Scanning Microscopy

Volume 3 | Number 2 Article 2

9-11-1989

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Scanning Microscopy, Vol. 3, No. 2, 1989 (Pages 411-418) 0891-7035/89$3.00+.00
Scanning Microscopy International, Chicago (AMF O'Hare), IL 60666 USA

ANALYSIS OF INSULATOR SAMPLES BY SECONDARY NEUTRAL MASS SPECTROMETRY

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(Received for publication June 08, 1989, and in revised form September 11, 1989)

Abstract

Secondary Neutral Mass Spectrometry (SNMS) offers new possibilities for the analysis of insulators when the electron component of the postionizing SNMS plasma is employed for a precise compensation of sample charging. The compensation techniques for the three operation modes of SNMS, namely the direct, the separate and the external bombardment mode are described. Corresponding examples for bulk and depth profile analysis of insulating samples and dielectric thin film systems will be reported and discussed.

Key words: Surface, thin film and bulk analysis; Secondary Neutral Mass Spectrometry; analysis of insulating samples and thin films; charge compensation in surface and thin films analysis.

Introduction

Recent electron or mass spectrometric methods for surface and depth profile analysis employ, in general, charged primary or secondary particles which generate or deliver the analytical information. Therefore, considerable trade–offs arise in the performance of such techniques when being applied to poorly conducting or electrically insulating samples. Such drawbacks are primarily due to sample charging effects which have disturbing influences on the charged particle fluxes to and from the surface and, in many cases, field induced ion diffusion altering the surface–near composition of dielectric samples.

Surface charging leads to a fading of the analytical signals because of the deceleration or deflection of the primary electron or ion beam. In addition, in Auger– or Photoelectron Spectroscopy AES or XPS and UPS the characteristic energetic positions of the electron peaks containing the analytical information are shifting in a mostly uncontrollable manner. In Secondary Ion Mass Spectroscopy (SIMS) the kinetic energy of the analyzed positive or negative secondary ions, and subsequently via, e.g., transmission effects of the mass spectrometer the height of the analytical signal is also strongly influenced by any sample charging. Such effects remain even when neutralized primary beams are applied which, however, are difficult to implement quantitatively (Borchardt et al., 1987). In all cases, interfering charging effects require the application of additional precisely controlled charged particle fluxes. This does not only complicate the experimental procedure considerably, but does in many cases not solve such problems in a quantitative manner.

The development of Secondary Neutral Mass Spectrometry (SNMS) as a novel method for quantitative surface and depth profile analysis (for a recent review see Oechsner 1988) offers a reliable solution of the problems encountered in the analysis of insulating materials: Firstly, in SNMS neutral atoms and molecules leaving the sample surface are employed, i.e., some of the problems arising in SIMS from sample charging are avoided. For their subsequent mass spectrometric analysis the ejected neutral particles are postionized when traversing a dense hot electron gas ("Electron Gas SNMS", Oechsner 1984). Hence, secondly, a sufficient electron reservoir for the compensation of positive sample charging due to the bombarding ions involved in SNMS is immediately available from the analytical technique itself. It is the aim of the present paper to describe readily manageable techniques for a precise

and controlled compensation of sample charging in SNMS, and to demonstrate the experimental practicability of insulator analysis with this technique by different experimental examples.

The neutral surface particles to be analyzed are produced by the bombardment of the sample either with a conventional ion gun or by extracting the bombarding ions from the SNMS–plasma. The latter is the most essential component of the SNMS technique, and consists of a high purity electrodeless rf–discharge generated by electron cyclotron wave resonance mostly in Argon at working pressures in the 10^{-4} mbar regime. The electron component of this homogeneous low pressure plasma with densities of 10^{10} to 10^{11} cm^{-3} at electron temperatures corresponding to 10 – 20 eV represents a Maxwellian electron gas by which an effective postionization of the ejected neutrals with postionization factors α^0 of several 10^{-2} is established.

For the three subsequently described operation modes of SNMS, different techniques for the compensation of positive sample charging due to the ion bombardment are employed. In the following sections the corresponding compensation techniques, which always involve the electron component of the SNMS–plasma, are described with respect to their experimental performance and discussed together with corresponding experimental results.

