4-8-2016

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ABSOLUTE ELECTRON EMISSION CALIBRATION: ROUND ROBIN TESTS OF Au AND GRAPHITE

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ABSTRACT

This study presents a round robin comparison of absolute electron yields of conducting and insulating materials performed in four international laboratories. Measurements are reported for two standard elemental conducting samples with reproducible sample preparation for Au and highly oriented pyrolytic graphite (HOPG). The primary objectives of the study were to determine the consistency and uncertainties of these absolute yields measurements, and to investigate the effects of the similarities and differences of the diverse facilities. In this initial round robin study, HOPG total electron yield results were found to be more reproducible than those for Au. Surface contamination significantly affected the total electron yield, even for relatively inert conductive samples like Au and HOPG.

1. INTRODUCTION

Accurate determination of the absolute electron yields of conducting and insulating materials are essential for models of spacecraft charging and related processes involving charge accumulation and emission due to electron beam and plasma interactions. Measurements of absolute properties require careful attention to calibration, experimental methods, and uncertainties. The primary objectives of the study were to determine the consistency and uncertainties of these absolute yields measurements, and to investigate the effects of the similarities and differences of the diverse facilities.

This study presents a round robin comparison of such tests performed in four international laboratories. Apparatus using low-fluence pulsed electron beam sources and various methods to minimize charge accumulation have been developed and employed at these facilities. An outline of measurement and analysis techniques used by each laboratory is presented, along with methods used to calibrate the incident energies and absolute yields measured with their different electron detectors. The effects of different charge minimization and neutralization methods are compared.

Absolute electron yield measurements for various materials are necessary to determine absolute charging levels and hence to predict possible electrostatic breakdown and injection of charges into plasmas. They have direct application to spacecraft charging, high voltage direct current (HVDC) power and transmission lines, ion thrusters, plasma deposition, multipactors, semiconductor metal-oxide interfaces, and nanodielectrics.

2. ELECTRON EMISSION TEST FACILITIES

Measurements were made of the absolute total, secondary and backscattered electron yields at normal incidence over the full range of incident energies accessible with each group's instrumentation (a full range of ~5 eV to ~6 keV).

2.1. CSIC SEY Facility

The CSIC SEY Facility of the Surface Nanostructuring for Space and Terrestrial Communications Group of ICMM-CSIC does research on dielectric, magnetic and metallic materials for space applications [1-3]. A main goal of these research activities is surface characterization by UHV spectroscopic techniques and low-secondary emission surfaces to avoid multipaction effects in RF high-power devices for satellite communications systems [3].

The CSIC SEY facility at ICMM (see Fig. 1) [1] has four interconnected UHV chambers (10^{-7} Pa) chambers equipped with:

- Four electron guns (pulsed/continuous),

- Ion gun (Ar, O, CH_x, …),
- VUV source (pulsed/continuous),
- X-ray source (Mg/Al anodes),
- Hemispherical electron analyzer,
- Quadrupole residual gas analyzer,
- Flexible sample size (12 - 250 mm),
- Sample Manipulation
  - Sample rotation: -90º to +90º,
  - UHV helium cryostat-micrometric manipulator, XYZθ (4 K-900 K)
  - UHV Micrometric manipulator XYZθ (<900 K),
  - UHV X(Zθ) nanometric manipulator,
  - UHV XZθ manipulator (1.8 m length),
- Temperature range: 4 K to 900 K.

Measurements capabilities include:

- **SEY (true secondaries and backscattered):**
  - Continuous method with total primary currents of <5 nA/cm^2,
  - Single pulse method with <180 ns pulses,
- **Energy Distribution Curves** (EDC). Primary energy (0-5 keV) and relative emission angle-dependence,
- **X-Ray Photoemission Spectroscopy** (XPS),
- **Auger Electron Spectroscopy (AES),**
- **Reflection High Energy Electron Loss Spectroscopy** (RHEELS),
- **Intensity-Voltage and Capacitance-Voltage** characterization**,**
- **VUV Photoemission** quantum yield,
- **Thermal desorption** processes**,**
- **Versatile sample conditions:**
  - Flexible sample sizes (<250 mm),
  - Extensive sample manipulation,
  - Simultaneous temperature range 4-900 K.

