

## THE EFFECT OF DEPOSITION OF METAL CHARGE COMPENSATION COATING ON SURFACE MORFOLOGY OF SAMPLES FOR SEM

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### Abstract

The paper deals with the study of layers applied by magnetic sputtering on samples for electron microscopy to obtain electric conductive surface. It describes the effect of different deposition parameters on the change of the surface morphology of the samples. The reference substrate is pure Si wafer; as the coating material was selected the most commonly used gold and platinum. Scanning Electron Microscopy and Atomic Force Microscopy were used for qualitative and quantitative evaluation of the structural changes.

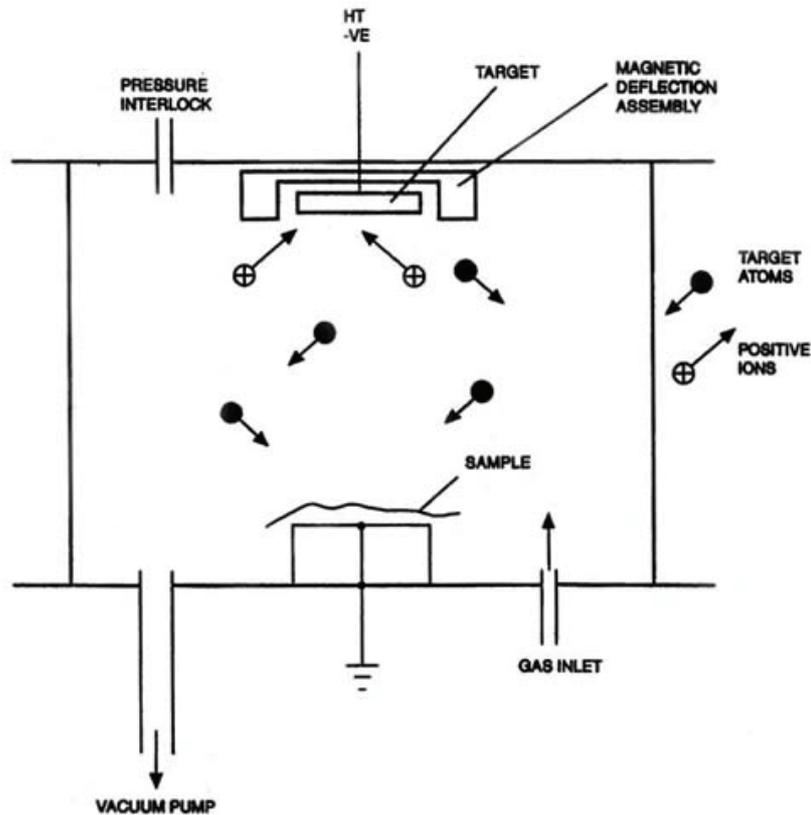
**Keywords:** Scanning electron microscopy, charge compensation, sputter coating

### 1. INTRODUCTION

The basic principle of Scanning Electron Microscopy is the bombardment of specimen's surface by accelerated electrons, which interact with the surface. To avoid static charge buildup on the surface of non-conductive samples and the creation of image artifacts, it is necessary to introduce such procedures, which eliminate the accumulation of the charge, resp. make sample modifications, thanks to which the surface becomes electrically conductive. To reduce or eliminate charging, without any modification the sample, it is also possible to use e.g. reduced current for scanning, resp. lower accelerating voltage. If this does not affect the resolution, the charge accumulation can be reduced by increase of scanning speed. More advanced SEMs are equipped by so called charge-compensator, which locally injects nitrogen to the scanned area, which removes the charge. Where these steps are not applicable, it is necessary to proceed with the treatment of the samples to make them conductive. Samples can be impregnated chemically (biological samples, osmium reactions with tannic acid), coat them using chemical vapor deposition (metal or carbon is heated under high vacuum), or coating by ion sputtering, which is based on a regulated discharge in argon atmosphere by the effect of electrical voltage (during discharge, gas ionization occurs, the resulting ions are attracted to the cathode - target - of coated metal, from which the accelerated ions ejects metal particles - they then cover the surface of the sample - substrate - by thin layer of metal). [1-3]

The following text deals with the method of ionic sputtering, which in practice is the most commonly used method of sample coating. It examines possible influences of the parameters of this coating method on the surface morphology of the studied samples.

