ISSUE : 1120/09

HIDEN ANALYTICAL

Customer Research REVIEW

Thin Films, Plasma & Surface Engineering

Customer Contributions:

Absolute Quantification of Deuterium Ion Fluxes
A .Manhard, T. Schwarz-Selinger, and W. Jacob | Max-Planck-Institute für Plasmaphysik

Ti and Al ion irradiation during $Ti_{1-x}Al_xN$ alloy film growth
G. Greczynski | Linköping University

Negative oxygen ion impingement on SiO_2 thin films during growth
A. Palmero | CSIC/US

The origin of oxygen in oxide thin films
Dr. Christof W. Schneider | PSI

Decomposition of Lignin from Sugarcane Bagasse
J. Amorim | CTBE

Related Products:

EQP - Plasma Sampling Mass Spectrometer

PSM - Plasma Ion Analyser

EQS - Bolt on SIMS Analyser

HPR-60 - Molecular Beam Mass Spectrometer

In the press:

NEW Compact SIMS - a design breakthrough for surface analysis

A very big thank you to all who have contributed:

Absolute Quantification of Deuterium Ion Fluxes from an ECR Plasma Source

Plasma discharges are widely used in technical processes such as surface modification and thin-film deposition and etching. In this sense, the corresponding quantitative characterization of plasma sources is a topic of particular importance for a fundamental understanding of plasma sources as well as for a technological optimization of processes. Hydrogen plasmas play a certain role in technical discharges so that a precise determination of the absolute hydrogen fluxes to a plasma-treated surface is very desirable. This is important for understanding both the discharge mechanism and the plasma-induced processes at the surface.

Our Reference: **AP0317**

PROJECT SUMMARY BY:

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REFERENCE:

A. Manhard, T. Schwarz-Selinger, and W. Jacob (2011) "Quantification of the Deuterium Ion Fluxes from a Plasma Source" *Plasma Sources Science and Technology* **20** 015010 doi:10.1088/0963-0252/20/1/015010

PAPER REFERENCE:

P. Wang, W. Jacob, M. Balden, A. Manhard, T. Schwarz-Selinger (2012) "Erosion of tungsten-doped amorphous carbon films in oxygen plasma" *Journal of Nuclear Materials* **420** (1-3), 101-109

HIDEN PRODUCT:

EQP Mass and Energy Analyser

A custom built electron-cyclotron-resonance (ECR) plasma source with a biased sample holder was thoroughly characterized with respect to the deuterium fluxes impinging on the substrate holder. The mass distribution of the impinging ions was measured with a HIDEN plasma monitor (Model HIDEN EQP 300). The plasma monitor was inserted in a mock-up sample holder thus allowing the measurement of the flux arriving at the usual sample position. In order to shield the plasma monitor from the magnetic field necessary for the ECR discharge, the mock-up sample holder was constructed from soft iron. An additional soft iron shielding cylinder was placed around the ion optics of the plasma monitor. The plasma monitor is differentially pumped in order to maintain a pressure of about 10^{-7} to 10^{-6} Pa during operation. The aperture in the mock-up sample holder was 10µm. For absolute quantification of the mass-integrated ion flux to the sample holder a retarding field analyser (RFA) was used.

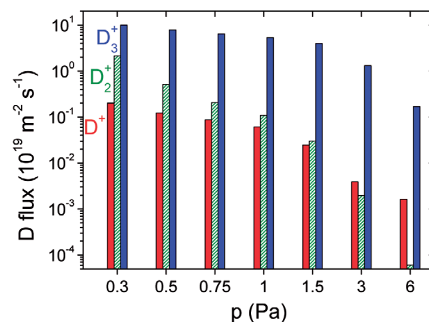


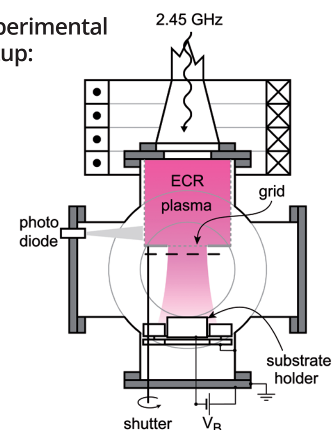
FIGURE 1: (ABOVE) *Ion-species-resolved deuteron flux as a function of the D_2 gas pressure, measured at a constant microwave input power of 144 W and with the sample holder at floating potential.*

