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ADVANCED VACUUM SYSTEMS FOR ANALYTICAL ELECTRON MICROSCOPY

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Abstract

Recent technological advancements such as significantly improved power supply stability and polepiece design, as well as increased accelerating voltage all contribute to the primary objective of the scanning electron microscope (SEM): higher resolution. Similarly, the advent of analytical electron microscopy (AEM) has also expanded the scope of applications to include energy-dispersive spectrometry, wavelength-dispersive spectrometry, and electron probe analysis. Specimen contamination and damage has become increasingly problematic as SEMs are used as analytical instruments. In an effort to minimize contamination and etching, and thereby benefit from these advances, cleaner and more efficient vacuum systems are required. Such benefits were realized when, for the first time, a Cameca microprobe's diffusion pump was replaced by a low vibration cryogenic vacuum pump, resulting in faster pump down and lower ultimate vacuum pressures.

Key Words: Cryogenic vacuum pump, high-vacuum technology, contamination reduction, analytical electron microscopy, vibration isolation, ultimate vacuum pressure.

Introduction

A high-energy beam of electrons used for electron microscopy and microanalysis can have significant impact on the specimen. Beam damage is usually more severe in organic and biological specimens (8); it has been reported by numerous investigators that this damage ranges from loss of organic material (6), and removal of volatile elements to significant hydrocarbon contamination (2,10) and etching. Hydrocarbon contamination can, by absorbing radiation, decrease X-ray intensities, thereby degrading analysis. This effect is much more noticeable during light element analysis.

While specimen cooling (4,7,12) has been shown to reduce mass loss considerably, specimen cooling itself can create problems. If the microscope's vacuum system is not generally free of hydrocarbon contaminates, the cooled specimen can then act as a contamination trap, causing further complications. It is advantageous, therefore, to not only reduce the pressure (14) in the specimen chamber but also to insure that the vacuum system itself is not a source of contamination.

This paper describes the achievement of excellent vacuum levels, high pumping speeds, reduction of the water vapor partial pressure (11) and, most importantly, the total elimination of vacuum system-induced contamination (1) through the replacement of the original vacuum pump with an Air Products Low Vibration Cryopump.

Original Vacuum System

A Cameca Camebax-MBX combined scanning electron microscope (SEM) and microprobe, consisting of three Cameca wave-length dispersive (WDS) spectrometers and a single energy-dispersive spectrometer (EDS), was chosen for modification. The SEM (see Figure 1) had been designed and installed with a fully automated vacuum system which included all the necessary safety and control devices. The vacuum system consisted of a 700 liter/sec diffusion pump, a water-cooled baffle, a liquid nitrogen trap, a vacuum ballast tank and two 12m^3/h mechanical vacuum pumps.

The elimination of all vacuum system-induced contamination, meaningful pressure reductions and a substantial lowering of the present partial pressure of water vapor would significantly improve the microprobe's analytical capabilities. A table of the advantages and the disadvantages of major vacuum systems is shown in Figure 2. Of these systems, only the cryopump is totally contaminant free; it also reduces substantially the water vapor partial pressure (6) because of its high intrinsic pumping speed for water vapor, approaching the theoretical limit.

