Research Report

The Use of Ultraviolet Lasers for Sealant Polymerization in the one Drop Fill Liquid Crystal Display Assembly Process

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ABSTRACT:

We describe experimental results comparing the use of pulsed ultraviolet lasers with ultraviolet lamps for assembling liquid crystal panels. Pull tests were also made to obtain data regarding the total fluence needed for UV lasers vs UV lamps. We found that the UV lasers are more efficient and the short UV laser pulses are advantageous in that heating of the liquid crystal is kept at a minimum due to the short pulse widths. Experimental data also showed that laser pulses lead to less shadowing of the portions of the panel that are shielded from the radiation that causes photoinititors to bring about polymerization of the glue seal.

Introduction:

To improve the manufacturing throughput of liquid crystal display panels, there is a trend to make use of the so called 'one drop fill' or ODF method. This method involves a substantial departure from the conventional process for constructing liquid crystal displays. In standard processing, a thermally activated sealant is applied near the entire periphery of one substrate except for a small section, then affixed to a second substrate to form the panel. The pair is baked at ~ 180C to cure the sealant after which the panel is filled with liquid crystal through the small section left without glue seal. The ODF technique, first introduced by Matsushita[1] consists in affixing the substrate containing thin film transistors (TFT's) to the color filter substrate after the liquid crystal, in the form of a series of precisely measured droplets, has been deposited on the color filter. This change in the manufacturing process avoids the tedious and time consuming effort required for filling the panel after the two substrates have been thermally bonded to one another. ODF also minimizes contamination inherent in the conventional liquid crystal filling process in which the panels are dipped into a container of the liquid crystal. On the other hand, the ODF process offers a substantial challenge in that the glue seal must now be cured rapidly, using a UV source and a glue seal containing photo initiators (or both photo and thermal initiators) to avoid intermixing of the liquid crystal with uncured glue seal material. Should intermixing between these two components take place prior to curing, serious performamce degradation of the display may occur either immediately or over time. To minimize curing time (and hence the time that intermixing might occur) we have experimented with several pulsed UV laser systems which have the following characteristics: 1) the UV output beam can be confined to the desired region, in this case, near the periphery of the panel. Shaping of a laser

beam to a desired dimension is generally routine and avoids unnecessary and possibly unwanted heating of adjacent regions that may not tolerate high temperatures; 2) the output UV intensity (W/cm^2/pulse) is much larger than conventional cw UV sources so that the total time for curing the glue seal of an entire panel with a high repetition rate laser may be reduced; 3) beam steering of a laser can be accomplished through the use of high speed scanning mirrors so that any glue seal pattern requiring UV curing can be readily traced; 4) as will be shown, very localized heating by the absorption of pulsed laser energy incident on a metallized portion of the panel can be advantageously utilized to enhance diffusion of photoinitiators underneath the 'shadowed' regions. The local heating enhances polymerization in what would otherwise be shadowed areas not capable of receiving incident radiation.

In our investigations we used two UV lasers, a pulsed XeF excimer (at 351 nm, manufactured by Lambda Physik, repetition rate up to ~ 50 pps) and a diode pumped, frequency tripled Nd^{3+}:YLF laser (UV output at 349 nm, repetition rate up to 10.05 kHz). Several UV lamp sources were also used, varying in intensity from ~6 mW/cm^2 to ~ 200 mW/cm^2 in order to compare data obtained with lamp and laser sources.

In this paper we report the results of experiments undertaken to examine the following: The comparative tensile pull strengths between two glass substrates affixed to one another by a droplet of one of several glue sealants, cured by UV lamp or UV laser irradiation; the degree of curing that occurs underneath metallization that causes optical shadowing of the glue; the effect on the curing process of adding thermal initiators to a sealant initially containing only photoinititors; the optical effects on twisted nematic liquid crystal near the glue-liquid crystal

interface (~ 4 x 4 inch test display panels) using ODF. Here observations were made with various glue seals after sealant curing with laser or lamp irradiation. One of the key differences among glue seals is the type of initiators used to promote polymerization. For the ODF process it is generally necessary to have photoinitiators in order to obtain photo polymerization of the glue seal. However, as will be shown, a combination of photo and thermal initiators, commonly referred to as a 'dual-cure' sealant appears to be especially useful for laser curing and in reducing the 'shadow' effect. This type of sealant made it possible to compare pull strengths between thermally cured (typically 120C) and photo cured samples using several UV sources including lasers.

Several sealants from different suppliers were tested, all containing proprietary chemical components and/or additives and hence, it is not possible to draw definitive conclusions concerning details of the polymerization reactions. Our experiments used two different UV pulsed laser sources to polymerize the sealants, a XeF excimer and a frequency tripled Nd^{3+}:YLF. The XeF laser has a wavelength of 351 nm with a repetition rate of up to 50 pulses per second with ~ 20 ns wide pulses. The laser output was focused to deliver between 30 and 60 mJ/cm^2/pulse over an area of several square centimeters. The second laser source, a Q-Peak Nd^{3+}:YLF laser is capable of delivering 0.5 mJ/pulse at a rate of up to ~ 10 kHz, pulse width 40 ns and a wavelength of 349 nm. The Q-Peak beam was always stationary, generally directed onto the samples without further focusing. In some experiments with this laser, samples were mounted on a translation stage capable of moving up to ~ 4 cm/s. The effective UV dwell time (laser Gaussian beam radius

~ 1.5 mm) incident on a substrate moving at 4 cm/s results in an effective incident fluence of

~ 5J/cm^2 . This is the approximate fluence normally used in manufacturing to cure sealants containing only photoinitiators.

With a mechanical shutter, it was possible to control the total laser fluence from the Q-Peak beam to very low values even when operating the laser at 10 kHz.This enabled pull test measurements to be made as a function of relatively low fluences. The data show that a considerably smaller total fluence than that typically used for UV liquid crystal panel sealing results in effective polymerization, determined by pull strength measurements as well as microscopic probing of the sealant after the samples were pulled apart.

