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ORIGINAL PAPER

Testing the efficiency of the Si_3N_4 membranes for charged particles registration

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Abstract Testing Si_3N_4 membrane windows is a preliminary research for the single-ion–single-cell irradiating programme prepared at the Cracow nuclear microprobe facility. The present investigation is concerned with finding a method to register every single particle of the microprobe beam when passing through a membrane to the atmosphere. The 200 nm thick membranes covered with different layers were investigated. The alpha particles, after passing the membrane window, were registered by a particle detector. Secondary electrons ejected from the membrane by alpha particles were registered by a channeltron. The channeltron signals were collected in coincidence with the silicon detector signals. The detection efficiency is the ratio of the fast coincident channeltron signal number to the total number of the silicon detector signals. The results of investigation of the membranes with different coverages are reported in the present work.

Key words Si_3N_4 • membrane • detection • secondary electrons • CsI

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Introduction

Recently a proton microprobe was installed at the Institute of Nuclear Physics in Cracow. A beam spot size of about 3 μm in diameter was obtained with 2.5 MeV protons from a Van de Graaff accelerator. One of many applications of the microprobe facility is using it as an analytical tool in biophysics. At present, the accelerated ions are focused inside a vacuum chamber, which does not permit to perform biological experiments on viable cells. We intend, however, to develop that setup focusing the proton beam in the atmosphere and to use it in the single-ion–single-cell irradiation research programme with a spatial resolution better than the size of the cell. Examples of the possible research are: the process of apoptosis (a "programmed" cell death [9, 10]), its occurrence as a function of the number of hits of the cell by protons, the cell repairing ability, DNA and RNA mutations, and others. Such investigations have already been developed in Gray Laboratory [3, 4] and are in preparation e.g. in JAERI [7] or in GSI (Darmstadt) [2].

Another interesting subject to study is the so called "bystander effect" [1, 8], in which the dying cell sends a signal to its neighbours and stimulates them to commit the apoptosis too. Hitting the chosen cells by protons and observing their non-damaged neighbours, one can learn about the range and kinetics of that process.

Vital problems in developing the external microbeam setup with a single-cell–single-ion hit facility are to find an appopriate window withstanding the atmospheric pressure (but sufficiently thin to neglect the scatterings or energy loss of the beam) and registering every particle of the beam passing through it. As windows, we used thin membranes made of Si_3N_4. Three methods of counting the charged

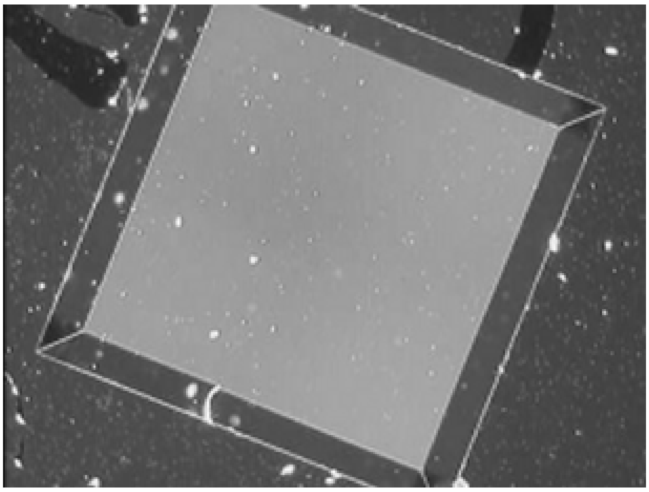

Fig. 1. Microscope image of the window with the Si_3N_4 membrane covered with a gold layer.

particles were considered: registration of secondary electrons ejected from the membrane when the ion was passing the window, registration of ions using the bottom of the Petri dish made of thin plastic scintillator, and counting the ions with a surface-barrier detector placed behind the Petri dish. Only the first method of registration (counting the secondary electrons) would enable to observe the cells with an optical microscope simultaneously with irradiating them. The aim of the present work was the evaluation of the efficiency of the Si_3N_4 membranes for light, low energy charged particle registration by counting the ejected secondary electrons. To make the system useful for our purpose, the efficiency of particles registration must significantly exceed 90%.

