Beam Induced Chemistry in the ORION® PLUS

Product Information

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Date: June 2009

Application

This note outlines the capabilities of the integrated gas chemistry delivery system in the helium ion microscope and how they may be utilized to create nano-fabrication processes.

ORION® PLUS Capabilities

Beam induced chemistry, high spatial resolution, low sputtering, inert gas beam technology.

Background

Beam induced machining has been carried out in charged particle optical tools for some time. In the simplest case, focused ion beam technologies allow fine scale material removal through sputtering. This capability has been augmented by the addition of gas injection systems (GIS) that provide a locally high concentration of chemically active species at the beam landing point. These gases can be decomposable molecules that leave deposits of desired material on the surface where induced by the beam. Conversely, an etching gas can be introduced, which is chemically reactive – but not spontaneously so – with material in the sample. The beam provides the energy locally to drive the reaction forward so that sample material is volatilized and removed only where addressed by the beam. More recently, SEM technology has been coupled with GIS. Besides providing depositions that are free from the ion contamination coming from (gallium) FIB liquid metal ion sources, etch chemistries provide material removal usually not possible in SEM.

Challenge

There are limitations to both FIB and SEM beam chemistry processes. Any process carried out in a traditional FIB will leave beam atoms in the machined area. Gallium is a metallic contaminant that alters electrical, optical, and chemical properties of materials into which it is implanted. There is also a strong sputtering action due to the high mass of this ion. To provide an example consider tungsten deposition, a common GIS process. For a 30 keV Ga beam, the sputtering yield for the W component in a deposit with a composition along the lines reported in the literature is about 3 atoms/ion. This means that there is a competition between metal addition and removal in the process. Indeed it is common for a non-optimized "recipe" to actually remove material. Finally, the FIB conditions when configured for beam chemistry lead to a beam size of about 10 nm. This imposes an lower limit on the feature size that can be created.

Chemistry carried out in SEM alleviates some of these problems. There is no sputtering or contamination, and the beam size can be smaller. Electron induced chemistry processes, however, are much slower than with FIB. The chemistry proceeds more efficiently at low beam energies – on the order of 1 keV – which means that the user must trade off spot size performance (feature size) for throughput. Concerning etch in particular, there are many materials for which appropriate chemistries have not been found – leaving no ability to perform material removal.

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ORION® PLUS Solution

We are developing an option for the ORION® PLUS which provides a flexible set of tools for creating focused helium beam induced processes. This package includes full integration of the OmniGIS™ product (from Omniprobe, Incorporated) into the microscope. Three crucibles for chemistries and two carrier gases can all be controlled from within the microscope's user interface. The capabilities provided allow the user control over all the parameters needed to develop deposition and etch recipes with the microscope. The system is undergoing beta testing with chemistries for both deposition and etch. We provide here a brief description of the hardware and software capabilities. We also give an illustrative example of a first round of optimization for platinum deposition that was carried out on the machine through a designed experiment.

There are two main functions provided in the system software for carrying out helium beam induced processing. All software features required for running beam chemistry recipes are available in the ORION® PLUS user interface, specifically those for setting up the process gas parameters in the OmniGIS™. This level of integration allows safety protocols to be interlocked as well. The first control panel is for the GIS unit itself. Figure 1 shows two tab pages of the configuration window. In this Figure, XeF_2 etchant is set up for delivery with a nitrogen carrier gas. One can set the temperature of the chemical in its crucible from 0° C to 40° C as well as the duty cycle of the delivery valve at the exit of the crucible into the injection needle. This determines the mass flow rate of the material. The carrier gas flow can be set independently of this in order to allow various degrees of dilution for the chemistry. Real time feedbacks allow the user to see when the delivery system is at the desired parameters. The second tab page, shown on the right of Figure 1, allows the operator to define preset delivery configurations for the GIS control, allowing quick and repeatable selection of chemistry conditions.

