Chapter 3 Experimental Procedure and Sample Preparation

3.1 Experimental

3.1.1 Differential Interferometer Setup

The optical setup used for measurement of interferometric fringes is shown in figure 3.1. A 5mW maximum output at 632.8nm Helium-Neon laser was used for the laser source. The laser beam first passed through an isolater and then a polarizer. The polarized beam passed through the first beam splitter (50/50) and seperated into two portions. One portion of the beam (the reference beam), is modulated by passing through a Bragg cell which will be discussed later at 80MHz. The phase-modulated beam is steered by the second mirror into the detector. The second portion of the beam (the sample beam) is transmitted through the second beam splitter and then steered by the galvano mirror to the sample. The galvano mirror oscillates at a frequency ω_g perpendicular to the main beam on the sample at same frequency. For film thickness measurement ω_g=3932Hz and fore electric field effect measurement ω_g=0. The reflected beam from the rocking mirror passes through a 5X objective and focuses on the reflective sample surface. The focus of the objective lens can be determined by moving it up and down and find the smallest and also the brightest image of the laser beam on the sample surface. The reflected beam from the sample reflects back into the 5X objective and retraces its path to the detector. The signal from the detector is amplified by a preamplifier and analyzed for its power spectrum. The signal is analyzed using an Agilent E4401B spectrum analyzer. Agilent E4401B spectrum analyzer is connected to a computer for data acquisition. A microscope was setup with a digital camera.

