An enhanced X-ray imaging contrast using a grating-based talbot interferometry

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**AN ENHANCED X-RAY IMAGING CONTRAST
USING A GRATING-BASED
TALBOT INTERFEROMETRY**

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Summary

We often deal with objects made of low-density materials in various settings, such as soft biological tissues in the medical sciences and manufacturing of food, and low-density polymeric substances in the manufacturing of medical devices and automotive parts. Inspection of low-density materials using the conventional X-ray imaging that is based on the principle of X-ray absorptions often results in a lack of image contrast because these materials do not generate satisfactory absorption-contrast under the irradiation of typical range of hard X-rays. Furthermore, in many cases, the objects always come in irregular form of shapes and sizes, this makes the X-ray imaging challenging as different X-ray energies are required to penetrate through different range of object thickness in order to provide a satisfactory visibility of fine defects and structural profiles residing beneath the surface.

In this thesis, we demonstrated the potential of a low-brilliance X-ray Dark-field imaging in achieving significant contrast enhancement, and picking up fine details in different types of low-density materials and across a range of object thicknesses. We have developed an X-ray Dark-field imaging prototype system based on the principle of fringe-shifting using a 2-grating X-ray Talbot interferometry. Under the illumination of low-brilliance micro-focused X-rays, we showed that our proposed system yielded higher CNR (Contrast-to-Noise Ratio) than the conventional method in the imaging of low-density materials. In addition, we introduced visibility transformation functions (both linear and non-linear approaches) to further enhance the contrast of the acquired Dark-field image, thus elevate the visibility of the sub-surface details and structural edges.

Three kinds of experiments have been designed to demonstrate the potential of X-ray Dark-field imaging system as a diagnostic tool for the inspection of biological and manufacturing samples, and for material characterisation. In the imaging of biological samples, the CNR in our X-ray images remained satisfactory across a large variation of object thicknesses which then allowed us to pick up both surface and sub-surface details in the micrometer length scale, which are not detectable using the

absorption-contrast. In the inspection of manufacturing part such as a glass panel sandwiched in the opaque covers, our system successfully revealed the sub-surface cracks which have interrupted the propagation of X-rays. Finally, in the last experiment, we showed that our method can visualize the bundle of soft cottons which are not detectable by the absorption-contrast, and it complemented the conventional X-ray imaging by differentiating cotton, grains and seeds in terms of various grey levels.

From the experiments, we realized that X-ray Dark-field imaging is a promising technique that utilizes the (U)SAXS power of material structures to achieve higher detectability, resolving power and structural discrimination. Furthermore, we have demonstrated that our method produced good CNR even using a low-brilliance microfocus X-ray tube, paving the way for multi-contrast diagnosis and inspection in the medical and manufacturing settings.

1. Introduction

X-ray imaging is a well-known diagnostic technology in the medical field as well as in non-destructive inspection industries since the past decades. In comparison with optical light, X-rays can penetrate through objects; giving them an advantage of “seeing through” thicker objects and also objects with surface opaque to the visible light.

Due to wave-particle duality, X-ray exhibits the characteristics of being a particle (photon) as well as a wave (electromagnetic wave). Firstly, from the particle perspective, an X-ray photon can be described as a particle carrying discrete amount of energy and momentum in the range of hundreds of eV to hundreds of keV. Such high energy allows the photon to penetrate through objects. When X-ray travels through a matter, X-ray photon interacts with the electrons of the matter via photoelectric effect, i.e. if the X-ray photon energy is higher than the binding energy of electrons in the atom, the photon can be absorbed by releasing an electron from the atom. This quantum mechanical process results in a reduction of the total number of X-ray photons to a certain degree, depending on the “absorption capability” of the material, and its intensity is governed by the linear attenuation law¹:

$$I(x) = I_0(x)e^{-\mu x} \quad (1.1)$$

where $I_0(x)$ is the initial intensity, μ is the wavelength-dependent linear attenuation coefficient, and x is the material thickness, i.e. the distance X-ray travelled in the material. The absorption difference in different material generates an absorption contrast in the X-ray image. This is why an X-ray image shows good contrast of a bone or metal (strongly absorbing) and soft tissues or plastics (weakly absorbing). This technology has been broadly adopted in the conventional X-ray radiography^{2,3}.

Secondly, researchers had already shown that X-ray behaves as a form of electromagnetic (EM) wave. X-ray has a much higher frequency ($>10^{18}$ Hz), in another

words, it has very short wavelength as compared to the visible light. Similarly, in the X-ray regime, a material refractive index can be described as⁴⁻⁶:

$$n(\lambda) = 1 - \delta(\lambda) + i\beta(\lambda) \quad (1.2)$$

where n is the refractive index of material, δ is the phase coefficient (real part) and β is the absorption coefficient (imaginary part). As X-ray travels through a matter, the wavelength-dependent refractive index of the material causes X-ray to experience a change of wave vector, hence a deviation in the propagation direction. This event results in a phase-shift of the X-ray wavefront, given by^{7,8}:

$$\phi(x) = \frac{2\pi}{\lambda} \int \delta(x, z) dz \quad (1.3)$$

where $k = \left(\frac{2\pi}{\lambda}\right)$ is the wave number, δ is the phase coefficient of the material refractive index, H is the X-ray travelled distance in the material. Furthermore, the X-ray characteristic wavelength and frequency can be demonstrated through interference effects when two X-ray beams interfere in space.

Thirdly, another process in the interaction between X-ray and matter has been actively explored is the scattering process. In this microscopic view, X-ray is being treated as a photon (particle), but instead of being absorbed by the atom, a scattered photon merely changes its wave vector and propagation direction. Because of the change of wave vector, the X-ray will experience a phase-shift. There is no reduction of photon number in this sense as long as the scattered photon arrives at the detector.

The phase-shift effect coming from the X-ray refraction and scattering has led researchers to explore a new image contrast formation, i.e. phase contrast and visibility contrast (scattering contrast). X-ray Dark-field imaging (Figure 1) exhibits several advantages over conventional absorption contrast. Firstly, the generation of image contrast does not depend on the photoelectric process in the material. For a

weakly-absorbing material (usually made up of light elements with low atomic number, such as oxygen, hydrogen and carbon) like thin layer of soft tissue and polymer, the probability of photoelectric process is small in proportion with the lower density of electrons in the atoms, giving poor absorption contrast in the X-ray image. As a result, the objects appear almost transparent in the image. But in the hard X-ray regime (X-ray energy in the range of 10-100 keV), the phase-shift experienced by the X-ray is of 3 order of magnitude higher sensitivity than the absorption, suggesting that phase contrast is superior and can generate better X-ray image contrast in terms of edge enhancement and improved visibility⁹. Secondly, as the image contrast is formed independent of the photoelectric absorption process, the material under inspection does not need to absorb as much X-ray photons as required in conventional X-ray imaging. Consequently, the damage to the biological tissues and polymeric specimen will be proportionately reduced, even though in some cases, the specimen is subjected to longer exposure when using a low-brilliance X-ray source or during a CT (Computed Tomography) scan.

Figure 1 X-ray images of chicken wing (left) Conventional X-ray: absorption-contrast (right) Visibility-contrast (Dark-field). Extracted from F.Pfeiffer., et al, "Hard-X-ray dark-field imaging using a grating interferometer", Nature Materials, Vol. 7 (2008)

1.1 Motivation, scope and objectives

There has been an increasing interest to enhance the potential of X-ray imaging to achieve higher detectability and imaging resolution for medical and manufacturing applications. Using conventional X-ray imaging that is based on absorption-contrast to detect microstructures in low-density material matrix has raised several issues. Firstly, the absorption-dependent imaging method requires the microstructures to have different absorption coefficients from the surrounding material. However, this condition is very difficult to meet especially in the imaging of material composition of almost similar absorption coefficients. Secondly, in order to detect the microstructure, the detector cell size in principle has to be at least two times smaller than the size of the microstructure, which currently is limited by the technology development of a detector to have a pixel size in the range of sub-micrometer.

The conventional absorption-contrast imaging method generates different grayscale values in the image as X-rays penetrate through various specimen thicknesses. However, when imaging an object composed of weakly-absorbing material with non-uniform spatial thickness, two major concerns have arisen. Firstly, in cases where the object is formed from a matrix of materials with weak and almost similar absorption cross-sections (e.g. biological tissues, food and polymer), the resulted X-ray absorption-contrast is very poor. Secondly, in the case of objects with large varying thicknesses, the X-ray energy needs to provide sufficient penetration through the thickest part of the object, yet the X-ray energy has to be kept low because low X-ray energies are more efficiently absorbed by the thinner regions of the object so as to generate a detectable contrast in the X-ray image, as dictated by the Linear Attenuation Law.

Several alternative imaging modalities have been proposed to overcome the above challenges and to enhance the X-ray image contrast. Among these methods are the Phase-contrast and Visibility-contrast (Dark-field) methods which provide complementary information not previously obtained in the conventional method.

The phase-contrast^{6,10-14} imaging methods had shed new light in the diagnosis and inspection of light materials such as biological tissues, mammography, angiography as well as polymer. This method is sensitive to the deviation of X-rays that happened at the edges and interfaces of materials where X-ray propagation has been disrupted.

X-ray Dark-field (visibility-contrast) imaging, by contrast, reveals sub-pixel information in low-density elements in the micrometer and sub-micrometer scales that were inaccessible via both absorption and phase contrast¹⁵⁻¹⁹. The Dark-field signal strength occurs most exclusively through small- and ultra-small angle X-ray scattering (SAXS and USAXS, respectively) from microstructures on a scale below the spatial resolution^{15,16,20} of the imaging system. The imaging method has evolved from previously analyser-based method using crystals²¹⁻²⁴ to using a compact grating interferometer^{16-18,25-29}. From initial setup using the high-brilliance synchrotron facilities^{10,30,31} the grating-based Dark-field imaging has increasingly gained its popularity because the system is tolerant to divergent and polychromatic beams, and hence is fully compatible with the low-brilliance benchtop-sized laboratory X-ray tubes^{15,16,32-37}. In terms of achieving a high X-ray imaging contrast and resolution, X-ray dark-field imaging has emerged as a promising approach.

The aforementioned observations motivated us to further investigate the potential of X-ray Dark-field imaging for exploring micro and sub-micro structures of specimens made of low density and weakly-absorbing materials such as polymer and biological tissues. The primary focus of this research is to develop a grating-based Talbot interferometry capable of feature detection at 10 μ m or lower, that can reveal details on the surface as well as under the surface of the specimen, which has not always been able to be analysed by using the conventional absorption-based X-ray radiography because the materials do not absorb X-ray photons sufficiently to produce satisfactory absorption contrast. Hence, we are exploring other properties of X-ray such as the (ultra) small-angle X-ray scattering to open up another avenue for us to generate X-ray image of better contrast. In addition, X-rays have always been used for imaging sub-surface structural details. Here, we extend our interest to also

the imaging of features on the surface, i.e. the external of specimen body, where there exists an interface between air and specimen.

Through this research, we aim to achieve the following objectives:

1. to develop a grating-based X-ray Darkfield imaging (XDI) prototype system to achieve an improved contrast compared to the conventional X-ray absorption-contrast in the imaging of low-density materials
2. to adopt phase-stepping mechanism of an analyser grating to allow sub-pixel resolving of fine details at micrometer and sub-micrometer scales
3. to perform assessment on the CNR (Contrast-to-noise ratio) of fringes generated by the gratings and the performance of phase-stepping
4. to design and conduct experiments using a variety of materials, such as biological samples, manufactured glass panel, soft cotton, grains and seeds
5. to evaluate the visibility of fine details (surface and sub-surface) across a large variation of object thicknesses

We proposed to develop a two-grating Talbot imaging prototype system using a low-brilliance X-ray microfocus tube. The system operates based on the principle of Talbot self-imaging effect and relies on the ability of the first grating, a phase grating, to diffract X-ray beams into fringe pattern with sufficient intensity, generating images with higher contrast and visibility. A second grating which is a binary absorption grating, also known as analyser grating is positioned downstream of the phase grating and right before the X-ray detector. Through phase-stepping procedure, this grating is linearly translated across the detector pixels, which serves to equivalently subdivide the detector cell into smaller unit as a means to increase the signal detectability and imaging resolution. Finally, a sequence of images are acquired and processed to generate Transmission (equivalent to absorption-contrast) and Darkfield images simultaneously. The two types of images are compared in every imaging experiment to find out the advantage of each imaging method.

