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Advancing Frontiers of Condensed Matter Physics**

3 - 7 July 2006

Clusters on surfaces: Matter in the non-scalable size regime

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Clusters on Surfaces: Matter in the Non-Scalable Size Regime

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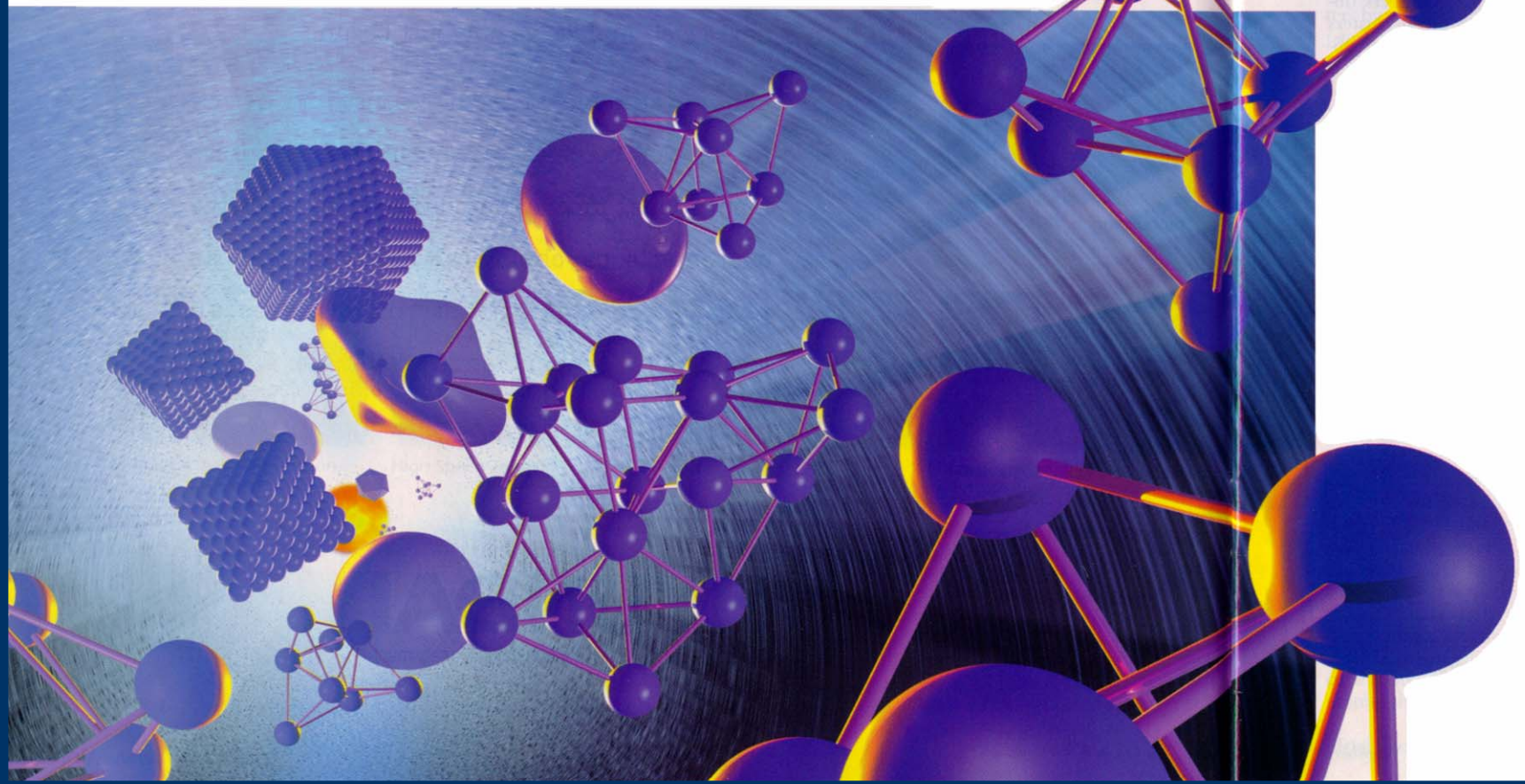
Technische Universität München



Outline

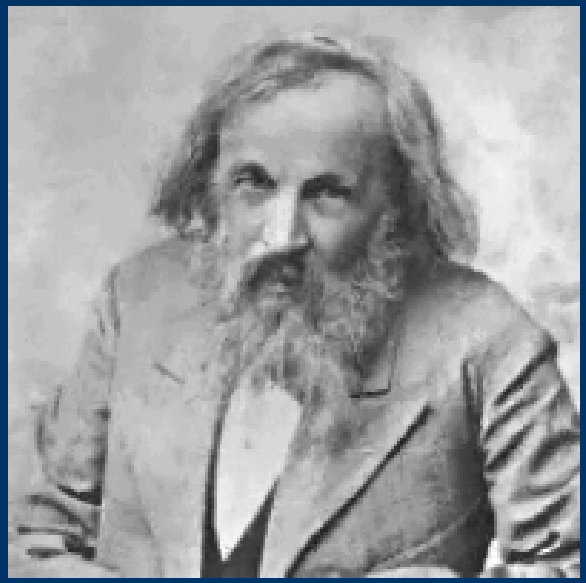
Introduction:

- Nanoparticles: The scalable size regime
- Clusters: The non-scalable size regime

Experimental Techniques

Guiding Principles for Understanding Cluster Chemistry

Mendelejev's Periodic Table

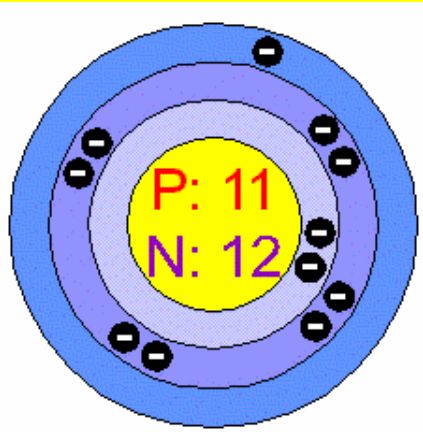


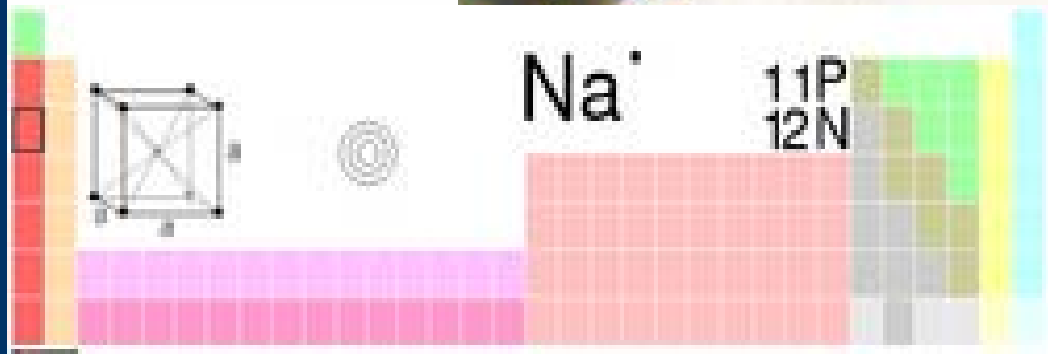
Mendelejev, Dmitri Ivanovitsj

8. Feb. 1834 (Tobolsk) -
2 Feb. 1907 (St.- Petersburg)

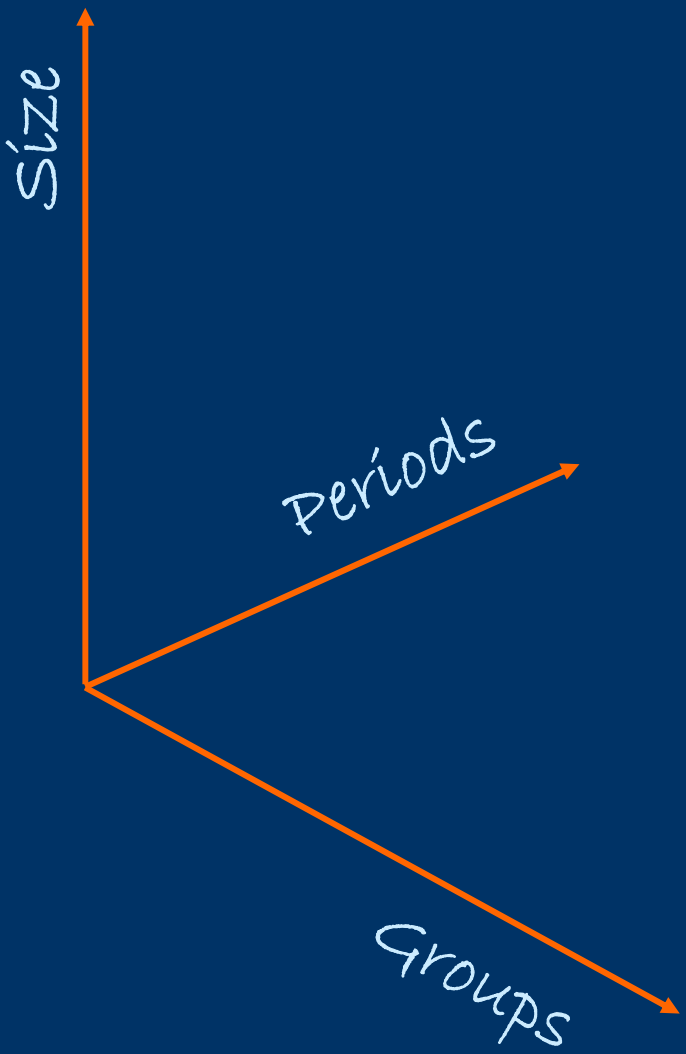
In ´*On the Relation of the Properties to the Atomic Weights of the Elements*´, received by the Russian Chemical Society in 1869.

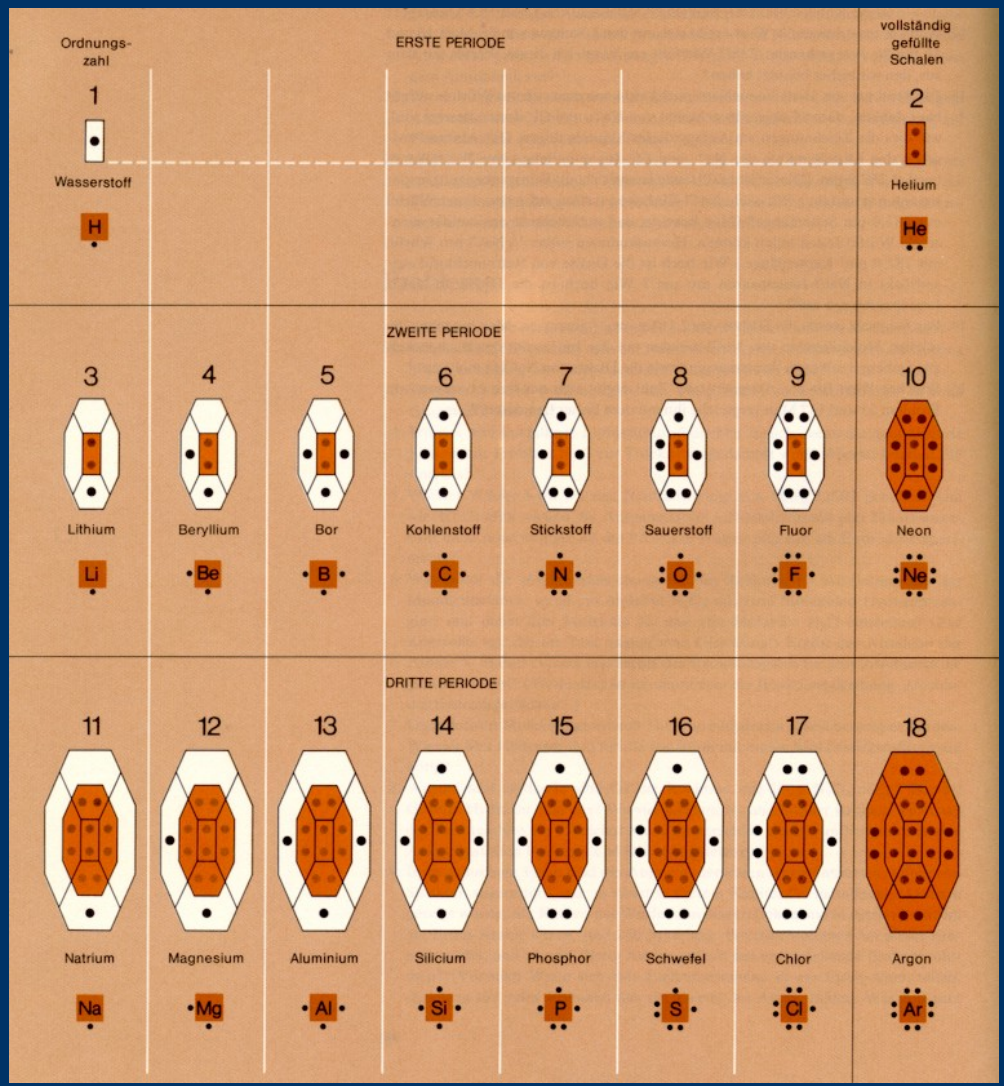
	Gruppe I.	Gruppe II.	Gruppe III.	Gruppe IV.	Gruppe V.	Gruppe VI.	Gruppe VII.	Gruppe VIII.
	-	-	-	RH^4	RH^3	RH^2	RH	-
Reihen	R^2O	RO	R^2O^3	RO^2	R^2O^5	RO^3	R^2O^7	RO^4
11		(Au = 199)	Hg = 200	Tl = 204	Pb = 207	Bi = 208	-	-	
12	-	-	-	Th = 231	-	U = 240	-	- - - -



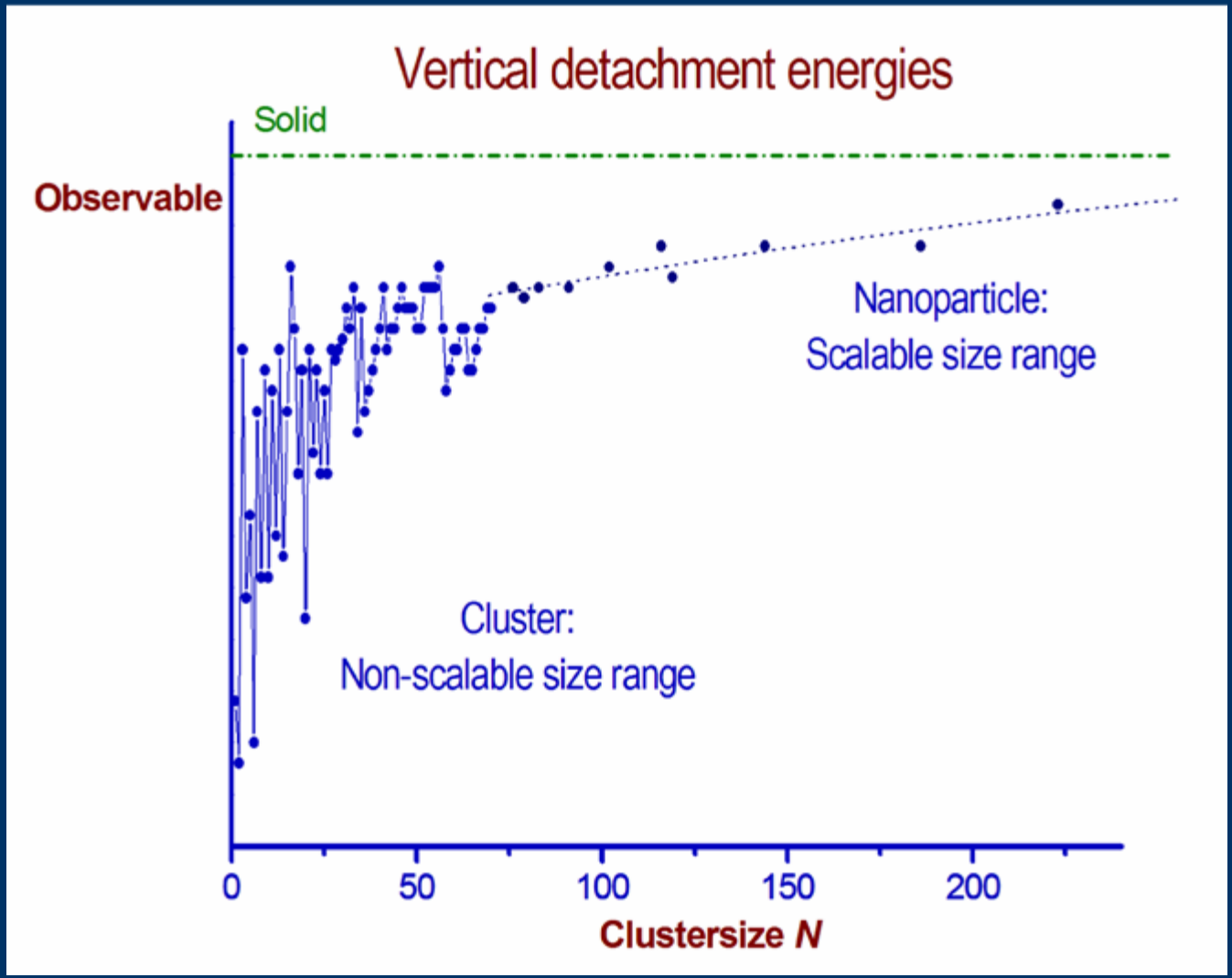


The 3rd Dimension of the Periodic Table





Non-Scalable – Scalable Size Regime



The Scalable Size Regime

Scaling Laws

- In the regime of large sizes (N > ~100), many particle properties (*e.g.* ionization energy, electron affinity, melting temperature and cohesive energy) show a smooth variation with cluster size.

- The following scaling laws apply for a general property (G)

$$G(R) = G(\infty) + aR^{-\alpha}$$

$$G(N) = G(\infty) + bN^{-\beta}$$

Usually $\alpha = 1$, $\beta = 1/3$.

The Scalable Size Regime

Examples

- **Ionization energies of potassium nanoparticles (N≤100):**

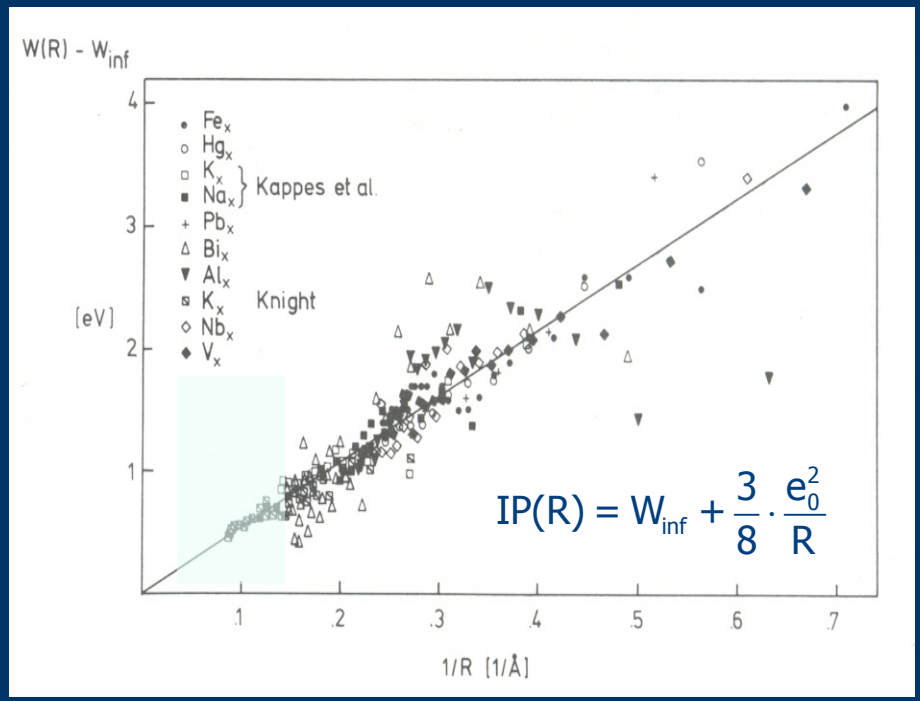
$$\mathrm{IP(R)}/eV = \; 2.3 + 5.35\left(R/\text{Å}\right)^{-1}$$

$$\mathrm{IP(N)}/eV = \; 2.3 + 2.04N^{-1/3}$$

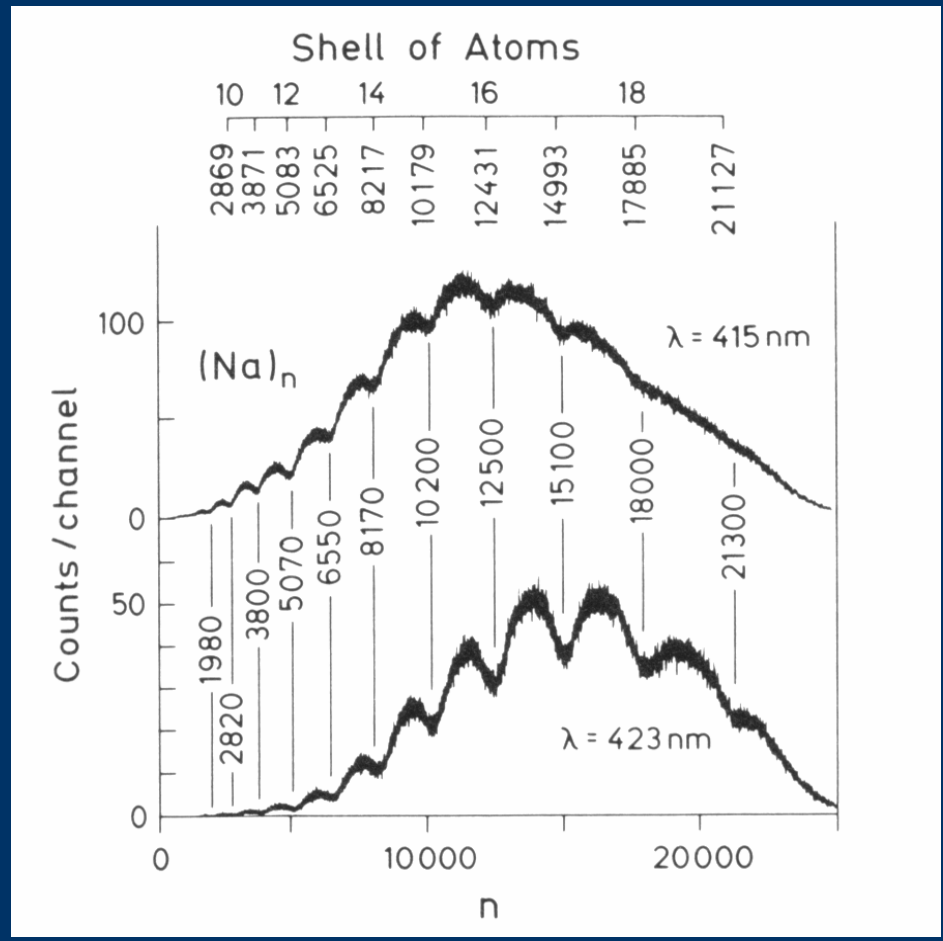
- **Melting temperatures of gold nanoparticles:**

$$\mathrm{T}_m\left(\mathrm{R}\right)/K = \; 1336.15 - 5543.65\left(R/\text{Å}\right)^{-1}$$

The Scalable Size Regime: Nanoparticles of Sodium (Na_n with n>2000)



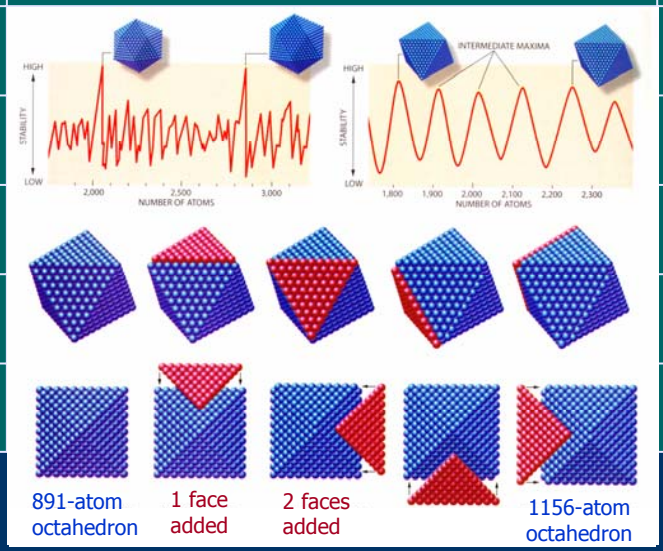
E. Schumacher , U. Heiz et al.
*Chimia **42** (1988) 357-376*



T.P. Martin et al.
*Z. Phys. D **19** (1991) 25*

Nanoparticle's Periodic Table: Geometric Shells

Shell (K):	Open Shells			Closed Shells
11	Na_{2870}	Na_{2871}	…	Na_{3871}
12	Na_{3872}	Na_{3873}	Na_{3874} …	Na_{5083}
13	Na_{5084}	Na_{5085}		Na_{6525}
…				
17	Na_{12431}	Na_{12432}		Na_{14993}
18	Na_{14994}	Na_{14995}		Na_{17885}
19	Na_{17886}	Na_{17887}		Na_{21127}



