doi:10.1017/S1431927618012928
Microsc. Microanal. 24 (Suppl 2), 2018

Nondestructive Multiscale X-Ray Tomography by Combining Microtomography and High-Energy Phase-Contrast Nanotomography

Akihisa Takeuchi[1,*], Kentaro Uesugi[1], Masayuki Uesugi[1], Fumiyoshi Yoshinaka[2] and Takashi Nakamura[2]

[1.] Japan Synchrotron Radiation Research Institute (JASRI) / SPring-8, Sayo, Hyogo 679-5198, Japan
[2.] Hokkaido University, Kita 13, Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan
* Corresponding author, take@spring8.or.jp

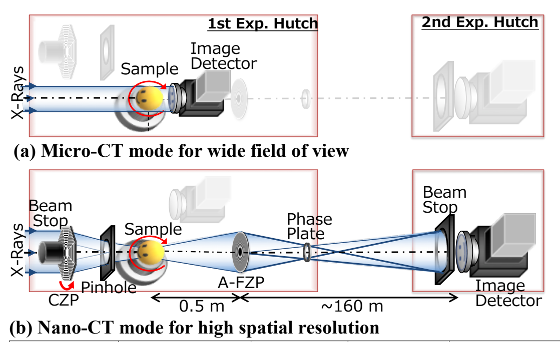
Multiscale tomography is a kind of three-dimensional (3D) imaging methods that combines multiple tomographic systems with different field of view and spatial resolution to perform a high spatial resolution measurement with a wide field of view. A wide-field and low-resolution (WL) system is used to capture entire object, and its region-of-interest (ROI) is precisely measured with a narrow-field and high-resolution (NH) system. In general, it is difficult to perform such measurement nondestructively because a probe is required to have two contradictory characteristics such as high-penetration to transmit a large object and high-interaction with matter for high-resolution and high-sensitivity. On the other hand, in the various fields such as metallic material, battery, and devices, nondestructive multiscale 3D/4D imaging is required in order to perform in-situ observation of bulky sample with mesoscale resolution. Here, we propose a nondestructive multiscale x-ray computed tomography (CT) consists of a x-ray micro-CT as a WL system using a simple projection optics and a newly developed phase-contrast high-energy x-ray nano-CT as a NH system. Its application example is also presented.

X-ray nano-CT based on a full-field x-ray microscope is now widely used for non-destructive 3D imaging with 100 nm or better spatial resolution. In most cases, Fresnel zone plate (FZP) is used as an objective. Its x-ray energy range is restricted to around 10 keV or lower because it is difficult to realize high efficiency at high energy region where high aspect ratio of zone structure is required. We have developed an apodization FZP (A-FZP) with which comparably high efficiency is obtainable at the high energy x-ray region [1]. Whereas conventional FZP has a uniform zone depth, A-FZP can be designed to have a deeper zone structure in the central region, that makes possible to realize relatively high efficiency at high-energy x-ray region. Using the A-FZP as an objective, a Zernike phase-contrast x-ray nano-CT for 20 keV x-rays has been developed at the Beamline 20XU of SPring-8 [2]. This system realizes both high transmittance to materials and high sensitivity by employing a phase contrast method at high energy x-ray region. Therefore, a high-resolution interior observation is available even for a millimeter-size bulk sample. It enables to realize a nondestructive multiscale-CT measurement by combining with a micro-CT.

Experimental setup of the multiscale-CT is shown in Fig. 1. X-ray energy of 20 keV is chosen with a double-crystal Si 111 monochromator. X-ray nano-CT used as a NH system is set across the 1st and 2nd experimental hutches. A hollow-cone illumination system using a condenser zone plate (CZP) [3], sample stages, an A-FZP objective, and a Zernike phase plate are installed at the 1st hutch. The A-FZP was fabricated at NTT Advanced Technology by using electron beam lithography technique. Tantalum zone patterns with the maximum depth of 2 µm was drawn on a Ru/SiC/SiN membrane with 2 µm thickness. Diameter of the A-FZP is 310 µm, outermost zone width is 100 nm, focal length at 20 keV is 500 mm, and measured diffraction efficiency at 20 keV is 9.1 %. A visible-light conversion type x-ray image detector (C12849-SY69701, Hamamatsu Photonics) which consists of a scintillator $Gd_2O_2S:Tb^{3+}$ 20 µm thickness, a fiber optical plate, and a scientific complementary metal-oxide semiconductor (sCMOS) is installed at the 2nd hutch located ~160 m downstream from the 1st hutch. The pixel size of the detector is 9.52 µm. Another x-ray camera, which consists of a scintillator $Lu_3Al_5O_{12}$: Ce^+ with 10 µm thickness, visible-light microscope objective (x10), and a sCMOS camera (C13440-20CU, Hamamatsu Photonics), can be installed between the sample and the A-FZP objective, to observe projection image of sample. Therefore, two measurement mode of the WL system (micro-CT) and the NH system (nano-CT) can be easily switched only by inserting and removing the hollow-cone illuminating system and the x-ray camera for micro-CT (see Figs. 1a and 1b). Typical parameters of these two systems are shown in Fig. 1c.