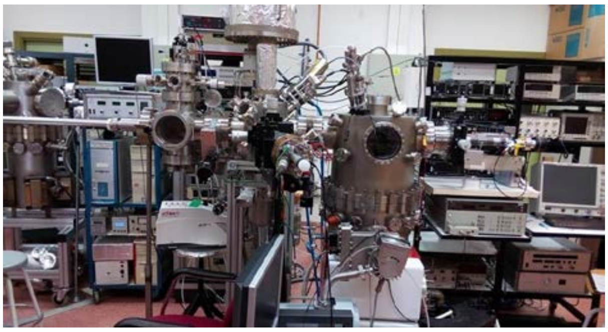
Electron yield results for conductors reported here were measured using the continuous sample current method [see Fig. 1(b)]. The sample current, I_s, and pulse current, I_p, are as-measured values to ground. The total electron yield is calcualted as

$$\sigma = 1 - \frac{I_S}{I_P} \quad .$$

Typical uncertainites for this method are $\sigma \pm 0.005$, $E_{max} \pm 1$ eV and $E_1 \pm 0.1$ eV. The sample is typically biased to -30 V to inhibit reabsorption of emitted low energy secondary electrons. For the pulsed method, yields are calculated by integrating currents over a single pulse duration [see Fig. 1(c)].

2.2. LaSIENE Facility

The Laboratory of Spacecraft Engineering INteraction Engineering (LaSEINE) at Kyushu Institute of Technology has studied spacecraft charging and discharging [4]. The Total Electron Emission Yield (TEEY) measurement facility was developed to acquire electron yield properties for a materials database for the charging analysis tool *MUSCAT*. TEEY of space conductive materials, as well as insulating material, have been measured. TEEY has also been measured after irradiation of samples with ionizing radiation, atomic oxygen, and ultraviolet light [5].



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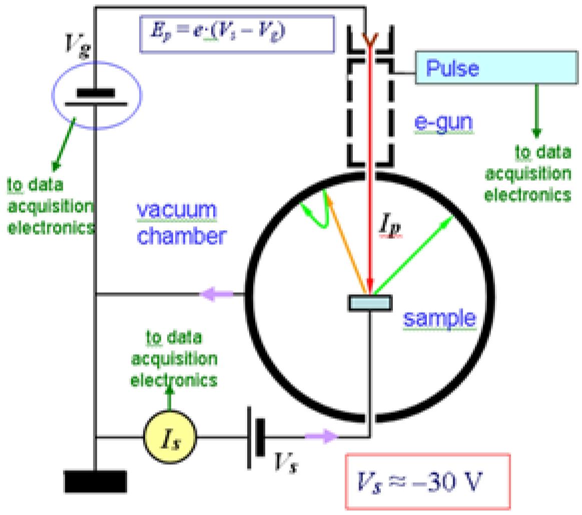

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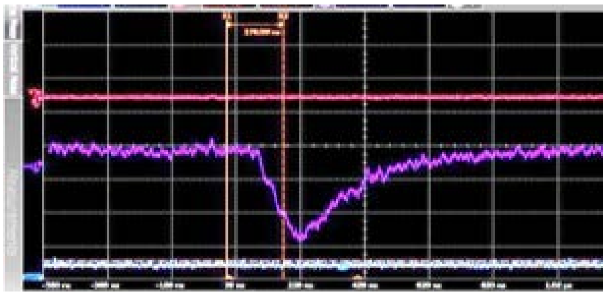
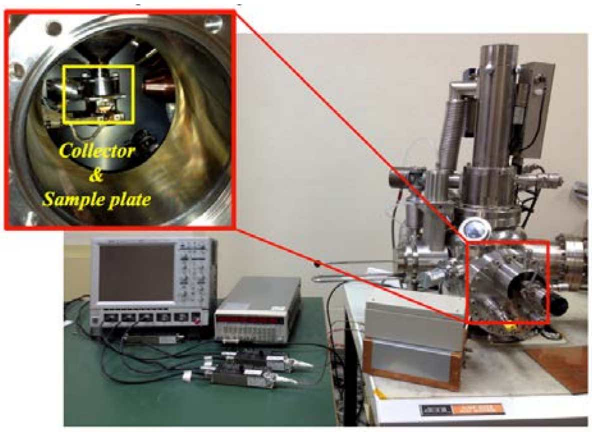


