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By

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Secondary Electron Yields of Carbon-Coated and
Polished Stainless Steel*

PPPL--1674

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ABSTRACT

To increase the power throughput to a plasma of an existing lower hybrid waveguide, secondary electron production on the walls and subsequent electron multiplication must be reduced. Since carbon has a low secondary electron coefficient (δ), measurements were performed for several UHV compatible carbon coatings (Aquadag[®], vacuum pyrolyzed Glyptal[®], and lamp black deposited by electrophoresis) as a function of primary beam voltage (35 eV to 10 keV), surface roughness (60 through 600 grit mechanical polishing and electropolishing), coating thickness, and angle of incidence (θ). Also measured were uncoated stainless steel, Mo, Cu, Ti, TiC, C as soot and ATJ graphite. The yields were obtained by varying the sample bias and measuring the collected current while the samples were in the electron beam of a scanning Auger microprobe. This technique allows δ measurements of Auger characterized surfaces with ≤ 3 mm spatial resolution. Results show δ to have a typical energy dependence, with a peak occurring at 200 to 300 eV for normal incidence, and at higher energy for larger θ . In general, δ increases with θ more for smooth surfaces than for rough ones. 90% of the secondary electrons have energies less than 25 eV. Some carbonized coating and surface treatment combinations give $\delta_{\max} = 0.88 \pm 0.01$ for normal electron beam incidence—a reduction of almost 40% compared to untreated stainless steel.

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I. Introduction

The use of waveguides in plasma heating experiments has recently received much attention.^{1,2} The waveguide power throughput may be limited by effects which cause an electron density $\geq 10^9 \text{ cm}^{-3}$ within the waveguide. This could occur if the peak RF electric field, which reaches 3 kV/cm in the PLT lower hybrid waveguides, causes sharp points or surface contaminants to field emit. Higher electron densities would develop as gasses desorbed from the surfaces were ionized. These problems are exacerbated by secondary electron production on the walls. Most free electrons in the guide will strike the walls at normal incidence ($\theta=0^\circ$). To avoid a multipactoring electron cascade³ a secondary electron coefficient (δ) less than unity at $\theta=0^\circ$ is necessary and a $\delta < 1$ at other θ is desirable.

The existing waveguide arrays for the PLT tokamak lower hybrid heating experiments are 304 stainless steel, for which, as shown by the authors, $\delta > 1$ at many energies (see figure 1 and 2). Surface treatments that can be readily implemented in the existing arrays are preferred. Sanding, electropolishing and cleaning the surface can be done to reduce arcing and gas desorption. As noted by A. A. Dorofeyuk *et al.*,⁴ carbon coating will reduce δ .

The purpose of this experiment was to test which combinations of sanding grit size, sanding direction, electropolishing and carbon coating type would produce the lowest δ . An application of a scanning Auger microprobe to measure δ 's over a large range of primary electron energies (E_p) similar to M. Padamsee and A. Joshi's⁵ is also reported.

II. Apparatus and Procedure

The measurements were performed with a Physical Electronics Model 590 Scanning Auger Microprobe (see figure 3). The electron emitter of this device is a LaB_6 crystal. It is at a high potential with respect to a nearby extractor grid. The grid is attached to the

vacuum vessel which is not externally grounded. The accelerated electrons are then focussed onto a specimen stage. Measurements of the beam size with a Faraday cup show that the beam diameter is less than 0.3 mm at a $E_p = 50$ eV and decreases to about 10^{-4} mm at 9.8 keV. Deflection plates can raster the beam across the sample. The base pressure in the system was $\approx 2 \times 10^{-9}$ torr.

Three devices are also aimed at the bombardment area: an ion sputter gun; an Auger spectrometer; and an electron multiplier for secondary electron microscopy (SEM). SEM can also be performed by monitoring the current absorbed by the sample.

To measure δ a variable voltage power supply (0 to ± 90 V D.C.) and an ammeter were connected in series between the specimen stage and the vacuum vessel. This allows the stage to be biased with respect to the surrounding areas thereby attracting or repelling secondaries. A voltmeter was connected between the LaB_6 crystal and the specimen stage to measure E_p .

The typical procedure to obtain δ is as follows: A sample is selected and the electron optics focussed to yield maximum total current to the positively biased sample. A SEM image of the irradiated portion of the sample is formed to assure the quality of focussing. This is a particularly important parameter in the low voltage range for the electron gun. An Auger spectrum is obtained. The electron gun is then returned to the desired voltage. With the stage biased to ≈ -2 V with respect to the vacuum vessel all the secondary electrons are repelled and the current (I_-) of the electron beam minus the secondary current is measured by the ammeter to an accuracy of better than 2%. With a positive bias, secondaries are attracted back to the sample and the total current (I_+) is measured.

Then $\delta(\bar{E}_p)$ is found from:

$$\delta = \frac{I_+ - I_-}{I_+},$$

where $\bar{E}_p = \frac{1}{2} [E_p \text{ (positive bias)} + E_p \text{ (negative bias)}]$. This device is capable of measuring $\delta(\bar{E}_p)$ at $\bar{E}_p = 35 \text{ eV to } 9.8 \text{ keV}$. Use of a positive bias voltage also allows measurement of the secondary electron energy distribution. A typical current vs bias voltage curve is shown in figure 4. For carbon, about 90% of the secondary electrons return to the sample when the stage is biased by +25 V. The energy distribution obtained was similar to F. Pellerin and C. LeGressus's secondary electron spectral data.⁶ For stainless steel the authors determined a bias voltage of +35 V was necessary to attract 90% of the secondary electrons. The error in determining δ due to collecting only $(1-\epsilon)$ of the emitted secondary electrons is $(\delta-1)\epsilon$. From collecting only 90% of the secondaries the error in δ is 1% for $.9 < \delta < 1.1$.

The data and sample reproducibility vary less than 2% from day to day even using different primary electron beam currents and other operating conditions.