Experimental Results and Discussion:

Pull Strengths

Shown in **Figure 1a** are values of tensile pull test strengths for a dual cure sealant (actually sold as a photocurable sealant but shown also to polymerize thermally as determined by scanning calorimetry) as a function of total incident UV fluence (J/cm^2). All pull tests for the present experiments used an Instron^{TM} pull tester with a special fixture to hold and position the samples for applied tensile force [2] . Samples consisted of two glass substrates, each 15 x 30 mm in areal dimensions, ~ 1.7 mm thicknesses, affixed to one another at right angles with a small glue spot, Figure 1b. The samples were clamped together until UV irradiation was completed. A comparison of the pull strengths is shown for two UV sources, a cw UV lamp stage with an output flux of 5.7 mW/cm^2, measured at the sample surface incident to the radiation, and the 10 kHz Q-Peak laser, ~ 0.5 mJ/pulse (equivalent to ~ 1.25 x 10^4 W /pulse, average power density

of ~ 1.8 x 10^5 W/cm^2 over the area corresponding to ~ 1.5 mm beam radius). For all fluences in Figure 1a, it is apparent that the pull strengths resulting from laser irradiation are considerably larger than those obtained with the cw UV source. It is widely known that pull strengths are influenced by the cross sectional area (CSA) of the glue spot. Generally, larger CSA's for a given sealant result in smaller pull strengths/area (the area of the glue sealant) due to stresses within the cured sealant. For the data shown in Fig 1a the UV lamp irradiated samples have a CSA of ~ 10 mm^2 while the effective CSA for the laser irradiated samples equals the size of the laser output beam directed onto the sample, ~ 6 mm^2 (the beam is not perfectly circular, rather slightly elliptical with major axes ~ 2.2 x 3 mm). Even though the actual initially uncured CSA's in some cases were larger than ~6 mm^2 , we found that only the 6 mm^2 spot was cured while the remainder outside the area defined by the laser beam area remained uncured. Therefore, for these laser pull strength measurements, a 6 mm^2 CSA was used .

Although the CSA'a for the samples cured with the laser and lamp irradiation sources are not precisely equal, these differences were not found to account for the systematically higher pull strengths of the laser irradiated samples. To illustrate the effect of glue spot cross sectional area we used the same glue seal to obtain a plot as a function of cross sectional area. Based on these data, it is reasonable to conclude that the difference in pull strengths for the two sources of UV irradiation shown in Figure 1a are not mainly attributable to differences in CSA but more likely to fundamental differences in the radiation sources and the subsequent interaction of the radiation with the sealant. A major difference between a UV lamp and the pulsed laser is the ability of the laser to deliver a very large photon flux for short durations which affects the

number of available free radical photoinitiators along with possible differences in reaction rates in the photochemistry of photopolymerization.

Some of the samples (Fig. 2) were pull tested within 1 hour after irradiation, (indicated by diamonds). Also shown are values of pull strengths for two samples tested approximately 21 hours after irradiation to determine the extent of latent curing, that is curing that occurs after the irradiation is terminated. As reported for some polymers, especially photoresists, there may be latent curing that occurs over a period of seconds or even hours after the radiation has terminated [3]. From our data, it would appear that latent curing is not a significant factor for this dual cure sealant. There may be some very long time effects that could occur over a period of days which generally, however are quite small and tend to take the extent of cure from 90% to nearly 100% (near full cure) [4].

Figure 3 shows a comparison between the pull strengths as a function of fluence for another photoinitator glue sealant for which the XeF laser and a high intensity UV lamp (200 mW/cm^2) were used. This glue contains photoinitiators but no thermal initiators. Here, again, the laser pull strengths are on average considerably higher for the laser than for the high intensity lamp irradiation. While the cross sectional areas are not indicated in this graph, the pull strength for the laser at a fluence of 4.5 J/cm^2, has a CSA ~27 mm^2 while the CSA value for the lamp irradiated sample at 6 J/cm^2 is ~20 mm^2 . These results give further evidence that while cross sectional areas do effect pull strengths, this parameter alone is not sufficient to determine why,

for most samples measured, the laser curing results in a stronger bond (higher pull strength) than samples cured by UV lamp irradiation.

The effect of adding thermal initiators (3% Trigonox 21) to this same glue sealant, initially containing only photoinitiators is shown in the pull test data, Figure 4. These samples were baked for various durations in a oven at 120C. It is clear from these data that sealants transformed to provide dual cure glue capabilities (that is, containing both photo and thermal initiators) are useful when both light and heat are available for the curing process. The data also show that a steady state (in terms of pull strength) is reached after ~ 30 minutes of baking while very little curing occurs in less than eight minutes. Pull tests of samples affixed with this same sealant, but with no thermal additives indicated no curing at 120C for one hour, the glue seal remaining liquidus. Thus, for this sealant without the added thermal initiators the application of heat (up to 120 C) in the absence of light does not result in polymerization.

Shadowing:

In liquid crystal panel fabrication, 'shadowing' refers to the inability of the photopolymerizing UV photons to interact directly with the glue seal due to opaque structures covering portions of the glue seal leaving the glue seal underneath partially or even totally uncured. UV radiation is generally blocked in the region where the metal gate and signal lines exit the interior of the panel to connect to the outside driving circuitry. A diagram of this portion of the thin film wiring, referred to as the fan-out region, is shown in Figure 5. Experiments have shown that for several glue sealants there is a greater degree of curing underneath the metal lines when a pulsed laser rather than a UV lamp is used for curing. This improvement in polymerization has been verified

both by pull tests, microscopic examination of glue seals after pull testing as well as by microscopic FTIR measurements [5].

