Beam-Induced Damage to Thin Specimens in an Intense Electron Probe

Raymond F. Egerton,1,* Feng Wang,1 and Peter A. Crozier2

1Physics Department, University of Alberta, Edmonton, AB T6G 2J1, Canada
2Center for Solid State Science, Arizona State University, Tempe, AZ 85287-1704, USA

Abstract: We have investigated the changes produced in single-element and two-layer transmission electron microscope (TEM) specimens irradiated by an intense nanometer-sized electron probe, such as that produced in a field-emission or aberration-corrected TEM. These changes include hole formation and the accumulation of material within the irradiated area. The results are discussed in terms of mechanisms, including electron-beam sputtering and surface diffusion. Strategies for minimizing the effect of the beam are considered.

Key words: radiation damage, electron sputtering, aberration-corrected probe

INTRODUCTION

A modern field-emission transmission electron microscope (TEM), operating at an accelerating voltage of typically 200 kV, can produce a subnanometer electron probe containing a current density approaching 10^5 A/cm^2. The current density may exceed 10^6 A/cm^2 if a spherical-aberration corrector is added to the illumination system (Lupini et al., 2001). Few materials can survive for long under such intense radiation, particularly if the probe is stationary, as required for elemental analysis by X-ray energy-dispersive spectroscopy (XEDS) or core-loss electron energy-loss spectroscopy (EELS) at high spatial resolution. We were therefore curious to determine the response of different materials systems to such conditions and to investigate possible steps that can be taken to minimize the changes produced by the electron beam.

Organic specimens are notoriously beam sensitive and even inorganic compounds degrade by radiolysis to various degrees, as a consequence of the energy transfer arising from inelastic scattering. Single-element samples are probably the most beam resistant, due to the absence of a radiolytic mechanism. However, elastic (nuclear) scattering of electrons can transfer appreciable energy if the scattering angle is large, as is the case for a small fraction of the scattering events. Such high-angle scattering gives rise to displacement damage in crystalline materials that are examined in a medium- or high-voltage TEM. However, this same scattering process can produce mass loss by electron-induced sputtering, as verified by studies in which ejected gold atoms were collected on a carbon foil (Cherns et al., 1976). Whereas displacement damage is most often reported in studies using a high-voltage microscope, the threshold incident energy for electron sputtering is below 200 keV for about half of the elements in the periodic table (Egerton et al., 2004). This leads to the pessimistic conclusion that few specimens are immune to electron-beam damage in the focused probe of a modern TEM.

To test the validity of this conclusion, we chose relatively radiation-resistant materials in which radiolysis is unlikely to occur. Carbon films are commonly used as a support for small particles and provided an obvious test specimen for our study. Although it has a high binding energy, carbon is a light element and is believed to undergo sputtering even by 100-keV electrons (Leapman & Andrews, 1992). We also examined multilayer specimens composed of carbon and metals. Aluminum and silver have low cohesive energies and should undergo sputtering by electrons of kinetic energy above 130 keV. Tungsten has a high cohesive energy and high atomic number and is not expected to sputter at incident energies below 500 keV.

These simple elements are believed to be immune from radiolytic processes that arise from inelastic scattering, allowing the sputtering process to be studied on its own. In contrast, compounds often suffer from radiolysis, which can greatly increase the thinning rate produced by an electron beam (Salisbury et al., 1984; Egerton et al., 1987; Hobbs, 1987; Hollenbeck & Buchanan, 1990). The use of a single element at the specimen surface also simplifies the interpretation of mass loss in terms of sputtering, avoiding complications due to differential sputtering rates and bulk diffusion processes (Mansfield et al., 1987; Medlin et al., 1989; McCartney et al., 1990).
Although rates of electron-induced sputtering can be predicted from calculated cross sections for elastic scattering (Bradley, 1988a, 1988b), some of the assumptions involved in these calculations are open to question (Sigmund, 1969). Therefore, experimental determinations of sputtering rate are valuable in addressing these questions.

**MATERIALS AND METHODS**

Most of our measurements were made in a JEOL-2010F TEM, at 200 kV accelerating voltage and with the Schottky-emission electron source operating at an anode (A2) potential of 7.4 kV. The condenser lenses were operated in STEM mode (1 nm HR probe) with a 40-μm-diameter condenser aperture.

