# A Technique for Calibrating an Electron-Beam Evaporator X-Ray Source

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An electron-beam metal evaporator was modified to act as an x-ray source for simulating x-ray lithography induced radiation damage in semiconductor devices. Besides x-rays, such a set-up generates secondary and backscattered electrons. Simulation of radiation damage induced during a typical x-ray lithography processing step requires that secondary and backscattered electrons be removed from the incident radiation.

A common method to separate the electrons and x-rays is to block the electrons with a thin beryllium film. However, thin beryllium films are expensive and are not commercially available in large sizes. Instead, a relatively inexpensive polymer pellicle was used to block the electrons. A disadvantage of the pellicle is that its x-ray transmission characteristics are unknown and must be determined experimentally. Previous works calibrated the pellicle x-ray transmission and system electron/x-ray flux ratios using the known x-ray transmission characteristics of beryllium film. To check the accuracy of the beryllium calibration results, a new calibration method employing two pellicles was developed. A comparison of the calibration data found a large discrepancy between the two calibration methods. The discrepancy was attributed to ultraviolet light absorption in the radiation dosimeters. A correction for the ultraviolet light absorption is presented for determining the true electron/x-ray flux ratios and pellicle x-ray transmission.

**Key words:** Controlled x-ray source, x-ray lithography, x-ray radiation damage, do-simetry

# INTRODUCTION

A paper by Bhattacharya et al. described a procedure for setting-up a controlled x-ray source using an electron-beam metal evaporator. With aluminum as the electron-beam target material, the x-ray output is peaked at the characteristic aluminum  $K\alpha$  x-ray energy of 1.5 kev. Aluminum  $K\alpha$  x-rays are ideal for simulating the radiation damage from an x-ray lithography processing step, since the aluminum  $K\alpha$  x-ray energy lies in the middle of the proposed energy range of 1 to 2 keV for synchrotron x-ray aligners.

Unfortunately, in addition to aluminum  $K\alpha$  x-rays, secondary and backscattered electrons are produced. As a result, the radiation incident on the sample is a mixture of electrons and x-rays. The mixture of electrons and x-rays is not representative of the radiation in an x-ray lithography process. It has been shown<sup>2</sup> that the fixed positive charge in the gate insulator of a insulated gate field effect transistor (IGFET) tends to saturate when irradiated with both x-rays and electrons simultaneously, and that the electrons generate significant quantities of fixed negative charge in the gate insulator. Therefore, to simulate x-ray induced damage, the electrons must be removed from the incident radiation.

The simplest method to eliminate electrons from the incident radiation is to block, or filter the electrons with a thin film of beryllium. Beryllium film is commonly employed as an electron-filtering material since its low atomic number causes little x-ray absorption. However, beryllium film has several disadvantages as an electron filter. First, thin films of beryllium are only available in sizes up to 50 by 50 mm. As a result, only small portions of a silicon wafer can be irradiated, unless multiple films are used. Secondly, thin films of beryllium are relatively expensive. A 50 mm by 50 mm by 12.5 micron film costs approximately 1000 dollars.

Bhattacharya<sup>1</sup> demonstrated that a polymer pellicle is also a suitable electron filter material. The pellicles are designed to act as dust covers for projection aligner photomasks. The advantages of the pellicle over the beryllium film as an electron-filtering material are: 1) the pellicle frames are available in sizes up to 150 mm in diameter, thereby allowing larger samples to be irradiated, and 2) the pellicles are relatively inexpensive; approximately 75 dollars for a 100 mm square, uncoated pellicle.

A disadvantage of the pellicle is that its x-ray transmission is unknown and must be determined experimentally, whereas the beryllium film's x-ray transmission is well known at all x-ray energies. In this experiment, the pellicle x-ray transmission and the electron/x-ray flux ratios were determined at various electron-beam currents using two different calibration methods. The first method was previously explained by Bhattacharya et al. and employs the beryllium film. The second method was developed as a check on the accuracy of the first calibration method results. A significant discrep-

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ancy between methods was found and attributed to ultraviolet (UV) light absorption in the radiation dosimeter. A method is presented to correct for the calibration error caused by the UV light absorption.

