International Tables for Crystallography (2006). Vol. C, Section 4.2.1.3, pp. 193–195.

$$\eta_c = bZE_0/2. \qquad (4.2.1.9)$$

Crystallographers are more accustomed to thinking of the spectrum in terms of wavelength. Equation (4.2.1.7) can be transformed into

$$dN_\lambda = hcbZ(1/\lambda^2 - 1/\lambda\lambda_0)\, d\lambda, \qquad (4.2.1.10)$$

which has a maximum at $\lambda = 2\lambda_0$. In practice, the *emerging* spectrum is modified by target absorption, which is greatest for the longer wavelengths and moves the maximum more nearly to $1.5\lambda_0$.

It is of interest to compare the X-ray flux in a narrow wavelength band selected by an appropriate monochromator with the flux in a characteristic spectral line, in order to examine the practicability of XAFS (X-ray absorption fine-structure spectroscopy) or optimized anomalous-dispersion diffractometry experiments. For these purposes, the maximum permissible wavelength band is about 10^{-3} Å. From equation (4.2.1.10), we see that, for a tungsten-target X-ray tube operated at 80 kV, dN_λ is about 1.1×10^{-5} photons with the $K\alpha$ energy electron^{-1} steradian^{-1} $(10^{-3}\,\delta\lambda/\lambda)^{-1}$ for an X-ray wavelength in the neighbourhood of 1.5 Å. By comparison, from equation (4.2.1.2), a copper-target tube operated at 40 kV produces about 5×10^{-4} $K\alpha$ photons electron^{-1} steradian^{-1}. In spite of this shortcoming by a factor of about 45, laboratory XAFS experiments are sufficiently common to have merited at least one specialized conference (Stern, 1980; see also Tohji, Udagawa, Kawasaki & Masuda, 1983; Sakurai, 1993; Sakurai & Sakurai, 1994).

The use of continuous radiation for diffraction experiments is complicated by the fact that the radiation is polarized. The degree of polarization may be defined as

$$p = (I_\parallel - I_\perp)/(I_\parallel + I_\perp), \qquad (4.2.1.11)$$

where $I_\parallel$ and $I_\perp$ are the intensities of radiation with the electric vector parallel and perpendicular to the plane containing the incident electrons and the direction of the emitted photons. For an angle of $\pi/2$ between the electrons and the emitted beam, p varies smoothly through the spectrum; it is negative for the softest radiation, approximately zero at $\nu/\nu_0 \sim 0.1$ and reaches values between +0.7 and +0.9 near the Duane–Hunt limit (Kirkpatrick & Wiedmann, 1945). Since practical use of white radiation is likely to be in the vicinity of $\nu/\nu_0 \sim 0.1$, the effect is not a large one.

It should also be noted that the spatial distribution of the white spectrum, even after correction for absorption in the target, is not isotropic. The intensity has a maximum at about 50° to the electron beam and non-zero minima at 0 and 180° to that beam (Stephenson, 1957).

4.2.1.3. *X-ray tubes*

The commonest source of X-rays is the high-vacuum, or Coolidge, X-ray tube, which may be either demountable and pumped continuously when in operation or permanently sealed after evacuation. The vacuum tube contains an electron gun that incorporates a thermionic cathode, which produces a well defined electron beam that is accelerated towards the anode or target, formerly often called the anticathode. In most X-ray tubes intended for crystallographic purposes, the anode is massive, *i.e.* its thickness is large compared with the range of the electrons; it is usually water-cooled and its surface is normal to the incident electron beam. Usually, it is desirable for the X-ray source to be small (between 25 µm and 1 mm square) and for the X-ray intensity from the tube to be the maximum possible for the amount of power that can be dissipated in the target. These objectives are best achieved by

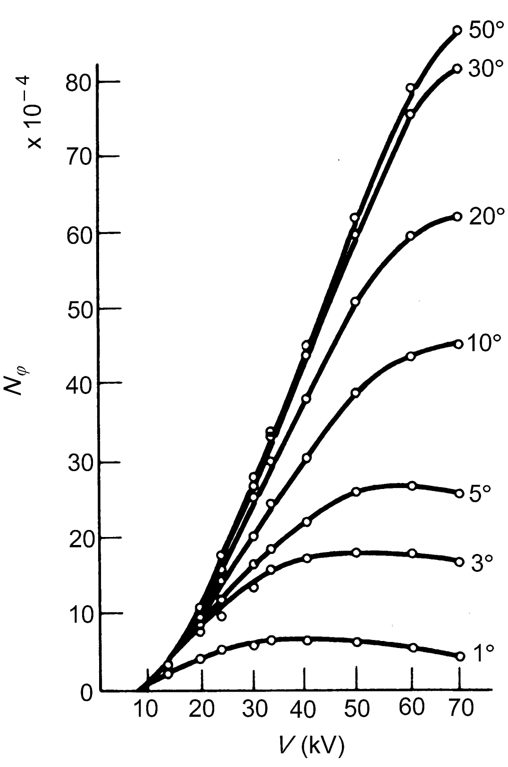

Fig. 4.2.1.2. Experimental measurements of N_φ for Cu K-L_3 as functions of the accelerating voltage for different take-off angles. From Metchnik & Tomlin (1963).