1.2 Organization of the thesis

The rest of the thesis is organized as follow:

In Chapter 2, we studies the principle of a grating-based X-ray Dark-field imaging (XDI) which is necessary before we started to design and develop the prototype system. The relationship between (U)SAXS signal and visibility function is given, and the retrieval method of Dark-field signal from the visibility function is explained.

In Chapter 3, we provide the schematic diagram and a detailed description on the XDI system design and development. The specifications and functions of every sub-system are given, and the experimental conditions are highlighted.

In Chapter 4, we present the evaluation outcome on the image and phase-stepping quality. In particular, the influence of image integration on image quality and fringe visibility is studied. Further, the intensity consistency and impact of integration number across phase-step positions is also presented.

In Chapter 5, we demonstrate that using a low-brilliance XDI, we achieved satisfactory X-ray Dark-field image contrast and visualized additional details in the biological, manufacturing samples not accessible via the conventional absorption-contrast. In addition, we also present the potential of XDI in the aspect of material characterisation.

In Chapter 6, we conclude the thesis based on the experiment observations and findings. In addition, we provide suggestions on possible future research direction for medical and manufacturing settings.

2. Theory of grating-based X-ray Dark-field imaging

2.1 X-ray Dark-field signal as a function of (Ultra)Small-Angle X-ray Scattering

X-ray dark-field imaging, also known as scattering-based imaging, is able to detect fine features which is spatially inaccessible by X-ray detector, and it can discriminate materials based on the (Ultra)Small-Angle X-ray Scattering ((U)SAXS) properties of the elemental compositions. It utilizes the advantage of the short wavelength of X-ray. Since hard X-ray wavelength typically falls under 1nm, the microstructures with a mean size from micrometer down to sub-micrometer and even nanometer will contribute to (U)SAXS cross-section. When X-ray penetrate through sample and interact with these structures, the (U)SAXS effects that occurred usually leads to a reduction of detected signal intensity, which contribute to the so called X-ray dark-field signal. Hence, analytic formulae and mathematical concepts have been constructed to relate the microscopic physics to the resultant reduction of signal intensity^{16,25,29,37-40}. A variety of dark-field imaging modalities and experimental validation had been reported to detect the scattering effects using a crystal analyzer⁴¹⁻⁴⁴, based on DEI⁴⁴ and grating-based Talbot interferometer^{25,29,37,38}. Among all these methods, the grating-based Talbot interferometer is more appealing because it allows the use of conventional detector with pixel size in the range from micrometer to tens of micrometer and a laboratory x-ray tube. In this thesis, the central of discussion is placed on the Dark-field imaging using grating-based Talbot interferometer.

2.1.1 Relationship between (U)SAXS and visibility function

There has been an active investigation into how X-rays interact with “particulate” to generate (U)SAXS effect which eventually causes a reduction of detected X-ray intensity (visibility). From the literature, a number of theoretical frameworks are available to define the relationship between the (U)SAXS effect and intensity reduction. Furthermore, a visibility function has been established to describe and measure the fluctuation of intensity variation as a result of (U)SAXS. As

X-rays interact with the structures, the reduction of intensity can be measured as a visibility degradation, which gives the Dark-field signal.

Z. Wang *et. al.* describe the (U)SAXS event as an angular broadening of X-ray beam. The scattering-contrast is explained in terms of a quantitative relationship between 2nd moment of scattering angular distribution and visibility degradation²⁹. The 2nd moment of scattering angular distribution, σ^2 , is written in the form of line integral:

$$\sigma^2 = \int_0^L S(l)dl \quad (2.1)$$

where $S(l)$ is the sample-thickness dependent scattering parameter of the X-ray, which is related to the sample density, scattering cross-section and angular X-ray broadening due to scatterers in the sample. The mathematical derivation also showed that σ^2 is related to visibility degradation:

$$\sigma^2 = -\frac{1}{2\pi^2} \left(\frac{d}{z_0}\right)^2 \ln\left(\frac{V'}{V}\right) \quad (2.2)$$

with

$$\frac{V'}{V} = \frac{a'_1 a_0}{a'_0 a_1} \quad (2.3)$$

where d is the period of the phase-stepping intensity oscillation pattern in a grating-based Talbot interferometer, z_0 is the distance of intensity pattern from X-ray source and the ratio $\frac{V'}{V}$ represents the visibility degradation.

In the model derived by G.H. Chen *et. al.*, an intensity loss is also observed as a result of small-angle scattering (SAXS). Scatterer in material with a particle's mean size typically from ten of nanometer to hundreds of micrometer which is larger than the X-ray wavelength will contribute to the SAXS cross-section. This information is encoded in Talbot-Lau interferometer as an intensity profile which then corresponds to the modulation in visibility function³⁷. A physically measurable relationship between the visibility function $\ln V_s$ and the microscopic theory of SAXS:

$$\ln V_s = \int \frac{\sigma_s(l) \rho_s(l)}{\left(\frac{2R(l)}{r}\right)^2} dl \quad (2.4)$$

where σ_s is the small-angle scattering cross-section, ρ_s is the number density of scatterers, R is the characteristic size of scatters, and r is the spatial transverse distance measured from the incident wave direction.

Similarly, based on small-angle scattering, *M Bech et. al.* introduced a mathematical formalism that describes the visibility as a function of the material-dependent linear diffusion coefficient²⁵. The detected intensity loss in a grating-based Talbot interferometer is quantified as visibility decays where the visibility function is expressed in a relation with the scattering width:

$$V = \exp\left(\frac{-2\pi^2 z_s^2}{p_2^2} \int \epsilon(l) dl\right) \quad (2.5)$$

where z_s is the distance from the sample to the analyzer grating G2, p_2 is the period of analyzer grating and ϵ is the linear diffusion coefficient which is defined as a function of the scattering width per unit length:

$$\epsilon \equiv \frac{\sigma^2}{\Delta l} \quad (2.6)$$

where σ represents the width of small-angle scattering and Δl is the sample thickness.

Finally, *W. Cong et. al.* developed a physical model based on the principle of energy conservation to arrive at an expression that describes the balance of photon energy before and after the small-angle scattering event³⁸. The intensity loss due to SAXS is extracted for a specific X-ray propagation angle which is then integrated over a range of angles of interest. The visibility function is related to the intensity loss in terms of:

$$\frac{T_s}{T_a} = 1 - V \quad (2.7)$$

where T_s is the small-angle scattering transport and T_a is the transmitted photon.

2.2 Retrieval of X-ray dark-field signal from visibility function

A variety of grating-based dark-field imaging has been explored and experimental results have yielded higher X-ray image contrast and provided complementary information inaccessible by the absorption-contrast and phase-contrast. It is reported that the (U)SAXS imaging can achieve higher detectability as it is sensitive to interfaces and structural changes. The retrieval of the dark-field signal, i.e. the visibility degradation is realized by the grating-based Talbot interferometer which is an identical setup with the differential phase contrast imaging.

2.2.1 Principle of grating-based Talbot interferometry

The Talbot self-imaging effect is a diffraction effect in the near-field regime, named after Henry Fox Talbot who first observed the effect in 1836, when a plane visible light illuminated a periodic grating. It was observed that the wavefield downstream of the grating is an exact replica of the grating's transmission function, and the image of this pattern repeats itself at special distances (called the Talbot distances).

This phenomenon is therefore called "self-imaging". At the half of each Talbot distance, the image shows a self-image as before but now with a lateral shift of half of the grating period. Further, at some fractional distances, similar image appeared but now shows pattern periodicity at half the period of the grating. Later in year 1881, Lord Rayleigh in his article ("On copying diffraction gratings and on some phenomenon connected therewith" Philos. Mag. 11) provided a mathematical explanation and showed that the Talbot effect is a natural consequence of Fresnel diffraction.

2.2.2 Paraxial-approximated Fresnel Diffraction

Figure 2 Fresnel diffraction

As illustrated in Figure 2, Plane P₀ is the grating plane and Plane P₁ is the observation plane. When Plane P₀ is illuminated by a monochromatic light, the diffracted wavefield propagates from Plane P₀ for a distance r in the free-space to arrive at the observation plane P₁. In general, the incident wavefront is expressed as $E_0(x, y) = \frac{e^{ikr_s}}{r_s}$. From the Rayleigh-Sommerfeld diffraction integral, the diffracted wavefield at plane P₁ can be written as⁴⁵:

$$E_1(x', y') = \frac{1}{i\lambda} \int E_0(x, y) \frac{\exp(ikr)}{r} \cos(\vec{n}, \vec{r}) dx dy \quad (2.8)$$

To obtain the Fresnel diffraction pattern, we assume that all the wavefronts are making small angle with respect to the optical axis of the system, such that the paraxial approximation holds. Under this approximation, the cosine term is approximately 1 and the diffraction integral becomes

$$E_1(x', y', z) = \frac{1}{i\lambda} \iint E_0(x, y, z = 0) \frac{\exp(ikr)}{r} dx dy \quad (2.9)$$

The propagated distance $r = \sqrt{(x - x')^2 + (y - y')^2 + z^2}$ is comparable to z , and z^2 is relatively large compared to $(x - x')^2 + (y - y')^2$. The paraxial approximation further allows us to make the denominator of the equation as z . However, the exponential term in the equation is much more sensitive to the small variation in the values of $(x - x')^2$ and $(y - y')^2$. Hence, to ensure the validity of propagation in the

near-field region, Fresnel approximation first expresses $\mathbf{r}$ as a Taylor series, and by considering that z is large such that the higher order terms are negligible. With the above conditions hold true, the original Rayleigh-Sommerfield diffraction integral can now be approximated to Fresnel approximated diffraction integral as follow:

$$E_1(x', y', z) = \frac{1}{i\lambda} \iint E_0(x, y, z = 0) \frac{\exp(ikz) \exp(\frac{ik}{2z} [(x' - x)^2 + (y' - y)^2])}{z} dx dy \quad (2.10)$$

Rearranging the equation, we arrive at the convolution formalism of the Fresnel diffraction integral:

$$E_1(x', y', z) = \frac{\exp(ikz)}{i\lambda z} \iint E_0(x, y, z = 0) \exp(\frac{ik}{2z} [(x' - x)^2 + (y' - y)^2]) dx dy \quad (2.11)$$

This equation can be categorized into two terms, i.e. one term is dependent on x while another term is dependent on y :

$$E_1(x', y', z) = \frac{\exp(ikz)}{i\lambda z} \int E_0(x) \exp(\frac{ik}{2z} (x' - x)^2) dx \int E_0(y) \exp(\frac{ik}{2z} (y' - y)^2) dy \quad (2.12)$$

In this research, a one-dimensional grating is used and the structure periodicity is dependent on x -axis. For simplicity, we assume $E_0(y) = 1$. Hence, the second term in this equation which is dependent on y can be evaluated using a Gaussian integral.

Recall that the integral of a Gaussian function is given by:

$$\int \exp(-a(x + b)^2) dx = \sqrt{\frac{\pi}{a}} \quad (2.13)$$

Hence, the second term of the equation can be evaluated as:

$$\int \exp\left(\frac{ik}{2z}(y' - y)^2\right) dy = \sqrt{\frac{\pi}{\left(\frac{-ik}{2z}\right)}} = \sqrt{i\lambda z} \quad (2.14)$$

With that, we have now reduced the equation to a one-dimensional expression:

$$E_1(x', z) = \frac{\exp(ikz)}{\sqrt{i\lambda z}} \int E_0(x, z = 0) \exp\left(\frac{ik}{2z}(x' - x)^2\right) dx \quad (2.15)$$

Now, with the plane P_0 being replaced by an infinitely periodic amplitude grating which has a period a , and one-dimensional transmission function $T(x)$, the wavefield immediately after the grating can be assumed to be equal to $T(x)$. This allows us to write the Fresnel integral in the form of:

$$E_1(x', z) = \frac{\exp(ikz)}{\sqrt{i\lambda z}} \int T(x) \exp\left(\frac{ik}{2z}(x' - x)^2\right) dx \quad (2.16)$$

However, it is impractical to calculate the diffraction field in the form of infinite integral, but by converting it into the finite sum⁴⁶, we arrive at a general analytical expression which describes the wavefield's amplitude distribution at the observation plane:

$$E_1(x', z) = \frac{1}{\sqrt{i2mn}} \sum_{l=0}^{(2mn-1)} T\left(x + l\frac{p}{n}\right) \exp\left[i\frac{\pi l^2}{2mn}\right] \quad (2.17)$$

where m and n are integers, l is the running variable, and p is the grating period. The intensity modulation is simply the modulus square of this equation. By substituting distance $z = \left(\frac{m}{n}\right) \frac{2p^2}{\lambda}$ into the equation, it allows us to observe amplitude modulation describing the Talbot and fractional Talbot effects at various z distances.

