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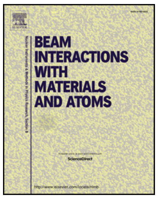

Helium ion beam induced electron emission from insulating silicon nitride films under charging conditions

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ABSTRACT

Secondary electron emission from thin silicon nitride films of different thicknesses on silicon excited by helium ions with energies from 15 to 35 keV was investigated in the helium ion microscope. Secondary electron yield measured with Everhart-Thornley detector decreased with the irradiation time because of the charging of insulating films tending to zero or reaching a non-zero value for relatively thick or thin films, respectively. The finiteness of secondary electron yield value, which was found to be proportional to electronic energy losses of the helium ion in silicon substrate, can be explained by the electron emission excited from the substrate by the helium ions. The method of measurement of secondary electron energy distribution from insulators was suggested, and secondary electron energy distribution from silicon nitride was obtained.

1. Introduction

Electron emission from the surface of solids is widely used for the characterization of bulk materials, thin films and nanostructures by various methods, such as scanning electron microscopy [1], scanning ion microscopy [2], electron-electron and photoelectron spectroscopy [3] and so on. When secondary electrons (SE) are emitted from insulating materials, a charge might accumulate in the sample influencing the emission process and distorting in this way the results of the investigation. The sign and the value of the charge depend on the exciting particle charge, secondary electron yield (SEY) and the excitation time. For the electron excitation it might be negative or positive depending on the SEY value that varies with the primary electron energy providing the possibility to avoid the charging by proper choice of the latter. In contrast to that, the charging caused by positively charged ions is unavoidable and strong being per ion a sum of its own charge and charge of emitted SE, the yield of which might frequently exceed one. Thus, the sample charges positively under positive ion irradiation and secondary electrons are attracted by this positive charge. In that case several techniques of charge compensation developed for other methods described above become not applicable, and a deeper understanding of the physics of charging process is needed to obtain reliable information about the insulating material by using SE detection.

Recently developed focused helium ion beam tool – helium ion microscope (HeIM) [4] uses the secondary electron emission signal for the image formation [5]. Besides imaging, the helium ion beam could be used for high resolution ion beam lithography [6] and ion beam induced deposition [7]. In both latter processes the interaction with SEs play an important role [7,8] and charging of the sample surface might influence the result.

The secondary electron emission in the helium ion microscope was being investigated extensively during last ten years both experimentally [9,10] and theoretically, with the help of numerical simulations [11–18]. The impact of the charging of insulating materials on image formation in scanning ion microscopy was analyzed by means of Monte-Carlo simulation in papers [15–18]. In particular, a possibility of a finite steady state secondary electron emission from thin insulating films on conductive substrates was predicted, but no experimental evidence of that has been presented up to now for the best of our knowledge.

In this paper we present experimental results of the investigation of the charging effects on the SE emission from thin insulating films in the helium ion microscope. The silicon nitride thin film was chosen as the object of the measurements due to its widespread usage in microelectronics and its well-known electronic properties [19–22]. We found that secondary electron signal decreased with the irradiation time because of the charging of insulating films tending to zero or reaching a finite value for relatively thick or thin films, respectively. The finiteness of secondary electron signal value in a case of thin films is in qualitative agreement with the theoretical predictions [17,18]. It was found that finite value of SEY from thin films is proportional to the ion electronic losses in silicon substrate that might be explained by the electron emission from the substrate caused by the helium ions penetrated through the film.

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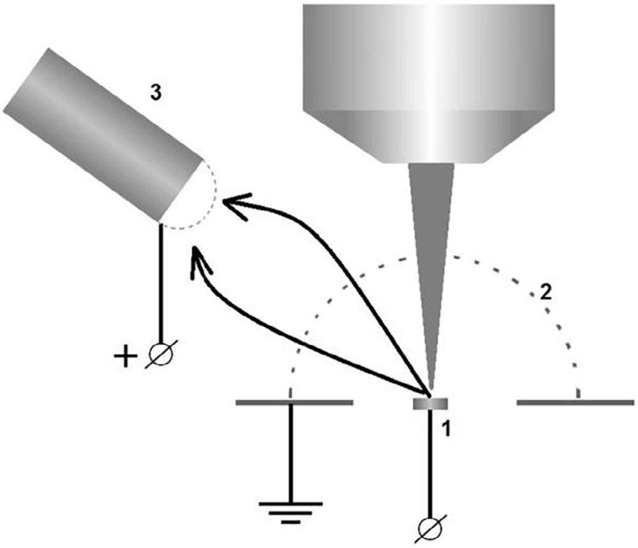

Fig. 1. Measurement set-up.

2. Materials and methods

We investigated silicon nitride films (Si_3N_4) on silicon substrate (p-Si, 4.5 Ohm*cm). 540-nm-thick silicon nitride film was obtained by low-pressure chemical vapor deposition (LPCVD) from silane and ammonia. Films with the thickness of 350 nm, 220 nm and 100 nm were obtained by wet etching of initial film in 49% hydrofluoric acid. All samples were sonicated in acetone and isopropanol and then cleaned with remote oxygen plasma to remove organic contaminations. The backside of the silicon substrate was covered with In-Ga eutectic to produce low-ohmic contact.

