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Investigation of argon ion sputtering on the secondary electron emission from gold samples

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ABSTRACT

Secondary electron (SE) yield, δ, is a very sensitive surface property. The values of δ often are not consistent for even identical materials. The influence of surface changes on the SE yield was investigated experimentally in this article. Argon ion sputtering was used to remove the contamination from the surface. Surface composition was monitored by X-ray photoelectron spectroscopy (XPS) and surface topography was scanned by scanning electron microscope (SEM) and atomic force microscope (AFM) before and after every sputtering. It was found that argon sputtering can remove contamination and roughen the surface. An "equivalent work function" is presented in this thesis to establish the relationship between SE yield and surface properties. Argon ion sputtering of 1.5keV leads to a significant increase of so called "work function" (from 3.7 eV to 6.0 eV), and a decrease of SE yield (from 2.01 to 1.54). These results provided a new insight into the influence of surface changes on the SE emission.

1. Introduction

When a solid is bombarded by electrons, it will respond by emitting secondary electrons (SE). This phenomenon has generated considerable interest since its discovery by Austin and Starke [1] in 1902. There has been much work on secondary electron emission (SEE) because of its important role in many fields [2,3], such as scanning electron microscopy (SEM). However, SEE is serious constraint to the development of electron accelerator, high power microwave and space microwave device performance [4–7]. SEE is the origin of multipactor effect, which sets one of the main limits to the working power of RF devices under vacuum.

Since the emitted SEs have a low energy (a few eV) and are generated along a few nanometers depth, SE yield is a very sensitive surface property. The literature devoted to this topic is extensive. Unfortunately, as reported by various authors, the values of SE yield are confused and often are not consistent for even identical materials [8]. In order to solve this problem, further work has been undertaken to investigation how may such a characteristic be affected?

It is the fact that a contamination effect leads to an increase in the SE yield [9,10]. The purpose of this article is to investigate experimentally argon ion sputtering on secondary electron emission of Au. Argon ion sputtering is used to remove contaminations. The surface composition is monitored by X-ray photoelectron spectroscopy (XPS) and the surface topography is scanned by scanning electron microscope (SEM) and atomic force microscope (AFM) at each sputtering step. The SE yield and the energy distribution of the emitted electrons were measured in suit.

2. Materials and experiments

Gold is not easily oxidized and it was therefore used in this study. The samples with purity 99.99% and thickness 0.3 mm were ultrasonically cleaned for 10 min each using anhydrous alcohol and de-ionized water. In order to study the SE yield and the SE spectra on the surface properties, the duration of sputtering energies with argon ions was varied from 0.1keV to 1.5keV. The experimental relation between the ion beam current I^+ and Ar^+ energy E_{ion} can be fitted well to an exponential function, $I^+ = \exp\left(-2.11 + 5.17E_{ion} - 1.507E_{ion}^2\right)$, where I^+ and E_{ion} are in μA and keV. Every sample was sputtered with fixed sputtering time T_{ion} of 10 min. During the process, the pressure was set to 1.6×10^{-6} Pa. The SE yield, SE spectra and BSE yield were measured before and XPS, SEM, AFM were also carried out to monitor the surface after every sputtering.

The measurements of yields and XPS were performed in an ultrahigh vacuum (UHV) chamber. It has a base vacuum pressure of 2×10^{-7}Pa and includes electric gun, argon ion sputtering, SE spectrum and XPS. The principle of the sample treatment and

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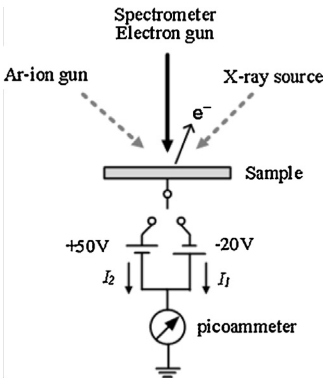

Fig. 1. Schematic representation of experimental system for Ar⁺ sputtering and surface analysis. The total SE yield σ, SE spectra, BSE yield η and XPS were measured before and after every sputtering. Here, the sample current I_1 and I_2 represent $(I_{PE} - I_{tot})$ and $(I_{PE} - I_{BSE})$ at a bias of −20 V and +50 V, respectively. The primary current I_{PE} is measured by a Faraday cup. The yields can be calculated from $\sigma = 1 - I_1/I_{\mathrm{PE}}$, $\eta = 1 - I_2/I_{\mathrm{PE}}$.

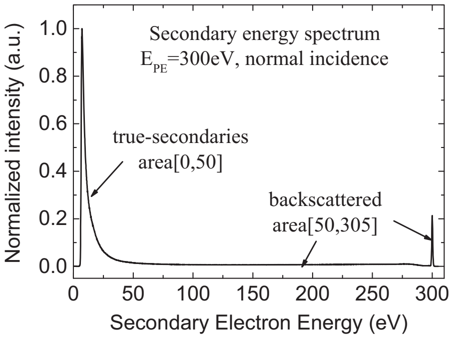

Fig. 2. A sample of measured energy spectrum for gold at E_{PE} = 300eV, normal incidence. The energy range of true-SEs is chosen to be 0–50 eV conventionally.

