Tubes For X-Ray Structure Analysis

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In this work considered the basis of the method of measuring X-ray diffraction analysis and the properties of soft X-ray radiation. Shown the practical problems of the Wulf-Bragg relation and studied the properties and operation of the X-ray diffraction tube.

Keywords: X-ray diffraction analysis, tube, target, soft X-rays, K-lines

X-ray diffraction analysis is based on the study of the diffraction pattern that occurs when X-rays interact with crystals. The structure of the diffraction pattern depends on the structure of the object under study. Since the atoms in crystalline bodies are arranged regularly, in certain order, forming spatial crystal lattice, where it acts on X-rays as kind of diffraction grating.

When soft X-rays pass through a crystal, the lattice nodes become sources of scattered radiation of the same wavelength as the incident one. There is coherent scattering of rays. The interference of waves scattered by individual nodes leads to the appearance of diffraction maxima in certain directions. In other directions, the scattered waves cancel each other out.

As professor of Moscow University G. V. Vulf and English physicist V. L. Bragg showed in their works, X-ray diffraction in crystal can be considered as a reflection of rays from the planes of spatial crystal lattice. Let us consider a family of atomic planes of a crystal (Fig. 1), the distance between which is the same and equal to *d*.

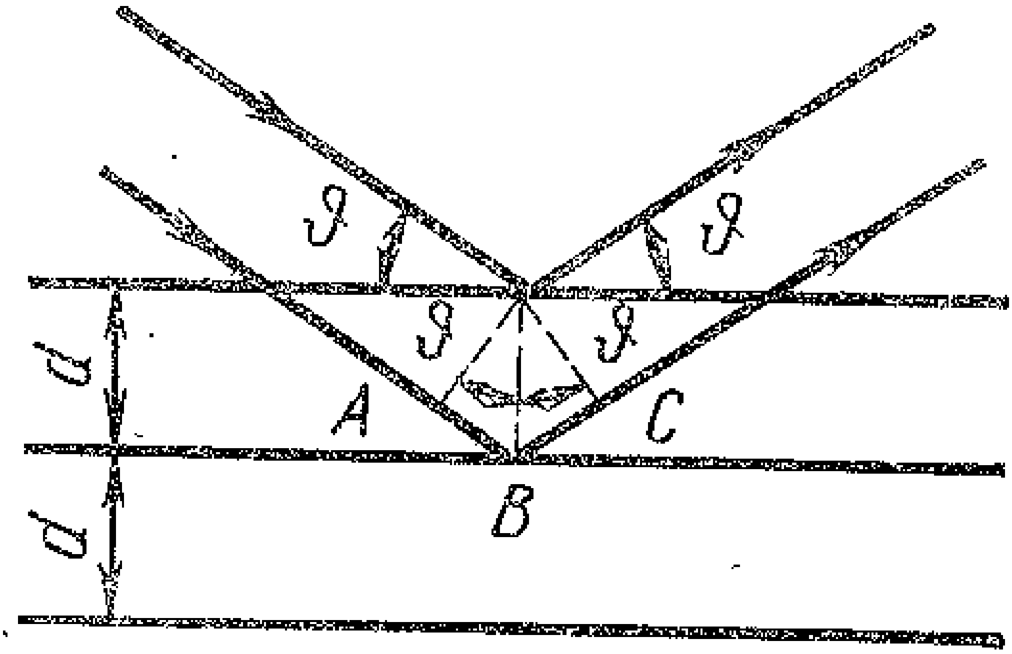

Fig. 1. - Reflection of X-rays by the atomic planes of the crystal.

A beam of X-rays with wavelength λ falls on the crystal at an angle ϑ. The rays experience mirror reflection from large number of atomic planes; reflected rays interfere. They will reinforce each other if the difference between their paths is equal to an integer number n of wavelengths. From fig. 1 it can be seen that the difference in the path of rays reflected from neighboring atomic planes is equal to the sum of the segments

$$AB + BC = 2d\sin\vartheta$$

Consequently, the mutual amplification of the rays, i.e., the formation of diffraction maximum, will occur under the condition

$$2d\ \sin\vartheta = n\lambda,$$

where n=1, 2, 3 ... is the order of reflection.