Materials

The Si_3N_4 membranes, manufactured using the lithographic method had a size of 1.5×1.5 mm^2 within a 5×5 mm^2 frame of thickness of 200 μm (Fig. 1). The investigated membranes were 200 nm thick. These membranes were commercially available from Silson Ltd (U.K.) [5]. In most cases, a 10 nm thick gold or indium layer was evaporated onto the membrane surface in order to remove the static charge accumulated during the interaction with charged particles. To improve the results, sandwich structures were used: Si_3N_4 membranes covered with a thin Au or In conducting layer were then coated with a 30 nm thick layer of good secondary electron emitters CsI, GaAs or ZnS [11]. The CsI covered membranes were measured also without the conducting layer of Au or In.

The TRIM [12] evaluation proved that this 60 nm thick sandwich membrane scatters more than 85% of the alpha particles by an angle of less than 0.3° and only 1.1% by more than 1°. The TRIM results for proton beam were even better, so according to the above results these membranes seemed to be very appropriate for external microbeam experiments.

After the evaporation, the surfaces of membranes were investigated using a scanning force microscope (SFM).

The SFM images of the surface structures of CsI, ZnS, GaAs evaporated on the Au or In layer are shown in Figs. 2 and 3. The structures were not uniform but were composed of islands of evaporated material; each of them had a different size and height. The CsI island sizes when evaporated on Au were of about 300 nm; when CsI was evaporated on In the islands sizes were about 50 nm. The SFM results might explain the difference in the registered efficiency between membranes with CsI evaporated on Au and In.

Method

A stainless steel chamber has been designed to measure the efficiency of detection of charged particles passing through the membrane (Fig. 4).

The membranes were tested using alpha particles from a ^{241}Am radioactive source (alpha particles energies of

