# MATERIALS CHARACTERIZATION BY AUGER ELECTRON SPECTROSCOPY SPUTTER DEPTH PROFILING

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**Abstract:** Depth profiling by ion sputtering in combination with Auger electron spectroscopy (AES) has become a valuable analytical tool in microelectronics and those areas of research and technology where the chemical composition of surfaces, interfaces and thin films is of importance. Its application range from fundamental surface and interface studies to thin-film structures for electronic and microelectronic devices, wear-and corrosion-resistant coatings, surfaces modified by plasma technique or ion implantation, etc. Reactions at surfaces as well as the resulting electrical and mechanical properties can only be understood if the relevant chemical and structural changes in the investigated thin-film structures are known. This requires a quantitative spatially resolved analysis and in-depth distribution of chemical composition with a high depth resolution. However, ion sputtering is not an ideal layer-by-layer erosion but is the result of a complex ion beam-sample interaction process. Various phenomena, the most important of which are due to ion beam induced changes of surface roughness and composition, limit the experimentally achievable resolution. The principles of the method and its fundamental capabilities and limitations will be discussed. The applicability of the AES depth profiling in microelectronics and some other technical fields will be illustrated by depth profiles of the characteristic samples.

# Karakterizacija materialov z Augerjevo elektronsko-spektroskopsko (AES) profilno analizo

**Ključne besede:** IBS naprševanje s curki ionskimi, karakterizacija materialov, lastnosti materialov, IBS naprševanje s curki ionskimi, AES Auger spektroskopija elektronska-profiliranje globinsko, polprevodniki, mikroelektronika, MBE rast molekularna žarkovna epitaksialna, MOCVD nanosi kemični s paro kovina-snov organska, plasti tanke, strukture večplastne

**Izvleček:** Profilna analiza v kombinaciji ionskega jedkanja s spektroskopijo Augerjevih elektronov je postala zelo uporabno analitsko orodje v mikroelektroniki in na tistih področjih preiskav in tehnologij, kjer je pomembna kemična sestava površin, faznih mej in tankih plasti. Uporablja se za temeljne preiskave površin in faznih mej, kakor tudi za preiskavo tankoplastnih struktur za elektronske in mikroelektronske naprave, za obrabno in korozijsko obstojne prevleke, površine obdelane s plazemsko tehniko ali implantacijo, itd. Reakcije na površinah in z njimi povezane električne in mehanske lastnosti lahko razumemo samo, če so poznane ustrezne kemične in strukturne spremembe v tankoplastnih strukturah. To zahteva kvantitativno prostorsko ločljivo analizo in globinsko porazdelitev kemične sestave z veliko globinsko ločljivost. Pri tem moramo vedeti, da ionsko jedkanje ne predstavlja idealnega primera odstranjevanja plasti za plastjo, ampak je to rezultat kompleksne interakcije ionskega curka s preiskovanim vzorcem. Različni pojavi, od katerih sta najpomembnejša sprememba hrapavosti in sestave, povzročena z ionskim curkom, omejujejo eksperimentalno dobljeno ločljivost. V delu so obravnavani principi metode in njena zmogljivost ter omejitve. Uporabnost AES profilne analize v mikroelektroniki in na nekaterih drugih tehničnih področjih je ilustrirana z globinskimi profili karakterističnih vzorcev.

## 1. Introduction

Advanced thin-films and composite materials based on progress in thin films and interface science /1/. Properties of materials interfaces on the nanometer scale influence on a great number of materials properties and macroscopic phenomena. The resulting properties as well as reactions at surfaces and interfaces, such as oxidation, corrosion, interdiffusion and segregation can only be understood if the relevant changes of the chemical composition at the interfaces and of regions around them are known. The rapid advancement of semiconductor material growth technologies such as molecular-beam epitaxy (MBE) and metalorganic chemical vapor deposition (MOCVD) has greatly improved the microelectronic devices with atomically abrupt interfaces and ultra-shallow doping profiles. In view of the planar structure of thin films, analysis of the in-depth distribution of chemical composition with high resolution is of primary importance. Numerous nondestructive and destructive methods have been developed for this purpose /2,3/. Among the various techniques, surface analysis methods in combination with ion sputtering are most frequently applied because they are applicable to almost any kind of solid material and allow the attainment of concentration depth profiles over a wide depth range from atomic thin films up to several micrometers. The combination of microsectioning by ion sputtering with surface analysis techniques based on Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), is most frequently applied /4/. The main reason is the small depth of information of these techniques of the order of 1 nm, which is a prerequisite for obtaining depth profiles with a high depth resolution. However, ion sputtering is not an ideal layer-by-layer erosion but is the result of a