(c)

Fig. 1. CSIC SEY facility. (a) Vacuum chambers. (b) Block diagram of system. (c) Current traces of pulse and sample currents.

The LaSIENE TEEY facility has a high vacuum (10^{-5} Pa) chamber [see Fig. 2(a)] [4]. Measurements capabilities include:



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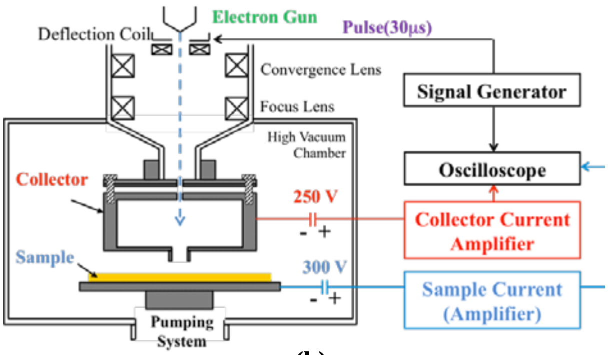

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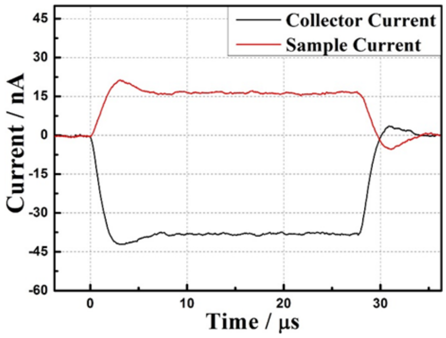

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Figure 2. LaSEINE TEEY facility. (a) Vacuum chambers. (b) Block diagram of system. (c) Current traces of collector and sample currents.

- **Electron Gun**: 300 eV-10 keV,
- **Electron Irradiation:** continuous or pulsed,
- **Sample Stage** movable in X-Y directions,
- **Temperature** sample holder control of 240-370 K.

Electron yield was measured using the pulsed sample current method [see Fig. 2(b)]:

- Pulses of ~20 nA with ~30 μs duration are typically used.
- The emitted current was measured with a collector designed to fully capture all emitted electrons.
- Sample currents were measured directly.

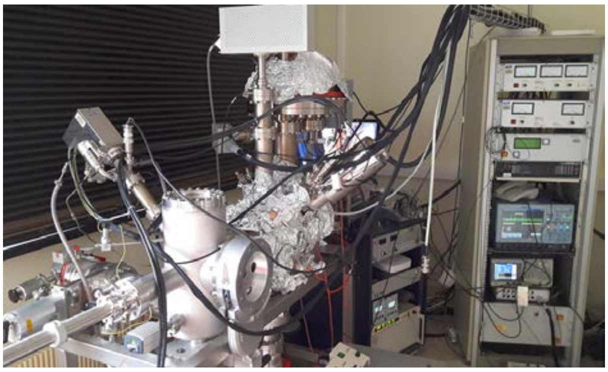

Figure 3. ONERA DEESSE Facility

- The incidendent current is calculated as the sum of the sample and collector currents.
- The collector can be biased negatively to adjust the landing energy of electrons from the electron gun. The sample was biased -50 V relative to the collector to inhibit reabsorption of emitted low energy secondary electrons.