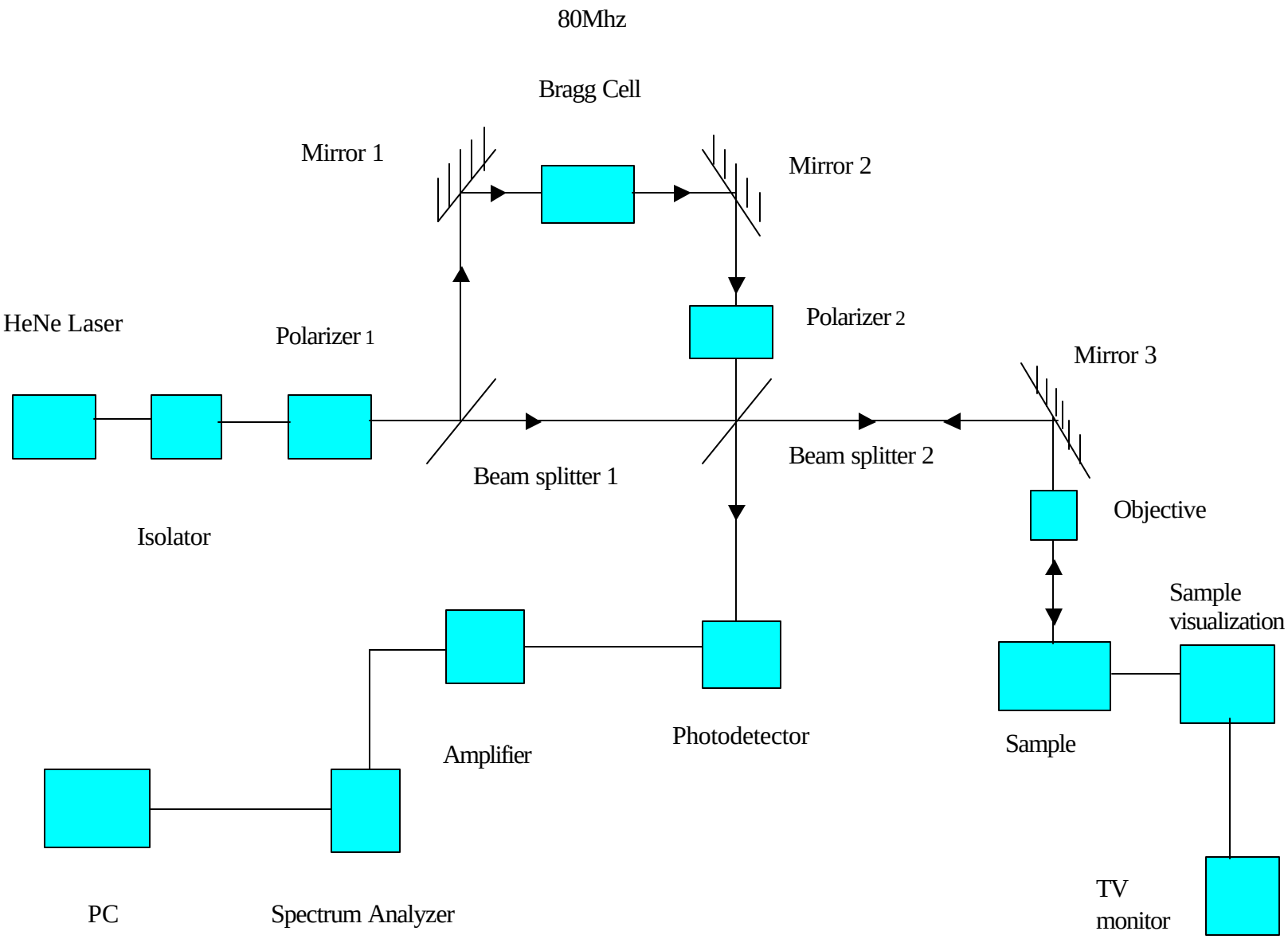

Figure 3.1 Differential Interferometer Setup

Heterodyne interferometer operates either with two frequencies lasers or with single-band frequency modulators to produce traveling interference pattern. With two frequencies lasers, the test medium and all the optical elements are slightly different from each other, which brings some limitation to the measuring system. In this interferometer, the reference beam and sample beam share the same laser source. The reference beam is modulated by Bragg cell, which has much higher modulating frequency and interaction length compared to Raman-Nath modulator. Raman-Nath modulator is another type of very commonly used modulators in heterodyne interferometery which was discussed in chapter 2. This special design permits to diminish the role of low-frequency noise and greatly enhances the signal-to-noise ratio. The resolution of this interferometer is about 0.05nm. The distance maybe changes due to the film structure or external stimuli.

Due to the complexity of the system, it requires careful alignment. For instance, the laser beam has two diffracted order beam after passing through the Bragg cell. Special attention has to be paid to find the exact Bragg angle of the incident laser beam into the Bragg cell by adjusting the right amount of power to maximize the 1^{st} order beam intensity. Proper care is also required to make the sample and reference beam congruent, i.e. parallel and coincident, for optimum interference signal.

Another critical alignment is the objective lens. In order for sample beam to trace its path to the photodetector and interfere with the reference beam, the sample beam has to pass through the center of the objective and also focus precisely on the reflecting sample surface. Both the objective holder and the galvano mirror are attached to three dimensional translation stages. The three dimensional freedom for both the galvano mirror and the objective allows the alignment of the laser beam at the rotational center of the galvano mirror and the center of the objective.

Adjusting the sample-to-objective lens distance is critical. Finding the right focal plane for the sample is an iterative process. There are two focal planes for the objective with the total distance between the laser spot at the center of the galvano mirror and the sample. The focal farther away from the sample surface is unstable, whereas the one near the sample surface gives stable signal. To define stable signal, we have to use a flat silicon wafer as the sample. For perfect flat sample, the laser beam travels at the same distance from the galvano mirror to the sample which is at the focal plane of the objective

lens as discussed above. The phase of the sample beam does not change, thus there should be no modulation of the reference beam. However, the sample holder and sample itself cannot be perfectly flat. There will always be some modulation about 80MHz (the reference beam modulating frequency) leading to sidebands observed at the spectrum analyzer. If the sidebands are very small, we call the signal is stable; otherwise, we call the signal unstable. Choose the right focal plane is very critical for the system to have stabilized signal.

The laser spot is about 7μm after passing through 5X objective. The gold line samples used to probe electric field effect are at 10μm pitch. Polymer films were deposited on and between gold lines by spin-casting process. In order to investigate the effect of electric field on the polymer film, the laser is focused on the film in between two gold lines. Such small dimensions for both the laser spot and the films require high-resolution visualization system. A high-resolution microscope shown in figure 3.1 is installed for visualization.

The systems shown in figure 3.1 can be used to measure both film thickness with galvano mirror rocking frequency of ω_g =3932Hz. When galvano mirror is stationary (i.e. ω_g =0), the system can be used to measure the changes of the film under external field. Furthermore, if the two polarizers in figure 3.1 were removed and galvano mirror rocking frequency were set at ω_g =0, the system can be used to measure directly the film thickness change under external field. With polarizers and ω_g =0, the dynamic birefringence due to E-field can be also probed.

Bragg cell provides the phase shifting in this interferometer. The principle of Bragg cell is illustrated in figure 3.4. A Bragg cell is an acousto-optic modulator. The device consists of a glass block with a piezoelectric transducer bonded to it. When the transducer is excited at a frequency $\nu_{M,}$ it sets up acoustic pressure waves that propagate through the glass block, causing a periodic variation (wavelength Λ) in its refractive index. A laser beam (wavelength λ) incident at the Bragg angle θ_B (where $\Lambda\sin\theta_B=\lambda$) on this moving phase grating is diffracted with a frequency shift $\nu_{M.}$ The Bragg modulator beams either of one diffraction order or of two diffracted orders depending on the accuracy of adjustment and on the power applied to the modulator. The modulator can

shift the laser frequency by 80MHz at a low electric power input of up to 2 or 3W. The frequency difference between the diffracted reference beam and the undiffracted sample beam is equal to the modulation frequency.

