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Application of polycapillary optics for dual energy spectroscopy based on a laboratory source

Yu. Cherepennikov^{a,*}, I. Miloichikova^a, A. Gogolev^a, S. Stuchebrov^a, D. Hampai^b, S. Dabagov^{b,c,d}, A. Liedl^{b,e}

^a Tomsk Polytechnic University, Lenin Avenue 30, Tomsk 634050, Russia

^b INFN Laboratori Nazionali di Frascati, Via E. Fermi 40, 00044 Frascati, Italy ^c Nuclear University MEPhI, Kashirskoye Shosse 31, Moscow 115409, Russia

^d RAS P.N. Lebedev Physical Institute, Lenin Avenue 53, Moscow 119991, Russia

^e Università di Roma 3, Dip. di Scienze, L.go San Leonardo Murialdo 1, 00146 Roma, Italy

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1. Introduction

Nowadays, X-ray absorption spectroscopy is widely used for X-ray structural analysis as well as for elemental analysis of a sample under investigation. A special group of absorption analysis methods implies measurements of the radiation absorption coefficients at several energy lines that allow estimating various characteristics of a test object. The most widely used methods are the so-called "dual" techniques, in which the analysis is carried out by two adjacent measurements. Similar methods are used in a number of applications, such as bed depth control of sandwich products, determination of component concentrations in multicomponent mixtures, acid concentrations in chemical productions, substance definition of analyzed objects, etc. [1-3] A priori knowledge about the test object, such as the number of layers or components, their linear attenuation coefficients, etc., requests for detailed examination of the objects that is typically related to the complexity of the measurements processing as well as of the results interpretation. Moreover, solving real technological problems based on utilization of the objects without well defined their characteristics becomes impossible.

* Corresponding author. *E-mail address:* che@scalpnet.ru (Yu. Cherepennikov).

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ABSTRACT

This paper describes experimental investigation aimed at the development of X-ray tube based source with a spectrum characterized by two different monochromatic lines (double-line spectrum). The modification of the tube spectrum to be a double-line one is suggested by means of a two crystals kit. The possibility to increase the beam intensity was experimentally studied via the use of a polycapillary semilens that collects divergent X-ray radiation from the source.

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Implementation of "dual" techniques requires X-ray beams with spectrum that contains two or more energy lines. Nowadays, radioactive isotopes are typically used for production of these beams. For another approach, X-ray tube spectra obtained at different values of voltage are used for "dual" measurements [1,2]. Both techniques have a number of advantages and disadvantages. First of all, radioactive sources might lead to the environmental risks and additional radiation dose for the personnel, even beyond the immediate operation time. On the other hand, the analysis of the experimental results is simplified due to the line spectrum. The advantages of X-ray tube approach include both higher radiation intensity and safety of the staff beyond the source operation time. However, the use of continuous spectrum radiation decreases the measurement accuracy, for example, because of the impossibility for precise recording the spectrum shape, as well as for distinguishing the scattered radiation registered by the detector. Intense X-ray beams with a double-line spectrum or monochromatic energy-tunable X-ray beams from conventional X-ray tubes might be used in "dual" technique measurements in order to improve the statistics of collected data (to increase the S/N ratio), to simplify the data processing and to provide the personnel radiation safety.

In this work we propose [4] the combination of X-ray tube and crystal monochromators, which reflect X-ray lines of different energies in one direction, to be used for production of double-line spectrum beams. This approach might be an alternative to

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the currently applied ones. In addition, the use of polycapillary optics has been suggested for getting X-ray radiation flux increased. The optics structure represents very precise shaped bundles of hollow inner cavities, i.e., capillaries, used as channels (or waveguides) for efficient radiation transmission [5,6]. Diffraction applications of polycapillary optics are topical issues well reported in many publications over the past 30 years, e.g. [7–15]. Application of a polycapillary semilens in the dual energy spectroscopy aims at the resonance enhancement for the final double-line spectrum due to the possibility to collect much more radiation from the source keeping rather small beam divergence.