To rid the surface of contaminants, each sample was sputter cleaned with a 4 keV Ar^+ beam immediately before analysis. About 40 Å of the carbon surfaces were removed. To some extent this mimics the conditioning procedure generally used in tokamaks to eliminate adsorbed gasses. Since the RF conditioning and tokamak environment could not be duplicated in the scanning Auger microprobe system, the samples were briefly sputter cleaned between each measurement. Each of these cleanings removed about 10 Å from the surface. In this way effects such as electron-bombardment-induced rapid oxidation⁷ and

electron induced "conditioning" (adsorption and polymerization of hydrocarbons)^{8,9} are further reduced. The original reduction being accomplished by rapid data acquisition (<30 sec per point), low base pressure (1 to 3×10^{-9} torr), low primary electron current density J_p (<20 $\mu\text{A}/\text{cm}^2$), and by observing no change in δ as a function of J_p . Further secondary electron coefficient experiments which mimic the tokamak environment more completely, such as Deuterium loading, e^- bombardment, and Hydrogen glow discharge cleaning, should be pursued.

As a control on the experimental method the δ of Mo at $\theta = 0^\circ$ (normal incidence), Cu at $\theta = 0^\circ$ and Ti at $\theta = 0^\circ, 30^\circ, 45^\circ$ and 60° were measured. These results are shown in figure 5 and agree with published results^{10,11} with 1%.

III. Surface Treatments

The δ measurements were performed on approximately 1 cm x 1 cm pieces of 2.5 mm thick 304 stainless steel. These were cut from larger pieces that underwent some or all of the following treatments:

Sanding: The first sanding was always unidirectional with either a grit size of 60, 80, 120, 240, 400 or 600. The samples were ultrasonically cleaned in a hot Alconox[®] solution to remove leftover grit, rinsed in deionized water, and oven dried in air at 100°C.

Resanding: The second sanding was also unidirectional but perpendicular to the original direction. This sanding was continued only until a visually uniform "grid" pattern emerged. Again the samples were cleaned.

Electropolishing: The samples served as the anode in 40 C Summa Processing[®] solution using a current density of $0.1\text{A}/(\text{cm})^2$ for six minutes. The samples were rinsed and cleaned.

Coating: Several methods were tried. Samples were dipped into a 50-50 solution of Glyptal[®], a commercial varnish, and xylene, and then air dried. The resultant film was $\approx 1 \mu$ thick. Other samples were dipped into Aquadag[®]. A third coating method¹⁰ was electrophoretic deposition (ED) of lamp black suspended in the 50-50 solution (1/2 gm carbon/(100 ml Glyptal[®] + xylene)). A voltage of 2 kV was applied across the solution yielding a current density of $0.075 \times 10^{-6} \text{ A}/(\text{cm})^2$. After 20 minutes the samples were removed and dried.

Vacuum Pyrolysis: In all cases the coatings were baked at 400°C at $\approx 1 \times 10^{-5}$ torr for one hour to pyrolyze the hydrocarbon compounds. The temperature of 400°C was chosen to avoid phase changes in the steel, carbon diffusion and braze failure. After baking, the thickness of the plain Glyptal[®] coatings was determined to be $\approx 300 \text{ \AA}$ by sputtering (see figure 6). The thickness of the ED lamp black coating was $\approx 1400 \text{ \AA}$ (see figure 7).

IV. Results

As seen by others¹³ on a variety of materials, higher incident angles produce higher δ 's, and the maximum δ (δ_{max}) occurs at larger values of E_p . The lowest δ_{max} for a Glyptal[®] coating (0.88 ± 0.01) at $\theta = 0^\circ$ was obtained with the surface treatment consisting of 400 grit size sanding, resanding and electropolishing. These results for all angles of incidence measured are shown in figure 8. Auger spectroscopy showed that after sputter cleaning the surface region of pyrolyzed Glyptal[®] consisted of 96% carbon, 2% oxygen and 2% others. Representative Auger spectra before and after sputter cleaning are shown in figure 9. A two hour residual gas contamination at

$p = 1.5 \times 10^{-9}$ torr typically raised δ by 3% and increased the oxygen concentration to 4%. After sitting in atmosphere for two months the remeasured surface had a δ_{\max} 5% higher than the cleaned sample and a 5% Oxygen surface contamination.

Surface roughness noticeably affected δ_{\max} values for Glyptal[®] coated surfaces. Smoother surfaces produced larger δ_{\max} values at large θ than rough surfaces did. At normal incidence δ_{\max} was nearly independent of grit size (see figure 10). If only one unidirectional sanding was performed, δ_{\max} measurements varied as much as 12% for large θ depending on the direction of the electron beam with respect to the sanding direction. Perpendicular incidence gave the lowest values.

Pretreating the Glyptal[®] coated samples by electropolishing decreased δ only slightly. The reduction in δ was 3% at $\theta = 0^\circ$ and 12% at $\theta = 60^\circ$. Redipping the Glyptal[®] samples after the first pyrolysis and then rebaking did not change the shape or magnitude of the secondary electron curve.

The results for other coatings are shown in figure 11. These surfaces had the same pretreatment as described for the results shown in figure 8. Aquadag[®] has a δ_{\max} of 0.91 ± 0.01 but was hard to apply uniformly and did not adhere well. The ED lamp black samples had much lower δ for higher θ . Their δ_{\max} at normal incidence was the same (0.88 ± 0.01) as the Glyptal[®] sample. By not electropolishing the ED lamp black samples a thicker carbon coat can be deposited and the values of δ are not affected. However, electropolishing may reduce field emission and gas desorption. In any case, sanding and cleaning are necessary since the δ values of unpretreated ED lamp black samples rose quickly between sputter cleanings (see

figure 12). The δ 's for all of the carbon coatings decreased slowly above an \bar{E}_p of 1.5 keV. The entire range of δ values for Glyptal are shown in figure 13.

Data at $\theta = 0^\circ$ and 60° for TiC are also shown in figure 11. The TiC was deposited by chemical vapor deposition¹⁴ onto blocks of graphite. The thickness of the TiC coating was between 15 μ and 20 μ . The δ_{\max} at $\theta = 0^\circ$ was 0.87 ± 0.01 , and the rise of the δ curve with θ was moderate.