The total electron yield is calculated as

$$\sigma = 1 - \frac{I_S}{I_C + I_S} \quad .$$

2.3. ONEREA DEESSE Facility

The DEESSE (Dispositif d'Etude de l'Emission Secondaire Sous Electrons) facility of the Space Environment Department (DESP) of Onera works on many projects closely related to space applications dealing with electron emission, such as charging effects of spacecraft and Hall Effect Thruster technology (HET) [6].

The DEESSE facility at ONERA [7] has a UHV Analysis chamber (10^{-7} to 10^{-8} Pa) with a high vacuum Transfer chamber (10^{-6} Pa) equipped with (see Fig. 3):

- 3 electron guns (pulsed/continuous),
- Ion source (Ar, Xe, H) from 25 eV to 5 keV,
- VUV source and X-ray (Mg/Al) sources,
- a hemispherical electron analyzer,
- a surface potential probe.

Measurements capabilities include:

- **Electron Guns:** Kimball Physics 1 eV-2 keV; Kimball Physics 50eV-5 keV; Staib 1 keV-22 keV.
- **Incident Current** measured by Faraday cup.
- **Surface Analysis** Auger Electron Spectroscopy (AES).
- **X-Ray Photoemission Spectroscopy** (XPS),
- **Electron Energy Loss Spectroscopy** (EELS),

- **Sample Rotation** -90° to +90° to study incidence angle effects.
- **Temperature Control** of sample holder from ambient to 500 °C.

Electron yield was measured using the pulsed sample current method:

- Incident current was measured as function of incident energy using the Faraday cup (polarized to +24 V).
- Thereafter, sample current was measured as function of incident energy.
- The sample holder was biased to -18 V in order to avoid the collection of the low energy tertiary electrons by the sample surface.
- After that, incident current stability was confirmed for select energies. With the Kimball Physics electron gun, the observed variation is <2%.
- To limit conditioning effect, electron beam was pulsed (5 µs pulse for conducting materials).

The total electron yield is calculated as

$$\sigma = \frac{[I_B - I_S]}{I_B}$$

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2.4. USU SEEM Facility

The Utah State University Materials Physics Group (MPG) Space Environment Effects Materials (SEEM) test facility performs state-of-the-art ground-based testing of electrical charging and electron transport properties of both conducting and insulating materials, emphasizing studies of electron emission, conductivity, luminescence, and electrostatic discharge. They have studied how variations in temperature, accumulated charge, exposure time, contamination, surface modification, radiation dose rate and cumulative dose affect these electrical properties—or related changes in structural, mechanical, thermal and optical properties—of materials and systems.

The SEEM facility has a high vacuum (10^{-5} Pa) chamber [see Fig. 4(a)] [8,9]. Measurements capabilities include:

- **Total/Secondary/Backscattered Electron Yields** using <20 eV to 50 keV mono-energetic pulsed beams with <5% absolute uncertainty.
- **Electron Emission Spectra** versus energy (0-5 keV with ~0.1 eV resolution) and emission angle-dependence.
- **Ion-Induced Electron Emission** spectra and yields for various <300 eV to 5 keV mono-energetic inert and reactive ions.