3.1.2 AC Electrical Field applying device

The gold electrode sample with spin-cast polymer film is placed under the beam. The gold electrodes applying device is adjusted to firmly contact the gold pad on the sample. Apply an AC field to the sample as shown in figure 3.5 with $\omega_g = 0$. The changes in the film under electric field can be probed.

3.1.3 AFM thickness measurement

AFM (Atomic Force Microscopy) MMAFM-2 made by Digital Instruments is used to measure film thickness. MMAFM-2 can be operated at both Tapping mode and contact mode. Tapping mode is used to measure film thickness in this study because tapping of soft surfaces by a fast oscillating probe prevents damage by virtually eliminating the lateral forces inherent to contact mode AFM. In tapping mode, the tip-sample contact area is minimal, providing the best resolution (a few nanometers lateral and sub nanometer vertical) in imaging of non-periodic topographical features. Height images recorded in tapping mode also most accurately reproduce the true topography of soft samples.[1] For examining polymer materials, tapping mode offers the ability to perform a high-resolution profiling of surface morphology and nanostructures.[1]

3.1.4 Thin film thickness measurement

To calibrate interferometer thickness measurement, the measurement results were compared with AFM thickness measurement. Thin films of PS were spin-cast on half of the silicon wafer as shown in figure 3.2. The laser beam focused at the edge of 0.9% PS film is shown in figure 3.3. By focusing the laser on the edge of thin film, two first order sidebands appeared due to the phase change of the thin film. According to the theory derived in section 3.2, film thickness can be calculated from the ratio of first order sidebands to versus carrier. The following figure 3.2 shows spin-cast PS thin from 0.9% polystyrene (PS) solution in toluene at 3000rpm (rotations per minute). Figure 3.3 shows

the laser beam focusing at the edges of above PS thin film. The interferometer measurement of the 0.9% PS film thickness is 30nm, and the AFM thickness measurement gives about 30-35nm.

