Sample handler for x-ray tomographic microscopy and image-guided failure assessment

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X-ray tomographic microscopy (XTM) yields a three-dimensional data model of an investigated specimen. XTM providing micrometer resolution requires synchrotron light, high resolution area detectors, and a precise sample handler. The sample handler has a height of 270 mm only, is usable for 1 μm resolution, and is able to carry loading machines with a weight of up to 20 kg. This allows exposing samples to load between scans for image-guided failure assessment. This system has been used in the XTM end station of the materials science beamline of the Swiss Light Source at the Paul Scherrer Institut. © 2005 American Institute of Physics. [DOI: 10.1063/1.1979475]

Usually systems for x-ray tomographic microscopy (XTM) are operating with a parallel beam without magnification and provide a spatial resolution between 1 and 2 μm . Special targets often determine the design of these experimental stations: For instance, the system at HASYLAB¹ supports phase contrast tomography based on a Bonse-Hart-type interferometer using a sample chamber filled with liquid. This makes a vertically flipped arrangement of the sample handler necessary. The high-throughput x-ray microtomography system at the APS² is operating with short scan times. Thermomechanical drifts are therefore negligible and the overall height of the sample handler is not critical. The system at Bessy II³ is dedicated to refraction enhanced microtomography. This requires the sample handler to be placed between two monochromator crystals. In fact this system at Bessy II is using the diffraction enhanced imaging (DEI) technique but the designers prefer to call it refraction enhanced. The XTM system at the Materials Science beamline of the Swiss Light Source (SLS) provides micrometer spatial resolution and is supporting *in situ* experiments such as image-guided failure assessment (IGFA).⁴ In the IGFA mode the system operates with the vertical rotation axis and meanwhile the horizontal rotation axis will be used with the Bragg magnifier detector⁵ or when elongated samples have to be scanned.

For tomographic imaging the sample handler has to provide movements according to the six degrees of freedom x, y, z and Φ_x, Φ_y, Φ_z (cf. Fig. 1). Adjustments of Φ_x and Φ_z fit the projection of the orbit of a sample mass point in a charge-coupled device (CCD) line. Movements in the x and y direction will center the sample in the beam. The sinogram will be taken by stepwise increasing Φ_y . The system has to provide 1 μm absolute and 1% relative resolution on its main translation axis x . The translation stages allow travels

of 50 mm and the stages for tilting adjustments should have tilting ranges of $\pm 2^\circ$. The load capacity of the vertical rotation stage has to be 5 kg of balanced load onto the rotor and 20 kg of balanced load onto the stator. The horizontal rotation stage has to bear 0.5 kg. The following specifications are derived from the basic resolution requirement: the positional stability and reproducibility of the rotation axis has to be $< 0.5 \mu\text{m}$ and the accuracy of a 5 mm translation movement $< 1 \mu\text{m}$. The acceptable variations of pitch and yaw depend on the Abbe levers, see Fig. 1. The rotation axes have to provide an accuracy of $< 0.02^\circ$ as well as concentricity and axial runout of $< 0.3 \mu\text{m}$.

FIG. 1. Orbit of a sample mass-point. The small roll, pitch, and yaw movements have an influence on the orbit depending on the distances h_r, h_t , and u . Small values of these distances allow larger tolerances for roll, pitch, and yaw movements.

FIG. 2. Design and construction of the sample handler. The plan (top) view shows the way how the total height could be kept low by placing two lifting gears laterally.

A tripod consisting of a fixed support and two lifting gear drives adjust the angles Φ_x, Φ_z adding less to the overall height than two goniometer cradles. The x and y stages mounted on the stage z have large openings in order to host the next stage, Φ_y being the topmost, as shown in Fig. 2. By this way an overall height of 270 mm is sufficient to integrate the air bearing with a length of 100 mm and the Abbe errors are kept as small as possible. The static air bearing meets the runout and wobble specifications of $0.3 \mu\text{m}$ and $5 \mu\text{rad}$. A directly coupled torque motor with a sensor having 0.001° resolution is driving the rotation axis. The translation stages are equipped with crossed-roller rails, spindles with ball screws, dc motors with gears and linear encoders with $0.1 \mu\text{m}$ resolution. The thermal drifts could also be reduced

FIG. 3. XTM system, consisting of beam (1), base plate (2), detector (3), sample handler (4), IMCSD device (5), shutter and slits (6).

