

Method for Auger Analyses of Thick Insulators Samples

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Abstract – We introduce a simple method for Auger analysis of thick insulators samples. Its main point is formation, over the probe's surface, of a conductive layer with approximately square orifices. The last are obtained by two consecutive vacuum evaporations (of C, Al, Ag, Au, etc.) as before the first one a few threads with diameter 20-40 μm are put on the probe (approximately parallel), while before the second these threads are removed and new ones perpendicular to the first are put. The performed Auger analyses, of volumetric or thick insulators samples with known composition, prove the method applicability.

Keywords - Auger electron spectroscopy, AES, thick insulators analyses

I. INTRODUCTION

Micro- and nanoelectronics are a main field of application of Auger analyses. The reason for that is the unique opportunity for surface element analysis, enriched with depth profiling and quantification. Unfortunately, the analyzed materials are limited to conductors and semi-conductors. Insulators can be analyzed only through "layer over conductive surface" variant (with layer thickness not exceeding 50-300nm according to the conductivity).

During the electron bombardment of thick insulators samples the "charge-up" phenomenon occurs. It inhibits the analyses which include bombardment. For example it is present in Electron Probe Micro-analysis (EPMA), where the solution is to apply carbon covering with level of thickness lower than the informational EMPA-depth. Yet, for AES this doesn't apply, because it is surface-analytic method and any interference towards the surface is inadmissible (therefore any photolithographic methods are also inapplicable). Reducing of charging in AES is primary accomplished by influencing the total secondary electron emission coefficient, s , or by reduction/ neutralizing/ conducting the charge [1, 2, 3, 4]. The following are suggested methods for the first type: I. Reducing the accelerating voltage of the primary electron beam; II. Increasing the specimen tilt angle near to glancing incidence of the primary beam; III. Rotating the sample with the same goal. To the second type the following methods pertain: Ion bombardment [5] covering the specimen with a grounded metal mesh [1, 6, 7]. Irradiation of the sample with another electron beam (at low acceleration voltage and glancing incidence) [5], reducing the primary electron beam current and the current density and bias the surface with a negative potential affect on both

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types.

Characteristic for all of these methods is that they are not always successful. Furthermore experimental optimization of each analyzed sample is usually required. In addition the commercial apparatuses often don't allow any upgrade interference. Perhaps the metal mesh method is relatively most universal, but it also has limits related to the mesh thickness, the Auger analysis geometry and the surface of the sample.

The method we are using in the current work doesn't have the fore mentioned flaws. Furthermore it is universal and can be applied with ease.

II. EXPERIMENTAL

Base setting of the suggested method

A few approximately parallel well-cleaned threads with diameter 10-40 μm are put on the surface of the sample, which will be analyzed. Their material is of no relevance as long as it can withstand the next vacuum evaporation; the last is also in effect for the way they are attached. A conducting material is evaporated (C, Al, Ag, Au, etc.) as the surface thickness is about 200nm. Now the threads are removed. New threads are placed on the evaporated surface, relatively perpendicular to the direction of the first ones. With the same approach material evaporation is performed. After the threads are removed, the sample is metalized, except a few areas with relatively square shape. Then it is put for analysis, as the metalized surface is grounded. The analysis is performed in the "squares". Their small size (side length around the diameter of the used threads) allows smooth leakage of the primary electron beam current – there is not "charge-up".

Particularly in our experimental demonstrations of the method, we used 40 μm Al bonding' wire (we experimented with an organic fiber also) and evaporated $2 \times 170 \text{ nm}$ Au, forming 12 squares.

Auger analyses

The Auger microprobe used has an energy resolution $\Delta E/E \sim 0.3 \%$ and a beam energy E_p up to 10 keV. The analytic regime was E_p 3 keV and a modulation voltage V_{mod} 4 V_{pp} . The spectra are monitored in a differential mode.

In the next part of the work experimental proofs of the method applicability are presented. For this purpose a few typical insulator materials which can't be analyzed through standard Auger methods were selected. Volumetric or thick layer samples are prepared with the fore mentioned method, and are Auger analyzed after. The obtained Auger spectra demonstrate the applicability of this strategy. We

are presenting them in the next part of the article together with a brief comment.

III. RESULTS AND DISCUSSION

Above all we underline that there were no problems during all the analyses and no sample “charge-up” was present at any time.

1. *K-glass analysis.*

The analyzed glass is used as substrate for laboratory research of technological processes etc. In the observed spectrum in Figure 1 – as expected – mainly oxidized silicon and oxygen are present, and at lesser extend phosphorus, potassium and calcium. In order to show the applicability for surface analyses, the spectrum shown here is without preliminary ion milling. Therefore there is significant carbon pollution, some amounts of Mo and significant noise.