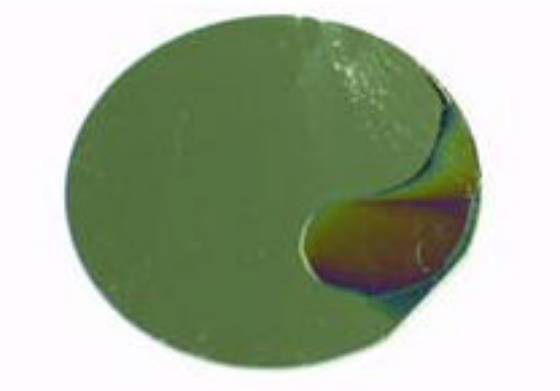

Figure 3.2 0.9% Polystyrene (PS) Film Spin-cast on Silicon Wafer Sample

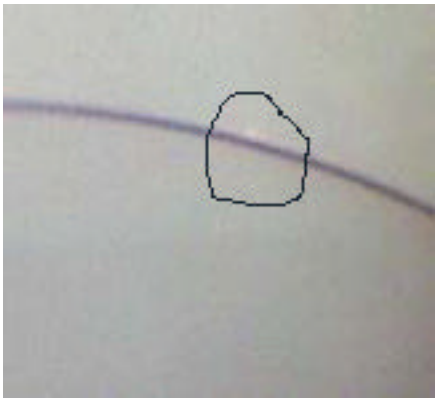

Figure 3.3 Laser Beam Focused on 0.9% PS Film Edge

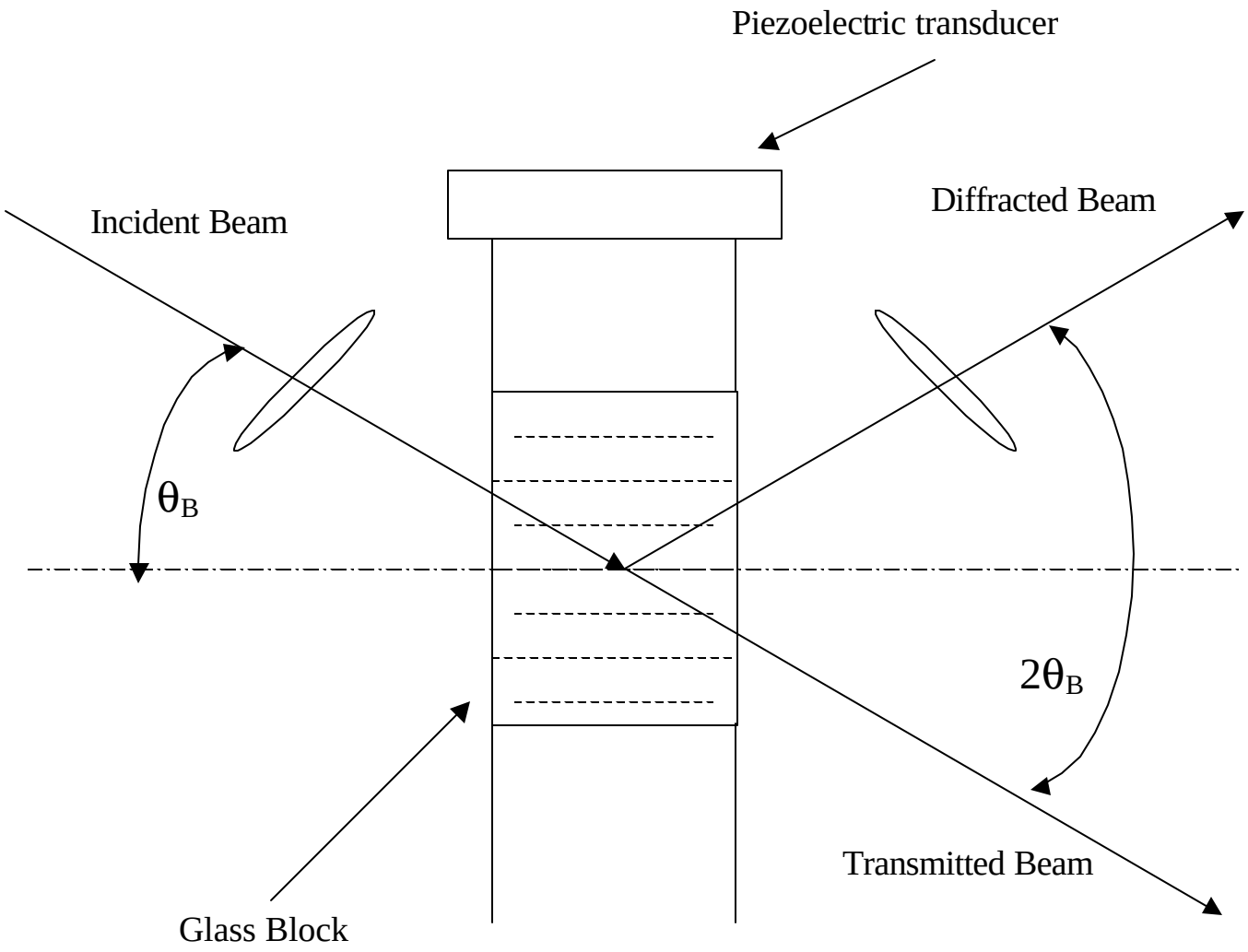

Figure 3.4 The Bragg Cell

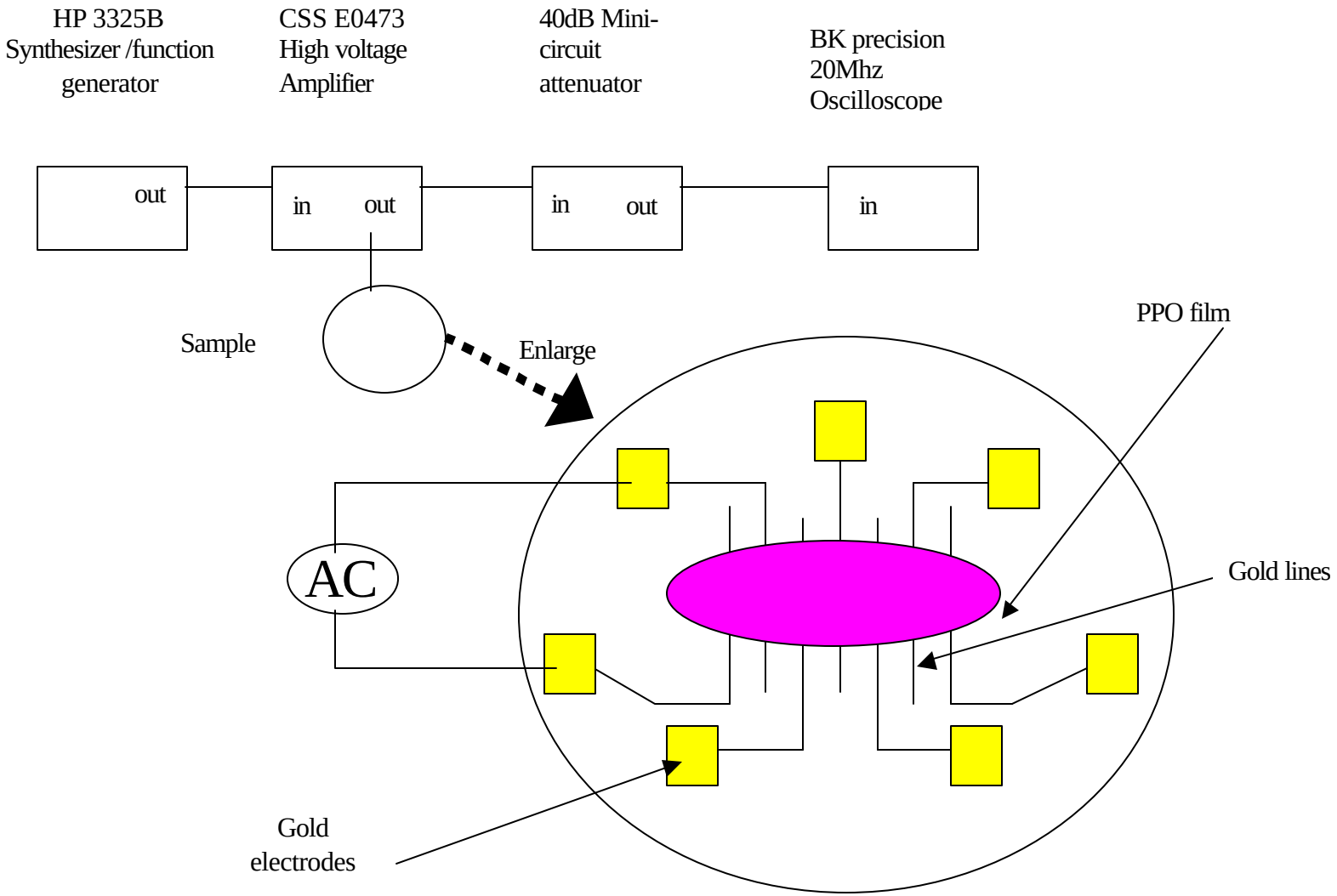

Figure 3.5 Apply AC Field to Polymer Film

3.2 Materials and Sample Preparation

3.2.1 Spin-casting Film Sample preparation

Sample preparation procedure is shown in figure 3.6. The silicon wafer was immersed in ethanol solvent for 24 hours and then followed by air-drying. Subsequently, the wafer was exposed into a piranha solution (in the volume ratio of H_2SO_4: H_2O_2=3:1) for 30 seconds. The piranha solution was constantly stirred to break air bubbles and let fresh O_2 react with silicon wafer and then air-dry the wafer. The clean wafer was dipped into deionized water and tested if the wafer is hydrophilic or polar. If the wafer is polar, the water spreads very nicely on the wafer and air-dry the wafer again; otherwise, the water will de-wet and form droplets on the wafer. In this case, immerse the wafer into the piranha solution and repeat the above procedure until the wafer finally becomes polar. To treat the wafer into hydrophobic, expose the clean hydrophilic wafer into 0.1% HF solution in deionized water for 10 seconds and then air-dry the wafer and dip the wafer into deionized wafer to test if the wafer is hydrophobic. If the wafer is nonpolar, the water will dewet from the wafer. If so, air-dry the nonpolar wafer. Otherwise, immerse the wafer back into the HF solution until the wafer becomes nonpolar and then air-dry the wafer.

The materials in different concentrations were used for study. 2%, 3%, 4% PPO 2000, 2%, 3%PPO 8000 solution in ethanol, 0.9%, 2% Polystyrene (PS) in toluene. For polar polymer PPO, the wafer is treated to obtain hydrophilic surface. For nonpolar polymer PS, the wafer is treated to obtain hydrophobic surface. To spin-cast polymer film, the vacuum connected to the spinner sample holder has to be turned on. Then place the treated wafer on top of spinner sample holder. A few drops of polymer solution were placed onto the wafer and then were allowed to spread before the spinner was turned on. The power and vacuum were turned off until the film thickness is uniform. The whole procedure is shown in figure 3.6.