Analysis of Insulating Samples by the Direct Bombardment Mode (DBM)

In this operation mode ion bombardment of the sample is established by extracting positively charged noble gas ions from the SNMS plasma onto the analyzed surface by means of a simple ion optics mounted in front of the sample. The bombarding energy can be varied from a few keV down to values as low as a few 10 eV at current densities of the normally incident ions in the order of 1 – 2 mA cm^{-2}. Under appropriate operation conditions extremely high lateral homogeneity of the bombarding ion current can be established. Since then any crater effects can be avoided, and atomic mixing effects can be minimized for the available low bombarding energies in the order of 10^2 eV, the DBM displays unique properties for high resolution depth profile analysis (Oechsner 1984, Oechsner et al. 1985). A schematic diagram of the Direct Bombardment Mode DBM is shown in Fig. 1.

When a sufficiently thin insulating layer is backed by a conducting or semiconducting substrate, the DBM can be applied without additional measures for charge compensation. A corresponding example is shown in Fig. 2 for a 106 nm thick SiO_2–layer on Si (Paulus 1984, Geiger et al. 1987). During the analysis under normal bombardment with 330 eV Ar^+–ions the SiO–peak of the corresponding SNMS spectrum has been continuously recorded as the most characteristic signal from the oxide. The positive charge transferred to the sample is supposedly transported to the conducting Si–substrate via defect–induced conductivity. The decrease of the SNMS–signal of SiO at the beginning of the depth profile is ascribed to the deceleration of the bombarding ions in consequence of sample charging. The arising electric field and presumably the photon flux from the neighbouring SNMS–plasma may then cause a defect induced conductivity in the layer. Since the ion acceleration voltage is applied between the Si substrate and the plasma, the effective ion bombarding energy is reduced by the potential drop across the oxide

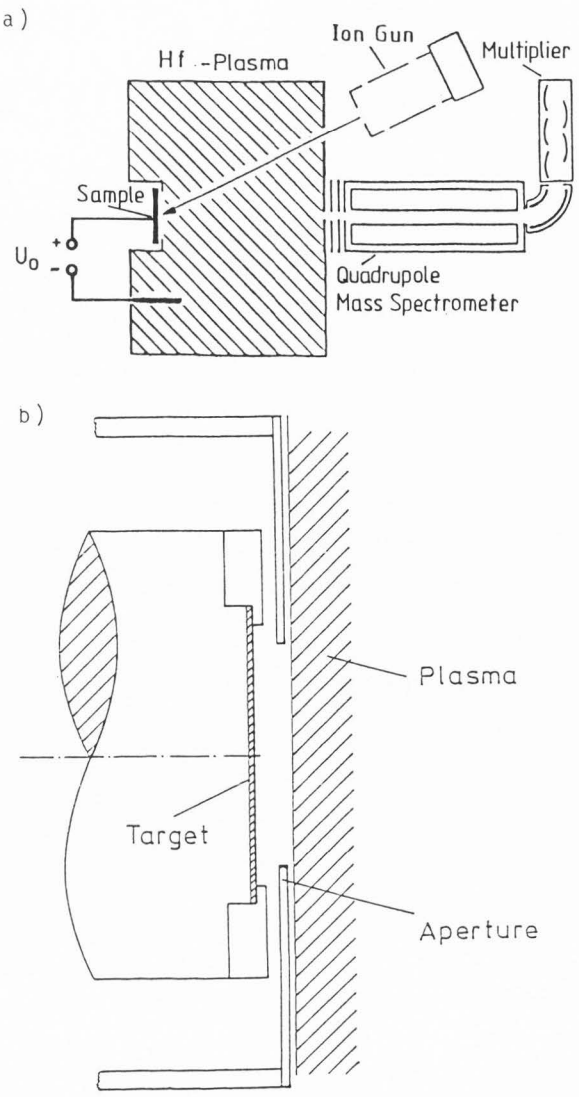

Fig. 1. a) Schematic diagram of the Direct Bombardment Mode DBM and the Separate Bombardment Mode SBM of Secondary Neutral Mass Spectrometry SNMS. The voltage U_0 applied between the sample and a reference electrode inside the SNMS plasma is used for DBM. In SBM the sample is bombarded through the plasma with a rastering ion gun; U_0 is switched off in this case.
b) Ion optical system for the extraction of plasma ions with high lateral homogeneity in DBM.

layer. This explains the slow increase of the SiO–signal with decreasing layer thickness. When the SiO_2/Si–interface is reached, the SiO–signal passes through a slight maximum, and then decreases sharply. This behaviour is understood from the so–called Direct Emission Model which applies to the molecule formation in oxide sputtering (Oechsner 1982). According to this model the surface oxygen concentration c_0^s at the actual surface from which the monoxide molecules are ejected is given by:

$$c_O^s = 0.5 \left[1 \pm \left[1 - \frac{I(MeO^0)}{I^{max}(Me^0)}\right]^{0.5}\right]. \quad (1)$$

$I(MeO^0)$ describes the SNMS MeO–signal before and after its maximum value $I^{max}(MeO^0)$ referring to $c_O^s = 0.5$.

