



Pulsed irradiation for high-throughput curing applications



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ARTICLE INFO

Article history:

Received 23 August 2016

Received in revised form 6 December 2016

Accepted 7 December 2016

Keywords:

Intense pulsed light
Photopolymerization
Multifunctional acrylates
Coating
Infrared spectroscopy

ABSTRACT

A xenon flash lamp was used to create intense pulsed light (IPL) irradiation to cure acrylate coatings at very high rates and ambient temperature; we investigate whether the achievable conversion can be increased compared to conventional irradiation and as the dose rate is increased with IPL parameters – pulse intensity, pulse duration, and pulse repeat period – and with the photoinitiator (PI) concentration and the curing temperature. Conversion of acrylate double bonds was measured using Fourier transform infrared (FTIR) spectroscopy. In all cases, cure conversion increases with the number of pulses until it plateaus at the maximum achievable value, when the reduced mobility of reactive species prevents further reaction. Changing the pulse intensity, the pulse duration, and the pulse period – and even changing the photoinitiator loading – do not appreciably affect the achievable conversion at ambient temperature. Increasing the temperature can result in somewhat higher achievable conversion, but with the loss of advantages of ambient temperature cure. Consistent with the expectation that the cure rate should depend strongly on intensity, a single measured pulse with high dose rate can achieve millisecond-timescale cure of acrylate coatings, providing adequate curing dose while avoiding damage to the substrate from excessive exposure. In multiple pulse patterns, the achievable cure can be attained more quickly with higher dose rate; as opposed to a single pulse irradiation, the introduction of dark periods can reduce the total irradiation dose needed to attain the achievable cure conversion.

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1. Introduction

UV curing processes are of interest in many industrial sectors, from the traditional packaging industry to the emerging electronic device manufacturing [1]. High-powered mercury lamps are widely used to provide high UV intensities in order to increase the rate of cure. However, mercury lamps can result in temperature increases – both from nonselective irradiation and from high reaction rate – ozone production and excessive power consumption [2]. Lasers, attractive for emitting monochromatic high-intensity light, produce reactions in a localized area, so are limited to applications like microlithography and 3D printing. With recent advancements in the UV LED technology, UV LED systems can provide UV light of a tight, specific range of wavelengths without ozone production or IR radiation, but they put forward more stringent requirements on formulating compatible UV curable coatings.

A promising alternative light source that might avoid the disadvantages of mercury lamps is the xenon flash lamp, which can produce intense pulsed irradiation at ambient temperature over

large areas. In this approach, termed “Intense Pulsed Light” (IPL) [3], the high pulse intensity enables rapid generation of reactive species, while the dark periods between pulses can allow dissipation of the heat of reaction and of nonselective light absorption. These systems are used in the manufacture of medical devices, optical fibers, and electronics, and it has been suggested that xenon flash lamp can reduce the curing time to a few minutes (in some cases within seconds) for automotive coatings [2,4,5]. Recently, IPL curing has become attractive to researchers working on the roll-to-roll processing of printed electronics, where fast cure and low temperature are highly valued. Xenon flash lamps have been employed in the fabrication of dielectric capacitors [6] and electrically conductive adhesives [7] on a laboratory scale, achieving complete cure much faster than the currently mainstream methods of continuous UV curing.

There are only few studies, though, quantitatively examining the factors that affect the speed and the extent of cure in IPL curing processes. Previous IPL curing investigations show the curing processes are completed within several minutes, determined by the number of pulses necessary for the achievable conversion [2,4,5]. We aim to explore whether the curing speed can be further enhanced by carefully tuning a full range of processing variables in a model UV curable system. Work so far [4,5] illustrates a general pattern that the cure conversion increases with the number of pulses, but it

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reaches a plateau at a maximum “achievable” conversion when the mobility of reactive groups becomes too limited for further reaction. Following the excess free volume hypothesis suggested by Kloosterboer and coworkers [8], we are interested in whether the achievable conversion might be higher with higher irradiation dose rate. Previous work by Decker et al. [9] suggested that increasing intensity with continuous mercury lamp irradiation might raise the achievable conversion, but they noted the strong possibility that temperature rise due to reaction and to radiation absorption might be the cause.

In this work, we studied the ambient temperature IPL curing of an acrylate monomer system that is known to present a limited attainable cure with low-intensity UV irradiation. The capability of xenon flash lamps for fast photopolymerization of acrylate resins was investigated using a single pulse and a multiple pulse pattern. We wished to determine whether the achievable cure and cure rate can be increased with IPL and as the IPL parameters are changed to increase the net rate of irradiation dose – with more intense pulses, with longer pulses, and with shorter pulse periods (i.e., more closely timed pulses). We also examined whether the achievable cure depends on the photoinitiator (PI) concentration and the curing temperature.

2. Experimental

2.1. Coating formulations

In the present study, trimethylol propane triacrylate (TMPTA; Sigma-Aldrich, St. Louis, MO) was the monomer and 2,2-dimethoxy-2-phenylacetophenone (DMPA; Sigma-Aldrich, St. Louis, MO) was the PI. Their chemical structures are shown in Fig. 1. DMPA has UV absorption peaks at 252 nm and 335 nm, the molar absorption coefficients of which are $16510 \text{ LM}^{-1} \text{ cm}^{-1}$ and $266 \text{ LM}^{-1} \text{ cm}^{-1}$, respectively [10]. Coating formulations were prepared by dissolving PI into monomers and then stirring in the dark for 1 h. The amount of PI varied from 0.05 mol% to 5 mol% (expressed as the ratio of moles of DMPA to the moles of double bonds in acrylate monomers).

