Auger Electron Spectroscopy in high vacuum: 
Nanocharacterisation in the Scanning Electron Microscope

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Abstract. It is generally regarded as impossible to carry out Auger Electron Spectroscopy (AES) analysis in a scanning electron microscope (SEM) due to the high ambient gas pressure in an SEM. This is because standard electron energy analysers such as the Concentric Hemispherical Analyser (CHA) or Cylindrical Mirror Analyser (CMA) are devices that acquire data in a serial manner that can last up to few minutes. This is considered too slow for high vacuum ($10^{-6}$ mbar) and a previously cleaned surface would be re-contaminated before a spectrum could be completed. This has led to AES being traditionally carried out under ultra-high vacuum (UHV) environment. We report on two devices for fast acquisition of AES data characterising nanoscale objects by the use of AES in an SEM.

1. Introduction
The characterisation of samples at the nanoscale is an important aspect of nanotechnology. Characterisation at the micron scale has been carried out for several decades using X-ray microanalysis – usually with the aid of Energy Dispersive Spectroscopy (EDS) as an add-on technique in scanning electron microscopy (SEM). In order to approach nanoscale characterization using EDS, the primary electron energy needs to be reduced. This means that the K shells of many elements can no longer be excited and the analysis needs to rely on L and M shell excitations. The peaks of these levels are often close together and quantification is difficult. An important recent advance in EDS is the use of Silicon Drift Detectors (SDDs) which have higher energy resolution and allow the L and M peaks to be separated. However, even at lower primary energies, the excitation volume is defined by the primary beam energy and is larger than the primary electron beam spot on the sample. In order to characterize samples at such a scale, it is necessary to resort to Auger electron spectroscopy (AES). In addition, AES could provide complementary information to EDS such as the nature of the chemical bonding, as well as electronic information provided by the plasmon losses. However, a major obstacle to the use of AES in standard SEMs is that AES normally requires Ultra High Vacuum (UHV) conditions. This is because at poorer vacuum conditions, the surface would be covered with a layer of mostly carbon and oxygen compounds in just a few seconds. Since AES normally requires at least a few minutes to acquire a spectrum, then this renders AES impractical in SEMs at their usual operating pressure. This paper discusses two approaches to the fast acquisition of Auger electron spectra – the Hyperbolic Field Analyser (HFA) [1] and the Magnetic Electron Energy Spectrometer (MEES) [2] which overcome these restrictions.
The first device for parallel AES data acquisition is the Hyperbolic Field Analyser (HFA) which has been previously described [1]. In this paper we will show AES spectra that have been acquired using the HFA on an SEM with a pressure of ~10^{-6} mbar (JEOL 6400F). The surfaces of the used samples are in-situ cleaned with energetic Ar ions. The rate of recontamination is determined by studying the rates of growth of Carbon and Oxygen Auger electron peak heights.

Another device for fast acquisition of AES data is the Magnetic Electron Energy Spectrometer (MEES). This device would be better suited to SEMs with higher magnetic field strengths in the sample region and can acquire electrons over a wide range of electron take-off angles. Theoretical background to the MEES will be presented as well as AES spectra obtained using a custom built UHV system operating at ~10^{-7} mbar and using a novel Active Pixel Sensor as electron detector.

2. The Hyperbolic Field Analyser (HFA) in the Scanning Electron Microscope

The HFA was mounted in the specimen chamber of the SEM. The sample surface was cleaned using an Ar ion gun (IQE 12/38 by SPECS). A number of materials were studied, but Silicon is the example presented here. The sample is rotated between the position for Ar ion cleaning and that for AES. The sample was cleaned using 5keV Ar ions (specimen current 1-2 μA) for 10-20 minutes prior to analysis. Figure 1 shows the Auger spectrum of the Si just after cleaning. Acquisition started within 10s of finishing the Ar ion cleaning. Fifty spectra were taken with each spectrum taking 1s. These spectra were then averaged. Several such averaged spectra were taken over subsequent minutes.

![Figure 1. An Auger spectrum of Silicon taken using the HFA showing the Si LVV and C KLL Auger spectra.](image)

The depth of the layer C_i is related to the atomic concentration of an overlayer of element i by

\[ X_i = 1 - \exp \left( -\frac{C_i}{\lambda \cos \theta} \right) \]

where \( \lambda \) is the inelastic electron mean free path, \( \theta \) is the angle between the surface normal and the detector [3].

The atomic concentration \( X_i \) is given by...
\[ X_i = \frac{\alpha_i I_i}{\sum_j \alpha_j I_j} \]

where \( \alpha_i \) is the inverse relative sensitivity factor, \( I_i \) is the intensity of the Auger peak of element I \([4,5]\).

The rate of growth of Carbon on the Silicon could be determined by measuring the depth of the layer Si and C in relation to elapsed time.

![Graph showing the rate of growth of Carbon with time.](image)

Figure 2. Rate of growth of Carbon with time as estimated using Si and C Auger signals.

3. The Magnetic Electron Energy Spectrometer (MEES)

The MEES uses a magnetic field to disperse the electrons. The magnetic field direction is arranged to be parallel to the primary beam. Electrons emitted from the surface pass through a small slit and travel in a helical fashion until they strike a 2D detector. The detector used in this experiment was an Active Pixel Sensor \([6]\). Electrons of the same energy but varying take off angles form a curve as shown in Figure 3. In this figure the intense line is caused by elastic peak electrons being emitted at a range of take off angles. A spectrum of the output can be determined by integrating in a line from top left (= low energy) to bottom right (=high energy) and the result can be seen in Figure 4.
4. Conclusions

Two different approaches to rapidly acquiring Auger spectra using two entirely different electron energy analysers have been presented. These class of analysers are >x50 faster than conventional AES analysers. The ability to acquire Auger spectra in high vacuum has been shown to be possible and that Auger spectroscopy can be carried out in a standard SEM. This is most relevant in applications requiring elemental and chemical analysis at high spatial resolution that conventional X-ray
spectroscopy (EDS) cannot provide. In addition, the magnetic spectrometer allows the possibility to acquire Auger spectra even in the presence of relatively strong magnetic fields and in confined spaces.

**References**

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