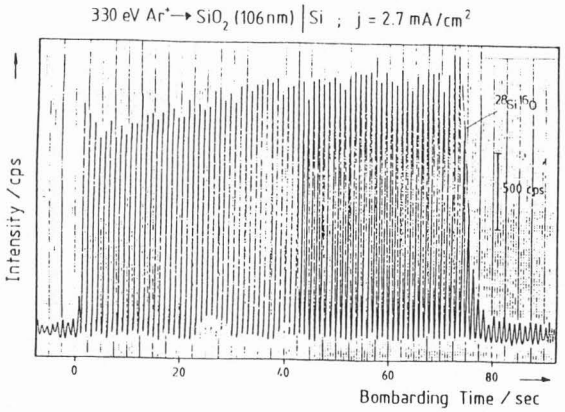

Fig. 2. SNMS–sputter depth profile of a 106 nm SiO_2–layer on Si measured with DBM. The $^{28}Si^{16}O$–peak has been continuously recorded during sputter profiling with normally incident Ar^+–ions of 330 eV.

When this model and equ. 1 are applied to the results in Fig. 2, the variation of c_O^s with the sputter time is obtained (see Fig. 3). The oxygen concentration obtained within the layer agrees very well with the SiO_2 stoichiometry. When the different sputter rates for the oxide and the substrate are considered, an SiO_2/Si–interface width in the order of 2 nm is obtained from the results in Figs. 2 and 3.

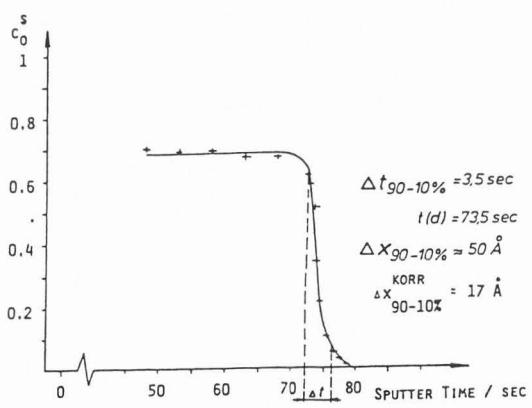

Fig. 3. Oxygen surface concentration c_O^s determined from the SNMS depth profile in Fig. 2. Only the range around the SiO_2/Si–interface is shown. When the experimental interface width is corrected for experimental influences, a corrected 90–10% width of only 17 Å is obtained.

Similar results are obtained for a thin e⁻–beam evaporated HfO_2 layer of approximately 200 nm even when a highly insulating substrate of fused silica is used. In this case defect induced charge transport parallel to the film plane out to the surrounding metallic sample mount has to be assumed (Stewart et al. 1988).

Such results demonstrate that sufficiently thin dielectric films can in principle be investigated with the DBM of SNMS even with high depth resolution without any additional measures for charge compensation. For thicker insulating films or for bulk insulators appropriate charge compensation, however, has to be established. This requirement can be met by the arrangement shown in Fig. 4 (Kopnarski 1984). The insulating target is covered with a mask in the form of a circular

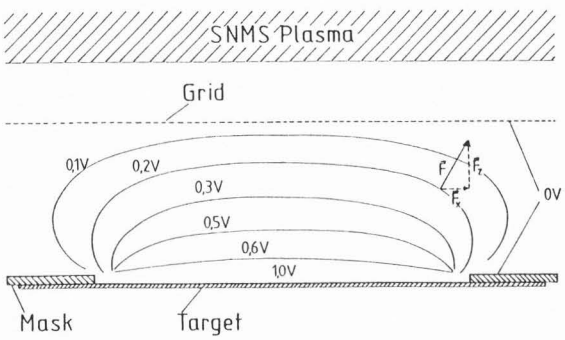

Fig. 4. DBM target arrangement for the SNMS analysis of insulators using ion induced electron emission from a circular metallic mask for charge compensation at the target. The potential lines refer to a potential drop of 1 eV from the insulating target surface to the circular mask and the metallic grid facing the SNMS plasma.

aperture, and a plasma free equipotential space confined by a grid facing the SNMS–plasma is established above the target. When the (negative) bombarding potential is applied to this system, plasma ions are accelerated within the space charge sheath in front of the grid and penetrate the grid down to the target and the mask. Low energy electrons produced at the circular mask by such ions via potential emission experience an attractive force from the neighbouring insulating target which is charging slightly to a positive potential in the order of 1 volt.

