Physics 441/2: Transmission Electron Microscope

Introduction

In this experiment we will explore the use of transmission electron microscopy (TEM) to take us into the world of ultrasmall structures. This is the regime between 1000 Å and atomic dimensions in which the continuing miniaturization of integrated electronics is being pursued. It is also a very important region for structural biology. These length scales are far below the limit where the resolution of conventional optical microscopy becomes dominated by the wavelength of visible light (~5000 Å). The transmission electron microscope (or TEM), first invented in the late 1930's, has now developed into the technique of choice for microstructral studies in a wide range of fields: materials research, biophysics, polymer science, mineralogy, and health sciences, to name a few.

In the first part of the experiment, you will get a feel for the capabilities and immense resolving power of TEM by imaging some samples of DNA. We will measure the diameter and pitch of DNA's famous double-helix structure. In the second part of the experiment, we will use TEM to study some "quantum well" structures made from ultrathin layers of Silicon and alloyed Silicon-germanium. We will determine the point spread function of the Philips 420 electron microscope and measure, at the highest magnification, the width of a quantum well and the abruptness of its boundaries. These are key quantities which determine the spectrum of electronic energy levels of the quantum well. The Si/Si-Ge samples will also demonstrate the capability of TEM to obtain selected-area diffraction patterns, from which detailed structural information can be obtained at atomic dimensions.

Basic Principles

The uncertainty principle, $\Delta p.\Delta x = h$, sets a fundamental limit on the spatial resolution, Δx, that can be obtained by probing a system with a beam of particles having de Broglie wavelength λ (= $h/\Delta p$). Thus $\Delta x \cong O(\lambda)$. In this way, the resolving power of an optical microscope is limited by the wavelength of the light employed: ($\lambda > 3000$Å). In the **electron microscope,** much shorter de Broglie wavelengths are possible by using highly energetic electrons, thereby pushing the resolution limit down to the Å level.

Since they are charged, electron beams can be deflected by electrostatic or magnetic fields. In virtually all commercial electron microscopes nowadays, **magnetic lenses** are used to focus the electron beam carrying out the functions that glass
lenses serve in a conventional optical microscope. The Philips 420 was one of the first high-resolution ($\Delta x < 5$Å) microscopes on the market. Although this instrument was built

in the early eighties, the designs of the column and the magnetic lenses remain state-of-the-art.

The principle of operation is entirely analogous to the optical microscope. A collimated beam of electrons, emitted from a hot LaB_6 filament, is accelerated to ~120 keV. After passing through a **condenser lens** the beam is incident on the sample. The size of the beam can be varied, but is typically ~ 1 micron in diameter. Thus small selected areas of the sample can be probed. The beam passes through the sample (which is thinned to ~ 200 Å to permit **transmission** of electrons in the 100 keV range) An **objective lens**, situated immediately below the sample**,** then produces a magnified image of the sample. Finally, this image is projected onto a **fluorescent screen** at the base of the column.

One of the powerful features of electron microscopes is their ability to display diffraction patterns of the sample. In this case the wave-like nature of the electrons is utilized to diffract the incident beam from the atomic structure within the sample. This can provide information on the crystal structure of the sample and is particularly useful when the atomic arrangement is regular and periodic, as in a crystal. It is very straightforward to switch from a real image to the diffraction pattern: In **diffraction mode** the current to the objective lens is turned off so that the diffraction pattern of the sample is simply projected onto the fluorescent screen.

Imaging a "superlattice"

Introduction - The "superlattice" sample we're going to investigate is an example of an artificial crystal structure made by depositing alternating layers of two different (but chemically related) materials on the flat surface of a semiconductor wafer. In this case the two materials are Silicon and an alloy of Si and Ge, $Si_{0.8}Ge_{0.2}$. The confinement of electrons within one of these layers is referred to as a "quantum well". The deposition process, carried out in an ultrahigh vacuum chamber ($p < 10^{-10}$ Torr), is called "Molecular Beam Epitaxy" (MBE). MBE is one of the most important and widely used fabrication techniques in modern electronics technology; it is capable of producing devices with precisely defined layer thicknesses of less than 10nm! For example, most laser diodes used in DVD players are made by MBE. Another important example is the sample used for the discovery of the fractional Quantum Hall Effect, which was recognized by the 1998 Nobel Prize for physics.

