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MEASUREMENT OF FILM THICKNESS ON INTEGRATED CIRCUITS USING ENERGY DISPERSIVE X-RAY ANALYSIS (EDXA)

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Abstract

The need for a rapid, non-destructive failure analysis of complex integrated circuits has led to the use of energy dispersive x-ray analysis (EDXA), to measure the localized thickness of SiO_2 and Al films on integrated circuits, by detecting the penetration threshold of the electron beam through the film. The accuracy (10%-20%) is determined by actual Scanning Electron Microscope (SEM) measurements, traceable to a secondary length standard, of the film thicknesses of sectioned samples. In contrast, another SEM based thickness measurement technique, such as Yakowitz-Newbury method, gave results that were 50-100% larger, required higher accelerating voltage that would damage the integrated circuit and took longer to setup/perform measurement. Typically, the thickness of the films range from 0.3 to 1.5 microns and the e-beam energy varies from 4 keV to 20 keV.

KEY WORDS: Film Thickness on Integrated Circuits, Scanning Electron Microscope (SEM), Energy Dispersive X-ray Analysis (EDXA), Penetration Voltage, Thickness Measurement, Length Standard.

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Introduction

During the course of failure analysis or quality inspection and evaluation of microelectronic parts, a distinct need to measure metallization or oxide thickness is required to evaluate the processing of chips and to perform failure analysis. Thus, a program was developed to evaluate available laboratory instrumentation to perform film thickness measurements on packaged integrated circuits. A laser ellipsometer and an optical interferometer were evaluated and found to be unsuitable for this application. The available ellipsometer was unsuitable due to the large spot size and nonuniformity of the integrated circuit surface. The Nomarski interferometer, although better at localizing steps on the surface of chip and other chip details, was also unsuitable because of the sharpness of the steps and merging of the fringe lines.

As a result of these investigations, techniques were examined that use the Scanning Electron Microscope (SEM) in conjunction with Energy Dispersive X-ray Analysis (EDXA). The Yakowitz-Newbury method (Yakowitz-Newbury, 1976) was used initially, but proved too time consuming in practice, required high accelerating voltage and consistently estimated thickness of films at a higher value than the actual measured value. The inaccuracy of the Yakowitz-Newbury method for this application is attributed to the relatively large thickness of the integrated circuit films, e.g., approximately 1 micron for aluminum films and 0.5 to 0.8 microns for silicon dioxide/silicon nitride.

In order to overcome the difficulties with the Yakowitz-Newbury method, an alternative technique was developed in this laboratory that is relatively simple to implement, uses lower accelerating voltages and gives better accuracy in the estimation of the film thickness on the integrated circuits.

Description of Measurement Technique

Basically, this technique uses a variable accelerating voltage to detect the penetration of the electron beam through

the thin film, using EDXA. Once the electrons have sufficient energy to penetrate the film, the transmitted electrons can interact with the substrate material to generate x-rays. By plotting the relative x-ray intensities of the film material to the substrate material, an estimate of the accelerating voltage required for film penetration can be made. This threshold value for the film penetration voltage can be used in conjunction with various range-energy formulas to estimate the electron range and correlate to the film thickness.

Implementation of this method requires localization of the electron beam on the area of interest. This can be done with high accuracy and resolution, in contrast to the optical methods. A minimum of three (preferably more) x-ray intensity readings beyond penetration is required to give an estimate of the penetration voltage.

It is found that reasonable statistics can be obtained for all three readings within ten to fifteen minutes. A simple calculation is then employed to obtain the film thickness.

The implementation of this technique is relatively insensitive to the electron microscope operating conditions and can tolerate variations in the electron beam parameters, provided the accelerating voltage is stable and accurate during measurement and data accumulation.

A comparison matrix showing the relative differences between the various methods is given below in Table 1.

Table 1
Comparison Matrix

	Penetration Voltage Method	Sectioning Method	Yakowitz-Newbury Method
TIME	10-15min	1/2 day	1/2 day
TYPE	non-destructive	destructive	electric damage
ACCURACY (0.5 to 1.5 micron range)	10%-20%	5%-10% (Standards)	50%-100%
ACCELERATING VOLTAGE	4-20 keV	30 keV	30 keV
ANALYSIS METHOD	Spreadsheet	Visual	Quant. EDXA
EQUIPMENT	SEM+EDXA	SEM	SEM+EDXA
COST	100K	70K	110K

This comparison matrix displays, in an abbreviated format, the main areas that contributed to the usefulness or disadvantage of the respective measurement technique.