To avoid the possibility of hydrocarbon contamination in the beam, specimens were left for 30 to 60 min under a heat lamp before insertion in the TEM. In addition, each specimen was flooded with a broad-area electron beam (condenser aperture removed) for 30 min before the start of measurements, to polymerize any hydrocarbon molecules adsorbed on the surface and prevent their diffusion toward the electron probe. The liquid nitrogen cold finger of the TEM was used to reduce the partial pressure of hydrocarbons and water vapor around the specimen, and thereby reduce the risk of beam-induced contamination or etching.

It is not easy to measure the current density in a small high-intensity electron probe. In fact, the current density falls continuously from the center of the probe as a result of spherical aberration of the condenser lenses and objective pre-field. The total probe current was measured by recording a slightly defocused image on a Gatan CCD camera, with short (0.1 s or 0.05 s) integration time and assuming 1.0 diode counts per incident electron (estimated from a previous calibration). The resulting values (in the range 1.6–5.1 nA) were within 15% of those deduced from the current absorbed by the focusing screen of the TEM (previously calibrated using a Faraday cup). The beam current remained stable (to within 10%) over several hours.

The effective diameter of the probe was estimated to be about 1.5 nm, based on the diameter of the fast Fourier transform (FFT) of several STEM images of a tungsten-coated carbon film, and also from scans of a high-magnification image of the probe across the edge of the TEM focusing screen. The current density at the center of the probe was thereby estimated to be \( 5 \times 10^4 \) A/cm\(^2\).

Reimer (1997, p. 81) gives the current density for a 100-kV Schottky source (virtual source diameter 15 nm) as 500 A/cm\(^2\). At 200 kV and after demagnification to 1.5 nm, the current density should therefore be \( 1 \times 10^5 \) A/cm\(^2\) in the absence of aberrations. If spherical aberration predominates, the fraction of current lying outside a radius \( r \) is

\[
f = \left( \frac{r}{r_s} \right)^{2/3} \quad \text{(Egerton & Crozier, 1997)}
\]

where \( r_s = C_a \alpha^3 \) = 1.2 nm for \( C_a = 0.5 \) mm and \( \alpha = 13.5 \) mrad, giving \( f = 0.87 \)

for \( r = 1 \) nm and a current density \( 8.7 \times 10^4 \) A/cm\(^2\), in rough agreement with our experimental estimate.

With fine-probe measurements, drift of the probe or of the specimen during a measurement is always a potential problem. We therefore made careful observation of the STEM image to ensure that the drift rate was sufficiently low.

**EXPERIMENTAL RESULTS**

Our first test consisted of focusing the probe on a vacuum-evaporated carbon film and recording the time \( T \) required to form a hole in the film. Due to the diminishing current density in the tails of the electron probe, the hole diameter increases with increasing irradiation time, allowing the hole diameter to be plotted as a function of time, as in Figure 1a.

Measured by EELS, the thickness of the carbon film was

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**Figure 1.** a: Hole radius as a function of time, for irradiation of a 20-nm carbon film in a 200-keV Schottky-emission probe. b: Carbon thinning rate as a function of radial distance from the center of the probe, estimated from a.
15 nm. Dividing this value by the time to form a hole of a given radius, the thinning rate can be plotted as a function of the radius from the center of the beam; see Figure 1b. The thinning rate close to the center of the beam is seen to be about 2 nm/s.

An alternative way of measuring a thinning rate is from core-loss EELS. The areal density of a particular element can be found by recording an ionization edge characteristic of that element, subtracting a noncharacteristic background (by extrapolation of the pre-edge intensity) and integrating the core-loss intensity over a 50-eV window above the ionization threshold. This procedure was applied to 15-nm carbon films that had been coated on one side with a 10-nm layer of evaporated silver or on both sides with approximately 1 nm of tungsten. The K-edge was used to monitor carbon and the M-edge for silver.

The results are shown in Table 1. The thinning rate for carbon (0.65 nm/s) is lower than shown in Figure 1b, probably reflecting a slightly lower current density but also the protective effect of the silver, which prevents carbon being removed from one of the two surfaces. It is generally believed that electron-induced sputtering from a thin specimen takes place mainly at the beam-exit surface, because the initial momentum transfer by a high-angle backscattered electron is in the direction of the incident electron (Crozier et al., 1990). However, the carbon data in Table 1 do not support this assumption; essentially the same carbon-sputtering rate was observed when the specimen was inverted, placing the silver on the top (beam-entrance) surface.

