Palacký University Olomouc

Scanning electron microscopy methods in study of micro objects

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OUTLOOK

- INTRODUCTION;
- SEM VS. LIGHT MICROSCOPE;
- CONSTRUCTION OF THE SEM;
- BEAM-SPECIMEN INTERACTIONS;
- EQUIPMENT;
- RESULTS AND DISCUSSIONS;
- CONCLUSION

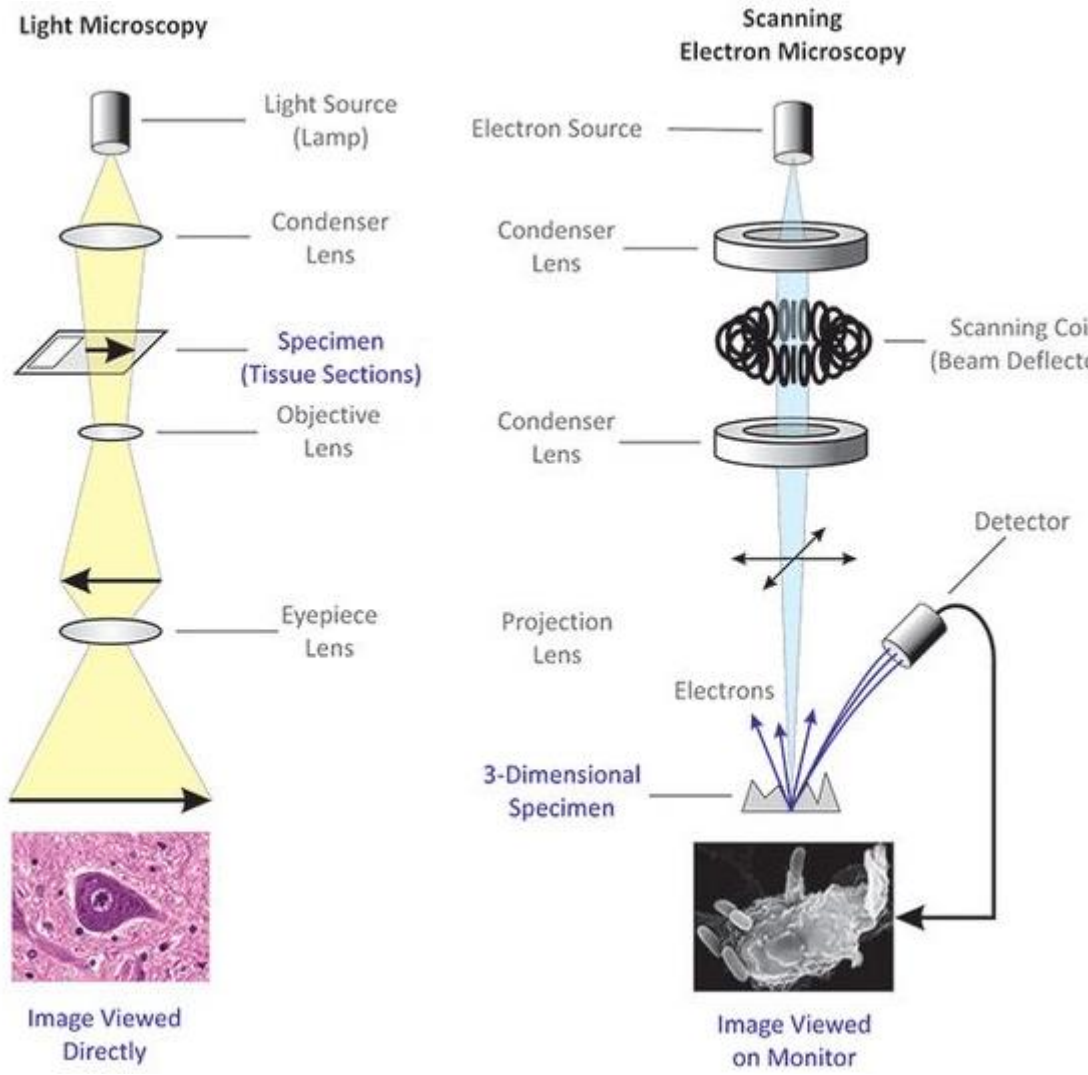
INTRODUCTION

The scanning electron microscope (SEM) is a device that produces a beam of accelerated electrons used to examine objects on a very fine scale.

WHAT CAN WE STUDY IN A SEM?

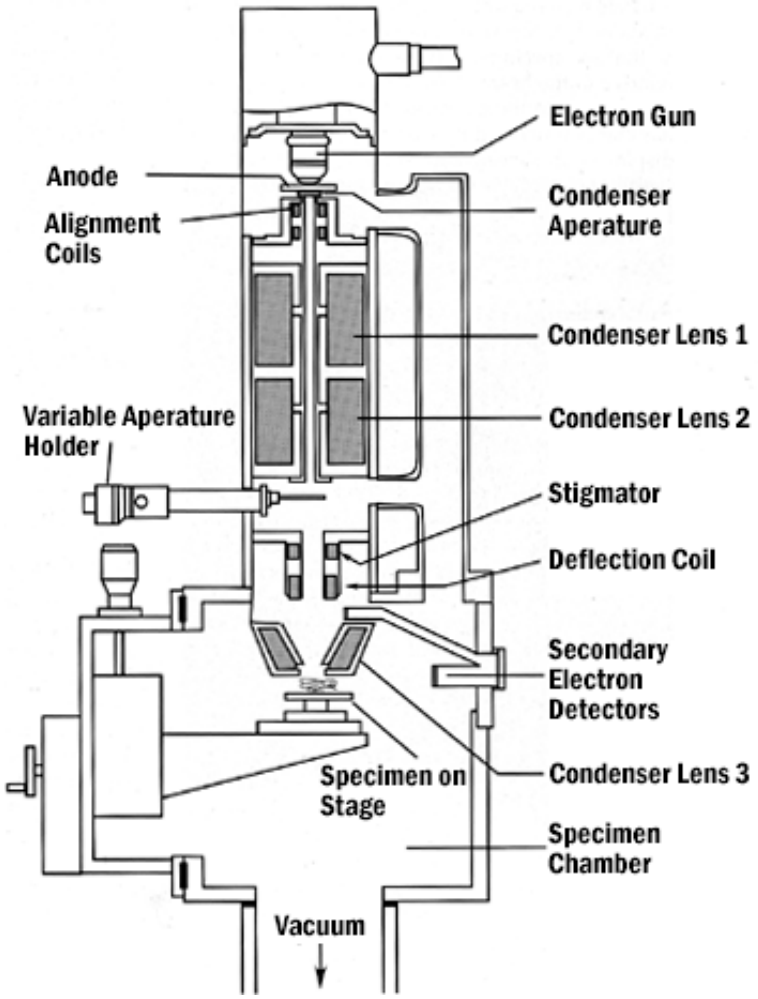
- TOPOGRAPHY
- MORPHOLOGY
- COMPOSITION
- CRYSTALLOGRAPHY
- ELEMENTAL ANALYSIS
- ORIENTATION OF GRAINS

SEM VS. LIGHT MICROSCOPE



Item	SEM	Light Microscope
Illuminating beam	Electron beam (wavelength:0.006-0.087 nm)	Light beam (wavelength: 200 -750 nm)
Medium	Vacuum	Atmosphere
Lens	Electron lens (for probe demagnification)	Optical lens (for enlargement)
Resolution	Secondary electron image: 4.0 nm	visible : 200nm
Depth of field	30µm (at 1000x)	0,1 µm
Magnification	10X to 300 000X (electronically)	10X to 2 000X (lens exchange)
Focusing	Electrical	Mechanical
Contrast	Geometrical shape, physical and chemical properties	Absorbtion and reflection of the light (color and brightness)
Obtainable images	Secondary electron and backscattered electron images	Transmission and reflection images

SCANNING ELECTRON MICROSCOPE



- **electron gun -** located at the top of the column where free electrons are generated by thermoelectric emission from a tungsten filament
- **a system of lenses -** which act to control the diameter of the beam as well as to focus the beam on the specimen;
- **a series of apertures** - which the beam passes through and which affect properties of that beam;
- **controls for specimen position -** (x,y,z-height) and **orientation** (tilt, rotation);
- **an area of beam/specimen interaction -** that generates several types of signals that can be detected and processed to produce an image or spectra;
- **vacuum system**

BEAM-SPECIMEN INTERACTIONS

SIGNALS

- Beam of electrons can interact with atoms of both the specimen nucleus and electrons and can produce a multitude of signal types: backscattered electrons, secondary electrons, X-Rays, Auger electrons, cathodoluminescence.

SECONDARY ELECTRON (SE)

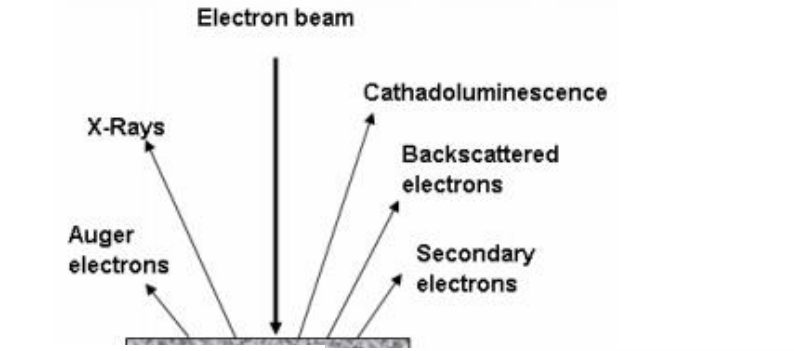
- beam of electrons interacts with the electric field of a specimen atom electron → inelastic events → a transfer of energy to the specimen atom and a potential expulsion of an electron from that atom (**secondary electron SE)**

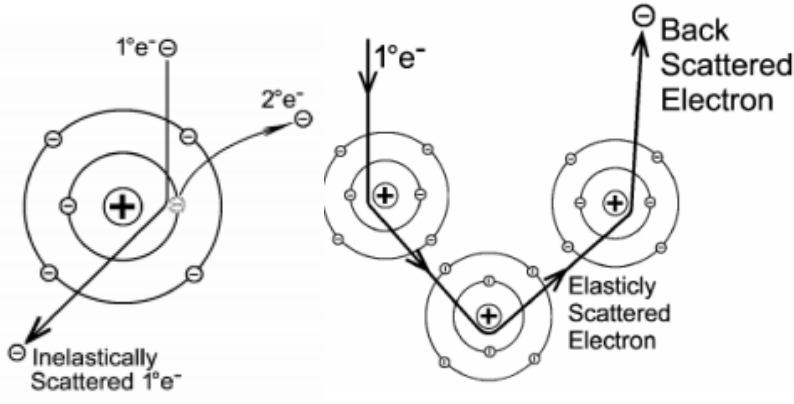
BACKSCATTERED ELECTRON (BSE)