As a typical measurement example of the multiscale-CT, nondestructive observation of interior-originating fatigue cracks in a titanium alloy Ti-4Al-4V is shown in Fig. 2. Although Ti-4Al-4V has excellent mechanical properties, it may cause internal fatigue fracture in very high cycle exceeding 10^7 termed as very high cycle fatigue [4]. The origins and propagation processes of such cracks have not been revealed yet because of following three difficulties; (1) initial cracks are generated not from the surface but from the inside, (2) their very small length (a few tens of µm) and further smaller opening, and (3) sample must not be unnecessarily small, in other words, more than several hundreds of µm in diameter is required for sample in order to examine and to observe "internal" crack. In the experiment, entire body of a sample with 0.45 mm diameter was measured with micro-CT mode at first (Fig. 2a). Next, position of the internal crack was identified from the micro-CT data (circled region in Fig. 2a). Finally, the identified interior region was observed nondestructively with the nano-CT mode (Fig. 2b). Although the micro-CT data can be used for a precise reconstruction of nano-CT, simple filtered back-projection method is used here. One can find from the micro-CT image where the internal cracks are located, whereas the spatial resolution is insufficient to observe their detail. Nano-CT image clearly shows the 3D positional relations between the (α + β) dual phase microstructure and the initial cracks. It is also possible to reveal their propagation process by employing the in-situ measuring method.

[1] A. Takeuchi, et al., J. Synchrotron Rad. **24** (2017) 586.
[2] A. Takeuchi, in preparation.
[3] Y. Suzuki, et al., AIP Conf. Proc. **1365** (2011) 160.
[4] F. Yoshinaka, et al., Int. J. Fatigue **93** (2016) 397.
[5] The experiment has been done with the approval of JASRI (Proposal Nos. 2015B1404, 2015B1993, 2016B1459, 2017A1268, 2017B1407). This work was supported in part by RIKEN.



Mode	View field [µm]	Res. [µm]	Voxel [µm]	Pixel format
Micro-CT	1000	~2	0.5	2048 x 2048
Nano-CT	63	0.2	0.03	2048 x 2048

(c) Typical parameters of micro-CT and nano-CT

Figure. 1. Schematic drawing and typical parameters of multiscale-CT at BL20XU.

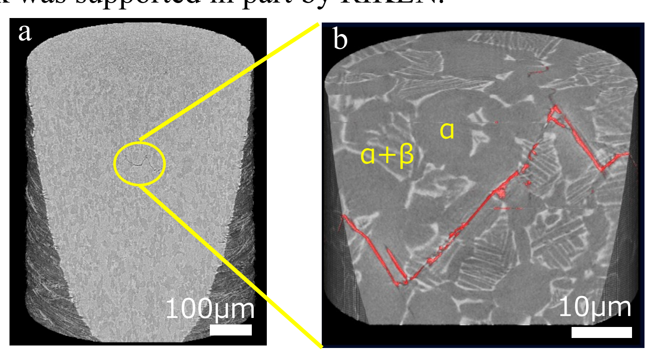


Figure. 2. Multiscale-CT images of interior fatigue cracks in Ti-4Al-4V. (a) Micro-CT image (200 ms exposure, 900 images/180 deg). (b) Nano-CT image of the circled region in Fig. 2a (2 s exposure, 3600 images/180 deg). X-ray energy is 20 keV.

https://doi.org/10.1017/S1431927618012928 Published online by Cambridge University Press