ThreeBond
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Main Seal Adhesive for Liquid Crystal

Introduction

A display device has become an essential part of the computer and other equipment. Especially, the market for liquid crystal displays (LCDs) is steadily expanding, and segregating itself from CRT and PDP. Every LCD manufacturer is seeking higher resolution, higher reliability, and lower cost.

To improve the productivity and reliability of LCDs, Three Bond has proposed various new products: main seal adhesives, end seal adhesives, mold agents, pin-lead fixing adhesives, anisotropically conductive adhesives, and interlayer conductive adhesives (see “Materials for Peripheral Equipment of Liquid Crystal” in ThreeBond Technical News No. 43). Among these, main seal adhesives and end seal adhesives are the most important, because they contact the liquid crystal materials directly.

Various adhesives with different curing methods are reviewed elsewhere. This issue discusses the development of the ultraviolet-curing main seal adhesive.

Contents

1. Main seal adhesive for LCD

Today, solvent-type, heat-curing epoxy resin is used as the main seal adhesive for most LCDs. However, in shifting to higher LCD resolutions, misalignment due to heat has been observed. As a result, ultraviolet adhesive has been investigated for its potential as a sealing agent. The conventional ultraviolet-curing sealing adhesives that have been used as the LCD main seal adhesive have presented several problems:

(1) They can contaminate the liquid crystal.
Because the sealing agent and the liquid crystal materials are mutually soluble, irregular orientation can arise around the sealing agent.

(2) They cure slowly, and high heat is required.
To secure reliability, heat is applied after UV irradiation. But there is a problem of peeling (separation) during the heating process. Besides the heat, the UV-curing process itself requires light with an intensity of over 10,000 mJ/cm^2.

(3) The bonding strength they achieve is insufficient.
Because the sealant has insufficient resistance to the stress given in the scribe-cut process, peeling can occur.

(4) Their moisture resistance is insufficient.
Because of the high permeability of the sealant, moisture can penetrate into the liquid crystal panel, and cause an orientation failure.

(5) Their heat resistance is insufficient (low Tg).
Because Tg is low, the heating process that is required to achieve isotropy can give rise to several problems, such as peeling and uneven clearance.

Three Bond has developed an entirely new main seal adhesive for the liquid crystal, one that solves all these problems.

2. Examining the mutual solubility with liquid crystal

Ultraviolet adhesive has conventionally been used for the end seal adhesive (sealant). Most of the end seal adhesives produced today are in fact ultraviolet adhesive. Panel manufacturers, however, are not satisfied with these sealing agents. The end seal adhesive contacts liquid crystal before it hardens, and dissolves itself in the liquid crystal while curing. This causes an orientation failure.

The main seal adhesive was reviewed from the viewpoint of the mutual solubility with liquid crystal before curing. If a sealing agent before curing showed low mutual solubility with liquid crystal, it would probably not dissolve in liquid crystal while curing. If this were found to be so, this method could be used to develop an end seal adhesive.

