

Investigation of the electron emission properties of silver: From exposed to ambient atmosphere Ag surface to ion-cleaned Ag surface



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ABSTRACT

Electron emission properties of materials are highly dependent to the surface and the first nanometres subsurface. Technical materials, i.e. used within applications are ordinarily exposed to atmosphere, which interacts with the surface. The contamination layer building up at the surface of materials and/or oxidation layer affects dramatically the electron emission properties. In this paper, starting from 99.99% pure silver sample, exposed 4 years to ambient atmosphere, we monitored the variations of the electron emission properties and the surface composition during step by step ion etching procedure.

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1. Introduction

When a solid is hit by incident electrons, the energy transfer can result in the emission of secondary electrons (SE) and backscattered electrons (BSE). Electron emission (EE) is a physical phenomenon involved in several scientific and technical fields such as micro-analysis [1], particle accelerator [2,3] space technology (spacecraft charging [4], hall effect thrusters [5]), nuclear Physics [6]. Electron emission is also at the origin of multipactor effect that can occur in several radiofrequency (rf) devices operating under vacuum (waveguides, circulators). Multipactor effect can be defined as the generation of an electron cloud by resonance between electron emission and rf field [7]. The consequences of this adverse effect are a power limitations and additional constraints in the design of rf components. The estimate of its onset threshold is highly needed. In many situations, the threshold could be estimated from simulations [8]. In the framework of a multi-laboratory project [9], it was clearly established that all tested multipactor models and simulation software are extremely sensitive to the electron emission properties of the material components. Therefore, to improve the modelling of this effect and prevent occurrence, it is necessary to determine accurately some properties of the emitted electrons such as (i) the secondary electron emission yield (SEY), (ii) the electron

backscattered yield (BSEY) and (iii) the energy distribution $N(E)$. These properties are ordinarily extracted from measurements performed on materials of high purity evaporated [10] or ion-cleaned [11] under ultra-high vacuum conditions (UHV). However, the materials used in rf devices are usually exposed days and months to ambient atmosphere. Thus, their surface properties are extremely different from that of the bulk. Since the emitted secondary electrons have a low energy (a few eV) and are generated in the first few nanometers in depth; obviously, the tabulated EE properties of pure materials are far from being representative of technical materials. For instance, the maximum of the electron emission yield (EEY) of pure aluminum is lower than 1 [10], while that of technical aluminum is higher than 3 [12]. The aim of this work is to study experimentally the electron emission of exposed to atmosphere silver and to establish the relation between the EE properties and the surface composition. Technical Ag surface (exposed to atmosphere) is supposed to be composed by Ag_2S [13] and other deposited compounds, like water and hydrocarbons [14]. A special experimental protocol was developed to extract relevant quantities. The electron emission yield (EEY) is investigated from very low incidence energy (some eV) to 2000 eV. A step by step Argon ion etching was performed in situ. The surface composition is monitored by Auger electron spectroscopy (AES) during cleaning process. The EEY was measured at each step. An overall drop of the EEY was observed during the ion cleaning process. The maximum of the EEY decreases from 2.26 to 1.67 and the first crossover energy increases from 18 to 130 eV during the ion cleaning process. The effect of the

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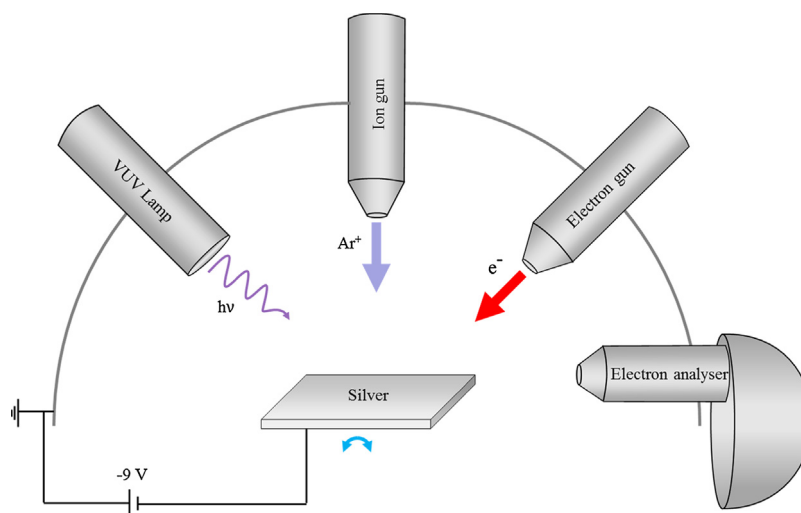


Fig. 1. Schematic representation of experimental setup CELESTE.

ion-cleaning on the EEY tends to be suppressed after venting of the vacuum chamber. The EEY variations were linked to the surface composition.