A sample of ATJ graphite was analyzed and found to have a δ_{\max} at $\theta = 0^\circ$ of 0.89 ± 0.01 at 270 ± 12 eV. The shape was intermediate between the $\theta = 0^\circ$ Glyptal sample (figure 8) and the $\theta = 0^\circ$ Aquadag sample (figure 11). The published¹⁵ value for graphite (type not specified) is $\delta_{\max} = 1.0$ at $E_p = 300$ eV.

Soot, produced from an oxygen poor methane flame, was deposited on stainless steel and found to have a δ_{\max} of 0.38 ± 0.02 at $\theta = 0^\circ$ (see figure 14).

V. Summary

By modifying a scanning Auger microprobe, secondary electron coefficients (δ) were determined to an accuracy of $\pm 2\%$. The technique was verified by using standards. Surface treatment, consisting of sanding, resanding, electropolishing, carbon coating and vacuum pyrolyzing reduced the δ of stainless steel below unity at all primary beam energies. In particular ED lamp black reduced δ_{\max} from 1.23 to 0.88 ± 0.01 at $\theta = 0^\circ$, and to below 1.00 at all θ .

Recent lower hybrid heating results on PLT show at least a factor of 3 increase in power throughput to the plasma using the coated waveguides.¹⁶ Other modifications were also implemented in the waveguide

at the same time, but it is believed that the coating process increased power throughput significantly.

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Note: A condensed version of this report can be found in JVST
(April 1982).

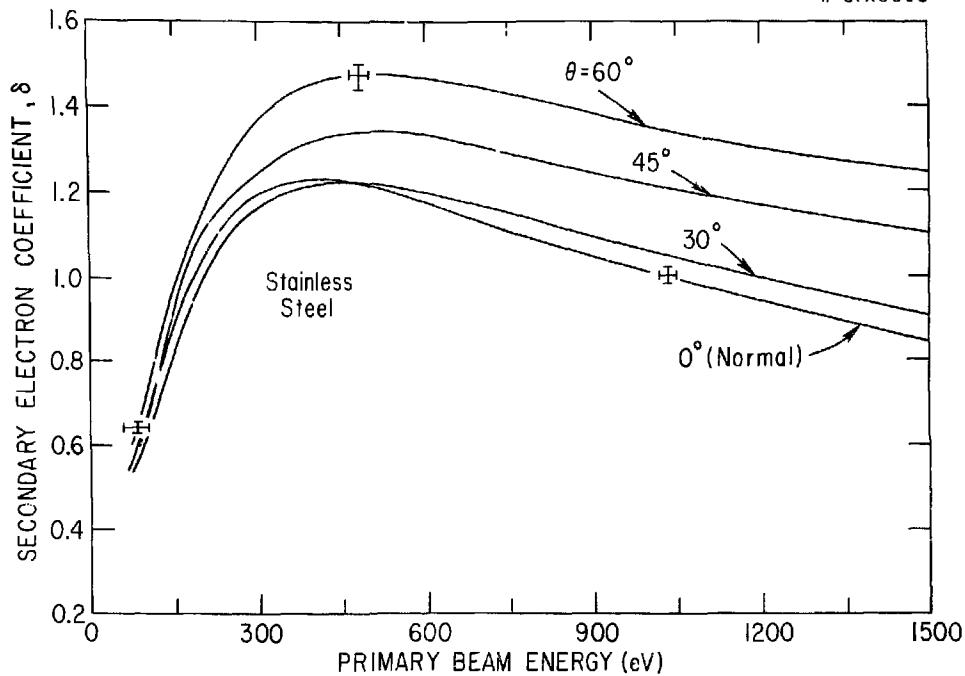


Fig. 1. $\delta(\bar{E}_p)$ for 304 stainless steel at four angles of incidence. The surface was sanded with a 400 grit in a grid pattern and electropolished. Representative error bars shown.

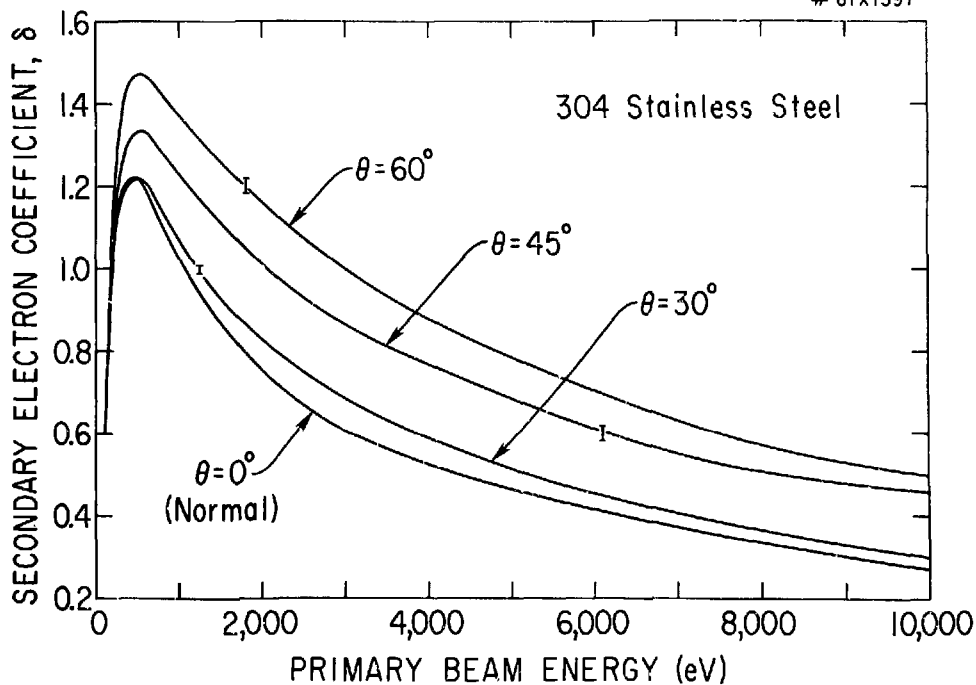


Fig. 2. $\delta(\bar{E}_p)$ for 304 stainless steel at four angles of incidence. The surface was sanded with a 400 grit in a grid pattern and electropolished. Representative error bars shown.

