Model PC 150

Plasma Cleaning of Various Specimens for use in TEM

Applications Laboratory
Report 36

Sample
Preparation

1.0: Purpose

Plasma cleaning is becoming a commonly used technique for both preventing contamination from entering into the microscope and to prevent it from building up on the specimen during analytical electron microscopy. To determine where most contamination originates and how successful the cleaning process is, several different types of specimens were subjected to the plasma cleaning process. Standard TEM specimens, specimen holders, as well as microscope pole pieces and other parts were all cleaned using the PC 150 to determine how well contamination was removed.

2.0: Experiments and Procedures

Most of the aim for this evaluation was trying to track down the main source of contamination when performing focused probe microanalysis in the TEM. Most of the cleaning was attempted using pure O_2 as the process gas, since hydrocarbons are easily removed with pure O_2. Oxygen reacts with the hydrocarbons to form CO, CO_2 and H_2O which are pumped away by the vacuum system. The Ar component of plasma cleaning which is normally recommended was not used in this experiment (in most cases) since we have found the contamination rate of O_2 plasma cleaned specimens to be much less than those treated with Ar plasma of equal time and power. Initially a few sample holders for Philips TEM's were cleaned to see the effectiveness of the pure O_2 process on contamination reduction. After a few of these tests were evaluated and seen to reduce the contamination rate to a level of acceptability, all other processes were run with confidence.

Below is a list of all of the samples and process parameters which we ran using the PC 150. Everything from TEM specimen holders, TEM specimens, tweezers, specimen handling equipment, TEM stages, Hot Cell TEM stage for in situ heating experiments, apertures and other TEM holder parts were cleaned in the unit.

Plasma Cleaning Parameters for ASU Experiments
PC 150

#	Sample Description	Gas	Power (Watts)	Time (minutes)	Pressure (mTorr)	DC Bias (volts)
1	Plain holder; CM 20	Oxygen	10	15	208	-395
2	Plain holder; CM 400	Oxygen	30	15	210	-444
3	Holder + specimen; Si plan view	Oxygen	10	15	204	-398
4	Holder + specimen; AlCu alloy	Oxygen	30	15	208	-380
5	Holder + specimen; Si sample	Oxygen	13	20	197	-388
6	Tweezers, tube tips; holder	Oxygen	13	30	202	-413
7	Holder + XTEM Si specimen; epoxy sandwich	Oxygen	13	30	202	-415
8	Plain holder; CM 200	Oxygen	14	30	194	-422
9	Glass jar; Be parts	Oxygen	14	30	197	-395
10	Si; AlCu; Stainless Steel samples	Oxygen	13	15	200	-400
11	Si/Si; SiNb; Stainless Steel samples	Oxygen	14	15	200	-398
12	Contaminated specimen; Si w/beam spots	Oxygen	15	10	201	-399
13	Contaminated specimen; Si w/beam spots	Ar/O2; 50/50%	15	30	204	-401
14	Contaminated specimen; Si w/beam spots	Argon	15	10	200	-399
15	Various samples	Oxygen	13	15	201	-400
16	TEM holder parts	Oxygen	13	15	235	-385
17	TEM holder parts; double tilt parts	Oxygen	18	20	250	-436
18	Hot cell for TEM stage; heating stage	Oxygen	15	20	209	-405
19	Heating holder; stage parts	Oxygen	15	20	210	-409
20	Plain holder	Oxygen	15	10	200	-398
21	Au film on Holey Carbon Film	Oxygen	10	2	200	-398

3.0: Results and Discussion

In general, all of the cleaning processes were successful in the removal of hydrocarbons from the surface of the cleaned sample and drastically reduced the contamination rate when tested in the microscope. Some of the observations made are discussed below.

