Low Voltage Transmission Electron Microscopy

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The reasons for increased contrast with decreased accelerating voltage in the transmission electron microscope (TEM) are explained. The low voltage TEM (accelerating voltage 5 keV), as developed by Armin Delong, is described and assessed. This microscope, currently being produced by Delong Instruments in Brno, Czech Republic, is a combination of a miniaturized TEM and a standard light microscope. Further benefits of this microscope over the conventional TEM – including price, field of view, installation space, distortion, and sample damage – are addressed. Finally, current and possible future applications, as explored by Lawrence F. Drummy, are discussed.

I. INTRODUCTION

It can be shown[1] that the atomic scattering factor (for an scattering angle θ, for electrons with energy E_0, incident on a material with atomic number Z) is given by

$$f(\theta) = \frac{\left(1 + \frac{E}{E_0}\right)}{8\pi^2 a_0}\left(\frac{\lambda}{\sin\frac{\theta}{2}}\right)^2 (Z - f_X). \quad (1)$$

In a TEM image, the contrast ΔI/I is proportional to the single-atom scattering cross section, which is given by

$$\sigma(\beta) = \int_{\beta}^{\infty} |\, f(\theta)|^2 \, d\Omega \quad (2)$$

for a bright field collection half angle β. So contrast increases with atomic number and decreases with incident electron energy. In order to increase the contrast when imaging low Z materials (such as organics) in a transmission electron microscope, it is then advantageous to lower the accelerating voltage.

The accelerating voltage cannot be lowered without limit, however, since resolution decreases with decreasing accelerating voltage. With a spherical aberration coefficient of 1 mm and an objective aperture semiangle of 0.01 rad, the optimal accelerating voltage for thin film carbon is approximately 5 keV.[2] When compared with 100 keV, an accelerating voltage of 5 keV produces images with about an order of magnitude greater contrast, and a resolution about 3 times worse (See Figure 1).

Following the realization by Nixon[3] that increased inelastic scattering at lower energies can increase contrast, attempts were made to construct a low voltage electron microscope[4] in the 1960's for the imaging of biological (low Z) materials. However, at

that time, there were technological limits on electron gun brightness, robustness against sample contamination, sensitivity of the fluorescent screen, and insensitivity to stray electric and magnetic fields.[5] In 1992, Armin Delong utilized improvements on these limits to make a usable low voltage transmission electron microscope (LVTEM).[2]

II. LVTEM DESIGN[2]

The LVTEM as constructed by Delong consists of two main parts: a miniaturized transmission electron microscope with relatively low magnification onto a fluorescent yttrium aluminum garnet (YAG) screen, and a light microscope to further magnify the image from the structureless YAG screen onto a CCD camera for resolvable recording and viewing. There is thus a conversion from an electron image to a light image prior to viewing or recording.

A. Electron optical system[6]

The miniaturized TEM (150 mm long) has a combination of electrostatic lenses and permanent magnet lenses. Electrons are emitted from a Schottky emission cathode. The condenser and objective lenses are made of eight small shielded $SmCo_5$ magnets 30 × 9 × 3 mm. The beam passes through these lenses before penetrating the sample. After the sample, the beam is corrected for astigmatism by stigmator-deflectors and magnified and projected onto the YAG screen by three electrostatic lenses. Figure 2 depicts the miniaturized TEM schematically.

B. Light microscope[2]

The YAG screen has a quantum efficiency of only 5%. This is about a fifth of conventional polycrystalline screens, so that one might start to worry about the brightness of the image from a YAG screen. However, the light microscope allows for a large gain in brightness so that even with the light flux from the YAG screen into the light microscope objective lens reduced to one third, the brightness gain is greater than 1500. This means that the beam current can be reduced 300 times and still produce an image as bright as that from a conventional TEM with a polycrystalline screen.

III. OTHER BENEFITS

Price – Because half of the microscope is a light microscope, the lenses and other optical components have a large market and, thus, are very precise and commercially available at relatively low cost.

Field of view – Wide field eyepieces are available which can give a field of view of 280 mm (three times greater area than conventional TEM's).[2]

Distortion – For the same reason that prices are lower, well worked out and precise optical components result in lower distortion.

