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RAPID SURFACE MICROANALYSIS USING A LOW TEMPERATURE PLASMA

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Abstract

There is a need for rapid, high-resolution (micron or sub-micron) scanning of surfaces of special nuclear materials (SNM) and surrogate materials to locate and identify regions of abnormalities. One technique that is commonly used to analyze the composition of solid surfaces and thin films is secondary-ion mass spectrometry (SIMS). SIMS devices are very complex and expensive. We propose to develop a simpler, less expensive surface analysis devices, based on glow-discharge optical emission spectroscopy (GOES) that can provide excellent spatial resolution. Ions from a plasma discharge sputtered atoms from the surface and the discharge electrons effectively excite and ionize the sputtered atoms. GOES uses the light emitted by the excited particles for quantitative analysis. In the GOES device, the ion flux is extracted from the gas-discharge plasma and focused to a micron size on the sample, providing very local sputtering and local elemental analysis. The radiation from the sputtered atoms is passed through an optical fiber to an optical spectrometer and recorded. To register the distribution of elements over the sample, the sample is scanned electro-mechanically as a polarized deuteron injector for a future electron-ion collider (EIC).

INTRODUCTION

Convenient, inexpensive methods for nanoscale surface composition measurements are needed for microelectronic manufacturing, advanced nanotechnology, chromatography and many other fields. Sputtering occurs when particles of a solid material are ejected from its surface by energetic particles from a plasma. The degradation of the solid material and the subsequent deposition of the ejected material onto vulnerable surfaces are the usual subjects of sputtering studies. However, plasma science has yet to be combined with sputtering to create new diagnostic applications.

In this report we will propose a plasma beam technique to 1) rapidly scan large targets to find areas of interest for high resolution investigation, and 2) provide an inexpensive method for high resolution surface composition measurements.

Sputtering occurs when particles of a solid material are ejected from its surface by energetic particles from a plasma. A small change in the design of the plasma discharge device makes it possible to localize sputtering on a small portion of the target and to obtain the distribution of ejected elements over the surface of the sample with micron resolution. Another change, to be developed in this project, will generate a line of plasma that will allow rapid scans of single square cm area samples.

For surface analysis, optical glow discharge spectroscopy (OGDS) is used [1]. Ions from the plasma discharge sputter the atoms from the surface and the electrons from the plasma discharge excite and ionize the sputtered atoms. OGDS uses the light emitted by the excited particles for quantitative analysis.

GRIMM LAMP FOR OPTICAL GLOW DISCHARGE SPECTROSCOPY (OGDS)

The Grimm discharge lamp shown in Fig. 1 [2] is typically used for OGDS. The radiation of sputtered atoms is recorded by a spectrometer through a window and fiberglass cable. The anode is separated from the cathode by an insulator. The anode has an inner diameter of 7-10 mm. It is covered by a diaphragm with a 3 mm hole. The anode to cathode gap is set to within 0.25-2.5 mm. Helium, Neon, Argon, Krypton and/or Xenon gases are fed into the cathode-anode gap through a leakage channel with gas pressure of about ~1-5 Torr. A high voltage of 1 to 2.5 kV is applied to the anode. Light is recorded along the axis of the device through window and fiber optic cable. The emitted spectrum can be recorded by an inexpensive Ocean Optic spectrometer with a fiberglass light gate.

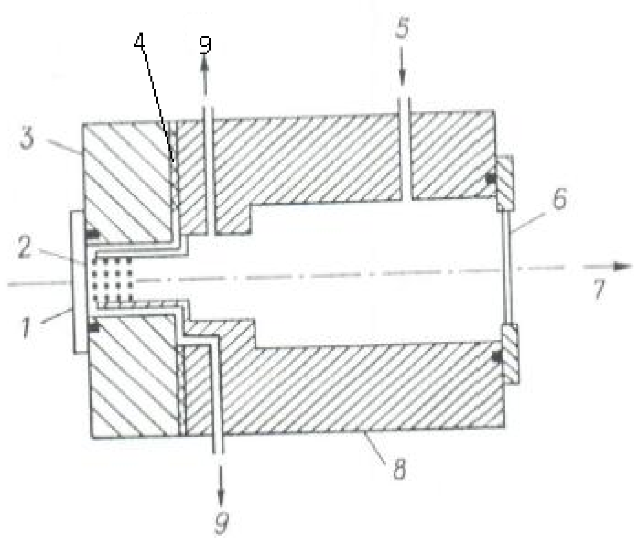

Figure 1: Grimm lamp for OGDS. UPPER: 1- sample; 2- discharge; 3- cathode; 4- insulator; 5-gas supply; 6-quartz window; 7 - monochromator and detector; 8- anode; 9- pumping.

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An example of an emission spectrum from a Grimm-type glow discharge lamp is shown in Fig. 2. In this case, the diameter of the anode is 4–10 mm, and the gap between the anode and sample is 0.2 mm. Gas pressure 10 Torr. The discharge burns between the inner wall of the anode about the part of the sample adjacent to the cylindrical cavity of the anode. Light is observed along the axis of the device.