Three cases have been analysed to show the Talbot self-imaging effect at various z distances, see Figure 3:

- Case 1: At Talbot distance, i.e. $z = (1)(\frac{2p^2}{\lambda})$
The wavefield, $E(x, z) = T(x)$, exact replica of grating transmission function
- Case 2: At odd integer (m') multiple of Talbot distance, i.e. $z = (m')(\frac{2p^2}{\lambda})$
The wavefield is also an exact replica as in Case 1, i.e. $E(x, z) = T(x)$
- Case 3: At Half Talbot distance, i.e. $z = (\frac{1}{2})(\frac{2p^2}{\lambda})$
The self-image is now translated by half-period, i.e. $E(x, z) = T(x + \frac{p}{2})$

The same result can also be obtained by another approach⁴⁷ in which the grating's complex amplitude transmittance is represented by a Fourier series. This means that the wavefield right behind the periodic grating is decomposed into a series of angular spectrum of plane waves, each traveling at individual direction making an angle with respect to the grating surface. The grating transmittance is then the summation of all these plane waves. The calculation has shown that the self-image of the grating appears at even multiples of $\frac{2p^2}{\lambda}$, which agreed with the results obtained by Guigay⁴⁶.

Figure 3: Intensity distribution of self-imaging effect for an amplitude grating with period a . Adapted from T. Weitkamp, et al, "Tomography with grating interferometers at low-brilliance sources", Proc. of SPIE Vol. 6318, 63180S (2006)

2.2.3 Fractional Talbot effect produced by a phase grating

All the preceding discussions are based on the assumption of a periodic grating with complex amplitude transmission function. If $T(x)$ is a pure phase transmission function for which $T(x) = \exp[i\varphi(x)]$, i.e. no absorption in the grating but a real and periodic phase modulation function $\varphi(x)$, then the above results cannot be entirely applied.

For instance, at the Talbot distance $z = (2p^2)/\lambda$, if we directly substitute the pure phase transmission function $T(x) = \exp[i\varphi(x)]$ into the Fresnel diffraction integral equation, there is no intensity modulation that can be detected by the sensor. Instead, a constant intensity image is obtained without any observable Talbot self-image of the phase grating, see Figure 4. On the other hand, if we shift the distance to the quarter of the Talbot distance, i.e. $z = \left(\frac{1}{4}\right) * \left(\frac{2p^2}{\lambda}\right)$ we obtain the wavefield amplitude as follow:

$$E_1(x', z = \frac{p^2}{2\lambda}) = \frac{1}{\sqrt{i8}} \sum_{l=0}^7 T(x + l \frac{p}{4}) \exp[i \frac{\pi l^2}{8}] \quad (2.18)$$

It was also shown that the Talbot effect with half a grating period can be obtained at 1/16 of the Talbot distance, where $z = \left(\frac{1}{16}\right) * (2p^2/\lambda)$ ⁴⁶.

Figure 4: Intensity distribution of self-imaging effect for a phase grating with period a . Adapted from T. Weitkamp, et al, "Tomography with grating interferometers at low-brilliance sources", Proc. of SPIE Vol. 6318, 63180S (2006)

2.2.4 Phase-stepping and Dark-field image reconstruction

Among the many detection methods, phase-stepping⁴⁸⁻⁵¹ and Moire analysis⁶ are the commonly used methods in X-ray phase contrast imaging system. For each of these processes, the data analysis algorithm that follows will be different. Further, the advantages and limitations of the methods will determine the performance of the 2D phase reconstruction. A comparison between the two methods has been generally summarized⁵², and tabulated in Table 1.

Table 1: A comparison between fringe analysis methods of (a) phase-stepping (b) Moire analysis

Analysis method	Advantage	Disadvantage
Phase-stepping procedure	Higher spatial resolution, Generation of 3 kinds of information in a single experiment, i.e. differential phase contrast, dark-field and absorption images	Need for high-precision actuators in nano-meter with satisfactory reproducibility Multiple-shot, slow for tomography
Moire analysis	Single-shot, very fast image acquisition	Lower spatial resolution (resolution is very much dependent on the Moire fringe density)

With the advancement of the mechatronics technology today, the phase-stepping method appears to be a promising approach to gear towards the development of higher resolution X-ray Phase Contrast Imaging (XPCI). The need for high-precision actuators in nano-meter scale is no longer an unachievable target. Further, the advantage of the phase-stepping procedure in generating three kinds of images in a single experiment has further enhanced its potential to offer richer information that can never be achieved by conventional X-ray imaging methodology todate. The phase grating transformed the phase information into intensity modulation captured by the X-ray detector. The fundamental idea of the phase-stepping method is to evaluate the local changes of the intensity modulation induced by the sample, and determine from these several images two new information in X-ray imaging methodology, i.e. differential phase contrast (edge enhancement) and Dark-field (structural visibility), while preserving the conventional absorption contrast information.

The phase-stepping procedure can be illustrated by Figure 5:

Figure 5: The principle of phase-stepping procedure observed by detector. (a) System diagram of major components, (b) fringes pattern when sample is absent, (c) fringes pattern distorted when sample is placed in front of phase grating (G1), (d) illustration of 4-step linear translation of G2 to resolve distorted fringe pattern.

The absorption grating (G2) is scanned in ten steps over one grating period. Without the sample in place, a periodical intensity signal is observed. At 10 different grating positions, the same detector pixel captured 10 different intensity levels. Plotting the 10 intensity levels across the 10 stepping positions yields an intensity oscillation (also known as phase-stepping signal) at the pixel. In each pixel, the oscillating phase-stepping signal can be written as a Fourier series:

$$I(p_x, p_y, x_g) = a_0(p_x, p_y) + \sum_{m=1}^{\infty} a_m(p_x, p_y) \cos\left(\frac{2\pi m x_g}{g_2} + \phi_m(p_x, p_y)\right) \quad (2.19)$$

where $x_g \in [0, kp_2]$ over a grating's period and g_2 is the period of G2.

Using a laboratory micro-focus X-ray tube, the low coherence of the source only allows significant magnitude of the 1st order. Hence in practice, only a cosine function is observed¹⁶:

$$I(p_x, p_y, x_g) \approx a_0(p_x, p_y) + a_1(p_x, p_y) \cos\left(\frac{2\pi x_g}{g_2} + \phi_1(p_x, p_y)\right) \quad (2.20)$$

When the sample is in place, the X-ray refraction induced by the sample creates a refraction angle, which in turns caused local changes (transverse shifts) of the cosine intensity curve. From the measured phase-stepping signal oscillation in each pixel, the resultant transverse shift due to X-ray refraction which is induced by the sample is given by:

$$S(p_x, p_y) = \frac{\phi_1(p_x, p_y)}{2\pi} g_2 \quad (2.21)$$

In each pixel, the resultant X-ray angular refraction can be deduced by tangent (with small-angle approximation):

$$\alpha(p_x, p_y) \approx \tan \alpha(p_x, p_y) = \frac{S(p_x, p_y)}{d} = \frac{g_2 \phi_1(p_x, p_y)}{2\pi d} \quad (2.22)$$

Hence, the relation between X-ray angular refraction and differential phase-shift can be established⁵³:

$$\propto (p_x, p_y) = \frac{\lambda}{2\pi} \frac{\partial \Phi(p_x, p_y)}{\partial x} \quad (2.23)$$

Finally, the information of interest, i.e. the differential phase-shift of the X-rays after passing through the sample can be expressed as

$$\frac{\partial \Phi(p_x, p_y)}{\partial x} = \frac{2\pi}{\lambda} \alpha(p_x, p_y) = \frac{g_2}{\lambda} \frac{\phi_1(p_x, p_y)}{d} \quad (2.24)$$

A further step can be employed to determine the actual phase-shift of X-ray by taking an integration of $\frac{\partial \Phi}{\partial x}$ along the x-axis. As previously mentioned, one of the advantages of the phase-stepping procedure is that it can generate three information simultaneously. By means of one-dimensional Fourier analysis, considering just the 1st order term, the information of interest can be extracted from the measured phase-stepping signal (see Figure 6):

(a) Differential phase-shift:

$$\emptyset_1 = \emptyset_1^s - \emptyset_1^r; \quad (2.25)$$

(b) Dark-field (visibility):

$$V = \frac{v^s}{v^r} = \frac{a_0^r a_1^s}{a_0^s a_1^r} \quad (2.26)$$

(c) Absorption:

$$a_0 = \frac{a_0^s}{a_0^r} \quad (2.27)$$

Figure 6: Phase-stepping procedure retrieves DPC (Differential Phase Contrast), DF (Dark-field) and ABS (Absorption) information

2.2.5 Measurement of visibility contrast

In Dark-field imaging, the visibility contrast is formed through the mechanism of SAXS from microstructures with a scale much smaller than the spatial resolution of the imaging system. The visibility contrast is encoded in the intensity oscillation curves obtained via phase-stepping. A reference intensity oscillation I^r , is acquired without specimen in place. This is followed by a repeated scanning of the analyzer grating (G2) but now with specimen placed in front of the phase grating (G1) to generate a specimen intensity oscillation, I^s . A complete cycle of phase-stepping provides transmission and Dark-field images simultaneously. The normalized average transmission of the specimen in each detector pixel is given by^{16,29,49,53}:

$$T(p_x, p_y) = a_0^s(p_x, p_y)/a_0^r(p_x, p_y) \quad (2.28)$$

where a_0^r and a_0^s are the means of the reference and specimen intensity modulation respectively.

The Dark-field signal refers to the normalized oscillation amplitude, i.e. the normalized visibility of the intensity modulation^{16,29,49,53}:

$$V^r(p_x, p_y) = a_1^r(p_x, p_y)/a_0^r(p_x, p_y) \quad (2.29)$$

$$V^s(p_x, p_y) = a_1^s(p_x, p_y)/a_0^s(p_x, p_y) \quad (2.30)$$

where V^r and V^s are the normalized visibilities, and a_1^r and a_1^s are the amplitudes.

To provide a quantitative measure related to the SAXS and USAXS of the microstructures at micrometer and sub-micrometer length scale in the specimen, V^s is compared against V^r to give a relative visibility reduction as a normalized visibility:

$$V = V^s(p_x, p_y)/V^r(p_x, p_y) \quad (2.31)$$

For a homogeneous specimen, weak internal density fluctuation does not produce a detectable reduction in visibility, hence the value V remains almost equals to 1. However, when the specimen is porous and contains strongly fluctuating density such as inhomogeneous material and disruptions in the structural details, an obvious decrease of the intensity amplitude can be observed, giving value V of less than 1.

3. X-ray Dark-field imaging system instrumentation

This chapter details the system design and instrumentation of the proposed 2D X-ray Dark-field imaging system. The system consists of five major sub-systems (see Figure 7 and Figure 8), namely the X-ray source, X-ray detector, sample manipulator, grating and 2D phase reconstruction sub-systems. Each of these sub-systems independently plays important roles in the whole assembly.

In Section 3.1, we introduce the X-ray source used in the experiments and the necessary safety elements required to ensure safe operation of the system. The X-ray properties (photon energy range, spot size, beam divergence, tube voltage and current) are highlighted as they are closely related to the grating design and imaging results of low-density sample. The main reason for selecting such an X-ray source is also discussed.

In Section 3.2, the functionality and characteristic of the X-ray detector are outlined to provide a foundation towards understanding the image acquisition process. In addition, the properties of the X-ray detector are related to the motivation of using an absorption grating (G2) and the implementation of phase-stepping procedure.

In Section 3.3, we present the specifications of sample manipulator that we integrate into the system. The main purpose of using the sample manipulator is to allow angular tuning of the sample to ease adjustment of the sample to a desired angular position. However, in this research, we do not aim to perform X-ray Computed Tomography scan which captures X-ray projections at multiple angles.

In Section 3.4, we document the design of phase grating (G1) and absorption grating (G2). As explained in the previous section, the phase grating is used to form a periodic Talbot self-image at a specific distance downstream of the X-ray source, while the absorption grating (G2) serves to carry out the phase-stepping procedure. In this sub-system, the precision of gratings positioning and translation is taken into

consideration. Hence, precise and repeatable motorized stages and nano-positioners are used to offer sufficient degree of freedom for the grating alignment and positioning.

