Epalloy™ 7200 for use as an Accelerator in Elevated Temperature Cure Epoxy Systems

Epalloy 7200 has been shown to provide enhanced cure of resin formulations designed for ambient and sub-ambient temperature cure. Unmodified aromatic and cycloaliphatic amines designed for elevated temperature cure were tested to see if Epalloy 7200 would accelerate cure. The amines used in this phase of the study were;

- Ancamine 9360 – Methylene Dianiline based hardener, from Air Products
- Ethacure 100 – Diethyltoluenediamine from Albermarle Corporation
- Amicure PACM -- bis-(p-aminocyclohexyl) methane, from Air Products
- Vestamin IPD – Isophorone Diamine from Hüls America

Time to cure studies where performed using a Perkin-Elmer DSC7 Thermal Analyzer to compare Epalloy 7200 and LER with each of the above-mentioned curing agents. Kinetics determinations were made using the Scanning Kinetics option in the Pyris 6 software package from Perkin-Elmer. Scanning Kinetics computes results from a single cure scan.

To generate Scanning Kinetics data, samples were mixed at ratios shown in Table 1 below.

TABLE 1 – Weight Ratios for Epalloy 7200 and LER with Aromatic and Cycloaliphatic Amines for Kinetics Determinations

	A	B	C	D	E	F	G	H
7200	100	0	100	0	100	0	100	0
LER	0	100	0	100	0	100	0	100
Ancamine 9360	36.5	38.4	-	-	-	-	-	
Ethacure 100	-	-	22.4	23.6	-	-	-	
Amicure PACM	-	-	-	-	26.4	27.8	-	
Vestimin IPD	-	-	-	-	-	-	21.4	22.5

Small aliquots of mixed material were added to stainless steel sample pans and crimped closed. Cure scans were run from 50 to 230°C at 10°C/min. Scans were analyzed to generate onset temperatures, peak exotherm temperature and Delta H. From the cure scans, the scanning kinetics software can compute a variety of data including % reaction vs. time for various isothermal cure temperatures. From this data, we can make comparisons of times to cure at various temperatures. Figure 1, below, shows the comparison between Epalloy 7200 and LER for time to cure to 90% full cure at temperatures from 110 to 190°C.

Figure 1

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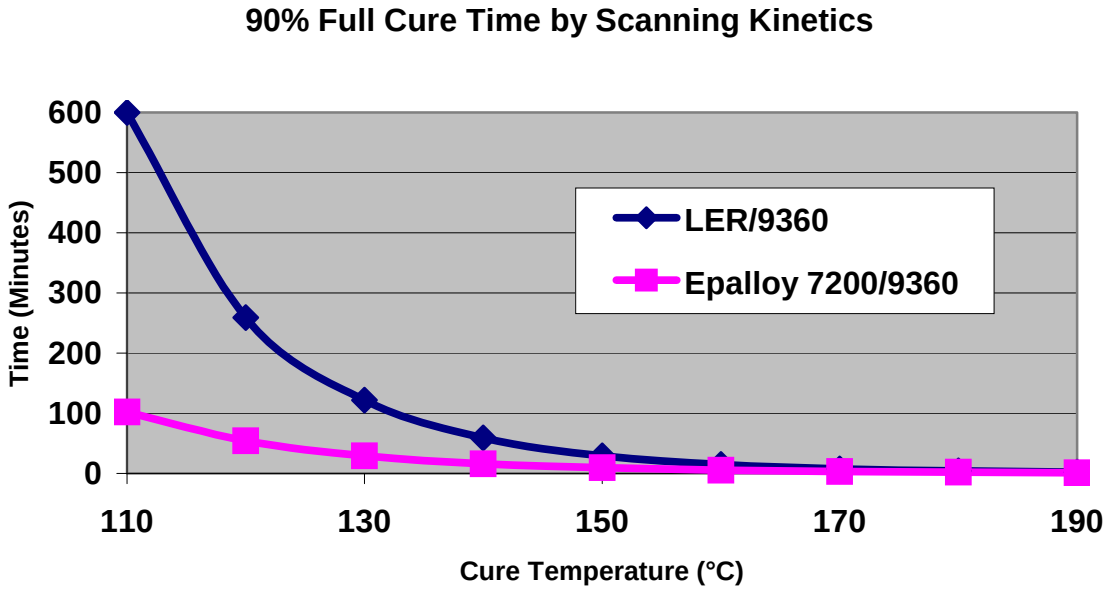

Figure 1 shows a clear advantage in cure time with the Epalloy 7200 at lower temperatures, diminishing in effect as the temperature increases. However, as shown in Table 2 below, even at 150°C the time advantage is still over 3-fold with Epalloy 7200.

