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Progress on the ANL Advanced AEM Project*

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PROGRESS ON THE ANL ADVANCED AEM PROJECT.

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Commercial instrumentation on the market today, although labeled as "analytical electron microscopes", are in fact transmission or scanning transmission electron microscopes with analytical attachments. While the manufacturers always optimize their instruments, they inevitably do so at the expense of analytical performance, and instead optimize for the best possible image resolution. In 1980 the Electron Microscopy Center (EMC) at Argonne National Laboratory (ANL) began a development project to specify and acquire a true analytical electron microscope (AEM). The instrument specifications were devised with the primary goal of attaining the best possible analytical sensitivity, resolution and versatility consistent for state of the art materials research and still provide moderate imaging capabilites. Early in the conceptual design it was determined that the instrument was sufficiently involved that it could not be built by the research staff at ANL alone, and the decision was made to enter a development contract with a commercial manufacturer. After spending 3 years raising funds, 2 years visiting manufacturers to discuss and detail the specifications, and 1 year in bidding and negotiating a contract, the project was finally awarded to VG Scientific Instruments, (now VG Microscopes Ltd.) of East Grinstead, England. Progress has been slow but deliberate and it appears that the instrument is nearing completion and may actually be delivered this year.

During the preliminary stages of this project, the most difficult task was to convince the manufacturers of the need and advantages of developing an analytical microscope operating at accelerating voltages of ~ 300-500 kV and having an integral cold field emission gun (CFEG). In 1980, when the EMC began this project, instruments operating at greater than 200 kV were few and far between (excluding HVEM's), and commercial FEG's only operated at 100 kV. Today, 11 years later, medium voltage (300-400 kV) and 200 kV-FEG's instruments are standard items in nearly all the manufacturers repertoires; however, when the EMC began this project the issue was in fact a major obstacle. The other important hurdles were the concept that image resolution was not the prime objective of the instrument; that true UHV (< 5×10^{-10} Torr) at the specimen was an absolute requirement; and that the instrument must be modular in nature to allow the EMC to develop and install analytical detectors for future work, yet to be specified.

The general specifications of the instrument which were finally developed are listed in Table 1. Basically, the system consists of a CFEG with gun lens, a triple condenser, objective and quadruple projector

(Figures 1-2). While high resolution imaging of the specimen is not important, imaging of the probe is considered essential, thus the four projectors allow sufficient magnification to do so under all conditions. All apertures (VOA, Cond, Obj, Diff, EELS) are electrically isolated and have 8 position stops for electron dosimetry, together with a faraday cup in the probe forming system. A conventional electrostatic beam blanking system is integral and is located between C2 and C3 lenses. One of the most important features in the system design was the development of an objective lens configuration which satisfied the ANL requirements. Figures 3 and 4 sketch out the geometrical configuration of this area, which includes 7 experimental ports for analytical equipment development, and 3 for electrical connections. The pole piece gap is sufficient to allow complete inversion of the stage, although in practice due to the need for gearing the motor drives the actual rotation about the primary tilt axis is limited to $\pm 85.^\circ$ Initially 3 types of specimen stages are being constructed: ambient double tilt Be, LN_2 double tilt Be, and a single tilt 1000°C heating stage. Image detection is accomplished by using conventional NTSC video in the CTEM and BF/ADF imaging in STEM, using one of four operator-selected YAG screens, the signals from which are flash digitized (8, 16, 32 bit) and routed to two independent frame stores (512 x 512 x 8 bit). A comprehensive specimen preparation chamber is directly interfaced to the column (Figure 2) which allows complete extensive cleaning, characterization and preparation of the specimen surface (see Table 1). A "parking lot" for 12 specimens is also provided for in the specimen prep chamber. Two 400 l/s and two 60 l/s Ion Getter, four Titanium Subliminators, 1 Turbomolecular comprise the evacuation pumps for the system.