The sputter coating is based on the dispersion of the cathode material formed by the coated metal (e.g. gold, platinum or palladium), by ions of the working gas (typically Ar) extrapolated from the glow discharge plasma, which is located by a magnetic field close to the cathode. Plasma electrons are captured in the "tunnel" of magnetic field and drift along the tunnel. This considerably extends the trajectory will increase the number of collisions and create a dense plasma. Positive ions come from plasma to target. The atomized target particles pass through the plasma towards the substrate at which the negative charge is. Scheme of sputter coating see on **Figure 1**. [1,2,4]



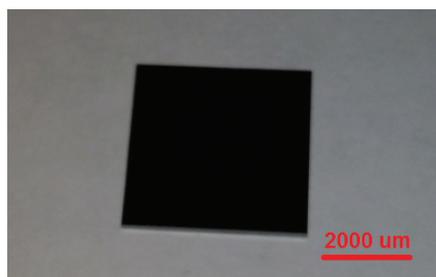
**Figure 1** Scheme of sputter coating [1]

The main advantage of sputter coating is the homogeneous deposition of layers. One of the appreciable disadvantages of sputter coating is, that in the case of extra fine sample surfaces, the coating layer could affect not only chemistry but also the morphology of surface of the samples that we need to examine. The most noticeable effect will be the thickness of the layer (depends on sputtering time), often applied at a thickness of up to 10 nm, and the material (metal) used for coating. Other variables that may affect, even minority, are current, vacuum level and distance from target to substrate. The goal of this paper is to evaluate and describe the morphology of deposited layers with respect to thickness and sputtered material. [1,5,6,7]

## 2. EXPERIMENTAL

### 2.1. Samples - preparation and parameters

As reference samples to study the effect of coating on surface morphology were used identical Si wafers with dimensions approx. 5x5 mm (see **Figure 2**). These samples were thoroughly cleaned with isopropyl alcohol and by the Ar ion beam. Reference samples were scanned without coating; others were sputter coated by layers of gold (Au) and platinum (Pt) at thicknesses of 1, 2, 5 and 10 nm. With 1 nm as the minimum thickness of the coating, requiring the simultaneous use of the procedures for elimination of electrical charge described in chap. 1, thickness 5 nm is standard, by manufacturers of coating devices recommended and default preset layer thickness, 10 nm then the generally reported coating thickness of the samples, in which, however, is expected higher rate influence of surface morphology, and 2 nm is used in practice as the compromise between the thickness of the layer providing sufficient charge compensation and affecting of the surface morphology. Each of the sample variants have been prepared and tested in 3 specimens, in total, 27 samples were used, the parameters of which are shown in **Table 1**.



**Figure 2** Preview of the reference sample - Si wafer

**Table 1** Samples and their parameters

| Sample  | 1a   | 2a      | 3a      | 4a      | 5a      | 6a      | 7a      | 8a       | 9a       |
|---------|------|---------|---------|---------|---------|---------|---------|----------|----------|
|         | 1b   | 2b      | 3b      | 4b      | 5b      | 6b      | 7b      | 8b       | 9b       |
|         | 1c   | 2c      | 3c      | 4c      | 5c      | 6c      | 7c      | 8c       | 9c       |
| Coating | none | Au 1 nm | Pt 1 nm | Au 2 nm | Pt 2 nm | Au 5 nm | Pt 5 nm | Au 10 nm | Pt 10 nm |

For the sample preparation were used ion polisher Fischione SEM Mill and the sputter coating device Quorum Q150R ES with Au and Pt targets. Deposited layer thicknesses have been measured by integrated FTM crystal, the sputtering rate is 2 nm per 1 min of sputtering time. Both devices use Ar gas as the ionization medium.