FIGURE 2: (RIGHT) *Schematic view of the experimental set-up. To quantify the particle flux to the substrate the substrate holder is replaced by a mock-up containing the EQP plasma monitor (not shown).*

The resulting ion fluxes impinging on the substrate holder for a microwave power of 144 W are presented in Fig. 1 as a function of operating pressure in the range from 0.3 to 6 Pa. Even at the lowest investigated pressure of 0.3 Pa the dominating ion species hitting the sample surface is D_3^+. The dominant production pathway for this ion is an ion-molecule reaction in the bulk plasma. A D_2^+ ion collides with a D_2 molecule producing a D_3^+ and a D atom. With increasing pressure the probability for this reaction increases so that the relative ion species composition changes. With increasing pressure more and more D_3^+ ions are produced at the expense of the primary D_2^+ ion which is produced in the bulk plasma by electron-induced ionization of D_2 molecules. At pressures higher than about 1.5 Pa the total target current decreases due to a decrease of the plasma density, but still the relative contribution of D_3^+ continues to increase. At the highest investigated pressure of 6 Pa the contribution of D_2^+ is almost negligible (less than 10^{-3}). In contrast to D_2^+ the fraction of D^+ does not significantly change. D^+ is, as D_2^+, produced by electron-induced ionization of D_2 molecules and additionally by direct ionisation of atomic D (D plasma concentration is about 1%), but in contrast, the cross section for an ion molecule reaction of D^+ is much lower than that of D_2^+.

For the defined standard operating parameters of our plasma source, i.e. a D_2 plasma at p = 1.0 Pa, P_{MW} = 144 W and the sample holder at floating potential, the total deuteron flux in the form of ions is 5.6×10^{19} Dm^{-2}. This corresponds to a total ion flux of 1.9×10^{19} ions m^{-2}. 94% of these ions are D^{+3}, 3% are D^{+2}, and 3% are D^+ ions. The ion flux increases monotonically by about a factor of 2 if the DC bias voltage is increased to −600 V.

Experimental setup:



Role of Ti^{n+} and Al^{n+} ion irradiation (n = 1, 2) during $Ti_{1-x}Al_xN$ alloy film growth in a hybrid HIPIMS/magnetron mode

Thin films of metastable NaCl-structure $Ti_{1-x}Al_xN$ exhibiting high hardness (typically ~30 GPa) and good high-temperature oxidation resistance are used for wear protection in a wide variety of applications ranging from cutting tools to mechanical components in the aerospace industry. Enhanced performance is obtained at elevated temperatures for alloy films with high AlN content. The calculated $Ti_{1-x}Al_xN$ enthalpy of mixing is positive over the entire range of composition and reaches a maximum (corresponding to the largest driving force for decomposition) at $x = 0.68$.[i] Nevertheless, metastable NaCl-structure alloys can be obtained by physical vapor deposition due to kinetically-limited low-temperature growth and dynamic low-energy ion-irradiation-induced mixing in the near-surface region.

We investigate the growth of polycrystalline $Ti_{1-x}Al_xN$ films by magnetron co-sputtering from separate elemental metal targets, Al and Ti, in a hybrid approach in which high-power pulsed magnetron sputtering (HIPIMS) is combined with dc magnetron sputtering (DCMS). The former sputtering method is known to produce significant ionization of the metal ion flux incident at the growing film surface, while very little metal ions are present in the later case. Thus, this hybrid configuration allows us to selectively control the composition of metal ion fluxes during ion-assisted growth.