complex ion beam-sample interaction process. Various phenomena, the most important of which are due to ion beam induced changes of surface roughness and composition, limit the experimentally achievable depth resolution /5,6/. In this work the principles of the AES depth profiling and its fundamental capabilities and limitations will be discussed and illustrated by depth profiles of the characteristic samples.

## 2. AES depth profiling: instrumentation and experimental procedure

Any commercial equipment for AES depth profiling comprises an electron energy analyser, an electron source, an ion gun and the sample stage all mounted in a stainless steel chamber in which an ultra-high vacuum (UHV $\leq 10^{-7}$ Pa) can be maintained (Fig. 1). The regime of UHV prevents the contamination of the sample surface during analysis with reactive gases such as CO, $H_2O$ and $C_xH_y$. Inert gases (e.g. argon, xenon) showing no chemisorption can be tolerated up to pressures that my be several orders of magnitude higher than those of reactive gases /5/.

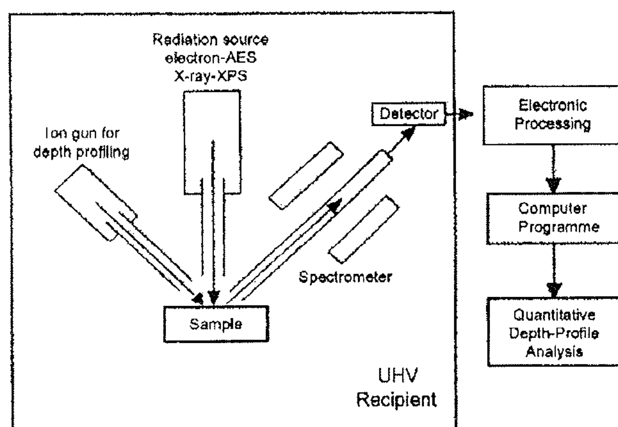


*Fig. 1. Schematic showing the arrangement of instrumental parts of a typical AES (XPS) depth-profile analysis instrument (adapted from Ref. /7/).*

Instrumentation for AES depth profilng requires a source of energetic ions impinging on the sample surface and an AES analysis of the residual surface after a certain sputtering time. Auger analysis can be done either discontinuously after subsequent sputtering steps or by continuous sputtering and simultaneous electron spectroscopy.

The most frequently used ion guns in AES instruments are simple electrostatic devices where the inert Ar ions are generated by collisional excitation with electrons from a hot filament. The positive ions are accelerated to energies between 0,2 and 5 keV and are focused on the sample surface, creating a sputtering spot of typically 1-5 mm in diameter. The pressure in the ion formation chamber should be about $4\times10^{-3}$ Pa. For precise depth profiling, an ion gun with x/y beam deflection capability should be used. This enables the exact matching of analysed and sputtered areas. Rastering of the well-focused ion beam over an area up to 10 x 10 mm greatly improves the uniformity of the ion density and leads to a flat bottom of the sputtering crater which is necessary for optimum depth profiles. To optimize the sputtering conditions with respect to a specific sample topography, a sample stage with both tilt and rotation capabilities was introduced.

## 3. Evaluation of depth profiles

In AES depth profiling a continuous sputtering and simultaneous electron spectroscopy is most frequently used together with multiplexing of different element peaks and a display of the respective Auger peak-to-peak heights (APPH) as a function of the sputtering time. Sputter erosion and surface analysis are complex processes and a variety of experimental factors generally impede a straightforward data evaluation /5/.

The raw data of a depth profiling experiment consists of an elemental signal intensity, I , as a function of the sputtering time, t. The aim of the quantification of a depth profile is to obtain the original in-depth distribution of the composition, c(z), from the measured data, I(t). Therefore the following three fundamental procedures are necessary /7/: (a) conversion of the measured sputtering time t into sputtered depth, z=f(t), (b) conversion of the measured elemental intensity I to the elemental concentration, c = f (I), (c) deconvolution of the measured profile by means of a depth-resolution function (DRF).