Theory

To examine the theoretical basis of the penetration voltage method and to determine the appropriate formalism for implementing this method, two empirical/theoretic based formalisms were investigated, namely, Yakowitz-Newbury (Yakowitz-Newbury, 1976) and the Everhart-Hoff (Everhart & Hoff, 1971) formulations.

In the Yakowitz-Newbury formulation, k defines the ratio of the x-ray intensity from the film; If, to the x-ray intensity of the bulk sample of the same material as the film; Ibf:

$$k = \frac{If}{Ibf} . \qquad (1)$$

Further, taking the case of the electron beam penetrating the film, then it follows that If < Ibf. In this case, to a first approximation, the x-ray intensity generated in the substrate material, Isub, can be expressed as:

$$Isub = C \, (Ibf - If) , \qquad (2)$$

since any part of the electron beam not dissipated in the film will be dissipated in the substrate. C is an arbitrary constant to account for other effects, such as those arising at the interface. Dividing by If, the following useful expression is obtained:

$$\frac{Isub}{If} = C \, (Isub/If - 1)$$

$$= C \, (1/k - 1) \qquad (3)$$

This expression (Eq. 3) will allow the determination of the penetration energy by plotting (1/k- 1) versus the electron beam energy. For the calculations, it was assumed that a one micron thick aluminum film was deposited on a silcon substrate. The k factor was calculated for various energies and (1/k - 1) plotted, as shown in Figure 1.

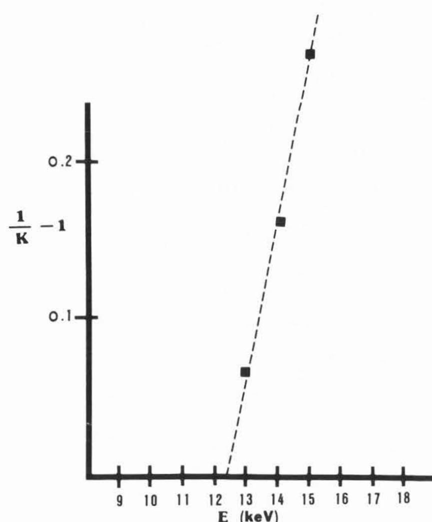

Figure 1. ($^{1}/_{k}$ -1) versus Beam Energy (Yakowitz-Newburg calculations)

The penetration energy obtained at the intersection of the line with the energy axis is approximately 12.3 keV. Using various range-energy formulas, such as depth-dose (Everhart & Hoff, 1971) or Heinrich formula (Yakowitz-Newbury, 1976), the respective thicknesses obtained for the penetration voltage are 1.2 microns and 1.58 microns. Clearly, to obtain the correct value for the film thickness, a correction factor of 0.83 or 0.63 respectively would be required. Further, actual experimental measurements on aluminum films gave similar results, where the Yakowitz-Newbury method estimated the film thickness much higher than the actual value of the film thickness.

To improve on the high estimation bias obtained with the above method, another thickness estimation method was employed, based on the Everhart-Hoff formulation. Using the depth-dose formalism, the beam energy dissipated in the material is given by:

$$P = \int_{0}^{y} Z(y)\, dy \qquad (4)$$

where | represents an integral sign, Z is the normalized energy loss parameter, and y is the normalized depth. Using the simplified assumption that the energy of the electron beam, not dissipated in the film, will be dissipated in the substrate, the following expression is obtained for the x-ray intensity ratio:

$$\frac{Isub}{If} = C\,\frac{1 - Pf}{Pf} \qquad (5)$$

$$\text{where } Pf = \int_{0}^{yf} Z(y)\, dy\,, \qquad (6)$$

and yf is the normalized thickness of the film. Using this expression, (Eq. 5), a plot of Isub/If versus electron beam energy can be obtained. Assuming as before, a one micron thick aluminum film on a silicon substrate, the plot of Isub/If is shown in Figure 2.