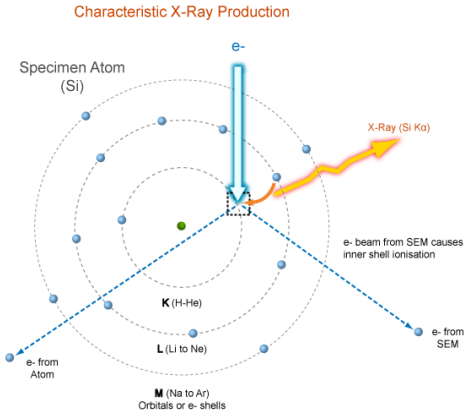
- Beam of electrons interacts with the electric field of a specimen atom → a change in direction of the beam electron → the elastically scattered beam is deflected back out of the specimen → **backscattered electron BSE**

X – RAYS

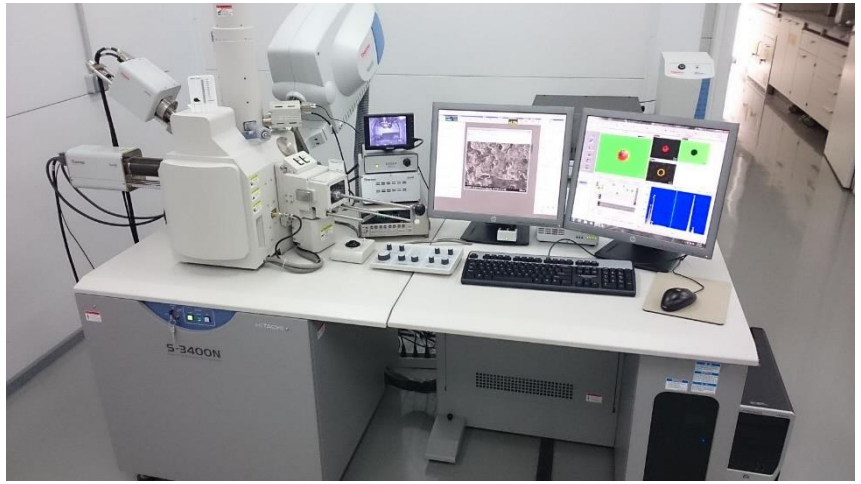
- When a vacancy in an electron shell is filled with electron from higher level a photon with energy characteristic for every element is released. This can be used for an element analysis of the sample.







EQUIPMENT:HITACHI S-3400N



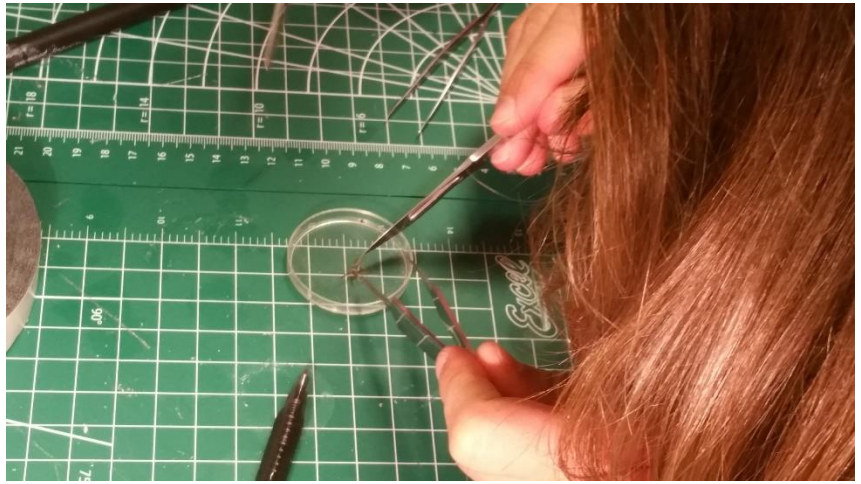
Instrument specifications:

- The Hitachi 3400N VP-SEM is a Scanning Electron Microscope with Tungsten Filament allowing accelerating voltages up to 30kV.
- It is equipped with both Secondary (SE) and Backscatter (BSE) Electron Detectors, energy dispersive spectrometr (EDS) and has a fully eucentric 5 axis motorized stage that allows samples up to 25cm in diameter.

sputter JFS1100

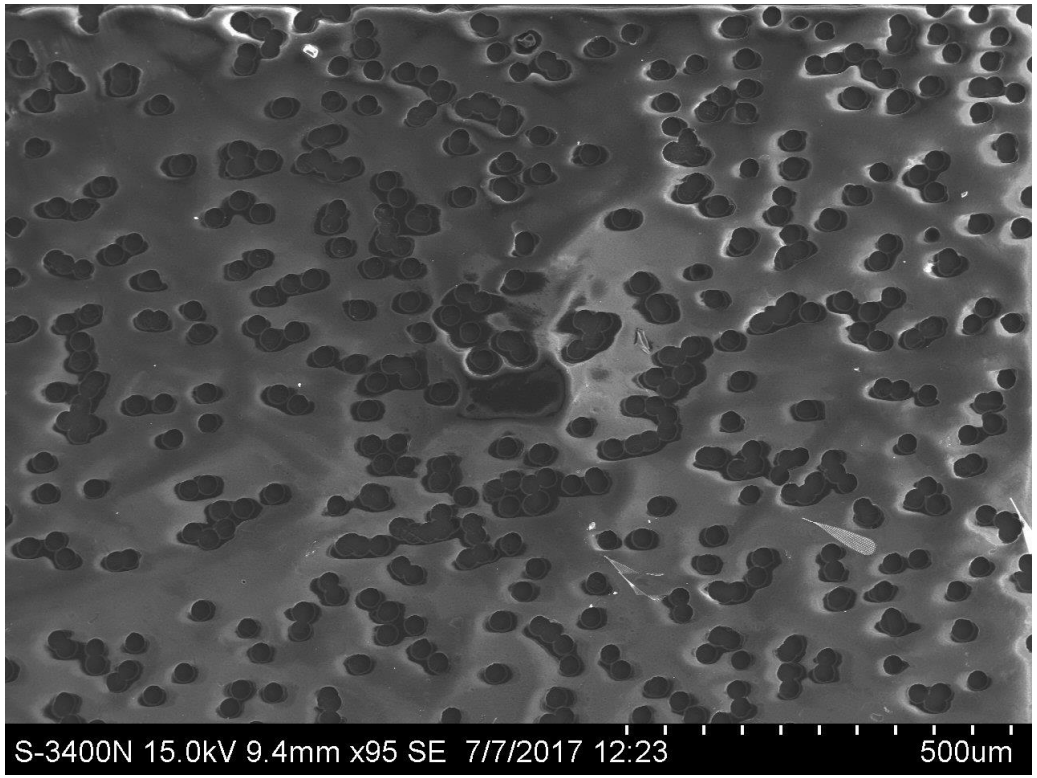
PREPARING THE SAMPLE

- The samples are fixed on the clean specimen stage using silver conductive glue or carbon / copper conducting stickers;
- Biological samples must be dried and fixed with special preparation to prevent shriveling of the samples;
- Non conductive samples must be coated with a conductive layer (Au and Cr in our case) with the aim to increases the secondary emission coefficient and to prevent the target from heating, avoids the charging of the sample;
- Conductive samples can be mounted on the specimen stage without special coating.