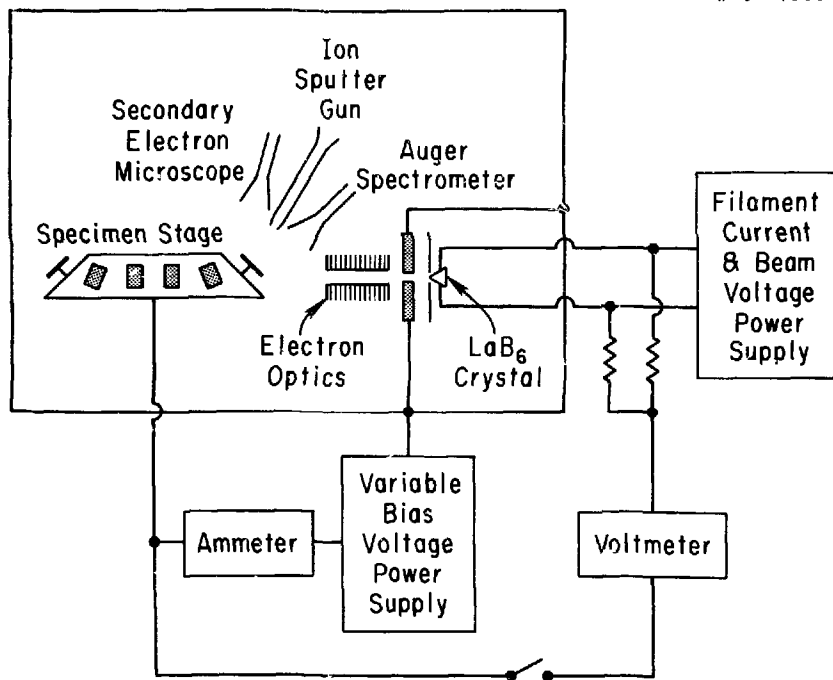


Fig. 3. A Physical Electronics model 590 scanning Auger microprobe was slightly modified by the addition of a variable voltage supply, an ammeter and a voltmeter.

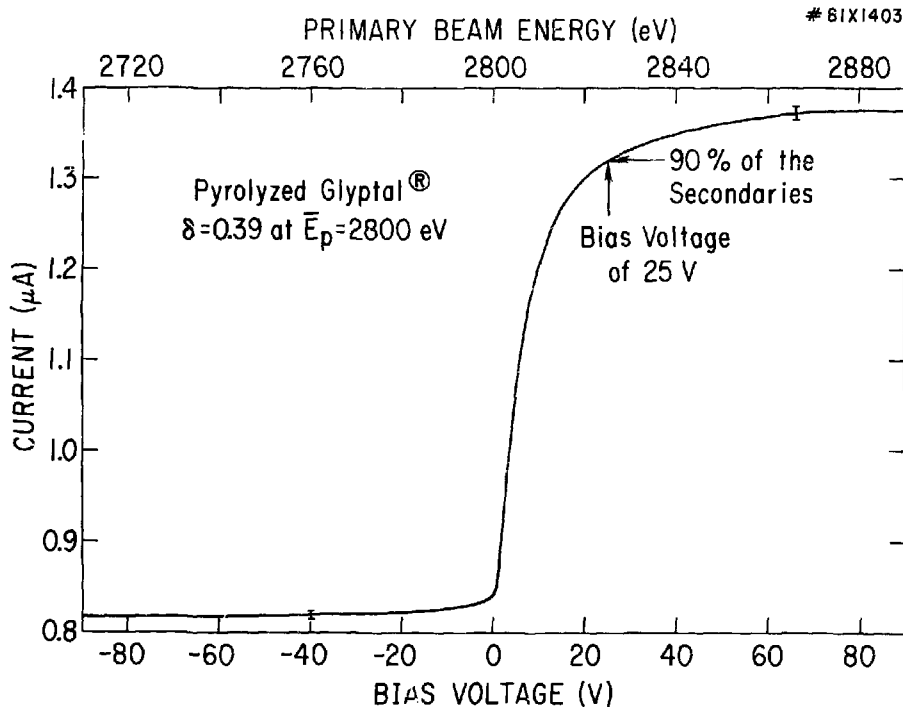


Fig. 4. Current to the sample stage vs bias voltage. Ninety percent of the secondaries are collected with a bias voltage of +25 V for carbon coatings. In stainless steel, +35 V are necessary to collect 90% of the secondaries.

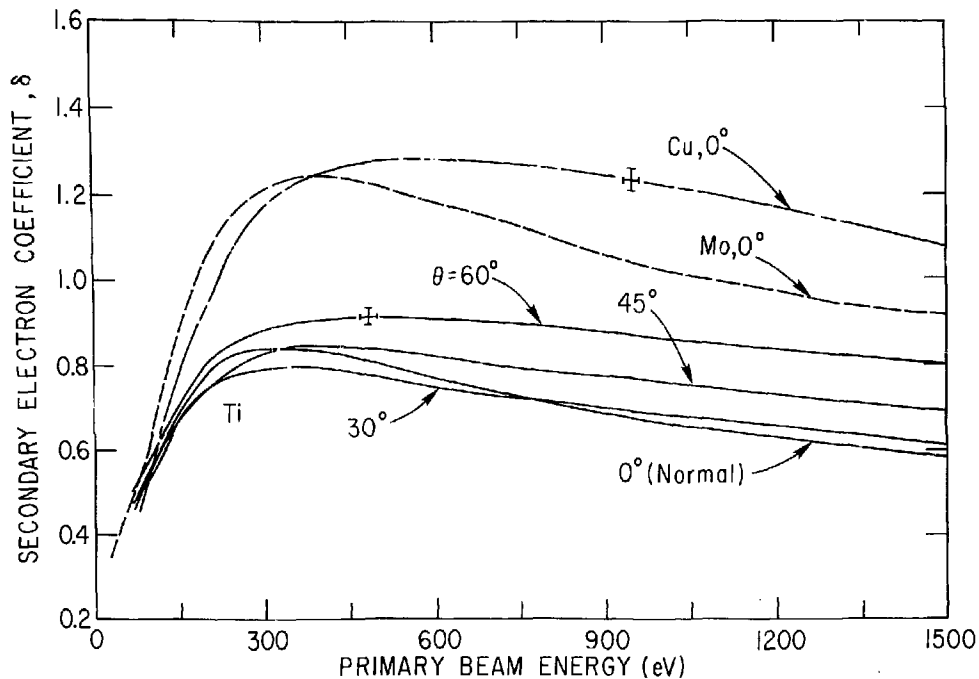


Fig. 5. $\delta(\bar{E}_p)$ for titanium at 4 angles of incidence and copper and molybdenum at normal incidence. Within the error bars these curves agree with published data.

