Development of scanning transmission ion microscopy computed tomography at Fudan microbeam line Postprint

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Abstract

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Full Text

Preamble

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Development of Scanning Transmission Ion Microscopy Computed Tomography at the Fudan Microbeam Line

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Abstract

Computed tomography was applied to STIM (Scanning Transmission Ion Microscopy) projections recorded at the Fudan Ion Beam Laboratory. In this work, to visualize the three-dimensional mass density distribution in several specimens, a test structure composed of a hollow gold cylinder was presented together with a detailed description of the developed system, including data reconstruction code (TomoRebuild 2) and image display software (AMIRA®). Future development will enable particle induced X-ray emission tomography for elemental analysis of micrometer-sized samples.

Key words: STIM-CT, TomoRebuild, Density distribution

Introduction

During recent years, particle induced X-ray emission (PIXE) and scanning transmission ion microscopy (STIM) microprobe analysis have been used to produce two-dimensional distributions of elements or mass density. These analytical methods can simultaneously measure all elements for total concentration and homogeneity. With the help of computed tomography (CT) techniques, energy loss data at many angles are acquired by rotating the sample, making it possible for STIM to determine the three-dimensional distribution of mass density and shape, as well as the internal structure of samples from a set of projections at different orientations [**?**]. CT development was initially driven primarily by medical applications [**?**]. In 1973, Godfrey Hounsfield presented the first medical scanner using X-rays (later he was awarded the Nobel Prize together with Allan Cormack) [**?**, **?**]. Because of the effective detection of transmitted ions and the inherently low beam current, the STIM-CT method is quantitative and relatively non-destructive, and provides mass normalization information for accurate elemental concentration determination with PIXE.

Recently, the micron beamline at the Fudan Nuclear Microprobe Laboratory has aimed to develop a micro-tomography system to provide important three-dimensional information of micrometer-sized samples [**?**]. The STIM tomography system has been implemented and will be expanded for PIXE tomography [**?**]. STIM-CT has become a versatile analytical technique in materials and life sciences [**?**]. In this paper, the system is described, and the STIM-CT characterization of a gold cylinder for three-dimensional density is presented, demonstrating its micro-tomography application in nuclear analysis techniques in China.

2 Experimental

The experiment was carried out at the Fudan Nuclear Microprobe Laboratory using a 3-MeV proton beam. An adjusted rectangular collimator is utilized as a configuration object, with another placed before the triplet quadrupole, 3.5 m away from the former, to prevent scattering. For these test experiments, the beam size in the focal plane is approximately 1- m diameter.

A windowless Si-PIN diode (Hamamatsu 1223-01) with a resolution of 25 keV at full width half-maximum (FWHM) is placed behind the sample holder, approximately 6 mm away from the sample [?], to measure the protons' residual energy [?] after traversing the sample at 0°. The detector suffers damage during the experiment since the protons are implanted into the crystal. As soon as the deviation of the measured proton energy in vacuum decreases, the detector is whirled to expose a fresh region [?]. A sample manipulator based on an x-y-z target stage (x, y: $±$12.5 mm, z: 0–50 mm, step: 5 m) with a steel needle is utilized as the rotation axis and attached to a PC-controlled magnetic rotary drive unit to correct the specimen position in case it moves out of the center view during specimen rotation at the beginning of data acquisition [?].

During the experiments, the projection angle of the tomography axis is controlled by a computer-controlled precision step motor capable of rotation in minimum steps of 0.05°. The control and data acquisition systems in the microprobe facility run on a PC under MS Windows 2000. The STIM detector, which is connected to OM1000, converts the analog signal to digital signal. The computer controls the scanning size of 2 mm × 2 mm with Oxford Microbeams Ltd. The data acquisition and 3D-scan analysis are fully automated. A schematic diagram of a STIM-CT experiment is shown in Fig. 1.

[Figure 1: see original paper]

Fig. 1 Schematic diagram of a STIM-CT experiment. The spot size of the incident beam at the specimen surface was adjusted to 2 m. The proton energy is measured by the Si-PIN detector.

A single hollow cylinder sample composed of gold was prepared for the experiment. This non-complex, well-characterized structure was chosen as a model for STIM-CT study due to its regular shape, good columnarity, and concentricity characteristics. The specimens, which were thin enough for transmission of 3 MeV protons, were mounted on top of a steel needle using cyanoacrylate adhesive or super glue, dried in air for approximately 12 h, and aligned along the vertical axis.