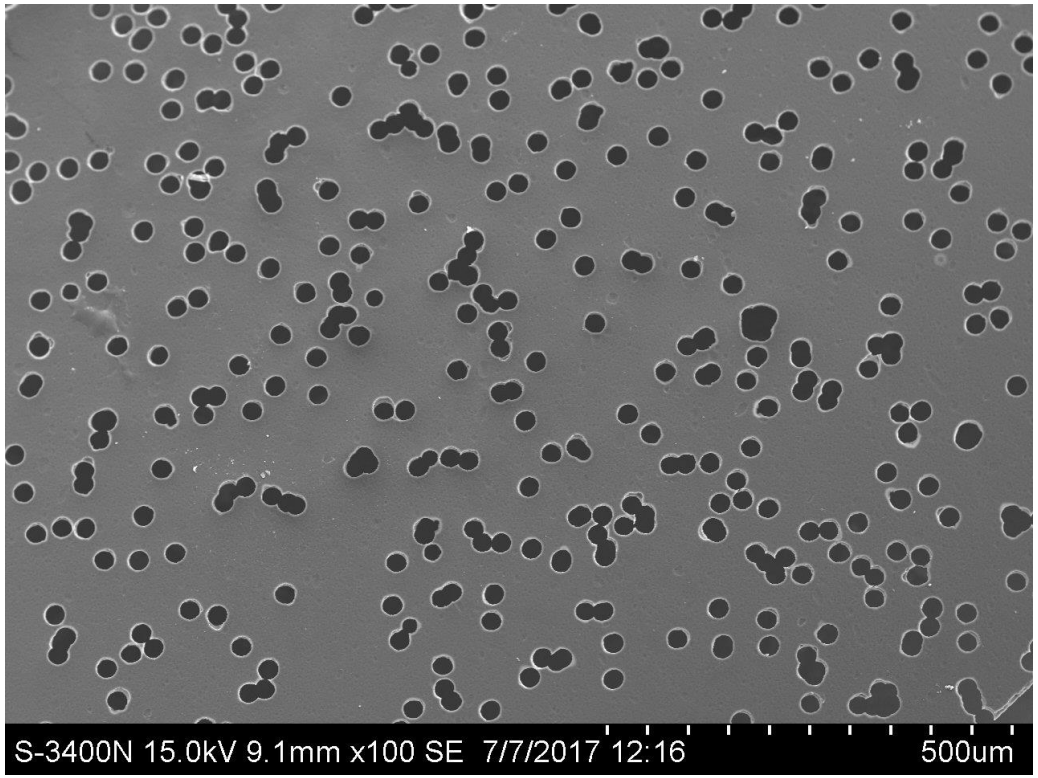


RESULTS AND DISCUSSIONS

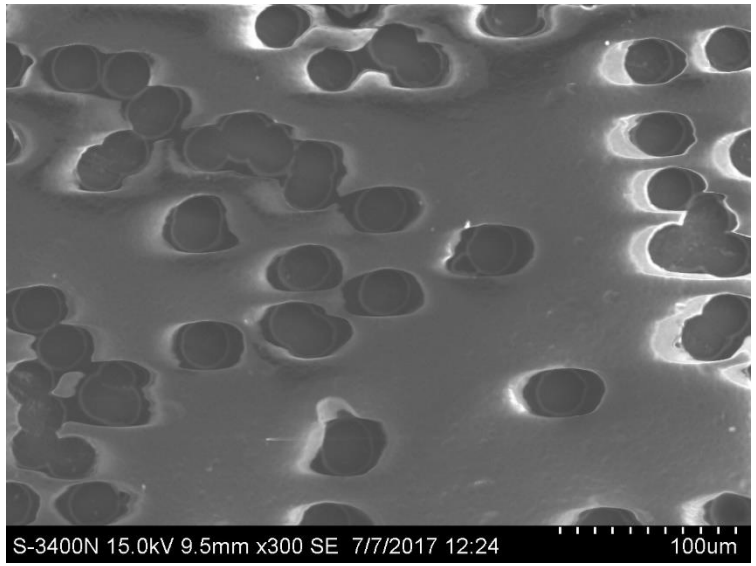
Uncoated sample



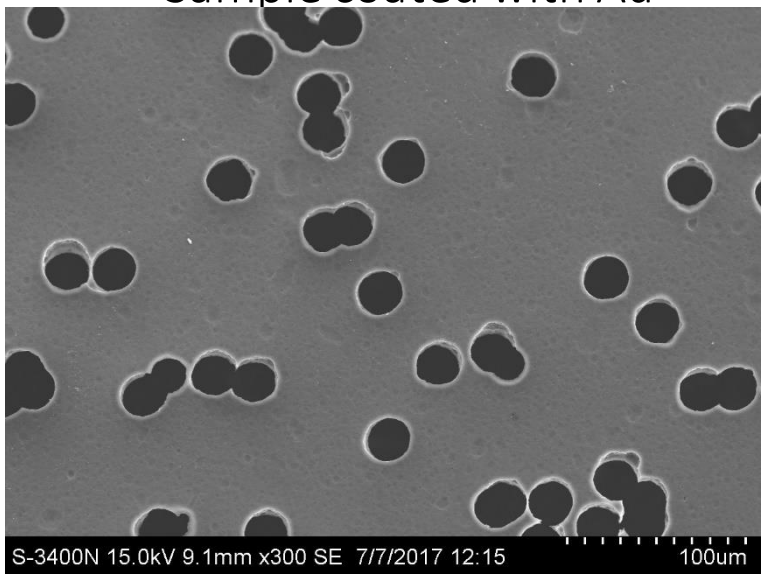
Sample coated with Au

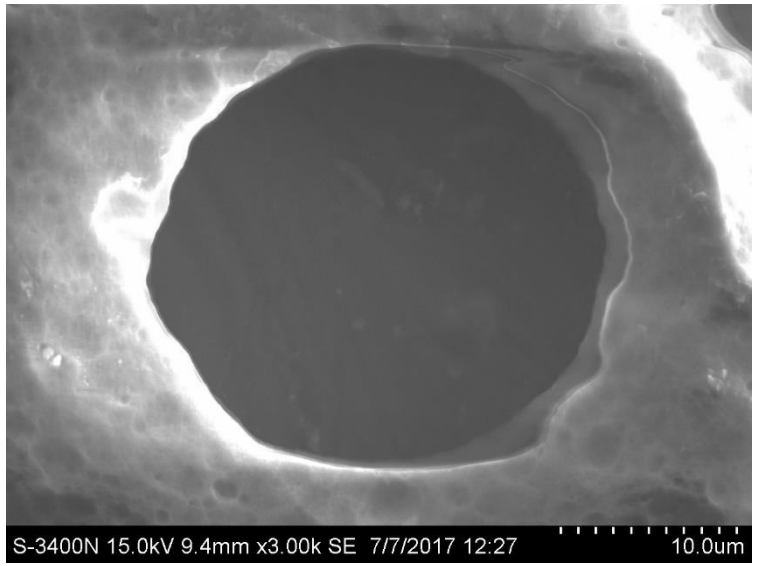


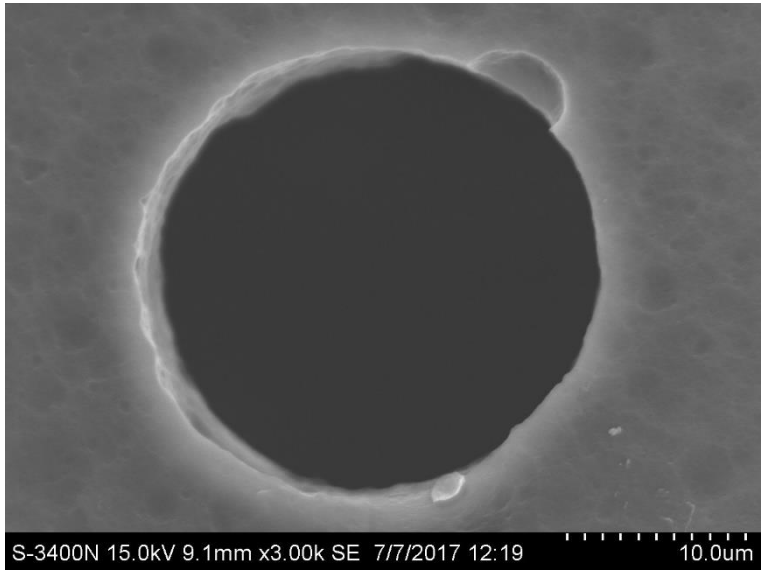
Uncoated sample



Sample coated with Au

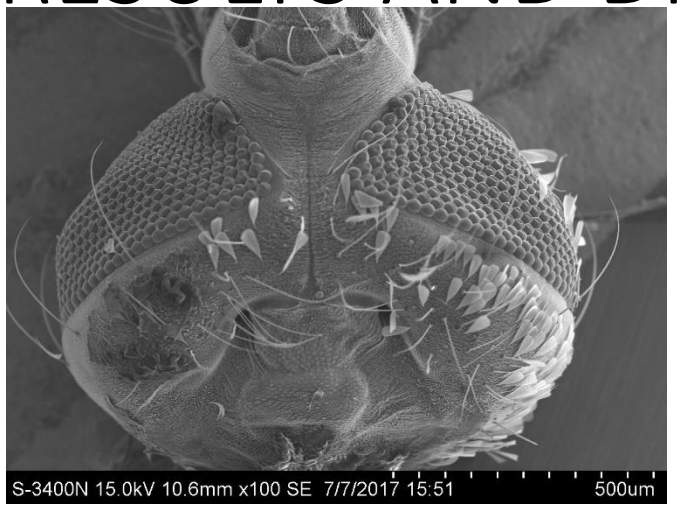






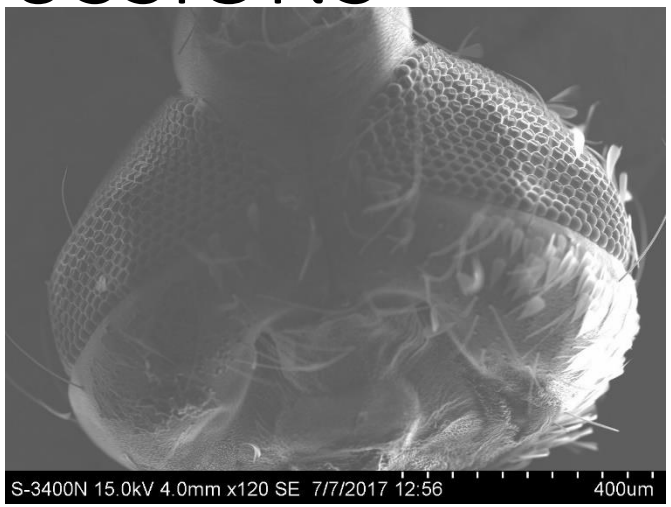
RESULTS AND DISCUSSIONS

A1



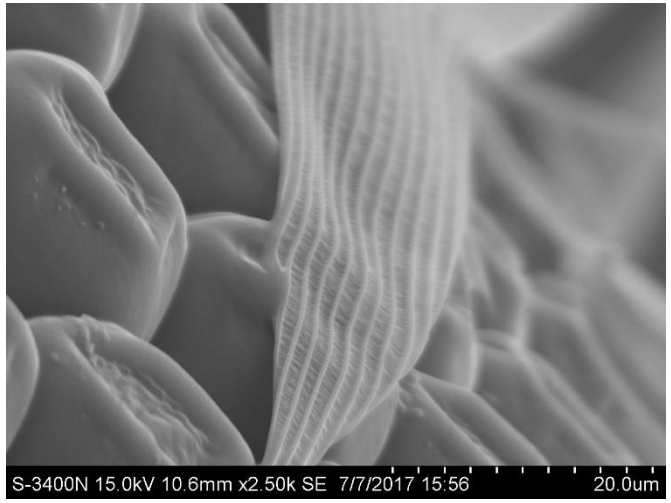
Mosquito head, biological sample coated with a conductive layer (Au) x3

A2

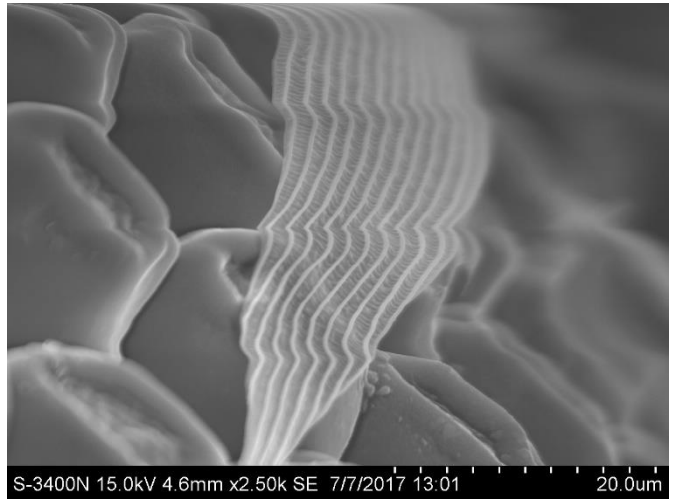


Mosquito head, biological sample coated with a conductive layer (Au) x1

B1



B2



The idea of the coating the specimen is to increase its conductivity in the Scanning Electron Microscope and to prevent the built-up of charge on the specimen.

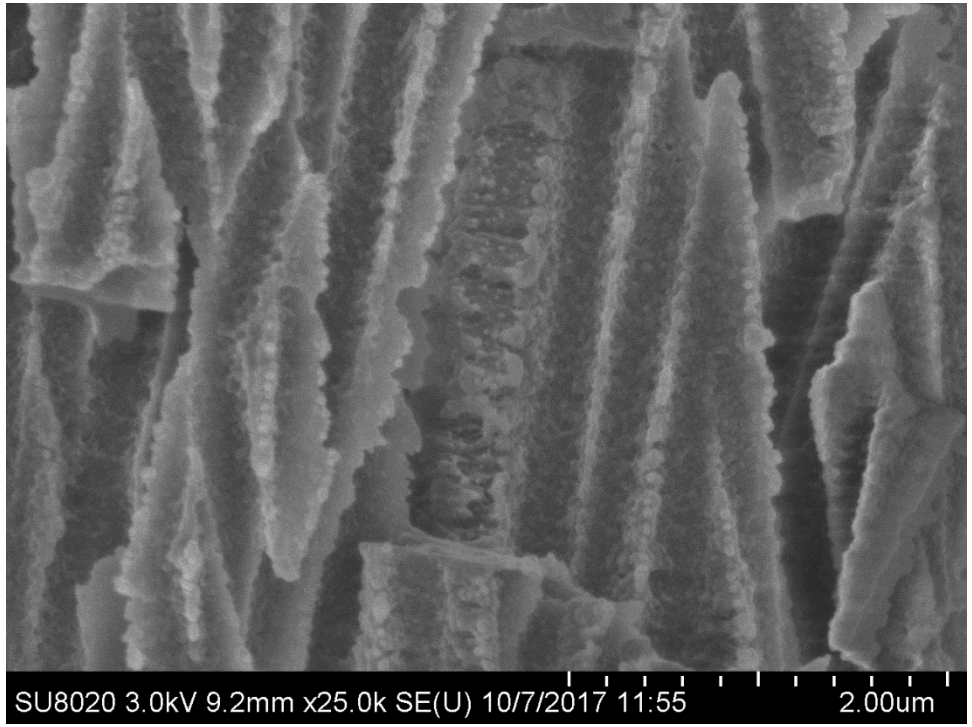
A1 and A2 show the difference of contrast as a result of quality of covering layer.