## 4. Depth resolution

The aim of any AES depth profiling is to get a depth profile which can be comparable as closely as possible to the original elemental distribution with depth. A measured depth profile generally differs from the true in-depth distribution of composition owing to a large number of distortional effects /5-7/. The factors which contribute to the broadening of the interfaces can be roughly categorized as instrumental factors, effects depending an sample characteristics and the effects of ion beam-sample interactions. The most important causes of profile distortions include original surface roughness and sputtering-induced roughness, atomic mixing, information depth, preferential sputtering and nonuniform ion beam intensity.

A measure of profile broadening is the depth resolution $\Delta z$. By convention, the depth resolution corresponds to the distance over which a 16% to 84% (or 84% to 16%) change in signal is measured /5,8/. The generally accepted definition for the depth resolution is $\Delta z = 2\sigma$, where $\sigma$ is the standard deviation of the corresponding Gaussian resolution function /5/. The depth resolution, $\Delta z$, can be directly determined in the depth profile of a sharp A/B interface as shown schematically in Fig. 2.

All contributions $\Delta z_j$ from different physical phenomena mentioned so far caused the profile broadening. Assuming depth resolution functions of approximately Gaussian shape and mutual independence, these contributions add up in quadrature /5,7/: $\Delta z = [\Sigma (\Delta z_j)^2]^{1/2}$. For the single sandwich layers (A/B/A) and multilayer

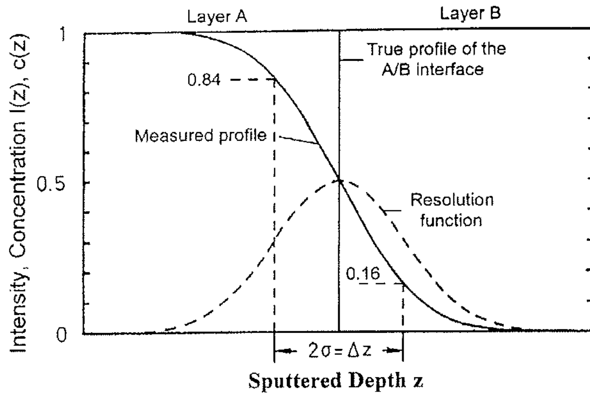


*Fig. 2. Broadening of the measured profile and definition of the depth resolution, Δz (84-16%) ≈ 2σ, for a Gaussian resolution function; σ = the standard deviation (adapted from Ref. /5/).*

structures (A/B/A/B...), Δz can be extracted from the amplitude of the concentration profiles /5/.

The depth resolution of the multilayered samples with atomically flat interfaces is mainly determined by the influence of the following three fundamental parameters: atomic mixing (M), roughness (R) and information depth (I). Recently, this influence is quantitatively described by the so-called mixing-roughness-information depth model (MRI-model) /9/. Optimized depth profiling with Δz ≅ 2 nm can only be achieved if these three parameters are minimized /11/.

## 5. AES depth profiles of selected samples

Multilayer structures are of great importance in the fabrication of semiconductor devices and also have become indispensable as reference materials in depth profiling, particularly for the determination of depth resolution Δz as a function of the sputtered depth /10-12/. Quite often, Δz is degraded mainly by the sample surface roughness, and this occurs both on samples with an initial roughness of the surface /13,14/ and on originally smooth samples with roughness induced by ion sputtering during depth profiling /15,16/. It was shown that the decisive parameter influencing the dependence of Δz on surface roughness is the angular distribution of the differently oriented microplanes /17,18/. Sputtering induced surface roughening is generally caused by local differences in the sputtering rate, because the sputtering yield is dependent on the ion incidence angle with respect to the crystallographic orientation of polycrystalline metallic materials /19/. However, even for homogeneous samples with smooth surfaces, the dependence of the sputtering yield on the angle of ion incidence remains a distortional effect.

The effect of an initial roughness on Δz was studied on two different multilayer structures sputter deposited on polished single-crystal (111) silicon substrates /20/. The first sample (No. 1) consisted of an Ni/Cr multilayer with a total of 16 alternating Ni and Cr layers with a single layer thickness of 30 nm. The second sample (No. 2) had on the top of the Ni/Cr multilayer, an additional crystalline Al layer with an average thickness of ~ 40 nm.

