Investigation of secondary electron emission from selected nanomaterials

Report by: Marian Cholewa, *Univ. Cologne*

A1. SCIENTIFIC AIMS

A proposed project will concentrate on investigation of a basic properties of a secondary electron emission (SEE) from selected nanomaterials. The proposed materials will express enhanced SEE under radiation including ions, electrons, X-rays and photons. This project will concentrate on following four elements:

- Selection of materials and nanomaterials with enhanced SEE properties.
- Characterization of selected materials for SEE properties under influence of radiation.
- Theoretical modeling of SEE properties of selected nanomaterials as a function of material properties and radiation.
- Development and characterization of a novel radiation detector.

The Author with collaborators performed a pilot studies [1-5] on selected materials including: (a) polycrystalline CVD diamonds doped with boron and (b) nanomaterials including ZnO/AlN and ZnO/GaN (zinc oxide nanorods covered with thin layers of AlN and GaN. These materials have shown enhanced SEE properties when compared with thin layers on Au (gold) and CsI (cesium iodide).

A2. SIGNIFICANCE

It is still unknown what properties of material will influence enhancement of SEE under influence of ionizing radiation. In this project, the Author is proposing to fill the gap in basic understanding of above mentioned materials and other carefully selected nanomaterials. The concept of developing materials and applications based on their superior SEE properties is new. And we organized a powerful and experienced team of researchers and institutes to fill the existing gap in science. There has been vast amount of new materials developed around the globe. Most of them have never been tested for SEE properties and as potential candidates for development of new class of radiation detectors. These detectors are of great interest for both, analytical industry and scientific community.

A3. PRESENT KNOWLEDGE

The newly developed materials will be studied by secondary electron emission measurements using ions, X-rays, electrons and photons. The followings are the deliverables of the project:

1. **Development** of new materials including one-dimensional (1-D) heterostructure nanomaterials. The field enhancement factor inherited from 1-D nanomaterials such as carbon nanotubes and nanorods together with a coating using negative electron affinity materials (eg. MgO, diamond) should lead to enhanced secondary electron emission properties.
2. **Development of the test facilities** for the assessment of a quality of the thin film nanomaterial during and after production process. This work will be done in Germany.
3. **Characterisation and testing** of these new materials for: (a) secondary electron efficiency, (b) resistance to radiation damage, (c) resistance to external conditions (e.g. atmosphere, humidity, etc.), (d) regeneration protocols. This work will be done in Germany.
4. **Theoretical modelling** of the production and emission of the secondary electron emission from investigated nanomaterials as a function of ionizing radiation.

Similar developments led by Dr Cholewa in Germany enabled his group to develop a commercial detector for ions by using Boron-doped CVD (chemical vapour deposition) diamond [1-3] as a secondary electron emitter. However, these new nanomaterials based on nanotubes and nanorods look even more promising than Boron-doped CVD diamond and hence would enable detectors to be produced with improved properties. And preliminary research, performed by Dr Cholewa and his collaborators in Singapore [4], produced very promising results and obtained a patent in USA [5] in this area.

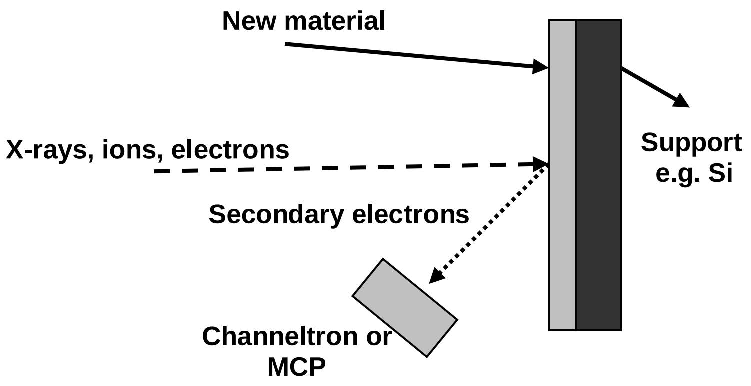

Figure 1: This figure shows the schematic diagram of the new detector and experimental set-up with the new material. The secondary electrons generated by the ionizing radiation are detected either with electron multiplier (channeltron) or micro-channel-plate (MCP) detectors connected with the proper electronics.

Vast amount of work has been performed for development and characterization of physical properties of CNTs [6-8] and NNs [2-3,9] and also their field emission capabilities. And this research has been mostly driven by the need for development of new generation flat screen displays. However, to the best of our knowledge there has been no research on these nanomaterials for their secondary electron emission properties and as potential candidates for development of new detectors for ionizing radiation. This project will be the first of its kind.

Detectors used for detecting X-rays, ions and electrons play an important role in market of analytical instruments (e.g. SIMS, MALDI-TOF). Several companies have been delivering detectors and components for

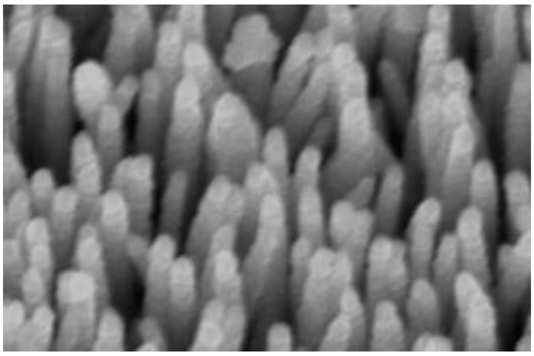

Figure 2: SEM image of AlN coated ZnO nanoneedle coaxial heterostructure. More details in [12].