In Section 3.5, we discuss the “2D Dark-field image reconstruction sub-system” which is made up of two key functional elements, i.e. the image acquisition procedure (phase-stepping) and image analysis (Dark-field signal extraction). Finally, the actual experimental setup and imaging conditions are presented in Section 3.6.

Figure 7 Components of 2D X-ray Dark-field Imaging Technique

Figure 8 Schematic of XPCI system and its associated sub-systems

3.1 X-ray source sub-system

A polychromatic microfocus X-ray tube with an energy range of 10 keV to 100 keV has been chosen as the source of illumination. As reported in the literature, X-ray Dark-field imaging that works based on the Talbot interferometry requires a minimum transverse coherence length $l_c = L\lambda/w_x$, where L = source-to-phase grating distance, λ is the wavelength and w_x is the source size. The requirement on the longitudinal coherence length is less stringent. Working with a realistic polychromatic source, the fringe visibility and contrast reduces owing to the integrated Talbot effect of various wavelengths in the spectrum. However, as reported in most of the grating-based X-ray Dark-field imaging results, the system works well with polychromatic source and accepts a wide X-ray energy (E) bandpass tolerance. The focal spot measures 5 μm at an output power of 4 W with X-ray beam emission angle of 42 degree.

Working with a microfocus X-ray tube greatly reduces the number of X-ray photons that can penetrate through the sample and finally reach the detector because a microfocus tube produces lower tube voltage and current which determine the number of X-ray photons it can generate. This consequently decreases the probability of X-rays' interactions with the sample and the visibility of the phase-sensitive information that can be captured by the X-ray detector. However, increasing the speed of X-ray Dark-field imaging and data analysis is not the final goal of this research. Hence, optimizing the detector's exposure time may improve the situation by allowing acceptance of more X-ray photons in a longer integration timeframe.

Further, the motivation of choosing a microfocus spot size is that it can fulfil the spatial coherence mentioned above, which in principle will generate image of better quality. This is an added advantage as compared to other mode of grating-based XPCI systems which uses a larger spot size (typically tens of milli-meter) but needs a third grating (absorption) in front of the X-ray tube which acts as a "slicer" that "slices" the X-ray beam into multiple micro-meter beams, in order to achieve the required spatial coherence. Adding a third grating not only adds to the complication

of the system assembly, it also introduces more uncertainties in data analysis and increases the cost of system development.

3.2 X-ray detector sub-system

The X-ray detector is a CMOS photodiode array sensor placed in direct contact with the sensitive $\text{Gd}_2\text{O}_2\text{S}$ scintillator which converts the incident X-ray photons to light, which in turn is detected by the photodiodes. The sensor has 1024×1024 pixels, with pixel size of $48\mu\text{m}$, offering an active area of 49.2 mm^2 which determines the imaging field-of-view. With a detector pixel size of $48\mu\text{m}$, it is impossible to resolve the diffraction fringe pattern with period that is of several microns. Hence, a second grating (G2) is needed to enable fringe detection. The details will be discussed in the 2D phase reconstruction sub-system.

The detector is aligned to the centre of the X-ray emission and is mounted onto a precise stage so that it can be translated along the optical axis to find the optimum Talbot distance that yields the highest visibility of Talbot self-image. Once the position is fixed, the detector stays in the position throughout the entire course of operation. There is no need to change the position unless there is a change in the experiment parameters, such as sample type, X-ray energy and grating specifications. The image acquisition unit is conveniently connected to the detector via USB communication. The key detector properties (offset, gain and exposure time) are calibrated once before the experiments, or whenever necessary. This procedure is taken prior to experiments to ensure that the detector defects are accounted for and that they will not be reflected on the final X-ray image.

There exists X-ray camera with much smaller pixel size, typically in the range of tens or a few micro-meter. However, these products are usually expensive and their costs are three or four times higher than the X-ray detector mentioned above. In principle, a smaller pixel size is favourable in application that aims to achieve higher imaging resolution. However, working with larger pixel size like in this case is not entirely a bad thing because it allows collection of more X-ray photons which will

subsequently increase the detection sensitivity. The problem of insufficient resolution can be resolved by stepping a second grating (G2) across one detector pixel to enable the effect of sub-pixel detection which provides more information that will be explained in Section 4.4.

3.3 Sample Manipulator sub-system

The sample manipulator is made up of a linear translational stage and a rotation stage. The linear stage allows sample translation along the optical axis to offer various imaging magnification levels. It has a travel range of up to 500 mm, a resolution of 20 μm and bi-directional repeatability of 6 μm . The rotation stage is mounted onto the linear stage to provide angular adjustment of the sample which provides X-ray imaging of the object at different perspective. However, it is not meant to do a 360 degree scanning of Computed Tomography as this research's scope focuses only on 2D imaging methodology. The rotation stage has a 360° continuous rotation with minimum incremental motion of 2.19 arcsec. The repeatability measure is less than 1 arcmin.

The samples used in the experiments are small and light objects that are made of low-density polymeric material. Hence, mounting of sample onto the stages is very straightforward and the issue of overloading can be eliminated. Further, the sample remains stationary in all experiments, motion artefacts can be neglected. For sample with more complicated shape or geometry, special fixtures can be designed to hold the sample stable in place.

3.4 Grating sub-system

In this research, the X-ray Dark-field imaging system (Figure 9) employed a microfocus X-ray source. As the requirement on spatial coherence is fulfilled, the third grating usually placed directly in front of the X-ray source is no longer needed. Here, only two gratings are used in the setup. Hence, the grating sub-system contains primarily a phase grating (to generate Talbot effect), an absorption grating (to resolve the Talbot fringe pattern) and high precision motorized stages to enable careful alignment and positioning of the gratings.

In our Talbot interferometer, we used a cone-beam X-ray source. Due to the magnifying cone-beam geometry, the final fractional distance, z' , is re-adjusted according to the geometrical magnification factor^{54, 55}:

$$z' = \left(\frac{l}{l-z}\right)z \quad (3.1)$$

where l is the distance between the X-ray source and G1, and z is the Talbot distance before re-adjustment. In this case, the second grating (G2), which is a binary analyser grating with periodicity matched with the re-scaled fringe period, is placed at the plane where the Talbot fringes are formed. The positions of G1 and G2 are shown in Figure 9.

Figure 9 Schematic of gratings positions in the XPCI setup

The design specifications of phase grating, G1 (period 4.5 μ m, duty cycle 50%) are illustrated in Figure 10. The trench is made of Nickel and measures 7.1 μ m. This phase grating is designed to work with X-ray energy of 21 keV. The G1 induces a phase-shift of π rad to the X-rays that pass through the grooves of G1. As mentioned

in Section 3, a self-imaging effect will occur at the specific Talbot distances, which in this case, the self-image (periodical pattern) of phase grating can be observed at 0.045 m downstream of G1.

Figure 10 Phase grating, G1

Under a plane wave illumination, based on the calculation, the periodical pattern should have a periodicity of half the G1 period, i.e. 2.25 μm. However, due to the fact that the microfocus source is generating a diverging (cone) beam, X-rays with curved wavefront is propagating towards the phase grating, hence the resultant fringe at the observation plane has to be corrected to be 2.4 μm, by taking into consideration of the magnification factor created by the curved X-ray wavefront.

The absorption grating (G2) has a periodicity designed to match closely with the resultant Talbot fringe period, i.e. 2.4 μm. The design specifications are illustrated in Figure 11. In order to preserve good visibility of the fringe pattern, gold, a material with high X-ray absorption coefficient has been used to fill the trenches. The repeating bars made of Gold/polymer material combination generate sufficient contrast of the fringe pattern in the X-ray image.

Figure 11: Absorption grating, G2

Figure 12: (top) phase grating G1 (bottom) absorption grating G2

The system definitely performs optimally when the two gratings G1 and G2 are well fabricated according to specifications and well-aligned. Most critical are the translation and the rotation of gratings along the optical axis of G1 with respect to G2, which require an alignment precision in the order of 10 μm and 0.5 milli-degrees, respectively.

The phase grating (G1) is mounted onto a precision stage to allow movement along the z-axis and maintain a specific distance from the X-ray source. More importantly is the position of absorption grating (G2) which plays a critical role in the

phase-stepping procedure. G2 is placed as close as possible to the X-ray detector. It is mounted onto a precise PZT nano-positioner that enables G2 translation along the x -axis at a step size of $0.48\ \mu\text{m}$ (for a 5-step approach) or $0.24\ \mu\text{m}$ (for a 10-step approach).

In terms of angular alignment, each of the gratings G1 and G2 is mounted onto a goniometer to allow angular adjustment with respect to each other, and around the z -axis. The angular adjustment around the y -axis can be manually rotated whenever necessary.

3.5 Two-dimensional Phase Reconstruction sub-system

This sub-system consists of two important elements, namely the phase-stepping image acquisition unit with the necessary motorized stages to carry out the acquisition process, and a two-dimensional phase reconstruction software. The purpose of this sub-system is to extract phase-sensitive information from the distorted Talbot fringe pattern, as a result of X-ray wavefront that changed its shape after interacting with the sample placed in the X-ray propagation pathway.

The X-ray image formed by the X-ray detector is an intensity image that is composed of the effects of X-ray absorption, refraction or phase-shift and ultra-small-angle scattering occurred in the sample. The phase-stepping procedure serves to separate these information. As highlighted in Section 3.3, in a single experiment, the phase-stepping procedure can generate two new information, i.e. Differential Phase Contrast (DPC) corresponding to the refraction or phase-shift of X-rays, and Dark-field (DF) corresponding to the ultra-small-scattering, while preserving the conventional Absorption (ABS) information.

Phase-stepping procedure is carried out by laterally translating the G2 in the x -axis over a G2 period of $2.4\ \mu\text{m}$ while G1 remains stationary. In a five-step approach, G2 is displaced by a step-size of $0.48\ \mu\text{m}$. Whereas in a ten-step approach G2 is then displaced by a step size of $0.24\ \mu\text{m}$. For every step taken, an X-ray intensity

image is recorded by the X-ray detector. Upon completion of the scanning process, the image sequence is sent to the 2D phase reconstruction unit for phase-sensitive information extraction and analysis.

3.6 Experimental Setup and Imaging Conditions

In this thesis, the X-ray Dark-field imaging experimental platform operates based on Talbot grating interferometry is instrumented at the Singapore Institute of Manufacturing Technology, Singapore. Figure 13 shows the actual experimental setup which was developed based on the design specifications outlined in Chapter 4. All components shown in the photograph are mounted onto an optical bench which provides a stable platform in the shielded X-ray chamber. The experiments are carried out on a Hamamatsu microfocus X-ray tube (L10101) operated at X-ray energy range from 40kV-80kV with tube current of 100uA. The tube is composed of a conventional filament-based tungsten (W) tube with a minimum focus size of 5µm which satisfied the minimum spatial coherence requirement in a Talbot grating interferometer⁸.

The two gratings, namely phase grating (G1) and analyser grating (G2) were fabricated by Microworks, Germany, using techniques such as lithography, deep etching and electroplating of gold. The active area is 50 x 50 mm². The G1 groove is made of Nickel while G2 groove is made of polymer and filled with gold. Both gratings are supported by polymer substrate. The G1 and G2 have periods of $p_1=4.5\mu\text{m}$ and $p_2=2.4\mu\text{m}$ respectively. The design of gratings is optimized for a mean X-ray energy of 21 keV, with groove heights of 7.1µm and 20µm for G1 and G2 respectively. G1 is placed at 0.65m downstream of the source, and the specimen is placed as close as possible in front of G1. The distance between G1 and G2 corresponds to the 1st fractional Talbot distance. The images are recorded using a Radlcon area detector. The detection module consists of an array of 1024 x 1024 pixels, with a pixel size of 48 x 48 µm² and the resulting field-of-view is 50 x 50 mm².

Figure 13: Photograph of experimental setup in the X-ray laboratory showing the key hardware components and associated control units

A commonly available computer with 4 GB RAM, 32-bit OS and processing power of 2.40 GHz can meet the data processing requirement. The computer is connected to the X-ray source and an X-ray safety interlock mechanism which is important to ensure that the system is properly shielded before any operation and that no leakage of X-rays is allowed during the operation.