Figure 3 shows the AES depth profiles of two different multilayer structures obtained by ion sputtering of stationary samples by using two symetrically inclined ion

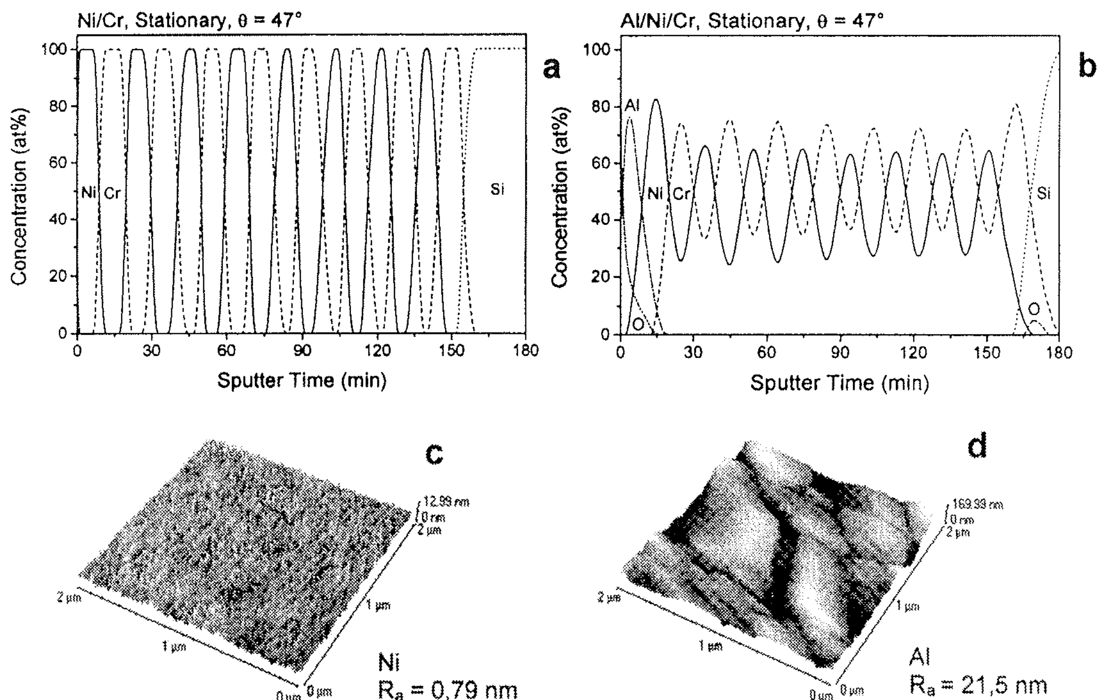


*Fig. 3 a,b,c,d. AES depth profiles (a) of the Ni/Cr and (b) Al/Ni/Cr multilayers obtained by ion sputtering of stationary samples with 1 keV $Ar^+$ ions at an incidence angle of 47°. Atomic force microscopy images showing the surface features of (c) as-deposited Ni and (d) Al surface layers lying on the top of two Ni/Cr multilayer samples /20/.*

