Analysis Method for the Curing Degree of Fiber and Fiber Coil

Abstract: The analysis method for the curing degree of fiber and fiber coil is deeply introduced by introducing the principle of FTIR, common test methods, curing degree and curing process analysis.

Keywords: Potassium bromide (KBr) pellet method, liquid film method, ATR reflection method, curing degree and curing process analysis

Introduction:

In this article, Fourier Transform Infrared (FTIR) spectrometer is used to analyze the curing degree of the fiber and fiber coil produced by the company, to improve the stability and reliability of the end product.

1. FTIR principle

Infrared and visible light are both electromagnetic waves, and infrared is an electromagnetic wave with a wavelength between visible light and microwave. According to the wavelength range, infrared light can be divided into three wave regions: near infrared, medium infrared and far infrared, among which the medium infrared region (2.5~25μm; 4000~400cm-1) can well reflect the various physical processes carried out inside the molecule and the characteristics of the molecular structure, and is the most effective for analyzing various problems in molecular structure and chemical composition. Therefore, the medium infrared region is the most widely used in the infrared spectrum. Generally speaking, the infrared spectrum mostly refers to this range. The infrared spectrum belongs to the absorption spectrum, which is produced by absorbing infrared light of a specific wavelength when compound molecules vibrate.

The FTIR spectrometer is designed according to the principle of light coherence, so it is an interference spectrometer, which is mainly composed of a light source, an interferometer, a detector, a computer and a recording system. Most FTIR spectrometers use a Michelson interferometer, so the original spectrum measured in the experiment is the interferogram of the light source, and then the fast Fourier transform calculation of the interferogram is carried out by the computer to obtain the spectrum as a function of wavelength or wavenumber. Therefore, the spectrum is called FTIR spectrum, and the instrument is called FTIR spectrometer.