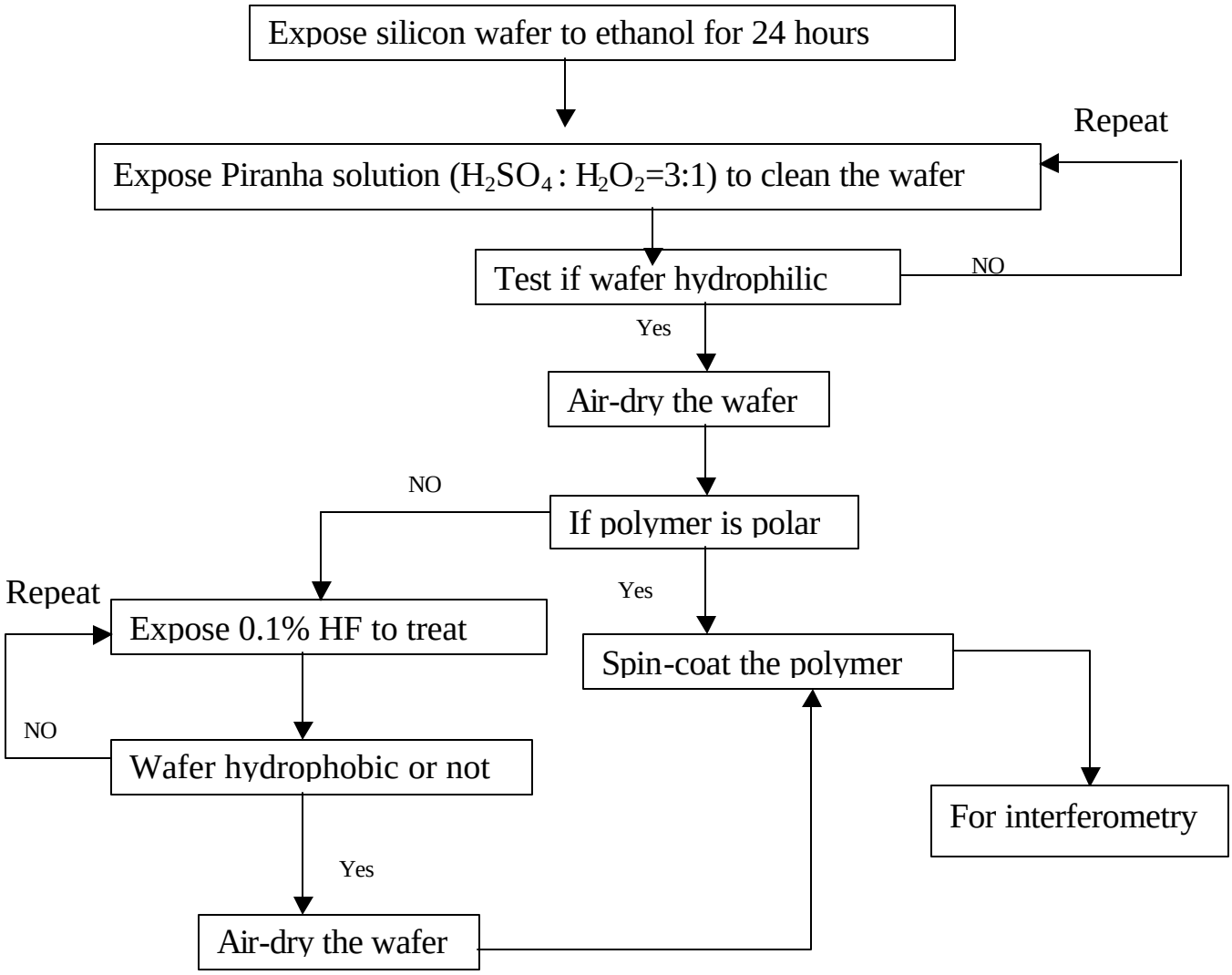

Figure 3.6 Flow chart of sample preparation

3.2.2 Gold electrode sample preparation

The gold electrode samples were made by lithography method by IBM. The basic fabrication sequence for ultra deposition process is shown in Figure 3.7. Followed by cleaning, dehydration baking, the wafers are coated with photo-resist. Spin coating is used to apply photo-resist films. The photo-resist (PR) is subsequently exposed through a

mask. The mask contains clear and opaque features that define the pattern to be created in the PR layer. The areas in the PR exposed to the light are made either soluble or insoluble in a specific solvent known as a developer. In the case of when the irradiated regions are soluble, a positive image of the mask is produced in the resist. Such material is therefore termed a positive resist. If the developer dissolves the non-irradiated regions, a negative image results. The resist in this case is called a negative resist. Following development, regions of SiO_2 no longer covered by resist are removed by etching, thereby replicating the mask pattern in the oxide layer and finally all the residue are cleaned.[2] The resultant gold electrode lines on Si sample are shown in figure 3.9.