2. Experimental setup

In the experiment, an Oxford X-ray tube (Apogee 5500 Series) with a molybdenum anode was used as X-ray source [16]. The voltage was fixed at 49 kV, while the current - at 0.9 mA. A quasiparallel beam was formed by polycapillary semilens with the input focal distance equals to 59 mm, while the output divergence counts 1.4 mrad. A lead mask with 3 mm hole diameter was installed in front of the entrance end to screen all useless radiation out of acceptance cone of the optics. An additional lead mask with 4 mm hole diameter was mounted in front of the silicon crystals on the axis of the beam. The hole diameters of input and output masks correspond to both entrance and exit semilens diameters, respectively. The second mask allows decreasing the beam divergence contribution to the results of measurements without semilens, which were performed to estimate the efficiency of polycapillary semilens use. In this case, the whole experimental geometry is similar to that shown in Fig. 1 but without the semilens 3 on the beam propagation axis.

The beam generated by X-ray tube (Fig. 2) hits the silicon crystals (100) and (111), which are set parallel and close to each other at some angle to the beam propagation axis. The crystals have circular shapes with a large (turned to the observer in Fig. 2) and a small (turned to the X-ray source in Fig. 2) edges. They are stuck in the kit by double-sided tape contacting along large edges and set in the holder that can move by motor SR50PP Newport (θ angle scan) [17]. Thus, both crystals can be rotated synchronously by the same angle by means of one motor. The advantage of this scheme that after the crystals kit there is only one diffracted beam with double-line spectrum while for two independently rotated crystals we deal with two beams reflected at different angles. Therefore, one does not have to find the crossing point of two beams to set the test object and detector. Thus the test object and detector can be moved freely along the direction of diffraction. In addition, a polycapillary semilens in this scheme provides equal paths of Xrays with both energies through the test medium in practical application.



Fig. 1. Experimental geometry: X-ray source (1); pinholes (2, 2'); polycapillary semilens (3); Si crystals (4); spectrometer XGL-SPCM-8100 (5); CCD camera (6).



Fig. 2. The picture of experimental setup.

The X-ray reflection by the crystal planes is investigated for a wide range of angles. The spectrometer XGL-SPCM-8100 with 5 mm² active area manufactured by XGLab is used to record the radiation [18]. The detector is set at about 30 degs to the initial X-ray beam axis, and it records X-ray radiation with energy up to 40 keV. The kit of crystals can be rotated defining the θ angular scan, while the detector is linearly moved as shown in Fig. 1.

Besides, the Photonic Science CCD-camera is placed behind the crystal along the initial beam propagation axis [19]. The CCD-camera is used to align semilens as well as to precisely set all the units of the optical scheme. In addition, the camera is used to reveal a "zero position" when the crystals are parallel to the beam (Fig. 3).

After alignment of the optical scheme, the scanning by the crystals-to-beam angle was performed. The position, in which one of the two diffraction lines corresponds to K_{α} (Mo)-line is determined experimentally. In the experimental scheme, large crystal edges are initially oriented to the beam, and radiation diffraction is expected on (111) and (400) planes of Si crystals. However, during scanning the correct position for diffraction from these planes was not defined that could be explained by the alignment problems. Nevertheless, a dual diffraction line is recorded when a large crystal edge oriented along the initial beam axis and the θ angle (marked in Fig. 1) is small enough and equal to \sim 1 deg. In this case, the beam hits a small side of the crystal (as seen in Fig. 2), and diffraction from other plane families becomes observable.

3. Results and discussions

In the experiment, a dual diffraction line was recorded at a small θ angle. The maximum intensity of diffraction for the Mo characteristic K_{\alpha}-radiation has been observed at θ = 0.59°



Fig. 3. X-ray beam image in a "zero position" of Si crystals recorded by the CCD-camera.

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