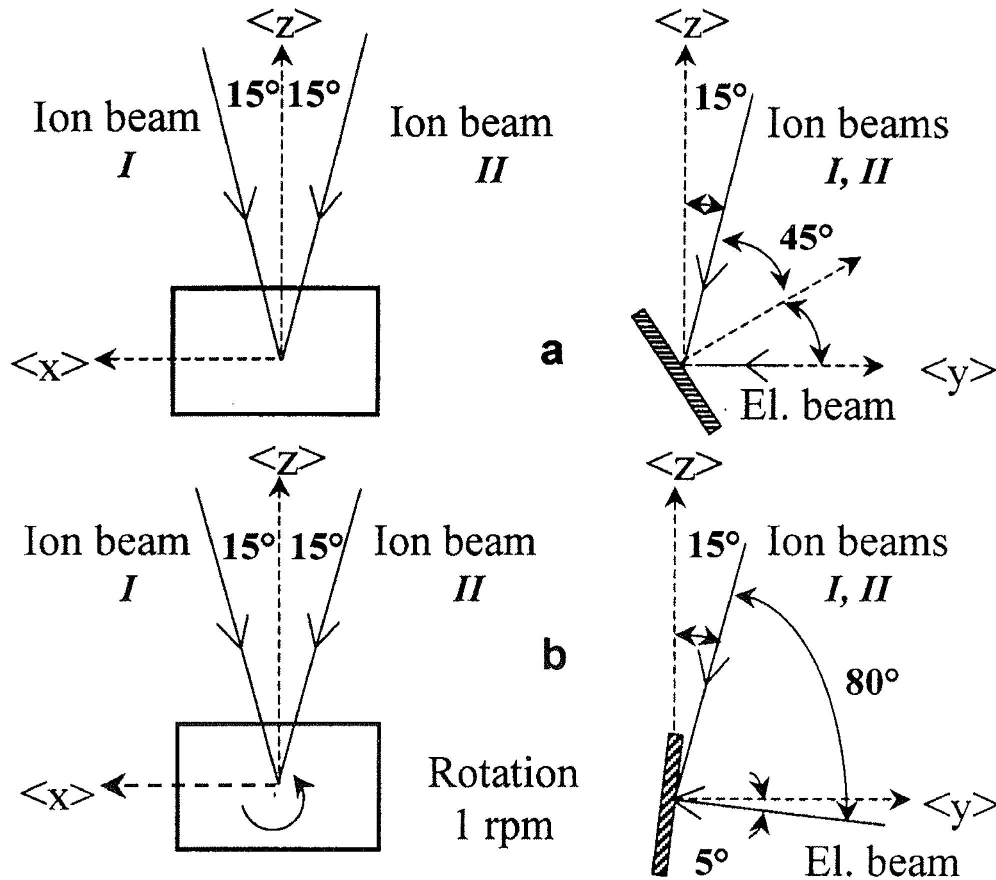


*Fig. 4 a, b. Geometry of the experimental arrangement in rectangular x, y, z coordinates in a plane perpendicular (left) and parallel (right) to the electron beam, respectively; (a) stationary sample, $\theta = 47°$ and (b) sample rotation and grazing incidence ion beams, $\theta = 80°$.*

guns at an incidence angle of 47° (Fig. 4a); note $\theta = \arccos(\cos 15° \times \cos 45°) \cong 47°$. The AES depth profile of the uncovered Ni/Cr multilayer (Fig. 3a) shows relatively sharp interfaces. In contrast, the depth profile in Fig. 3b obtained on the Ni/Cr sample covered with the crystalline Al layer shows a strong broadening of the interfaces. The surface topography of both as deposited multilayers (No. 1 and No. 2) was examined by atomic force microscopy (AFM). The AFM data reveled that the Ni/Cr multilayer has a smooth surface with a roughness amplitude $R_a = 0{,}79$ nm (Fig. 3c) and the Ni/Cr multilayer covered with a crystalline Al layer exhibited a rough surface with $R_a = 21{,}5$ nm (Fig. 3d). Obviously, the rough crystalline Al surface layer caused in-depth topographical changes of the Ni/Cr multilayer and strongly deteriorated the depth resolution of the originally sharp internal interfaces.

The degradation of the depth resolution due to angle-dependent effects like surface roughness and crystalline orientation can be reduced by multidirectional ion bombardment which is achieved by sample rotation during depth profiling /21/. For the Al/Ni/Cr sample the optimized depth profile similarly to that shown in Fig. 3a was reached only with rotational depth profiling at a grazing incidence angle of about 80° (Fig. 4b). Multidirectional ion bombardment during rotational AES depth profiling at grazing incidence angle removes the crystalline orientation effect, promotes the smoothing effect and decreases the microroughness of the initially rough Al/Ni/Cr sample surface /20/.

It is often necessary to determine the layer composition or interfacial contamination within metallization structures such as vias and contacts during semiconductor device characterization or failure analysis. Compucentric rotation of the sample during ion sputtering allows the acquisition of depth profiles on specific features which do not reside directly over the stage rotation axis /22/.

Aluminum, a common conductor used in semiconductor devices, is particularly prone to cone formation during sputter etching. Fig. 5a shows as-received circuit and Fig. 5b shows an Al via before AES depth profiling. Figs. 5c,d show the topographies obtained at the Al vias after depth profiling without (Fig. 5c) and with sample rotation (Fig. 5d) during sputter etching. In Fig. 5 are shown the corresponding AES depth profiles obtained without (S) and with (R) sample rotation. The sample rotation prevent the cone formation (Fig. 5c) and the depth profile (R) has a much sharper Al/Si interface, which permits a better estimation of possible interdiffusion or reaction between layers.