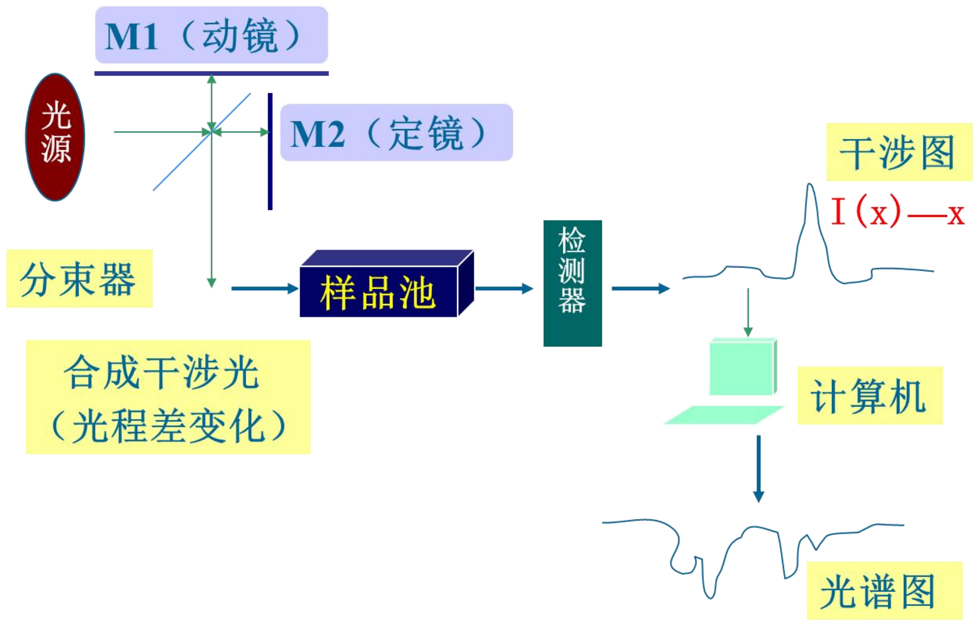

Figure 1 Schematic diagram of FTIR work

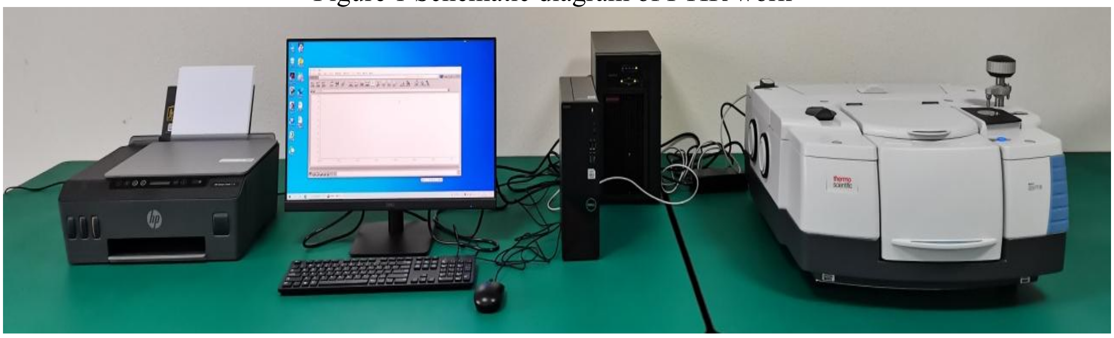

Figure 2 Nicolet™ iS50 FTIR spectrometer of YOEC

2. Common test methods

2.1 Potassium bromide (KBr) pellet method

This is a classic traditional infrared spectrum sample preparation method. Spectral pure KBr is used as the dispersion medium. After the sample and potassium bromide are fully ground and placed on the tablet press, the sample can be prepared. It is widely used in infrared qualitative analysis and structural analysis. The main disadvantage of this method is that potassium bromide itself easily absorbs moisture in the air, and the tested spectrum is prone to moisture interference and the baseline is easy to tilt, which affects the test results. Figure 3 shows the spectrum of pure potassium bromide containing a small amount of water. Through the test, it can be seen that the main impurity is water. Therefore, the use of the potassium bromide pellet method for sample preparation requires high-purity and very dry potassium bromide.

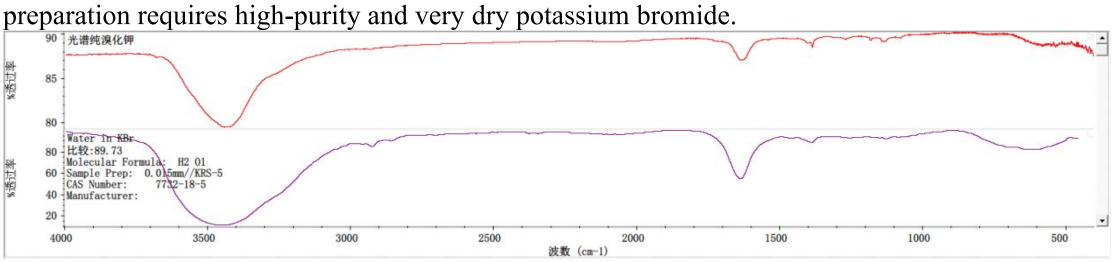

Figure 3 shows the spectrum of pure potassium bromide containing a small amount of water.

2.2 Liquid membrane method

If the sample to be tested can be dissolved with a specific solvent or the sample itself is liquid, the potassium bromide sheet can be used as the background, the sample can be thinly coated on the potassium bromide side, the solvent is dried, and then placed in the infrared spectrometer for testing. The advantage of this method is that the purity of potassium bromide does not need to be so high because the impurity peak has been scanned into the background in the process of testing the background. In this way, the spectrum obtained is generally flat with no impurity peak. It should be noted that the coated samples should not be too many, otherwise the test process may exceed the instrument range and cause a test failure. As shown in Figure 4, the coating concentration of the sample shown by the red line is too large, resulting in changes in the peak shape near 1200cm-1 and 1800cm-1, affecting the test results, while the green line is the normal spectrum.

