size 200 µm and active area 400 × 400 mm was used as imager. The CT reconstruction with 24 µm voxel size was done utilizing the Volex 6 software (developed in Fraunhofer Development Center for X-ray Technology EZRT, Germany).

It was found, if only flat field correction is applied see figure 7 left, only a relatively low contrast (30%) is achieved between the delamination and the surrounding material. This contrast can be improved twofold (60%) utilizing a beam hardening correction method [2] applied on all CT radiograms, see figure 8 right. The density profiles in a reconstructed vertical slice labelled by the red dashed line are plotted on top of the related reconstructed slices. One can see that the peaks corresponding to the open delaminations are clearly visible and these are better emphasized if the beam hardening correction is applied. It was estimated that distance between faces of the open delamination is ∼ 85 µm evaluating signal profile in figure 7. It is the same value as it was
Figure 7. Open delamination detected by the computed tomography. Using data just flat-field corrected (left), contrast is relatively low due to significant CFRP density variations. The contrast improves twofold when the beam hardening effect is corrected (right).

Figure 8. Tomographically reconstructed specimen. The whole reconstructed volume (left). Detail of this volume where open delamination is clearly visible (right).

proven utilizing phase contrast effect measurement depicted in figure 3. Distance between faces of the closed delamination has to be much smaller than tomographic resolution as this delamination type is not visible in the CT reconstruction.

The reconstructed volume is depicted in figure 8 left for which the related projections were beam hardening corrected [2]. It was proven that the open delamination (i.e. delamination surfaces are not in contact) can be resolved in this reconstruction, see figure 8 right. Note that the inner CFRP structure has two orders high relatively density variations, therefore the identification of the delamination is quite demanding task.

It was proven that the presence of open delamination and its shape can be identified utilizing computed tomography, however the closed delamination detectable by the phase contrast techniques is not visible in the tomographic reconstruction due to its practically zero volume.

6 Conclusions

Delaminations in CFRP composite materials were successfully identified utilizing several X-ray techniques. As demonstrated, the phase contrast effect can help identifying the delamination even in the situation when the tomography fails (especially when delamination surfaces are in contact). The phase image can be simply resolved from the attenuation image performing two measurements with two different energy thresholds. These results were confirmed using the single grating phase
contrast technique. In the case of open delamination, we have demonstrated that these features are detectable in the tomographic reconstruction. The key procedure providing a sufficient image contrast in the reconstruction is a proper signal to equivalent thickness calibration technique applied on all tomographic projections. We have demonstrated that using this technique, the contrast between open delamination and surrounding CFRP material was twofold improved compared to the reconstruction based just on data after flat-field correction.

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References

[1] X. Llopart, R. Ballabriga, M. Campbell, L. Tlustos and W. Wong, Timepix, A 65k programmable pixel readout chip for arrival time, energy and/or photon counting measurements, Nucl. Instrum. Meth. A 581 (2007) 485 [Erratum ibid. A 585 (2008) 106].
[2] J. Jakubek, Data processing and image reconstruction methods for pixel detectors, Nucl. Instrum. Meth. A 576 (2007) 223.
[3] F. Krejci, J. Jakubek and M. Kroupa, Single grating method for low dose 1-D and 2-D phase contrast X-ray imaging, 2011 JINST 6 C01073.
[4] T.J. Davis, D. Gao, T.E. Gureyev, A.W. Stevenson and S.W. Wilkins, Phase-contrast imaging of weakly absorbing materials using hard X-rays, Nature 373 (1995) 595.
[5] S.W. Wilkins, T.E. Gureyev, D. Gao, A. Pogany and A.W. Stevenson, Phase-contrast imaging using polychromatic hard X-rays, Nature 384 (1996) 335.
[6] J. Jakubek et al., Large area pixel detector WIDEPIX with full area sensitivity composed of 100 Timepix assemblies with edgeless sensors, 2014 JINST 9 C04018.
[7] I. Jandejsek et al., X-ray inspection of composite materials for aircraft structures using detectors of Medipix type, 2014 JINST 9 C05062.
[8] A. Olivo et al., An innovative digital imaging set-up allowing a low-dose approach to phase contrast applications in the medical field, Med. Phys. 28 (2001) 1610.
[9] A. Olivo et al., Preliminary study on extremely small angle X-ray scatter imaging with synchrotron radiation, Phys. Med. Biol. 47 (2002) 469.