

Solid state mass spectrometry for materials science

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Abstract - High technology is the key to industrial development. It is largely based on material science. Development and production of materials with new or improved properties pose new challenges for Analytical Chemistry : Ultra trace analysis, micro and nano distribution analysis, surface analysis as well as three-dimensional stereometric analysis are very important for materials research. In this paper the significant contributions of solid state mass spectrometry are discussed.

ULTRA-TRACE BULK ANALYSIS

The development of pure and ultrapure metals is of great significance for developing materials with optimal properties, e.g. high electrical and thermal conductivity. The analytical problem is then to achieve multi-element ultra trace methods for the pg g^{-1} to ng g^{-1} range. Methods providing very high detection power and minimum chemical interferences (e.g. contamination) are required. Among the direct physical techniques SIMS and GDMS offer a great potential [1]. Among these techniques, SIMS is the most versatile because signal generation can be optimized by choosing various reactive or unreactive primary ion species (oxygen, caesium, argon, etc.) as well as sputtering conditions (primary beam energy and density); and additional information can be obtained about the homogeneity of a material through ion images and depth profiles.

Quantitation is achieved by means of relative sensitivity factors (RSF) obtained from measurements of matrix matched reference materials [2].

Ultrapure refractory metals like W, Mo, Ta are used in the microelectronics industry as sputter targets to prepare silicide electrode lines in VLSI devices. The specifications regarding purity include : concentration of each alkali element and of U and Th $< 1 \text{ ng g}^{-1}$, concentration of metal impurities (e.g. Fe, Cr, Cu) $< 10 \text{ ng g}^{-1}$. These trace elements can cause malfunction of a transistor cell through diffusion (such electrode lines carry current densities of about 1 million Acm^{-2}) or emission of α -radiation. The detection power of SIMS for multi-element trace analysis of such refractory metals is in the order of pg g^{-1} to ng g^{-1} for most elements, if both positive and negative secondary ions are measured (through bombardment with O_2^+ and Cs^+) (Table 1).

TABLE 1 : DETECTION LIMITS IN ng g^{-1} FOR ULTRA TRACE ANALYSIS FOR MOLYBDENUM BY GDMS AND SIMS. FOR SIMS THESE HAVE BEEN EXTRAPOLATED FROM SIGNALS MEASURED FOR IMPURITY CONCENTRATIONS IN THE LOW ng g^{-1} RANGE UNDER CONDITIONS ELIMINATING SPECTRAL INTERFERENCES.

	GDMS	SIMS		GDMS	SIMS
Al	0,5	0,004	Ni	0,9	11,0
B	0,2	2,0	Sb	0,4	12,0
Ba		0,3	Si	0,5	0,2
Ca	7,7	0,003	Sn	2,1	
Co	0,2	4,0	Sr	23,0	0,17
Cr	0,6	0,3	Ta		13,0
Cu	3,3	13,0	Th	0,5	7,0
Fe	2,6	0,8	Ti		10,0
K	3,6	0,0007	U	0,5	
Mg	0,6	0,01	V	2,3	0,3
Mn	0,2	0,5	Y	29,0	0,3
Na	0,5	0,0007	Zr	36,0	0,9

The accuracy of the quantitative results is in the order of 50 % rel. [3]. GDMS offers similar figures of merit, but has the advantage that non-matrix matched reference materials can be used for quantitation. Both methods open up a new domain in the analysis of ultra pure materials and due to their large detection power. The major drawback of these techniques is their small sample consumption in analysis (typically 1 – 10 μg per element). This may cause significant problems with the representativity of results when inhomogeneous materials are analyzed. Therefore such ultra-trace analyses of metals should include statistical data on the materials homogeneity as obtained from repeated analyses with one method, or better application of GDMS and SIMS. For inhomogeneous materials chemical dissolution of larger amount (e.g. 1g) with subsequent elemental analysis including enrichment of ultra-trace components is necessary [4]. ET-AAS, ICP-OES and ICP-MS are the techniques of choice. All chemical steps should be carried out in cleanrooms using ultra pure reagents.

MICRO AND NANO DISTRIBUTION ANALYSIS

Micro and nano distribution analysis is obtained by recording ion micrographs or step scan profiles with a finely focussed ion beam. These techniques are particularly valuable for the characterization of trace elements at interfaces in heterogeneous materials, e.g. composites. Trace elements can influence the mechanical properties strongly if enriched at the interface. Often the question is posed for the mechanism of the influence of the trace elements which demands for an analytical characterization with high spatial (lateral) resolution and high detection power. Unfortunately these two analytical figures of merit have an inverse relation with each other : an increase in spatial resolution decreases the detection power due to the smaller number of atoms sputtered. Ways have to be sought which allow to generate analytical ions with a maximum useful yield.

