Attachment : 4003 Determination of Internal Stress for Glass Containers

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Internal stress refers to the residual stress inside the object when the external factor (force or humidity, temperature changes, etc.) is removed, which caused object deformed and induced internal force of the object that interacts between various parts to resist such external factors（also called permanent stress）. It is caused by the heterogeneous volume change of the macro or micro structure inside the material. If the uneven internal stress persists in a glass container, it can reduce the mechanical strength of the container, making it prone to cracking during production, use, and storage. Annealing is a technological process to eliminate or reduce the internal stress of glass containers. The measurement of internal stress is mainly used to control the annealing quality of glass containers for pharmaceutical Packaging.

Determination Principle：Usually, glass is an isotropic homogeneous material. When there is internal stress, it will exhibit anisotropy and cause light birefringence. This method uses a polarizing stress meter to measure the birefringence optical path difference, and the value of the optical path difference per unit thickness δ is used to represents the internal stress of the product. The measurement principle of birefringence optical path difference is that the white light emitted by the light source becomes linear polarized light after passing through the polarizing mirror. After the linear polarized light passes through the sample with birefringence optical path difference and quarter-wave plate, its vibration direction will rotate by an angle θ. The value of angle θ (the unit is degree) is directly proportional to the birefringence optical path difference T of the sample, and its formula is T=565θ/180=3.14θ. Thus, when there is internal stress in the glass sample, the angle θ can be measured by rotating the polarizer, and the birefringence optical path difference T of the sample can be calculated.

Instruments：The technical requirements for a polarizing stress meter include the following specifications: The brightness at the edge of the light field should not be less than 120 cd/m^2 when using polarized light elements and protective parts for observation. The polarizing elements must ensure that the degree of polarization is not less than 99% at any point within the bright field. Polarization field should be no less than 85 mm. A 565 nm full-wave plate (sensitive color plate) and a quarter-wave plate shall be between the polarizer and analyzer, respectively, with the slow axis of the wave plate is at 90°to the polarization plane of the polarizer. The analyzer should be able to rotate relative to the polarizer and the full-wave plate or quarter-wave plate, and it should be equipped with a device for measuring the rotation angle.

Determination: The samples should not have undergone other tests after annealing. It must be placed in the laboratory for more than 30 minutes under the laboratory temperature in advance. Gloves should be worn during the test to avoid contacting with the sample by hands directly.

1. Tests of the colorless sample

Inspection of the bottom of the colorless sample: Place the quarter-wave plate into the field of view, and adjust the polarization stress meter to zero point to present a dark field. Put the sample into the field of view and observe the bottom from the mouth. At this time, a dark cross will appear in the field of view. If the sample possesses small internal stress, the dark cross will be blurred. Rotate the analyzer to separate the dark cross into two arcs moving in opposite directions. As the dark area moves out, a blue-grey color appears on the concave side of the arc, and a brown color appears on the convex side. To measure the internal stress value of a selected point, rotate the analyzer until the blue-gray color of this point is just replaced by brown. Rotate the sample around the axis to find the point of maximum internal stress, then rotate the analyzer until the blue-gray color is replaced by brown. Record the rotation angle of the analyzer or the birefringence optical path difference at this time, and measure the thickness of the point.

Inspection of the sidewall of the colorless sample: Place the quarter-wave plate into the field of view and adjust the zero point of the polarization stress meter to make it a dark field of view. Put the sample into the field of view, so that the axis of the sample is at 45 °to the polarization plane. At this time, areas with different brightness and darkness appear on the sidewall. Rotate the analyzer until the dark areas on the sidewalls converge and just completely replace the bright areas. Rotate the sample around the axis to determine the maximum internal stress area. Record the rotation angle of the analyzer or the birefringence optical path difference of the maximum internal stress area, and measure the thickness of the two side walls (record the sum of the thickness of the two side walls).

2. Tests of the colored sample

The inspection procedure is the same as that of the colorless sample. It is more difficult to determine the rotation endpoint of the analyzer when there is no obvious blue-grey and brown color, and the glass transmittance is low. At this time, the average method can be used to determine the accurate endpoint. That is the average value of the total angle of the rotation angle (or the birefringence optical path difference) when the dark area replaces the bright area and the rotation angle when the bright area reappears.

Result Calculation

$\delta = T/t = 3.14\theta/t$

δ is the optical path difference per unit thickness, nm/mm. (used to characterize the magnitude of internal stress).

T is the optical path difference of the tested part of the sample, nm.

t is the total thickness of the light-transmitting part of the tested part of the sample, mm.

θ is the rotation angle of the analyzer (when the maximum internal stress is measured).

The 3.14 is a constant when a white light source (the effective wavelength is about 565 nm) is used, and 1° rotation of the analyzer is equivalent to an optical path difference of 3.14 nm.

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