



Kinetic and mechanical properties of dual curable adhesives for display bonding process



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ABSTRACT

UV–thermal dual curable adhesives were prepared using an acrylic resin with 2,2′-azobis(4-methoxy-2,4-dimethyl valeronitrile), which exhibits ten hour half-life decomposition temperature of 30 °C, in order to address issues related to monomers that cannot be cured in a shaded area during the display bonding process. We investigated the dual curing behavior of the resin with variations in the UV dose and in the thermal radical initiator content, and we also assessed the influence of the dual curing on the mechanical properties. The curing behavior was assessed through the use of photo-DSC and FT-IR conversion. In addition, the degree of crosslinking was investigated via gel fraction, and the thermal properties were measured via DSC and TGA. Peel strength, probe tack and pull-off tests were performed to evaluate the bonding performance of the dual curable adhesives. The adequate level of thermal radical initiator (TRI) content to obtain a high conversion and degree of crosslinking was approximately 0.1 phr. These were the optimum values to adjust the balance between the unstable state and the insufficient contents, and the thermal curing behavior was promoted with an increase in the UV dose and in the thermal initiator content. The gel fraction and the FT-IR ATR results clearly indicate that an enhanced network structure was formed under low UV and high thermal radical initiator content conditions. Therefore, in a low UV dose condition, the mechanical properties improved, even with a low molecular weight due to a high network formation.

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1. Introduction

The display bonding process involves coalescing the window and touch screen panel (TSP) of a device using PSAs or transparent adhesives, such as optically clear resin (OCR) or optically clear adhesive (OCA) [1,2]. The reason to apply OCR or OCA on the entire surface of the display is to adhere the TSP and the window and to protect the TSP from an external impact. In addition, this also reduces the loss of light since it minimizes the reflection of light by filling up the air gap between the glass and the panel [3]. The window is divided into two parts: a transparent part of the display and a black matrix (BM) through which light cannot pass. UV light cannot penetrate under the BM area, and so unreacted monomer can cause reliability issues and pollution near the circuit board. The process to simply use light to cure the adhesive is therefore limited in that it cannot be used to cure the shaded area. A side that is overshadowed by the flexible printed circuit board (FPCB)

does not achieve fully cure, even if the device goes through a side curing process that irradiates light in different directions.

Industry has been increasingly using UV curing because doing so has various advantages including a fast curing speed, low energy consumption, processability at room temperature, low VOCs emissions, etc. [4,5]. In addition, the UV curing process is being considered to replace traditional solvent-borne processes because it is more ecologically friendly and has excellent properties. In particular, acrylate is widely used for the UV radical polymerization because it has a high reactivity [6]. However, the use of a UV curing process has a major disadvantage in that the shaded areas where light cannot reach cannot be cured [7]. For example, the curved portion of an automobile coating and the BM area for a display bonding process cannot be completely cured by only using a UV curing process [8,9]. This insufficient conversion can result in the elution of materials and can limit the mechanical properties of the polymer due to its low molecular weight [10–12].

As a result, various studies have been carried out on dual curing materials that not only respond to a UV curing process but also incorporate other methods of curing. Park et al. [13] described the

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dual curing mechanism for epoxy acrylate by forming crosslinking between the hydroxyl group and epoxy group. Studer et al. [14] observed the network formation of an acrylic copolymer containing isocyanate and a hydroxyl group in the branch during thermal curing. Hwang et al. [9] studied dual curing behavior and rheology using a thermal radical initiator for automotive applications. Shan et al. [15] explored the application of a UV–thermal dual curing system for electrically conductive adhesives (ECAs).

In this study, 2,2'-azobis(4-methoxy-2,4-dimethyl valeronitrile), which decays by 1st order chemical kinetics and has 30 °C of ten hour half-life decomposition temperature, was used with an acrylic prepolymer, photo radical initiator and a crosslinking agent. An initiator with a low decomposition temperature was used because the touch screen panel could be damaged at a high temperature during thermal curing, and it was also used to make it possible to cure the material with the ambient temperature of the metal halide UV lamp chamber. A minimum amount of initiator was added because the resin becomes unstable as excessive thermal radical initiator (TRI) is added, and other problems may occur, such as self-curing, undissolving and yellowing during storage. In previous studies, adding more than 0.2 phr of the initiators resulted in an unstable state [16]. On the other hand, a photo radical initiator was added in a sufficient amount to cure the shaded area during side curing with a low UV dose and to reduce the process time as much as possible. During the process, the dual curing behavior was measured in a UV dose of 0, 400, 800 and 1600 mJ/cm² because curing can proceed in a variety of pre-UV curing conditions due to the randomness of UV penetration. Table 1 provides the sample names of each of the dual cured adhesives. Isobornyl acrylate, 2-ethylhexyl acrylate, and N-vinyl caprolactam were used as monomer. Poly(ethylene glycol (200) dimethacrylate) was used as a crosslinking agent. Fig. 1 shows the brief procedure to prepare the sample. Then, FT-IR was used to investigate the chemical reactions within the adhesive. The formation of a cross-linked structure was evaluated through a gel fraction. The curing behavior was also evaluated via DSC and TGA, and peel strength, probe tack and pull-off tests were conducted to assess bond strength.