B1 and B2 show drifting as a result of quality of covering layer.

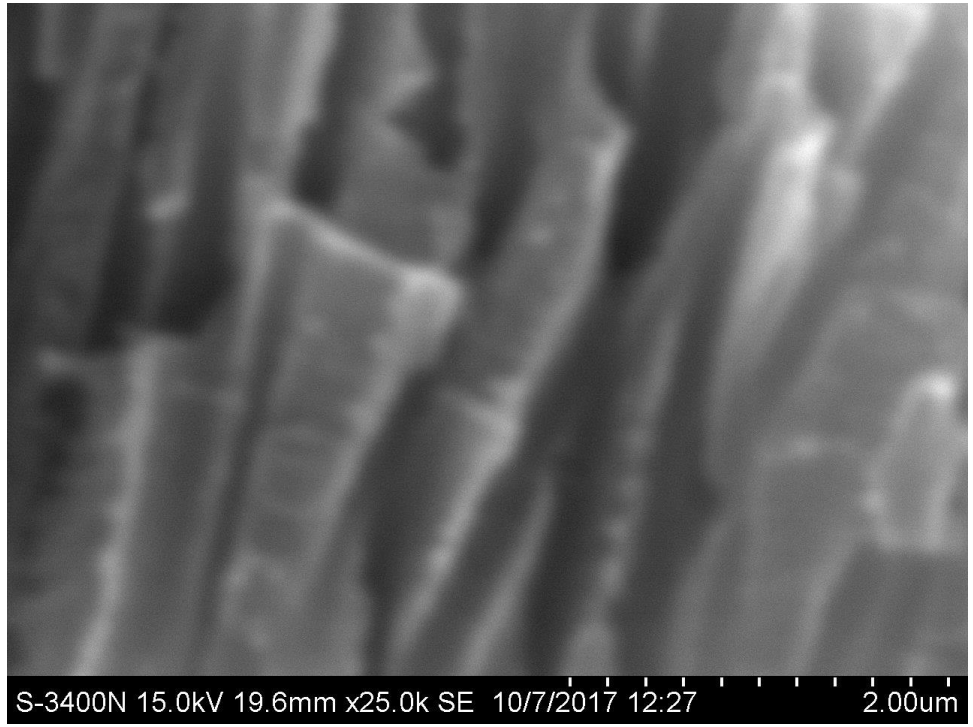
RESULTS AND DISCUSSIONS

Comparison of a different instrumentation.