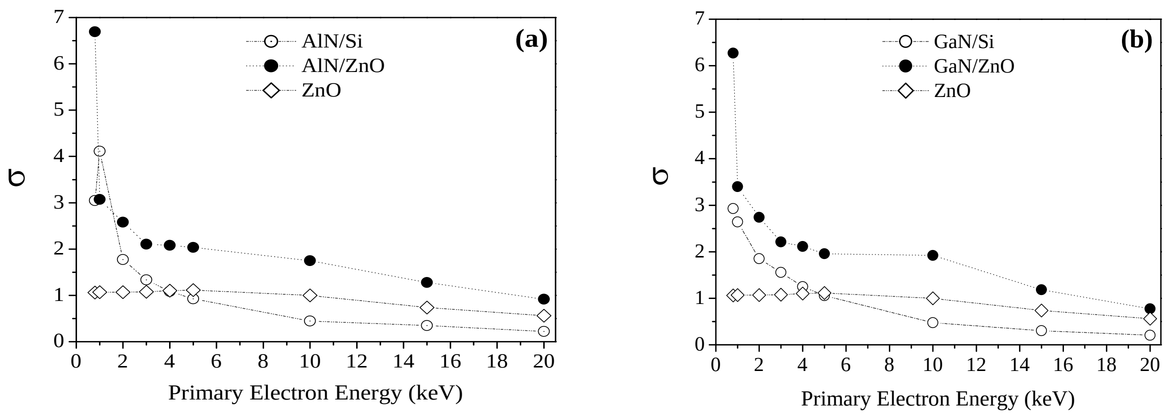

Figure 3: Secondary electron efficiency for different materials as a function of energy of primary beam of electrons. ZnO nanoneedles (NNs) with AlN and GaN coating shows the highest efficiency. A ZnO nanoneedles sample shows lower efficiency. Detail data has been reported in [4].

these markets. In the last 50 years, the methods for the detection and measurement of ionizing radiation (e.g. x-rays, ions and electrons) have undergone significant evolution [11]. The old detectors are being replaced recently by new generation detectors. And these new detectors are more frequently based on newly developed materials and technologies (e.g. diamond, high purity germanium, Peltier cooled devices, etc…).

Figure 2 shows SEM image of AlN coated ZnO nanoneedle coaxial heterostructure. And Figures 3 and 4 shows results for selected samples irradiated with electrons and X-rays. The SEE properties of ZnO-coated carbon nanotubes (CNTs) using biasing technique in a scanning electron microscope [12] has been investigated.

In order to investigate the SEE emission for X-rays we used the SINS (surface, interface and nanostructure science) beamline at the SSLS in Singapore, details of which can be found in [13]. We performed X-ray photoemission spectroscopy (XPS) using a beam of 1 keV X-rays. The results of the SEE yield are shown in Fig. 4 as a function of the electron energy. It is clearly visible that GaN/ZnO exhibits the highest SEE yields.

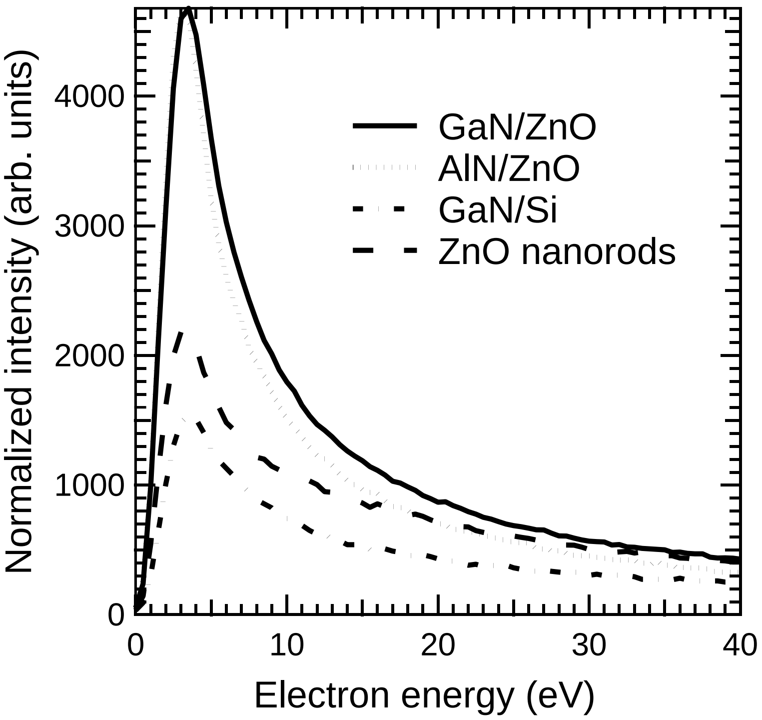

Figure 4: XPS (X-ray Photoemission Spectroscopy) spectra of 4 different samples at 1 keV photon energy. The energy of the electrons was measured between 0 and 40 eV. The data for different samples were normalized to the electron beam current of the storage ring, i.e., to the incident photon flux. No bias on the sample. More details in [4].

