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Surface modification of glass using xenon flashlamps

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An assessment study is reported using a xenon flashlamp of pulse length 1.8 msec and 25 J cm^{-2} energy for the beneficial modification of glass surfaces. Small diameter glass fibres (6 μm), preheated to 270° C, which fused and melted following flashlamp treatment, provided direct evidence that significant surface heating is possible. Scratches in bulk samples showed only limited evidence of surface healing when preheated to 500° C and flashlamp treated. However, bending and edge rounding of these samples provided indirect evidence for softening of a surface layer. A computer model was used to predict the effect of higher lamp power. The model data indicate that the system used would heat the surface significantly to a depth of 30 μm. However, the time duration, ~3 msec, is too short for significant flow with bulk samples. Extended to longer pulse lengths (20 msec) and higher lamp incident energies (50 J cm^{-2}), the model predicts similar treatment depths but the duration of near maximum temperature rise (300° C) is increased from 3 msec to 15 msec.

1. Introduction

Incoherent light as a continuous source of heating has many applications including medical heat treatment of muscle strains, drying of saturated liquids and paints, and warmers in the poultry industry. More specialized applications include light focused to fuse glass in the manufacture of glass to metal seals [1], and tungsten halogen lamps [2] to heat GaAs samples to 1000° C after 8 sec exposure. Flashlamp light sources, which provide, on a repetitive basis, much higher peak intensities than are available with continuous light sources, have a more recent history. In semiconductor processing, flashlamp annealing provides a viable alternative to conventional furnace annealing. Heinig *et al.* [3] and Bentini [4], using flashlamps to anneal ion-implanted silicon, predicted surface temperature increases of ~1300° C for incident light energies of 50 J cm^{-2} for 0.5 msec pulse duration. Loisel *et al.* [5] reported flashlamp recrystallization of large-area amorphous silicon films.

The capacity of xenon lamps to heat the surface of glass to temperatures sufficiently high for flaw healing to occur is experimentally assessed as a possible method for the modification of glass surfaces. In addition, a number of flashlamp parameters were measured and used with a computer model to predict surface temperature profiles as a function of lamp energy and pulse duration.

2. Absorption and emission characteristics

For satisfactory surface heating of glass and retention of bulk shape, the incident energy must be quickly absorbed in the first tens of micrometres of the surface. The output pulse characteristics of flashlamps [6] satisfy this short time criterion. However, the absorption depth and corresponding temperature rise will be determined by lamp energy, pulse duration and the relationship between the lamp spectrum and the glass absorption and heat-loss characteristics. Clear glass, transparent in the visible spectrum, absorbs in the infrared and ultraviolet as shown in Fig. 1a. As the

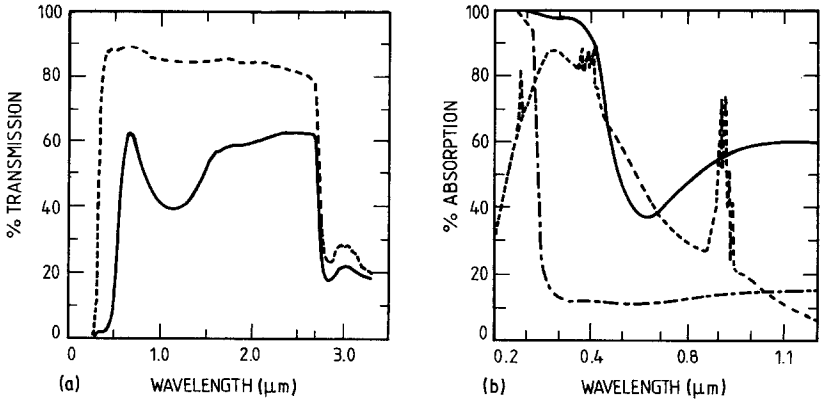

Figure 1 (a) Transmission intensity plotted against wavelength for (——) amber and (– – –) white glass. (b) (——) Amber and (– – –) white glass absorption and (– – –) flashlamp emission plotted against wavelength.