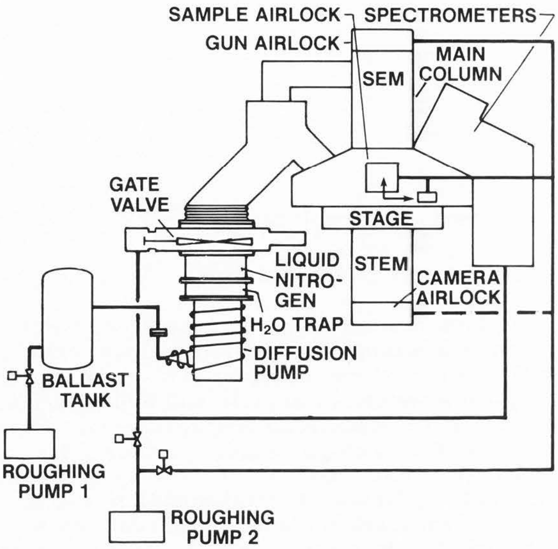

Fig. 1. Original Cameca SEM

Low Vibration Cryopumps

Advantages

- Requires no continuous fore-pumping
- No LN_2 required
- Highest pumping speeds for all gases
- Contamination free
- High throughput
- Pumps dangerous gases
- No moving parts exposed to vacuum chamber
- Field maintainable
- Cost comparable with turbo pump
- Low maintenance intervals (10,000 hr)

Disadvantages

- Regeneration required
- Longer initial startup times
- Vertical installation only

Diffusion Pumps

Advantages

- Low initial cost
- No vibration

Disadvantages

- Requires LN_2 to prevent contamination and to pump condensables
- Restricted to vertical mounting
- Requires continuous forepumping
- Lower pumping speeds than cryopump
- Potential for catastrophic failure
- Oil creep problem can be significant
- Not contamination free

Turbomolecular Pumps

Advantages

- No LN_2 required
- Fast initial startup
- Horizontal or vertical installation

Disadvantages

- Lower pumping speeds than cryopump for water and hydrocarbons
- Potential for oil backstreaming from roughing pump or bearings
- Requires continuous forepumping
- Frequent bearing maintenance
- Cost comparable with cryopump
- Potential self-destruction if particle falls into turbine blades
- Vibration must be considered
- Splinter shield recommended but does decrease net pumping speeds
- Not contamination free

Ion Pumps

Advantages

- Requires no continuous fore-pumping
- No LN_2 required
- No vibration
- Contamination free

Disadvantages

- Low throughput
- Gas re-emission, memory effects
- High cost
- Difficult to start
- Strong magnetic field
- Electrical isolation required for some applications
- Extremely heavy
- Not field maintainable

Fig. 2. Pumping Systems Comparison

Vacuum System Testing and Modification

Prior to the modification, the diffusion pump system was evaluated. Testing determined that the existing primary vacuum gauge, chosen by Cameca and located directly on top of the pumping stack, was correctly indicating and recording specimen chamber pressures. The maximum obtainable vacuum levels using the diffusion pump were evaluated and are shown in Figure 3, which shows baseline pressure readings at the primary vacuum gauge over several days of continuous pumping with and without LN_2 in the primary trap.

The final series of tests involved pump down cycles from atmosphere to high vacuum, obtaining normal operating conditions of $5x10^{-6}$ torr with and without LN_2 in the primary trap. These test results are shown in Figure 4.

