Electron Energy-Loss Spectroscopy in an Environmental TEM

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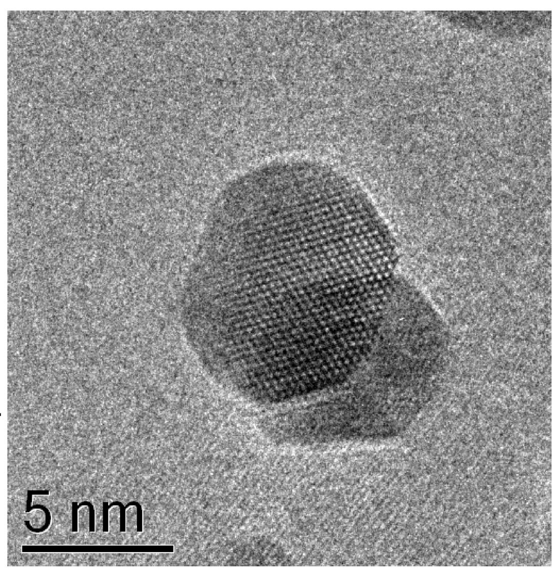
Introduction

Since the advent of environmental transmission electron microscopy (ETEM), numerous reports have appeared in the literature with topics ranging from catalytic applications such as carbon nanotube (CNT) growth, particle reshaping and reduction/oxidation phenomena to nanowire growth.

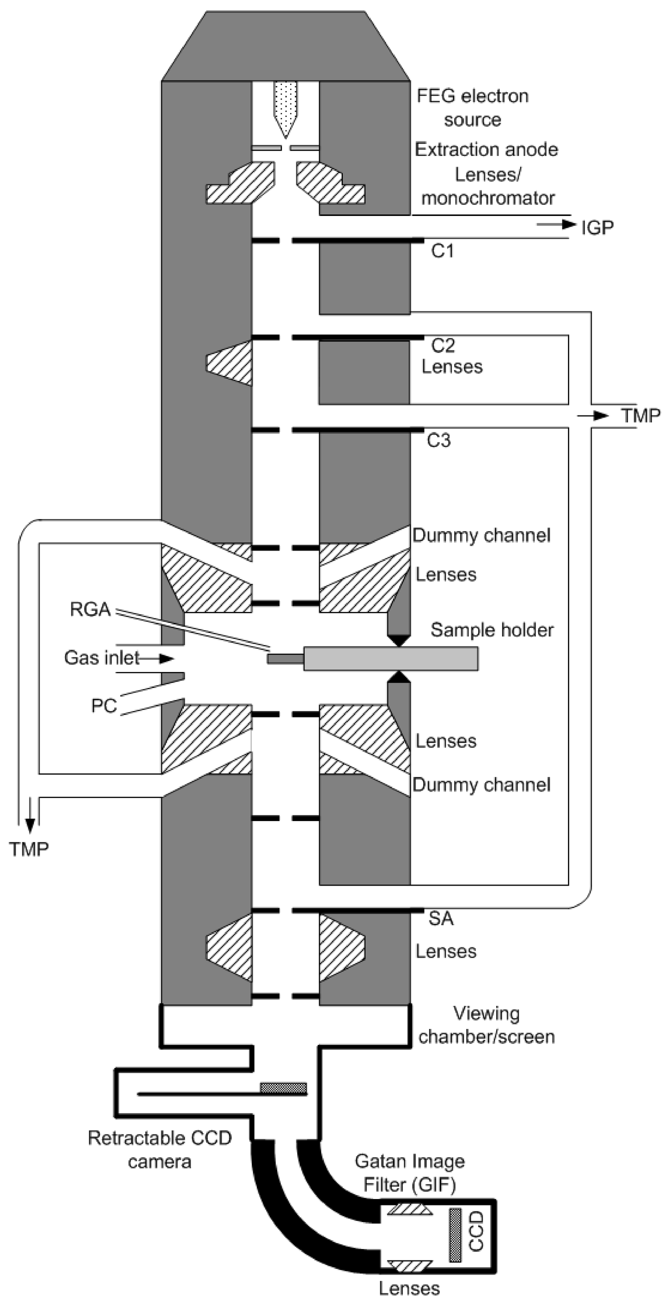
Here we access how the presence of gas in the objective pole piece gap affects spectra and images. This is important in the light of recent improvements in TEM performance in the form of aberration correctors and monochromators.