The use of 1-D heterostructure material as a material of choice for development of new detectors is a new concept. There has been a lot of work with these new materials to develop with their use excellent field emission devices [6-10].

Preliminary experiments performed by Cholewa et. al [4] with low energy (from 50 eV to 1.2 keV) X-rays, electrons and ions clearly show a higher secondary electron emission from NNs and CNTs with different coating.

The proposed new nanomaterials are highly efficient generators for secondary electrons and are known to be much better than Carbon, metals (e.g. Gold) and cesium iodide (CsI) which have been routinely used in the past. However, the understanding of the mechanisms that contribute to its excellent properties are still not understood, and therefore it can be difficult to produce material with reproducible properties. Therefore, we are creating a network of research institutes to solve remaining problems associated with this development, such as:

- development of the technology for a reproducible production of highly efficient layers of nanomaterials with thickness of 1 micrometer or less.
- development of a test facilities for monitoring the quality of layers of nanomaterials as the electron emitters
- development of theoretical model(s) for understanding the physical phenomena governing production and emission of secondary electrons from these new materials and nanomaterials.
- development of different models of detectors and associated electronics

This project requires a close collaboration within mixed teams of specialists. This new detector will have following properties:

- Efficiency close to 100% which is much higher when compared with existing traditional systems.
- The new system will be able to work with different vacuum (10^{-5} torr and better) and wide range of temperatures.
- Detection system will offer a possibility of a very fast counting of detected ions with a speed in excess of 10 MHz (e.g. 10^7 ions/s) which is very important for many scientific and commercial applications.
- A detection system for ns accuracy time of flight of low energy (5-40 keV) very heavy molecular ions (10^2 – 10^6 Daltons).

A4. DEVELOPMENT OF HIGH EFFICIENCY AND HIGH SPEED RADIATION DETECTOR

There is a constant demand in the scientific community and industry for highly efficient and high speed detectors systems. To answer this call we decided to develop new class of detector(s) based on newly developed nanomaterials. Additionally we are proposing the following:

- These new detectors will be characterized for sensitivity to radiation damage and their behaviour under different conditions (e.g. temperature, storage, vacuum, etc.).
- Testing with x-rays, electrons and ions (from low mass as proton to heavy molecules up to 10^6 daltons) as a function of energy and count rate to determine efficiency of a new detector.
- We will explore possibilities to develop 2-dimensional (2D) detector based either on 2D MCP or 2D position sensitive device such as CCD.

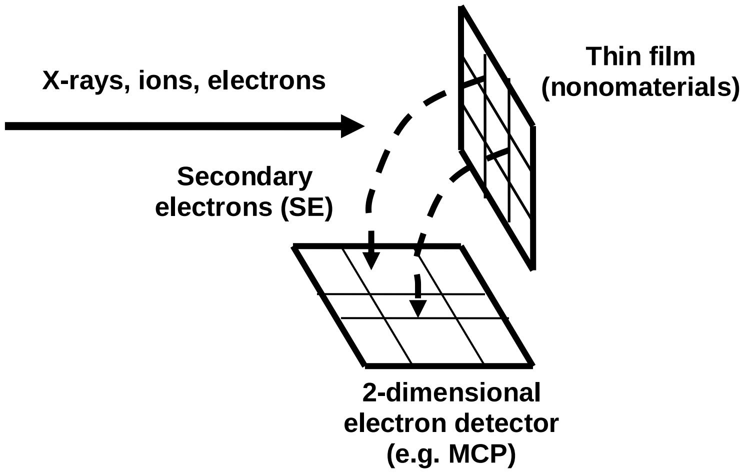

Figure 5: Schematic view of the 2-dimensional (2-D) detector. The secondary electrons emission (SEE) generated from the thin film nanomaterials are collected by the 2-dimensional electron detector (e.g. MCP). Secondary electron trajectory is formed by a combination of electric and magnetic fields (ExB). A signal from the 2-D electron detector will be processed by a custom-build fast electronics.

A major avenue of development is to combine electric and magnetic fields, so that electrons produced by radiation impact on a rather large area (>5 cm^2) are brought to a two-dimensional electron detector (e.g. MCP) surface with tens pico-second accuracy. Fig. 5 shows the principle of the method. An electric field of 1-3 kV/cm accelerates the electrons from the nanomaterial plate. A weak magnetic field (100 to 200 Gauss) normal to the electric field causes the electrons to move in a semi-circle until they hit the electron detector area that is perpendicular or parallel to the emitting surface. High time coherence is maintained for all electron paths. Only the distribution of initial secondary electrons energies contributes to minimal time spread in the 1.5-4 ns electron flight duration. Increase in counting efficiency is obtained through the much increased electron emission from the nanomaterials, compared to detecting directly with MCP that has about 50% dead area and lower secondary emission response for radiation (e.g. ions or X-rays).

Newly developed detectors (1-D or 2-D) will require state-of-the-art fast electronics. There are several scientific institutions and companies specializing in development of custom-build electronics. This electronics will be designed and developed to our specification and purchased from the market.