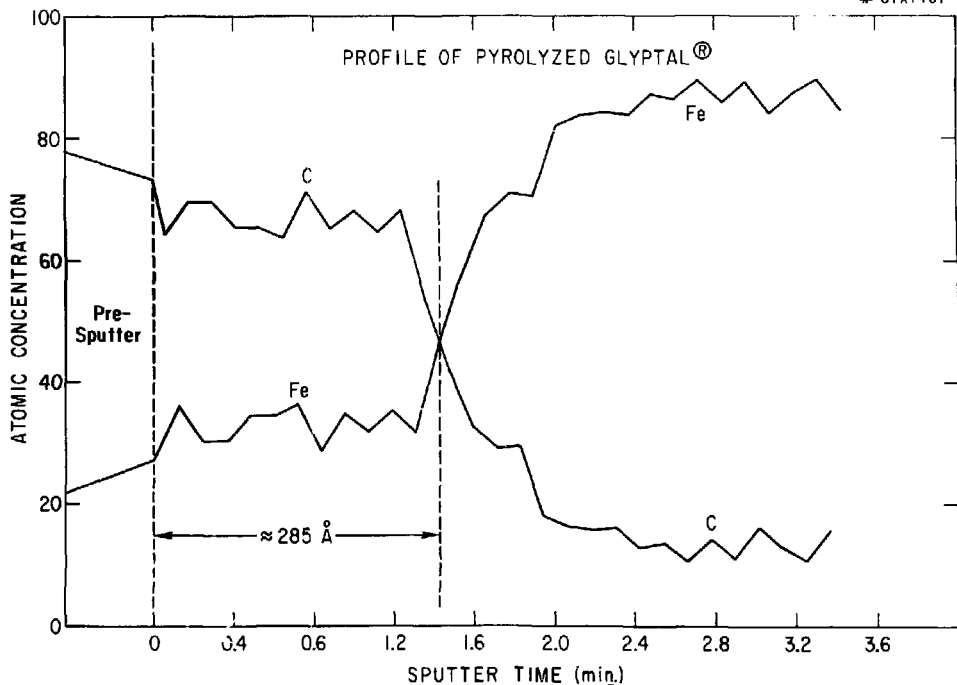


Fig. 6. Profile of pyrolyzed Glyptal®. The surface is not 30% Iron. When only two elements were surveyed the noise level registers near 30%. The coating depth given assumes that the sputter yield of 4 keV Ar⁺ on C about equals the sputter yield of 4 keV Ar⁺ on Ta₂O₅.

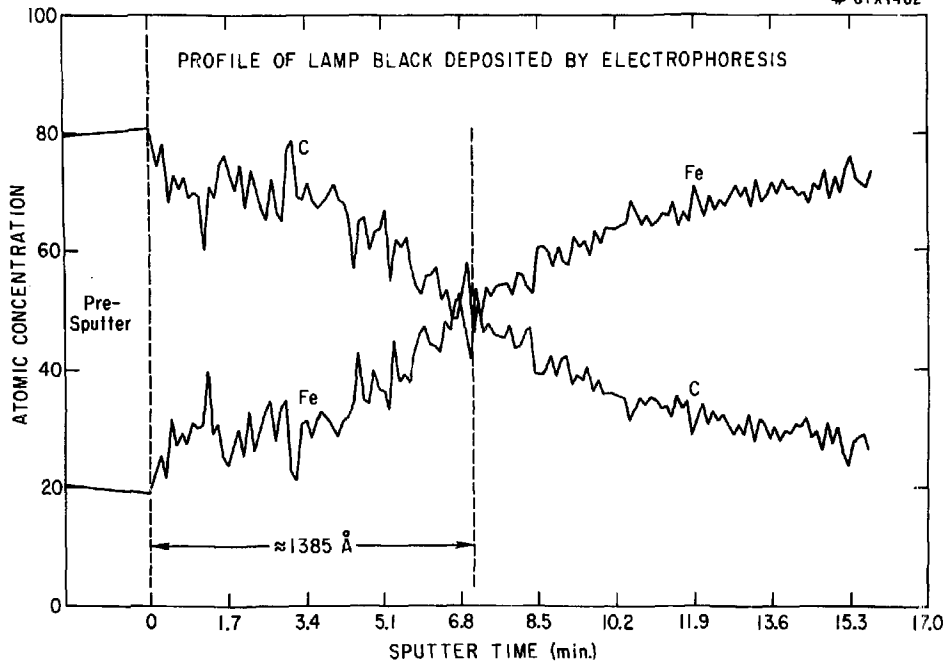


Fig. 7. Profile of ED lamp black. The surface is not 30% Iron. When only two elements were surveyed the noise level registers near 30%. The coating depth given assumes that the sputter yield of 4 keV Ar^+ on C about equals the sputter yield of 4 keV Ar^+ on Ta_2O_5 .