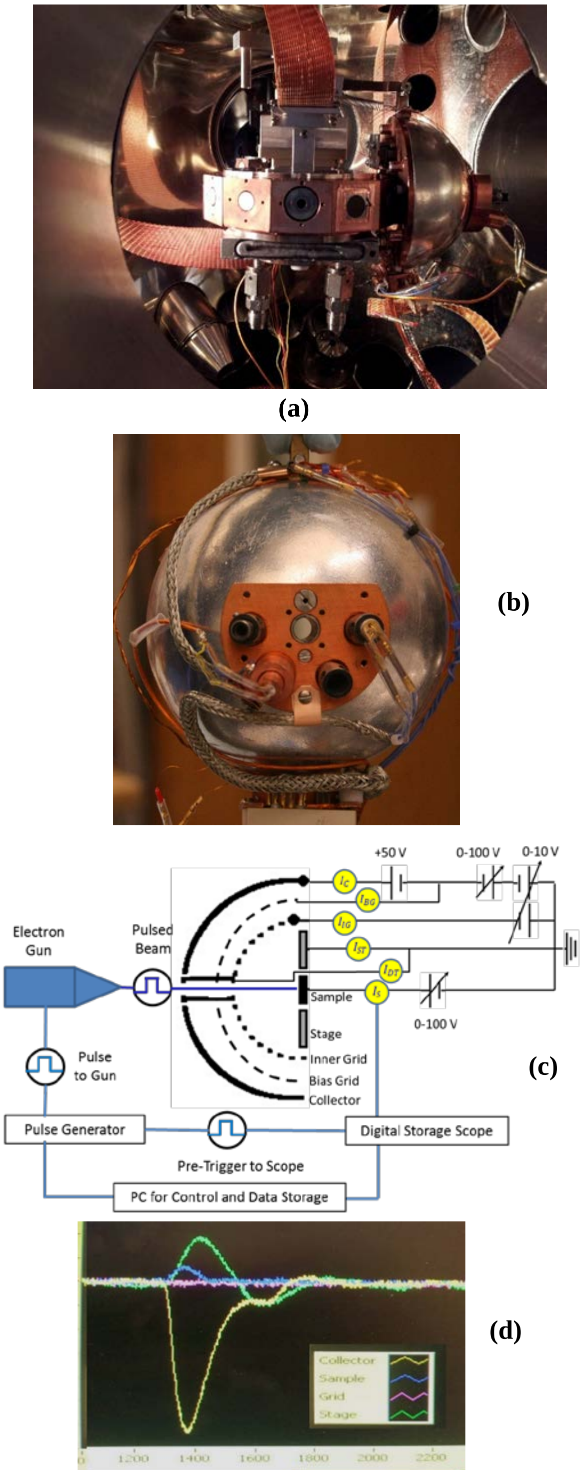

Figure 4. USU SEEM Facility. (a) Chamber interior, showing the sample carousel and hemispherical grid retarding field analyser. (b) Electron flood gun and UV LED on the exterior of the hemispherical grid retarding field analyser. (c) Block diagram of system. (d) Current traces of collector, sample, bias grid and Faraday cage (inner grid, stage and drift tube) currents.