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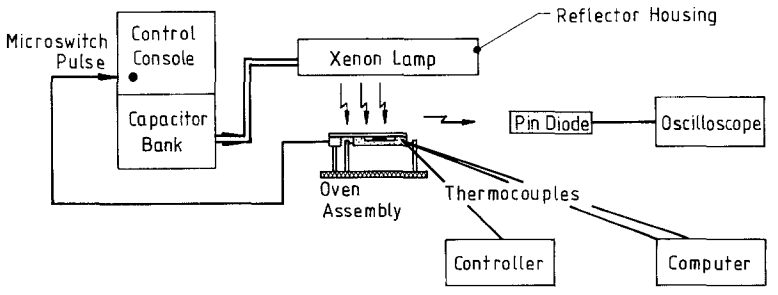

Figure 2 Schematic diagram of experimental apparatus.

flashlamp energy increases, the peak wavelength spectrum shifts towards the ultraviolet indicating a possible favourable match with the absorption spectrum for clear glass, which strongly absorbs below 0.2 μm. With amber glass, absorption is enhanced over a greater region of the spectrum (Fig. 1a). The optical intensity output from air-cooled flashlamps [6] having envelopes made from synthetic fused silica is shown together with the absorption characteristics for amber and white glass in Fig. 1b. The ultraviolet output would be absorbed in most glasses, whereas absorption of visible light would depend on the glass composition (Fig. 1b). The extent of this match of lamp emission and glass absorption characteristics for glass surface heating is significant.

3. Experimental methods

The flashlamp system consisted of a capacitor bank, flashlamp assembly (model 13F6, ILC Technology, Sunnyvale, California), preheating oven and monitoring equipment (Fig. 2). Maximum output into the lamp of 5300 J was achieved when the bank was charged to 2400 V. The lamp was located in a polished semi-cylindrical aluminium reflector housing, allowing the output to be brought to a line focus 45 mm from the lamp.

The flashlamp output was characterized using a storage oscilloscope to monitor the response of a fast PIN diode exposed to the lamp light by a partially reflecting mirror. Pulse width was constant at 1.8 msec over the range of bank voltages employed. A calorimeter was used to measure the lamp energy incident on a sample positioned at the focal plane of the reflector as a function of capacitor bank energy.

At the bank energy (corresponding to 2400 V) used in these studies the incident energy was 25 J cm^{-2}.

Using a simple oven, samples were heated to predetermined temperatures before exposure to the flashlamp. The oven, a 300 W nichrome heater winding embedded in a ceramic disc, surrounded by an aluminium sheath and incorporating a solenoid operated shutter/cover, is shown schematically in Fig. 3. When the solenoid is activated, the shutter swings aside exposing the glass samples and closes a microswitch to fire the lamp. The time lapse between the shutter opening and the lamp firing is less than 0.1 sec. The oven design ensures that the temperature difference between the bottom and top surfaces of the samples is $< 30°$ C at 500° C and that there is a minimal fall in the top surface temperature before the lamp is fired. Three Cr–Al thermocouples were used to monitor the sample temperature and control set temperatures: two embedded in the ceramic body (one connected to the temperature controller, the other computer logged) and the third (also computer logged) located on the sample surface. Temperatures were accurate to $\pm 5°$ C at 500° C.

Samples were prepared from 3 mm thick amber bottle glass, 1 mm thick microscope slides, 0.16 mm thick cover slides and 6 μm glass fibres. Bulk samples were usually cut into 25 mm × 10 mm rectangles and cleaned in ethanol. Scratches, $\sim 12\,\mu$m wide × $\sim 0.3\,\mu$m deep, were produced by a ruling machine operated with a 100 g load. Samples were generally examined in groups of three; untreated, oven heated and flashlamp treated (FLT). Two samples were placed on the oven ceramic surface (Fig. 3). When the desired set temperature was reached (about 1 min after switch on), the glass surface temperature was noted until equilibrium was reached (about 1 min). After 8 min at equilibrium, one sample was removed and following the re-establishment of equilibrium (about 0.5 min), the capacitor bank was charged to 2400 V and the remaining sample FLT. This sample was then removed and compared with the others using optical microscopy under interference contrast and interferometry conditions.

Rutherford backscattering (RBS), a nondestructive surface analysis method based on the rebound energy of incident 2 MeV helium ions [7], was used to determine if any changes in composition accompanied treatment.