2. Experimental

2.1. The experimental setup

The principle of the experimental protocol is represented in Fig. 1. EEY measurements, AES and electron emission spectra were recorded before and after each erosion step. Similar protocol has been used by Contini et al. [15] to study electron emission loss spectra of aluminum at only three etching steps. All measurements were performed in the CELESTE facility at ONERA. This facility is entirely dedicated and designed to the study of electron emission. A dry turbo-molecular pump associated with an oil-free primary pump allows the system to be maintained at a vacuum level down to 5×10^{-9} mbar. The tank is grounded. The sample holder allows the variation of the electron incidence angle from 0° (normal incidence angle) to 60° . An ELG-2 electron gun from Kimball Instrument was used. The electron beam was pulsed during EEY measurements to limit the surface conditioning effect [12] and was continuous during spectra acquisition. The Tectra ion gun used has an energy range from 50 to 5000 eV. Argon gas is injected through a microleak valve and atoms are excited by microwaves generated by a microwave magnetron with a 2.45 GHz frequency. The Omicron hemispherical electron analyzer can record spectra from 2 to 2000 eV with an accuracy of 0.5 meV. The sample was negatively biased to -9 V during EEY and AES measurements to avoid experimental noise in the very first eV of the emission spectrum in the AES case and to avoid low energy secondary electrons to be recaptured by the sample. It should be noticed that due to the -9 V sample biasing, all the AES spectra are shifted by 9 eV to high energies. Prior to measurements, the tank was baked to 180°C for 16 h.

2.2. Sample

The sample was Ag (Ag00470/31) of high purity (99.99%) provided by Goodfellow Company. It was exposed for more than 4 years to ambient atmosphere. It has a square shape of 32×20 mm² area and a 0.5 mm thickness. Roughness measurements performed on the sample using a Zygo interferometer prior to the experiments revealed an average roughness of 160 nm. The sample has

been washed by ethanol before mounting in the measurement system, where it was outgassed in UHV for 10 days before the first measurement. It was also heated to 100°C for 4 h during the UHV exposure.

2.3. Erosion parameters

The sample was etched with Argon ions in 14 steps. Short ion irradiations durations were chosen for the first steps to avoid a too fast composition evolution. The irradiation duration of each step was progressively increased, starting with 30 s for the first one until 30 min for the last step. The same parameters (Ar ions energy of 1 keV, $3.5 \mu\text{A cm}^{-2}$, normal incidence) for each erosion step were applied; only the erosion duration of each step was varied. The ion current, measured using a Faraday cup was adjusted to $3.5 \mu\text{A cm}^{-2}$. The results presented in this paper are focused on (a) the as received sample (before erosion), (b) the sample at an intermediate state corresponding to an erosion time of 62.5 min and (c) the sample considered as completely cleaned after 132 min of erosion (final state). The final state is considered reached when both the AES spectrum, due to contaminants and EEY have reached a steady state. The thickness of the removed layer was estimated considering the etching rate (atom/ion) found in literature [16–18]. Two major hypothesis were used to estimate removed thickness as a function of the etching time:

1. The measured ion current is not totally representative of the incident Ar flux, neutral Ar may also impact the sample surface and therefore lead to underestimate of removed thickness.
2. The etching rate considered is that of pure silver. This hypothesis is partially incorrect, especially for the first etching steps when the major portion of ejected atoms are contaminants and not silver. This may lead to uncertainty in the removed thickness estimation because the etching rate of contamination layer is unknown.

3. Results

3.1. Surface composition

The AES microanalysis technique was used to monitor the surface and near surface chemical composition (few nanometers in depth). After each erosion step, several AES peaks (as Ag, C, O,

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