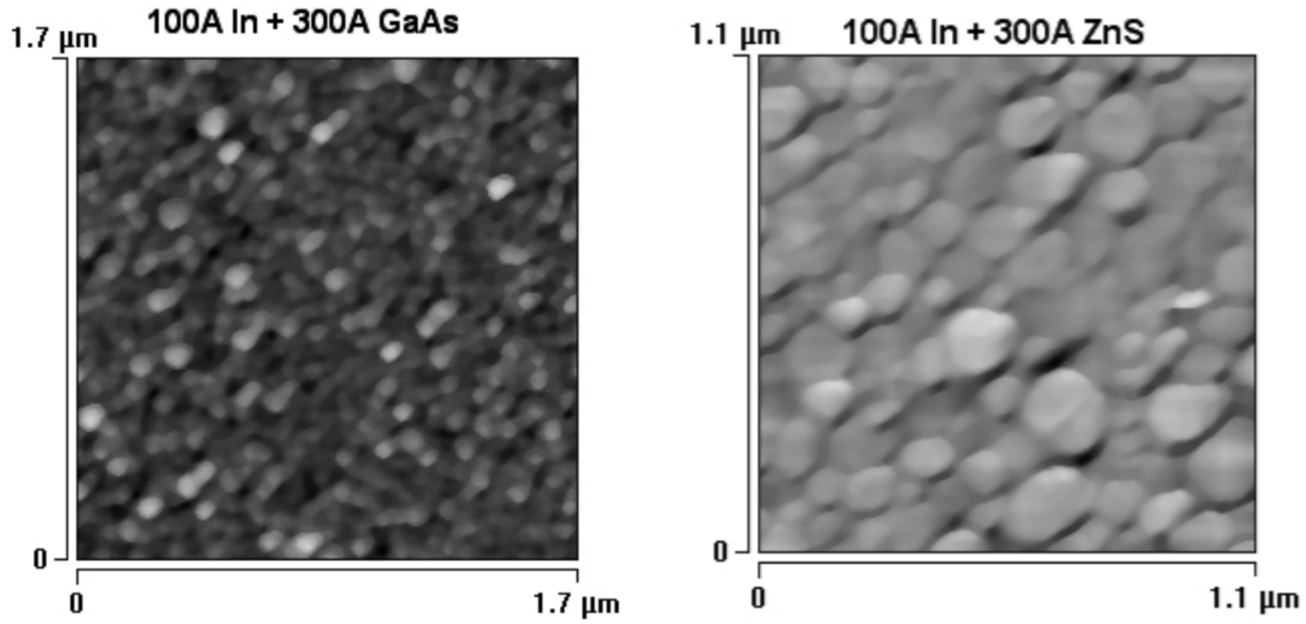

Fig. 2. SFM images of a Si_3N_4 membrane surface covered with GaAs (left) and ZnS (right). Please note the different scanning areas.

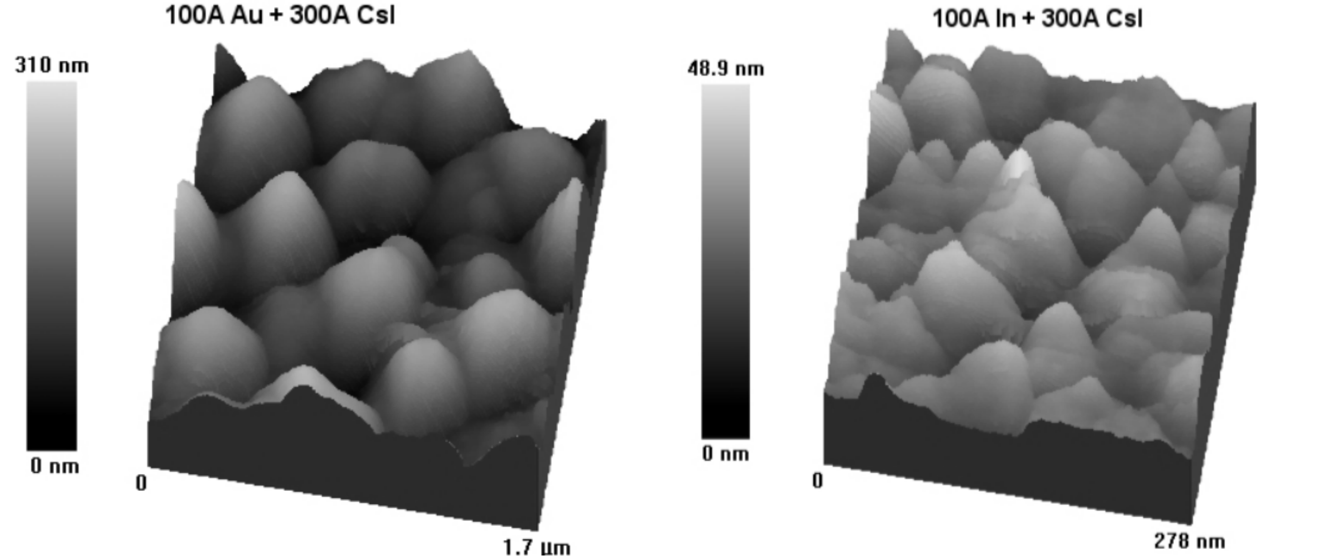

Fig. 3. Difference between the surfaces of the Si_3N_4 membrane covered with the CsI layer evaporated on In (right) and on Au (left) conductive layer. Please note the significant difference in scale.

5.442 MeV (12.5%) and 5.484 MeV (85.2%), activity of 300 μC/cm^2). Due to less effective ejection of secondary electrons by protons, the results for a proton beam (which will be used in real experiments) may be different, but at this moment the alpha source was the only way to make any estimations without serious modification to the chamber at the microbeam setup. From the other point of view however, the overall background in the case of single protons beam will be lower. The alpha particles were passing through the collimating system of diaphragms, Si_3N_4 membrane, and then were registered by a semiconductor, ion implanted silicon detector characterized by the 100% efficiency for the particle registration. The alpha particle passing through the window were ejecting secondary electrons from the membrane. Those electrons were registered by a channeltron [6]. To protect the channeltron against the gamma emission, the source was covered with a 3 mm thick lead screen. The channeltron signals were collected in coincidence with the silicon detector signals. The alpha particle detection efficiency, using secondary electrons registered by the channeltron, was the ratio of fast coincident events to the total number of the silicon detector signals.