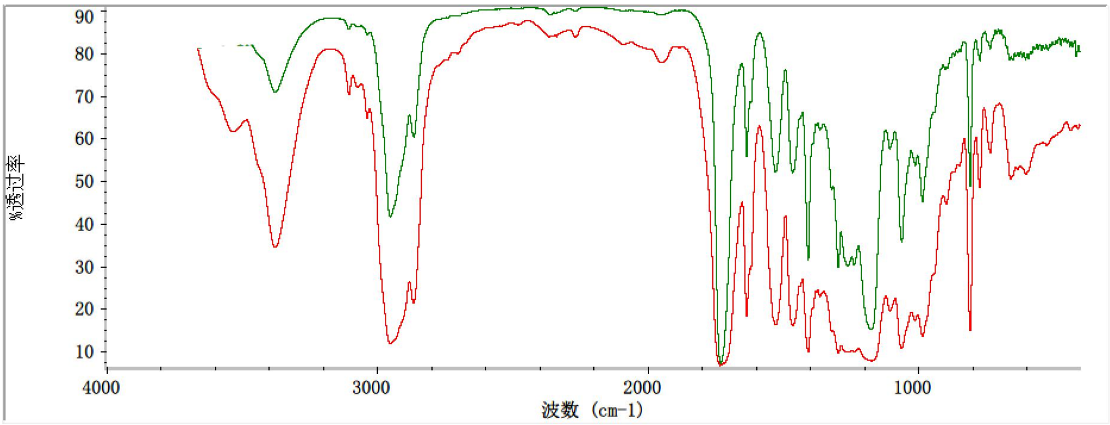

Figure 4 Infrared spectrum of samples with excessive concentration

2.3 ATR reflection method

The Attenuated Total Reflection (ATR) accessory is designed based on the principle of optical internal reflection. The infrared light emitted from the light source passes through a crystal with a high refractive index and then is projected onto the surface of a sample with a low refractive index. When the incident angle is greater than the critical angle, the incident light will be totally reflected. The infrared light is not all reflected, but penetrates to a certain depth in the sample surface and then returns to the surface. The sample has selective absorption in the frequency region of the incident light, and the intensity of the reflected light is weakened, resulting in a diagram similar to the transmission absorption, to obtain the structural information of the chemical composition of the sample surface. Compared with conventional infrared technology, there is no need for sample preparation and sample damage, and solid, liquid, and fiber surfaces can be detected.

Since the penetration depth of light of different wavelengths to the sample is different, the penetration depth at the high-frequency end and the low-frequency end can differ by an order of magnitude (usually the penetration depth does not exceed 2μm), and the absorption peak intensity at the low-frequency end is much stronger than the peak intensity at the high-frequency end. Therefore, the spectrum measured by the ATR method cannot be directly compared with the spectrum measured by the transmission method. When detecting changes in the absorption peak intensity, such as measuring the curing degree, the same method needs to be used to test the samples before and after curing. Figure 5 shows the comparison between the perspective spectrum and the ATR spectrum of the same sample, and it can be seen that the absorption peak intensity of the spectrum is significantly different.

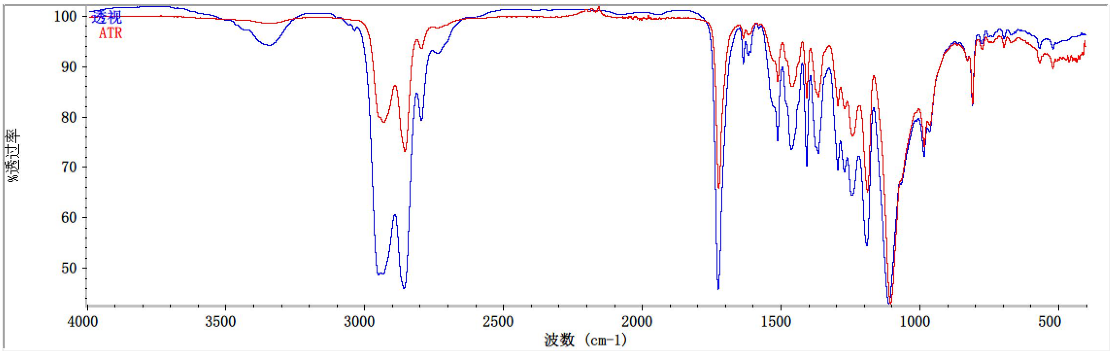

Figure 5 Comparison between perspective spectrum and ATR spectrum

3. Curing degree and curing process analysis

(1) Curing degree test

Ultraviolet curing materials (including coatings for the inner and outer layers of optical fibers, coloring inks for optical fibers, light-curing coil adhesive, etc.) are mainly composed of acrylate, photoinitiators, and additives. The curing principle is that the photoinitiator generates free radicals under the excitation of ultraviolet light, and the free radicals initiate acrylate chain growth and polymerization.

