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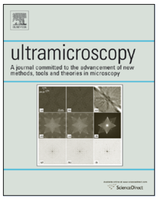

Choice of operating voltage for a transmission electron microscope

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ABSTRACT

An accelerating voltage of 100–300 kV remains a good choice for the majority of TEM or STEM specimens, avoiding the expense of high-voltage microscopy but providing the possibility of atomic resolution even in the absence of lens-aberration correction. For specimens thicker than a few tens of nm, the image intensity and scattering contrast are likely to be higher than at lower voltage, as is the visibility of ionization edges below 1000 eV (as required for EELS elemental analysis). In thick (> 100 nm) specimens, higher voltage ensures less beam broadening and better spatial resolution for STEM imaging and EDX spectroscopy.

Low-voltage (*e.g.* 30 kV) TEM or STEM is attractive for a very thin (*e.g.* 10 nm) specimen, as it provides higher scattering contrast and fewer problems for valence-excitation EELS. Specimens that are immune to radiolysis suffer knock-on damage at high current densities, and this form of radiation damage can be reduced or avoided by choosing a low accelerating voltage. Low-voltage STEM with an aberration-corrected objective lens (together with a high-angle dark-field detector and/or EELS) offers atomic resolution and elemental identification from very thin specimens. Conventional TEM can provide atomic resolution in low-voltage phase-contrast images but requires correction of chromatic aberration and preferably an electron-beam monochromator.

Many non-conducting (*e.g.* organic) specimens damage easily by radiolysis and radiation damage then determines the TEM image resolution. For bright-field scattering contrast, low kV can provide slightly better dose-limited resolution if the specimen is very thin (a few nm) but considerably better resolution is possible from a thicker specimen, for which higher kV is required. Use of a phase plate in a conventional TEM offers the most dose-efficient way of achieving atomic resolution from beam-sensitive specimens.

1. Introduction

Use of the de Broglie relation ($\lambda = h/mv$) shows that an electron wavelength $\lambda = 0.12$ nm, sufficient for atomic resolution, is achieved by accelerating electrons through a potential difference of only 100 V. The fact that transmission electron microscopes operate at much higher voltages is partly a reflection of the aberrations of their imaging lenses. In the presence of such aberrations, the resolution of a conventional (CTEM) or scanning (STEM) instrument can be represented by

$$\textit{resolution} \sim aC_s^{1/4}\lambda^{3/4} + b\lambda^{1/2}(C_c\Delta E/E_0)^{1/2} + \textit{parasitic aberrations}$$

where C_s and C_c are spherical- and chromatic-aberration coefficients; *a* and *b* are numbers that depend on the imaging mode but do not differ greatly from 1. *Parasitic aberration* refers to misalignment of electron-optical column or the effect of stray magnetic fields, and can be generalized to include mechanical and electrical instabilities.

For the first 50 years of TEM development, the quest for improved (and eventually atomic) resolution involved reducing λ by raising the accelerating voltage. The final result of this philosophy was the high-voltage microscope (HVEM) operating at 1 MeV or even 3 MeV. High-voltage stability and parasitic aberrations proved to be limiting factors, although 0.1 nm resolution was eventually achieved [1]. High-voltage microscopes can image relatively thick specimens (of the order of 1 μm) but they tend to produce knock-on radiation damage and were in fact used to simulate neutron damage in metallic specimens [63]. In the end, atomic resolution was more easily obtained by using an *intermediate* voltage (in the range 200–400 kV), by careful design and machining of polepieces, and by using a short objective focal length to reduce the aberration coefficients C_s and C_c.

Two recent developments have challenged the predominance of these medium-voltage TEMs. First, the availability of nanotechnology specimens such as nanotubes (with dimensions of the order of a few nm) followed by the discovery of self-supporting monatomic layers of carbon (graphene) and other materials, which have provided ideal test specimens for microscopy at lower voltage, where the increased contrast proves useful.

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A second development was the commercial introduction of multipole correctors that make C_s close to zero (or even negative) and allow atomic resolution at lower operating voltages, such as 60 kV in STEM mode [2,3]. Even without aberration correction, Crewe in 1979 used the STEM at 25–30 kV to obtain 0.25 nm resolution, sufficient to resolve isolated heavy atoms on a thin carbon support [4].

In a CTEM, the objective lens must deal with energy losses introduced by the specimen, making ΔE larger than in STEM mode (where it represents only the energy spread of the electron source), so the effect of chromatic aberration becomes severe at lower voltage. To achieve atomic resolution, this aberration must be corrected simultaneously over some reasonable field of view, using various techniques [5–7].

The impact of aberration correction in biology has been minimal because the resolution obtainable from biological specimens is limited by radiation damage rather than by the TEM optics. We discuss dose-limited resolution (DLR) later in this article, together with the choice of operating voltage for energy-dispersive x-ray (EDX) analysis, electron energy-loss spectroscopy (EELS) and energy-filtered imaging (EFTEM).

