

Enhanced Pultrusion Using Photocure to Supplement Standard Thermal Cure

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Abstract

The rapid cure that occurs when photoinitiated resins are exposed to light of the appropriate wavelength offers the potential for the use of photocure to improve the processing efficiency for the pultrusion process. The objective of this research is to demonstrate the feasibility of a photocure pultrusion process that utilizes standard pultrusion tooling and equipment. In order to accomplish this objective, a hybrid thermal/photo initiator process which uses photocure to supplement standard thermal cure was developed. Results demonstrate the potential offered by this hybrid thermal/photo initiator process; exposure to UV energy lead to an increase in degree of cure and mechanical properties in the thermal/photo initiated composites.

Introduction

Composites which utilize photoinitiators to achieve polymerization during processing are found in a wide range of applications today. The introduction of bis-acylphosphine oxide (BAPO) photoinitiators that can achieve cure in relatively thick sections up to approximately ½ inch thick has expanded the potential uses of photocure for composites [1, 2]. The use of BAPO and alpha-hydroxy ketones (aHK) in conjunction with each other enables photocure composites to achieve good through cure and surface cure. Current commercial applications for photocure composites range from sporting goods to infrastructure retrofit [3 - 6]. A variety of open-mold composites processing techniques such as hand lay-up, vacuum assisted resin transfer molding (VARTM), and filament winding are utilized commercially to manufacture photocure composites. However, open-molding processing techniques are not appropriate for all applications.

The pultrusion process is well-known as a versatile, cost-effective composites manufacturing process, and the utilization of photocure techniques with the pultrusion process offers an area for additional development. Photoinitiated resins offer the potential for increased process efficiency because they are not limited by the rate of heat transfer from the heated die to the resin. However, the use of photoinitiated resins with the pultrusion process is complicated by the need to reach a level of cure as the fiber/resin mass moves through the enclosed pultrusion die. Reaching a proper level of cure while inside the die is necessary since the pressure developed within the die influences the consolidation of the fiber/resin mass. However, the closed pultrusion die makes it difficult to expose the photoinitiated resin to UV energy as it travels through the pultrusion die.

Patent and literature searches reveal that various proprietary techniques have been developed in attempts to apply the benefits offered by photoinitiated resins to the pultrusion process [7 - 17]. To accommodate the requirement of having the photoinitiated resin exposed to UV energy of the appropriate wavelength to initiate cure, the use of special tooling such as UV transmittent materials for the die [7, 8, 15] or metal dies that incorporate fiber optic cables to transmit the UV light to the interior of the die [10] have been examined. Based on the patents and published reports concerning the use of photoinitiated resins with the pultrusion process, it is apparent that there is significant interest in the possibility of taking advantage of the characteristics offered by photoinitiated resins such as rapid cure in conjunction with the process advantages offered by the pultrusion process. However, one limitation associated with all of these approaches to photocure pultrusion is the need for special tooling and special pultrusion equipment.

The objective of this research is to demonstrate the feasibility of a photocure pultrusion process that takes advantage of the desirable characteristics of photocure while utilizing standard pultrusion tooling and equipment. A hybrid thermal/photo initiation process which uses photocure to supplement standard thermal cure has been developed to accomplish this objective. The use of standard pultrusion dies, resin formulations, and processing procedures was desired to make the adoption of photocure technology by the pultrusion industry as easy as possible. The successful integration of thermal and photocure techniques has been reported by other researchers for manual, laboratory-scale composites processing, but no discussion of the application of this approach to automated, commercial-scale processing such as pultrusion was provided [18].

