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Abstract: Laura Maria Bingham, Development of nanoparticle catalysts and total internal reflection (TIR) Raman spectroscopy for improved understanding of heterogeneous catalysis

Laser Raman spectroscopy has been used very effectively for some time to probe heterogeneous catalytic reactions *in situ*.

TIR Raman is a variant of non-resonant Raman spectroscopy which uses a totally internally reflected light, *i.e.* an evanescent electric field acts as the excitation source. TIR Raman reduces or avoids some key limitations of bulk Raman spectroscopy, including reduction of laser induced sample damage. This thesis has therefore been investigating the possible application of TIR Raman spectroscopy to studying heterogeneous catalysts, in particular films of size controlled metal nanoparticle catalysts. Potential catalytic materials (such as platinum, supported either on optical elements or in mesoporous silica) were synthesised, characterised by techniques such as TEM, UV-VIS, deposited on substrates both at monolayer and thicker coverages, and subjected to both conventional and TIR Raman spectroscopy. This included a significant amount of synthetic development, in particular of copper nanoparticles.

Results indicated that TIR Raman enables acquisition of spectra with improved sensitivity, compared to bulk Raman, below the damage threshold of the materials, even at low levels of surface coverage. Specifically, Raman bands indicating the presence and removal (by plasma cleaning) of organic capping agents on nanoparticles have been detected for a number of systems. This was not achieved using confocal Raman spectroscopy. This was extended to develop a system for studying gas/solid catalytic reactions *in situ* using a specially constructed gas cell to enable application of the technique under reaction conditions.

Finally, the nanoparticles synthesised primarily for the TIR Raman study have also been demonstrated as catalysts in improving the understanding of several catalysed reactions, in particular direct amide bond formation from amines and alcohols, cascade oxidations, and selective furfural hydrogenation.