Optimizing and applying high-resolution, in-line laboratory phase-contrast X-ray imaging for low-density material samples

Robert Zboray

Center for X-ray Analytics, Department Materials Meet Life, Swiss Federal Laboratories for Material Science and Technology, Empa, Überlandstrasse 129, 8600 Dübendorf, Switzerland

Correspondence
Robert Zboray, Center for X-ray Analytics, Department Materials Meet Life, Swiss Federal Laboratories for Material Science and Technology, Empa, Überlandstrasse 129, 8600 Dübendorf, Switzerland
Email: robert.zboray@empa.ch

Present address
Center for X-ray Analytics, Department Materials Meet Life, Swiss Federal Laboratories for Material Science and Technology, Empa, Überlandstrasse 129, 8600 Dübendorf, Switzerland

Funding information

In-line, or propagation-based phase-contrast X-ray imaging (PBI) is an attractive alternative to the attenuation-based modality for low-density, soft samples showing low attenuation contrast. With the growing availability of micro- and nano-focus X-ray tubes, the method is increasingly applied in the laboratory. Here, we discuss the technique and demonstrate its advantages for selected low-density, low attenuation material samples using a lab-based micro- and nano-computed tomography systems Easytom XL Ultra, providing micron and sub-micron range resolution PBI images. We demonstrate a multi-step optimization of the lab-based PBI technique on our scanner that includes choosing the optimal detector-source hardware combination available in the setup, then optimizing the imaging geometry and finally the phase retrieval process through a parametric study. We point out and elaborate on the effect of noise correlation and texturing due to phase retrieval. We demonstrate the overall benefits of using the phase image and the phase-retrieval for the selected samples such as improved image quality, increased contrast-to-noise ratio while only marginally influencing the spatial resolution. The improvement in image quality also enables further image processing steps for detailed structural analysis of the samples, which would
Introduction

X-ray micro and nano computed tomography (CT) is a powerful tool to examine the 3D morphology and internal structure of materials. A good overview of recent trends in the application of X-ray micro-CT is given in Stock (2008) and in Maire and Withers (2014). Studying the internal structure of low-density, soft materials can however be challenging using the conventional attenuation-based X-ray imaging modality. Instead, the use of phase-contrast or phase-sensitive X-ray imaging techniques can be especially beneficial in material science for characterizing such materials including polymers, fibres, scaffolds, foams, cellulose- and wood-based materials just to mention a few examples of industrial interest. By means of phase-sensitive techniques it is possible to generate contrast in relation to the phase shifts imparted by the sample and to extend the capabilities of X-ray imaging to those details that lack sufficient visibility in conventional attenuation X-ray imaging. The refraction of the X-ray wavefronts and the corresponding phase shift imparted by the sample is governed by the decrement of the real part, $\delta$, of its complex index of refraction $n = 1 - \delta + i\beta$.

A review and comparison of X-ray phase-contrast imaging techniques is given in (Wilkins et al., 1996; Diemoz et al., 2012; Mayo et al., 2012; Endrizzi, 2018; Salditt et al., 2017)

In-line, or propagation-based, X-ray phase-contrast imaging (PBI) is especially promising for high-resolution micro- and nano-CT investigations of low-density and low-attenuation samples. It is finding more and more widespread applications due to the increased availability of CT scanners with micro- and nano-focus laboratory X-ray sources (Bidola et al., 2017; Zabler et al., 2020). This makes the technique much more accessible to researchers outside of the synchrotron realm. The wavefronts of coherent, or in case of laboratory sources, partially coherent X-rays are distorted by inner interfaces or structures of the sample and propagating a certain distance they interfere and form inline phase-contrast patterns. If the propagation distance between sample and detector is sufficient, this effect appears as edge enhancement (Wilkins et al., 1996; Pogany et al., 1997; Salditt et al., 2017). Edge enhancement effects are practically unavoidable, using tubes with focal spots below 5 $\mu$m full-width at half maximum (FWHM) and operating at high magnifications (Mayo et al., 2012), which is the typical case for high-resolution, cone-beam X-ray micro- and nano-CT in a laboratory setup.

Several comprehensive overview papers cover the PBI technique explaining its basics and putting it into context with respect to other phase-contrast imaging modalities that are feasible using laboratory X-ray sources (Wilkins et al., 2014; Diemoz et al., 2012; Endrizzi, 2018). Without repeating all those details here, we just highlight the main benefits and some potential difficulties of the PBI method. Advantages include, in the first place, the technical simplicity of PBI compared to e.g. grating-based methods. It does not require any beam shaping device. The PBI method tolerates and works for non-monochromatic X-ray spectra such as of micro-focus X-ray tubes (Myers et al., 2007). Simple phase retrieval methods exist for PBI (Paganin et al., 2002) and the retrieved phase signal can increase contrast-to-noise (CNR) and signal-to-noise ratios (SNR) up to two orders of magnitude compared to the attenuation signal (Beltran et al., 2011; Kitchen et al., 2017). This is especially important for low-intensity laboratory micro-focus setups, where otherwise unfeasible long exposure times might be needed to improve image quality. Some disadvantages of PBI on laboratory sources include the inherently low intensity of micro- and nano-focus tubes and the correspondingly long
exposure times. This can be aggravated by using relatively small pixel-sized detectors to resolve the edge enhancement, diffraction patterns and thus the phase effect better. High-resolution, small pixel detectors are typically coupled with relatively thin and therefore low-efficiency scintillator layers.