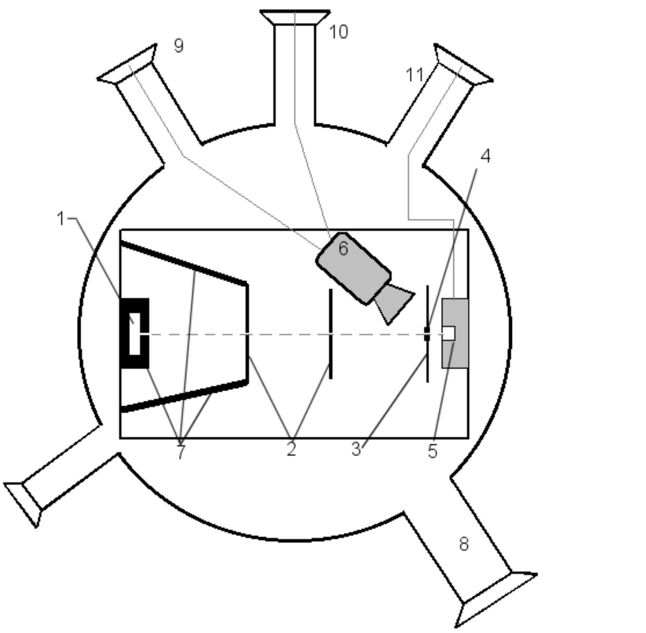

Fig. 4. Schematic view of the measurement chamber. 1 – ^{241}Am alpha source (screened with lead); 2 – diaphragms collimating alpha particles; 3 – steel frame for Si_3N_4 window; 4 – window with Si_3N_4 membrane; 5 – particle detector (ORTEC semiconductor ion implanted silicon detector); 6 – channeltron (Sjuts Electronics) [6]; 7 – lead screens; 8 – connection to vacuum pump; 9 – BNC feedthrough for channeltron signal; 10 – BNC feedthrough for delivering voltage channeltron; 11 – BNC feedthrough for Si detector signal.

The measurements were performed using a data acquisition system of the microprobe setup (schematically shown in Fig. 5). A proper window of alpha particle energy was used as a software gate to the fast coincident spectra (Fig. 6a).

Measuring procedure

The acquisition time of the single measurements was about 3 h. For every coverage, two membranes were prepared and each of them was investigated 4 times in order to check the consistency of the results. The vacuum was below 10^{-6} hPa. Discriminator thresholds were set above the noise level. We measured alpha particles by the silicon detector and the secondary electrons registered by the channeltron. Time spectra were recorded using SILENA 4418/T TDC with 1200 ns base. From the measured spectra we knew the number of alpha particles and the number of coincident events (Fig. 6b). Comparing these two values we were able to estimate the detection efficiency of the alpha particles (registering secondary electrons ejected from membrane) passing through the membrane.

Results

The results of the experiment are summarized in Fig. 7. Two test measurements were performed: i) without the steel frame, ii) with the empty steel frame.