We perform two independent sets of experiments. In the first set, the Al target is powered with HIPIMS, while the Ti target is operated with DCMS. The positions of the targets are then switched for the second set of experiments. Thus, we separately probe the role of intense Ti^{n+} and Al^{n+} ion fluxes (n = 1,2) from HIPIMS-powered targets on film growth kinetics, microstructure, and physical properties over a wide range in metastable alloy compositions. We measure the ion flux distribution functions in situ with the PSM 003 Hiden Analytical Mass Spectrometer. The distinctly different flux distributions obtained from targets driven in HIPIMS vs. DCMS modes (see Fig. 1) allow the effects of Al^{n+} and Ti^{n+} (n = 1, 2) ion irradiation on film growth kinetics, and resulting properties, to be investigated separately. Bombardment with Al^{n+} ions (primarily Al^+ in the Al-HIPIMS/Ti-DCMS configuration) during film growth leads to NaCl-structure $Ti_{1-x}Al_xN$ ($0.53 \le x \le 0.60$) films which exhibit high hardness (> 30 GPa) with low stress (0.2 – 0.7 GPa tensile). In contrast, films with corresponding AlN concentrations grown under Ti^{n+} metal ion irradiation (with a significant Ti^{2+} component, cf. Fig. 1) in the Ti-HIPIMS/Al-DCMS mode have much lower hardness, 18-19 GPa, and high compressive stress ranging up to 2.7 GPa. The surprisingly large variation in mechanical properties results from the fact that the kinetic AlN solubility limit x_{max} in $Ti_{1-x}Al_xN$ depends strongly on the target power configuration during growth and hence the composition of the ion flux. AlN with x_{max} ~ 64 mol% can be accommodated in the NaCl structure under Al^{n+} ion flux, compared with ~ 40 mol% for growth with Ti^{n+} flux. The strong asymmetry in film growth reaction paths is due primarily to the fact that the doubly-ionized metal ion flux is approximately two orders of magnitude higher from the Ti target, than from Al, powered with HIPIMS.

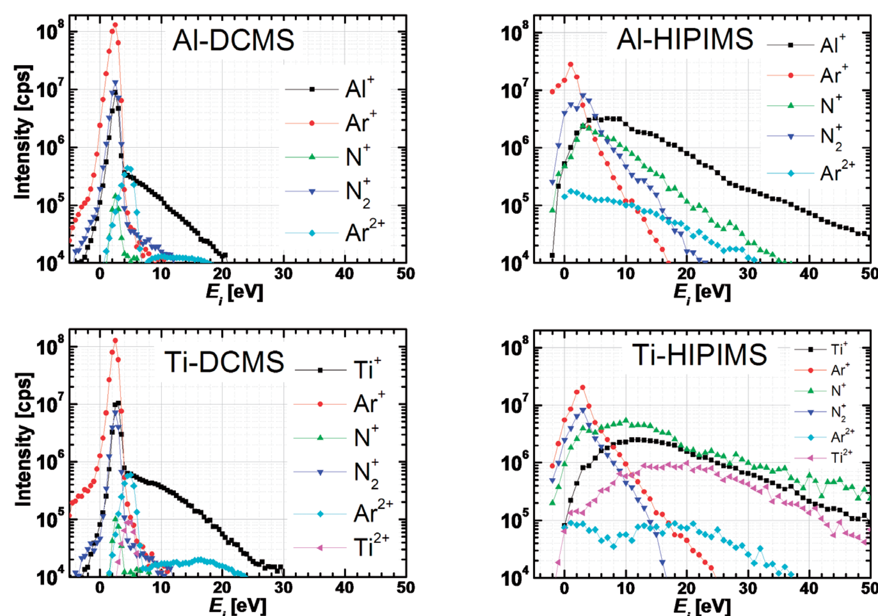


FIGURE 1: *Ion energy distribution functions (IEDFs) obtained with PSM003 mass spectrometer from Hiden Analytical, UK. Data are presented for Al and Ti targets operated in both DCMS and HIPIMS modes. The DCMS data are time-averaged. For HIPIMS, the IEDFs correspond to the highest target current density, 20-~s, portion of the 200 ~s pulses.*

Our Reference: **AP0381**

PROJECT SUMMARY BY:

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PAPER REFERENCE:

G. Greczynski et al. (2012) "Role of Ti^{n+} and Al^{n+} ion irradiation (n = 1, 2) during $Ti_{1-x}Al_xN$ alloy film growth in a hybrid HIPIMS/magnetron mode" *Surface and Coatings Technology* **206**, 4202-4211

