Issue Number: 1120/02

HidenLife

A world of products connected by science & imagination

Hiden Quadrupole Mass Spectrometers *in action*:

Thin Films, Plasma and Surface Engineering

Hiden Quadrupole mass spectrometers are used for process, environmental and research applications throughout the world.

This newsletter includes a selection of the most recent application stories from referenced published sources.

Our contributors to this newsletter caught our eye with published articles of the highest quality.

Key data from the Hiden ESPion Langmuir Probe, MAXIM SIMS/SNMS Workstation, IG-20 Ion Gun and HPR-30 Vacuum Process Gas Analyser are included.

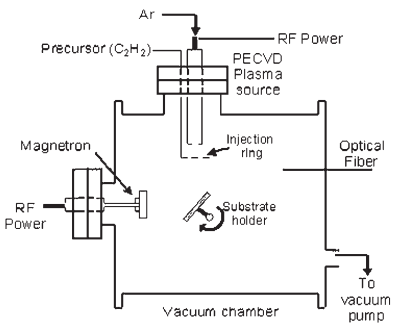
We are delighted that they have shared a brief synopsis of their research for our newsletter.

A very big thank you to all who have contributed.

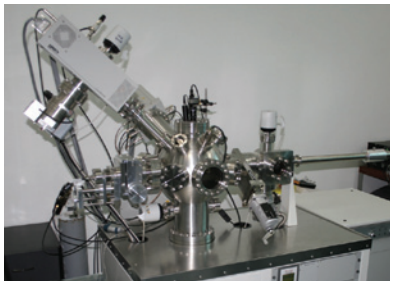
In this issue:

HIDEN ANALYTICAL

Customer Stories:



▲ Schematic of plasma assisted sequential deposition setup



▲ The Hiden MAXIM SIMS/SNMS Workstation

Our Reference: AP0088

Project Summary by:
Ph.D Tomy Acsente,
National Institute for Lasers, Plasma and Radiation Physics,
Bucharest Romania

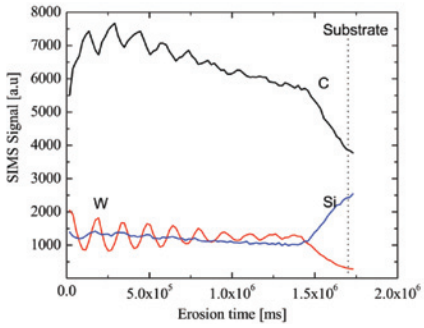
Paper Reference:
"*Properties of composite a-C:H/metal layers deposited by combined RF PECVD/magnetron sputtering techniques*" Thin Solid Films , Volume 519, Issue 12, 01-Apr-11, Pages 4054-4058

Hiden Product:
MAXIM SIMS/SNMS Workstation

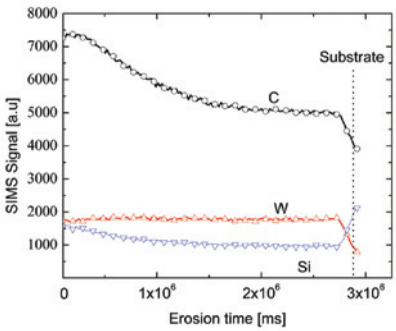
Metal-carbon composites and multilayer thin films prepared by plasma assisted sequential deposition

Thin films incorporating mixed metal and carbon elements (like carbon-metal nanocomposites or multilayer thin films) find applications as functional coatings in many fields of modern technology. A novel plasma assisted deposition method (namely sequential deposition), combining magnetron sputtering (MS) and plasma enhanced chemical vapor deposition (PECVD) was implemented. Using the same deposition setup, the sequential deposition method proved to be able in producing either a-C:H/W nanocomposite or carbon tungsten multilayers.

Most of the plasma deposition methods used currently for production of metal carbon nanocomposite or multilayers involve simultaneously addition of the film constituents on the substrate. Using our approach it is possible to obtain composite films from separately deposited elemental constituents. The method consists in cyclic (sequential) exposure, for predefined time intervals, of a substrate to totally independent two plasma deposition sources, working in alternative sequences: MS in Ar for metal deposition and PECVD, in Ar and C_2H_2, for deposition of hydrogenated amorphous carbon (a-C:H). One deposition cycle consists in the following steps: metal deposition by MS, transport of the substrate in the front of PECVD, deposition of a-C:H, and backward transport of the substrate in the front of MS. The substrate is transported between the plasma sources using a stepper motor; during the substrate movement the plasma sources are not energized and proper gas composition for the following deposition step is prepared. Besides the plasma processes parameters (discharges power and gas composition) important process parameters are the temporal ones (duration of MS and PECVD processing steps, transport time, and the number of cycles).