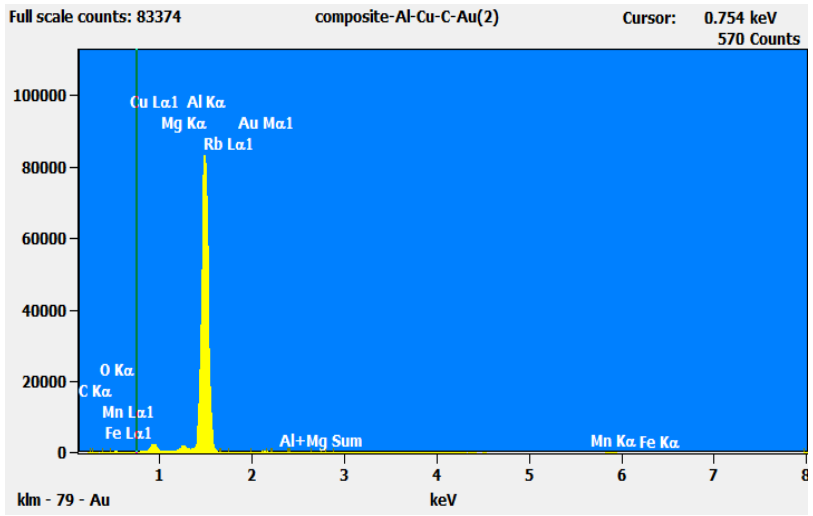
Field emission

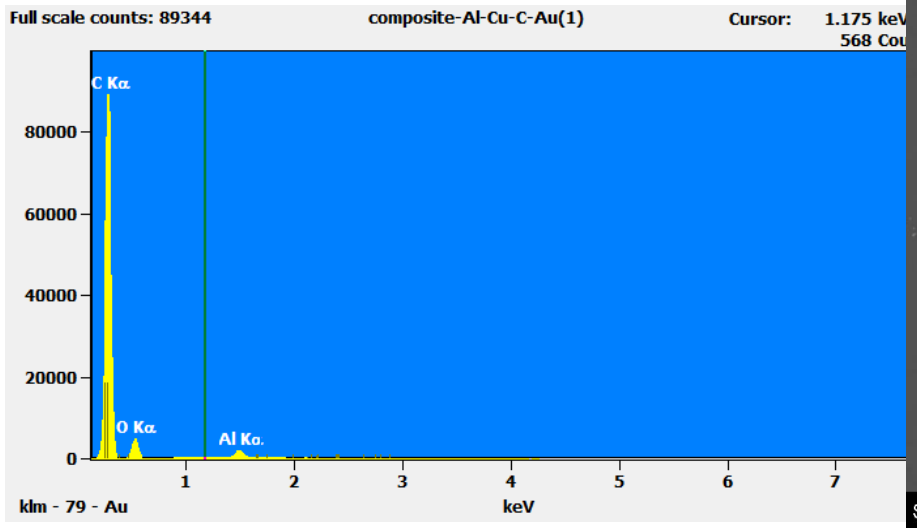


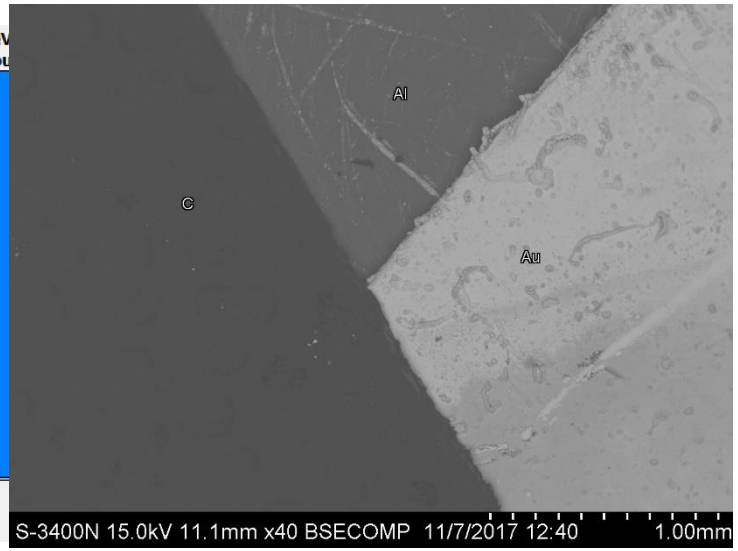
Thermal emission

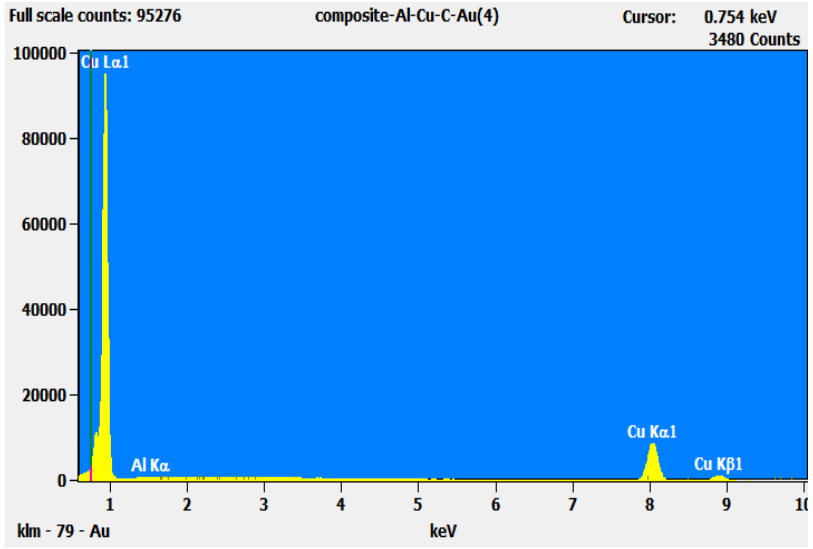


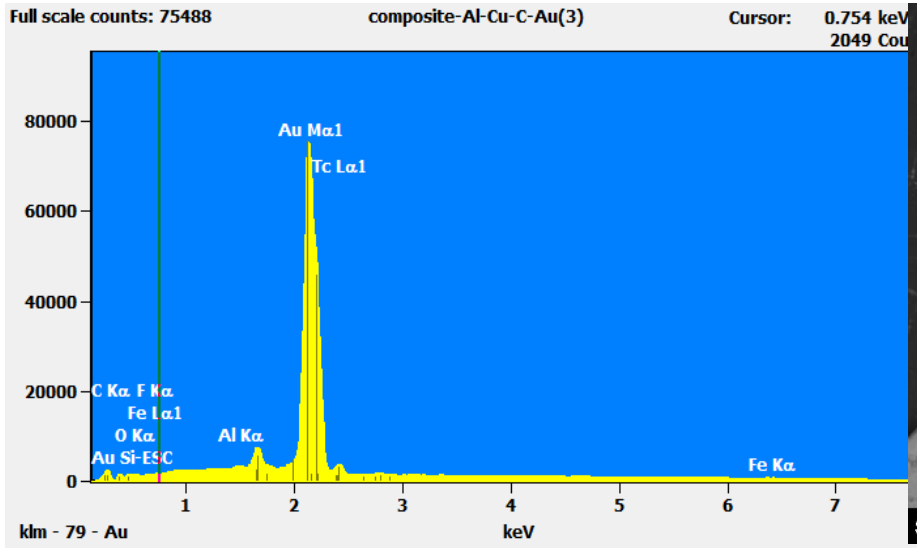
RESULTS AND DISCUSSIONS

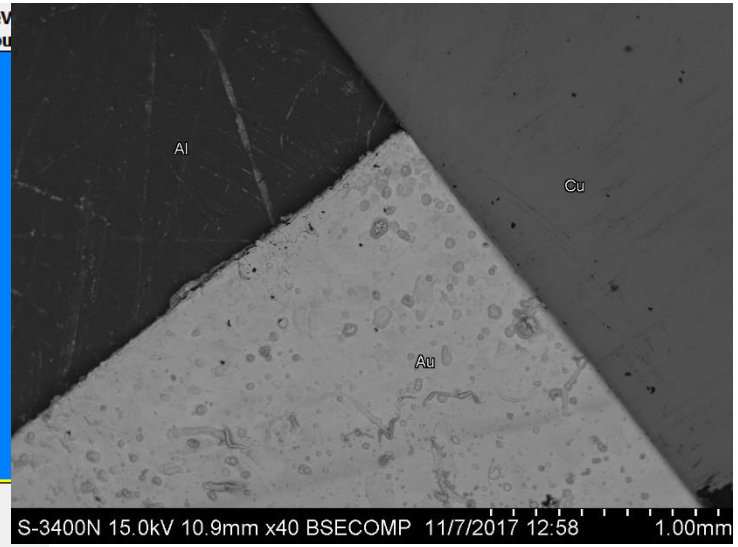








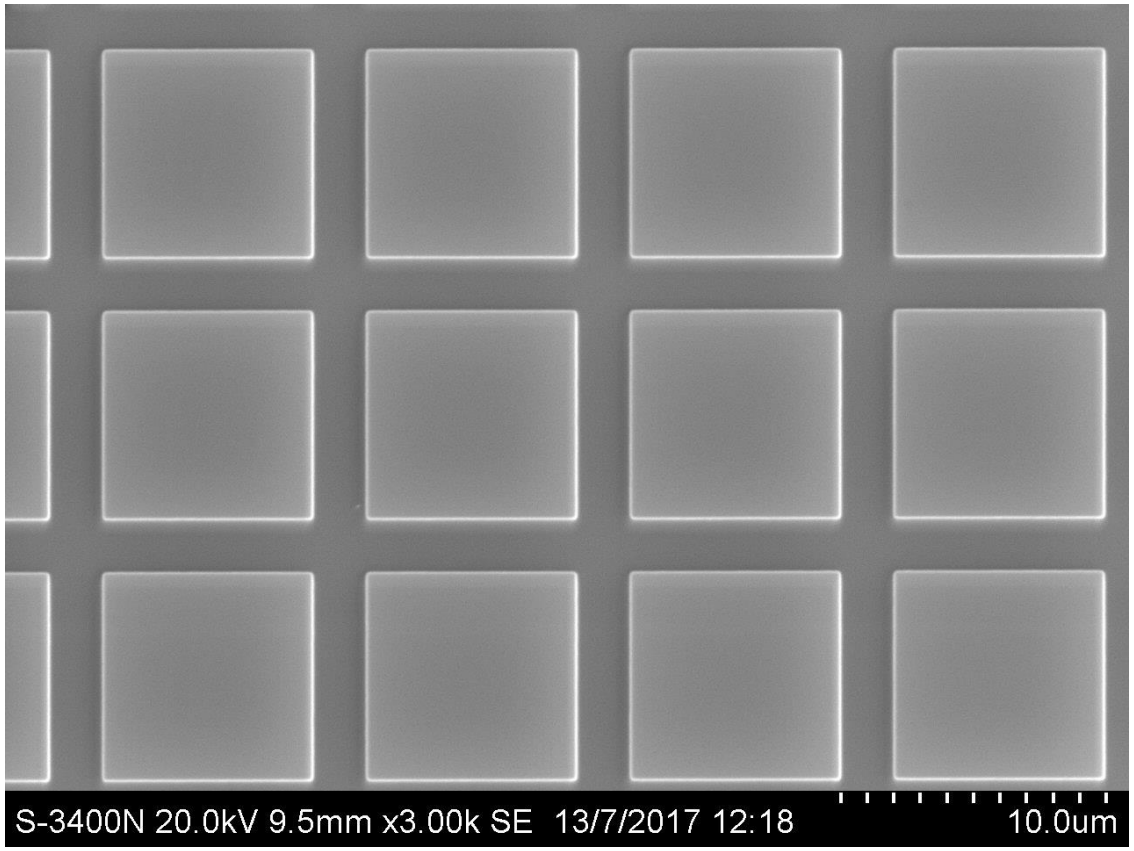


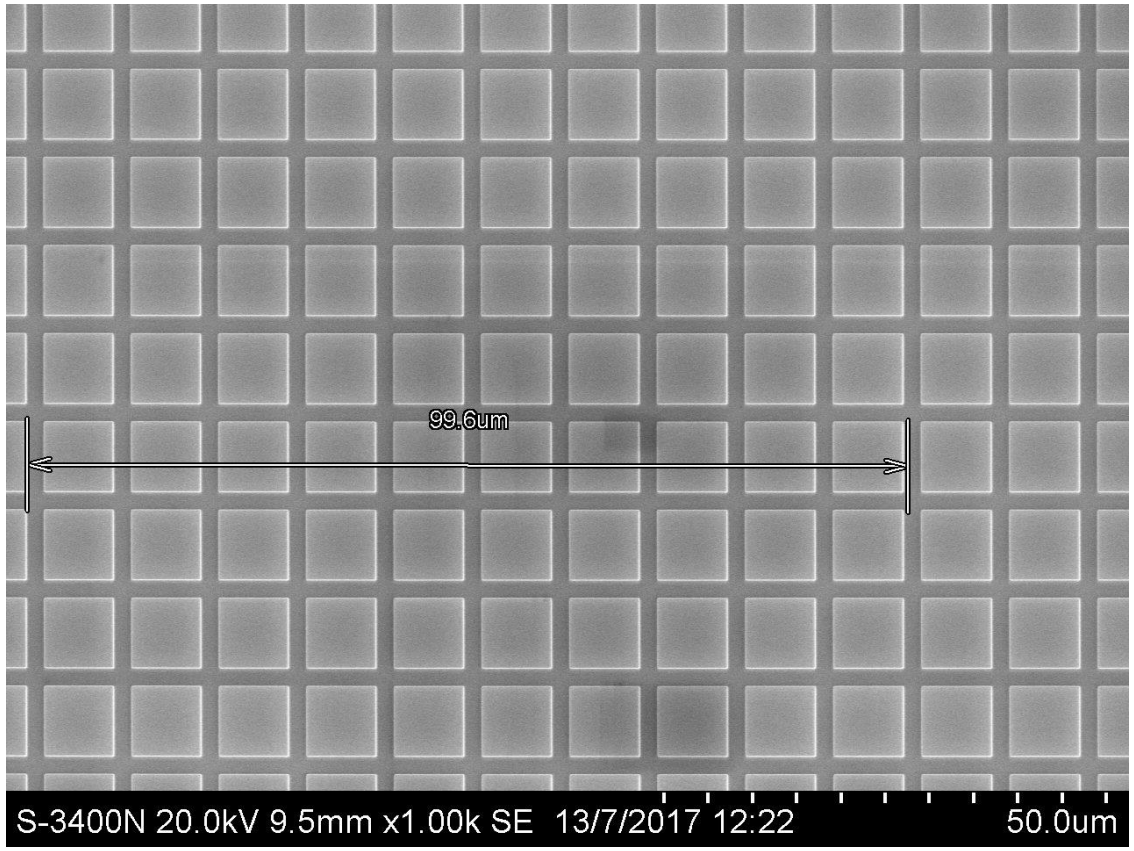


Back scattered electrons, X-Ray analysis of Au, Al, C, Cu.

RESULTS AND DISCUSSIONS

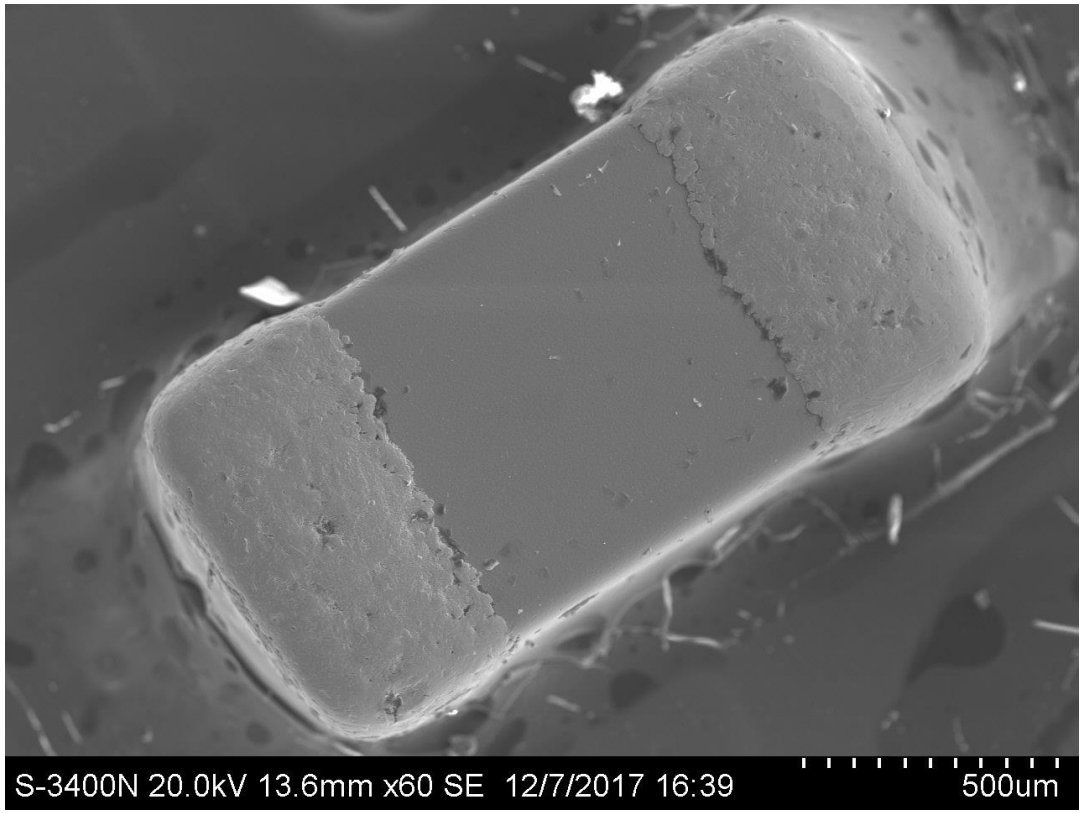
Measuring of parameters of silicon standard for calibration of electron microscope.





RESULTS AND DISCUSSIONS

Element analysis via EDS.