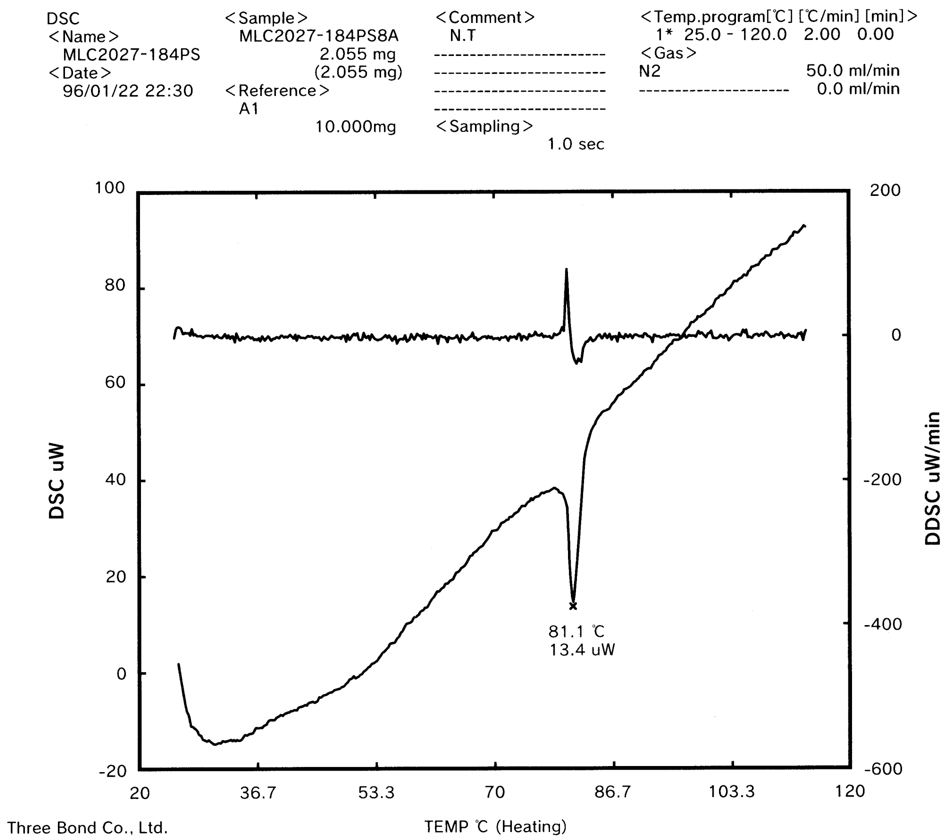
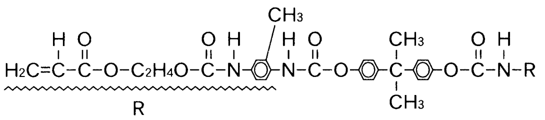


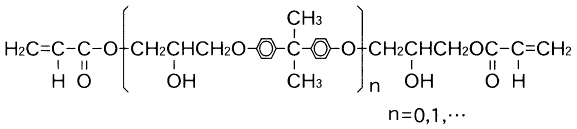
Fig. 1. DSC measurements of the liquid crystal

When a liquid crystal is heated to a specific temperature, called the “phase transition temperature,” it becomes an isotropic liquid. This is one of the unique characteristics of liquid crystal. If the property of a liquid crystal changes because of its mixture with another substance, its phase transition temperature may change. Liquid crystal absorbs heat during phase transition, as can be confirmed by recreating the phase transition with high probability using a DSC (Differential Scanning Calorimeter). When liquid crystal is in contact with different substances or is undergoing a weather resistance test, the heat absorption peak shown on the DSC would move. In other words, the heat absorption peak would shift when a foreign substance is dissolved in the liquid crystal or when the liquid crystal itself is changed (Fig. 1). The measuring method is as follows.

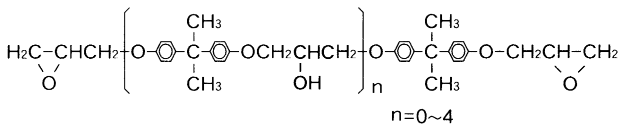
Three different sealant materials were measured: urethane acrylate, epoxy acrylate, and epoxy (UV curing).



Urethane acrylate



Epoxy acrylate



UV epoxy

(1) Preparing a specimen

1) Put 0.05 g of sealing agent gently into an ampoule, and leave it for 30 minutes.
2) Pour the liquid crystal material slowly into the ampoule so that the sealing agent constitutes 10% of the entire liquid, and leave it for 15 minutes.
3) Using ORC SPOT UV QRU-2266, irradiate ultraviolet rays through the bottom of the ampoule at the intensity of 10 mW/cm^2 for 40 seconds for curing (Fig. 2).

(2) Test method

1) Take about 1 mg of liquid crystal out of the ampoule, and set it on the DSC (SII DSC110).
2) Heat it at the rate of 2˚C per minute, and monitor the temperature until it reaches the phase transition temperature.
3) Seal the ampoule containing the remaining liquid crystal, place it in the high-temperature tank, and leave it at 100˚C for one hour.
4) After one hour has passed, repeat procedures 1) and 2).