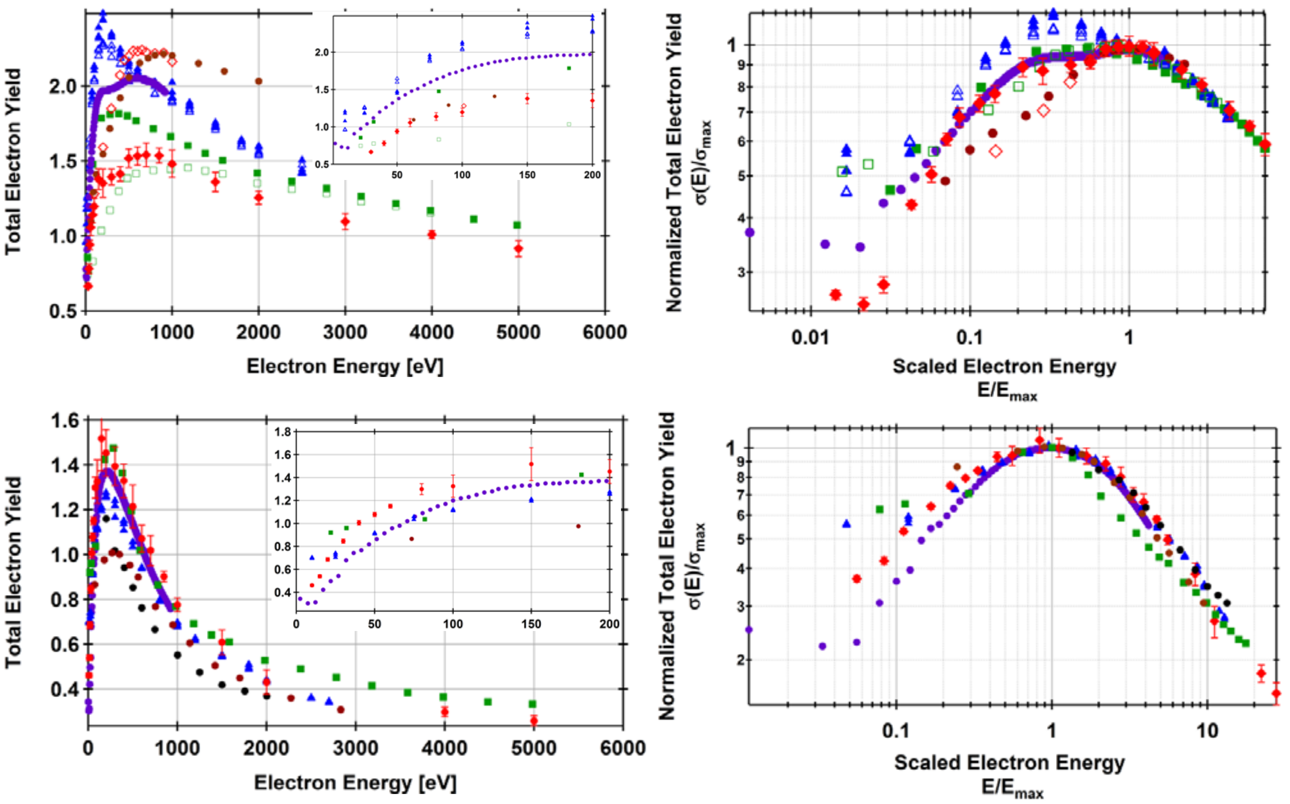

Figure 5.Total electron yield versus incident electron energy for Au (a and b) and HOPG graphite (c and d). (a) and (c) are linear plots with low energy detail insets. (b) and (d) are log-log plots of scaled yields $\sigma(E)/\sigma_{max}$ versus scaled energy E/E_{max} Refer to Tables I and II for definitions of the plotting symbols.

- **Photon-Induced Electron Emission** spectra and yields for <0.6 eV to >6.5 eV (165-2000 nm) monochromated photons.
- **Surface Voltage** simultaneous measurements of 0-10 kV with <0.2 eV resolution.
- **Induced Electrostatic Breakdown** simultaneous current and NIR/VIS/UV optical measurements.
- **Sample Manipulation:** XYZθ motion of a 10 sample carousel.
- **Temperature** capabilities from <60 K to >450 K [8].

Electron yields calculated from integrated current traces from six detector elements [see Fig. 4(d)] of a fully enclosed hemispherical grid retarding field analyser used for emission electron energy discrimination [see Fig. 4(b)] [9]. The total electron yield is calculated as

$$\sigma = 1 - \frac{\int_{pulse}[I_S + I_{St} + I_{IG} + I_{BG} + I_{DT}]dt}{\int_{pulse}[I_C + I_S + I_{St} + I_{IG} + I_{BG} + I_{DT}]dt}$$

Refer to Fig. 4(c) to identify each current element. Pulses used are typically 3-5 µs duration at <1 nA-cm^{-2} beam current densities for 1-3 mm diameter beam spots.

Both a low energy ~5 eV flood gun and a ~5 eV UV LED are used for a few seconds between each incident electron pulse to neutralize charged insulating samples [9,15].

3. RESULTS

Measurements were made of the absolute total electron yields at normal incidence over the full range of incident energies accessible with each group's instrumentation (a full range of ~5 eV to ~6 keV). Measurements were made for identical samples with reproducible sample preparation of three standard materials:

- the elemental conductor Au (25 µm thick 6N high purity Au foils)
- the elemental semimetal graphite (DOW highly oriented pyrolytic)
- the polymeric insulator polyimide (25 µm thick Kapton HN™)

Total electron yield results for Au and HOPG are reported here.