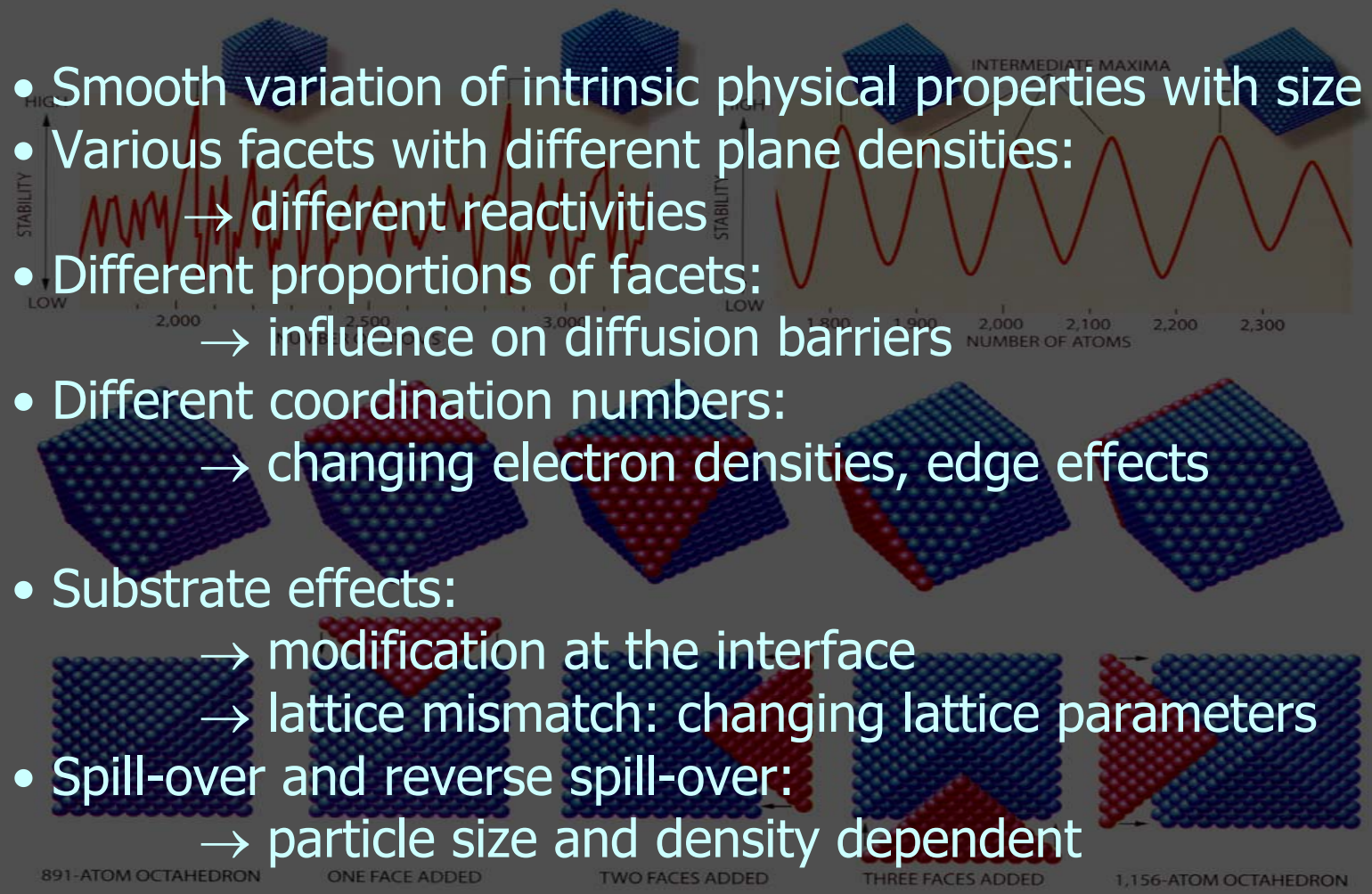
$N=1/3\,(10K^3-15K^2+11K-3)$

Mackay Icosahedra

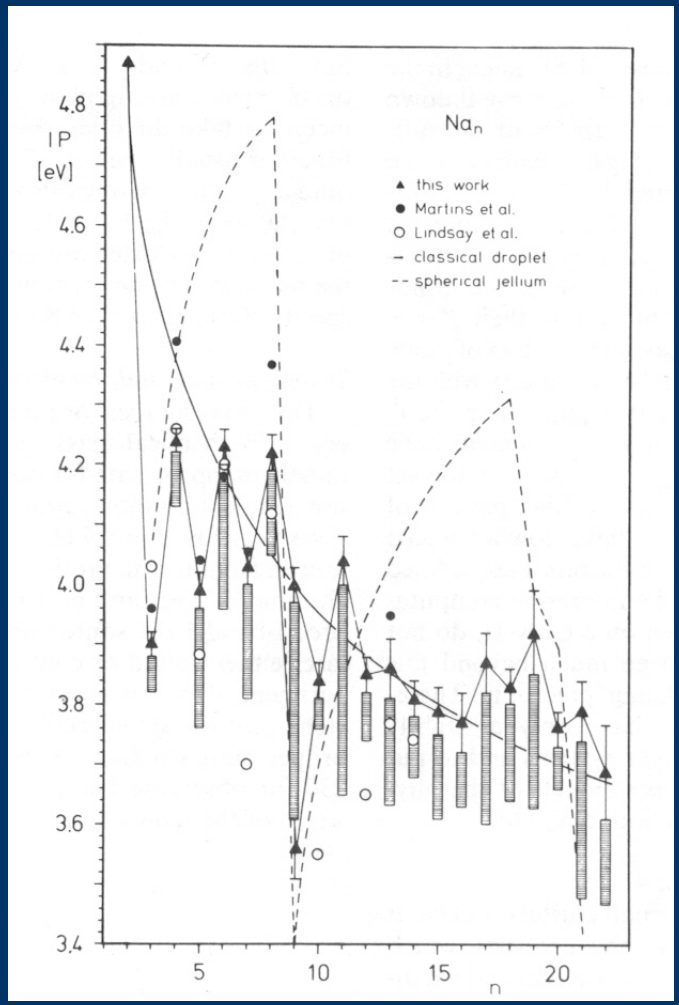
Acta Cryst. ***15*** *(1962) 1916*

Size Effects of Nanoparticles: Some Guiding Principles

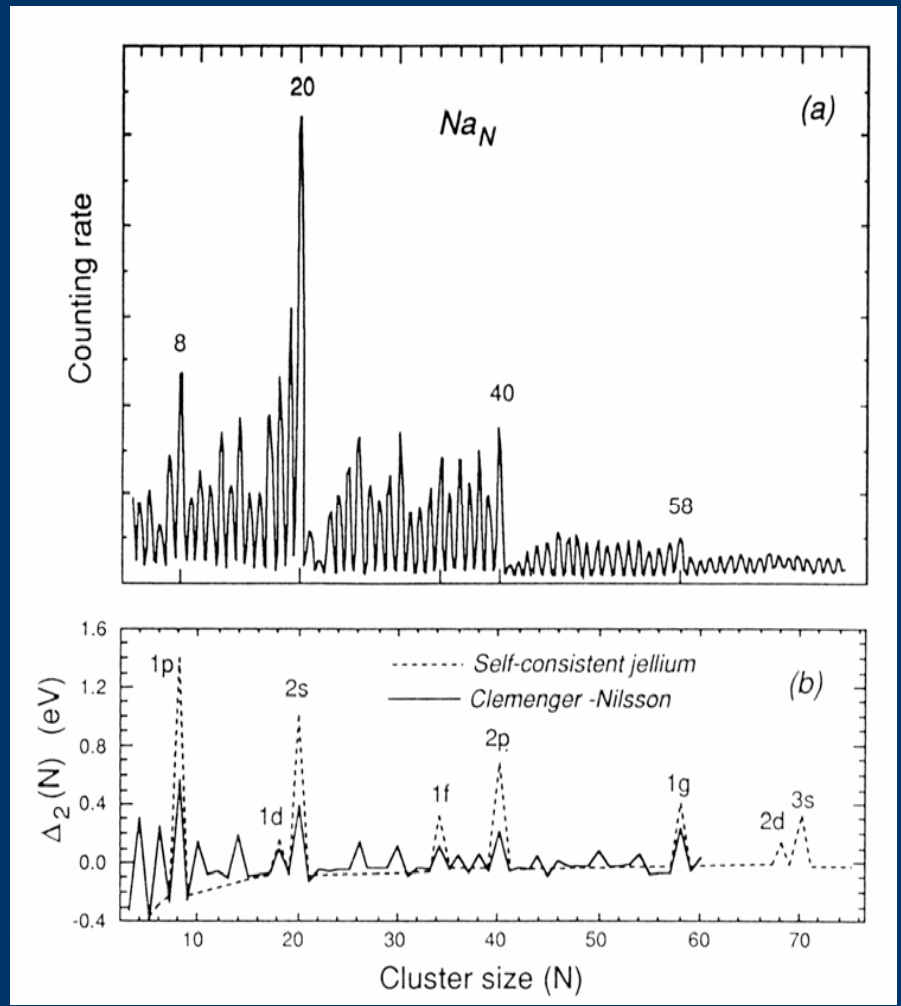
- Smooth variation of intrinsic physical properties with size
- Various facets with different plane densities:
  → different reactivities
- Different proportions of facets:
  → influence on diffusion barriers
- Different coordination numbers:
  → changing electron densities, edge effects
- Substrate effects:
  → modification at the interface
  → lattice mismatch: changing lattice parameters
- Spill-over and reverse spill-over:
  → particle size and density dependent



The Non-Scalable Size Regime: Sodium Clusters (Na_n with $n < 150$)



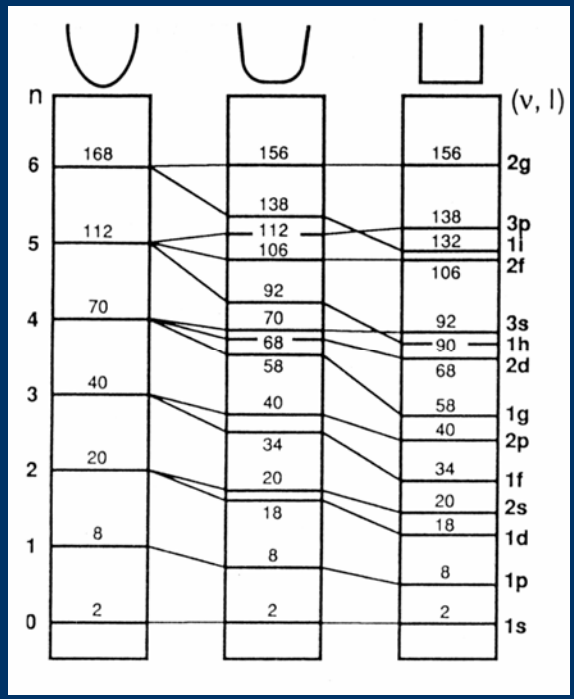
E. Schumacher, U. Heiz et al. Chimia **42** (1988) 357



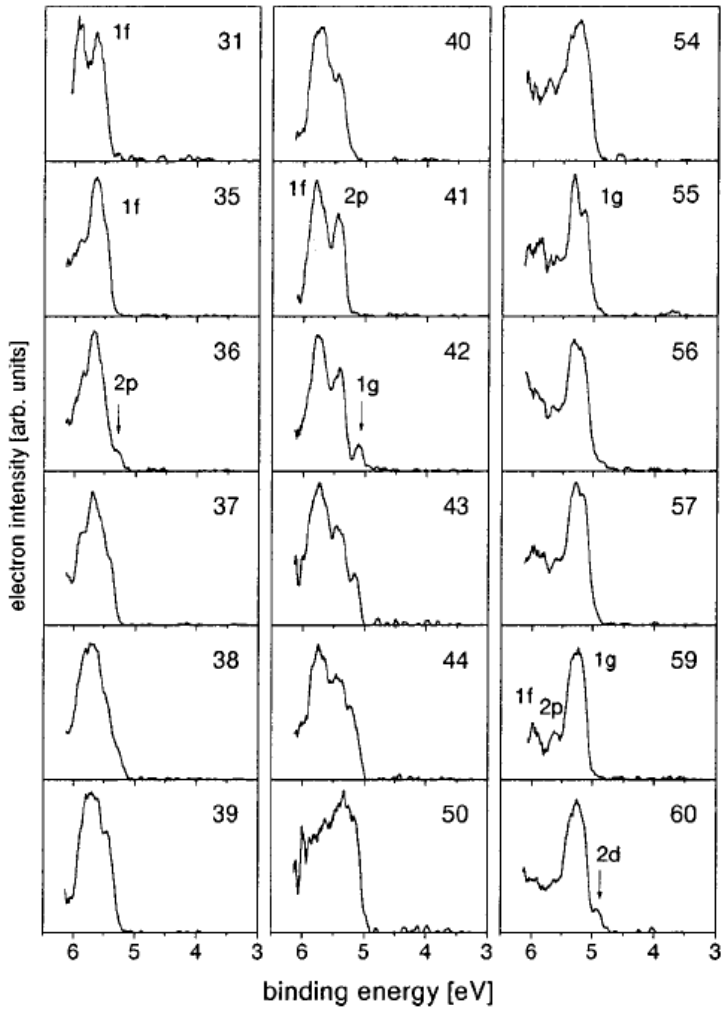
de Heer Rev. Mod. Phys. **65** (1993) 611

Cluster's Periodic Table : Electronic Shells

Shell	Monovalent				Closed shells
1s	Na				Na_2
1p	Na_3	Na_4	⟶	Na_7	Na_8
1d	Na_9	Na_{10}	⟶	Na_{17}	Na_{18}
2s	Na_{19}				Na_{20}



The Non-Scalable Size Regime: Sodium Clusters (Na_n with $n < 150$)



Photoelectron spectra of Na_n (n=31-60) obtained with a photon energy of 6.42 eV. The peak labels give the quantum numbers of the corresponding electron shells. Closed shells are observed at:

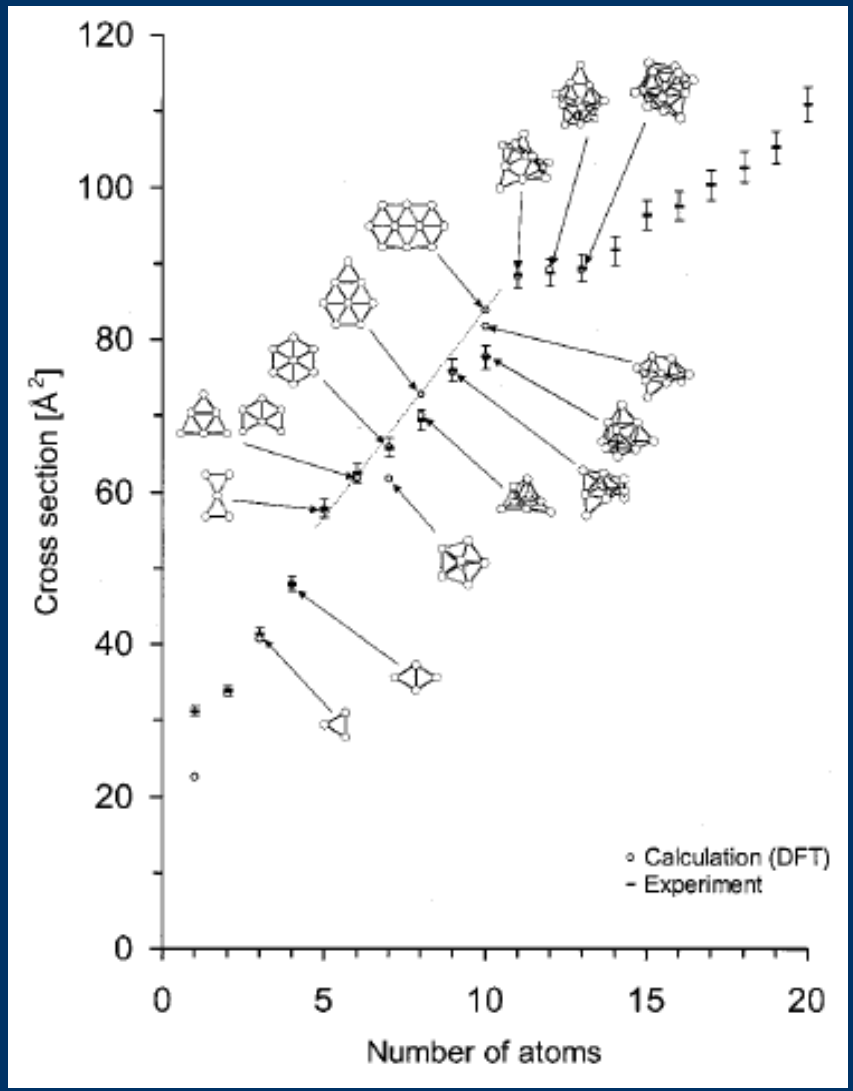
Na_{35}^+ (34 electrons): 1f

Na_{41}^+ (40 electrons): 2p

Na_{59}^+ (58 electrons): 1g

v. Issendorff, B. Phys. Rev. A 65 (2002) 63201

The Non-Scalable Size Regime: Geometric Structure of Gold Clusters

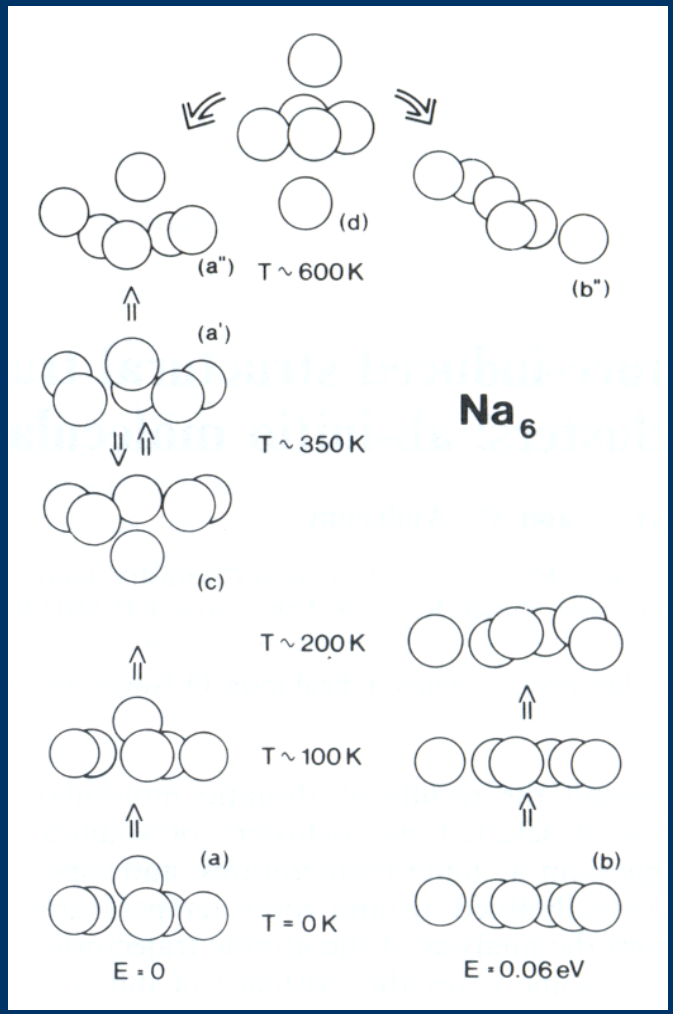


Ion mobility measurements and *ab initio* calculations reveal gold clusters to be planar up to Au_n with n~10 for the cations and n~13 for the anions.

Strong relativistic effects in gold are responsible for these unique geometric structures

S. Gilb et al. J. Chem. Phys. 116 (2002) 4094
H. Häkkinen et al. Phys. Rev. Lett. 89 (2002) 33401

The Non-Scalable Size Regime: Dynamic Structural Fluxionality



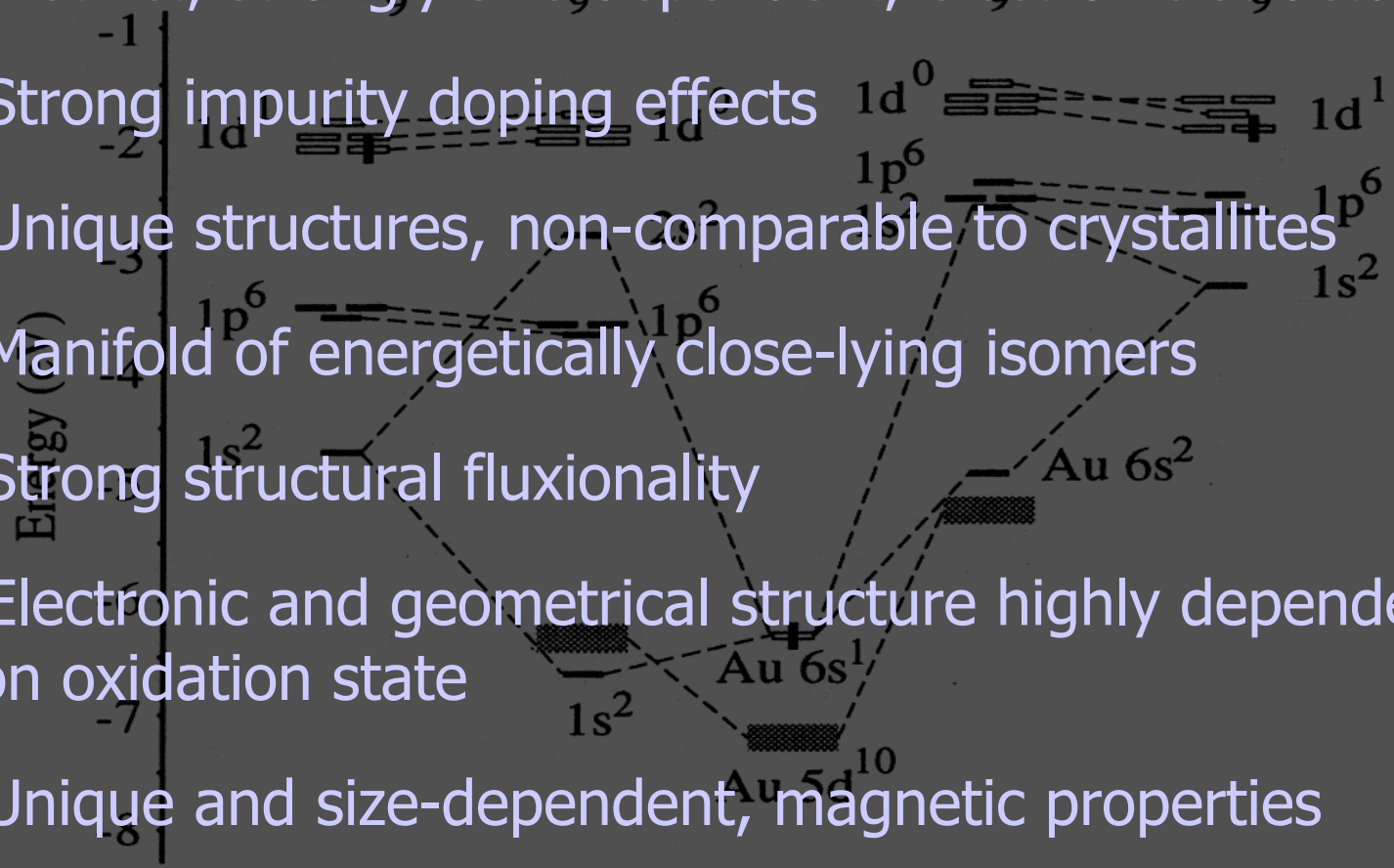
*U. Röthlisberger et al. Atoms, Molecules and Clusters **20** 243 (1991)*

Many clusters reveal several energetically close lying isomers.
At finite temperatures these isomeric structures can be populated and interconvert into each other.