As electrons are accelerated, their angle relative to the optic axis decreases and the electron-optical brightness (current per unit area per unit solid angle) increases in proportion to a relativistically-corrected accelerating voltage [64], making it easier (in the presence of lens aberrations) to obtain high current in a small electron probe. But high voltages make electrical insulation and voltage stability more difficult. The required lens currents also increase, making the instrument bigger and more expensive (a HVEM requires a specially constructed building).

Phosphor screens are still used in a conventional TEM, as a viewing screen or as part of the phosphor-screen/CCD-camera combination. An electron beam spreads laterally as it travels through the phosphor, reducing the spatial resolution at low and medium magnifications. The angular spreading is less at higher beam energy (see Section 4) but a thicker phosphor is then required for efficient detection, so beam spreading remains a problem. Direct-exposure diode arrays are now available for use in the TEM. They are restricted to very low image intensities but are capable of fast readout [65] and can have better DQE than a scintillator/CCD camera [61].

Since most TEMs are designed to operate at several different voltages, the user has a choice. But operating the instrument at lower voltage usually results in lower CTEM-screen brightness (even in the absence of a specimen) and a noisier image from the CCD camera. A decrease in electron-optical resolution can also be expected, because of the increased scattering angles and the greater effect of lens aberrations.

Provided the specimen is thin enough, lower voltage gives higher image contrast, due to the larger elastic-scattering cross section (proportional to $1/v^2$ where v is electron speed). For thick specimens, and especially at low kV, the intensity of a bright-field diffraction-contrast image becomes low because the fraction F of unscattered electrons that pass through objective aperture decreases, reducing the signal/noise ratio and potentially the resolution. This aspect will be discussed later in relation to organic samples.

2. TEM of conducting specimens

The principal reason for operating at low kV is to reduce or avoid knock-on displacement, the radiation-damage process that predominates in electrically-conducting specimens (which do not damage by radiolysis). Knock-on damage arises from high-angle “elastic” scattering (deflection by the electrostatic field of atomic nuclei), which can transfer several eV of energy direct to a nucleus and result in the displacement of an atom within a crystal or at its surface. The different possibilities are listed below in order of *decreasing* activation energy E_d required to displace an atom.

Atomic displacement within a crystal.
Atom displacement at grain boundaries.
Atom displacement *from* a surface.
Surface-atom displacement *along* a surface.
Adatom displacement along a surface.

Displacement within a crystalline specimen leads to interstitial-vacancy pairs, which at elevated temperature aggregate into dislocation loops that give black–white diffraction contrast [63]. Atoms at a grain boundary are generally more loosely bound and their displacement is observable using phase-contrast atomic-resolution TEM. Atom displacement from a surface results in electron-beam sputtering of material into the vacuum, seen as a crater formed on (usually) the beam-exit surface of the specimen and eventually as a complete hole. The motion of heavy adatoms on a thin substrate has been observed in the STEM [3,4,8] and may be either a beam-induced or a thermal effect, as discussed below.

2.1. Bulk displacement damage

The energy that an electron can lose by elastic scattering through an angle θ is $E = E_{max}\sin^2(\theta/2)$, where E_{max} is the maximum energy transfer that corresponds to a scattering angle of 180°, which is given in eV [9] by

$$E_{max}(\mathrm{eV}) = (1.1/A)[2+E_0/(511\ \mathrm{keV})]E_0(\mathrm{keV}) \tag{1}$$

Here A is the atomic (mass) number of the atom and the incident energy E_0 is in keV.

There is a threshold incident energy E_0^{th} below which displacement cannot occur because $E < E_d$ even for 180° scattering:

$$E_0^{th}(\mathrm{eV}) = (511\ \mathrm{keV})\{[1+AE_d/(561\ \mathrm{eV})]^{1/2}-1\} \tag{2}$$

Large A or large E_d implies high E_0^{th} but the relationship is nonlinear.

For bulk displacement in crystals, the activation energy E_d is relatively large; see Table 1. E_d is related to the binding energy of the solid but varies somewhat with the direction of momentum transfer, so E_0^{th} depends somewhat on the crystal orientation, even in a cubic material. The values given in Table 1 are therefore approximate. Because E_{max} varies inversely with A, the threshold energy for a compound is usually determined by displacement

Table 1
Measured activation energy E_d and threshold energy E_0^{th} for bulk displacement in crystalline solids [10,11].

Material	E_d (eV)	E_0^{th} (keV)
Diamond	80	330
Graphite	31	150
Magnesium	10	101
Aluminum	16	169
Silicon	21	221
Iron	17	327
Copper	20	412
Zinc	14	318
Gold	34	1330
Al_2O_3	18	187
MgO	60	330
GaAs	9	233
CdTe	5.6	233

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