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Analysis of vibration-based degradation of the spatial resolution of a nanometer-X-ray fluorescence analysis setup

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Abstract
The Physikalisch-Technische Bundesanstalt (PTB) operates its own laboratory at the Berlin Electron Storage Ring for Synchrotron Radiation (BESSY II) for the analysis of surfaces, solids, liquids, nano-layers and structures with respect to physical and chemical properties. An ultra-high vacuum measuring station is being used for reference-free X-ray spectrometry (XRS), which allows for a quantitative elemental analysis of different samples. This instrument can also be used for X-ray fluorescence (XRF) analysis with high spatial resolution in the nanometer range. However, different kind of perturbations in the vicinity of the measuring station can deteriorate the spatial resolution.

The presented work involves a frequency and amplitude-dependent analysis of the impact of vibrations on the spatial resolution of the nanometer X-ray fluorescence analysis (nm-XRF) setup of the PTB. The vibrations originating internal or external to the experimental setup were recorded by an acceleration sensor and the sources of vibration have been identified. Two complementary experiments were performed: two different nm-XRF instruments were characterized at the same site of the PTB PGM beamline at BESSY II and in addition, one of the nm-XRF instruments was characterized at the PETRA III P04 beamline.

Based on this new knowledge, constructional changes are foreseen to improve the experimental station of the PTB for future quantitative elemental analysis at the nanoscale.

Keywords: Finite element method (FEM), Metrology, Vibration, Analysis

1. Introduction

Element concentrations and chemical binding states can be investigated with high spatial resolution down to the nanoscale by using synchrotron radiation based scanning X-ray spectroscopy. The nanometer X-ray fluorescence analysis (nm-XRF) is one of the upcoming tools for a further development in relevant application fields such as life science and energy storage to enable experiments with high spatial resolution. The adaptation of traceable methodologies is important to quantify the elements in samples relevant for nanoelectronics and pharmaceuticals. PTB's vibration reduced nm-XRF setup can be used for transmission and fluorescence measurements. The fundamental idea of PTB's novel nm-XRF setup is to reduce independent vibrations by mounting everything on a single platform. The compact-setup can used in any experimental chamber at the sample holder position. The aim was to optimize the spatial resolution, which is currently at 100 nm[1].

2. Setup of the nm-XRF

The synchrotron radiation (SR) is diffracted in different orders by the zone plate (ZP). The first diffraction order is selected by the order sorting aperture (OSA). A silicon drift detector (SDD) is detecting the element-specific fluorescence radiation and a photodiode the transmitted radiation.

The experimental setup consists of an 8-axis piezomanipulator for the optics (ZP and OSA) and the transmission sample as well as a 4-axis piezomanipulator for the fluorescence samples. Both are attached to a mounting plate with a transfer system, which is installed in an ultra-high vacuum chamber of the PTB [2]. The 8-axis manipulator contains two translatory axes for the ZP and three translatory axes each for the order OSA and the transmission sample. Every piezo stage of the manipulators has a resolution of 1 nm and is encoder controlled.

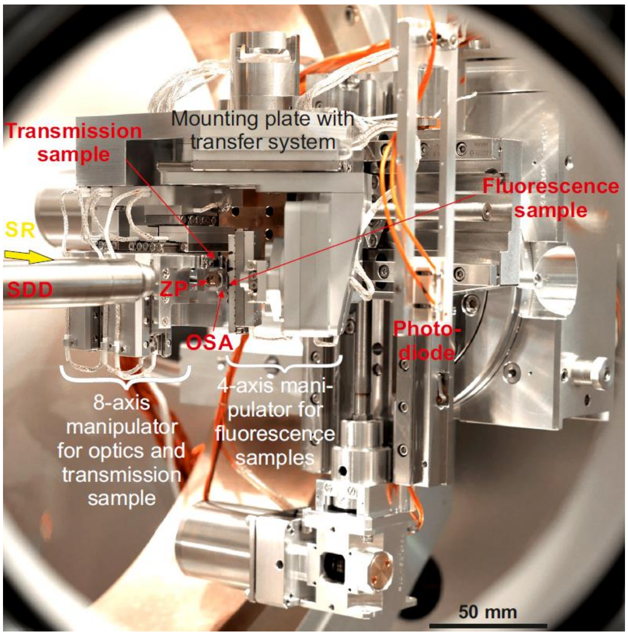

Figure 1. The nm-XRF setup installed in the UHV-chamber of the PTB[1,2]

3. Internal and external vibrations

To analyse the frequencies of the PTB experimental chamber, an accelerometer of PCB Synotech (PCB 356M132) was installed. The critical vibrations in x, y and z directions were determined with the fast Fourier transform (FFT) in python (black lines in Figure 2), which are the speeds of the turbopumps (820 Hz & 1000 Hz), the associated inlet pressure pumps (25 Hz & 50 Hz) and the vibrations in the ground.

Then the vibration was reduced by changing the vacuum pump system to a combination of non-evaporable getter and ion pump (NEXTorr2000 [3]) (red lines in Figure 2).