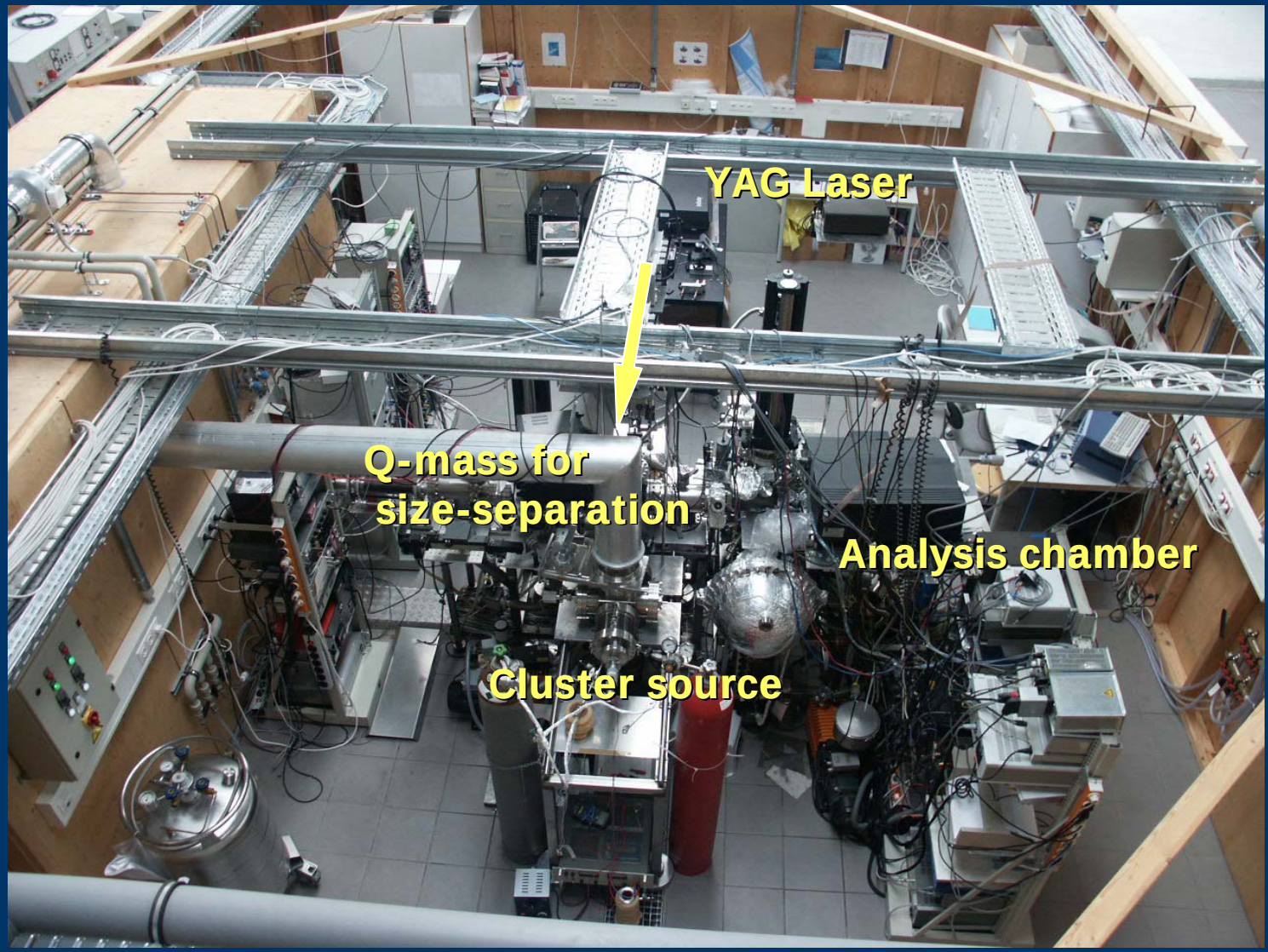
 # Dynamic structural fluxionality

Cluster Properties

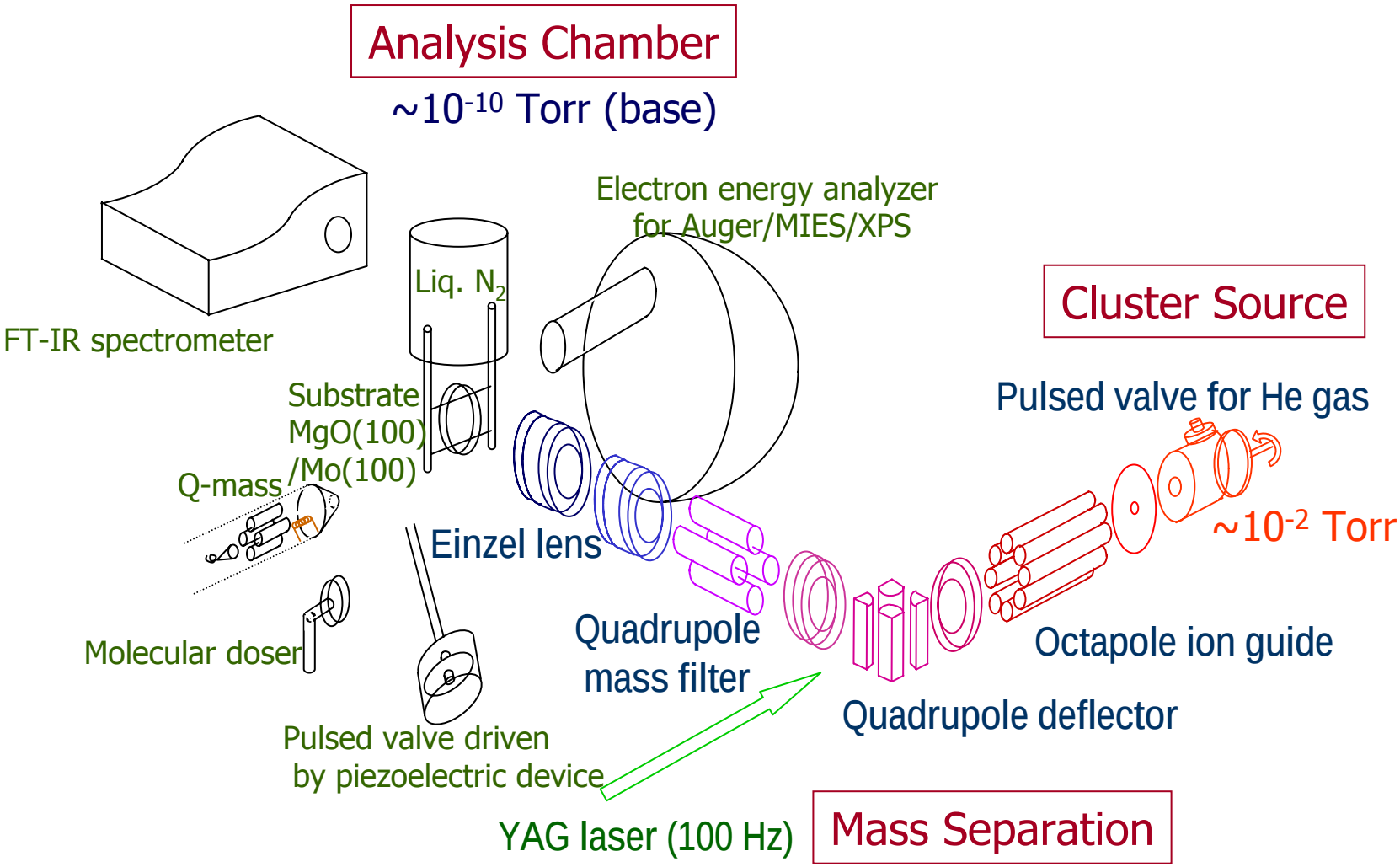
- Distinct, strongly size-dependent, electronic structures
- Strong impurity doping effects
- Unique structures, non-comparable to crystallites
- Manifold of energetically close-lying isomers
- Strong structural fluxionality
- Electronic and geometrical structure highly dependent on oxidation state
- Unique and size-dependent, magnetic properties



Experimental Techniques

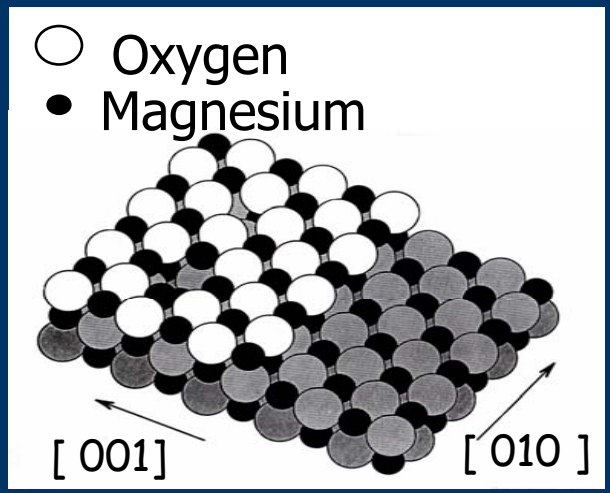


Experimental Techniques: Cluster Preparation



Experimental Techniques: Support Materials

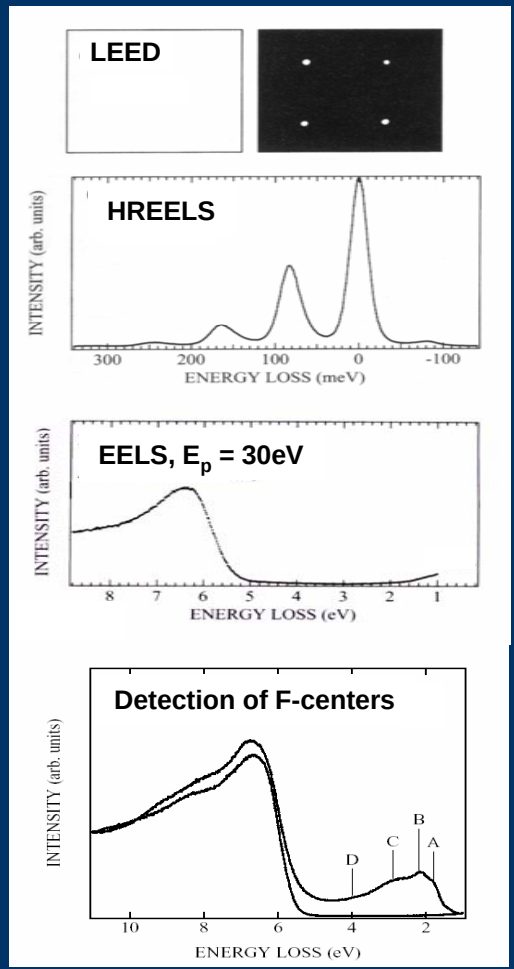
Preparation:

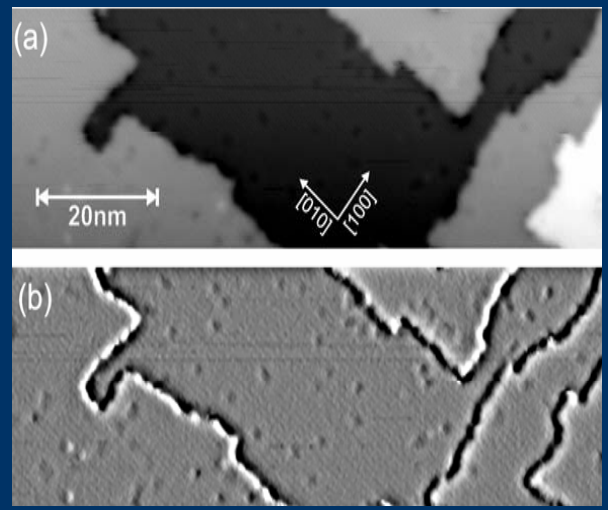


Epitaxially grown on Mo(100) or Ag(001) by evaporation of Mg in 10^{-6} Torr O_2 at RT.

Wu et al. Chem. Phys. Lett. ***182*** *(1991) 472*

Characterization:





Heiz et al.
J. Phys. D.: Appl. Phys. ***33*** *(2000) R85-R102*

Schaffner et al.
Surf. Sci. ***417*** *(1998) 159*

Experimental Techniques: Characterization

Typical cluster densities: 10^{-12} -10^{-13} clusters/cm^{-2}

⌗ Detection limit of many classical surface science techniques!

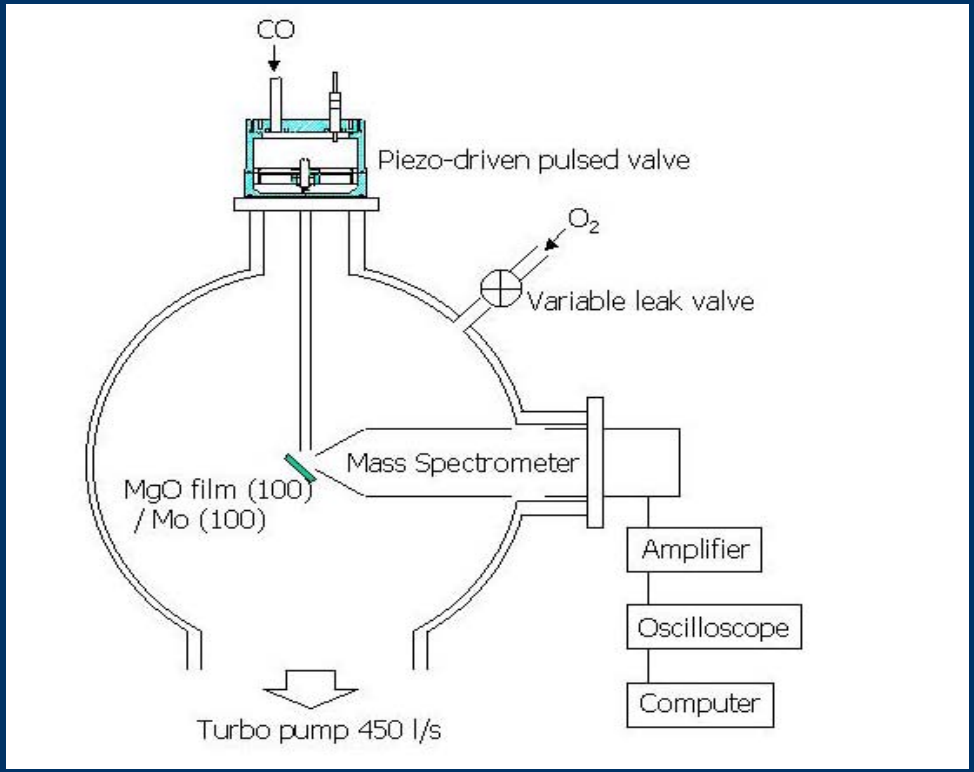
Integral methods:

- **Temperature programmed desorption**
- **Temperature programmed reaction**
- **Fourier transform infrared**
- **Electron energy loss**
- **Ion surface scattering**
- **X-ray photoelectron spectroscopy**
- **Ultraviolet photoelectron spectroscopy**
- **Metastable impact electron spectroscopy**
- **X-ray magnetic dichroism**

Local methods:

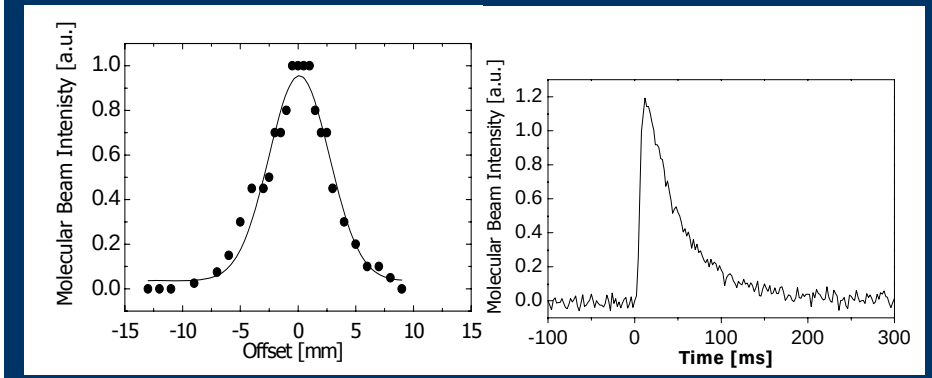
- **Scanning tunneling microscopy**
- **Scanning tunneling spectroscopy**
- **Atomic force microscopy**

Experimental Techniques: Pulsed Molecular Beam Reactive Scattering

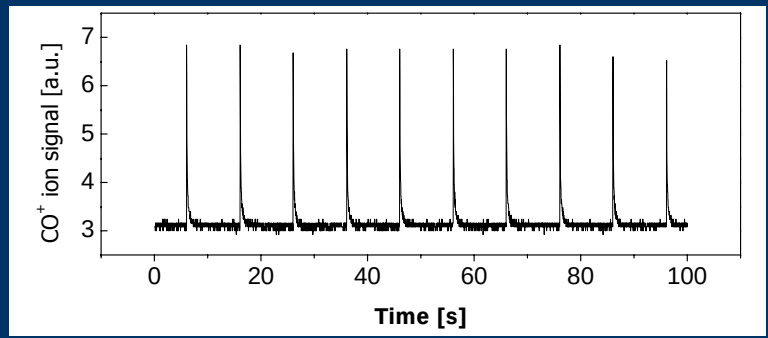


Beam profile:
8 mm (diameter of crystal)

Time profile:
~70 ms
Maximal local p:
$1x10^{-3}$ mbar



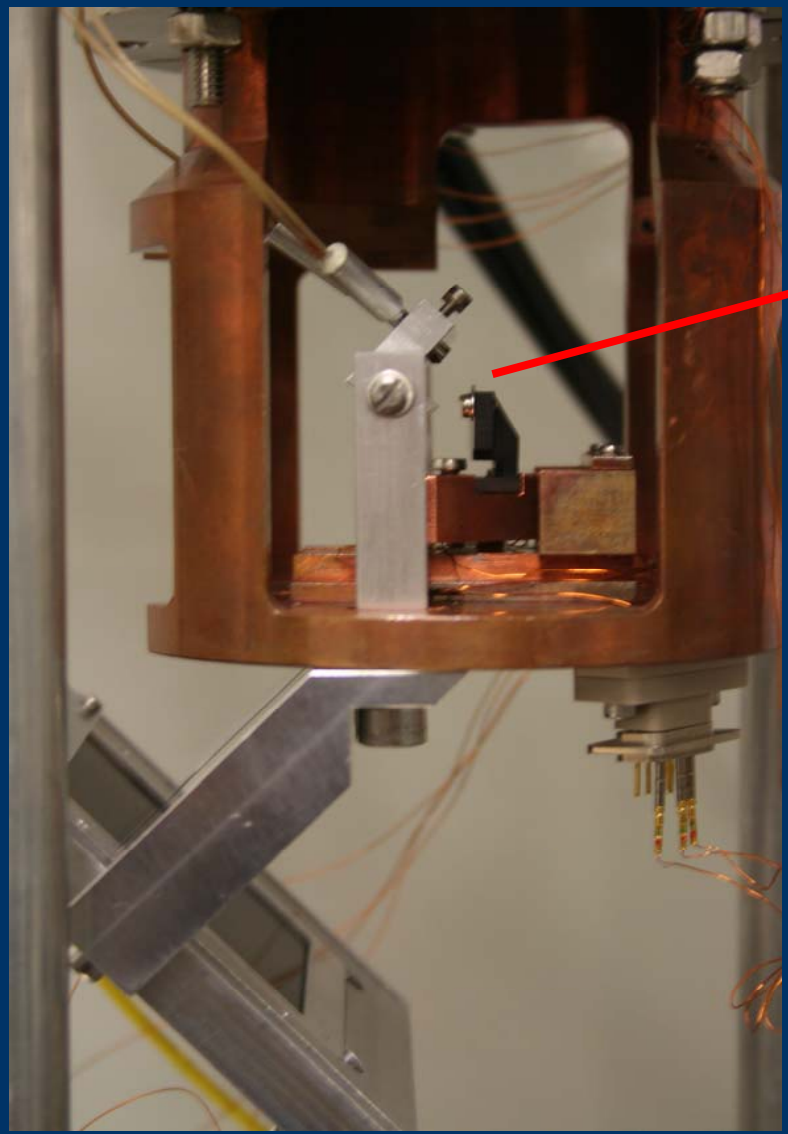
Stability: < 1% pulse to pulse

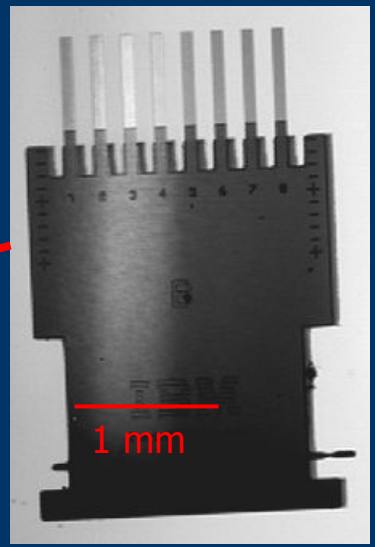


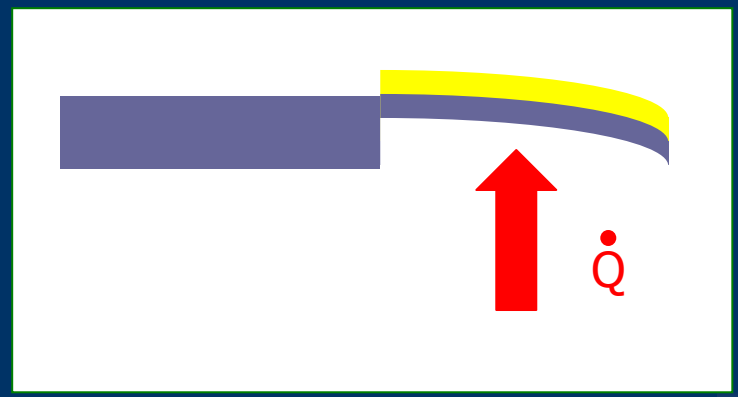
Experimental conditions:
$p_{O2} = 5x10^{-7}$ mbar,
$p_{CO}(max) \sim 1x10^{-3}$ mbar,
repetition rate = 0.1 Hz
$\Delta t_{pulse} = 0.07s$

$$TOF = \frac{C_{calib} \times \int_{peak} I_{CO_2}}{n[Pd_n] \times \Delta t_{pulse}}$$

Experimental Techniques: Microcalorimetry

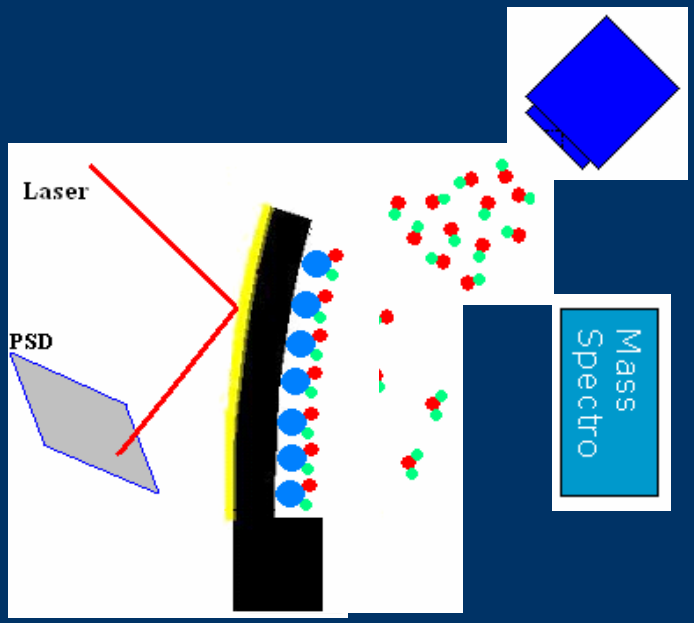


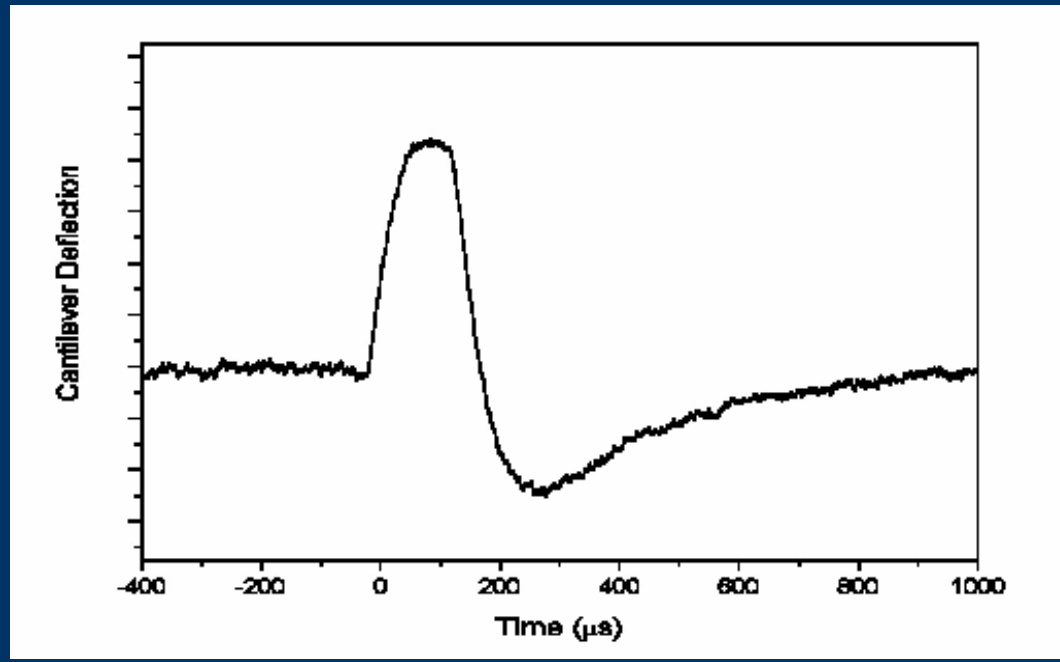




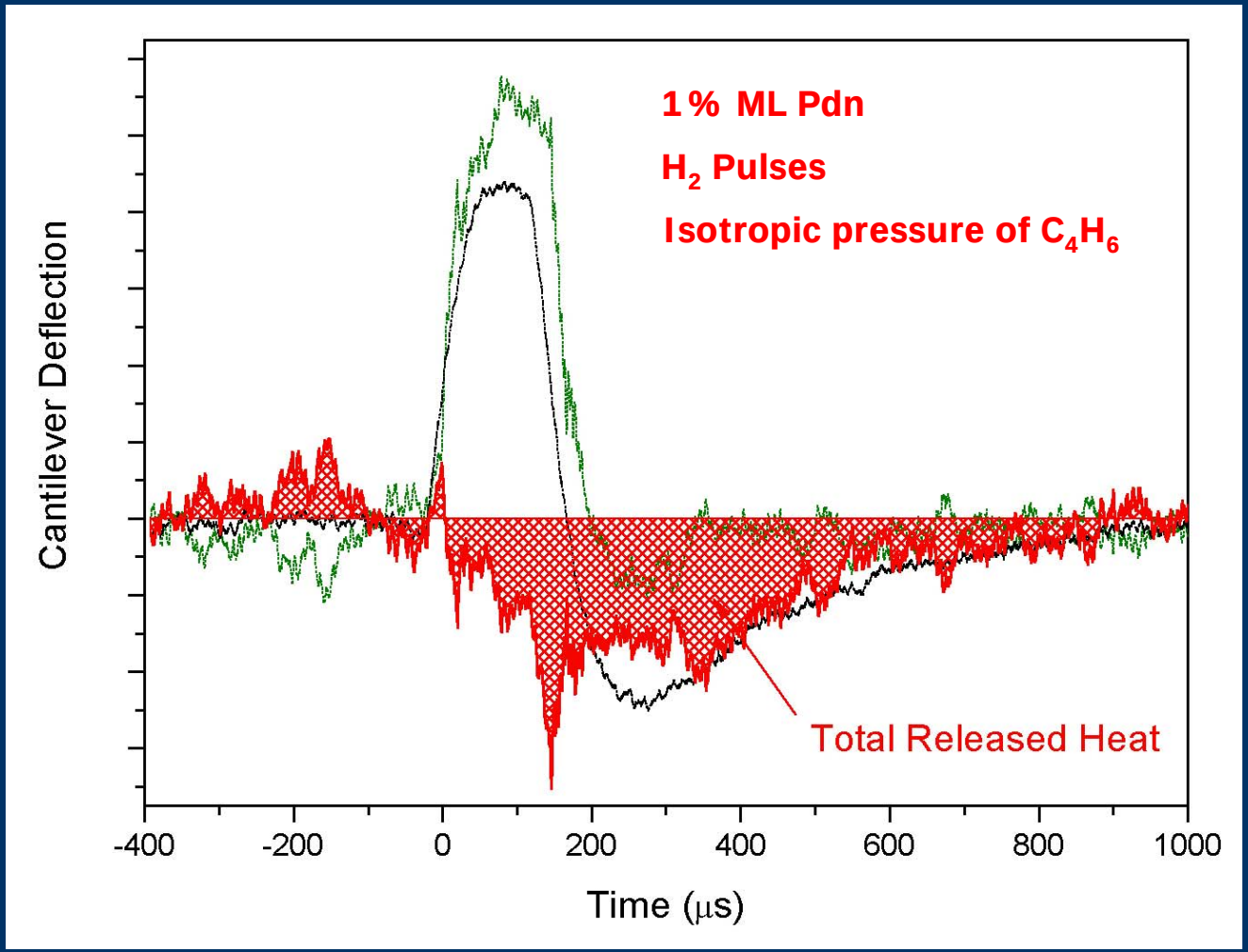
Experimental Techniques: Microcalorimetry