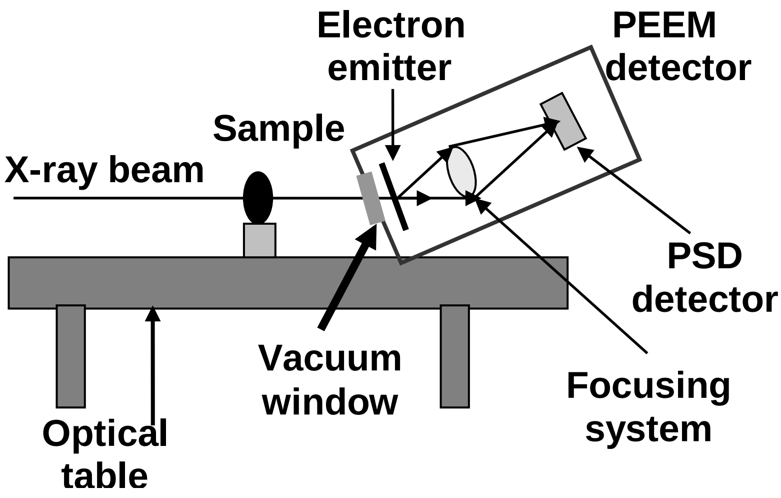

Figure 6: Schematic diagram of the modified PEEM imaging system for the synchrotron imaging beam line. The X-rays are passing through the sample and a very thin window at the front of the PEEM detector. Secondary electrons are emitted from the thin electron emitter. The PEEM detector is slightly tilted to prevent the high energy γ radiation from residual gas scattering in the storage ring to reach the detector. The sample is mounted on a high positioning accuracy XYZ stage.

As shown in figure 6, it might be possible of improving the spatial resolution to below 100 nm in X-ray imaging by modifying a standard photo electron emission microscopy (PEEM) system to be used as a high resolution X-ray detector called X-ray Electron Emission Microscopy (XEEM). A 300 nm spatial resolution [14] was already demonstrated with this concept. A further reduction to the sub 10 nm range may be possible using a proposed time-of-flight PEEM approach [15].

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A10. COLLABORATORS

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Experiments at the University of Cologne, Germany in 2017

B.1 EXPERIMENTAL SET-UP

In May and June 2017 we have performed series of experiments at the facilities of the University of Cologne. We performed these experiments with a ^{241}Am α-source with dose delivery of 1.4 MBq (measured source intensity was about 0.3 MBq). The experimental setup is presented in figures 7 and 8. The electronic set-up for the experiment is presented in figure 9. The set-up realizes the idea presented in figure 5.
The alpha particles hit a 200µm thin silicon nitride (Si_3Ni_4) sample of size 2x2 mm² covered with metal films or nano-materials. The in backward direction emitted secondary electrons were transported by an electrostatic system composed of an accelerating stage and a 45° electrostatic mirror to an 100x80mm² MCP. The samples were biased with -2.0 kV for the extraction of the secondary electrons. The electron multiplication inside the MCP generates a signal which was fed in a constant fraction discriminator with a given threshold. Signals above the threshold are counted as detected alphas. The signal height and the number of detected alphas is tightly related to the emitted number of secondary electrons per ion. Hence this is a measure for the quality of the secondary electron yield of the material. Behind the foil was silicon surface barrier detector for registering the total number of alphas (N_α) which traversed the sample. It has a counting efficiency of practically 100%. To discriminate between false MCP signals (e.g. from the dark counts) and real alphas the time of flight between the sample and the silicon detector was used. Only coincident events within a given time difference were considered as real alphas (N_{COINC}).

The detection efficiency was calculated using formula:

$$E = N_{COINC}/N_\alpha \qquad (1)$$

The experimental set-up is a comparable set-up for a future application of the secondary electron emitters as transparent ion tracking devices.

Figure 7: Vacuum chamber with associated equipment and electronics. During experiments the vacuum was about 0.5-2. x 10^{-6} mbar.

