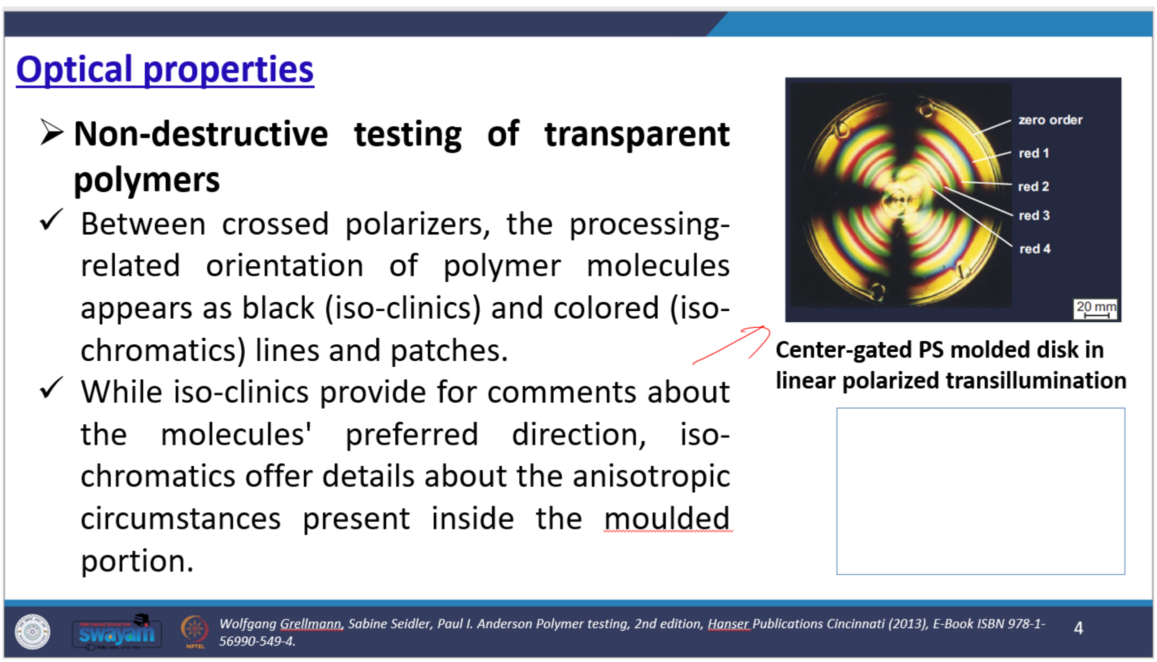
Lecture 45- Polymer Testing 10

Hello friends, welcome to the next segment of optical physical properties. The previous segment covered various thermal properties like heat conductivity and diffusivity, differential scanning, calorimetry, thermogrammetry, and thermo-mechanical analysis. Apart from this, we discussed the optical properties in the refractive index, refractometer, polarization, and polarization optical tests; all these things were covered. In this particular segment, we will discuss the other optical properties like non-destructive testing, transparent polymers, transmission, absorption, reflection, glass, intrinsic diffuse reflectance and haze, infrared spectroscopy color, and laser technology.

Let us talk about non-destructive testing or transparent polymers. So, between the crossed polarizers, the processing-related orientation of polymer molecules appears as black or isoclinics and colored lines and patches.



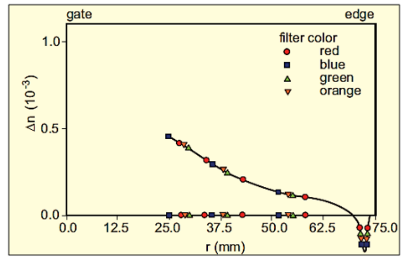
Isoclinics provides comments about the molecule's preferred direction and isochromatic details of the anisotropic circumstances inside the molded portion. A circular polystyrene device with a center gate is shown in this particular figure in the linear polarized translation without any external loads.

$$\Delta n = \frac{k\lambda}{d}$$

According to this particular figure and equation, the isochromatics can be used to determine the position of isochromatics and their assignment to a specific color order, starting with the black isochromatics of the 0 order. This black cross from the isoclinics indicates the molecules' symmetrical star-shaped mean orientation direction.

Cont…

- ✓ A circular PS device with a center gate is shown in linear polarized transillumination without any external loads.
- ✓ According to Figure and equation, the iso-chromatics can be used to determine the position of the iso-chromatics and their assignment to a specific color order, starting with the black iso-chromatics of the zero order.
- ✓ This black cross formed by the iso-clinics indicates the symmetrical, star-shaped main orientation direction of the molecules.



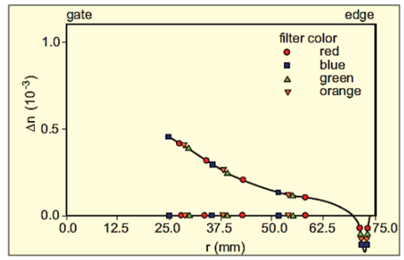
Anisotropy change in the moulded PS disk along its mould flow path

Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4. 5

The interference caused by light waves moving through anisotropic plastic causes the color orders to appear. The light rays are not separated if there is no anisotropy, and when polarizers are crossed, the darkness predominates, and the 0-order isochromatic is present. Therefore, retardation does not occur. When anisotropy rises, a specified wavelength in white light is extinguished, and the remaining light component takes on color. Now, a particular equation can be used to compute the amount of refinance along the circular center gated flow route as per the figure, and this is delta n is equal to k lambda d where delta n is by reference and k is the color order number starting on the outside of this circular disk with always black 0 order isochromatic and lambda is the wavelength of the isochromatic used for the evaluation, d is the thickness and k and lambda yields the optical retardation.