The other item needed for recipe development is a beam scanning pattern. Figure 2 shows the control tab page for this function. From here the user can set all the parameters to define an exposure. The total dose applied

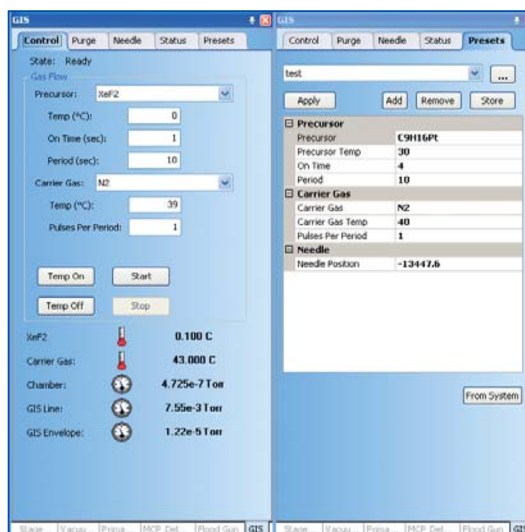

Figure 1. Two tab pages of the GIS control panel.

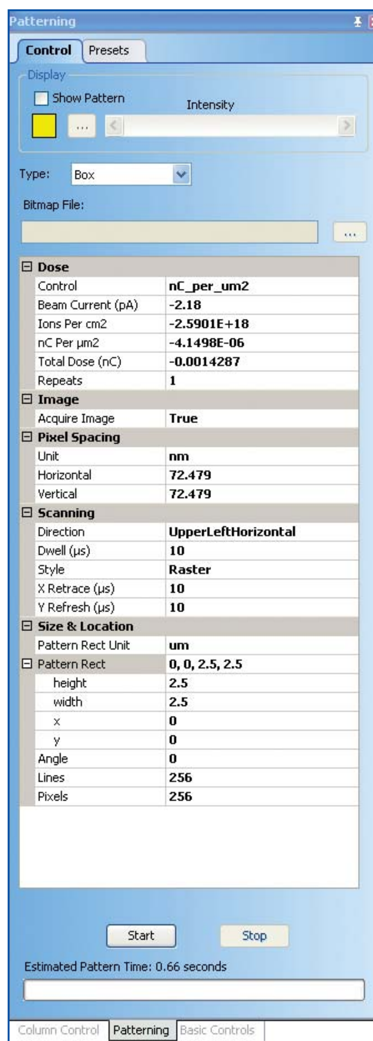

Figure 2. The control tab page of the patterning panel.

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can be set using one of several measurement units, with the beam current being provided from the beam blanking unit in the ion column. The user defines a rectangular scanning area with arbitrary size and orientation. Either pixel density or spacing can be set within the box. The scanning pattern is also user selectable, with raster and serpentine scans in any orientation within the box. One can also acquire imaging signal during the operation. This is useful, for example, when looking for end-point during an etch operation. These scan parameters can also be saved as presets, for quick recollection and re-use. It is also possible within the user interface to import a bitmap with 256 gray levels to drive the scan. In this case, the relative dose at each pixel is defined by the gray level of the bitmap. For more complex patterns, or for more flexibility, the system is capable of being driven by an external pattern generator. There are several commercially available generators that offer advanced functionality. The combination of gas flow and scan strategy defines a complete recipe for a processing application.

We present now an example of this system in operation for developing a process. An experiment was carried out to do initial characterization of platinum deposition rate and material purity. A full factorial statistically designed experiment was used. The experimental design was a 3 factor, 2 level full factorial design with a full replicate and center points. The JMP software package was used for analyzing the resulting data. The center point of the experimental parameters was empirically selected based upon several screening experiments conducted in advance. The recipe experimental parameters were beam current, deposit area, and pixel spacing. All other known variables were held fixed. Figure 3 shows a top-down survey image of the deposits created for the experiment. The randomized variation of the deposit area parameter can be noted from the results. The volume of the deposits was determined by atomic force microscopy, in order to determine the deposition rate. Energy dispersive spectroscopy (EDS) was utilized to determine the atomic percentage of platinum in the deposits. A molybdenum substrate was used in order to avoid any EDS peak overlap with Pt, C, or O signals.

