Hard X-ray phase imaging and tomography using a grating interferometer

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Received 6 January 2006; accepted 11 March 2007
Available online 16 March 2007

Abstract

An interferometric technique for hard X-rays is presented. It is based on two transmission gratings and a phase-stepping technique, and it provides separate radiographs of the phase and absorption profiles of bulk samples. Tomographic reconstruction yields quantitative three-dimensional maps of the X-ray refractive index and of the attenuation coefficient, with a spatial resolution down to a few microns. The method is mechanically robust, it requires little monochromaticity, and can be scaled up to large fields of view. These are important prerequisites for use with laboratory X-ray sources. Numerous applications ranging from wave front sensing to medical radiography are presently under investigation.

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Keywords: Synchrotron radiation; Phase contrast; Radiography; Interferometry

1. Introduction

X-ray radiography and tomography are important methods for the non-destructive investigation of bulk samples, as the penetration depth of hard X-ray beams is rather high, and the low level of diffuse scattering allows for the recording of sharp projections of the attenuation coefficient. However, for such important classes of samples as biological tissue, polymers, and fiber composites, the use of conventional X-ray radiography is limited because these objects show only weak absorption. Therefore, X-ray radiographs or tomographic data sets with sufficient amplitude contrast are often difficult to obtain. In particular for medical applications such as mammography, a high radiation dose is required.

The cross section for elastic scattering of hard X rays in matter, which causes a phase shift of the wave passing through the object of interest, is usually much greater than that for absorption. For example, 17.5-keV X-rays that pass through a 50-μm-thick sheet of biological tissue are attenuated by only a fraction of a percent, while the phase shift is close to \( \pi \). Recording the X-ray phase shift rather than only the absorption thus has the potential of substantially increased contrast. A variety of X-ray techniques are employed to detect the phase contrast of a sample, i.e. to convert it into an amplitude contrast in the image plane [1]. Some techniques use the Fresnel diffraction of coherent hard X-rays at the edges of a phase object to significantly improve the visibility of an object in microradiography (see for example [2]). In first approximation, the obtained intensity distribution is proportional to the Laplacian of the refractive index distribution [3] and in certain cases a reconstruction of a phase object from a single micrograph is possible [4]. Quantitative information on arbitrary phase objects can be obtained by numerically evaluating series of images acquired with the detector placed at different distances from the sample [5].

The most sensitive method to measure the phase shifts introduced to a wave front is interferometry. A set-up for a Mach-Zehnder type interferometer operated in the hard X-ray range was introduced by Bonse and Hart [6] about four decades ago. It consists of three partially transmitting Bragg crystals used as beam splitter and recombining elements. The incoming light is split into two separated branches one of which passes through...
the sample while the other serves as an unperturbed reference beam. The two beams interfering at the exit of the interferometer give an intensity distribution that represents the difference in optical path and thus – if perfectly aligned – of the phase shift caused by the object. Ando and Hosoya pioneered phase contrast imaging with such a device in the early seventies [7], and more recent set-ups have produced large numbers of excellent phase contrast images and computer tomograms, e.g. of biological specimens [8,9]. The main technical difficulty lies in the extreme demands on the mechanical stability of the optical components, as the relative positions of the optical components have to be stable within a fraction of a lattice constant, i.e. to sub-Ångstrom dimensions. Therefore, Bonse-Hart interferometers are very difficult to handle especially when made big enough to investigate large samples.

We developed a different kind of interferometer, in which the interfering beams are not completely separated but merely sheared by a small angle so that they pass through different, closely spaced parts of the sample. The principle of this device is depicted in Fig. 1. It consists of a phase grating $G_1$ and an absorption grating $G_2$, a scheme which was first proposed by us in 2002 [10]. The first grating acts as a beam splitter and divides the incoming beam essentially into the two first diffraction orders. Since the wavelength $\lambda$ of the illuminating hard X-rays ($\approx 10^{-10}$ m) is much smaller than the grating pitch ($\approx 10^{-6}$ m), the angle between the two diffracted beams is very small. Downstream of the beam-splitter grating, the diffracted beams interfere and form linear periodic fringe patterns with a periodicity $g$ that equals half the period of $G_1$. Note that the period and the lateral position of these fringes do not depend on the wavelength of the X-rays. Perturbations of the incident wave front, such as those induced by refraction on an object in the beam, lead to local displacement of the fringes. However, since the pitch of the phase grating (and thus the spacing of the interference fringes) does not exceed a few microns, an area detector placed in the detection plane will generally not have sufficient resolution to resolve the fringes. Therefore, a grating $G_2$ with absorbing lines and the same periodicity and orientation as the fringes is placed in the detection plane, immediately in front of the detector. This analyzer grating acts as a transmission mask for the detector and transforms local fringe position into signal intensity variation. The detected signal profile thus contains quantitative information about the phase gradient of the object.