Consistent with the goal of maximizing the analytical sensitivity is minimizing spectral artifacts. Careful scrutiny has therefore been given to all parts of the probe forming system, with special attention to the various beam defining apertures. For example, multi-layer low/high Z material combinations have been employed in both beam and non-beam defining apertures and at all critical surfaces to minimize potential sources of uncollimated hard x rays which give rise to the hole count phenomenon. The windowless XEDS system has been optimized to maximize the subtending solid angle and allow retraction along a direct line of sight path to the specimen. This allows the instrument to achieve a continuously variable solid angle up to a maximum of 0.3 sR. A conventional hemispherical Auger spectrometer with extraction optics is interfaced to the center of the objective lens and both serial and parallel EELS detection capabilities will be present. In addition, not shown in Figure 3, on the incident beam side of the objective lens between C2 and C3 is a drift space for secondary and Auger electron spectrometers utilizing parallelizer optics installed within the objective prefield. The entire system is microprocessor controlled using a 386 level PC as system controller with a second 286 system for image handling using the two frame stores. The system may be controlled either directly by the operator using conventional multi-function dials and switches or using the PC and a mouse directed interactive graphical user interface.

The high voltage generator has already demonstrated combined ripple and noise of 150 mV at 300 kV (0.5 ppm!) under a full load bench test. With the column operating at 250 kV graphite 3.4 Å STEM lattice images have been achieved. The expected gun brightness is ~$4x10^9 A/cm^2/sR$ at 300 kV and the nominal image resolutions in both TEM and STEM should be < 3Å pt/pt BF, and < 2Å in HADF-STEM. Figure 5 plots a calculated contrast transfer function for the Objective Lens at 300 and 100 kV, while Figure 6 plots the calculated probe current/size relationships at 300 kV.

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Table 1. ANL-EMC Advanced AEM Project Goals

- **Cold Field Emission Electron Source**
  - V_O: 50 - 300 kV, ΔV_{step} = 25 V, $\Delta V/V_O \leq 1$ ppm
  - $\beta_{300kV} \sim 4 \times 10^9$ A/cm^2/sR and ultimate probes ≤ 3 Å

- **Ultrahigh vacuum (UHV) environment**
  - < 2×10^{-11} torr - Gun
  - < 2×10^{-10} torr - In the specimen region of the Objective Lens Column
  - < 5×10^{-10} torr - Specimen Preparation Chamber

- **Electron Optics capable of :**
  - STEM: Scanning Transmission Electron Microscopy
    All the usual imaging/scanning modes - Frame/Line/Spot...
    Mag : 50 X - 20 MX, Resolution BF STEM: ~3 Å, HADF ~1.85 Å
  - TEM: Transmission Electron Microscopy
    Condenser/Objective Configuration-
    Parallel probes down to ~ 100 Å, Convergent probes down to ~ 2-3 Å
    Mag : 10 kX - 900 kX, Resolution: BF Pt/Pt : <2.8 Å, BF Lattice : < 3.4 Å
  - SEM: Scanning Electron Microscopy
  - TSEM: Transmission Scanning Electron Microscopy
  - TSED: Transmission Scanning Electron Diffraction
  - CBED: Convergent Beam Electron Diffraction, 8 aperture positions!
    CL: 10 mm - 9 m , Distortion Free Mode: 10-50 mm
  - SAED: Selected Area Electron Diffraction : Only at > 30 kX
  - RHEED: Reflection High Energy Electron Diffraction

- **Side Entry Goniometer Stages**
  - Goniometer Stage capable of tilting to ± 85 ^{o} for special applications without hitting pole piece
  - RT Double Tilt Beryllium: ± 60 ^{o}, ± 45 ^{o} Primary/Secondary
  - LN_2 Cooled Double Tilt Be Stage: ± 60 ^{o}, ± 45 ^{o} Primary/Secondary , -165^o C to +100 ^{o}C
  - Single Tilt Heating Stage: ± 45 ^{o} Primary, RT to + 1000^o C

- **Analytical SubSystems on the E/O Column**
  - XEDS - Windowless Detector @ 0.3 sR retractable along a line-of-sight to specimen
  - EELS - Serial/Parallel modes
  - AES - Hemispherical Analyzer on Objective Lens and with Parallelizer Configuration
  - Imaging - Two independent frame stores

- **Specimen Preparation Chamber**
  - Main AirLock and Transfer Cell with Sputter Cleaning Facilities
  - High Pressure/Temperature Gas Reaction Cell
  - Vacuum Transfer Vessel
  - Thin Film Evaporation Chamber
  - Mini-SIMS system - Gallium LMIS, Quad Mass Analyzer
  - RV LEED
  - ANL MultiPort Station for development work.