Space – The microscope is significantly smaller than a conventional TEM, allowing installation on any regular desktop.

Sample damage – Because this microscope allows for the reduction of the beam current, as well as the accelerating voltage, both knock-on damage (from the momentum of the electron) and radiation damage (from exposure to charge) are reduced.

IV. APPLICATIONS

The most obvious application of Delong's LVTEM is to biological samples. Specifically, the imaging of organic thin films and polymers is greatly enhanced with better Z contrast and reduced beam damage. Drummy has shown[7] that the mass-thickness contrast of pentacene is quite good at the grain boundaries using the LVTEM at an accelerating voltage of 4.7keV (Figure 3). Images taken on the FEI Tecnai G20 TEM in Duffield Hall, shown in Figure 4, do not show the grain boundaries as clearly.

In imaging block copolymers, Drummy made use[7] of the LVTEM's enhanced contrast not only for low Z materials, but also for subtle differences in sample density. He obtained clear images of density differences on the order of 0.1 g/cm^3. This eliminates the necessity for staining with high density elements. Some block copolymers, such as blends of polystyrene and poly(methyl methacrylate) PS-PMMA are imaged by intentionally damaging the PMMA block so that the reduced mass contrasts with the PS block. With the LVTEM, it should be possible to see contrast between the blocks with mass reduction.

Further still, the LVTEM shows remarkable contrast for small changes in sample height (on the order of 1 nm). Drummy imaged[7] Generation 5 and Generation 7 dendrimers (highly branched polymers) at a height of 1-2 nm above the amorphous carbon substrate. The density of the substrate is approximately the same as that of the dendrimers so that the sharp contrast is due to that small change in sample height.

In situations where exceptionally fast turn-around is helpful, the LVTEM offers a solution in that the microscope is much more suitable for placement near a lab or production site. For example, in processing electrospun nanofibers,[7] the relationship

between processing conditions and outcome are not completely understood. In this case it is very helpful to be able to examine the sample immediately after production and adjust the processing conditions according to the results. The smaller LVTEM allows for this if placed next to the nanofiber production equipment.

V. CONCLUSION

High resolution electron microscopy (HREM) is an alternative method for imaging organic materials. Increased resolution may allow for better distinction of grain boundaries in pentacene thin films, for instance. However, the wide range of benefits described above, including reduced sample damage, favor the LVTEM for most organic sample imaging. The dual nature of the microscope (electron and light) means that it will benefit from improvements in both fields. This microscope should be strongly considered by anyone who images light elements, specifically organics.

VI. ACKNOWLEDGEMENTS

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VII. FIGURES

Fig. 1. Dependence of resolution and contrast of TEM on the acceleration voltage.[2] The sample is a 20 nm thick carbon film.

Fig. 2. A schematic drawing of the miniaturized TEM.[6]

Fig. 3. TEM image taken at 4.7 keV of thin film pentacene.[7]

Fig. 4. TEM image taken at 200 keV of thin film pentacene.

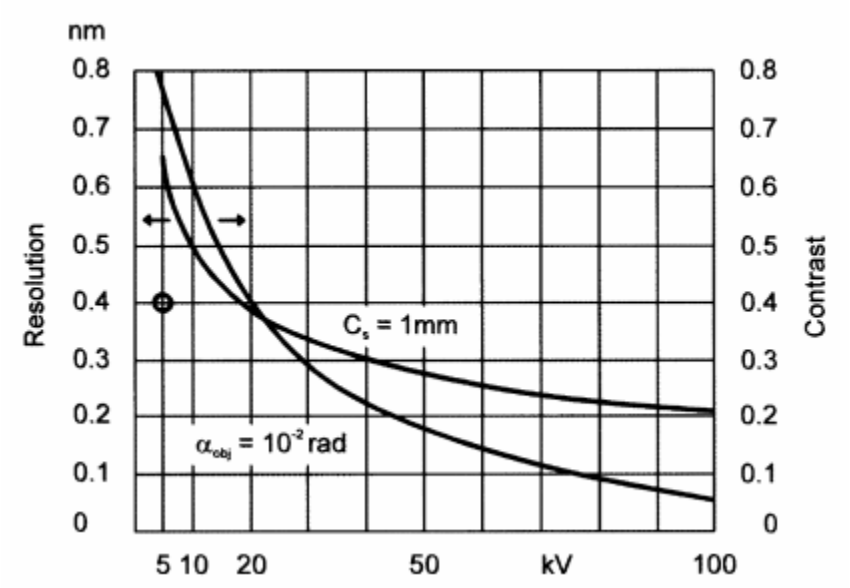

Fig. 1.