Cantilever response to the reactive gases





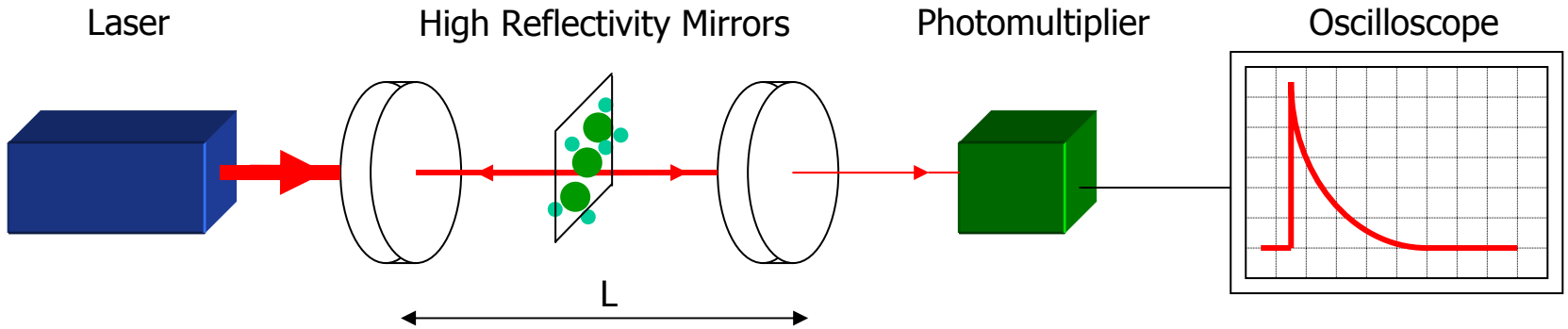
Experimental Techniques: Microcalorimetry



Experimental Techniques: Microcalorimetry

- **Development of a highly sensitive microcalorimeter**
  - **Sensitive to temperature changes of ~10^{-3} K**
  - **100 nWatt sensitivity**
  - **Response time ~ 0.1 - 1 ms**
  - **10 pJoule sensitivity**
- **Study of cluster deposition**
  - **Binding / rearrangement heats**
- **Study of binding energies of adsorbates**
- **Study of reaction heats on clusters on surfaces**

Experimental Techniques: Cavity Ringdown Spectroscopy



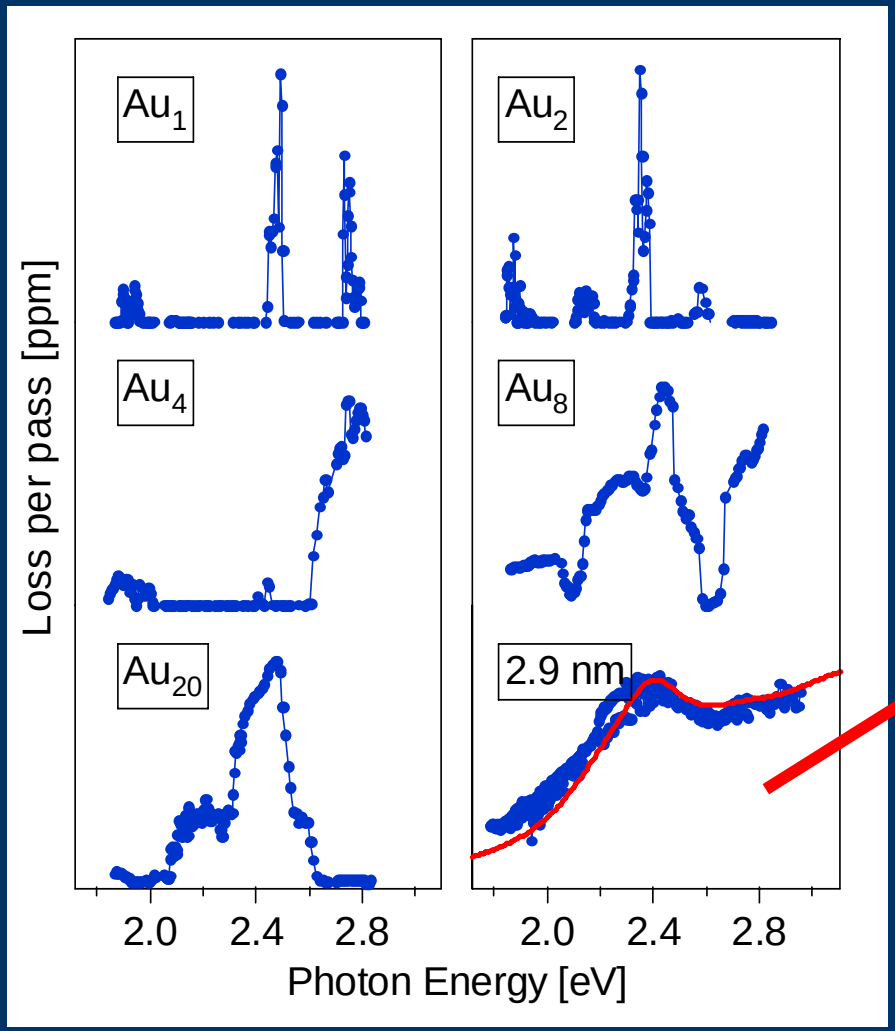
- Λ_0: Intrinsic loss of the cavity (transmission of the mirror, surface scattering, …)
- Λ_s: Additional loss due to the absorption of light by the sample

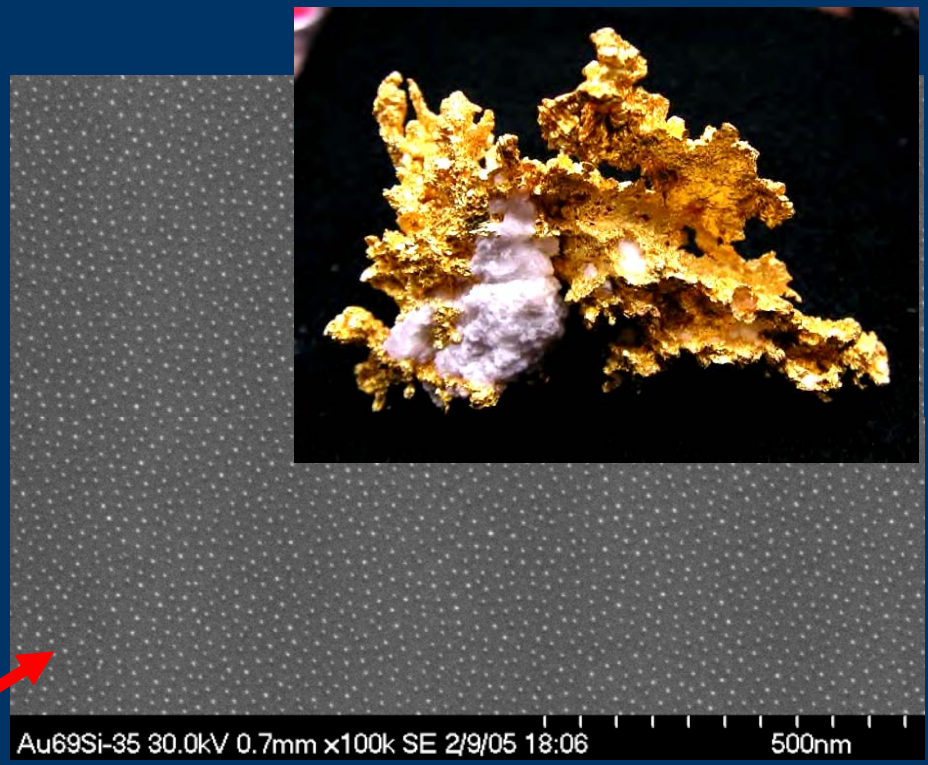
$$\tau_0 = \frac{L}{c\Lambda_0}$$

$$\tau_s = \frac{L}{c(\Lambda_0 + \Lambda_s)}$$

$$\Rightarrow \Lambda_s = \frac{L}{c}\left(\frac{1}{\tau_s} - \frac{1}{\tau_0}\right)$$

Experimental Techniques: Cavity Ringdown spectroscopy





J.-M. Antonietti *et al.*, Phys. Rev. Lett. **94** (2005) 213402

A. Del Vitto *et al.*, J. Phys. Chem. B ***109***, (2005) 19876

Experimental Techniques: Cavity Ringdown spectroscopy

- Softlanding of size-selected clusters
- Characterization of trapping sites
- Characterization of atomic and molecular transitions
- Information of cluster structure
- Transition from atom to bulk

Guiding Principles of Cluster Chemistry

When Gold is not Noble

Structural, Electronic, and Impurity-Doping Effects in Nanoscale Chemistry: Supported Gold Nanoclusters

Charging Effects on Bonding and Catalyzed Oxidation of CO on Au_8 Clusters on MgO

Stéphane Abbet , Ken Judai, Anke Wörz, Jean-Marie Antonietti and Ueli Heiz

Technical University of Munich, Lehrstuhl für Physikalische Chemie, D-85747 Garching

Hannu Häkkinen, Bokwon Yoon and Uzi Landman

Georgia Institute of Technology, School of Physics, Atlanta, Georgia 30332-0430

J. Phys. Chem. A *103* (1999) 9573

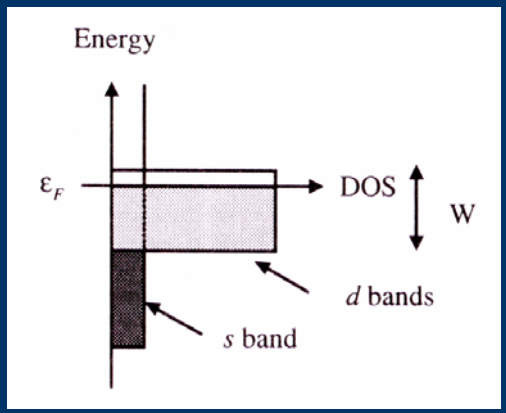
J. Am. Chem. Soc.,*125* (2003) 10437

Angewandte Chemie Int. Ed., *115* (2003) 1335

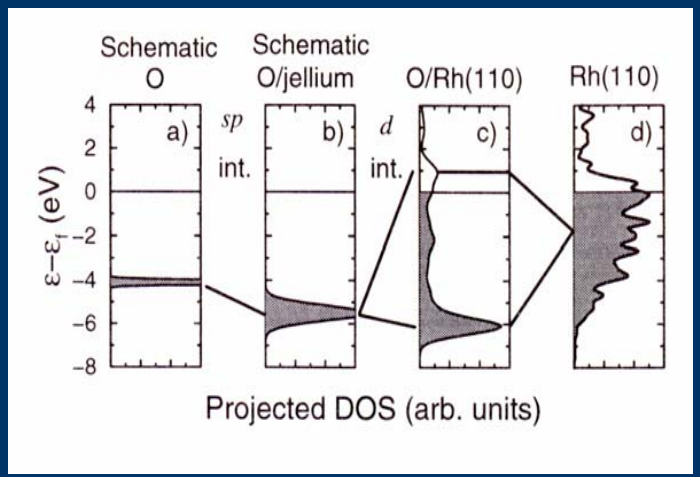
Science *307* (2005) 403

Why is Gold Noble in the Solid State?

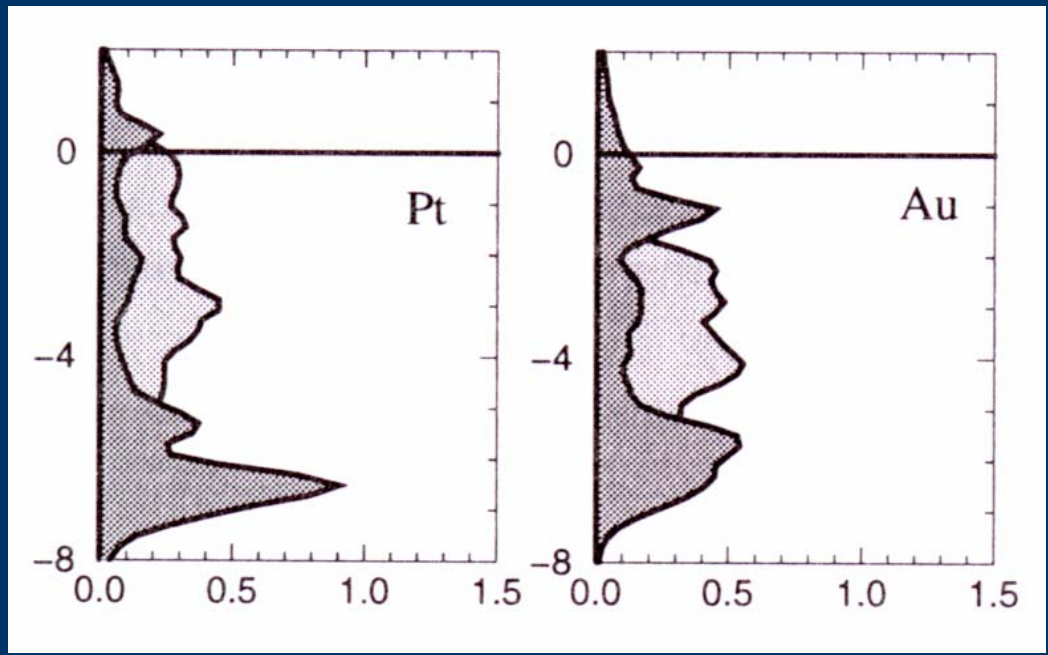
Typical DOS of Metals



Coupling of Molecular State with DOS of Metal



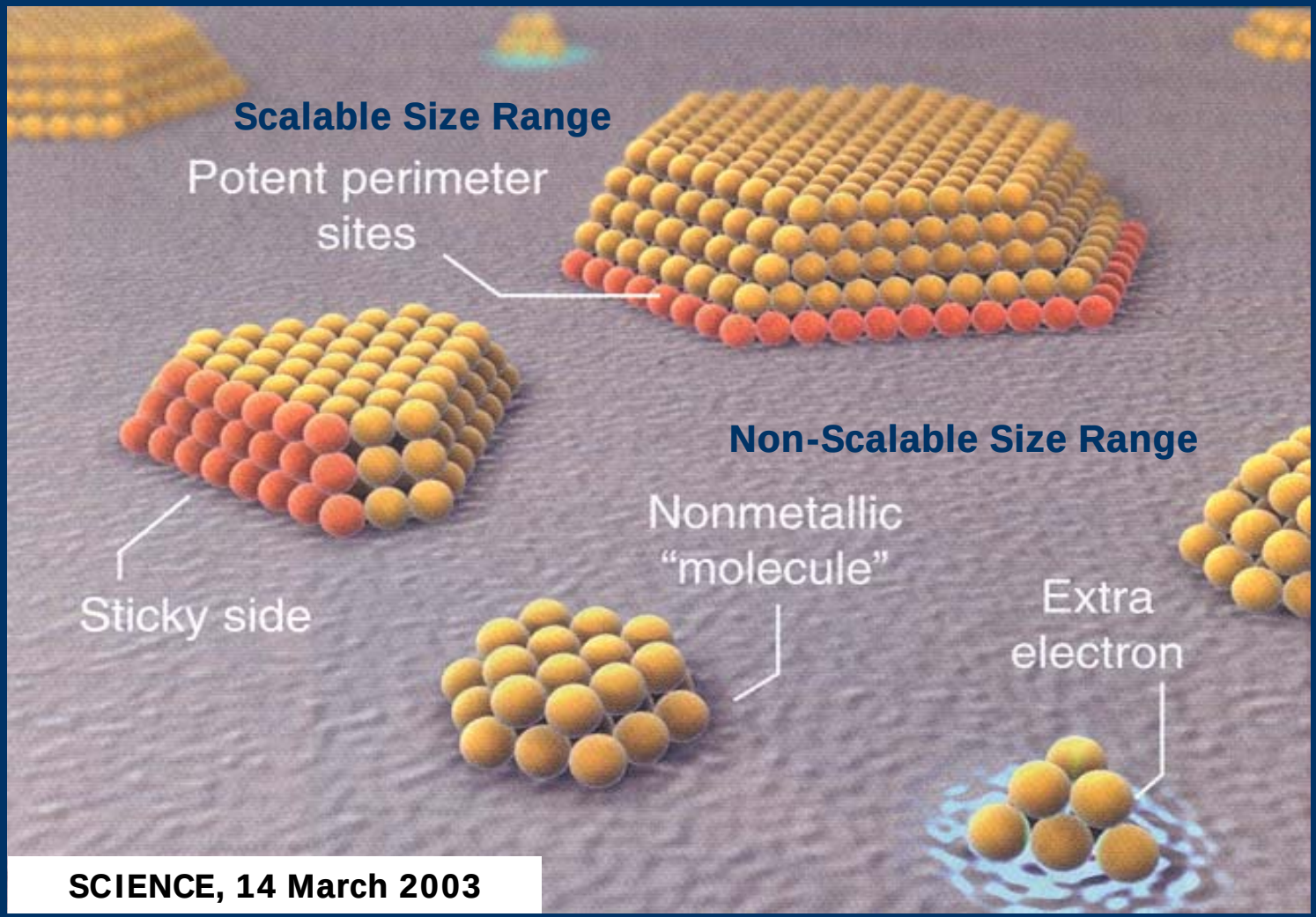
Why is gold noble?



LDOS projected onto the p_x state of adsorbed oxygen (Dark Area)

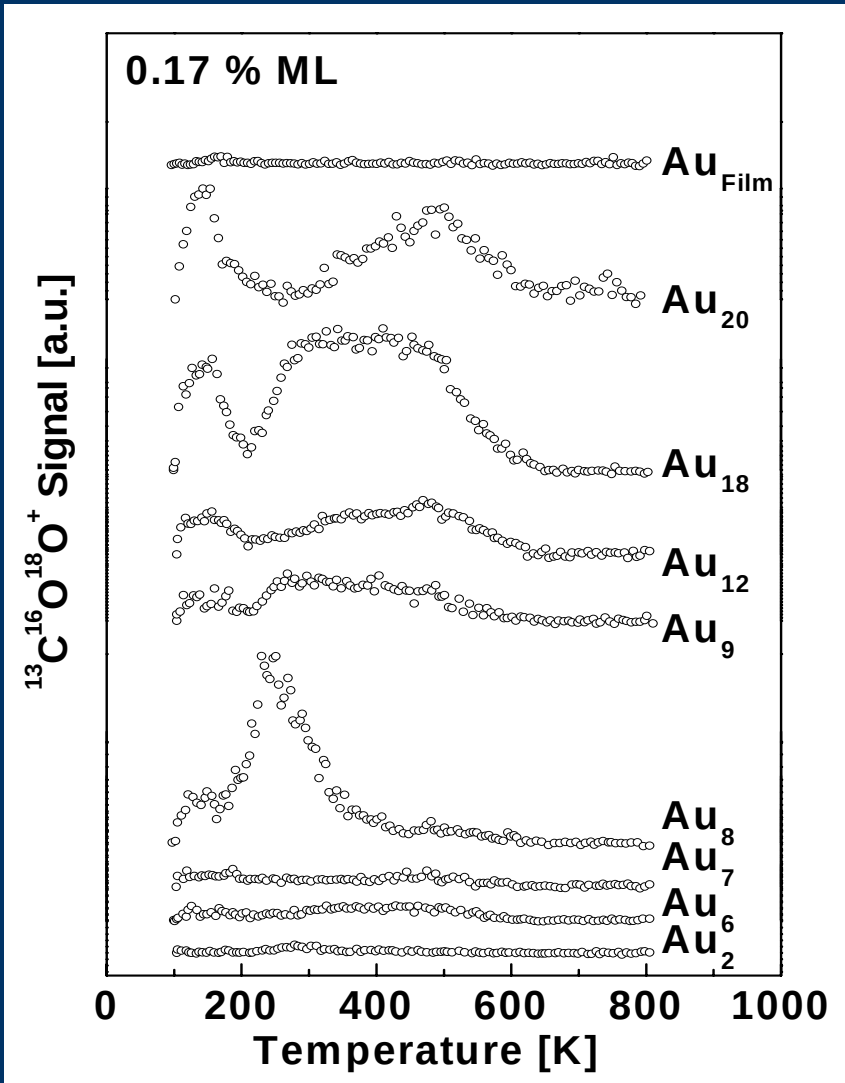
*Hammer et al., Nature **376** (1995) 238*

Gold Nanocatalysts



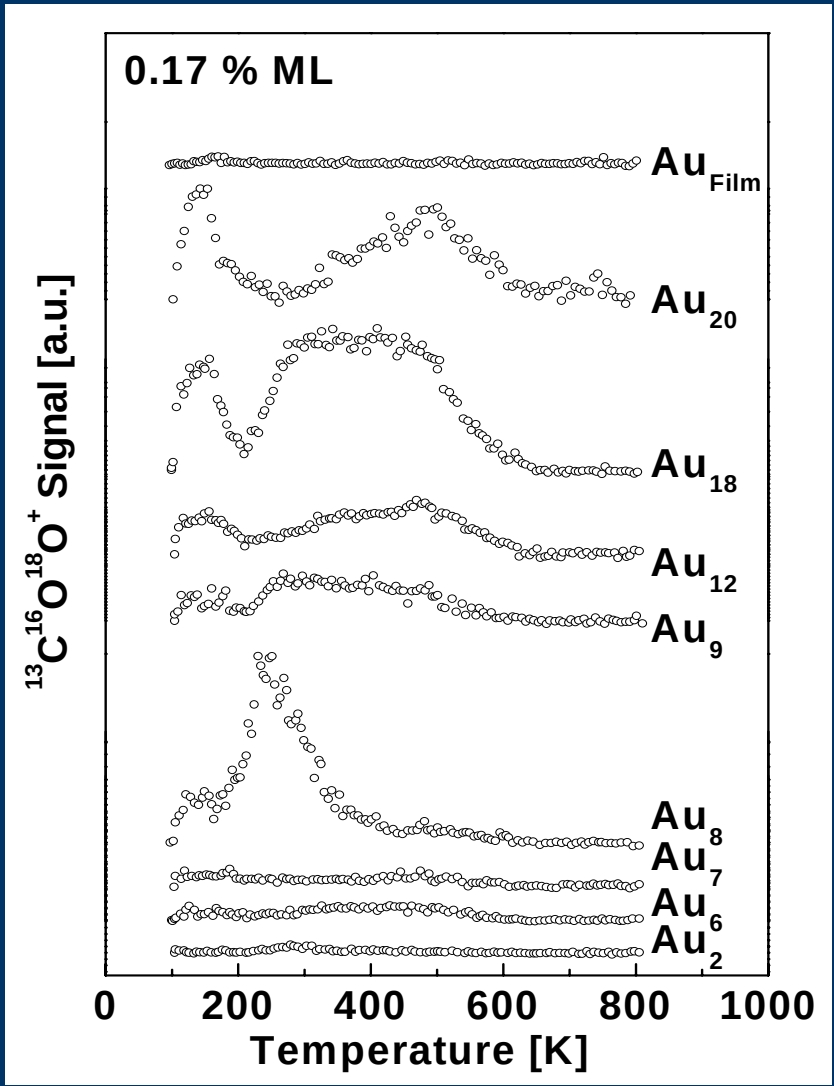
SCIENCE, 14 March 2003

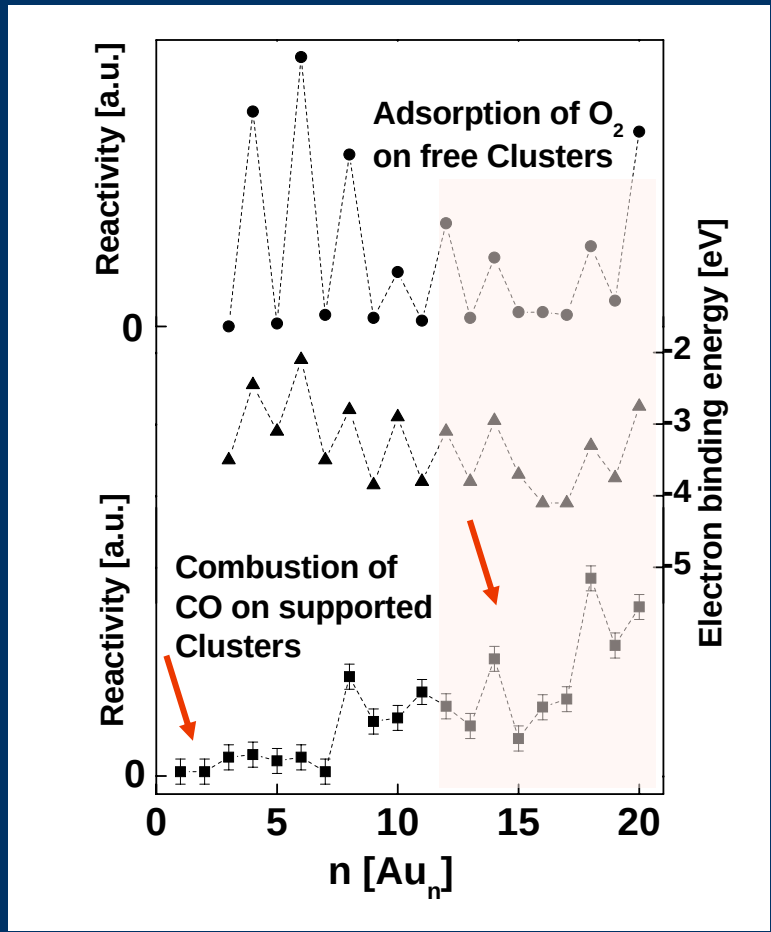
Formation of CO_2 on Supported Gold Clusters



Each Atom Counts !

