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NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH Section A

X-ray fluorescence analysis of a crystal surface oriented at the Bragg angle to the exciting radiation beam

V.B. Baryshev *, N.I. Chkalo, V.I. Kondratyev, G.N. Kulipanov

Budker Institute of Nuclear Physics, 630090 Novosibirsk, Russian Federation

Abstract

It is shown experimentally that in X-ray fluorescence elemental analysis of impurities on the crystal surface, the signal-to-noise ratio can be increased if the crystal to be examined is oriented at the Bragg angle to the monochromatized exciting beam.

In near-surface crystal impurities analysis, the effect of increasing the signal-to-noise ratio in energy-dispersive XFA spectra may be observed not only in the total reflection mode, but also in the case when the crystal to be analyzed is positioned at the Bragg angle to the monochromatized exciting beam. This statement was experimentally checked using the SR beam from the VEPP-3 storage ring in the 11.8–18.5 keV energy range. By the background in this type of experiments we mean both the presence of an intense peak of Compton scattering in the XFA spectrum and the noises caused by incomplete collection of the charge produced by Compton photons in the detector.

The experimental arrangement is presented in Fig. 1. The flat Si(100) crystal served as a monochromator. Copper films of 16 and 51 Å thick, produced by laser deposition on 3-mm-thick LiF(002) crystals, were analyzed using X-ray fluorescent analysis with synchrotron radiation excitation (SRXFA technique). The experiment was performed at the SRXFA station described in detail in Ref. [1]. The LiF crystal was installed on a rotator allowing one to rotate it about the vertical axis near the Bragg angle at the chosen energy of exciting radiation with a step equal to 5 arc min. At every step, the XFA spectrum of the copper film and the Compton scattering of the exciting radiation in the LiF crystal was detected using a Si(Li) solid state detector.

The measurement scheme also includes a photomultiplier with a NaI scintillator in order to measure the intensities of the reflected and incident (in the absence of the LiF crystal) beams of exciting radiation, which made it possible to define the integral reflection coefficient.

We use the "quality coefficient" $K=(k_1/k_0)$, where k_1 is the ratio between the copper and Compton peaks in the SRXFA spectrum for the current angle of the LiF crystal to the SR beam, and k_0 is the copper/Compton peaks ratio for a Bragg angle plus 1°. Fig. 2 show the experimentally measured values of the quality coefficient K near the Bragg angle for two LiF crystals at an excitation energy of 18.35 keV. The difference between the two

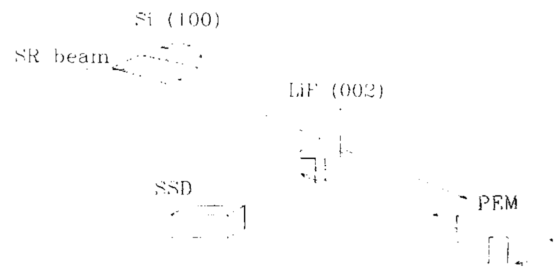

Fig. 1. The principal scheme of the experiment.

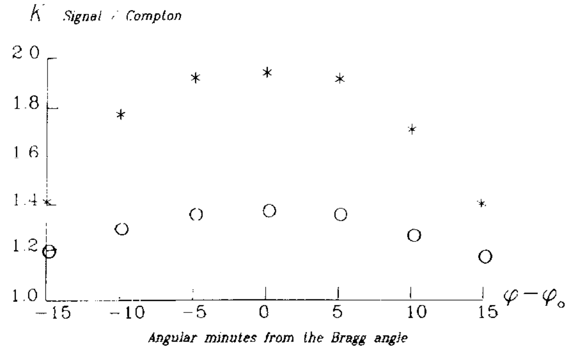

Fig. 2. The experimental values of the quality coefficient K near the Bragg angle for two LiF crystals at the 18.35 keV excitation energy. Here $\phi-\phi_0$ is the difference between the current and Bragg angles in arc min. * for crystal 1 (16 Å of Cu film); ○ for crystal 2 (51 Å of Cu film).

* Corresponding author.

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curves is caused by different coefficients of reflection of the LiF crystals (0.31 and 0.16 respectively).

The data obtained shows that when the crystal is located at the Bragg angle, the counting rate of the Cu-K-alpha fluorescence line becomes about $(1+k)$ times larger, whereas the intensity of the Compton scattering of the exciting radiation on the LiF crystal decreases proportionally to $(1-k)$, where k is the integral reflection coefficient of the crystal for the spectral-angular composition of the radiation coming from the monochromator. This is in accord with the simple model (see Fig. 3) in which the incident and reflected radiation beams excite X-ray fluorescence of a copper film on a surface, and only the non-reflected portion of the radiation is involved in Compton scattering inside the LiF crystal.

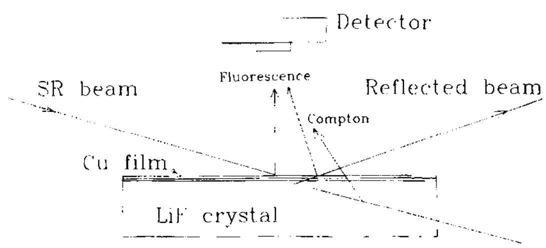

Fig. 3. The model accounting for the effect near the Bragg angle.

The signal-to-noise ratio in the XFA spectra for the cases of Bragg angle and grazing incidence measurements were experimentally compared. For the best of two LiF crystals, this ratio was increased by $K = 1.9$ at the Bragg angle, as compared to $K = 27$ in the case of grazing incidence, which is due to the low X-ray reflection coefficient ($k = 0.31$) of this material. However, the experimental results allow us to suppose that with the use of a narrow-band monochromator and perfect crystals and with a reflection coefficient close to 1, the signal-to-background ratio increase $K = (1+k)/(1-k)$ may be of importance for practical purposes.

Reference

[1] V.B. Baryshev, N.G. Gavrilov, A.V. Daryin, K.V. Zolotarev, G.N. Kulipanov, N.A. Mezentsev and Ya. V. Terekhov, Nucl. Instr. and Meth. A 282 (1989) 570.