The relative effectiveness of a pulsed laser compared to a lamp in curing a dual- cure glue seal can be considered in terms of heating effects that occur in the regions of metallization. Since fan-out thin film lines exiting the panel are numerous and closely spaced, it is difficult to calculate the temperature of a single line due to the incident radiation. However, an approximate temperature can be obtained and a comparison made between the UV lamp enclosure and a pulsed UV laser. We propose a model in which a major difference in the degree of polymerization of shadowed regions is due to the local temperature attained by the shadowed portion of the sample during the laser radiation process, a possible non-linear increase in the production of free radicals and an increase in diffusion (due to high temperatures) of radicals underneath the otherwise shadowed regions. For both UV lamp and Q-Peak laser samples, the temperature rise during optical radiation can be approximated using heat flow calculations published by Pittaway [6] for Gaussian beams incident on an absorbing surface in combination with the effects of 1-dimensional heat flow in a semi-infinite medium [7]. We have used the Gaussian beam calculations in two broad limits to apply to the lamp and laser cases. First, for absorption of the UV lamp radiation incident on the metal lines we assume a temperature gradient in the direction normal to the plane of the liquid crystal panel, i.e. the +/- **z** direction, to reach equilibrium during the radiation process. We assume that the lamp radiation causes a temperature increase of the fan out which is only a small fraction of the total panel area. The UV irradiating flux is ~ 6-8 mW/cm^2 typically for 600 seconds delivering a total of ~ 4.5- 4.8 J/ cm^2. The major portion of the panel is protected by a reflecting mask so that only the periphery

undergoes UV exposure. Masking also prevents the rest of the panel from any temperature increase when UV flood illumination is used. The thermal diffusion length in the panel is ~ $(\kappa t)^{0.5}$ ~ 1.9 cm where κ is the thermal diffusivity (5.8×10^{-3} cm^2 /s for crown glass) and t, the time for curing the glue seal (radiation flux of 6-8 mW/cm^2). Since the thermal diffusion length is large compared to the thickness of the panel (thickness typically ~ 0.15 cm), it is reasonable to assume that the temperature becomes equilibrated or uniform in the z direction in the region of shadowing long before the total irradiation time of 600 s.

To estimate the temperature rise of the shadowed region for this lamp irradiation , we made use of solutions published by Pittaway [6] for radial heat flow for a uniform absorbing surface (a metal thin film with an infinite absorption constant) in contact with the glass and glue to simulate the closely spaced fan-out lines. Since the fan-out lines are thin (~ 1000A Mo, with a reflection coefficient of ~ 50%) their thermal mass is negligible and can be ignored. We assume a single set of thermal parameters corresponding to that of crown glass [6]. Radial heat flow occurs in both the plus and minus z directions as the metal film is affixed on the underside of the upper substrate. Additional details of the calculations are found in the Appendix. Use is made of Eq. 9 of Pittaway [6] to determine the temperature at the center of the absorbed incident Gaussian beam irradiating for 600 s. In this approximation we assume the absorbing metal to extend on the order of 1 cm from the center of large Gaussian beam with radius of 1 cm simulating the uniform lamp flux. With this large beam radius the flux is almost constant over the fan-out area of the ~0.3 cm width. Using these parameters we find the maximum temperature rise of the absorbing film at the center of incident radiation is only ~ 1.5°C (with 50% optical reflection).

This very small temperature rise is expected to have no effect in activating thermal initiators nor in enhancing the diffusion of photoinitiators underneath the shadowed regions.

For comparison we consider the rise in temperature due to the pulsed Q-Peak laser on a region of metallization at least as wide as the near Gaussian laser beam diameter (~ 3 mm) to simulate the temperature rise of the fan-out lines. Since each pulse is only 40 ns in duration, thermal equilibrium is not attained in the z direction, even at pulse rates of 10 kHz. It can be shown from Eq 17 of Pittaway [6] that the center of the Gaussian irradiated metallization returns very close to ambient temperature after less than 1×10^{-4} seconds (the time between successive pulses). The rapid decay time implies that there is almost no thermal buildup from successive pulses (less than 10°C for 750 pulses for a dwell time of 75 ms, scanning at 4 cm/s) so that any significant temperature rise is due to each individual laser pulse. With the help of Eq. 15 of Pittaway [6], the temperature at the center of the irradiated spot increases by 80--150° C at the end of each pulse. This increased temperature is sufficient to enhance the diffusion of the photoinitiators that have been made active by the photons immediately adjacent to the shadowed region. The diffusion enhancement is caused by the expected lowered glue viscosity due to the higher temperature of the metal film and the region of glue adjacent to and in contact with the metal underside of the film. The increased temperature from the laser pulse is also sufficiently high to activate some of the thermal initiators (for a dual cure glue) immediately underneath the shadowed region, however we have no quantitative way of estimating the number of radicals excited in this manner.

The extent of enhanced diffusion is determined by Fick's second law,

$$\delta C/\delta t = D(T) \nabla^2 C \qquad (1)$$

where C is the concentration of the activated photoinitiators created next to the shadowed region. The intense pulsed irradiation causes large concentration gradients of photoinitiators immediately adjacent to the metallized regions which enhances diffusion.

The temperature dependent diffusion coefficient, D(T) for the present system is not available. However, diffusion of the radical photoinitiators created in the unshadowed region are expected to have a greater probability of diffusing underneath the shadowed regions due to the lowering of the glue viscosity since viscosity generally decreases exponentially with increased temperature.

We have found that the extent of uncured glue sealant underneath metallization depends very much on the particular sealant. In most cases we observed that pulsed laser irradiation leads to a more complete cure of shadowed regions, especially for relatively large shadowed areas. An additional advantage of the short laser pulses in the glue seal region is that the radial thermal spread is very limited. Thus the temperature at a distance equal to two Gaussian beam radii is only 2-3% of the temperature at the center. The rapid falloff with radial distance implies that the liquid crystal will not become heated substantially beyond two beam radii, minimizing possible thermal collateral damage to the panel. This analysis is found by using **Figure 6** (pg 976) of Pittaway[6]. The ability of the laser to cure very rapidly reduces the time for potential contamination between the glue seal and the liquid crystal. Such contamination can lead to

deterioration of the display quality at the glue-liquid crystal interface which tends to spread towards the center of the panel with time.

