

List of Figures

1.1	Active sites in an Au/TiO _x catalyst	4
1.2	Latimer diagram for the coinage metals	7
2.1	Helmholtz and Stern model of the electric double layer	13
2.2	Potential/time diagram used during cyclic voltammetry	14
2.3	Typical cyclic voltammogram of a gold electrode	14
2.4	Surface state of a gold electrode in dilute H ₂ SO ₄ at different potentials	15
2.5	Region definitions in a typical XAS spectrum	18
2.6	The transmission mode in X-ray absorption spectroscopy	19
2.7	A typical total electron yield setup	20
2.8	Scattering events forming EXAFS	22
2.9	TEY cell for studying the growth of Au ₂ O ₃	28
2.10	TEY cell for monitoring the decomposition of Au ₂ O ₃	28
2.11	Transmission setup in X-ray absorption spectroscopy	29
2.12	The UHV sample holder	30
2.13	Sketch of the UHV setup used in this work	31
2.14	Vacuum plan of the UHV system.	31
2.15	The electrochemical glass cell	33
2.16	Impurities detectable after the transfer process from electrochemical cell to UHV	35
2.17	Energy level diagram for XPS experiments	38
2.18	XP spectrum of a clean gold sample	40
2.19	Model for depth profiling <i>via</i> XPS	44
2.20	Depth profiles for different models	44
3.1	Typical sample current during a plasma discharge oxidation	47
3.2	Examples for the galvanostatic and potentiostatic oxidation processes.	48
3.3	Ratio between Q _{ox} and Q _{red}	49

3.4	Evolution of reduction peaks on a polycrystalline gold surface as a function of oxidation potential	49
3.5	Integral of the reduction peak as a function of the oxidation potential	50
3.6	Relation between oxidation charge and number of reduced Au ions	51
3.7	Determination of S_0^2 for different samples	52
3.8	Pre characterisation of the crystalline gold oxide sample. . .	54
3.9	Phase analysis of Au_2O_3	56
3.10	Results of the EXAFS analysis of Au_2O_3	57
3.11	Comparison between single and multiple scattering fits for Au_2O_3	61
3.12	EXAFS fit of electrochemically grown Au_2O_3	63
3.13	LC analysis of the growth of Au_2O_3 at 0.5 mA/scm	64
3.14	LC analysis of the growth of Au_2O_3 at 450 mA/scm	65
3.15	Phase composition analysis of gold oxides with FEFFIT . . .	67
3.16	Growth process analysed <i>via</i> all Au L edges	69
3.17	Analysis of different galvanostatic oxidation processes	70
3.18	Experiments with a fluorescence yield XAS cell using thin gold foils on mica	71
3.19	Decomposition of commercial Au_2O_3	72
3.20	Thermal decomposition of commercial and electrochemical Au_2O_3	73
3.21	TGA analysis of commercial Au_2O_3	74
3.22	Spectra taken during thermal decomposition of Au_2O_3	75
3.23	Different O_2 Sputtering times investigated <i>via</i> TPD	76
3.24	Heating rate variation of a Au/O phase obtained from plasma oxidation	77
3.25	Heating rate variation for different sputter energies	77
3.26	TPD experiments with potentiostatic and galvanostatic oxidised gold samples	78
3.27	Heating rate variation experiments with galvanostatic and potentiostatic oxidised samples.	79
3.28	TPD spectra acquired for different m/z values after a potentiostatic oxidation at 1.8V	80
3.29	UHV decomposition of an electrochemically prepared Au_2O_3 phase	82

3.30	XP spectra of polycrystalline gold samples exposed for short times to a 250 eV plasma discharge.	83
3.31	Influence of CO on the XP spectrum of a plasma oxidised Au sample	83
3.32	Influence of CO on the decomposition of oxidic oxygen species	84
3.33	XPS peak fits of a gold oxygen phase obtained <i>via</i> a plasma discharge	85
3.34	A typical XPS experiment following the potentiostatic oxidation of an Au sample	86
3.35	Thermal decomposition of an oxide layer prepared potentiostatically at $U_{ox}=+1.7$ V	86
3.36	Decomposition of a galvanostatically prepared Au_2O_3 phase	88
3.37	XPS peak fits of an electrochemically oxidised sample	90
4.1	XPS signal intensity ratios during thermal decomposition of Au_2O_3	103
4.2	XPS analysis of the decomposition of a potentiostatically grown Au_2O_3 sample	103