- **Computer Control**
  - PC Compatible 386 for Basic System Operation and Control
  - PC Compatible 286 for Frame Store and imaging

- **Special Objective Lens Port Configuration**
  - 7 Experimental Ports on Objective Lens for Analytical Equipment
  - 3 Additional Ports for Electrical Feedthrus etc...

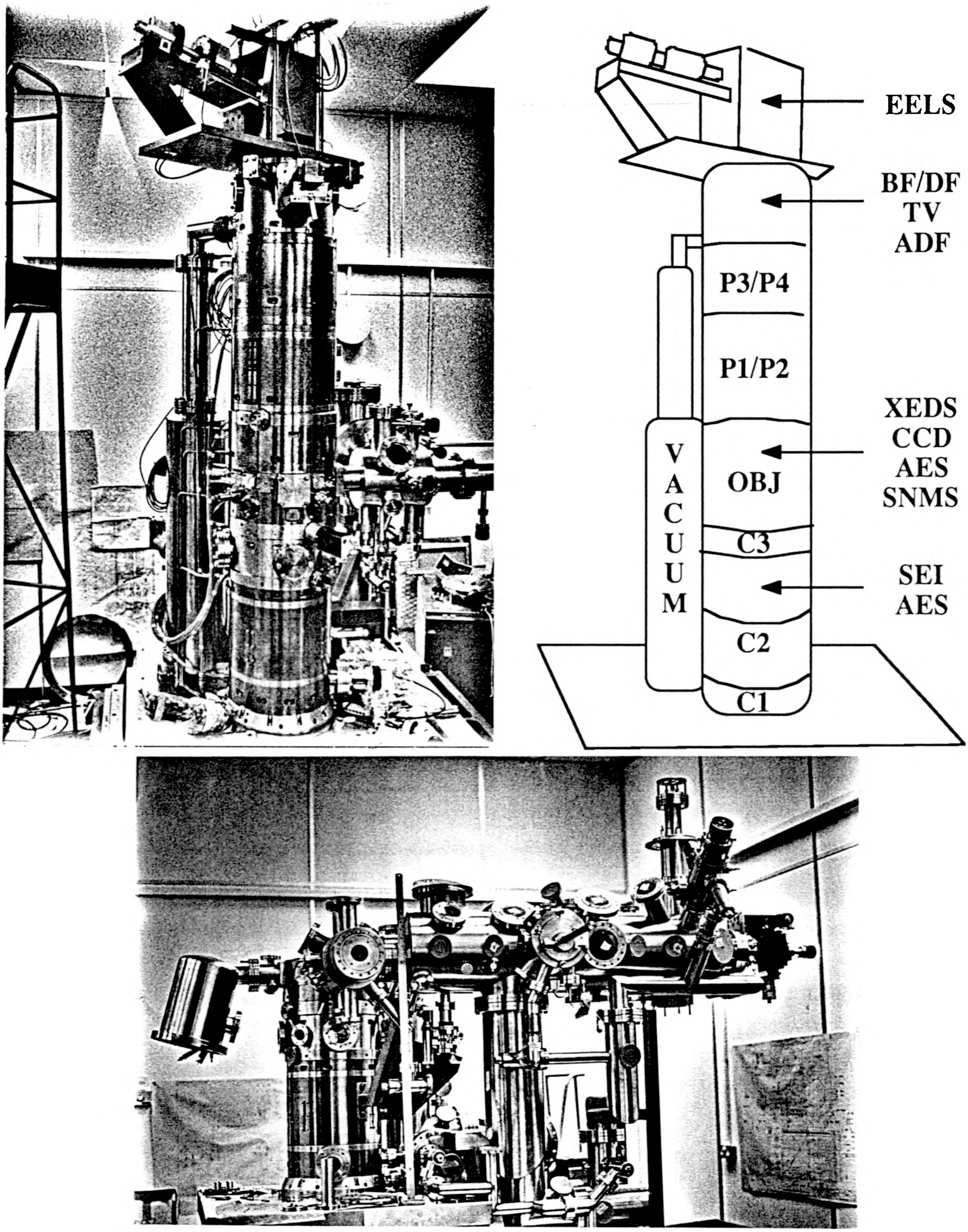

Figure 1. ANL AEM Column Assembly (top) .and Specimen Preparation Chamber (bottom).
Note on the lower figure the column is only assembled up to the objective lens. See figure 2 for plan view.