The computer controls the X-ray emissions, motorized stages and X-ray detector. The illumination control unit interfaces with the X-ray source to manipulate the X-ray tube voltage and current according to the needs of the experiment. The detector control unit enables the calibration and setting of X-ray detector parameters such as offset, gain and exposure time. The motion control unit interfaces with multiple motorized stages for various purposes, from sample position manipulation to gratings alignment and positioning, and importantly the phase-stepping process. The X-ray images recorded by the detector are then transmitted to the processing unit for 2D phase reconstruction.

The two gratings are positioned (see Figure 14) such that a slanted fringe pattern is observed in every individual X-ray image (Figure 15). In our experiment, we observed that there is no tight requirement on the relative angular deviation between the two gratings and the degree of tilting of the fringes. However, we ensured that these fringes are uniformly spaced across the detection area and the visibility of the fringes is sufficient to yield higher quality result in the data processing step that follows.

Figure 14 Schematic of X-ray Dark-field system

Figure 15 Fringes observed in the X-ray image when 2 gratings are in place

In the phase-stepping procedure, G2 is linearly stepped across a range of G2 period, i.e. $2.4\mu\text{m}$ with an incremental step of $0.24\mu\text{m}$. A series of 10 individual X-ray images is recorded at these discrete 10 positions, x_g . An integration of 2 images is adopted at each step to acquire a less noisy image. Hence, at each stepping position, every image is acquired at 5s exposure time and re-integrated twice, summing up to a total of 10s acquisition time per step.

The phase-stepping process takes place twice to generate intensity oscillation curves under the condition of with and without the specimen. The specimen intensity oscillation, $I^s(p_x, p_y, x_g)$, is compared against the reference intensity oscillation, $I^r(p_x, p_y, x_g)$, to allow us to extract key parameters and to compute mean ratio, visibility and normalized visibility for each image pixel. Transmission image is obtained by normalizing the mean value while the Dark-field image is obtained via normalizing visibility over mean value as explained in Chapter 2.

4. Evaluation of image and phase-stepping quality

In the proposed imaging system using a grating interferometer setup, the image quality, phase-stepping performance and fringe visibility play an important role in the final extraction of the Dark-field signal. Hence, experiments have to be conducted to evaluate the performance of the following aspects of the imaging process:

1. influence of image integration on the image quality and fringe visibility at a stationary position
2. intensity consistency and impact of integration number across phase-step positions

4.1 influence of image integration on image quality and fringe visibility at a stationary position

Having a good quality image is of high priority in the Dark-field imaging because the information extraction depends strongly on the amplitude and mean of the intensity oscillation curves obtained via the phase-stepping procedure. If the image is heavily corrupted by noises, the formation of a clear and accurate Dark-field image will be hindered.

In this experiment with the two gratings in position, a total of 30 images, with each looks like Figure 16, are acquired while the analyser grating remains stationary at the home position. First of all, a big ROI, i.e. ROI_A which contains 200x200 pixels is cropped. Then, two small ROIs, namely ROI_B and ROI_C, each with 3x3 pixels, are prepared to represent the background (blue) and fringe (yellow) regions respectively as shown in Figure 16.

Figure 16 ROI and pixel selection for evaluation of intensity consistency

A statistical evaluation of these images is quantified in terms of the trend of standard deviation and fringe visibility which are measured by contrast-to-noise ratio, CNR, which is described as

$$CNR = \frac{|\bar{S}_{fringe} - \bar{S}_{background}|}{(\sigma_{fringe}^2 + \sigma_{background}^2)^{1/2}} \quad (4.1)$$

where $\bar{S}$ is the mean intensity and σ^2 is the variance of the ROI.

The influence of image integration is also taken into account. This is done by continuously exposing the gratings for an extended period of time at a stationary position. For instance, an integration of two images leads to an acquisition duration of 10s which is twice of that needed by a single image (5s). In this context, “Int 2” refers to integration of two images, “Int 4” refers to an integration of four images and so forth.

From the analysis of the mean intensity of the selected big ROI, we observed that the higher the number of image integration, the lower is the standard deviation of the intensity, see Figure 17.

Figure 17 Influence of integration number on intensity of ROI_A

From the figure, we observed that without any image integration, a single-shot image shows the highest standard deviation, relating to highly fluctuating intensity values within the selected ROI. On the other end, with an integration of 10 images, the standard deviation value decreases by more than 5%. This shows that the images acquired with a higher integration number is more reliable and it is evident by a decreasing standard deviation with a higher integration number, see Figure 17.

Furthermore, we assessed the fringe visibility across different number of integration as a measure of the contrast-to-noise ratio. We calculated the CNR of ROI_A in each of the acquired 30 images and plotted the values against the image number from 1 to 30, see Figure 18. Firstly, we noticed that the fringe visibility fluctuates in a certain range which forms the basis of intensity variation that could be a consequent of inconsistent X-ray photons emission, electronic noise and mechanical jitter. We discerned a reduction of fluctuation amplitude as the number of integration grows. If we take the pattern of Int 10 (i.e. integration number is 10) as a reference, the fluctuation contributions from each of the integration number becomes obvious.

Secondly, in Figure 18, we obtained a decreasing magnitude of CNR while the mean value of CNR remains quite consistent regardless of the integration number used. The data explained that if without any image integration, we would be facing a largely varying fringe visibility that has CNR values ranging between 0.80 and 2.56. As we increase the integration number to 2 and 4, the fluctuation range has reduced by approximately 40%. When we use higher integration number, from 6 to 10, the fluctuation range diminishes by more than 60%, giving a more stable and consistent fringe visibility across all the 30 images we captured.

Figure 18 (a) Fringe visibility of ROI_A across 30 images, (b) Influence of integration number on fringe visibility

From the above data, apparently, reliable images are obtained at the expense of image acquisition time. A higher integration number corresponds to a longer overall imaging process. Without any image integration, the imaging process can be completed in less than 2 minutes. Although imaging speed is not of the primary concern in this thesis, we mainly used an integration number of 2 or 4 to keep the imaging time within a reasonable duration. However, if a very high quality assessment is required, an integration number of 6 to 10 is favourable in order to eliminate image artifacts due to intensity inconsistency.

The same experiment procedure is adopted to evaluate the intensity consistency at the pixel level. Two pixels are selected, one from the background (Pixel_1) and one from the fringe (Pixel_2). The intensity of the pixels are measured and plotted against the image number. Figure 19 shows the intensity pattern for Pixel_1 and Pixel_2 across the 30 images. Taking Int 10 as a reference, the intensity variation of different integration number can then be compared.

From the charts, we observed the intensity fluctuation at the pixel level falls under the acceptable range in our Dark-field imaging. Furthermore, we clearly noticed that as the integration number increases, the intensity variation decreases, consequently giving more reliable image content. We also learned that using zero integration will likely generate images with stronger background noise. Hence, this evaluation results provide a basis to select a suitable integration number based on the application requirement on image quality and image acquisition speed.

Figure 19 Intensity consistency of (a) background at pixel_1, (b) fringe at pixel_2

4.2 intensity consistency and impact of integration number across phase-step positions

In the previous experiment, we investigated the influence of image integration on fringe visibility at a stationary position. We also know that the fringe visibility measured in terms of CNR is closely determined by the intensity variance. Hence, a detailed evaluation on the intensity consistency becomes meaningful. As phase-stepping is an important procedure in Dark-field imaging, several experiments have to be conducted to evaluate the performance of phase-stepping in terms of intensity consistency and fringe visibility at various step-positions of the analyser grating, and lastly to assess the performance of the translation of the analyser grating.

In this experiment, the analyser grating is translated over the grating period of $2.4\text{ }\mu\text{m}$ at an incremental step size of $0.24\text{ }\mu\text{m}$ to generate intensity oscillations without sample mounted in the pathway of X-ray propagation. The experiment used an integration of 4 images at every step-position and is repeated three times. The mean intensity of ROI_B and ROI_C at various step-positions are plotted to create the intensity oscillation of background (Figure 20a) and fringe (Figure 20b).

The detectability of the oscillation amplitude is crucial in our Dark-field imaging which is strongly dependent on the visibility contrast, a measure of curve amplitude against the mean value. We observed that in Figure 20, the phase-stepping procedure has created reasonable sinusoidal curves with amplitude measured at approximately 5 units of image grey levels which can be clearly detected in digital image analysis. As we compared all data obtained from the repeated cycles, the curves are closely aligned with a variation remains below 3 units of grey level at each phase-step position.

Figure 20 Mean intensity oscillation obtained from phase-stepping:
(a) for background at ROI_C, (b) for fringe at ROI_B

In our actual imaging experiments, the extraction of Dark-field signal requires that every pixel in the phase-stepped images is analysed. In order to obtain a meaningful assessment of our imaging performance, the intensity oscillations for the background and fringe are analysed at the individual pixel level. For this purpose, two experiments are conducted separately with the phase-stepping procedure repeated 5 times. In the first experiment, an integration of 2 images is used. From the image, two pixels are selected, one from the background (pixel_1) and another one from the fringe region (pixel_2). The intensity of the selected pixel is plotted across the image sequence as shown in Figure 21a (background) and Figure 21b (fringe).

Figure 21 Five cycles of intensity oscillations obtained from phase-stepping with Int 2: from (a) background pixel, (b) fringe pixel

In the second experiment, the procedure remains the same except that an integration of 4 images is used this time. The intensity of the selected pixel is plotted across the image sequence as shown in Figure 22a (background) and Figure 22b (fringe).

Figure 22 Five cycles of intensity oscillations obtained from phase-stepping with Int 4: from (a) background pixel, (b) fringe pixel

This experiment allows us to understand the behaviour of the analyser grating movement by analysing the characteristic of the intensity oscillation. As we repeated the phase-stepping procedure 5 times, we obtained 5 sinusoidal curves that are aligned quite closely even at the pixel level. By comparing Figure 21 and Figure 22, we noticed a higher correlation in the experiment using an integration number of 4 than that with 2 because a higher integration number produces images with less intensity fluctuation and hence generates a better image quality.

From the above evaluation outcome, we gather that image integration indeed influences the reliability of the image content. The result shows that fringe visibility improves as the number of integration increases. In addition, the intensity oscillation curves at the background and fringe are reasonably sinusoidal and they provide easily detectable amplitude which contributes to obvious visibility contrast in the Dark-field image.

Furthermore, the oscillation curves obtained from the repeating cycles of phase-stepping agreed quite well with each other. This shows that the performance of grating movement falls within the acceptable tolerance for the Dark-field imaging, which consequently ensures that image artifacts due to misalignment of the oscillation curves obtained with and without sample are kept to the lowest possible.

The assessment results have provided with a guideline to configure our imaging parameters. In this thesis, we seek to achieve a balance between imaging duration and image quality. In the next chapter, we will show that using an integration number of 2 or 4 not only save us the image acquisition time, we are also able to obtain an enhanced image contrast even using a low-brilliance X-ray microfocus tube.

5. Applications and Imaging Results

5.1 Applications and Sample Description

We have identified two major limitations of the conventional X-ray imaging:

1. Poor X-ray image contrast in the imaging of object composed of light materials (low atomic number elements)
2. Unable to achieve equal X-ray visibility of internal features of an object with large-varying thickness

Light material in this context is defined as a material that is mainly composed of low atomic number elements such as Carbon (C), Hydrogen (H), Oxygen (O) and Nitrogen (N). These elements are commonly found in the air and biological tissues of human, animals and plants. Inherently, these elements and their compositions do not strongly absorb X-ray radiation because they have very low mass attenuation coefficient which makes them contribute very little photoelectric effect in the typical X-ray energies range between 10 and 100 keV. For instance, in Figure 23, we notice that the light material (adipose and skeletal muscle) exhibit X-ray absorption capability that is three order lower than heavy material (iodine). By using the conventional absorption-based X-rays imaging method, we are not getting satisfactory contrast in the X-ray image to allow us to efficiently separate these light elements.

Figure 23 X-ray absorption capability of high and low density material.
Extracted from "<http://www.xrayphysics.com/attenuation.html>"

From the Lambert-Beer's Law, we understand that X-ray absorption is proportional to the object thickness. This means that the object thickness will decide the X-ray energy and exposure time required to enable sufficient X-ray penetration through the object and allow photoelectric events to happen. However, in real life many objects do not have regular shape and thickness. Therefore, we need to have different range of X-ray energy and exposure time to accommodate the variances in order to achieve satisfactory image contrast at different regions of the object. This often leads to the need of taking multiple shots of X-ray images of the same object and then combines the information by various image processing methods. However, this approach falls short when the object has a large gradient of thickness. A continuous adjustment of the X-ray parameters is required to optimize the X-ray absorption as X-ray scans through various parts of the object.