Figure 3 shows the results for the three materials. Urethane acrylate showed the largest shift in the heat absorption peak, followed by epoxy acrylate and epoxy (UV-curing). This shows that urethane acrylate is inappropriate for this purpose. The shift in the heat absorption peak, however, should not be interpreted as the cause of the orientation failure. The shift in the phase transition temperature does not completely match the fall of the specific resistance. It suggests, however, that a substance affecting the properties of liquid crystal has dissolved into the solution.

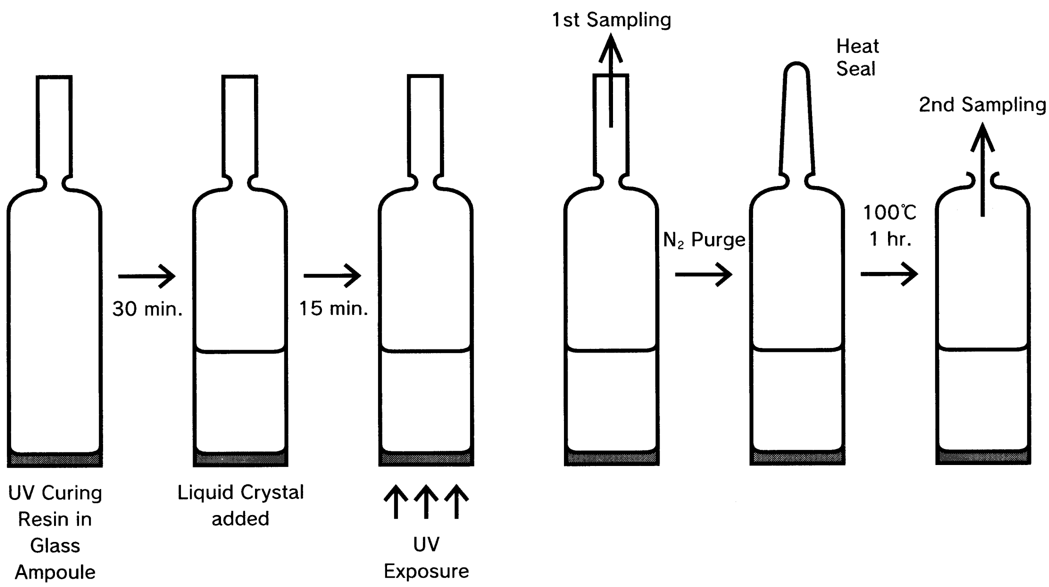


Fig. 2. Testing a base resin

The liquid crystal solution was analyzed by use of gas chromatography to identify the substances dissolved in the liquid crystal. Acrylic monomer and photo-polymerization-initiator substances were found in the urethane acrylate and epoxy acrylate specimens; the UV epoxy specimen contained only photo polymerization initiator. This finding suggests that substances dissolved in the liquid crystal can be reduced by using less of these substances in manufacturing the sealants.

Next, the shift in the heat absorption peak for epoxy acrylate and epoxy (UV-curing) was measured for different values of irradiation intensity. The result (Fig. 4) was better when the intensity was higher. When it was lower, epoxy acrylate showed better results than epoxy (UV-curing). The orientation performance, as indicated by the difference of irradiation intensity, resembles the orientation failure on the actual panel. Figure 5 shows the relationship between the amount of outgas increases during hardening and the orientation failure observed when epoxy acrylate was used as the sealant. From this result and that shown in Fig. 4, it can be suggested that when the irradiation intensity is reduced, the amount of outgas increases, and more resin dissolves in the liquid crystal. The resin causes a shift in the phase transition point, and thus causes the orientation failure.