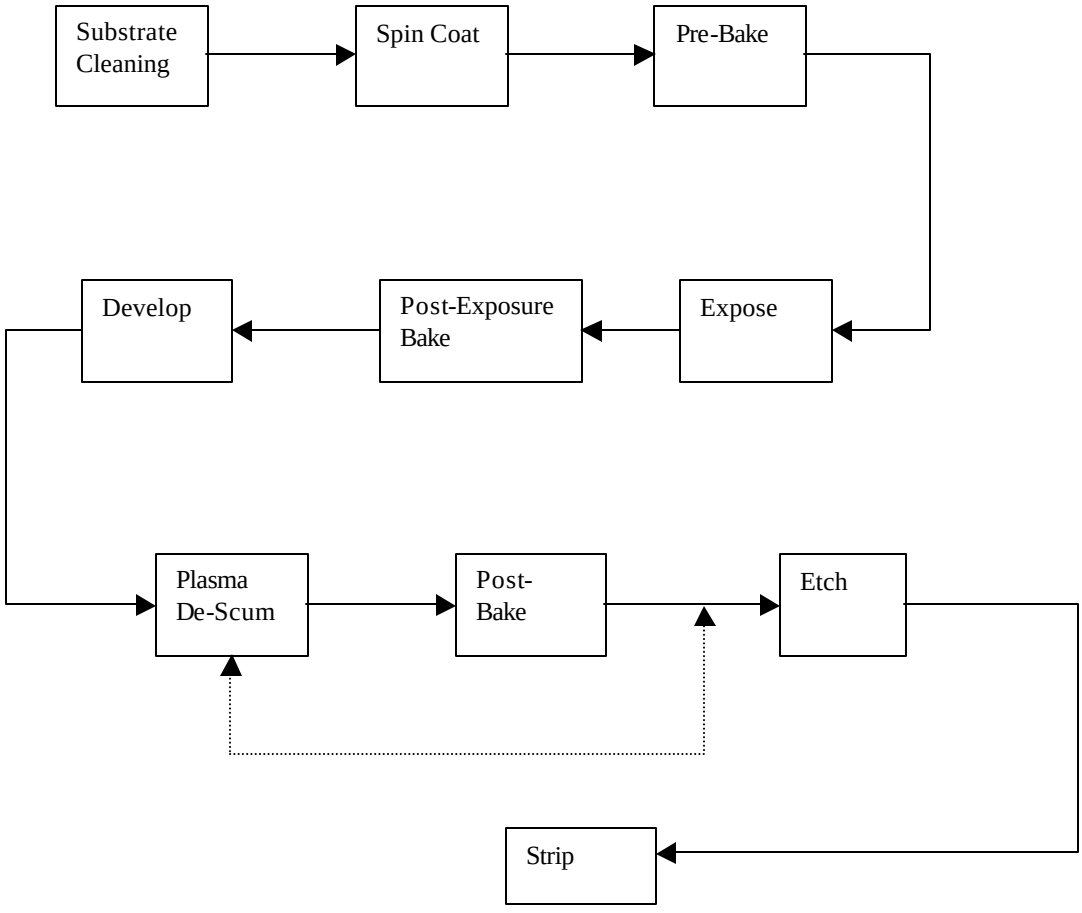

Figure 3.7 Flow chart of a typical resist process. Steps in broken lines are not the used for all materials.

