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Synchrotron Infrared Radiation for Spectroelectrochemical Microscopy

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Synchrotron infrared radiation has been successfully coupled through an IR microscope to a thin-cavity external reflectance cell to study electrochemical processes. Excellent signal-to-noise ratios were achieved even at aperture settings close to the diffraction limit. Comparisons of noise levels as a function of aperture size demonstrate that this can be attributed to the high brilliance of synchrotron radiation relative to a conventional thermal source. Time resolved spectroscopic studies of diffusion controlled redox behaviour have been measured and compared to purely electrochemical responses of the thin-cavity cell. Marked differences between the two measurements have been explained by analysing diffusion in both the axial (linear) and radial dimensions. Whereas both terms contribute to the measured current and charge, only species that originate in the volume element above the electrode and diffuse in the direction perpendicular to the electrode surface are interrogated by IR radiation. Implications for using ultramicroelectrodes and synchrotron IR (SIR) to study electrochemical processes in the sub millisecond time domain will also be presented.