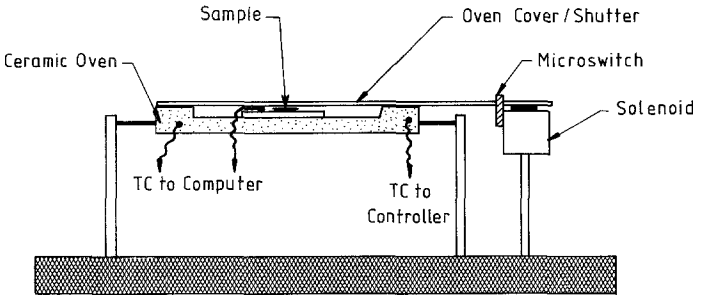

Figure 3 Schematic diagram of preheating oven.

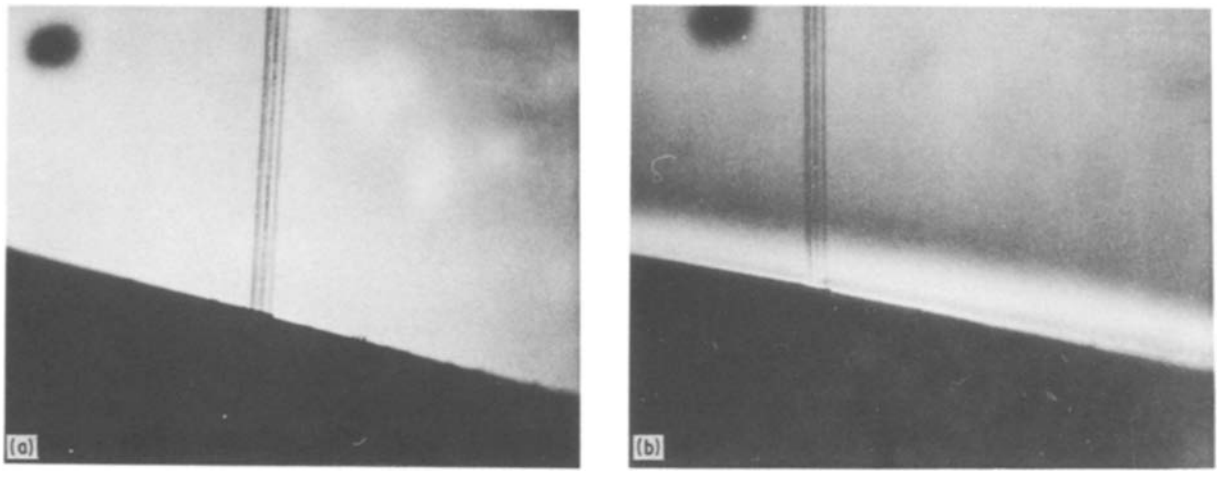

Figure 4 (a) Scratch near edge of microscope slide. (b) Same area as (a) following preheating to 460° C and flashlamp treatment.