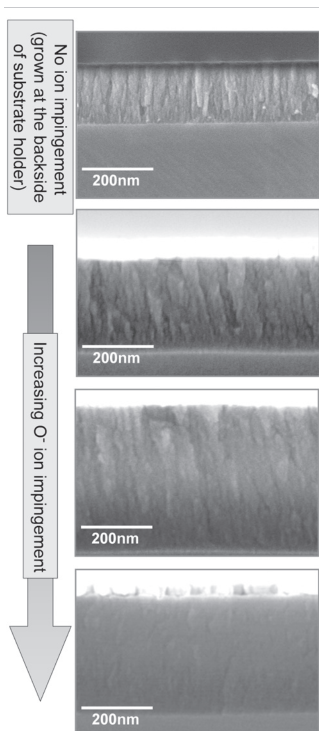
HIDEN PRODUCT:

PSM Plasma Ion Analyser

Influence of plasma-generated negative oxygen ion impingement on magnetron sputtered amorphous SiO_2 thin films during growth at low temperatures

Understanding the fundamental processes involved in thin film nanostructuration is nowadays a key issue in material science and technology. In this aspect, plasma-assisted deposition of thin films is relevant thanks to the strong interaction between plasma-generated species and the film during growth, which may induce the formation of metastable structures which optimize or provide singular properties for diverse applications. In project PLASMATER (P10-FQM-6900), funded by the Junta de Andalucía, we aim at studying these fundamental processes and their influence on the film nanostructure.

For this, film characterization techniques such as scanning electron microscopy, atomic force microscopy, optical response measurements, etc. have to be combined with plasma diagnosis techniques, such as optical emission spectroscopy, Langmuir probe measurements or mass spectrometry. We have available an energy-resolved mass spectrometer (EQP 500 HIDEN Analytical Ltd.) which has helped us understand the role of ion impingement on the growth of SiO_2 thin films deposited by DC pulsed magnetron sputtering.



Cross-sectional electron microscopy images of the films for increasing O^- flux.

In DC pulsed magnetron sputtering discharges, positively charged ions are produced in the plasma bulk and accelerated towards the film with kinetic energies of few electronvolts when the substrate is not biased. Although their role in thin film nanostructuration could be relevant in some cases, their typical energy is about the displacement energy threshold of the adatoms in the material, and, most likely, they heat up the film surface and weakly induce thermally activated relaxation processes. On the other hand, negative ions, which are present when an electronegative gas is introduced in the reactor, may be generated at the cathode surface and accelerated towards the film with energies of the order of the cathode potential fall (i.e., few hundred electronvolts). These ions possess enough energy to induce the mobilization of the film adatoms through collisions and cause important changes in the film nanostructure. Although from an energetic point of view negative ions are relevant, the question on whether their flux is high enough to actually produce changes in the film was still open until this paper appeared.

In this publication we show that negative oxygen ions are responsible for microstructural changes on magnetron sputtered SiO_2 thin films. For this, we have deposited several coatings under wisely-chosen conditions with different microstructures. After analyzing the role of the relevant processes affecting the growth of the films, such as surface shadowing, thermally activated processes on the surface as well as positive and negative ion impingement, we conclude that the obtained microstructural changes can only be attributed to the impingement of intermediate energy O^- ions. These ions appear in the discharge as a product of the dissociation of high-energy molecular oxygen negative ions produced near the cathode surface and that are accelerated towards the plasma. In the image we depict four cross-sectional scanning microscopy images of the films deposited for increasing O^- ion flux bombardment. There, the removal of vertical geometrical patterns and void percentage for increasing negative oxygen fluxes are apparent. Overall, we show the existence of two thin film nanostructuration regimes at low temperatures, controlled by the amount of O_2 in the deposition reactor and caused by the competition between the surface shadowing mechanism and the negative ion-induced adatom surface mobility processes.