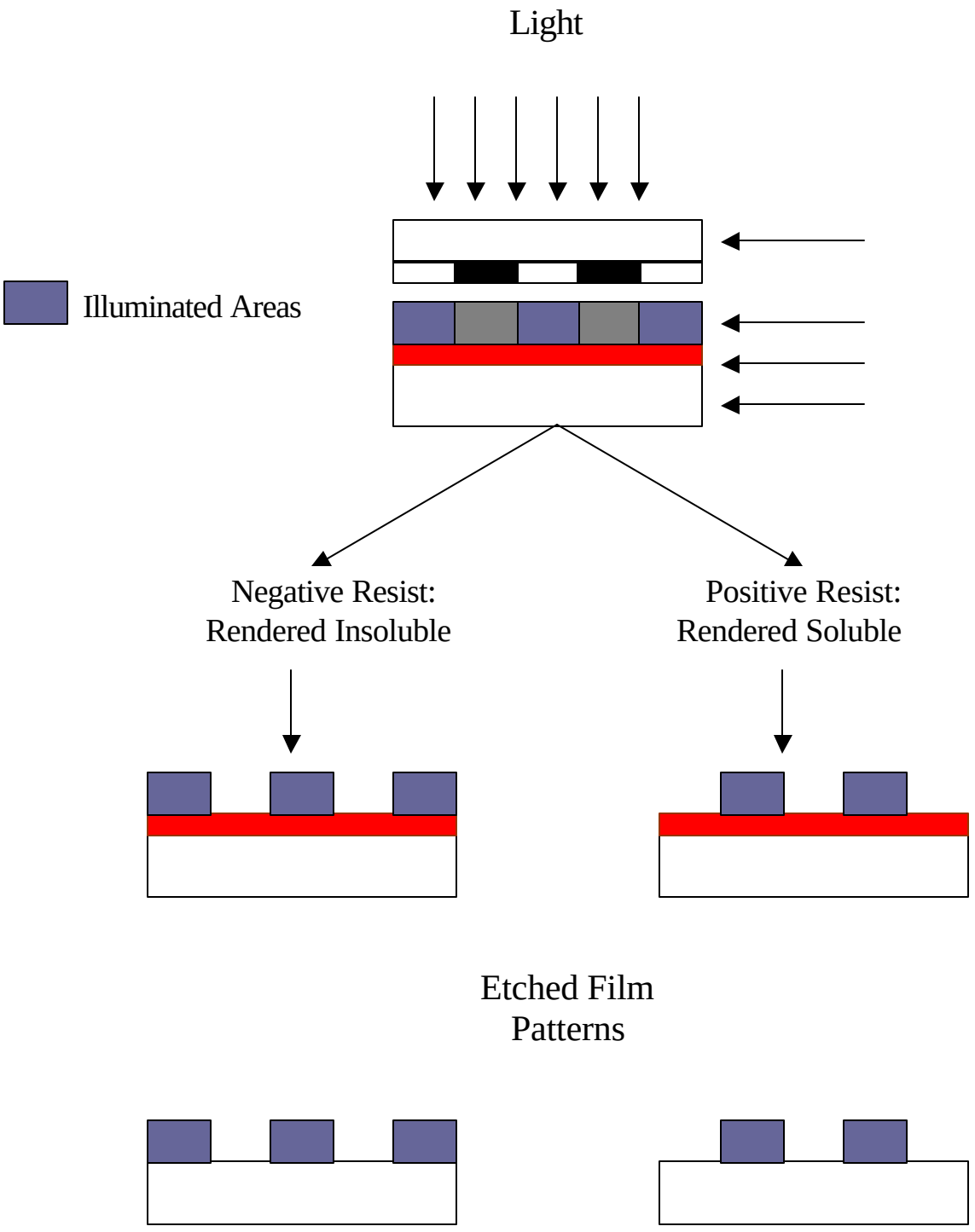

Figure 3.8 Exposure and development of negative and positive photo-resists

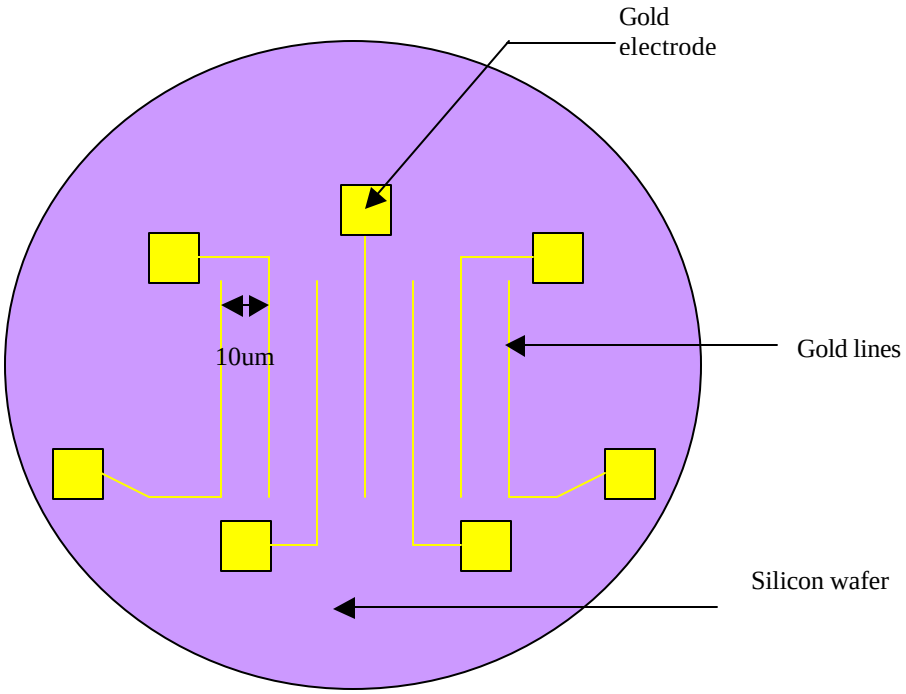

Figure 3.9 The gold line sample

References:

[1] S. N. Magonov, D. Reneker, Annul. Revs. Mat. Sci., Vol. 27, 175-200, (1997).

[2] Stanley Wolf, Richard N. Tauber, Silicon processing for the VLSI ERA, Volume 1: Process technology, 2nd edition, Lattice Press, Sunset Beach, CA. (1999).