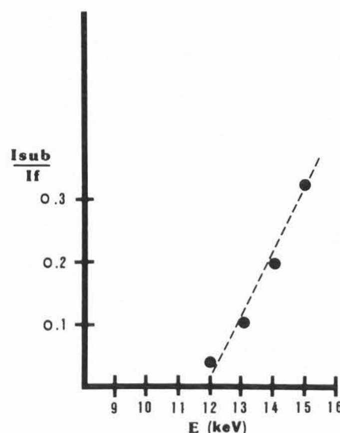

Figure 2. Isub/If versus Beam Energy (Everhart-Hoff calculations)

The penetration energy obtained from this plot is approximately 11.5 keV. By comparison, the Everhart-Hoff method gives a closer estimate for the actual film thickness. Using the range-energy formulas, the respective estimated film thicknesses are 1.05 microns or 1.45 microns. The respective correction factor for each estimate are 0.912 or 0.69. Obviously, the Everhart-Hoff formalism, in combination with the depth-dose range-energy relation, gives the best results for estimating the thickness of aluminum films on integrated circuits.

In conclusion, both formalisms confirm the correctness of the experimental method as an estimation technique for the penetration voltage. Based on the calculations, however, the Everhart-Hoff formalism is the most accurate for the range of thickness investigated in this paper. Further, the depth-dose range-energy formula gave results, based on these calculations, that was closest to the assumed film thickness values.

In the subsequent work, the depth-dose relation will be used to estimate the film thickness from the penetration voltage. This concludes the section on the theoretical discussion of the basis for the

voltage penetration method and, in turn, establishes the basis for the approach in the following section on experimental procedures.

Experimental Procedure

To calibrate this method with respect to a secondary length standard, an aluminum film of approximately 1 micron, was deposited on two silicon wafers. One wafer was coated with a thin film of gold and the other was left uncoated. These wafers were fractured and the thickness of the aluminum film was measured visually in the SEM. The calibrated micron marker of SEM was compared to the secondary length standard (diffraction grating). This secondary standard was, in turn, calibrated with respect to the NBS SRM 484. Based on this procedure, the SEM measurement of the film thickness is the most accurate of all the methods described in this paper, since it can be traced to a "primary" length standard from the NBS.

An optical interferometer was also employed on the aluminum film, using a standard optical microscope with a Nomarski interferometer attached to it. The results of this method are less accurate than the SEM analysis, since this technique essentially averages over a fairly large surface area compared to SEM analysis of the film. Thus, the optical technique is in contrast to using the SEM, which can localize the thickness of the film to a specific location. Due to the nature of samples and available equipment, the interferometer was only used to measure the thickness of the film on the aluminized wafers.

Finally, the EDX analysis was used to measure the film thickness in the vicinity of the optical and SEM thickness measurements. As described previously, the accelerating voltage of the SEM was varied until the e-beam penetrated the aluminum film and a weak silicon peak was obtained. At this point, the voltage was increased in steps of 1 keV and the x-ray spectra was taken at each step. The relative ratio of silicon to aluminum Isi/Ial was then plotted versus accelerating voltage. Enough data was taken to extrapolate back to the penetration voltage. Usually, this required a minimum of three to four data points beyond the initial detection of substrate material. Further, enough counts were accumulated at each point to give a peak count for the film above 30k. Typically, the plot of the data is as shown in Figure 3.

A least squares fit of the data points, above the minimum point, to a straight line, will allow an estimation of the penetration voltage at the intersection with the energy axis.

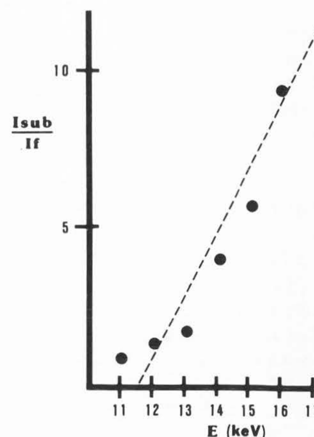

Figure 3. Experimental Data: Isub/If versus Beam Energy

Table 2

Estimated Penetration Voltage (keV)

	Al (unc)	Al (c)	IC#1 (unc)	IC#2 (unc)
Peak Ratio (Isi/Ial)	10.21	11.44	12.89	13.16
K Ratio (Ksi/Kal)	10.52	11.25	-----	-----

(Uncertainty of +/- 0.2 keV)
(unc-uncoated, c-coated with Au film)

The results of this procedure are given in Table 2, for several different samples. The K ratio was obtained for values produced after performing a semiquantitative analysis on each spectra. The quickest way for implementing above procedure was by using peak ratio (Isi/Ial), since it didn't require further analysis or comparison to standards. Also, the peak ratio was relatively insensitive to SEM operating parameters as compared to other ratios.