In some cases this can be done by applying a chemical surface modification of the material during analysis : e.g. for distribution analysis of trace amounts of phosphorus in a two-phase W-NiFe alloy a molecular ion PO_3^- can be generated by using primary oxygen ions in combination with oxygen flooding of the sample surface which is nearly 1000 times more intense than the atomic ion P^- [5]. Using a lateral resolution (diameter of analytical area in each point of the step scan profile) of ca. 2 μm only about 10^9 atoms are sputtered in each location. Generation of PO_3^- yields a useful yield for the determination of phosphorus of about 10^{-3} . Consequently a detection limit of roughly 1 $\mu\text{g/g}$ can be achieved for a microanalytical area of 2 μm in diameter. With this technique it was then possible to study the segregation of phosphorus to the grain boundaries between W and NiFe (binder phase). Fig. 1 shows these lateral distribution profiles for a material showing embrittlement. It is evident that there is a strong segregation of phosphorus to the grain boundaries causing this effect. It must be noted that information about the phosphorus species present can not be obtained by SIMS. TEM however allowed to determine that phosphorus is enriched at the grain boundary as nanoprecipitates of NiP_2 with a typical diameter of 5 nm [6]. This also shows that quantitation of the SIMS signals in the binder phase yields only an average content which is not representative for the nanodomains.

At the present state of development the practical lateral resolution of SIMS is limited to about 100 nm although 20 nm primary ion beam diameters have been applied [7]. For such high resolution analysis only the very sensitive elements yield signals for the trace level. Postionization of neutrals – e.g. by photons – may be feasible way to increase the useful yield in nanoanalysis [8].

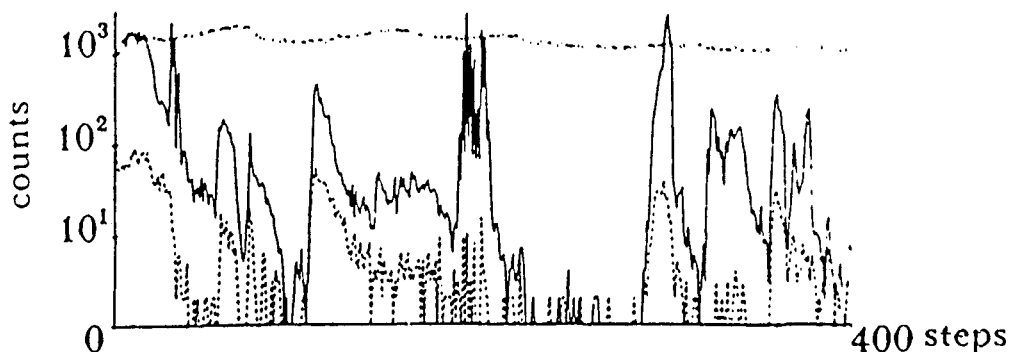


Fig.1: Microdistribution analysis of phosphorous in W-NiFe alloy. Step scan profiles of P measured as PO_3^- (—) and matrix elements measured as WO_3^- (....) and FeO_2^- (- - -). Sample sintered and slowly cooled, causing segregation of phosphorous to the grain boundaries [5].

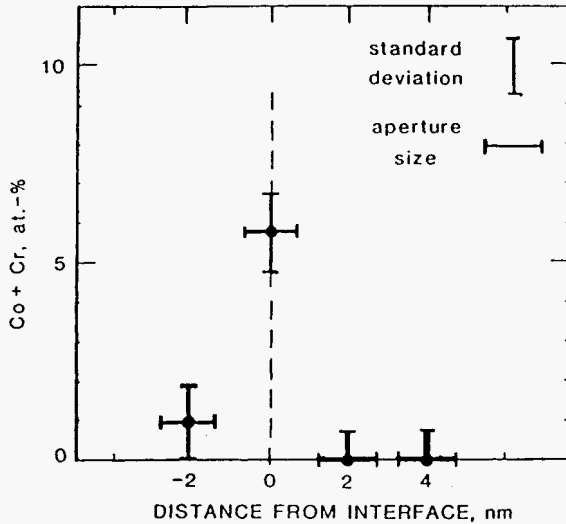


Fig.2:

Atom probe analysis : concentration profile of Co + Cr across the grain boundary WC/WC in a hard metal sample. Spatial resolution = 2 nm [9].