Cont…

- ✓ A circular PS device with a center gate is shown in linear polarized transillumination without any external loads.
- ✓ According to Figure and equation, the iso-chromatics can be used to determine the position of the iso-chromatics and their assignment to a specific color order, starting with the black iso-chromatics of the zero order.
- ✓ This black cross formed by the iso-clinics indicates the symmetrical, star-shaped main orientation direction of the molecules.

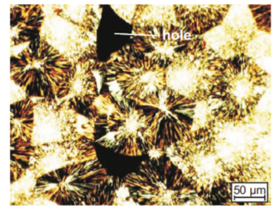


Anisotropy change in the moulded PS disk along its mould flow path

Now, a microscopic polarized light testing method for amorphous plastic. Amorphous polymers that are not filled do not form any structure that can be seen under a light microscope. However, polarization microscopy techniques can be used to find microscopic heterogeneity in variation in anisotropy. Point accurate measuring of retardation in a range from a few nanometers to approximately 80 meters is possible with the employment of tilting plates and rotary compensators. The key benefit of microscopic approaches is that measurement can be performed without requiring isochromatic line gradients across many orders in the molded item.

Microscopic polarization optical method for testing of semi-crystalline plastics

- ✓ In a light microscope, spherulitic structures are frequently apparent in semi-crystalline polymers.
- ✓ The spherulites, which are spatially composed of fibrils, can be tested in polarized transillumination to a minimum structural member size of 1 m by cutting them into semithin sections (section thickness to 10 m).



Spherulites structure of PP film in linear polarized light

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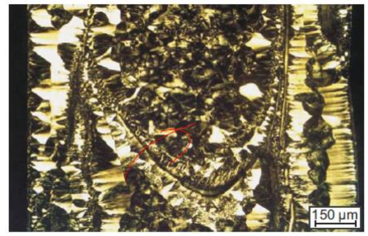
Thus, it is conceivable to manage anisotropy variations and differences within a minimal range, (≥ 4 µm), such as the phases of polymer blend on the one hand and film anisotropies with basically constant retardation on the other. Now, if we talk about semi-crystalline plastic in the light of a microscope, the spherulitic structures are frequently apparent in semi-crystalline polymers. The spherulites spatially composed of fibrils can be tested in polarized translumination to a minimum structure size of 1 meter by cutting them into a semi-thin section, a section of, say, 10-meter thickness.

Now, this figure depicts the spherulitic structure of a polypropylene film made by the crystallization of a liquid immediately from the melt. Due to the production process, holes have been formed at the limits of spherulities. Sample can be extracted from crucial areas of molded parts processed during the procedure and then examined using light microscopy.

Cont…

- ✓ The chosen processing conditions can then be inferred from this.
- ✓ A typical multi-phase structure of a PP part made from pellets is shown in Figure.
- ✓ At the granule borders, different PP modifications can be seen, each with a different set of optical and mechanical characteristics.
- ✓ It is necessary to measure optical data at the spherulites in order to determine these alterations, such as birefringence value and optical sign.



Structure selection from a part molded from PP

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Now, type from form distribution size and the number of occurring phases must all be considered in such an evaluation. Now, if the chosen processing conditions can be inferred from that particular aspect, this figure shows a typical polypropylene part's typical multi-phase structure made from pallets. Different polypropylene modifications can be seen at the granule borders, each with different optical and mechanical characteristics. Measuring the optical data at the spherulites is necessary to determine these alterations, such as birefringence values and optical signs.

Let us talk about the transmission absorption and reflection. Similar transparency, haze, and hiding power are optical qualities that primarily depend on two phenomena of incidence light;

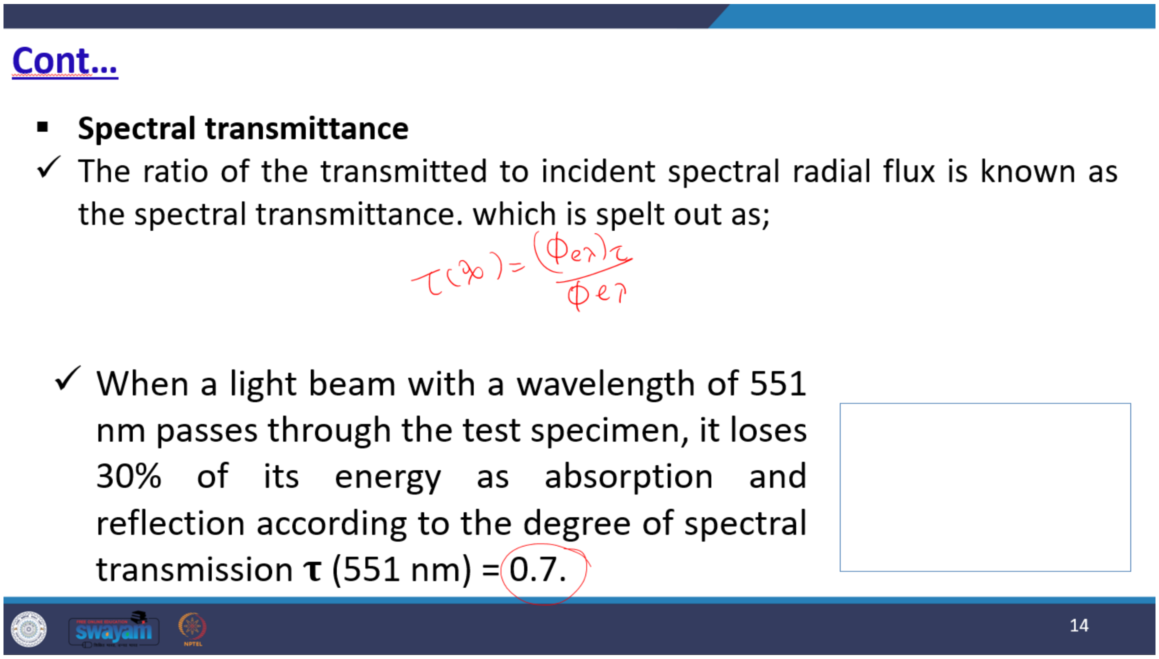
The material absorbs incident light, converting it into heat only by measuring the amount of reflection and transmission can this value be calculated.