When estimated penetration voltage of the film is obtained, an estimation of its thickness can be obtained using one of the available range-energy formulas described above. The depth-dose approximation formula (Everhart & Hoff, 1971) was used here :

$$T = 40 \times \frac{E^{1.75}}{p} \qquad (7)$$

where T = thickness of film (micrometers), E = electron-beam accelerating voltage (keV) and p = density (mg/cm3). Using this equation to estimate film thickness, results in Table 3 which compares the SEM and interferometer measurements.

Table 3
Film Thickness Measurement (Micron)

	Al Film (uncoated)	Al Film (coated)	IC#1 (uncoated)	IC#2 (uncoated)
Peak Ratio	0.863+/-.04	1.053+/-.04	1.298+/-.05	1.346+/-.05
K Ratio	0.909+/-.04	1.023+/-.04	---------	---------
SEM (visual)	1.00+/-.034	1.170+/-.04	1.550+/-.13	1.497+/-.12
Interferometer	0.79+/-.12	0.950+/-.09	---------	---------

These results using the penetration voltage method are reasonable and give a credible estimate for the thickness of aluminum films. In contrast, use of the Yakowitz-Newbury technique consistently gave film thickness estimates that were larger in value than the actual measured values, for the range of thicknesses and SEM operating parameters used for this experiment. Further, the Yakowitz-Newbury method requires much higher accelerating voltages to obtain thickness estimates than the penetration voltage method. The higher accelerating voltages required by Yakowitz-Newbury can damage the integrated circuit, causing the device to malfunction electrically. With the penetration voltage method, there is less likelihood of damage and, at worst, may only lead to degradation of parameters rather than complete destruction or malfunction of the integrated circuit. Also, damage or degradation can be further limited for sensitive circuits by restricting the probing and measurements to the peripheral area of chip where none of the critically active circuit elements are located. Based on the results obtained in this laboratory, the penetration voltage method gives reasonable and fairly accurate measurements of the film thickness on an integrated circuit with a minimum of damage to the electrical functionality of the device.

To obtain more precise estimates of the film thickness, a correction factor was applied to give a one micron thickness for the uncoated, peak ratio reading. This would correspond with the SEM visual analysis of that film. Applying the same correction to all the other samples, the results in Table 4 are obtained.

Table 4
Corrected
Thickness Estimates (Microns)

	Al (unc)	Al (c)	IC#1 (unc)	IC#2 (unc)
Peak Ratio	1.00	1.22	1.50	1.56
K Ratio	1.05	1.18	------	------
SEM (visual)	1.0	1.17	1.55	1.497

(unc=uncoated, c=coated)

These results correspond very closely with the SEM analysis values, which can be traced to secondary and NBS length standards. Based on the close correlation of the corrected penetration voltage thickness estimates with the SEM visual, the correction factor is valid for all four samples, taken from different sources. Based on these results, the correction factor can be utilized for other aluminum film samples to estimate the film thickness.

Similar results were obtained for silicon dioxide/silicon nitride films, such as, passivation on integrated circuits. Typical results for a hybrid integrated circuit chip are given in Table 5.

Table 5

Silicon Dioxide/Nitride
Film Thickness

	Penetration Voltage (keV)	Thickness (MICRON)
Peak Ratio	4.41	0.2429+/-.03
K Ratio	4.64	0.2655+/-.03
SEM (visual)	------	0.1980+/-.03

The estimated results were obtained assuming that the film was silicon dioxide. If silicon nitride was assumed, the penetration voltage thickness results would have been closer to the SEM visual results and within the error margin. Since results were reasonable and demonstrate the utility of the penetration voltage method, no further analysis was performed to determine the nature of the silicon dioxide film. It was initially planned to fabricate silicon dioxide films on an aluminum substrate, but deposition equipment was not available for that purpose at the time of this experiment. It is hoped that future experiments will determine whether any correction factors are needed for the silicon dioxide/silicon nitride films used on integrated circuit chips.

Experimental Error

Analysis of the sources of error in the employment of the penetration voltage method indicated that error propagation could arise from several areas: (1) surface contour irregularity; (2) e-beam energy variation; (3) minimum detection level of EDXA equipment; (4) estimation of the penetration voltage; and (5) estimation of film thickness.