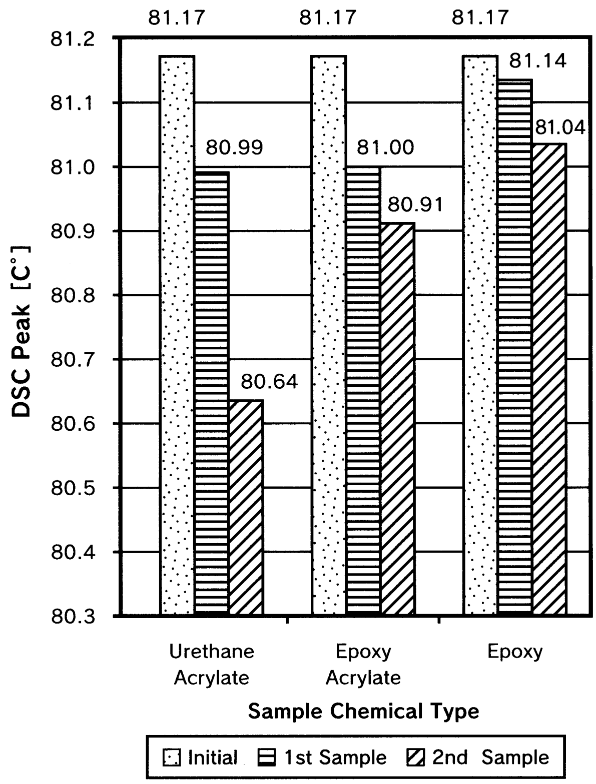


Fig. 3. DSC results

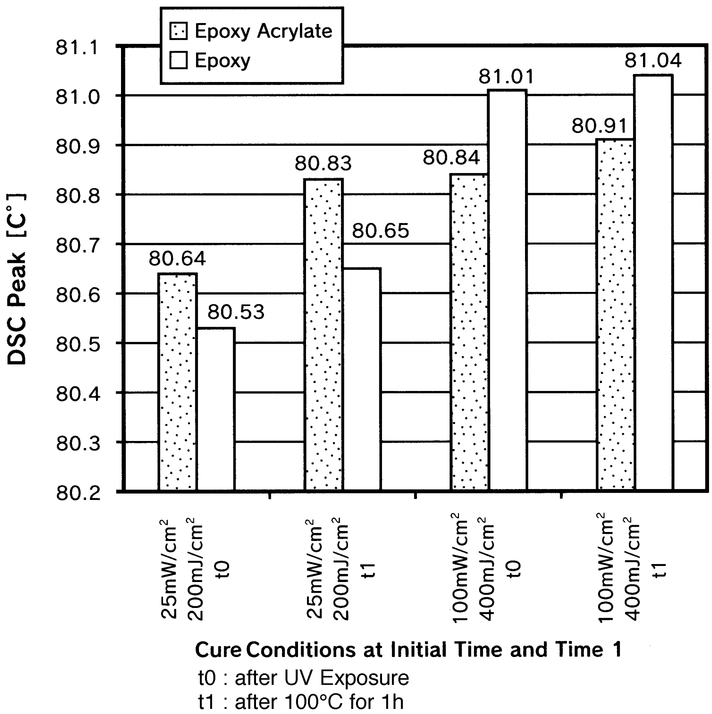


Fig. 4. DSC results for different UV-curing conditions

From these results, either epoxy acrylate or ultraviolet-curing epoxy would seem to be the best choice. However, physical properties, such as bonding strength, glass transition point and reliability, must be taken into account in the selection of a main seal adhesive. Three Bond therefore developed a main seal adhesive, based on UV-curing epoxy, that could be cured under high irradiation intensity (100 mW/cm²).

3. Development of a main seal adhesive

ThreeBond 3025G for liquid crystal displays is an ultraviolet-curing main seal adhesive in which epoxy resin is the primary component. It was developed to have high bonding strength, high Tg, and low moisture permeability. ThreeBond 3025G cures completely only under the irradiation of ultraviolet rays.

