PIKE TECHNOLOGIES
Spectroscopic Creativity

Installation and User Guide

FlexIR™ Hollow Waveguide Accessory

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Introduction

The FlexIR Hollow Waveguide Accessory is an excellent tool for remote and specific area analysis of a wide variety of samples. Visible surface contamination, small area material identification and bulky materials too large to fit into the FTIR sample compartment are a few of the many samples and application types for the FlexIR accessory.

The FlexIR Hollow Waveguide (HWG) Accessory is designed for high throughput, ruggedness and wide spectral range. The FlexIR utilizes a custom optical design with diamond-turned focus optics providing exceptional IR throughput. The new hollow waveguides are very durable and free from the typical fracture problems encountered with polycrystalline and glass core fibers. The highly reflective HWG fibers transmit maximum energy through the mid-IR spectral region – eliminating the need for multiple fibers for a complete spectrum.

Available probes include ATR, diffuse reflectance, and specular reflectance. The ATR probe may be configured with a zinc selenide, germanium, or diamond/zinc selenide composite crystal. The PIKE Technologies FlexIR Hollow Waveguide Accessory is built and tested for optimum performance for your FTIR spectrometer.

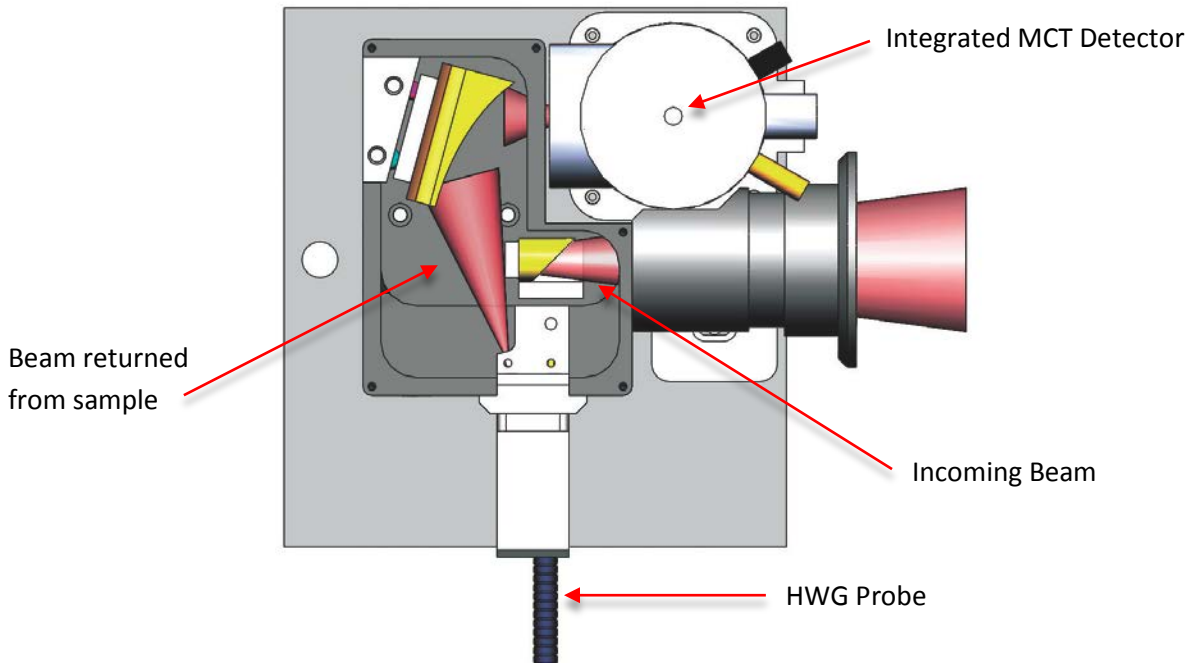

Figure 1: Optical Diagram of the MCT FlexIR Accessory

Unpacking Your FlexIR HWG Accessory

In order for you to quickly verify receipt of your accessory, we have included a packing list. Please inspect the package carefully. Contact PIKE immediately if any discrepancies are found.

