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ScienceDirect
Applied Surface Science 254 (2008) 2110–2113
applied surface science
www.elsevier.com/locate/apsusc

The effect of contamination of dielectric target surfaces under electron irradiation

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Received 8 June 2007; received in revised form 21 August 2007; accepted 21 August 2007
Available online 31 August 2007

Abstract

The influence of the contamination film formed under the electron bombardment of the sample surface on the conditions of experimental studies using analytical electron-probe apparatus (scanning electron microscopes, X-ray microanalyzers) is considered. The accompanying artifacts, namely the decreased effective value of the secondary electron emission coefficient and the shifted value of the second crossover energy of primary electrons are calculated.

PACS : 79.20.Hx; 29.30.Ep

Keywords: Secondary electron emission; Insulators; Contamination; Second crossover energy point

1. Introduction

In the standard electron-probe analytical apparatus, the sample surface irradiated by the electrons is covered with a hydrocarbon layer. The process of the layer formation consists in a number of consecutive reactions including formation of free radicals under the action of electron irradiation, adsorption of organic molecules and polymerization on the sample surface.

The phenomenon of growing contamination film of carbon compounds (mainly from vapor residues of the vacuum pump oils) on the target surface irradiated by electrons has been known for a long time [1–7]. Condensation of residual hydrocarbons C_nH_m with the subsequent electron-stimulated polymerization under the action of the electron beam occurs in conditions of relatively moderate technological vacuum in analytical electron-probe apparatus ($10^{-5} - 10^{-6}$ Torr). This spurious effect introduces considerable corrections into measurement results, the reported data, however, demonstrate a rather widespread of the estimates of this effect.

The most adverse role is played by the shifted value of the second crossover energy of primary electrons that can be twice as lower in the presence of the film contaminating the surface [8]. As the present study shows, this effect is more pronounced in dielectrics.

2. Estimation of the secondary electrons emission coefficient

Let us estimate the value of the shift in question and the effect of the polymerized film on the secondary electron yield in the ''bulk dielectric substrate-contaminated hydrocarbon layer'' system. The total coefficient of electron emission from the solid medium is equal [9,10] to:

$$\sigma = \delta + \eta \tag{1}$$

where δ is the coefficient of emission of secondary electrons (SEs) generated by both the primary electrons of the beam and the reflected electrons (REs), η is the coefficient of the REs emission.

For homogeneous media the coefficient δ is found from the semi-empirical formula [9,10]:

$$\delta = \frac{BE_0\lambda}{2E_iR_0}\left[1 - \exp\left(\frac{-R_0}{\lambda}\right)\right] \tag{2}$$

where the parameter $B = 0.5 \pm 1.0$ is the constant characterizing the probability of the surface barrier being overcome by the

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doi:10.1016/j.apsusc.2007.08.076

secondary electron before it is released into the vacuum, E_0 is the energy of primary electrons, E_i is the generation energy of secondary electrons (of an electron-hole pair in a dielectric), λ is the average emission depth of secondary electrons moving towards the surface (the distribution of secondary electrons throughout the target is taken to be isotropic). The path depth R_0 is uniquely connected with the energy of primary electrons and the target material characteristics: $R_0 = CE_0^n$, where C is the material parameter, the coefficient n ranges from 1.2 to 2, depending on the accepted model [9]. We used the following semi-empirical relation for δ which take into account the dependence of R_0 on E_0, with relation (2) containing the maximum values of δ_m that are achieved at a certain energy of primary electrons E_m [10]:

$$\delta = 1.31\delta_m\left(\frac{E_0}{E_m}\right)^{-0.8}\left(1-\exp\left[-1.45\left(\frac{E_0}{E_m}\right)^{1.8}\right]\right) \quad (3)$$

where Z, A are the atomic number and atomic weight of the target material, respectively.