#### EXPERIMENTAL APPARATUS

The beryllium film used for the calibration measured 50 mm by 50 mm by 12.5  $\mu$ m thick. This was the largest area and thinnest film available from Cerac, Inc. (P.O. Box 1178, Milwaukee, WI 53201). The pellicles were made by Dupont Tau Laboratories, Inc. (P.O. Box 2956, Poughkeepsie, N.Y. 12603) and were non-optical coated nitrocellulose, nominally 2.85 microns thick, with a thickness tolerance of plus or minus 0.2 microns.3 The electron filter material must be thicker than the maximum penetration depth of the highest energy electron. Bhattacharya et al. used previously reported electron penetraton data4 and equations for electron range as a function of electron energy<sup>5</sup> to calculate a maximum penetration depth in each filter material. The calculated electron range for 10 keV electrons was 1.55  $\mu$ m in the polymer film and 1.35  $\mu$ m in beryllium.

The electron-beam evaporator system was a Cooke Vacuum Products, model MK-VIIEB evaporator with a 5 kV, 750 mA electron-gun power supply. The 5 keV electrons had sufficient energy to create the 1.487 keV aluminum  $K\alpha$  x-rays, but insufficient energy to penetrate the beryllium or pellicle films.

The dosimeters used for all measurements were radiachromic dye films manufactured by Far West Technology, Inc. (330-D South Kellogg, Goleta, CA 93117). The dosimeters consist of a clear nylon film 1 cm by 1 cm by 50  $\mu$ m thick which is impregnated with a radiation-sensitive dye. Upon irradiation, the dosimeter becomes blue in color. The absorbed dose in the dosimeter is proportional to the change in the optical density at wavelengths of 605 or 510 nm. The radiachromic dye dosimeters have many excellent characteristics including: long-term stability after irradiation, a wide range of measurable doses (0.1 to 10 MRads), and nearly equal sensitivity to electrons and aluminum Kα x-rays for incident energy fluences up to 200 mJ/cm2<sup>6</sup> (all incident x-ray energy fluences were less than 100 mJ/cm2). The radiachromic dye dosimeters have several disadvantages however, the most important being a sensitivity to ultraviolet (UV) light. Exposure to UV light during or after irradiation will cause a significant error in determining the exact radiation dose due to x-rays or electrons. The sensitivity of the dosimeter to UV is unknown at this time, however, it was assumed that the UV response is linear over the exposure and energy fluence ranges encountered in the experiment.

#### EXPERIMENTAL PROCEDURE

The first calibration method used to determine the electron/x-ray flux ratios and the pellicle x-ray

transmission was based on the known x-ray transmission of the beryllium film. This method was previously described by Bhattacharya *et al.*<sup>1</sup> and is referred to here as the beryllium calibration method. The second, new calibration method did not use the beryllium film, but instead employed two pellicles simultaneously. Hence, the second method is referred to as the two-pellicle calibration method.

The beryllium calibration method consisted of three equivalent dose calibration runs at each electron-beam current investigated. Figure 1 and 2 show diagrams of the experimental set-up for the beryllium and two-pellicle calibrations, respectively.

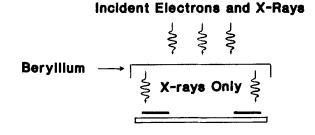
In all three calibration runs, the irradiation time and electron-beam current were closely monitored to assure equivalent total doses between runs. Two additional dosimeters outside the periphery of the beryllium or pellicle (not shown in Figs. 1 or 2) were used to measure the total dose of each run.

