Application Brief

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TMA Measurements of Glass

1. Introduction

Recently there has been an increase in developments of new types of glasses. Glass with peculiar characteristics such as photochromic glass, conductive glass and laser glass have been developed and some of these are already in commercial production. Unlike general chemical industry products, which usually require a number of manufacturing processes to obtain finished products, glass is produced by following a relatively simple process.

Various raw materials are mixed, fused, and then formed into finished glass products. In addition, glass has the major advantage of ready formation into free shapes.

Though the coefficient of expansion for glass is generally low, it is important from the quality control standpoint to accurately determine a glass's expansion coefficient and analyze the exact temperature levels at which its physical properties change.

This brief presents some examples of glass physical property measurements.

2. Expansion Coefficient Experiment and Results

Assorted types of probes are routinely used to measure the coefficient of expansion for various materials. h most cases a probe is applied to the sample and the variation in direct length is measured by various means; optical techniques, differential transformers or alternating optical interference patterns are measured (interferometer).

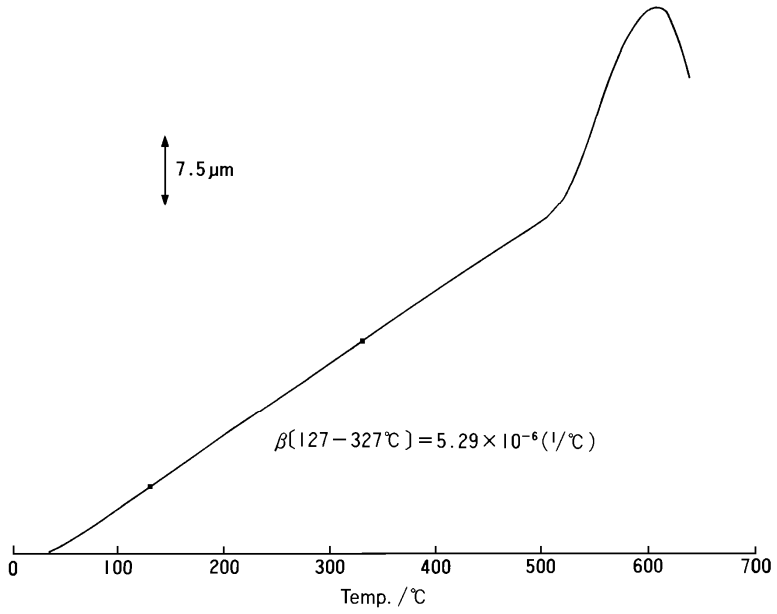

Figure 1 Expansion coefficient of SRM 731 glass

The TMA type of probe is just one of these probes but it is the most widely accepted method for measurement of expansion coefficients. The TMA method offers various advantages. One advantage is that precise sample geometry is not necessary and rough shaping is all that is required. Other advantages are smaller sample sizes (less than 20mm in length), ease of instrument operation, and much shorter measurement times.

Figure 1 shows TMA100 results for a NBS standard glass (SRM 73l, Borosilicate Glass). The mean coefficient of linear expansion value for the temperature range of l27℃ to 327℃ (400 to 600K) was calculated and determined to be 5.29×10^{-6}(l/℃). This value coincides up to the 10^{-7} order with the interferometer determined NBS value of 5.24×10^{-6}(l/℃). This evidence indicates that the TMA100 provides high-precision measurement of coefficients of linear expansion combined with extremely simple operation.

3. Strain, Softening and Glass Transition Point Experiment and Results

Normally, viscosity is used to define the strain point and Littleton point (softening point) of glass at 4×10^{-14} and 4.5×10^{-7} poises, respectively. The ASTM methods calculate these points based on the elongation rate of glass samples that are hung and heated gradually (ASTM C 336-7l and C 338-73). The disadvantage of these ASTM methods is that they require considerable time and labor due to preparation of samples of exact diameter and length and precise calibration before each measurement.