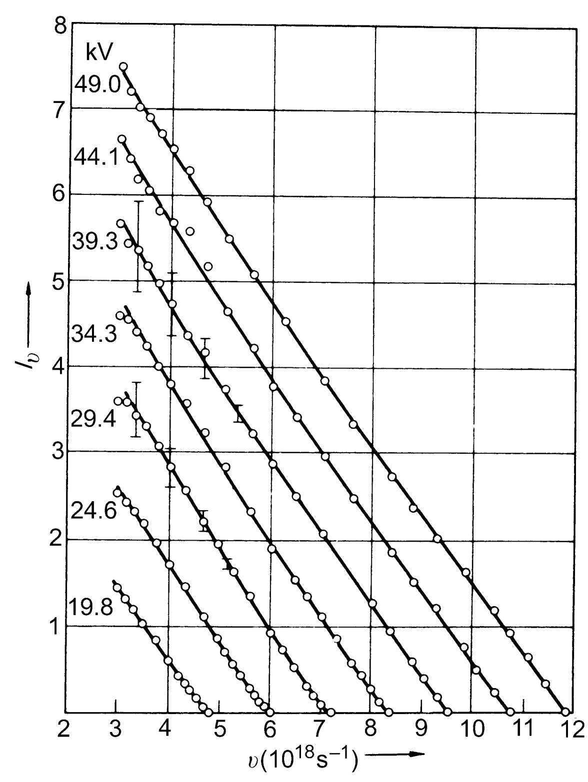

Fig. 4.2.1.3. Intensity per unit frequency interval *versus* frequency in the continuous spectrum from a thick target at different accelerating voltages. From Kuhlenkampff & Schmidt (1943).

Table 4.2.1.3. *Copper-target X-ray tubes and their loading*

X-ray tube	Anode diameter (mm)	Speed		$f_1 \times f_2$ (mm) (mm)	μ	Loading (kW)		Recommended specific loading (kW mm^{-2})
		r min^{-1}	mm s^{-1}			calc.	recommended	
Standard insert	–	–	–	8× 0.15	0.295	1.0	0.8	0.67
	–	–	–	8× 0.4	0.359	1.2	1.5	0.47
	–	–	–	10× 1.0	0.425	1.8	2.0	0.20
	–	–	–	12× 2.0	0.493	2.5	2.7	0.11
AEI-GX21	89	6000	28000	1× 0.1	0.425	1.4	1.2	12.0
				2× 0.2	0.425	3.95	3.2	8.0
				3× 0.3	0.425	7.3	5.2	5.8
				5× 0.5	0.425	15.6	15.0	6.0
AEI-GX13	457	4500	108000	1× 0.1	0.425	2.7	2.7	27.0
Rigaku-RU200	99	6000	31000	1× 0.1	0.425	1.5	1.2	12.0
				2× 0.2	0.425	4.2	3.0	7.5
				3× 0.3	0.425	7.6	5.4	6.0
Rigaku-RU500	400	1250	26200	10× 0.5	0.359	26.8	30	6.0
Rigaku-RU1000	400	2500	52450	10× 1	0.425	60	60	6.0
Rigaku-RU1500	250	10000	131000	10× 1	0.425	96	90	9.0
KFA-Jülich	250	12000	157000	14× 1.4	0.425	173	120	6.1

designing the electron gun to produce a line focus, that is the electron focus on the target face is approximately rectangular with the small dimension equal to the desired effective source size and the large dimension about 10 to 20 times larger. The focus is viewed at an angle between about 2 and 5° to the anode surface to produce an approximately square foreshortened effective source; and the X-ray windows are so positioned as to make these take-off angles possible. For some purposes, very fine line sources are required and windows may be provided to allow the focus to be viewed so as to foreshorten the line width. Higher power dissipation is possible in X-ray tubes in which the anode rotates: the line focus is now usually on the cylindrical surface of the anode with its long dimension parallel to the axis of rotation.

