An Energy Stabilized Post-Column Electron Energy-Loss Spectrometer For Transmission Electron Microscopy

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Abstract. An energy stabilizing system for electron energy loss spectrometers in a transmission electron microscope (TEM) is described. A wire-based fast response detector is used to position the zero-loss peak. A digital based real-time processor is used to stabilize the energy-dispersed electron spectrum and simultaneously to acquire a drift corrected electron energy loss spectrum. The performance of the stabilizing system is evaluated.

1. Introduction
Electron energy loss spectroscopy (EELS) has firmly established itself as an extremely useful analytical technique which can have single atom sensitivity [1]. The information content in EELS is a function of the energy resolution achieved. The practical energy resolution of electron energy-loss spectrometers for use in transmission electron microscopes (TEMs) is determined by a combination of factors such as the energy spread of the electron source, the instabilities of the electron accelerating voltages as well as mechanical vibrations [2]. There is already a large improvement in the intrinsic resolution of the electron source, especially with the introduction of source monochromators, and the energy spread can be reduced to less than 100 meV for a TEM operating at 200keV [3]. However, in many practically useful cases, the routine achievement of good energy resolution, particularly over extended recording periods, is limited by fluctuation in the high tension of the electron microscope.

Our approach builds on the 1993 proposal of McMullan, which aimed to stabilize the energy loss spectrum by stabilization of the zero-loss peak at the dispersion plane [4]. Initial results for implementation on a dedicated scanning transmission electron microscope (STEM) was quite encouraging [5], based on an all analogue electronic feedback circuit. We have developed a digital version of the energy stabilized spectrometer for a transmission electron microscope, which has the sophisticated post-sample electron optics in comparison with the STEM. By incorporating the up-to-date electronics and signal processing technique into the energy stabilizer, we not only have improved the energy drift detecting and feedback ability, but are also able to serially collect the spectral information in the neighbourhood of the zero energy-loss beam including plasmon or absorbed-edge peaks. In this paper, we will describe the system and report some preliminary results.

2. Dual-detector EELS
Our energy stabilized electron energy-loss spectrometer consists of a post-column prism energy analyser, followed by two beam detectors focusing on different spectral regions in the energy dispersing plane of the analyzer. The fast response detector, consisting of a narrow wire, is used to
track the movement of the zero-loss peak. The detected signal is used in the feedback loop to stabilize the position of the zero-energy-loss beam. The multi-channel detector, consisting of a multiplet-based lens system for magnification and a charge-coupled-device for efficient parallel EELS (PEELS) acquisition, is used to collect the energy stabilized electron energy-loss spectrum. Figure 1 shows the schematic block diagram. Its main advantages are long term drift corrected spectrum acquisition over a large energy and dynamical ranges with the energy resolution determined by the energy spread of the electron source used.

Figure 1. Schematic block diagram of the energy-stabilized post-column electron energy-loss spectrometer for transmission electron microscopy. 1- nanomotor, 2-tungsten nanowire, 3- scintillator, 4-photomultiplier tube (PMT).

A wire-based fast response detector consists of a tungsten wire of about 5 μm diameter strung across a metal slot which is driven by a nanomotor. The position of the electron beam is determined by its interaction with the wire. A YAG scintillator is coupled to a DM0045C photomultiplier tube provided by the Electron Tubes Limited UK, which is used to acquire the back-scattered electrons from the wire detector by converting the electron signal into a light signal. A Tektronix AFG320 signal generator is used to produce a periodical scanning signal to repeatedly transfer the spatial distribution of the electron beam into the time-domain distribution. The drift detection module measures the spectrum drifts by computing the cross correlation function between the spectrum of every successive oscillating period and the spectrum acquired at the first oscillating period. This has been proved to be very effective in detecting the drift even under the condition of very poor signal-to-noise ratio (SNR) as it is often the case in TEM with a monochromator source. The drift signals are fed to a proportional-integral-differential (PID) controller [6] and the output is added to the beam oscillating signal, so the energy drift can be corrected. A National Instrument PXI-1031 based real-time system is used to meet the requirements of large computing speed and sufficient bandwidth in the drift detection and feedback module.
3. Preliminary results
Currently, we have been testing out various capabilities of our EELS system using a JEM-2000EX TEM with a thermionic tungsten electron source. Because our drift detection and feedback system is digitally based, this gives us a great deal of flexibilities over the analogue system. Firstly, we can use the wire detector to serially acquire the low loss spectrum over the energy range where the wire sensing is taking place. An example is shown in Figure 2(a), where the data is taken from an ion-beam thinned silicon wafer. The zero-loss peak, followed by evenly spaced plasmon loss peaks at 16 and 32 eV, can be seen clearly. This is consistent with the plasmon excitation in bulk silicon.