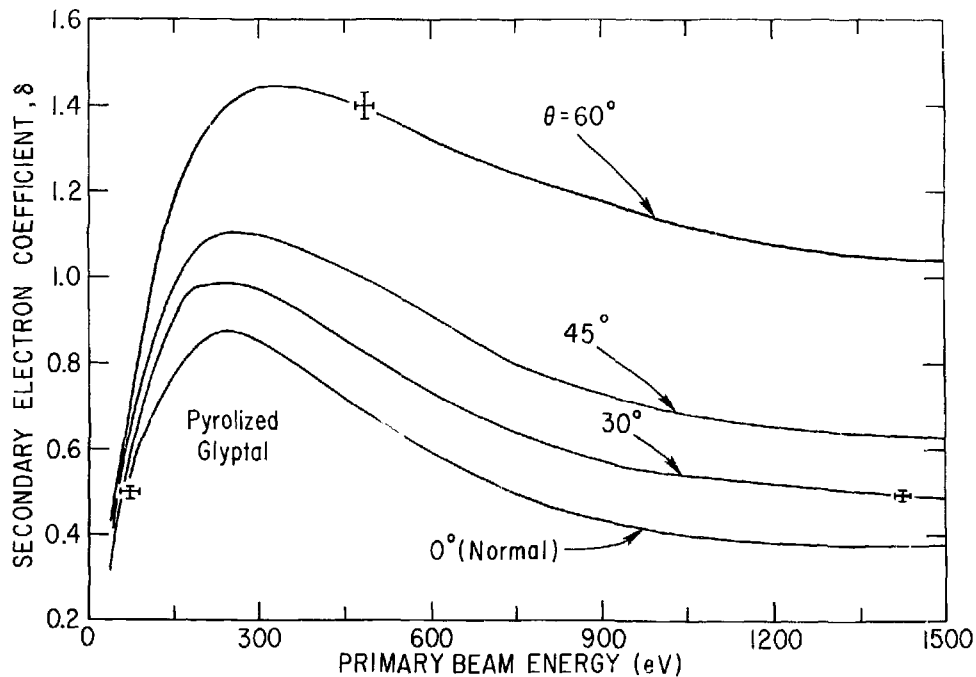


Fig. 8. $\delta(\bar{E}_p)$ for Glyptal[®] coated 304 stainless steel at four different angles of incidence. The sample was sanded with 400 grit in a grid pattern, electropolished, then dipped into Glyptal[®] and vacuum pyrolyzed. Representative error bars (\pm <2% in δ) are shown.

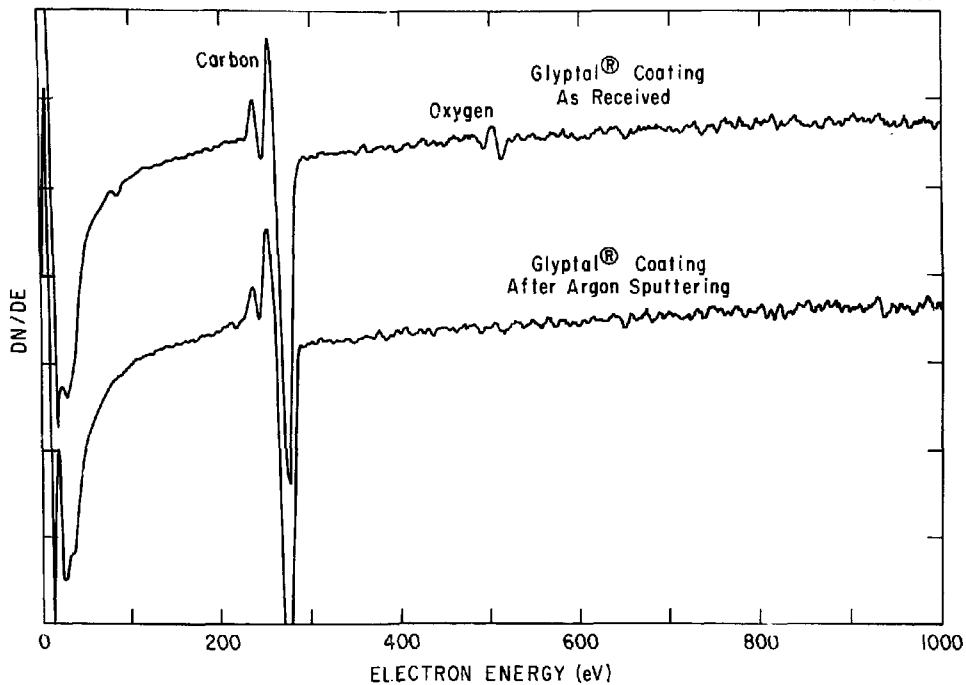


Fig. 9. Auger spectra of pyrolyzed Glyptal® before (upper trace) and after (lower trace) initial Argon sputter cleaning. About 40 Å were removed from the surface.