Environmental TEM

- Experiments were carried out in an FEI Titan 80-300 equipped with objective lens aberration corrector, a monochromator and a 3-step differential pumping system.
- In this microscope, imaging and spectroscopy is possible in the presence of up to ca.1500Pa gas pressure.
- Stable pressure is ensured by using digitally controlled mass flow controllers.

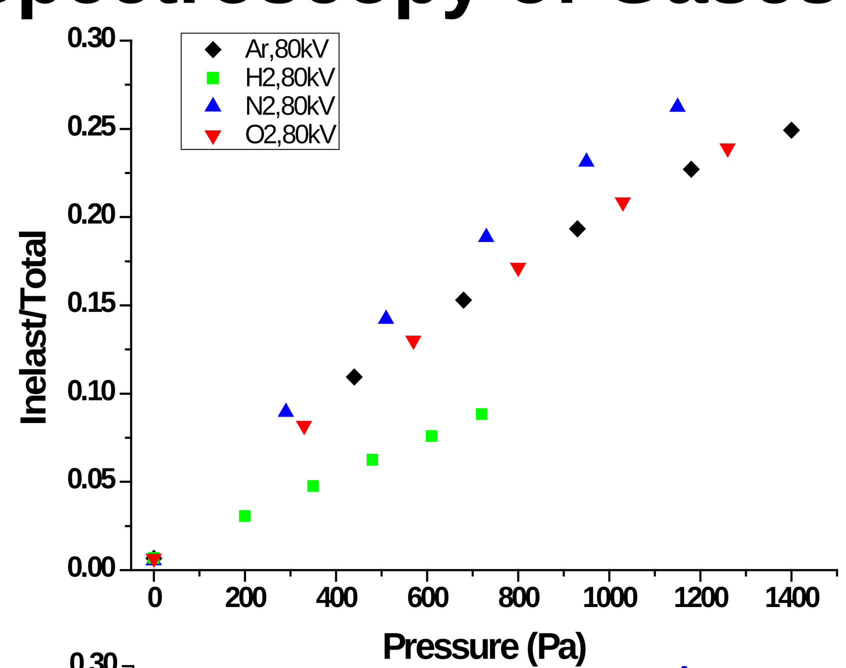


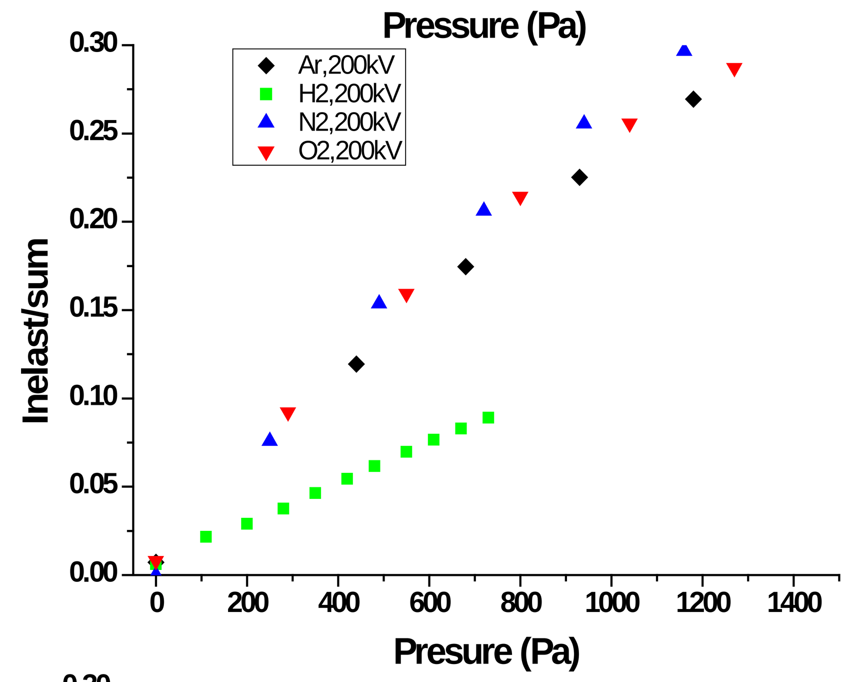
Au particle in 320Pa O_2 imaged at room temperature