Our Reference: **AP-EQP-0004**

PROJECT SUMMARY BY:

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PAPER REFERENCE:

M. Macias-Montero et al. (2012) "Influence of plasma-generated negative oxygen ion impingement on magnetron sputtered amorphous SiO_2 thin films during growth at low temperatures" *Journal of Applied Physics* **111** (5) 054312 *http://dx.doi.org/10.1063/1.3691950*

HIDEN PRODUCT:

EQP Mass and Energy Analyser

The origin of oxygen in oxide thin films: Role of the substrate

During the growth of oxide thin films by pulsed laser deposition, there is always the question from which source oxygen is incorporated in the as-grown thin film: Target, background or substrate. This is an essential question since the optimization of the deposition conditions will depend on it. Using ^{18}O exchanged $SrTiO_3$ and $LaAlO_3$ as substrates, a strong oxygen substrate to film transfer has been experimentally observed for epitaxially grown $SrTiO_3$ and $LaAlO_3$ thin films by SIMS depth profiling. This oxygen transfer effect can seriously change the respective thin film properties.

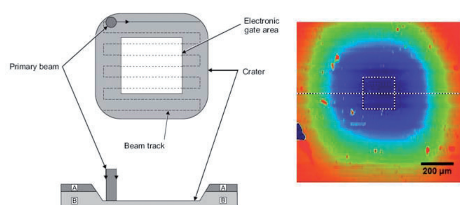

FIGURE 1: *Schematic representation of the raster crater (left) and a real crater measured with a profilometer (right). The different colours correspond to different depth levels.*

The preparation of high quality complex oxides thin films and heterostructures with atomic precision has considerably advanced in recent years with a crystalline quality comparable to semiconductors and oxide semiconductors structures. The challenge associated with growing oxides is to provide and control the amount of oxygen during the growth to obtain the desired oxygen stoichiometry and hence appropriate physical properties of the respective oxide. For a deposition using pulsed laser deposition (PLD) it often seems to be sufficient to create the correct amount of plasma species within an oxygen background to form the chosen compound as a thin film on a suitable substrate, and to supply the missing oxygen with a subsequent annealing step.

To investigate the role of oxygen supplied by the substrate during a deposition, the oxygen diffusion properties of $SrTiO_3$ and $LaAlO_3$ thin films grown on ^{18}O isotope exchanged $SrTi^{18}O_3$ and $LaAl^{18}O_3$ substrates were studied by dynamic secondary ion mass spectrometry (D-SIMS) which yields elemental depth profiles. $SrTiO_3$ and $LaAlO_3$ thin films have been prepared at three different deposition temperatures (nominal room temperature, 650°C and 750°C) at a background pressure p=1.5x10^{-5} mbar and a laser fluence F=4 Jcm^{-2}. SIMS spectra were recorded using a Hiden Analytical EQS quadrupole mass spectrometer operated with a 2.5 keV Ar ion beam focused to 150 µm diameter rastering over a square of 1x1mm with an effective sampling area of 500x500 µm. The etched area is subsequently measured with a Dektak 8 profilometer to convert etching time into depth. (see Fig. 1). In addition a kinetic energy selection scheme is used to separate species with the same mass.

The ^{18}O diffusion from $SrTi^{18}O_3$ into $SrTiO_3$ shows a pronounced dependence on the deposition temperature (Fig. 2a). Whereas for a room temperature deposition, no traceable ^{18}O diffusion into the film has been measured, the situation changes dramatically for elevated deposition temperatures. At 650°C, there is considerable ^{18}O diffusion from the substrate into the film, while at T_S=750°C there is no significant difference with respect to the amount of ^{18}O measured in the film and substrate. In the case of $SrTiO_3$ grown on $LaAl^{18}O_3$ a significant and homogeneous oxygen contribution is measured for the $SrTiO_3$ film prepared at T_S=750°C (Fig. 2b). Even at 650°C, there is still a significant ^{18}O intake which can be detected up to the film surface. Measuring Sr and Ti species from the substrate and film simultaneously with ^{16}O and ^{18}O, the elemental composition of film and substrate are very similar. This implies that oxygen in the $SrTiO_3$ thin film is supplied by the substrate and the oxygen provided by the target seems to play a minor role for this system. The results also indicate that the initially formed film is oxygen deficient and a chemical gradient is in favour of supplying oxygen via the substrate.