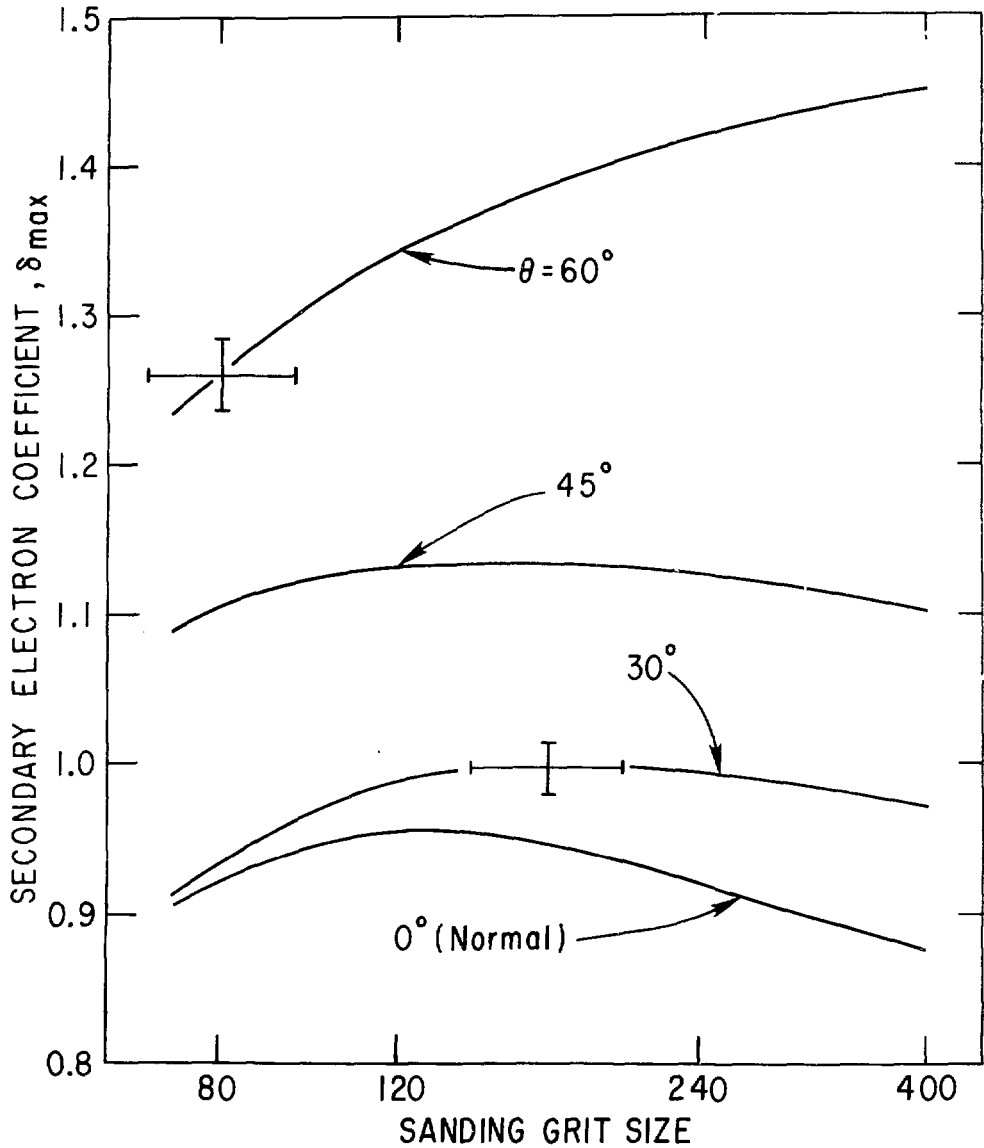


Fig. 10. δ_{\max} vs. sanding grit size at four angles of incidence for Glyptal[®] coated 304 stainless steel. The samples were sanded with various grit in a grid pattern, electropolished, then dipped into Glyptal[®] and vacuum pyrolyzed.

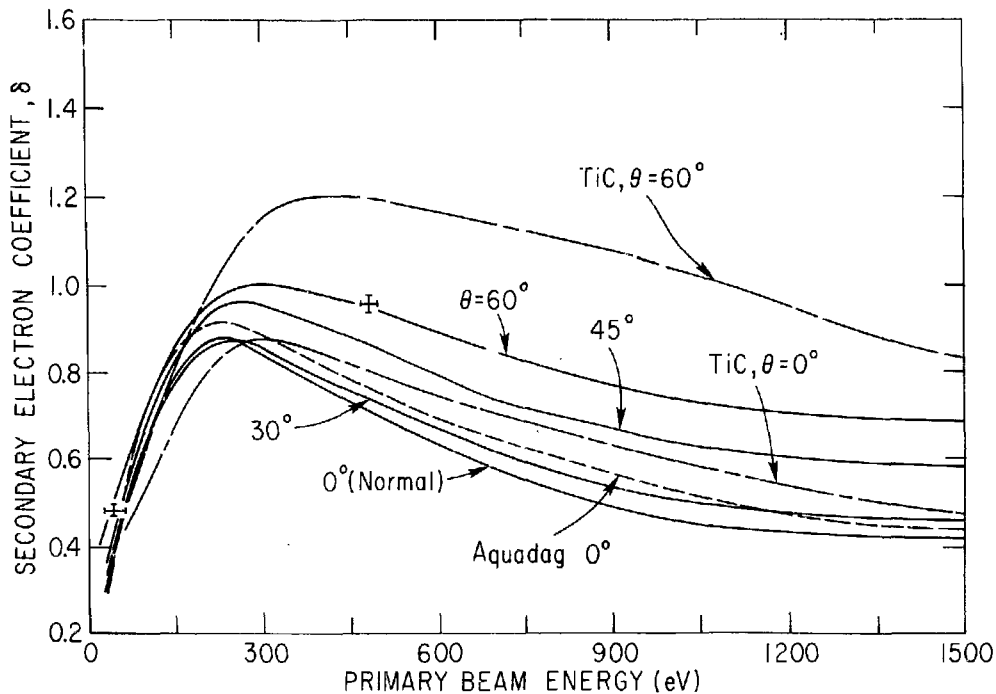


Fig. 11. $\delta(\bar{E}_p)$ for ED lamp black at four angles of incidence (solid lines), Aquadag[®] at normal incidence (broken line) and CVD titanium carbide at two angles of incidence (dot dashed lines). Except for the TiC, the pre-dipping surface treatments were identical to those in figure 3.

