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Strategies for x-ray analysis of non-conductive specimens in a conventional scanning electron microscope

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Identification (ID) of major elements within a small feature is a key requirement in forensic science, failure analysis and phase ID. Fig.1 exemplifies the problem with non-conductive samples. The accumulated trapped charge raises surface potential, reducing the landing energy for electrons; the Duanne Hunt Limit, (DHL) is lowered well below 20keV and useful lines such as GaK are not excited. Furthermore, the surface field can deflect incident electrons away from the intended analysis spot and spurious peaks such as AlK give a false positive ID. If the specimen surface is irregular, carbon coating the specimen may not be effective because isolated islands of coated material still charge up. Such areas can be identified by mapping the intensity of x-rays in a band just below the DHL. In fig. 3(b) the x-ray map identifies areas in the corresponding s.e. image 3(a) that are still charging despite the conductive coating. Fig.3(c) shows two spectra integrated from spectrum image pixels within the boxed areas in fig.3(a). Where coating is not an option, variable pressure SEM provides one route to neutralise surface charge. However, the beam "skirt" may excite elements well outside the analysed feature and give false IDs. Alternatively, in conventional SEM, reducing beam kV increases the current fractions lost by secondary emission and backscatter. As shown in fig.4, for the same beam current, the specimen will retain 60% less current at 5kV than at 20kV. This helps imaging but element ID is more difficult because of the greater loss of x-ray excitation. A more effective strategy is to tilt the sample. As fig.5 shows, while x-ray excitation falls off significantly at tilts beyond 30 degrees, the absorbed current fraction falls much more rapidly and at tilts of 70 degrees or more, charging may even be avoided if the absorbed fraction goes negative beyond the "E2" threshold. If the E2 limit cannot be achieved, landing energy will drop until increased emission and surface leakage is sufficient to neutralise residual current [1]. If charge does accumulate then analysis has to be completed before the surface potential begins to interfere with the beam [2]. 100pA for 10 seconds with a 6 msterad solid angle typically gives enough x-rays for qualitative ID and increasing solid angle reduces necessary current. Fig.2 shows an idealised electrostatic model for the effects of trapped charge. Rastering the beam will not alter the x-ray acquisition rate but by spreading the dose over a larger area, the rise in surface potential (drop in DHL) is slowed down by 1/R. If the sample is thin enough so that $d \approx R$, the potential is also suppressed by the grounded stub. (Alternatively, a ground plane on the sample surface surrounding the uncoated area reduces the magnitude of the surface potential for a given Q.) A charged sample can be discharged by slowly ramping down the kV so that the landing energy on all charged areas is always < E2. Below 1.5kV, the specimen can then be imaged and new areas for analysis selected before the beam kV is restored. In a practical example at 7kV , 300pA, an uncoated $Ba_2TiSi_2O_8$ specimen charged immediately at normal incidence. At 55 deg.tilt in spot mode, 30 seconds acquisition with a 5msterad collection angle was achieved before DHL dropped by 0.5keV. With a 10um raster width, analysis time extended to 480 seconds for the same effect.

[1] S. Fakhfakh et al, *Nucl.Instr.and Meth.* B 197 (2002), 114
[2] D.E.Newbury, *Scanning* 22, (2000), 345
[3] The assistance of Steve Pitman and Jenny Goulden in experiments is gratefully acknowledged.

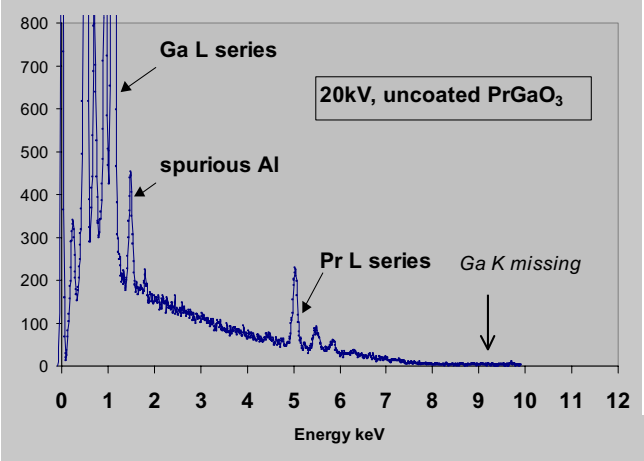
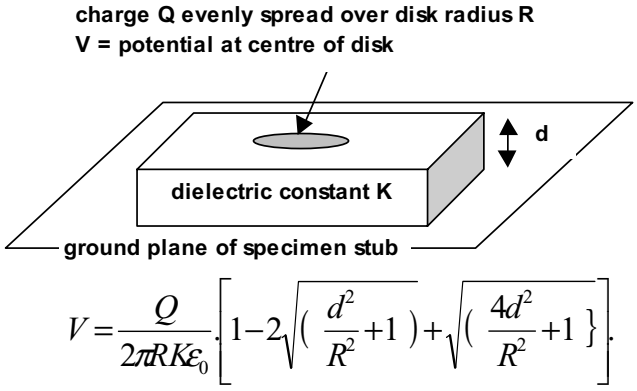
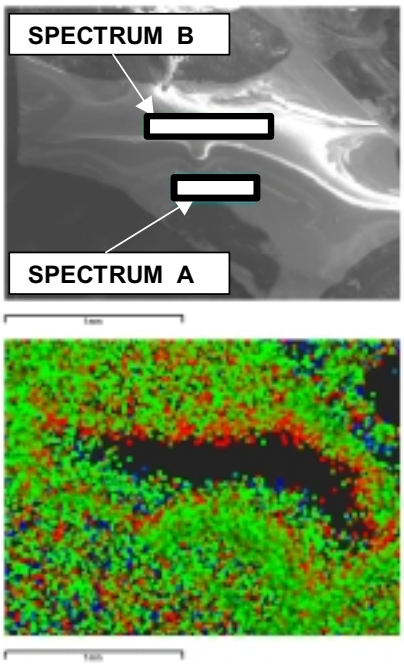