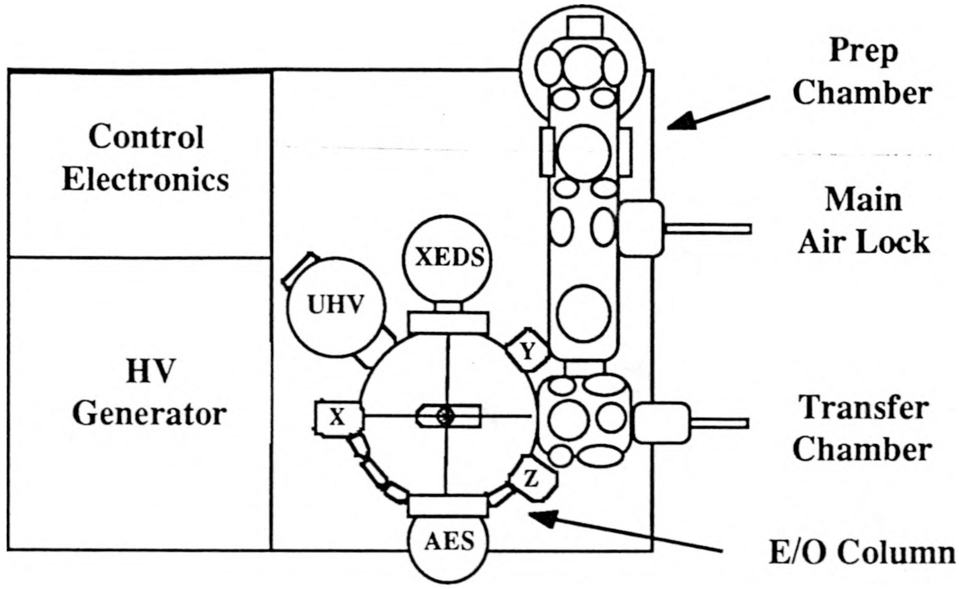

Figure 2 Plan View of System showing location of Preparation Chamber relative to Column