High technology requires access to even smaller dimensions and quantities, with the ultimate goal of characterizing single atoms in terms of chemical identity and spatial position (atomic resolution). Atom probe microanalysis via field ion microscopy seems promising. Through nanosecond voltage or laser pulses, sample material (shaped to a tip) is desorbed and ionized. Identification and quantitative data for the species are obtained by time-of-flight measurements combined with single ion detection. Spatial resolution is achieved by use of the imaging properties of the field ion microscope which allows ions to be extracted from a specific atomic position by means of a movable aperture in the imaging screen [9,10].

Fig. 2 shows as an example the concentration profile across a grain boundary WC-WC in a hard metal [9]. The diameter of the analytical volume of each point is only 2 nm. Quantitation is performed by simply counting the ions since the ionization yield is 1 for all atoms. In this example quantitative results for the trace elements Co and Cr found at the interface were obtained from measurement of only 20 ions in total.

The atom probe can not only perform quantitative nanoanalysis, but also two- and three-dimensional elemental distributions with a lateral resolution of less than 1 nm can be obtained using time-gated imaging devices and sequential removal of the top atomic layer of the material [10]. Fig. 3 shows as an example the three-dimensional representation of a nanoprecipitate in steel [10].

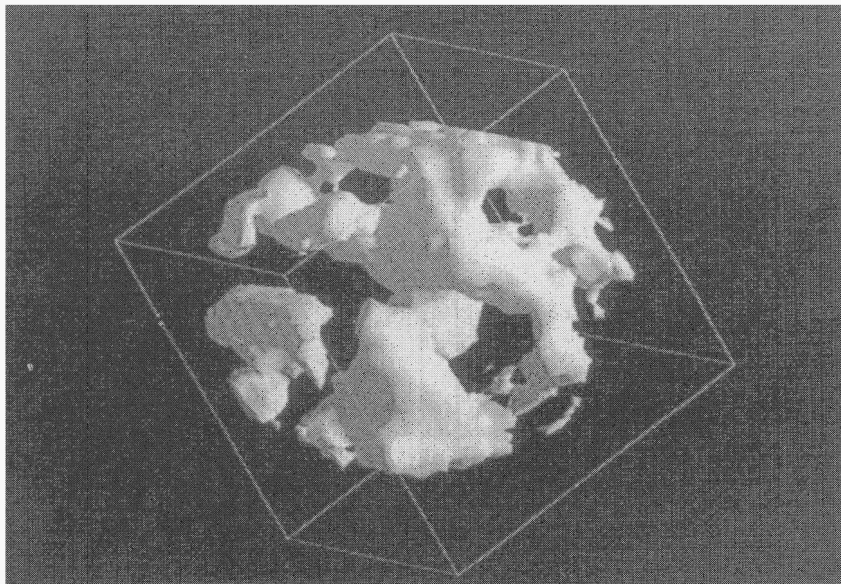


Fig.3: Three-dimensional image of a nanoprecipitate [ferrite] in stainless steel. The contours represent iso-concentration lines. Length of cube = 15 nm [10].

Such type of investigations are of great significance because the determination of trace elements at grain boundaries and interfaces will allow to obtain more information on the mechanism of the influence of trace elements on properties.

SURFACE ANALYSIS

In surface analysis the composition of a material is characterized with maximum spatial resolution perpendicular to the surface. Different optimized measurement techniques are used, depending on the spatial domain to be characterized: static secondary ion mass spectrometry for the selective analysis of the outermost atomic or molecular layer, quasi-static depth profiling with sputter rates of less than 1 atomic layer per second allow the characterization of extremely thin surface layers, dynamic depth profiling enables to characterize the depth distribution of trace elements up to micrometers. Special problems arise in the characterization of interfaces due to artefacts generated by the primary ion bombardment.

STATIC SECONDARY ION MASS SPECTROMETRY

In static SIMS the sample is exposed to an extremely low primary ion fluence (typically 10^{11} ions per cm^2). Thus only a fraction of the top monolayer is removed by sputtering and the original atomic and molecular structure is not disturbed by the primary ion impact. Static secondary ion mass spectra contain information on molecular structure, orientation of molecules, chain length of polymers etc. The unique feature is that this important information can be obtained from the top monolayer, while other molecular probes exhibit an information depth up to μm only.

The development of sophisticated TOF (time-of-flight) mass spectrometers exhibiting high mass resolution has prompted a significant and rapid development of molecular surface analysis [11].