Surface irregularities were particularly evident in the SEM analysis and the optical measurements. As indicated in Table 2, the thickness variation detected varied from +/-0.04 to +/-0.13 microns, depending on the sample. Due to the large variation in some samples, it is anticipated that surface irregularities will be the dominant source of error for the thickness measurement. This will be particularly true for integrated circuits, since these devices are subject to numerous processing steps during manufacturing. These steps include photoresist patterning, etching, heat treatment, high temperature oxidation, passivation and reflow processing. All these processes will put stress on film and its surface, contributing to the surface irregularity. Further, calibration with respect to the secondary and NBS length standard introduces an error, typically, +/-0.07 to +/-0.13 microns, depending on the magnification used. The surface irregularity is a precision error, while the calibration error is an accuracy error. However, both will impact the readings. The elimination of the calibration error would require the use/availability of thin film standards. In the absence of such standards, a correction factor was used. The surface irregularity error can't be eliminated and must be considered the primary source of error in the SEM visual analysis.

Variation in the electron beam energy was measured and proved to be less than +/-0.2 keV. This variation in energy will propagate into an error of +/-0.05 microns in the thickness estimate, using the penetration voltage method. This error is below the range of the surface irregularities detected with the SEM visual analysis.

Errors due to the minimum detection capability of the EDXA equipment and the least square estimation of the penetration voltage are minimized by taking a number of data points above the initial penetration of the film. A minimum of three data points was adequate, but more could be taken and may be necessary for thicker films. Goodness of fit criteria, such as the correlation coefficient, can be used to minimize the errors arising during curve fitting. Typically, the correlation coefficient obtained for the least square fit to the data used in this report was 0.9 or better. Further, the uncertainty of the actual electron range at penetration is overcome by taking a large number of x-ray counts at each data point. Typically, over 30K counts at the film peak were taken to ensure good statistics. More counts at each data point could be taken to give better statistics and minimize the error. Actual estimation of the error due to this procedure was not undertaken, due to the uncertainty in the electron range at the interface and the minimum detection capability of EDXA equipment. However, the experimental procedure minimizes the error by averaging out the various sources of error and uncertainties.

The error generated in the actual estimation of the film thickness will depend on the range-energy equation used for the thickness estimation. Of the several range-energy equations available, the depth-dose equation gave the best fit to experimental data obtained with the SEM visual examination. No estimate of error was made for comparison, since thick film standards were not available.

In conclusion, the results obtained with the penetration voltage method were reasonable, when compared to SEM visual measurement of the film thickness. The primary source of error is due to the surface irregularities of the samples used, particularly, for the integrated circuits.

Conclusions

The results obtained to date on a variety of different samples, both those specifically fabricated for this experiment and available integrated circuits in this laboratory, demonstrate the utility and accuracy of the penetration voltage method as a means of obtaining the thickness of aluminum and silicon dioxide/nitride films on integrated circuits, in the 0.5 to 1.5 micron range.

Further, this method allows the measurement of the film thickness with relatively low accelerating voltages, so that the possibility of damage to the electrically functional integrated circuit is minimized. This is very helpful during the failure analysis of integrated circuits, since many failures are detected only by means of the circuit electrical functionality. Also, the low accelerating voltage is required during inspection for process and quality control, so that the circuits are not damaged or degraded.

It is anticipated that this method will be a useful technique to be used with MIL-STD 883, Method 2018.2, where the integrated circuits are inspected with SEM for quality defects, such as metallization thickness and passivation coverage. The implementation of this method for thickness measurements would require only 10 to 15 minutes to acquire several spectra and the input of data into a program of 10 to 20 code lines to obtain an estimate of the film thickness to a reasonable accuracy.

Acknowledgements

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References

Everhart, T. Hoff, P.H., (1971), Determination of keV Electron Energy Dissipation versus Penetration Distance in Solid Materials, J. Appld. Phys. 42, 5837-5846.

MIL-STD-883C, (25 Aug 83), DOD, Test Methods and Procedures for Microelectronics, Method 2018.2, Scanning Electron Microscope (SEM) Inspection of Metallization (4 Nov 1980) (available from: Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, PA 19120).

Yakowitz, H. Newbury, D.E., (1976), A Simple Analytical Method for Thin Film Analysis with Massive Pure Element Standards, Scanning Electron Microsc. 1976; I:151-162.