Scattering inside the medium causes incident light to be reflected from its original direction.

The spectral transmission is the ratio of transmitted to incident spectral radial flux, is known as the spectral transmission, which is spread out as

$$\tau(\lambda) = \frac{(\phi_{e\lambda})_\tau}{\phi_{e\lambda}}$$

So, when a light beam with a wavelength of 551 nanometers passes through the test specimen, it loses 30 percent of its energy as absorption, and the reflection according to the degree of spectral transmission of tau 551 nanometer is equal to 0.7.



Now, if we talk about the absorptance (a_λ), it is defined as the ratio of the incident spectral radiant flux $\phi_{e\lambda}$ to the total radiant radiant flux absorbed by the medium.

$$a(\lambda) = \frac{(\phi_{e\lambda})_a}{\phi_{e\lambda}}$$

Similarly the spectral reflectance P(λ) can be determined as

$$P(\lambda) = \frac{(\phi_{e\lambda})_P}{\phi_{e\lambda}}$$

$(\phi_{e\lambda})_P$ is the total spectral flux reflected at the medium interface. This can only happen on one mirror surface or several clear media surfaces. Diffused reflectance results from a mirror surface when transmission does not happen and the luminance of the reflecting surface is compared to the luminance of a perfectly matte white body under equal illumination and observation conditions to establish the degree of diffused reflection.

Cont...

- **Absorptance a** (**λ**)
- It is defined as the ratio of the incident spectrum radiant flux ($\phi_{e\lambda}$) to the total spectral radiant flux absorbed by the medium.

$$a(\lambda) = \frac{(\phi_{e\lambda})_a}{\phi_{e\lambda}}$$

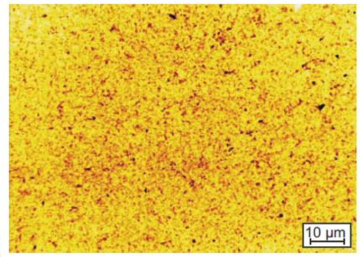
Similarly, spectral reflectance p (**λ**) can be determined as;

$$\rho(\lambda) = \frac{(\phi_{e\lambda})_\rho}{\phi_{e\lambda}}$$

Now, spectrophotometers like Minolta CR 40 are used to measure diffused reflectance and transmission. As a result of having little or no absorption in visible light, the majority of unfilled amorphous polymers are transparent, and transmittance is significantly changed by the addition of colors and process-related additives like heat stabilizers and UV stabilizers and due to their tinny size, these compounds are typically only detectable by electron microscopy. However, a light microscope can make a comparatively big particle of commonly used black stabilizers that can be visible. Now, the image allows for conclusions to be made regarding particle aggregation and distribution. This particular figure illustrates a 2-meter segment of a polyethylene matrix of a high-tension cable with nearly unagglomerated, very even carbon black distribution.

Cont…

- ✓ However, a light microscope can make the comparatively big particles of commonly used black stabilizers visible.
- ✓ The images allow for conclusions to be made regarding particle agglomeration and distribution.
- ✓ Figure illustrates a 2 m semi-thin segment of the PE matrix of a high-tension cable with nearly unagglomerated, very even carbon black distribution.

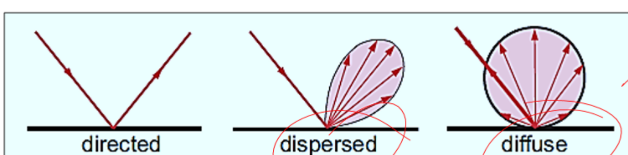


Carbon black distribution in a transilluminated semi-thin PE section

Let us talk about the glass's intrinsic diffused reflectance and haze. The surfaces of technical materials are always a little rough; due to this, they only partially reflect lights when illuminated directly as illustrated in this diagram. Here, you see the directed, dispersed, and diffused. The glass and the size of the surface roughness determine how the reflected light is distributed. The different intensities emerge when the resulting reflection is measured from various observation angles, and glass refers to this quality.

➢ Gloss, Intrinsic diffuse reflectance and Haze

- ✓ Surfaces of technical materials are always a little rough.
- ✓ Due of this, they only partially reflect light when illuminated directly, as illustrated in the diagram.



Figure; Types of surface reflection: **directed** (regular) reflection on a high-gloss smooth**, dispersed** or scattered (mixed reflection on a surface with low roughness and medium gloss, as well as **diffuse (totally scattered)** reflection on rough and matt surfaces

Surface haze is the word used to describe the diminished glass produced by such surface roughness. Let us talk about the intrinsic haze. When light strikes scattered centers, this is the light beam and it is scattered over the polymer; these are the fillers and certain pigments. So when the light strikes centers that scattered light like pigment or spherulites on the semi-crystalline polymer, internal diffuse reflection produces an inherent haze. The combined light reflected by a sample comes from both the surface and the inside.