WARNING: *The hollow waveguide probes have a limited bend radius of 150 mm. A smaller radius bend will break the probe.*

Packing List

FlexIR Manual
PN 045-3000
Quantity 1

FlexIR Hollow Waveguide Base
PN 045-30XX
Quantity 1

HWG Probe or Probes
(based upon your order)

Probe Attachment Screws
(for Mid-IR FlexIR)
Quantity 2

Wrench Set
Quantity 1

Purge Tubing Kit
PN 025-3055
Quantity 1

Funnel
(to fill MCT Detector)

Quantity 1

Reference Cap
(Diffuse and Specular Probes only)
Quantity 1

Installing the FlexIR HWG with MCT Detector

Accessory Setup

Turn off the power to the FTIR spectrometer. Connect the interface detector cable of the HWG accessory to the detector connector port of the spectrometer. Consult the FTIR manual for additional information regarding the detector connector port and the proper software settings to select an external detector.

The base optics of the Mid-IR FlexIR HWG Accessory fits into the sample compartment of the FTIR spectrometer. The FlexIR HWG Accessory will include the baseplate mount for your FTIR spectrometer. Place the accessory into the sample compartment of your FTIR and secure any lock-down screws.

MCT Liquid Nitrogen Filling and Stabilization

Fill the MCT detector with liquid nitrogen using the funnel included with the accessory.

WARNING: *Wear safety goggles and protective clothing when handling liquid nitrogen. Exposure to liquid nitrogen will cause severe skin or eye injury.*

Pour a small amount of liquid nitrogen into the funnel and wait for it to drain into the Dewar of the detector. Add additional small amounts of liquid nitrogen until the detector is filled. Allow the detector to stabilize for about 10 minutes and then top it off with a final amount of liquid nitrogen to completely fill the liquid nitrogen Dewar. A lit green LED light indicates the Dewar contains liquid nitrogen and is beginning the detector cooling process or is sufficiently cooled. It is not an indication that the accessory is necessarily ready for use. Be sure to wait a 10 minute equilibrium time after the Dewar has been completely filled. Observing a stable interferogram is another indication that the HWG accessory is ready for measurements. An insufficiently cooled detector will produce a low and unsteady signal. The red LED light indicates the accessory is not ready for use. If the red LED is illuminated during use check that the detector Dewar is filled with liquid nitrogen.

Installing the HWG Probe

The next step is to install the HWG probe. Insert the black probe connector end into the base optics and secure with hex screws provided with the accessory as shown on the following page.

Figure 2: Assembled FlexIR Accessory

Aligning the FlexIR Accessory

This accessory has been pre-aligned on the same make as your spectrometer. In most cases, little to no further alignment is necessary. To assess the need for additional alignment, check the energy when in the monitor mode of the spectrometer software. When using the specular or diffuse reflection probe, slide the reference cap over the probe tip before monitoring the energy. No reference cap required for ATR probes.

The alignment procedure requires that the IR beam pass through the hollow waveguide(s) and return the beam through the hollow waveguide to reach the detector. Prior to adjusting the mirrors as explained below a closed loop must be established. In other words, the alignment process for a diffuse reflectance or a specular reflectance probe requires that the probe tip be positioned gently touching the diffuse reference disk or a specular mirror embedded in the reference cap included with the probe, respectively. For an ATR probe, no reference or mirror is required. Simply be sure the ATR crystal surface is clean.

Open the FTIR bench setup software display to monitor the interferogram signal strength. Please refer to your FTIR software manual for details of this operation. Reduce the size of the FTIR aperture to achieve acceptable interferogram signal strength. If your FTIR does not have an aperture, use an IR beam-limiting screen placed at the right or left side of the FTIR sample compartment to reduce the energy to an acceptable level.

Optimize the signal by adjusting the input mirror (Figure 3), which directs the IR beam into the hollow waveguide(s), located below the installed probes. Rotate slowly the screws, positioned on the diagonal, one at a time while maximizing the signal by monitoring the energy displayed through the FTIR software.