FIG 1: Element ID on charging sample



$$V = \frac{Q}{2\pi R K \varepsilon_0}\left[1 - 2\sqrt{\left(\frac{d^2}{R^2}+1\right)} + \sqrt{\left(\frac{4d^2}{R^2}+1\right)}\right].$$

FIG 2: Idealised model of trapped charge



3(a)

3(b)

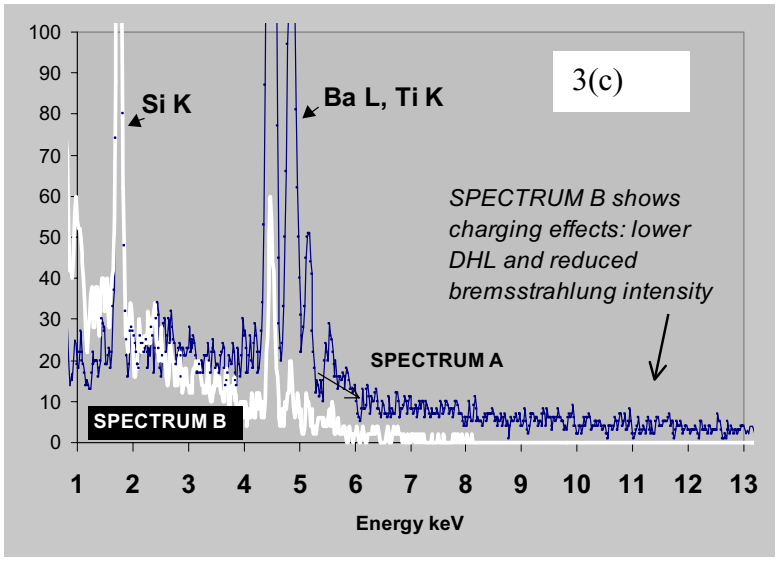


FIG 3: Coated $Ba_2TiSi_2O_8$ at 20kV (a) s.e. image 2mm field (b) x-ray map for 10keV-20keV (c) spectra for regions in (a)

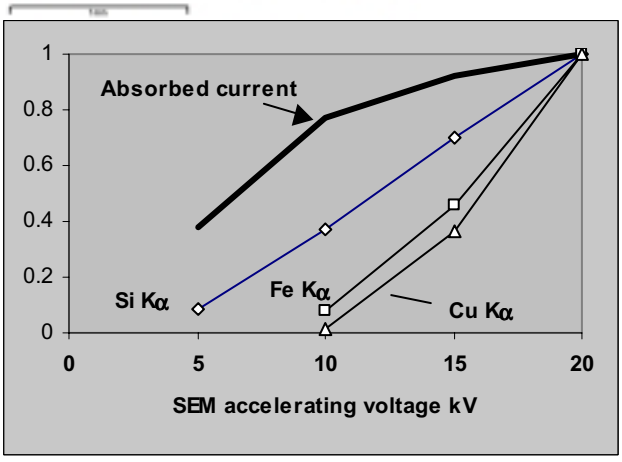


FIG 4: Excitation and dose relative to 20kV for fixed beam current at normal incidence

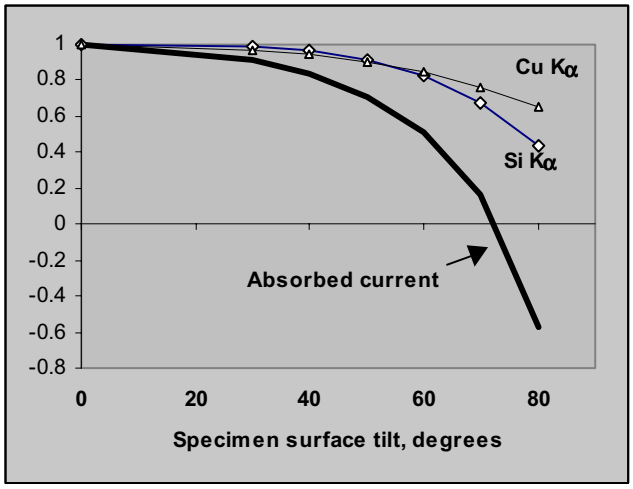


FIG 5: Effect of tilt for typical sample at 20kV

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