A 3D-STIM-tomography experiment consists of a number of 2D-STIM sample images, called projections, under different incident angles from 0–180° in the third dimension [?]. The current rate was adjusted to 1000–3000 Hz to avoid detector damage during analysis. Under these conditions, STIM-CT is considered a non-destructive technique [?]. The transmitted ions are collected by the detector and mechanically whirled at 0° on the incoming beam axis during acquisition [?]. The beam scan over the region under study is programmed at the beginning of the experiment. The sample was scanned over an area of 1800 m × 1800 m with a total of 128 horizontal slices, obtained from 128$×$128 pixels. The projections were recorded by horizontally scanning the beam over the sample from top to bottom [?]. Several rapid scans for every projection were performed at a speed of 10 s/pixel, with rotation controlled by a computer-controlled precision step motor. A total of 100 projections were recorded over a

180° angular range with 1.8° steps using 50 scans/projection. The total duration was approximately 5 h.

3 TomoRebuild Technique

Before data treatment, the experimental tomography data files from Fudan must be converted into the format of Centre d'Etudes Nucléaires de Bordeaux Gradignan (CENBG) using a specific procedure written in Fortran 90®. The TomoRebuild 2 data reduction program was used for data treatment. This code, developed at CENBG, processes STIM-CT data before reconstructing 2D or 3D tomography images. Based on the filtered back projection algorithm, this reconstruction is the most popular and least time-consuming technique for ideal data [**?**]. However, the reconstructed image may drastically deteriorate due to noise and misalignment problems in the raw data [**?**]. Details on how the TomoRebuild software operates can be found in Refs. [**?**, **?**].

The graphics software AMIRA® was used to display the 3D structure image of the sample by associating a color code with density values. The lowest density regions represent vacuum characteristic of the area surrounding the sample [**?**, **?**]. The specimen's outer surface was determined according to a threshold near low values. Several cutting planes are selected to visualize the internal structure at any orientation. Automatic calculations are carried out on selected materials to determine thickness, volume, and average density of the sample.

4 Results and Discussion

Figure 2 [Figure 2: see original paper] shows that the 3D structure of the target was reconstructed, and two iso-surfaces are displayed. The result shows there are many deviations from the ideal projection process [**?**]. The experimental conditions affect the ability to obtain an approximation closer to the ideal projection process, with specific ion beam characteristics being of concern.

(1) The energy dependence of the stopping power should be considered [**?**]. Described by the linear stopping power, the energy loss of ions passing through a specimen is related to the incident beam energy and is converted to the projected areal mass density along the trajectory prior to the reconstruction of the stopping power [**?**].

(2) Beam broadening within the specimen (lateral straggling) should be considered [**?**]. The resulting beam profile, which defines the interaction volume within the specimen, is a convolution of the incident beam profile and the broadening function due to multiple scattering with electrons in the specimen. It is important that the interaction volumes for each ion trajectory do not overlap; otherwise, the spatial resolution of the reconstructed specimen is degraded. For a given specimen, the incident energy and specimen thickness control the broadening of the proton beam [**?**]. A further effect, caused by the statistical nature of multiple interactions

between the ions and electrons of the material, is energy straggling. After interacting with matter [?, ?], a beam of defined energy has a Gaussian energy distribution.

The reconstruction of the corrected sinograms for the hollow cylinder is shown in Fig. 3. The density close to zero represents the limit characteristic between the sample and the surrounding vacuum. The highest densities distinguish the gold wall from the interior hollow region. The picture is strongly affected by artifacts represented as streaks outside the sample region. These artifacts originate from the insufficient number of projection angles in combination with the mean filtering of the energy loss values [?].

[Figure 3: see original paper]

Fig. 3 The reconstructed ion microtomography slice of the target from three orientations. (a) Axial orientation, (b) coronal orientation, and (c) sagittal orientation.

Figure 4 [Figure 4: see original paper] presents the possibility of stacking the reconstructed slices and performing three-dimensional tomography. An artificial cut was conducted along the symmetry axis to illustrate the object shape and its internal structure [?].

[Figure 4: see original paper]

Fig. 4 Target 3D image. An artificial cut along the symmetry axis illustrates the internal structure and the target wall surface.

5 Conclusion

This study on a hollow cylinder sample demonstrates the capability to perform 3D STIM-CT experiment setup in the micron beamline at the Fudan Nuclear Microprobe Laboratory. More accurate quantitative STIM tomography and rotation axis alignment with higher precision will be performed. Further, PIXE-T information will be conducted to characterize the elemental distributions within the sample and obtain a more accurate quantitative mass density determination.

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