Such an arrangement conserving the directional and the kinetic properties of the low energy bombarding ions in the DBM mode also for insulating samples has been applied to PTFE and to different glass samples. Fig. 5 shows a typical SNMS spectrum for PTFE. A number of C_nF_m fragments are found besides atomic C, while atomic F is missing. The SNMS spectrum in Fig. 5 contains interestingly mainly molecular fragments with $n = 4$ and m in the range from $1 - 10$ but without m equal to 5 or 6. This result indicates that the PTFE target contains preformed subunits with 4 C–atoms which selectively survive the sputtering process and also the electron impact postionization in the SNMS plasma. In addition, the $C_{12}F_6$ molecule is also identified as a very stable species.

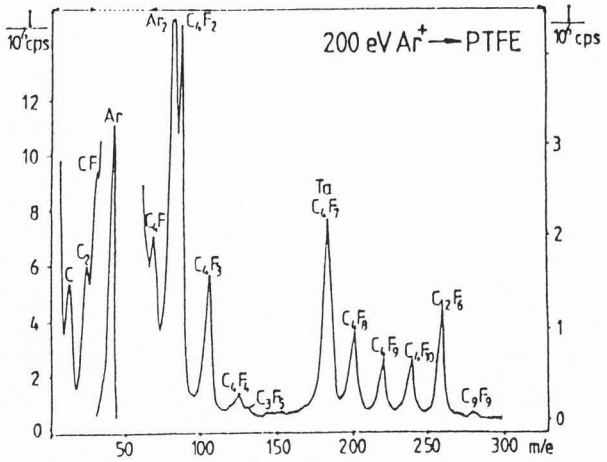

Fig. 5. SNMS spectrum of a PTFE sample (from Kopnarski 1984).

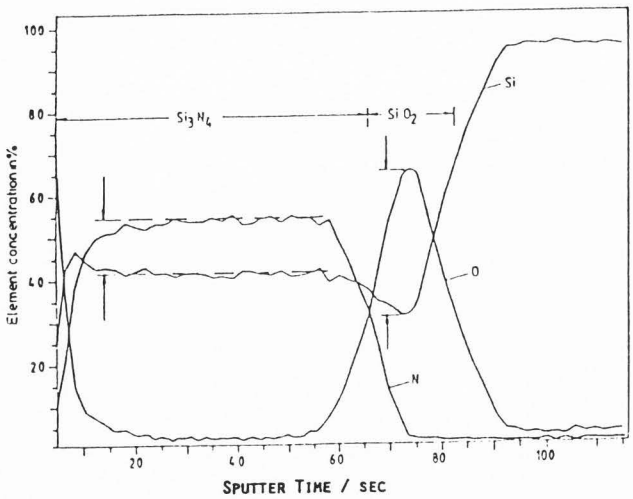

Fig. 6. Evaluated SNMS depth profile of a 70 nm Si_3N_4/30 nm SiO_2 layer system on Si (from Jede and Peters 1987).

As another experimental approach for space charge compensation in the DBM of SNMS a metallic grid is placed immediately upon the target surface (Jede and Peters 1987, Stewart et al 1988). The SNMS–depth profile obtained with this technique for an insulating layer system of 70 nm Si_3N_4 and 30 nm SiO_2 on Si is shown in Fig. 6. The bombarding voltage of 600 eV was applied between the SNMS plasma and the metallic grid placed on the dielectric sample surface. The removal rate under Ar^+ bombardment was around 1.4 nm/sec. Since the molecular contributions to the SNMS signals amounted only to about 10^{-2} in the present case, the evaluation according to the conditions for atomic sputtering (Oechsner 1984 and 1988) has been applied to the raw data of the SNMS depth profile analysis of this layer system. The obtained atomic concentrations in Fig. 6 describe very well the stoichiometry of the sample system.

Analysis of Insulators with the Separate Bombardment Mode SBM

The principle of this operation mode (Müller et al. 1985) is included in Fig. 1. The insulating sample is in direct contact with the SNMS–plasma, and automatically self–biasing to the so–called floating potential. This potential is negative against the plasma environment and determined by the temperature T_e of the plasma electrons and the mass ratio between plasma ions and electrons. For the characteristic operation conditions of the SNMS–plasma the corresponding negative potential drop between the plasma and the target amounts to 15 – 20 V. At floating potential the saturation current of plasma ions and the electron retardation current onto the insulating sample compensate each other precisely.

