Reprinted from **OPTICS AND LASER TECHNOLOGY** Printed in England

Automating the measurement of the softening point of glasses using a gas laser

N. KAWANISHI, Y. MINEGISHI, H. ISHIZUKA, T. MINOSHIMA, T. KISHII

Viscous elongations of thin glass fibres have been automatically traced and recorded near their softening points using an optical, mechanical, and electronic method. The technique allows tracings to be obtained by applying external forces of less than 5 mg. Measurements were repeatable to within ±1–2°C; this repeatability was critically dependent on the uniformity of the glass fibre diameter.

Nomenclature

T_S	Softening point
$\bar{x}$	Average diameter of each fibre
σ	Standard deviation in diameter of each fibre
$\bar{x}_1$	Average diameter of upper half of each fibre
$\bar{x}_2$	Average diameter of lower half of each fibre
$\bar{T}_S$	Average softening point
σ_{TS}	Standard deviation in softening point

Determining the softening point of glass is a useful measure for quality control, practical application, and scientific characterization. The history and details of these measurement up to 1927 have been described by Littleton.[1] The American Society for Testing Materials has specified the softening point and the method of measurement in ASTM C338-57. Similar specification have been applied in Japanese Industrial Standard JIS R3104-1970.

Method of measurement

A glass fibre from 0.55 to 0.75 mm in diameter, with a diameter uniformity of ±0.01 mm, and 23.5 cm long was suspended vertically. The upper half of the fibre was heated by an electric furnace, the temperature of which was raised at a rate of 4 ~ 6°C min^{-1}. Viscous elongation of the fibre caused by the weight of the lower half of the fibre was continuously measured. The softening point was determined as the temperature of the furnace at the time when the elongation rate reached 1 mm min^{-1}. The viscosity of glass at the softening point was presumed to be 4 x 10^7 poise.

Elongation of fibres has, in most cases, been measured by using travelling microscopes or magnifying projectors. Both types of apparatus require manual operation or manual recording during measurement. Automating the measurement rejects human error and assures higher precision and better efficiency, both in factories and in laboratories.

The authors are with the Tokyo Shibaura Electric Company, Kawasaki, Japan 210. N. Kawanishi, Y. Minegishi, H. Ishizuka and T. Minoshima are in the Glass Engineering Division, and T. Kishii is in the R & D Centre. Received 15 September 1975.

Apparatus

An electric furnace with an aluminium-chromium thermocouple was constructed following ASTM specifications. Elongation of glass fibres was measured using the apparatus shown in Fig.1. A CdS photoconductive cell is mounted on a bench. This bench could be raised or lowered along a threaded steel column by an electric motor driven precision worm gear arrangement. Two limit switches prevented over running of the bench. The motor was actuated by a CdS cell via an ac switch consisting of a silicon controlled resistor and diodes. One rotation of the worm gear corresponded to a bench movement of 1 mm. The gear carried five pins equally spaced along its periphery. These pins closed a microswitch five times per rotation. The microswitch, when closed, temporarily short-circuited the thermocouple output.

The beam from a He-Ne gas laser was introduced into the bench through diagonal prisms and focused with a convex lens. After focusing, the beam was detected by the CdS