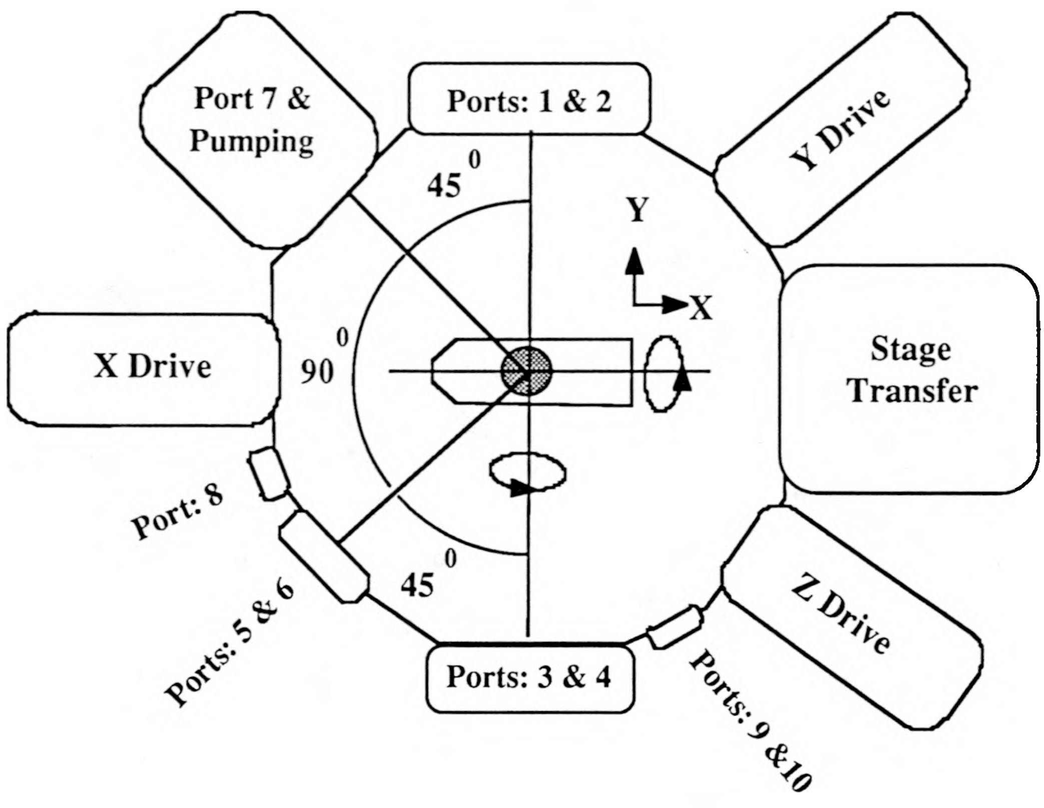

Figure 3. Plan View of Objective Lens Area Showing Experimental Port Configuration.

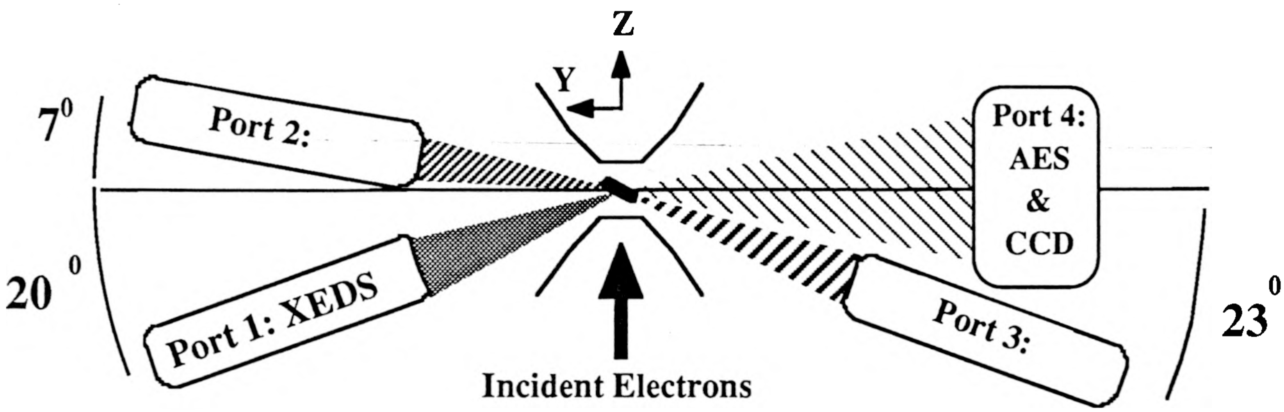

Figure 4. Cross Section through Ports 1/2 and Ports 3/4 of Figure 1. Ports 5/6 have the same geometry as Ports 1/2, Port 7 is similar to Port 4. Ports 8-10 are for electrical connections.