To separate this phase information from other contributions to the signal, such as absorption in the sample, inhomogeneous illumination or imperfections of the gratings, the phase-stepping approach used in visible-light interferometry [11] was adapted to this setup. We can derive two separate images of an object following a process described in more detail in [12]. The first one represents the amplitude contrast image that we would have received with no interferometer in the beam. It contains absorption contrast and in-line phase contrast caused by diffraction on the edges of the sample. The intensity signal of the second image is proportional to the phase shift gradient in the object, which is why we call it the differential phase contrast (DPC) image. The DPC image can be used to obtain the phase profile of the object by a simple one-dimensional integration. An example of an amplitude contrast image, a DPC image, and a phase contrast image of a simple test object obtained with the described method is shown in Fig. 2. One can already see a significant enhancement of the contrast in the phase contrast images. Moreover, it should be pointed out that a different physical quantity is used for the image formation: while the attenuation of the beam is related to the imaginary part of the complex refractive index, the phase shift is related to its real part.

2. Grating fabrication

Obviously, the quality of the gratings used in such an interferometer set-up is crucial. We use micro-fabrication techniques to define the grating structures with sufficient accuracy. It is essential that the two gratings have the correct ratio of periods. For a plane incoming wave, the period of $G_2$ should be two times smaller than that of $G_1$, whereas for a spherical incoming wave, a slight correction needs to be included. Microlithography techniques need to be used to define the grating line pattern on silicon substrates. The further processing depends on the individual properties required: the beam splitter grating $G_1$ should have low absorbing structures that introduce a phase shift of $\pi$ to the passing X-ray waves, whereas the analyzer grating $G_2$ should have highly absorbing lines.

![Fig. 1. Principle of the X-ray grating interferometer. The first grating splits the incoming wave into two waves which create a line pattern by interference. This pattern is analyzed using a second grating. An object with a phase gradient in the beam will act as a prism and slightly refract the beam, which in turn shifts the interference pattern downstream of the object in lateral direction. The resulting change in transmission through the analyzer grating depends on the sign and strength of the phase gradient.](image-url)
We use silicon as the phase shifting material for the beam splitter gratings. The structure height to obtain the required phase shift is proportional to the photon energy used. For 17.5 keV, a height of 22 μm is optimum. The period of our beam splitter gratings is close to 4 μm resulting in very high aspect ratios of the structures. The top part of Fig. 3 shows a cross section of such a grating. The structures are made by wet chemical etching in potassium hydroxide solution. As substrates, we use 250 μm thick silicon wafers with ⟨110⟩ orientation. The grating patterns are exposed using a high precision electron beam lithography process [13]. The orientation of the lines is along the ⟨112⟩ direction with a precision of better than 0.1°, this results in an anisotropic etching with vertical side walls. This process is also used for the fabrication of linear Fresnel zone plates, more details can be found in [14].

The fabrication of the analyzer gratings is even more challenging. Firstly, the period has to be two times smaller, i.e. 2 μm, and secondly, no simple etching process exists to pattern highly absorbing materials with high aspect ratios. The structure height again depends on the photon energy, at 17.5 keV using gold as absorbing material, a structure height of 10 μm is desirable for a high contrast of the DPC signal.

We first pattern a silicon grating using the method described above. Then, the gaps of the grating are filled with gold by electro-deposition. Using a shadow evaporation process and selective wet etching, it is possible to let the gold grow from the bottom of the silicon grooves [15], as any deposition on the side walls or the silicon ridges would result in an incomplete filling of the grooves. The lower part of Fig. 3 shows a cross section of a gold-filled silicon grating fabricated by the described process.

3. Experimental results

In the setup used at various synchrotron beamlines, the gratings are mounted into a holder unit, which has motorized actuators for translation perpendicular to the grating lines and rotation about the beam axis. The inter-grating distance is fixed to typically a few centimeters, and the interferometer is mounted directly onto an X-ray CCD camera. The gratings are then aligned with the lines parallel to each other using a moiré technique. The setup is very robust, it requires no special precautions to reduce vibrations, mechanical drift, etc.