As-recieved circuit

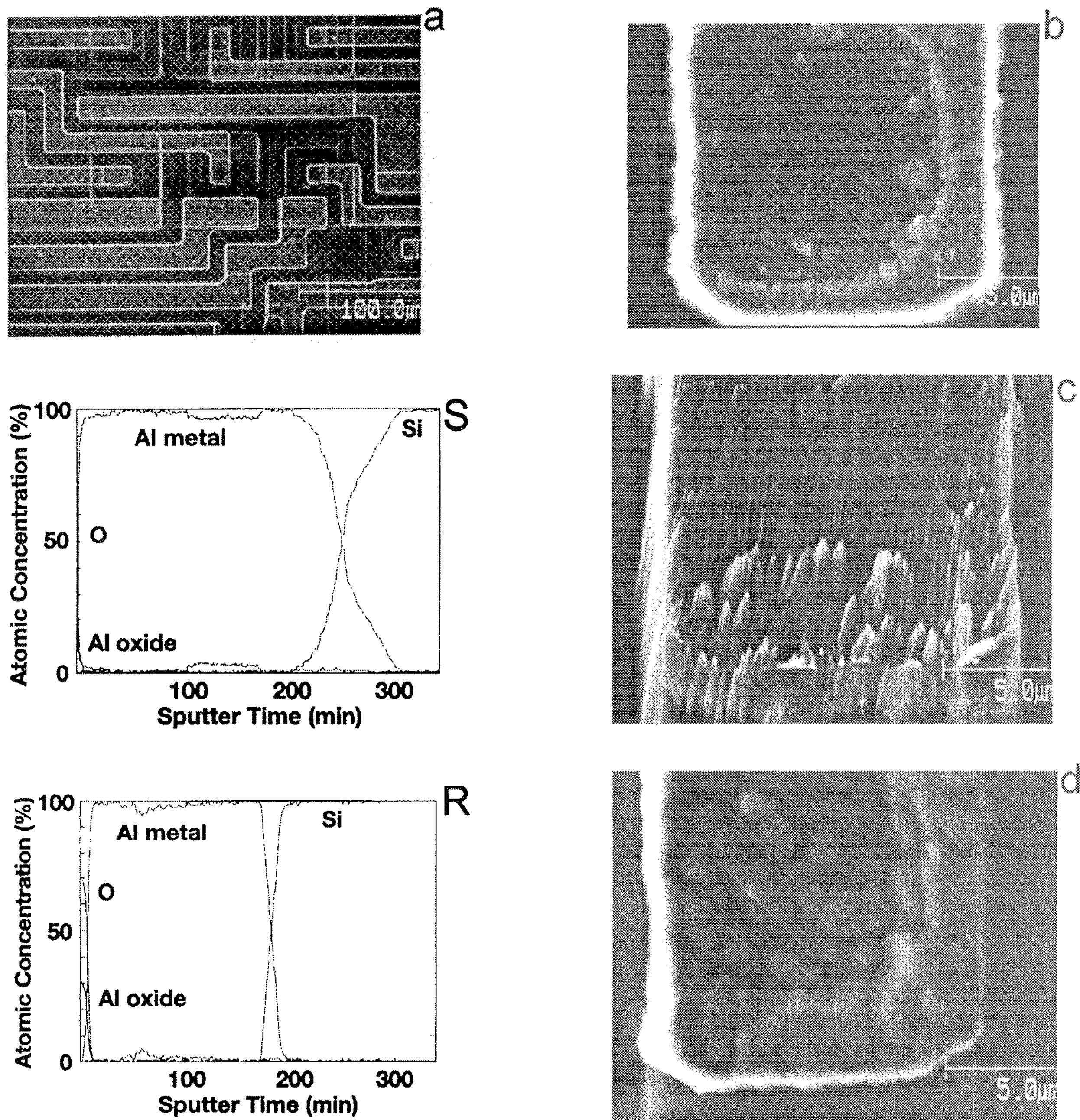


*Fig. 5 a, b, c, d. Secondary electron images show: (a) as-received circuit, (b) an Al via before ion etching, (c) an Al via after sputter depth profiling without and (d) with sample rotation. AES sputter depth profiles obtained without (S) and with (R) rotation of the sample are shown too /22/.*