2.2. Curing conditions

The light source utilized was a xenon flash lamp (Sinteron 2010, Xenon Corp., Wilmington, MA) in the burst mode, where a predetermined number of pulses was emitted. The time-varying irradiation pattern is determined by four processing parameters: the number of pulses, voltage (setting the energy level per unit time during pulses), the pulse width (the time duration of each pulse), and the pulse period (the delay from the leading edge of one pulse to that of the next pulse). According to the information provided by the manufacturer, the peak irradiation dose rate increases linearly with the lamp voltage. In addition to the pulsed irradiation mode, the xenon flash lamp is different from a conventional mercury lamp in both spectrum distribution and light intensity; it is energy rich

from 246 nm to 1000 nm with intensity on the order of 10 mW/cm^2 (Courtesy of Xenon Corp.). The lamp-to-sample distance was fixed at 25.4 mm.

2.3. Conversion measurement

An FTIR spectrometer (Magna IR-750, Nicolet Instrument Co., Madison, WI) in transmission mode was used to measure the extent of cure of TMPTA coatings. Spectra were recorded with 32 scans and a resolution of 2 cm^{-1} at room temperature. The Thermo Scientific OMNIC software package was used for spectra collection and data analysis. A background spectrum was collected before each sample measurement and subtracted from the sample spectrum. The uncured resin was sandwiched between two sodium chloride plates separated by a $0.025 \times 25 \text{ mm}$ steel spacer (McMaster-Carr, Chicago, IL), which was then exposed to pulsed irradiation. This arrangement permits UV transmission during curing due to the fact that sodium chloride is transparent to UV light from 200 nm to 400 nm [11] and effectively avoids oxygen inhibition problems associated with free-radical photopolymerization. Coating thickness was $25 \mu\text{m}$, controlled by the thickness of spacers but with certain variance among samples.

IR spectra of each sample were recorded before and after IPL curing, respectively. The extent of cure (α), measured by the fractional conversion of acrylate double bonds, was calculated from the drop in the absorption of C=C characteristic peaks at 1635 and 1619 cm^{-1} (symmetric C=C stretch and asymmetric C=C stretch):

$$\alpha = \frac{A_0 - A_t}{A_t} \times 100\% \quad (1)$$

where A_0 represents the absorption of double bonds before IPL curing and A_t represents that after IPL curing [12,13]. The absorption value, proportional to the double bond concentration in the sample, was calculated by integrating the area under both peaks from 1660 to 1580 cm^{-1} (where the photoinitiator has negligible IR absorption).

2.4. Heat transfer analysis

The heat transfer equations describing the heat generation during IPL curing of the TMPTA coatings were solved using a heat transfer model within the COMSOL software package (version 4.4) in a similar manner to [14]. The model substrate configuration used for simulations comprised, from top to bottom, a 4 mm NaCl layer, a $25 \mu\text{m}$ TMPTA layer, and a 4 mm NaCl layer. Upon these geometries, the maximum element sizes were limited to $0.1 \mu\text{m}$ in the TMPTA layer and $20 \mu\text{m}$ in the NaCl layers. The top surface of the structure was allowed to cool through convective heat transfer with the surroundings at a temperature of 293.15 K with an assumed heat transfer coefficient of $10 \text{ W/m}^2 \text{ K}$. The bottom surface of the model structure was assumed to be held at a constant temperature, $T = 293.15 \text{ K}$, due to its contact with a solid surface which acts as a heat sink. The pulse duration of $100 \mu\text{s}$ and the pulse period of 1 s were used to simulate heating from 10 lamp pulses during IPL curing. The heat generation from nonselective light absorption was negligible compared to the heat from reaction. The heat flux \dot{q} resulting from polymerization was calculated according to the following equations:

$$\dot{q} = \frac{\alpha \Delta H_{\text{TMPTA}}}{\Delta t} \quad (2)$$

$$\Delta H_{\text{TMPTA}} = \Delta H_{\text{C=C}} \times \frac{\rho}{M} \times f \quad (3)$$

where α is the degree of conversion (using results from FTIR experiments), ΔH_{TMPTA} is the heat of polymerization of TMPTA monomers per unit volume (0.872 kJ/cm^3), Δt is the flash duration

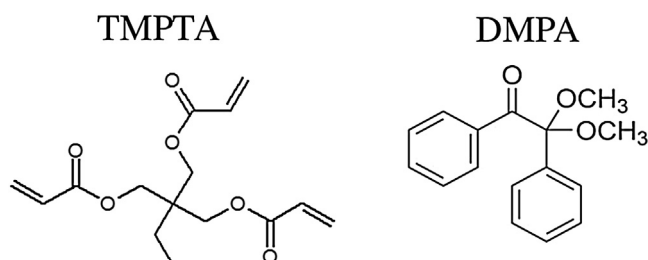


Fig. 1. Chemical structures of TMPTA and DMPA.

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