This relation is called the Wulf-Bragg equation.

It follows from the equation that monochromatic X-rays are reflected from the crystal not at any, but only at quite certain angles of incidence ϑ. At n=1, reflection of the first, n=2, second, etc. orders is observed. They correspond to the angles $\vartheta_1, \vartheta_2, \vartheta_3, \ldots$, and $\vartheta_1 < \vartheta_2 < \vartheta_3$, ...

Based on the Wulf-Bragg equation, two important practical problems can be solved:

1) determine the interplanar distance d of the crystal at a known wavelength λ (this is the purpose of X-ray diffraction analysis);

2) find the wavelength λ with known d (this is the goal of X-ray spectral analysis).

Most X-ray analysis methods require monochromatic radiation. Usually, almost uniform characteristic K-radiation of an X-ray tube or its K_α-line is used, which is isolated using a special filter.

X-ray diffraction analysis is based, as was pointed out, on the phenomenon of coherent scattering of rays by the object under study. Only soft rays experience such scattering.

Therefore, the characteristic radiation of the X-ray diffraction tube must be soft. It can be obtained if the target of the device is made of material with a relatively low atomic number. Typically, targets are made from the following metals: 24Cr, 26Fe, 27Co, 28Ni, 29Cu, 42Mo, 47Ag (the numbers in front of each chemical symbol indicate the atomic number of the corresponding element).

The quality of the radiograph depends on the degree of homogeneity of the radiation used. Since the characteristic beams consist of a number of wavelengths and always appear against the background of the bremsstrahlung spectrum, the homogeneity of the beam obtained from an X-ray diffraction tube may be insufficient for solving a number of problems. To improve the monochromaticity of the beam, the K_α-line of characteristic radiation is isolated from its spectrum. A beam of rays from the tube is passed through a selective absorbing filter. The filter is made from material whose atomic number is one less than the atomic number of the tube target material. Under this condition, the K-jump in the attenuation coefficient μ_f - of the filter material is located between the K_β- and K_α-lines of the spectrum (Fig. 2).

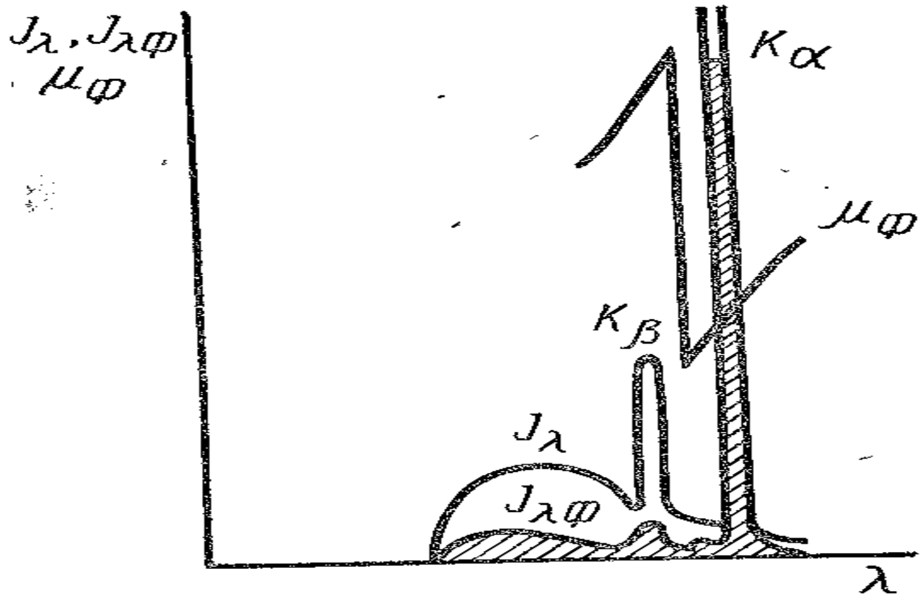

Fig. 2. Isolation of the K_α-line of the characteristic radiation of the tube using filter.