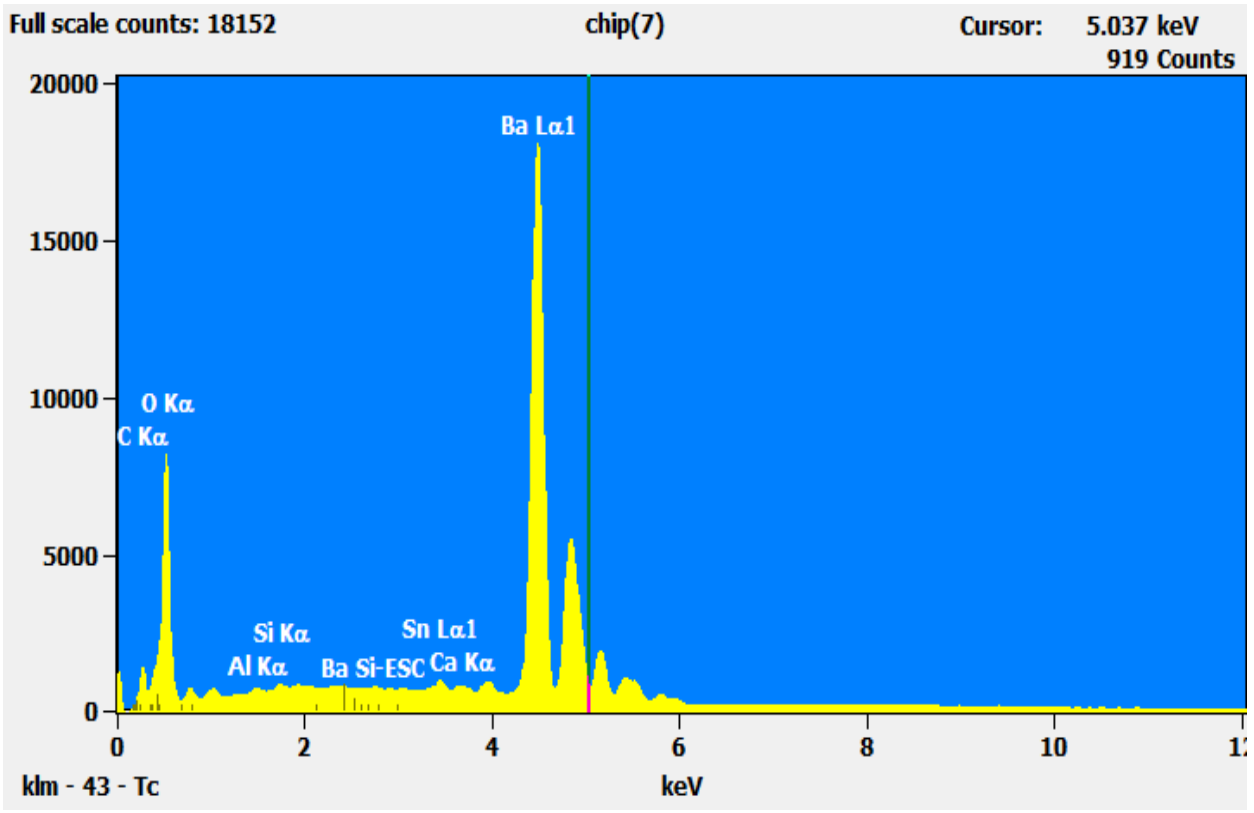
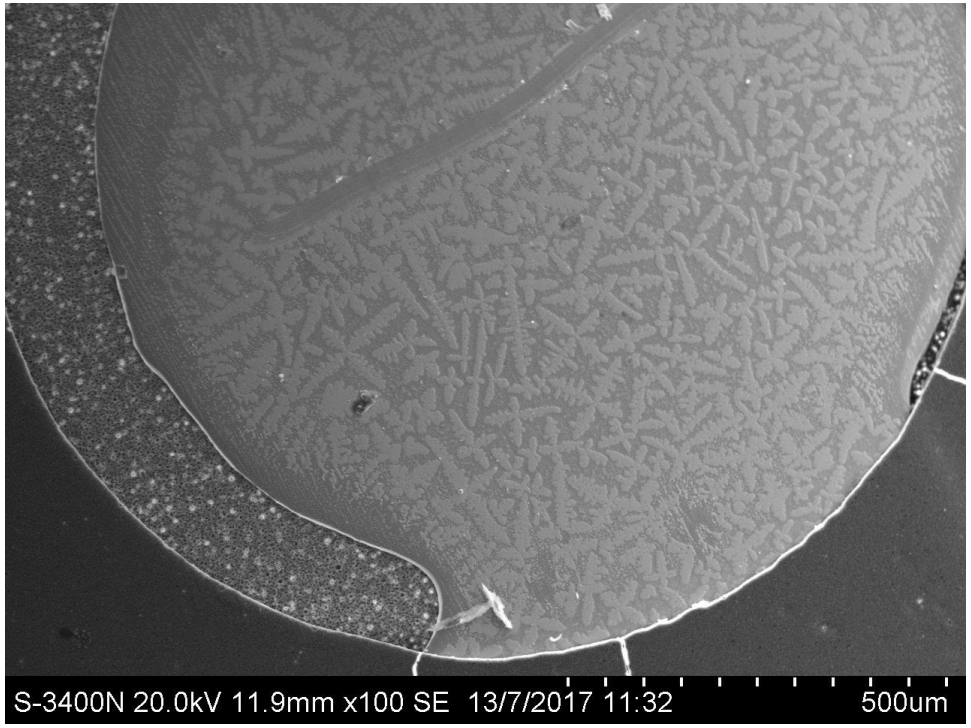


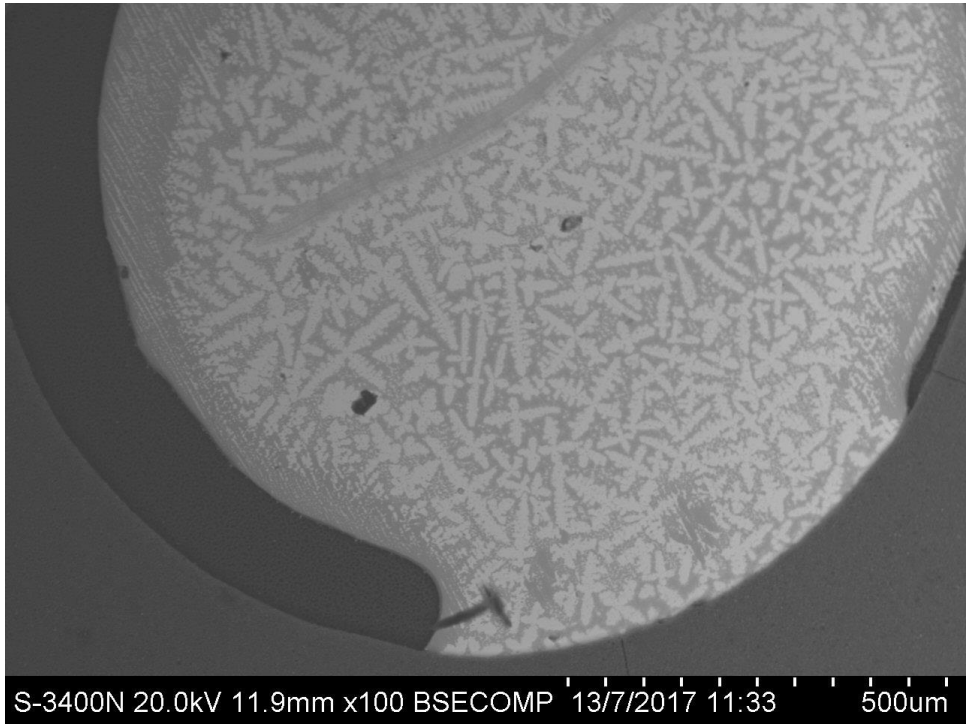
Image and X-Ray spectra of resistor from MP3 player.

RESULTS AND DISCUSSIONS



secondary electrons SE

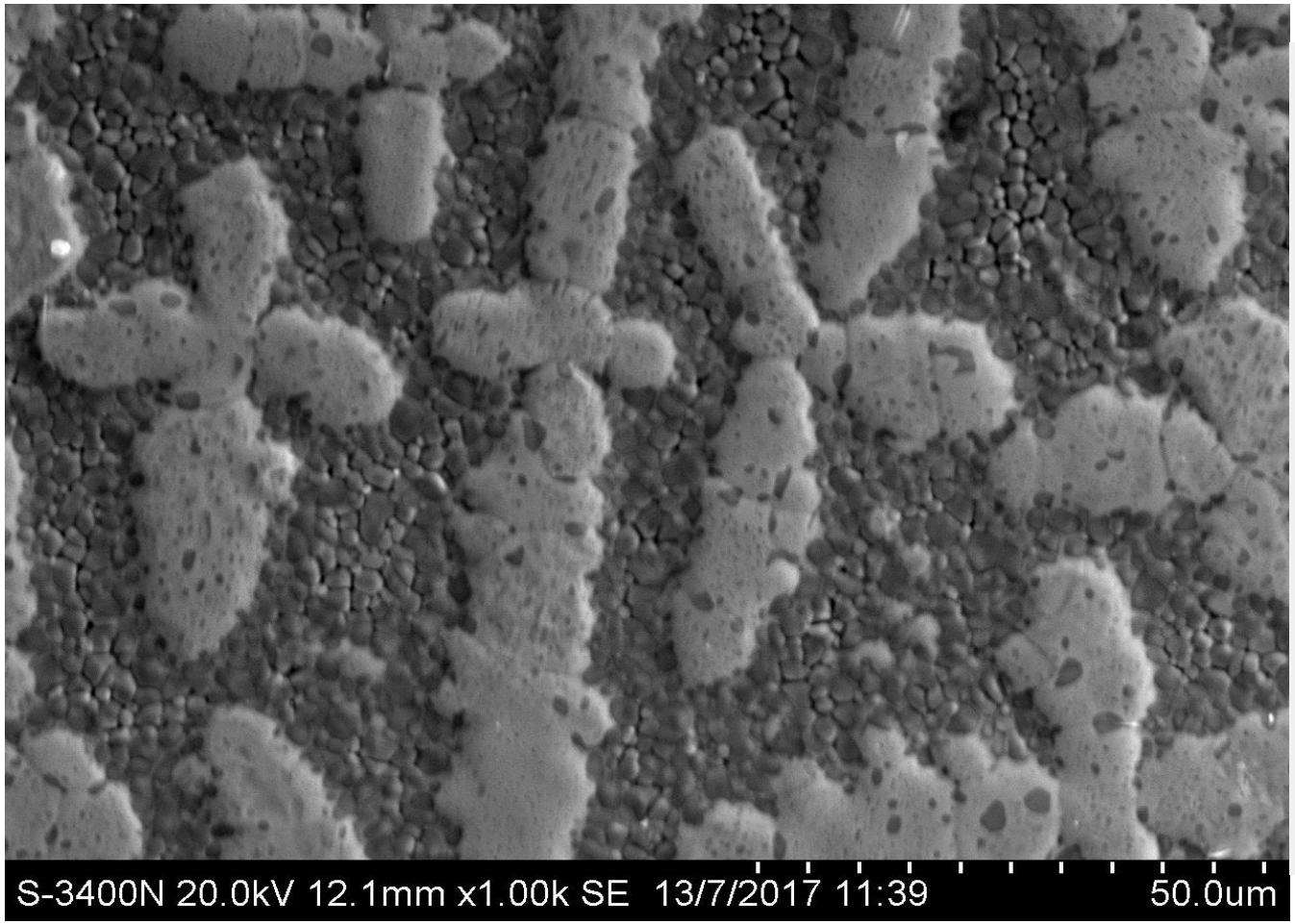
- Suitable for topographical observation.