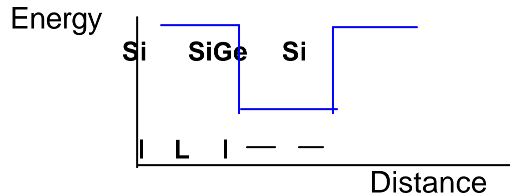
TEM measurements - In this experiment you will use Transmission Electron Microscopy (TEM) techniques to investigate the microscopic structure of a superlattice, including measurements of key structural aspects such as:

- individual superlattice layer thicknesses
- sharpness of interfaces between the different layers
- crystallographic orientation of sample
- interatomic distances within each layer

Each of these parameters is very important to the physics of real electronic devices based on superlattice materials. For example, in the example given above, energy

levels are established by forming a “quantum well” by means of the discontinuities in electronic potential that exist at the interface between the two different materials:

Fig. 1: Si -SiGe Quantum Well



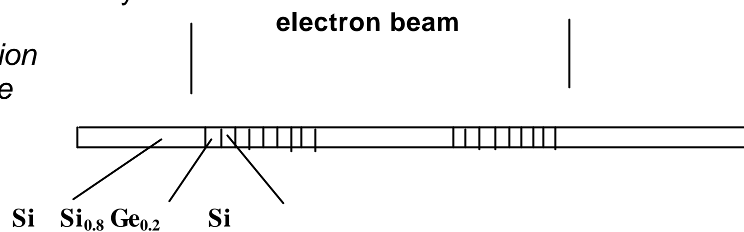
Clearly, it is important to minimize roughness, and smearing of the potential discontinuity, if the quantum well is to function as desired. Also, it is important that the width, L, is precisely defined.

Procedure - The power of TEM lies in its ability to provide direct, high-resolution, images of microscopic structures as well as “selected area” diffraction patterns of the *same region* that is being imaged. In this way one can routinely obtain both a ‘picture’ of the sample (which is really an electron absorption contrast map in a bright field image) as well as information about the crystallographic arrangement of the atoms. All of this information is obtained from the region probed by the electron beam (typically 1μm across, in this microscope).

In this part of the experiment you will try to obtain *as sharp an image as possible*, limited by the resolution of the microscope, and by the intrinsic chemical interdiffusion between Si and SiGe layers. With the microscope set in the “M” mode (button on right of front panel), set the magnification between 200,000x and 600,000x, and record (using the CCD camera) several images of the superlattice at different microscope focus settings to determine the optimal focus condition. Make a note of the magnification for each picture.

Note that the superlattice sample is prepared as a thin cross-section (in order to permit transmission of the electron beam, the thickness cannot be more than ~30nm) so that the beam is *parallel* to the layers:

Fig. 2: Cross-section TEM sample



At low magnification (~3000x!) you will be able to see how the sample was prepared. First, the thin-film stack was sliced vertically, and then the two resulting halves were glued together (face-to face):

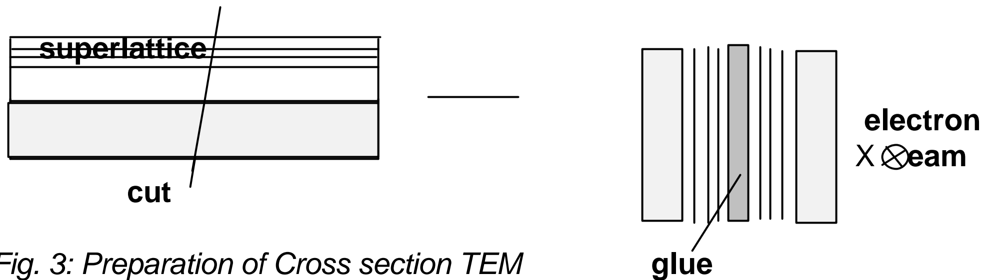
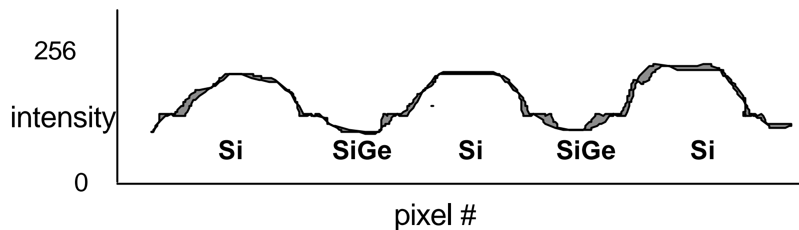