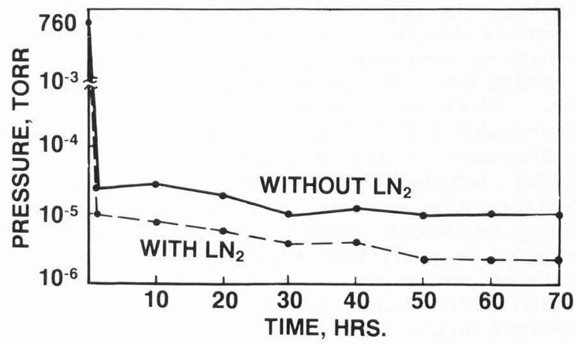

Fig. 3. Diffusion Pump Vacuum Levels

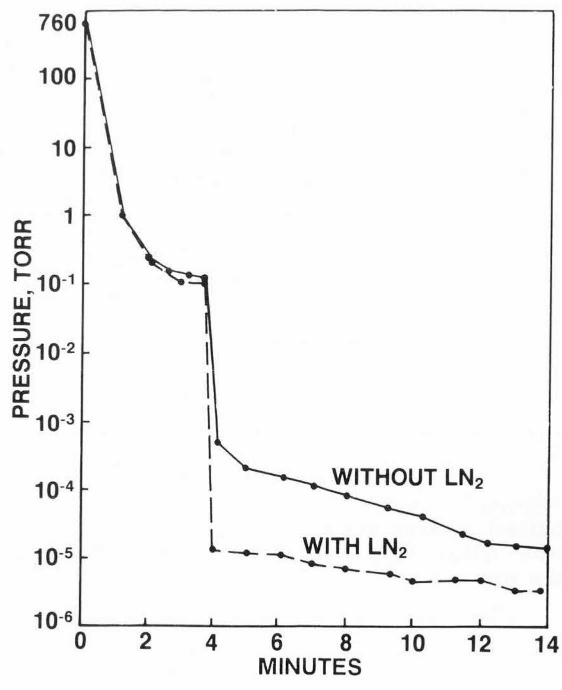

Fig. 4. Initial Diffusion Pump Vacuum Levels

After the entire microprobe was cleaned using standard ultra-high vacuum procedures (3,5,9), a low vibration cryopump, with a pumping speed of 680 liter/sec for N_2, was installed in place of the diffusion pump. In replacing the diffusion pump with the cyropump, the LN_2 (15) trap and the water baffle were no longer required, as these items were used on the original vacuum system to prevent oil backstreaming from the diffusion pump. It is important to note that their use in preventing backstreaming had the adverse effect of reducing the diffusion pump's pumping speed by approximately 40%. Figure 5 shows the modified system.

Vacuum System Data

The performance of the modified vacuum system is shown in Figure 6. This graph compares an average pump down cycle of the diffusion pump system with that of the low vibration cryopump. After 70 h, the final recorded vacuum levels were 5 x 10^{-6} for the diffusion pump, with the LN_2 baffle and water trap, and 1 x 10^{-7} for the cryopump. Though clearly the superior vacuum source, the cryopump was not available for use on vibration-sensitive equipment such as the microprobe until the development of the Air Products Low Vibration Cryopump. Its successful use on SEMs is well documented (13). In this installation and numerous others, including dual pumped transmission electron microscopes, no detectable vibrational interference has been recorded using this cryopump.

Conclusions

Successful installations of the Low Vibration Cryopump in the disciplines of electron beam microscopy and microlithography now include Hitachi SEM Models S570 and S800, and TEM Models H600 and S800. JEOL installations include TEM Models 100CX, 200CX, and 1200EX, and SEM Model 35C. ISI Microscopes SEM installations include Models SS-40 and IC-130.

Microlithography, Ion Beam, and X-ray Lithography all use and benefit from cryopumping. Residual gas analysis studies at a number of user sites have clearly shown the benefits of cryopumping through the significant reduction of hydrocarbons, improved vacuum levels and substantial reductions of water vapor partial pressures, all contributing to the growing acceptance of this technology.

In consideration of the on-going programs, cryopumping should help achieve new goals in microanalysis and electron microscopy.

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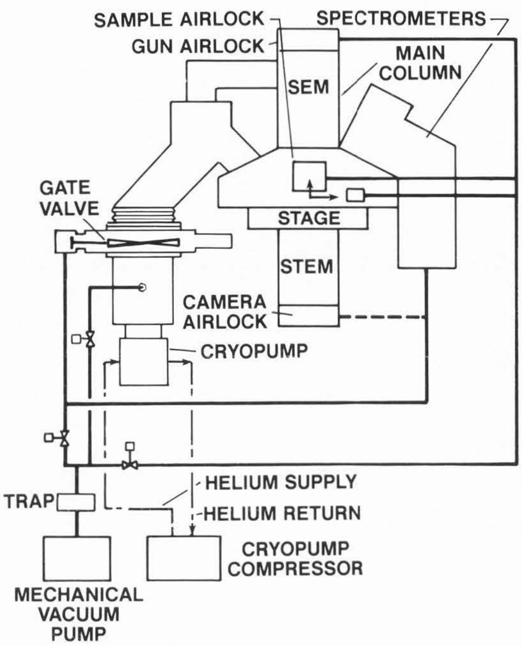