Fig. 4 shows as an example the negative secondary ion mass spectra of a polycarbonate film: original surface (a), after O_2 -plasma treatment (b), and after O_2 -plasma treatment and rinsing with water (c) [12]. The changes in chemical surface structure in the outermost monolayer of the material are clearly observable and demonstrate the high information content of such spectra.

Molecular surface analysis is rather in an initial state with exciting new developments certainly ahead. It is extremely important for many areas of high technology, like the development of polymers with defined surface properties, adhesion, lubrication, interface structures in composites.

QUASI-STATIC SECONDARY ION MASS SPECTROMETRY

Quasistatic SIMS can be applied for trace element analysis. Trace element analysis in the nm-depth domain is of particular significance in the production of silicon wafers. The trace elements originate mainly from surface preparation (cutting, polishing, cleaning etc.) procedures and may influence the growth of the thermal oxide or be a source for detrimental metallic contamination in a VLSI-chip.

The basic problem of trace analysis in such thin surface layers is the low amount of material available for analysis.

For sector field instruments optimized "quasistatic" measurement techniques had to be developed for the different elements to achieve the optimum between (high) detection power and (low) sample consumption per data point [13], since the amount of material available in analysis is very low: in 1 atomic layer only 10^{15} atoms ($\sim 10^{-8}$ g) are present per cm^2 . At a typical analytical area of 10^{-4} cm^2 only 10^{11} atoms are removed. This means for a relative detection limit of 1 $\mu\text{g/g}$ only 10^5 analyte atoms are sputtered. Only high efficiency measurement techniques with a useful yield in the order of $10^{-1} - 10^{-4}$ provide a sufficient detection power. These figures of merit must be achieved at a high mass resolution power to separate the analytical ions from the interfering hydrocarbon species which are present on the surface of every wafer.

For high performance SIMS sector field instruments the practical detection limits for a sample consumption of ca. 10^{10} atoms is about $1\mu\text{g/g}$ for aluminium (which serves as a tracer element for contamination). This corresponds to an absolute detection power of $5 \cdot 10^{-19}$ g, or ca. 10^4 aluminium atoms, and to a surface coverage of ca. 10^9 atoms/ cm^2 [13].