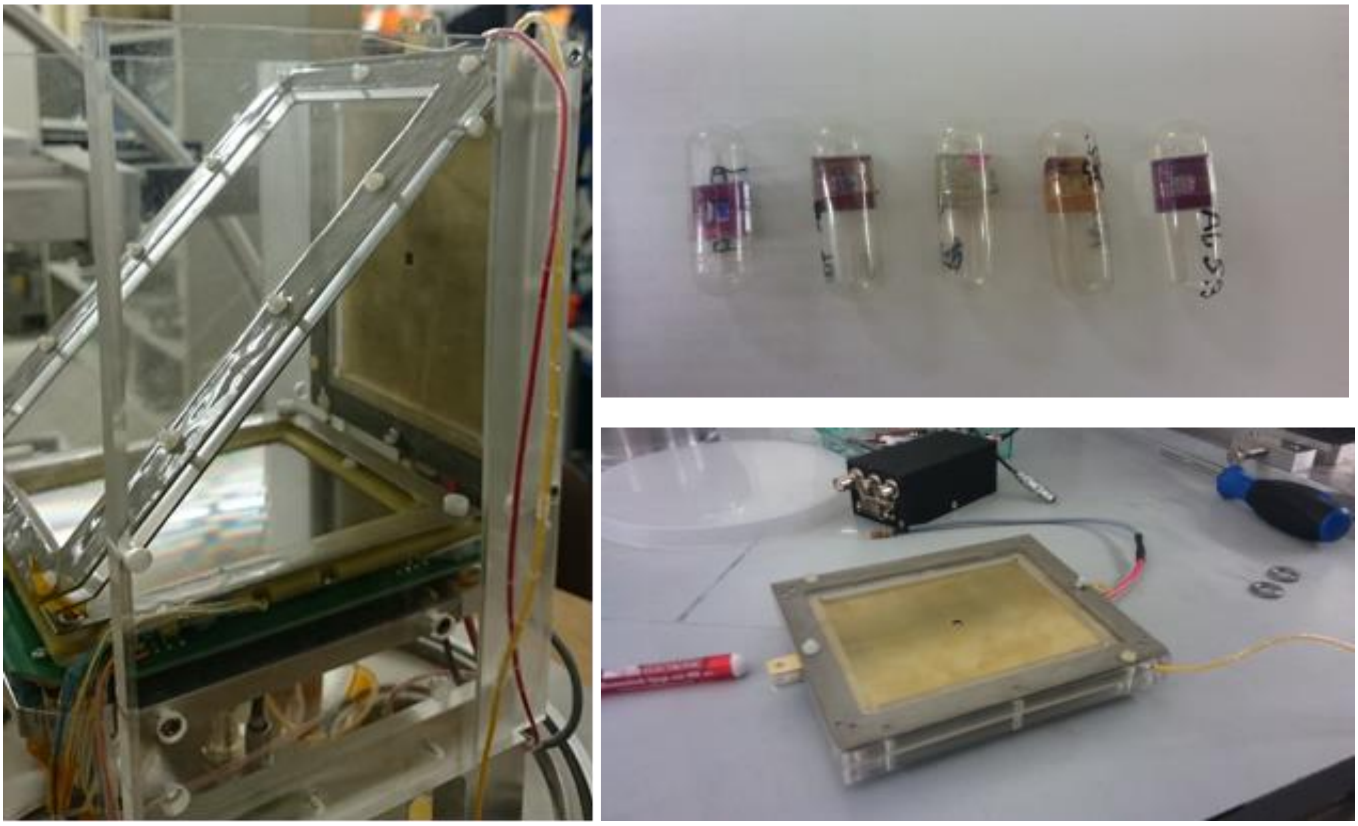

Figure 8: Microchannel-plate (MCP) detector with associated equipment and electronics. One Mesh has an open area ratio of 92.4% (left). Different samples (upper right) and setup with brass mounting to centre the foils and applying a quasi-homogeneous field in the sample region (lower right).

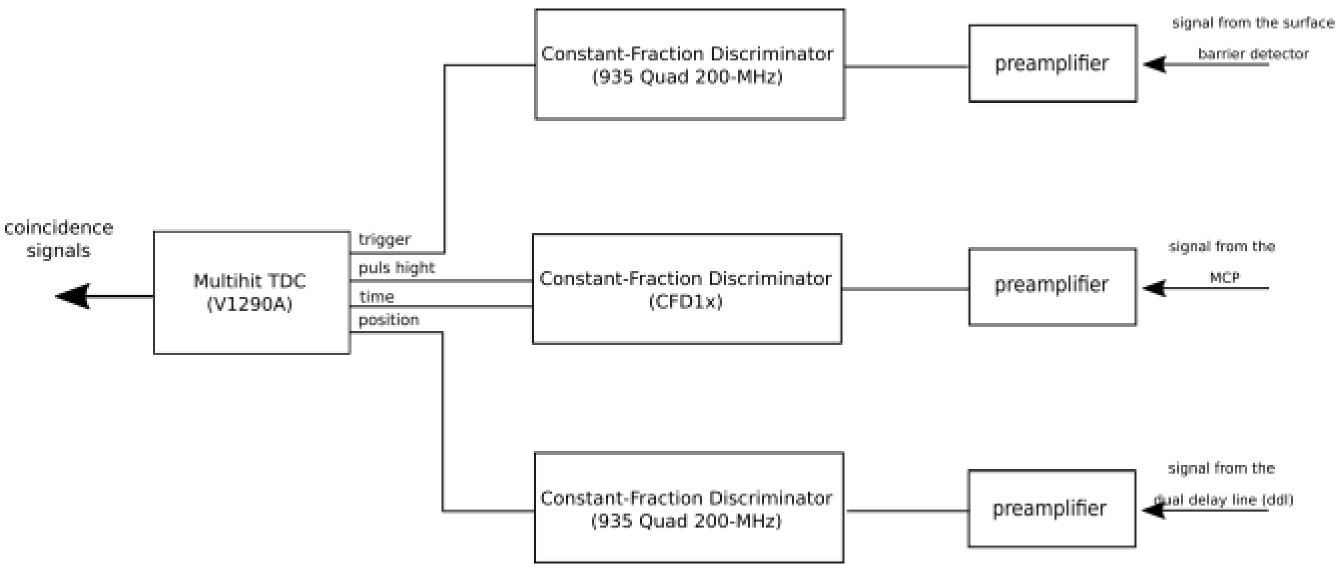

Figure 9: Electronic set-up for experiments.