Testing of 4 x 4 inch ODF panels.

Twenty-four panels were assembled consisting of two glass substrates with the following elements: stripes of indium-tin oxide (ITO) to which external driving voltages can be attached; a thin alignment layer deposited on each substrate of the panel consisting of either ion beam treated diamond-like carbon (DLC) or 'rubbed' polyimide [8, 9] ; metallized edges deposited around the periphery of one of the substrates of the panel comprising differing metal/open area spacings where portions of the metallized patterns coincide with locations at which the glue seal is deposited; 5 micron diameter spacer balls to maintain separation of the two panel substrates: droplets of twisted nematic liquid crystal dispersed onto one of the two plates (ODF method) after which the two substrates are joined in vacuum. Curing of the glue seal utilized either ~ 8mW/ cm^2 UV lamp exposure for 10 minutes or the Q -Peak 10 kHz UV laser with the panels moving on a table at 4 cm/s providing a fluence of ~ 5 J/cm^2 along the glue seal. Three different glue seals were used, one photoinitiator glue seal and two dual cure sealants; Panels after lamp or laser curing were subsequently baked for 30 minutes at 120C and examined around their edges with crossed polarizers for optical imperfections near the metallization. Subsequently, the samples were thermally stressed by baking at 70C for over 600 hours and then reexamined. Defects around the periphery could be detected using a microscope containing rotatable crossed polarizers with the least number of defects observed for one of the dual cure sealants for both lamp and laser curing. Simultaneous experiments to test the extent of glue

curing under shadowed regions using similar metal patterns in the aforementioned panels utilized micro-

FTIR [5] spectroscopy. The FTIR data revealed that shadowing effects on the glue underneath the largest metallized region are minimized with Q-Peak laser curing compared to several other UV lamp sources. Details of the FTIR results as well as the 4x4 panel data will appear in separate reports.

Conclusions:

We have shown that pulsed UV lasers can be used effectively to cure sealants used for liquid crystal panel assembly. In general we find that for most glue seals studied, the laser is efficient in curing the sealant leading to relatively stronger pull strengths compared to sealing with UV lamp radiation. For dual-cure sealants, the laser is generally more effective in preventing shadowing underneath metal data/gate lines. A further advantage of laser curing is the lack of heat spread to adjacent regions thereby minimizing the possibility of contamination between the liquid crystal and the glue seal. These features as well as the ease with which laser beam steering can be implemented make laser curing especially attractive for the ODF process.

Acknowledgments:

We are grateful for excellent assistance from Eric Liniger and Eileen Gallagher. Also, we wish to acknowledge advanced copies of the not yet published FTIR data from Chen.Cai and David Medeiros.

Appendix:

Equations used from Pittaway, Ref 6): 1) For the uniform temperature in the z direction, resulting from long time irradiation (600s) giving rise to thermalization, we utilize Eq. 9 pg 971:

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where T is the temperature increase

K, the thermal conductivity

D, the thickness of the panel

E_o, the total power of the Gaussian beam

$\lambda = \kappa\gamma t$ where κ is the thermal diffusivity, $\gamma = 1/d^2$ where d is the Gaussian radius and t is the time of the irradiation by the UV light source, UV light absorbed 50% by the thin film coating.

To determine the temperature for a short pulse of the Q-Peak Gaussian laser, we use Eq. 15 on page976:

$$TKd/E_o = 1/\pi^{3/2} \tan^{-1} 2\lambda^{1/2}$$

Equation 17 to calculate cooling after the radiation source has been turned off, page 977:

$$TKd/E_o = \frac{\tan^{-1}[\lambda^{1/2} - (\lambda - \lambda_1)^{1/2}]}{\pi^{3/2}[\lambda^{1/2}(\lambda - \lambda_1)^{1/2} + 1/2]}$$

Here $\lambda_1 = \kappa\gamma t_1$ where t_1 is the pulse width and t is the time between pulses or cooling time. κ and γ are defined as before.

Figure 6 of Pittaway has been used to determine the radial falloff in temperature away from the center of the beam.

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Figure Captions:

1) a) Pull strength of samples each consisting of two pieces of glass attached with a dual cure sealant and polymerized with the Q-Peak laser and inside the lamp enclosure (5.8 mW/cm^2 incident flux) as a function of total fluence. b) Schematic of two glass samples positioned for pull testing.

2) Pull strength for UV lamp cure (~5.8 mW/cm^2 incident flux) of the glue seal of Figure 1 as a function of the sealant cross-sectional area (CSA). Also shown is the absence of any significant latent cure.

3) Pull strength for a glue seal containing only photoinitiators, as a function of total fluence. Data are for two UV sources of irradiation: (1) XeF laser with 30mW/cm^2 (2) 200 mW/cm^2 UV lamp; both with incident flux over several square centimeters.

4) Pull strength data for the glue seal of Figure 3 containing thermal initiators through the addition of Trigonox 21, cured at 120C in an oven. Without added thermal initiators, there is no oven cure at 120C in the absence of UV radiation.

5) A schematic of the fan-out region capable of absorbing UV radiation in the open portions while opaque to UV radiation in other parts causing shadowing. The region in which the glue seal is coincident with the fan-out is shown.