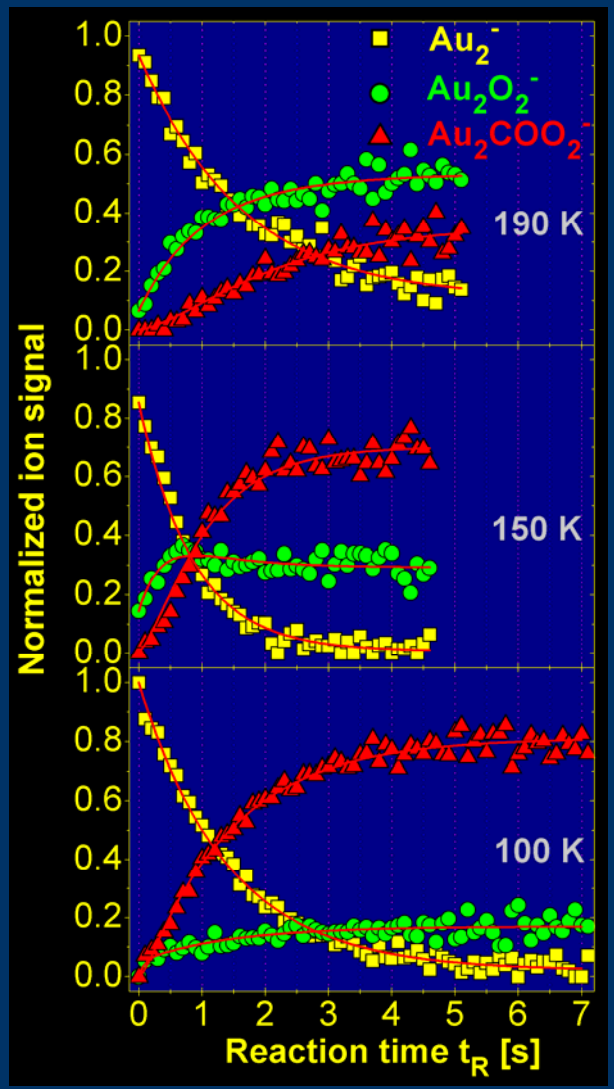
Comparison to Reactivity of Free Gold Cluster Anions





Note: No O_2 adsorption on neutral and cationic gold clusters !

Reaction Mechanism of the CO Combustion on Free Au_2^-



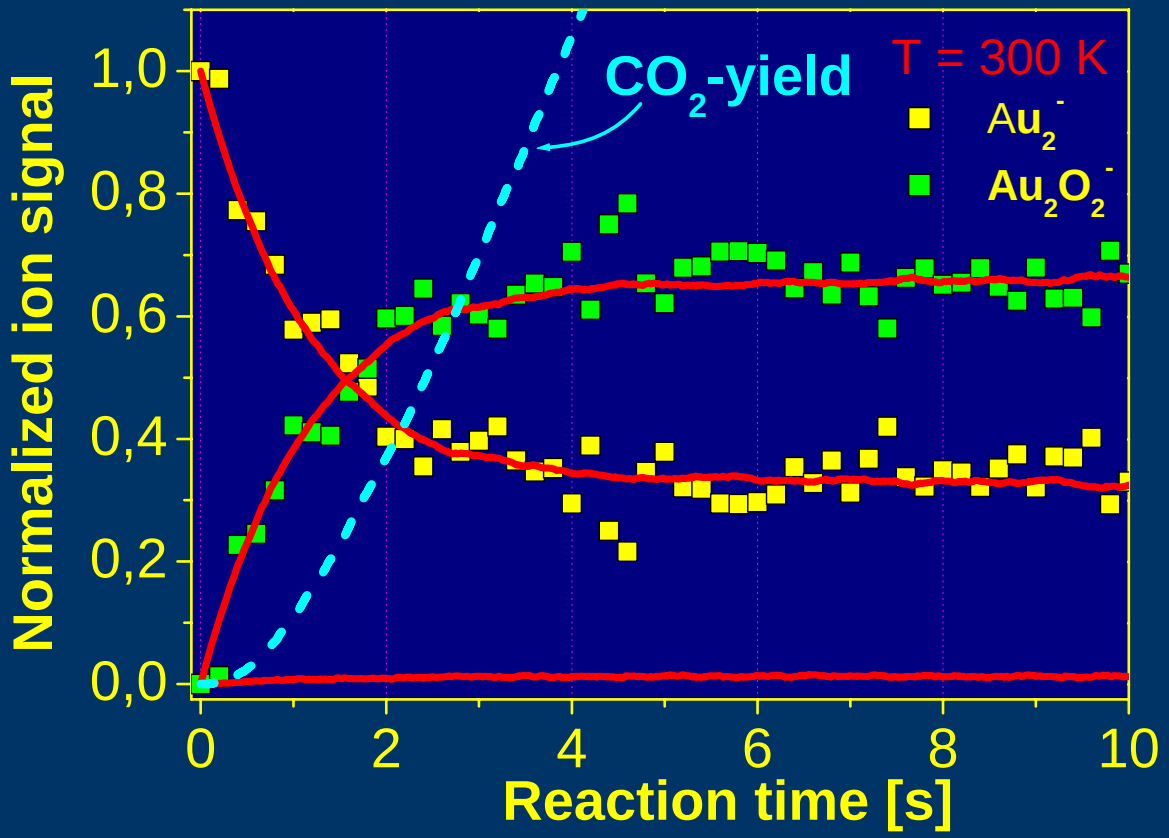
Elementary steps needed to fit data

$Au_2^- + O_2$	$\rightarrow$	$Au_2O_2^-$	k_1
$Au_2O_2^- + CO$	$\leftrightarrows$	$Au_2(CO)O_2^-$	$k_{2,3}$
$Au_2(CO)O_2^- + CO$	$\rightarrow$	$Au_2^- + 2\,CO_2$	k_4

with $k_{1\text{-}4}$ pseudo first order rate constants (pressure dependent).

L. Socaciu, J. Hagen, T. Bernhardt, L. Wöste, U. Heiz, H. Häkkinen, U. Landman
J. AM. CHEM. SOC. 2003, 125, 10437

Catalytic Turn-Over Frequency (TOF)



TOF ≈ 0.6 CO_2 molecules per gold cluster per second

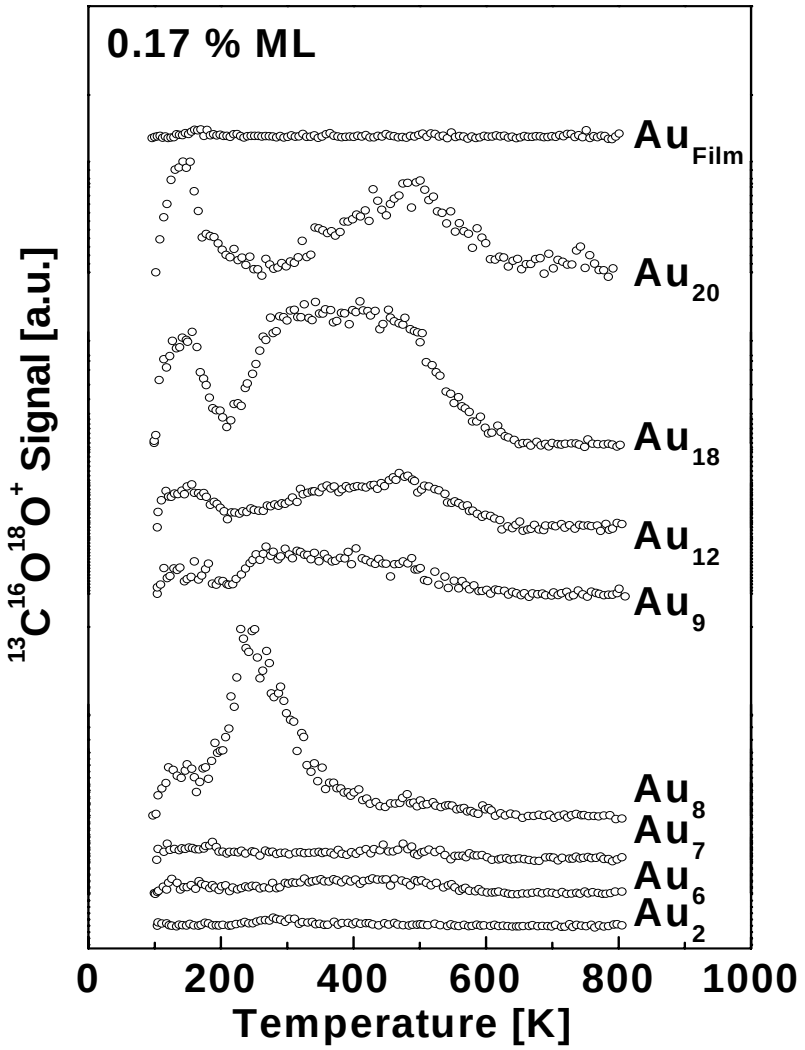
2 nm gold particles at 273 K: TOF = 0.2 s^{-1} per Au atom (Haruta et al.)
3.5 nm gold particles at 350 K: TOF = 4 s^{-1} per Au atom (Goodman et al.)

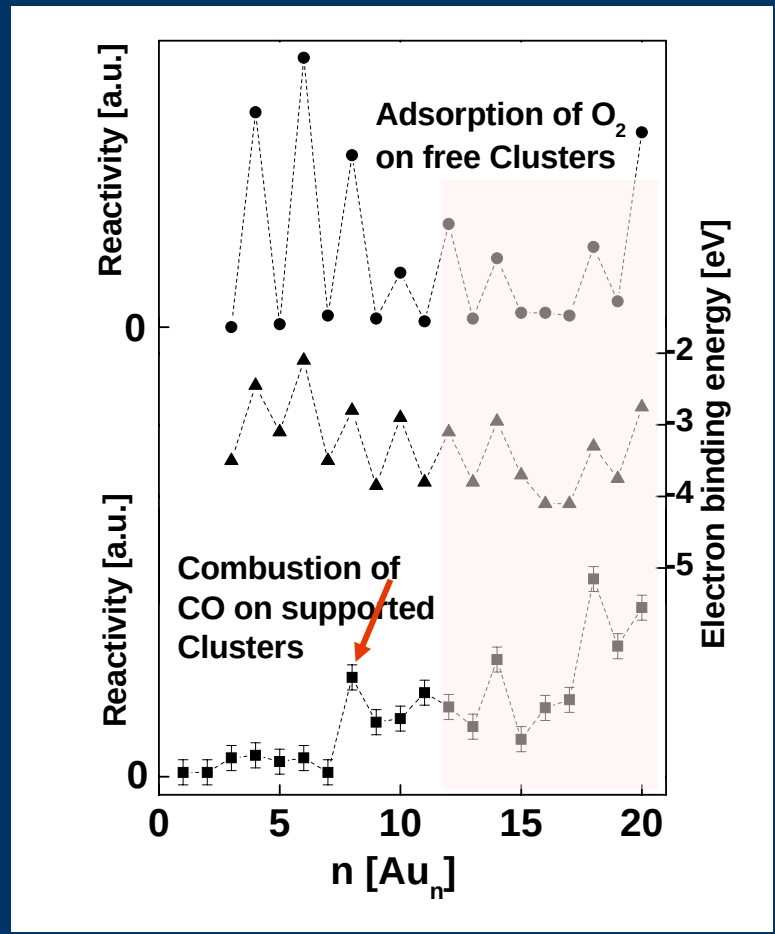
Free Clusters May Behave Differently Than Supported Clusters

Steric Hinderence

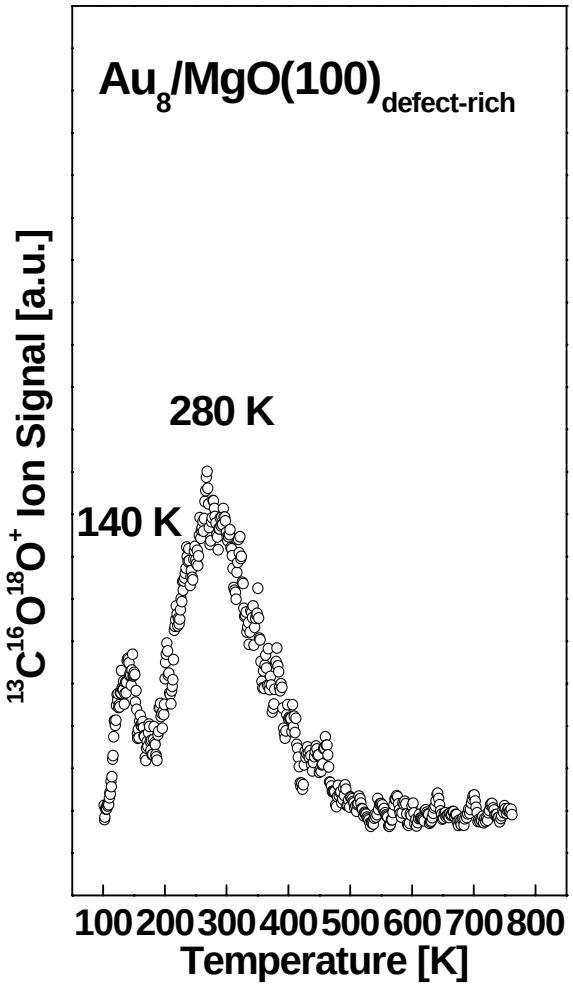
Change of Electronic Structure

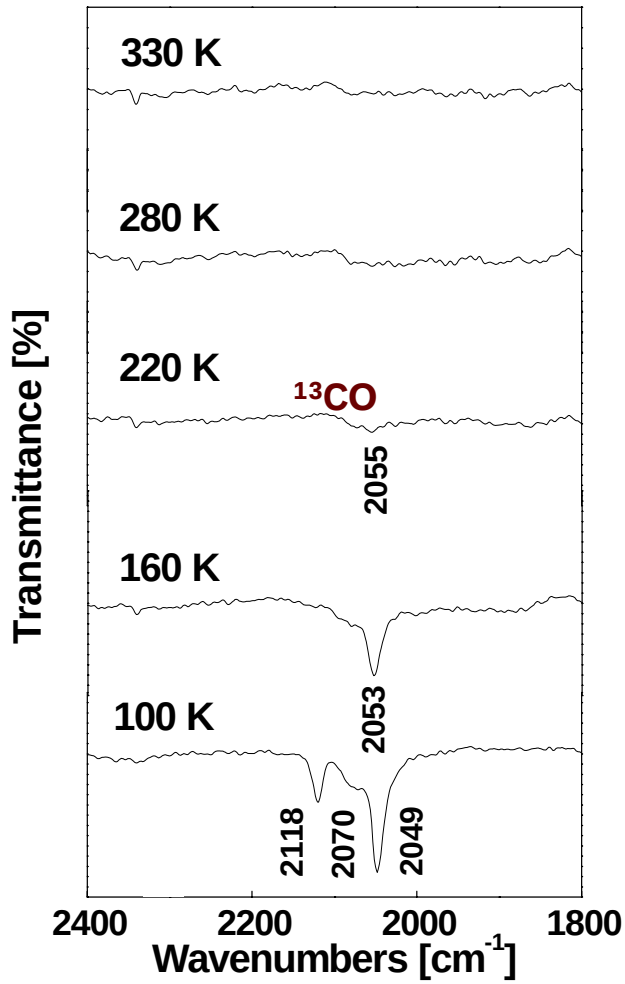
Reactivity of Free and Supported Nanoscale Gold

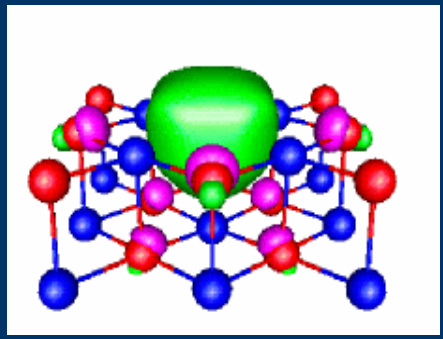




Influence of Defect Sites: Au_8 on MgO(100) F-centers



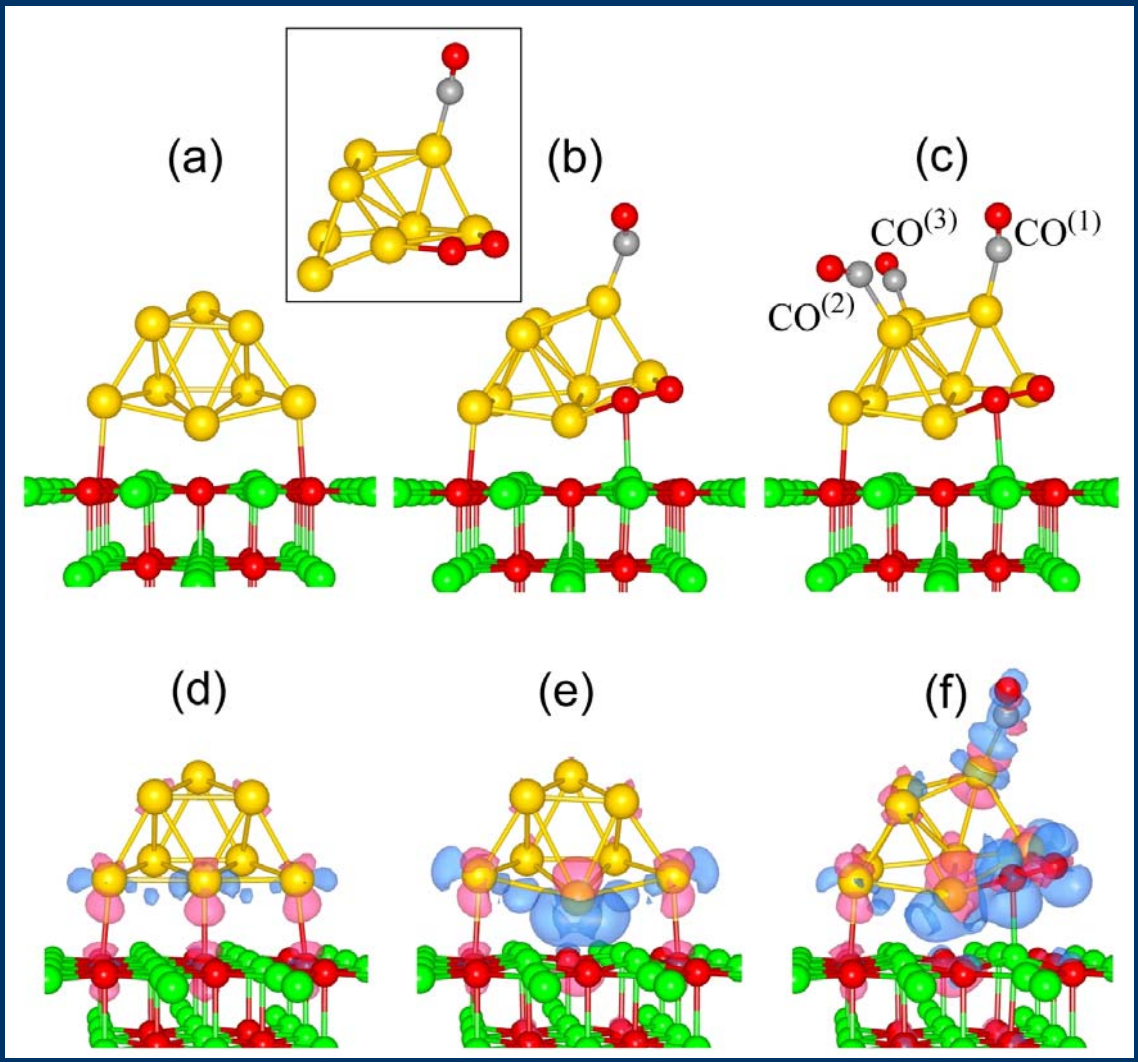




F-center
Oxygen vacancy

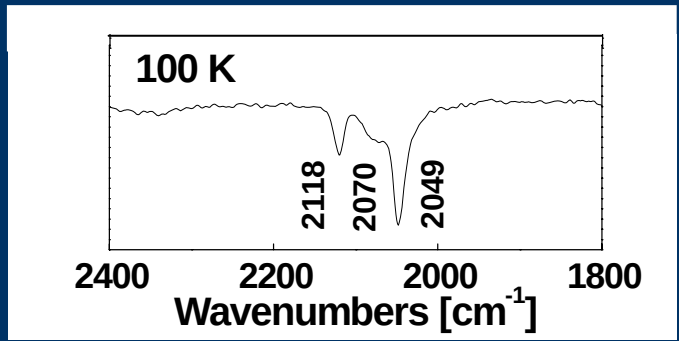
B. Yoon, H. Häkkinen, U. Landman, A. Wörz, J.-M. Antonietti, S. Abbet, K. Judai, U. Heiz, *Science 307 (2005) 403*

Theoretically Proposed Structure



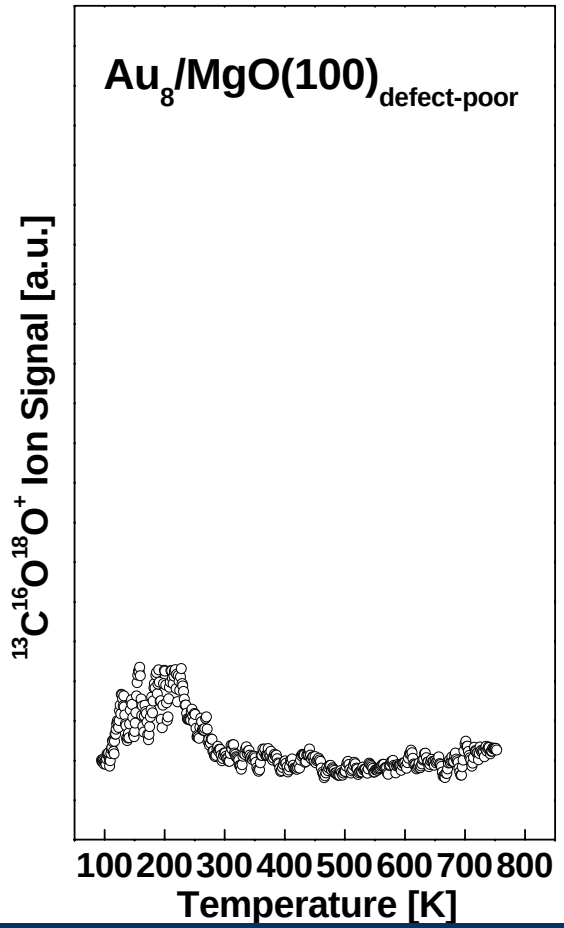
$^{13}CO/Au_8/MgO(FC)$:

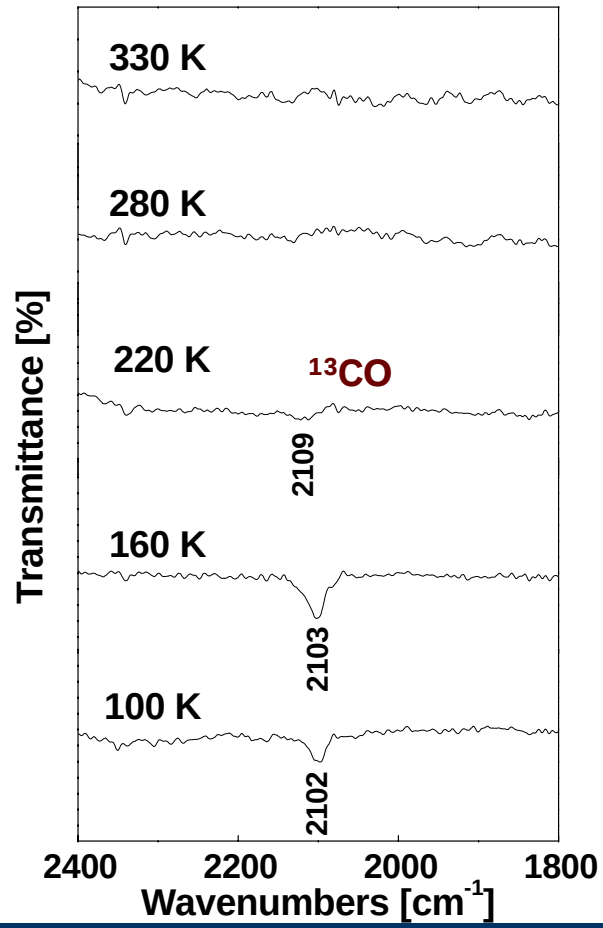
$\nu_{theor.}$: 2018 cm^{-1} (1)
$\nu_{theor.}$: 1931 cm^{-1} (2)
$\nu_{theor.}$: 2004 cm^{-1} (3)