Cont...

❖ **Intrinsic haze**

- ✓ When light strikes centers that scatter light, like pigments or spherulites on semi-crystalline polymers, internal diffuse reflection takes place, producing an inherent haze.
- ✓ The combined light reflected by a sample comes from both the surface and the inside.
- ✓ Because of this, a body's pigments help create the perception of color.

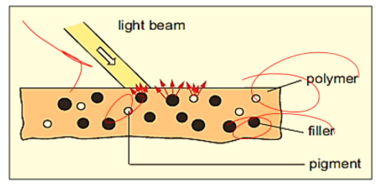


Diagram of internal diffuse reflectance

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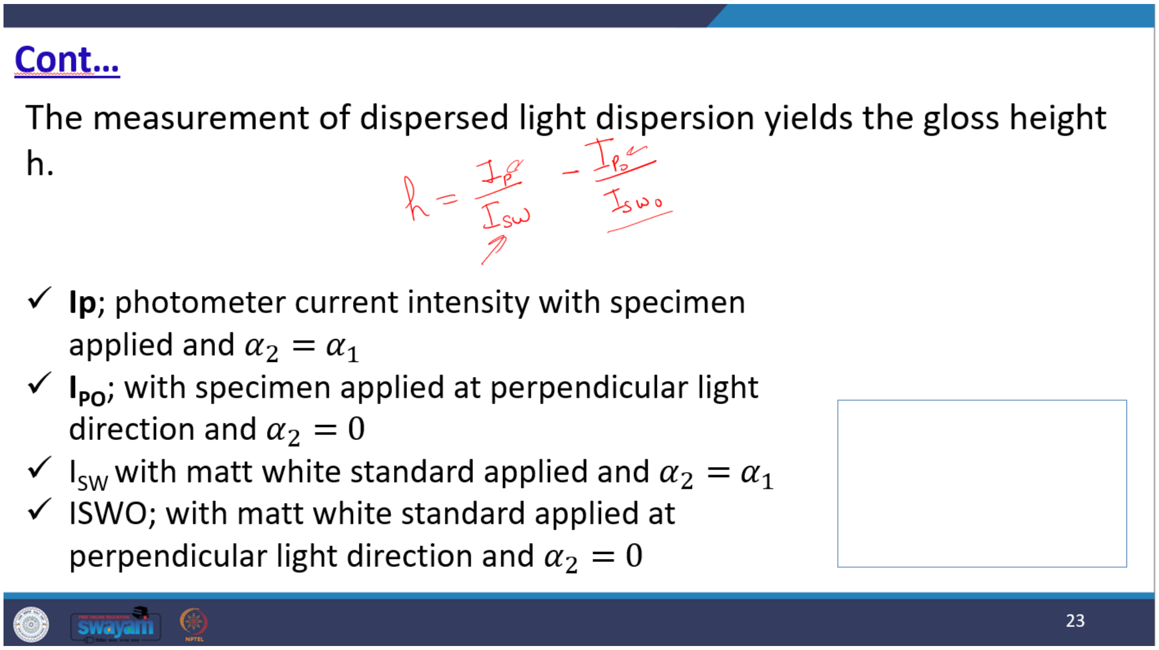
Because of this, the body's pigment helps to create the perception of color. Let us discuss the factors on which the glass generation in polymer and its evaluation depends. The specimen surface reflection and scattering characteristics are influenced by its structure roughness, curvature, and plane position. The incoming light spectral distribution intensity and geographical distribution. So, the observation angle and distance of the observer, the color and transparency of the specimen, and the color brightness and reflection properties of the surrounding surface were tested.



The measurement of dispersed light dispersion yields the glass height, say h, can be represented as

$$h = \frac{I_P}{I_{sw}} - \frac{I_{P_0}}{I_{SW_0}}$$

Ip; photometer current intensity with specimen applied and $\alpha_2 = \alpha_1$

I_{PO}; with specimen applied at perpendicular light direction and $\alpha_2 = 0$

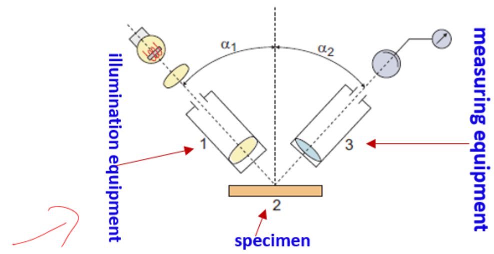
I_{SW} with matt white standard applied and $\alpha_2 = \alpha_1$

$\boldsymbol{I_{SW_0}}$; with matt white standard applied at perpendicular light direction and $\alpha_2 = 0$

Now, using the necessary goniophotometer under the angle α_2, the photometer current I p is determined. This is the schematic diagram of the goniophotometer for determining the glass height. A glass scale is created using the glass height, and it has a white standard with the glass of G=0 and a black standard made of polished black glass with a reflective index of n = 1.57 and a gloss of G = 100.

Cont…

- ✓ Using the necessary goniophotometer under angle α_2, photometer current Ip is determined.
- ✓ A gloss scale is created using the gloss height, and it has a white standard (tiff) with a gloss of G = 0 and a black standard made of polished black glass with a refractive index of n = 1.57 and a gloss of G = 100.

