X-RAY IMAGING IN MICRO-TO-NANO WORLD

J.H. Je^{1*}, B.M. Weon¹, S.K. Seol¹, J.M. Yi², Y. Hwu³, and G. Margaritondo⁴

¹X-ray Imaging Center, Pohang University of Science and Technology San 31 Hyojadong, 790-784 Pohang, Korea

² Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, USA

³ Institute of Physics, Academia Sinica, Taipei 115, Taiwan

⁴ Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015, Switzerland

Keywords: radiography, phase contrast, x-ray imaging, synchrotron, bright field imaging

*) e-mail: jhje@postech.ac.kr

In recent years, X-ray imaging has been literally revolutionized by the exploitation of the unique characteristics of synchrotron sources. In particular, the high spatial coherence of the radiation significantly contributes to the development of advanced and powerful X-ray imaging. The results are very high quality microradiology and microtomography images and movies - taken with a limited X-ray dose – that find a variety of applications in materials science, biology and medical research. In this talk we review basic theory and selected applications of phase contrast X-ray imaging to materials and biomedical sciences. Furthermore we introduce a new strategy of combining phase contrast radiology and diffraction X-ray microscopy to visualize atomic level defects such as misfit dislocations and micropipes in semiconductor single crystals. Finally phase contrast X-ray imaging in nanometer-resolution (< 30 nm) will be demonstrated.

1. Introduction

X-ray imaging methods based on phase contrast radiology are becoming important analytical tools for real-time processes in materials science, life science, medicine, physics, chemistry and other disciplines [1-4]. Even though theoretical background and the practical implementation were discussed recently [5], most of results had limitation in dynamic studies owing to significant losses of X-ray flux by using monochromatic X-rays.

In comparison we introduce white beam phase contrast X-ray imaging, enabling time-resolved dynamic studies in millisecond time resolution. We review basic imaging mechanism of white beam phase contrast imaging and demonstrate several applications in materials and biomedical sciences.

To further enhance spatial resolution in nanoscales, the development of X-ray nanoscopy based on using Fresnel zone-plates is discussed [6]. Furthermore we introduce a new concept of X-ray microscopy, brightfield X-ray microscopy, which has been for the first time developed in our group by combining phase contrast radiology and diffraction topography [7, 8].

2. Technical background

Coherence is the property of a wave that enables to produce visible diffraction and interference effects. For the X-rays traveling though a pinhole, the diffraction pattern may or may not be visible on the detector depending on the source size, its angular divergence and its wavelength bandwidth. The condition to see the edge diffraction fringes is $\Delta\lambda/\lambda < \sqrt{2}$. This condition is already satisfied without using any monochromator for synchrotron hard X-rays. The equivalent condition for "refraction" radiology is much more relaxed. This results in many consequences of the limited need for time coherence. First of all, no monochromator is necessary for phase contrast radiology. In other words white or "pink" beamlines are enough for phase contrast X-ray imaging. Therefore there is no monochromator-related X-ray flux loss, enabling time-resolved experiments with time resolution of 1 msec. Finally high time resolution together with high (better than 1 micrometer) lateral resolution can be achieved [9].

As for X-ray nanoscopy, Fresnel zone plates (FZPs) are widely used as focusing and magnifying optics devices and offer the highest imaging resolution in the entire electromagnetic spectrum. A FZP consists of concentric rings with decreasing width and increasing radius - and the outermost zone width approximately sets the resolution.

X-rays can yield bright-field (BF) images of crystalline systems similar to transmission electron microscopy (TEM); such images carry information both from diffraction/scattering phenomena and from absorption and phase contrast. For a strong reflection of (0001) 4H-SiC wafers in the Laue (transmission) geometry, synchrotron X-ray transmission micrographs simultaneously yield diffraction-based information on lattice distortions and radiographic information on structural inhomogeneities.

3. Applications in materials and biomedical sciences

The fabrication of 3D conducting polymer structures with high aspect ratios remains a challenge. Such structures are particularly important in a broad range of device applications in microelectronics, biomedical devices, and micro-systems such as actuators and sensors.



Figure 1. FESEM images of freestanding PPy HAR microstructures with dense and smooth morphology. The images show: a) a wire-like straight structure with aspect ratio = 70; b) a zigzag structure; c) a complex structure corresponding to the letters "XIC".



Figure 2. Sequence of phase contrast images revealing the evolution of the water film during X-ray irradiation.



Figure 3. The volume-rendered 3D structure of a piece of mouse aorta. Scale bar is $50 \mu m$.

Figure 1 shows several successful fabrication tests of freestanding polypyrrole(PPy) high-aspect-ratio microstructures with different shapes: straight (a), zigzag (b), and a complex geometry (c) using real-time monitoring of localized electropolymerization of 3D PPy growth [10].