Figure 2 shows TMA300 results for an NBS standard glass (SRM 715, Alkali-free Alumina-Silicate Glass). The Curves are labeled (l) and (2) in the figure and show measurement results for two different measurements. Curve (l) data is after the sample was gradually cooled from 820℃ down to room temperature and then reheated at rate of 10℃/min. Curve (2) data is from the Curve (1) sample after being left in the furnace to cool down to temperature and then reheated at the same 10℃/min rate.

When a sample is left to naturally cool down in the furnace, a rapid temperature drop occurs in the region of the glass transition point. This leaves a strain inside the sample glass, causing it to become a quenched glass.

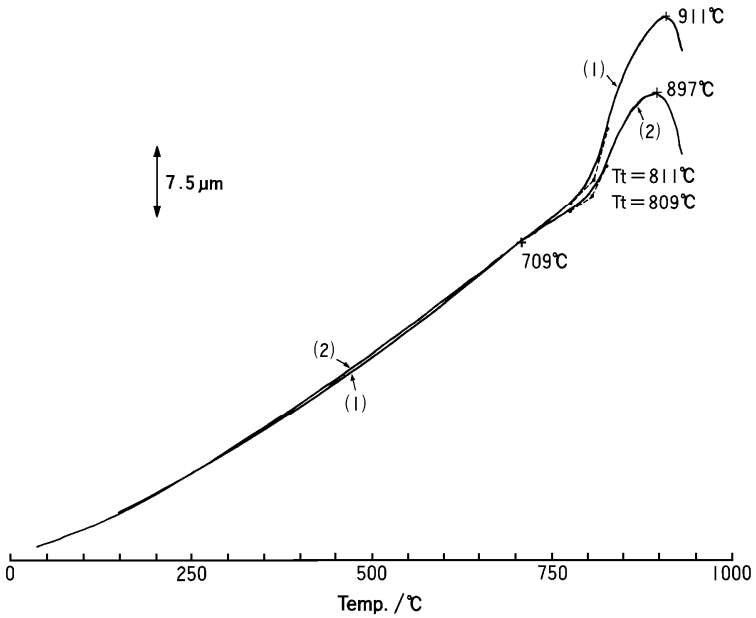

Figure 2 Gradually cooled glass(1) and quenched glass(2)

When the quenched glass curve (2) is compared with the gradually cooled glass curve (l), curve (2) shows slightly higher expansion than curve (l) in the low temperature region. Curve (2) crosses curve (l) at 709℃ and from this point on the quenched glass shows a lower expansion coefficient than the gradually cooled glass.

The relationship between quenched and gradually cooled glasses, described above, is a well known tendency and the temperature at the intersection of the two curves is known as the "Diversing temperature (Dt)". Reports state that the Dt appears at a temperature close to the strain point. The Dt shown in Figure 2 (709℃) is approximate to the strain point literature value (714℃) obtained using the ASTM methods.

If the softening point of glass is defined as the temperature at which the TMA expansion of that glass stops, curve (l) in Figure 2 shows that point at 911℃ for gradually cooled glass. According to the ASTM method, the softening point of grass relates to viscosity and is defined as the temperature level where the sample glass begins to soften at a determined rate. The literature value determined according to the ASTM method is 961℃. Better temperature control and accuracy is available with a TMA system, providing a temperature value at which the sample actually begins to soften.

Inspection of the TMA measurement data shows that the linear slope changes discontinuously. This is because the coefficient of expansion changes at the glass transition point. The data shown in Figure 2 indicates that the glass transition point for both gradually cooled and quenched glass samples is about 810℃. It has been reported that the strain in glass can be removed when the glass is held at a temperature 10 to 15℃ higher than its transition point for more than 15 minutes. This means that the glass transition point can be used as a guideline for heat treating quenched glass to remove strain (stress relief).

4. Summary

As shown in this brief, TMA systems provide relatively simple methods for analysis of the physical properties of glass. TMA should be an indispensable instrument for product quality control during manufacturing and heat treatment processes.