Table 2 – Time to 90% Cure v. Temperature – Epalloy 7200 v. LER w/ Ancamine 9360 by Scanning Kinetics (minutes)

	110°C	120°C	130°C	140°C	150°C	160°C	170°C	180°C	190°C
LER	600	259	122	59	29	15	7.6	4.1	2.3
7200	102	54	29	16	9.4	5.3	3.3	2	1.3
LER/7200	5.88	4.80	4.21	3.69	3.09	2.83	2.30	2.05	1.77

Knowing that significant differences exist in the rate of cure between the two resins, can we use the data generated by scanning kinetics to test for faster physical property development when using the Epalloy 7200/9360 system as compared to the LER/9360 system? Shown below are Scanning Kinetics curves for LER/9360 (Figure 2) and Epalloy 7200/9360 (Figure 3). To see if Epalloy 7200 will develop equivalent Tg to LER using a lower temperature, or shorter cure schedule, we note that in Figure 2 it appears LER/9360 needs to cure for approximately 28 minutes at 150°C to achieve 90% full cure. Figure 3 shows that Epalloy 7200/9360, requires 28 minutes at 130°C for 90% full cure.

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Figure 2 - % Reaction v. Time at Isothermal Temperatures for LER cured with Ancamine 9360 by Scanning Kinetics