![Figure 2](image)

Figure 2. The data from the wire-based fast response detector: (a) the stabilized spectrum acquired from a silicon single crystal (b) The characteristics of the zero-loss drift and the effect of drift-correction. (c) The power spectrum density (PSD) analysis of the drift (the sampling frequency); (d) The histogram of the energy drift detected.

At the end of each scan, we can compute the spectral drift by cross-correlation or other means. This can be used in at least three ways: i) it can be used to monitor the nature of the drift as shown in Figure 2b; ii) it can be used to drift correct the serially acquired spectra so that a drift corrected spectrum or an accumulation of such spectra can be recorded digitally as is the case in Figure 2(a); iii) the digital signal can be feedbacked to cancel the drift, so that the spectra acquired with the parallel detector is also drift corrected.

The advantage of the digital feedback system is huge as we can try out various signal processing routines without costly electronic circuit remodification. This allows us to easily fine tune the feedback system. Figure 2(b) shows the position drift of the zero-loss peak when a proportional-
integration and differential (PID) feedback system is turned on or off. We have all the information to analyze the response function of the feedback loop by taking the power spectrum of the drift signals as shown in Figure 2(c). It is clear to see that PID feedback can remarkably suppress the energy drift of zero loss peaks, especially the low frequency components.

Even if the cancellation is not perfect, we have a record of the remaining drift pattern of the zero loss by calculating the probability distribution of the zero loss position during an acquisition period. We can use this distribution to deconvolve out the remaining drift induced broadening in the PEELS spectra. An example of the drift probability distribution plot for the drift curve in Figure 2(b) is shown in Figure 2(d). FWHM of 0.36 eV is obtained when feedback is on, which means that the stability of at least 0.36eV can be attained in our stabilizing system.

4. Discussion
One of the chief factors limiting the drift-stabilization is the tungsten electron source. It has been shown that the wire detector is at least capable of 0.1 eV sensitivity in drift detection for a 5 μm wire detector [4]. We will move next to test the system using LaB₆ filament as electron source to improve the intrinsic energy resolution of the EELS spectrometer. An additional factor maybe in the optimization of the feedback loop algorithm which is underway. Eventually, we will test with a field emission gun or one with a monochromator.

The digital-based system has one disadvantage over the analogue one in terms of time delay introduced by signal processing. We have estimated the feedback loop requires 0.57 ms to complete. This places an upper limit of 1.75kHz as the sampling rate for the zero-loss position, and may limit our ability for responding to high frequency interference. However, we expect the use of a faster and dedicated digital signal processor (DSP) can further push away this limit.

5. Conclusion
A digitally based energy-stabilizing system provides us with at least the two following benefits. First, some numerical recipes or signal processing tools can be used, such as cross correlation, PID feedback, self-adaptive filter and power spectrum density(PSD), etc. and a great deal of flexibilities are given in fine tuning the drift detection and feedback system to stabilize the whole spectrum. Second, the wire-based fast response detector does not only provide the energy drift corrected signal, but also can collect the spectral information in the neighbourhood of the zero-energy-loss beam, including zero-loss peak and plasma peak.

Preliminary test results show that an energy stabilizing ability of 0.36 eV in our system is obtained, which could be further improved by optimizing the PID controller and increasing the sampling rate.

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