Development of Hybrid Thermal/Photo Initiated Pultrusion

For all photocure processing, a Fusion UV Systems F300S 300 watt/inch microwave powered electrodeless lamp was used. Based on the absorption characteristics of the photoinitiators used, a D-type bulb, with the output spectrum shown in Fig. 1, was selected for the initial experiments. Based on results from previous experiments conducted on photocure filament winding, a combination of BAPO and aHK photoinitiators at concentrations of 0.3 parts per hundred resin (phr) and 0.9 phr, respectively, was selected [19]. BAPO influences through curing, while aHK accomplishes surface curing of the composite [19, 20]. A multi-purpose, medium reactivity, isophthalic polyester resin commonly used with the pultrusion process (AOC P706) was used in this study [21]. A fiber volume of 68% of multi-compatible, 113 yield, E-glass roving (OC 366-AD-113 E-glass) was used for the manufacture of all composites in this study. The type and amount of reinforcement in the composite will affect the penetration of the UV energy into the composite for curing. For example, carbon fiber does not allow for sufficient penetration of the UV energy into the interior of composite for UV cure. It is suspected that not all E-glass reinforcements are equivalent in terms of allowing penetration of UV energy into the composite; however, this hypothesis has not yet been verified in this study. The E-glass roving initially used for these experiments gave acceptable results, so other types of roving were not examined in these experiments.

Both post-cure and on-line photocure pultrusion processes were developed in this research study. Prior to the application of the photocure processes to the pultrusion process, preliminary experiments were required to identify viable resin formulations to be used in the pultrusion experiments.

Preliminary Resin Formulation Experiments: Prior to the start of the UV pultrusion experiments, it was necessary to determine a workable resin formulation for the photocure pultrusion processing. Very little data concerning the formulation of resin systems for UV pultrusion is available in the open literature, so information learned from prior research on UV cure filament winding was used to establish a starting resin formulation. Previous work conducted at the University of Mississippi had demonstrated that filament wound polyester/glass composites showed the best curing characteristics when the concentrations of the photoinitiators BAPO and aHK were 0.3 and 0.9 phr, respectively [19].

For the pultrusion process, the addition of inorganic fillers to the resin system was desired since fillers are known to aid in processing in addition to acting as inexpensive, space-filling materials in the pultruded composites. However, selection of the type and amount of filler

was not straightforward. A variety of material characteristics such as particle size, brightness, and pH are provided by filler manufacturers, but data related to UV absorbance characteristics of fillers are not readily available. Because equipment to directly measure these characteristics was not available for this research, an experimental study that evaluated exotherm temperature, depth of cure, and Barcol hardness of the exposed surface of photocured resin samples formulated with various types and amounts of fillers was conducted.

The procedure used for these preliminary resin formulation experiments involved the filling of a volume of 4" x 4" x 0.125" on a glass substrate with resin samples formulated as in Table 1. The fillers and amounts used for each formulation are shown in Table 2. Prior to the addition of the resin, a thermocouple was secured to the lower, center surface of the volume. After the mixed resin was poured into the volume, the sample was exposed to the F300S UV lamp.

Results from these experiments demonstrated the influence of fillers in the formulation of resins subjected to UV cure. Data recorded for each sample are shown in Table 2. None of the kaolin clay formulations at either 20 phr or 10 phr were cured through the 0.125" thickness of the sample. Calcium carbonate showed some possibility for successful use, but the alumina trihydrate (ATH) formulations showed the best potential. Because the resin formulation with 10 phr of ATH showed the best combination of properties as shown in Table 3, this type and amount of filler was selected for use in the pultrusion processing experiments.

Pultrusion Processing: For the pultrusion processing experiments, a hybrid thermal/photo cure process was used. The resin formulation for these hybrid thermal/photo cure composites was similar to the formulation shown in Table 1 along with the addition of 0.5 phr Perkadox 16 and 0.25 phr TBPB for thermal cure. Based on the results of the initial resin formulation experiments, 10 phr ATH was used as the filler for the resin. Data from these hybrid cure experiments were compared to data of composites that used only thermal cure that were produced under the same processing conditions.

Two approaches were used for the hybrid thermal/photo cure pultrusion processing – off-line, post-cure exposure to UV energy and on-line UV exposure immediately following the pultrusion die. A 1" x 0.125" rectangular cross-section was pultruded for the off-line, post-cure experiments. This rectangular cross-section was selected because it was appropriate for mechanical test specimens. For the on-line UV experiments, a 0.375" diameter round cross-section was produced; this cross-section was used due to size limitations of the reflector used with the F300S UV lamp. Because it would have been very easy to reach full cure from thermal cure

alone for the relatively small cross-section that had to be used for these experiments, pultrusion processing variables of die temperature and line speed were selected to intentionally produce an undercured composite as it exited the heated die. This was done to simulate conditions in which a combination of thermal cure and photocure could be used to cure a thicker composite at faster line speeds than could normally be achieved if thermal cure alone were used. The hybrid thermal/photo cure techniques described here are not limited to geometries that can be accommodated by this reflector. For commercial applications requiring different part geometries, modification of the reflector, fabrication of a specialized reflector, or selection of larger UV lamps could easily be done. Modifications of the reflector were not done for this initial feasibility study due to budget and time constraints.