$\nu_{CO/MgO}$: 2118 cm^{-1}

Influence of Defect Sites: Au_8 on MgO(100)

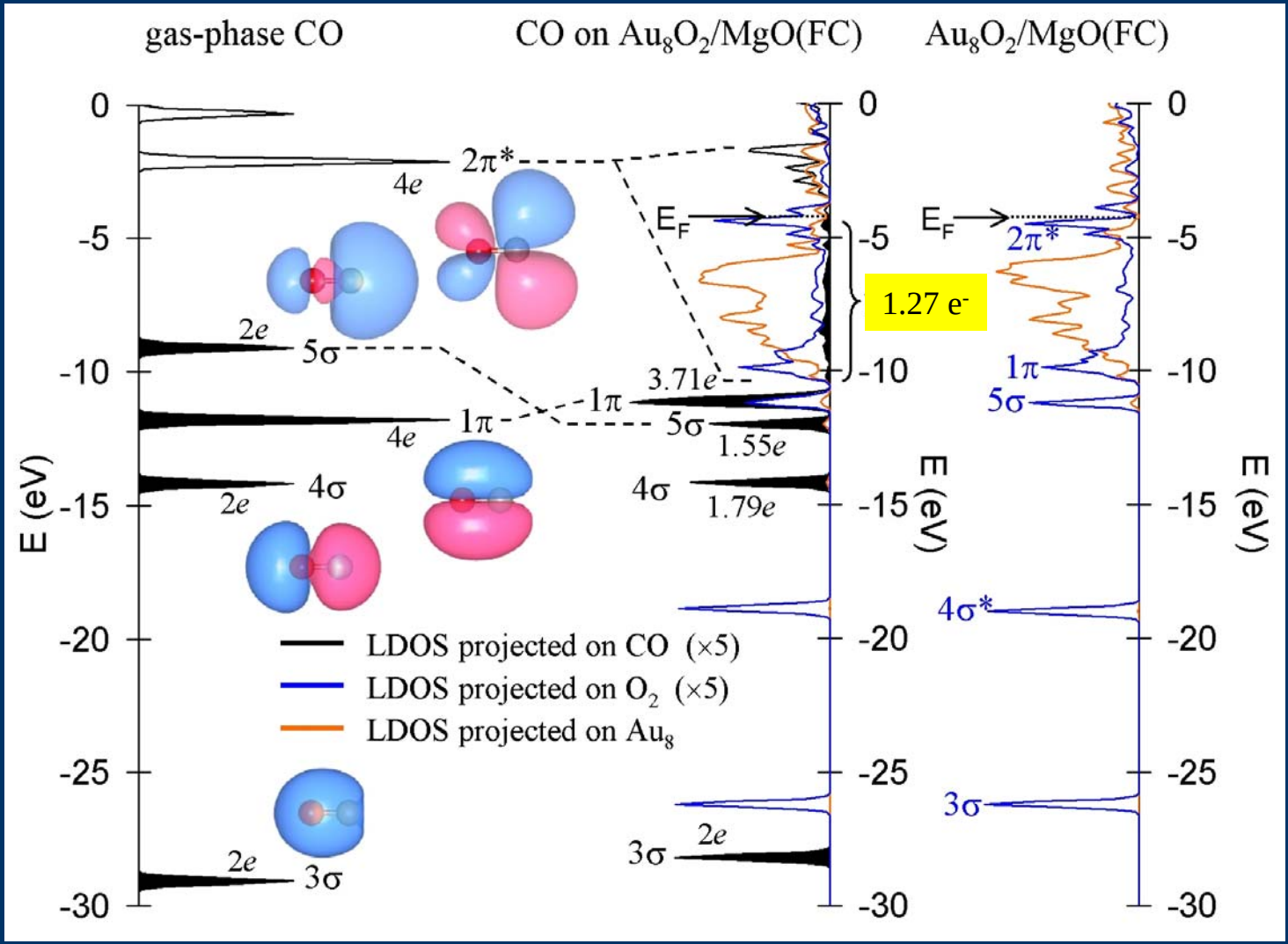




Redshift induced by F-center:

$\Delta\nu$ = 30-50 cm^{-1}

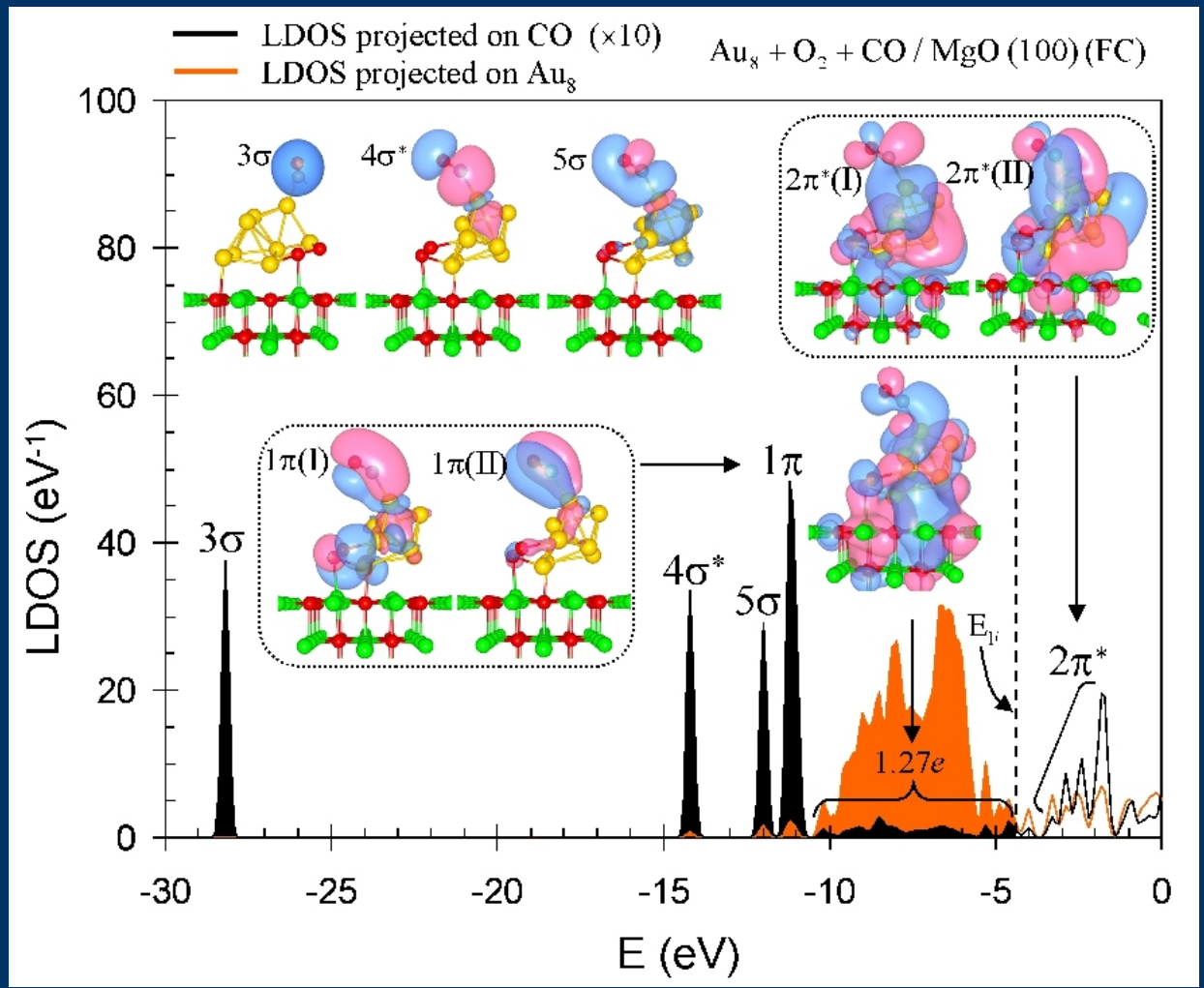
CO bonding on $Au_8O_2/MgO(FC)$



CO on Au_8O_2 / MgO:

1.18 e⁻

CO bonding on Au_8O_2/MgO (FC)



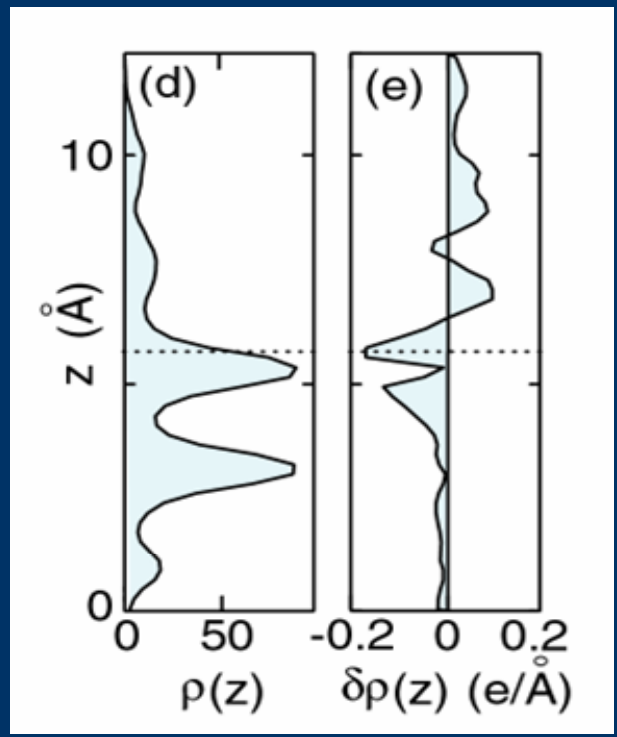
CO-bonding via backdonation into 2π* and donation of 5σ into cluster

Effect of F Centers: Cluster Charging

$\Delta \nu_{exp.}(cm^{-1})$: 30-50

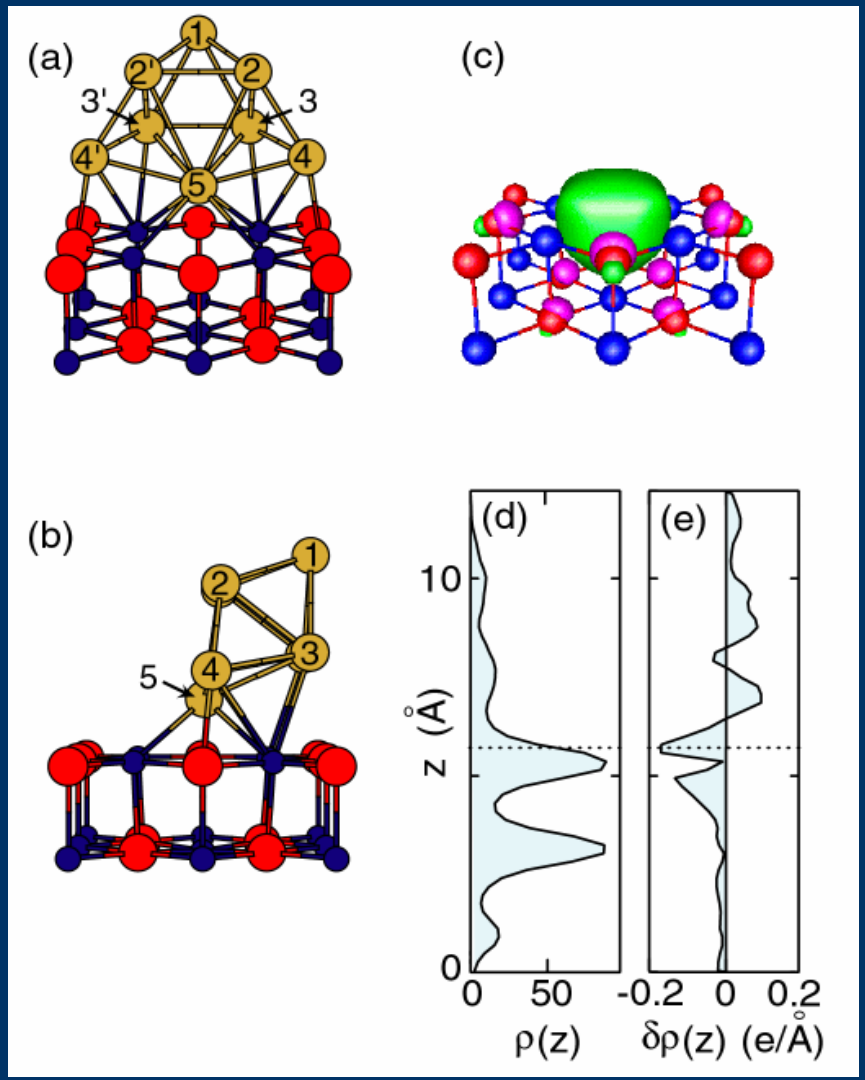
$\Delta Q\ Au_8O_2{}^{13}CO$	spin	$\Delta \nu_{theor.}(cm^{-1})$
0	1	0
0.25	0.875	18
0.5	0.75	37
0	0	0
0.25	0	19
0.5	0	34

Charging ⇔ Frequency shift



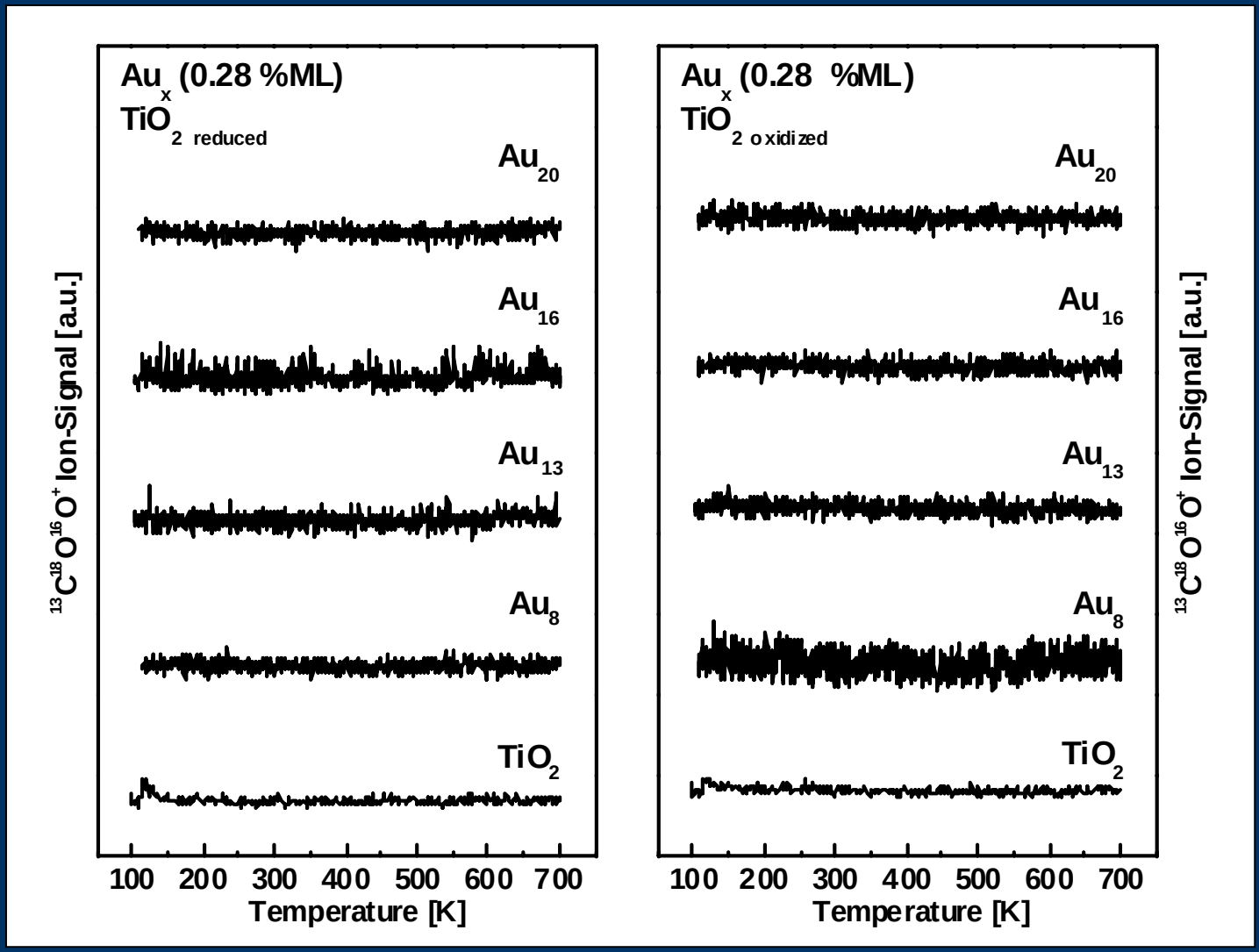
$\Delta Q = 0.5$

Effect of F Centers: Cluster Stabilization



- **Strong binding between cluster and F-center; 3.4 eV in comparison to 1.2 eV on regular terrace sites.**
- **Charge transfer to the cluster: ~ 0.5 e^-**

CO-Oxidation on Au_n on TiO_2

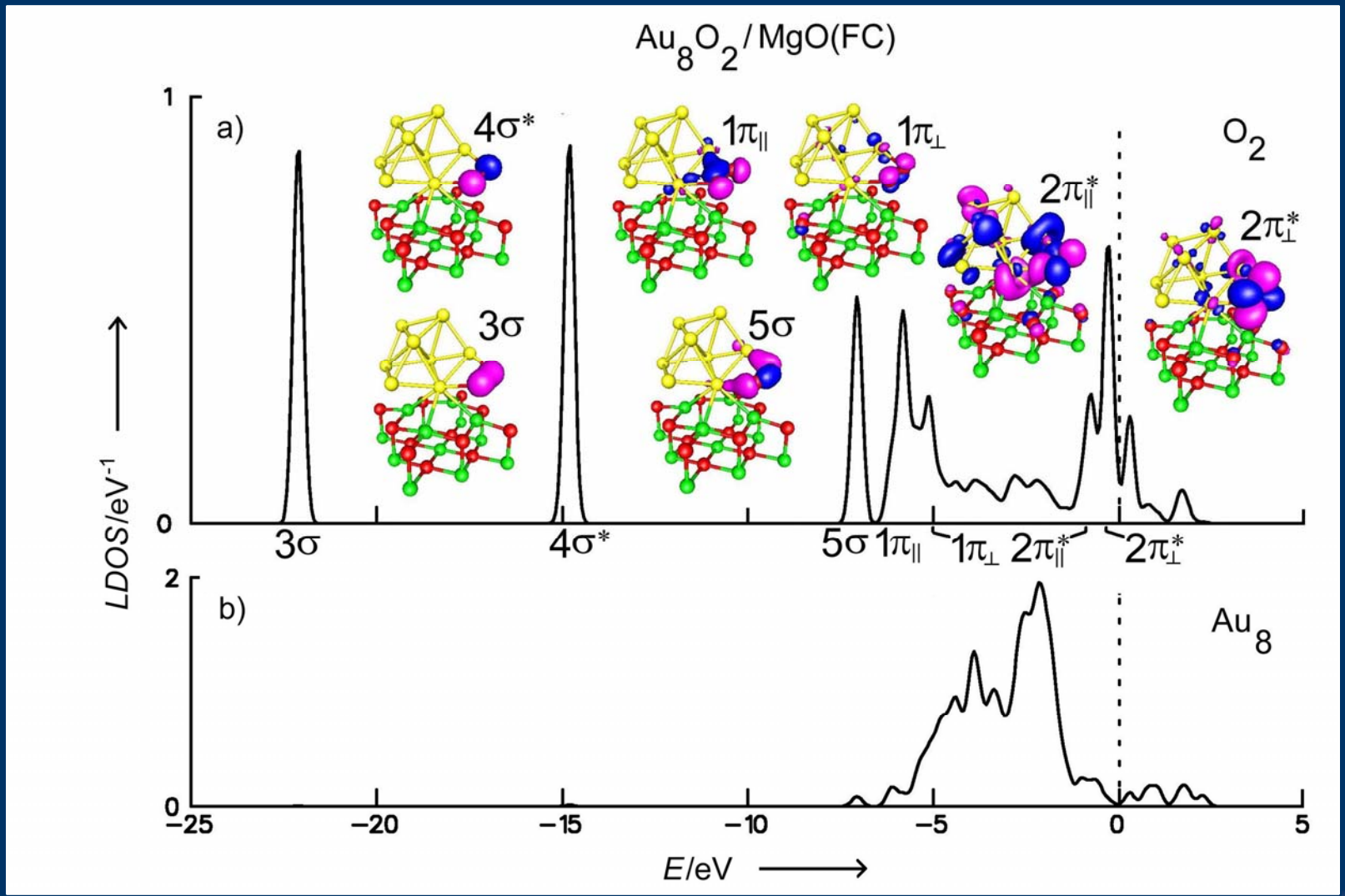


Cluster-Support-Interaction:

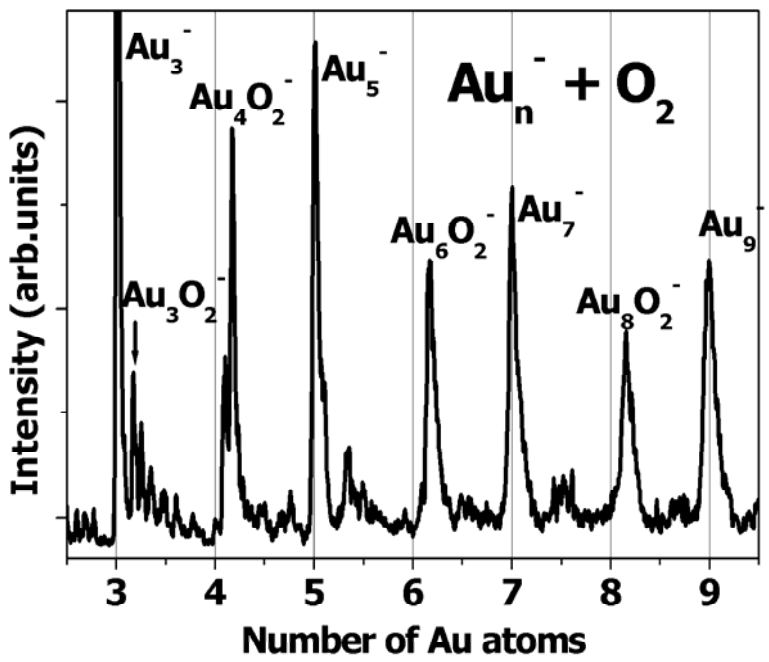
- Stabilization
- Charging

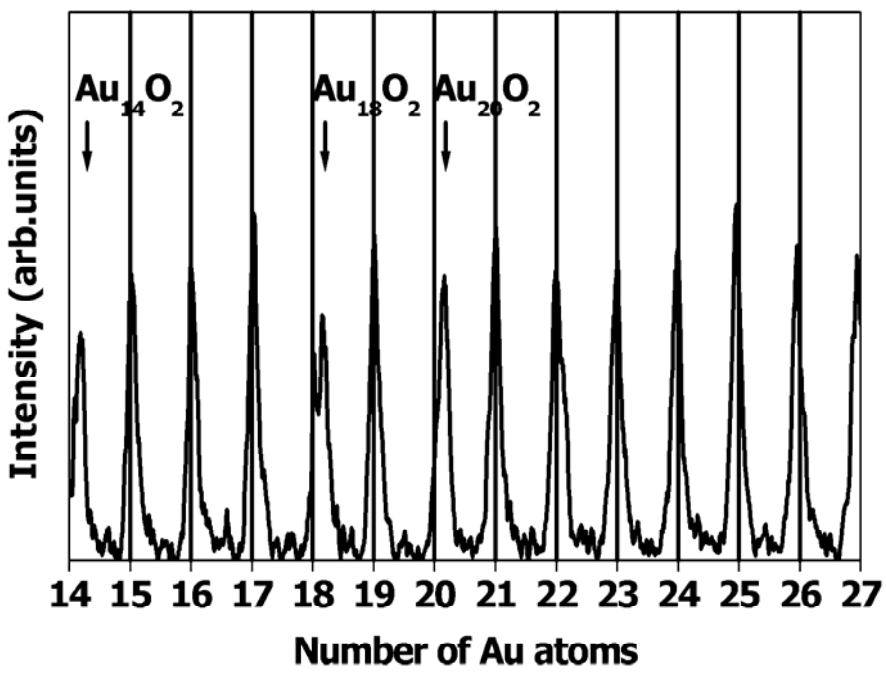
Why is Gold Active at low Temperatures ?