In this thesis, onion (Figure 24) is used as one of the key test objects for our imaging objective as it serves as a representation of a large gradient of thicknesses across a matrix of light material. The onion in a form of irregular shape has largely varying thickness from its growing tip to its body (bulb) and to the roots. The external very thin layers of tunic (outer skin) are not removed. Inside the onion, there exist layers of scale leaves that can be interpreted as interfaces between the tissues, where a disjoint of material and structure can be observed.

Figure 24 Picture of samples (left) Onion (right) Asparagus

Besides, asparagus is also used as a test object as it is made of a matrix of light elements and has an interesting structure at its crown. Inside the body of the asparagus are a large number of tubular structures that are responsible for nutrients transportation.

Using the conventional absorption-based X-ray imaging, these features cannot be easily captured without more effort. At the onion tip, the structure is formed by very thin layers of tunic which require very low X-ray energies to allow for sufficient absorption to generate clear visibility in the X-ray image. In contrast, the body (bulb) of the onion is composed of much thicker form of tissues, which according to the Lambert-Beer's law, needing a lot higher X-ray energy to ensure enough penetration and sufficient absorption to take place along the X-ray propagation path. Furthermore, majority parts of these light materials do have almost similar mass attenuation coefficients, and they are weak in X-ray absorption and not able to generate satisfactory absorption-contrast to be visualized in the conventional X-ray image.

In this section, we aim to show that even using a low-brilliance X-ray tube, the X-ray Dark-field imaging method can address the aforementioned challenges and complement the existing X-ray imaging by providing an enhanced image contrast and additional information at sub-pixel lengthscale. In our experiments, we first performed a 2D imaging of the test object under the conventional X-ray imaging using the same microfocus source and detector but without gratings in place. Subsequently, image processing techniques are applied to improve the quality and contrast of the conventional absorption image. Next, G1 and G2 are integrated into the system, and phase-stepping is carried out to obtain the normalized transmission and visibility (Dark-field) images simultaneously. Finally, the calculated Dark-field image is compared against the improved version of the absorption image and the intensity oscillations are interpreted to quantitatively measure the visibility reduction caused by the specimen.

5.2 Visibility enhancement approach

5.2.1 Sub-pixel fringe scanning procedure

Figure 25 Illustration of a 8-step sub-pixel fringe shifting procedure

Figure 25 illustrates the collection of data points as the fringe formed by G1 and G2 is translated across the length of G2 period. In the picture, the little red box represents a pixel of the detector, denoted as pixel A. Firstly, without a sample in the X-ray propagation pathway, during the sub-pixel stepping of G2, the fringe will shift its position accordingly. Hence, pixel A will experience a fluctuation in the grey value over time as shown a reference intensity fluctuation in the plot of the intensity of pixel A against the stepping position of G2. The process repeats for the case when the sample is positioned closely (typically a few millimeters) in front of G1 to produce a sample intensity fluctuation.

Specifically in our case, we adopted a 10-step procedure with an increment of sub-pixel step size (i.e. 0.24 μ m). This gave us two datasets, i.e. a collection of 10 data points for the reference intensity fluctuation and another set of 10 data points for the sample intensity fluctuation. In particular, we are interested in Darkfield imaging, hence, two important parameters are extracted from the datasets, i.e. amplitude and mean values from each dataset.

Taking an example from the imaging of Onion, one pixel has been selected and the changes in intensity of that pixel of interest has been evaluated (Figure 26).

Figure 26 A pair of reference and sample intensity curves for a given pixel of interest

Every pixel has two sets of data after a complete cycle of fringe shifting:

- (a) Reference intensity curve, $\{I_1^r, I_2^r, \dots, I_N^r\}$
- (b) Sample intensity curve, $\{I_1^s, I_2^s, \dots, I_N^s\}$

where N is the total number of steps in the fringe shifting process.

The intensity of pixel (x, y) is described as:

$$I_A(x, y) = A_0(x, y) \frac{a_0^r(x, y) (I_{max}^s(x, y) - a_0^s(x, y))}{a_0^s(x, y) (I_{max}^r(x, y) - a_0^r(x, y))} = A_0(x, y) V(x, y) \quad (5.1)$$

where the mean values are noted as $a_0^r(x, y) = \frac{1}{N} \sum_{i=0}^{i=N} I_i^r(x, y)$ and $a_0^s(x, y) = \frac{1}{N} \sum_{i=0}^{i=N} I_i^s(x, y)$ respectively, and $A_0(x, y)$ depends on the visibility enhancement methods used, which are explained in the following sub-sections.

5.2.2 Visibility transformation method

The main purpose of visibility enhancement (Figure 27) is to further improve the visibility of fine details in the Darkfield image. We propose global and local transformation to the visibility value, $V(x,y)$, calculated from the reference and sample intensity curves. We explored three categories of transformation functions, which can be achieved through a global constant, linear function or nonlinear function as depicted in Figure 28.

Figure 27 Visibility transformation is applied to further improve the original Darkfield image

Figure 28 Classification of various visibility transformation functions

5.2.3 Global visibility enhancement

Global transformation is the most straightforward approach to enhance the overall image visibility. It aims to scale up the contrast of an image by a constant factor, such as the ratio of the global mean values.

5.2.3.1 Ratio of global mean values

In this method, A_0 is a global constant which is defined as:

$$A_0 = \frac{1}{2}(I_{gmax}^r - I_{gmin}^r) \quad (5.2)$$

where I_{gmax}^r and I_{gmin}^r are the global maximum and global minimum values of all the reference curves in the Darkfield image, respectively. When A_0 is a constant, the visibility value, $V(x, y)$, in every pixel will be multiplied by the constant A_0 to form the final Darkfield image intensity:

$$I_A(x, y) = A_0(x, y)V(x, y) \quad (5.3)$$

The global approach worked well in our Darkfield images and it successfully produce final image with good contrast that make fine details become easily detectable. We also realized that the global approach is not sensitive to the intensity variations between detector pixels. Hence, the localized approaches were also investigated on the same set of images to allow us to be better informed about the performance of the two methods.

5.2.4 Local visibility enhancement

We understand that almost every pixel may have different values in their reference and sample intensity curves. The localized approaches are taken because they consider the variations of the intensity curves in every pixel and adapt to such

changes across the pixels. In this case, the value A_0 is determined by the intensity fluctuation of the pixel of interest.

The local approach to visibility enhancement is classified into two categories, namely linear and nonlinear transformations. The linear functions are defined by Max-min linear transformation, percentage transformation, and piece-wise transformation. The nonlinear functions that we investigated are logarithmic transformation and exponential transformation. The aforementioned methods are explained in the subsequent paragraphs and applied to the same set of Darkfield images for comparison as detailed in the next section.

5.2.4.1 Max-min linear transformation

The calculated visibility, $V(x, y)$, to form the Darkfield image usually has a very small value (typically a positive number of below 10 in our case). Hence, in the Max-min linear transformation, $A_0(x, y)$ serves as a linear scaling function that aims to lift up the visibility in individual pixel to a level that the embedded contrast can be easily visible in the final Darkfield image. In this case, A_0 is given by:

$$A_0(x, y) = b(I_{max}^r(x, y) - I_{min}^r(x, y)) \quad (5.4)$$

where b is a scaling factor which is a positive integer that depends on the bit depth of the image. In our experiments, the value of b that typically lies between 3 and 6 produced satisfactory imaging contrast for our specimen of interest.

5.2.4.2 Percentage transformation

In the percentage transformation, $A_0(x, y)$ serves the same purpose as in the Max-min linear transformation but now in a different way:

$$A_0(x, y) = b(I_{max}^r(x, y)) \quad (5.5)$$

where the value b can be any positive number from the range of $[0.5, 1.0]$. Sometimes, a lower value, such as 0.6 or 0.7 is preferred for a noisy Darkfield image. Otherwise, b can be set as 1.0 to optimize the image contrast.

5.2.4.3 Piece-wise transformation

We recall that the visibility-enhanced intensity, $I_A(x, y)$ of pixel (x, y) in the final Darkfield image is defined as:

$$I_A(x, y) = A_0(x, y)V(x, y) \quad (5.6)$$

where $A_0(x, y)$ is the visibility transformation function, and $A_0(x, y)$ is the calculated visibility value to produce the Darkfield image from the pair of reference and sample intensity curves. Here, using the piece-wise transformation, I_A is expressed as a function of V for a given image pixel:

$$I_A(x, y) = \begin{cases} 0, & V(x, y) < 0.5 \\ V(x, y) * I_{max}^r(x, y), & 0.5 \leq V(x, y) < 3 \\ 5 * V(x, y) * I_{max}^r(x, y), & V(x, y) \geq 3 \end{cases} \quad (5.7)$$

This multi-level function allows us to adaptively adjust the image contrast based on the visibility calculated at different regions of the image. As a result, the high visibility regions will be further enhanced while the low visibility regions are suppressed.

5.2.4.4 Logarithmic transformation

Besides the linear transformation functions mentioned in the previous sections, two nonlinear transformations (logarithmic and exponential) have been explored as well. In the logarithmic transformation, the intensity I_A is described as:

$$I_A(x, y) = b \log(V(x, y) + 1) \quad (5.8)$$

where

$$b = b_0 I_{max}^r(x, y), \text{ and } b_0 \in [0.5, 1.0]$$

The logarithmic function is adopted to enhance the high visibility region while suppressing the low visibility region. The value b_0 is chosen depending on the image condition. A value that ranges between 0.5 and 1.0 is suggested as these values work well in our experiment.

5.2.4.5 Exponential transformation

Using the exponential transformation, the intensity is given by:

$$I_A(x, y) = b e^{V(x, y) - 1} \quad (5.9)$$

where

$$b = b_0 I_{max}^r(x, y), \text{ and } b_0 \in [0.5, 1.0]$$

The exponential function can be seen as a complementary feature to the logarithmic transformation. Both methods serve to improve the visibility nonlinearly so that necessary attention will be given to the specific regions to achieve the desired contrast enhancement.

The adoption of global and local transformations in our experiments is detailed in the next section. Two specimens were used, namely onion (low density biological tissues with non-uniform thickness) and pen (assembly of plastic parts).

5.3 Comparison of visibility enhancement methods

This section focuses on comparing the performance of the aforementioned global and local visibility enhancement methods on improving the contrast of Darkfield image. Two specimens have been investigated, i.e. onion and pen.

Figure 29 Visibility enhancement of Darkfield image of onion (as low density component with non-uniform thickness). Global approach using (a) ratio of global mean values. Local linear transform using (b) Max-min, (c) Percentage, (d) Piece-wise functions, and local nonlinear transform using (e) Logarithmic, and (f) Exponential functions

From Figure 29, we can see the impact of various transformation functions on the visibility of fine structures in the onion. All methods appear to be able to highlight important features such as skin layers and internal scale leaves. However, in terms of image quality, the local approaches seem to be too sensitive to changes and that result in generating undesired noises in the image. Firstly, the top left corner of Figure 29(b) is affected by the defect of gratings, secondly, Figure 29(c-e) show a white vertical line that comes from the defective detector pixels, and lastly Figure 29(f) is heavily corrupted by noise. Hence, in this experiment, we find that the global approach has outperformed all other localized approach. Not only it is simple and easy to implement, it also successfully preserves most information and provides satisfactory contrast in the Darkfield image.

Figure 30 Visibility enhancement of Darkfield image of pen (as an assembly of plastic parts). Closed-up image based on Global approach using (a) ratio of global mean values, Local linear transform using (b) Max-min, (c) Percentage, (d) Piece-wise functions, and local nonlinear transform using (e) Logarithmic, and (f) Exponential functions

Figure 30 shows the outcome of visibility enhancement on the Darkfield image of pen as a representation of an assembly of plastic components. We are interested to observe the edge sharpening effect and identify sub-surface crack.

The global approach using the ratio of global mean values as a scaling factor (Figure 30(a)) has provided clear edges and internal crack in the visibility-enhanced image. Similarly, the local approaches (Figure 30(b-e)) also enhanced the external and internal edges, at the same time increased the visibility of internal crack. However, the exponential transformation (Figure 30(f)) does not produce useful result and corrupts the image with noise.