B.2 RESULTS

Detection efficiency of metal films

We studied silicon nitride substrates covered with different metals mainly of thickness of 20nm. The results are presented in table 1. To study the dependency of the layer thickness, we measured Pt samples of different thicknesses. The results are presented also in table 1.

Material	Z	Density [g/cm³]	Mass thickness [µg/cm²]	Thickness [nm]	N_{COINC}	N_{α}	Efficiency [%]
Li	3	0.53	4.8	90.0	8,678	10,523	82.5
Al	27	2.7	5.34	19.6	85,290	103,600	82.3
Au	79	19.3	38.5	19.9	60,637	78,844	76.9
Pt	78	21.4	18.0	8.4	19,554	31,088	62.9
			49.0	22.9	23,120	38,500	60.1
			103.0	48.1	20,947	33,391	62.7
substrate (Si_3N_4)		3.44	68.8	200	7,282	10,280	70.8
empty					9,288	209,200	4.44[1]

Table 1: Measured detection efficiency of different metals evaporated on silicon nitride substrates from SILSON Pty. Ltd. produced at the University of Cologne. Also shown are measurements for only the substrate and for an empty detector.

Detection efficiency of novel 2D nano-materials

Promissing novel 2D nano-materials were studied. The results are summarized in table 2. All samples from the Hong Kong Polytechnic University are 2-dimensional nano-materials with structure similar to Graphene.

Material	Thickness [nm]	N_{COINC}	N_{α}	Efficiency [%]
$ReSe_2$		28,329	39,749	71.3
Graphene (SILSON Pty. Ltd.)		40,130	55,815	71.9
Graphene		22,919	33,454	68.5
$PtSe_2$	2.8	21,852	30,624	71.4
$PtSe_2$	19.0	22,244	30,314	73.4
$MoSe_2$		23,733	34,598	68.6
VSe_2		22,980	33,191	69.2

Table 2: Measured detection efficiency of different 2D nano-materials on silicon nitride substrates from SILSON Pty. Ltd. produced at the Hong Kong Polytechnic University by Prof. Daniel Lau (if not stated otherwise).

Detection efficiency with different energy losses

To compare the efficiencies with different energy losses of the alpha particles in the samples we inserted in between americium source and sample an 8.0 mg/cm^2 thick gold foil to reduce the alpha energy. The mean energy of the alpha particles was estimated to be 4 MeV. Based on the program LISE++ we calculated the mean remaining energy after passing the foil to be 1.6 MeV. Finally the energy loss after 38.5 µg/cm^2 gold was calculated. The results from the measurement using alpha particles with reduced energy are shown in the table 3.

[1] Identified as electronic cross talk, distinguishable by time difference (cf. discussion)

Material	Thickness [nm]	Mean alpha energy [MeV] (estimated)	Energy loss after 19.9 nm Au [keV] (calculated)	N_{COINC}	N_{α}	Efficiency [%]
Au	19.9	1.6	9.94	38,180	44,695	85.4
		4.0	13.90	60,637	78,844	76.9

Table 3: Comparison of detection efficiency at different energy losses in the same sample.

B.3 DISCUSSION

Material	Thickness [nm]	Efficiency $_{\text{material +substrate}}$ / Efficiency $_{\text{substrate}}$	Efficiency $_{\text{material + substrate}}$ / Efficiency $_{\text{Al + substrate}}$
Al	19.6	1.162	1.0
Graphene (SILSON Pty. Ltd.)		1.016	0.874
Graphene (HK Poly U)		0.968	0.832
$ReSe_2$		1.007	0.866
VSe_2		0.977	0.841
$PtSe_2$	2.8	1.008	0.868
$PtSe_2$	19.0	1.037	0.892
$MoSe_2$		0.969	0.833

Table 4: Comparison of the relative yield from the investigated 2D nano-materials to the yield from the Si_3N_4 substrate (Efficiency $_{\text{substrate}}$) and a Si_3N_4 substrate with 20 nm Al layer. None of the studied materials shows significantly higher yield compared to Al or Si_3N_4.

The electron yield is proportional to the deposited energy in the surface layers. The thickness of the studied nano-materials is limited from the available production techniques. On the other hand the range of the particles which will be measured with the developed detectors is much larger that the nano-material thickness. Therefore it is sufficient to compare the number of emitted secondary electrons from the thickest available samples in order to select a promising secondary electron emitter. In our measurements the “detection efficiency” presented in tables 1 and 2 is proportional to the secondary electron yield. The coefficient of proportionality is determined by the MCP detector efficiency (cf. e.g. table 3).

A comparison of the yield from the studied nano-materials relative to the yield of a bare Si_3N_4 substrate foil and a foil covered with 20 nm Al is shown in table 4. None of the new materials stands with significantly higher secondary electron yield compared to Si_3N_4 or Al in this experiment. For the application as ion tracking devices we conclude to use Al films at the moment. This conclusion must be restricted to the above stated limitation of thickness and to possible future improvements of the nano-materials.

The reproducibility of the measurements were regularly checked by measuring former measured samples again. The results could be reproduced within less than 1%.