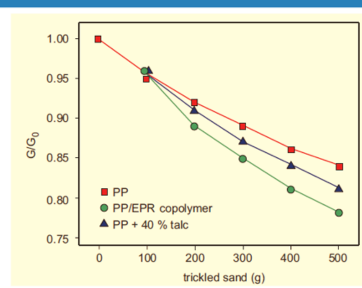


Schematic diagram of a goniophotometer for determining gloss height

Now, determining the surface roughness by glass measurement, glass value is a good tool for describing these surfaces because it heavily depends on the specimen surface roughness. Thus, this method can be used to evaluate the surfaces after testing for wear and scratch resistance. In the sand trickling test, the glass value g of a polypropylene material with varied scratch resistance is affected by the amount of sand used compared to the initial glass value (G_0).

➢ Determining Surface Roughness by Gloss Measurement

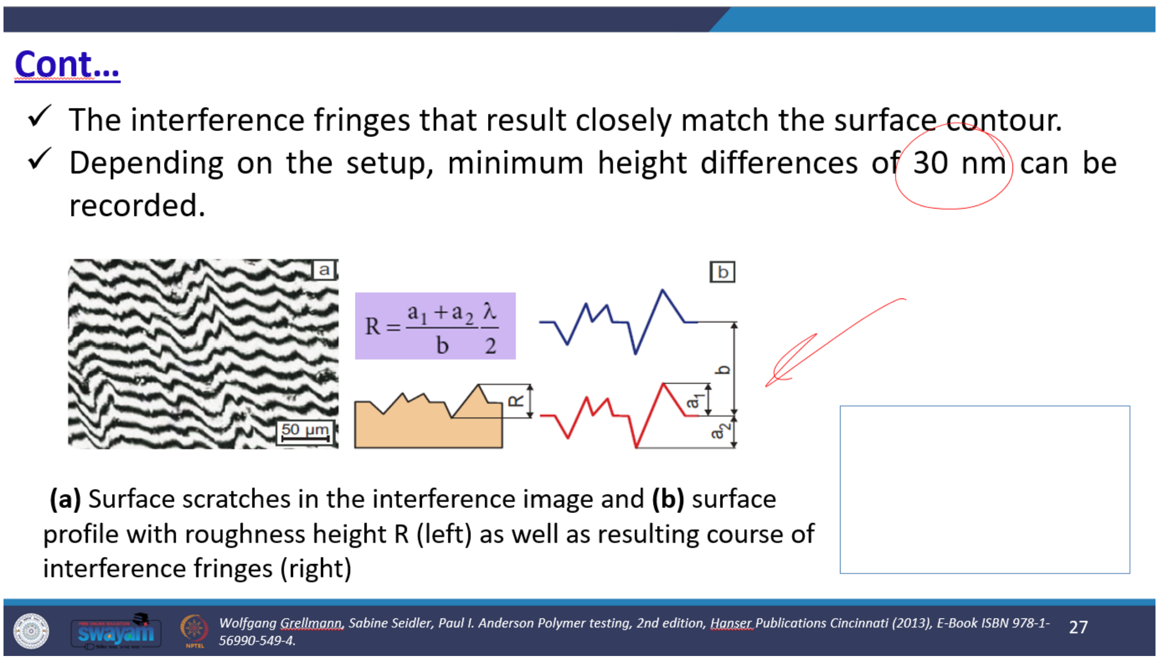
- ✓ Gloss value is a good tool for describing these surfaces because it heavily depends on specimen surface roughness.
- ✓ Thus, after tests for wear and scratch resistance, this method can be used to evaluate surfaces.
- ✓ In the sand trickling test, the gloss value G of PP materials with varied scratch resistance is affected by the amount of sand used, as compared to the initial gloss value G0.



Influence from sand-trickling on surface roughness and gloss value on sharp edge geometries

We are now determining the surface roughness using interference microscopy. We refer to overlapping two coherent light waves as interference and maxima and minima happen depending on the magnitude of the phase difference. The necessary retardation may be caused by minute height differences on reflective surfaces, inhomogeneities in a transparent material, and refraining in anisotropic phases. In the Tolensky interference setup, a very small air wedge is placed between a surface and a semi-transparent mirror in a microscope under the monochromatic light of a specific wavelength λ.

This is the typical figure of the surface scratches in the interference image and surface profile with the roughness height. Now, the interference fringes that result closely match the surface contour, and depending on the setup, a minimum height difference of 30 nm can be recorded.

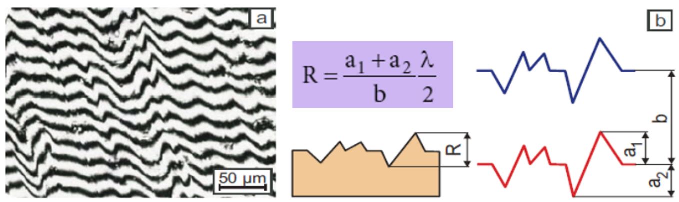


This demonstrates the interference picture of a scratched polystyrene specimen surface that had been gold coated in a vacuum before measurement to improve contrast. Metal interference filters are used to set a wavelength λ of the necessary monochromatic light. By tilting the interference mirror, the distance between the fringes can be altered, affecting the measurement precision. Measuring $\alpha_1 and\ \alpha_2$ fringe deflections with the microscope or on a photo is possible. By performing the differential interference contrast under the polarized light, it is possible to achieve very good three-dimensional contrast of slight surface height changes in the microscope. Now, black and white or a color variation or valence are created from a little variation in surface height.

When linear polarized light is employed, direction dependence develops, meaning that linear image patterns are suppressed in the direction that a Wollaston prism splits light. Circularly polarized light entirely eliminates this particular phenomenon.

