Observation of X-ray refraction contrast using multilayer mirrors with resonant absorption

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Abstract
X-ray refraction contrast in one-dimensional images of a copper wire, 75 μm in diameter, and a human hair was observed using a W/C multilayer mirror with resonant absorption at CuKα radiation. The multilayer structure consisting of 25 bilayers was designed for CuKα radiation so as to have narrow absorbing resonance at a grazing angle of 0.9°. A monochromatic probe X-ray beam with a divergence of approximately 7 arc s was obtained from a conventional X-ray tube and a double crystal monochromator set in a strongly dispersive configuration. This result proves the feasibility of X-ray refraction radiography using resonantly absorbing multilayer mirrors manufactured by conventional magnetron sputtering technology. © 2000 Published by Elsevier Science B.V.

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1. Introduction
This publication continues the discussion of the feasibility of X-ray refraction radiography using multilayer mirrors with resonant absorption initiated in the previous paper [1]. Conventional X-ray radiography is based on absorption contrast, i.e., on the variation of the absorption factor of different parts of a sample. Therefore, the smaller the dimensions of the details, the lesser is their contrast in the pictures obtained by absorption radiography methods. It is well known, however, that the refraction contrast originating from the variation of the refraction indices of different parts of a sample does not depend directly on the dimensions of the details, and, therefore, produces much better resolution in images of biological tissues. Today, a variety of imaging techniques for biological and medical investigations based on refraction contrast are being developed using both the coherent synchrotron radiation sources (see, e.g. Refs. [2,3], and citations therein), and laboratory X-ray sources [4,5]. In all these techniques, sometimes referred to as phase-contrast imaging, the commonly weak useful signal is detected against the background of a strong direct beam. Therefore, the detector shot noise produced by the direct beam decreases the signal-to-noise ratio, which is of a primary importance when an image is recorded electronically. Quite a different method based on depth-graded multilayer mirrors with...
narrow and deep absorption resonances was proposed in Ref. [1]. Here, the direct beam is suppressed by tuning the angle of incidence to a resonance, appearing like a dip in the reflectivity curve, while the refracted components of the transmitted beam slightly declined from their original direction are efficiently reflected by a mirror. The present short communication reports on the first successful experimental results displaying the practical feasibility of this technique.

2. The experimental setup

The scheme of the experiment is clear from Fig. 1. A standard 1.2 kW X-ray tube with a copper anode and long, fine focus was installed in a linear projection mode so as to form a linear X-ray source with visible dimensions of 4 mm in the horizontal plane and approximately 40 μm in the vertical plane. Estimations made in Ref. [1] show that the divergence of the probe beam must not exceed 10 arc seconds. Apart from the divergence, the spectral width of the probe beam also plays a significant role because the mirror’s resonant angle depends on the wavelength. The natural relative spectral width of the CuKα doublet including side wings is of the order of 4 × 10⁻³. Calculations show that such spectral widening increases the angular width of the resonance by a value of approximately 10 arc seconds. Therefore, the probe beam must not only have extremely small divergence, but also good spectral purity as well. This was achieved by coupling the source with a double crystal monochromator working in a strongly dispersive mode with both Si (111) reflections. According to Ref. [6], in this configuration, the horizontal divergence of the probe beam is equal to 7.4 arc seconds, while the relative spectral width is approximately 5 × 10⁻⁴, i.e., an order of magnitude less than the natural spectral width of the CuKα radiation. Thus, the effect of spectral widening can be neglected. The vertical divergence of the probe beam was of the order of several degrees.

The refracted (and, therefore, deflected from their original direction) components of the probe beam that passed through the object placed immediately after the monochromator were efficiently reflected by a specially designed depth-graded multilayer mirror positioned at a grazing angle of approximately 0.9° with respect to the probe beam, while the direct component was strongly attenuated. The W/C multilayer coating consisting of 25 bilayers was deposited onto a flat quartz substrate 40 × 40 mm² by means of the magnetron sputtering technique described earlier [7]. Its measured reflection curve is shown in Fig. 2 in comparison with the theoretical one, which is designed for the resonant angle of 0.9°. The angular width of the experimental reflection curve, measured with the monochromatic incident beam exiting from the double-crystal monochromator, is approximately twice the width of the theoretical reflection curve. The resonant angle of the experimental curve is shifted to a value of 0.846°, which may be due to an excess layer thickness of approximately 8% for both the tungsten and carbon layers relative to the calculated values. Measuring the difference between the experimental and theoretical values of the resonant angles makes it possible, in principle, to calculate corrections to the layers’ thickness and redo the mirror in order to obtain better results.

