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A new X-ray pinhole camera for energy dispersive X-ray fluorescence imaging with high-energy and high-spatial resolution

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ABSTRACT

A new X-ray pinhole camera for the Energy Dispersive X-ray Fluorescence (ED-XRF) imaging of materials with high-energy and high-spatial resolution, was designed and developed. It consists of a back-illuminated and deep depleted CCD detector (composed of 1024 × 1024 pixels with a lateral size of 13 μm) coupled to a 70 μm laser-drilled pinhole-collimator, positioned between the sample under analysis and the CCD. The X-ray pinhole camera works in a coaxial geometry allowing a wide range of magnification values.

The characteristic X-ray fluorescence is induced on the samples by irradiation with an external X-ray tube working at a maximum power of 100 W (50 kV and 2 mA operating conditions).

The spectroscopic capabilities of the X-ray pinhole camera were accurately investigated. Energy response and energy calibration of the CCD detector were determined by irradiating pure target-materials emitting characteristic X-rays in the energy working-domain of the system (between 3 keV and 30 keV).

Measurements were performed by using a multi-frame acquisition in single-photon counting. The characteristic X-ray spectra were obtained by an automated processing of the acquired images. The energy resolution measured at the Fe–Kα line is 157 eV.

The use of the X-ray pinhole camera for the 2D resolved elemental analysis was investigated by using reference-patterns of different materials and geometries. The possibility of the elemental mapping of samples up to an area of 3 × 3 cm^2 was demonstrated.

Finally, the spatial resolution of the pinhole camera was measured by analyzing the profile function of a sharp-edge. The spatial resolution determined at the magnification values of 3.2× and 0.8× (used as testing values) is about 90 μm and 190 μm respectively.

1. Introduction

In the last decade the interest for the non-destructive elemental analysis with high-spatial resolution has grown rapidly in a number of scientific disciplines and industrial applications [1–5].

The X-ray fluorescence (XRF) is distinguished among other analytical techniques for its capabilities in performing the non-destructive analysis of multi-elemental samples. Moreover, innovative micro-XRF spectrometers with a lateral resolutions of few tens of microns, were developed and applied for the determination of the elemental distribution of the samples [6,7].

Generally, the X-ray fluorescence micro-analysis is performed by using small-dimension beams obtained by focusing the primary radiation (emitted by an X-ray tube or delivered at a synchrotron facility) with advanced X-ray optics [6–8].

The scanning of the sample surface, through a series of local measurements, gives the possibility to determine the elemental distribution. The advantages in using this experimental approach were largely demonstrated, even if one limit can be found in the long measurement time necessary when large areas have to be investigated.

The recent development of X-ray pinhole cameras suited for the 2D resolved XRF analysis, represents a novel and promising technique in material studies [9–11].

The pinhole camera is a well-known imaging technique already used in different research works and applications [12–14]. In the case of the XRF imaging for the material analysis, it is based on the use of an external X-ray source (e.g. a tube) for inducing the X-ray fluorescence emission from the samples. The characteristic X-ray radiation is detected by means of a position- and energy-sensitive detector, through a pinhole-collimator positioned between the sample and the detector.

Because of the low-energy and low-spatial resolution available up to now, their use has been limited to few analytical cases.

Recently, a different imaging system (based on a 264 × 264 pixels pnCCD detector, equipped with X-ray polycapillary optics) was developed

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and tested for the elemental mapping of materials with high-energy and high-spatial resolution [15,16]. Some examples of its applications were also reported [15–17].

This work presents a new X-ray pinhole camera developed at the INFN-LNS laboratories (Istituto Nazionale di Fisica Nucleare — Laboratori Nazionali del Sud) in Catania (Italy).

A back-illuminated and deep depleted CCD camera is used as position- and energy-sensitive detector; it is composed of 1024 × 1024 pixels that allow the direct detection of X-rays in the energy range between about 3 keV and 30 keV. A 70 μm pinhole-collimator is placed between the sample and the CCD, allowing to perform the XRF imaging.

In the present set-up, the magnification of the X-ray pinhole camera (defined as the ratio between the CCD–pinhole and sample–pinhole distances) can be varied depending on the sample under investigation. Its range is mainly limited by the detection efficiency of the system.

Samples are irradiated by a medium-power X-ray tube. The use of a multi-frame acquisition in single-photon counting and an automated processing of the acquired frames, allowed to obtain the X-ray fluorescence spectra and the 2D elemental mapping. The energy resolution of the X-ray pinhole camera, measured at the Fe-Kα line, is 157 eV.

A spatial resolution of 90 μm and of 190 μm was measured at a magnification of 3.2× and 0.8× (the latter taken as test-values).

The experimental characterization of the X-ray pinhole camera (in terms of energy response, energy resolution and spatial resolution) as well as its capabilities in performing the 2D resolved XRF imaging of reference materials, are presented and discussed in the following sections.

2. The X-ray pinhole camera: equipment and experimental set-up

The X-ray pinhole camera installed at the LNS-INFN laboratories is shown in Fig. 1. The main components of the system are: the external X-ray source (a); the sample-holder equipped with XY micrometric stages (b); the pinhole-collimator (c); and the CCD detector (d).