After adjusting the input mirror optimize the position of the output mirror, which redirects the returned beam into the detector. Adjust the two screws located on the top of the accessory (Figure 3). This mirror and the resulting focus on the detector is extremely sensitive to small movements of the positioning screws. A turn of less than a quarter of a complete rotation will drastically change the amount of energy striking the detector, which may be monitored through the FTIR software.

Figure 3: Locations of mirror alignment ports

Repeat the adjustment of the input and output mirror two to three more times if increases in performance are observed, to fully optimize the accessory. The positioning of the MCT detector has been optimized for the specific bench and probe type. In general, the position of the MCT detector does not require adjustment. In the event different probe types are in use, the position/alignment of the MCT detector may slightly improve signal performance. Adjust the MCT detector position in the left or right direction by loosening the screw located on the detector base rigging as shown in Figure 4. After adjustment, retighten the screw to secure the detector.

Figure 4: Illustration of MCT detector alignment point

Optimizing the MCT Pre-Amp Gain

The MCT pre-amp gain has been adjusted at the PIKE manufacturing facility. It most likely will not require further adjustment. However, adjustments may be required especially if using different probe types which exhibit large relative energy differences reaching the detector. For example, the gain is set higher for a diffuse reflectance probe compared to an ATR probe.

Figure 5: Illustration of MCT detector alignment point

The gain should be adjusted to allow 50% of total energy counts or voltage registering on the detector. To adjust the gain, remove the gain cap from the front of the base optics. Access the gain screw using a small screwdriver.

Removing the FlexIR Accessory

Turn off the power to the FTIR spectrometer.

WARNING: *Do not disconnect the detector cable before turning off the FTIR. Failure to do this may cause damage to the FTIR and/or FlexIR electronics.*

Remove the signal/power cable from the FTIR spectrometer and remove the FlexIR accessory from the sample compartment.

Storage

Place the FlexIR fiber optic accessory into the plastic bag provided to protect its optics from dust.

ATR Probe

The ATR probe may be used to measure solids, liquids, and powders. Intimate contact between the sample and the ATR crystal is required to obtain a quality spectrum. To use, collect a background spectrum of a clean ATR crystal. Next, place the ATR tip on the sample and exert pressure to obtain good contact and collect the sample spectrum.

The ATR probe is sealed up to 60 mm beyond the ATR probe tip. Do not submerge the probe into a liquid deeper than 60 mm. Double score lines are etched on the probe head to indicate the maximum immersion allowed. Isopropyl alcohol, a mild solvent, is recommended for cleaning the ATR crystal using a cotton swab.

Zinc Selenide

Zinc Selenide (ZnSe) is a general-purpose ATR material. It has limited use with strong acids and alkalis. The acceptable pH range is 5 through 9. Additionally, complexing agents such as ammonia and EDTA will erode its surface because of the formation of complexes. The maximum temperature range for the ZnSe probe is ambient to 95 °C.

Germanium

Germanium (Ge) has been used extensively in the past as a higher refractive index material for samples that have a high refractive index such as carbon filled samples. Due to its higher refractive index, spectra collected with a Ge ATR crystal are weaker in spectral absorbance compared to spectra collected with ZnSe or diamond/ZnSe composite ATR crystals. The acceptable pH range is from 1 to 14. The maximum temperature range for the Ge probe is ambient to 95 °C.

Diamond/ZnSe Composite

Diamond is one of the most rugged optical materials. It can be used for the analysis of a wide range of samples including acids, bases, and oxidizing agents. Diamond is also scratch and abrasion resistant. Its disadvantage is the intrinsic absorption from approximately 2300 to 1800 cm^{-1}. The maximum temperature range for the diamond/ZnSe probe is ambient to 60 °C.

Diffuse Reflectance Probe

The diffuse reflectance probe is suitable for solid samples with diffuse surfaces only. Do not submerge the diffuse reflectance probe in powders or liquids as the tip is without a protective window and damage to the probe will result.