3.1. Phase contrast radiography

The method was applied to image an animal organ, a rat heart, which was placed in a container filled with a 4% aqueous formalin solution. The imaging of such a sample is very challenging, as the sample and the surrounding medium have very similar absorption and phase shift. Fig. 4 shows the amplitude image and DPC image, as well as an image of the projected phase shift of the sample. The phase image is again the result of a numerical integration of the DPC image. The
images are stitched together from two frames as the used X-ray camera was too small to cover the whole sample. In amplitude contrast, only some fatty tissue in the upper half of the image is visible, whereas the complete organ with many details can be seen in the DPC and the phase image. Note that the gray scale of the DPC image and the phase contrast image are quantitative, as can be seen from the units of the grayscale bar.

3.2. Phase contrast tomography

As mentioned before, the phase gradient images can be used to obtain phase images by integration. As the method yields quantitative information, a set of projections taken from an object under different viewing angles can be used to obtain a 3-dimensional map of the real part $\Re$ of the complex refractive index $n = 1 - \Im + i\Re$ for each voxel. Fig. 5 shows a small spider sitting on a loop of fishing line. This three-dimensional density-projection rendering of the refractive-index distribution reveals details of the internal structure of the animal that would be difficult to access with other techniques. Note the fine details inside the joints of the spider’s legs. The spatial resolution is a few micrometers.

3.3. Wave front sensing

The grating interferometer is very sensitive to minute deflections of an X-ray wave front. In case of the described radiography and tomography experiments, this is caused by refraction on phase gradients in the object. The high sensitivity of the method can also be used to monitor wave front distortions of synchrotron radiation to characterize the source itself or optical components. We have successfully applied this method to map out the errors of X-ray multilayer mirrors [16] simply by placing the interferometer directly behind the mirror. It was possible to detect slope errors with a sensitivity better than 100 nrad, which is below the fabrication tolerances of state-of-the-art mirror fabrication. A similar experiment was performed to reveal the fabrication errors of Beryllium refractive X-ray...
lenses. Besides the extreme sensitivity, the method has the great advantage that it can be used in-situ, meaning when the optical element is mounted in its operating environment including the heat load of the synchrotron radiation.

4. Conclusion and outlook

We have demonstrated how an interferometer consisting of two microstructured gratings can be used to record differential phase contrast images and phase contrast images in the hard X-ray range. Especially for light, low absorbing materials, the contrast can be enhanced significantly. The image formation process can also be used to acquire data sets suitable for a tomographic reconstruction of the X-ray refractive index inside an object. In addition, the interferometer can detect very small wave front distortions in the X-ray range, which makes it useful for the in-situ testing of X-ray optical elements.

Besides applications in materials science, and non-destructive testing, our future work will focus on the further development of the technique for medical diagnostics. Mammography appears to be one of the most promising applications, as the photon energies used in breast imaging are low compared to other medical radiography techniques, the absorption contrast between healthy tissue and a breast tumor is usually very small, and the reduction of the applied radiation dose is a significant issue.

This will require two major development steps. Firstly, the field of view of the interferometer – and thus the gratings – has to be scaled up from its present size of a few millimeters to many centimeters. It is therefore no longer efficient to use slow and expensive electron beam lithography for the grating fabrication. We have therefore developed a photolithography process that enables us to fabricate interferometer gratings over large areas. Fig. 6 shows a photograph of a 64 mm × 64 mm silicon beam splitter grating fabricated at the Laboratory for Micro- and Nanotechnology. The size is at present limited by the used 100 mm diameter wafers, but there is no fundamental problem to transfer the process to the 300 mm diameter substrates presently used in semiconductor industry.

The second development step is to transfer the technique away from the synchrotron to X-ray tube sources. Unlike a synchrotron, such sources provide too little flux to be used with a monochromator, and the radiation is much less collimated. We have demonstrated [12] that the grating interferometer technique can be used with broad-band X-rays and divergent radiation. These properties make the presented phase contrast imaging technique most promising for medical applications.

Acknowledgements

We would like to thank B. Haas, F. Glaus, and E. Deckardt of the LMN for help in the microlithography processes. We are also indebted to various staff members of the European Synchrotron Radiation Facility ESRF and the Swiss Light Source SLS.

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