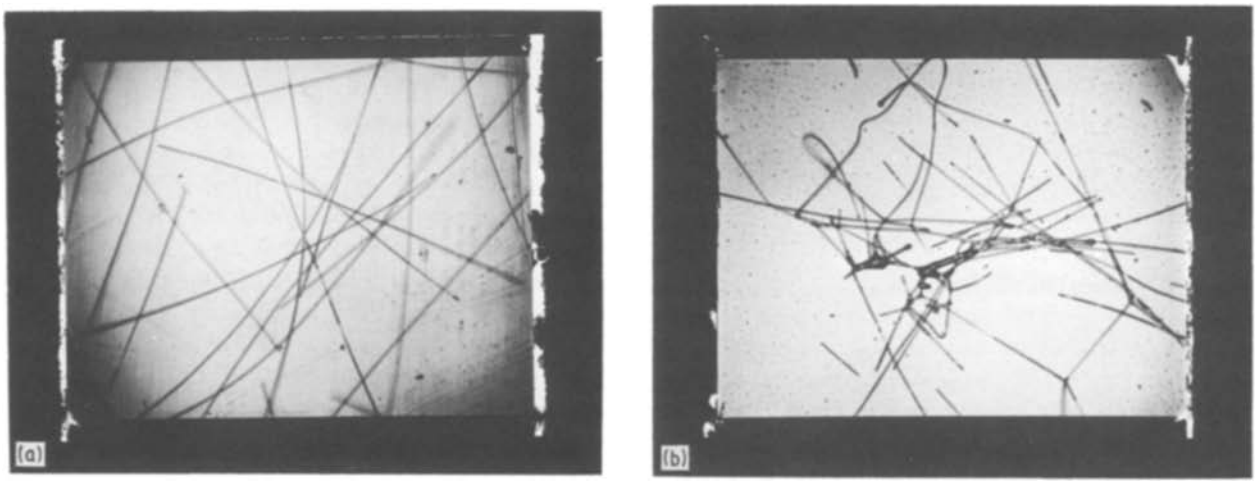

Figure 5 (a) Glass fibres, 6 μm, heated to 580° C for 8 min. (b) Glass fibres, 6 μm, heated to 580° C for 8 min, cooled, heated to 460° C and flashlamp treated. Same area as (a).

4. Flashlamp treatment results

The results are summarized in Table I. Surface rippling was observed for all bulk FLT glass samples preheated to 465° C. No change in the morphology of scratches in FLT microscope slides was observed until the preheated surface temperature was 460° C or higher. At these temperatures, scratches were smoothed in the region of the sample edge (Fig. 4), the surface developed ripples and previously sharp edges were rounded.

The results with amber glass were similar to those observed with microscope slides. Breakdown of the FLT sample surface into a scrapable powder occurred with samples preheated to 570° C (not observed with glass slides). The stronger absorption of the amber glass (Fig. 1b) may account for this.

An interesting effect was recorded with the thin (0.16 mm thick) cover slides. Below 500° C, surface rippling was the only observed difference between the preheated and the preheated/FLT samples. At 500° C, the FLT samples were bowed upwards on removal from the oven. This effect must be the result of differential thermal contraction implying a significant rise in the near surface temperature. No changes in scratch morphology were noted. The top and bottom surfaces of the cover glass slides, preheated (500° C) and preheated/FLT, were examined by RBS. No significant changes in surface composition were noted.

In summary, these observations with bulk FLT samples suggest a thin surface layer is heated for a short time to temperatures sufficiently high to generate change, perhaps by melting, but that these

TABLE I Summary of preheating and FLT sample results

Sample	Evidence for surface melting/softening	Minimum preheating temperature (° C)	Depth of melting/softening (μm)
Microscope slide	Rippling, edge rounding	460	< 0.1
Amber glass	Rippling, edge rounding	465	< 0.1
Cover slide	Concave bending	500	< 0.1
6 μm fibre	Fusing/beading	270	Through sample
2.5 μm fibre*	Fusing/beading	23	Through sample

*2.5 μm fibres received only cursory examination.

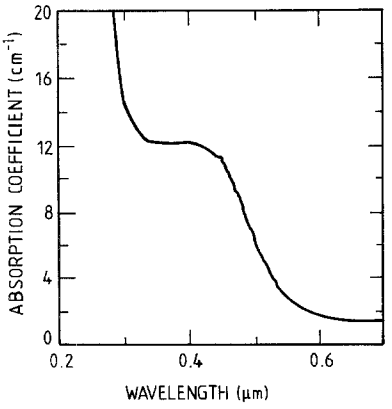

Figure 6 Absorption coefficient for amber glass plotted against wavelength.

temperatures are not sustained long enough to dramatically alter scratch morphology.

Finally, the effect of the outlined heating procedures on glass fibres was examined. Superficially coloured fibres, 6 μm diameter, were heated (oven surface temperature 580° C) for 8 min causing the colouring matter to burn off, but leaving clean straight glass rods with no signs of softening or fusing (Fig. 5a). The oven temperature was then reduced to 460° C and the fibres FLT. Melting and fusing occurred (Fig. 5b). Some fusing was observed for FLT fibres with final

TABLE II Input parameters used in computer modelling of flashlamp treatment

Thermal conductivity*	0.01 W cm^{-1} K^{-1}
Density	2.44 g cm^{-3}
Specific heat*	1.26 J g^{-1} K^{-1}
Latent heat of fusion	100 cal g^{-1} (1500 K)
Latent heat of vaporization	400 cal g^{-1} (2100 K)
Duration of flashlamp pulse	1.8 and 20 msec
Flashlamp intensity	25 and 50 J cm^{-2}
Absorption and coefficient	50 and 100 cm^{-1}

*Assumed no variation with temperature

preheating temperatures as low as 270° C. This clearly indicates that under the most favourable conditions, temperature rises in excess of 300° C can be generated.