Secondary electron emission in the helium ion microscope Zeiss Orion was excited by the focused helium ion beam of a diameter of about 1 nm, with ion energies from 15 to 35 keV and an ion beam current of 0.32 pA in the spot mode. The position of the beam spots was discretely moved from one point to another distant point with the help of microscope scan generator and specimen stage.

The scheme of the secondary electron signal measurement is depicted in Fig. 1. Sample **1** was installed in the center of grounded hemispherical grid **2** of a radius of 9 mm. The SE signal from Everhart-Thornley detector **3** was acquired periodically for 0.5 µs with the time interval of 0.5 µs. The product of the chosen acquisition time and beam current gives the elemental charge value of $1.6 * 10^{-19}$C corresponding, formally, to the impact of a single He^+ ion per 0.5 µs. In fact, as the ion emission from ion source is a stochastic Poisson process the number of ions hitting the sample at every step might be 0, 1 or 2. This, together with the noise of the electronic detection system, resulted in a significant scatter of experimental points around their averaged value. The averaging over the series of independent measurements was applied to improve the signal-to-noise ratio.

Two kinds of SE signal experiments were performed. In the first one the SE signal temporal changes were measured with a single ion charge step during the total time of 0.5 s that corresponded to the dynamic ranges of the impact of 10^6 ions in every point. Every measurement was performed at a new point of the sample separated by a distance of 100 µm from previous one that was established to make negligible the influence of the charge accumulated at previously irradiated points on SE emission. The backside of the silicon substrate was grounded in this experiment.

In the second kind of the experiments SE signal was measured as a function of the sample surface potential to obtain energy distribution of SE electrons. This was done by means of retarding field method described in Refs. [9,10] with the same hemispherical grid electrode (**2** in Fig. 1). The sample substrate was connected with a positive electrode of a bias voltage source whereas hemispherical grid was grounded. SE signal caused, again, by the impact of 1 ± 1 helium ion at every point of 10^4 points 600–700 nm distant from each other at a certain applied bias was acquired and averaged. This guaranteed to avoid the mutual impact of the charges of the irradiated points that appeared when the distance between points was less than 300 nm.

It should be noted that since the retarding bias voltage was applied to the silicon substrate the sample surface potential is generally less than the former one by the value of the voltage drop over the film, the film-substrate interface and the space charge region in semiconducting substrate. However, since the surface-to-grid distance was three orders of magnitude larger than thicknesses of the insulating film and of the space charge region at semiconductor-film interface one can believe that the surface potential coincided well with applied bias voltage.

3. Results

3.1. Kinetics of the SE signal under He ion impact (“single point” experiment)

The dependences of SE signal as a function of the number of helium ions hit in a single point for all investigated Si_3N_4 layers are depicted in the graphs of Fig. 2a–d. The results can be divided into two groups depending on the ion energy and on the film thickness.

SE signal from 100-nm-thick film (see Fig. 2a) decreases with the ion fluence but does not reaches zero to the end of the total time of measurement. It tends to a steady state value at primary ion energies of 15 keV and 20 keV, whereas at the higher ion energies SE signal exhibits a weakly pronounced minimum after an impact of approximately 2–3 * 10^5 ions and then slightly increases. In the case of 220-nm-thick film SE signal decreases to steady state values at the ion energies of 25 keV and 30 keV. At the ion energy of 35 keV SE signal monotonically decreases showing only a decrease of the slope at the last stage of the ion irradiation (see Fig. 2b).

In the case of 220-nm-thick film (Fig. 2b) and energy of primary ions of 15 keV and 20 keV as well as in 350-nm and 540-nm-thick Si_3N_4 films at all ion energies used SE signal decreases more rapidly than for the cases just described above and no SE signal is observed after a fluence of approximately 10^5 ions.

A comparison of all presented in Fig. 2 graphs reveals a general trend that SE yield increases with the energy of primary helium ions for any film thickness and decreases with the increase of the film thickness up to 350 nm at the same ion energy. The latter can be seen from comparison of Fig. 2a–c where the SE signal from 100 nm-thick film exceeds that from 220-nm-thick film that, in turn, is larger than one from 350-nm-thick film at the same ion energy. At the same time, SE signal kinetics obtained from 350-nm- and 540-nm-thick films are rather close to each other reflecting its independence on the further increase of the film thickness.

3.2. SE emission excited by single ion impact (“single ion” experiment)

Fig. 3 represents the dependence of an averaged SE signal obtained under single ion impact on the retarding bias voltage applied to the sample. One can see that SE signal decreases monotonically with the applied bias reaching 10% of initial zero-bias value at the retarding voltage of about 7 V.

4. Discussion

Let us discuss particular features of the experimentally observed temporal dependences of SE-yield upon the point irradiation of silicon nitride layers of different thicknesses on silicon substrate. The fact that SE signal increased with the energy of primary helium ions independent on the thickness is in agreement with the well-known increase of the SE