In spite of the strong fundamental and applied interest in water microstructure, so far, no technique was able to produce stable freestanding pure-water thin films, mostly due to rapid rupture caused by the very low viscosity and high surface tension of pure water. As can be demonstrated in Fig. 2 that shows real-time fabrication process of stable free standing thin films of pure water, we were for the first time able to fabricate free standing water film with a lifetime of 1 h after 54 min irradiation [11].

Figure 3 demonstrates the volume-rendered 3D structure of a piece of mouse aorta. The white arrows point to borders between neighboring cells, whereas the black arrow points to a cell nucleus that extrudes from the surface and whose outline is well preserved in the 3D reconstruction analysis. In this case, the imaging capability goes beyond the mere imaging of the outline and shape of the individual cell and provides subcell information [12].

4. Conclusions

White beam phase contrast X-ray imaging is expected to be significantly applied in a variety of sciences in the near future. Acknowledgments: This work was supported by the Creative Research Initiatives (functional X-ray Imaging) of MOST/KOSEF.

References

- [1] Y. Hwu, W.-L. Tsai, A. Groso, G. Margaritondo, J.H. Je, "Coherence-enhanced synchrotron radiology: simple theory and practical applications", *J. Phys. D: Appl. Phys.* 35 (2002) R105-R120.
- [2] A. Snigirev, I. Snigireva, V. Kohn, S. Kuznetsov, I. Schelokov, "On the possibilities of x-ray phase contrast microimaging by coherent high-energy synchrotron radiation", *Rev. Sci. Instrum.* 66 (1995) 5486-5492.
- [3] A. Momose, T. Takeda, Y. Itai, K. Hirano, "Phase-contrast X-ray computed tomography for observing biological soft tissues", *Nature Med.* 2 (1996) 473-475 & 596.
- [4] W.L. Tsai, P.C. Hsu, Y. Hwu, C.H.Chen, L.W. Chang, J.H. Je, H.M. Lin, A. Groso, G. Margaritondo, "Building on bubbles in metal electrodeposition", *Nature* 417, 6885, (2002) 139.
- [5] P. Cloetens, R. Barrett, J. Baruchel, J.-P. Guigay, "Phase objects in synchrotron radiation hard x-ray imaging", J. Phys. D: Appl. Phys. 29 (1996) 133-146.

- [6] Y.S. Chu, J.M. Yi, F. De Carlo, Q. Shen, W.-K. Lee, H.J. Wu, C.L. Wang, J.Y. Wang, C.J. Liu, C.H. Wang, S.R. Wu, C.C. Chien, Y. Hwu, A. Tkachuk, W. Yun, M. Feser, K.S. Liang, C.S. Yang, J.H. Je, G. Margaritondo, "Hard-x-ray microscopy with Fresnel zone plates reaches 40 nm Rayleigh resolution", *Appl. Phys. Lett.* **92** (2008) 103119.
- [7] J.M. Yi, Y.S. Chu, Y. Zhong, J.H. Je, Y. Hwu, G. Margaritondo, "X-ray bright-field imaging analyzes crystalline quality and defects of SiC wafers", *J. Appl. Crystallogr.* 40 (2007) 376-378.
- [8] J.M. Yi, J.H. Je, Y.S. Chu, Y. Zhong, Y. Hwu, G. Margaritondo, "Bright-field imaging of lattice distortions using x rays", *Appl. Phys. Lett.* 89 (2006) 074103.
- [9] G. Margaritondo, Y. Hwu, J.H. Je, Synchrotron light in medical and materials science radiology, *Riv. Nuovo Cimento* 27 (2004) 1-40.
- [10] S.K. Seol, JT Kim, JH Je, Y Hwu, G Margaritondo, "Three-Dimensional (3D) Polypyrrole Microstructures ", *Macromolecules*, **41** (2008) 3071–3074.
- [11] B.M. Weon, J.H. Je, Y. Hwu, G. Margaritondo, "Stable freestanding thin films of pure water", *Appl. Phys. Lett.* 92 (2008) 104101.
- [12] Y. Hwu, W.-L. Tsai, H.M. Chang, H.-I. Yeh, P.-C. Hsu, Y.-C. Yang, Y.T. Su, H.L. Tsai, G.-M. Chow, P.C. Ho, S.C. Li, H.O. Moser, P. Yang, S.K. Seol, C.C. Kim, J.H. Je, A. Groso, E. Stefanekova, G. Margaritondo, "Imaging cells and tissues with refractive index radiology", *Biophysical J.* 87 (2004) 4180–4187.