For the glasses used here the onset of oven softening was observed at temperatures near 600° C.

5. Pulse heating model

A computer model developed for nanosecond pulsed laser heating studies [8] was modified to run at time scales in the millisecond regime and applied to the flashlamp system. In this model, all energy is absorbed in a surface region whose depth is determined by the value of the absorption coefficient. As shown in Fig. 1b, a substantial fraction of the lamp output lies in the ultraviolet where amber glass, in particular, is

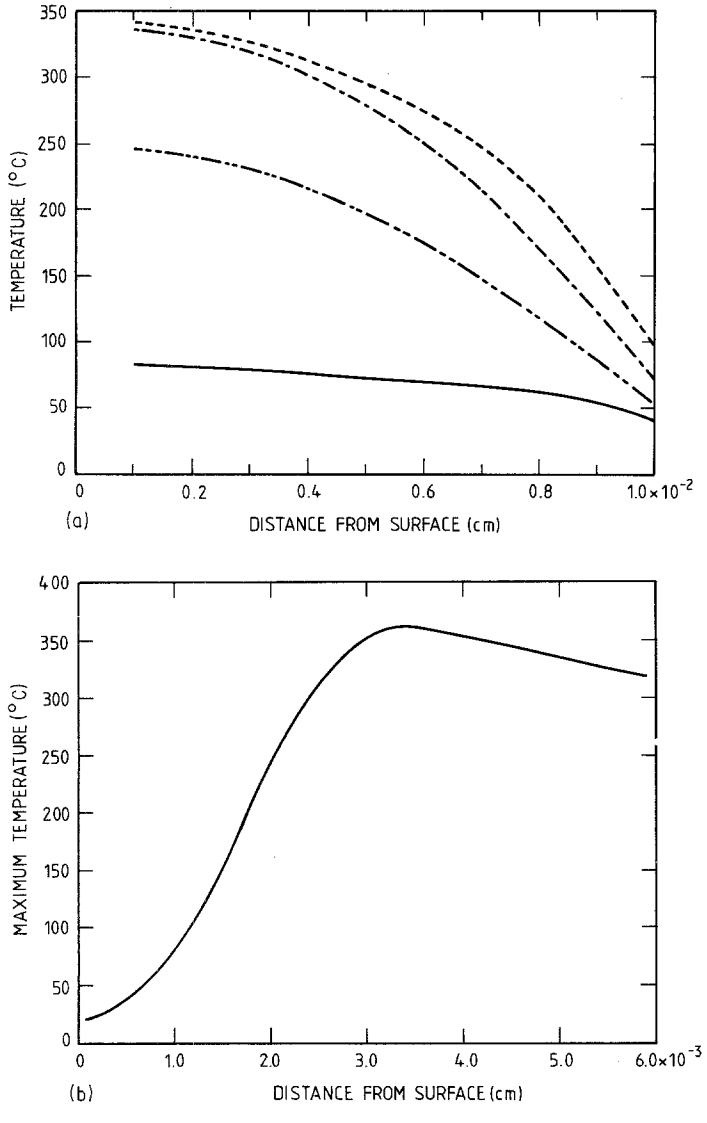

Figure 7 Computer calculated profiles for 1.8 msec pulse, 25 J lamp output and 50 cm^{-1} absorption coefficient. (a) Temperature against depth at increasing times from the start of the flash pulse. (——) 1.0 msec, (– – –) 2.0 msec, (– – –) 5.0 msec, (– – –) 10.0 msec. (b) Maximum temperature against depth.