Discussion with Reviewers

M. G. Rosenfield: Would this technique work if the film were of higher atomic number than the substrate (W or Au, for example)?
Author: To date, experimental measurements haven't been performed on metal of higher atomic weight than aluminum. To penetrate a micron gold film would require a prohibitively high accelerating voltage, which would damage the integrated circuit. Experiments on thin gold film (less than 1000 angstroms) using this technique were performed, but estimates were not verified by SEM visual analysis.

M. G. Rosenfield: The experimental points in Figure 3 do not fit a straight line. Why is this and would it be better to use a more accurate curve fit?
Author: The curvature of graph as the voltage approaches the penetration voltage of the film is probably due to straggling effects at the interface as seen in H. Kanter and E. J. Sternglass, Interpretation of Range Measurement for Kilovolt Electrons in Solids, Phys. Rev. 1962, Vol. 126, No. 2, p 620-626. With respect to curve fitting, other methods have been used, but were not as easy to implement as the straight line fit. Based on the Everhart-Hoff formalism, the straight line fit introduces approximately a 5% error in the estimation of the penetration voltage. Fit to a higher polynomial has problems due to the presence of background count. Elimination of the background count would require more measurements and should be considered for an automated system.

Reviewer III: Could you list a comparison of Yakowitz-Newbury versus Everhart-Hoff thickness for aluminum in $5 < E_o < 10$ keV range?
Author: Based on formalisms mentioned in the paper, calculations showed that Yakowitz-Newbury and Everhart-Hoff corresponded closely at film thickness below 2000-3000 angstroms, but started to diverge noticeably above that level, to give a significant error for aluminum film thickness in the 1 micron region and above. This was confirmed experimentally for aluminum films at 1 micron and up and for gold films in the 300 to 500 angstrom range.

J. W. Newkirk: Did author use Yakowitz-Newbury technique to perform measurements on film and what were the results?
Author: Yes. Typical results for the 1 micron aluminum film was 1.25-1.50 microns. Results for the 1.5 micron aluminum film gave even larger errors, 2.0 to 2.25 microns. However, the Yakowitz-Newbury technique did give results that corresponded with the penetration voltage method for film thickness of several hundred angstroms. These measurements were time consuming due to the instability of beam current. Long acquisition times were used to average out these fluctuations. However, relatively significant variations in individual readings were detected and sufficient quantity of readings were required to obtain consistent results with the Yakowitz-Newbury technique by averaging results.

J. W. Newkirk: Since the Yakowitz-Newbury technique is important to this paper, could you give a brief description of this technique?
Author: The Yakowitz-Newbury technique uses a combination of experimental measurements of the x-ray intensities of thin film and bulk samples of the same material as film, compared to calculated x-ray intensities, in order to determine the film thickness. Specifically, x-ray spectra must be obtained from 1) thin film on substrate, If, at a sufficient beam energy to give an electron range that is typically greater than 1.5 times the film thickness, and 2) bulk standard of same material as film, Ibf, at the same beam condition as used for thin film sample. The measured values are used to determine $k = If/Ibf$. To obtain the film thickness, calculations are performed using the Yakowitz-Newbury formalism to match the measured k, where the film thickness is the variable.

Reviewer III: With the samples having large surface topographical errors, were the measurements made with a point probe? Could averaging out be accomplished using a small screen raster?

Author: Some measurements were done using the spot mode and some using partial field (small raster). Both measurements were compatible. Small screen raster would certainly average out x-ray intensity over region scanned and provide an average thickness value on that basis. In the spot mode, care must be taken to position beam at same location for each successive measurement.

J. W. Newkirk: Would you comment on the error introduced by reducing the raster field and what resolution can be achieved at the count rates you are using?
Author: All measurements were performed in partial field mode (reduced raster) or spot mode. No significant difference was seen on smooth aluminized wafer, but considerable variation was noted on the integrated circuit samples with the rough surfaces. For the integrated circuits, the measurements must be made with small partial field or spot mode and care must be taken to position beam at same location for all successive readings.

SEM was operated at low count rate, with dead time between 20-30%. This condition gave highest detector peak resolution of 150-155 eV FWHM (full width half max), for the best resolution of substrate peak near the penetration voltage.