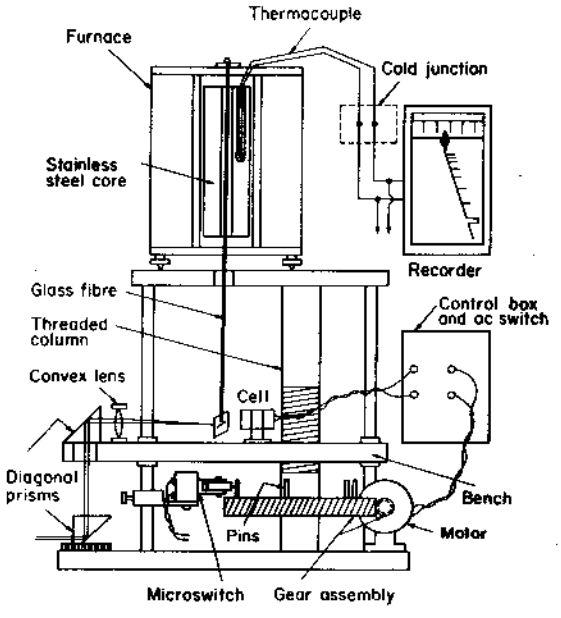

Fig.1 The apparatus

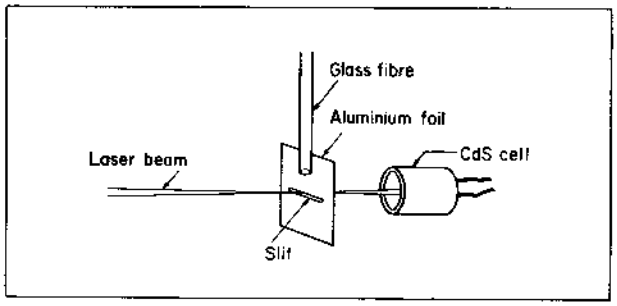

Fig.2 Arrangement of glass fibre, slit, laser beam, and photoconductive cell

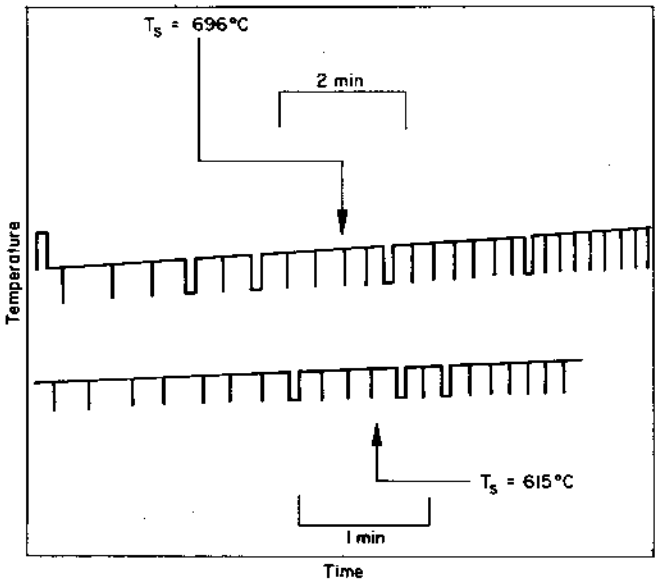

Fig.3 Chart recording traces

cell (Fig.2). The cell was fitted with a shield, a light diffuser, and an interference filter centred at the wavelength of the laser light. A sheet of aluminium foil 0.02 mm thick and about 7 mm square, in which was cut a narrow horizontal slit, was attached to the lower end of a glass fibre with a small quantity of glue. This foil was placed at the focal plane of the laser beam. At the start of measurements, the focal point of the beam was placed just below the slit. The force applied to the fibres during measurements was limited to a few milligrams.

As the furnace heated up the fibre elongated due to viscous flow. The foil moved downward, allowing the laser beam to pass through the slit. The beam actuated the motor, driving the bench downward until the focal point passed the slit. Thus, the focal point was always kept adjacent to the slit and the bench moved downward at precisely the same rate as the fibre elongation. The temperature of the furnace was continuously recorded by a chart recorder. The output of the thermocouple temporarily decreased when the microswitch was closed by the pins. This produced downward spikes on the temperature chart (Fig.3). One interval between traces corresponded to 0.2 mm of bench motion or to 0.2 mm of fibre elongation. The elongation rate was easily estimated from the chart recording. The temperature at which the rate was 1 mm min^{-1} was assigned as the softening point of the glass.

A control box allowed the following modes of operation:

1. Rapid heating of the furnace by an ac 100 V line or controlled heating by a voltage regulator.

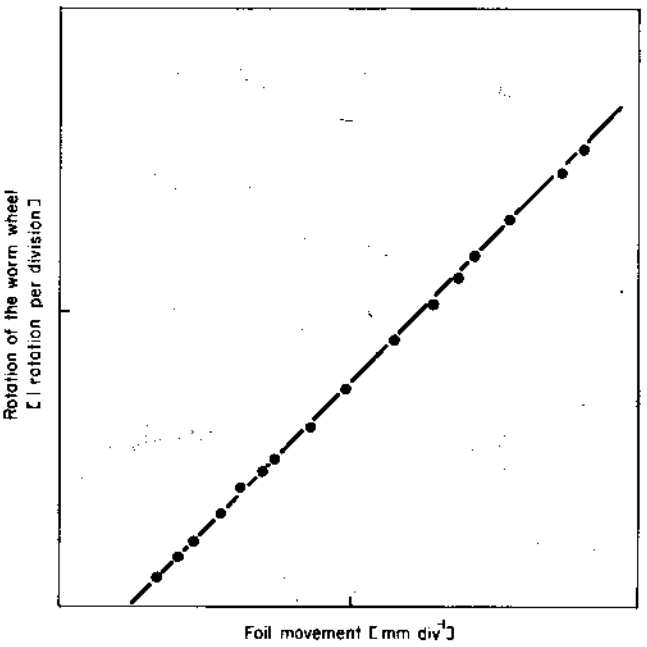

Fig.4 Relation between foil movement and worm wheel rotation

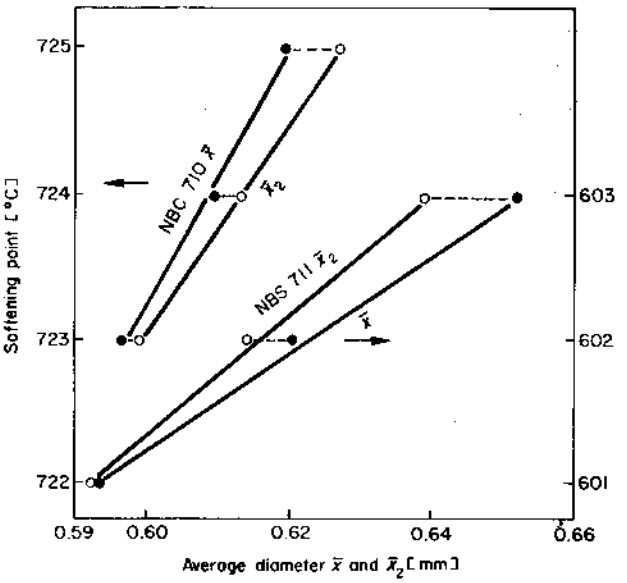

Fig.5 Effect of fibre diameter on softening point

2. Free motor running powered by an ac 100 V line or controlled motor running powered by the CdS cell and ac switch.

3. Upward or downward bench motion.

4. Limit switch release.

The thermocouple and the recorder were calibrated by the instrumentation office at the authors' factory.

Apparatus calibration

Fibre elongation tracing

An aluminium foil having a horizontal slit was moved vertically by a screw micrometer to simulate the movement caused by fibre elongation. The relation between the translations of the foil and the bench (Fig.4) was sufficiently linear for the purpose of accurate softening point measurements.

Effect of fibre diameter, diameter uniformity, and heating rate

Standard glasses, 710 and 711, available at The National Bureau of Standards, were used as samples. Nominal softening points are 624°C and 602°C, respectively. Fig.5 shows the effect of fibre diameter. A softening point change of 2°C corresponded to an approximate change of 0.05 mm in $\bar{x}$. Uniformity in x had a rather critical effect (Figs 6 and 7). The effect of heating rate was very small (Fig.8). Foil weight had very little effect (Fig.9).

Repeatability

Repeatability of the measurements was studied from the results shown in Table 1. The ASTM and JIS specifications forecasted the repeatability of ±1°C and ±2°C, respectively. The results were consistent with JIS, but σ_{TS}'s were not always less than 1°C.

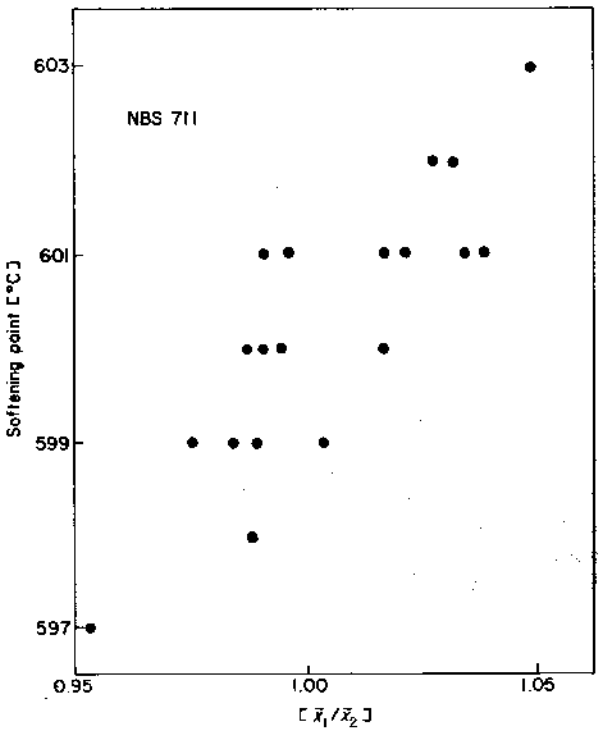

Fig.6 Effect of diameter uniformity on softening point (NBS 711 glass)