Temperature measurements, degree of cure measurements, Barcol hardness, and mechanical property measurements were used to evaluate the effects of photocure on these composites. By exposing the composite with this hybrid thermal/photo cure formulation to UV light following thermal exposure, it was expected that any portion of resin left uncured from the thermal exposure would cure due to the UV exposure. This was expected to generate two distinct exotherms in the thermocouple data. The first exotherm would occur due to partial cure within the die, and the second exotherm would occur due to exposure to the UV energy.

Details of selected experiments are discussed in this report; however, numerous other experiments were conducted to identify workable line speeds, lamp locations, and other processing parameters. Details concerning the other experiments required to identify the workable processing parameters can be found in another publication [Rahul's Thesis].

Results and Discussion of Hybrid Thermal/Photo Pultrusion

Off-line, Post-cure Photocure Pultrusion Processing: Prior to the start of the UV cure pultrusion experiments, data for thermal cure only composites were obtained for use as a baseline for comparison to the UV exposed samples in later experiments. These composites utilized a resin formulation similar to that shown in Table 1, but 0.5 phr Perkadox 16 and 0.25 phr TBPB were used instead of the BAPO and aHK. These composites were manufactured using typical commercial pultrusion processing conditions of 36 in/min and 48 in/min line speeds and die temperatures of 282°F, 327°F, 318°F. As shown in Table 4, the designation "UVPul-Baseline" was used for this experiment. Based on thermocouple data recorded during this experiment, peak exotherm occurred at approximately 2/3 of the distance into the 36 inch long

die. Short-beam shear data for the "UVPul-Baseline" composites are shown in Table 5.

To simulate the effects that UV post-cure would have on composites that were not able to achieve sufficient heat transfer while within the pultrusion die, the die temperatures that had been used for the baseline experiment were reduced and the pull speed was increased. For this experiment, designated as "UVPul-PostUV", processing conditions of 232°F, 277°F, 268°F die temperatures and a 96 in/minute pull speed were used to simulate the production of an undercured composite as it exited the heated die. Thus, even though this was a relatively small cross-section, it was not optimally cured as it exited the heated die. Shortly following the removal of the composites from the pultrusion line, the samples were exposed to light from the UV lamp for 30 seconds with the lamp held 4 inches above the samples. Because of the lack of published data related to properties of pultruded composites manufactured using hybrid thermal/photo cure, it was necessary to perform these initial experiments to determine the feasibility of this approach for the processing of pultruded composites.

Comparisons of experimental data shown in Tables 5 – 6 demonstrate the benefits that can be obtained through the use of UV energy for additional cure of pultruded composites. Both the short-beam data and the Barcol hardness data of the baseline experiment are significantly higher than the corresponding property data for the UVPul-PostUV composites prior to their exposure to the UV energy post-cure; however, following the UV post-cure, the properties of the UVPul-PostUV composites were essentially equivalent to the baseline properties. This comparison demonstrates that UV photo cure can be successfully used to improve the properties of composites that did not reach optimal cure during the initial pultrusion processing. This data suggests that a combination of thermal cure and photocure cure could be used to cure a thicker composite at faster line speeds than could normally be achieved if thermal cure alone were used.