In this experiment, it appears that both global and local approaches (except exponential transform) produced satisfactory outcome. But if we were to do a comparison in terms of image quality, the logarithmic transformation (Figure 30(e)) seems to outperform all other methods because not only it achieves improved image contrast, this method also generates the least noisy image.

5.4 X-ray Dark-field imaging results and discussion

In the following sections, all experiments are conducted using the grating-based X-ray Dark-field imaging system explained in Chapter 4. We conducted a total of 7 experiments with a summary of key features given in Table 2.

Table 2 A summary of key features in each experiment

Experiment	Specimen	Tube Voltage	Exposure Time	Integration Number	Key Feature to evaluate
Evaluation: Internal structures of biological samples					
1	Onion	60kV	5s	4	Short exposure
2	Onion	40kV	20s	2	Long exposure, Low integration
3	Onion pair	80kV	5s	2	High penetration
4	Asparagus body	40kV	5s	4	Low penetration, Short exposure
5	Asparagus crown	40kV	5s	4	
Evaluation: Internal defect of manufactured part					
6	Cracks in glass	40kV	5s	4	Short exposure, Sub-surface crack detection
Evaluation: (U)SAXS power of different material					
7	Seeds, grains, cotton	40kV	5s	4	Discrimination power based on (U)SAXS for material characterization

- **Experiment 1:**

We investigated the imaging outcome of onion using low exposure time of 5s. The tube current is set at 60kV to allow a decent penetration though the onion of varying thickness. The selection of an integration number of 4 is guided by the evaluation results detailed in Chapter 5.

- **Experiment 2:**

We increased the exposure time to 20s and compare the imaging results with that of Experiment 1. Both tube voltage and integration number are

decreased to a setting lower than recommended to allow us to observe the impact of exposure time in this application.

- **Experiment 3:**

We evaluated and compared the performance of absorption-contrast and Dark-field imaging under higher X-ray energy which allows higher penetration depth.

- **Experiment 4 & 5:**

Asparagus is used as a test sample because it offers interesting features at the internal and external surfaces. The asparagus crown and its body consist of fibrous structures at the inside and outside. In this experiment, we wanted to find out whether our imaging method gives an advantage in picking up fine structures in different parts of the asparagus of different thicknesses.

- **Experiment 6:**

It is common to have internal defects in the manufactured parts that are hidden beneath an opaque surface, which is not observable either by human eyes or under the optical microscope. In this experiment, we prepared a specimen to simulate sub-surface cracks by shielding the defective glass with opaque covers. The imaging results can then be verified with the actual image of the glass when the opaque covers are removed.

- **Experiment 7:**

X-ray Dark-field imaging is not only useful for surface and sub-surface visualization, it also has potentials in material characterization based on the (U)SAXS power of the specimen's material composition and geometrical details. This experiment serves to evaluate the discrimination power of our setup in the cases of various specimen characteristics, such as soft cotton, seeds and grains.

5.4.1 Experiment 1: Imaging of onion (low exposure time)

In this experiment, the X-ray source is operated at the 60kV tube voltage and 100 μ A tube current. The exposure time is 5s per image acquisition and 4 images are integrated to generate a less noisy image. Figure 31a shows the standard X-ray absorption-contrast image of the specimen imaged under the same X-ray settings but is acquired in a separate experiment without having the gratings positioned in the X-ray pathways. The exposure time is 5s and no integration is carried out.

Figure 31 shows the two typical problems with the imaging of irregular-shaped object using the absorption-contrast method. First of all, the onion tissues are made of light materials which inherently do not absorb X-ray radiation as strong as other dense material. Secondly, while we wanted to keep the details of the thin structure at the onion tip, we lost the details at the thick and bulky part of the object. By increasing the X-ray energy in order to allow more penetration through the onion body, we lost the image contrast at the onion tip because absorption cross-section decreases as the object thickness is reduced. On the other hand, as we decreased the X-ray energy to extremely low hoping to increase the contrast at the tip, we lost the contrast at the body and only observed a huge cluster of dark pixels. Using image processing methods to enhance the contrast (Figure 31b) we observed an improved visibility of the onion as a whole but not the visibility of the tunic and internal scale leaves. Figure 31c is the transmission image obtained from the grating interferometer, which shows similar problems as in the absorption-contrast image.

In contrast, the Dark-field imaging demonstrates a stronger discriminatory power for the detection of both external and internal features across various parts of the onion than that of the standard absorption-based images. From Figure 31d, we observed higher visibility of the scale leaves embedded in the thick region of the onion.

Figure 31 X-ray imaging of onion without grating (a) Absorption-contrast, (b) Enhanced absorption-contrast; with gratings (c) Transmission, (d) Dark-field; enlarged view of onion tip: (e) absorption-contrast, (f) Dark-field; enlarged view of onion body: (g) absorption-contrast, (h) Dark-field

Figure 32 Intensity oscillations at pixel (282, 576)

From the phase-stepping, we obtained the sample and reference intensity oscillation curves. Figure 32 describes the pair of intensity oscillations from pixel (282, 576). In a single experiment, we measured the mean and amplitude values for every pixel to construct the transmission and Dark-field images, respectively.

From the data, we observed that the mean value of the sample curve has reduced by approximately 5.2%, and the ratio of amplitude, i.e. a_1^r/a_1^s is approximately 7.33. By putting together the mean value of every pixel, we obtained the transmission image which is equivalent to the standard absorption-contrast image.

Besides, we also noticed a significant reduction of amplitude where (U)SAXS effect occurred. The visibility values V can be obtained from the same oscillation curves. Then, we arrived at the values $V^r = 0.0763$ and $V^s = 0.0109$, thus giving the resultant visibility of $V = 0.1428$, or equivalently 14.28%. By normalizing the visibility (amplitude) signal over the transmission (mean) signal, we obtained the Dark-field image.

Regions of the specimens with strong scattering effect, such as the interfaces and edges inside and outside of the specimen, produce strong visibility-contrast in the Dark-field image. This experiment shows that the scale leaves in the body of the onion consist of strongly scattered microstructures compared to its surrounding tissue matrix. In addition, external features such as the onion skin also produces strong Dark-field signal. We observed that the local small- and ultra-small angle scattering (SAXS and USAXS) power of the features can be detected across a large gradient of specimen thickness, which has not been possible using the conventional absorption-contrast imaging.

5.4.2 Experiment 2: Imaging of onion (long exposure time)

The onion is again investigated using the same platform but now under a lower X-ray energy (40kV/100 μ A) and much longer exposure time (20s per image) with integration over 2 images. Through phase-stepping process, the transmission image is calculated as shown in Figure 33a and the Dark-field image as in Figure 33c. Under a longer exposure time of 20s, internal features have become more prominent even in the transmission image. Using image processing techniques such as contrast enhancement and normalization, an improved version of the transmission image (Figure 33b) is obtained, and as expected, more features become visible in the thick region of the body. However, this comes at the expense of losing thin-layer structural details at the tip.

In comparison, the calculated Dark-field image (Figure 33c) obtained from this experiment keeps information at both the thin and thick regions. The experiment shows that not only Dark-field imaging retained the thin-layer structure in the image, it also revealed more internal details at the thick part at the onion body (see region-of-interest highlighted in yellow). As observed in the close-up view (Figure 33d) of the region-of-interest, multiple thin layers of scale leaves now appear in the image which were not observed at all in the equivalent transmission image (Figure 33a) and its enhanced version (Figure 33b).

Figure 33 X-ray imaging of Onion with gratings at 40kV/20s (a) Transmission, (b) Enhanced transmission, (c) Dark-field, (d) enlarged view of region-of-interest in Dark-field image

Figure 34 Intensity oscillations of pixel (401, 599)

The obvious visibility enhancement (Figure 34) in the Dark-field image is quantified by measuring the V values from the intensity oscillation curves. Taking pixel (401, 599) for analysis, we first observed a mean reduction by approximately 5.52%, and the ratio of amplitude, i.e. a_1^r/a_1^s is approximately 3.67. In terms of visibility measure, we obtain values $V^r = 0.0674$ and $V^s = 0.0194$, thus giving the resultant visibility of $V = 0.2886$, or 28.86%.

As compared to Experiment 1 conducted under a much shorter exposure time of 5s, an increment of the exposure time (having higher flux) did increase the visibility-contrast due to higher occurrence of (U)SAXS effect induced by the microstructures. This happens as more X-ray photon flux is introduced into the sample, there is a higher probability of microstructures interacting with the photons, resulting in a stronger re-distribution of the intensity or broadening effect in the intensity pattern. In Figure 34, we observed a strong reduction of oscillation amplitude and a “spreading” effect in the intensity pattern.

5.4.3 Experiment 3: Imaging of a Onion pair (high penetration)

A pair of Onions is tested using the same platform under a higher X-ray energy (80kV/100 μ A) to allow higher penetration. But the exposure time is reduced to 5s per image and the integration number is also kept at 2. Figure 35a shows an enhanced version of the original absorption-contrast image. Contrast enhancement techniques using image processing are applied in attempt to increase the visibility of inner features at the thick region of the onion body. Similarly, we are not able to discern all features at all parts of the onions simultaneously. In comparison, the Dark-field image as shown in Figure 35b have allowed much higher visibility of features across the large varying object thickness, giving a more complete representation of the test specimen.

The enlarged views of different parts of the onions are available from Figure 36, Figure 37 and Figure 38 to allow more detailed evaluation of the two imaging methods. We observed that Dark-field imaging is not a substitute of the absorption-contrast imaging but rather a complementary method that can provide additional information based on an alternative contrast, i.e. visibility-contrast. The strong contrast area noticed in the Dark-field image represent regions in the specimen that contain strong micro-scatterers. For regions having higher difference in material density but low number of micro-scatters, the absorption-based imaging system will generate contrast not visible in the Dark-field image.

a

b

Figure 35 X-ray imaging of Onion pair at 80kV/5s/Int2
without grating: (a) Enhanced absorption-contrast;
with gratings: (b) Dark-field

Figure 36 A closed-up view of region A of onion
without grating: (a) enhanced absorption-contrast;
with gratings: (b) Dark-field

Figure 37 A closed-up view of region B of onion
without grating: (a) enhanced absorption-contrast;
with gratings: (b) Dark-field

Figure 38 A closed-up view of region C of onion
without grating: (a) enhanced absorption-contrast;
with grating: (b) Dark-field

5.4.4 Experiment 4: Imaging of Asparagus Body

The stem of asparagus is halved and examined under the imaging setup to investigate the fibrous tubular vascular tissues in the stem, such as xylem and phloem. The imaging system operates at X-ray energy of 40kV/100 μ A, short exposure time of 5s per image with integration over 4 images. The generated transmission image is shown in Figure 39a. The image has been enhanced using image processing method such as normalization in order to improve the contrast along the edges of the tubular structures. However, the features of interest appear as a bundle of connected vertical lines. At regions where X-ray absorption is stronger, dark pixels fill up those areas and form blobs as seen in Figure 40a and Figure 40c. Hence, features residing in the thick parts of the asparagus can hardly be distinguished and potentially results in a misinterpretation of the test sample.

In comparison, the Dark-field image (Figure 39b) elevates the contrast and highlight features that can produce strong Dark-field signal which then make themselves distinct from the surrounding matrix. In the image, tubular structures become more identifiable. In Figure 40b, we see multiple vertical lines across the asparagus body, however, these lines have appeared disconnected in the absorption-images. This experiment shows that without Dark-field imaging, we might have thought that the lines are merged to form another structure of higher density (dark blobs).

In the analysis of the magnified Dark-field images (Figure 40b), we noticed obvious influence by noise. An attempt to reduce the noise using image processing methods such as median filter, de-speckle and outlier removal are applied to remove the artefacts (Figure 40d) but the results are not very satisfactory. Another possible solution is to increase the image integration number from 2 to 6 or higher as a means to minimize the pixel intensity fluctuation and ease the extraction of Dark-field signal.

Figure 39 X-ray imaging of halved asparagus at 40kV/5s/Int2:
with gratings (a) transmission, (b) Dark-field

Figure 40 region-of-interest A of asparagus body: (a) enhanced transmission, (b) Dark-field;
region-of-interest B: (c) enhanced transmission, (d) enhanced Dark-field

5.4.5 Experiment 5: Imaging of Asparagus Crown

In this experiment, the crown of asparagus is being examined under the same imaging setup to compare the visibility of leaves under conventional absorption and Dark-field imaging. Similarly, we used X-ray energy of 40kV/100 μ A, short exposure time of 5s per image with integration over 4 images. The transmission image generated (Figure 41a) is comparable to the conventional absorption contrast image. Using image processing, the contrast enhancement effect (Figure 41b) has sharpened the edges of some features, but most of the details at the thicker parts of the specimen have been “obstructed” due to high absorption of X-ray photons at those regions.