Reviewer III: The correction factor for aluminum film is different from silicon dioxide film. Is this due to density differences? Have you looked at differences in correction factors between SiO_2, phosphorsilicate glass (PSG) or SiOxNy passivation?
Author: To explain the difference in correction factors of approximately 1.1 for aluminum films and 0.9 for silicon dioxide film, some of the discrepancy must be attributed to the relative thickness of the films. The thinner film has a much faster rising curve (k versus energy), which will tend to give a higher penetration voltage estimate, using the procedure described in this paper. Fluorescent and absorption effects on the intensity of x-ray peaks were also calculated, but were found to require correction in the opposite direction to that required for this experiment.

With respect to the passivation materials (SiO_2, SiOxNy and PSG), density differences might be a factor. However, well defined films will be needed to determine if the density differences can be detected by having a significant effect on penetration voltage.

Reviewer III: Do you have any data on minimum thickness measurements with variation in Eo?
Author: Presently, experimental factors limit measurement to electron beam energies around 5 keV due to the low x-ray intensities attainable with tungsten source in the present configuration. Further, the depth-dose formula used for thickness estimation is only validated to 5 keV. An example of the thinnest film measured was the silicon dioxide film measured in Table 5 and the gold film on aluminized wafer (approximately 200 angstroms), which was not verified by a SEM visual analysis.

Reviewer III: The Yakowitz-Newbury formalism uses a function [f(x)] for calculation of k, which takes into account mass absorption, take-off angle and beam versus emitted x-ray line energies. Is this incorporated in your C constant?
Reviewer I: Your C constant is not arbitrary. Shouldn't it include factors accounting for different x-ray generation factors for substrate, film and bulk film materials?
Author: The k values were calculated directly from the Yakowitz-Newbury formalism, which include consideration of the factors mentioned in the questions. The function (1/k - 1) was then plotted versus electron beam energy and shown to approximate a straight line (Fig. 1). The C constant represents the proportionality factor in the straight line fit to the calculated data. The same is true for the Everhart-Hoff formalism, which is based on electron dissipation and fitted to empirical data. As discussed in the text, a straight line fit in conjunction with depth-dose range-energy formula gave approximately a 5% error for calculated data.

M. G. Rosenfield: Backscattered electrons, which can be significant for high Z materials, have been neglected in your assumptions concerning dissipation of electron energy in substrate versus the film. Will this affect any of your calculations?
Author: Both the Yakowitz-Newbury and Everhart-Hoff formalisms take into account backscattering in their calculations. This consideration applies to the film material. For the case of film on a high Z substrate, the calculations will probably have to be modified to correct the backscattering effect. This case has not been considered for this experiment.

Reviewer III: Would you expect peak intensity ratio to be different for LaB_6 electron beam source versus a tungsten hairpin?
Author: I don't anticipate any problem, provided the count rate is maintained at a level to obtain maximum resolution of substrate peak from the background level. It is anticipated that a LaB_6 source will provide better beam stability and will extend measurements to lower beam energies, where the x-ray count rate is insufficient using the tungsten source.

Reviewer III: Have you tried the peak ratio method on multi-element layers?
Author: No. The technique has not been applied to multi-element layers at the present time, other than SiO_2 film. An example of a multi-element material where this technique wouldn't be practical is tungsten silicide on silicon substrate, due to the overlap of the tungsten and silicon peaks.

Reviewer I: What about the difference in results for coated and uncoated wafers? Could you discuss this?
Author: Due to charging effects, some samples have to be coated with a conductive layer to obtain a high resolution scanning electron microscope micrograph. The purpose of the coated sample was to determine if this coating would distort the measurement of the film thickness. This was found to be the case, presenting problems both experimentally and in the estimation of the film thickness. As a result, subsequent measurements were only performed on the uncoated samples.

Reviewer I: With respect to experimental errors, only the error for accelerating voltage measurement was quantified, and probably the least important. Wouldn't other errors be more important?
Author: Experimental factors precluded a direct measurement of these errors, other than the correlation to the NBS standard and the surface roughness. Both the correlation to NBS standard and the surface roughness were measured and quantified to approximately +/-0.1 microns. With respect to curve fitting and minimum detection limits, calculations indicate that straight line curve fit procedure introduces a 5% error approximately, and a 1% minimum detectability level introduces a 4% error. Since these errors were not determined experimentally, the procedure followed was to minimize and stabilize the errors due to these sources by using low count rates to increase resolution and a minimum of data points above the initial detection of substrate peak. This procedure gave consistent results.