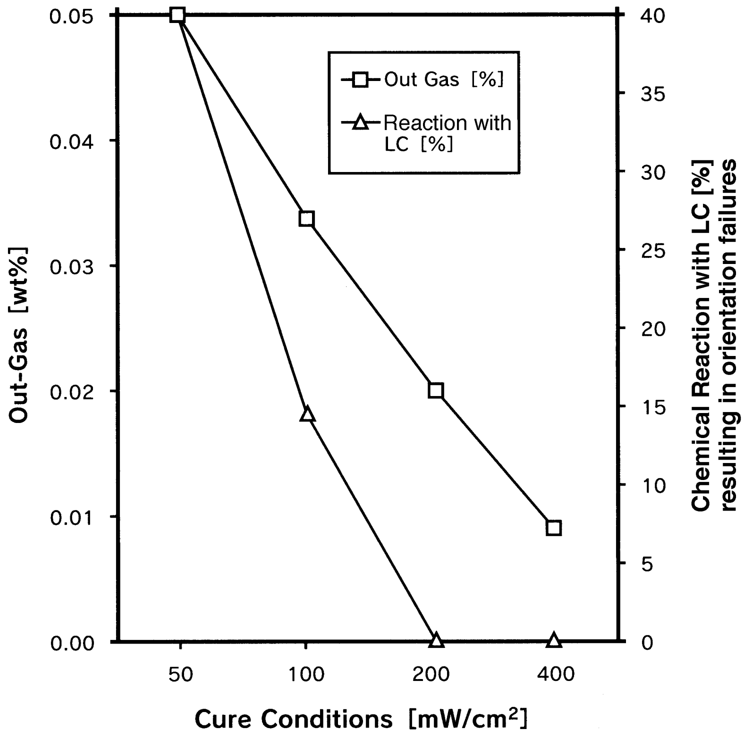


Fig. 5. Out gas and chemical reaction with LC vs. curing condition

Table 1. Physical properties of ThreeBond 3025G

Test item	Unit	ThreeBond 3025G	Test method	Note
Appearance	——	Milky brown liquid	3TS-201-01	
Viscosity	Pa·s {P}	45 {450}	3TS-210-02	25˚C
Specific gravity	——	1.38	3TS-213-02	25˚C

Test item	Unit	ThreeBond 3025G	Test method	Note
Hardness	——	90	3ST-215-01	JIS-D 25˚C
Glass transition point	˚C	140	3ST-501-04	
Water absorption	%	1.0	3ST-233-01	Boiling 2 hours
Water vapor permeability	g/m²·24h	5.0	JIS-Z-0208	40˚C × 95%RH
Hardening shrinkage ratio	%	3.0	3ST-228-01	
Peeling adhesive strength	MPa {kgf/cm²}	0.93 {9.5}		Corning 7059
	MPa {kgf/cm²}	0.62 {6.4}		Corning 7059 30 Ω ITO evaporation
Ion density			3ST-511-01	PCT × 48-hour extraction
Cl^-	ppm	25		
Na^+	ppm	1		
K^+	ppm	1		

Hardening condition : 4kw high-pressure mercury-vapor lamp (Oak Corp. HMW-244-11CM)
Irradiation distance : 15cm
Accumulated quantity of light : 40kJ/m² {4000mJ/cm²}

ThreeBond 3025G can be either screen-printed or applied with a dispenser. Table 1 shows its physical properties. The curing rate is 4,000 mJ/cm^2, which is fast for an ultraviolet-curing epoxy resin. The bonding strength is sufficient to resist scribe cutting. The graphs below show the results of tests of peeling adhesive strength, glass transition point, retention of elasticity at temperature up to 120°C, and moisture resistance (85°C, 90%RH).