Fig. 3: Preparation of Cross section TEM Sample

After you are satisfied that have recorded the sharpest image possible, press the "D" button (right hand side of panel) to go into diffraction mode. Adjust the spot size knob to bring the diffraction spots into focus. Record a diffraction pattern of the area you are imaging, noting the "camera length" reading on the from panel.

Analysis - (Direct image). From the magnification value, and a scale factor to take account of the CCD camera magnification (which you can calibrate using the 4cm diameter circle etched onto the TEM screen), calculate the thickness of the Si and SiGe layers in Å. Now measure the sharpness of the interfaces between the Si and SiGe layers. There are various ways to do this. One of the neatest is as follows: make a line profile across the layers using the KSA image analysis software. It should look something like this:



You can make all distance measurements in pixels initially, and then convert them to Å later, using a scale conversion factor. Now with this profile window open on the screen, perform a Fast Fourier Transform (FFT) of the profile intensity using the FFT tool in the KSA software (top tool bar). The FFT gives you the Fourier coefficients, c_k, of the sinusoidal frequencies that, when added together will reproduce the observed profile, f (r):

$$f(r) = \int c_k e^{ikr} dk \qquad (1)$$

where k is the spatial frequency (wavenumber).

The *high frequency* values of k are the ones that are important in defining the sharpness of the profile (the rapidly varying part). Therefore by examining the large-k region of the profile's FFT, one can evaluate how sharp the interface between the layers is.

Quantitatively, there are various ways to do this, including fitting an exponential envelope to c_k vs. k, and finding the 1/e point. The inverse of k at this point would be a measure of the sharpness of the interface. Another measure would be to determine the maximum k at which there is a significant value of c_k (i.e. significantly above the noise signal). This approach is the basis of the Modulation Transfer Function, or MTF, a curve which is often used to characterize the spatial resolution of imaging instruments. Note: in order to determine the resolution of the microscope itself, take a line profile of a sharply focused edge of the sample at the high magnification (~500,000), and perform a line profile.

Analysis of Diffraction - use Bragg's Law, $\lambda = 2d \sin \theta$, to estimate the interatomic spacing, d, within the Si or SiGe layers. Here λ is the electron wavelength at the accelerating potential of the microscope (read off from the front panel), and θ is the deflection angle between the incident and diffracted electron beam for a particular diffraction spot.

In order to interpret the value of d that you obtain, in terms of interatomic spacing, you will also need to determine the crystallographic orientation of the sample relative to the incoming electron beam. Is the orientation (100), (110) or (111)? You can determine this from the symmetry of the diffraction pattern. If the (100) axis is parallel to the beam, then the diffraction pattern should have 4-fold rotation symmetry (repeats every 90^0). If (111) orientation, then should have 3-fold symmetry (triangular diffraction pattern). Another hint: the orientation of the Si wafer on which the sample was grown is (100).

Other experiments (Optional) - try to record the diffraction pattern in the exact region where the superlattice is. This requires very careful placement of the smallest selected area diffraction aperture (~100nm in diameter). Why do the diffraction spots in this region appear to have such a peculiar shape?

Questions:

1. Calculate the de Broglie wavelength of the electrons at the operating potential of the microscope.

2. Estimate the wavelength spread, and therefore the monochromaticity of the electron beam, given that the electrons are emitted from a filament at 1000K. Assume a Boltzmann distribution of energies.

3. Calculate the width, L, of a quantum well which will emit visible light (λ_{light}~5000Å) between the first excited state (n=1) and the ground state (n=0) of the electrons confined within the quantum well. Hint: the electronic energy levels of a quantum well are given by $E_n = h^2 k_n^2/2m$, where $k_n = 2\pi n/L$.