Once the deposition rate and atomic percentage for each experimental run had been determined, the data was statistically analyzed using the JMP statistical analysis package. A model was fit to the data, including all interaction terms up to degree 2. After determining which experimental parameters were statistically significant, a mathematical model was created to model the data. In this way predictions can be made of the influence of the chosen recipe parameters on the resulting deposition. The analysis for deposition purity (at. % Pt) revealed that pixel spacing and beam current are the primary determinants of purity. There was detected, however, an interaction between beam current and deposit area that influenced the outcome. Therefore the experimenter must take all three of these factors into account when optimizing this recipe. Figure 4 plots the deposition purity vs. rate. There is a noticeable increase in the at. % Pt for slower deposition processes. The model predicts that 40 at. % should be achievable. Further parametric studies are required in this area. The influence of beam current on

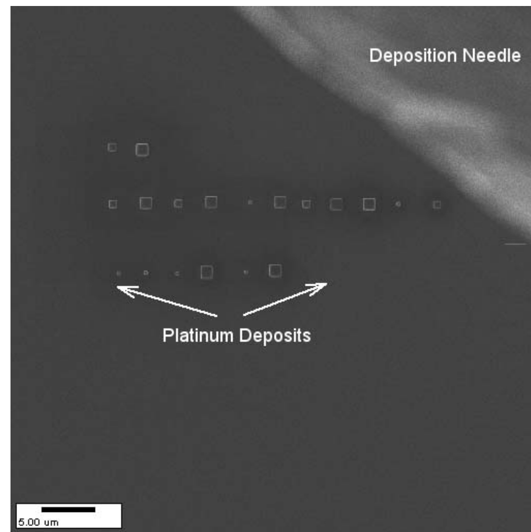

Figure 3. Platinum deposits created for the designed experiment.

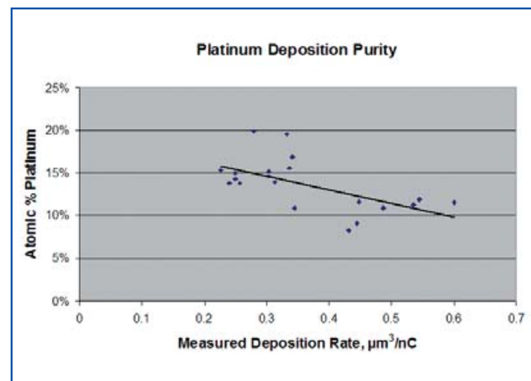

Figure 4. Dependence of deposit purity on deposition rate.

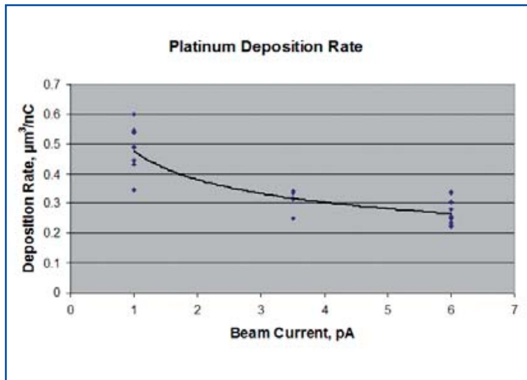

Figure 5. Dependence of deposition rate on helium ion beam current.

deposition rate was also characterized, as is summarized in the plot of Figure 5. The rates are comparable to Ga FIB processes and an order of magnitude higher than electron beam induced growth. We also see a greater rate for lower ion beam current. This is an indication that the work thus far has probably been carried out in a gas depleted regime. Thus there is again more opportunity to optimize this process.

The above exercise demonstrates a system that is fully capable of being used for beam chemistry process development. Four different chemistries have been tested in the ORION® PLUS, to varying degrees. In no instance was there disruption to the gas ion source or optics, so the system is stable for use with such processes. The OmniGIS™ has been used with a wider variety of chemistries; see Table 1 for the list of released chemistries on that product. Several others have been tested experimentally. Most of these have yet to be characterized for ORION® PLUS use, so there are a significant number of options for further research.

Chemistry	Purpose
$(CH_3)_3(CH_3C_5H_4)Pt$	Platinum deposition
Tungsten precursor $W(CO)_6$	Tungsten deposition
TEOS	SiO_2 deposition
TMCTS	SiO_2 deposition
Styrene	Carbon deposition
Naphthalene	Carbon deposition
XeF_2	Etching

Table 1: Released chemistries on the OmniGIS™.

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