In contrast, the EELS measurements of the areal density of silver showed a distinct top/bottom effect. The Ag removal rate was positive on the beam-exit surface and negative (increase of Ag M-edge signal with time) with silver on the beam-exit surface. This accumulation of silver implies some form of diffusion from surrounding areas of the specimen.

Figure 2 shows EELS measurements of the removal of aluminum from a bilayer sample prepared by vacuum evaporation of 32 nm of Al onto a 20-nm carbon film. These tests were performed with a JEOL-2010 TEM equipped with a LaB₆ electron source, which provides a lower current density (about 2000 A/cm²) in the focused probe. With Al on the beam-exit surface, the Al thinning rate was around 0.2 nm/s. With carbon on the beam-entrance surface, the Al K-edge signal remained constant for about 2 min, then decreased at about the same rate (~0.3 nm/s). Evidently the carbon acts as a sacrificial layer, giving temporary protection against sputtering from the beam-exit surface.

We had hoped that a thin layer of tungsten on both surfaces might completely protect a light-element sample from sputtering by 200-keV electrons, because the threshold energy required for sputtering tungsten is as high as 500 keV. Instead, our EELS results indicated a loss of carbon, at a rate that varied considerably between individual measurements but generally higher than for silver coating a single surface; see Table 1. Examination of the film at high magnification showed that the tungsten does not uniformly coat the surface but occurs in small (<2 nm) crystallites. There is, therefore, a high probability that a gap between crystallites occurs within the area of the electron probe, allowing carbon atoms to escape into the vacuum. In addition, we believe that the tungsten was partially oxidized; its oxide layer would almost certainly damage radiolytically.

Our silver and aluminum films also showed an island structure but on a coarser scale. The EELS measurements were made by positioning the electron probe on an island rather than in a gap between islands.

### The Sputtering Process

Sputtering occurs when momentum is transferred to an atomic nucleus by Coulomb interaction with an incident electron, generally referred to as elastic scattering. The pro-
cess is less efficient than with incident ions because only high-angle electron collisions (which are relatively rare) can transfer enough momentum to eject a surface atom. Even so, electron-induced sputtering has been invoked to explain the thinning of a variety of materials, including gold (Cherns et al., 1976), zinc (Bradley & Zaluzec, 1988), TiC (Medlin et al., 1989), TiO₂ (Crozier et al., 1990), and carbon (Leaman & Andrews, 1992), as well as transition metal carbides, oxides, and nitrides (Thomas, 1985).

During elastic scattering by an atom, an electron that undergoes an angular deflection θ transfers to the nucleus an amount of energy equal to

\[ E = E_{\text{max}} \sin^2(\theta/2), \]  

(1)

where \( E_{\text{max}} \) is the maximum possible energy transfer, corresponding to \( \theta = \pi \) rad. Relativistic kinematics gives

\[ E_{\text{max}} = 2E_0(E_0 + 2m_0c^2)/Mc^2, \]

(2)

where \( M \) is the mass of the scattering atom, assumed initially at rest, and \( E_0 \) is the kinetic energy of the incident electron (rest mass \( m_0 \)); \( c \) is the speed of light in vacuum and \( m_0c^2 = 511 \text{ keV} \) is the electron rest energy.

Neglecting screening of the nuclear field, which is a good approximation for large scattering angles, the differential cross section for such Rutherford-type scattering is

\[ \frac{d\sigma}{d\theta} = \frac{e^2Z^2/(8\pi e_0 E_0)^2 [(E_0 + m_0c^2)/(E_0 + 2m_0c^2)]^2}{2\pi \sin \theta / \sin^4(\theta/2)}. \]  

(3)

This expression can be integrated over the scattering angle, from \( \theta = \pi \) to a minimum value given by \( \sin^2(\theta/2) = E/E_{\text{max}} \) to give a cross section for energy transfer in the range \( E_{\text{min}} \) to \( E_{\text{max}} \):

\[ \sigma = \left(2.45 \times 10^{-29} \text{ m}^2\right)Z^2 \left[1 - \frac{\nu^2/c^2}{(v^2/c^2)}\right] \times [(E_{\text{max}}/E_{\text{min}}) - 1], \]

(4)

where \( \nu \) is the speed of the incident electron. By setting \( E_{\text{min}} \) equal to the surface binding energy \( E_s \) (tabulated for elements as their sublimation energy), equation (4) can be used to calculate the sputtering rate, assuming that sputtering involves only the transfer of energy to surface atoms.