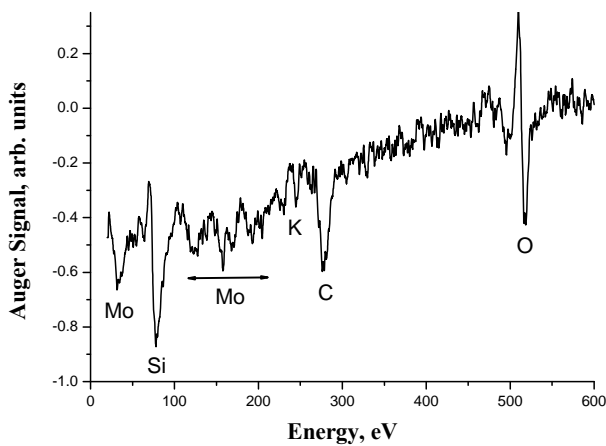


Fig. 1. Auger spectrum of the K-glass surface without ion milling.

2. *Analysis of metal ceramic (sital).*

The main application of sitals in microelectronics acts like a substrate for Hybrid IC and specialized printed circuit boards (PCB) for Surface-mount technology. In the observed spectrum in Figure 2 mainly oxidized silicon and

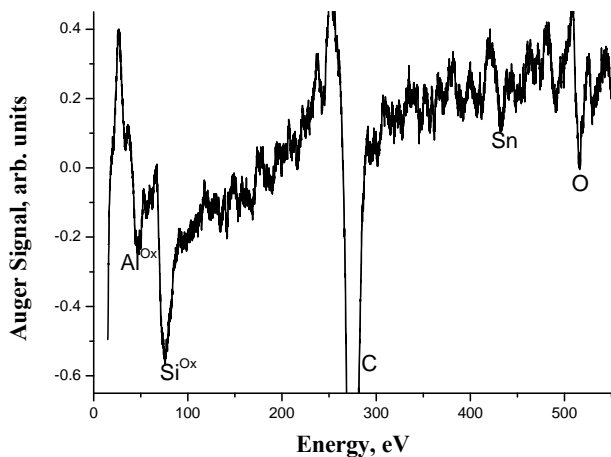


Fig. 2. Auger spectrum of the Sital surface without ion milling.

aluminum and oxygen are present. Since a failure ICs is analyzed in the spectrum is present also a tin. As in the previous analysis without preliminary ion milling there is significant carbon pollution and noise.

3. *Analysis of polyethylene terephthalate.*

The polyethylene terephthalate, $C_{10}H_8O_4$, is used in electronics in the flexible display manufacturing. Often it is commercially available with deposited layer of ITO (Indium tin oxide) on it, as important semiconductor for OLED technology. In AES of the organic it should be noted, that hydrogen isn't Auger identifiable. In the spectrum of Figure 3 there is only C and O. Rough estimation with our laboratory coefficient of element sensitivity gives 79 at.% C, therefore there is around 10 % C-surface pollution. (Actually it is lower, if its shielding effect over the lower layer is taken in consideration.)

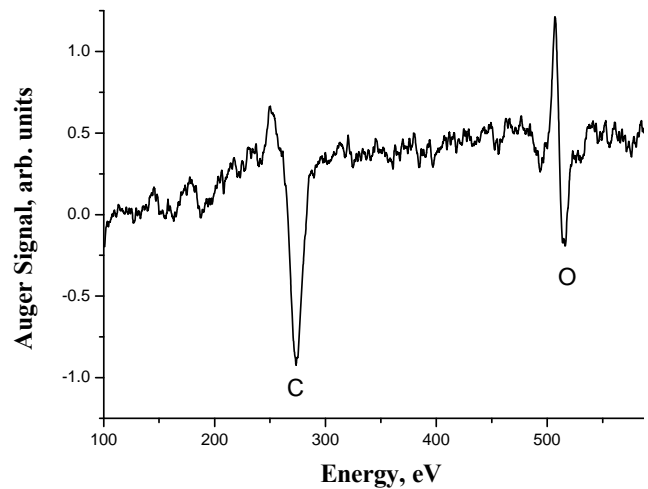


Fig. 3. Auger spectrum of the polyethylene terephthalate surface without ion milling.