The output intensity distribution was measured in the mirror’s plane of incidence by a linear detector array developed at the Institute of Automatics and Electrometry (Siberian Branch of the Russian Academy of Sciences) in the laboratory of Prof. P.E. Tverdokhleb. It is composed of 2580 reverse biased silicon photodiodes with individual amplifiers. The geometry of its sensitive area is shown in Fig. 3. The sensitive array is assembled
on a silicon substrate which is cooled and stabilized to a temperature of approximately 5°C with a Peltier refrigerator. The dark current and gain differences between single photodiodes are compensated for by taking account of the reference values measured and stored beforehand in the computer memory. The isolation of a single sensitive element from the neighboring ones can be judged by Fig. 4, which shows the detector output signal only when a single element is irradiated through a narrow slit by CuKα radiation. It is important to be sure that the neighboring elements do not produce any effect which could be interpreted as the result of refraction contrast.

The signal-to-noise ratio was the crucial issue in the experiment. The inner noise of the detector has
two components: variations of the average current drain from different sensitive elements and shot noise. The first component can be compensated for by subtracting the average output noise signal stored in the computer before measurements from the real-time signal. The second one can be reduced by averaging the output signal over both the time and the number of realizations. The averaging time interval was limited by the charge leakage current, and in the experiment could not exceed 1 s. With an average photon flux at the detector of approximately 50 photons per second at a single element, a time interval of 1 s was insufficient to obtain a high enough signal-to-noise ratio. Therefore, additional averaging over 100 realizations was applied, so that the total duration of one measuring cycle was 100 s.

3. Observation procedure

The probe beam cannot be ideally uniform in its cross-section due to inevitable imperfections of both the collimating crystals and the multilayer mirror. Besides, spatial variation of the amplification of the detector's sensitive elements introduce additional distortion into the output signal distribution. In order to compensate for these effects, the following procedure was applied. The mirror was slightly mismatched from the resonant angle in order to obtain a sufficient detector output signal, and the object was inserted approximately into the middle of the probe beam as is shown in Fig. 1. Two types of objects were used in the experiment: a copper wire, 75 μm in diameter, and a human hair, approximately 60 μm in diameter. Despite weak absorption of X-rays in the objects, especially in the hair, their careful positioning was possible due to a high enough detector signal. Later, the mirror was adjusted to the resonant angle so as to minimize reflection, and the detector signal was averaged over 100 realizations and stored in the computer memory. Finally, the object was removed, and the detector signal was averaged over 100 realization with the subtraction of previously stored data. Thus, the data obtained in the experiment were of reverse sign compared to what could be expected during direct measurements.

A numerical simulation was performed prior to the experiments in order to estimate the possible refraction contrast that could be obtained using the multilayer mirror with the reflection curve shown in Fig. 2. For this purpose, the experimental reflection curve was interpolated in a narrow region around the resonance (shown by an arrow in Fig. 2), and this approximation was used in the numerical model. The finite spatial resolution of the detector was taken into account by dividing the image region into an appropriate number of elements, in which random traces were summed separately. From Fig. 4, it follows that the total width of such an element should be equal to approximately double the size of a single detector element, i.e., about 30 μm. The results of the numerical simulation are shown in Fig. 5. The common features can be learned from Fig. 5a, which presents a simulated signal for a copper

![Fig. 5. Simulated signals of refraction contrast for (a) a copper wire, 75 μm in diameter and (b) a human hair, 60 μm in diameter. Spatial resolution of the detector is equal to 30 μm.](image)
wire. The central peak marked by number 1 arises due to absorption, while two side peaks of the opposite sign marked by number 2 arise due to refraction. Fig. 5b presents a simulated signal for a human hair. The main chemical component of a human hair is a protein referred to as β-keratine (C₄₀H₅₆). In this case, absorption is much smaller; therefore, the central peak practically vanishes.

4. Results

The experimental results obtained with a copper wire are presented in Fig. 6a and b in two arbitrarily chosen realizations. The arrows show the regions of refraction contrast, which are qualitatively consistent with the numerical simulation. In order to prove that the marked regions really correspond to a refraction contrast, the multilayer mirror was removed from the probe beam, thus, leaving the optical path to the detector free (Fig. 6c). One can distinctly see the absence of side reflexes in this case. This mode of operation corresponds to traditional absorption radiography.

A human hair is a far less absorbing object with respect to a copper wire at CuKα radiation. Therefore, as can be seen from Fig. 7 obtained with a hair of 60 μm in diameter, the central peak, originating from absorption contrast, is much less

![Graph](image1)

![Graph](image2)

![Graph](image3)

Fig. 6. Detector signals obtained with a copper wire of 75 μm diameter in the refraction contrast mode (a,b) and (c) the absorption contrast mode.

![Graph](image4)

![Graph](image5)

![Graph](image6)

Fig. 7. Detector signals obtained with a human hair in the refraction contrast mode. (a,b) Two arbitrarily chosen realizations. The regions of refraction contrast are marked by arrows.
5. Conclusions

Many groups throughout the world are attempting to create an operating facility for X-ray refraction radiography using both synchrotron and laboratory-based sources [2–5]. A new approach in the development of a laboratory-based facility using multilayer mirrors with resonant absorption was introduced in Ref. [1]. Now, we have experimentally proved that it is possible to create such a mirror using the standard magnetron sputtering technique, to form a probe beam with the necessary divergence, and to observe as small an object as a human hair using a linear detector array.

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