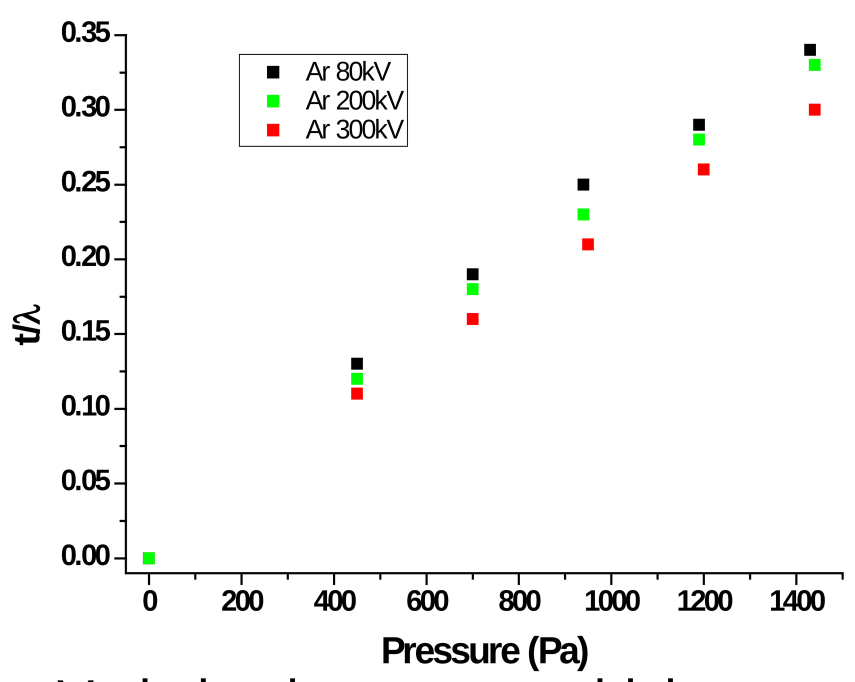


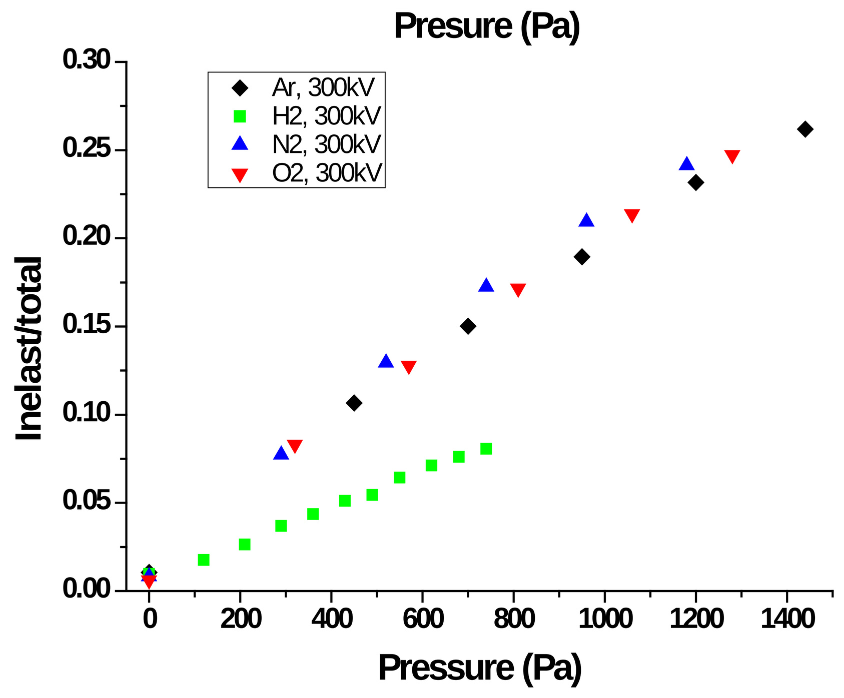
Electron Energy-Loss Spectroscopy of Gases