The sample rotation during depth profiling was introduced also in other surface analytical techniques such as photoelectron X-ray spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS). The desire to provide hard disk media with higher storage density has caused the development of magnetic media with more complex multilayer structures with different individual layer thicknesses. In Fig. 6 are shown the components of a typical hard disk /23/. XPS depth profiling has been routinely applied to thin film systems up to a few hundred nanometers in thickness. The modern ion guns and the ability to change sputter rates, under computer control, during a depth profiling make it possible to efficiently depth profile thicker films. In Fig. 7 is shown a sputter depth profile through all the layers on the hard disk, approximately 14 $\mu m$ in thickness /23/. During depth profiling of the thinner layers the ion gun was set at 2 kV and the sputter rate was approximately 6 nm/minute. For the thicker layers, the ion gun was set to 4 kV and adjusted for a sputter rate of 46 nm/minute. This type of flexibility allowed one depth profile to be used to collect detailed information from the thin mag-

netic layers and the thick NiP layer in a reasonable amount of time. Additionally, the spectra from the detailed depth profile made it possible to examine the chemistry at interfaces and within layers /23/.

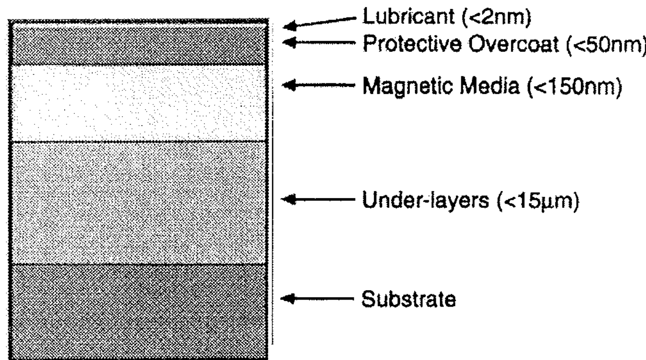


*Fig. 6. The scheme of the multilayer structure of a typical hard disk /23/.*

The preparation of well-characterized silicide thin films for microelectronics needs a control of interfacial reactions and diffusion processes during heat treatment of metal semiconductor systems. The precise observation of AES depth profiles at interfaces and quantification of the interface width of heat treated samples enables the determination of the main moving species in the early stage of reactions, the calculation of the effective interdiffusivity and the characterization of the new formed reaction products /24,25/.

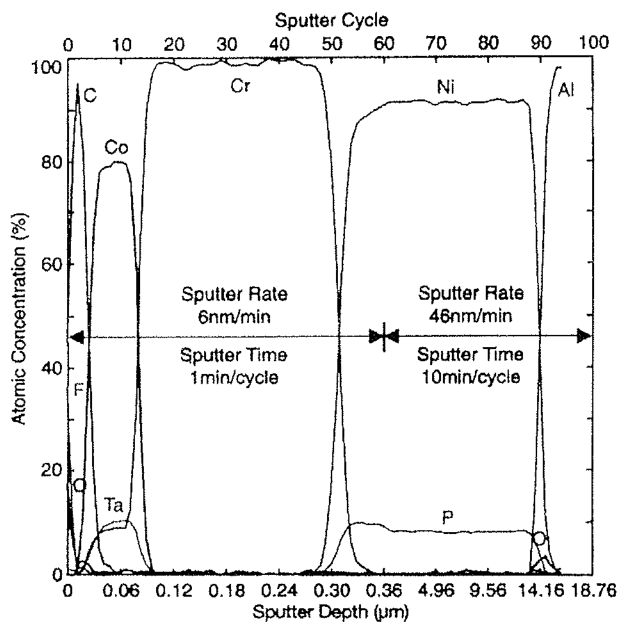


*Fig. 7. Sputter depth profile of the thin lubricant film, the thin magnetic layers and the thick underlayers obtained by programming a change in etch rate and sputtering time during the depth profiling /23/.*