The threshold incident-electron energy, \( E_{\text{th}} \), that is just sufficient to cause ejection of surface atoms is given by setting \( E_{\text{max}} \) equal \( E_s \) in equation (2), giving

\[ E_{\text{th}} = m_0c^2\left[1 + \left(M/m_0\right)E_s/(2m_0c^2)\right]^{1/2} - 1]. \]  

(5)

The sputtering cross section \( \sigma \) is zero at \( E_0 = E_{\text{th}} \) and increases to a maximum somewhere between 1.5\( E_{\text{th}} \) and 2\( E_{\text{th}} \), then gradually decreases with increasing incident energy.

Equations (3) and (4) ignore effects due to electron spin. Solution of the Dirac equation indicates that the Rutherford cross section given by equation (5) should be multiplied by a factor \( r(\theta) \), given approximately (McKinley & Feshbach, 1948) by

\[ r(\theta) = 1 - \left(\frac{\nu}{c}\right)^2 \sin^2(\theta/2) \]

\[ + \pi(Z/137)(\nu/c)\sin(\theta/2)[1 - \sin(\theta/2)]. \]  

(6)

By incorporating \( r(\theta) \) and integrating equation (3) over the angular range, we obtain Mott cross sections for sputtering of aluminum that are within 1% of those calculated by Bradley (1988a) using more exact procedures. However, equation (6) does represent a low-Z approximation and could underestimate the cross section by as much as a factor of 2 for a heavy element such as gold (Bradley, 1988b).

Taking the minimum energy transfer for sputtering as the surface-binding energy \( E_s \) implies that escape of a surface atom does not depend on the direction of the momentum transfer, in other words, the angle \( \phi \) (relative to the incident direction) at which the scattering atom starts to move. This assumption implies that neighboring atoms elastically reflect an excited surface atom or that the surface atom is subject to a radially symmetric (spherical) potential, an approximation that is defended by Sigmund (1969). Elastic reflection may imply that sputtering should occur equally from the top and bottom surfaces of a thin specimen.

An alternative assumption is that there is a planar potential step at the surface, in which case only the momentum component \( (2ME)^{1/2} \cos \phi \) perpendicular to the surface (corresponding to an energy \( E \cos^2 \phi \)) can be used to overcome the surface-potential barrier (height \( E_s \) and escape of the atom requires \( E \cos^2 \phi > E_s \). Because \( \theta = \pi - 2\phi \), equation (1) implies \( E = E_{\text{max}} \cos^2 \phi \) and escape will occur if \( E_{\text{max}} \cos^2 \phi > E_s \). In this case, escape involves a maximum value of \( \phi \) given by \( \cos(\phi_{\text{max}}) = (E/E_{\text{max}})^{1/4} \), rather than \( \cos(\phi_{\text{max}}) = (E_{\text{th}}/E_{\text{max}})^{1/2} \), which would apply if the direction of the momentum were unimportant. The minimum energy exchange for sputtering is now

\[ E_{\text{min}} = (E_sE_{\text{max}})^{1/2} \]

(7)

and the cross section for sputtering is given by equation (4) with \( E = E_{\text{min}} \) rather than \( E = E_s \). Interestingly, measured cross sections for electron sputtering of gold (Cherns et al., 1976) lie intermediate between the values we calculate assuming \( E_{\text{min}} = (E_sE_{\text{max}})^{1/2} \) and those calculated with \( E_{\text{min}} = E_s \).

There is also the possibility that momentum transferred to subsurface atoms can be transmitted to a surface atom. In the case of a crystalline material, this process leads to focused collision sequences in which energy is channeled in particular directions. Such effects are known to be important in determining the sputtering yield from a target bombarded with ions (Sigmund, 1969). But molecular-dynamics
modeling of electron-induced sputtering from crystalline (111) gold foils has indicated that subsurface collisions increases the sputtering yield by no more than a factor of 2, and only for incident electron energies well above the threshold (Cherns et al., 1977). We therefore neglect the contribution of subsurface collisions in our single-element specimens.