## 2.2. Experimental methods

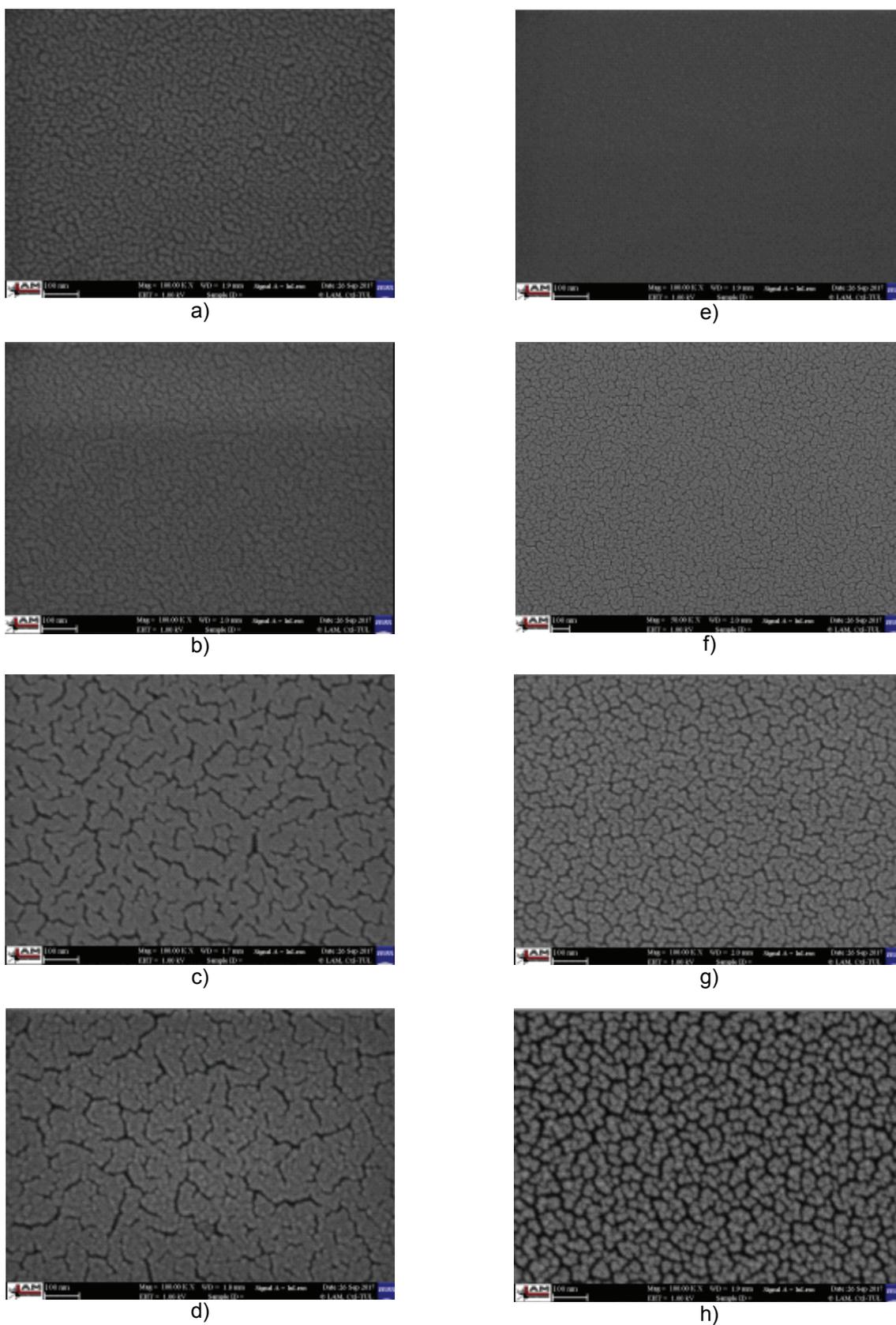
The acquisition of sample surface images was utilized by UHR Scanning Electron Microscope Zeiss Ultra Plus. Images were taken in magnifications of 100 000x and under accelerating voltage of 1 kV in topographical contrast, using secondary electrons (SE), through Inlens Zeiss detector. The data were processed by Smart SEM SW. The surface morphology of the samples was evaluated. Additionally, sample surfaces were scanned with use of Atomic Force Microscope JPK, in Contact mode, with scanned area of 1x1 μm, resolution of 0.98 nm/pixel, with data processing in JPK data Processing and Gwyddeen SW. Cantilever used for scanning was Pointprobe Silicon SPM-Sensor (thickness 2 μm, length 450 μm, width 50 μm, resonance frequency 13 kHz, force constant 0,2 N/m). Besides qualitative evaluation of surface morphology of samples was performed also quantitative evaluation of surface roughness.