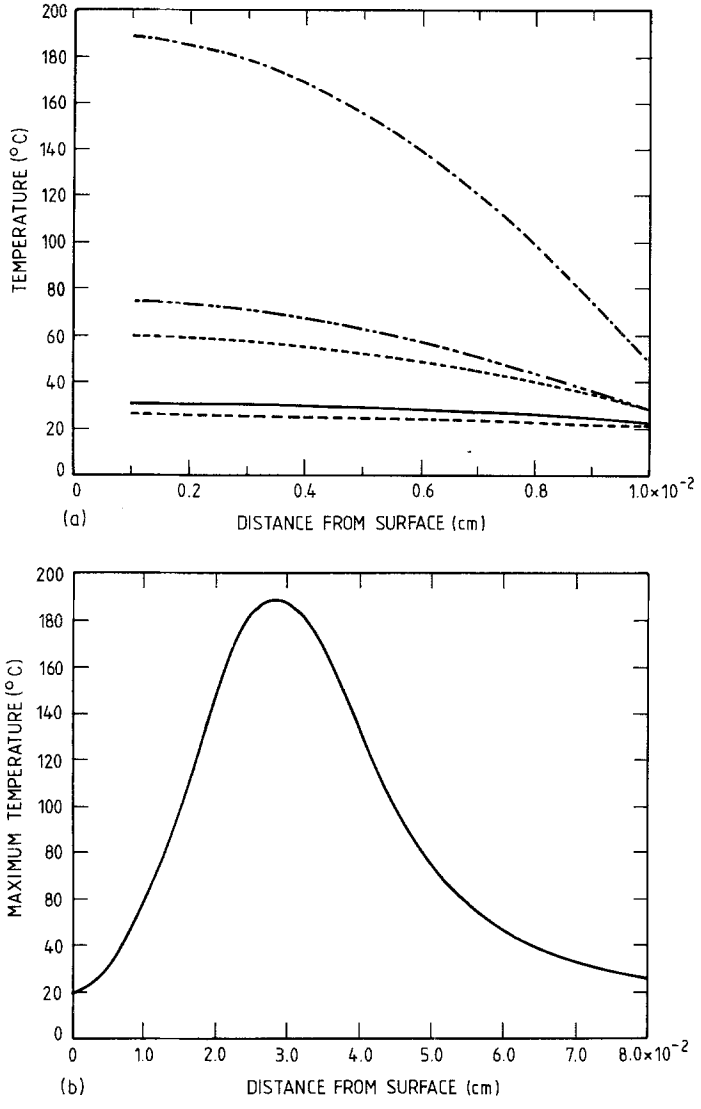

Figure 8 Computer calculated profiles for 20 msec pulse, 25 J lamp output and 50 cm^{-1} absorption coefficient. (a) Temperature against depth at increasing times from the start of the flash pulse. (——) 5.0 msec, (- - -) 10.0 msec, (— - —) 28.0 msec, (— — —) 50.0 msec, (— — —) 80.0 msec. (b) Maximum temperature against depth. Note increase in depth profiled due to longer pulse length.

strongly absorbing. These absorption characteristics for amber glass are presented in Fig. 6 as plots of absorption coefficient against wavelength. On the basis of the data two coefficients, 50 and 100 cm^{-1}, where selected for separate model calculations.

Calculated temperature profiles, using input parameters taken from the literature [9, 10] and listed in Table II, are presented in Figs 7 and 8, where the flashlamp pulse widths assumed were 1.8 and 20 msec, respectively, for a flashlamp energy of 25 J cm^{-2} and absorption coefficient of 50 cm^{-1}. In all cases the initial sample temperature was 20° C. In both figures, plots of temperature against depth at various times following the flash commencement (Figs 7a and 8a) and maximum temperature against depth (Figs 7b and 8b) may be comparatively assessed. If we assume, on the basis of the experimental results, that samples experience a surface temperature increase of 300° C, then the model provides satisfactory agreement (Fig. 7b).

Figs 7 and 8 also show that the maximum surface temperature occurs shortly after pulse completion. Near maximum temperatures extend to depths of about 30 μm; however, with the present 1.8 msec pulse system they are only maintained for about 3 msec (Fig. 7a). The model data suggest that if a 20 msec pulse were available, similar temperatures over similar depths could be maintained for 15 msec (Fig. 8b). Increasing the absorption coefficient from 50 to 100 cm^{-1} has the major effect of almost doubling the maximum preheated temperature (Fig. 9b) and highlights the importance of this parameter.

6. Discussion and conclusions

The flashlamp system used had inadequate power to remove defects from the surface of bulk samples. However, sufficient evidence of surface softening (Table I and Fig. 4) has been gathered to indicate that flashlamps warrant further investigation for the surface modification of glass.

Clear evidence of the capacity of flashlamp systems to melt glass comes from results with glass fibres where sufficient energy was available to fuse $\sim 6\,\mu$m diameter fibres with preheating temperatures as low as 270° C (Fig. 5). These samples lose heat (unless in good contact with the substrate) by radiation (negligible), air conduction and self conduction. Clearly the latter two conduction paths did not prevent the melting point of the fibre being exceeded. As the diameter of the fibre decreases, the fibre will, below some critical diameter, only be able to lose heat to the air. If this cannot be done quickly the fibre may vaporize. Some evidence for this phenomenon was noted with smaller diameter fibres ($< 3\,\mu$m) which were briefly studied.