Figure 8a shows the sandwich structure of Si(33 nm)/Ni(50 nm)/Si(33 nm), with a total thickness of 116 nm which was sputter deposited onto smooth silicon (111) substrate. The reaction of Ni with amorphous silicon thin films was activated in a differential scanning calorimeter (DSC), at heating rate of 40 °C $min^{-1}$. Already with a heating of the Si/Ni/Si sample from room temperature to 320 °C the reaction between Si and Ni layers is almost accomplished (Fig. 8b), and the reaction product shows a composition close to $Ni_3Si_2$. However, the selected area diffraction pattern of this cross-sectioned sample showed the presence of two polycrystalline and amorphous phases, namely $Ni_2Si$ and NiSi /24/. The mixture of these two phases is in agreement with an Ni/Si ratio of 3:2, recognized in the AES depth profile in Fig. 8b. The results show the importance of compositional and structural data obtained with different analytical techniques for the precise characterization of reaction products.

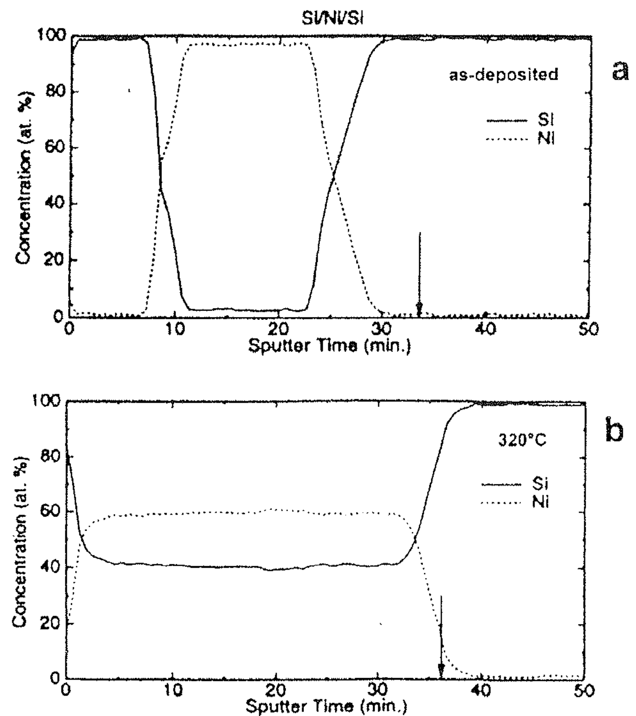


*Fig. 8 a, b. AES depth profiles of Si/Ni/Si sandwich layer: (a) as-deposited and (b) after dynamic heating in a DSC instrument at a rate of 40 °C $min^{-1}$ from room temperature to 320 °C.*

## 7. Conclusions

Compositional depth profiling of thin films and multilayer structures by AES in combination with ion sputtering is the most frequently applied method to obtain the in-depth elemental distribution. A precise quantitative depth profiling requires a quantification of both the analysed elemental signal intensities in concentration units, and the sputtering time in a depth coordinate. The large number of physical parameters, depending on

sample characteristics, ion beam-sample interactions, and instrumental factors, lead to a broadening of a measured profile. A measure of profile broadening is the depth resolution $\Delta z$. By convention, the depth resolution is $\Delta z = 2\sigma$, which corresponds to the difference of the depth coordinate between 84 and 16 % of the intensity change at an interface. Depth resolution in depth profiling of the multilayer structures with atomically flat interfaces is determined by three parameters: atomic mixing, surface roughness and information depth and their influence is quantitatively described by the MRI-model. Optimized depth profiling with $\Delta z \cong 2$ nm can only be achieved if these three parameters are minimized /11/. Quite often, on technological samples $\Delta z$ is degraded mainly by the sample surface roughness. The degradation of the depth resolution due to angle-dependent effects like surface roughness and crystalline orientation can be reduced by multidirectional ion bombardment. As illustrated by AES depth profiling of the Ni/Cr and Al/Ni/Cr multilayer samples, and by the Al/Si metallization structure of a semiconductor device, the sputtering of the rotated sample at large ion incidence angle and relative low ion beam energy ($\leq$ 1 keV), promotes the smoothing effect and decreases the microroughness of the initially rough sample surface, thus enabling us to achieve the improved depth resolution. Further, the applicability of the depth profiling is illustrated by the depth profile of the multilayer structure of a typical hard disk and by the characterization of reaction products in the heat treated Si/Ni/Si thin-film structure.

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