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# New Methods for Measuring Chemistry and Temperature Using Scanning Ion and Electron Beams

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We describe two new methods we have developed for measurement of local chemistry and tempertaure using scanning ion and electron beams.

In the first method, we implement ion-induced Auger Electron Spectroscopy (AES) using a focused ion beam (FIB), with the eventual goal of developing a new high spatial resolution and high chemical sensitivity tomographic technique. While FIB tomography has emerged as a powerful and widely-used method for recreating 3D reconstructions of the internal structure of materials ranging from length scales of tens of nm to tens of microns, corresponding quantitative chemical methods are lacking. Integration of secondary ion mass spectroscopy (SIMS) with FIB is hampered by the fact that secondary ionization yields with $Ga^+$ ion beams in the FIB are very low ($10^{-5}$ or less) for many elements [e.g. 1,2]. This is further accentuated by the relatively low transmission factors of standard SIMS detectors such as quadrupole mass spectrometers, although higher transmission factor detectors such as time-of-flight (TOF) systems have been successfully applied [e.g. 2,3]. These factors compromise the ability to generate chemical information from small volumes of material. The fundamental advantage of coupling AES to the FIB is that Auger electron yields per incident ion can be relatively high, of order $10^{-1}$ [4]. Coupled with the high transmission factors of Auger electron detectors, such as hemispherical analysers, this provides the potential for extracting high sensitivity chemical information from small volumes of material. We have successfully integrated an Orsay Physics Cobra mass-selecting FIB column into an ULVAC-PHI VersaProbe system, aligning the focal points of the FIB and of the detector optics with the necessary 3D precision. We use an Au-Si alloy source separated into $Si^+$, $Si^{2+}$ and $Au^+$, $Au^{2+}$ beams. Depending on the atomic number of the target specimen, this is necessary to ensure that the majority of the Auger transitions are from the atoms of the sample rather than in the primary beam. Example spectra for multiple elements with a primary 60 keV $Si^{2+}$ ion beam are shown in Figure 1. Strong Auger peaks are observed for each element, but for the Mg, Al and Si samples, additional extremely sharp peaks are observed (arrowed). These peaks arise from Auger emission from atoms that have been sputtered from the surface before Auger decay, and thus are much more spectrally sharp than for Auger emission from the typical case of atoms that remain in the near surface region of the sample. The fact that these free atom peaks are only observed in a subset of the samples can be understood in terms of the product of the kinetic energy of the sputtered excited atoms and the substantially longer vacancy state lifetimes, and thus probability of escape of the target atom from the surface, for the relevant elements and vacancy states. In our experimental geometry and conditions, we observe an average Auger yields of 0.06 for Cr and 0.09 for Al per incident 60 keV $Si^{2+}$ ion. This compares very favourably to comparable FIB-SIMS yields. The instrumental yields are also higher: our estimate for the product of the typical transmission factor for our spectra-collection conditions and the detector efficiency for the channeltron-type detector employed in the analyser, is 0.6. The biggest reduction in yield actually arises from the solid angle over which Auger electrons are captured, yielding an additional attenuation factor of 0.03. The total collected yields are thus in the $10^{-3}$ range, enabling atomic concentrations of order 0.1-

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1.0 % to be detected within a $(50\ \text{nm})^3$ voxel.

In the second method, we have developed a new non-contact approach to measurement of temperature with nanoscale spatial resolution, thermal scanning electron microscopy (ThSEM), by quantifying the effects of thermal diffuse scattering in electron back scattered diffraction (EBSD) patterns. Figure 2 shows the result of intensity traces across a (400) Kikuchi line in an EBSD pattern recorded from a Si(001) sample with 20 keV electrons in a JEOL 840 SEM [5]. As expected, the intensity of the excess Kikuchi line decreases monotonically with increasing temperature, while the intensity of diffuse scattering increases. The peak intensity in the Kikuchi line can be correlated to the predicted curve from measured Debye Waller factors using neutron scattering [6] with a coefficient $R^2 = 0.986$. The spatial resolution we have estimated from the 20 keV beam used for these experiments is c. 80 nm; employing finely focused low energy electron beams in a state-of-the-art field emission instrument should enable 10 nm resolution to be attained. We estimate the temperature sensitivity from our measurements to be 5-10$^{o}$ C. Most importantly, while other techniques such as scanning thermal microscopy have comparable spatial resolution and nominally better temperature sensitivity, the over-arching advantage of the current technique is that is essentially non-contact. This is a crucial advantage in making temperature measurements of nanoscale objects, such that the act of measurement itself does not substantially modify the local temperature. [7]

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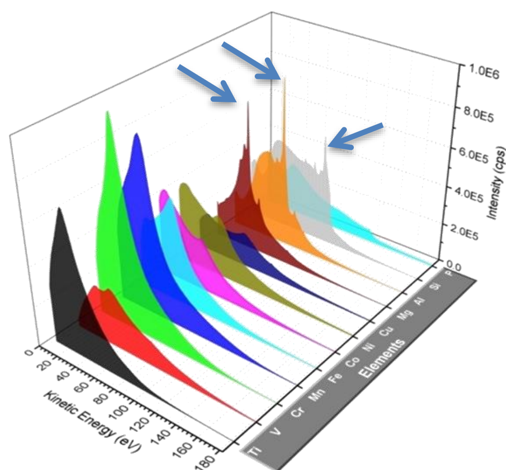


**Figure 1.** Auger electron spectra from an incident 60 keV $Si^{2+}$ ion beam for different target elements (Mg, Al, Si, P from $L_{23}$ shell vacancy, others from $M_{23}$ shell vacancy).

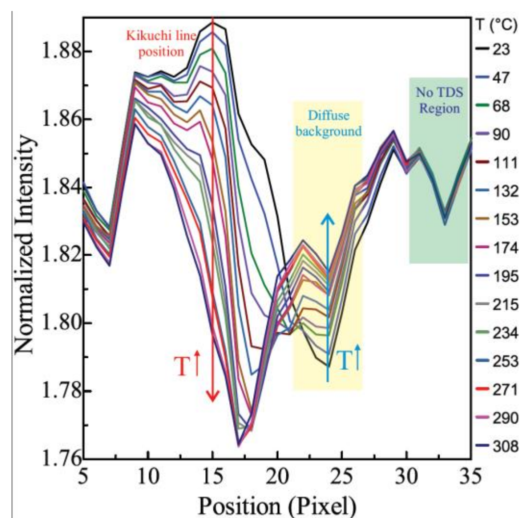


**Figure 2.** Intensities of the (400) excess line in an EBSD pattern from Si(100) as a function of sample temperature

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