The present paper discusses how lab-based PBI and the phase retrieval can be optimised for low-density, low-attenuation materials to improve or enable the study and characterization of their internal structures. The benefits of the technique are demonstrated by using it for three illustrative sample materials. We examine how the physical setup, the imaging geometry and the phase retrieval parameters should be chosen to optimize the imaging results in terms of the visibility of morphological features and of CNR in order to facilitate further image analysis steps such as segmentation, feature extraction etc. Here we use the retrieval method by Paganin et al. (2002) underlying a single-material constraint. We compare its performance to the Bronnikov-Aided-Correction (BAC) algorithm (De Witte et al., 2009), which is based on the modified Bronnikov algorithm by Boone et al. (2009) and has been shown to produce good results for soft samples especially in case of using partially-coherent, laboratory sources. We compare the two retrieval algorithms and show their benefits and drawbacks with respect to one another. We also examine and discuss the potentially adverse effect of noise correlation and texturing on image quality for strongly filtering phase retrieval.

Methods

PBI in the present study has been carried out using an EasyTom XL Ultra 230-160 micro- and nano-CT scanner (RxSolutions SAS, Chavanod, France). The scanner features a Hamamatsu reflection target micro-focus and a Hamamatsu L10711 nano-focus, transmission X-ray tube with an 1 \(\mu m\)-thick tungsten target on a diamond window. The latter tube, used in this study, was equipped with a LaB\(_6\) cathode. The tube can be operated at small-, mid- and large-focus mode up to 100 kVp. Depending on the actual kVp used, FWHM of the emission spot size varies between 0.6-0.8 \(\mu m\) for small-, 1.5-2.2 \(\mu m\) for mid- and is around 3 \(\mu m\) for large-focus settings as was confirmed by measurements using JIMA and Siemens-star patterns. The lower limits of the previous figures are typically achieved for high voltages of \(\geq 65\) kVp due to the tube characteristics. To enhance the edge (phase) effect and to suppress the high-energy part of the broad X-ray spectrum a high-resolution, 11 Mpix, 14 bit CCD-based detector (xiRay from XIMEA) was used for the study. It features a 20 \(\mu m\)-thick GadOx (P43) scintillator, fiber-optically coupled directly on the CCD chip. The camera has 9 \(\mu m\) native pixel size, however as a reasonable compromise between detection efficiency and resolution for the given thickness of the converter, it is used in 2x2 binning mode. At 2x2 binning and \(px=18 \mu m\) pixel size, the image size is 2016x1344 pixels. The conversion efficiency of the detector drops below 10% at around 35 keV, essentially filtering out any contribution of photons above that energy. Another detector option available in the system is a flat-panel (FP) detector with a thick, high-efficiency CsI converter, however it has a much larger (127 \(\mu m\)) pixel size and does not feature the aforementioned spectral suppression effect. These properties of the different system components motivate to use it at relatively high kVps (to approach the smallest possible tube emission spot for the given focal mode) in combination with the CCD (spectral filtering) to get high-quality PBI. Using unnecessarily high kVp, even though high-energy photons do not contribute to the detector signal, was avoided in the study. It does not increase the tube output in the useful, low-energy part of the spectrum for such thin-target transmission tubes, just the thermal load on the target with potential adverse effects on the imaging. Preliminary investigations on simple, single-material test objects, like a small-diameter Polymethylmethacrylat (PMMA) tube, have proven that such an optimal combination of the available hardware (both detector and source side) as explained above can bring more than factor two improvement in phase sensitivity compared to other choices of source and detector. In these investigation, which are not presented here for brevity, we followed an approach similar to e.g. (Balles et al., 2016). Note that our scanner has been developed and commercialised primarily aiming at attenuation micro- and nano-CT investigations.
Nonetheless, as the present paper proves, with the appropriate hardware options it can be broadly deployed for PBI.

The scan parameters for the different samples are summarized in Table 1. For the CT scans, the samples did not require a specific preparation. They were securely fixed on appropriate sample holder for each scan. The scans were completed by taking 1568 projections over 360° of rotation. The tomographic reconstruction of the images (both transmission and phase) has been carried out by standard filtered back-projection algorithm for 3D cone-beam geometry, FDK (Feldkamp et al., 1984).