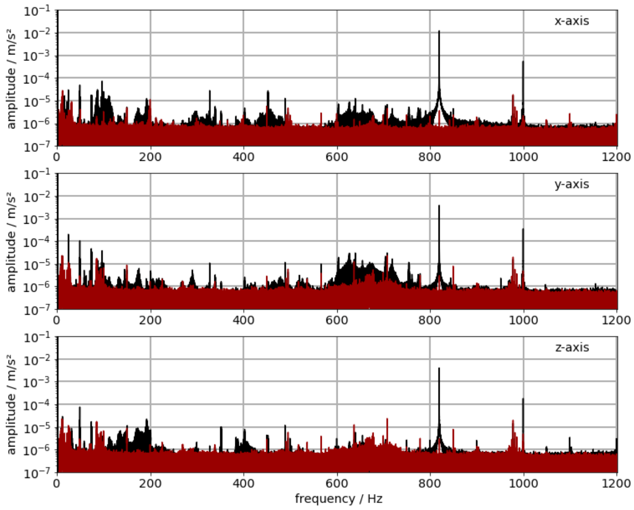

Figure 2. Frequency spectrum of PTB's chamber

With a second nm-XRF instrument [4], the vibrations at the PETRA III P04 beamline at DESY and at the PTB PGM beamline at BESSY II could be compared. In summary the ground vibrations at DESY are lower than at BESSY II, but are in general negligibly small compared to the internal perturbations. Any vibration in the vicinity of the chamber affects the measurement, which changes the FFT slightly, but not the critical vibrations.

3.1. Critical vibration analysis of the nm-XRF setup

To identify the characteristic frequencies (red lines in Figure 3), the nm-XRF setup was simulated with a simplified manipulator of the fluorescence samples in ANSYS which was validated by experimental modal analysis. The nm-XRF setup was mounted in a frame and placed on a foam mat. The acceleration sensor was installed on a holder of the transmission sample and a force excitation was performed with an impact hammer. The black lines in Figure 3 are the Frequency Response Function (FRF) of 10 measurements.

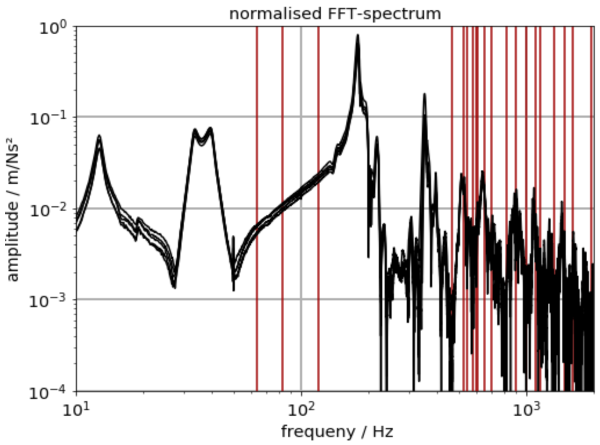

Figure 3. Simulated (red) eigenfrequencies and measured (black) FRF of the nm-XRF instrument in x-axis

The measured eigenfrequencies mostly correspond to the simulation. Unknown frequencies were also identified, which are probably caused by the 4-axis manipulator of the fluorescence samples, which is replaced in the simulation by a simple L-profile to reduce the calculation time.

3.2. Influence of the vibrations on the high spatial resolution

Three knife edge scans with the transmission sample were performed to determine the actual spatial resolution [5]. The first measurement was made with the turbopumps in operation and the second when they were turning off. The last scan was only performed by using the new vacuum system based upon a NEXTorr-pump.

Table 1 Spatial resolution by the different vacuum systems

Vaccum system	Spatial resolution / nm
Turbopumps	207
Turbopumps turning off	409
NEXTorr-pump	150

The spatial resolution was improved by using the NEXTorr pump.

4. Constructional changes of the nm-XRF setup

The ANSYS simulation shows different vibrations (natural frequencies and amplitudes) for the three spatial directions. This disproves the basic idea of the nm-XRF setup, because the OSA, ZP and samples vibrate differently to each other [1].

Based on this new knowledge a rectangular setup with a new positioning design of piezo stages was constructed with less eigenfrequencies and better damping of the external vibrations. The first ANSYS simulations confirm this hypothesis.

5. Summary and outlook

First of all, the UHV chamber of PTB was examined with regard to the vibrations. Next, the natural frequencies were determined by an ANSYS simulation of the nm-XRF setup. The eigenfrequencies were validated by experimental modal analysis. Subsequently, the nm-XRF setup was reconstructed, which was also investigated in ANSYS. The results show lower vibrations compared to the former design.

The new vacuum system reduces external vibrations and thus improves the spatial resolution

For better ANSYS simulations of the former setup, the complete 4-axis manipulator of the fluorescence samples must be imported to determine all natural frequencies. Next, a validation of the new designed nm-XRF setup with the experimental modal analysis has to be carried out and finally the new spatial resolution of the updated design has to be determined.

It is not possible to dampen the nm-XRF setup separately from the chamber of the PTB, but the vibrations of the ground can be reduced with suitable vibration isolation techniques. The characteristic frequencies of the chamber should be identified, because the eigenfrequencies affect the nm-XRF setup.

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