Spectral resolution of x rays using diffraction focusing

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A principally new method of investigating x-ray spectra is demonstrated, using the diffraction focusing that occurs in a doubly diffracted beam of a U-shaped x ray interferometer. The spectral-line parameters were measured for the Mo$K_{\alpha_{1,2}}$ doublet as well as for the Mo$K_{\beta_{1,3}}$ doublet that is difficult to resolve with spectrometers of the ordinary type. The spectral width of the $K_{\alpha_{1,2}}$ lines agrees with the better results obtained with the aid of incomparably more complicated and cumbersome instruments, turns out to be somewhat less than the theoretical value.

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In 1973 one of us (V. L. Indenbom) lectured at the Erevan State University on the dynamic scattering of x-rays and advanced the idea of a new type of x-ray spectrometer, based on the use of the phenomenon of diffraction focusing of spectral lines. The theory of the effect was briefly described in[1] and can be illustrated by Fig. 1. Each narrow monochromatic beam, when diffracted in the first interferometer plate of thickness d, acquires a width $2d\tan\theta$, where θ is the Bragg angle. After a second diffraction in a second plate, according to the theory,[1] rather than expanding to $4d\tan\theta$, the beam experiences a diffraction compression and reconstructs the image of the initial narrow slit with accuracy $\Delta = (\Lambda/\pi)\tan\theta$, where Λ is the extinction length for the given reflection.

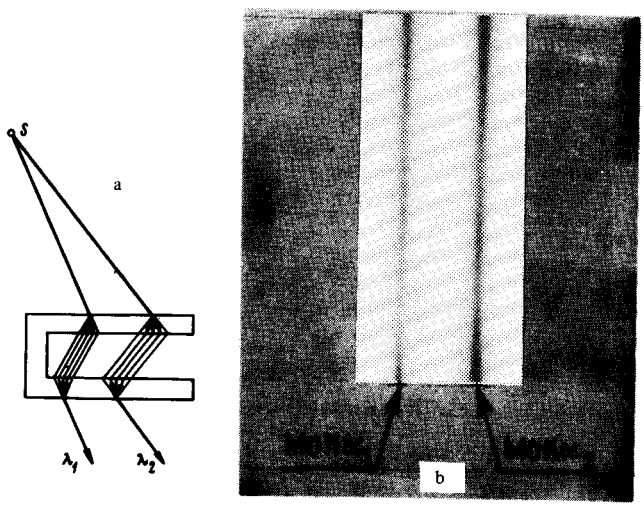

FIG. 1. Spectrometer with diffraction focusing of x-ray lines: a—experimental setup; b—x-ray photograph of Mo$K_{\alpha_{1,2}}$ doublet.

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Observation of the effect of diffraction focusing in experiment[2] and clarification of the factors that ensure the possibility of obtaining the theoretically predicted sharp focusing[3] have made possible a practical realization of the spectrometer with diffraction focusing.

The setup was assembled in accordance with the scheme of Fig. 1a, except that the first slit was missing, inasmuch as the radiation source was the microfocus tube D-4C of the Rigaku-Denki firm with a focal-spot projection on the order of 3 μ. The U-shaped interferometer was cut from a dislocation-free single crystal of silicon and mounted on the table of the A-3 camera. The plates were usually cut along the (111) plane, perpendicular to the growth axis of the ingots. The plate thicknesses were equalized with accuracy 1—2 μ by mechanical and chemical polishing. The reflecting planes were (220) and (224). The diffraction broadening in the region of the Mo K_α radiation was 1.4 and 3.5 μ, respectively, while the calculated resolution of the spectrometer reached, according to[1], 1.2×10^{-5} and 1.7×10^{-5} Å. The spectrograms were registered with type IK photographic plates, and the microphotometry was with the aid of a Carl Zeiss G-II recording microdensitometer.

Figure 1b shows a photograph of the doublet Mo$K_{\alpha_{1,2}}$ for the (220) reflection. The distance between the doublet lines (0.48 mm) corresponds to the calculated spectral intervals between the lines K_{α_1} and K_{α_2} ($\lambda_2-\lambda_1=4.28\times10^{-3}$ Å). It is seen from the microgram of Fig. 2a that the half-width of the photographic image of the doublet lines is approximately 0.15×10^{-3} Å, which is noticeably less than the data obtained on the natural widths of these lines with the aid of two-crystal spectrometers ($\delta\lambda=(0.24—0.29)\times10^{-3}$ Å for Mo K_{α_2}).[6]