In the SBM particle ejection from the sample surface occurs by ion bombardment with a conventional ion gun through the SNMS–plasma as diagrammed in Fig. 1. Because of the comparable geometrical arrangement the collection sensitivity for postionized sputtered neutrals is the same in the DBM and the SBM of SNMS.

When the rastered ion beam from the separate ion gun mounted at the opposite side of the SNMS plasma transfers additional positive charge to the insulating sample, the negative floating potential of the sample is automatically decreasing in such a way that the additional ion current from the ion gun is exactly compensated by a corresponding increase of the electron retardation current. A minor disadvantage of SBM is that positive secondary ions which can energetically overcome the well of the floating potential superimpose the flux of postionized sputtered neutrals. Such ions have to be suppressed or eliminated by additional experimental measures. Since the ion energy from the separate ion gun is in general higher than the low bombarding energies established with the DBM, SBM has a reduced depth resolution compared to DBM.

A corresponding SNMS spectrum of a highly insulating SiO_2–BaO–B_2O_3–glass with approximately 30 wt% SiO_2, 47 wt% BaO, 20 wt% B_2O_3 and with minor components of ZnO, Al_2O_3 and SbO_3 is shown in Fig. 7. The SNMS spectrum is not expected to display the undisturbed bulk concentration of the sample, since the surface had been pretreated with a polishing slurry which is known to cause a subsurface region of altered composition. Nevertheless, the main peaks of the SNMS spectrum reflect well the ratio of the main components of the investigated glass (from Müller et al. 1988).

An example for the experimental flexibility of SNMS is given in Fig. 8 which shows an SNMS spectrum of an Al_2O_3 sample. In order to determine impurity concentrations of calcium and potassium with atomic masses interfering with argon, krypton was chosen for operating the SNMS plasma and the separate ion gun in this case. The relatively small O–peak in Fig. 8 is due to the fact that oxygen is mainly ejected as O^-–ions.

SNMS–Analysis with the External Bombardment Mode EBM

In this operation mode, being schematically shown in Fig. 9, the sample is mounted outside the postionizing plasma chamber and bombarded with ions from an external ion gun. The target is separated from the SNMS plasma by an electrical diaphragm which can be

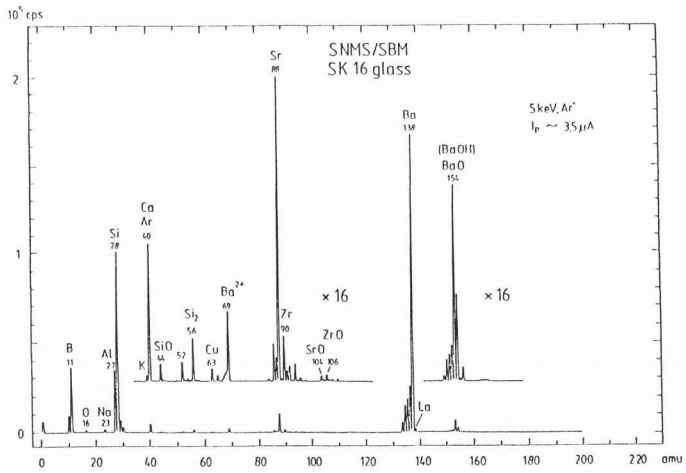

Fig. 7. SNMS spectrum with the SBM mode of an SK16 glass containing 30 wt% SiO_2, 47 wt% BaO, 20 wt% B_2O_3 and minor components of ZnO, Al_2O_3 and SbO_3. An energy window was set for suppression of the low energy particles from the SNMS plasma (from Müller et al. 1988). Sample by courtesy of Dr. H. Bach, Mainz.

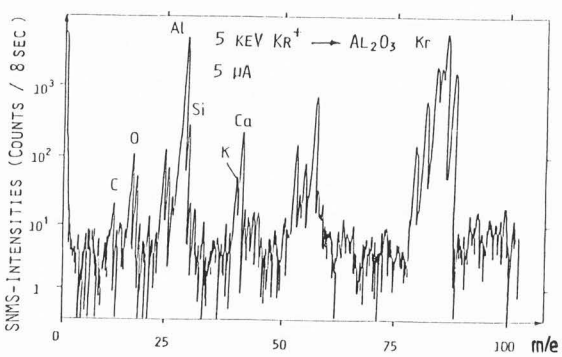

Fig. 8. SNMS spectrum in the SBM mode of an Al_2O_3 sample. Kr was used as working gas in the SNMS plasma and the bombarding separate ion gun for circumventing mass interferences with K and Ca.

opened in an arbitrary direction for charged particles of arbitrary sign.