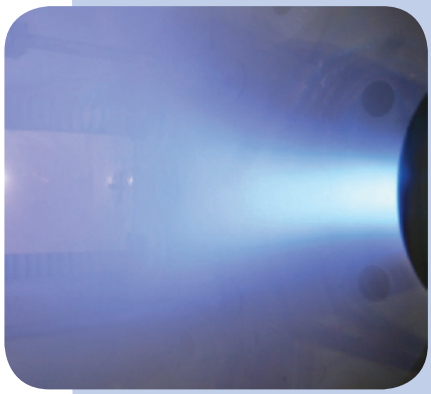


▲ SIMS profile of a carbon tungsten multilayer structure

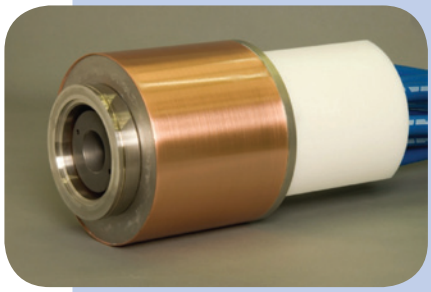


▲ SIMS profile of a carbon tungsten nanocomposite structure

The durations of plasma processes determine the composition and the degree of intermixing of the constituents in the deposited film, while its thickness is controlled by the number of successive deposition cycles. Such as, quasi-homogeneous nanocomposite films with



▲ Fig. 1 Hollow cathode plasma plume

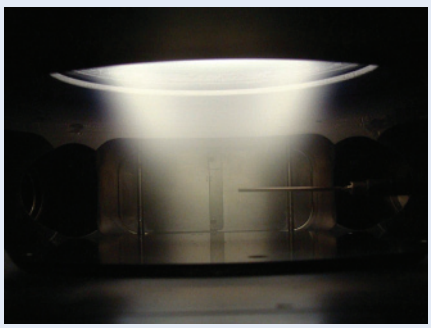


▲ Fig. 2 The hollow-cathode based plasma source

Hiden ESPion plasma probe measurements on a hollow-cathode based large-volume plasma source

Vacuum-based surface engineering and coating processes require plasma sources generating a high charge carrier density in order to achieve enhanced sputter and activation effects. Fraunhofer FEP is providing a hollow-cathode based plasma source, which produces homogeneous large-volume plasmas of up to 1 m^3 with maximum plasma densities of more than 10^{12} cm^{-3} at chamber pressures between 0.1 and 10 Pa. The cathode tube is flown through by the working gas such as argon with flow rates between 8 and 100 sccm. A diffuse arc discharge with currents up to 200 A between the inner cathode tube surface and the surrounding annular anode is ignited and amplified by an axial magnetic field with field strengths up to 100 mT.

In order to characterize the plasma plume and to study the discharge mechanisms of the hollow cathode, extensive measurements with the Hiden ESPion plasma probe have been carried out. The probe tip was moved by a linear Z-motion drive up to 60 cm into the chamber at three axial distances from the hollow cathode device (30 cm, 70 cm, and 125 cm) yielding spatial distribution of plasma parameters. It was found that reducing the gas flow through the cathode tube results in drastically increased plasma density and range. Furthermore, the measurements show the pressure dependence of the axial plasma density decay and of the electron temperature. At high gas pressures, the plasma is more concentrated near to the hollow cathode, and the Maxwell-distributed electrons



▲ Picture of the Langmuir Probe in the $Ar/0_2$ plasma with a titanium target

O^- density measurements in the pulsed-DC reactive magnetron sputtering of titanium

The importance of electronegative species in technological plasmas is currently of great interest, both from a theoretical and experimental point of view, with much of the recent work concentrating on reactive ion etching discharges.

However, other very important plasma systems have been somewhat neglected by scientist and engineers in terms of the role of negative ions, for instance, pulsed magnetized discharges (the sputter magnetron) used for the reactive deposition of thin films which contain electronegative gases such as oxygen.