If sputtering arises only from electron collisions with exit-surface atoms, corresponding to a sputtering cross section $\sigma$, the sputtering rate $S$ for an element of atomic weight $A$, density $\rho$ can be calculated as

$$S = \frac{J}{e} \sigma \left( \text{thickness of 1 monolayer} \right)$$

$$= \left( \frac{J}{e} \sigma \right) (\text{volume per atom})^{1/3} = \left( \frac{J}{e} \sigma [u A / \rho] \right)^{1/3} \left( \frac{J}{e} \sigma [u A / \rho] \right)^{1/3} \left( \frac{J}{e} \sigma [u A / \rho] \right)^{1/3} \left( \frac{J}{e} \sigma [u A / \rho] \right)^{1/3} \left( \frac{J}{e} \sigma [u A / \rho] \right)^{1/3},$$

where $J$ is the incident-electron current density, $e$ the electronic charge, $u$ the atomic mass unit, and $A$ the atomic weight of the element. Calculated values of $S$ for $J = 5 \times 10^4$ A/cm$^2$ and an amorphous carbon specimen ($\rho = 1.8$ g/cm$^3$) are shown in Figure 3, for comparison with our experimental data. The calculated thinning rate (exceeding 1 nm/s at $E_0 = 200$ keV) indicates that sputtering should be significant for a high-intensity electron probe. The approximate agreement with the experimental data points lends support to the interpretation of the observed thinning effect in terms of sputtering.

**Discussion**

Our experiments suggest that, under a focused electron beam, carbon is removed at a similar rate from the entrance and exit surfaces of an uncoated foil. Therefore it appears that equation (7) does not apply to amorphous carbon. Because carbon films are known to be slightly porous, a planar-potential approximation at the surface may be inappropriate. On the other hand, taking $E_{\text{min}} = E_s$ gives a calculated sputtering rate higher than the observed thinning rate, especially if sputtering is assumed to occur equally from both surfaces; see Figure 3. Although we took steps to minimize contamination effects, it is possible that the carbon loss might be partially compensated by surface diffusion of hydrocarbons toward the beam.

An alternative explanation of the carbon-film thinning is oxidation: reaction of carbon atoms with residual water vapor, catalyzed by the presence of the electron beam. This could explain the equal thinning rate from both surfaces, but it raises two questions: Why does sputtering not occur at the rate expected and why does the oxidation rate approximately match the calculated sputtering rate? In any event, oxidation is unlikely to account for the thinning of silver and aluminum, which have solid oxides.

In the case of a silver surface, calculations based on Mott cross sections predict a sputtering rate (for $J = 5 \times 10^4$ A/cm$^2$ and $E_s = 2.95$ eV, the Ag sublimation energy) of 17 nm/s if we take $E_{\text{min}} = (E_s E_{\text{max}})^{1/2}$ and even higher if $E_{\text{min}} = E_s$. Our measured thinning rate with silver on the beam-exit surface was only 0.32 nm/s, and with silver on the entrance surface we observed a net accumulation of Ag within the beam. These results make sense if sputtering from the surface is nearly compensated by the diffusion of silver from the surroundings. The different behavior with silver on the top and bottom surface implies a higher Ag removal rate in the latter case, consistent with sputtering from a planar-potential surface, $E_{\text{min}} \approx (E_s E_{\text{max}})^{1/2}$.

The activation energy for surface diffusion of silver is reported to be 0.3 eV for Ag on Ag (Hummel & Geier, 1975) and 0.32 eV for Ag on SiO$_2$ (Kim et al., 2002). These low values seem consistent with the relatively low sublimation energy of silver (1.95 eV). Diffusion of Ag on Pd(100) has an activation energy of 0.35 eV and a surface diffusion coefficient around $D_s = 25$ nm$^2$/s at room temperature (Felix et al., 1995). This would provide a diffusion length $\approx (D_s T)^{1/2} = 22$ nm for $T = 20$ s, the time period involved in our experiments. Therefore it seems reasonable to expect radial surface diffusion of silver into a 1-nm-diameter beam, driven by the local curvature of the surface produced by sputtering. Considering a steady-state solution of the radial diffusion equation (Wall, 1980), the inward diffusion rate would nearly balance the sputtering rate (taken to be 17 nm/s) if Ag atoms migrate toward the beam over a radial distance of about 4 nm.

For an amorphous carbon film, whose thermal conductivity is modest ($\kappa \approx 1.6$ W/m/K), the temperature rise in a 5-nA 1-nm-diameter beam (due to inelastic scattering) can be calculated to be less than 2 K (Egerton et al., 2004). Because silver has a higher thermal conductivity, the temperature rise in the Ag/C sample should be even less and
therefore heating by the electron beam is unlikely to affect the diffusion rate. Radiation enhanced diffusion (Ma & Marks, 1986) is a possibility; however the materials we are studying are good electrical conductors, so atomic migration is probably not driven by a local electric field, as postulated for some oxides (Humphreys et al., 1990; Cazaux, 2004).