In EBM the positive ion charge transferred to the sample from the ion gun can be compensated by an additional electron current being extracted from the SNMS plasma through the electrical diaphragm. As shown in Fig. 10 this compensation becomes directly visible by the variation of the SNMS signals from an insulating sample as a function of the potentials applied to the appropriate diaphragm electrodes. As demonstrated by the behaviour of the Ba–signal from a glass sample, no SNMS signal is obtained when the diaphragm is completely closed for charged particles. Then no compensating plasma electrons arrive at the sample and the ion bombardment is interrupted due to sample charging. By appropriate variation of the voltages U_2 and U_3 (see Fig. 10) the SNMS signals appear, because now an electron current from the plasma penetrates to the sample surface. Exact charge neutralization can be readily determined from the break point of the intensity curves in Fig. 10 when saturation is reached.

By this technique thin insulating MgF_2–films of 90 nm on two different glass substrates have been analyzed. The samples were bombarded under 45^0 with argon ions of 2.5, 3.5, and 5 keV, and those particles leaving the surface in the plane of incidence under 90^0 against the bombarding beam enter the postionizing chamber through the electrical diaphragm for subsequent mass analysis. The corresponding results are shown in Fig. 11. Quite surprisingly, the SNMS depth profiles for Mg behave differently for the two substrates when the bombarding energy is changed. The profiles for the different bombarding energies coincide for the Pb–rich glass substrate consisting of 74 wt% PbO and 24 wt% SiO_2. On the other hand the profile tails split up for the substrate with only 0.1 wt% PbO but 69 wt% SiO_2, and additional 11 wt% B_2O_3 and 10 wt% Na_2O. The bombarding time scale is always normalized to the time t_0 for which the interface between the MgF_2–layer and the substrate is reached.

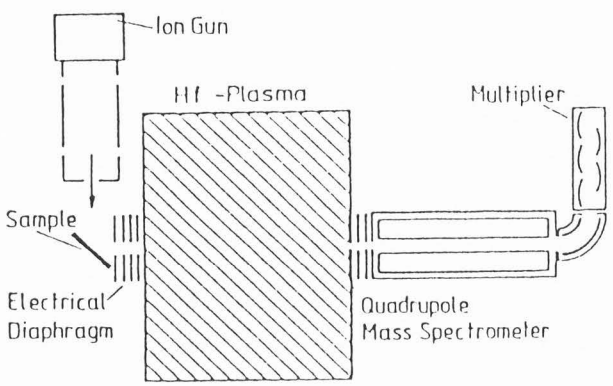

Fig. 9. Schematic diagram of the external bombardment mode EBM of SNMS.

The different behaviour of the results in Fig. 11 has to be ascribed to the different cross–sections for recoil implantation of Mg and for atomic mixing in the two different systems. While the range distribution for Mg implantation in a matrix with the heavy element Pb as the major component is found to depend only a little on the knock–on energy, a clear dependence on the kinetic properties of the Mg particles is found for the low mass matrix. The higher the bombarding energy, the deeper is the range along which Mg from the overlayer is deposited. Besides the energy dependent range of the recoil atoms, the influence of the depth of atomic mixing which decreases with the energy of the primary projectiles becomes clearly visible in this case.