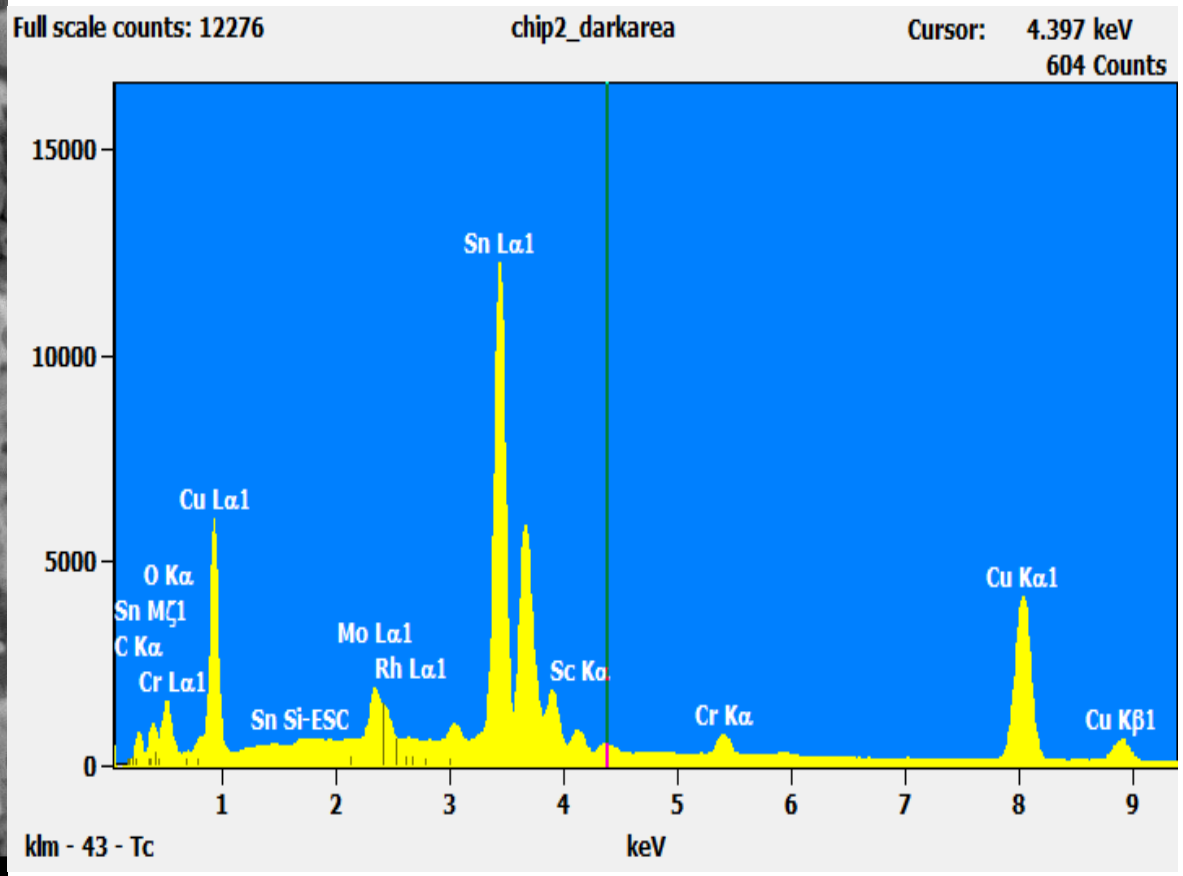
backscattered electrons BSE

- Suitable for contrast of different components (Z-contrast).

RESULTS AND DISCUSSIONS

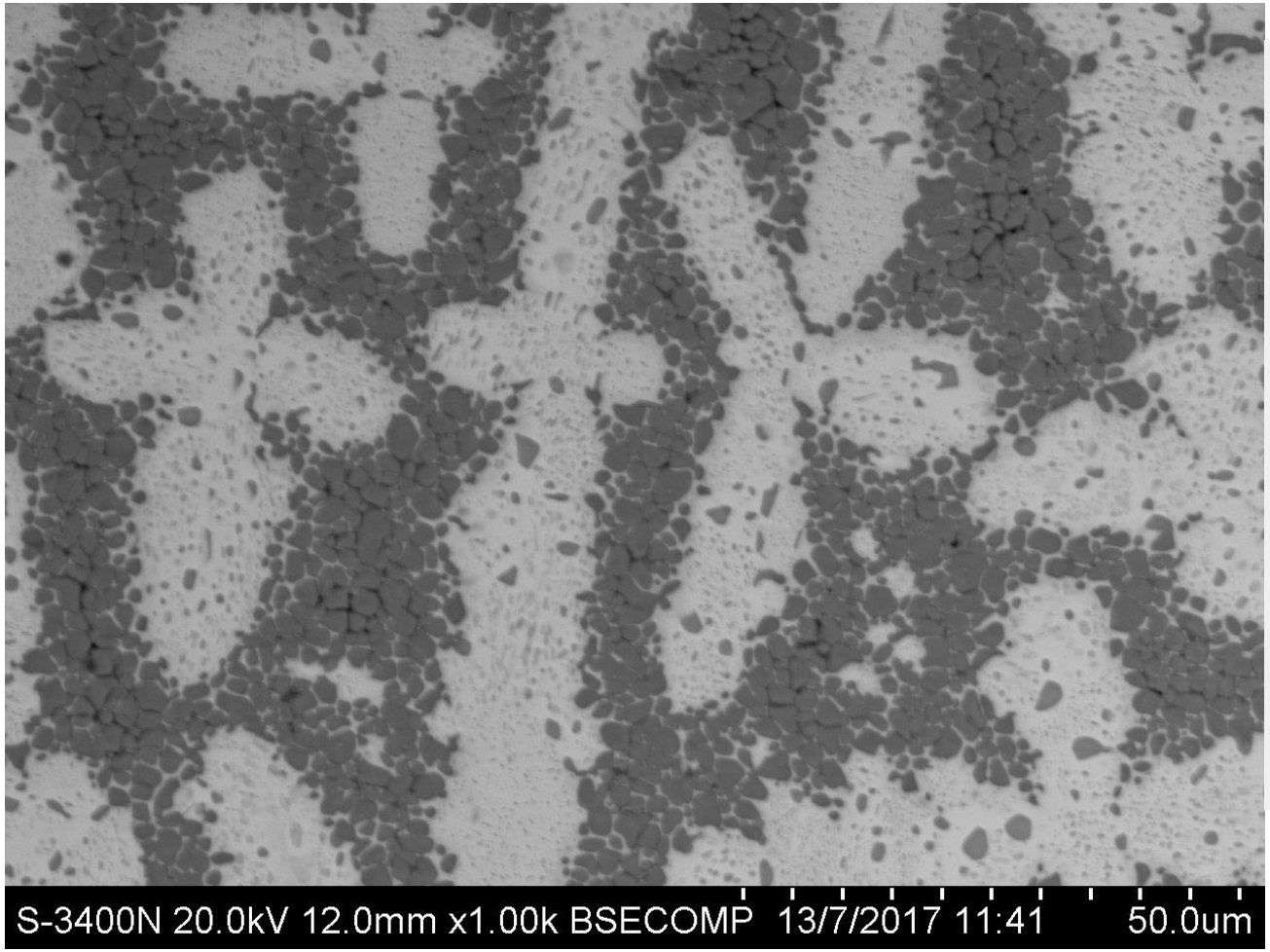


Detail of previous image captured in SE mode.

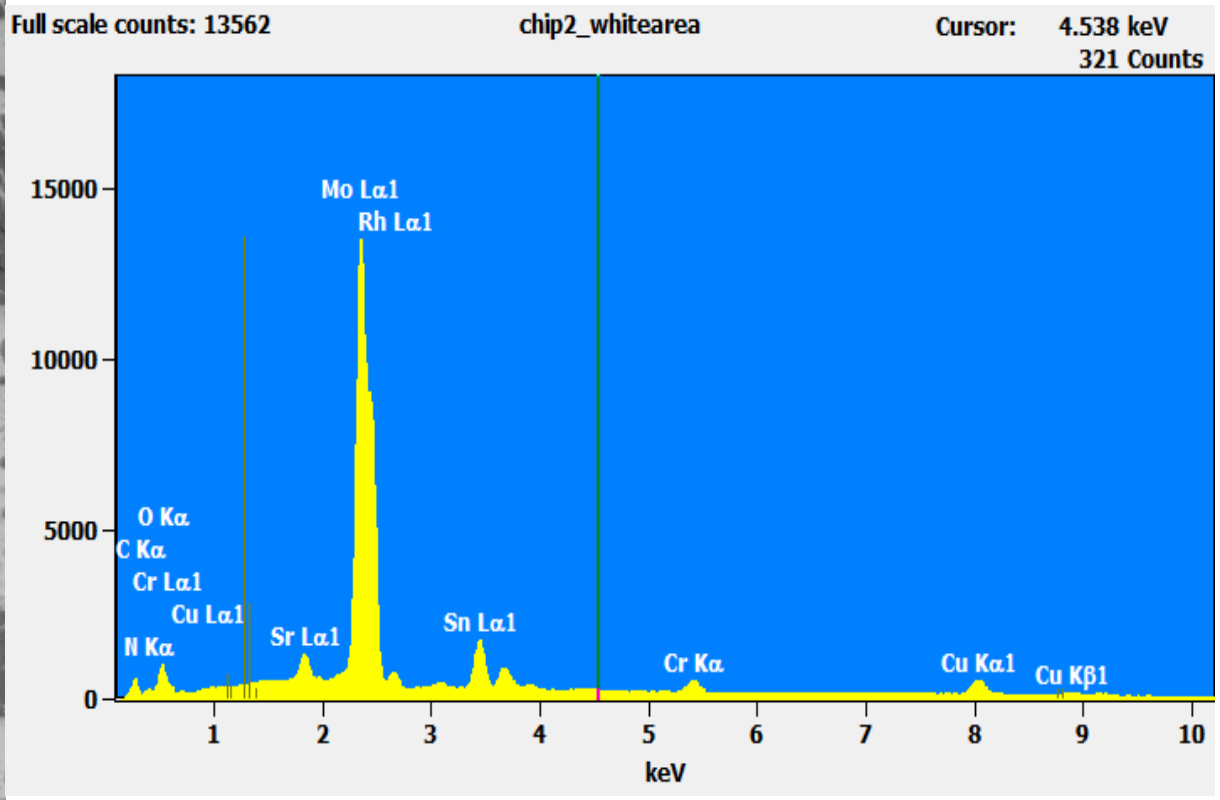


X-Ray spectrum of the “dark” area.