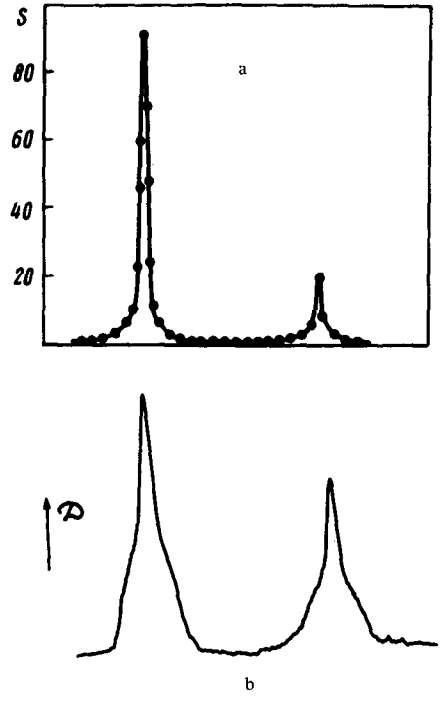

FIG. 2. Line shapes of the Mo$K_{\alpha_{1,2}}$ doublet; a—plot of the photographic image; b—the same for the optical image density corresponding to the x-ray intensity.

Account must be taken, however, of the nonlinearity of the photogrammetric curve. Figure 2b shows a plot of the $MoK_{\alpha_{1,2}}$ doublet recalculated into optical density $D=\log(1-S)$, which corresponds approximately to the x-irradiation density. The recalculation reveals clearly the pedestal of the central peak, and the heights of the peak and of the pedestal turn out to be comparable. It appears that the line shape is influenced also by the dimension of the x-ray tube focus and by the sphericity of the wave reaching the crystal surface. (Estimates show that the two indicated effects make an additional contribution of about 3 μ each to the line broadening).

Subtraction of the pedestal yields the line shape shown in Fig. 3a. The half-width of the MoK_{α_1} line amounts here to 0.24×10^{-3} Å, and that of the MoK_{α_2} line to 0.23×10^{-3} Å, corresponding to the best results obtained with the aid of the incomparably more cumbersome and complicated installations. A theoretical calculation of the x-ray term widths, unfortunately, does not give the required accuracy. According to the latest data[7] the summary broadening (both radiative and that due to Auger transition) of the K, L_2, and L_3 terms of Mo ($Z=42$) should be 4.29, 2.60, and 2.37 eV, respectively.[7] This yields for the MoK_{α_1} line ($K\rightarrow L_3$ transition) a half-width 6.66 eV or $\delta\lambda=0.27\times10^{-3}$ Å and for MoK_{α_2} ($K\rightarrow L_2$ transition) a half-width 6.89 eV or $\delta\lambda=0.28\times10^{-3}$ Å, which exceed the experimental data, both those cited in the literature and those obtained in the present study.

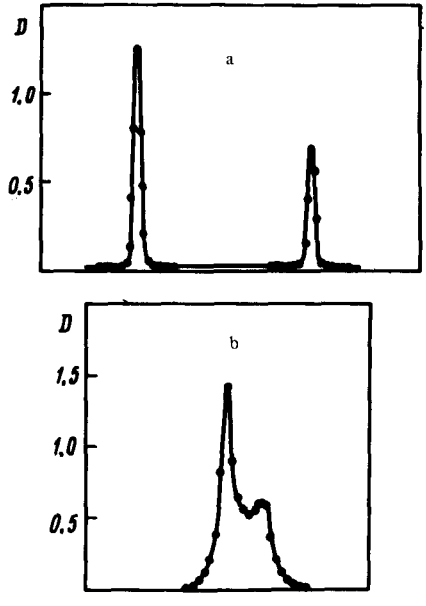

FIG. 3. Line shape after subtraction of the background: a—of the $MoK_{\alpha_{1,2}}$ doublet; b—of the $MoK_{\beta_{1,3}}$ doublet.

The high resolution of the spectrometer with diffraction focusing can be demonstrated with the observation of the $MoK_{\beta_{1,3}}$ doublet as an example. The tabulated distance between the lines amounts in this case to only 0.58×10^{-3} Å, while the theoretical line half width reaches 7 eV or 0.23×10^{-3} Å ($K\rightarrow M_{1,2}$ transitions; the total width of the M term is 2.7 eV).[7] As seen from Fig. 3b,

spectrometer with diffraction focusing, even at small dimensions that make it fficult to suppress the effects of spherical aberration and the diffusion of the cal spot, separates the $MoK_{\beta_{1,3}}$ doublet quite well. The distance between lines approximately 0.57×10^{-3} Å, and the half-width of the lines (after subtracting e background and taking the line overlap into account) is of the order of 0.23 10^{-3} Å, which agrees with the theoretical estimate.

The results demonstrate clearly the possibility of realizing in practice a ectrometer with diffraction focusing of the x-ray spectral lines, the high solution of the instrument, and its advantages over x-ray spectrometers of e usual type, both as applied to the solution of ordinary problems (for exam- e, problems of x-ray spectral analysis) and as applied to extending the range applicability of x-ray spectrometers the construction of small-size auto- atic spectrometers, or to the contrary of large-size high-resolution spec- ometers that make it possible to investigate the influence of various factors the shape, width, splitting, and shift of x-ray spectral lines.

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The callouts of the spectral lines $K\alpha_1$ and $K\alpha_2$ in Fig. 1(b) should be interchanged.