TABLE I. Changes of pitch $\Delta\Phi_z$ and yaw $\Delta\Phi_y$, flatness f , straightness s , accuracy a , precision as standard deviation σ_x and largest deviation Δx .

	$\Delta\Phi_z$ (μrad)	$\Delta\Phi_y$ (μrad)	f (μm)	s (μm)	a (μm)	σ_x (μm)	Δx (μm)
Result	5	10	0.3	0.2	1	0.4	0.9
Goal	10	12	1	1	1	0.5	1

to a very low value by using ferritic steel with a thermal expansion coefficient of $11 \times 10^{-6}/\text{K}$. This design enables the mounting of an *in situ* mechanical compression and staining device (IMCSD) weighing 18 kg which is described elsewhere.⁶ Figure 3 shows the whole tomography system consisting of a high resolution detector,⁷ sample handler with IMCSD device, and other components in the experimental hutch.

Distance and angular measurements were made with a laser interferometer system consisting of laser HP 5519A, interferometer (beam divider) HP 10565B, and two retroreflectors, HP 10556A as well as with an Elcomat 2000 autocollimator. These measurements address the x translation stage when the projection image will be assembled by two partial images within a distance of 5 mm in order to get a 10 mm frame. The measurements cover a displacement range of 40 mm with 5 mm wide steps in both directions. The total number of steps was 45. Table I is presenting the results.

Figure 4 shows the results of the radial run-out measurement taken with a Precitec CHR 150 E instrument for contact-free scanning distances to a reference sphere. A sine wave of $0.9 \mu\text{m}$ amplitude was the best fit to the oscillations caused by the eccentric mounting of the sphere. The deviations from the fitted sine wave were less than $0.2 \mu\text{m}$. The axial run-out was measured in a similar way and was found to be less than $0.3 \mu\text{m}$. The wobble was not measured but it cannot be larger than radial run-out divided by the axial distance from the center of the bearing to the reference ball. This distance is 120 mm. So the wobble is below $5 \mu\text{rad}$.

The total random translation error as a geometric sum of position controller error ($0.3 \mu\text{m}$ for the first picture after in

FIG. 4. Plot of the radial run-out of the vertical rotation axis understood as the deviation of the measurements from the fitted sine wave. A sine wave fit compensates for the remaining eccentricity of the reference ball after being centered as good as possible with a centering head mounted on the rotating axis.

FIG. 5. Edge scan of a platinum bolt moved into and out of the beam. Movements of the sample into and out of the beam are needed during sinogram scan in order to get the actual flat intensity distribution of the image. The bar represents $10\ \mu\text{m}$. The pixel size in the image is $0.35\ \mu\text{m}$.

beam movement), the yaw related error ($0.4\ \mu\text{m}$) and the pitch related error ($0.2\ \mu\text{m}$) is $0.5\ \mu\text{m}$ and the linear sum of all error terms may be as high as $1\ \mu\text{m}$, which was confirmed by the interferometer measurements. Figure 5 demonstrates the effects on an x-ray scan of a $\varnothing 0.5\ \text{mm}$ bolt of platinum. The bolt was moved into the beam for one image and then moved out again for 40 times. The bolt was not turned, so that the shadow edge of the bolt in the scan should be a straight line. The standard deviation of the position error derived from the shadow edge scan is $0.6\ \mu\text{m}$ with noise taken into account. In a tomography scan the number of projections between two flat updates is usually about 50. The situation improves insofar as the position controller error becomes $<0.1\ \mu\text{m}$, due to feedback controller characteristics. The system is usable for resolutions of $1\ \mu\text{m}$.

In Fig. 6 a typical result from the IMCSD is presented: the development of damage in a trabecular bone sample that has been scanned and loaded between the scans. For the scans the photon energy was selected at $17.5\ \text{keV}$ and the nominal resolution in the image was $3.5\ \mu\text{m}$.

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FIG. 6. 3D representation of a bovine trabecular bone sample. Left: prior to mechanical compression. Right: after 1000 cycles of 2.5% compressive strain and subsequent static compression to 5%. The bar represents $500\ \mu\text{m}$.

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