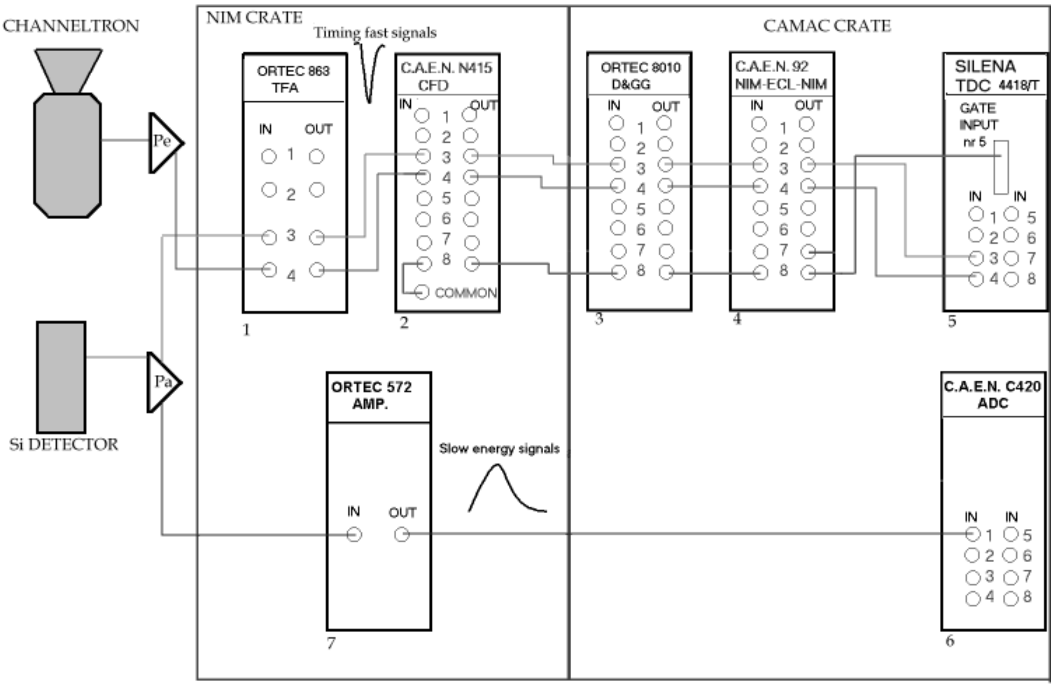

Fig. 5. Data acquisition system. Pe – preamplifier for channeltron signals. Pa – preamplifier for timing and energy signals from Si detector (ORTEC 2398). 1 – timing filter amplifier; 2 – constant fraction discriminator; 3 – delay and gate generator; 4 – NIM–ECL––NIM translator; 5 – time to digital converter; 6 – amplitude to digital converter; 7 – active filter amplifier.

In the first case, the alpha particle detection efficiency was close to 46% but in the second one only – 1%. This can be explained as follows: during the experiment without the frame, the channeltron registered electrons ejected by alpha particles from the silicon detector. With frame installed (but without the Si_3N_4 window), the secondary electrons ejected from the silicon detector by alpha particles passing through a 3 mm-diameter hole in the steel frame were stopped in the frame before reaching the channeltron. These tests proved that the channeltron was sufficiently well screened against the secondary electrons ejected from the particle detector and was counting only the electrons ejected from the membrane.

The detection efficiency measured for the uncoated Si_3N_4 membranes was about 50%, that is not sufficient for the single-ion–single-cell irradiation setup. The efficiency was not improved even after covering the membranes with a gold layer. The detection efficiency measured for the membranes with GaAs and ZnS layers did not reach 60% and, therefore, were disqualified (although the results for ZnS layer were slighty superior). Much better efficiency was achieved for CsI layers – covering directly the membrane or evaporated on the gold or indium buffer. All the results were not worse than 75% with small discrepancy, while the results for In case exceeded 80%. For the pure CsI layer, the average efficiency was almost the same as for the sample with the In or Au layer but the differences beetwen the single measurements were bigger (from 69% up to 80%). This might be explained by the absence of the conducting layer. Grounding this sample by a silver glue decreased that discrepancy. The best efficiency of 85% was achieved for the 30 nm CsI layer evaporated on a 10 nm In layer. The differences between the efficiencies for the membranes covered with CsI evaporated on indium and CsI

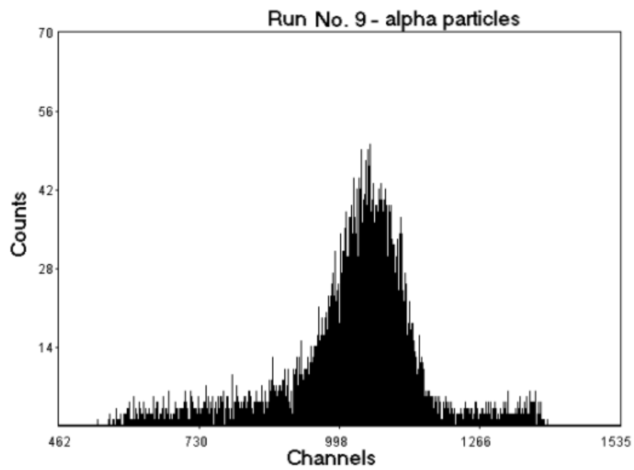

Fig. 6a. Energy spectrum of ^{241}Am alpha particles detected after the passage through membrane. Channel width is 5.37 keV.