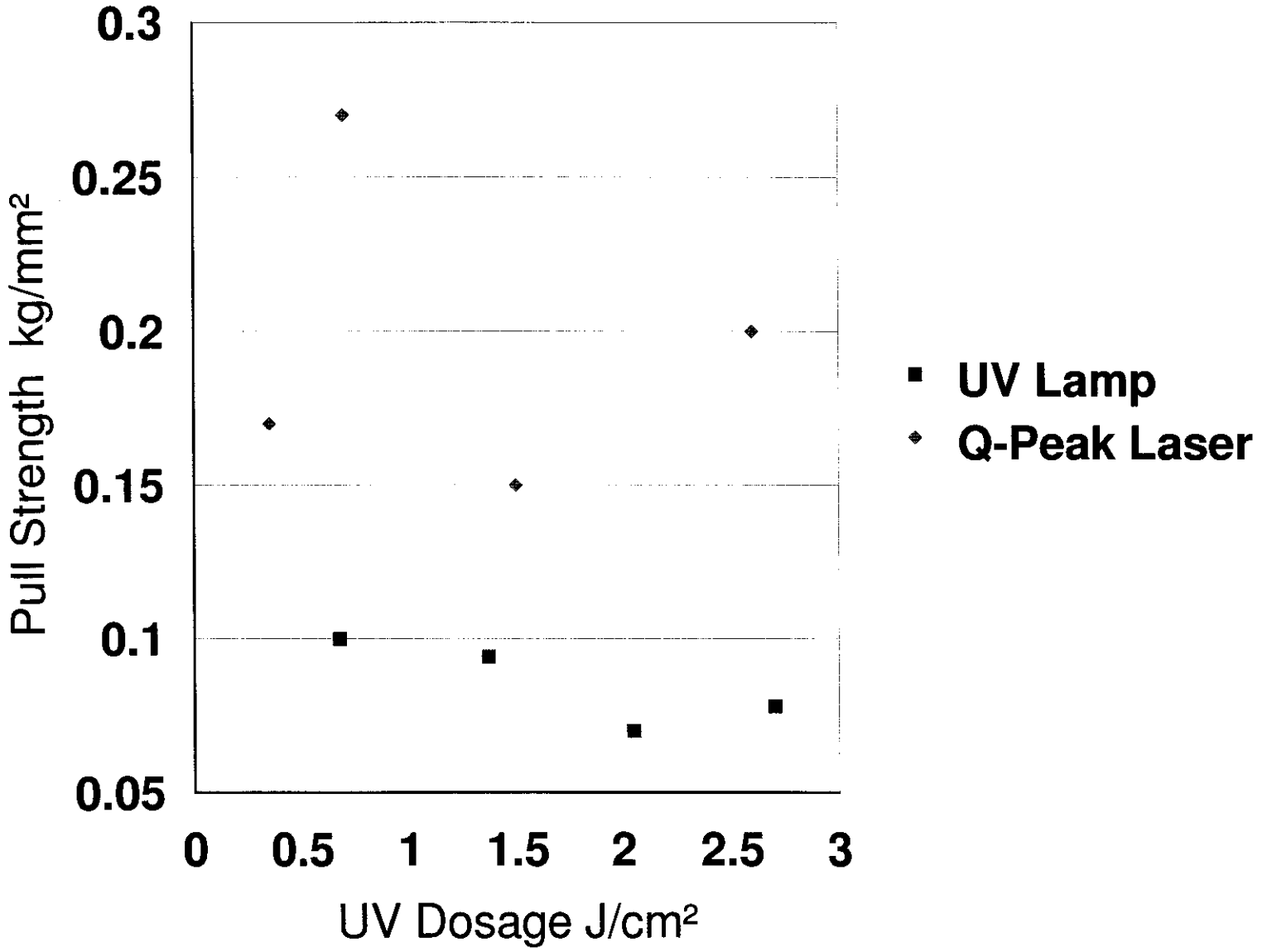

Fig. 1(a) Pull strength vs. UV dosage for lamp and Q-Peak laser irradiated glue seal samples. The CSA for the lamp irradiated samples was 10 mm² and the CSA for the laser irradiated samples was 6 mm².