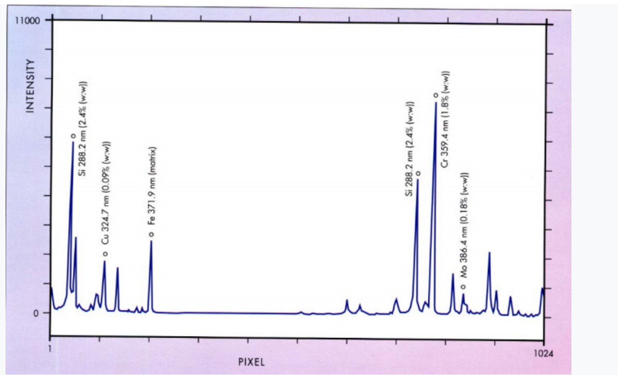

Figure 2: An example of a spectrally segmented emission spectrum of glow discharge using a Grimm-type glow discharge lamp with floating restrictor (8-mm diameter): 50 mA, 3.5 Torr, 2 kV; 22 Plasma array spectrometer (LECO); steel sample: 217A (Research Institute, CKD, Prague).

SECONDARY-ION MASS SPECTROMETRY (SIMS)

Secondary-ion mass spectrometry (SIMS) is a technique used to analyze the composition of solid surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam and collecting and analyzing ejected secondary ions [1]. The mass/charge ratios of these secondary ions are measured with a mass spectrometer to determine the elemental, isotopic, or molecular composition of the surface to a depth of 1 to 2 nm. A schematic of SIMS is shown in Fig. 3.

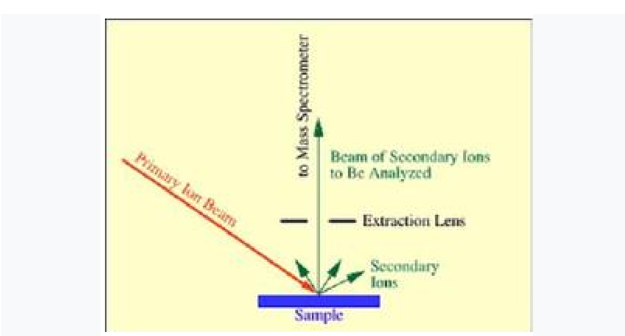

Figure 3: Schematic of SIMS.

Due to the large variation in ionization probabilities among different materials, SIMS is generally considered to be a qualitative technique, although quantitation is possible with the use of standards. SIMS is the most sensitive surface analysis technique, with elemental detection limits ranging from parts per million to parts per billion. A schematic of a typical SIMS system is shown in Fig. 4, where the functional components are described in the figure caption.

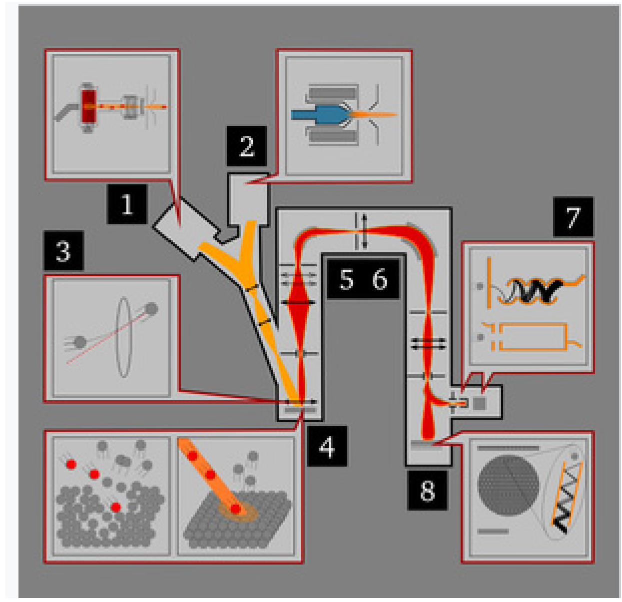

Figure 4: Schematic of a typical dynamic SIMS instrument: High energy (usually several keV) ions are supplied by an ion gun (1 or 2) and focused on to the target sample (3), which ionizes and sputters some atoms off the surface (4). These secondary ions are then collected by ion lenses (5) and filtered according to atomic mass (6), then projected onto an electron multiplier (7, top), Faraday cup (7, bottom), or CCD screen (8).

The components of a SIMS device include (1) a primary ion gun generating the primary ion beam, (2) a primary ion column, accelerating and focusing the beam onto the sample (and in some devices an opportunity to separate the primary ion species by a Wien filter or to pulse the beam), (3) a high vacuum sample chamber holding the sample and the secondary ion extraction lens, (4) a mass analyser separating the ions according to their mass-to-charge ratio, and (5) a detector.

In short, SIMS devices are complex and expensive.

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In the device shown in Fig. 5, the ion flux 6, extracted from the gas-discharge plasma 9, is focused by a spherical emission surface to micron sizes on sample 8, providing very local sputtering and local elemental analysis. The radiation of sputtered atoms is recorded by a spectrometer through window 7 and fiberglass cable 10. The anode is separated from the cathode by an insulator 4.