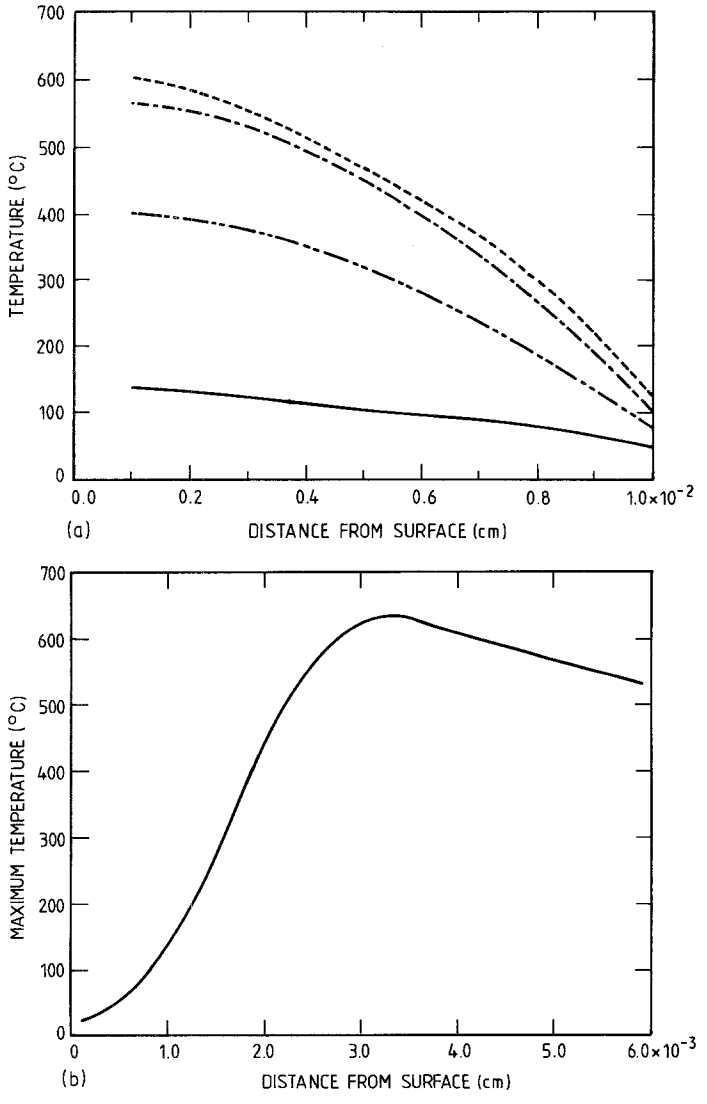

Figure 9 Computer calculated profiles for 1.8 msec pulse, 25 J lamp output and 100 cm^{-1} absorption coefficient. (a) Temperature against depth at increasing times from the start of the flash pulse. (——) 1.0 msec, (– – –) 2.8 msec, (– · –) 5.0 msec, (– · · –) 10.0 msec. (b) Maximum temperature against depth.

The bending of cover slides and edge rounding of other bulk samples is further evidence of surface softening occurring, albeit for a time too short for flaw healing.

The computer model provides general support for the experimental results and is the basis for predictive forecasts. The model indicates that with the power (25 J cm^{-2}) and pulse characteristics (1.8 msec) employed, softening will occur in a surface layer 20 to 30 μm deep. However, with bulk samples the short duration of maximum temperature (3 msec), would not be sufficient for extensive flow and flaw healing because surface heat is lost by conduction to the air and to the relatively large bulk of the glass. With glass fibres, where the surface-to-bulk ratio is such that only air conduction is important, the predicted surface temperature rise is supported by the experimental results which also imply temperature increases of 300° C or greater. Finally the model predicts that a flashlamp system with an incident energy of 25 J cm^{-2} and a pulse length of 20 msec would generate a temperature profile (Fig. 8a) of sufficient duration (< 15 msec) that annealing of surface defects to depths in excess of 30 μm is likely.

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