Effect of F centers: Activation of O_2 (peroxo state)



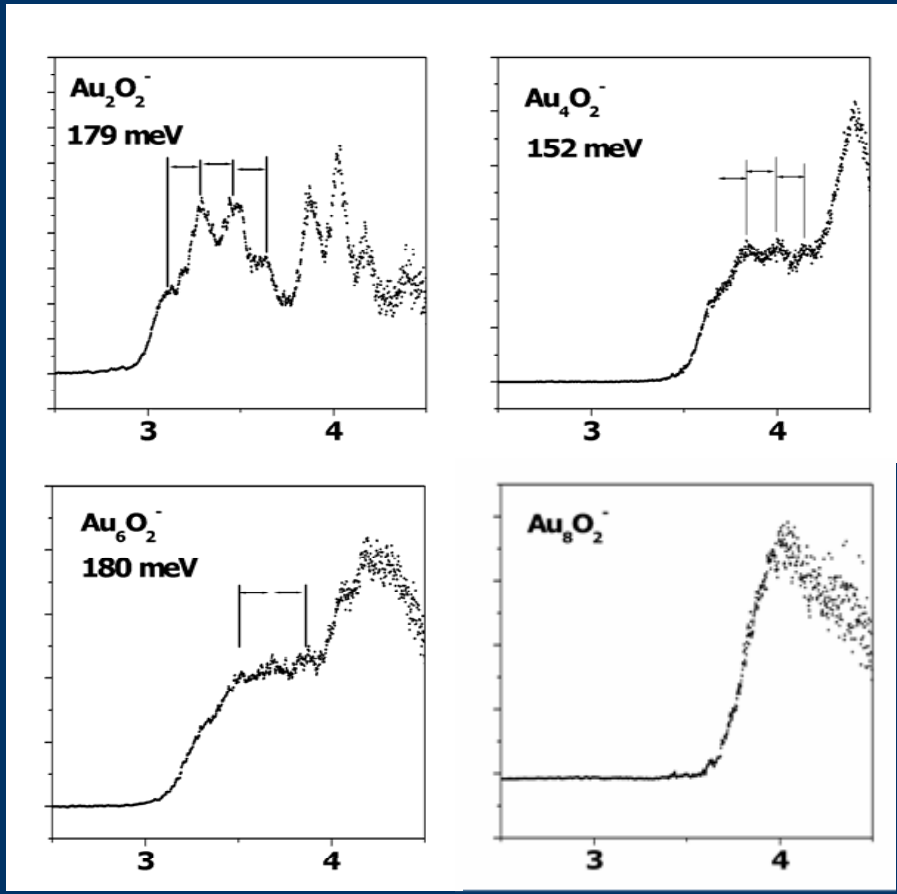
Comparison with Gas Phase Studies





Stolcic, Fischer, Ganteför, Kim, Sun and Jena *J. Am. Chem. Soc. 125, (2003) 2848*

Experimental Evidence of Molecular O_2 Adsorption



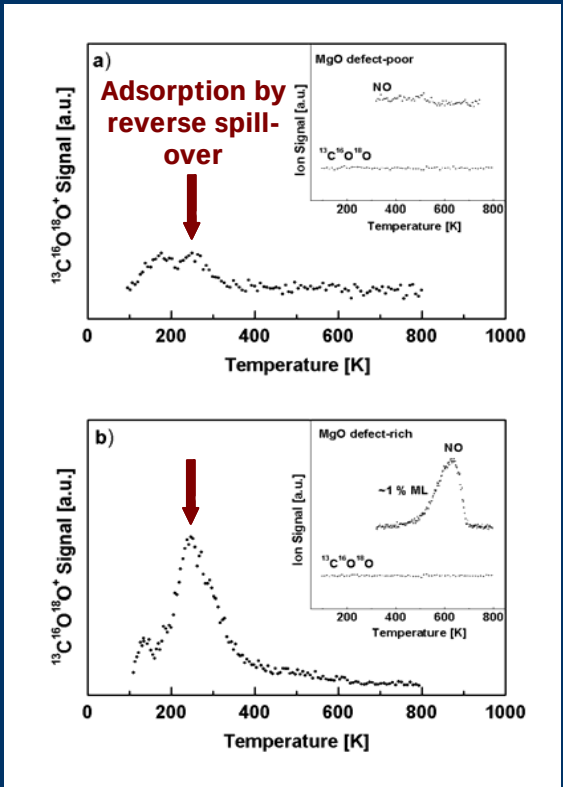
Stolcic, Fischer, Ganteför, Kim, Sun and Jena
J. Am. Chem. Soc. 125, 2848 (2003)

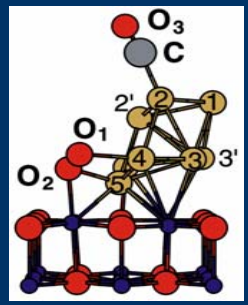
Unique Activation of Reactants on Clusters !

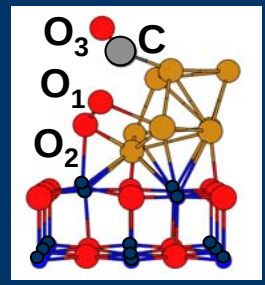
Identification of possible Reaction Mechanisms

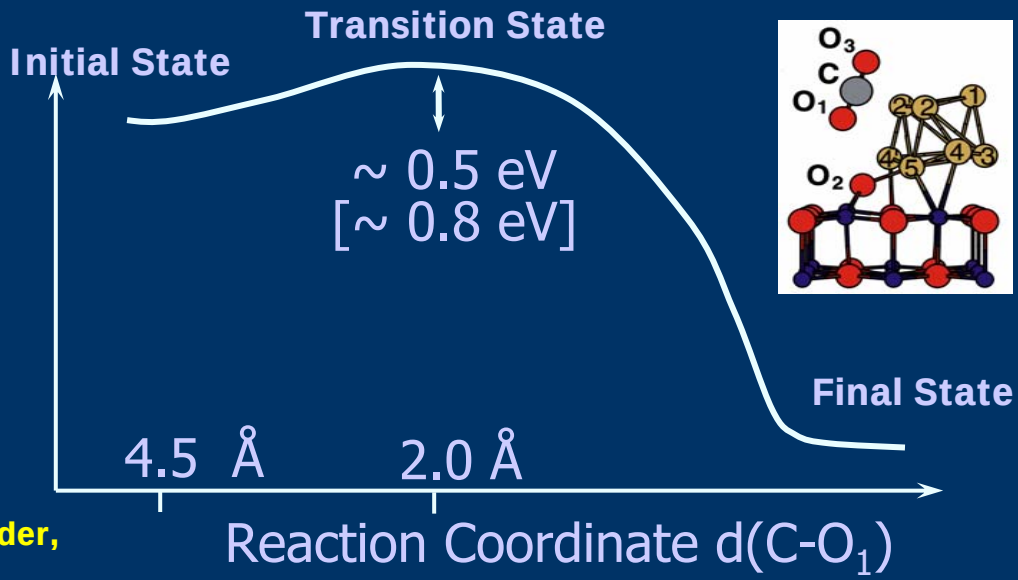
Oxidation of CO on Au_8 Bound to Defect-Poor and Defect-Rich MgO(100) Surfaces

Langmuir-Hinshelwood-Periphery Mechanism





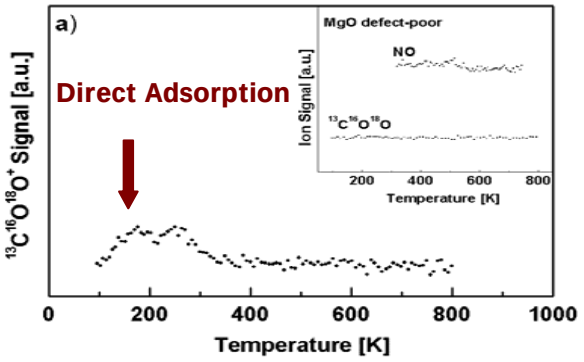


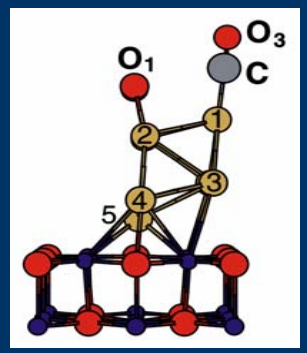


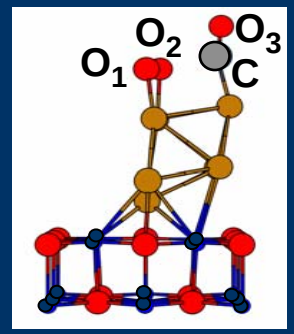
A. Sanchez, S. Abbet, U. Heiz, W.-D. Schneider, H. Ha1kkinen, R. N. Barnett, Uzi Landman
J. Phys. Chem. A **1999**, *103*, 9573

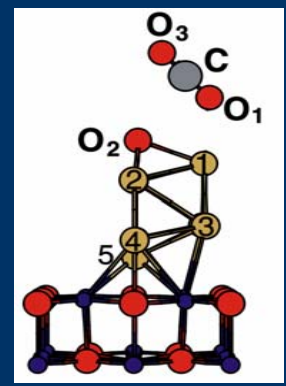
Oxidation of CO on Au_8 Bound to Defect-Poor and Defect-Rich MgO(100) Surfaces

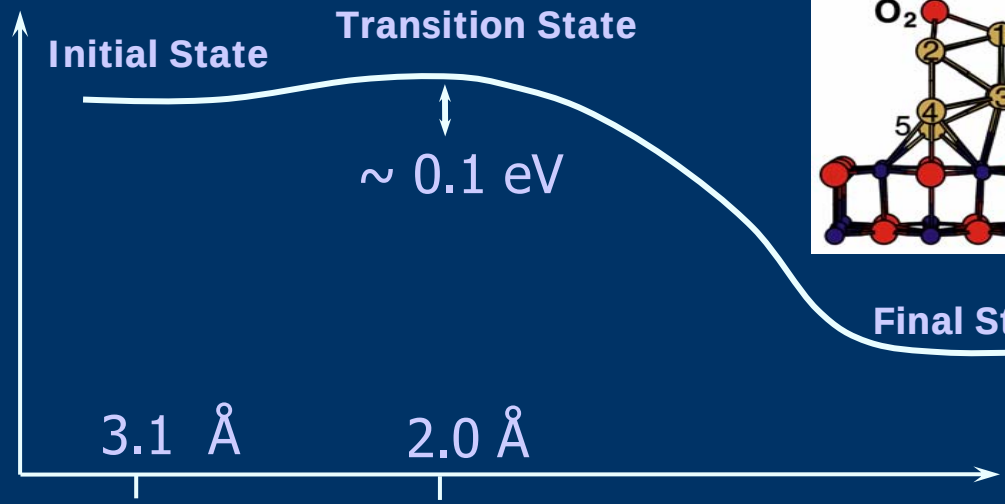
Langmuir-Hinshelwood-Top Mechanism



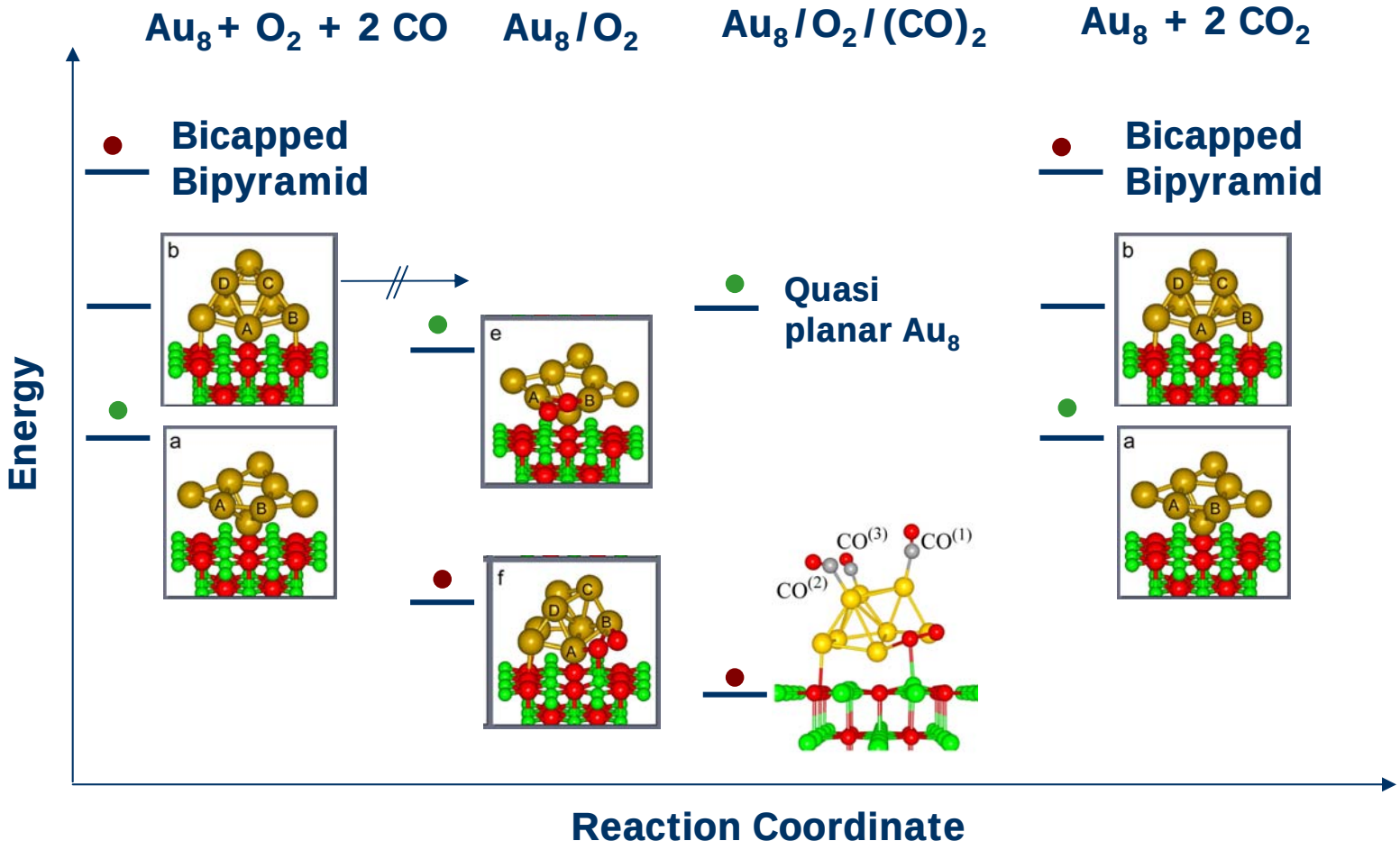






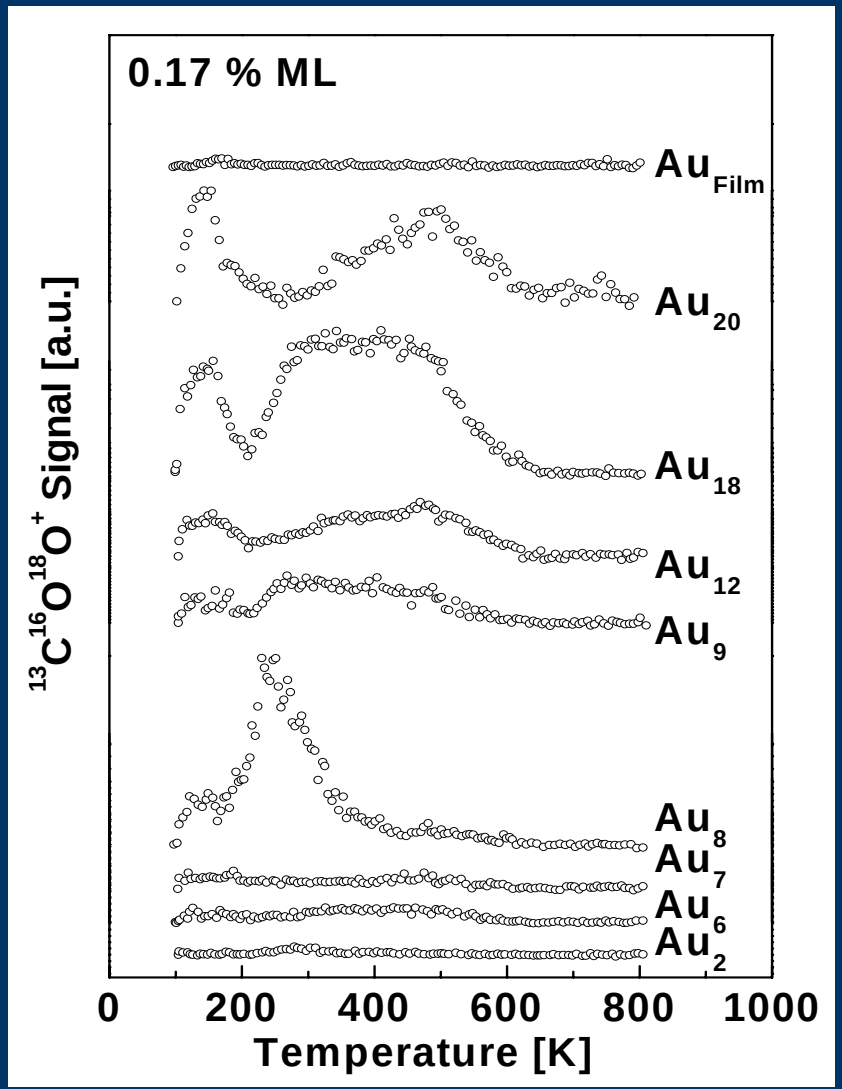


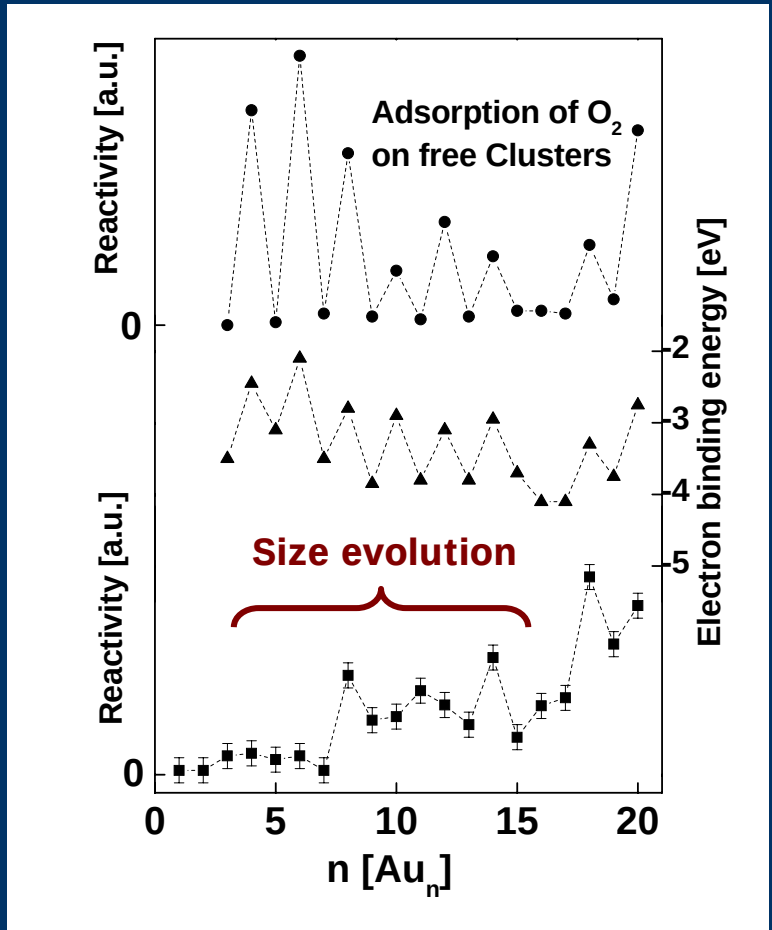
Dynamic Structural Fluxionality



Dynamic Structural Fluxionality

Evolution of Reactivity with Size and Elemental Composition

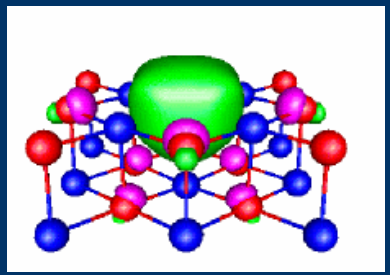




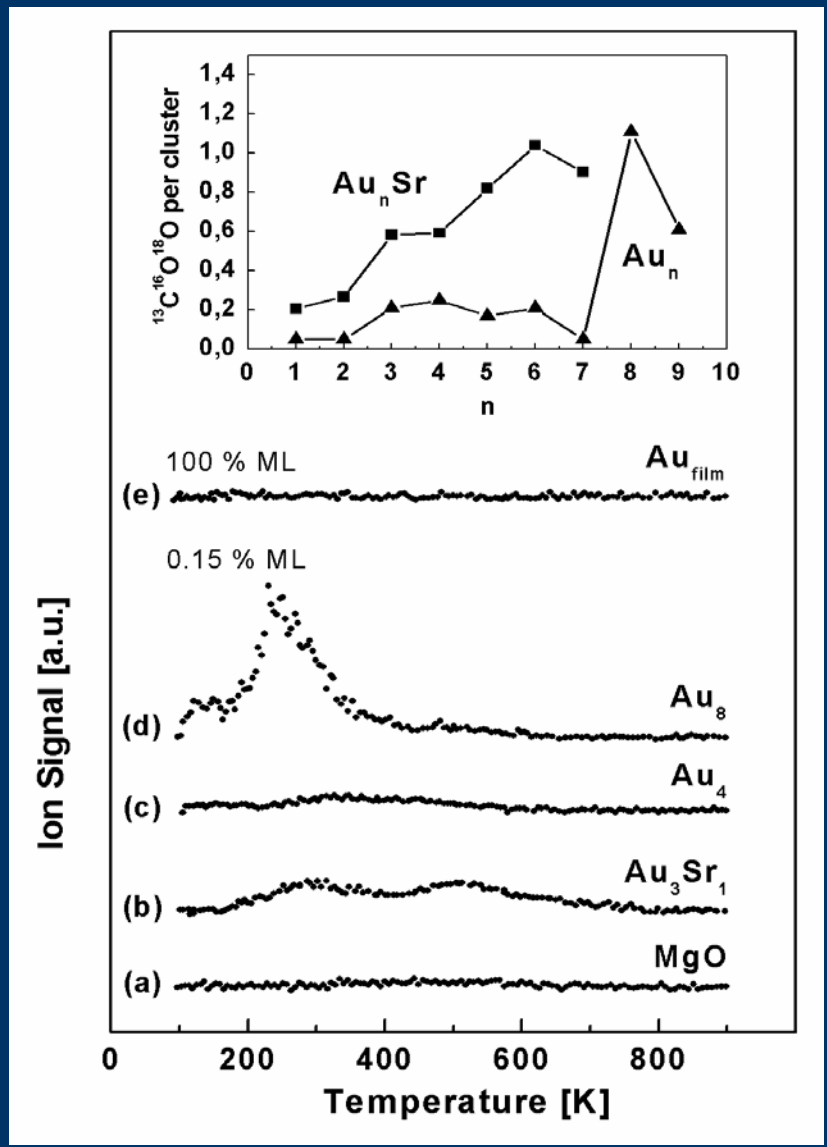
Activation by Impurity Doping
Understanding Size-Evolution of the Reaction

Gold Cluster Reactivity

Cluster deposition of FC/MgO(100)



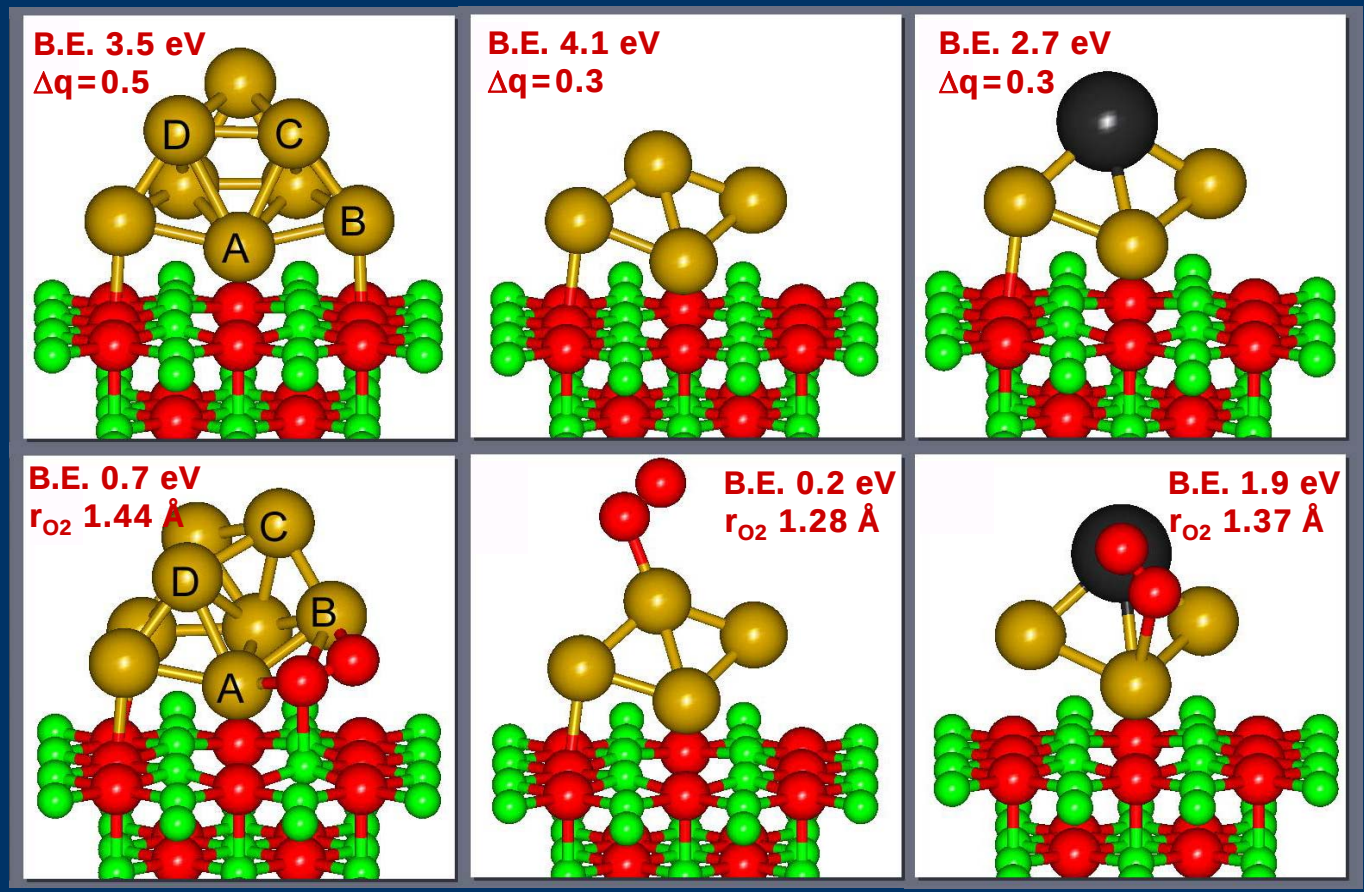
- Au_n (n<8) inert
- Au_8 smallest gold catalyst
- Au_3Sr smallest doped cluster
- MgO and Au_{film} inert



i. Sanchez, S. Abbet, U. Heiz, W.-D. Schneider, H. Häkkinen, R. N. Barnett and U. Landman
When gold is not noble: Nano-scale gold catalyst.
J. Phys. Chem. A *103* 9573-9578 (1999)

