CHAPTER III

EXPERIMENTAL

3.1 Diamond micro-ATR sensor

3.1.1 Materials and equipments

Homemade accessory for adjusting the reflecting plane

1. Sample holder for diamond micro-ATR sensor
2. Flat tip diamond micro-ATR sensor head (0.10 carat)
3. Sharp tip diamond micro-ATR sensor head (0.10 carat)
4. Nicolet Magna 750 Series II FT-IR spectrometer and NICPLAN infrared microscope

3.1.2 Acquisition parameters of FT-IR experiments

Nicolet Magna 750 Series II FT-IR spectrometer equipped with NICPLAN infrared microscope

Experimental setup

Spectral resolution, cm^{-1}	4.0
Number of scans	256
Result spectrum	Absorbance

Spectrometer parameters

Source	Globar®
Detector	Mercury-cadmium-telluride (MCT)
Beam Splitter Setting	KBr

3.2 Efficiency study of the diamond micro-ATR sensor for various types of samples

3.2.1 Materials and equipments

1. Soft polymers

1.1. Flat polyethylene film

1.2. Thick polypropylene film

2. Hard and rigid polymers

2.1.Thick polystyrene piece

2.2. Thick polycarbonate film

3. Coating polymer on metal

Soft drink can

4. Coating polymer on polymer

4.1. Compact disc

4.2. Adhesive tape

5. Small sampling area

5.1. 50 baht bank note on blue region

5.2. 50 baht bank note on white region

3.2.2 Methodology for spectral acquisition using diamond micro-ATR sensors

Steps in preparation a diamond micro-ATR sensor:

1. The adjustable plane-homemade accessory was placed on the stage of IR microscope.

2. The piece of sample was placed against with the flat tip and then an applied pressure was employed.

3. The diamond sensor equipped with the sample was put into the homemade accessory.

4. The table facet of diamond was adjusted perpendicular to IR incident beam in order to achieved high energy throughput before ATR spectra were taken.

5. The sample procedure as described in second step was repeated using sharp tip diamond as an IRE instead of flat tip diamond and ATR spectrum was acquired.

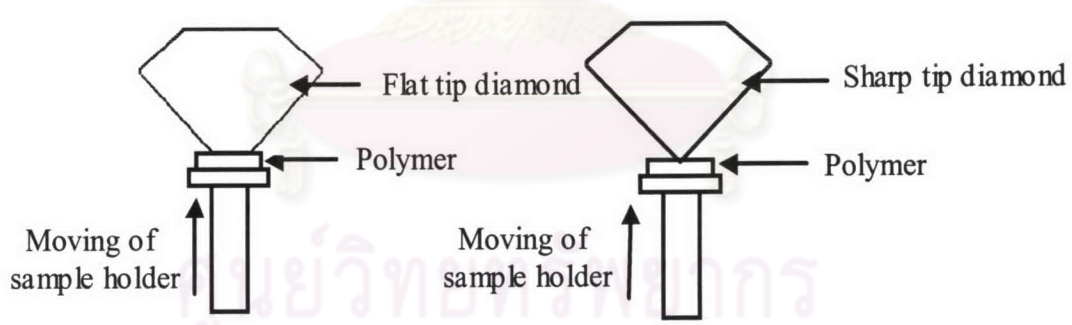

Figure 3.1 Experimental procedure for acquiring optical contact between a polymer and the two types of diamond tip.

3.3 Conventional ATR experiment

3.3.1 Materials and equipments

1. **Soft polymers**

    1.1. Flat polyethylene film

    1.2. Thick polypropylene film

2. **Hard and rigid polymers**

    2.1. Thick polystyrene piece

    2.2. Thick polycarbonate film

3. **Coating polymer on metal**

    Soft drink can

4. **Coating polymer on polymer**

    4.1. Compact disc

    4.2. Adhesive tape

5. **Small sampling area**

    5.1. 50 baht bank note on blue region

    5.2. 50 baht bank note on white region

6. **Single reflection attenuated total reflection (SATR) accessory (45° IRE from Spectra Tech.) with seagull accessory**

    Hemisphere ZnSe IRE (diameter 2.50 cm)

7. **Nicolet Magna 750 Series II FT-IR spectrometer**

3.3.2 FT-IR spectrometer condition

Nicolet Magna 750 Series II FT-IR spectrometer

Experimental setup

Spectral resolution (cm^{-1})	4.0
Number of scans	256
Result spectrum	Absorbance

Spectrometer parameters

Source	Globar®
Detector	Mercury-cadmium-telluride (MCT)
Beam Splitter Setting	KBr

3.3.3 Methodology for spectral acquisition using conventional ATR experiment ZnSe IRE system

Steps in preparation a diamond micro-ATR sensor:

1. The incident angle of seagull accessory was set at 45°.
2. A hemisphere ZnSe prism had placed into the accessory before the background spectrum was aquired.
3. The sample was placed against ZnSe IRE by an applied and then ATR spectra of the samples were acquired.
4. The same procedure as described in step 3 was repeated using other samples and then ATR spectra were acquired.

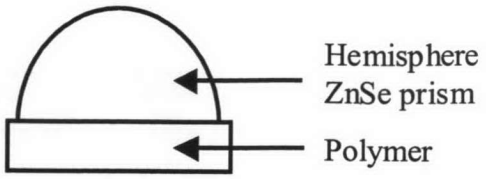

Figure 3.2 Experiment procedure for acquiring optical contact between a polymer and the ZnSe prism.