Cont...

- ✓ The interference fringes that result closely match the surface contour.
- ✓ Depending on the setup, minimum height differences of 30 nm can be recorded.



(a) Surface scratches in the interference image and **(b)** surface profile with roughness height R (left) as well as resulting course of interference fringes (right)

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Let us talk about the color. The majority of the plastic is transparent or translucent, making it easy to color with the color of all intensities. The thickness, quantity, and distribution of the pigment in a final item and the processing all affect the color quality. The color of a surface has a significant impact on how it is perceived. Red, green, and blue are three primary colors of light. Their intensities are measured separately to determine the color of light. A matte white ball with an internally coated coating provides a diffused illumination to the surface. By employing an optical system with an optical wedge, the light that has been diffusely reflected is directed onto a photocell.

The intensities obtained are assigned to the color red (X), green (Y), and blue (Z). So, the equation we are going to write is used to derive the standard color x and y from these values,

$$x = \frac{X}{X + Y + Z}$$

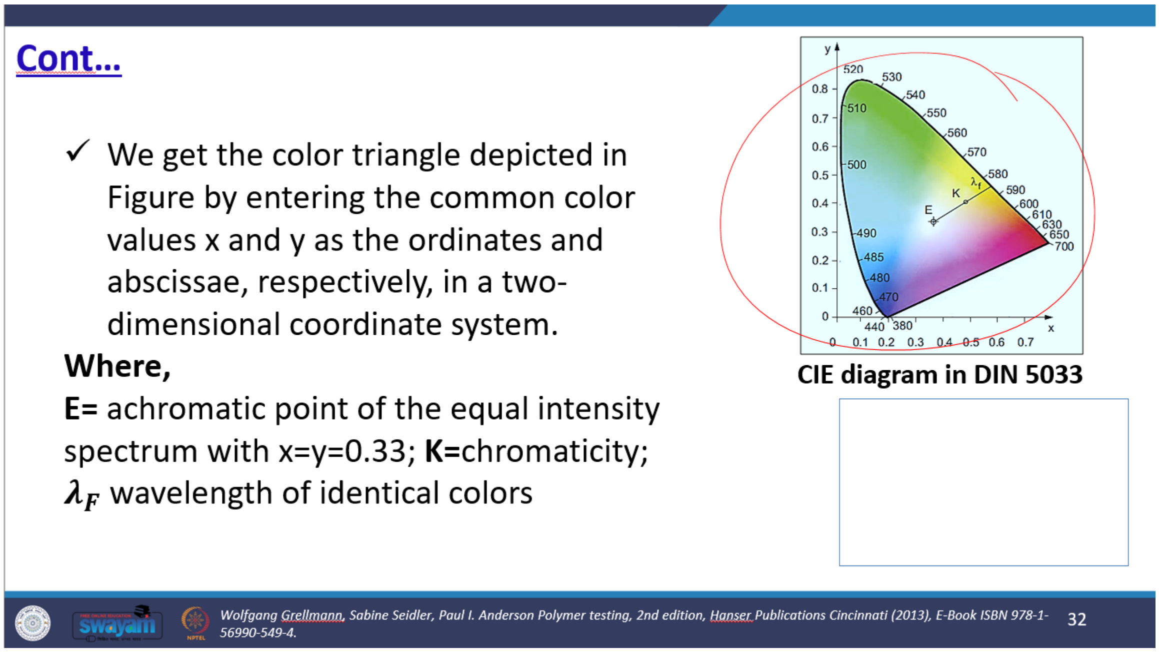
$$y = \frac{X}{X + Y + Z}$$

Now, we get the color triangle depicted in this figure by entering the common color values x and y as the ordinates in the two-dimensional coordinate system

Where,

E= achromatic point of the equal intensity spectrum with x=y=0.33; **K=**chromaticity; $\boldsymbol{\lambda_F}$ wavelength of identical colors

Now, measurement precision and reproducibility are strongly influenced by the nature and appropriation of a specimen. The representative object chosen for the mesh must be robust and small.



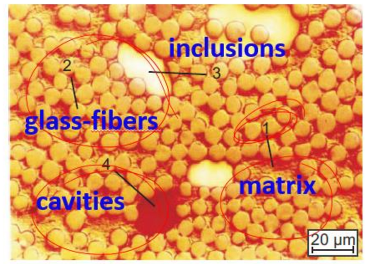
If the specimens exhibit orientations, they lead to dichromism. Now, color is processed in reflected light when tests are conducted on completely opaque material. Depending on the background choice, color registration issues happen when using this technique or translucent specimen.

Now, two procedures are to be used: (i) a color contacting agent is used to register the color value on a smooth white background. (ii) On a rough surface, color values are registered without the use of a color contacting agent.

Strongly scattering specimens are tested in reflected light mode with a white or a black background both with and without contact medium, while transparent weakly scattering specimens are measured in transmitted light.

Cont…

- There are numerous light microscopy techniques used.
- e.g., Figure illustrates distribution errors of glass fibers in a colored EP resin that were made apparent by polishing against the direction of fiber orientation.
- Here, a combination of the dark-field illumination of the microscope and the inherent color of the matrix material is combined to produce effective contrast.



