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Simultaneous Specimen & Stage Cleaning for Analytical Electron Microscopy

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An innovative technique has been developed which allows simultaneous cleaning of a sample and a specimen stage which minimizes and, in some cases eliminates, contamination of inorganic specimens analyzed via SEM, TEM, STEM and AEM.

The technology involves subjecting the specimen and the specimen stage to a reactive plasma gas which efficiently removes a wide range of contaminants from critical surfaces. The procedure may be carried out prior to inserting the specimen and specimen stage into the EM via an externally produced accessory unit and can be extended for use on all types of electron microscopes where the sample and stage are loaded external to the instrument. Such an instrument was produced and implemented first by Dr. Nestor Zaluzec at Argonne National Laboratory[1]. His system modified a commercially available plasma etching system which included adapting it to accept a side entry holder. Commercialized systems are now available which are designed specifically for this application, some of which are also capable of accepting top entry holders. An alternative approach would be to incorporate the technology directly into the electron column instrumentation itself, where not only the specimen and stage should be cleaned, but also the relevant interior parts of the microscope[1,2].

Background

Microcharacterization of materials by electron microscopy is ultimately limited by the ability to observe, detect and analyze the constituent materials present in microvolumes of specimen. In the environment of the electron microscope, the interaction of surface borne contaminants with the high energy incident electron probe can create deposits on the surfaces of a sample which can prohibit these analyses. These contaminants may be introduced directly by the specimen, deposited on the specimen via the specimen stage or deposited on the specimen via the microscope system. Electron microscope manufacturers attempt to minimize the last of these by judicious design. However, the first two sources of contaminant introduction are out of their direct control.

Surface borne contamination is introduced in the preparation of inorganic specimens (metals, ceramics, semiconductors, etc.) for EM work. The materials to be studied are frequently subjected to chemical or electrochemical polishing followed by solvent rinsing and air drying. This typically leaves residual organic material on the specimen surface. Attempts to clean the specimen stage include rinsing the stage with various solvents. Improper or poor techniques used to store both specimens and/or stages prior to insertion in the microscope can introduce additional organic residue. Finally, contamination derived within the instrument can mitigate analysis. While the majority of the organics involved in these processes dissipate, a small amount generally remains on the surfaces and is sufficient to cause problems when the specimen is subsequently examined in modern analytical microscopes. Although these organic residues are widely distributed and generally at low concentrations on the various surfaces, they can become mobile in the microscope environment and are attracted to the periphery of any focused electron probe, forming deposits. Since these contaminants can travel large distances over the surface of the specimen, it is important to remove or immobilize them as much as possible prior to an analysis without disturbing the microstructure of the specimen. It is important to note that once the deposits are formed, they are not easily removed. Analysis of a corrupted area is precluded.

Procedure

It has been well documented that low temperature/energy plasmas can be used to reactively etch/ash organic materials found on the surface of inorganic materials[3,4]. This has been used by the industrial community to clean semiconductor wafers and other bulk materials for many years. In plasma cleaning

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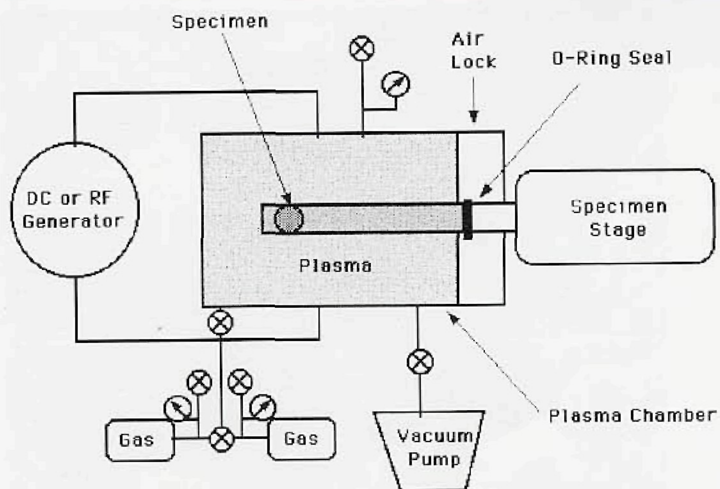

Figure 1. Schematic of a Plasma Coater for TEMs.

procedures for electron microscopy, a related technique is employed for cleaning EM specimens, however, instead of subjecting only the material of interest (i.e., specimen) to the plasma, the entire stage and sample is plasma cleaned.

This last aspect is critical to the application of the technology to the modern AEM. The ANL patented technology[2], permits this by the incorporation of an airlock system into a plasma reaction chamber. The seal for this airlock is the 'o-ring' of the typical side entry stage (Figure 1), thus permitting direct access of the plasma to not only the specimen, but also the entire area of the side entry stage which is introduced into the vacuum environment of the electron microscope. The result of this is that hydrocarbon contaminates are removed completely from the immediate sample environment. For the special case of top-entry HREM stages, where an o-ring seal is not present, the entire stage and specimen can be introduced into the reaction chamber. This latter arrangement has also been facilitated in some commercial implementations.

The nature of the gas used to create the plasma selected is based upon the desired effect. Although an argon/oxygen mix is generally used for hydrocarbons, specific gases (bc13, cf4, ...) may be used to tailor a reaction.

Different gases have pronounced effects, some remove hydrocarbons, while others can be used to gently thin specimens by removing surface layers using a surface sensitive plasma/chemical reaction. Cleaning time for hydrocarbon removal is typically 10-30 minutes. Specimens that previously contaminated in minutes can be studied for several hours after treatment.

As this is a relatively new technique as applied to electron microscopy, procedures have not yet been documented for every sample set. These procedures, the effects of power settings, thermal control requirements and the selection of gas species will be the subject of future articles.

1. Dr. Nestor J. Zaluzec, Argonne National Laboratory - personal communication.

2. Untied States Patent No. 5,510, 624 by Argonne National Laboratory and the University of Chicago

3. The Use of Plasma Cleaning for the Preparation of Clean Electron Transparent Thin Foils, Zandbergen et al, icem 13-paris, 17-22 July 1994 pp 1003-1004.

4. Use of Chemically Reactive Gaseous Plasmas in Preparation of Specimens for Microscopy, Richard S. Thomas, Techniques and Applications of Plasma Chemistry, Wiley-Interscience publication, 1974, Chapter 8, pp 255-346.

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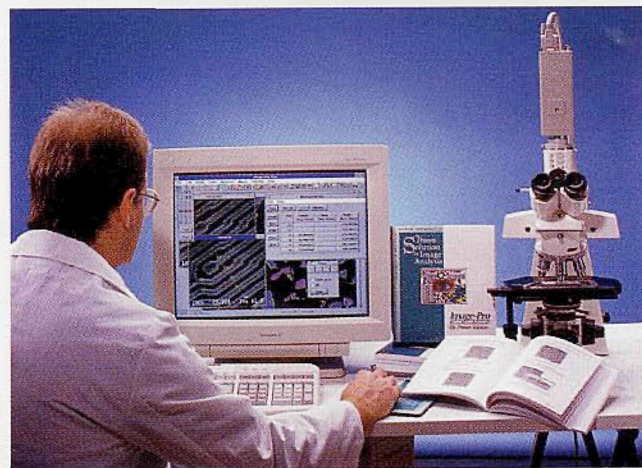

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