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doi:10.1017/S1431927611003904

Microsc. Microanal. 17 (Suppl 2), 2011

X-ray Microanalysis Artifacts Visualized

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X-ray microanalysis is a powerful research tool whose results, unfortunately, quite often suffer from misinterpretation. Time and again journals publish papers with artifact-influenced results [1,2] and peer review sometimes fails to filter out such papers. Proper interpretation of X-ray microanalysis requires a deep understanding of the physics of electron beam-specimen interaction which is not a required subject for most researchers. In order to demonstrate for researchers and graduate students the possible pitfalls and to highlight the necessity of proper communication with a properly trained operator, we have prepared a few demonstrations, based on visualization rather than on theoretical discussion.

An important artifact of X-ray maps and line scans is the contrast due to changes in the background, which is proportional to the average atomic number Z [3]. Therefore, even if a map was acquired for an element not present in a specimen, variations in background intensities for different phases will be recorded and false conclusions about specimen composition could be made. Fig. 1a shows an X-ray map with the superimposed line scans (P, U and Al) of a cross section of dental enamel (high Z) embedded in resin (low Z). Maps as well as line scans show similar X-ray intensity distribution for all three elements, but only P is present in the specimen. In contrast to maps and line scans, the X-ray spectrum (Figure 1b) can be used to prove the presence of P and absence (within detection limits) of U and Al.

Auto scaling of X-ray maps can cause skewing of image contrast due to the background effect. [3]. Auto scaling makes the map of Mg fairly bright in normal dentin, which only has traces of magnesium (Fig. 2a). On the other hand, the Mg map of caries affected dentin show brighter spots for the Mg-containing mineral in tubules and much darker (because of auto scaling) dentin with the same amount of Mg (Fig. 2b). A comparison of such maps can lead to the incorrect conclusion that caries affected dentin is depleted in magnesium.

The dramatic difference in the resolution (2-3 orders of magnitude) of secondary electrons (SE) imaging and of the X-ray signal can lead to a misunderstanding and incorrect interpretation of the results. When an electron beam is scanned across a specimen with a visibly sharp vertical edge, the X-ray signal generated by the beam changes gradually as the signal production volume crosses the edge. Fig. 3 represents resin embedded enamel with the superimposed profiles of P obtained at 25, 15 and 8 kV; the widths of profiles demonstrate the dependence of signal production volume on kV value. Occasionally, profiles like these are incorrectly interpreted and false “findings” of “diffusion/intermediate zones” are reported.

References

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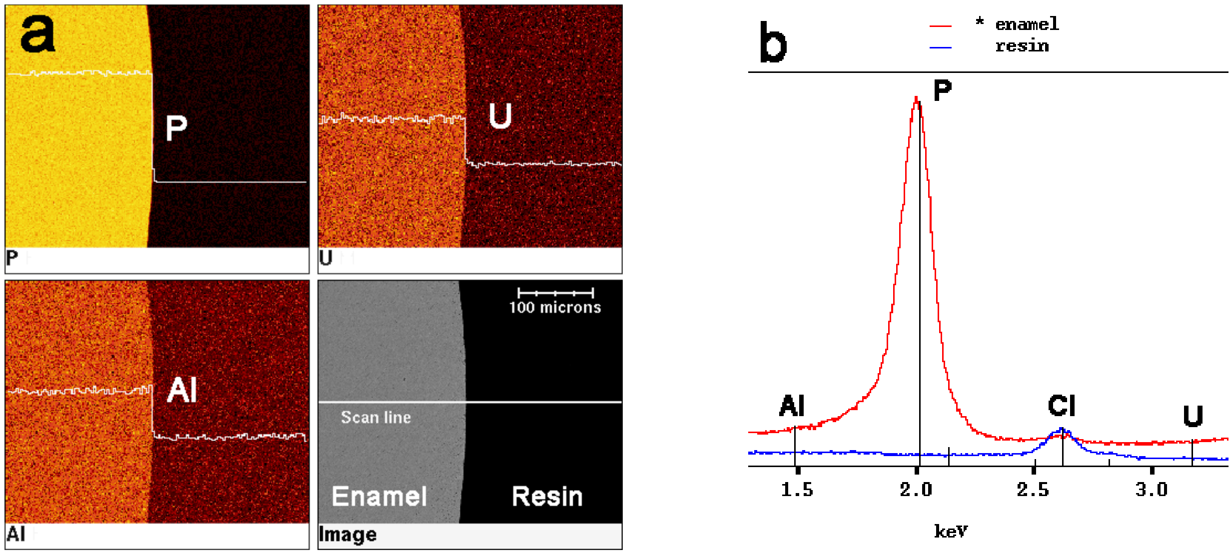

Figure 1. a) Maps and line scans of P, U, and Al obtained from the polished cross section of a resin embedded tooth. b) Superimposed spectra of enamel (red) and of embedding resin (blue); substantial difference in background intensities recorded; No U and Al were detected.

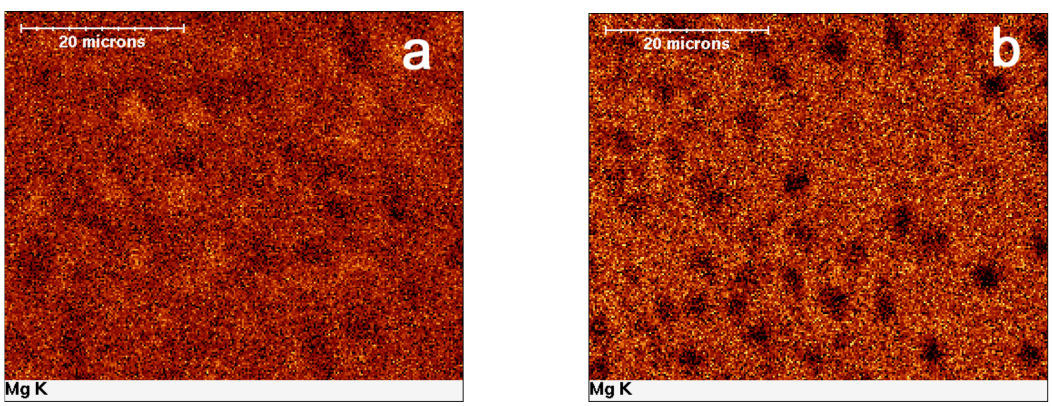

Figure 2. Mg maps of cross sections of caries-affected (a) and normal (b) dentin.

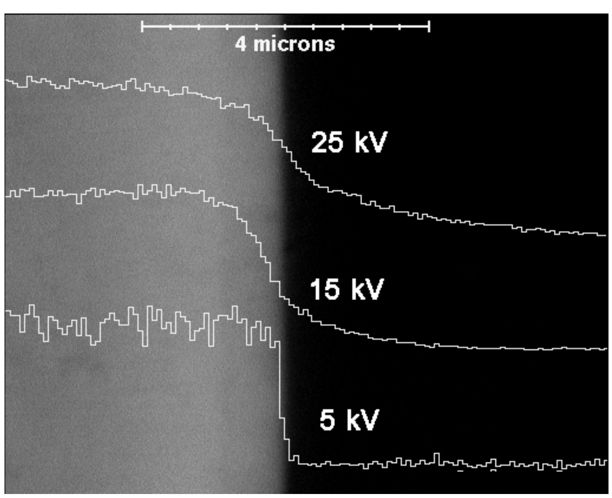

Figure 3. Phosphorus profiles obtained at different accelerating voltages from the same scan line on enamel (left) – embedding resin (right) boundary.

https://doi.org/10.1017/S1431927611003904 Published online by Cambridge University Press