### Material samples

Three different low-density, low-attenuation material samples are used in our study. The first one is an experimental pure cellulose porous foam with low density (ca. 18-20 mg/cm³) and high porosity (=99%). Typically it is used as adsorbent or filter in aqueous condition because of its high porosity. The available sample size for this sample was relatively large (ca. 1 cm across) and due to its very soft and fragile structure it could not be trimmed to a smaller size without destroying its internal structure. This limited the maximum magnification when capturing the full sample cross section and thus explains the relatively large voxel size for this experiment in Table 1. For a better resolution, local tomography on a region of interest (ROI) could be done for such a sample. The second sample is a carbon-fibre reinforced polymer-matrix (CFRP) composite made of (fibre type IM7, nominal diameter about 5.2 µm) and a toughened epoxy (matrix polymer type 977-3). The density of the CFRP sample was 1.79 g/cm³. The third one is a biodegradable, porous polymer foam (PF) scaffold, foamed up using supercritical CO₂ an utilised in the biomedical field. The density of such foams is typically around 40 mg/cm³. Note that detailed and comprehensive investigations of the two foam materials will be presented elsewhere in the near future. Here they are used just as illustrative examples for our study and are not meant for quantitative evaluation of these materials.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Voltage [kV]</th>
<th>Target current [µA]</th>
<th>Tube focus mode</th>
<th>Exposure time per projection [s]</th>
<th>Effective voxel size [µm]</th>
<th>z_{eff} [mm]</th>
<th>M [-]</th>
<th>NFr [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>cellulose foam</td>
<td>65</td>
<td>30.4</td>
<td>mid</td>
<td>5</td>
<td>4.2</td>
<td>54.4</td>
<td>4.3</td>
<td>23.2</td>
</tr>
<tr>
<td>CFRP</td>
<td>80</td>
<td>15.7</td>
<td>small</td>
<td>18</td>
<td>0.3</td>
<td>5.0</td>
<td>59.3</td>
<td>1.8</td>
</tr>
<tr>
<td>PF</td>
<td>60</td>
<td>30.1</td>
<td>mid</td>
<td>5</td>
<td>1.3</td>
<td>20.3</td>
<td>13.8</td>
<td>4.6</td>
</tr>
</tbody>
</table>

**Table 1** Experimental parameters for the different samples scanned in this study. z_{eff} is the effective propagation distance for divergent, cone-beam geometry, M is the geometrical magnification and NFr is the Fresnel number (see Eqs. 1 and 2).

### PBI and Phase retrieval methods

Next, we briefly review some basics of PBI and of the phase-retrieval methods used here. Denoting the source-to-sample distance by z₁ and the sample-to-detector distance by z₂, where z is the optical axis of the setup, the geometric...
magnification of the setup is given by:

$$M = \frac{z_1 + z_2}{z_1}$$  \hspace{1cm} (1)

Furthermore we define the Fresnel number ($N_{Fr}$) as:

$$N_{Fr} = \frac{p x^2_{\text{eff}}}{z_{\text{eff}} \lambda}$$  \hspace{1cm} (2)

where $p x_{\text{eff}} = p x / M$ is the effective pixel size, $z_{\text{eff}} = z_2 / M$ is the effective propagation distance for divergent, cone-beam geometry and $\lambda$ is approximated by an effective (mean) wavelength for polychromatic sources. In PBI typically three regimes are distinguished (Salditt et al., 2017) based mainly on the propagation distance and by the corresponding value of the Fresnel number. The first one is the contact regime with the detector placed directly behind the object ($z_2 \approx 0$). This is the regime for which phase effects have not yet been transformed into measurable intensities. The second is the direct contrast or edge enhancement regime, featuring larger propagation distances and $N_{Fr} \geq 1$. In this regime the contrast is governed by the Laplacian of the phase shift (Pogany et al., 1997; Diemoz et al., 2012; Salditt et al., 2017) and this is the regime used in our study (see Eq. 5 below). The third regime is for very large propagation distances and $N_{Fr} \approx 1$, where one obtains a Fraunhofer (far-field) diffraction pattern, which loses resemblance to the object.

The relationship between the measured intensity data and the object attenuation and phase shift properties in PBI is non-linear. In order to obtain analytically tractable reconstruction algorithms for phase-contrast imaging, the model is typically linearised in the frame of a so-called weak-object approximation, assuming low attenuation and small phase shift by the object, i.e. the object’s optical properties are linearised (Guigay, 1977; Pogany et al., 1997; Gureyev et al., 2004; Salditt et al., 2017). The object optical properties are described by the object transmission function, $q$, as (Guigay, 1977; Pogany et al., 1997; Gureyev et al., 2004; Salditt et al., 2017):

$$q(r) = \exp(-\mu(r) - i\phi(r))$$  \hspace{1cm} (3)

where $\mu(r)$ and $\phi(r)$ describe the attenuation and phase shift by the object and are the integrals along $z$ of the imaginary and real parts of the objects complex refraction index ($n = 1 - \delta + i\beta$) in the projection approximation. Under weak-object conditions the contrast transfer function (CTF) of x-ray PBI in the frequency domain can be derived as (Guigay, 1977; Pogany et al., 1997; Gureyev et al., 2004; Salditt et al., 2017):

$$I(k, z_{\text{eff}}) = 2\pi \delta_D(k) + 2\sin(\chi) \tilde{\phi}(k) - 2\cos(\chi) \tilde{\mu}(k)$$  \hspace{1cm} (4)

where $\delta_D(k)$ is the Dirac-delta function, $k(k_x, k_y)$ is the wave vector in the detection plane perpendicular to the optical axis and in terms of the spatial frequencies ($\nu_x, \nu_y$) = ($k_x, k_y$)/2$\pi$ with $\chi(v, z) = \pi \lambda z_{\text{eff}} v^2$, where $v^2 = \nu_x^2 + \nu_y^2$. 