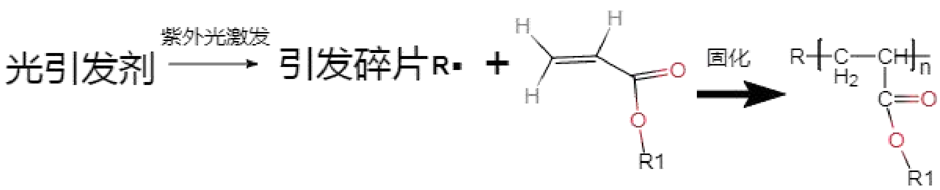

Figure 6 Principle of ultraviolet curing

During the curing process, the C=C double bond disappears and becomes a C-C single bond, so we define the curing degree as the ratio of double bonds to single bonds. Among them, the absorption peak of the C=C double bond is used as the characteristic peak, and the absorption peak of the ester bond is used as the reference peak because the ester bond does not participate in the reaction. Take the coating as an example, test the infrared spectrum before and after curing, and calculate the curing degree, as shown in the figure,

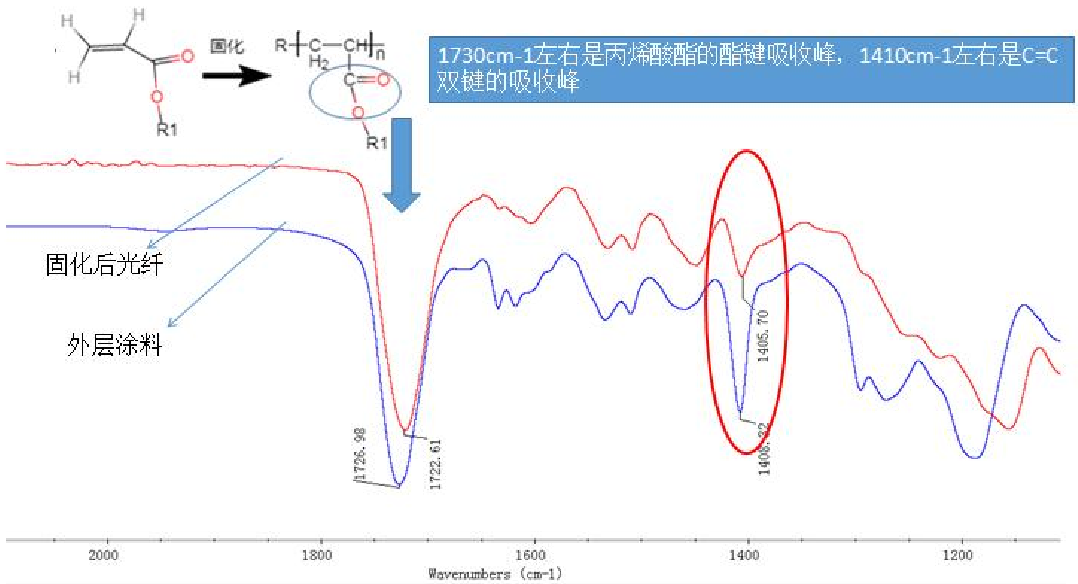

Figure 7 Changes of the infrared spectrum before and after coating curing

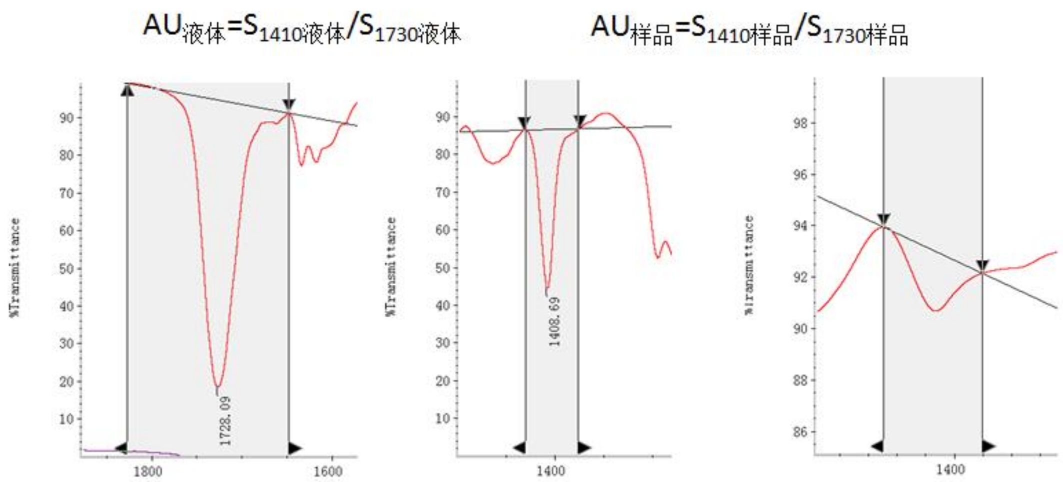

Figure 8 Schematic diagram of peak area measurement

The software is used to measure the corresponding peak area. Due to the different accuracy of the instrument, there may be some deviation in the measurement of absolute curing degree with different instruments, including the measurement of peak area.
The absolute curing degree RAU is calculated according to formula (1):

$$RAU = \frac{AU_{液体} - AU_{样品}}{AU_{液体}} \times 100\% \qquad (1)$$

where:
RAU - absolute curing degree
AU liquid - peak area ratio of two absorption peaks of liquid;
AU sample - peak area ratio of two absorption peaks of sample.

Generally, the concept of relative curing degree is introduced in engineering. Since the cross-linking network of materials cannot be ideal, the absolute curing degree of many materials cannot reach 100%, so every material will have a limited curing degree. Relative curing degree refers to the ratio of absolute curing degree to ultimate curing degree of material.

If the ultimate curing degree of material is 90.5%, when the absolute curing degree of the material reaches 90%, the relative curing degree is 90%/90.5% = 99.45%.