Fake coincidences

To determine the origin of the coincident events we measured without any sample (4.4%), we made further investigations. In order to rule out the possibility to measure the 59.94 keV gamma-quanta with the silicon surface barrier detector and alpha-particles hitting the MCP coincidently, we first covered the americium source with a thin plastic. No significant gamma-quanta were measured.
After replacing the plastic we also run the experiment without applying voltage on the electrostatic mirror.
The MCP didn´t measure significant event rates.

We uncovered an electronic noise in the signal from the silicon surface barrier detectors pre amplifier which was caused by the MCP. This noise leads to a coincident fake rate at the order of the 4.4 % events we measured previously. Our conclusion is that the events are not related to physically events. The false coincidences shows a specific time difference which makes them distinguishable from real coincidences. Hence a correction for N_{COINC} was possible.

Remarks and open questions from experiments at Cologne University:

1) Very high efficiency for Al, Li (above 80%), Au (above 75%) and Silicon nitride (above 70%).
2) Lower efficiency for Pt (ca. 60%).
3) Efficiency depends on the material of the sample in a complex way.
4) Efficiency may depend on the sample thickness. The mean free electron path of the secondaries has to be studied.
5) Efficiency may depend on the sample uniformity.
6) Efficiency depends on the energy loss in the active thickness of the material.
7) Efficiency may depend on the absorbed rest gas on the surface of the sample.

Remarks and open questions from the measurements with 2-dimensional materials from Hong Kong:

1) The efficiencies of the 2-dimensional materials are comparable to the efficiency of the pure silicon nitride sample.
2) The efficiencies of the 2-dimensional materials at the given thicknesses are lower than Al and Li.
3) The samples may be too thin to have a measurable effect.
4) The 2-dimensional $PtSe_2$ seems to behave different to the Pt samples.

B.4 OUTLOOK

We expect to receive further samples in the end of July. These samples will have a so called 1-dimensional nano structure (see below).
Additional to the experiment with the alpha-source, we will investigate the samples behaviour using protons or heavy ion beams.
We are also interested in measuring samples covered with Ag, CsI and samples on a different substrate than silicon nitride or without substrate, to differ strictly between effects caused by the evaporated materials and those who are caused by the silicon nitride.
Measuring the unknown thicknesses of the nano-materials with the Rutherford backscattering method will allow us to normalize the detection efficiency to an estimated energy loss.

Experiments at GSI, Darmstadt, Germany in 2015

In January 2015 experiments have been performed at GSI with thick sample of ZnO/GaN (Zinc oxide nanorods covered with thin layer of GaN). Sample was obtained from South Korea. The ZnO nanorods were deposited on ca. 1 mm thick silicon. So the sample was non-transmitting for α particles from ^{241}Am α-source. And because of the sample we were not able to conduct experiments with the set-up at Cologne University in September, 2014. For experiments at Cologne University we need transmitting samples. The picture of the sample was taken with SEM microscope at GSI and is presented in figure 10.

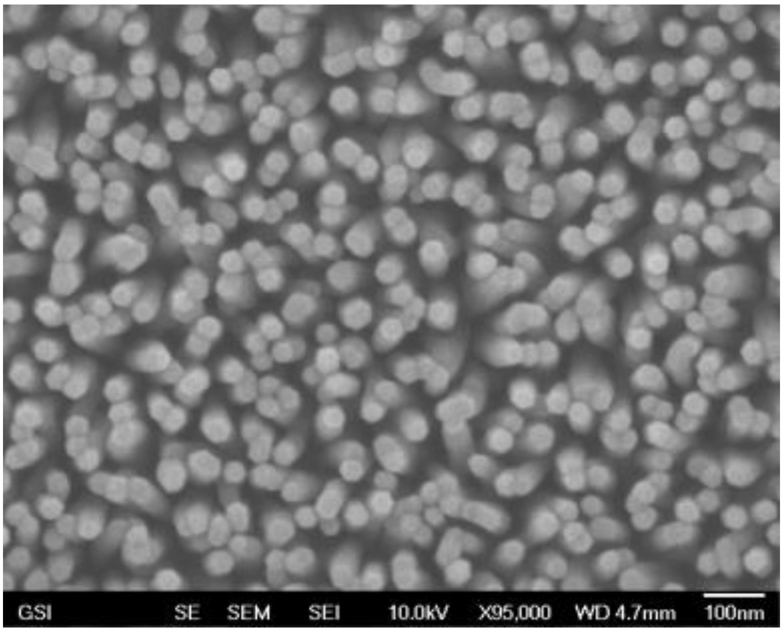

Figure 10: SEM image of GaN coated ZnO nanoneedle coaxial heterostructure. Picture obtained at GSI by Dr. Bernd Fischer and Dr. Kay Voss.