The first run of the beryllium calibration measured the total energy fluence as registered by the radiachromic dye film. In the second run, the dosimeters were shielded from the incident electrons

### Run #1: No Beryllium or Pellicle

# Incident Electrons and X-Rays Doslmeters Base Plate

# Run #2: Beryllium Film Only



# Run #3: Pellicie Only

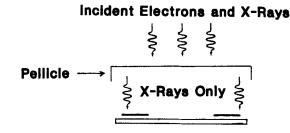
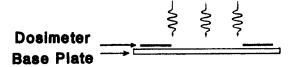


Fig. 1 — Beryllium calibration of pellicle.

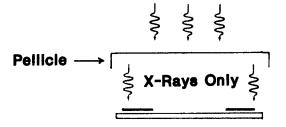
### Run #1: No Pellicle

# Incident Electrons and X-rays



#### Run #2: One Pellicie

# Incident Electrons and X-Rays



# Run #3: Two Pellicles

# Incident Electrons and X-Rays

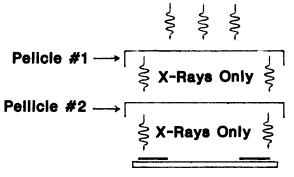


Fig. 2 — Two-pellicle calibration.

by the beryllium film. Hence, the energy fluence measured in run two is due entirely to x-rays. After the second run, the incident x-ray energy fluence is determined with the following exponential relation:

$$E_{xb} = E_x \cdot \exp(-\rho_b \cdot X \cdot (\mu/\rho)_b) \tag{1}$$

where:  $E_{xb}$  is the x-ray energy fluence transmitted through the beryllium film,  $E_x$  is the incident x-ray energy fluence,  $\rho_b$ , is the density of the beryllium film, X is the thickness of the beryllium film, and  $(\mu/\rho)_b$  is the mass absorption coefficient of the beryllium film. Solving for the incident x-ray energy fluence yields:

$$E_{x} = \frac{E_{xb}}{\exp(-\rho_{b} \cdot X \cdot (\mu/\rho)_{b})} \tag{2}$$

Once the incident x-ray energy fluence is known, the fraction of the incident energy fluence due to electrons,  $E_e$  (hereafter referred to as the electron fraction), can be determined by:

Electron Fraction = 
$$E_e = \frac{E_T - E_x}{E_T}$$
 (3)

where:  $E_T$  is the total energy fluence measured by the radiometer in run one. In the third calibration run, the beryllium film was replaced with a single pellicle. Since all three calibration runs were carried out at equivalent electron gun currents and irradiation times, the incident x-ray energy fluence is the same for all calibration runs. Once the values of the incident x-ray energy fluence and the x-ray energy fluence transmitted through the pellicle were known, the x-ray transmission fraction,  $T_x$ , and mass absorption coefficient of the pellicle were determined by re-arranging Eq. 1:

$$T_x = \frac{E_{xp1}}{E_x} \tag{4}$$

where:  $E_{xp1}$  is the x-ray energy fluence transmitted through the pellicle. The pellicle mass absorption coefficient is given by:

$$(\mu/\rho)_p = \frac{-\ln(E_{xp1}/E_x)}{\rho_p \cdot X} \tag{5}$$

The two-pellicle calibration, like the beryllium calibration, also used three equivalent-dose calibration runs at the same beam currents as the beryllium calibration. Figure 2 shows a diagram of the experimental set-up for the three calibration runs. The first two-pellicle calibration run was identical to the first beryllium calibration run. The second calibration run used a single pellicle to measure the x-ray energy fluence transmitted through the polymer film. The third calibration run employed two stacked pellicles over the dosimeters. The purpose of pellicle number 1 was to filter the electrons from the incident radiation and pass only x-rays on to the second pellicle. The x-ray energy fluence transmitted through the first pellicle in run three was assumed to be equivalent to that measured with the single pellicle in run two. Therefore, the pellicle xray transmission was calculated with the following relations:

$$T_x = \frac{E_{xp2}}{E_{xp1}} \tag{6}$$

where:  $E_{xp2}$  is the x-ray energy fluence measured through both pellicles in run three. Furthermore, the mass absorption coefficient equals:

$$(\mu/\rho)_p = \frac{-\ln{(E_{xp2}/E_{xp1})}}{\rho_p \cdot X}$$
 (7)