Structure of a EP/GF composite perpendicular to fiber orientation, obtained in vertical illumination with dark-field illumination

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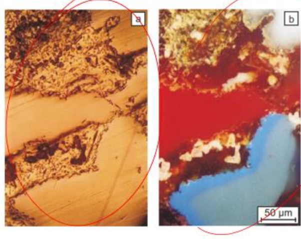
So, after being compacted in dishes, the powder or, grain or fibers are assessed for their color values. The color values of liquid are measured in cuvettes. The glass covers on solid porous or liquid samples can be problematic, and the amount of the value contamination that results is influenced by the cover materials' reflective index self-absorption and the glass thickness. The frequently ill-defined optical contact between the sample and cover leads to further inaccuracies. Then, talk about the color determination for filled polymers and polymer blends. Both material science and plastic engineering concentrate on the following structural factors to test the optical characteristics of filled and reinforced polymers like the orientation of the fillers and reinforcers filler or fiber distribution and agglomeration, as well as the influence on morphological formation in the matrix and various methods of light microscopy are utilized. Numerous light microscopy techniques are being used. This figure illustrates the distribution errors of a glass fiber-colored EP resin that were made apparent by polishing against the direction of fiber orientation.

You see that the glass fibers have different types of cavities, and this is the matrix. Now, here, a combination of a dark-filled illumination of the microscope and the inherent color of the matrix material is combined to produce effective contrast. In experiments employing the incoming light and the bright-filled illumination, the real colors for the specimen component cannot be reflected because the spectral makeup of the light changes under the microscope. Now, front surface mirrors are utilized in the dark field to illuminate without a spectral shift. The true specimen colors they are presented in this microscopic image demonstrate the variation in color representation using an unscratched polished piece of a recycled saw dust-packed polyolefin blend as an example.

Cont…

- ✓ The true specimen colors are presented in the microscopic image.
- ✓ demonstrates the variations in color representation using an unscratched polished piece of a recycled sawdust packed polyolefin blend as an example.
- ✓ Both a pure contrasting method and a way to identify colors can be employed with tiny lighting.



Polished section of a vertically illuminated sawdust-filled polyolefin material in **(a)** bright-field and **(b)** dark-field illumination

Now both the pure contrasting method and a way to identify colors can be employed with the teeny lighting. This is a polished section of a vertically illuminated saw dust-filled polyolefin material. This is the bright field, and this is the dark field illumination.

Let us talk about the infrared spectroscopy. Infrared spectroscopy is an absorptive spectroscopy technique that uses a wavelength between approximately 780 nanometers and 1 mm. The mid-range infrared spectrum, which encompasses wavelengths between 2.5 and 2.25 meters, is the most crucial for studying polymers. The term wave number is used to express the spectral range as this reciprocal of the wavelength is centimeter inverse approximately 4000 to 400-centimeter inverse in the wave number corresponding to the median infrared. The infrared spectrum absorption band may be related to the oscillation of specific valencies inside the polymer molecule to complete atomic groups that are functional groups.

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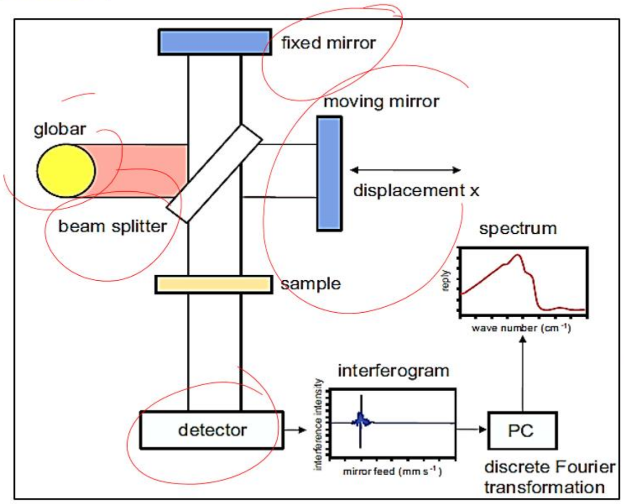

Configuration diagram of an FTIR spectroscope

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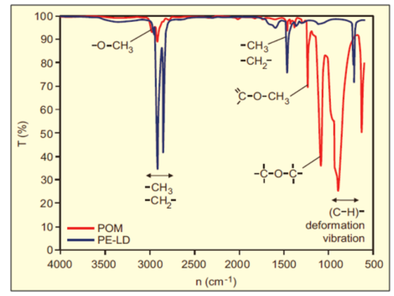
In light of this, infrared spectroscopy is an appropriate technique for examining the polymers and their additives. Using infrared spectroscopy, the qualitative and quantitative analysis is based on the wavelength-dependent interaction of infrared rays with molecules or a group of molecules. Now, this interaction generates distinctive bands in the absorption spectrum and IR spectroscopes; they are categorized based on how they choose wavelengths to analyze the light. The Fourier transform infrared and the dispersive IR spectroscope are the most popular. This is the configuration diagram of an FTIR spectroscope.

Detectors, beam splitters, GloBAR, Flix mirrors, and moving mirrors exist. So, this is the anatomy of FTIR spectroscopy. In transmission reflection, the test specimen's attenuated total reflection or ATR modes can be measured. This method can be applied to many films because the specimen thicknesses for transmission measurement range from 5 to 50 depending upon the IR transparency. The signal from the specimen that is either too thin or too thick is too weak for analysis. The fillers and colorant can only be identified if one particle size exceeds the resolution limit, and the particles can be pushed into the FTIR microscope measuring window.

Along with the trans illuminating the specimen, the material to be measured powder can be created under nitrogen cooling and crushed with KBR or NaCl powder as an IR ray neutral embedding substance. ATR, or reflection spectroscopy, is used to measure specimens that are too thick or unsuitable for transmission measurement, such as fiber-reinforced plastic. An IR neutral optically highly reflective crystalline substance, that is, a diamond, is softly pressed onto the surface of the ATR measurement. The total reflection can be seen at the edge layer at the right angle. Now, by using this technique, surfaces can be described without extensive preparation.