In this project, we have made the first detailed study of the time-evolution of the density of negative ions in pulsed reactive magnetron plasmas (Ar/O_2) and their effect on the structure and dynamics of the plasma itself. The main negative ion species we observe close to the substrate is O^- with densities approaching the electron density in the pulse on time, but with densities much greater than the electron density when the off-times are greater than about 50 μs. About 105 of the total O^- density are very energetic, with energies up to the equivalent of the cathode target potential. The results are useful in understanding thin film growth dynamics and the fundamental data can be used in other more surface engineering orientated projects.

elemental concentration ranging from 10 at % W and 80 at % C up to 40 at % of both W and C were obtained using plasma short exposures of a few seconds. Increasing the duration of plasma processes up to tens of seconds there were deposited multilayer films, consisting of successive C and W layers. The in-depth compositional profiles of both composite and multilayer structures were investigated using a MAXIM SIMS/SNMS Workstation from Hiden Analytical.

Our Reference: AP0067

Project Summary by:

UNIVERSITY OF LIVERPOOL

Professor James Bradley
Department of Electrical Engineering and Electronics, University of Liverpool, Liverpool UK

Paper Reference:

"*O^- density measurements in the pulsed-DC reactive magnetron sputtering of titanium*" Thin Solid Films, Volume 519, Issue 5, 30-Dec-10, Pages 1705-1711

Hiden Product:

ESPion Advanced Langmuir Probe

have lower energies. On the other hand, low gas pressures lead to a beam-like character in the vicinity of the hollow cathode and to a low axial and lateral density decay, exhibiting pronounced homogeneity. Comparable results have been found in nitrogen and oxygen atmosphere, where additional optical and mass-spectrometric analysis revealed strong excitation and dissociation of the reactive gas molecules.

The high efficiency of the plasma source was already shown in various applications such as high rate plasma etching for substrate pretreatment, plasma-activated evaporation of metal and oxide layers for corrosion protection, plasma-assisted reactive magnetron sputtering of hard and wear-resistant coatings, and plasma-enhanced chemical vapor deposition. The Hiden ESPion plasma probe has been shown to be an excellent tool for the quantification of plasma effects and the development of new plasma processes.

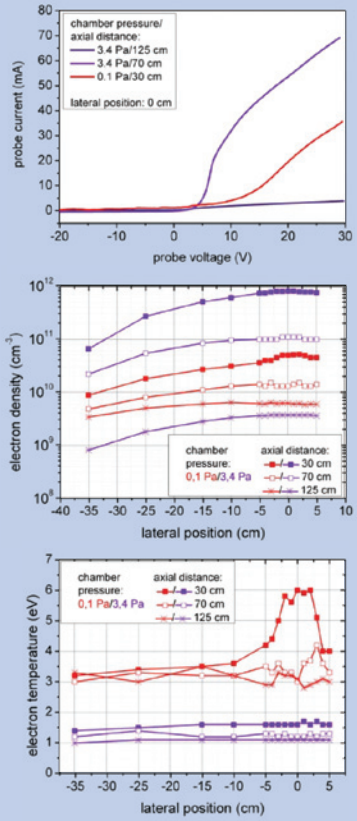


Fig. 4: Plasma probe measurement results; (a) Probe characteristics (b) and (c) Lateral density and electron temperature distribution at different axial positions and chamber pressures (discharge current: 50 A, cathode gas flow: 10 sccm)

Our Reference: AP0121

Project Summary by:

Fraunhofer FEP Burkhard Zimmermann, Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik FEP, Winterbergstraße 28 01277 Dresden, Germany, burkhard.zimmermann@fep.fraunhofer.de

Paper Reference:

B. Zimmermann, F. Fietzke, W. Möller: *"Spatially resolved Langmuir probe measurements of a magnetically enhanced hollow cathode arc plasma"* Surface and Coatings Technology, Volume 205 Supplement 2 (2011), Pages S393-S396

Hiden Product:

ESPion Advanced Langmuir Probe

Surface characteristics of parylene-C films in an inductively coupled O_2/CF_4 gas plasma