- Electron energy-loss spectra from gas with no specimen in the column were acquired at 0.1eV/ch dispersion.
- Zero loss peaks were extracted from the spectra by fitting them to a Gaussian/Lorentzian^2 model.
- The plots on the right show the fraction of inelastically scattered electrons as a function of gas pressure.





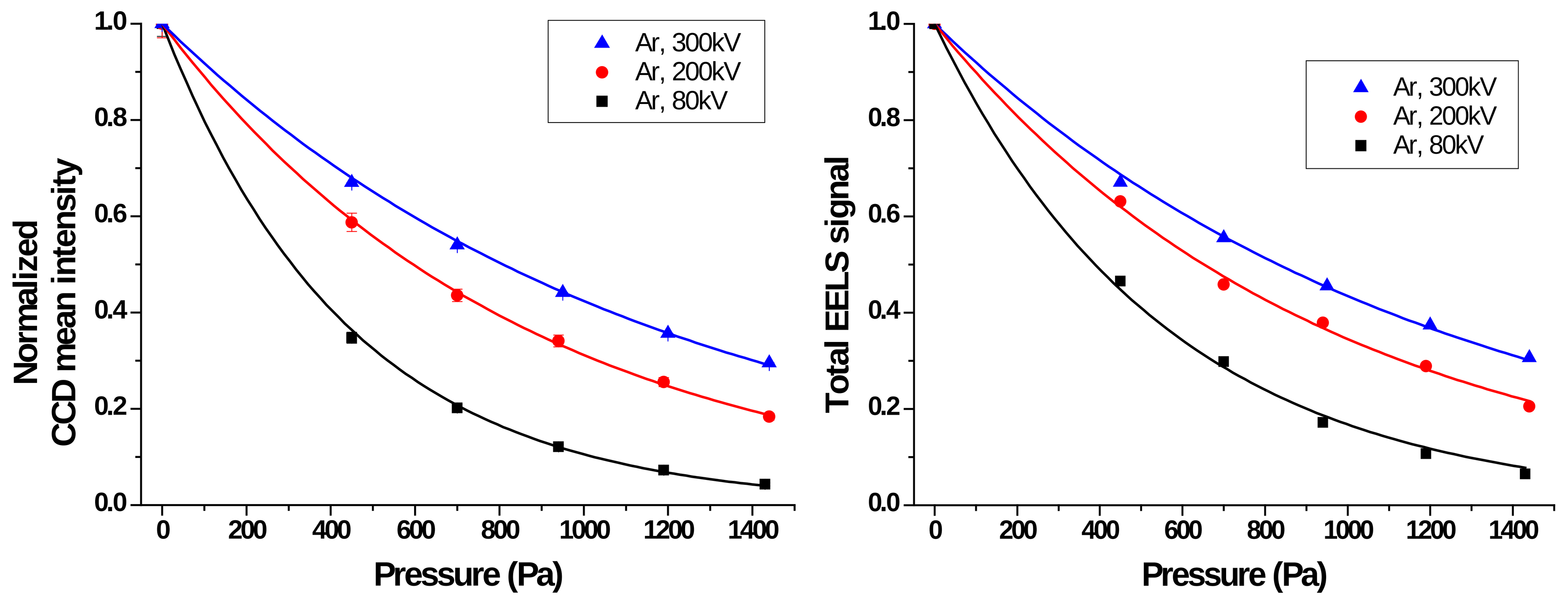




- Variation in apparent thickness of gas plotted in units of the inelastic mean free path as function of pressure and acceleration voltage. Note that t is fixed while λ varies.