On-line Photocure Pultrusion Processing: Based on the encouraging results from the off-line, post-cure photocure pultrusion experiments, the use of online photocure processing was examined. Due to equipment geometry limitations, a 3/8 inch diameter cross-section was used for all composites produced in these experiments. After some experimentation, it was determined that line speeds of 60 in/min and 72 in/min could be successfully used for these experiments; slower line speeds resulted in scorching of the composite surface as the composite was exposed to very intense UV energy for a relatively long period of time as the composite traveled through the UV lamp reflector at the focal length of the lamp. The time of UV exposure for the on-line photocure processing was much shorter than the 30 seconds of exposure used in the post-cure photocure experiments,

but the intensity of the light was much greater as the samples passed through the lamp's reflector.

The approach used for these experiments was similar to the approach used for the UV post-cure experiments, except that the UV exposure occurred during the pultrusion processing. Approximately 25 inches after the composite exited from the pultrusion die, it traveled through the UV lamp and reflector. After traveling through the UV lamp and reflector, the pultruded composite continued down the pultrusion line, through the pullers and through the cut-off saw. As the processing conditions given in Table 4 show, the "UVPul-OnlineUV" experiment was intended to simulate the production of an undercured composite as it exited from the heated die; however, the addition of the UV energy exposure following the heated die was intended to increase the cure of the composite online before it was removed from the pultrusion machine. For comparison, composites were also produced under these processing conditions with the UV lamp turned off. Based on the thermocouple data for the 60 in/minute processing conditions shown in Fig. 2, it is apparent that the composite experienced additional exotherm as it traveled through the UV lamp/reflector. This additional cure is also reflected in the shear strength of the samples measured using a torsion test and in the heat of reaction data measured using differential scanning calorimetry (DSC). The average shear strength of the UVPul-OnlineUV composites with no UV exposure (4.4 ksi) was 1.9 ksi lower than the shear strength of the UVPul-OnlineUV composites that traveled through the UV light at 60 in/minute (6.3 ksi). As illustrated in Fig. 3, the heat of reaction data for these composites show similar effects. The heat of reaction for the UVPul-OnlineUV composites with no UV exposure was 9.6 J/g compared to 4.8 J/g for the UVPul-OnlineUV composites that traveled through the UV light at 60 in/minute. The data indicates that the use of a pull speed of 72 in/minute for UVPul-OnlineUV composites did not provide sufficient time for exposure to the UV light used in this experiment. For example, the shear strength for the UVPul-OnlineUV samples processed at 72 in/min was 4.9 ksi. However, to accomplish sufficient UV exposure at higher line speeds, additional lamps could be used in series to provide a longer exposure distance for the composites.

Because the exposure of the hybrid thermal/photo cure composites to the UV energy results in an additional exotherm, it is thought that this technique can be used to enhance the cure of relatively thick composites or composites containing constituent materials such as various fillers that cannot be easily penetrated by UV energy. Even if the UV energy cannot penetrate through the entire thickness of the composite, the additional exotherm triggered by the exposure of the composite to UV energy has the potential to trigger additional cure due to the thermal initiator if sufficient unreacted thermal initiator

remains after the composite exits the heated die. Additional experiments are necessary to verify this hypothesis.

Based on the results of these experiments, it was determined that the use of hybrid thermal/photo cure techniques can provide benefits for pultrusion processing. While the experiments discussed in this report demonstrated the viability of this technique, additional data is necessary to identify the optimal processing conditions, resin formulation, and UV exposure for pultruded composites. As part of this study, additional experiments have been conducted to investigate some of these parameters. For example, experiments have been conducted to investigate the effects of the use of different types of ATH fillers and the effects of the use of different spectrum bulbs with the UV lamp. Experiments using type H and type V bulbs with output spectra shown in Fig. 1 have also been conducted. Due to space constraints, discussions of these experiments are available in a separate publication [23].

Conclusions and Summary

Based on the results obtained from this study, it is apparent that the use of photocure in conjunction with thermal cure can be used with the pultrusion process. This study has been shown that a dual thermal/photo cure strategy has the potential to increase line speeds that can be achieved while producing well-cured composites. The exposure of the composites to UV light of the appropriate intensity, appropriate spectrum, and for the appropriate time is important. Also, the UV absorption characteristics of the constituent materials of the composite must be considered.