Figure 41 X-ray imaging of asparagus crown at 40kV/5s/Int4 without grating: (a) absorption-contrast, (b) enhanced absorption-contrast

In contrast, the Dark-field image (Figure 42a) has improved the visibility of most features in any part of the specimen as long as the features can produce strong (U)SAXS effect when interacting with the X-rays. The noise is minimized using noise reduction method such as de-speckle (Figure 42b), and image brightness and contrast adjustment (Figure 42c).

Figure 42 (a) Dark-field image, (b) Dark-field image enhanced by noise reduction (de-speckle), (c) Dark-field image with improved brightness and contrast

From the imaging results, we observed that the Dark-field image is indeed not a substitution but rather a complementary solution that provide additional information not accessible using the conventional absorption-contrast. Figure 43a shows the enlarged absorption-contrast image of the asparagus crown. Comparing with the enlarged version of the Dark-field image (Figure 43b), the absorption-contrast gives information about thickness variation across the specimen while the Dark-field image outlines the features with strong (U)SAXS effect.

Figure 43 Enlarged view of asparagus crown: (a) enhanced absorption-contrast, (b) Dark-field

5.4.6 Experiment 6: Imaging of Sub-surface crack

In this experiment, the sample (Figure 44) was prepared to mimic a sub-surface crack which is commonly found in the manufacturing industry. The sample setup consists of a transparent glass sandwiched between 2 sheets of opaque and thin paper covers as shown in Figure 44. In the cutting process to create an opening window through the center of the glass, cracks were accidentally introduced at the edges of the window.

Figure 44 Defective glass sandwiched between opaque covers

The top and bottom opaque covers are temporarily removed to allow a camera snapshot of the crack for verification with the X-ray result later on. From Figure 45, we noticed a crack at the bottom left corner of the window. In addition, at other corners of the window, we observed minor disruptions to the structure as outlined in red circles. These features on the glass surface can be easily picked up by optical camera, but may not be the case in the event when they are residing beneath an opaque surface.

Figure 45 (a) snapshot of cracks on glass, (b) enlarged view of region-of-interest A

Now with the defective glass embedded between the opaque covers, using the same setup, we obtained the transmission and Dark-field images as shown in Figure 46. From the transmission image, we are not able to visualize the features of interest. A further step has been taken to enhance the image contrast using image processing techniques, typically histogram equalizer and normalization. From the enhanced version of the transmission image, the window edges at the interface between the glass and air are sharpened. We observed an irregularity at the corners of the window but we are still unable to clearly trace the trail of the crack.

Figure 46 X-ray imaging with gratings of crack at sub-surface, 40kV/100uA
(a) Transmission, (b) enlarged view of region-of-interest A,
(c) transmission image with contrast enhancement, (d) enlarged view of region-of-interest B

Figure 47 X-ray imaging with gratings of crack at sub-surface, 40kV/100uA
(a) Dark-field image, (b) enlarged view of region-of-interest,
(c) extracted region 1, (d) extracted region 2, and (e) extracted region 3

In the Dark-field image (Figure 47), we obtained an improved visibility of the features. From the enlarged view near the window (see yellow box), we noticed a number of cracks are extended outward from different corners of the window which are not visible in the absorption-contrast images. At region 1, a short crack is detected as seen in Figure 47c, and at region 2, an even shorter rupture is identified (Figure 47d). At region 3, a long and curved fracture can be traced (Figure 47e). The shape and locations where the cracks are found match with those in Figure 45. However, only a fraction of the cracks are shown in the Dark-field images. We clearly see that additional effort is needed to reduce noise in the image and enhance the visibility of features so that the complete trail of the cracks can be visualized.

5.4.7 Experiment 7: Characterisation of materials

The main objective of this experiment is to show that besides using the X-ray Dark-field imaging for inspection purposes, it can also serve to characterize material based on the scattering power the material has when interacting with the incident X-rays. A variety of samples with different characteristics, such as soft cotton, seed, raw sugar, sago, and ginseng grain powder are inserted and arranged in the opaque polymer tubes as illustrated in Figure 48. A portion of the tube is left empty so that air in the final X-ray images can serve as a reference for comparing the scattering effect.

From the absorption-contrast image (Figure 49a) and its contrast enhanced version (Figure 49b), we clearly see the distribution of seeds, sugars, sago and grains inside the tubes. Their shapes and outlines are clear in the conventional X-ray images. However, what is missing is the soft cotton. Due to extremely low cotton density and X-ray absorption, the cotton cannot be discerned at all. In comparison, we obtained a lot more information from the X-ray Dark-field image.

Firstly, all materials are found and distinguishable in the Dark-field image (Figure 49c) and its enhanced version (Figure 49d). Even though cotton does not

absorb X-ray, the microstructures in the cotton scatter X-rays and re-distribute the intensity spatially, hence generating an alternative contrast in the image. Secondly, the Chai seeds and raw sugars which are not distinguishable in the absorption-contrast can now be clearly differentiated under two different shades of grey levels. The distribution of these two materials can also be picked up as highlighted in the yellow box.

Figure 49 Characterization of materials (a) absorption-contrast, (b) enhanced absorption-contrast, (c) visibility-contrast (Dark-field), (d) enhanced visibility-contrast

Lastly, the different sizes of sago and ginseng grains are reflected in the Dark-field image too although they share almost the same grey level. Even though the features outline have become not clear and they appear smeared as a result of (ultra) small-angle-scattering by the microstructures within, this experiment has nevertheless demonstrated the strength of Dark-field imaging in characterizing materials that are useful in the detection of certain structural details in a matrix of material with almost similar X-ray absorption capability.

6. Conclusion

In this thesis, we reported the development of X-ray Dark-field imaging using a grating-based Talbot interferometer to achieve significantly enhanced X-ray imaging contrast as compared to the conventional X-ray imaging method. The operation of conventional X-ray imaging is based on the principle of Lambert-Beer's Law. It states that X-ray energy's attenuation is proportional to the material's mass attenuation coefficient, which highly depends on the probability of the occurrence of photoelectric absorption and inelastic Compton scattering of X-rays in the material to generate absorption-contrast in the X-ray image. An object that contains materials of high mass attenuation coefficient, such as elements with high atomic number, will attenuate X-rays more and generate grey values that are different from their surroundings in the image. In addition, X-rays propagating through a thicker object will experience higher chances of attenuation as compared to a thinner object. Therefore, the conventional absorption-contrast discriminates materials based on their variations in the mass attenuation coefficients.

Our proposed X-ray Dark-field imaging system is based on the principle of grating-based Talbot interferometry. A phase grating is used to produce fringes pattern at a distance downstream from the grating and an analyser grating is used to resolve the object-induced X-ray wavefront distortion via a phase-stepping procedure. As X-ray propagates through the sample, it encounters (ultra)small-angle scattering that causes X-rays to deviate from its original path, thus resulting in a redistribution and broadening of detected intensity signal at the sensor. The visibility-contrast strongly relies on the ability of the material and its structural details to distort the X-ray wavefront. In addition, we introduced visibility transformation functions (both linear and nonlinear) as part of the image processing procedure to further improve the contrast of the acquired Dark-field image.

6.1 Current achievement

A grating-based Talbot interferometer has been instrumented to enable experimental validations of the principle and effectiveness of Dark-field imaging. In this research, we found out that X-ray Dark-field imaging is suitable for the visualization of features inside and outside of low-density materials (biological sample, polymer device), and it also opens an avenue for possible material characterization by its ability to detect the (ultra) small-angle-scattering effect.

Table 3 A summary of experimental validations of X-ray Dark-field imaging

	Conventional X-ray Absorption-contrast	X-ray Dark-field Visibility-contrast
Biological sample: Onion with non-uniform thickness		
(a) Visibility of external feature	Image contrast at the thin layers of onion skin is poor and visibility is low.	Visibility of onion skin is significantly enhanced and the contrast is satisfactory.
(b) Visibility of internal feature	Internal features are visible at thinner regions of the body but very poor visibility at thicker parts even with high X-ray energy.	Achieved good visibility of internal scale leaves across a wide variation of body thickness, without sacrificing the contrast at thin tunic layers.
Manufactured sample: Crack in Glass panel sandwiched between opaque covers		
(a) Visualization of sub-surface cracks	A small part of the deep crack is visible, but not complete visibility. Other minor fractures are not detectable.	All cracks are detectable and can be identified in the Dark-field image. However, some shallow parts, especially the tail ends of the cracks are not clearly visible.
Material characterization: Cotton/Seed/Grains		
(a) Discrimination of different samples	Chai seed and raw sugar are not distinguishable. Cottons are not detectable.	Different kinds of seeds/grains are differentiated in terms of different shades of grey values in the Dark-field images. Cottons are visible.
(b) Visualization of sample shape and size	Clearly shows the outlines of shapes and sizes of seeds/grain, except for the soft cottons.	Our X-ray Dark-field imaging method is not able to capture outlines of shapes and sizes of the samples.

Through experimental validations we summarize in Table 3 a few comparisons between the conventional X-ray absorption-contrast and the visibility-contrast (Dark-field). We also conducted a number of experimental evaluations to investigate the visibility of fringes in the image and the quality of images acquired from phase-stepping procedure. We gathered that the number of image integration required indeed influences the reliability of the image content. The results showed that fringe visibility is improved (i.e. smaller variation of contrast-to-noise value) with the number of integration. In addition, the intensity oscillation curves at the background and the fringes are reasonably sinusoidal and hence they provide easily detectable amplitudes which contribute to good visibility contrast in the Dark-field image. Furthermore, the oscillation curves obtained from the repeating cycles of phase-stepping agreed quite well with each other. This means that the performance of grating movement is within acceptable tolerance for the Dark-field imaging, which consequently ensures that image artifacts due to misalignment of the oscillation curves obtained with and without sample are kept to the lowest possible. The assessment results also provided a guideline to configure our imaging parameters. In this thesis, we seek to achieve a balance between imaging duration and image quality. Hence, in the imaging experiment, we showed that using an integration number of 2 or 4 not only save us the data acquisition time, and we still can generate an enhanced image contrast even with a low brilliance X-ray microfocus tube.

In conclusion, we demonstrated that by using a low-brilliance X-ray source such as a laboratory microfocus tube, our X-ray Dark-field imaging that operates based on the principle of Talbot grating interferometry can discern structural detail within a matrix of low X-ray absorbing material and maintain a consistent contrast-to-noise ratio over a large variation of object thickness. Furthermore, the resultant visibility-contrast outperforms the conventional absorption-contrast in terms of revealing the fine details embedded in a matrix of weakly-absorbing material over a large field-of-view of $5 \times 5 \text{ cm}^2$. The finding complements the conventional X-ray absorption-contrast imaging and will open new and broader avenues for diagnostic and inspection applications in both medical and manufacturing settings that often have to deal with objects of non-uniform thickness.

6.2 Future work

Our X-ray Dark-field imaging system demonstrated advantages in terms of contrast and feature visibility enhancement. It also demonstrated limitations in terms of material discrimination power and imaging resolution.

The future plan would include the effort in improving the sensitivity of the X-ray Dark-field imaging system and enhancing the imaging specificity to strengthen the material discrimination power and imaging resolution. In addition, it would also be worthwhile to explore 3D reconstruction of the information derived from X-ray refraction and (ultra)small-angle-scattering. The potential research directions may include two major aspects:

1. 3D XPDT (X-ray phase-contrast and dark-field tomography) system design and instrumentation
2. Design and development of tomographic algorithm for XPDT

Through the design and implementation of X-ray grating or zone plate in the XPDT system, it may be promising to detect feature size of a few micrometer towards sub-micrometer within a sample dimensions of at least 50mm³. The system would be designed such that it is mechanically stable and scalable to enable fast-speed scanning of larger sample area.

Following that, the tomographic algorithm would be designed to reconstruct volumetric phase-contrast and dark-field datasets that represent the virtual 3D model of the real object. From virtual slicing through the 3D models, we could visualize the internal structural details at micrometer and sub-micrometer length scales. Further, the algorithm would also aim to handle large amount of image sequence in a shorter timeframe.

7. List of author's publication

Submitting a manuscript to a specialized journal in the field of X-ray imaging.

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