

2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

dispersive diffraction. This is done with one or more of the following techniques:

- crystal monochromators;
- single or balanced filters; and the
- detector system.

Special methods such as total reflection from a highly polished surface are rarely used in powder diffraction.

2.3.5.4.1. Crystal monochromators

Reflection from a single-crystal plate is the most common way to obtain monochromatic X-rays. Although the reflected beam is not strictly monochromatic because of the natural width of the spectral line and the rocking angle of the crystal, it is sufficient for practical powder diffraction. The crystal reflects λ and may also reflect subharmonic wavelengths $\lambda/2, \lambda/3, etc.$, and higher-order hkl 's depending on its crystal structure. Crystals can be selected to avoid the subharmonics, for example, Si and Ge cut parallel to (111); they have a negligible 222 reflection and $\lambda/3$ can be easily rejected with pulse-amplitude discrimination. Crystals are selected with relatively small Bragg angles to minimize polarization effects. Virtually all monochromators are of the reflection type. Transmission monochromators such as thin mica have been used occasionally in X-ray spectroscopy but not in powder diffraction.

When a crystal monochromator is placed in the direct beam from an X-ray tube or synchrotron-radiation source, the crystal also reflects other wavelengths from the continuous radiation. It is necessary to take a photograph of the reflected beam to see if Laue spots may be close to the spectral line and might pass through. If Laue spots are a problem and a flat crystal is used, a small rotation will move the spots. The entrance and exit slits should be made as narrow as possible for the experiment and a

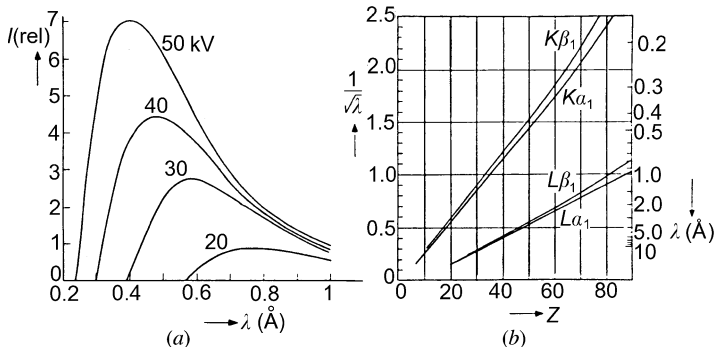


Fig. 2.3.5.4. (a) Continuous X-ray spectrum of tungsten target X-ray tube as a function of voltage and constant current. Full-wave rectification, silicon (111) crystal analyser, scintillation counter. (b) Plot of Moseley's law for four characteristic X-ray spectral lines.

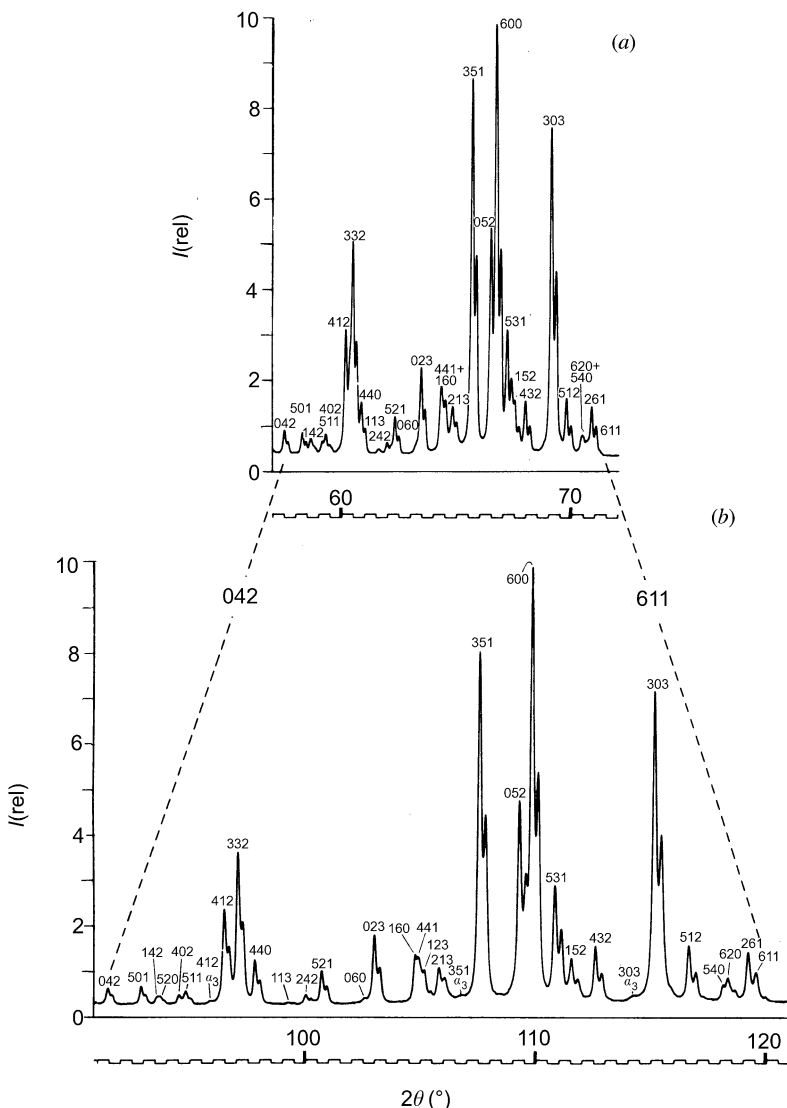


Fig. 2.3.5.5. Portion of diffractometer pattern of topaz showing effect of increasing dispersion on separation of peaks. (a) Cu $K\alpha$, (b) Cr $K\alpha$.