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New opportunities in X-ray tomography

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Abstract

We discuss standard X-ray-imaging techniques. Phase-imaging methods and a new class of nano-focus and nano-resolution laboratory systems offer new opportunities in true laboratory-based X-ray microtomography with a host of possible applications that have mainly been demonstrated only at synchrotron sources. Notwithstanding these advances, the diffraction limit for X-ray-imaging methods is a long way off. We preview the link between high-resolution 'standard' imaging schemes and the new field of coherent diffractive imaging.

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1. Introduction

X-ray tomography based on absorption contrast has been a staple of medical and materials science for many years. The use of CT scanners in medicine and in non-destructive testing of materials is well known. Nevertheless, various aspects of tomography have remained at the forefront of scientific research. Consequently, reconstruction algorithms continue to be improved as do methods for locally reconstructing samples that extend beyond the detection apparatus field of view. Improvements in detector technology continue to improve the resolution and detection efficiency of experiments (although modern detectors are only now beginning to approach the workhorse of X-ray science over the last century—X-ray film).

The advent of synchrotron sources has allowed the exploitation of materials properties as a function of energy. In particular, the differential contrast between proteins and water based on the difference between the carbon K edge (284 eV, 4.4 nm) and the oxygen K edge (532 eV, 2.3 nm) has been well explored (Spiller, 1994). However, the field has been limited mainly to frozen or dried samples or to other radiation tolerant samples due to the high dose received during the imaging process (Attwood, 1999).

Due largely to the improvement in synchrotron sources, recent years have seen the development of low-dose methods of obtaining sample information via measurement of the phase of the wavefield. Techniques include: interferometry (Momose et al., 1995; Kohmura et al., 2003; Beckmann et al., 1997). Zernike phase contrast (Schmahl et al., 1988), differential interference contrast (Wilhein et al., 2003), segmented detectors

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(Palmer and Morrison, 1991) or Shack–Hartmann arrays (Lane and Tallon, 1992), and diffraction enhanced imaging (Chapman et al., 1997). Phase variations in the wavefield can be detected directly as a result of the evolution of contrast in the intensity of the wavefield with propagation. Approaches that have been demonstrated include in-line holography (Jacobsen et al., 1990), multiple defocus methods (Coene et al., 1992) and Wigner deconvolution methods (Chapman, 1996), while the simpler technique of optics-free propagation was realized only in the latter half of the 1990s (Snigirev et al., 1995; Cloetens et al., 1996; Wilkins et al., 1996). Several methods of inverting the equations governing the propagation of the wavefield have now been developed. These include contrast transfer function methods, which require the assumption of a weak object (Wu and Li, 2003) or of a slowly varying phase with a weakly absorbing object (Turner et al., 2004), transport of intensity methods (Nugent et al., 1996, Paganin et al., 2002), holographic methods (Jacobsen et al., 1990), and far- (Miao et al., 1999) and near- (Allen et al., 2004) field iterative methods.

Whether phase or absorption contrast is used for imaging, the resolution attainable in the images is limited. In radiography where a contact image of the object is made or a parallel beam is used to image the sample, as in Fig. 1, the resolution is limited by the detector (Peele et al., 2005). This is typically of the order of tens of microns for CCDs. Scintillator or film detectors can attain of order 1 μm resolution. However, to image at high resolution some form of projection

Fig. 1. Bee sting imaged using phase contrast and parallel beam (Peele et al., 2005). The width of the sting is about 50 μm at its thinnest.

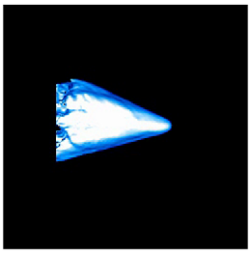

Fig. 2. AFM tip imaged at sub-micron resolution using phase imaging and point projection (McMahon et al., 2003). The radius of curvature at the tip is 900 nm.

geometry, such as that used in obtaining the image in Fig. 2 (McMahon et al., 2003), must be used. This typically uses some form of optic (zone plates, curved mirrors or refractive lenses) to obtain magnification from a simple point source projection or the optic is implemented in a microscope configuration. In this form resolution is limited by the optics. The current best resolution attained with a zone plate in first order is about 15 nm (Chao et al., 2005). At higher energies resolution is several times coarser.

The great advances that have been made in synchrotron technology have perhaps obscured the fact that laboratory sources can offer similar performance for X-ray imaging in some cases. For X-ray energies above the Cu K edge (8 keV, 1.54 Å) a laboratory source can offer similar resolution at comparable data acquisition times for a tomographic data set. This is due to the fact that for imaging, particularly phase imaging, a relatively coarse energy bandpass can be used and still provide quantitative results (Mayo et al., 2002; Pogany et al., 1997). Additionally, some of the high flux advantage from a synchrotron source is lost as the bulk of the acquisition time is lost in detector readout and stage motion and settling. Furthermore, recent years have seen the development of relatively high-power micro- and nano-focus sources and nano-resolution imaging systems. These provide sub-micron resolution in the laboratory setting. Indeed low-power laboratory systems with source resolution in the 100 nm range have been demonstrated (Mayo et al., 2002).