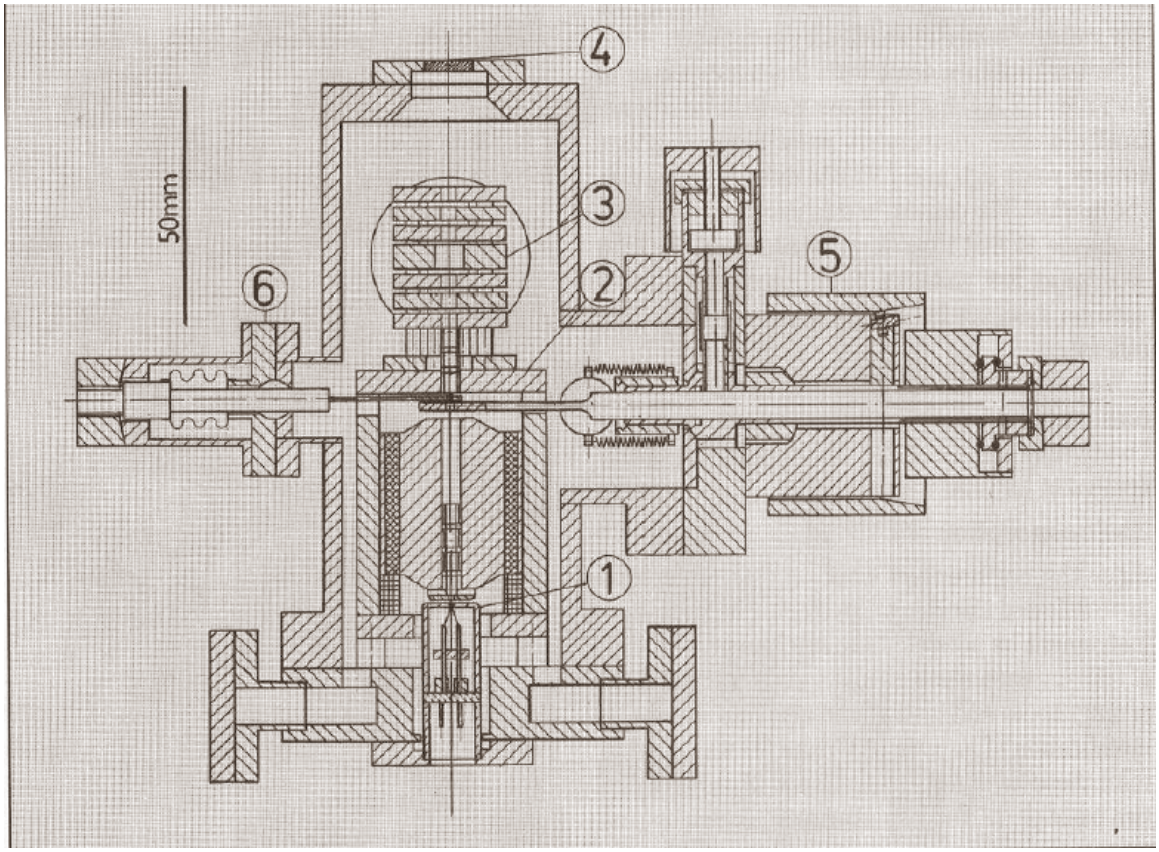

Fig. 2.