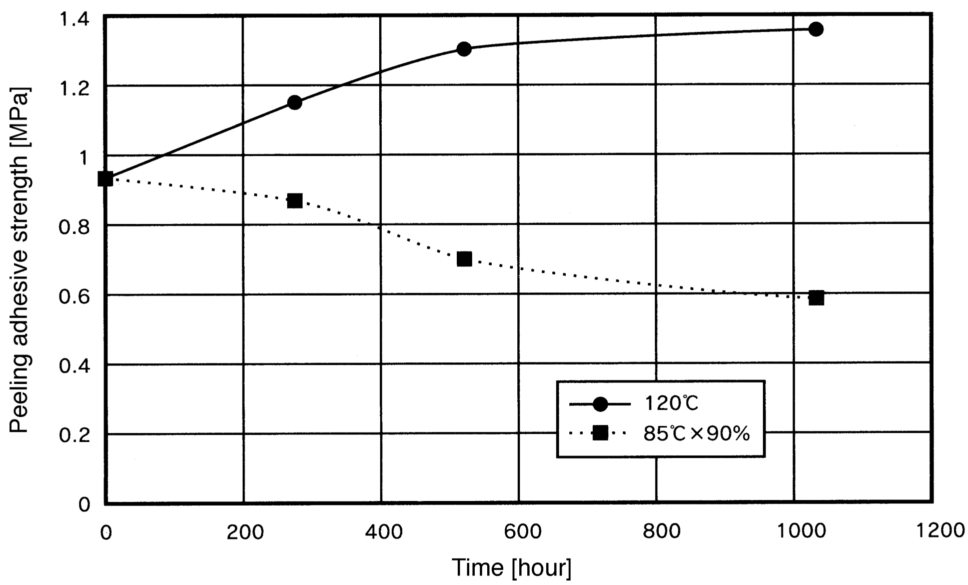
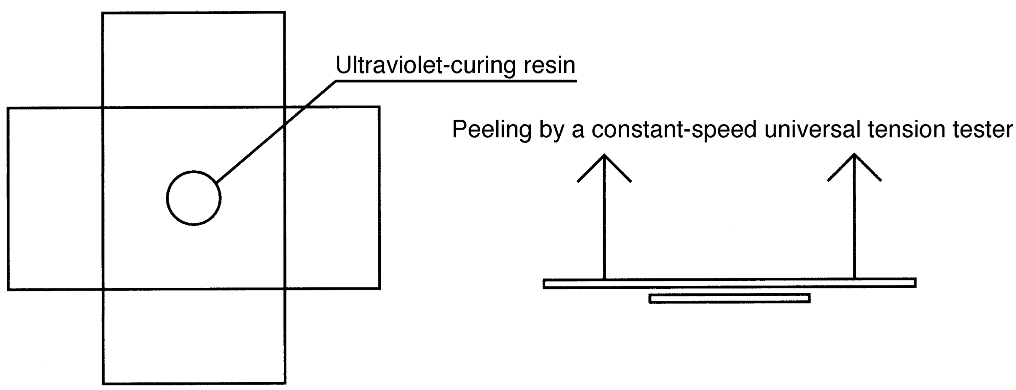


Fig. 6. Results of peeling adhesive strength test

Test for peeling adhesive strength

1. The spacer agent was added to the ultraviolet adhesive. Two sheets of glass were placed in a form of a cross, with the resin in between, as shown below.
2. The assembly was irradiated and cured at 100 mW/cm^2 for 40 seconds.
3. The bottom glass sheet was fixed, and the two end sides of the top sheet were lifted for the peeling test.
4. The peeling adhesive strength per unit area was calculated by measuring the space of the bonded surface area.



Glass size 25×50×1.1 (mm)