The CCD, the pinhole-collimator and the sample are arranged in a coaxial geometry in a 50 cm long linear-guide. The samples can be moved along the linear-guide (Z direction) by using manual movements; also, the pinhole–CCD distance can be varied through a telescopic system between 10 mm and 80 mm. This set-up allows the easy framing of the samples and the proper selection of the magnification to be used during the measurements.

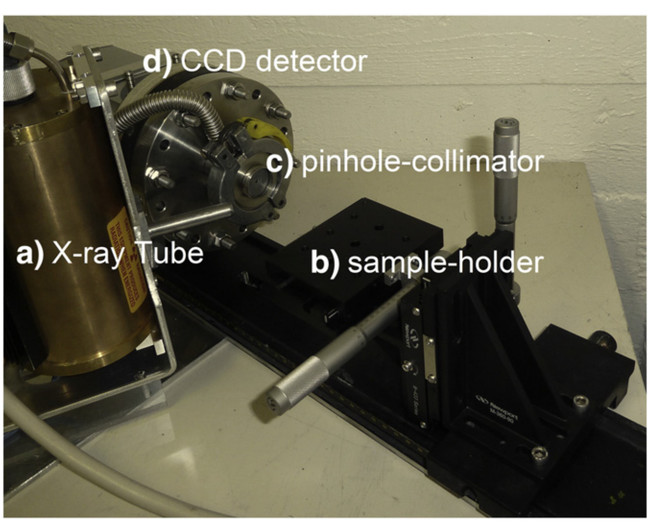

Fig. 1. The new X-ray pinhole camera.

The primary X-ray source for irradiating the samples is a W anode X-ray tube (Mod. Neptune produced by OXFORD INSTRUMENTS); it presents maximum working parameters of 50 kV and 2 mA (max. power = 100 W). The primary beam is collimated on the samples by means of lead collimators of different sizes: a 5 mm collimator is used at higher magnifications, where the area framed by the pinhole-camera is limited to few square millimeters; alternatively, a 20 mm Pb collimator is installed on the system.

In order to verify if the sample under analysis is homogeneously illuminated by the tube, a fluorescent screen (ORTHO 400 by CAWO) is placed at the sample's position at the beginning of each measurement.

Irradiation geometry of the X-ray source (i.e. angles and distances) is not fixed but it is determined by the position of the sample along the linear guide.

Samples are positioned in front of the pinhole camera by using a sample-holder allowing fine movements (the minimum step is 10 μm). This way a precise framing of the region to be investigated is obtained.

A 70 μm pinhole-collimator is axially placed between the sample and the CCD detector. The pinhole was obtained by a laser-drilling procedure on a 75 μm thick tungsten disk. Since the transmission of fluorescence and scattered X-ray radiation through the tungsten disk can produce blur effects on the images, the pinhole-collimator is included between two lead foils of 50 μm thickness and presenting a 500 μm central hole. This way transmission effects are strongly minimized.

A back-illuminated and deep depleted CCD camera (IKON-M by ANDOR) is used as position- and energy-sensitive detector; it consists of a 13.3 × 13.3 mm^2 chip divided into 1024 × 1024 pixels each presenting a lateral size of 13 × 13 μm^2. The thickness of each silicon pixel is 40 μm.

The CCD allows direct X-ray detection in the energy range between 0.2 and 30.0 keV. Quantum efficiency of the CCD camera is between 5% and 50% in the interval 0.2–1.0 keV; it increases up to 85–90% in the interval 2.0–10.0 keV; and finally it decreases down to 3% at 30.0 keV.

A removable 25 μm Be window, acting as light-tighter, is installed in the front of the CCD; this absorber strongly reduces the detection efficiency of the CCD at the lower energies (i.e. below 3 keV).

The CCD is equipped with a knife-edge sealed CF152 flange that allows in-vacuum operations. A thermo-electric platform operating on the CCD, allows its cooling under vacuum down to a temperature of −100 °C. This ensures a strong reduction of dark current that is reported to be 0.0012 e^-/pixel/s.

Since the lowest noise of the CCD camera ensures the best energy resolution, we maintain the CCD under vacuum during the measurements. This is obtained by interfacing the above CF152 flange with a vacuum component consisting of a cylindrical chamber 4 cm in diameter and 8 cm long. The separation-window between air and vacuum is an 8 μm Kapton foil.

Vacuum operations are performed by the use of a turbo molecular pump (HiPace 80 by Pfeiffer) suited for ultra high vacuum applications (down to 10^{-6}–10^{-7} mbar). A vacuum of 10^{-3} mbar (enough to cool the CCD chip) is obtained in about 1 min. When necessary, the CCD can work in air, cooled at a temperature of −45 °C. The dark current increases to a value of 0.09 e^-/pixel/s and the degradation of the energy resolution of the detector was measured to be about 20%.

All the acquisition parameters of the CCD are software-controlled. Exposure time can be fixed down to a minimum value of 10 μs. The readout process of the CCD is based on clocking values that can be fixed at 50 kHz and at 1, 3 and 5 MHz. Maximum operating frame rate is 112 fps (frames per second).

Finally, the 1024 × 1024 pixels can be binned up to 128 × 128 pixels. This way measurement time can be strongly reduced, even if the spatial resolution results slightly degraded.