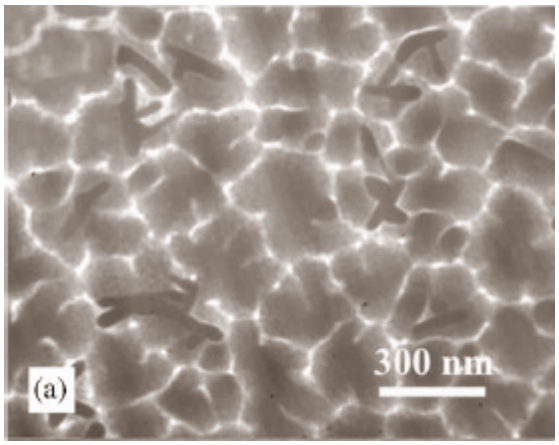

Fig. 3.

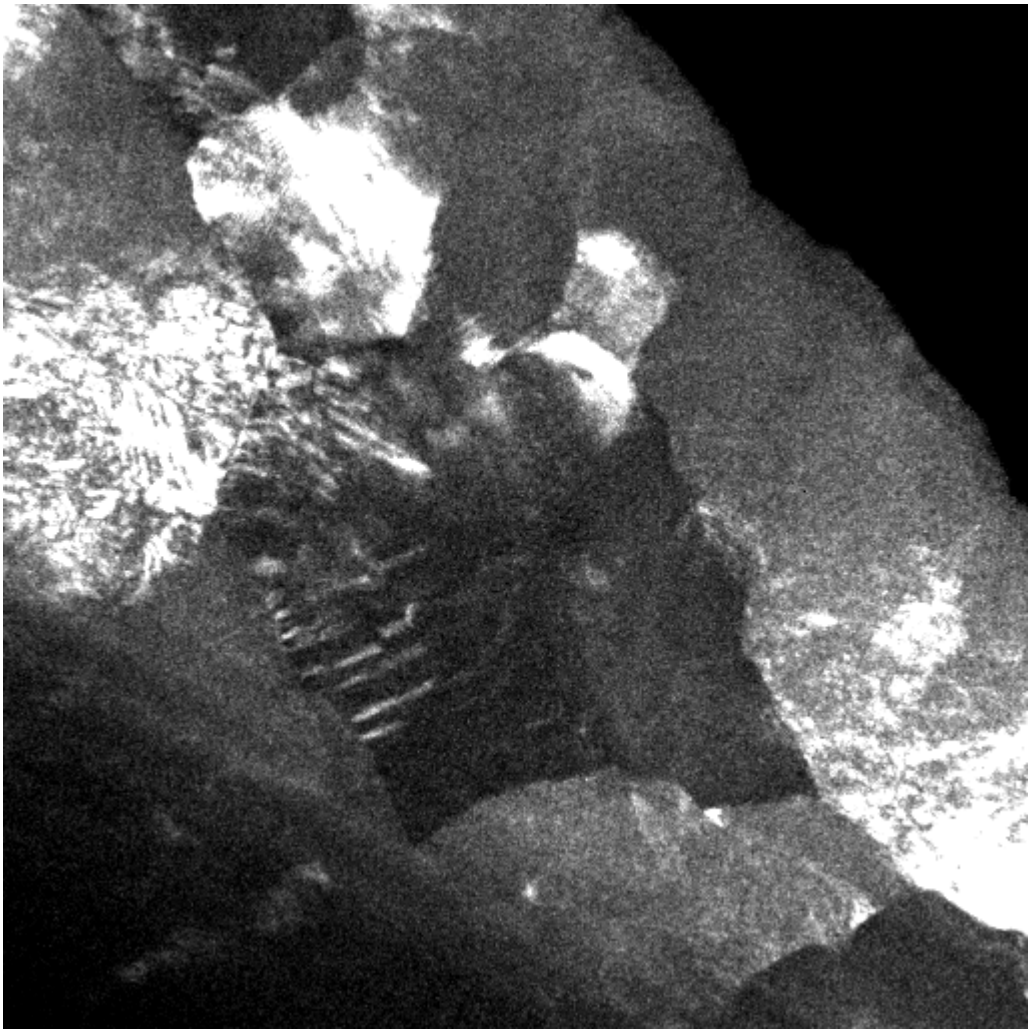

Fig. 4.

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