Having determined the pellicle x-ray transmission, the incident x-ray energy fluence was then calculated using the single pellicle data from run two.

$$E_x = \frac{E_{xp1}}{T_r} \tag{8}$$

The electron fraction was calculated in the same manner as the beryllium calibration method:

$$E_e = \frac{E_T - E_x}{E_T} \tag{9}$$

#### RESULTS

#### A) Electron Fraction

The electron fraction as a function of the electron gun current for both the beryllium and two-pellicle calibration methods is shown in Fig. 3. It is readily seen from Fig. 3 that there is a large discrepancy between the two calibration methods. This discrepancy has been attributed to sensitivity of the dosimeter to UV light. The UV light is generated by the tungsten filament electron source in the electrongun, which is in direct line-of-sight with the dosimeters, and by the electron scattering process of Bremsstrahlung at the surface of the electron target. The correction for the UV light error is differ-

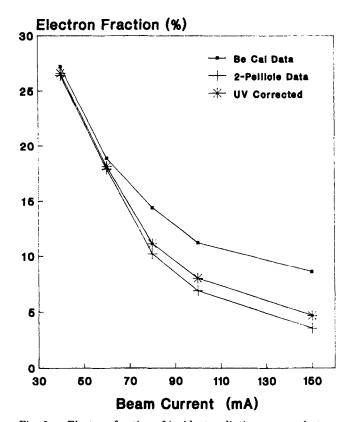


Fig. 3 — Electron fraction of incident radiation versus electron beam current using the beryllium and two-pellicle calibration methods. The UV corrected calibration data shows the extent of the calibration error.

ent in each calibration method because the beryllium film is opaque to UV, while the pellicle transmits a high percentage of the UV light. The average UV light transmission of the uncoated pellicle is 92% between the wavelengths of 350 and 450 nm.<sup>3</sup> The error caused by UV can be corrected by including a UV energy fluence variable and the UV transmission of the pellicle in the electron fraction and pellicle x-ray transmission calculations. Figure 4 shows the composition of the incident and measured energy fluences including UV, for all three calibration runs in both the beryllium and two-pellicle calibration methods.

For both calibration methods, the total energy fluence measured in the first calibration run is a combination of the x-ray, electron and UV energy fluences:

$$E_{\mathcal{T}} = E_x + E_e + E_m \tag{10}$$

The energy fluence measured in the second beryllium calibration run is due entirely to x-rays since the beryllium film blocks both the electrons and the UV light. As a result, the calculated incident x-ray energy fluence has no UV contribution. However, the UV energy is included in the calculated electron energy fluence:

$$E_e = E_T - E_x = (E_x + E_e + E_{uv}) - E_x$$
  
=  $E_e + E_{uv}$  (11)

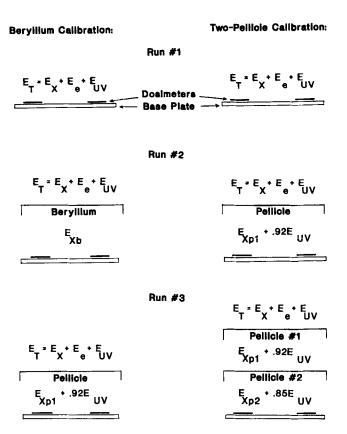


Fig. 4 — Beryllium and two-pellicle calibrations including UV energy.