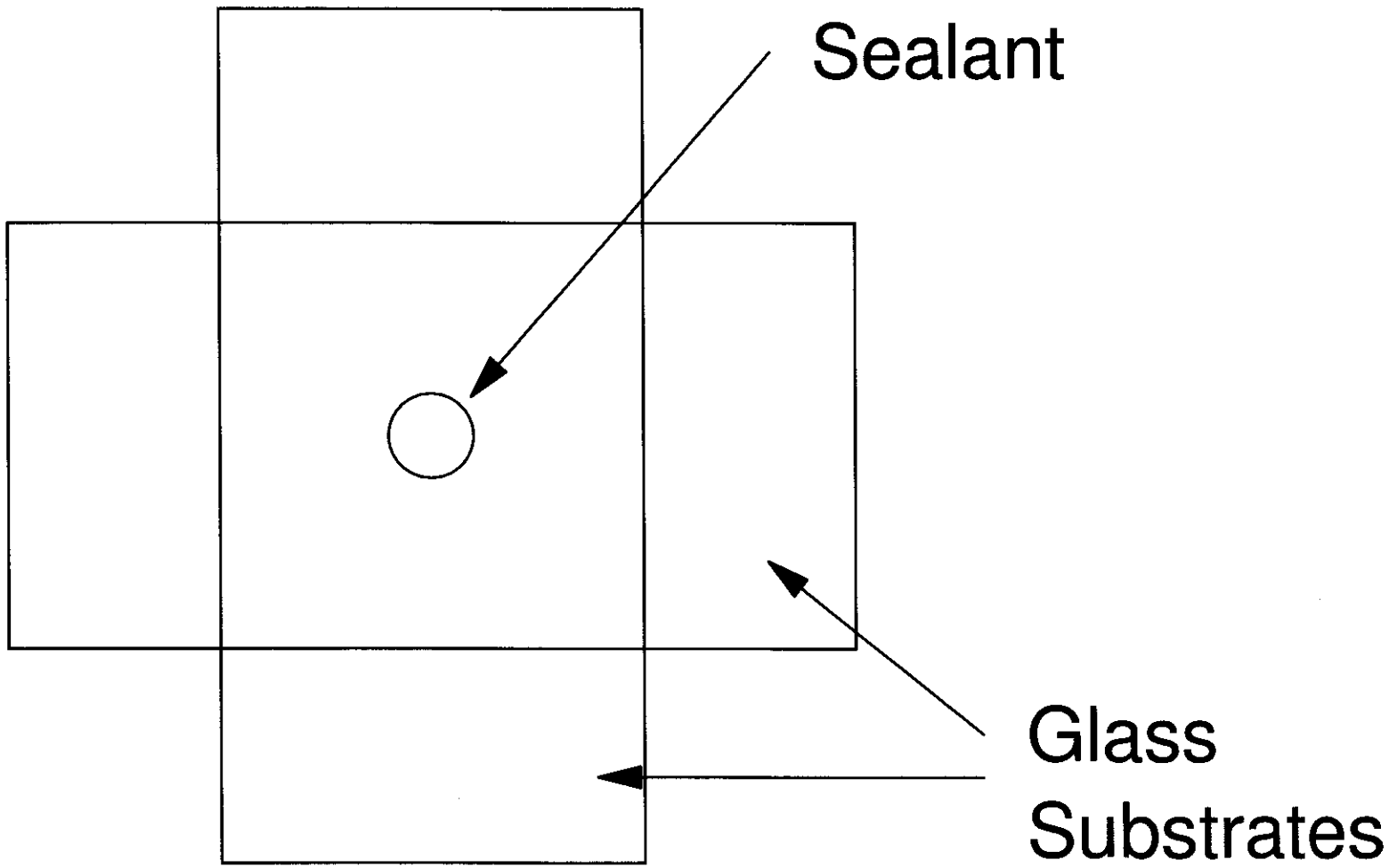

Fig. 1b Two glass slides for pull test

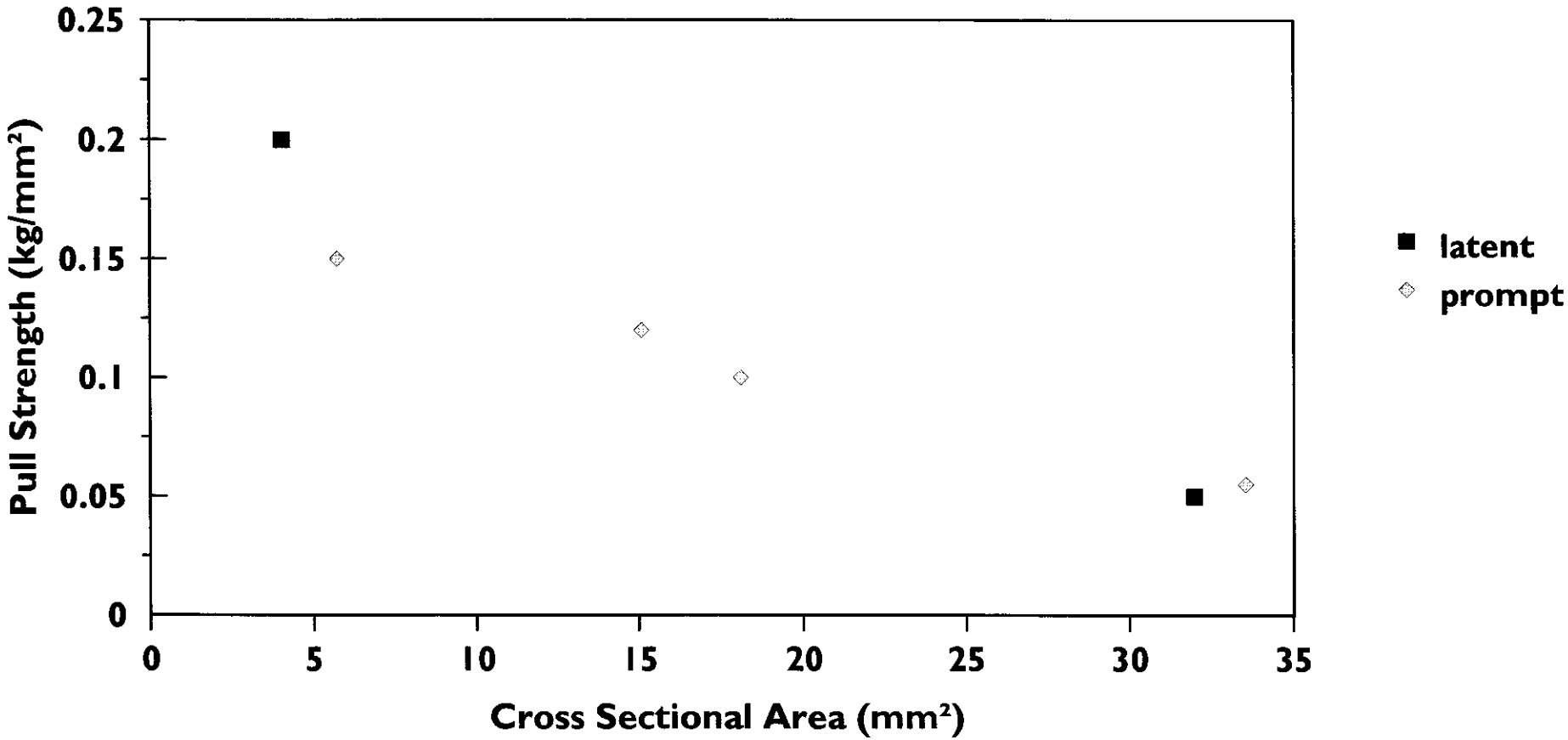

Fig. 2 UV lamp irradiated latent cure investigation of a dual-cure adhesive. Prompt samples are pull tested 1 hour following oven curing while latent samples are pull tested 21 hours following oven curing.