As a result, the intensity of the K_β peak will be attenuated by the filter to a large extent, and the K_α peak will be relatively weak. Simultaneously, the intensity of the bremsstrahlung spectrum also decreases significantly. Therefore, the beam passing through the filter turns out to be more homogeneous in composition than the initial one: it contains mainly the K_a-line of the characteristic radiation of the tube.

The anode of the tubes for X-ray diffraction analysis is grounded and cooled with running water directly from the water supply. The anode target is placed at an angle of 90° to the instrument axis. Such an arrangement of the target makes it possible to use several working beams simultaneously, i.e., to take X-ray diffraction patterns of a number of samples at once, in one step. Long-wavelength characteristic radiation is emitted from the tube through thin beryllium windows, the number of which is usually two, three or four. The axis of the working beams passing through the windows makes an angle of 3-6° with the target plane.

In order to reduce the time of taking X-ray images, the tubes are operated at the maximum allowable power, the value of which depends (ceteris paribus) on the target material.

In x-ray diffraction analysis, the beam of rays acting directly on the object under study is narrow, since it first passes through diaphragms with a small (0.3–1.0 mm) aperture or narrow slits. If the size of the focal spot of the tube is much larger than the size of the slit, then the beam intensity turns out to be low, since only radiation from a small focus area is used. To obtain high beam intensity, it is necessary to have sharp focus. Tubes for X-ray diffraction analysis are usually made with a linear focus. The dimensions of the actual focal spot are on average 1.0x10 mm, which makes it possible to obtain an effective spot at an angle of 6° in the form of a square with a side of 1 mm.

When the tube for X-ray diffraction analysis is used for long time, foreign materials can be deposited on the target surface, the characteristic radiation of which often has wavelengths close to the wavelengths of the main radiation. As a result, as they say, the emission spectrum of the tube is contaminated with lines of foreign elements. For example, there may be contamination of the spectrum by the L-lines of tungsten evaporating from the filament. The presence of extraneous lines in the characteristic radiation spectrum of the tube leads to complication and errors in the interpretation of X-ray patterns.

The degree of spectrum pollution can be characterized by the following parameters:

$$\eta_1 = J_1/J_{K_a} \ , \eta_2 = J_2/J_{K_a}, \qquad \eta_m = J_m/J_{K_a} \ ,$$

where J_1, J_2, J_m- is the intensity of the lines of foreign elements in the spectrum of the characteristic radiation of the tube; $J_{K\alpha}$-intensity, K_α-lines of the characteristic radiation of the target material.

The largest (at the moment) of the parameters in this series is taken as the value of the degree of pollution η. Over time, the degree of contamination increases and may exceed the permissible value, which is assumed to be 2% for pipes of domestic production.

In order to reduce the rate of contamination, the filament in the cathodes of modern X-ray tubes is made of tinted tungsten carbide. Such cathodes have lower operating temperature and lower evaporation than pure tungsten cathodes.

However, the degree of contamination of the radiation spectrum depends not only on the temperature of the cathode, but also on the pressure of the residual gases in the device. During the operation of the tubes, the gas is ionized by thermo electrons and secondary electrons. Bombardment of the cathode assembly with positive ions formed leads to sputtering of its materials and their condensation on the target surface. As a result, lines of iron, tungsten and other elements appear in the spectrum. In tubes with insufficiently high vacuum, intense contamination of the spectrum by extraneous lines occurs already in the first 100-150 hours of continuous operation. A getter is used to improve the vacuum in the tubes.

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