Therefore, the electron fraction becomes:

Electron Fraction = 
$$\frac{E_e + E_{uv}}{E_T}$$
 (12)

Similarly, the effect of the UV on the two-pellicle calibration data also was determined. The first calibration run yielded the same results as the beryllium calibration. The second run employed a pellicle, therefore, the energy fluence measured is due to both x-rays and UV. The energy fluence measured in run two can be written as:

$$E_{xp1} = E_x \cdot C + 0.92 \cdot E_{uv} \tag{13}$$

where:  $E_x$  is the total incident x-ray energy fluence and C is the x-ray transmission fraction determined by the ratio of the dosimeter readings in run three to run two:

$$C = \frac{E_{xp2} + 0.85 \cdot E_{uv}}{E_{xp1} + 0.92 \cdot E_{uv}}$$
 (14)

For simplicity, it is assumed that the energy fluence due to x-rays is much greater than the energy fluence due to UV, and that a first approximation of the magnitude of C is 0.70. Substituting this value back into Eq. 13 yields:

$$E_{xp1} = 0.70 \cdot E_x + 0.92 \cdot E_{uv} \tag{15}$$

The calculated incident x-ray energy fluence,  $E_{xcalc}$ , is determined by dividing  $E_{xp1}$  by C:

$$E_{xcalc} = \frac{E_{xp1}}{C} = \frac{0.70 \cdot E_x + 0.92 \cdot E_{uv}}{0.70}$$
$$= E_x + 1.31 \cdot E_{uv}$$
(16)

This yields a calculated electron energy fluence of:

$$\begin{split} E_{ecalc} &= E_T - E_{xcalc} \\ &= (E_x + E_e + E_{uv}) - (E_x + 1.31 \cdot E_{uv}) \\ E_{ecalc} &= E_e - 0.31 \cdot E_{uv} \end{split} \tag{17}$$

The electron fraction then becomes:

Electron Fraction = 
$$\frac{E_e - 0.31 \cdot E_{uv}}{E_T}$$
 (18)

Thus, the true electron fraction is measured by neither the beryllium nor the two-pellicle calibration methods. The true electron fraction can be calculated from the difference between the two curves, D:

$$D = (Electron Fraction)_{Be}$$

$$-$$
 (Electron Fraction)<sub>2Pell</sub> (19)

$$D = \frac{E_e + E_{uv}}{E_T} - \frac{E_e - 0.31 \cdot E_{uv}}{E_T}$$
 (20)

$$D = \frac{1.31 \cdot E_{uv}}{E_T} \tag{21}$$

Solving for  $E_{uv}$  yields:

$$E_{uv} = \frac{D \cdot E_T}{1.31} \tag{22}$$

 $E_{uv}$  can be quantified and corrected for with Eq. 22 since the magnitude of D and  $E_T$  are known. The UV corrected electron fraction curve is shown in Fig. 3 along with the beryllium and two-pellicle curves.

#### B) Pellicle X-ray Transmission:

Figure 5 shows the pellicle x-ray transmission results obtained with the beryllium and two-pellicle calibration methods. Theoretically, the pellicle x-ray transmission should be constant for all beam currents, since the energy distribution of the x-rays is independent of the beam current. The data appears to confirm this, although there is some scatter due to measurement inaccuracies. The average x-ray transmission calculated with the beryllium calibration method was 78.3% while the two-pellicle method averaged 71.4%. This discrepancy between calibration methods also is attributed to the UV light sensitivity of the dosimeter. A UV light correction analogous to that derived for the electron fraction calculation is described below.

For the beryllium calibration, the x-ray transmission including UV effects is given by:

$$T_x = \frac{(E_{xp1} + 0.92 \cdot E_{uv})}{E_x} \tag{23}$$

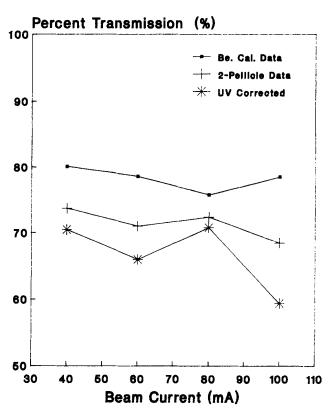


Fig. 5 — Pellicle percent x-ray transmission versus electron beam current using the beryllium and two-pellicle calibration methods. UV corrected calibration data shows the extent of the calibration error.