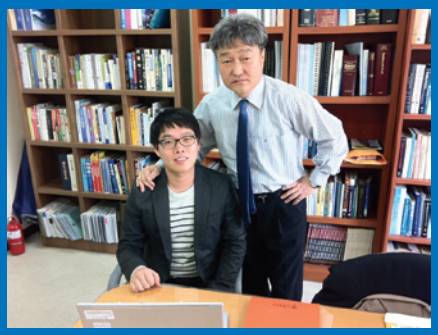


Fig. 2 Discussing the extracted data with co-worker
Left: Dr Yong-Hyun Ham
Right: Professor Kwang-Ho Kwon

We investigated the degradation mechanism of OTFT performance in the plasma processing. In order to examine the origin of the degradation mechanism due to the plasma exposure, the relationships between operating parameters and plasma species should be analyzed for fabrication of high performance transistor in the plasma processing. So, we extracted the plasma species using quadrupole mass-spectrometry (QMS). Then, we applied this result for analyzing a degradation mechanism of OTFT devices.

Our Reference: AP0002

Project Summary by:

Dr Yong-Hyun Ham and Professor Kwang-Ho Kwon Korea University, College of Science & Technology, Dept. of Control & Instrumentation Engineering, Plasma Application Research Laboratory, Choong-nam Korea

Paper Reference:

"*Surface characteristics of parylene-C films in an inductively coupled O_2/CF_4 gas plasma*" Thin Solid Films, Volume 518, Issue 22, 01-Sept-10, Pages 6378-6381

Hiden Product:

HPR-30 Vacuum Process Gas Analyser

Fig. 1 Photograph of the Hiden Mass Spectrometer in use in our lab

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**

In the Press:

Hiden Products referenced in our Customer Stories in this issue:

MAXIM SIMS/SNMS Workstation

A state of the art secondary ion mass spectrometer for static and dynamic SIMS and SNMS applications:

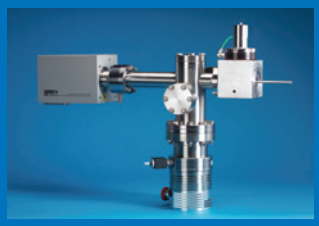
- Integral energy filter for ion acceptance at 30° to the probe axis
- High transmission SIMS extraction ion optics
- High efficiency electron impact SNMS ionizer
- Triple mass filter
- Pulse ion counting detector
- Control electronics with Windows MASsoft PC software
- Raster control for imaging and depth profiling

ESPion Advanced Langmuir Probe

The ESPion advanced Langmuir probe for rapid, reliable and accurate plasma diagnostics for industry and academia:

- Etching / Deposition / Cleaning Plasma Processes
- Pulsed plasma operation
- Ion density (N_i & G_i)
- Electron retardation (T_e & EEDF)
- Electron density (N_e)
- Plasma Potential
- Debye Length, floating potential
- Ion flux

HPR-30 Vacuum Process Gas Analyser



The HPR-30 process gas analysis system is a compact gas analysis system for monitoring residual gases and vacuum processes:

- CVD / PECVD / RIE / LPCVD / MOCVD
- Vacuum Coating / Plasma Etching
- Sputter Deposition
- Contamination Studies
- Base Pressure Fingerprint
- Leak Detection / Virtual Leaks / desorption
- Outgassing / Bakeout / Pump Performance
- Chamber / Process gas contaminants

The Hiden TPD Workstation for UHV Thermal Desorption Studies

(Our Reference: PR0041)

The TPD Workstation is a complete turnkey station for UHV temperature programmed desorption studies and is suited to both quality control and to research applications. Applicable technologies include photovoltaics, metallurgy and semiconductor and thin-film studies

The HPR60-EQP Atmospheric Plasma Monitor

(Our Reference: PR0050)

The system is configured specifically for measurement of atmospheric plasma using three stages of pressure reduction, with orifice sampling providing fast transfer of neutrals and of reactive species direct to the mass spectrometer stage. The system enables measurement of positive ions, negative ions and neutral species with molecular weights up to 2500 amu. A custom design service is offered, if required, to engineer a suitable system interface to adapt the sample extraction zone to the reactor.

Typical applications include analyses of atmospheric plasma jets (APD), dielectric barrier discharges (DBD), flame chemistry.

Hiden Applications

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 Science

- 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

Sales Offices:

We have sales offices situated around the globe.
Visit our website for further information.

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