and $\chi$ is the reduced spatial frequency. $\bar{\mu}(k)$ and $\bar{\phi}(k)$ are the Fourier transforms of $\mu(r)$ and $\phi(r)$.

The intensity in Eq.4 is thus given by amplitude and phase of the object transmission function, however altered by the oscillating sine and cosine terms, meaning that different frequencies are transmitted with very different strength at different propagation distances. A simpler formulation for the normalized intensity in real space can be derived based on the so-called transport-of-intensity (TIE) equation, which rather than linearising the object’s optical properties is based on linearisation with respect to the propagation distance (Teague, 1983; Salditt et al., 2017; Endrizzi, 2018):

$$\frac{I(r, z_{\text{eff}})}{I(r, 0)} = 1 - \frac{z}{k} \bar{\chi} \bar{\phi}(k)$$  \hspace{1cm} (5)

Note that Eq.5 for a pure phase object ($\mu(r)=0$) and approximating the sine term for low frequencies ($\chi \ll \pi/2$) can be derived from Eq.4. The TIE-based form approximates the phase CTF in Eq.4 very well in the low-frequency region i.e. for moderate propagation distances (Salditt et al., 2017), and gives thus satisfactory results in the direct contrast regime for most practical cases. Phase retrieval methods based on the TIE are therefore used in our study.

Note that in the above and following equations, though not explicitly indexed for brevity, all real and Fourier-space coordinates $r$ and $k$ are in the detection plane perpendicular to the optical axis.

We consider two single-distance phase retrieval methods. The simplest form of TIE-based phase retrieval is obtained by inversion of Eq.5 via Fourier transform ($F^{-1}$) as

$$\bar{\phi}(r) = -\frac{k}{z} F^{-1} \left[ \frac{F[I(r, z_{\text{eff}})/I(r, 0) - 1]}{k^2} \right]$$  \hspace{1cm} (6)

Eq.6 is the phase retrieval step of the original Bronnikov algorithm (Gureyev and Nugent, 1997; Bronnikov, 1999). In the original work of Bronnikov the phase filter was integrated together with the ramp filter into the tomographic reconstruction, however the phase retrieval can be also performed prior to reconstruction on the projection images. This is actually done mostly in practice, which we also follow in the present paper. The contact image $I(r,0)$ is experimentally challenging and not practical to measure. For pure phase objects or negligible attenuation it represents a normalization by an open-beam (no object) image, $I_0$, instead of $I(r,0)$ in Eq.6. Finite object attenuation is typically taken into account adding an empirical regularization parameter, $\alpha$, to the denominator of Eq.6 also to compensate the singularity at zero frequency ($k=0$) and stabilize the reconstruction. This leads to:

$$\bar{\phi}(r) = -\frac{k}{z} F^{-1} \left[ \frac{F[I(r, z_{\text{eff}})/I_0 - 1]}{\alpha + k^2} \right]$$  \hspace{1cm} (7)

Eq.7 is the modified Bronnikov algorithm (MBA) (Boone et al., 2009). De Witte et al. (2009) proposed an alternative approach the so-called Bronnikov-Aided-Correction (BAC), which is a two-step algorithm. In the first step, an approximation for the phase distribution, $\phi(r)$ is obtained by applying Eq.7. Then the contact image is estimated by expressing it with the help of Eq.5 as

$$I(r,0) = \frac{I(r, z_{\text{eff}})}{1 - \gamma \bar{\chi} \bar{\phi}(r)}$$  \hspace{1cm} (8)
where \( z/k \) was replaced by a control parameter \( \gamma \) that is typically determined empirically together with \( \alpha \) (Salditt et al., 2017). A common strategy for determining \( \alpha \) is to examine in \( \phi(r) \) obtained in the first step profiles through edges and material boundaries and decrease \( \alpha \) starting from larger values until edge effects are eliminated. Then adjust the strength of the phase correction in second step increasing \( \gamma \) starting from small values and its final value is determined by visual inspection. We also apply this strategy here. The result is an effective attenuation image containing both attenuation and phase contrast.