686 Kinzig

where:  $(E_{xp1} + 0.92 \cdot E_{uv})$  is the x-ray energy fluence transmitted through the pellicle in run three.

For the two-pellicle method, the calculated x-ray transmission including UV effects is given by:

$$Tx = \frac{(E_{xp2} + 0.85 \cdot E_{uv})}{(E_{xp1} + 0.92 \cdot E_{uv})}$$
(24)

where: the numerator is the energy fluence measured in run three and the denominator is the energy fluence measured in run two. The magnitude of the UV energy correction is determined from the difference between the beryllium and two-pellicle curves, similar to the electron fraction calculations.

$$D = T_{rRe} - T_{r2Pell} \tag{25}$$

$$D = \frac{(E_{xp1} + 0.92 \cdot E_{uv})}{E_x} - \frac{(E_{xp2} + 0.85 \cdot E_{uv})}{(E_{xp1} + 0.92 \cdot E_{uv})}$$
(26)

Obtaining a common denominator:

$$D = \frac{(E_{xp1} + 0.92 \cdot E_{uv})^2 - E_x \cdot (E_{xp2} + 0.85 \cdot E_{uv})}{E_x \cdot (E_{xp1} + 0.92 \cdot E_{uv})}$$
(27)

But,

$$E_x = \frac{E_{xp1}}{K} \tag{28}$$

and

$$E_{rp2} = E_{rp1} \cdot K \tag{29}$$

where: K is the x-ray transmission of the pellicle and is assumed to be 0.70 as a first approximation. Substituting these relations into Eq. 27 and reducing algebraically yields:

$$D = \frac{0.63 \cdot E_{xp1} + 0.85}{(1.43 \cdot E_{xp1}^2)/E_{uv} + 1.31 \cdot E_{xp1}}$$
(30)

Solving for  $E_{uv}$ :

$$E_{uv} = \frac{D \cdot E_{xp1}^2}{E_{xp1} \cdot (0.44 - 0.92 \cdot D) + 0.59}$$
(31)

The UV corrected pellicle x-ray transmission data is plotted along with the uncorrected data in Fig. 5. The average UV corrected x-ray transmission of the pellicle was 67.9 percent.

#### CONCLUSION

An electron-beam metal evaporator was set-up as an x-ray source to simulate x-ray lithography induced radiation damage in semiconductor devices. The electron/x-ray flux ratios and pellicle x-ray transmission were calibrated at various electron-beam currents using the previously described beryllium calibration method and a new two-pellicle method. A significant error, caused by the sensitivity of the dosimeter to ultraviolet light, was found in both calibration methods. A procedure is presented for correcting the UV light absorption error in the electron/x-ray flux ratios and pellicle x-ray transmission results.

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#### REFERENCES

- P. K. Bhattacharya, A. Reisman and M. C. Chen, J. Electron. Mater. 17, 273 (1988).
- A. Reisman and C. J. Merz, J. Electrochem. Soc. 130, 1384 (1983).
- Dupont Tau Laboratories, Inc. data sheet for type 1 uncoated nitrocellulose pellicles.
- D. R. Beaman and J. A. Isasi, "Electron Beam Microanalysis—Part I," Mater. Res. and Standards, (Vol. 11, p. 8, 1971).
- T. E. Everhart and P. H. Hoff, J. Appl. Phys. 42, 5837 (1971).
- J. R. Maldonado, A. Reisman, H. Lezec, B. Bumble, C. K. Williams and S. S. Iyer, J. Vac. Sci. Technol. B5, 248 (1987).