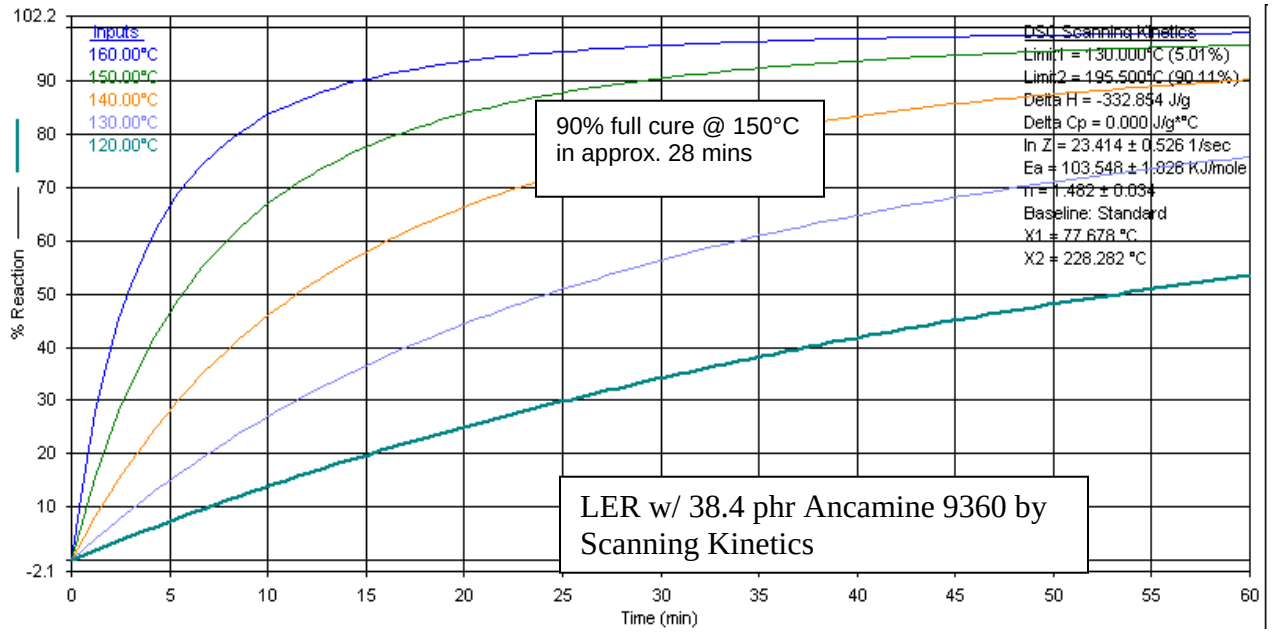
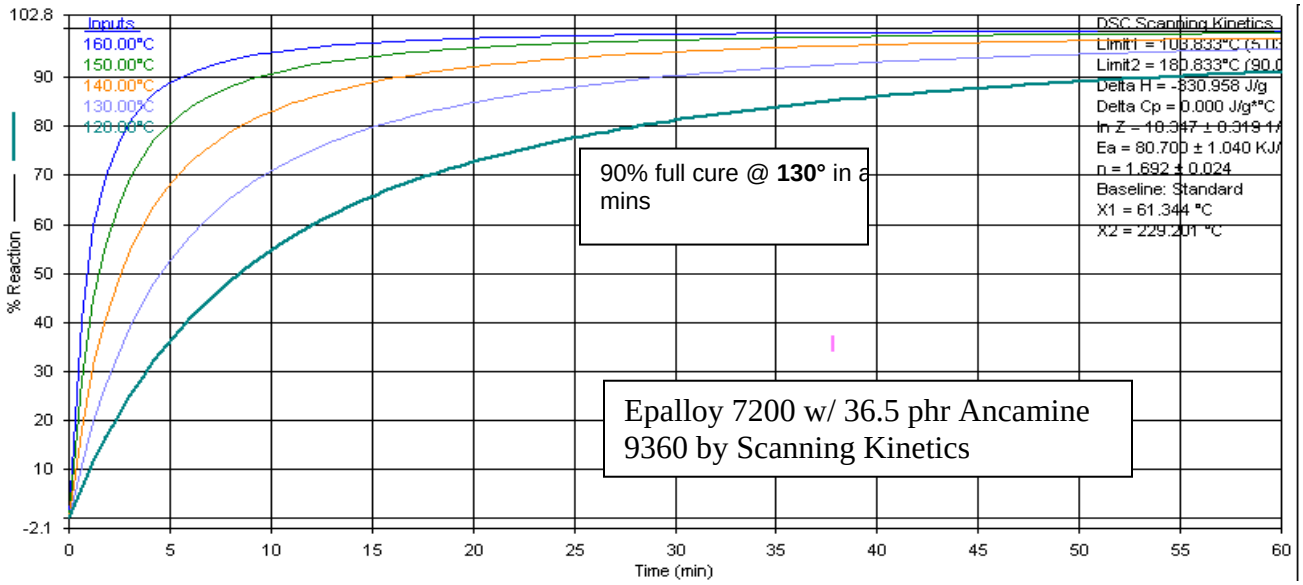


Figure 3 - % Reaction v. Time at Isothermal Temperatures for Epalloy 7200 cured with Ancamine 9360 by Scanning Kinetics



In order to test the theory that Epalloy 7200 can provide equivalent Tg with a lower temperature cure, or shorter cure cycle at the same temperature, samples of each system were cured under the conditions shown in Table 3, and then checked for Tg.

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Table 3 – Comparison of Tg with Epalloy 7200 v. LER cured w/Ancamine 9360 under Different Time/Temperature Conditions

Cure Schedule	LER/9360 - Tg	7200/9360 - Tg
25 mins @ 150°C	120°C	-
10 mins @ 150°C		114°C
25 mins @ 130°C	-	119°C
25 mins @ 120°C	71°C	109°C
30 mins @ 120°C	-	118°

The data in Table 3 shows that the 7200 formula can cure to a similar Tg as the control using cure schedules for similar times as the control, but at temperatures 20 to 30 degrees lower than the control. However, curing at the same temperature for a shorter period of time (10 minutes at 150°C), does not quite give an equivalent result.

In the same manner to that described above, work was done to show similar effects when using Ethacure 100, Isophorone Diamine, and PACM as the curing agent.

Figure 4 shows the time to 90% full cure curve as developed from scanning kinetics data for LER vs. Epalloy 7200 cured with Ethacure 100. Table 4 shows results comparing Tg with varying cure schedules, which indicates the 7200 system can achieve the same Tg as the LER system by curing for the same time but lower temperature than the control (130°C for 74') or shorter cure time at the same temperature as the control (150°C for 41 minutes).