Interaction of Fast Electrons with Gases

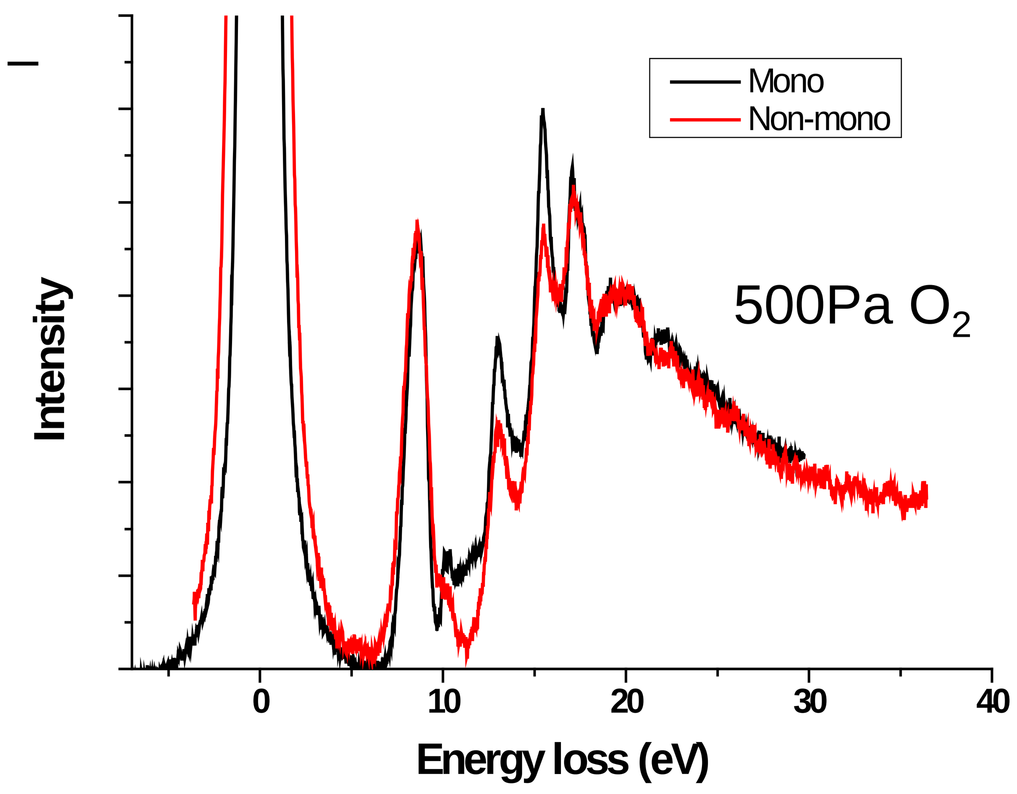
- Image intensity in the presence of gas was measured on a Pre-GIF US1000 CCD camera without a specimen and without varying the parameters of the illumination system of the microscope.
- Variations in image intensity were used to determine total (elastic and inelastic) scattering cross sections and mean free paths.
- Total intensities in the energy-loss spectra were also measured up to 150eV energy-loss.



	80kV		200kV		300kV	
	σ /m^2 (x10^{-22})	λ/10^{-3}m (500Pa)	σ /m^2 (x10^{-22})	λ/10^{-3}m (500Pa)	σ /m^2 (x10^{-22})	λ/10^{-3}m (500Pa)
H_2	1.804	46	-	-	-	-
He	0.969	85	-	-	-	-
N_2	8.871	9	4.105	20	3.164	26
O_2	9.111	9	4.129	20	2.989	28
Ar	12.61	7	6.003	14	4.474	19

Monochromated EELS

- Non-monochromated – FWHM of ZLP 0.7eV.
- Monochromated – FWHM of ZLP 0.2eV.
- Minor features are resolved in the monochromated spectra.



Conclusions

- Electron energy-loss spectra from gases were acquired.
- High-resolution imaging is possible in the presence of gas.
- Intensity is lost due to scattering from gas molecules.
- More studies are needed for understanding the path and scattering of the electron wave in the high pressure area of the ETEM.