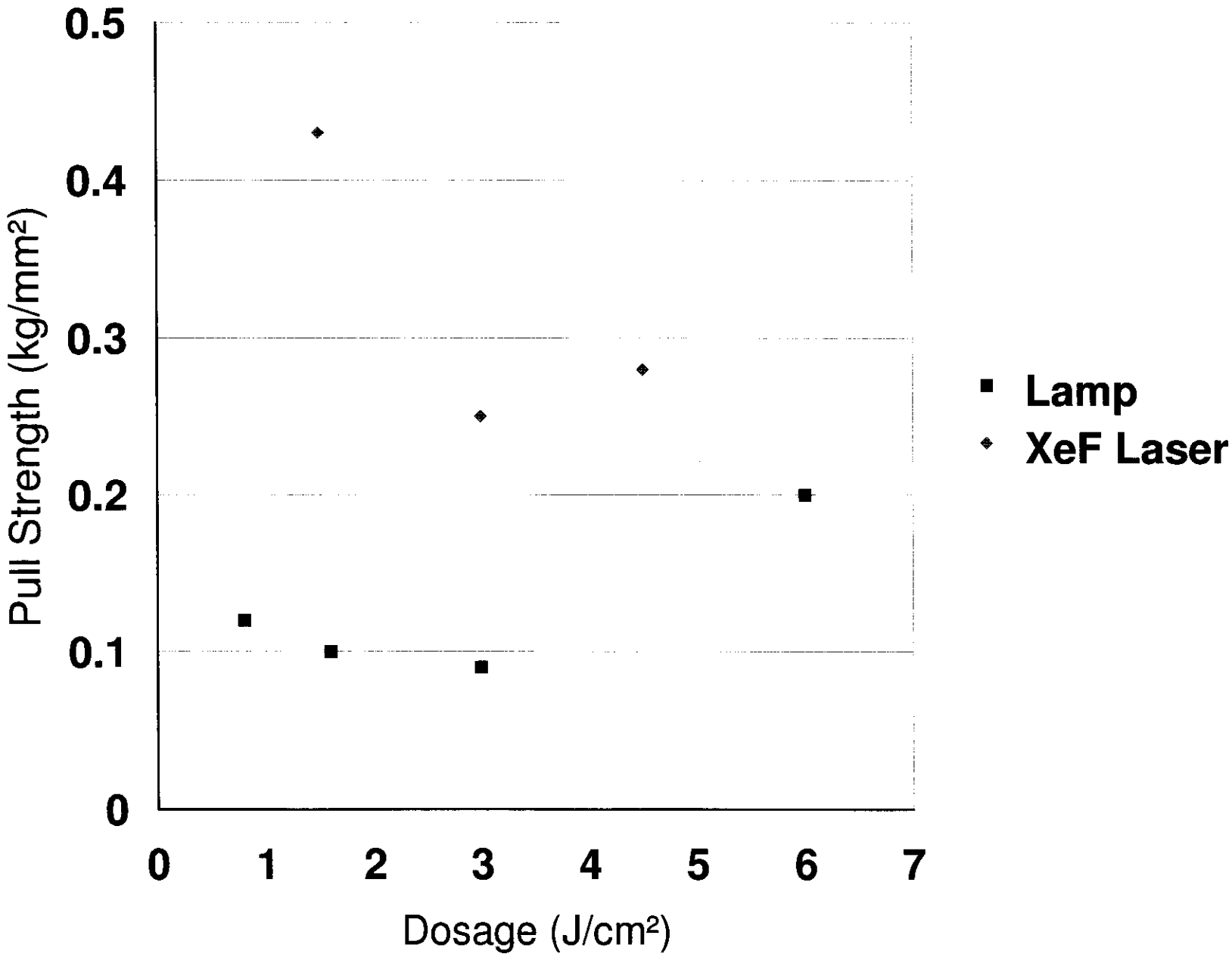

Fig. 3 Pull strength vs. UV dosage of lamp and laser irradiated samples of a commercial photolytically curing adhesive. Lamp flux was 200 mW/cm² and the laser fluence was 30 mJ/cm² with a repetition rate of 10 hz.