3.1: Specimen Holder Cleaning

- Specimen holders were cleaned using only pure O_2 for almost every process step. The holders were found to be completely free of contamination following the cleaning process of around 15-20 minutes.
- Holders of various microscope types (CM20, CM 200FEG, CM 400) all were cleaned using the PC 150. Every holder was processed for approximately the same amount of time (~ 15–20 minutes per holder).
- A heating stage holder was also cleaned using the O_2 process, which eliminated the contamination of this holder completely.

3.2: Specimen Cleaning

- An Al / Cu alloy specimen was cleaned to remove the contamination present on the specimen. The specimen was left under the focused probe of the electron beam for over 3 hours and no further contamination developed. There did appear to be some radiation damage from the electron beam, however this was found to be existing beam damage from previous evaluations in the TEM. The specimen was prepared around 1977, therefore the damage and contamination seen was already present on the specimen.
- A Si specimen was plasma cleaned and then placed into a clean TEM holder to see if there would be any contamination build up following plasma cleaning. The Si specimen was allowed to sit under the focused probe of the electron beam at high current density and no further contamination of the specimen resulted.
- The next experiment was to find out which source of contamination contributes the most to C spot formation: the specimen, specimen holder, or microscope. This was done in the following manner: Si specimens at different stages of cleaning were prepared: a) as received from the ion mill (a contamination free ion mill); b) non-plasma cleaned; c) plasma cleaned. The same was done with sample holders: a) as received (uncleaned); b) non-plasma cleaned; c) plasma cleaned. After the samples and holders had been prepared in the manner described above, the following experiment was carried out:
- A dirty specimen and dirty holder were evaluated in the TEM using a focused electron beam probe and were found to contaminate badly (as expected).
- A dirty specimen holder and clean specimen were evaluated in the same fashion and the specimen did contaminate as a result of the dirty specimen holder being used.
- A clean holder and dirty specimen also were evaluated in the same manner and the specimen did NOT contaminate
- An as received specimen (ion milled) was then placed into a clean holder and again evaluated in the same fashion; the specimen did NOT contaminate
  - ➢ **NOTE: These results are still pending, the observations discussed above are preliminary in nature.**
- A cross section TEM specimen was prepared using the standard dimpling/ion milling techniques, which incorporates epoxy adhesives for gluing materials together during preparation. Concern about the epoxy being affected during plasma cleaning led to the investigation of this. The specimen was first evaluated in the TEM prior to plasma cleaning, with a focused probe allowed to dwell on the epoxy / Si interface for over 5 minutes. There were no C spot formations neither created nor was the epoxy damaged. Following this TEM evaluation the specimen was plasma cleaned for 30 minutes to evaluate if any changes in the epoxy take place (i.e. is the epoxy intact?). Upon evaluation, the specimen itself did not contaminate and the stacks of material were left intact, however, C spot formation occurred in the epoxy area AFTER plasma cleaning. The specimen itself did NOT contaminate however, and it remained intact during the microanalysis.
- A Carbon Holey Film containing a thin Au film on the grid was also cleaned. Although the film itself was going to be attacked by the O_2 plasma, it was run for a short time to remove the contamination present on the sample.

3.3: Miscellaneous Part Cleaning

- Several different stage parts were cleaned using the PC 150, mostly anti-twist washers, clamping rings, small TEM probes, etc. to eliminate any sources of contamination possible.
- Tweezers, specimen holders, vacuum tips, etc. also were cleaned in the PC 150.

- A Hot Cell stage for the TEM was also cleaned. This was a major source of contamination inside the microscope and therefore the hot cell stage was removed and cleaned using the O_2 plasma.
- The actual stage of the TEM (goniometer portion) was also removed from the TEM and cleaned. This again was done to track down the original source of contamination.

4.0: Conclusions

It has been shown that the PC 150 is unique in it’s capability to handle oddly shaped and size parts for cleaning, ranging from small TEM specimens up to large pole pieces and specialized stages. This wide range in flexibility allows the instrument to be applied for locating contamination sources, routine cleaning of all microscope parts which are exposed to vacuum, and for performing precision cleaning of specimens prior to high resolution or analytical transmission electron microscopy.