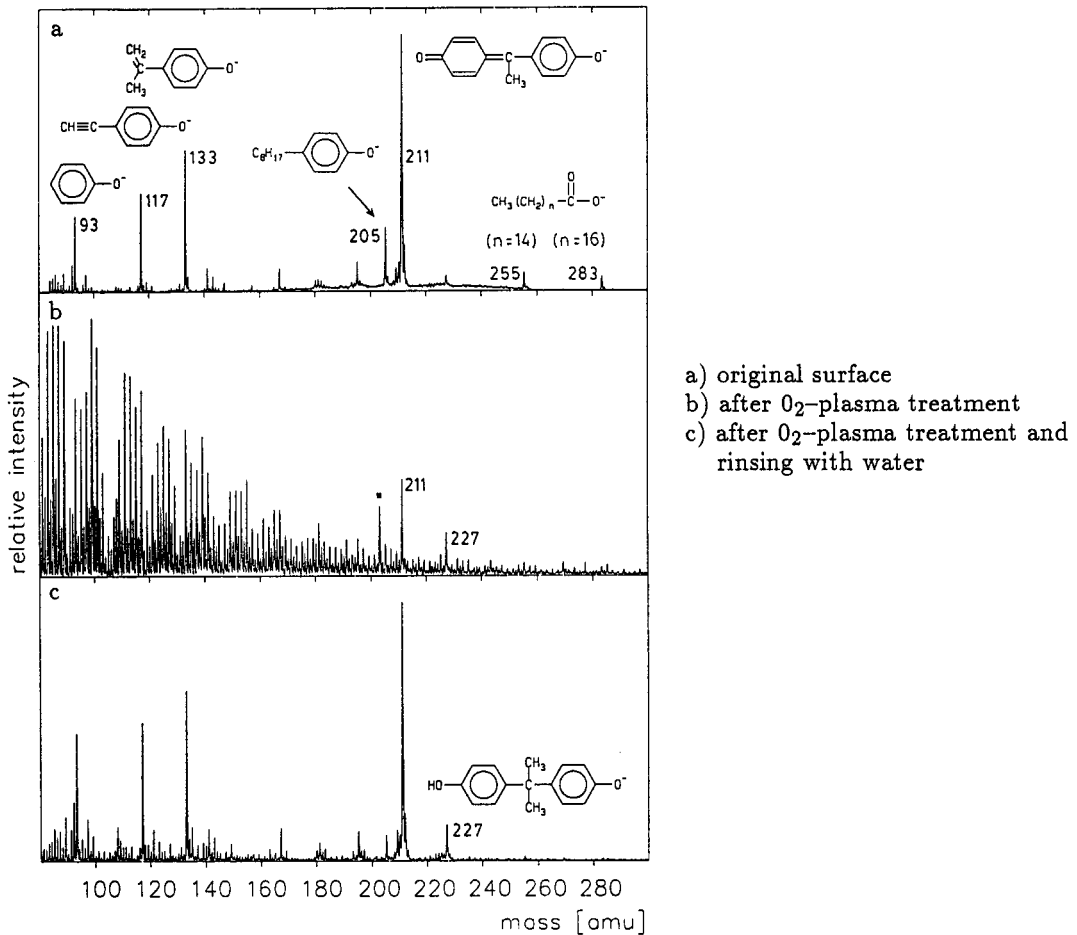


Fig.4: Static secondary ion mass spectra (negative secondary ions) of polycarbonate film [12]

Other elements which must be measured at a higher mass resolution like Fe are about a factor 100 less sensitive. An increase in detection power can be achieved by using SIMS techniques based on high resolution time-of-flight (TOF) mass spectrometers [14]. In general the minimum detectable surface coverage is in the range of $10^8 - 10^9$ atoms/cm².

Laser resonance ionization SNMS can still lower the detection limits due to the possibility of selective and highly efficient ionization of atoms by a multiphoton absorption process. This technique is particularly useful for elements which have a low ionization yield and which exhibit strong interferences, like Fe. By this technique a detection limit for Fe of about 2 ng/g with a material consumption of about 1 monolayer can be obtained [15]. This corresponds in absolute amounts to ca. 40 iron atoms sputtered during measurement of 1 data point or a minimum detectable surface coverage of 10^6 atoms/cm².

Systematic investigations of the surface contamination of wafers – performed mainly with SIMS – have shown that these trace elements are concentrated in the native oxide and that their content depends strongly on the type of cleaning procedure [13].

DYNAMIC SURFACE ANALYSIS

The major goal of dynamic surface analysis is quantitative distribution analysis with a high analytical accuracy. This means in the first instance consideration of the matrix and selective sputter effects, then combination of various analytical techniques to study and reduce systematic errors and to increase the information content of the analysis. Different problems and consequently analytical procedures occur for (laterally) homogeneous (like semiconductors) or heterogeneous materials (like most metals).

TABLE 2 : ANALYTICAL FIGURES OF MERIT FOR DEPTH PROFILING OF DOPANT ELEMENTS IN SILICON WITH SIMS (DATA OF TECHNICAL UNIVERSITY OF VIENNA, OBTAINED WITH CAMECA IMS 3f).

dopant	primary ion species	secondary ions	detection limit [cm^{-3}]	[ppba]	inaccuracy
B	O_2^+	$^{11}\text{B}^+$	$1 \cdot 10^{14}$	2	$\pm 5 - 10 \%$
P	Cs^+	$^{31}\text{P}^-$	$1 \cdot 10^{15}$	20	$\pm 20 \%$
As	Cs^+	$^{75}\text{As}^{28}\text{Si}^-$	$1 \cdot 10^{14}$	2	$\pm 10 \%$
Sb	Cs^+	$^{121}\text{Sb}^{28}\text{Si}^-$	$4 \cdot 10^{13}$	1	$\pm 20 \%$
		$^{123}\text{Sb}^{28}\text{Si}^-$	$3 \cdot 10^{14}$	5	$\pm 5 - 10 \%$

In the study of semiconductors one of the major areas of interest is the characterization of the distribution of the dopant elements within surface layers of typically less than $1 \mu\text{m}$ [16].

The major requirements to be fulfilled are large dynamic range of analysis and high detection power (concentration range ca. $10^{14} - 5 \cdot 10^{21} \text{ atoms cm}^{-3}$); large spatial (depth) resolution (a few nanometres); high accuracy of analytical information (concentration vs. depth).

With optimized analytical conditions detection limits for distribution analysis are obtained which are in the range between $4 \cdot 10^{13} \text{ cm}^{-3}$ and 10^{15} cm^{-3} (Table 2). The analytical accuracy is sufficient for the characterization of the redistribution of dopant elements taking place during the various production steps for microelectronic devices [17].