Table 1
Formulations and operating conditions for dual curable adhesives.

Name	UV dose (mJ/cm ²)	TRI contents (phr)
DCA-0000	0	0
DCA-0002		0.02
DCA-0005		0.05
DCA-0010		0.1
DCA-0400	400	0
DCA-0402		0.02
DCA-0405		0.05
DCA-0410		0.1
DCA-0800	800	0
DCA-0802		0.02
DCA-0805		0.05
DCA-0810		0.1
DCA-1600	1600	0
DCA-1602		0.02
DCA-1605		0.05
DCA-1610		0.1

2. Experimental

2.1. Materials

2-Ethylhexyl acrylate (2-EHA, Sigma Aldrich, USA), isobornyl acrylate (IBA, Sigma Aldrich, USA) and N-vinyl caprolactam (VC, Tokyo Chemical Industry, Japan) were used as monomers. Poly(ethylene glycol (200) dimethacrylate) (PEGDMA 200, Miwon Specialty Chemical, Republic of Korea) was used as a difunctional monomer to produce the network structure in the adhesives. Hydroxydimethyl acetophenone (Micure HP-8, Miwon Specialty Chemical, Republic of Korea) was used as a photo radical initiator. 2,2'-Azobis(4-methoxy-2,4-dimethyl valeronitrile) (V-70, Wako Pure Chemical Industries, Japan) was used as the thermal radical initiator. All reagents were used without any further purification. The chemical structures of each monomer and initiator are shown in Fig. 2.

2.2. Characterization methods

2.2.1. Prepolymer synthesis

The prepolymer was prepared using 2-EHA (61%), IBA (35%) and VC (4%) via bulk radical polymerization under UV irradiation with 1 wt% of photo radical initiator. Polymerization was performed in a 500 ml four-necked rounded-bottomed flask equipped with a mechanical stirrer, N₂ inlet, thermometer and LED UV lamp with wavelength of 365 nm. The temperature was maintained at room temperature with constant stirring with 100 rpm. After purging with N₂ for 30 min with constant stirring, the monomer mixtures were exposed to a UV lamp (20 mW/cm²) for 70 s under N₂ purging [17].

2.2.2. Preparation of adhesive film

The dual curable adhesives were prepared by blending prepolymer with 1 phr of crosslinking agent and thermal radical initiator. The mixture was mixed with a paste mixer at 600 rpm and 500 rpm for 3 min. The acrylic resin was coated to a 100-μm thickness on corona-treated polyethylene terephthalate films (PET, SKC Co. LTD., Republic of Korea). The coated resin was cured by UV curing using a UV conveyor equipped with medium pressure mercury UV-lamps with an intensity of 154 mW/cm² and main wavelength of 365 nm. The irradiated UV doses were of 200, 400, 800 and 1600 mJ/cm². The UV doses were measured using a UV radiometer (IL 390 C Light Bug, International Light Inc.). Each of the pre-cured adhesives with various curing degrees was cured in an oven at 80 °C for 5, 10 and 30 min.

2.2.3. Fourier transform infrared spectroscopy (FT-IR)

The IR spectra of the samples were observed using Fourier-transform infrared spectrometry (JASCO FTIR-6100) equipped with an attenuated total reflectance (ATR) accessory. The spectra were collected with 32 scans at a resolution of 4 cm⁻¹ between 4000 and 400 cm⁻¹. The acrylate double bond conversion was calculated using Eq. (1)

$$\text{Conversion(\%)} = \frac{(A_{810})_0 / (A_{1720})_0 - (A_{810}) / (A_{1720})}{(A_{810})_0 / (A_{1720})_0} \times 100 \quad (1)$$

where $(A_{810})_0$ is the intensity for 810 cm⁻¹ at the initial time and (A_{810}) is the intensity for 810 cm⁻¹ after curing [18].

2.2.4. Gel fraction

The gel fraction of the dual curable adhesives after UV curing and thermal curing was determined by soaking the samples in toluene for 1 day at room temperature. The amounts of the sample were about 5 g. In addition, the insoluble part of the adhesives was filtered with a 40 mesh net and dried at 70 °C.

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