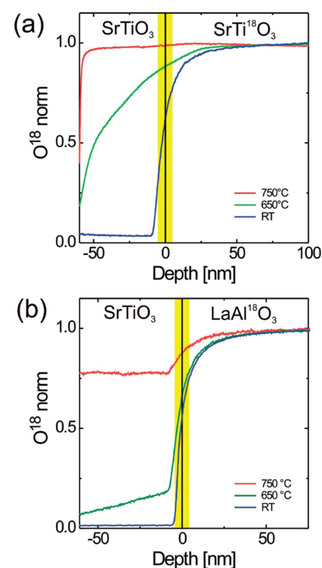

FIGURE 2: *a) ^{18}O SIMS depth profile of $SrTiO_3$ on $SrTi^{18}O_3$ grown at T_S=750°C, 650°C and room temperature. The sharp drop of the ^{18}O signal near the $SrTiO_3$ surface for the film grown at T_S=750°C could be related to a back-exchange of ^{16}O at room temperature. b) ^{18}O SIMS depth profile of $SrTiO_3$ on $LaAl^{18}O_3$ grown at T_S=750°C, 650°C and room temperature.*

Our Reference: **AP-EQS-0006**

PROJECT SUMMARY BY:

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PAPER REFERENCE:

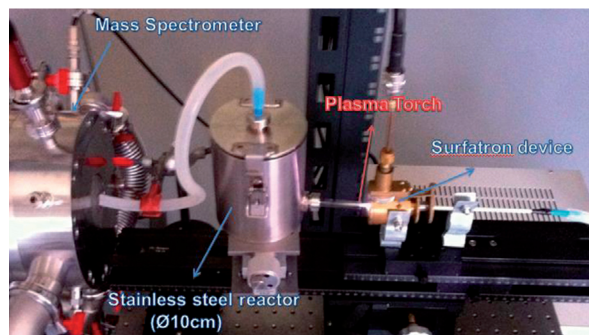
C. W. Schneider, M. Esposito, I. Marozau, K. Conder, M. Doebeli, Yi Hu, M. Mallepell, A. Wokaun, and T. Lippert (2010) "The origin of oxygen in oxide thin films: Role of the substrate" *Appl. Phys. Lett.* (online) http://dx.doi.org/10.1063/1.3515849

HIDEN PRODUCT:

EQS Secondary Ion Mass Spectrometer
Bolt on SIMS Analyser

Decomposition of Lignin from Sugarcane Bagasse during Ozonation Process Monitored by Optical and Mass Spectrometries

Mass spectrometry measurements were employed to study the interaction of ozone with sugarcane bagasse. The aim was to remove lignin prior to bagasse hydrolysis exposing the lignocellulosic matrix to the additional enzymatic attack. The reaction mechanisms proposed in the literature are sometimes controversial. In this study the yield of more important radicals were recorded by mass spectrometry, using a Hiden HPR-60 MBMS, where three phases could be observed during the treatment. The first one was characterized by the presence of CO molecules. During the second phase radicals such as O_2, CH_3OH, OH and H_2O appeared. The third stage was characterized by the removal of these radicals and the appearance of the HCOOH, CO_2 and H_3O radicals. All these results have supported the Criegee's mechanism in the explanation of ozone attack of the lignin.



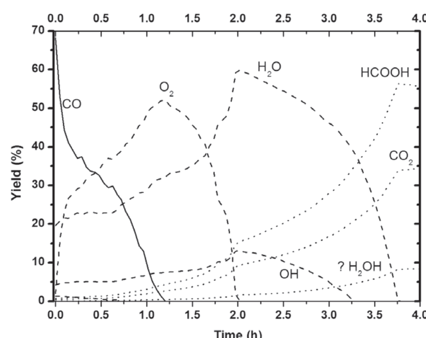
Sugarcane Bagasse Plasma Pretreatment

TREATMENT CONDITIONS:

- *~25g of dry sugarcane bagasse (50% moisture) - milled at 500μm*
- *Gas flow Mixture: 95% Ar (1.9 SLM) and 5% O_2 (0.1 SML)*
- *$\Delta t_{treatment}$ = 3h.*

Experimental setup for microwave plasma biomass pre-treatment

Sugar cane bagasse, raw material (left) and after 6 hours of treatment (right).