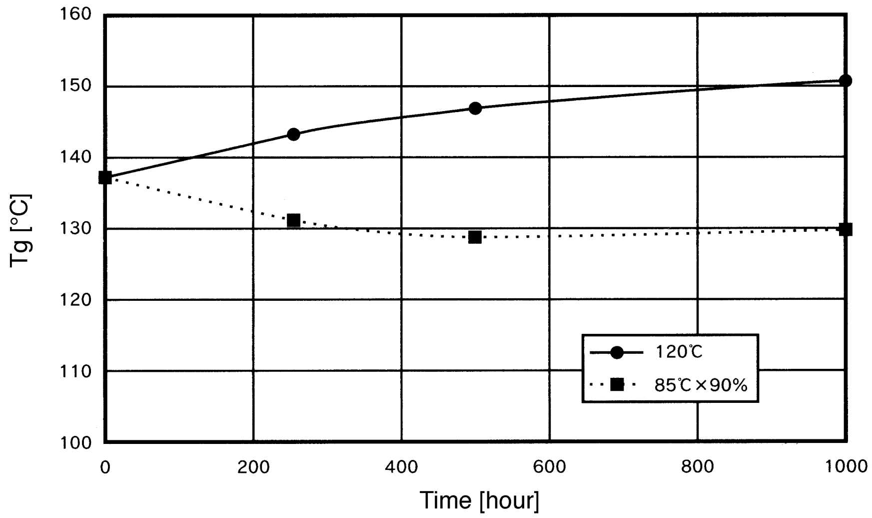


Fig. 7. Variation of Tg over time

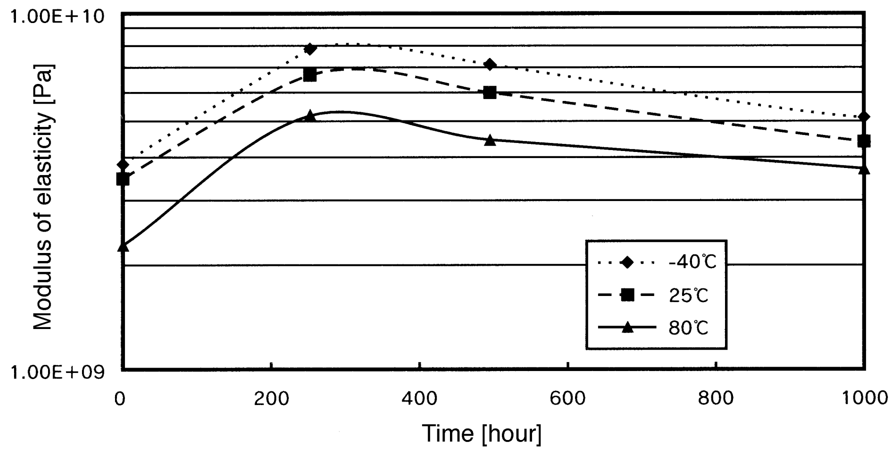


Fig. 8. Variation of elasticity over time at constant temperature of 120˚C

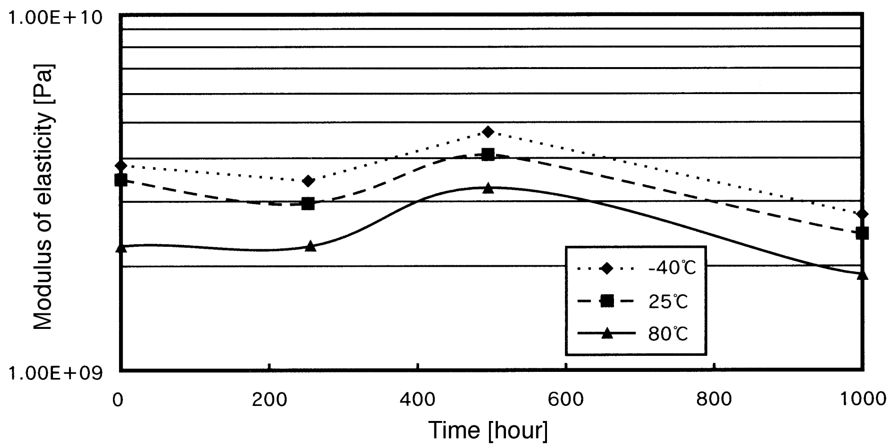


Fig. 9. Variation of elasticity over time at temperature of 85˚C, 90%RH

4. Prospects

Taking the advantage that it can cure only under ultraviolet irradiation, ThreeBond 3025G, a UV sealant for liquid crystal, is used for small poly-Si panels of view finders and projectors. For application to larger panels, some problems remain to be solved:

(1) Finding the curing method for shaded area such as under the aluminum wiring
(2) Further improving the bonding strength
(3) Improving applicability to the lamination unit
(4) Coping with the narrower lamination gap

In addition, further research is necessary on applying the sealant to the liquid crystal film, improving the end-sealant performance, and developing a UV interlayer conductive agent.

Conclusion

The application of UV-curing agent to the LCD main seal has just started. Already, however, the technology is becoming indispensable for improving the throughput, yield, and resolution of the liquid crystal panels. A technical breakthrough is still required to apply this method to larger panels, but in collaboration with the manufacturers of the panels and other equipment, it will not be too long before an ultraviolet-curing agent can be put to this use as well.

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