For focal-spot sizes down to about 100 μm, an electrostatic gun is adequate; this consists of a fine helical filament and a Wehnelt cathode, which produces a demagnified electron image of the filament on the anode. For most purposes, the Wehnelt cathode can be at the same potential as the filament but cleaner foci and adjustment of the focal spot size are possible when this electrode is negatively biased with respect to the filament. The filament is nearly always directly heated and made of tungsten. Lower filament temperatures, and smaller heating currents, could be achieved with activated heaters but the vacuum in high-power devices like X-ray tubes is rarely hard enough to permit their use since they are easily poisoned. However, Yao (1992) has reported successful operation of a hot-pressed polycrystalline lanthanum hexaboride cathode in an otherwise unmodified RU-1000 rotating-target X-ray generator.

Very fine focus tubes, with foci in the range between 25 and 1 μm, require magnetic lenses. At one time, the all-electrostatic X-ray tube of Ehrenberg & Spear (1951), which achieved foci between 20 and 80 μm, was very popular.

Sealed-off X-ray tubes for crystallographic use are nowadays made in the form of inserts containing a target of one of a range of standard metals to produce the desired characteristic radiation. A series of nominal focal-spot sizes, shown in Table 4.2.1.3, is commonly available. The insert is mounted inside a standard shield that is radiation- and shock-proof and that is fitted with X-ray shutters and filters and often also with a standardized track for mounting X-ray cameras. The water-cooled anode is normally at ground potential and the negative high voltage for the cathode, together with the filament supply, is brought in through a shielded shock-proof cable. The high voltage is nowadays generally of the constant-voltage type, that is, it is full-wave rectified and smoothed by means of solid-state rectifiers and capacitors housed in the high-voltage transformer tank, which also contains the filament transformer. The high tension and the tube current are frequently stabilized. Only the simplest X-ray generators now employ an alternating high tension that is rectified by the self-rectifying property of the X-ray tube itself.

A demountable continuously pumped form of construction is nowadays adopted mainly for rotating-anode and other specialized X-ray tubes. The pumping system must be capable of maintaining a vacuum of better than 10^{-5} Torr: filament life is critically dependent upon the quality of the vacuum.

Rotating-anode tubes have been reviewed by Yoshimatsu & Kozaki (1977). The first successful tube of this type that incorporated a vacuum shaft seal was described by Clay (1934). Modern tubes mostly contain vacuum-oil-lubricated shaft seals of the type due to Wilson (1941) and are based on, or are similar to, the rotating-anode tubes described by Taylor (1949, 1956). In some tubes, successful use has been made of ferro-fluidic vacuum seals (see Bailey, 1978). The main problems in the operation of rotating-anode tubes is the lifetime of the seals and of bearings that operate *in vacuo*. In successful tubes, *e.g.* those manufactured by Enraf, Rigaku-Denki, and Siemens, these lifetimes are about the same as the lifetime of the filament under good vacuum conditions, that is, of the order of 1000 h.

Phillips (1985) has written a review article on stationary and rotating-anode X-ray tubes that contains many important practical details.

4.2.1.3.1. *Power dissipation in the anode*

The allowable power loading of X-ray tube targets is determined by the temperature of the target surface, which must remain below the melting point. Müller (1927, 1929, 1931) first calculated the maximum loading both for stationary and for rotating anodes. His calculations were refined by Oosterkamp (1948) who considered, in particular, targets of finite thickness, and who also treated pulsed operation of the tube. For normal conditions, Oosterkamp's conclusions and those of Ishimura, Shiraiwa & Sawada (1957) do not greatly differ from those of Müller, which are in adequate agreement with experimental observations.

For an elliptical focal spot with axes f_1 and f_2, Müller's formula for the maximum power dissipation on a stationary anode, assumed to be a water-cooled block of dimensions large compared with the focal-spot dimensions, can be written

$$W_{\rm stat} = 2.063(T_M - T_0)Kf_1\mu(f_1,f_2), \qquad (4.2.1.12)$$

where K is the specific thermal conductivity of the target material in W mm^{-1}, T_M is the maximum temperature at the centre of the focal spot on the target, that is, a temperature well below the melting point of the target material, and T_0 is the temperature of the cold surface of the target, that is, of the cooling water. The function μ is shown in Fig. 4.2.1.4. For copper, K is 400 W m^{-1} and, with $T_M - T_0 = 500$ K,

$$W_{\rm stat} = 425\mu f_1. \qquad (4.2.1.13)$$

For $f_2/f_1 = 0.1$, and $\mu = 0.425$, this equation becomes

$$W_{\rm stat} = 180 f_1. \qquad (4.2.1.14)$$

In these last two equations, f_1 is in mm.