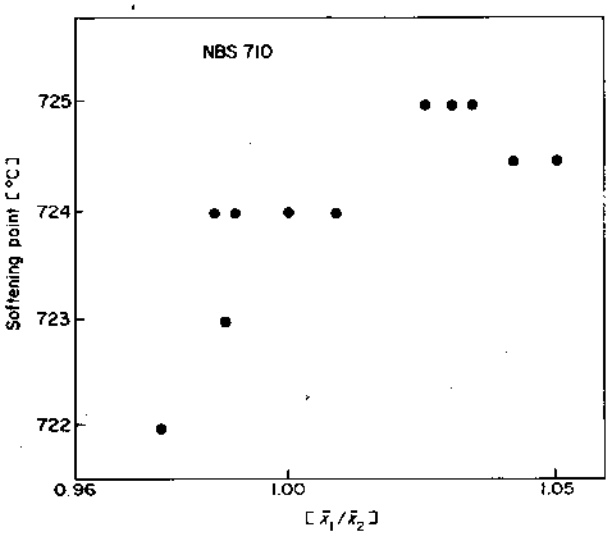

Fig.7 Effect of diameter uniformity on softening point (NBS 710 glass)

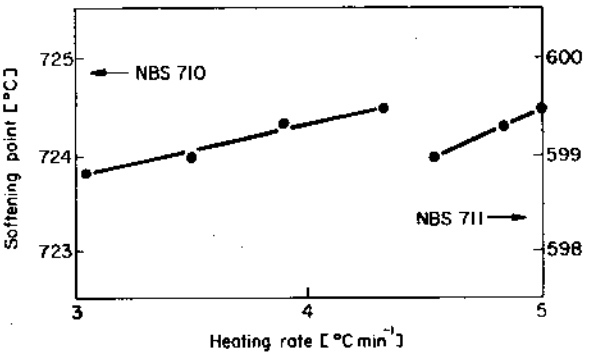

Fig.8 Effect of heating rate on softening point

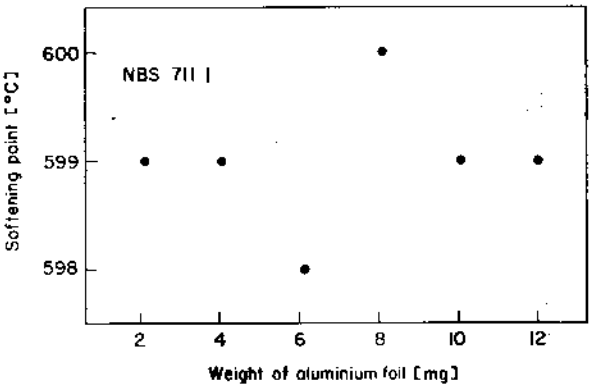

Fig.9 Effect of aluminium foil weight on softening point

The authors believe that the discrepancy was caused by a lower accuracy of the recorder in temperature measurement compared to a potentiometer.

Conclusion

Glass softening point measurements have been automated using a simple optical, mechanical, and electronic method. Measurement repeatability was estimated to be ±1–2°C. The results for NBS standard glasses gave values close to the nominal ones. The only drawback of the method was the use of a chart recorder for temperature measurement instead of a potentiometer. This presumably resulted in a slightly lower repeatability.

Reference

1 Littleton, J. T. *J Am Ceram Soc* **10** (1927) 259

Table 1 Experimental results on repeatability of measurements

Glass		NBS 711			NBS 710		
Experiment serial no.		1	2	3	4	5	6
Number of samples		10	10	11	12	11	12
$\bar{X}$ [mm]	min	0.632	0.587		0.587	0.613	
	max	0.683	0.660		0.681	0.667	
σ [mm]	min	0.010	0.013		0.005	0.011	
	max	0.019	0.045		0.018	0.033	
$\bar{x}_1/\bar{x}_2$	min	0.953	0.882		0.987	0.956	
	max	1.037	1.057		1.048	1.105	
Heating rate [°C min^{-1}]	min	3.94	4.55		3.05	3.05	
	max	5.76	5.00		3.89	4.12	
T_S [°C]	min	595	597	598	724	724	722
	max	601	604	601	725	728	724
$\bar{T}_S$ [°C]		599.0	601.8	599.6	724.1	725.2	723.0
σ_{TS} [°C]		1.94	2.10	1.03	0.88	1.58	0.84