It must be emphasized however that these processes are studied not in devices but in non-structured model specimens, since the required figures of merit cannot be achieved at sub-micrometer lateral resolution as would be required for today's highly integrated devices (e.g. the 4 MB DRAM). The highly accurate depth profiles obtained from specimens subjected to the various technological steps (e.g. oxidation, implantation, annealing) allow to establish physical models which enable the calculation of the distribution (x, y, z) of dopants in actual devices [18,19]. In order to achieve a sufficient accuracy of the models and a substantial range of applicability also "small effects" have to be characterized. For these - (as shown in Fig. 5) the ultimate accuracy of SIMS in concentration and depth scale is required. The

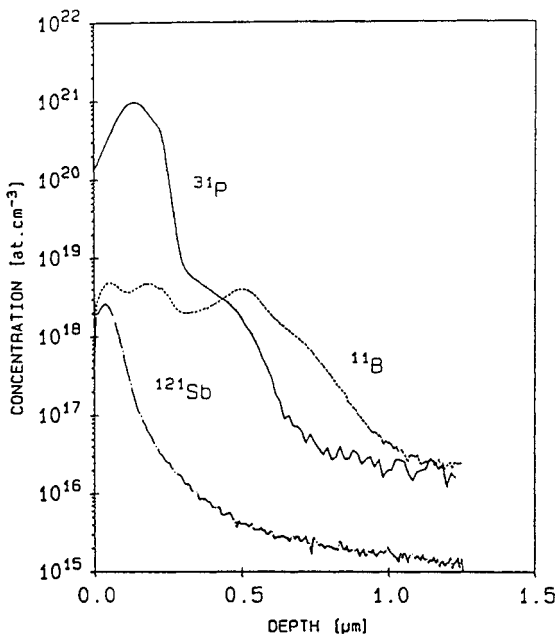


Fig.5: Quantitative depth profiles of P, B and Sb in silicon after annealing. High accuracy measurements for multielement depth profiling allow to exhibit small but significant effects in redistribution of the dopant elements, like the small peak in the profile of B at 230 nm depth corresponding to a 'kink' in the profile of P [20].

values achievable for the concentration scale are contained in Table 2. Depth calibration is made by crater depth measurements using largely stylus techniques. An accuracy of several nanometers can be obtained.

Fig. 2 shows as an example for high accuracy depth distribution analysis the profiles of Sb, B and P in silicon after annealing. At a depth of 230 nm the boron distribution exhibits a slight enhancement. It corresponds to a kink in the phosphorous profile. With TEM a distinct defect band can be observed precisely at 230 nm distance from the surface. Therefore it can be concluded that boron is segregated at the dislocation band. The kink in the phosphorus profile indicates also the redistribution of this element near the dislocation band. It is also suspected that P-B complexes are formed [20].

SIMS being the major technique for depth profiling of trace elements is applied for many different material systems : dopant elements in III-V and II-VI semiconductors [21]; impurities in thin layer systems [22]; corrosion processes of metals [23]; ion exchange phenomena in glass [24]; influence of trace elements on quality of coatings (ceramic, diamond, etc.) [25]; surface modification of materials by chemical reactions, ion implantation, etc. [26]

For these tasks SIMS is frequently combined with EPMA, SEM, XPS, AES, RBS, TEM, and other techniques.

For the study of fundamental processes in dynamic surface analysis with SIMS the combination with STM and AFM has proven to be particularly useful. Sputter craters generated by impact of single primary ions can be observed with STM under UHV conditions and allow to derive important conclusions about the energy transfer, displacement of surface atoms and emission of particles [27]. Topographic changes introduced during depth profiling are in the nm-dimension for single-crystals. These can be studied with STM and AFM which both exhibit an excellent vertical discrimination power in imaging and allow to study quantitatively the development of sputter induced surface roughness and its influence on the quality (dynamic range) of the depth profiles.