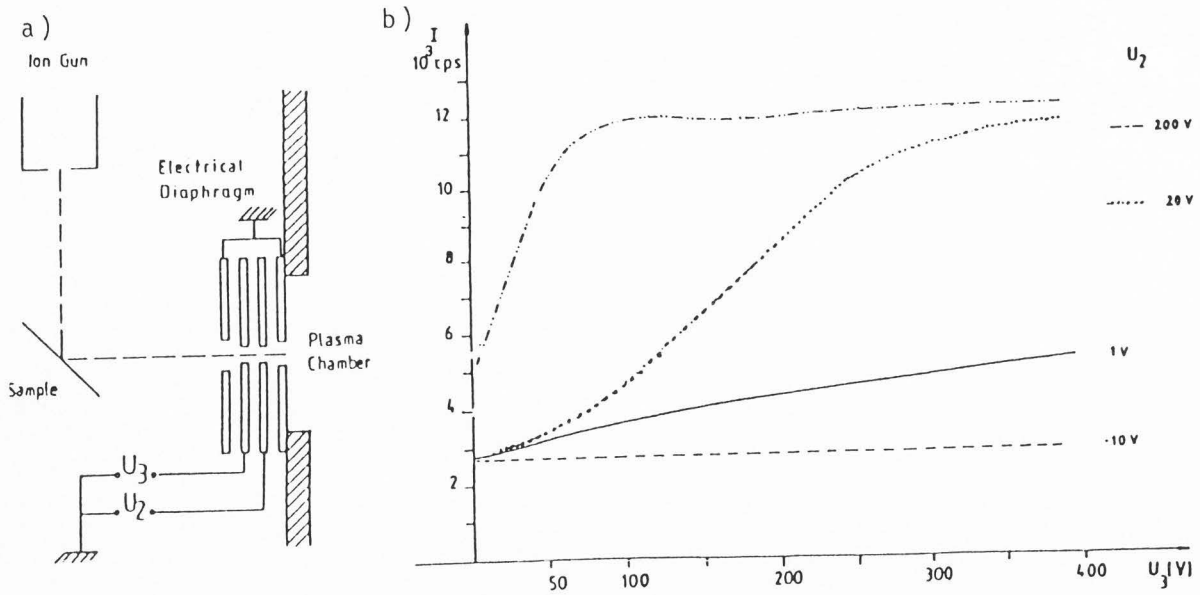

Fig. 10. a) Scheme of the electrical diaphragm applied in EBM.
b) Variation of the ^{138}Ba SNMS signal from a glass sample as a function of the diaphragm voltages U_2 and U_3 (from Geiger et al. 1987)

Conclusions

The two characteristic features of electron gas SNMS, namely the exclusive use of neutral atoms and molecules for analytical purposes, and the availability of a dense electron gas have been shown to make SNMS a very appropriate tool for the analysis of insulating samples and films. When in the Direct Bombardment Mode (DBM) the sample is replaced by an equipotential space system with the insulating sample as the base plate, sample charging can be compensated by potential electron emission from a co–bombarded metallic mask at the sample. Under appropriate conditions the ultimate depth resolution achieved with low energy SNMS for conducting and semiconducting sample systems is expected to be almost conserved also for electrically insulating samples. The Separate Bombardment Mode (SBM) provides a very simple and elegant possibility by which sample charging under different bombarding conditions is automatically compensated by the physical system itself. Finally, the External Bombardment Mode (EBM) supplies a sophisticated possibility for a precise and controllable charge compensation via an electron current through the electrical diaphragm. The neutralization point can be determined with high accuracy. This technique also allows the investigation of physical details for the implantation and mixing processes in highly insulating systems.

As a common feature of these three operation modes, exact compensation of sample charging can be much easier and much more reliably established for SNMS than for other techniques for surface and depth profile analysis. This makes SNMS to the presently most appropriate technique for the quantitative analysis of insulating samples. An additional method for charge compensation which promises the complete transfer of the advantages of the direct bombardment of

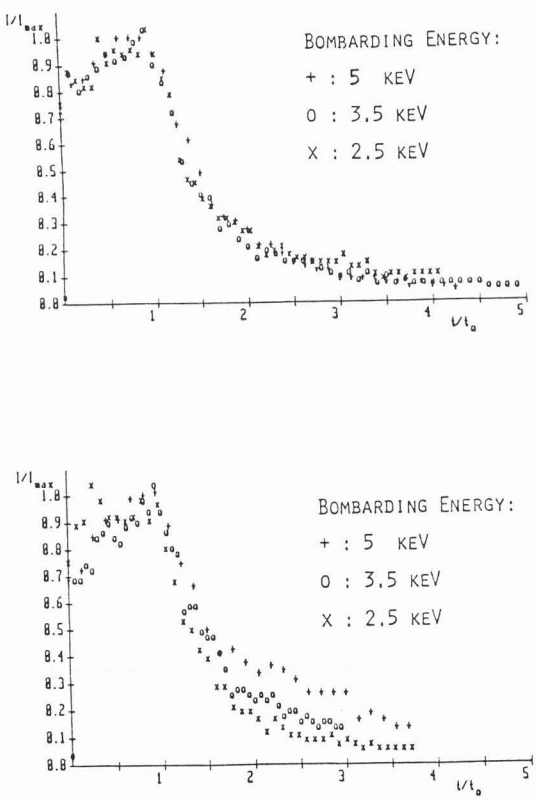

Fig. 11. SNMS sputter depth profiles of Mg for MgF_2 layers of 90 nm on two different glass substrates. The sputter time t is always normalized to the time t_0 for which the interface between the MgF_2 layer and the glass substrate is reached. The measurements have been performed with Ar^+–ions of different energies at a bombarding angle of 45° (from Geiger et al. 1987).

low energy SNMS to insulating samples without the additional measures described above is presently being investigated in the author's laboratory. In this mode a high frequency potential is applied to an insulating sample which is then self–biasing against the SNMS plasma in a controlled manner similar to the target in an rf–sputtering diode.