Besides the BAC algorithm, we use here the likely most often applied single-distance phase retrieval algorithm by Paganin et al. (2002). It is derived assuming a fixed-stoichiometry object, i.e. with a constant \( \delta/\beta \) ratio, as

\[
\phi(r) = -\frac{\delta}{2\beta} \ln \left( \mathcal{F}^{-1} \left[ \frac{\mathcal{F}\{I(r, z_{\text{eff}})/I_0\}}{1 + \frac{\lambda z_{\text{eff}} \delta}{4\pi\beta |k|^2}} \right] \right) \tag{9}
\]

This is in effect very similar to the MBA algorithm representing a second-order low-pass filter in the frequency domain. For coherent, mono-chromatic radiation and single-material sample the parameters in Eq.9 are uniquely defined. However, for partially coherent, broad-spectrum laboratory setup the parameters in the denominator in front of the quadratic frequency term are typically lumped into a single coefficient that is determined empirically (Brombal et al., 2019; Bidola et al., 2015; Salditt et al., 2017). Empirical determination of the coefficient is also done if the Paganin retrieval is applied in the above form for multi-material samples, which is often the case in practice. Due to the low-pass filtering, the Paganin retrieval has been demonstrated to greatly improve the SNR and CNR of the images without significantly blurring the image and influencing its spatial resolution (Kitchen et al., 2017; Nesterets and Gureyev, 2014; Brombal et al., 2018). This property of the retrieval method has been recently placed on a firm theoretical basis (Gureyev et al., 2017). We elaborate on the relation between CNR and spatial resolution with respect to the strength of the low-pass filtering effect for our samples in detail in the next section. Note that the Paganin method was later extended for multi-material samples (Beltran et al., 2010, 2011). An extensive comparison of different phase retrieval methods including the ones detailed above and so-called Fourier-type methods based on the CTF in Eq.4 can be found in Burvall et al. (2011) and in Chen et al. (2013).

Results and Discussion

After choosing the optimal hardware combination available on the setup as explained under Methods, one can also optimize the imaging geometry to obtain high-sensitivity phase imaging. Quite a few studies dealt with the optimization of the imaging geometry for PBI both for monochromatic, coherent synchrotron radiation and for partially coherent, broad-spectrum sources (Gureyev et al., 2008; Nesterets et al., 2005, 2018). A propagator-based approach was introduced in (Balles et al., 2016) for laboratory scanners. Especially for the latter case, either the physical dimension of the device or considerations for affordable scan times limit the maximum practically applicable source-to-detector distance \((z_1 + z_2)\). In general, with increasing of this distance the phase-contrast increases (Gureyev et al., 2008). Then one typically optimises the magnification, \( M \). Nesterets et al. (2018) points out that there is always a trade-off between the spatial resolution and the CNR. Both cannot be fully optimized at the same time. If both are equally important, they suggest to find an intermediate value for \( M \) degrading both CNR and spatial resolution equally. The theoretical formulations for optimal \( M \) in Gureyev et al. (2008) and in Balles et al. (2016) result in somewhat differing
values. In our aforementioned preliminary tests on simple, single-material test objects, we found a closer match with the former study, thus we also tried to optimise $M$ accordingly for our study here, which resulted in the values given in Table 1. Note that some boundary conditions like the size of ROI in the sample needed to be captured might further limit the ability and parameter space for optimizing the geometry.

As a last step of the optimization of our PBI imaging, we examine the effect of the Paganin phase retrieval on the CT reconstruction by adapting the -6dB cut-off frequency, $v_c = f v_{Ny}$, of the second-order low-pass filter term in Eq. 9 as

$$G(v_x, v_y) = \frac{1}{1 + |v_x^2 + v_y^2|/v_c^2}$$  \hspace{1cm} (10)$$

where $f$ is the ratio of the cut-off and the $v_{Ny}$ Nyquist frequencies. It has to be emphasized here again that the goal of the optimization is the best possible visualization and CNR of the sample internal structures to enable or improve subsequent image processing steps such as segmentation, structural analysis, porosity, wall-thickness evaluation etc. rather than performing quantitative PBI.

We have elaborated for all three samples the CNR and the spatial resolution as a function of the cut-off frequency in a parametric study varying the ratio $f$, to find an optimal value in terms of these two image parameters. The CNR between two different materials in the samples or between the sample material and surrounding air is calculated as:

$$CNR = \frac{|I_1 - I_2|}{\sqrt{\sigma_{I_1}^2 + \sigma_{I_2}^2}}$$  \hspace{1cm} (11)$$

where $I_{1,2}$ are the reconstructed image intensities in a ROI for material 1 and 2. The spatial resolution is determined by taking azimuthally-averaged radial power spectra based on the 2D Fourier transform of the reconstructed images. Following Modregger et al. (2007); Schulz et al. (2010); Mizutani et al. (2016), the noise level has been estimated from the high-frequency, flat end of the radial power spectra and the resolution was estimated from the maximum spatial frequency at which the power spectrum surpasses twice the noise level.