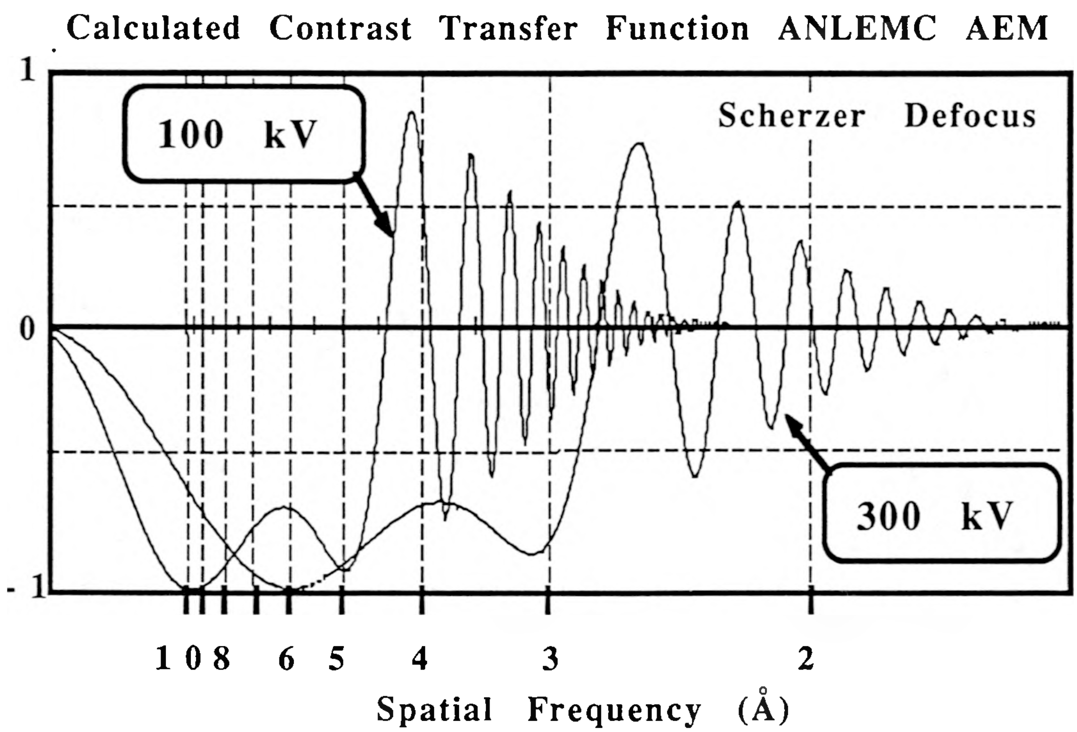

Figure 5. Calculated Contrast Transfer Function for the ANL AEM at 300 and 100 kV; Scherzer Defocus, Cs=4.4 mm, CFEG Source

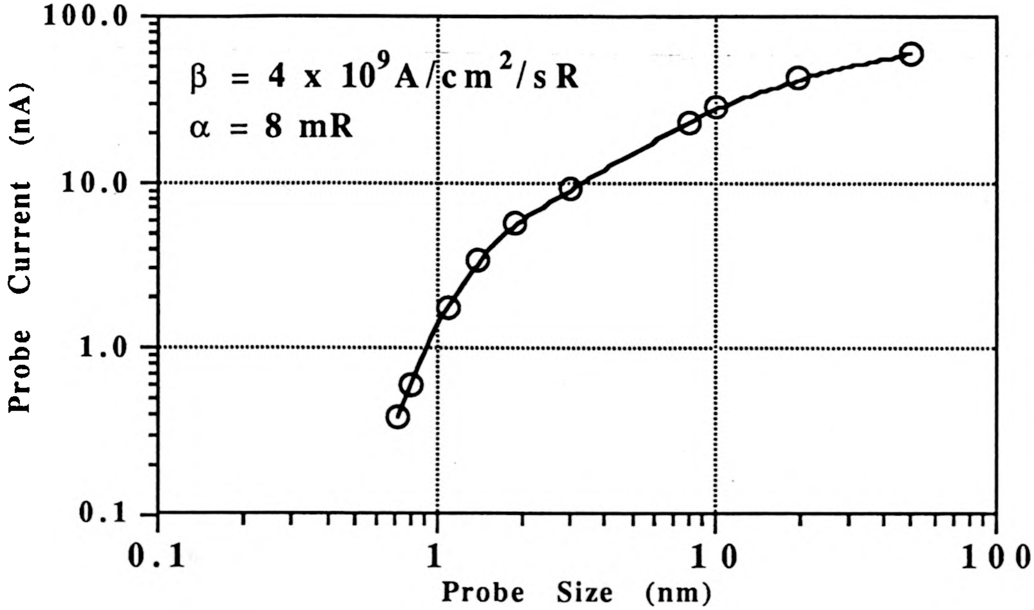

Figure 6. Calculated Probe Size/Current Relationship for the ANL AEM at 300 kV