When processed using conditions that resulted in undercured composites as they exited from the heated pultrusion die, both off-line and on-line UV exposure were seen to successfully increase the degree of cure of the hybrid thermal/photo cure composites compared to that for thermal cure only composites. Thus, this study has demonstrated the feasibility of the use of a hybrid thermal/photo cure strategy with standard pultrusion tooling and equipment to cure a thicker composite at faster line speeds than could normally be achieved if thermal cure alone were used.

While the use of hybrid thermal/photo cure techniques to enhance pultrusion processing is not as easy as simply throwing some photoinitiator into a standard resin formulation and then applying a UV cure post-cure after production, this approach can be successfully applied in selected commercial applications. Because this research utilized standard pultrusion tooling and equipment with a resin formulation very similar to a typical pultrusion resin formulation, the feasibility of these techniques for commercial pultrusion applications has been demon-

strated. However, the effects of parameters such as filler type and amount, reinforcement type, and part geometry must be considered when adopting this technique for commercial applications.

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Biographies

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Table 1. Resin Formulation

Component	Description	Amount (phr)
Polyester Resin	AOC 706-101	100
Styrene	Used to dissolve powdered components	1
BAPO (Irgacure 819)	Photoinitiator for through thickness cure	0.3
AHK (Irgacure 184)	Photoinitiator for surface cure	0.9
Filler	As per Table 2	0, 10, or 20
Internal Mold Release	Zelec - UN	1

Table 2. Filler Types and Amounts Used for the Preliminary Experiments

Experiment Number	Filler Type (Description)	Filler Amount (phr)
P1	None	0
P2	Kaolin Clay (Coarse hydrous kaolin clay)	20
P3	Calcium Carbonate (Dry ground)	20
P4	Alumina Trihydrate (Bayer ATH)	20
P5	Kaolin Clay (Intermediate hydrous kaolin)	20
P6	Kaolin Clay (intermediate particle size, delaminated kaolin clay)	20
P7	Kaolin Clay (fine particle size, intermediate aspect ratio kaolin clay)	20
P8	Kaolin Clay (ultrafine particle size, low aspect ratio kaolin clay)	20
Additional experiments conducted based on the outcome of Experiments 1 - 8		
P9	Kaolin Clay (Coarse hydrous kaolin clay)	10
P10	Calcium Carbonate (Dry ground)	10
P11	Alumina Trihydrate (Bayer ATH)	10

Table 3. Characteristics of Preliminary Experiment Samples

Experiment Number	Filler Type (Amount)	Appearance	Average Barcol Hardness
P1	None	Cured through thickness	20
P2	Kaolin Clay (Coarse hydrous kaolin clay) (20 phr)	Only top surface cured, liquid on bottom	28
P3	Calcium Carbonate (Dry ground) (20 phr)	Almost cured top and bottom, bottom somewhat sticky	25
P4	Alumina Trihydrate (Bayer ATH) (20 phr)	Almost cured through thickness	32
P5	Kaolin Clay (Intermediate hydrous kaolin) (20 phr)	Top surface cured, liquid on bottom	24
P6	Kaolin Clay (intermediate particle size, delaminated kaolin clay) (20 phr)	Top surface cured, liquid on bottom	26
P7	Kaolin Clay (fine particle size, intermediate aspect ratio kaolin clay) (20 phr)	Not cured through thickness	26
P8	Kaolin Clay (ultrafine particle size, low aspect ratio kaolin clay) (20 phr)	Not cured through thickness	28
P9	Kaolin Clay (Coarse hydrous kaolin clay) (10 phr)	Top surface cured, liquid on bottom	26
P10	Calcium Carbonate (Dry ground) (10 phr)	Almost cured through thickness, slightly sticky on bottom	24
P11	Alumina Trihydrate (Bayer ATH) (10 phr)	Cured through thickness	30

Table 4. Processing Conditions for Pultrusion Experiments

Experiment Name	Line Speed (in/min)	Die Temperatures (°F)
UVPul-Baseline	36 and 48	282, 327, 318
UVPul-PostUV	96	232, 277, 268
UVPul-OnlineUV	60 and 72	232, 277, 268