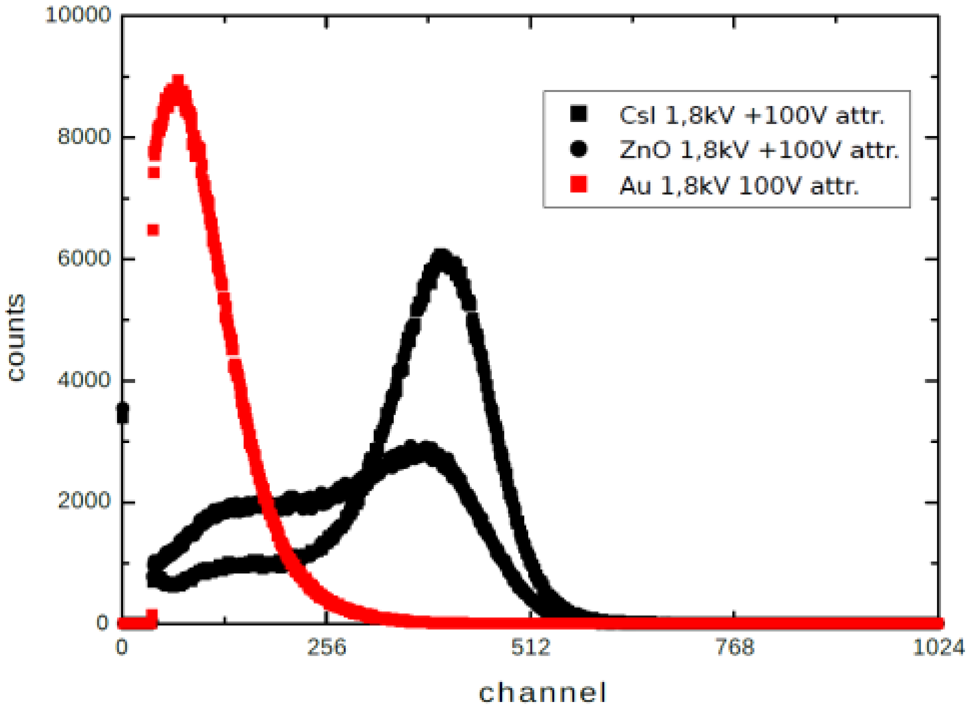

Figure 11: Signals collected from channeltron detector from Au and CsI samples. Data obtained at GSI by Dr. Bernd Fischer and Dr. Kay Voss.

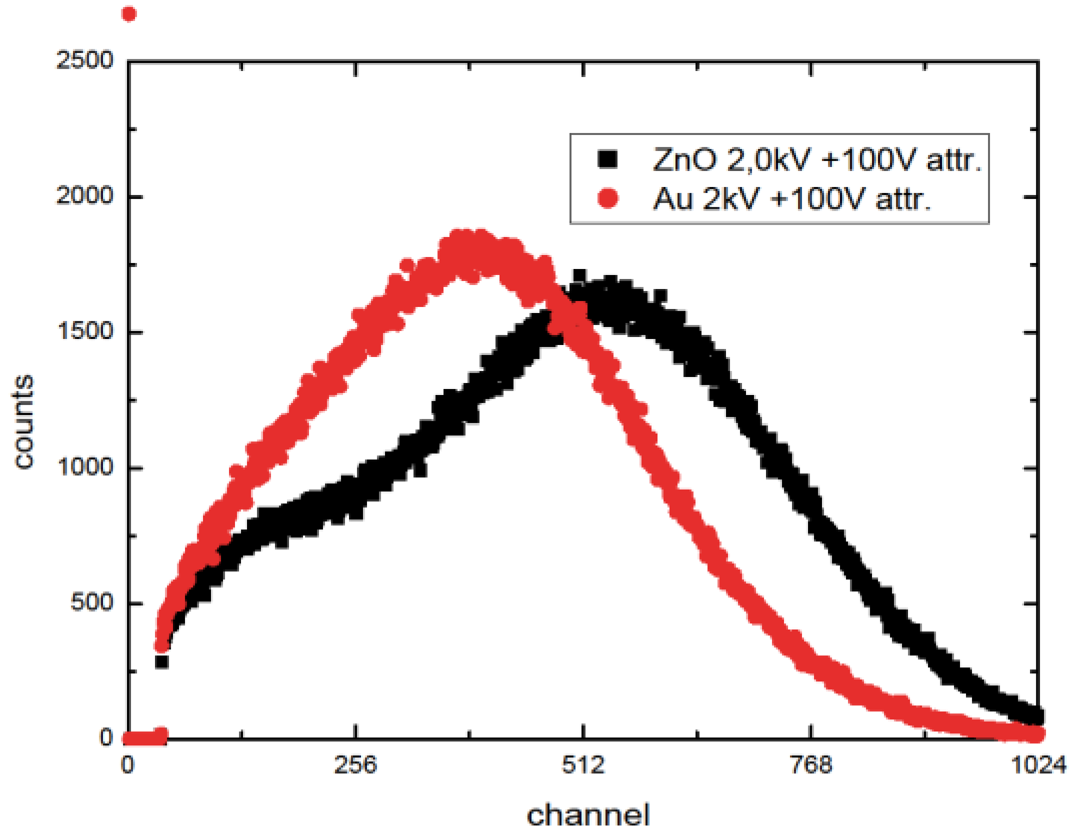

Figure 12: Data collected from channeltron detector from GaN coated ZnO nanoneedle coaxial heterostructure and from Au samples. Data obtained at GSI by Dr. Bernd Fischer and Dr. Kay Voss.