Physics of Nanotechnologies

Lecturer Muhammad Amer Mustafa

UOS Sub Campus Bhakkar

Low-energy electron diffraction

Low-Energy electron diffraction (**LEED**) is a technique for the determination of the surface structure of single-crystalline materials by bombardment with a collimated beam of low energy electrons (20–200 eV) and observation of diffracted electrons as spots on a fluorescent screen.

LEED may be used in one of two ways:

- Qualitatively, where the diffraction pattern is recorded and analysis of the spot positions gives information on the symmetry of the surface structure. In the presence of an adsorbate the qualitative analysis may reveal information about the size and rotational alignment of the adsorbate unit cell with respect to the substrate unit cell.
- Quantitatively, where the intensities of diffracted beams are recorded as a function of incident electron beam energy to generate the so-called I-V curves. By comparison with theoretical curves, these may provide accurate information on atomic positions on the surface at hand.

Development of LEED as a tool in surface science

- Though discovered in 1927, low energy electron diffraction did not become a popular tool for surface analysis until the early 1960s. The main reasons were that monitoring directions and intensities of diffracted beams was a difficult experimental process due to inadequate vacuum techniques and slow detection methods such as a Faraday cup.
- Also, since LEED is a surface sensitive method, it required well-ordered surface structures. Techniques for the preparation of clean metal surfaces first became available much later.
- Nonetheless, H. E. Farnsworth and coworkers at Brown University pioneered the use of LEED as a method for characterizing the absorption of gases onto clean metal surfaces and the associated regular adsorption phases, starting shortly after the Davisson and Germer discovery into the 1970s.

- In the early 1960s LEED experienced a renaissance, as ultra high vacuum became widely available and the post acceleration detection method was introduced by none less than Germer and his coworkers at Bell Labs using a flat phosphor screen. Using this technique diffracted electrons were accelerated to high energies to produce clear and visible diffraction patterns on a fluorescent screen. Ironically the post-acceleration method had already been proposed by Ehrenberg in 1934.
- In 1962 Lander and colleagues introduced the modern hemispherical screen with associated hemispherical grids. In the mid sixties, modern LEED systems became commercially available as part of the ultra-high vacuum instrumentation suite by Varian Associates and triggered an enormous boost of activities in surface science. Notably future Nobel prize winner Gerhard Ertl started his studies of surface chemistry and catalysis on such a Varian system.[7]

- It soon became clear that the kinematic (single scattering) theory, which had been successfully used to explain X-ray diffraction experiments, was inadequate for the quantitative interpretation of experimental data obtained from LEED.
- At this stage a detailed determination of surface structures, including adsorption sites, bond angles and bond lengths was not possible.
- A dynamical electron diffraction theory which took into account the possibility of multiple scattering was established in the late 1960s. With this theory it later became possible to reproduce experimental data with high precision.