RESULTS AND DISCUSSIONS



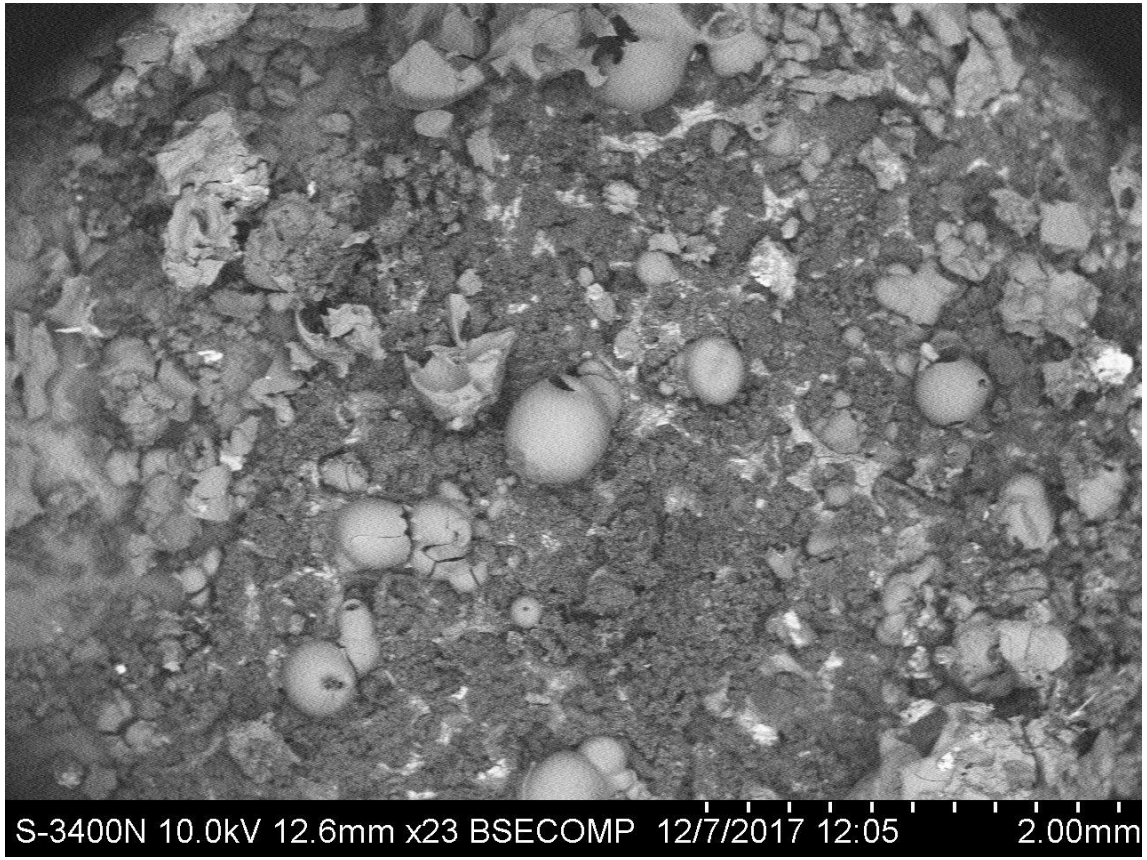
Detail of previous image captured in BSE mode.



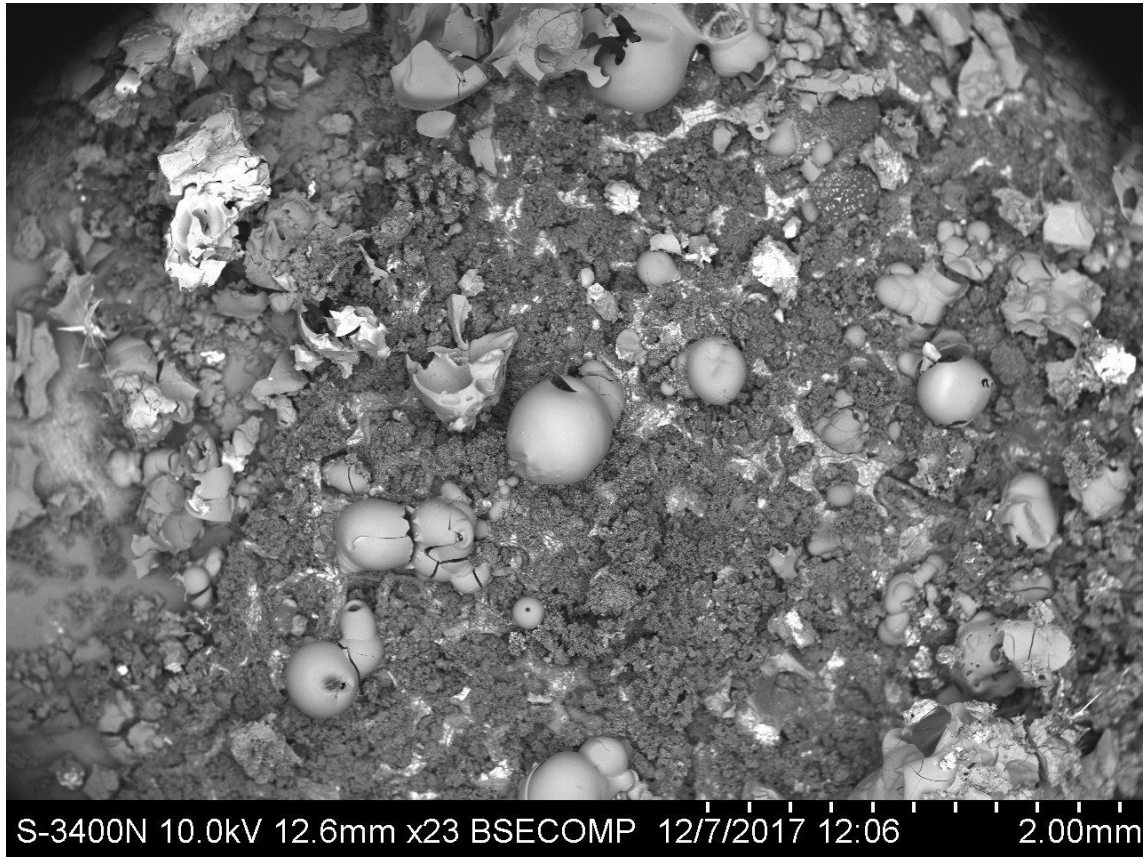
X-Ray spectrum of the “bright” area.

RESULTS AND DISCUSSIONS

Example of capturing images with different scanning speed.



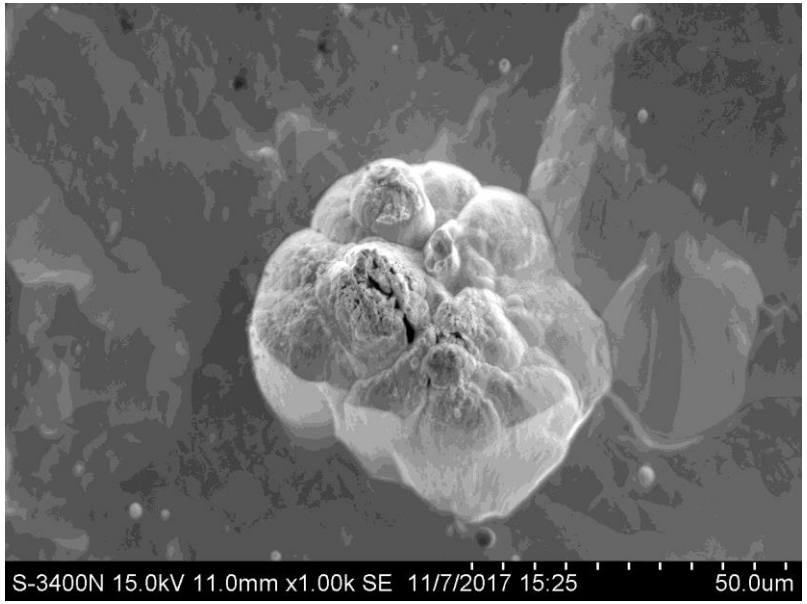
Fast scanning speed.



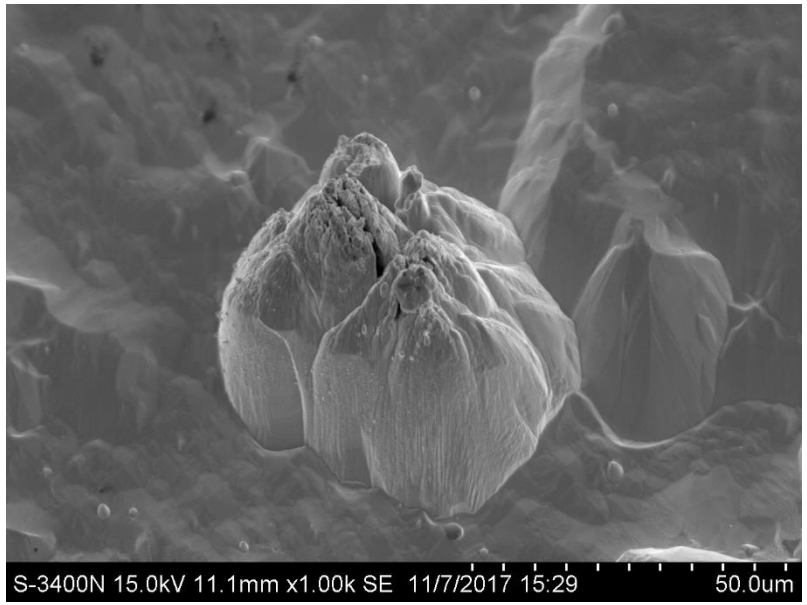
Slow scanning speed.

RESULTS AND DISCUSSIONS

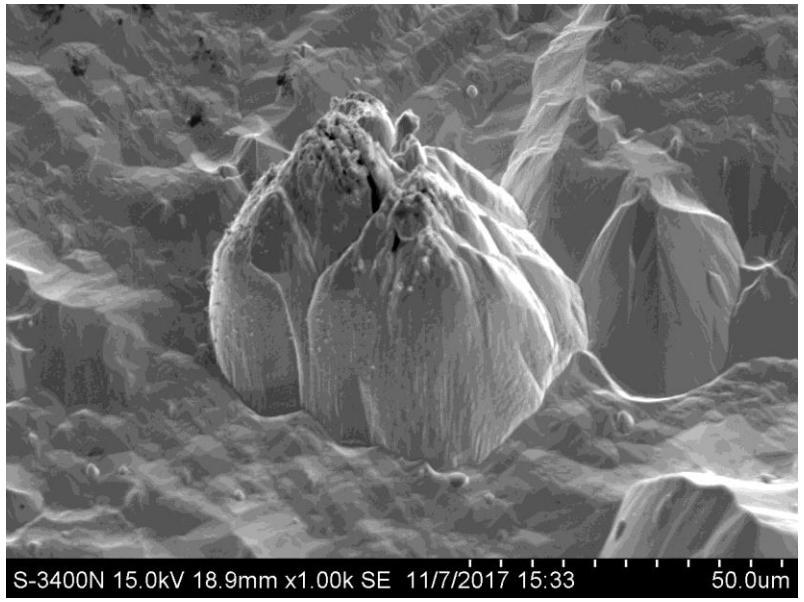
TILT 0



TILT 30



TILT 45



- Tilting the sample holder increases visible topography details and the brightness of the surface.

CONCLUSION

- SEM is an important tool for investigation of particles and small object which are not possible to be observed with other types of microscopes;
- SEM allows us to observe samples with high magnification and resolution;
- The quality and resolution of SEM images are function of three major parameters: instrument construction, selection of imaging parameters (e.g. operator control), and nature of the specimen. All three aspects operate concurrently;
- The SEM image is a 2D intensity map in the analog or digital domain each image pixel on the display corresponds to a point on the sample, which is proportional to the signal intensity captured by the detector at each specific point.

Thank you for your attention.