Figure 4

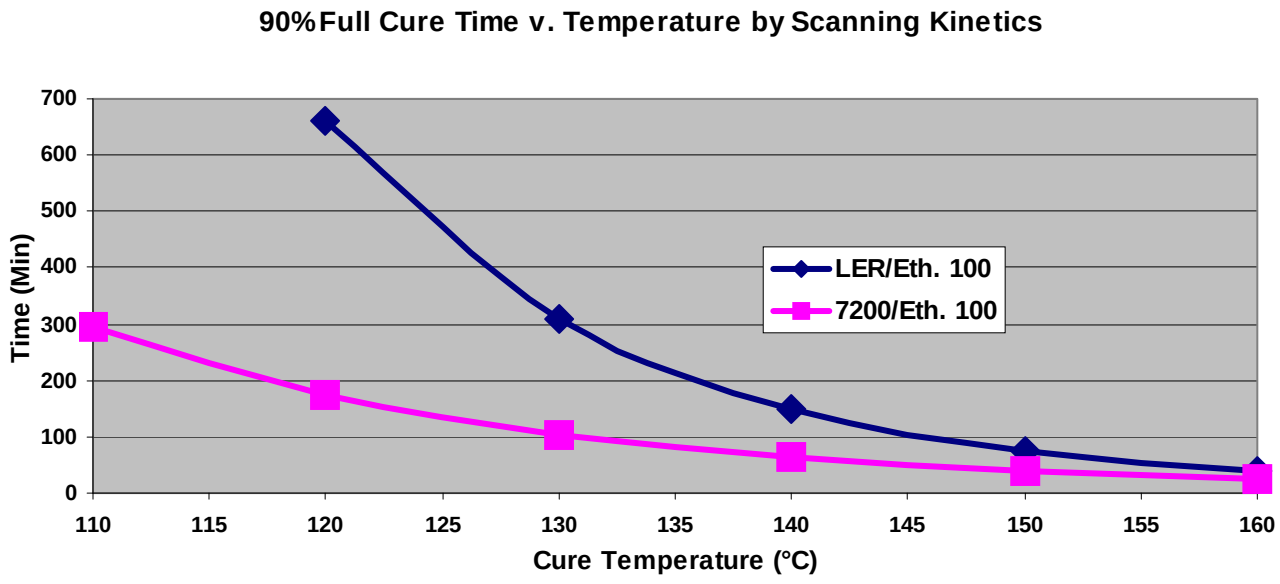

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Table 4 – Comparison of Tg with Epalloy 7200 v. LER cured w/Ethacure 100 under Different Time/Temperature Conditions

	LER	7200
150°C/74'	127°C	
150°C/41'	98°C	130°C
130°C/74'		129°C

Figure 5 shows the time to 90% full cure curve as developed from scanning kinetics for LER vs. Epalloy 7200 cured with IPD (Isophorone Diamine). Table 5 shows data comparing Tg with varying cure schedules, which indicates the 7200/IPD system can achieve the same Tg as the LER system with shorter cure cycles, but not at significantly lower temperatures.