## 3. RESULTS AND DISCUSSION

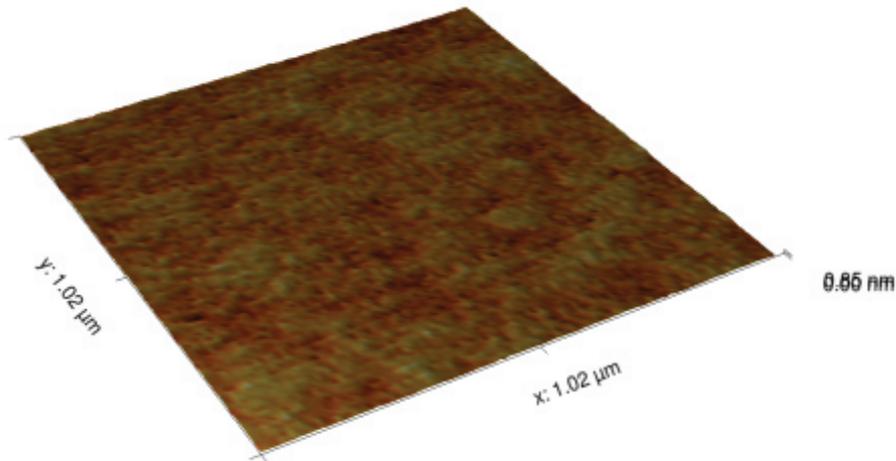
**Figures 3 - 5** illustrate the morphology of the examined surfaces. **Figure 3** shows the sample surfaces, scanned by SEM at a magnification of 100 000x. The left column includes gold coated samples, the right column of the platinum coated samples. Below, in **Figure 4**, is the surface of reference sample without coating, and on **Figure 5** are the selected results with the most significant differences obtained with the AFM. **Table 2** lists surface roughness values and measured mean values of agglomerate sizes for individual materials and layer thicknesses. The values are the average values of the three specimens examined (a, b, c) of the given sample type.