For a rotating target, Müller found that the permissible power dissipation was given by

$$W_{\rm rot} = 1.428\,K(T_M - T_0)f_1(f_2\rho Cv/2K)^{1/2}, \qquad (4.2.1.15)$$

where f_2 is the short dimension of the focus, assumed to be in the direction of motion of the target, v is the linear velocity, ρ is the density of the target material, and C is its specific heat.

For a copper target with f_1 and f_2 in mm and v in mm s^{-1},

$$W_{\rm rot} = 26.4 f_1(f_2 v)^{1/2}. \qquad (4.2.1.16)$$

Equation (4.2.1.16) shows that for very narrow focal spots rotating-anode tubes give useful improvements in permissible loading only if the surface speed is very high (see Table 4.2.1.3). The reason is that with large foci on stationary anodes the isothermal surfaces in the target are planar; with fine foci, these surfaces become cylindrical and this already makes for very efficient cooling without the need for rotation. Rotating anodes are thus most useful for medium-size foci (200 to 500 μm) since for the larger focal spots it becomes very expensive to construct power supplies capable of supplying the permissible amount of power.

Table 4.2.1.3 shows the recommended loading for a number of commercially available X-ray tubes with copper targets, which will be seen to be in qualitative agreement with the calculations. Some of the discrepancy is due to the fact that the value of $K(T_M - T_0)$ for the copper–chromium alloy targets used in actual X-ray tubes is appreciably lower than the value for pure copper used here. To a good approximation, the permissible loading for other targets can be derived by multiplying those in Table 4.2.1.3 by the factors shown in Table 4.2.1.4. It is worth noting that the recommended loading of commercial stationary-target X-ray tubes has increased steadily in recent years. This is largely due to improvements in the water cooling of the back surface of the target by increasing the turbulence of the water and the effective surface area of the cooled surface.

In considering Table 4.2.1.3, it should be noted that the linear velocities of the highest-power X-ray-tube anode have already reached a speed that Yoshimatsu & Kozaki (1977) consider the practical limit, which is set by the mechanical properties of engineering materials. It should also be noted that much higher specific loads can be achieved for true micro-focus tubes, *e.g.* 50 kW mm^{-2} for a 25 μm Ehrenberg & Spear tube and 1000 kW mm^{-2} for a tube with a 1 μm focus (Goldsztaub, 1947; Cosslett & Nixon, 1951, 1960).

Some tubes with focus spots of less than 10 μm utilize foil or needle targets. These targets and the heat dissipated in them have been discussed by Cosslett & Nixon (1960). The dissipation is less than that in a massive target by a factor of about 3 for a foil and 10 for a needle, but, in view of the low absolute power, target movement and even water-cooling can be dispensed with.

4.2.1.4. *Radioactive X-ray sources*

Radioactive sources of X-rays are mainly of interest to crystallographers for the calibration of X-ray detectors where they have the great advantage of being completely stable with time, or at least of having an accurately known decay rate. For some purposes, spectral purity of the radiation is important; radionuclides that decay wholly by electron capture are particularly useful as they produce little or no β or other radiation. In this type of decay, the atomic number of the daughter nucleus is one less than that of the decaying isotope, and the emitted X-rays are characteristic of the daughter nucleus. In some cases, the probability of electron capture taking place

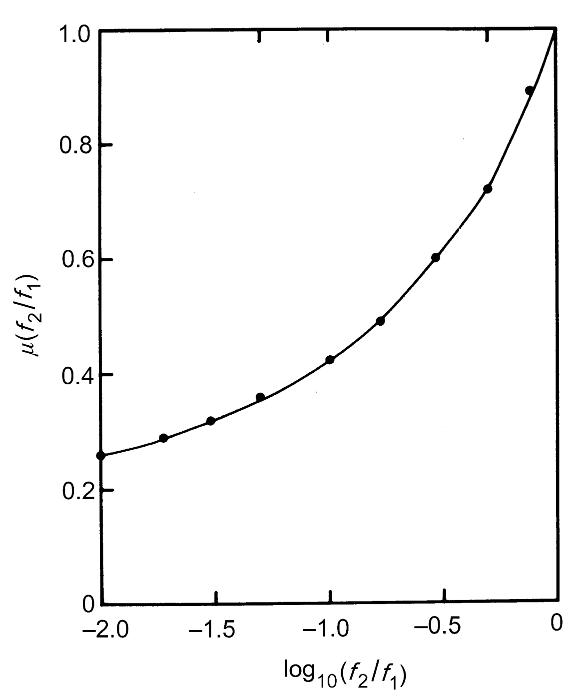

Fig. 4.2.1.4. The function μ in Müller's equation (equation 4.2.1.12) as a function of the ratio of width to length of the focal spot.

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