Cont…

- ✓ Total reflection can be seen at the edge layer at the right angles.
- ✓ By using this technique, surfaces can be described without extensive preparations.
- ✓ Figure displays characteristic spectra from a POM and a PE-LD that were produced using ATR.
- ✓ Different bands in the spectrum that are also referred to as fingerprints arise as a result of changes in chemical structure.



ATR-IR spectra of POM and a PE-LD

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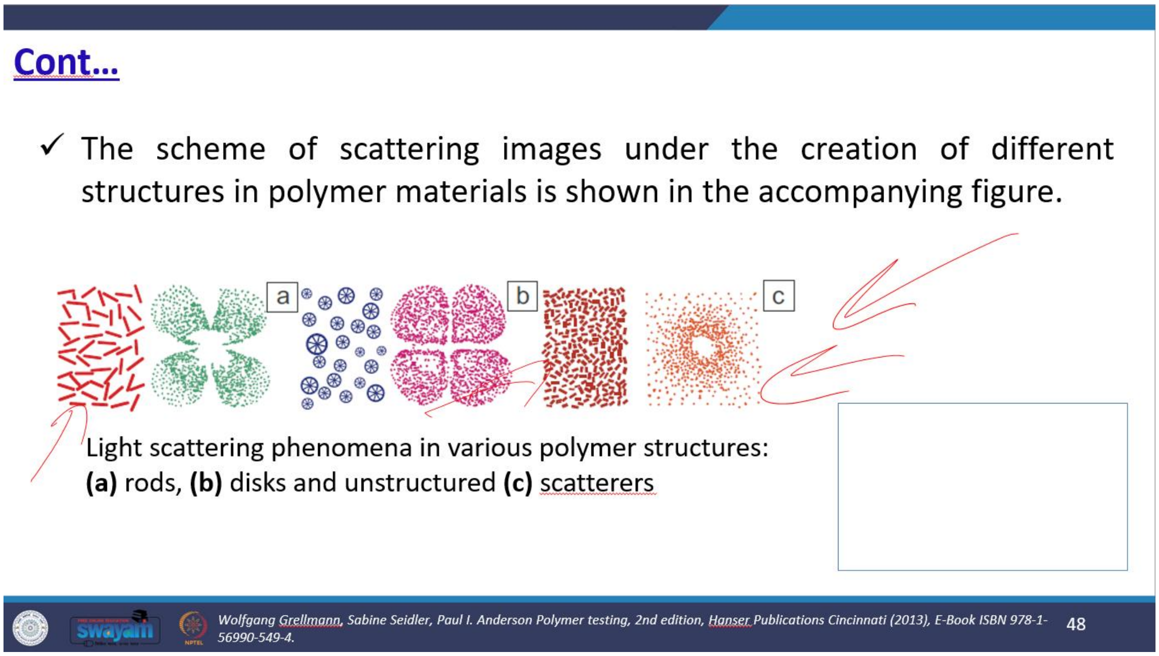
This figure displays the characteristic spectra from polymers or PE-LD produced using ATR. Now, different bands in the spectra that are also referred to as fingerprints arise as a result of changes in chemical structure. Let us talk about laser technology when using a light microscope tool on fine spherulitic semi-crystalline polymers with particle sizes between 100 and 5 mm. There are restrictions on how precisely one can determine the size, shape distribution, and anisotropy. For a structural component ranging in size from 100 to 10, these values can be determined using optical deflection with a linearly polarized light. The measurement is carried out using a separate optical laser light scattering apparatus or a light microscope with a laser or a linear polarized light from a strong commercial microscope illumination at a very small illumination aperture.



Now, with the aid of a Mekey Bertrand lens, scattering images that appear in the back lens focal plane can be captured on a conoscopic beam path, and afterward, a session on a photometer using the microscope heating stage and this method extremely high optical resolution measurements may be made as spherulites are formed from the melt. Additionally, it often has opinions on growth and nucleation at different cooling gradients. Now, you can see the light scattering phenomenon of various polymer structures like rods and another type of disc that is unstructured and scattered. So, this is the scheme of spreading the image under the creation of a different structure polymer material shown in this figure. The advantage of using a light microscope for a small angle light scattering is that it can capture the scatter and a microphotographic image of the specimen section that needs to be examined.

Cont…

The advantage of using a light microscope for small-angle light scattering is that it can capture scatter and microphotographic images of the specimen sections that need to be examined.

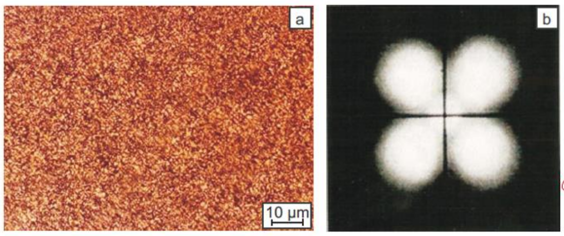


Figure (a) Shows the structure of PE-ND and **(b)** the corresponding scattering image.

(a) Structure image of a PE-ND specimen and
(b) scattering image taken with light microscopy

Now, this figure shows the PND structure and the corresponding scattering image. So, dear friends, in this particular segment, we conclude the polymer testing, and in this segment, we discussed the optical testing different forms of optical testing in a more specific way for the convenience of the student. We enlisted various references that you can utilize as per requirement. Thank you very much.