Figure 5

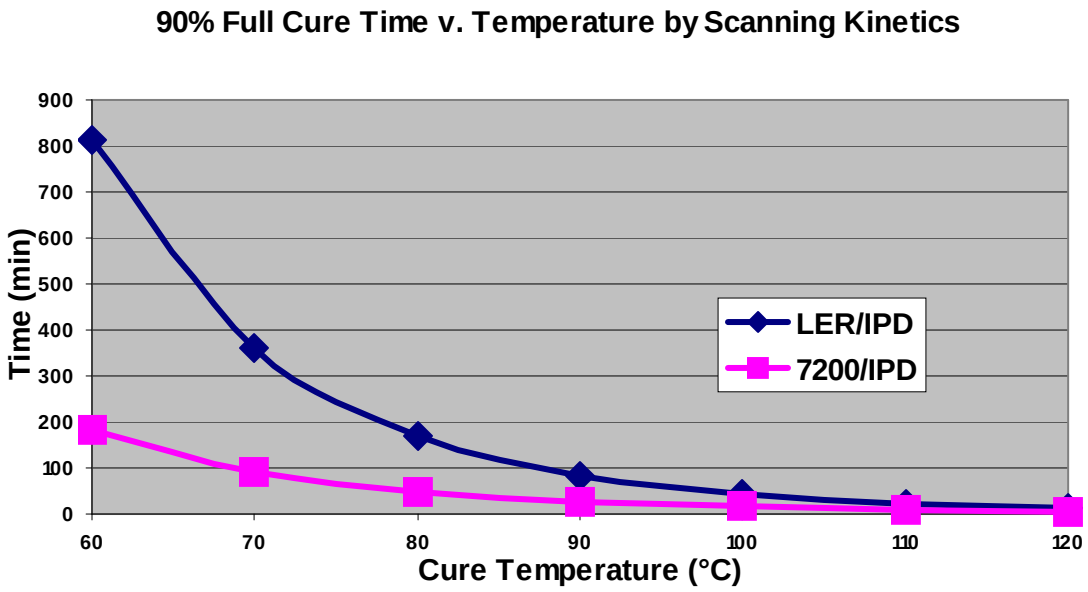

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Table 5 – Comparison of Tg with Epalloy 7200 v. LER cured w/Isophorone Diamine under Different Time/Temperature Conditions

	LER	7200
90°C/85'	112°C	
90°C/30'		107°C
90°C/40'		111°C
65°C/90'		86°C

These data clearly indicate Epalloy 7200 can be used to achieve advantageous cure cycles with curing agents designed for elevated temperature cure such as Methylene Dianiline, Ethacure 100, Amicure PACM, and Isophorone Diamine. These advantages can result in savings in time and energy to cure products utilizing these curing agents.

Figure 11 shows the time to 90% full cure curve as developed from scanning kinetics for LER vs. Epalloy 7200 cured with PACM. Table 16 shows data comparing Tg with varying cure schedules, which indicates the 7200/PACM system can achieve a similar Tg to the LER system with lower temperature cure cycles.

Figure 11

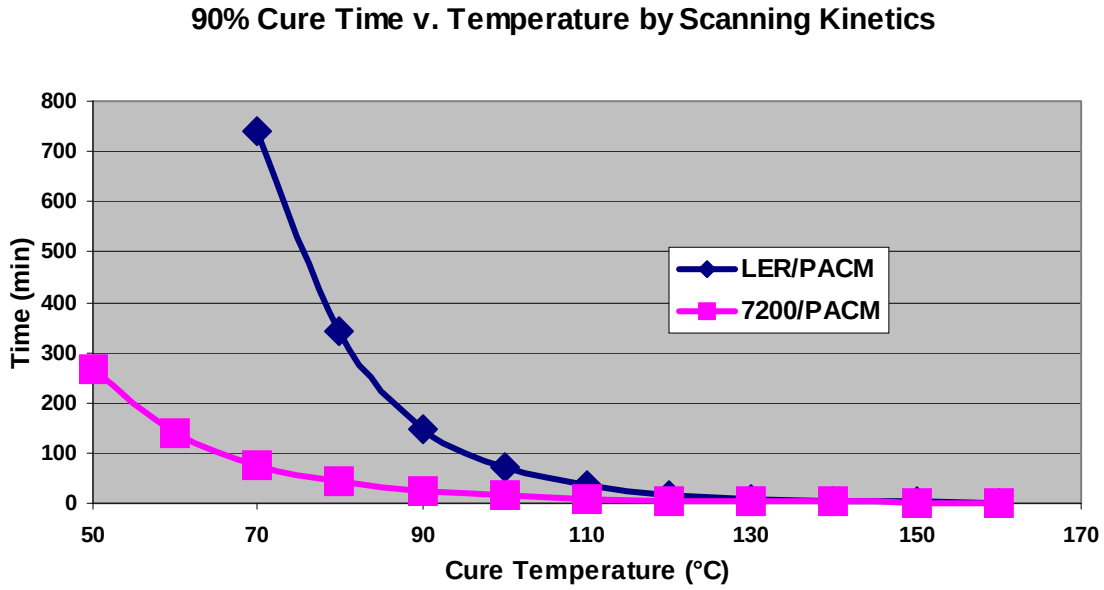

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Table 16 – Comparison of Tg with Epalloy 7200 v. LER cured w/PACM under Different Time/Temperature Conditions

	LER	7200
110°C/35’	112°C	
110°C/18’		107°C
90°C/35’		111°C
85°C/35’		86°C