Fig. 1 shows the results for the three samples. It is clear from the figures that the spatial resolution is only marginally influenced with increasing strength of the phase retrieval filtering. Note that accuracy of the spatial resolution is ±3.5% for CFRP, ±1.4% for cellulose foam and ±0.6% for PF sample (at 2σ-level). Furthermore, we can also see that it is safe to decrease the cut-off frequency as low as at least $f=0.4-0.5$, which results in an at least 5-6-fold increase in the CNR. We show below PBI images compared to transmission images using an $f$ in this range for the CFRP and the PF samples (Figs 6 and 7) and even lower for the cellulose sample (Fig. 4). Those figures also illustrate that such a choice of $f$ is also sufficient to practically fully eliminate the edge effect.

Although the CNR would increase even further for further lowering $f$, a trade-off has to be taken due to another consideration, highlighting a further interesting aspect of the Paganin phase retrieval, which we discuss here. As pointed out by Nesterets et al. (2018), due to the phase retrieval the noise in the images (both projection and CT) becomes more correlated. There is a trade-off between decreasing the noise amplitude by increasingly stronger low-pass filtering and the increase of its spatial correlation length. The phase retrieval reduces the magnitude of the noise but makes it correlated (textured). This is confirmed by plotting the Fourier Ring Correlation (FRC) of the CT images
FIGURE 1 Parametric study of the influence of the Paganin phase retrieval and $\nu_c$ on the CNR and the spatial resolution for the three samples. The CNR between the foam materials and air is shown for the cellulose foam and PF scaffold, whereas the CNR between the fibre material and epoxy is given for the CFRP sample. The accuracy of the spatial resolution is $\pm 3.5\%$ for CFRP, $\pm 1.4\%$ for the cellulose foam and $\pm 0.6\%$ for the PF (at $2\sigma$-level).

for different $\ell$ factors as is shown in Fig.2. The FRC has originally been introduced in electron microscopy (Van Heel and Schatz, 2005) but has been later also used to evaluate the spatial resolution of X-ray images (VilaComamala et al., 2011; Guizar-Sicairos et al., 2012; Wakonig et al., 2019). It is defined as the normalised cross-correlation coefficient between two corresponding rings of radius $\nu$ in the Fourier transform of image 1 and 2:

$$\text{FRC}(\nu_i) = \frac{\Sigma_{\nu\epsilon\nu_i} F_1(\nu) F_2^*(\nu)}{\sqrt{\Sigma_{\nu\epsilon\nu_i} F_1^2(\nu) F_2^2(\nu)}} \quad (12)$$

It measures the correlation between two independent realizations (1,2) of the same image. To produce the FRCs in Fig.2, we have divided the original CT data set into two, separating every even projection to set 1 and every odd projection to set 2.
The FRCs clearly show a strongly increasing correlation in the high frequency region, directly below $\nu_{Ny}$, where practically noise dominates the image and practically no contribution from actual sample structure is present. The frequency band of high correlation is also gradually increasing and extends towards lower frequencies for decreasing $f$. In the low-frequency region ($\nu \leq 0.4 \nu_{Ny}$), where the contribution from actual sample structures dominates, the correlation increases much less (CFRP sample) or not at all (PF sample) for decreasing $f$. To investigate how the noise correlation length increases, we separate the reconstructed images into a high frequency image containing primarily noise and a low-frequency image containing predominantly the actual sample structures by 2D band-pass filtering in Fourier domain. This ideal filter is defined for the low-frequency image as

\[
H(\nu) = \begin{cases} 
1 & \text{for } \nu \leq c\nu_{Ny} \\
0 & \text{for } c\nu_{Ny} < \nu \leq \nu_{Ny}
\end{cases}
\]  

(13)

with $0 < c < 1$ and vice versa for the high-frequency image. The factor $c$ has been estimated by examining the behaviour of the RMS deviation between the low-frequency image and the image containing the full spectrum as a function of $c$ and by visual inspection. The RMS is typically large for low values of $c$ as the low-frequency image does not fully contain all the dominant, actual sample structures present in the full image. At a certain $c$ value the RMS drops relatively quickly and levels off with further increasing $c$ (see the inset in Fig.3c for a typical example). The value of $c$ where the levelling off starts is chosen and confirmed by inspection of high-frequency (noise) image that it contains no visible remaining sample structures. This resulted in $c=0.9$, 0.8 and 0.65 for the CFRP, the cellulose and for the PF samples, respectively. Fig.3 illustrates the increase in noise correlation length for the case of the CFRP sample. It shows the autocorrelation function (ACF) of the separated noise component for varying $f$ fraction of the cut-off frequency of the Paganin filter. The effect and the trend is clearly visible: apart from the white noise peak at 0,
for decreasing $f$ there is a broadening of the ACF indicating increasing correlation length of the noise. The separated, high-frequency noise component of CFRP image is shown in Fig. 3b for $f=0.5$ for the phase and in Fig. 3c for the transmission image, respectively. These images also deliver a visual impression of how noise correlation and texturing increases by the Paganin filtering. The same trend in the ACF of the high-frequency noise component was observed for the other samples as well.