Species yield (%) as a function of bagasse ozonation treatment time (moisture of 50%).

Physico-chemical analyses has shown that the lignin content was reduced as a function of ozonation time, achieving the maximum delignification efficiency of about 65% during 4h of treatment.

By optical spectroscopy, an investigation of the influence of typical bagasse sizes and of moisture content in bagasse during the ozonation was performed. The results have indicated that the best condition for the ozone diffusion in the lignocellulosic matrix was for particle bagasse sizes to be less than or equal to 0.5mm with wetness of 50%. This last result (related to moisture content effect) was in agreement with the one obtained via mass spectrometry, corroborating the consistency of both techniques employed.

The FTIR studies revealed that several lignin bonds were strongly affected by the ozonolysis process giving support for the mass detection results related to Criegee's mechanism as well. The SEM technique demonstrated an alteration on the rugosity of bagasse surface morphology after the ozonation pretreatment procedure. Therefore in conclusion all of the results obtained from several analytical methodologies have strongly indicated that oxidative delignification of sugarcane bagasse via ozone has a great potential for applications on biomass pretreatment methods, in particular for sugarcane bagasse.

Our Reference: **AP-HPR-60-0002**

PROJECT SUMMARY BY:

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PAPER REFERENCE:

(2013) "Decomposition of Lignin from Sugar Cane Bagasse during Ozonation Process Monitored by Optical and Mass Spectrometries" *The Journal of Physical Chemistry B* **117** (11) 3110-3119
dx.doi.org/10.1021/jp3121879

HIDEN PRODUCT:

HPR-60 MBMS Molecular Beam Sampling Mass Spectrometer

Related Products:

EQP – Plasma Sampling Mass Spectrometer

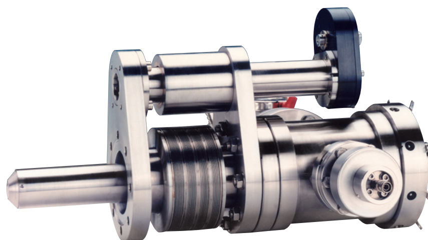
The Hiden EQP is a combined mass/energy analyser for the analysis of positive AND negative ions, neutrals, and radicals from plasma processes:

- Analysis of positive ions, negative ions, neutral radicals and neutrals
- Etching/Deposition Studies
- Ion Implantation/Laser Ablation
- Residual Gas Analysis/Leak Detection
- Plasma electrode coupling - follow electrode conditions during operation
- Analysis through a viewport, grounded electrode, driven electrode

PSM – Plasma Ion Analyser

The Hiden PSM is a differentially pumped mass spectrometer for the analysis of secondary ions and neutrals from plasma process:

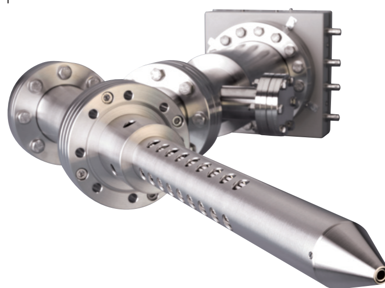
- Reactive Ion Etching – End Point Detection
- Plasma Deposition Studies
- Ion Implantation/Laser Ablation
- Residual Gas Analysis
- Leak Detection



EQS – Bolt on SIMS Analyser

The EQS is a secondary ion mass spectrometer bolt on SIMS analyser for the analysis of secondary positive and negative ions from solid samples:

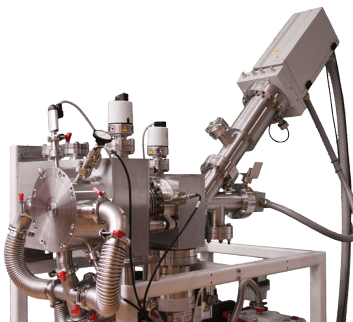
- Static/Dynamic SIMS with Energy Analysis
- Integral Front End Ioniser for RGA
- Composition/Contamination Analysis
- Depth Profiling
- Leak Detection & Desorbed Gas Analysis
- Compatible with Hiden SIMS Workstation
- Suitable for FIB-SIMS Integration



HPR-60 – Molecular Beam Mass Spectrometer

The Hiden HPR-60 Molecular Beam Sampling Mass Spectrometer is a compact gas analysis system for analysis of neutrals, radicals and ions:

- Reaction Kinetics
- Plasma Diagnostics
- Combustion Studies – Flame Ionisation Analysis
- Catalysis Studies
- CVD/MOCVD – Diamond Growth Studies
- Flash Desorption Studies
- Atmospheric Glow Discharge Analysis
- Cluster Analysis



In the press:

OUR REFERENCE: HAPR0107

NEW Compact SIMS

A design breakthrough for surface analysis

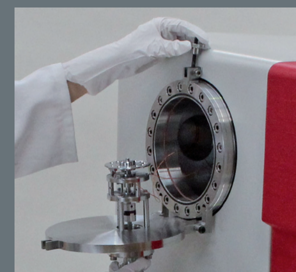
The **Hiden Compact SIMS** tool is designed for fast and easy characterisation of layer structures, surface contamination and impurities with sensitive detection of positive ions being assisted by the oxygen primary ion beam and provides isotopic sensitivity across the entire periodic table. The ion gun geometry is optimised to be ideal for nanometre depth resolution and near surface analysis.

A rotary carousel enables 10 samples to be simultaneously loaded for measurement into the dry-pumped vacuum chamber. The instrument has a small footprint and is exceptionally easy to use, it boasts the same control software and ion gun system as the fully featured Hiden SIMS Workstation family, providing depth profiles, 3D and 2D images and mass spectral data. The MAXIM-600P detector is based around the highly reliable Hiden 6mm triple quadrupole mass filter with pulse ion detection. An electron gun option is available for analysis of insulating samples.

In addition to SIMS, the Compact SIMS has an SNMS facility that is useful for quantification of high concentration elements, such as alloys.

Applications:

- Solar cells
- Glass coatings
- Metallic thin films



Features:

- Small footprint
- Easy user friendly layout
- Positive SIMS and SNMS
- Depth Profiling
- 3D characterisation and imaging
- Mass spectra
- Isotopic analysis
- Analysis on the nanometre scale

For further information on these or any other Hiden Analytical products please contact Hiden Analytical at **info@hiden.co.uk** or visit the main website at **www.HidenAnalytical.com**

If you would like to submit a project summary for consideration in our next Newsletter, please email a brief summary (*approx. 500 words*) and corresponding images to **marketing@hiden.co.uk**

HidenAPPLICATIONS

Hiden's quadrupole mass spectrometer systems address a broad application range in:

GAS ANALYSIS

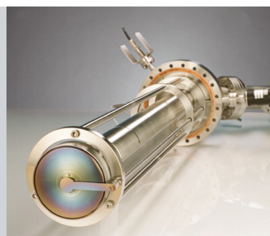
- dynamic measurement of reaction gas streams
- catalysis and thermal analysis
- molecular beam studies
- dissolved species probes
- fermentation, environmental and ecological studies

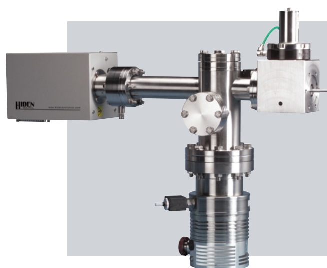
SURFACE ANALYSIS

- UHV TPD
- SIMS
- end point detection in ion beam etch
- elemental imaging - surface mapping

PLASMA DIAGNOSTICS

- plasma source characterisation
- etch and deposition process reaction kinetic studies
- analysis of neutral and radical species





VACUUM ANALYSIS

- partial pressure measurement and control of process gases
- reactive sputter process control
- vacuum diagnostics
- vacuum coating process monitoring

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