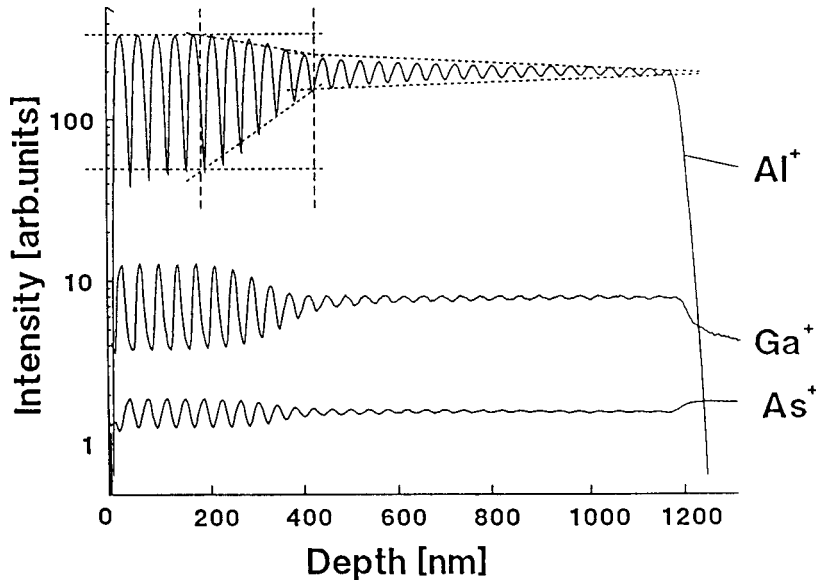


Fig.6: SIMS depth profiles of MBE grown $\text{Al}_{0,35}\text{Ga}_{0,65}\text{As}$ superlattice consisting of 30 bilayers ($d_1 = d_2 = 20$ nm) deposited on a GaAs substrate [28].

Fig. 6 shows as an example the depth profiles of Al, Ga and As in a multiquantum well structure consisting of alternating 20 nm layers of $\text{Al}_{0,35}\text{Ga}_{0,65}\text{As}$ and GaAs. A dramatic decrease in the dynamic range of the profiles can be observed after sputter removal of ca. 200 nm (10 layers). This corresponds to an increase in the roughness of the surface analyzed as shown in Fig. 7 [28]. Cone and ripple formation is observed.

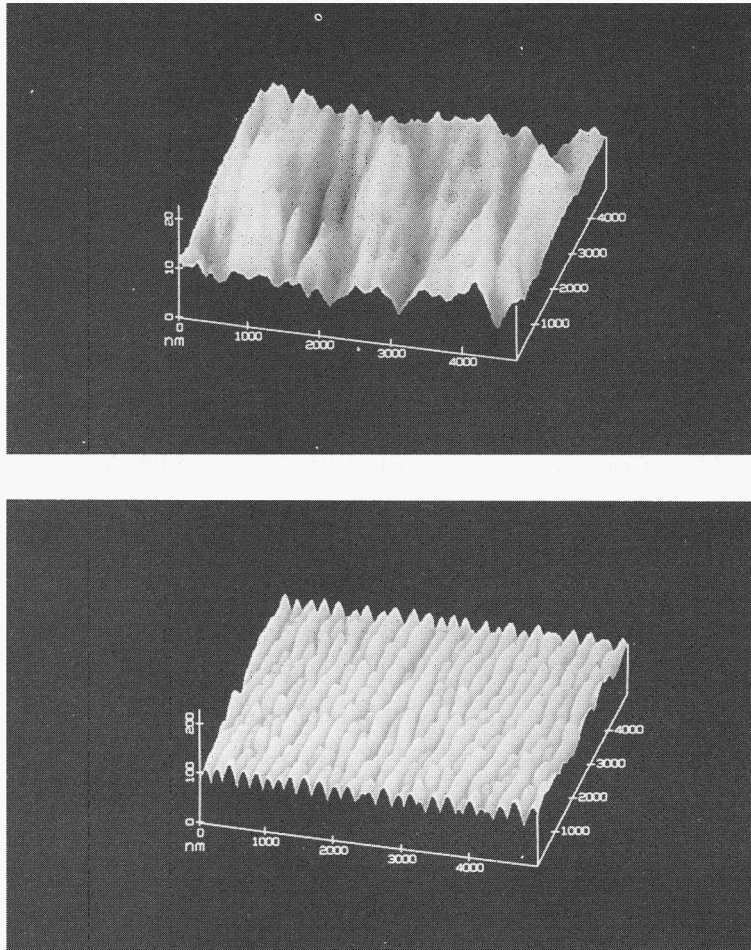


Fig. 7: AFM image of original surface and after sputter removal of 390 nm of AlGaAs/GaAs superlattice structure. At the original surface the roughness is a few Ångstroms. At a depth of 390 nm ripple formation with an average roughness of 58 nm is observed causing the severe loss of depth resolution in the SIMS profile [28].