The contribution of correlated noise compared to the large-scale, dominant structures of the samples in the image is in general modest. However, correlated and textured noise can decrease the visibility of or camouflage small and weak structures in the object having sizes comparable with the noise correlation length and therefore diminish the quality of the imaging analysis. Texturing of the noise can indeed be observed as shown above for the CFRP samples, therefore our results indicate that decreasing $f$ significantly below 0.4-0.5 can potentially have and adverse for the image quality and for the visibility of small and weak sample structures. This should be kept in mind when choosing the value of $f$ for the Paganin retrieval.

**FIGURE 3** (a) Autocorrelation function (ACF) of the separated noise component for the CFRP sample for varying $f$ fraction (shown in the legend) of the cut-off frequency of the Paganin filter. For decreasing $f$ there is a clear broadening of the ACF indicating increasing correlation length of the high-frequency noise. Note that the vertical scale has been adjusted for better visibility of the effect. The separated noise component of CFRP image is shown for $f=0.5$ retrieval (b) and for the transmission image (c). These images also deliver a visual impression of how noise correlation and texturing increases by the Paganin filtering. The inset in (c) shows a typical example of RMS curve determining the parameter $c$ for separating the low- and high-frequency (noise) images.

### Cellulose foam

Fig. 4 shows the transmission CT image of the cross section of the cellulose foam sample compared to the corresponding PBI based on the phase retrieved by the Paganin filter with $f=0.2$. In spite of the noise correlation and texturing considerations explained above, for this sample we had to choose such a low $f$ value to obtain images of sufficient quality for the subsequent image processing steps. The grey scale images demonstrate the immense improvement in structure visibility. Without such a strong Paganin filtering those structures are hardly discernible even visually not to mention for automated segmentation algorithms. Clearly, such a sample can only be analysed using the phase-contrast image. Note that this sample was very fragile and soft and the higher intensities along the periphery of the
sample on the reconstructed images are very likely due to physical compression even in case of extremely careful sample handling and fixing on the scanner.

Fig. 5 shows a 3D rendering of a small internal ROI of the segmented cellulose foam sample based on the PBI shown in Fig. 4b. It shows its highly complex, entangled porous structure. The cellulose material is shown in light brown on the left image whereas the pores are transparent and to increase the visibility of the structures, the volume shown is clipped along a slanted plane (the flat looking parts). The right image illustrates in colour the pore volumes (colour scale is according to the pore size) and in semi-transparent, grey the cellulose material demonstrating that phase retrieval enables pore-size distribution analysis to be done.

| CFRP |

Fig. 6 compares the transmission CT image of the cross section of the CFRP sample with the corresponding PBI based on the phase retrieval by the Paganin filter with $f = 0.5$. Similar to the case of the cellulose foam, the visibility and CNR is greatly improved by using the phase image and phase retrieval (>factor 6 as is shown Fig. 1). Here we determined the CNR between the epoxy matrix and the carbon fibers.

| PF scaffold |

Fig. 7 depicts the transmission CT image of the cross section of the PF sample compared to the corresponding PBI based on the phase retrieval by the Paganin filter. Here similar to the CFRP and opposed to the cellulose foam sample also a more modest filtering with $f = 0.5$ is applied. The grey scale image and the histograms show a significant improvement in image quality and CNR increase by more than a factor 7. Fig. 8a shows a 3D rendering of the pores in an internal ROI of the segmented PF sample based on the phase image in Fig. 7b where the foam wall structures are made transparent. Fig. 8b depicts the pore walls (struts) of the foam structures. The quality of the PBI enables also for this sample automated pore size distribution and wall thickness distribution analyses as proven by these figures. Note that the image analysis shown in the paper have been carried out by the commercial software package VGStudio Max 3.3. As with the most commercial image processing packages, the details of the algorithms applied are proprietary. Nonetheless, the porosity analysis algorithm used here and for the PF sample (VGDefX), to the author's knowledge, is based on searching for local minima of the grey value inside the sample and applying a local watershed method adjusting the threshold value locally. The wall thickness is calculated based rolling sphere method by taking the maximum possible inscribed sphere in the wall at the point of thickness measurement.

For the PF sample in Fig. 7b and c, we compare the BAC algorithm with the Paganin retrieval to see what benefit one might offer with respect to the other. It shows clearly that both the Paganin and the BAC phase retrieval improves the CNR and the image quality. While in the transmission image, diffraction (edge) effects are clearly visible making the further image processing steps much more difficult, these are practically fully eliminated in the phase images. Although the BAC image appears to be somewhat more noisy than the Paganin-based one, it preserves small-scale structures somewhat better as is demonstrated by the small pores at the tip of orange arrow in Fig. 7b and c, which can only be discerned as separate on the BAC image. Note that the size of these features is only a few pixels being close to the resolution limit. On the other hand, the image based on the Paganin retrieval visualizes better the small differences and variations in the material in the rim of the sample (see green arrows). Though it is not shown here for brevity, if we increase the cut-off frequency of the Paganin phase retrieval filter to around $f = 0.7-0.8$, then these two small
pores can be discerned as separate. However the image quality, grey value histogram and correspondingly the image segmentability becomes much worse than for BAC image and the edge effects become visible and are not eliminated fully as for the BAC image. In other words, our results indicate that if the priority is to visualize the smallest possible details in the sample at the cost of some extra noise, the BAC algorithm should be preferred. Whereas if maximizing the overall image CNR is important, it is preferable to use the Paganin retrieval using a low $r$ in the range of 0.4-0.5.