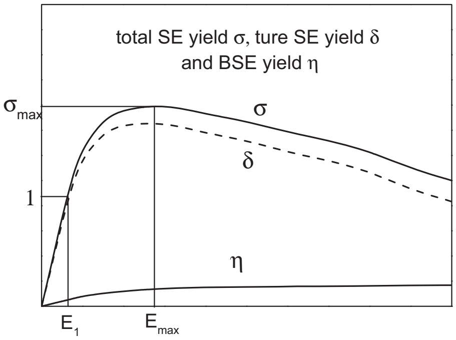

Fig. 3. An example of total SE yield σ, true SE yield δ and BSE yield η.

measurements is illustrated in Fig. 1, and some details of the related UHV facility have been described in Ref [11]. The X-ray source was newly-installed and the spectra were obtained by electron spectrometer DESA 150. It is note that, the argon ion and the X-ray incident at a 45° angle.

The general process [12,13] of secondary electron emission are well understood. In general, primary electrons (PE) impinge upon the surface of the material and secondary electrons emit from the surface. When impinge on the surface, the PEs are split into two parts, some of which are backscattered elastically (backscattered electrons, BSE) and the others penetrate the surface. A large part of the penetrating electrons interact with the material to produce true-secondary electrons (true-SE), while the others are reflected back out (rediffused electrons, RE). Each collision cause further interactions or scatterings. The collision will cascade through the material ending with the ejection of electrons (including BSE, true-SE and RE) from the surface.

The SE yield is defined by $\sigma = \mathrm{N_{se}}/\mathrm{N_{pe}}$, where the $\mathrm{N_{pe}}$ is the incident electrons number and the $\mathrm{N_{se}}$ is the secondary electrons number, i.e. the electrons emitted from the surface. The secondary electrons are composed of true-SEs, BSEs and a small or even a negligible amount of REs. We choose conventionally the emitted electrons with energies below 50 eV to be true-secondary electrons [13,14] and the ones with energies above 50 eV to be backscattered electrons (see Fig. 2). Although in some early work, the energy for true-SEs is 0–30 eV or even 0–100 eV. This is maybe another reason of the discrepancies between varies true-SE yield values.

The picoammeter (Keithley 6487) is used for current measurements. For all cases, the primary electron beam current was held at or below 1 nA, which was necessary to prevent inadvertent surface conditioning [15]. Measuring the primary electron beam current with the Faraday cup is a more accurate way. Bias on the sample of −20 V is needed during the sample current and the SE spectroscopy, preventing tertiary electrons from the chamber retuning to the sample [16]. With a negative bias of −20 eV, the electrons ejected from the surface are repelled. Thus, the sample current I_1 represents $(I_{PE} - I_{tot})$, in which I_{tot} is the total secondary electrons current. With a positive bias of +50 V, all the ture-SEs (with energies lower than 50 eV) are attracted back to the sample and only the BSEs electrons (with energies higher than +50 eV) can escape. The sample current I_2 represents $(I_{PE} - I_{BSE})$. However, in measurements, tertiary electrons from the chamber are attracted towards the sample and may decrease a little the value of BSE yield. Total SE yield σ, BSE yield η and the SE yield δ can be calculated from

$$\sigma = 1 - I_1/I_{PE} \tag{1a}$$

$$\eta = 1 - I_2/I_{PE} \tag{1b}$$

$$\delta = \sigma - \eta = (I_2 - I_1)/I_{PE} \tag{1c}$$

As shown in Fig. 3, $\sigma(E_{PE})$ and $\delta(E_{PE})$ increase at very low $\mathrm{E_{PE}}$ and then decrease with the increase of primary energy. $\sigma(E_{PE})$ is characterized by the parameters: E_1, the first primary electron energy at which σ = 1, $\sigma_{\max}$, the maximum yield, and E_m, the primary electron energy at the maximum yield.

3. Results and discussion

The sample was sputtered with argon ions in several steps. Fig. 4 illustrates the measured SE yield for Au samples, due to the argon ion sputtering at various energies. The energy of E_1 and the total SE yield maximum ($\sigma_{\max}$) for every step sample are given in Fig. 4 (b). The samples were sputtered for 10 min at energies of 0.1 keV to 1.5 keV.

Apparently, increasing E_{ion} can reduce $\delta_{\max}$ and increase E_1. For example, E_1 at normal incidence increases from 44.18 eV for the sample as received to 193.62 eV after 1.5 keV sputtering, while $\delta_{\max}$ decreases from 2.01 for as received to 1.54 after 1.5keV sputtering. Here, the values of $\delta_{\max}$ are from experiment immediately, while the values of E_1 are calculated by linear interpolation process. Note that $\delta_{\max}$ decreases and then increases a little with increasing E_{ion}. The evolutions in $\delta_{\max}$ and E_1 become gentle at higher sputtering energies. True-SE yield curves are compared to that of literature of pure Au at normal incidence from Bronstein [17] and Rothwell [18] (see Fig. 5). These experimental results have been retrieved from the database of Joy [8]. The comparison shows they were in reasonable agreement, and maybe the results of Bronstein and Rothwell were for an intermediate state gold.

The X-ray photo-electron spectroscopy was used to monitor the surface composition and contamination of the samples before and