HREELS Laboratory

Introduction

High-resolution electron energy loss spectroscopy (HREELS) is a powerful surface analytical technique which provides unique vibrational analysis of metal and semiconductor surfaces in an ultra-high vacuum environment.

EELS readily provides important information on:

- ► adsorbate vibrational frequencies
- ► molecular structure of adsorbates (decomposition, polymerization)
- ► bond strengths at surfaces
- ► adsorption geometry—surface-bonding sites
- ► surface chemistry (oxide formation, reduction, intermediates, etc.)
- overtone and combination vibrational bands
- ► surface acoustic and optical phonons.

Importantly, EELS affords higher surface sensitivity and wider spectral range than infrared spectroscopy. For example, a spectral range of 200-5000 cm⁻¹ can be scanned in a few minutes, and less than 10⁻³ monolayers of adsorbed CO may be detected. In contrast to IR spectroscopy, EELS is not limited by strict dipole selection rules, which often hinder observation of important modes and adsorbates. In EELS both long-range dipole and short-range "impact" scattering mechanisms are operable and may be effectively studied as a function of scattering angle and impact energy. For example, molecular adsorbates which exhibit weak dipole activity can be detected in the impact scattering regime.

Complementary technics as Auger electron spectroscopy (AES) and Temperature Programmed Desorption (TPD) technics are available in the same UHV system, giving the opportunity to the mapping of the adsorption/desorption processes, reaction pathways and elementary steps/intermediates of the surface catalytic reactions in the 100-1100 K temperature range.

Moreover, information obtained from HREELS ideally complements data obtained with other technics available in our research group (LEIS, XPS, STM), as well.

The experimental work could perform in a two-level UHV chamber with a routine base pressure of 5 x 10^{-10} mbar produced by a turbomolecular pump (Fig1. A). The chamber is equipped with facilities for AES, HREELS, and TPD methods. The HREEL spectrometer (LK, ELS 3000) was placed in the lower level of the chamber and has a resolution of 20–40 cm⁻¹ (FWHM). The count rates in the elastic peak were typically in the range of $1x10^4 - 1x10^5$ counts-per-second (cps). Spectrum could be recorded with a primary beam energy of 6.5 eV and at an incident angle of 60° with respect to the surface normal in the specular direction. However, investigation of surface intermediates is possible also in off-specular geometry.



Fig. 1. The HREELS-AES-TPD ultra-vacuum system (a), and the Rh(111) sample (b).

| Methods | High Resolution Electron Energy Loss Spectroscopy (HREELS) Auger Electron Spectroscopy (AES) Mass spectrometry (Temperature Programmed Desorption measurements) |
|---------------------------------|---|
| Samples and preparation methods | Rh(111) single crystal (or other single crystal surfaces e.g.: Au(111), Mo₂C/Mo(100), Mo(100), TiO₂(110) Metal evaporation by PVD technic 2D monolayer preparation by CVD (e.g.: boron nitride or graphene layers) |
| Capabilities | Ar ion gun for sputtering of surface (up to 5kV) Sample heating and cooling (100K – 1100 K) Adsorption of gas molecules from UHV gas dosing system (through a capillary). Substance could be gas, liquid or solid (depending on the vacuum pressure of the solid material). |

Specifications

Sample examples



Fig 2. Effect of annealing on the HREEL spectra after adsorption of 4 L ethanol at 170 K on the clean Rh(111) surface (a) and HREELS spectra obtained at different angles of incidence after the exposure of h-BN/Rh(111) to 100 L of azobenzene at 300 K (b).

Fig 2. (a).

After adsorption of ethanol on clean Rh(111) surface we observed several characteristic vibrations which very likely belong to η^2 -(O,C)–CH₃CHO at 670, 1105, 1195, 1340, 1410, 2890 and 2980 cm⁻¹. Another remarkable finding at room temperature (RT) is that the clean rhodium surface acts as a good C-C breaking catalyst. We can recognize the clear sign of this, as the adsorbed CO appears at 2040 cm⁻¹ at 300 K.

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Fig 2. (b).

We adsorbed azobenzene at 300 K on 2D h-BN nanomesh covered Rh(111) surface and we collected the HREELS results at different angle of incidence (off-specular geometry). The presented angle-resolved measurements confirm that the adsorption geometry of azobenzene is predominantly parallel to the h-BN monolayer.

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Contact

If your research interests require access to this HREELS system and have any questions on the instrument capabilities, please contact Professor Zoltán Kónya (konya@chem.u-szeged) or the technical contact Arnold P. Farkas (arnold.farkas@eli-alps.hu).Research application submission can be done through the online form.