THREE-DIMENSIONAL (3D) DISTRIBUTION ANALYSIS

3D characterization is an inherent feature of SIMS due to the sequential removal of atomic layers during depth profiling. The lateral distribution information can either be obtained with a finely focussed ion beam rastering across a specified area on the sample or by exploiting the stigmatic imaging properties of an ion microscope. In the latter case the secondary ion image is recorded with either a position sensitive resistive anode encoder or a CCD-camera. In any case a large number of sequentially taken ion micrographs is stored and processed using various mathematical routines (e.g. for smoothing, filtering, background subtraction, quantitation, color encoding [29]). The three-dimensional distribution of secondary ion signals is displayed in various modes, e.g. as cross sections, sections or in transparent cylinders. Fig. 8 shows as an example the distribution of oxygen in a high purity chromium. The micrograph represents the intensity distribution of the secondary ion CrO^+ [30]. The 3D-micrograph shows clearly the native oxide layer on the surface and the enrichment of Cr at the grain boundary leading to a deterioration of the corrosion resistance of this high purity material [31]. The sensitivity of 3D-analysis with SIMS becomes evident from the fact that the bulk oxygen concentration in the material is only $30 \mu\text{g/g}$.

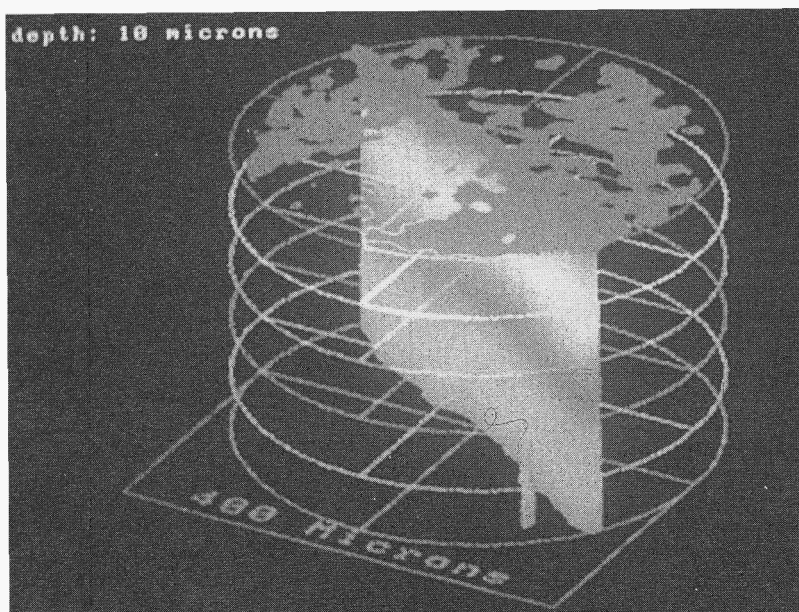


Fig.8: 3D-distribution of oxygen in high purity chromium sample (oxygen content = $30 \mu\text{g/g}$) as obtained by imaging SIMS [30].

For a typical 3D analysis about 100 MByte of data are collected and processed. This number demonstrates also the huge increase in potential information compared to a normal (laterally non-resolved) depth profile which contains about 100 kByte of data.

In principle a semiquantitative evaluation of these representations is possible with RSFs – but the situation is much more complicated than for other typical SIMS results since the trace elements are locally enriched (thus sputter yields and ionization probability change compared to the matrix) and the sputter induced artefacts can be substantial.

One of the other most important areas of application of 3D-analysis will be the stereometric characterization of inclusions or precipitates combining chemical and positional information. This will lead to greatly enhanced possibilities for a property relevant characterization of materials (particularly metals) and microelectronic devices.

CONCLUSION

Solid state mass spectrometry plays a key role in the development of high technology. The variety of techniques available gives access to a large amount of analytical information and to dimensions down to the atomic scale. For a given characterization problem, a strategy based on the combination of different analytical techniques complementary to each other has to be established. These complex analytical procedures are time-consuming and expensive, but usually valuable because unique information about material properties and process parameters can be gained.

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APPENDIX A: LIST OF ABBREVIATIONS AND ACRONYMS

AES	Auger Electron Spectrometry
AFM	Atomic Force Microscopy
DRAM	Dynamic Random Access Memory
EPMA	Electron Probe Micro Analysis
ET-AAS	Electro Thermal Atomic Absorption Spectrometry
GDMS	Glow Discharge Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
RBS	Rutherford Backscattering Spectrometry
SEM	Scanning Electron Microscopy
SIMS	Secondary Ion Mass Spectrometry
SNMS	Secondary Neutrals Mass Spectrometry
STM	Scanning Tunnelling Microscopy
TEM	Transmission Electron Microscopy
TOF	Time-of-Flight (Mass Spectrometer)
VLSI	Very Large Scale Integration (devices)
XPS	X-Ray Photoelectron Spectrometry