Fig. 5. Modified Cameca SEM

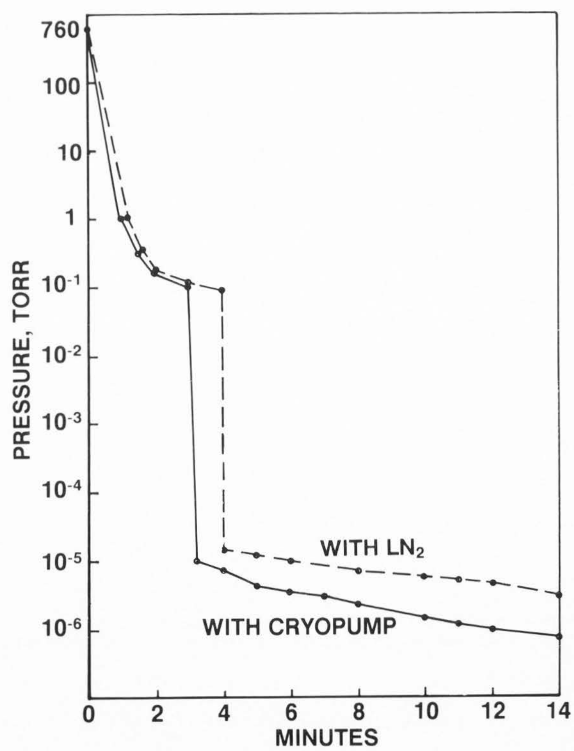

Fig. 6. Vacuum Levels: Diffusion Pump vs. Cryopump

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Discussion with Reviewers

J.T. Thirlwall: Will the author be more specific about the cleaning of the entire microprobe using "standard ultra-high vacuum procedures", in particular, to what extent were elastomer seals, thin windows and other components replaced or processed to reduce the release of contaminant materials?
Author: The entire microprobe was totally disassembled once all base line data had been completed. This included all optics, coils, and spectrometers. Our goal at this time was to remove all residual contamination that was visible or invisible. The actual method then utilized begins with a thorough washing of all items, using hot water and "Alconox" detergent. Once air dried, a chlorinated solvent, such as Chlorethylene NU, is used and all parts again washed carefully. We then rinsed all parts with methyl alcohol and allowed them to dry. The final cleaning step involved the ultrasonic cleaning of all components in Freon TF, wrapping each finished part in clean aluminum foil until ready for reassembly. All processes were done wearing gloves to prevent introduction of finger contaminants. This process was performed on all components removed from the microprobe and includes plastic components o-rings and glass.

J.T. Thirlwall: Can the author provide some measure of the reduction in contamination rates achieved in their microprobe?
Author: Unfortunately, we did not have an opportunity to take residual gas analysis of this system either before or after the modification. However, it is important to point out that the cryopump system is totally oil-free and, therefore, in no way could it contribute to the system contamination. Actual RGA analyses have been performed by both end-users as well as manufacturers and have, indeed, proven substantial reductions in contaminants and the actual reduction of the important water vapor partial pressures.

J. Wall: What are the main sources and magnitudes of gas loads in your system?
Author: In this particular installation and in a majority of today's vacuum systems, very few systems are allowed to be brought up to atmospheric pressure using ambient air. In fact, 90% of vacuum systems are now backfilled with dry nitrogen gas in order to reduce both the water vapor partial pressure and contaminant problems. This installation included the incorporation of a dry, nitrogen-backfill system and, therefore, the majority of our present gas load is now nitrogen.

J. Wall: Have you used Viton O-rings?
Author: Yes, in fact, we insured that all o-rings were comprised of the Viton composition. Each was thoroughly cleaned, as previously described, and then prior to installation, sparingly lubricated with low vapor pressure grease (perfluoropolyether) to aid in their sealing integrity.