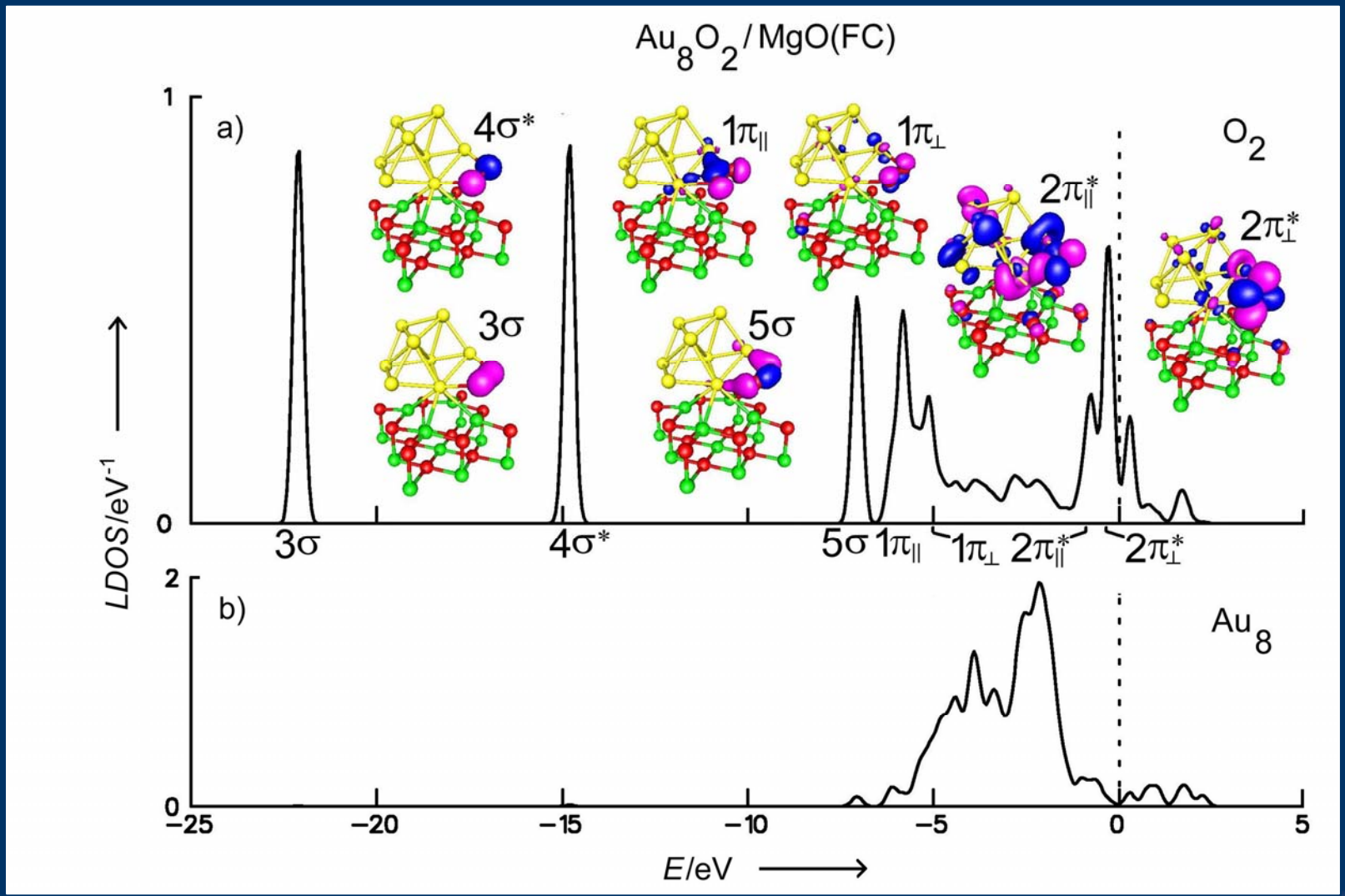
ii. H. Häkkinen, S. Abbet, A. Sanchez, U. Heiz, and U. Landman
Structural, electronic, and impurity-doping effects in nanoscale chemistry: Supported gold nanoclusters.
Angewandte Chemie Int. Ed., *42* 1297-1300 (2003)

Optimized Atomic Structures of Pure and Mixed Gold Nanocatalysts

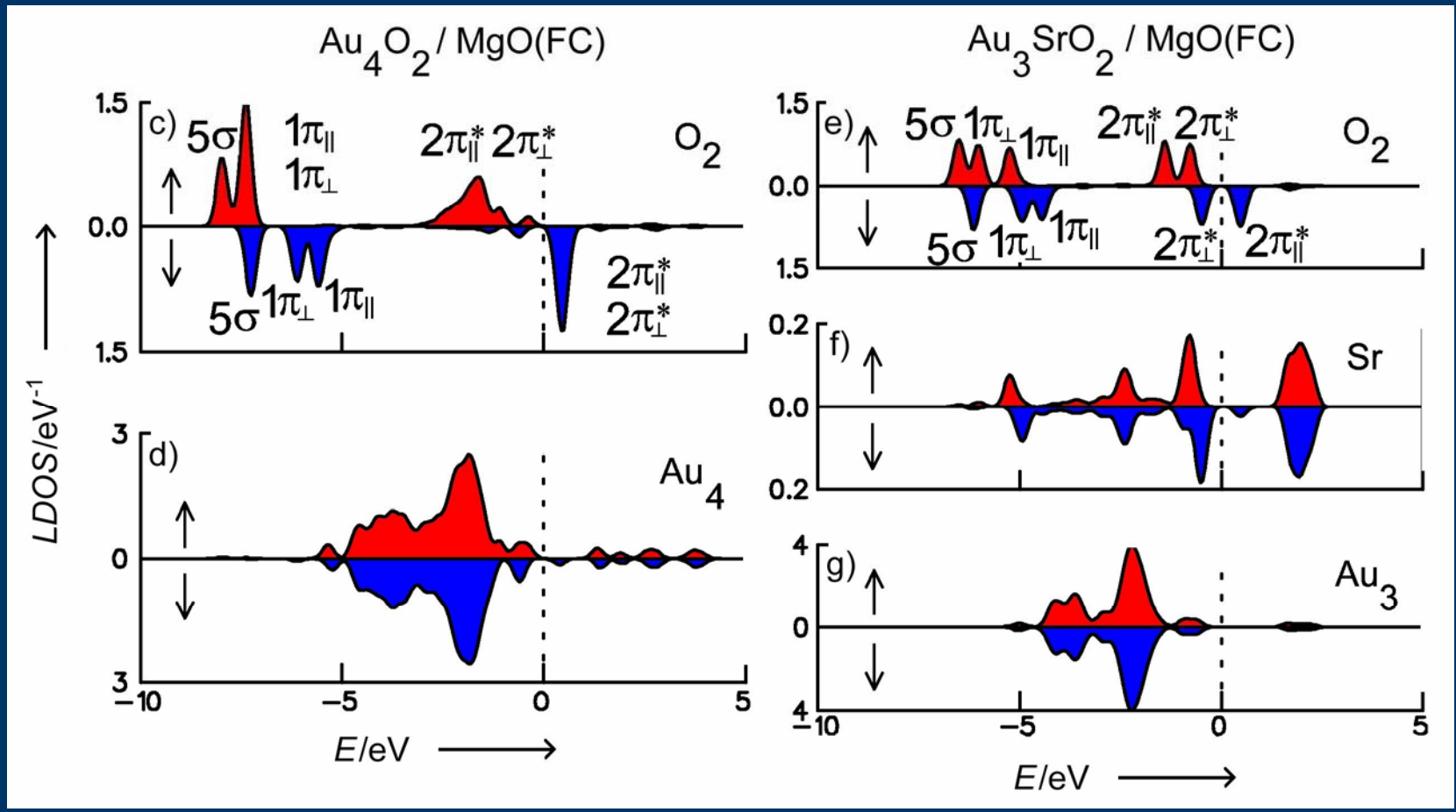


Peroxo **Molecular** **Superoxo**

$Au_8/Mg(100)/FC$: LDOS Projected on the O_2 Molecule and the Metal Part



Au_4, Au_3Sr/Mg(100)/FC: LDOS Projected on the O_2 Molecule and the Metal Part



Electronic Structure (Au_8, Au_4, Au_3Sr) Impurity Doping Effects

The Role of Moisture

Heterogeneous Catalysis

Vital Role of Moisture in the Catalytic Activity of Supported Gold Nanoparticles**

Masakazu Daté, Mitsutaka Okumura, Susumu Tsubota, and Masatake Haruta*

Why can inert gold become catalytically active only when dispersed in the form of nanoparticles?—This simple question has attracted growing interest in the field of not only catalytic and industrial chemistry,[1–4] but also cluster and theoretical science.[5–7] To answer this question, CO oxidation has been intensively studied as a model reaction.[8–14] The reaction is known to be greatly influenced by moisture in the reactant gas.[10,15] However, only a few recent studies discuss the reaction mechanisms taking water into account.[16,18] Even in these studies on the effect of moisture, for practical reasons, the addition of water vapor has been examined only at high concentrations.

The Effect of Moisture on Gold Catalysts

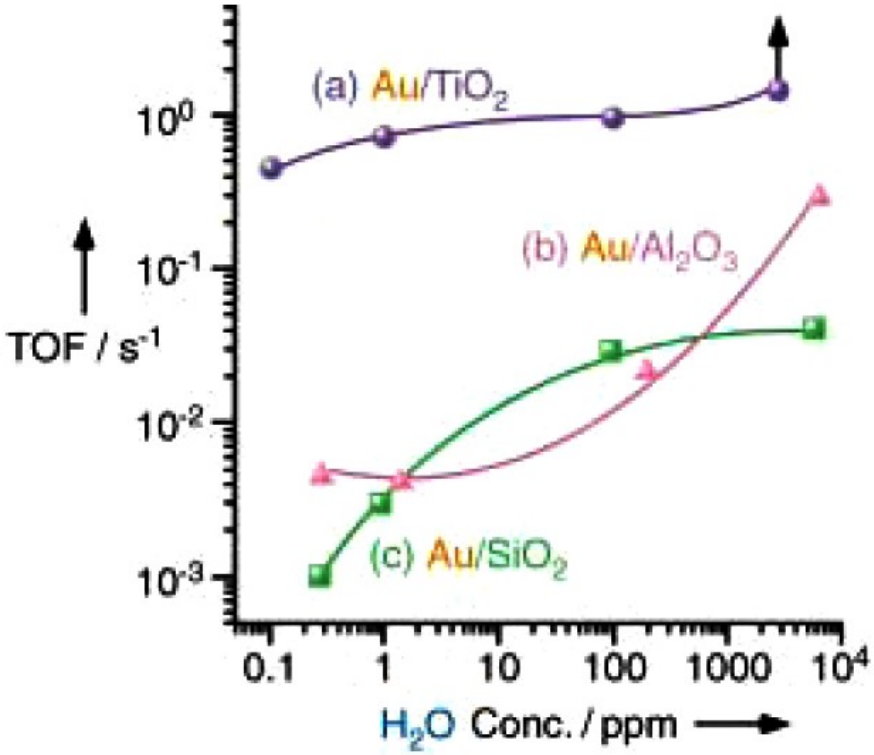
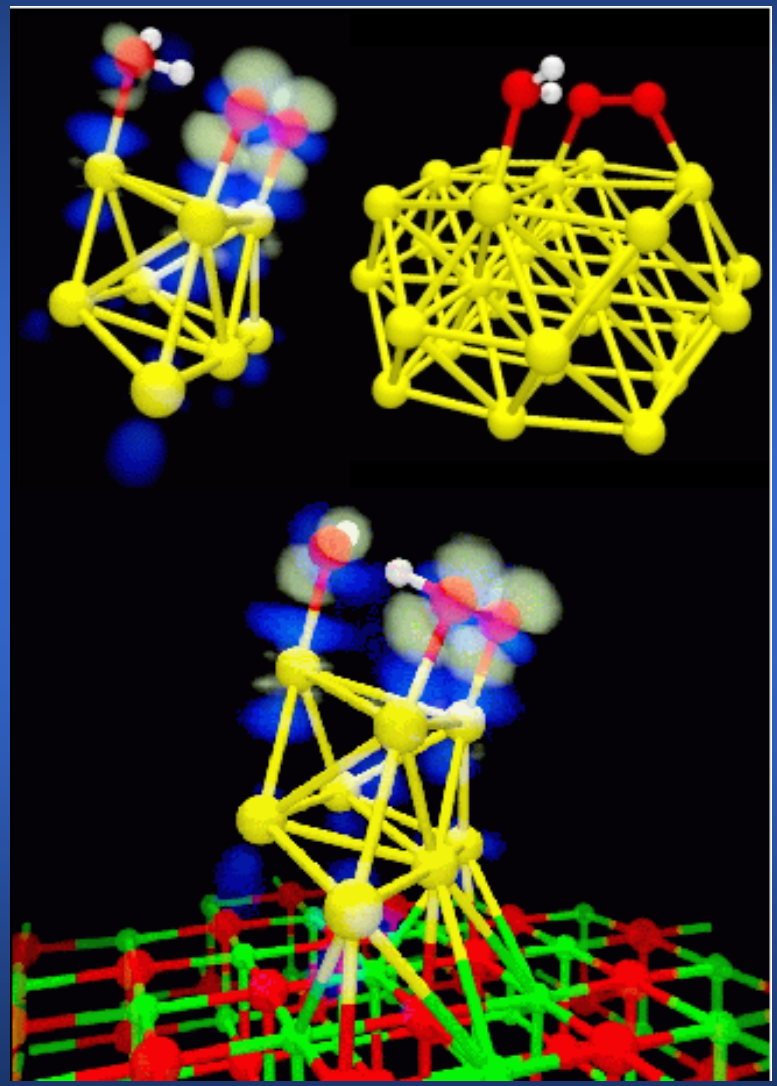


Figure 2. Turnover frequencies per surface gold atom at 273 K for CO oxidation over a) Au/TiO_2, b) Au/Al_2O_3 and c) Au/SiO_2 as a function of moisture concentration. Upright arrow indicates the saturation of CO conversion.

Mechanism Predicted by Theory



Angelo Bongiorno & Uzi Landman
Phys. Rev. Lett. 95, 1061021 (2005)

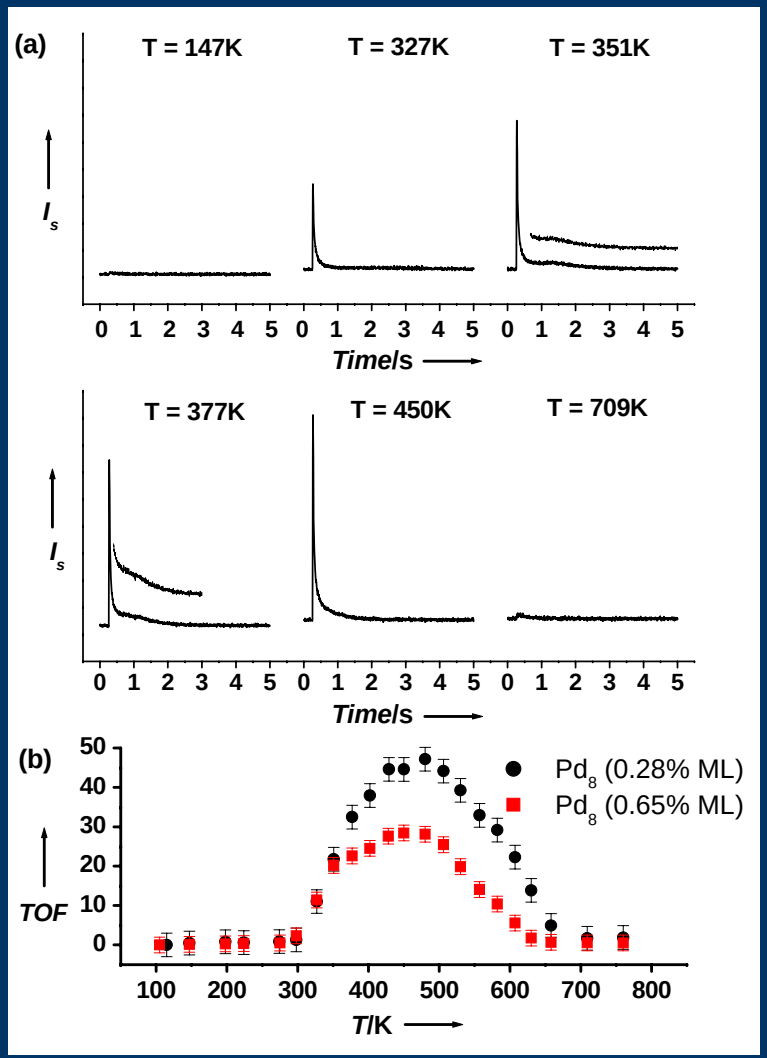
Cooperative Adsorption of H_2O and O_2

TABLE I. Energies (in eV) for the adsorption and coadsorption of O_2 and H_2O on free (Au_8 and Au_{30}) clusters and on a gold octamer supported on MgO(100), i.e. Au_8/MgO. In the case of the Au_8/MgO system, results are given for both the adsorption on the top-facet of the gold cluster cluster (-T) and at the peripheral interface of the cluster with the substrate (-P).

	O_2	H_2O	O_2-H_2O
Au_8	unbound	~0.3	0.4–0.9
Au_{30}	≤0.4	0.3–0.6	0.7–0.9
Au_8/MgO-T	≤0.1	0.2–0.3	0.5–1.2
Au_8/MgO-P	0.3–0.8	0.4–0.6	1.3–2.1

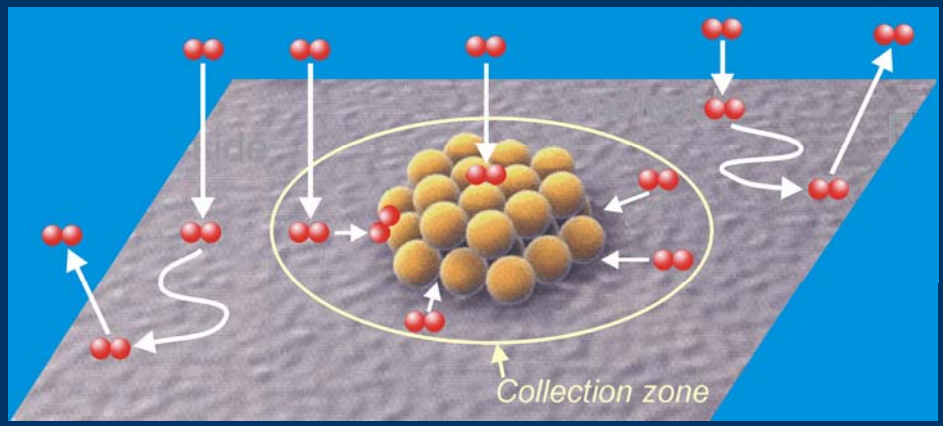
Cooperative Adsorption and Activation by Coadsorbants

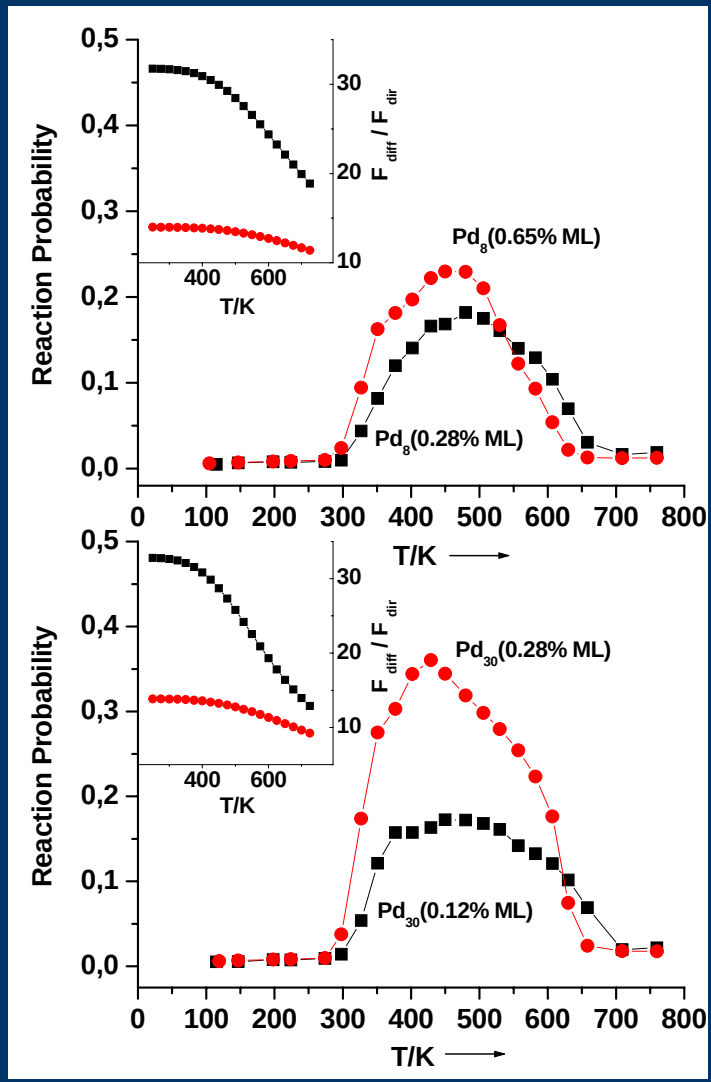
Chemical Properties in the Nonscalable Size Regime: Reverse Spill-Over



Sample	E_{LH} [kJ/mol]	Conditions	Ref
Pd(111)	105 59	Low O_2 cov., T> 500K high O_2 cov., T<420K	[18]
Pd(111)	45 – 64	High O_2 cov., T<500K	[17]
Pd [a] (27nm)	32 – 45	High O_2 cov., T<500K	[13]
Pd [b] (5-6nm)	57 ± 8 62 ± 9	CO rich regime, low T O rich regime, low T	[17]
Pd [a] (2.5nm)	19 - 20	High O_2 cov., T<500K	[13]
Pd_{30} [c]	36 ± 3	O saturated, T<400K	
Pd_8 [c]	25 ± 3	O saturated, T<400K	

Chemical Properties in the Nonscalable Size Regime: Reverse Spill-Over





Reaction probability: TOF normalized by total flux.

Low cluster coverage: High contribution of reverse spill-over (F_{diff})

Pd_8: No discrimination between F_{direct} and F_{flux} → a) identical activation energies at periphery and on cluster; b) no diffusion barrier at periphery.

Pd_{30}: F_{diff} less effective for reactivity → a) different activation barriers at periphery and on cluster; b) high diffusion barrier at periphery.

 # Effect of reverse spill-over is size dependent !

Effect of Reverse Spill-Over is Cluster Size Dependent

Nanocatalytic Factors

1. **Each cluster has its characteristic electronic structure: Intrinsic quantum size effects**

2. **Each cluster size has characteristic cluster-support interaction (stability, mobility, charging, steric effects ...)**

3. **Clusters are fluxional: Low-temperature reactivity**

4. **Effect of reverse spill-over is size dependent**

Thank You

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Post-Doctoral Position in Cluster Chemistry and Surface Science at the Technical University of Munich

A post-doctoral position is available in the group of Prof. Ueli Heiz at the Technical University of Munich in the field of Cluster Chemistry and Surface Science. In this project the size evolution of the chemical and catalytic properties of size-selected clusters at oxide surfaces will be studied by state-of-the-art surface science techniques (microcalorimetry, pulsed molecular beams, Fourier transform infrared spectroscopy, thermal desorption and reaction spectroscopy, metastable impact electron spectroscopy). There is an exceptional infrastructure available in the group to study other properties of nanostructures at surfaces as for instance with local probes or cavity ringdown spectroscopy. In addition, the Technical University of Munich offers a very active and stimulating scientific environment.

The succesful candidate is expected to have experiences in at least one of the used experimental techniques as well as in UHV technology.

The position is open in September 2006 and can be occupied for one year with an option of a second year.

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