In collecting a background spectrum, insert the probe tip into provided reference cap. If probe fits tightly or cannot bottom out on reference loosen set screw on side and insert probe cap. Tighten set screw to provide enough drag/friction to hold the reference cap in place, yet allow the cap to be removed. Next the sample spectrum is collected after positioning the probe either touching or within 0.4 mm from the sample. The probe tip is delicate. Avoid exerting a strong pressure on the tip end. The maximum temperature for the diffuse reflectance probe is 95 °C.

Specular Reflectance Probe

The specular reflectance probe is ideal for smooth non-diffuse surfaces. In collecting a background spectrum, insert probe into provided reference cap. If probe fits tightly or cannot bottom out on reference loosen set screw on side and insert probe cap. Tighten set screw to provide enough drag/friction to hold the reference cap in place, yet allow the cap to be removed.

Generally, a specular reflectance spectrum is collected by collecting a background spectrum using the reference cap with an embedded mirror. Next the sample spectrum is collected after positioning the probe either touching or within 0.4 mm from the sample. The probe tip is delicate. Avoid exerting a strong pressure on the tip end. Do not submerge the probe tip in a powder or a liquid as this will result in damage to the probe. The maximum temperature for the specular reflectance probe is 95 °C.

Suggested Measurement Settings

Wavelength Range: 4000 to 700 cm^{-1} (actual range of HWG Accessory MCT version: 5000 to 700 cm^{-1})
Velocity: Select appropriate speed for MCT detector for your spectrometer Resolution: 4 to 8 cm^{-1}

Actual measurement parameters will vary based on the experimental conditions and sample material.

Example Applications

Analysis of Coatings on Intractable by Panels by Diffuse and Specular Reflectance Probes

Spectra of coated metal samples collected with diffuse and specular reflectance FlexIR probes are illustrated on the following page. Figure 6 shows a spectrum of a coating on a smooth reflective surface (coating on the outside of a soda can), conducive to specular reflectance measurements. The second sample featured a painted surface with diffuse reflectance characteristics (Figure 7).

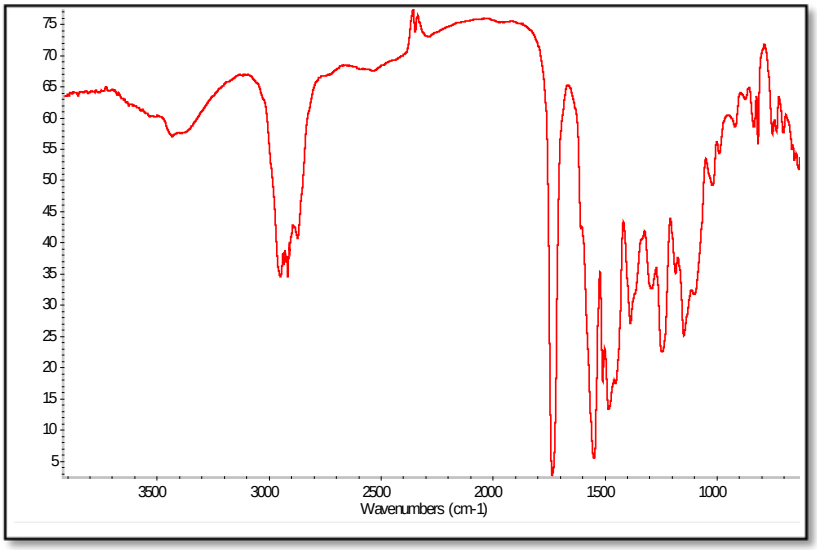

Figure 6: Spectrum of a coated aluminum surface using the specular reflection probe