Table 5. Property Data for UVPul-Baseline Composites

Sample Number	Barcol Hardness		Short-Beam Strength (ksi)	
	36 in/minute	48 in/minute	36 in/minute	48 in/minute
1	60	57	5.5	5.3
2	59	55	5.2	5.4
3	60	56	4.8	5.4
4	59	56	5.2	5.0
5	60	57	5.2	5.4
6	60	58		
7	59	57		
8	60	56		
9	60	57		
10	61	57		
Average	60	57	5.2	5.3
Standard Dev.	0.6	0.8	0.3	0.2

Table 6. Property Data for UVPul-PostUV Composites

Sample Number	Barcol Hardness		Short-Beam Strength (ksi)	
	96 in/min – No UV	96 in/min – UV Post	96 in/min – No UV	96 in/min – UV Post
1	49	58	4.6	5.3
2	50	59	4.6	5.3
3	49	58	4.5	4.8
4	50	60	4.8	5.1
5	49	59	4.6	5.1
6	48	59		
7	49	60		
8	50	58		
9	50	60		
10	48	58		
Average	49	59	4.6	5.1
Standard Dev.	0.8	0.9	0.1	0.2

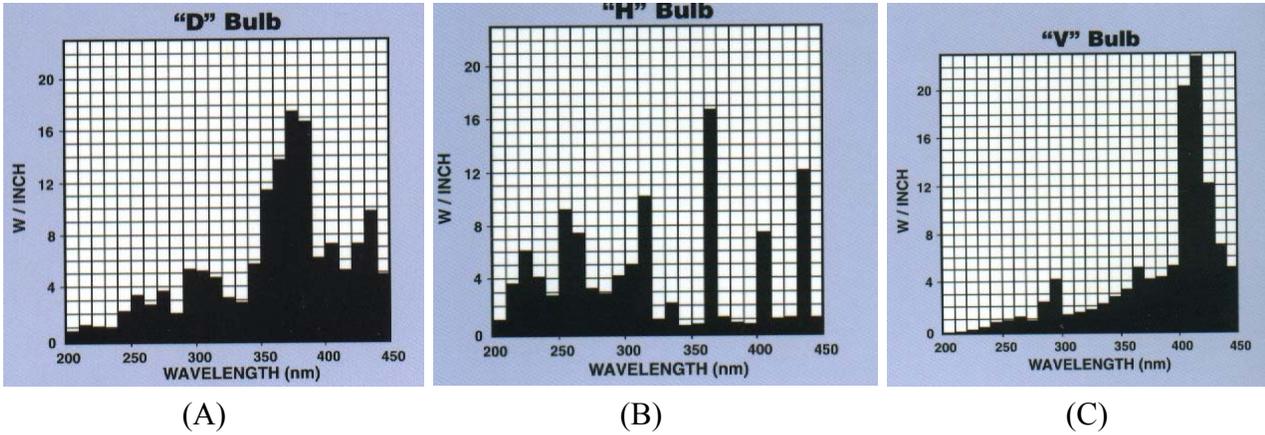


Figure 1. Output spectra of electrodeless bulbs used with the Fusion UV F300S lamp. (A) D type bulb, (B) H type bulb, and (C) V type bulb. (Data from UV Fusion F300S sales brochure.)