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Discussion with the Reviewers

A. Vertes: What is the sensitivity of the EBM arrangement and how does it compare with the capabilities of DBM and SBM – SNMS and SIMS? What is the author's opinion on possibilities of improving SNMS sensitivity?

Author: Because of the smaller solid angle by which sputtered neutrals are collected with EBM the detection sensitivity is by $10^{-2} - 10^{-3}$ below that with DBM or SBM. Depending on the geometric and experimental conditions, the useful yield of the 2 latter operation modes of SNMS is in the order of $10^{-7} - 10^{-9}$. For an in–situ comparison of SIMS and SNMS as being possible with EBM and SBM, the detection sensitivity of SNMS is in general above that of SIMS, since the postionization probability in electron gas SNMS is mostly above the secondary ion formation probability. For pure SIMS arrangements the collection efficiency for secondary ions is quite obviously above that of SNMS arrangements due to the much smaller distance between the sample and the spectrometer entrance and the possible application of an ion collecting electrical field.

A. Vertes: What is the reason for the surprisingly broad peaks in Fig. 5? How was it possible to uniquely assign them and how can one cope with H containing compounds?

Author: The (home–made) high transmission quadrupol mass spectrometer was operated at moderate mass resolution in the present case after having made sure that no hydrogen containing fragments appear in the sputtered flux from the PTFE sample. Of course, hydrogen containing compounds can be identified when the mass resolution is increased on account of the very high fragment signals displayed in Fig. 5.

A. Vertes: In Fig. 11 the curves corresponding to the low Pb glass substrate seem to split not only in the tail region but also in the $t << t_0$ domain. If this effect was significant it would indicate surprisingly intense atomic mixing.

Author: In comparision to the systematic behaviour in the tail region it cannot be safely decided whether the mentioned effect is purely due to a scattering of the SNMS signals. However, as for both glass substrates investigated in Fig. 11 the Mg signal increases before the tail region, an accumulation of the overlayer components due to knock–on implantation or bombardment induced diffusion seems possible.

D.S. Simons: Is the unusual stability observed for the C_4F_m fragments from PTFE also seen in conventional SIMS analysis of PTFE under keV argon bombardment, or is it unique to SNMS?

Author: From a comparison with the very scarce SIMS measurements the latter seems to be the case which presumably has to be ascribed to the differences between the formation processes of charged and neutral PTFE fragments.

A.R.E. Lodding: a) Could you comment on the kinetic energy distributions of the secondary ions as issuing from postionization? b) How wide is the energy acceptance window of the mass analyzer usually employed with SNMS, and can the energy position of the window be adjusted? c) In your example of the DBM mode you record a profile through a film of SiO_2, and find that the SiO signal varies as sample potential changes. Can this be due to the energy distribution "moving past" the acceptance window?

Author: a) As demonstrated by our early work on the energy distribution of sputtered neutrals in which the same positionization technique has been employed (Z. Phys. (1970) 238, 433–451), the energy distribution of the sputtered neutrals is not disturbed by the SNMS–plasma. The postionzation probability is inversely proportional to the velocity of a sputtered

particle. b) As usual the energy acceptance window of the quadrupole mass spectrometer varies with the mass resolution being actually chosen. More important, low energy particles can be cut off by an adjustable potential step in our spectrometer entrance optics. c) The behaviour of the SiO_2 depth profiling measurements has not been found to be determined by a variation of the energy window or the applied potential step.

A.R.E. Lodding: What is the "normal" and "maximal" rate of erosion by sputtering in the DBM mode?
Author: With the ion bombarding current density for DBM in the order of $1-2$ mA cm^{-2} the erosion rate can be readily determined from the total sputtering yield which varies typically between 0.1 and 1 atoms/ion in the bombarding energy range employed for DBM. For Ar^+ ions of 250 eV typical erosion rates are 1 monolayer/sec.

A.R.E. Lodding: Are there significant, mechanism–dependent differences in postionization factors as obtainable by either DBM, SBM, or EBM?
Author: No, as far as the postionization probability per particle entering the SNMS–plasma is concerned.

A.R.E. Lodding: Will there be an "imaging SNMS", and if so, when?
Author: The possibility to produce SNMS images has been already demonstrated with a rastered ion gun supplying with a lateral resolution of a few 10 μm. An SNMS microprobe for matrix effect free surface imaging with a lateral resolution below 1 μm and a detection sensitivity below 10^{-3} should be feasible and is just under development in our laboratory.