The relative curing degree R-RAU is calculated according to formula (2):

$$R - RAU = \frac{RAU_{样品}}{RAU_{极限}} \times 100\% \qquad (2)$$

where:
R-RAU - absolute curing degree
RAU sample - absolute curing degree of sample material;
RAU limit - ultimate curing degree of the sample material.

The ultimate curing degree of the material can be obtained by continuously increasing the irradiation power in the absence of oxygen and testing the absolute curing degree of the sample. When the absolute curing degree cannot be continuously improved, the ultimate curing degree can be obtained.
(2) Test analysis of fiber coil curing process

The fiber coil is composed of coil wrap adhesive and fiber. The size and volume of different types of fiber coils are different, so the curing of fiber coil is very different from that of simple coil wrap adhesive. Generally, how long the fiber coil needs to be cured is mostly obtained through experience. If the curing time is too short, it may cause poor internal curing of the fiber coil. If the curing time is too long, it will cause excessive curing and aging of the outer ring fiber coil and tail fiber, affecting the long-term reliability. Therefore, FTIR can be used to test the curing degree at different positions of the fiber coil to obtain the best curing process. Figure 9 is a schematic diagram of the position of the test curing degree of a certain fiber coil. The curing process can be evaluated by testing the curing degree of the coil wrap adhesive at these positions.

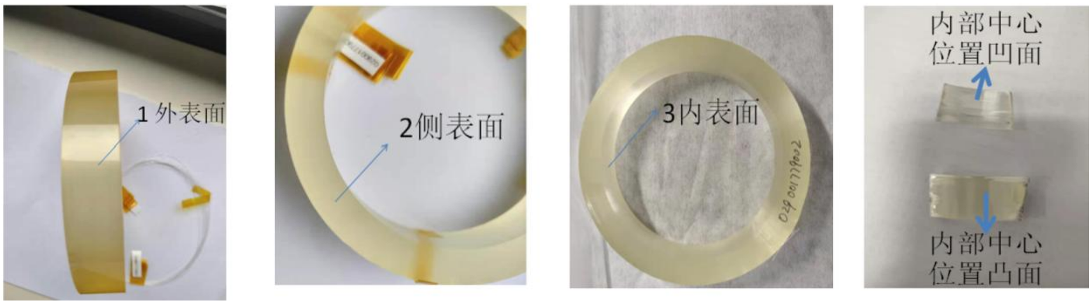

Figure 9 is a schematic diagram of the position of the test curing degree of a certain fiber coil

Conclusion

This article introduces the method and principle of measuring the degree of curing of optical fiber and optical fiber coils by using infrared spectrum technology, which provides a basis for better determining the curing process of the product.