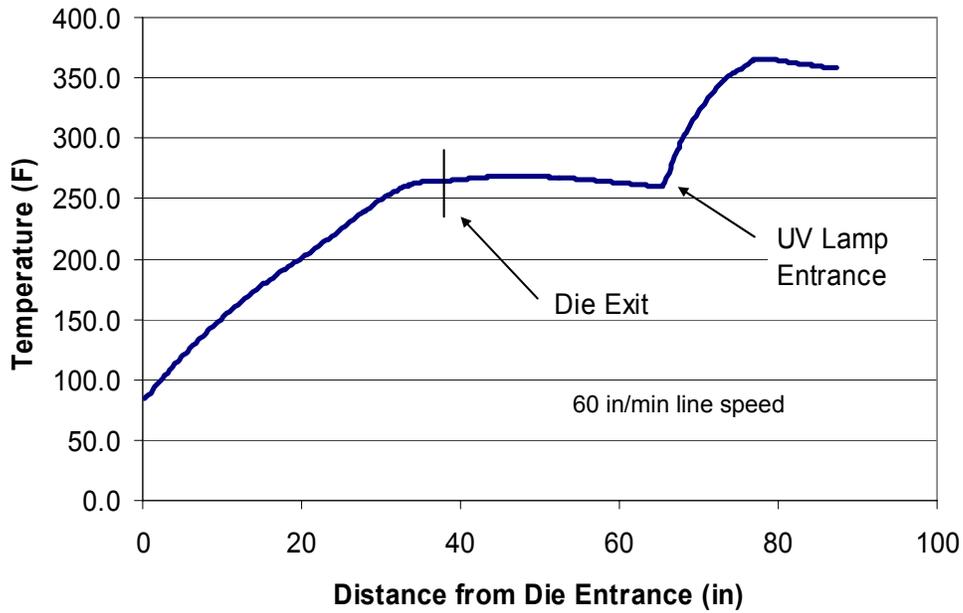


Figure 2. Thermocouple data from the UVPul-OnlineUV experiment showing exotherm from hybrid thermal/photo cure pultrusion using online photocure exposure after the heated pultrusion.

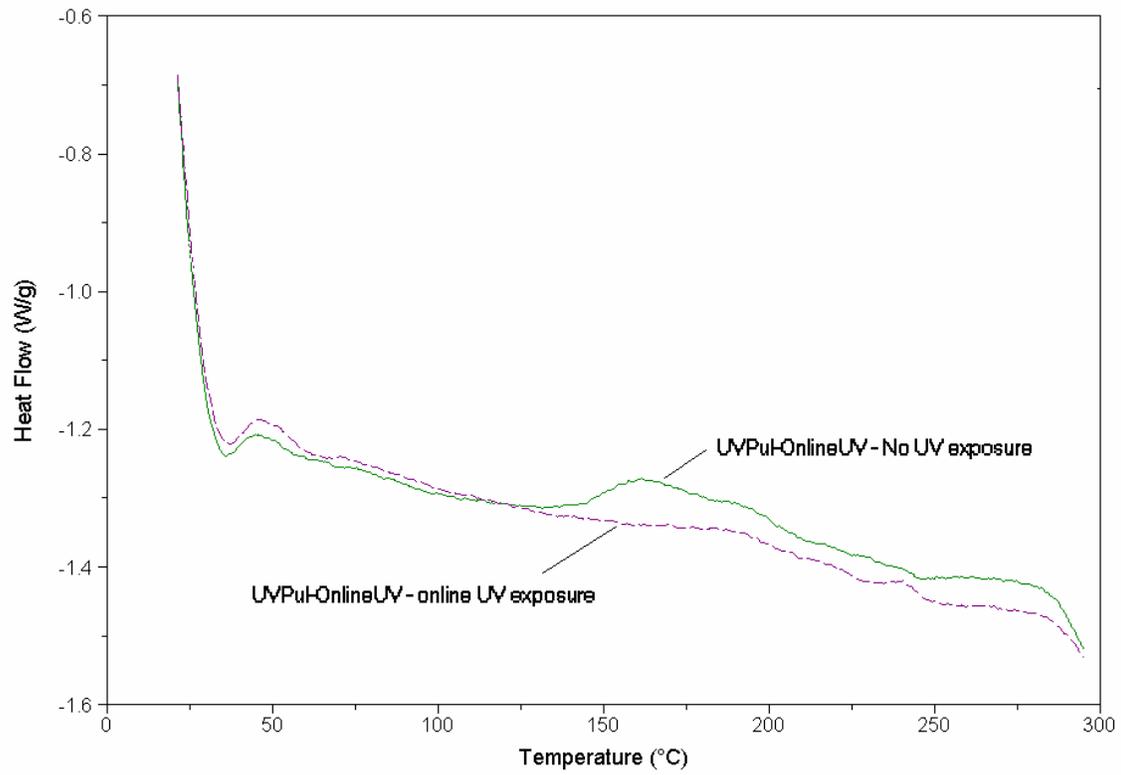


Figure 3. DSC thermograph illustrating differences in heat of reaction data for UVPul-OnlineUV samples with and without online exposure to UV energy.