Experimental Setup

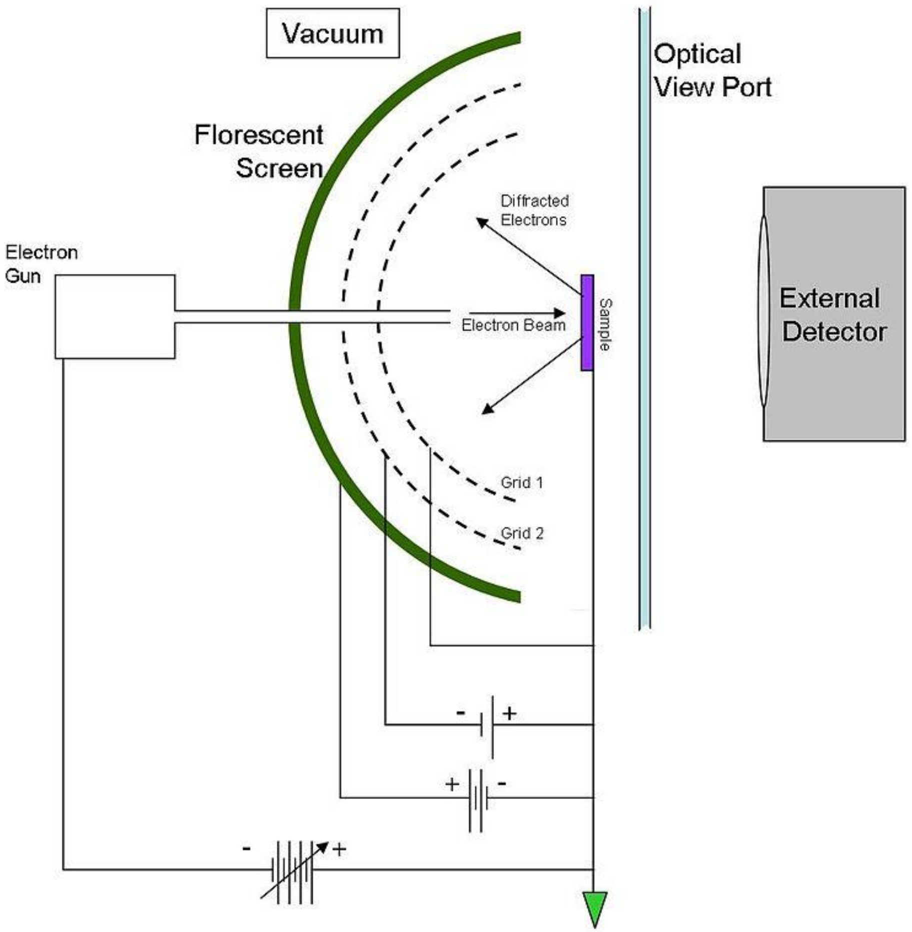

Figure 2 Diagram of a LEED optics apparatus.

- In order to keep the studied sample clean and free from unwanted adsorbates, LEED experiments are performed in an ultra-high-vacuum environment (10^{-9} mbar).
- The most important elements in a LEED experiment are:[2]
- A sample holder with the prepared sample
- An electron gun
- A display system, usually a hemispherical fluorescent screen on which the diffraction pattern can be observed directly
- A sputtering gun for cleaning the surface
- An Auger electron spectroscopy system in order to determine the purity of the surface.
- A simplified sketch of an LEED setup is shown in figure 2

Sample preparation

The sample is usually prepared outside the vacuum chamber by cutting a slice of around 1 mm in thickness and 1 cm in diameter along the desired crystallographic axis. The correct alignment of the crystal can be achieved with the help of x-ray methods and should be within 1° of the desired angle.[9] After being mounted in the UHV chamber the sample is chemically cleaned and flattened. Unwanted surface contaminants are removed by ion sputtering or by chemical processes such as oxidation and reduction cycles. The surface is flattened by annealing at high temperatures. Once a clean and well-defined surface is prepared, monolayers can be adsorbed on the surface by exposing it to a gas consisting of the desired adsorbate atoms or molecules.

Sample preparation

Often the annealing process will let bulk impurities diffuse to the surface and therefore give rise to a re-contamination after each cleaning cycle. The problem is that impurities which adsorb without changing the basic symmetry of the surface, cannot easily be identified in the diffraction pattern. Therefore, in many LEED experiments Auger electron spectroscopy is used to accurately determine the purity of the sample

Electron gun

In the electron gun, monochromatic electrons are emitted by a cathode filament which is at a negative potential, typically 10-600 V, with respect to the sample. The electrons are accelerated and focused into a beam, typically about 0.1 to 0.5 mm wide, by a series of electrodes serving as electron lenses. Some of the electrons incident on the sample surface are backscattered elastically, and diffraction can be detected if sufficient order exists on the surface. This typically requires a region of single crystal surface as wide as the electron beam, although sometimes polycrystalline surfaces such as highly oriented pyrolytic graphite (HOPG) are sufficient.

Detector system

A LEED detector usually contains three or four hemispherical concentric grids and a phosphor screen or other position-sensitive detector. The grids are used for screening out the inelastically scattered electrons. Most new LEED systems use a reverse view scheme, which has a minimized electron gun, and the pattern is viewed from behind through a transmission screen and a viewport. Recently, a new digitized position sensitive detector called a delay-line detector with better dynamic range and resolution has been developed.