Note the trend in the grey value histograms becoming more pronouncedly bimodal or even multimodal in Figs.4,6,7, which is the direct consequence of the phase retrieval improving significantly the CNR of the images. In Fig.6 it appears to a smaller extent showing a little shoulder on the left of the phase-retrieved histogram being less unimodal as for the attenuation image. This is consistent with the fact that the least improvement in CNR is observed for the CFRP sample compared to the others (see Fig.1).

Conclusions
We have discussed in the present paper the application of the propagation-based phase-contrast technique for laboratory micro- and nano-CT scanners. We have applied a multi-step optimization of the technique for our commercial CT scanner starting with choosing an optimal combination of the available hardware options for the detector (small pixel size, selective spectral sensitivity) and source (small-mid focal spot). In a next step we optimized the geometry of the imaging for the given physical boundary conditions of the scanner and finally the phase retrieval process by performing parametric studies. We compared the Paganin retrieval with the BAC algorithm and pointed out the benefits of each one with respect to the other. We also demonstrated the increase of the noise correlation length and corresponding noise texturing effect of strongly filtering Paganin phase retrieval. This might have an adverse influence on image quality and decrease the ability to distinguish and recognize small and weak features and structure in the sample. We have clearly demonstrated the benefits of laboratory inline phase-contrast imaging for different, industrially relevant low-density, low-attenuation material samples: a porous cellulose and a PF scaffold and a CFRP composite. A significant improvement in the image quality in terms of CNR, up to almost a factor 60 (for the cellulose foam sample) have been obtained, accompanied by only marginal changes in the spatial resolution. This enabled further automated image processing steps for detailed structural analysis of the samples, such as segmentation, pore-size and wall-thickness distribution analysis, which would have been much more difficult or not feasible at all based on the transmission image.

Acknowledgements
The author is grateful to Dr. M. Rottmar, Dr. A.J. Brunner, T. Wu and Dr. G. Nyström of Empa for providing the samples.
Figure 4  (a) Transmission CT image of the cross section of the cellulose foam sample. The fine internal porous structures of the very low density sample are hardly visible and distinguishable from the background on the transmission image. (b) The corresponding PBI based on the phase retrieved by the Paganin filter with $f=0.2$. The internal porous structure of the sample is very well visible. The subsequent segmentation of the pores from the cellulose material is only possible based on the phase image (see Fig5). The insets show the respective image grey value histograms both in linear (black) and logarithmic scale (grey). (c),(d) show a zoom-in on the yellow ROI for each of the corresponding images in the upper row to make the fine details of the sample structure more visible.
FIGURE 5  (a) 3D rendering of a small internal region of the segmented cellulose foam sample showing its highly complex, entangled porous structure based on the PBI in Fig4b. The cellulose material is in light brown and volume is clipped along a slanted plane corresponding to the flat parts. The image shows very clearly the porous cellulose structures. (b) Porosity analysis of the cellulose sample. The colour scale shows the pore size. The cellulose walls and structures are shown in grey and are made semi-transparent.

FIGURE 6  (a) Transmission CT image of the cross section of the CFRP sample. (b) The corresponding PBI based on the phase retrieved by the Paganin filter with $r=0.5$. The insets show the respective image grey value histograms both in linear (black) and logarithmic scale (grey).
FIGURE 7 (a) Transmission CT image of the cross section of the PF foam sample. (b) The corresponding PBI based on the phase retrieved by the Paganin filter with $f=0.5$ and (c) by the BAC algorithm using $\alpha=0.05$ and $\gamma=5$. (d),(e),(f) show a zoom-in on the yellow ROI for each of the corresponding images in the upper row. They illustrate clearly how both the Paganin and BAC phase retrieval improves the CNR and the image quality. In the transmission image, diffraction (edge) effects are clearly visible making the further image processing steps much more difficult. Though the BAC image appears to be somewhat more noisy than the Paganin-based one, it preserves small-scale pores better, as indicated by the orange arrow, that can only be discerned as separate on the BAC image. However, the Paganin-based image visualizes better the differences in the rim of the sample (green arrows). The insets show the respective image grey value histograms both in linear (black) and logarithmic scale (grey).
FIGURE 8  (a) 3D rendering of an internal ROI of the segmented PF sample based on the phase image by the Paganin retrieval shown in Fig7b. The image shows the pores while the foam wall structures are made transparent and the colour scale reflects the pore volume. (b) 3D rendering of the pore wall (strut) structures coloured according to the local wall thickness. Large merged pores have been separated by the image processing algorithm and the separation faces are shown in magenta. For a better visualisation of the latter walls are shown semi-transparent.
References