The dependence of the coefficient σ on the energy of primary electrons E_0 calculated by formula (3) is presented as an example in Fig. 1 for three dielectrics investigated by us. For comparison this figure also shows the experimental values (denoted by dots, triangles and squares) that were obtained in [11–14] under pulsed irradiation regime satisfying the case of uncharged samples. These results as well as the averaged numerical values of δ_m and E_m used for calculations by Eq. (3) have been reported in these works and are equal, respectively, to: $\delta_m = 6.3$, $E_m = 0.63$, $\eta = 0.18$ for Al_2O_3; $\delta_m = 3.5$, $E_m = 0.53$, $\eta = 0.17$ for SiO_2; $\delta_m = 1.65$, $E_m = 0.6$, $\eta = 0.11$ for PMMA. It follows from the plots presented that the values of the second critical energy E_2, where $\sigma = 1$, must be equal to 1.8 keV for PMMA, 4.4 keV for SiO_2 and 10.1 keV for Al_2O_3. According to the earlier assumptions (see, for example, [9,10]), if the initial energy $E_0 > E_2$, the total number of outgoing electrons is smaller than that of ingoing ones, which is

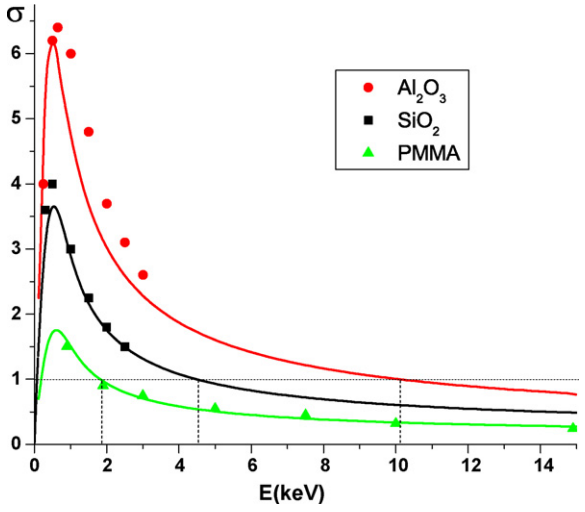

Fig. 1. Dependence of the net electron emission coefficient σ on the irradiating beam energy E for three dielectrics in the absence of their charging and contamination. Solid lines represent the calculated dependences, symbols – the experimental data.

responsible for (with the leakage currents being neglected) the sample as a whole being charged negatively. In the case of E_0 being chosen in the interval $E_1 < E_0 < E_2$, $\sigma > 1$ and the sample must ultimately be charged positively. It should be noted that the above-simplified treatment of the phenomenon of charging dielectrics based on the theory of secondary electron emission (SEE) is inadequate to provide a complete picture of physical processes responsible for negative charging of the dielectric targets by electron beams [14,15]. This fact, however, does not affect significantly the essence of the contamination problem under consideration; therefore the calculations given below are based solely on the SEE theory.

Variations of the SEE effective coefficient δ_{ef} of a composite target consisting of a bulky substrate with the secondary emission coefficient δ_s and a thin hydrocarbon film with the coefficient δ_c depending on the thickness d_c of the film growing during the time t is expressed by the following relation [2,10]:

$$\delta_{ef} = \delta_c + (\delta_s - \delta_c)\exp(-d_C/\lambda_C) \quad (4)$$

where $\lambda_c = 10$ nm is the average depth of SE emerging from the hydrocarbon film [2].

The growth rate of the contamination layer and, hence, its thickness d_c depend on various factors, in particular, on the pressure of the residual oil vapors in the microscope column, the temperature, the current density of the primary electron beam j, the primary electron energy E_0, the time t and the irradiated area [1–3,7].

For a stationary electron probe with the diameter of 1 μm and the current $I_0 = 10^{-9}$ A, the pressure 10^{-5} Torr at room temperature, it can be assumed, with a good approximation confirmed by experiment [2,16], that the film thickness growth rate (at the electron beam current density $j_0 = 0.1$ A/cm^2) is equal to 0.05 nm/s, i.e. the empirical expression for evaluation of the contamination thickness d_c as dependent on the irradiation time t is as follows:

$$d_C[\text{nm}] = 0.05\frac{j}{j_0}t[\text{s}] \quad (5)$$

Then Eq. (4) has the form:

$$\delta_{ef1} = \delta_c + (\delta_s - \delta_c)\exp\left(-0.005\frac{j}{j_0}t\right) \quad (6)$$

Now, the value of δ_{ef} can be adjusted when it is taken into account that a part of secondary electrons are generated by the flow of electrons reflected from the substrate. This part is equal to $\delta_{ef2} = \eta_s\delta_c$. Assuming a partial absorption of electrons in the d_c layer, the net expression for the δ_{ef} contribution, according to [9,10], at $j = j_0$ is written as:

$$\delta_{ef2} = \eta_s\delta_c\left[1-\exp\left(\frac{-0.05t}{0.5R_0}\right)\right] \quad (7)$$

As the contamination film thickness increases, the RE effective coefficient for the target (substrate) materials