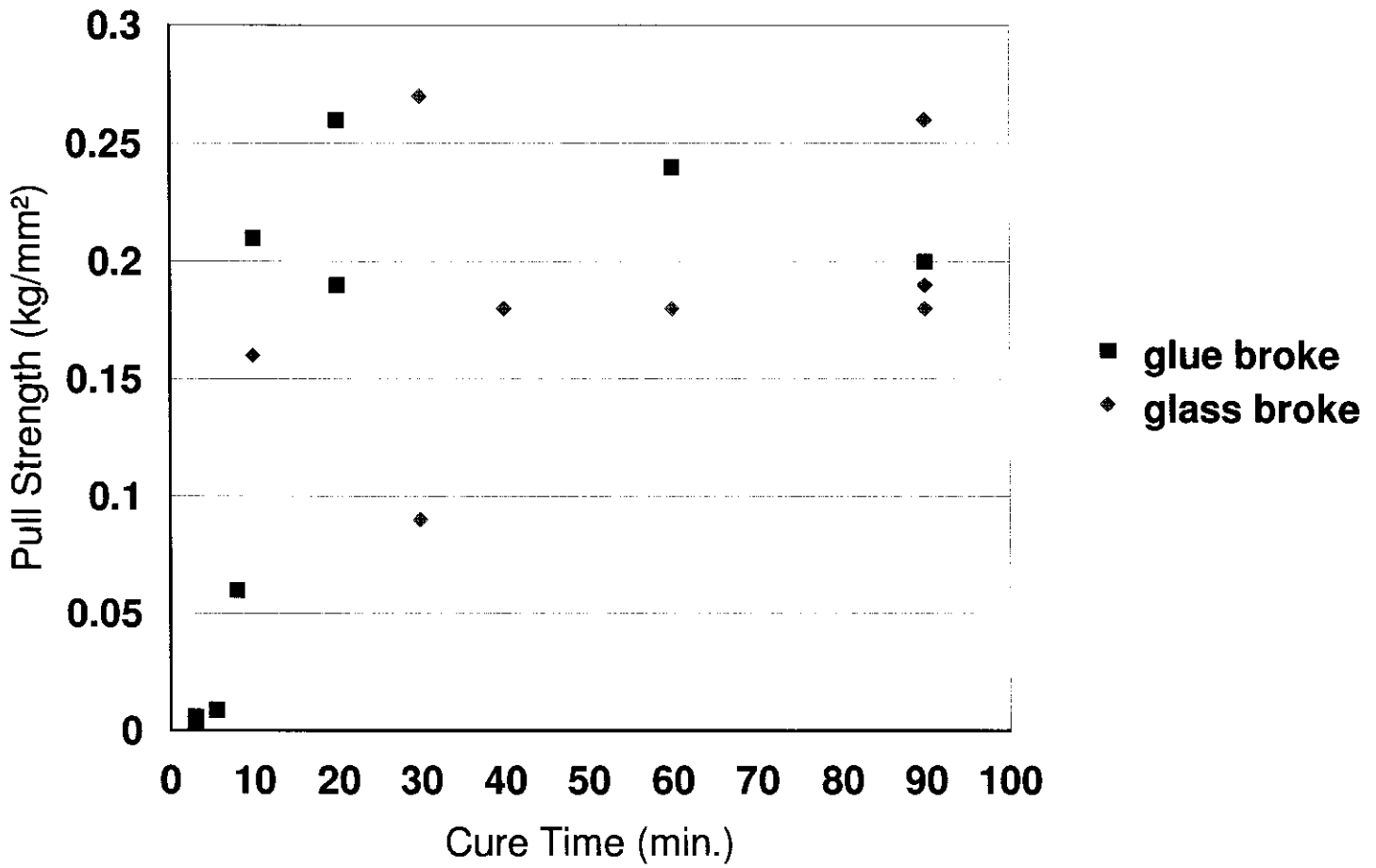

Fig. 4 Pull strength data for the glue seal of Fig. 3 containing thermal initiators through the addition of Trigonox 21,cured at 120C in an oven. Without added thermal initiators, there is no curing at 120C (in the absence of UV radiation).

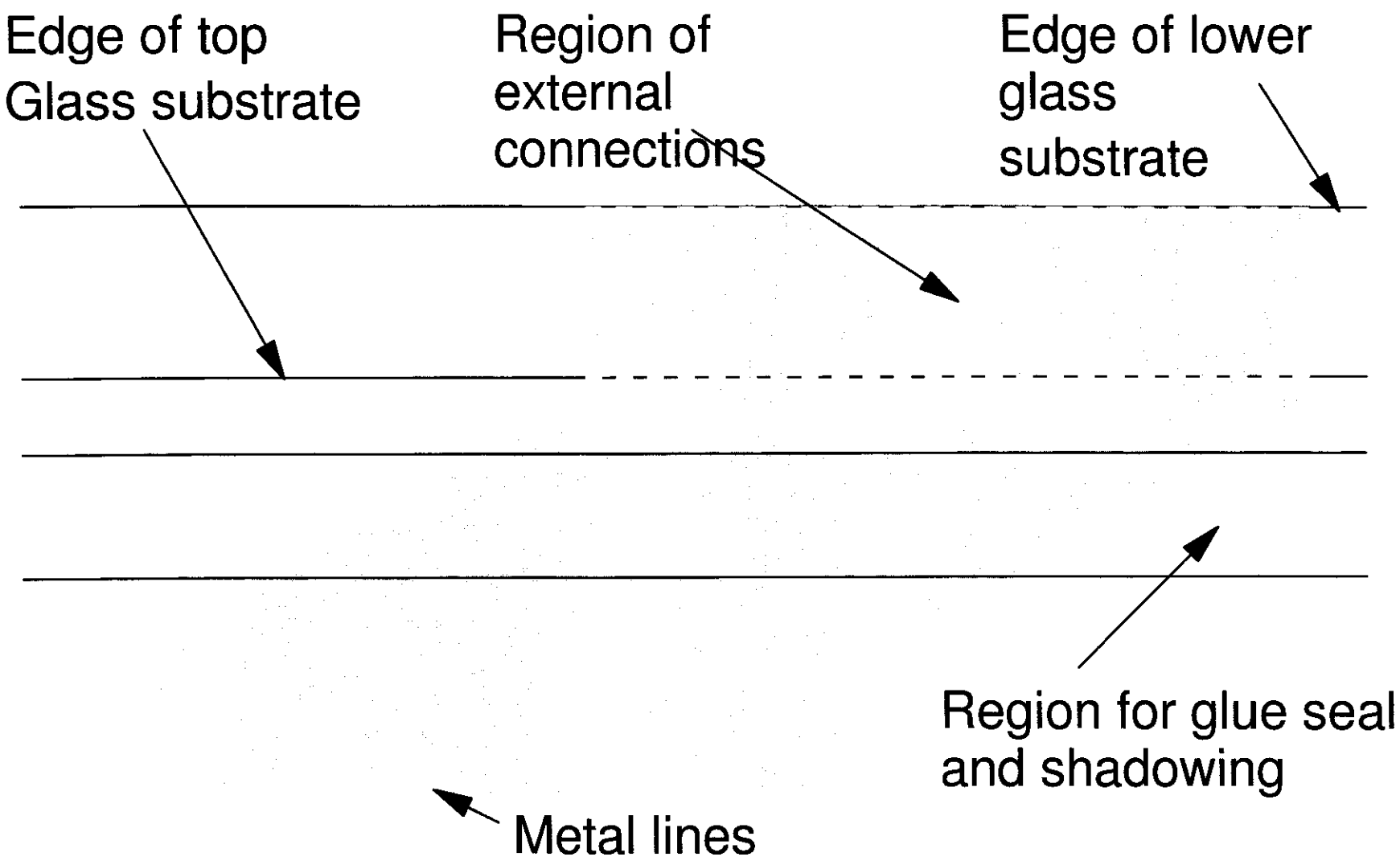

Fig. 5 Portion of fanout