4. *Analysis of PVC.*

We analyze PVC (polyvinyl-chloride, C_2H_3Cl) because its wide spread as insulation material also in common life. The sample with thickness of ~2mm is cut from volumetric material. Here, as in the fore mentioned comment for H, there is only carbon and chlorine in the spectrum, Figure 4, and the C-surface pollution could be evaluated.

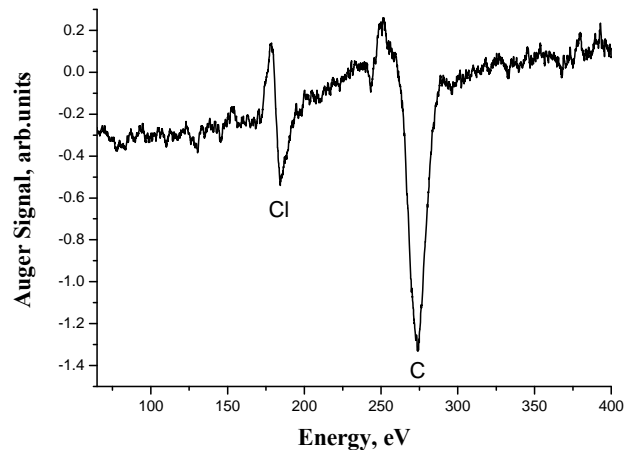


Fig. 4. Auger spectrum of the PVC surface without ion milling.

5. *Analysis of layer As₂S₃ with thickness 0.5 μm on silicon.*

As₂S₃ is promising material used in the manufacture of sensory and ion-selective membranes, in integral optics and photonics [8], as hard photoresist, and as component of more complex chalcogenide glasses [9]. For undoped As₂S₃, deposited on conductive substrate, the “charge-up” begins at layer thickness of 100-130 nm – i.e. the analyzed layer, Figure 6, is certainly insulating. The analysis can be used for determining the stoichiometry – important parameter in the compound synthesis – by the intensities of the S and As (1228 eV) peaks.

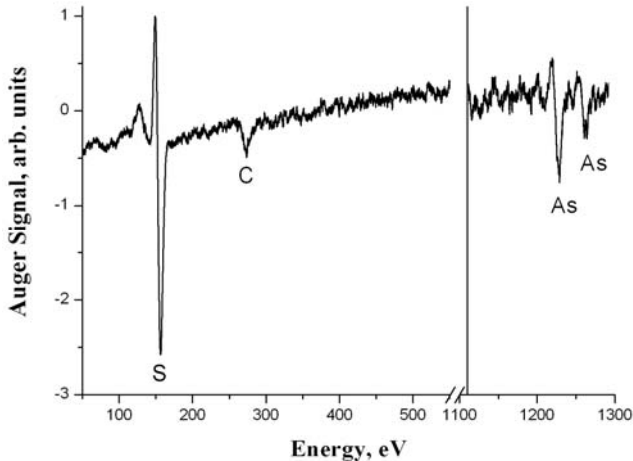


Fig. 5. Auger spectrum of the As₂S₃ surface after an ion milling with 3 kV Ar⁺.

General method comments

The suggested method is universal. We haven't registered any cases of unsuccess when used for analysis of different insulator samples. Common characteristic of the method is the relative ease of use and reliability during its execution. Its main technological operation is vacuum evaporation, which isn't problematic.

The sample is already prepared when it is putting in the analytic equipment (its treatment is performed outside the apparatus) thus removing the necessity for interventions in the analytical combine.

The preparation of the sample with the suggested method is exactly suited for technics for surfaces analyses. With the method the surface of the sample is neither polluted, nor are any changes introduced, provided the used

fibre is well defatted. Another advantage of the method is its applicability is not dependent from the surface form or type.

We want to explicitly underline that the suggested method is also applicable for Auger depth profiling. This is of particular importance, since this analytic variant is essential for the microelectronics.

We also want to highlight an important result for quantitative Auger analyses: The standardizing possibility for elements lacking elemental standards. It is known that in such case, the standards are determined based on analysis of binary, triple, etc. multiple compounds, sometimes minerals – always volumetric insulators.

Everything said shows that the suggested method is of great interest for the analytical practice.

IV. CONCLUSION

1. We suggest a universal method for avoidance of “charge-up” problems, appearing in Auger analysis of massive insulator samples. It is also applicable for other surface analysis methods having analogical problem.

2. The essence of the method is vacuum metallization of the sample surface, obtaining relatively square areas (side up to 40 μm) with unchanged surface, where the analysis is performed.

3. In order to prove the method applicability, 5 analyses of volumetric and thick layer samples from typical insulating material are performed, by applying the method itself.

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