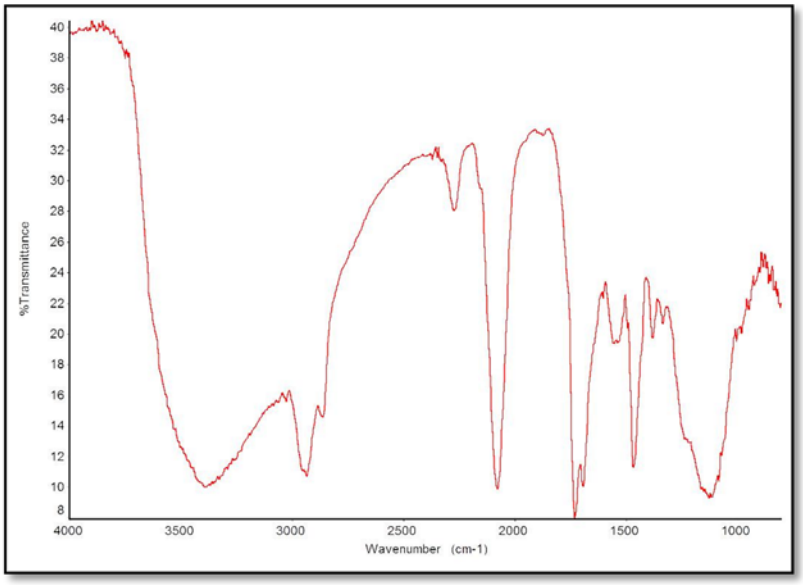

Figure 7: Spectrum of a painted panel collected using the diffuse reflection probe

Use of ATR HWG Probe to Investigate Defects in Tape

ATR is an ideal probe to investigate defects on adhesive tape. In this example a background was collected while the ATR probe was clean. The sample spectrum was then collected after adhering the tape sample to the ATR crystal. The resulting spectrum is shown in Figure 8. Other applications for the ATR accessory include reaction monitoring, skin analysis, and small spot defects.

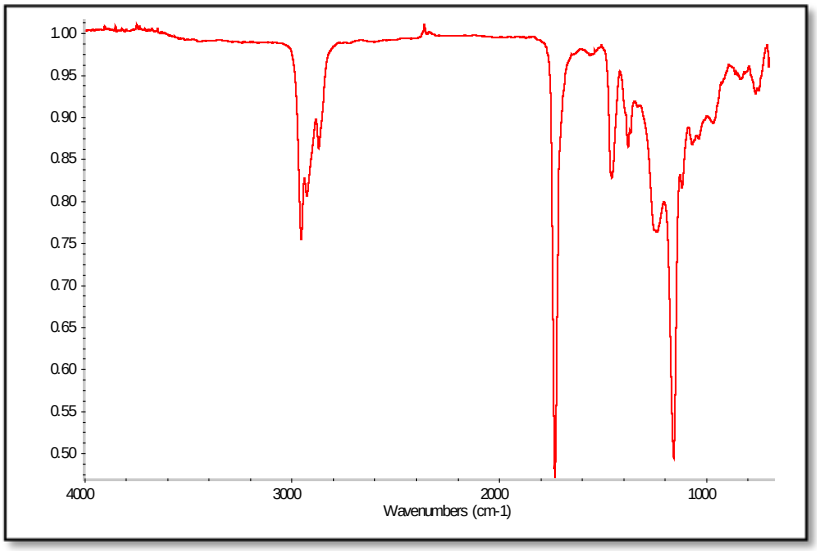

Figure 8: Spectrum of transparent tape (adhesive side) collected with the ZnSe ATR probe

Precautions

SAFETY

Caution should be used when handling and using ATR crystals since some of the materials can be hazardous. Specifically, zinc selenide is a heavy metal material and should be handled with this in mind. If the crystal is broken or pulverized, the dust may be harmful by inhalation, ingestion, or skin absorption.

HWG Probe Assembly Handling

Hollow waveguide assemblies are fragile. Do not bend past their minimum bend radius of 150 mm. Do not expose probes to shock. Do not drop as they may get damaged. Do not flex or stress the probe and waveguide interface. Use caution when analyzing powder samples with diffuse and specular reflectance probes. Stay within the prescribed temperature limit for each probe.

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