The anode 1 has an inner diameter of 7-10 mm. It is covered by a diaphragm with a 3 mm hole. The distance between the anode 1 and cathode 2 is set within

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0.25-2.5 mm. Helium, neon, argon, krypton, and/or xenon gases are fed into the cathode-anode gap through the leakage channel 5. The gas pressure is ~1-5 Torr. Better special resolution can be obtained with He gas. A high voltage of 1 kV to 2.5 kV is applied to the anode. Light is recorded along the axis of the device through window 7 and a fiber optic cable 10.

To register the distribution of elements over the sample, the sample is scanned mechanically. An analysis of dielectric samples is possible. The emitted spectrum will be recorded by an Ocean Optic spectrometer with a fiberglass light gate. Related software will be used for spectrum identification. Rapid scanning can be produced with low resolution ~1 mm. A chosen area can be scanned with high resolution ~1 micron. Ions produced in the plasma from sputtered atoms can be extracted and analyzed by a mass spectrometer.

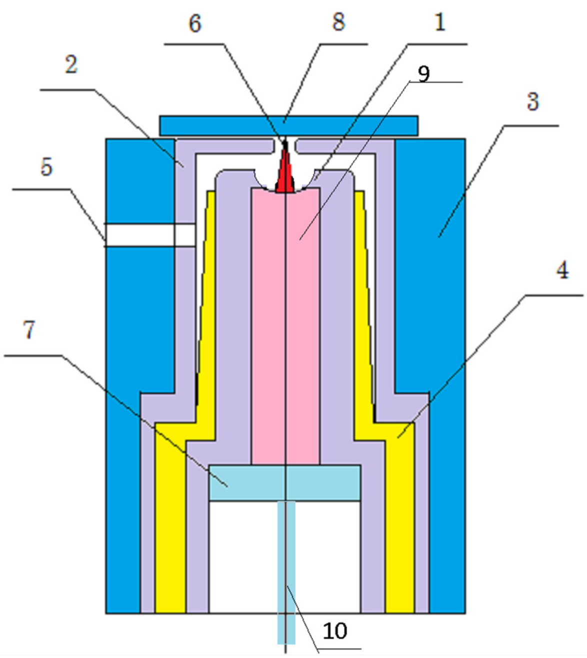

Figure 5: The scheme of the discharge device with self-focusing ion beam. 1- anode; 2- cathode; 3- case; 4- insulator; 5-gas supply; 6-focused ion beam; 7-window; 8-sample, 9-discharge plasma, 10-fiberglas optic.

Figure 6 shows a computer simulation of fine focused ion beam formation.

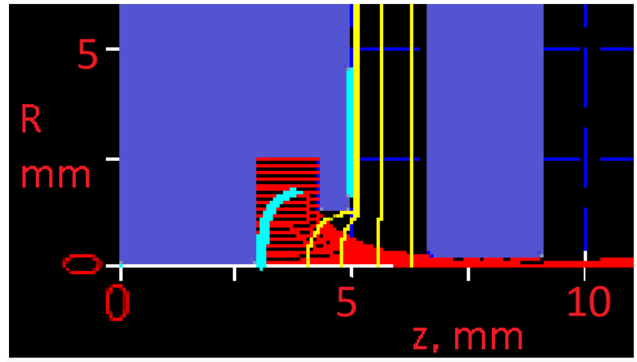

Figure 6: Computer simulation of fine focused ion beam formation.

It is possible to use helium as a working gas, which has a simple optical spectrum that is shown in Fig. 7. Metastable He atoms have high probability for excitation and ionization of impurity atoms.

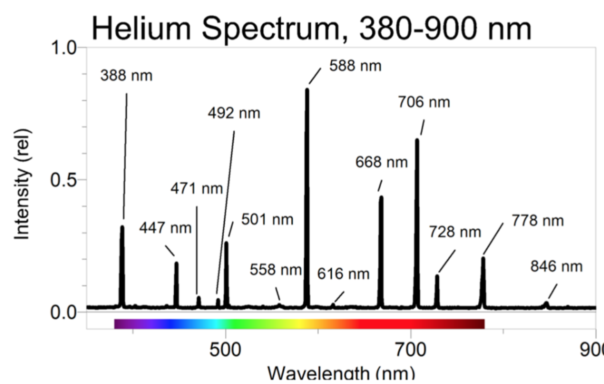

Fig. 7: Optical spectrum of He gas.

The microanalysis methods developed in this project will find broad application in different fields. Advanced surface diagnostics are essential for many fields: in microelectronics fabrication, in geology, in biology, in advanced nanotechnology, and in national security.

REFERENCES

[1] R. Behrisch and K. Wittmaack, Eds., *Sputtering by Particle Bombardment III, Topics in Applied Physics*. Heidelberg, Berlin, Germany: Springer-Verlag, 1991.

[2] J. C. Broekaert, "Glow Discharge Atomic Spectroscopy", *Applied Spectroscopy*, vol. 49, no. 7, p. 12A-19A, 1995.