**Table 2** Quantitative parameters of scanned surfaces

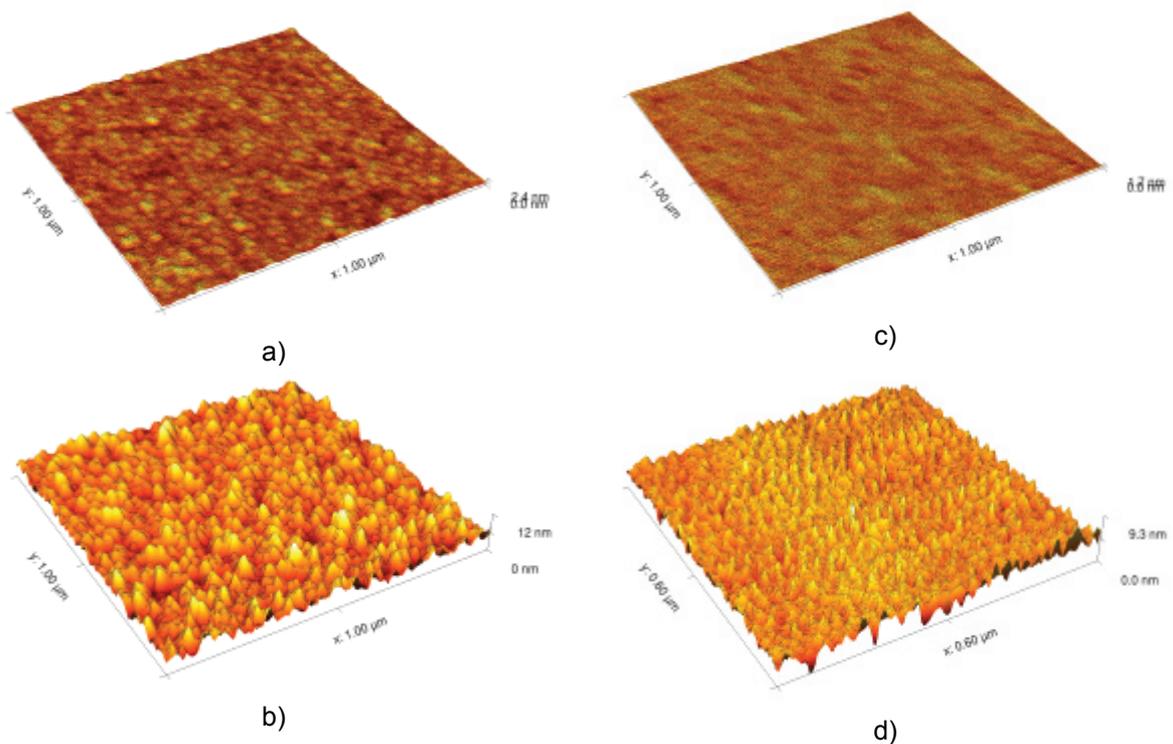
| Sample                                  | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|---|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Ra [nm]                                 | 0.084 | 0.208 | 0.126 | 0.264 | 0.144 | 0.489 | 0.295 | 1.171 | 0.603 |
| Average size of Au/Pt agglomerates [μm] | -     | 0.01  | 0.004 | 0.012 | 0.006 | 0.018 | 0.01  | 0.03  | 0.02  |



**Figure 3** Topography of samples after coating, SEM, InLens, 100 000x magnification; a) Au 1nm, b) Au 2nm, c) Au 5nm, d) Au 10nm, e) Pt 1 nm, f) Pt 2 nm, g) Pt 5 nm, h) Pt 10 nm



**Figure 4** Topography of the reference sample - uncoated Si wafer (sample 1a). AFM, contact mode, scanned area of 1x1  $\mu\text{m}$



**Figure 5** Topography of samples coated by a) 1 nm Au, b) 10 nm Au, c) 1 nm Pt, d) 10 nm Pt. AFM, contact mode, scanned area of 1x1  $\mu\text{m}$

**Figures 3 and 5** and the quantitative results show a considerable increase in agglomerate sizes with increasing layer thickness, also associated with the increase in surface roughness values. The results also differ with respect to the coating material. Au shows significantly larger agglomerates compared to Pt, up to 50% for the same thickness of the layer. In the case of Au, the surface roughness is higher by 150% at 1 nm, by 500% at 5 nm and by up to 1300% (!) at 10 nm over the reference, uncoated sample. In the case of Pt, the surface roughness is higher by 50% at 1 nm, by 250% at 5 nm and by up to 600% at 10 nm over the reference, uncoated sample.

#### 4. CONCLUSION

It is obvious that the increase in the thickness of the coated layer is accompanied by a noticeable increase in the agglomerate size of the coated material on the surface and by a significant roughness of the surface structure whose fine morphology is greatly distorted. This is more noticeable in the case of gold coating; changes are noticeable even at a thickness of 1 nm. The surface roughness Ra of coated samples compared to the uncoated sample was increased from 0.084 nm to 1.171 nm in the case of the 10 nm Au layer, in the case of 10 nm Pt to 0.603 nm. It can be recommended, especially in case of nanomaterials, not to coat the samples and use other methods for charge compensation, or coat them only with minimum possible thicknesses. The further research will be focused on investigation of the influence of other variables during coating, especially the distance of the sample from the target and the coating current.

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