Taking $E_{\text{min}} = E_e = 2.95$ eV for aluminum, we calculate a sputtering rate of 1.2 nm/s per surface for $J = 2000$ A/cm$^2$, the measured current density at the center of our LaB$_6$ electron probe. But assuming $E_{\text{min}} = (E_e E_{\text{max}})^{1/2}$, the sputtering rate is 0.30 nm/s, which matches fairly well our observed Al thinning rate of 0.2 nm/s. Therefore sputtering from an exit surface with a planar potential appears to be appropriate for aluminum. The complete (although temporary) protective effect of a carbon layer on the exit surface is consistent with this conclusion. In a microscope with poor vacuum, a contamination layer at the surface would provide similar protection.

The tungsten-coated carbon specimen suffered thinning rates not appreciably different from those of uncoated carbon, which we attribute to the fact that the coating appeared discontinuous on a few nanometers scale. The only common elements that have a sputtering-threshold energy above 200 keV are Zr, Nb, Mo, Ta, W, Pt, Au, and Pb. The last two form thin films that are discontinuous up to a quite large thickness, making them unsuitable for protecting a surface from sputtering. The other elements have a high melting point and form fine-grained films, but our experience with tungsten suggests that they might also be ineffective in providing a sputtering barrier.

Recently, Sherman and Chiu (2003) deposited 1.5-nm coatings of Ti$_4$Si$_{12}$ alloy on both surfaces of a frozen-hydrated TEM specimen to prevent electrostatic charging. Their coating was amorphous and did not significantly interfere with the TEM image or diffraction pattern. Unfortunately both Ti and Si have sputtering thresholds below 100 keV, so such a coating would not form a protective layer.

In the case of a binary compound, the low-Z element is likely to sputter preferentially, resulting in nonstoichiometry in the irradiated area and a concentration gradient (Medlin et al., 1989). Unless diffusion is fast enough, sputtering of the lighter element is then reduced. However, most compounds undergo decomposition via a radiolytic mechanism, which can result in a thinning rate much faster than that provided by sputtering.

Expressed in terms of the electron dose $D$ required to form a hole in a 20-nm sample, our thinning rates of carbon and aluminum are equivalent to $D \approx 3 \times 10^5$ C/cm$^2$. This is high compared to the dose required for radiolysis of organic compounds, which is typically below 1 C/cm$^2$. For microanalysis purposes, the damage rate due to the beam can be reduced by reducing the beam current whenever the probe is stationary. In the case of core-loss EELS, the core-loss acquisition rate can exceed $3 \times 10^8$ e/channel/s, equivalent to $10^8$ core-loss electrons/s if the core-loss spectrum is spread over 300 channels. Elemental analysis under high-flux conditions is therefore possible, but will be limited in practice because of mass loss due to sputtering or radiolysis.

**Conclusions**

Subjected to the high-intensity probe produced by a field-emission source, even single-element TEM specimens are likely to undergo mass loss, leading to hole formation. For a thin carbon specimen in a 200-kV Schottky-emission TEM, our observed thinning rate exceeded 1 nm/s. With an aberration-corrected probe, the thinning rate could be more than a factor of 10 higher. The sputtering threshold for carbon is below 40 keV, so thinning by sputtering is not avoided by reducing the beam energy.

We observed carbon disappearing from both surfaces, at a rate intermediate between that calculated for sputtering with planar and spherical surface potentials. Possibly the sputtering conditions are different from those of a metal because of the porous nature of the carbon. Oxidation is also a possibility, if the vacuum system contains traces of water vapor.

Aluminum appears to sputter from the beam-exit surface only. As a result, a carbon or contamination layer on this surface will provide temporary protection. The Al thinning rate was in rough agreement with sputtering theory, assuming a planar potential step at the surface.

Silver sputters preferentially from the beam-exit surface but at a low rate, which we attribute to surface diffusion of material from the surroundings. We actually measured an accumulation of silver at the beam-entrance surface.

A 1-nm layer of tungsten on both surfaces of a carbon film failed to protect the carbon from erosion in the beam, apparently because of the nanocrystalline nature of the coating. Possibly a thicker coating layer would be more effective, but more likely to interfere with TEM imaging and diffraction. Complete protection from sputtering appears to require a truly amorphous coating of high-Z material.

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