Figures show linear plots with low energy detail insets [Figs. 5(a) and (c)] and log-log plots of scaled yields

Table I. Total electron yield measurements of Au.

Symbol	Facility	Refs.	Measured Values			
			σ_{max}	E_{max} (eV)	E_1 (eV)	E_2 (eV)
●	CSIC SEY Facility (contin.)	1-3	2.06±0.02	600±20	24±1	NA
■	Onera—DEESSE Facility	4-5	1.81±0.03	360±20	28±1	~6000
□	Onera—DEESSE (Etched)	4-5	1.46±0.03	1250±50	NA	~6000
▲	LaSeine—TEY Facility (March 2)	6-7	2.48±0.03	200±20	NA	NA
Δ	LaSeine—TEY Facility (March 6)	6-7	2.3±0.1	240±30	12±2	NA
♦	USU—SEEM Facility (pulsed)	8-9	1.54±0.08	700±30	55±3	4000±200
◊	USU—SEEM Facility (contin.)	8,14	2.24±0.02	650±50	NA	NA
	Round Robin Average Values		2.0±0.4	600±460	24±9	5000±1000
●	Standard: Thomas & Pattinson	10	2.21±0.02	900±30	50±10	NA

Table II. Total electron yield measurements of HOPG graphite.

Symbol	Facility	Refs.	Measured Values			
			σ_{max}	E_{max} (eV)	E_1 (eV)	E_2 (eV)
●	CSIC SEY Facility	1-3	1.38±0.02	215±5	68±1	629±10
■	LaSeine—TEY Facility	4-5	1.48±0.05	270±20	60±2	610±40
▲	Onera—DEESSE Facility	6-7	1.28±0.05	190±10	69±1	552±20
♦	USU—SEEM Facility	8,9,14	1.4±0.1	200±30	40±2	895±20
	Round Robin Average Values		1.39±0.08	220±36	59±13	670±150
●	Standard 1: Whetten	11	1.01±0.01	300±20	250±30	350±20
●	Standard 2: Wintucky	12	NA	NA	NA	330±20

$\sigma(E)/\sigma_{max}$ versus scaled energy E/E_{max} [Figs. 5(b) and (d)]. The values determined at each laboratory for the maximum yield, σ_{max}, and energy at this yield, E_{max}, and the first and second crossover energies, E_1 and E_2 are also compiled and compared in Tables I and II.

4. CONCLUSION

In summary we offer the following conclusions:

- The total electron yield is highly sensitive to surface contamination [14],
- The shape of the normalized yield curves are very consistent,
- There is very good agreement of absolute yield for $E>E_{max}$, but less agreement for $E<E_1$,
- Agreement of HOPG total electron yields between facilities are ~5% for σ_{max} and ~20% for energies E_{max}, E_1 and E_2,
- HOPG has the advantage that clean smooth surfaces are easy to prepare with tape cleaving,
- The agreement for total electron yield is not as good for Au,
- Au samples exhibit differing degrees of contamination, as evidenced by surface analysis tests. They exhibit two TEY peaks near 700 eV (clean Au) and 200 eV (C contamination) [14].

Topics of future round robin analyses, based largely on measurements already made at the four facilities, include:

- Discussions of the relative strengths and weaknesses of the various facilities' methods,
- Energy discriminated measurements, including, secondary and backscattered yields and emission spectra,
- Charge sensitive measurements of dielectrics; specifically, polyimide (Kapton HN™) results,
- Surface sensitivity, as related to surface cleanliness tests, effects of contamination, surface roughening, and Ar sputtering.

Studies of the effects on electron yield of Au surface contamination—as measured with Auger Electron Spectroscopy and other surface analysis techniques—will be studied for samples: (1) as received, (2) subject to a simple standard cleaning procedure, (3) subsequently baked out under ultrahigh vacuum, and (4) after Ar ion sputter cleaning and thermal annealing. Similarly, studies of the effects of absorbed water and volatile compounds on electron yield will be made for polyimide samples: (1) unbaked and (2) subjected to a vacuum bake out.

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