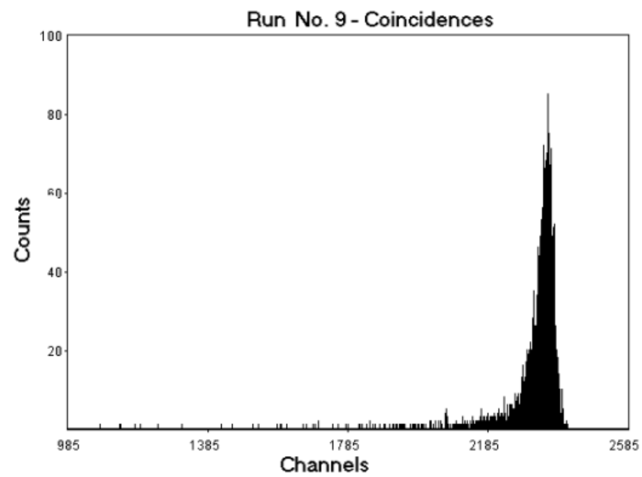

Fig. 6b. Time spectrum (coincidences between alpha particles and electrons). Channel width is 250 ps.

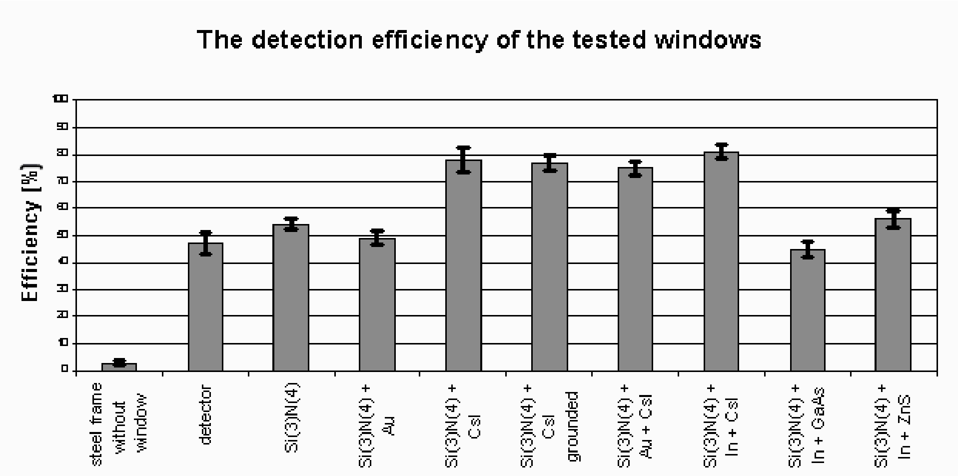

Fig. 7. The chart of the membranes efficiency for the alpha particles registration – all Si_3N_4 membranes were 200 nm thick. The Au and In conducting layers were 10 nm thick. The CsI, ZnS, GaAs layers were 30 nm thick. In most cases two different windows of the same covering were investigated.

evaporated on gold may come from the different surface characteristics.

Conclusions

The investigation performed in the present work proves that for commercially available membranes without additional treatment one cannot achieve efficiency high enough for alpha particle registration. Also, covering the membranes with ZnS or GaAs layers does not produce acceptable results. However, a thin CsI layer evaporated on the In buffer enabled us to detect more than 80% of the passing alpha particles. Although, this value is still too low for the designed single-hit microbeam setup, the CsI layer proves to be the best one for further investigations. The channeltron collected the secondary electrons only from 10% of the hemisphere. We expect that the use of a channelplate or additional channeltrons in a close geometry will permit us to detect above 90% of the passing alpha particles.

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