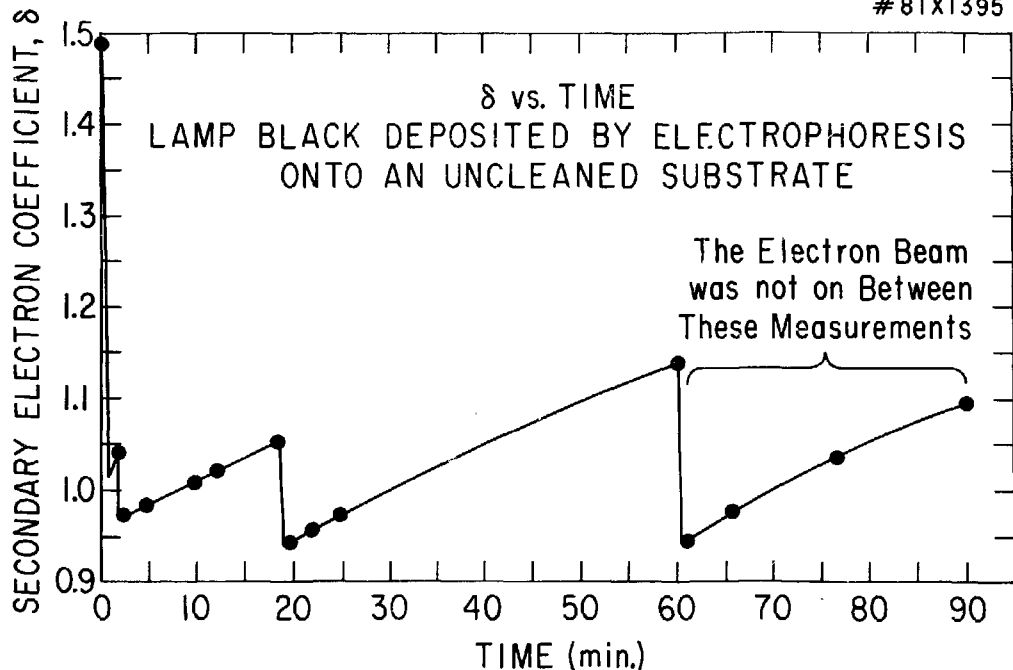


Fig. 12. $\delta(t)$ for ED lamp black deposited on to an uncleaned substrate. The vertical lines represent 40 sec. at Ar sputtering. Approximately 40 Å are removed from the surface each time. The rise in δ was not from electron bombardment. From 0 to 60 minutes the electron beam was on. From 60-100 minutes it was off. The same rise in δ can be seen in both cases.

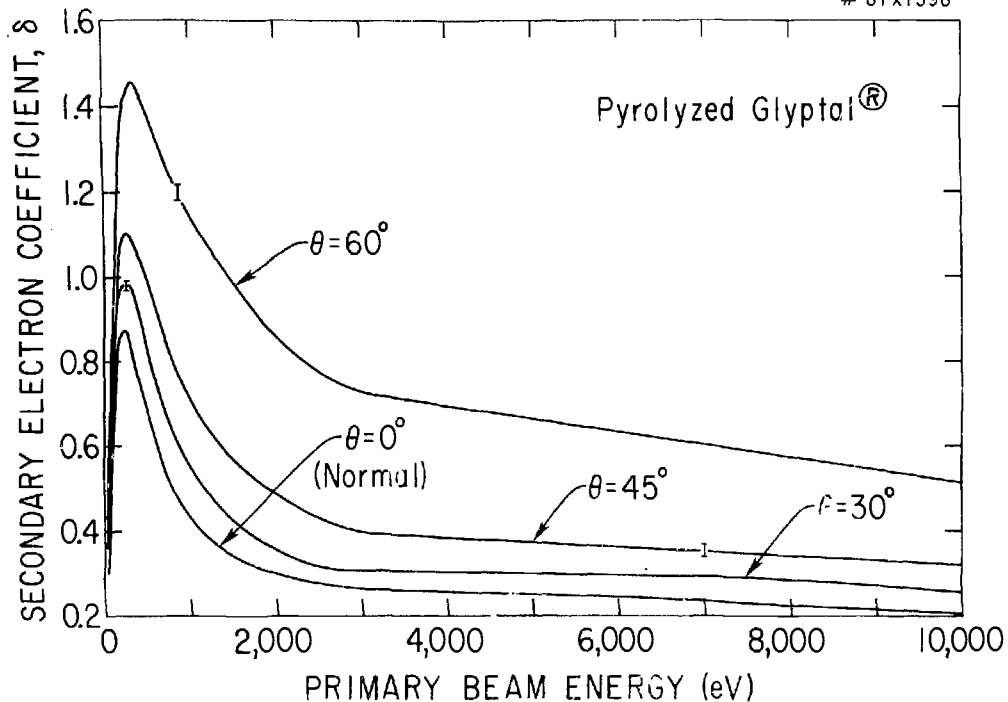


Fig. 13. $\delta(\bar{E}_p)$ for Glyptal[®] coated 304 stainless steel at four different angles of incidence. The sample was sanded with 400 grit in a grid pattern, electropolished, then dipped into Glyptal[®] and vacuum pyrolyzed.

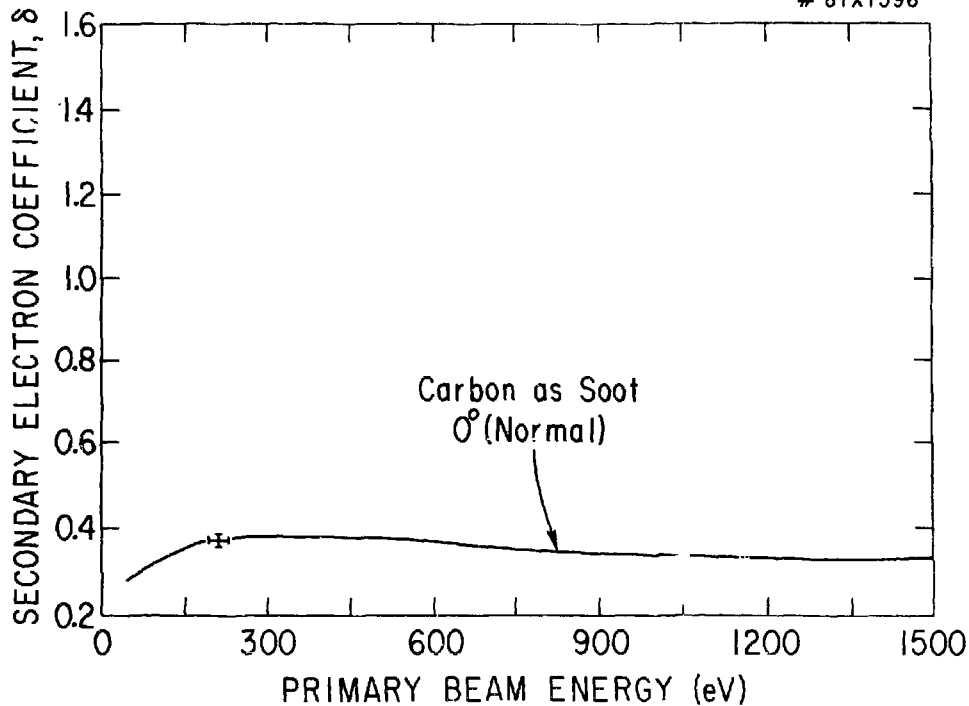


Fig. 14. $\delta(\bar{E}_p)$ for carbon deposited as soot from an oxygen poor methane flame. A representative error bar is shown.