2. New opportunity 1

Consider a homogeneous sample in the sense that it is described by a single refractive index, $n = 1 - \delta - i\beta$. In general a sample can be Fourier decomposed into a sum of components with various spatial frequencies. Consider a single spatial frequency, $\mathbf{u}$. The strength of the sinusoidal features at a measurement distance, z, for X-rays with wavelength, λ is given by the contrast

transfer function (Arhatari et al., 2004), CTF:

$$\mathrm{CTF} = \frac{4\pi}{\lambda}\left[\beta\cos(\pi\lambda z u^2) - \delta\sin\left(\pi\lambda z u^2\right)\right]. \quad (1)$$

This can be written (Turner, 2005)

$$\mathrm{CTF} = \frac{4\pi}{\lambda}\sqrt{\beta^2+\delta^2}\sin\left(\pi\lambda z u^2 + \arctan\left(\frac{\beta}{\delta}\right)\right). \quad (2)$$

For optimal contrast for a given spatial frequency the argument of the sin function in Eq. (2) equals π. For high energy X-rays for materials away from absorption edges the ration β/δ is small (Spiller, 1994; Attwood, 1999) so that for optimal imaging

$$z \approx \frac{1}{\lambda u^2}. \quad (3)$$

For a high-magnification point-projection imaging system z is approximately the source to sample distance. Thus, at an energy of 8 keV and for Nyquist sampled features ($u = 2/D$, where D is the feature size corresponding to the spatial frequency u) the source to sample distance for 1 μm features should be approximately 1.6 mm. This is feasible with so-called 'open source' micro- and nano-focus X-ray sources. For resolutions of the order of 100 nm, separations of order 10 μm are required. This has been achieved by incorporating the sample holder into the vacuum volume for the X-ray source (Mayo et al., 2002).

Phase imaging of features in the 1 μm range can therefore be made optimal using the newer nano-focus sources or using nano-resolution systems. Possible applications that we intend to pursue in the near future include: cracking and delamination in composite materials; corrosion studies; morphology of filtration particles; morphology in animal anatomy including eyeballs and vascalature; inclusions in materials ranging from gemstones to machine coatings and tissue scaffolding. All of these applications have scale lengths of interest in the 1 μm range and the need for non-destructive and/or 3D information precludes the use of standard high resolution but 2D methods such as SEM or AFM.

However, this ability to image features and therefore to undertake true microtomography in the laboratory setting only highlights the actual limitations of working with X-rays. While submicron imaging is possible and systems exist at both synchrotron sources and in the laboratory to image below 100 nm, the *diffraction* limit at X-ray energies is of the order of 10^{-10} m! Is there a way to enhance the images that are currently attainable using standard optical methods?

3. New opportunity 2

Recent developments in coherent diffractive imaging (Miao et al., 1999) (CDI) suggest the answer to the previous question may well be yes. In CDI the far field diffraction pattern from a sample is phased and thereby inverted via an iterative technique. In recent work we have investigated the effect of illuminating the sample with curved wavefields (Nugent et al., 2003; Nugent et al., 2005; Quiney et al., 2005). Our results indicate that appropriately chosen curved-beam illumination will encourage faster and more accurate inversions of the diffraction pattern, ensure a unique solution and can allow a solution for extended objects. This latter feature is particularly useful as in the standard CDI scheme samples must be contained within a finite region. Furthermore, the point projection magnification scheme we envisage for the 'standard' microtomography work is eminently suited to the curved beam CDI approach. As can be seen in Fig. 3 the magnified image forms the central or holographic part of the overall diffraction pattern. Higher spatial frequency information is scattered outside the illumination cone and this data forms the basis of the CDI inversion. The 'standard' image can therefore provide an excellent starting guess for the iterative solution method. Our recently funded ARC

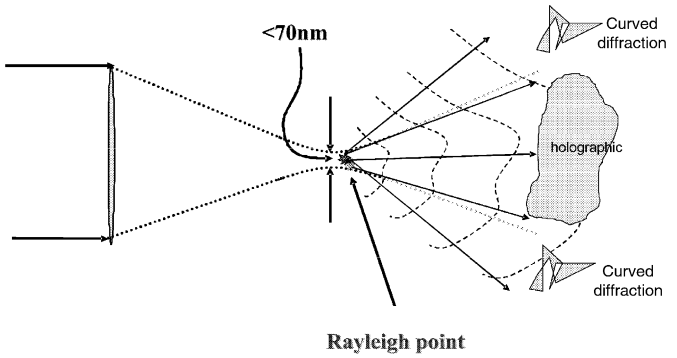

Fig. 3. Schematic showing point illumination of a sample and the resulting features of the image/diffraction pattern. The central region contains the magnified image of the sample. Depending on the geometry this image will appear as a magnified image or as a hologram. Outside this region the diffracted signal carries high spatial frequency information (Nugent et al., 2003, 2005; Quiney et al., 2005).

Centre of Excellence in Coherent X-ray Science is now in a unique position to investigate this natural evolution from 'standard' imaging methods to the diffraction-limited promise of CDI.

4. Conclusion

New laboratory-based sources offer the promise of standard phase and absorption imaging at the sub-micrometre level with a host of materials and biological science applications. Of even greater promise is the technique of CDI which can in principle provide imaging at the sub-nanometre scale. While the technical issues involved in imaging the shape and function of proteins as they go about their business inside a living cell are immense, the rewards are commensurate with the effort. Additionally, the steps along the way to this goal, such as imaging of fabricated nanostructures and micro- and nano- crystal imaging would in themselves represent valuable achievements.

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